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LUBRICATING GREASES: Their Manufacture and Use

BY

E. N. KLEMGARD

CONSULTING LUBRICATION ENGINEER

A book of practical and scientific data on the manufacture and use of lubricating greases, for grease makers, refinery executives, research chemists, lubricating engineers, salesmen, mill and power plant superintendents, and others interested in the efficient manufacture and utilization of lubricating greases. Includes the chemical analyses of many commercial lubricants, abstracts of important patents, formulae and practical processes for the manufacture of greases.

BOOK DEPARTMENT

REINHOLD PUBLISHING CORPORATION

330 WEST FORTY-SECOND STREET, NEW YORK, U. S. A.

1937

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THIS BOOK IS DEDICATED TO

J S K
and
R L K

WHOSE COURAGEOUS EXAMPLES AND INTENSE INTEREST IN TRUTH AND THE ACQUIREMENT OF KNOWLEDGE HAVE BEEN INSPIRATIONAL TO THE AUTHOR

Preface

Lubricating Greases: Their Manufacture and Use, while based on an earlier work, has been so greatly enlarged and completely revised that it hardly resembles the small book published under the name "Lubricating Greases" in 1927. The present volume of nearly 900 pages is more than five times the size of the earlier book. That the new work is warranted is substantiated by the facts that the earlier book was soon exhausted and that many new and important advances in the manufacture of lubricating greases have been made. These have served as the incentive to record many of the researches which have been carried out during the past decade, particularly those having to do with the new automotive lubricating greases, wheel bearing greases, steering gear lubricants, water pump greases, hypoid gear greases, and extreme pressure gear lubricants, as well as the most important advances in industrial lubricants, especially those made with extreme pressure ingredients. The patent reviews have been completed and arranged so that they may be readily consulted. From the data given, an inventor with comparatively little effort in searching may determine what is novel in connection with new lubricating grease developments.

To those not familiar with the intense activity in the development of lubricating greases since 1927, it may come as a surprise that a volume of such pretensions may now appear, but it is considered that many technologists now working in this field will appreciate the efforts made in compiling this volume and will welcome a complete reference book on lubricating greases as a necessary addition to their technical libraries. To quote from the preface to the previous book, "Lubricating Greases," published in 1927: "The art of making grease, first developed by Raecz more than sixty-five years ago, has made rapid strides in the last few years.

"The first greases of which there is knowledge were simple mixtures of lime soaps and oil, used for greasing axles on carts and wagons. Crude petroleum and rosin oils were mixed by hand in a kettle heated over an open coal fire. When the temperature was thought to be about right, a milk of lime solution was added and the batch stirred until it began to thicken. It was then poured into containers and allowed to cool, setting to a consistent grease. Frequently this type of grease was adulterated by additions of terra alba, talc, chalk, graphite or an excess of water.

"Today, axle greases are manufactured from refined pale and black oils, carefully chosen fats, and high quality hydrated lime. The old hand method of mixing has given way to continuous mechanical mixers, steam-jacketed or otherwise provided with a method of heating which is closely controlled. With the growth of the petroleum industry considerable investigation has been undertaken, with the objects of standardizing the processes already known and developing more satisfactory products. In addi-

tion to sett greases (as axle greases are now generally termed) there have been developed many special greases, varying from the original in both composition and application, and designed to meet particular requirements of color, gloss, texture, stability and consistency, at widely varying working temperatures.

"The processes used by various manufacturers for the production of these special greases have as a rule been carefully guarded, being considered as trade secrets. Little of value can be found in the literature on the subject. The material found in the technical journals is incomplete and often misleading. Many of the books on lubrication devote a few pages to grease, the information being out of date and not applying to present practice. The present volume is believed to be the only book now in print which deals solely with grease and its uses. In this work will be found the results of many years of research with the largest manufacturers of lubricants in this country, much of which has never before been printed. While the book does not pretend to be exhaustive, it does contain a fairly comprehensive exposition of the theory, manufacture and application of lubricating greases."

The present book has been prepared with a view to meeting the needs of grease research chemists and lubrication technologists throughout the world. Although the earlier book was based on a relatively limited experience with American lubricating greases, many copies were distributed in foreign countries. In this volume, as complete a discussion as possible of the lubricating greases marketed in Europe and Asia has been included. Entire chapters on the chemical and physical characteristics, specifications of grease making materials, and the design and management of grease plants have been included in the new work. The fundamental theories underlying the manufacture of greases and the dispersion of soaps in oil have been developed in so far as has been possible and many factors not discussed in prior literature have been presented.

The authors of most technical reference books do not attempt to discuss costs, profits, and the economic significance of the chemical products they deal with. It seems logical, however, that if it is true that the first and most important fact about grease is the requirement that it must lubricate and reduce wear of machine parts, then the second most important factual item is that it must be produced and sold at a profit by its manufacturers. In response to the need for information concerning this latter item many cost calculations, methods of computing material and compounding costs, and discussions of grease plant economics have been included.

ACKNOWLEDGMENTS

Appreciation is expressed for the permission of the Controller of His Britannic Majesty's Stationery Office to reproduce extracts from the British Abridgments of Patent Specifications, Group III. I am also grateful to Mr. F. T. Brearley of the United States Patent Office for permission to reproduce extracts from patents and items from the Official Gazette. Much of the data dealing with grease kettles and autoclaves has been obtain-

able through the courtesy of the Sowers Manufacturing Company, Buffalo, N. Y., and is the result of the efforts of Mr. F. E. Huggins, Jr. Grateful acknowledgments are made to Mr. M. B. Chittick of The Pure Oil Company for photographs of modern grease compounding equipment.

Many collaborators have cooperated in the preparation of the manuscript for the new book, and some of the thoughts expressed in this volume have been suggestions originally made by former associates for which I am duly thankful. Professor E. C. Colpitts, of the Mathematics Department of Washington State College, assisted in the development of some of the mathematical relationships. I wish particularly to express my appreciation to R. C. Bennison, Elsie Butts, Ellen K. Adams, and other members of my laboratory staff who have been of material assistance in the preparation of the manuscript.

E. N. KLEMGARD

Pullman, Washington
October 1, 1937.

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Introduction

A conservative estimate places the average total annual production of all forms of lubricating greases in the United States at 250,000,000 pounds.[1a] Appreciably more than one-half of this amount is made up of greases used for the lubrication of automotive equipment; the balance is consumed in the bearings of a wide variety of industrial machinery. Data collected by the National Association of Grease Manufacturers [1] in 1934, from forty-six lubricant manufacturers, indicated that the total annual sales were only about 130,000,000 pounds and that this represented a value of $7,329,378. The average cost including labor, materials, and packages was estimated

Production of Lubricating Greases and Petrolatum: 1925 to 1933 *

Census year	Number of establishments	Gallons	Value
		Lubricating greases other than axle grease	
1925	41	26,301,450	$ 8,262,744
1927	46	26,524,534	9,051,121
1929	53	34,605,242	12,729,391
1931	43	27,283,804	9,640,707
1933	(a)	(b) 27,212,000	(b) 7,396,000
		Axle grease	
1925	22	5,656,686	$2,098,676
1927	21	3,052,889	935,612
1929	23	3,748,383	1,368,936
1931	19	1,573,899	512,720
1933	(a)	(c)	(c)
		Petrolatum, mineral jelly, etc.	
1925	30	13,862,225	$3,559,207
1927	37	15,334,428	2,974,472
1929	28	13,663,503	2,547,531
1931	33	16,958,628	2,222,226
1933	26	(d) 14,711,000	(d) 1,855,000

(a) Not tabulated

(b) Includes data for axle grease.

(c) Included in lubricating greases.

(d) Incomplete, but probably within 5 per cent of true total. Derived from returns on standard schedule. (Small establishments were canvassed by means of an abridged schedule.)

* Data from: "Biennial Census of Manufacturers," U. S. Department of Commerce, Aug. 19, 1936.

at 2.919c per pound. The average selling price was 5.735c per pound. The total capital invested in grease manufacturing equipment in this country was greater than 5,000,000 dollars.

The trend towards special greases for the lubrication of automobile

[1a] The data compiled by the U. S. Bureau of the Census are probably the most authentic compilations available at this time. These data are given in the table entitled "Production of Lubricating Greases and Petrolatum: 1925 to 1933."

[1] Bulletin No. 2, National Association of Lubricating Grease Manufacturers, issued Dec. 12, 1934, by D. S. Hunter & Associates.

chassis bearings has greatly increased the production of such specific lubricants. It is evident, however, that many service stations do not yet market the best types of greases for automotive shackles, wheel bearings, steering gears, universal joints and water pumps.[2]

Investigators have shown [3] that by improved design and standardization of automotive bearings many of the special greases could be dispensed with. The problem is beset with many technical and commercial complications and automotive engineers find it difficult to select the most desirable lubricating greases for their units. They are, therefore, tending to change their designs, in many instances, so that oils and greases that are much less special in their nature will be more generally used. In any case, it is believed that the lubrication engineers and chemists employed by the grease manufacturers will continue to keep pace with the designers of machinery and that, as in the past, each year it will be necessary to develop special lubricating greases for special mechanisms. For this reason, many special and seemingly unimportant greases are discussed in the following pages, which may serve as the starting point for the development of lubricants for specific new purposes.

The secrecy and mystery which surrounded the activities of the early grease makers and the limited number of technical articles which they published limits the possibilities for compiling an extensive historical sketch of the industry. It may be said that during the past sixty years many of the "rule of thumb" and "trial and error" grease makers were able to turn out remarkably satisfactory products; at least, they met the requirements of their day.

One of the first intimations [4] we have of lubrication practice dates back to 1650 B.C. On the inner wall of an Egyptian tomb of Tehuti-Hetep is a decoration by an artist which shows the methods of lubrication employed in moving large stones, statues, and building materials. It is claimed that there is absolute evidence that the movement of stone from the quarries was aided by the application of olive oil poured on wooden planks. The first evidence we have of bearing lubrication is that of applying lubricants to the axles of the chariots used by the Egyptians. It has been reported by a chemist of the Cairo Museum, that deposits taken from the axle of a chariot used during the time of Yuss, about 1400 B.C., indicated the presence of quartz, iron, and a sufficient quantity of fat and lime to prove the use of lime soap at that time.

According to Lewkowitsch,[5] Raecz first proposed the production of lubricating greases by dissolving various lime soaps in mineral oil, about the year 1845. Maass [6] considers that the first simple greases, consisting of the soaps of potassium, sodium and calcium mixed with lubricating oils, were produced about 1875; however, there is considerable evidence to indicate

[2] Matthews, R. R., *Proc. Am. Petroleum Inst.*, **III**, 60 (Nov. 11, 1935).

[3] Blackwood, A. J., and Spencer, Jr., C. C., *S. A. E. Journal*, (34), **2**, 52-8 (1934).

[4] Lange, A. R., "Grease Making—Past, Present and Future," 3rd Annual Convention of the Lubricating Grease Manufacturers Association, Chicago (October, 1935); quoting from publications of William F. Parish.

[5] Lewkowitsch, "Chemical Technology and Analysis of Oils, Fats and Waxes," 5th Ed., **III**, 65.

[6] Maass, W., "Ole, Fette, Wachse, **4**, 19-22 (1936).

that the art was established long before this. On April 3, 1773, John Liardet [7] invented a combination of white or red lead with "drying oil" intended as "a grease for friction preserving steel, iron and various other uses."

He thus carried out the pioneer work for the later establishment of the lead base lubricants which are even yet preferred, in some instances, as for railroad journal box lubrication. The lead soaps of rape oil,[5] obtained by saponification with lead oxide and containing appreciable quantities of emulsified water, were extensively used in Germany as railway wagon greases from 1850 to 1860. In 1835, Henry Booth [8] proposed a grease "for the axle bearings of carriages and the axle spindles and bearing parts of machinery in general." It consisted of "soda or other alkaline substance with oil, tallow, or other grease and water—in such portions that it shall not be of—a corrosive nature when applied to iron." It is probable that the beginning of our present art of cup grease manufacture can be found in the Partridge patent of 1835 [9] which refers to a "composition paste—as an antifriction, applicable to the bearings of wheels and machinery." It was made by bringing together lime water and olive oil, whale oil or tallow. Holcombe, in 1841, suggested the use of naphthalene [10] as the basis of lubricating greases and curiously enough, in 1935, nearly a hundred years later, manufacturers have offered naphthalene modified by chlorination, to American grease makers as an extreme pressure base for their lubricants. The grease proposed by Denne [11] in 1847, was prepared by adding a solution of lime to the soda soaps of tallow and vegetable oils to obtain a precipitate of lime soaps, which were mixed with olive oil.

It is of note that none of these early grease patents, obtained between 1773 and about 1850, make use of mineral oil as during this period it was not possible to obtain commercially the petroleum derived oils. It will be recalled that the Drake well in Pennsylvania was not drilled until 1859, although oils from Burmese crudes were available in 1852. It is the author's belief that the first printed reference to the use of greases made with soda soaps and petroleum products is found in William Little's patent of 1849.[12] This inventor first distilled crude petroleum for the production of lamp oil, and the greasy oils next obtained were mixed with the soda soap of tallow and boiled together to make a grease "suitable for lubricating machinery." In 1853, Little [13] obtained a similar patent in which heavy oil from the distillation of coal was utilized in place of the petroleum product. The manufacture of a consistent lubricating grease from a sort of petrolatum ("greenish-yellow grease separated from heavy oil in the manufacture of lubricating matters") was described by Brockelbank [14] in which soda soap was boiled with this lubricant. The term "paraffine oil" was

[7] British Patent 1,040 (1773).
[8] British Patent 6,814 (April 14, 1835).
[9] British Patent 6,945 (December 7, 1835).
[10] British Patent 8,949 (May 6, 1841).
[11] British Patent 11,674 (April 27, 1847).
[12] British Patent 12,571 (April 16, 1849).
[13] British Patent 1,678 (July 14, 1853).
[14] British Patent 1,250 (June 5, 1854).

first applied to lubricating distillates from crude oil by William Kilgour in 1855, at which time he was experimenting with "earth oil from the Burman Empire." At this time in England, however, lubricating oils from coal tar were much cheaper than the petroleum products.

The first mention in English literature of lime base-mineral oil greases may be found in the British patent application of Richard A. Brooman,[15] in 1857, several years following the original suggestion of Raecz. From 1860 to date the patent literature is replete with references to lubricating compositions consisting of various soaps, fillers and lubricating oils. The most important of these patents will be mentioned in the following chapters as they may prove of value to research chemists as starting points for the development of new lubricants.

It is quite evident that the art of grease making has secured the attention of investigators for nearly one-hundred years. The fact that during this time grease manufacturing processes have not been fully understood and the art reduced to a science at an earlier date, may be attributed to the complexity of the materials used in their manufacture, the inclination of the grease makers to guard their secrets, and the reluctance of institutions and petroleum refiners to spend large sums for fundamental researches on products not having such enormous production and value as many other commodities; for instance, gasoline, fuel oils, and lubricating oils. The chemistry of petroleum is making rapid advances and with the vast amount of research being done along colloidal chemistry lines and synthesis, grease making processes are well on the way to becoming scientifically controlled procedures, rather than trial and error methods.

A new era is being approached. One in which lubrication problems involve higher speeds, higher bearing pressures, new metallurgical compositions in bearings, and closer bearing clearances. These conditions apply equally to both the automotive and industrial branches of lubrication engineering. In this connection, the following chapters should be useful to those interested in meeting these new requirements. The progressive grease manufacturer realizes that grease intended to most satisfactorily meet the new conditions must have greater stability to oxidation than many of the products of the past. Separation of the soap base, objectionable gumming and discoloration of the grease due to oxidation and evaporation of moisture, are items which may be readily overcome by research. Greases which are resistant to water, have a low yield point and at the same time a high melting point are in demand. Film strength and oiliness are also receiving considerable attention by investigators. The chief reason for utilizing a grease is to prevent wear, and at last many grease manufacturers are beginning to make tests to prove the wear resisting qualities of their lubricants.

[15] British Patent 181 (January 21, 1857).

LUBRICATING GREASES

Chapter I

The Colloidal Nature of Lubricating Greases

Mineral oils, fats and fatty oils saponified by various alkalis on first thought may constitute such a complex variety of combinations and properties that it would appear impossible to describe and classify them in a direct and simple manner. In general, it may be stated that lubricating greases consist of two phases, both exceedingly complex in nature, and both exhibiting definite colloidal properties not only when associated with each other, but when existing by themselves. Ordinary chemical analysis while indicating the components of a grease, lacks a great deal as an explanation of grease properties. Knowing that a grease was composed of sodium-tallow soap and a certain grade of mineral lubricating oil it might appear to be a simple matter to bring these materials together to produce a lubricating grease. One chemist not fully informed on grease making technic violently stirred the granular soap and oil together for many hours at room temperature, and when no plastic grease was formed, correctly concluded the soap was essentially insoluble in the oil. Heating the mixture to temperatures of 300° F., or higher, would have caused the soap to dissolve and on cooling, a grease of some sort would have been easily obtained. The application of heat brings about disintegration of the large soap aggregates and, if carried far enough, a homogeneous solution is attained which, on cooling, becomes a gel or smectic paste, due to the dispersion of the soap and the reassembly of the soap particles in a colloid condition, frequently termed the micelle structure. Others term the structure a space lattice.

The rule which holds true for all gels is that they must be built up from two components, a liquid at the temperature under consideration and a solid known as a gelator. The mineral oil is, of course, the liquid phase and the soap is the solid gelator. In general, gels are produced from super-saturated solutions,[1] and commercial grease manufacturers seldom depart from this fundamental rule. Greases are ordinarily classified as heat-reversible elastic gels, *i.e.*, the temperature solubility curve for the soap in

[1] Waterproof greases, axle greases, and railroad greases produced by the cold sett process, in which abietic or fatty acids are brought together in the cold in the presence of mineral oil with a solution of the desired alkali, may be exceptions in that as the super-saturated solution is approached gel formation proceeds rapidly. In some cases the grease is formed in less than fifteen seconds so that the period of super-saturation is not so well defined as with the boiled lime base cup greases. See Lloyd, D. J., "The Problem of Gel Structure," "Colloid Chemistry," by Alexander, J., I, 768, The Chemical Catalog Co., Inc. (Reinhold Publishing Corp.), New York (1926). Gels may also be formed by changes in the following properties of a colloidal system: a) Formation of an insoluble precipitate, b) the extent of hydration, and c) the concentration of the gelator. See Ostwald, "Practical Colloid Chemistry" (1924).

the oil is a steep one. At low temperatures the soap is relatively insoluble. Some investigators view greases as colloidal suspensions or emulsions. Neither of these theories is entirely correct as will be shown later. In substantiation of the emulsion classification, is the fact that most greases contain two liquids, oil and water, and frequently glycerol. From this standpoint, the soap is considered merely as an emulgator of the two liquid phases. The emulsoid theory is not universally applicable, however, as it is quite possible to prepare dehydrated greases and greases in which the only liquid phase is mineral oil. The theories of Bancroft and Langmuir, relative to oil and water emulsions are, however, readily applied to some greases. A more plausible theory of grease structure which will be discussed in greater detail later is that the soap exists in the form of liquid crystals dispersed in the oil, any emulsified water being considered as extraneous.

Liquid crystals, in general, may be formed from swarms of molecules, in each swarm of which groups of molecules are arranged in a more or less parallel condition. Such crystals give rise to colloidal properties and differ from true crystals in that it is quite likely that the ends of the molecules fall in parallel surfaces and the axes of the molecules are quite heterogeneous with regard to direction. If this were not the case and the atoms of the molecules caused them to become arranged in an orderly geometric form on a definite lattice we would have normal solid crystals.

The consistency characteristics of greases may vary from those of an oil of low viscosity to those of a dense plastic solid, depending on the quantity of calcium soap present in the mixture. The external phase of these greases is the mineral lubricating oil, the water being the inner or dispersed phase. It is known that mixtures of mineral oil and calcium salts of such higher fatty acids as oleic and stearic may be heated and the soaps will be dissolved in the oil; on cooling a gel will be obtained. This mixture, being anhydrous or containing only a small percentage of water, is unsatisfactory for lubrication purposes because of its tendency toward "sweating" oil (syneresis) and its instability to mechanical agitation, particularly if there is an excess of lime present. On stirring such a gel the soap is precipitated and the gel characteristics destroyed. If water is introduced into the mixture at a temperature of 190° F. a stable product may be obtained. It appears probable that since the lime soap is more soluble in the mineral oil than the water an emulsion of the water-in-oil type is formed.

Bancroft [2] connects the type of emulsion with the phase which is the best solvent for the emulsifying agent as follows:

"If the absorption of the emulsifying agent lowers the surface tension on the water side of the interface more than it does on the oil side, the interface will tend to curve so as to be convex on the water side, and we shall have a tendency to emulsify oil in water.

"If the absorption of the emulsifying agent lowers the surface tension on the oil side of the interface more than it does on the water side, the

[2] Bancroft, W. D., "Applied Colloid Chemistry," McGraw-Hill Book Company, New York (1922).

interface will tend to curve so as to be concave on the water side and we shall have a tendency to emulsify water in oil. The simplest way then to emulsify oil in water is to add a water soluble colloid which is absorbed strongly at the interface, and the simplest way to emulsify water in oil is to add an oil-soluble colloid which is absorbed strongly in the interface."

Calcium oleate and calcium stearate are, from a practical consideration, insoluble in water but have a slight solubility in mineral lubricating oils. Cup greases, then, conform to the general rule that the external phase is the one which is the best solvent for the emulsifier, that is, the calcium soaps being more soluble in oil than water produce a water-in-oil emulsion.

Bhatnagar [3] has arrived at the following theory of emulsification: "All emulsifying agents having an excess of negative ions absorbed on them and wetted by water will yield oil-in-water emulsions, while those having an excess of absorbed positive ions and wetted by oil give water-in-oil emulsions." By applying this theory to cup greases we arrive at a water-in-oil emulsion because the calcium salts of the higher fatty acids appear to be more readily wetted by oil than by water.

Langmuir [4] explains the mechanism by which colloids are formed as follows:

"If a film of closely packed oleic acid molecules covers the surface of water to which sodium hydroxide has been added, OH groups are absorbed by the COOH radicals, causing an expansion of the lower side of the film without a corresponding expansion of the upper side. This results in the bulging of the film downwards in spots so that it finally detaches itself in the form of particles, the outer surfaces of which consist of COOH groups together with the absorbed OH, while the interior consists of the long hydrocarbon chains. The size of the colloid particles is determined by the difference in size between the two ends of the molecules, just as the size of an arch is dependent upon the relative sizes of the two ends of the stones of which the arch is constructed."

An explanation of the phenomena of emulsification is advanced in the "oriented wedge" theory of Finkle, Draper and Hildebrand [5] and Harkins.[6] Taking Harkins' view of oriented adsorption, the hydrocarbon end of the soap will direct itself toward the oily liquid, while the metallic end will point toward the aqueous phase. If the fatty ends of the molecule can pack together closer than do the metallic ends, the interfacial film bends toward the oil, and we have an emulsion of oil in water. This is the case with sodium and potassium soaps. If, however, a soap of a bi-, or trivalent metal, such as calcium or aluminum, be used, which has several hydrocarbon chains attached, then the interfacial film bends towards the water, and we have an emulsion of water in oil.

Some idea as to the complexity of greases may be gained by merely listing the components which may appear in the oil and soap phases.

[3] Bhatnagar, *J. Chem. Soc.*, **119**, 61, 1760 (1921).

[4] Langmuir, Chem. Met. Eng., **15**, 468 (1916).

[5] Finkle, Draper, and Hildebrand, *J. Am. Chem. Soc.*, **45**, 2780 (1923).

[6] Harkins, "Second Colloid Symposium Monograph," 141, The Chemical Catalog Co., Inc. (Reinhold Publishing Corp.), New York (1924).

OIL PHASE

Lubricating Oils

The lubricating oil fractions of petroleum are considered as being mixtures of naphthenic, aliphatic and aromatic hydrocarbons. Usually they are contaminated with small quantities of nitrogen and sulfur derivatives which, as far as has been demonstrated, have little effect on the colloidal characteristics of greases. The relative proportions of aromatic, aliphatic and naphthenic hydrocarbons, however, influence the yield and transparency of the finished greases in accordance with the polarity of the compounds. The more polar compounds are, apparently, better soap solvents and while they tend to render grease making easier and reduce the chances for batch failures, the yields in some cases are slightly less and in the case of aluminum stearate base greases it is more difficult to obtain perfectly transparent greases.

According to Mikeska,[7] the chief differences in the lubricating oils derived from naphthenic crudes and those from paraffinic crudes may be found in the difference in hydrogen deficiency of comparable fractions and the nature and extent of cyclization. Mabery [8] has reported that the hydrogen deficiency shown for oils deviating from the type formula C_nH_{2n+2} may be as much as thirty hydrogen atoms due to the presence of aromatic and naphthenic material. Such investigators as Mabery and Bestuschew [9] have concluded that paraffine base oils contain a small number of rings and that comparable fractions from asphaltic crudes are composed largely of compounds having highly condensed nuclei, long paraffinic side chains being attached to the latter. Since the number of carbon atoms may vary from 10 to 60, and since different structures may be attributed to any of the following empirical formulae ordinarily assigned to lubricating oils, it is quite evident that the oil phase of grease is alone a most complex material:

$$C_nH_{2n+2}\ ;\ C_nH_{2n}\ ;\ C_nH_{2n-2}\ ;\ C_nH_{2n-4}\ ;$$
$$C_nH_{2n-8}\ ;\ C_nH_{2n-10}\ ;\ C_nH_{2n-12}\ ;\ C_nH_{2n-14}\ ;\ C_nH_{2n-16}.$$

Asphaltenes (85.2% C; 7.4% H; 0.7% S and 6.7% O).

Organic Petroleum Acids (Naphthenic Acids)—$C_{12}H_{23}COOH$; $C_{15}H_{29}COOH$; $C_{18}H_{25}COOH$; $C_{14}H_{27}COOH$.

Waxes—$C_{21}H_{44}$ to $C_{34}H_{70}$.

Organic Sulfur Compounds—Some are the same as in asphaltenes. The sulfur is probably a member of the ring structures.

Organic Nitrogen Compounds—Nitrogen bases $C_nH_{2n-15}N$.

The above mentioned components which are used generally in grease manufacture are mentioned in order to emphasize their extreme complexity. Obviously, a thorough phase rule study of greases would be complicated by an enormous number of eutectics and chemical individuals. It is believed

[7] Mikeska, *Ind. Eng. Chem.*, **28**, 970 (1936).
[8] Mabery, *Am. Chem. J.*, **19**, 419 (1897).
[9] Bestuschew, *Erdöl u. Teer*, **7**, 192 (1931).

that a start should be made by considering relatively pure greases free from extraneous substances.

Soap Phase

Alkali or positive metals are used for forming the soap: Sodium, potassium, calcium, aluminum, lead, zinc, barium, copper, magnesium, mercury, bismuth, chromium, manganese, iron, cobalt, nickel and the ammonium group NH_4. Not all of these 17 materials are used commercially in greases, but all have been used experimentally and are mentioned in the patent literature.

Triglycerides as follow combined with any of the above mentioned positive elements:

Saturated Series:		
Caproin	$C_3H_5(OC_6H_{11}CO)_3$	Cocoanut and palm oils
Caprylin	$C_3H_5(OC_7H_{15}CO)_3$	Cocoanut and palm oils
Caprin	$C_3H_5(OC_9H_{19}CO)_3$	Cocoanut and palm oils
Laurin	$C_3H_5(OC_{11}H_{23}CO)_3$	Cocoanut and palm oils
Myristin	$C_3H_5(OC_{13}H_{27}CO)_3$	Cocoanut and palm oils and spermaceti
Palmitin	$C_3H_5(OC_{15}H_{31}CO)_3$	Animal and vegetable fats
Stearin	$C_3H_5(OC_{17}H_{35}CO)_3$	Animal and vegetable fats
Arachidin	$C_3H_5(OC_{19}H_{39}CO)_3$	Peanut oil
Behenin	$C_3H_5(OC_{21}H_{43}CO)_3$	Ben oil

Unsaturated Series:		
Olein	$C_3H_5(OC_{17}H_{33}CO)_3$	Liquid animal and vegetable fats
Linolein	$C_3H_5(OC_{17}H_{31}CO)_3$	Drying oils
Linolenin	$C_3H_5(OC_{17}H_{29}CO)_3$	Drying oils—rape and linseed oils
Recinolein	$C_3H_5(OC_{17}H_{32}OHCO)_3$	Castor oil

Fatty acids having less than 8 carbon atoms are of little value in producing a consistent grease. The colloidal properties of the salts of the fatty acids decrease with molecular weight. Highly unsaturated fats give low yield values in most cases.

Any free alkali or uncombined metal or metal salt, solid or in solution.

Any uncombined fat or fatty acid.

Glycerol $C_3H_5(OH)_3$.

Water: free, bound to soap, or hydrating free alkali.

Even ordinary cup grease, containing a lime soap base, may consist of 500 to 1000 different chemical compounds. It is, therefore, plain why most investigators attempting to establish fundamental laws pertaining to colloidal science have avoided the study of such complex commercial materials as lubricating greases.

COLLOID PROPERTIES OF THE OIL PHASE

Mineral oils show definite Brownian movements (kinetic motion of small particles visible under the microscope). The Brownian Movement is usually attributed to collisions between the particles and the molecules

composing the liquid medium. The theoretical formula expressing the Brownian Movement [10] may be stated as:

$$D^2 = \frac{R}{N} \times \frac{Tt}{3\pi n} \times \frac{1}{a}$$

in which

D = displacement parallel to any direction during time t, due to molecular collisions.
a = radius of spherical particle
R = gas constant = 83×10^6
N = gas constant = 7×10^{23}
T = Absolute temperature
n = viscosity
t = time

a, n, and t have not been simultaneously determined for greases as yet.

The Brownian movement of particles in the oil is not ordinarily considered of any practical importance, but it should not be overlooked that the bombardment of the soap structure and the collision of minute particles may play some part in the stability of the grease during storage, syneresis and the character of the grease during its processes of coagulation.

Electron interferences in oils have been investigated by Buhl and Rupp.[11] Electrons energized by 100-400 volts were reflected on the surface of the oil and their intensity of reflection at a definite angle was determined with a test electrode connected to an Edelman string electrometer. The wave length of the electrons is changed by varying the potential. The order of reflection and a lattice constant corresponding to the length of one molecule can be determined from the diffraction maxima by Bragg's relation for x-rays. The following results were obtained:

	Lattice Constant d A. U.
Oleic acid	12
Paraffin oil	5–6
Glycerol trioleate	12 3
Veedol	9.9
Texaco	3–15–6.65
Valvoline	8.72

These results apparently indicate that oils of the paraffin series have molecules much longer than the naphthenes.

Mineral oils behave as colloids, particularly when cooled to low temperatures. At temperatures down to their pour point their flow characteristics are those of a fluid obeying the laws of viscous flow, but at lower temperatures the plastic state is attained.

According to Ostwald,[12] mineral oils have long been considered as sols. It is suggested that they be termed oleio-sols. (The dispersed particles have sufficient Brownian movement to keep them in solution.) It has been stated that in lubricating oils rod-shaped micelles occur, and that mechanical

[10] Alexander, J., "Colloid Chemistry," I, The Chemical Catalog Co., Inc. (Reinhold Publishing Corp.), New York (1926).

[11] Buhl, A., and Rupp, E., *Z. Physik.*, **67**, 572 (1931).

[12] Ostwald and Lohre, *Kolloid, Z.*, **45**, 166 (1928).

treatment may alter their absolute viscosities to some extent. Dunstan considers that oils are polyphase systems in which the disperse component is of the same chemical nature as the dispersion medium.

Kyropoulos [13] assumes that the long straight chain molecules of non-polar hydrocarbons have their electrical fields spread along the chain, and therefore form felt-like molecular aggregates which for high molecular weights have very high viscosities. Oils flowing along metal surfaces, as in making viscosity measurements, are subject to an orienting action by the metal, this being more pronounced for the longer chains. The orientation decreases with temperature so that at high temperatures the viscosities of the short and long-chain hydrocarbons become nearly equal. From the standpoint of satisfactory lubrication the viscosity of mineral oil component is no doubt its most important characteristic. This is also true when the oil is compounded with soap to form a grease.

Berl and Umstatter [14] have developed an electrodynamic theory of viscosity which assumes highly polymerized substances and pseudo-crystalline phases. Their results of a study of dielectric constants at different temperatures, molecular weights, and viscosity measurements of several oils indicate that petroleum lubricants are polymerized, but disassociate in solution on heating.

COLLOID PROPERTIES OF THE SOAP PHASE

Usually the soap phase is associated with water; in some cases the soap is hydrated. The later case nearly always applies to the fatty acid salts of lime, when used as grease bases. According to McBain,[10] sodium soap and water mixtures can be made to assume five forms; two crystalline forms, two liquid crystals (anisotropic or doubly refracting liquids) and a series of solutions ranging from crystalloidal to colloidal but which are ordinarily isotropic or singly refracting solutions.

Lamellar Crystals of Soap

The pure anhydrous sodium salts of the fatty acids never occur in commercial greases. In the pure form they are usually colorless or white powders. At about 392° F. they melt to a liquid or liquid crystal condition. At 572° F. they turn to mobile isotropic liquids, miscible with water at high pressures. The lamellar crystals frequently have sharp edges, x-ray examination showing numerous fine lines and indicating that they are typical crystals. During the grease-making process such crystals are seldom met, but where relatively pure stearic acid is employed crystals are encountered and special technic involving the use of protective agents and control of the pH value must be developed.

Curd Fibers

These are very frequently encountered in the preparation of fiber or sponge greases. It is quite likely that the tendency of the potassium and

[13] Kyropoulos, S., *Physik. Z.*, **29**, 942 (1928).

[14] Berl and Umstatter, *Kolloid-Beihefte*, **34**, 1-79 (1931).

sodium soaps to form fibers or curd fibers in aqueous mixtures is doubtless related and probably responsible for the fibrous condition often visible in the fiber greases. McBain [15] concludes that soap curd fibers consist of soap solution which has partially or wholly crystallized out in the form of multitudinous fibers which enmesh mother liquor or soap solution.

In greases the soap fibers also enmesh mineral oil, depending on such factors as the degree of unsaturation of the mineral oil, presence of protective materials such as glycerin and asphaltic matter, quantity of water present and type of soap. Agitation during the coagulation period also has a bearing on quality of the fibers formed.

Curd fibers from aqueous solutions can be readily seen under the ultramicroscope, and in some cases are viewed without magnification. The stability of grease in storage, when subject to high rates of shear, and extremes of temperature, is doubtless influenced to a great extent by the previous history of the fiber formation.

Where the soda soap greases have been heat treated during manufacture in order to remove all but mere traces of bound water, the grease on cooling assumes a buttery or gelatinous condition often quite free from fibers. The moisture content is quite important in promoting fiber formation in the grease.

Double Refracting Liquid Crystal Soap

It is known as "Neat Soap" and contains no fibers. It is homogeneous, plastic and has very little elasticity. This type of soap no doubt occurs in some types of soda base grease.

"Middle Soap." This was discovered by McBain. It is a colorless, transparent, plastic material, resembling vaseline. It is immiscible in Neat Soap and isotropic soap solution. When examined under polarized light Middle Soap frequently shows typical conic anisotropic configurations.

Friedel [10] in his discussion of the mesomorphic state of matter (between the crystalline state and the amorphous) refers to the soaps as being smectic materials. Generally speaking, smectic substances are pasty and are closely associated with nematic substances which are threadlike. With increasing temperatures soaps may pass through the following conditions of state:

Up to T_1 = Crystalline Form
T_1 to T_2 = Smectic Form
T_2 to T_3 = Nematic Form
Above T_3 = Amorphous Form

BEHAVIOR OF SOAP-OIL COMPOUNDS

It is well known that mineral oil of high polarity (i.e., having a large percentage of highly polar molecules) is the best solvent for producing stable soda soap greases; however, less polar paraffinic oils may also be used if protective agents are employed. Oils of low polarity are best for making aluminum stearate greases, while the reverse is true for calcium base greases.

[15] Laing and McBain, *Trans. Chem. Soc.*, **117**, 1507 (1920).

The fact that the aluminum stearate greases are isotropic whereas soda and calcium greases are anisotropic may have some bearing on this matter.

Lubricating greases may consist of:

Mixtures of mineral oil with solids such as graphite, talc, asbestos, etc.
Blends of residuum, waxes, uncombined fats, rosin or pitch.
Soap thickened mineral oils. This is the type we are particularly concerned with and the soaps of Na, K, Ca, Al, Pb or Zn are frequently used.

In the investigation of the gel structure of greases the following questions arise:

Does the soap precipitate or separate from the oil? (stability of the dispersion)
Do greases possess an ordered structure of molecular dimensions, or coarse molecular aggregates?
Are greases thixotropic, *i.e.*, do they "set up" or increase in viscosity or consistency after being broken down?
Are greases subject to syneresis and how can this form of instability be avoided?
What is "melting point"?

The answer to these and other questions will be brought out in the following discussion.

Colloid investigations of greases may be grouped as follows:

Microscopical investigations.
Ultra-microscopic investigations.
X-ray analyses.
Mechanical separation of the oil and soap phases.
Removal of the liquid or oil phase by means of capillary action.
Stability of greases (syneresis, thixotropy).
Swelling, volume changes, density, refractive index, evolution of heat on gelling.
Diffusion in grease gels and conductivity.
Dropping point.
Investigations of the viscous flow of greases.

Microscopic Investigations

J. R. Babb and others have attempted to use the microscope for investigating grease structure. The early efforts to study the structure of lubricating greases by means of the ordinary microscope were unsuccessful on account of the peculiar diffraction effects and the transparency of the soap particles. Farrington [16] has made use of the dark field system of illumination and has had encouraging results. A standard microscope with 3 mm. apochromatic objective lense, iris, 10-magnification eye piece, and a Cardioid condenser gave fairly satisfactory results. A 500-watt Mazda projection lamp served as a light source using a water filter and condenser lenses. To eliminate color fringes a green glass filter was found desirable. At magnifications of one-thousand diameters, particularly observing portions of the grease containing liquid petrolatum stirred into it in order to produce a black background needed for contrast with the lighted fibers, a fairly clear idea of the length of soap fibers was accomplished. Using this method of examination the following classification of lubricating greases may be made:

[16] Farrington, B. B., and Davis, W. N., *Ind. Eng. Chem.*, **28**, 414-16 (1936).

Micro-fiber Greases

The length of fibers in this case is less than one micron and the texture of the grease is buttery and smooth. Under this heading fall nearly all lime base cup greases and aluminum stearate base greases. The latter show no appreciable granular particles as is the case with the calcium soap greases.

Short-Fiber Greases

The length of the fibers in these greases is from one to ten microns. Ordinarily, they will range from five to ten microns in length and are about 0.4 micron in diameter. Driving Journal Compounds, smooth Ball Bearing Greases, and Paper Mill Dryer Roll Bearing Greases, all made with sodium soaps, fall in this classification.

Medium-Fiber Greases

These greases usually contain single, apparently flat and twisted fibers from 10 to 100 microns in length. They are about one micron in width and 0.3 micron thick. Some Wheel Bearing Greases and milled soda base greases, as well as some soda soap base greases made at the highest possible temperatures in open steam kettles, fall in this classification.

Long-Fiber Greases

These contain bundles of fibrils which are more than one-hundred microns in length. The fibrils are about 0.5 microns in diameter. These are the usual long fiber gear greases and chassis lubricants made with soda soaps.

When some greases are employed for lubrication of antifriction bearings at high surface speeds (1000 feet per min.) a structural change may occur and a form of aerogel may be formed. The flock patches of the aerogel can be readily observed on the surfaces of the normal grease by means of the microscope.

Aeration of certain transmission greases may occur and the microscope can be readily utilized for the study and evaluation of this service change in greases.

Microscopic examination with crossed Nichols of greases, which the author has carried out in collaboration with Dr. L. W. McBain of Stanford University (January, 1934), has indicated that normal No. 3 Fiber Grease (sodium-tallow soap base) and No. 3 Cup Grease (Calcium-tallow soap base) are anisotropic.

The smooth lime base cup grease was found to be more uniform in nature, the doubly refracting areas being smaller and of more uniform size than in the case of the soda soap base fiber grease. The fiber grease, containing about 0.75 per cent of water, did not appear as a normal emulsion, and if the water was in an emulsified condition, rather than in close chemical association with the soap, the state of division was exceedingly fine. On the other hand, small droplets of water were visible in the cup grease, indicating that in addition to water bound to the soap, free water was present. It is this emulsified water which influences to a considerable extent the

transparency of the grease. A very transparent lime base grease containing less than 0.5 per cent of water contained no observable droplets.

The fact that soda and calcium base greases are anisotropic, and relatively free from particles of a crystalline nature, substantiates the theory that greases are colloid systems, composed of viscous liquids (oil) in which are enmeshed and entangled immiscible fibrils (micelles or chains) composed of liquid crystals of soap. The liquid crystals of soap may be arranged in conic form, as McBain has proposed for soap in water systems, or are planar (axis of molecules nearly parallel). The evidence available indicates the planar condition as conics were not visible under the crossed Nichols using very high magnification. By special technic, however, it may be possible to view the conic condition and it is considered that further work along this line would yield very interesting results. Since the boundary between sols, jellies [18] and smectic pastes encountered as greases, is not definite, generalized statements regarding grease structure should be interpreted with this in mind.

Arveson [17] states that the soap particles are undoubtedly quite irregular in shape, but under flow conditions and under shear they act as bristles attached to the back of a comb. He considers that the areas between the bristles are occupied by the mineral oil.

The author has observed that aluminum stearate greases under the polarizing microscope are isotropic. This characteristic no doubt is responsible for their attractive clear appearance, and renders partially true the sales claim sometimes heard that such greases are "true solutions." The fact that commercial aluminum stearates may contain large quantities of free stearic acid may have some bearing on the degree of solution or the nature of the liquid crystals.

Ultra-Microscopic Investigations

Babb has used the Ultra-microscope on very soft liquid type greases, and observed definite Brownian movements which he could not distinguish from the movement observed in the oil phase without soap additions.

The author used both a Cardioid Ultramicroscope and a Lietz Slit Type Ultramicroscope for studying consistent greases and found that Brownian movement was not apparent unless they were melted. From this, it is concluded that in many greases the soap structure is so dense that Brownian movement of particles is prevented. This observation is in agreement with Arveson's that molecular bombardment of particles may play a part in grease structure. Fluid greases or melted consistent greases show pronounced Brownian movement.

It is not at all surprising that the Slit-type Ultramicroscope fails to reveal the structure of greases, as many substances of a similar nature have been found to be optically void with this instrument. A very dry calcium grease of soft consistency and containing calcium stearate was examined

[18] McBain, J. W., *J. Chem. Education*, (12), **6**, (1929).

[17] Arveson, M. H., "Flow of Petroleum Lubricating Greases II," Meeting of the American Chemical Society (1933).

with the Cardioid Ultramicroscope. Definite particles of soap were seen. On heating the grease Brownian movement was observed. This was not a normal type of cup grease, however, as it had a transparent but opalescent appearance. Farrington's results with the Cardioid microscope have already been discussed.

X-ray Analysis

In so far as the writer is aware, Bragg's method of x-ray analysis has never been applied to greases. Katz and Gerngross (1925) have reported well defined interference bands for gelatin gels and conclude that gels may contain matter in the semi-crystalline or smectic state. Work along this line would no doubt do much to establish the structure of various types of greases and might prove that the soap structure in greases is in the smectic condition or more definitely that greases are liquid crystal complexes. A study of grease films on bearing surfaces, utilizing the x-ray, is desirable.

Mechanical Separation of the Oil and Soap Phases

Experiments with various filters for testing the stability of greases have shown the ease with which oil could be squeezed from them. Filter paper placed between fine screens was effective but soon clogged with the soap base, also the area (about $\frac{3}{8}$" diameter) was too small. Alundum thimbles were found to be much more satisfactory.

Using a pressure of 10 pounds per square inch on a popular brand of automobile chassis lubricant, 17.8 per cent of soap free oil was readily obtained. Under the same conditions another grease of similar character, containing a lime soap base was found to lose 9.3 per cent by weight of oil. It is obvious that the process of manufacture, character of materials used and formula may greatly influence the stability of the grease. A grease made with 9 per cent by weight of commercial aluminum stearate was found to separate 25 per cent of oil. It is concluded that the attractive forces between oil and soap are not so great, but that it is possible to easily separate large quantities of the more loosely bound oil phase.

The centrifuge and ultra-centrifuge are very useful in determining the tendency of the oil and soap phases to separate.

Removal of the Oil Phase by Means of Capillary Action

The possibilities of using capillary forces for separating mineral oils from greases have been studied by several investigators. Bradford defines gels as two-phase liquid-solid systems, in which the mother liquid is held by molecular and capillary forces. From this standpoint, it is not surprising that liquid oil can be readily drawn from greases by substances offering greater capillarity than the soap base of the grease itself. It is well known that dust and dirt collecting on the outer surfaces of bearings will draw the oil from the grease, leaving a relatively dry soap residue within the bearing. The harmful effects of such residues in clogging oil channels in bearings are obvious. It is, therefore, highly desirable that grease lubricants should

be developed with a view to preventing this form of separation. Careful formulation and the selection of soap stocks, oil stocks, mixed bases, stabilizing agents, rubber latex, polymerized or oxidized hydrocarbons, etc., are possible means to preventing a high susceptibility to separation due to capillary action of surrounding solids. Naturally, the proper material for accomplishing this purpose must affect the inherent capillary forces of the soap base. In the case of lime soap greases, it is possible that the soap and added water are loosely bound together, thus increasing the capillarity of the dispersed soap. Evaporation of the water from such greases will in most cases cause breakdown.

Researches have been conducted having as their object the development of a method or test procedure for predicting whether or not greases will bleed oil on storage or in service. In one instance, a given volume of the grease surrounded by a small metal cylinder open at both ends, is placed on filter paper for a given period of time. The area covered by the oil drawn from the grease by the paper is taken as a measure of the stability of the grease and its characteristics with regard to resisting capillary attractions. By dehydrating the grease prior to filling the cylinders a further indication of the stability of the grease is revealed. Typical tests of this kind on a series of cup greases containing lime-tallow soap are listed below:

Cup Grease No.	Percent Soap	Area of Oil Spot on Filter Paper	
		Normal Grease	Grease after Dehydration
00	9.8	15.2 sq. cm.	18.1 sq. cm.
0	13.6	14.1 sq. cm.	28.6 sq. cm.
1	11.5	12.6 sq. cm.	18.1 sq. cm.
2	14.3	10.7 sq. cm.	12.6 sq. cm.
3	17.7	8.6 sq. cm.	12.0 sq. cm.
4	25.0	5.3 sq. cm.	28.3 sq. cm.
5	35.6	2.8 sq. cm.	6.2 sq. cm.

It is evident that the normal greases containing greater quantities of soap are less subject to releasing oil to the filter paper. After dehydration the greases are not dependent on the quantity of soap so much as on its character, free alkali content, etc.

Viscosity of the oil constituent plays an important part in controlling separation due to capillarity. Other factors being equal, the more viscous oils will not penetrate through the filter paper at as high a rate as less viscous oils.

Syneresis, Thixotropy

Syneresis, "weeping," or "bleeding," of greases is frequently noticeable and many developments have been made in their manufacturing technic to prevent this phenomenon from reaching objectionable proportions.

Factors affecting the syneric properties of greases are enumerated below:

Water Content
- Quantity present in grease.
- Relative amounts of water bound to soap and in a relatively free emulsified condition.

Nature of Soap Stock—relative amounts of stearic and oleic acid soaps

Kind of Soap and Quantity

Special Stabilizing Materials:
Glycerin
Unsaturated hydrocarbons
Emulsion agents—degras, sulfonated oils, saponin, etc.
Oxidized and polymerized hydrocarbons

All greases are thixotropic to a more or less extent. Soda soap greases made with viscous oils may continue to harden over periods of a year or more. Calcium greases increase appreciably in consistency after being freshly prepared, after which they may remain fairly stable or become softer with age. Oxidation and loss of water may harden greases, but this can hardly be termed a thixotropic phenomenon. On heating and reworking the thixotropic cycle may be repeated.

Swelling

When heated, greases frequently swell slightly, due to other causes than simple expansion. This may be explained theoretically by assuming that direct combination, sorption or the polarization of molecules or groups of molecules of the oil with exposed groups of the soap micelle for which the oil may have a great affinity. Another view is that free molecules of the oil penetrate between the interlacing micelle structure to release strains. Sodium and aluminum soap greases swell appreciably, while for calcium greases this phenomenon is not so apparent.

In certain types of calcium soap greases slight swelling and increase in consistency has been observed on slowly heating, apparently due to dehydration of the soap, and a change in structure of the soap phase.

Diffusion and Conductivity

Experimental work on the diffusion of various dyes through greases, should be of considerable interest in determining the nature of their structure. It is possible that in greases containing considerable quantities of water, sub-freezing temperatures will produce minute ice crystals, their formation through the grease being a form of diffusion.

Evidences of diffusion of water through dehydrated soda soap greases are frequent in industrial applications where such greases come in contact with water. In some transparent soda base grease the path taken by the water is readily observed. Diffusion of water through calcium or aluminum base grease is also possible, although from a practical consideration these products are considered as being relatively unaffected by water.

Diffusion of alcohol, glycerin, etc., through greases used for lubricating pumps on automobile cooling systems, has occurred in some instances.

Diffusion and conductivity are closely allied. It might be expected that the more viscous greases would offer greater interference to the migrating ion. Completely dehydrated greases should be very poor conductors, but it is quite difficult to prepare the completely dry mixtures of soap and oil. If it is true that the migrating ion travels through the liquid, which in the case of a dehydrated grease is the mineral oil, the rate would obviously be of a very low order due to its poor conductivity and high viscosity.

Dropping Point

The melting or dropping point of greases has long been considered as a criterion of their resistance to liquefaction by heat and consequently leakage. This is not always the case, however, as surface tension, adhesion and other factors are not similar in service applications to the same extent as in laboratory tests.

With the application of heat, greases which may be smectic, will pass into what may be termed the nematic state, and finally into an amorphous or liquid state. These transitions are gradual, and very often no definite melting point exists. It is often the case with gels that the terms "melting point" and "setting point" have no exact meaning.

The rates and extent of heating and cooling greases affect their "melting points." No doubt the size of the soap aggregates is greatly affected by the processes of heating and cooling during manufacture. In some cases a definite soap grain structure is intended to produce high melting points.

Viscous Flow

The viscous flow of greases has been studied and reported on by Arveson [17] and will not be discussed here in detail. Incidentally, flow studies of grease at various shear rates and temperatures are of considerable value in predicting service performance. In the laboratory, flow studies before and after greases have been subjected to agitation and stirring are extremely valuable as indications of service stability.

Several theories have been advanced in explanation of the type of flow which takes place. Some refer to it as the flow of "telescoping layers." Arveson visualizes flow of grease as being similar to that of two continuous brushes with bristles pressed together, the space between being filled with viscous oil. The bristles are supposed to represent the projections of the maze of soap particles in the grease.

COLLOID CHEMISTRY

The following definitions of units, colloid terms, and disperse systems, and brief discussion of suspensions, colloids and crystalloids, will be useful to anyone interested in the colloid nature of lubricating greases. The definitions as given will apply throughout the following chapters.

Definition of Units

μ	Micron	$\frac{1}{1{,}000}$ mm.	10^{-3} mm.
mμ	Millimicron	$\frac{1}{1{,}000}$ μ	10^{-6} mm.
Å	Ångstrom Unit	$\frac{1}{10}$ mμ	10^{-7} mm.
R. U.	Rutherford Unit	$\frac{1}{100{,}000}$ Å	10^{-12} mm

Definition of Terms

Disperse System (dispersion, dispersoid). A system composed of more or less finely divided matter distributed throughout a gaseous, liquid, or solid medium. The components or phases of the system are known respectively as the *dispersed phase* and the *continuous phase.*

Homogeneous System. A system in which there are no apparent surfaces of discontinuity (Lewis and Randall).

Heterogeneous System. A system consisting of two or more distinct homogeneous regions or phases, which appear to be separated from each other by surfaces of discontinuity (Lewis and Randall).

Interface. This is the surface between two phases (Alexander).

Specific Surface. This signifies external, free, or rind surface per unit weight (Alexander).

Suspension (mechanical suspension). A dispersion of particles sufficiently large that physical forces control their behavior.

Colloid (colloid solution). A dispersion of particles so finely divided that surface phenomena and kinetic energy control their behavior.

Crystalloid (true solution). A dispersion of molecules or ions.

Emulsion. A suspension of one liquid within another.

Suspensoid. A colloid in which the dispersed phase is a solid (Alexander).

Emulsoid. A colloid in which both phases are liquid.

Iso-colloid. A substance which is colloidally dispersed in itself, e.g., as colloidal ice in water (Alexander).

Sol. This signifies a colloidal dispersion whose particles have sufficient Brownian Motion to keep them in solution. The dispersing medium is often employed as a prefix, *e.g.*, hydrosol—in water (Alexander).

Gel. This is an aggregation of colloidal particles into practically non-motile larger groups (Alexander).

Order of Complexity of Matter

Material Unit	Order of Complexity	Approximate Size Limits
Proton	0 ?	2×10^{-8} Å
Electron	0 ?	3.8×10^{-5} Å
Atomic nucleus		H = 2×10^{-8} Å Light atoms = 5×10^{-5} Å
Atoms	1	0.1 to 0.6 mμ
Molecules	2	0.2 to 5 mμ
Molecular groups	3	0.5 to 10 mμ
Primary colloidal particles	4	2 to 20 mμ
Secondary colloidal particles	5	5 to 100 mμ
Microscopically resolvable particles	6	Over 100 mμ
Visible particles	7	Over 50 μ

Subdivision and Increase of Specific Area

To illustrate the enormous increase of specific surface (ratio of total volume) with subdivision, the following tabulation shows the dimensions resulting from the successive decimal subdivision of a 1 cm. cube until particles of molecular proportions are obtained.

Length of Edge	Number of Cubes	Total Volume	Total Surface	Specific Surface
1 cm.	1	1 cu. cm.	6 sq. cm.	6
1 mm.	10^3	1 cu. cm.	60 sq. cm.	6×10
0.1 mm.	10^6	1 cu. cm.	600 sq. cm.	6×10^2
0.01 mm.	10^9	1 cu. cm.	6000 sq. cm.	6×10^3
1 μ	10^{12}	1 cu. cm.	6 sq. m.	6×10^4
0.1 μ	10^{15}	1 cu. cm.	60 sq. m.	6×10^5
0.01 μ	10^{18}	1 cu. cm.	600 sq. m.	6×10^6
1 mμ	10^{21}	1 cu. cm.	6000 sq. m.	6×10^7

Approximate Limits of Dispersion Phenomena

1Å	1mμ	10mμ	100mμ	1μ	10μ	100μ	1mn
			Wave length ⟵ ⟶ of light				
Crystalloid ⟶ Dispersions	⟵ Col Dispe	loid ⟶ rsions	⟵ Suspe	nsions			
	Ultramic ⟵ Fi Particle but not r	roscopic eld s visible esolvable	→| ←Part	Microsco icles visible	pic Field and resolv	able	
Particle very r	s move apidly	Particle slo	s move wly ⟶	Brow ⟶|⟵	nian Move not visible	ment	
Particles pass through ⟶ membranes	Parti ⟵ filter pap	cles pass thr er but not m	⟶|⟵ ough embranes	— Part	icles retain filter paper	ed by	
Quart	z no longer	settles ⟶	⟵ Quartz	settles			
Oil emuls	ion forms no	cream layer	⟶|⟵	Oil emulsio	n forms cr	eam layer	

Classification of Disperse Systems

Dispersed Phase	Continuous Phase	Examples
Gas	Gas	No example; gases miscible in all proportions.
Gas	Liquid	Fine foam, air in gear grease or in ball bearing grease.
Gas	Solid	Gaseous inclusion in metals and minerals.
Liquid	Gas	Atmospheric fog, clouds, gases at critical state. Grease fog in gear cases.
Liquid	Liquid	Emulsions of oil and water, oil and water greases stabilized by soaps, etc., liquid crystal greases.
Liquid	Solid	Mercury in ointments, water in paraffin wax, oil in journal grease.
Solid	Gas	Cosmic dust, smoke, condensing vapors.
Solid	Liquid	Oildag, crystalline soap in greases.
Solid	Solid	Solid solutions, colloidal gold in ruby glass, coloring matter in gems.

Nature of Colloids

Alexander refers to colloid chemistry as "this fascinating twilight zone between physics and chemistry."

In defining colloid chemistry he says: "Colloid chemistry deals with the behavior and properties of matter in the colloidal condition, which as we now know, means a certain very fine state of subdivision. While there are no sharp limitations to the size of particles in colloidal dispersions, it may in a general way be stated that their sphere begins with dimensions somewhat smaller than a wave length of light, and extends downward well into dimensions which theory ascribes to the molecules of crystalloids."

Suspension—Colloid—Crystalloid

Concerning the distinction between suspension and solution Alexander says: "With the aid of the ultra microscope, which renders visible particles approaching in minuteness molecular dimensions, Zsigmondy has shown that there is no sharp line of demarcation between suspensions and colloidal solutions, but that with increasing fineness in the subdivision of the dissolved substance, there is a progressive change in the properties of the resulting fluids, the influence of gravity gradually yielding to that of the electric charge of particles, of surface tension and of other forms of energy.

On the other hand, there is no sharp distinction between colloidal and crystalloidal solutions, but as the particles in solution become smaller and smaller, the optical heterogeneity decreases correspondingly, finally vanishing as molecular dimensions are approached."

As the state of a dispersion passes from the suspended to the colloidal the well known Brownian Movement appears and increases as the particles become smaller until in a true solution it becomes enormous both in speed and amplitude.

As early as 1861 Graham pointed out some of the most marked differences in the properties of colloids and crystalloids. He noted the following properties of each:

Crystalloids. These have a high diffusive mobility, are chemically active, and are a statical condition of matter.

Colloids. These have low diffusive mobility, are relatively chemically inactive, have a wide sensibility to external agents, may exist as gelatinous modifications of fluid colloids, and may be considered as dynamical state of matter.

Graham's suggestion that the basis of colloidality is the composite character of the colloid molecule (as contrasted to molecular or ionic dispersion in crystalloids) is the idea now generally accepted.

Homogeneity and closeness of union in definite proportions by weight are suggested by Alexander as criteria of chemical combination. In this sense colloidal solutions are not chemical combinations, since such condition depends not upon the total mass involved but upon the total free or active surface.

For practical purposes colloid solutions may be considered as heterogeneous and crystalloid solutions as homogeneous. However, the distinction between homogeneity and heterogeneity depends entirely upon the refinement of our means of observation. Strictly speaking, no system is homogeneous, since combinations of the smallest known subdivisions of matter are discontinuous.

The Colloidal State

According to Alexander, the anomalous properties of colloidal matter may be attributed to the relative increase of specific surface and kinetic activity with fineness of subdivision.

He states: "In following the gradual dispersion of a substance from gross visible particles all the way down to true molecular subdivision, it is obvious that in the colloidal zone there is a transition from a state where the kinetic activity is small as compared to specific surface to a state where the kinetic activity is large as compared to the specific surface. In the heart of the colloidal zone there is usually observable an indefinite portion of the curve which has been termed the zone of maximum colloidality, wherein colloidal properties, such as viscosity, are most marked."

Further: "We may therefore pass upward into the colloidal state as molecules aggregate or become larger, or pass downward into the colloidal state as coarse particles are more finely ground or dispersed."

Bibliography to Colloid Chemistry Definitions

Alexander, J., "Colloid Chemistry," 3rd. Ed., D. Van Nostrand Co., New York (1929).

Banc oft, W. D., "Applied Colloid Chemistry," 1st Ed., McGraw-Hill Book Company, New York (1921).

Holmes, H. N., "Laboratory Manual of Colloid Chemistry," 2nd Ed., John Wiley and Sons, New York (1928).

Ostwald, W., "A Handbook of Colloid Chemistry," 2nd Ed., P. Blakiston's Sons and Company, Philadelphia (1918).

Kruyt, H. R., and van Klooster, H. S., "Colloids," 2nd Ed., John Wiley & Sons, New York (1930)

Freundlich, H., "Colloid and Capillary Chemistry," E. P. Dutton & Co., New York (1922).

Alexander, J., "Colloid Chemistry: Theoretical and Applied," 1, 2, 3, 4, The Chemical Catalog Co., Inc. (Reinhold Publishing Corp.) New York (1926-1932).

Chapter II

Materials Used in Grease Manufacture

In the preceding chapter some of the ingredients utilized in manufacturing lubricating grease were mentioned; however, no attempt was made to list in detail all of the most important materials or to describe them and record their physical tests. For the sake of convenience we shall list and discuss grease making materials under the following headings: lubricating oils, asphaltic residua, petrolatum and waxes, soap stocks including animal and vegetable fats and oils, fatty acids, perfumes, inorganic and organic fillers, and the alkali bases. Where cost data is given the figures are rough average values and are included not for the sake of their absolute value but to show the relative cost of ingredients for the period indicated. For this reason, such cost figures should be of interest to the formulator and grease plant management in selecting soap stocks, but must, of course, be used with greatest caution in costing as only the best current prices should be employed in estimating the cost of a given grease, making due allowance for freight, cost of storage and handling.

LUBRICATING OIL

The analyses of several thousand greases have shown that soap contents may vary from 0.25 per cent for certain textile and automotive crankcase oils to as much as 60 per cent by weight for some railroad driving journal compounds. Thus, it may be said that the lubricating oil portion of greases may range from 99.75 per cent to about 40 per cent. This is considering of course, that grease is a combination of a petroleum lubricant and a soap or mixture of soaps, suitable for the lubrication of certain types of bearing surfaces. Other greases are considered in the following chapters which contain no soap and may consist of residua blends, waxes, graphite and asbestos mixtures with oil and pitches combined with oil or inert fillers, all of which may produce lubricants which are non fluid and may be termed greases.

The mineral oil content is very important in that many of the properties possessed by the oil are imparted to the grease. When cup grease is subjected to violent disruptive forces such as are found between a journal and its bearing, or when stirred violently, it may lose much of its gel nature and break down to an oil and a soap. Inasmuch as the lime soap itself is not considered an efficient lubricant, it is the oil on which we must depend for satisfactory lubrication. Cup greases found on the market vary considerably in this property of stability, some being capable of withstanding vigorous agitation without excessive breakdown. In general, we find a wide variation in the properties of the mineral oil used. This is believed

to be due to the fact that users of greases have found from experience that their semi solid lubricants should be made up from oils of correct viscosity for particular conditions, in much the same way as it has been learned that there is an oil of most desirable body or grade for any given service.

The majority of cup greases are made with light lubricating oil. There are very few greases on the market having a Saybolt viscosity at 100° F. of less than 90. The greater proportion of cup greases are made with an oil of 90 to 180 viscosity at 100° F. A better grade of cup grease, having an oil of 180 to 300 viscosity, is manufactured by most of the larger concerns.

One well known manufacturer of cup grease in 1923 had for several years marketed a lubricant for the various bearings found on automobiles, which was termed a pressure lubricant. This term was derived from the method of applying the grease to the bearings under a pressure of several hundred pounds per square inch. In this process, which is now being used on nearly all automobiles and trucks as well as a wide variety of industrial machines, the grease is packed in a grease gun or "pressure gun" and forced to the bearings, through a flexible metal hose by means of a piston. In some grease guns a lever arrangement is employed to operate the piston. The chief point of difference between the grease marketed as pressure lubricant and the regular cup greases which had been used to a great extent in compression grease cups for automobile lubrication was the viscosity of the oil used as an ingredient of the grease. Whereas an oil of 100 to 180 viscosity at 100° F. was used in the regular cup grease an oil of 300-500 viscosity is employed in the manufacture of the pressure lubricant. The advantage of using the oil of higher viscosity is that the resulting grease when used for lubricating spring bolts and other bearings found on automobile chassis has a greater resistance to being forced from the bearings, one application of the pressure grease lasting much longer than the usual type of cup grease. Another advantage of the pressure grease is that it handles well in the pressure guns and has little tendency to change its characteristics of color and consistency due to the presence of air, with which it is often forced into contact under high pressure.

The advantages of the original pressure lubricant were soon apparent to other manufacturers and it was not long before a large number of special pressure greases were on the market. Some of these have been examined and the results show that oils having a viscosity over 500 at 100° F. are being used. One manufacturer is producing a special pressure lubricant from a Gulf Coast red oil having a viscosity of 1300 at 100° F. It is of note, however, that this product gave considerable trouble due to the fact that even when made with a small amount of soap to give a soft consistency, it is difficult to force it through the pressure gun and into the bearings.

Certain brands of cup grease to be used for crank pin lubrication on the donkey engines used in the lumber industry and for general construction work have been investigated and found to have been made with a heavy black oil of 100 to 250 Saybolt viscosity at 210° F.

Vroom [1] states that the oil contained in cup greases should conform

[1] Vroom, G. B., *J. Am. Soc. Naval Engrs.*, (3), **37**, 553.

in all respects to those of an oil that would be specified for similar operating conditions where oil instead of grease could be used. He suggests a series approximately as follows:

Specifications for Oil in Cup Greases (Vroom).

Contained oil, Saybolt viscosity at 100° F.	Flow point of grease, ° F.	Consistency of grease	A.S.T.M. penetration
180 to 200	140–150	Soft	300
180 to 200	165–175	Medium	200
180 to 200	210–220	Hard	125
250 to 300	140–150	Soft	300
250 to 300	165–175	Medium	200
250 to 300	210–220	Hard	125
360 to 410	140–150	Soft	300
360 to 410	165–175	Medium	200
360 to 410	210–220	Hard	125

The Society of Automotive Engineers have assigned numbers to Transmission and Rear Axle Greases which are based on the viscosity of the oil content and channeling requirements in service. This classification is essentially as given below:

Number	S. U. viscosity at 100° F. of the oil content only — Min.	Max	Grease must not channel in service at ° F.
80	....	800	Minus 10
90	800	1500	0
110	1500	3000	10
160	3000	6000	35
250	6000	....	..

Garlick[2] considers that ordinary cup greases made with light spindle oils will frequently give acceptable service results under conditions in which the lubricating oil alone would prove unsatisfactory. It is, of course, obvious that the quantity and nature of the soap in a lubricating film will affect its viscosity and lubricating value. The viscosity of the oil and the kind and quantity of soap used must be chosen with due regard for the service to be encountered. This is the reason we have such a wide variety of greases on the market, and it is certainly not sufficient to say that the lubricating value of greases may be graded by the viscosity of the oil content.

Gear greases, driving journal compounds, ball bearing greases and in many other grease lubricants viscous cylinder stocks or bright stocks are employed. The typical lubricating oils found in one of the largest grease factories in the Mid-Continent area (1925) were:

Name of Oil	S.U.V. at 100° F.	Pour ° F.	Cleveland Open Cup Flash ° F.	Fire ° F.	A.P.I. Gravity	N.P.A. Color
100 Ohio Neutral	110	30	340	380	28.5	3½
100 Texas Neutral	110	0	290	330	22.0	3
200 Texas Red Oil	210	0	330	380	21.5	7
500 Texas Red Oil	500	0	338	382	19.0	7
700 Texas Red Oil	710	0	365	420	18.8	8
FFF Penn Bright Stock	152/210	40	555	625	27.0	FFF
Black Oil	202	30	500	580	19.0	Black

[2] Garlick, H. S., *J. Inst. Petroleum Tech.*, (131), **20**, 829-883 (1934).

An Atlantic Coast (1928) grease manufacturer found it necessary to stock the following oils:

Name of Oil	S.U.V. at 100° F.	Pour ° F.	Cleveland Open Cup Flash ° F.	Cleveland Open Cup Fire ° F.	Gravity	Color *
100 Penn Neutral	100	35	360	410	28.0	2½
100 Gulf Coast	100	0	305	340	22.0	3
1300 Gulf Coast	1300	30	405	465	17.5	5½
Penn E Brt. Stk.	140/210	80	540	610	26.5	8 Dil.
Black Oil (Smackover)	80/210	0	370	430	15.0	Black

* S.U.V. indicates Saybolt Universal Viscosity. All gravity data will be given in terms of the A.P.I. scale unless otherwise noted. Colors are by the Union Colorimeter by the N.P.A., or A.S.T.M. scale.

A grease plant on the Pacific Coast producing lubricants which have been found quite satisfactory, stocks the following oils for the manufacture of one hundred and twenty different greases (1935).

Name of Oil	S.U.V. at 100° F.	S.U.V. at 210° F.	Gravity	Flash-Fire ° F.	Pour ° F.	Color A.S.T.M.	Approximate[3] Cost of Manufacture
100 Calif. Red	110	39.0	25.4	310–350	—0	4	4.5c per gal.
150 Calif. Pale	160	44.0	23.1	340–395	—0	2	4.5c per gal.
300 Calif. Pale	320	48.4	23.0	370–420	—0	3	5.9c per gal.
600 Calif. Red	645	58.8	21.8	400–450	—0	7	7.3c per gal.
1000 Calif. Pale	1025	68.2	21.3	430–495	0	4	7.8c per gal.
1000 Calif. Red	1025	69.5	21.5	425–495	0	7	8.1c per gal.
Naphthenic Bright Stock	4940	147	19.6	525–600	15	8	11.0c per gal.
Lub. Distillate	9600	162	14.6	465–545	40	Gr.-Blk.	3.5c per gal.
Mid-Con. SR Stk.	....	180	23.0	540–620	50	Drk.Gr.	14.0c per gal. (Purchased)
Penn. FFF Stock	....	140	26.0	540–605	20	8	23.0c per gal. (Purchased)
Black Oil *	870	...	18.3	245–280	0	Black	2.0c per gal.
Lt. Vacuum Dist.	220	43.0	19.5	345–395	0	Gr.-Blk.	3.0c per gal.
Med. Vacuum Dist.	940	60.0	17.0	385–435	0	Gr.-Blk.	3.2c per gal.
Hvy. Vacuum Dist.	2700	87.0	15.6	425–475	10	Gr.-Blk.	3.4c per gal.
No. 2 White Oil	105	...	30.2	325–365	..	30 Saybt.	50.0c per gal. (Purchased)

* Contains 3.32 per cent by weight hard asphalt by the Holde Method.

The following prices are of interest in indicating the bulk prices which must be paid at the oil terminals (refineries) for lubricating oils suitable for grease making, the figures being for the summer of 1936:

Kind of Oil	S.U.V.	Color	Cents per Gallon	
Texas Neutral (Unfiltered Pale)	100/100° F.	3	5.00– 5.75	F.O.B. Gulf Terminals
Penn Neutral	160/100° F.	3	23.5 –24.5	F.A.S. New York
Mid-Cont. Neutral	180/100° F.	3	16.0 –17.5	F.A.S. Gulf Terminals
Texas Red	100/100° F.	6	5.25– 5.50	F.O.B. Gulf Terminals
Texas Pale	300/100° F.	3	7.00– 7.50	F.O.B. Gulf Terminals
Texas Red	1200/100° F.	6	9.00– 9.50	F.O.B. Gulf Terminals
Texas Pale	1200/100° F.	4	9.00– 9.50	F.O.B. Gulf Terminals
631 Mid-Cont. SR Stk.	180/210° F.	Ol.Gr.	18.0	F.O.B. Gulf Terminals
Penn. Bright Stk.	140/210° F.	FFF	28.0	F.O.B. New York

[3] A rough estimate based on the market value of lubricating oils, includes cost of lubricating distillate, Edeleanu treatment, neutralization and redistilling under vacuum. It is interesting to note that the red oils are valued slightly higher than pale oils of equivalent viscosity. This is due to the fact that in some cases red oils are simply dyed pale oils and, therefore, the cost of coloring must be added.

Oils in the foregoing lists may be referred to in grease formulae in the following chapters. In many cases, however, special oils will be required for certain greases and in these instances tests on the oils will be given in as great detail as possible. In general the terminology utilized in the foregoing lists will be employed.

ASPHALTIC RESIDUA

(Bitumens, Air Blown and Steam Refined Asphalts)

Usually two or three grades of asphalt are needed in grease compounding plants for use in heavy greases for exposed gears, crankpin greases, and cable lubricants. These products derived from either Texas Gulf Coast, or California Crudes should have tests complying with the following requirements:

	100 to 120 Steam Refined Asphalt	50 to 60 Steam Refined Asphalt	10 to 20 Airblown Asphalt
Specific gravity 25° C., min.	1.018	1.020	1.014
Penetration at 77° F. (A.S.T.M. D5-25)	100–120	50–60	10–20
Ductility at 77° F., min. (A.S.T.M. D113-26T)	111	111	1
Flash point °F. C.O.C., min.	500	540	450
Melting point B. & R. method °F. (A.S.T.M. D36-26), min.	108	120	220
Fixed carbon, per cent by weight, (A.S.T.M. D168-30), max.	9.8	10.6	...
Percent loss on heating at 5 hr. 325° F. (A.S.T.M. D6-30), max.	.5	.5	.5
Penetration after heating, per cent of original penetration, min.	80	80	60
Percent ash, max.	.20	.20	...
Percent sulfur	1.50	1.50	...
Percent soluble in CS_2, by weight, min.	99.8	99.8	99.0
Percent soluble in CCl_4, by weight, min.	99.8	99.8	99.0
Percent soluble in 60 to 80° gasoline	92.0	90.0	60.0

PETROLATUM

A wide variety of petrolatums derived from wax bearing crudes in this country are employed in the manufacture of lubricating greases. As ball bearing lubricants, agents for decreasing the fiber length of soda soap greases, anti-rust compounds, slushing compounds, ingredients of greases intended for use in the food industry and as a component of automobile wheel bearing greases, petrolatums are utilized in appreciable quantities. Various grades are available at the following approximate prices:

Name or Color	Price, Cents per pound	A.S.T.M. Color Number
U.S.P.	...	1
Snow White	6¼–7½	1
Lily White	5¼–6½	1
Cream White	5 –6	1½
Light	3 –4	2
Medium	2½–3½	3
Dark Amber	2 –3	4
Red (Light)	2 –2⅞	5
Dark Green	1½–2½	4 Dilute

Color. This test is best made by means of the Union Colorimeter in accordance with the procedure recommended in A.S.T.M. Tentative Method D218-34T.

Consistency. The A.S.T.M. Tentative Method for Determining Penetration of Greases and Petrolatum, D217-33T., should be used for determining the consistency. The petrolatum should be melted, poured into a suitable can and cooled for several hours before making the penetration test.

Melting Point. This should be made by A.S.T.M. method D127-30, wherein a thermometer bulb is coated with the melted sample under specific conditions, and then mounted in a water bath the temperature of which is raised at the rate of 3° F. per minute until 100° F. is reached, and then at a rate of 2° F. per minute until the first drop of petrolatum leaves the bulb. This is noted as the melting point. The usual tests on several samples of dark green petrolatum ordinarily used in dark lubricating greases are listed below:

Sample Number	1	2	3	4	5
Specific gravity 15.5° C.	.920	.901	.921	.915	.870
A.S.T.M. color	8	4½ Dil	8	8	8
S.U.V. at 210° F.	89	112	90	75–100	51– 55
Flash ° F., O.C.	535	530	535	510–535	395–405
Fire ° F., O.C.	605	610	615	590–615	455–465
A.S.T.M. melting point ° F.	140	124	154	140–145	124–130
Percent water	.20	...	1.3	...	...
Percent ash	.036	...	.011	...	...

CERESIN WAX (Purified Ozokerite)

This wax is either white, yellow or orange. The latter two grades are sometimes used in engine greases and lubricating compounds. The range of characteristics are:

Specific gravity	.910–.950
Melting point ° F.	134–161

SOAP STOCKS

(Saponifiable Oils, Fats and Fatty Acids)

Cup and fiber greases may be prepared from any of the animal or vegetable oils or fats capable of being saponified by hydrated lime or caustic soda. The whole fats (triglycerides) or the fatty acids naturally occurring in them or derived from them may be employed. Such factors as color of finished grease, melting point, equipment available for process, quality and viscosity of mineral stock, odor of finished grease, and texture must be taken into consideration in the selection of the most appropriate soap stock for producing a particular grease. It is not our purpose to present extensive data on the chemical and physical properties of the fatty materials employed, as this information may be obtained in any of the standard works on oils, fats and waxes. However, for those who do not have such books available,

Table 1 gives a list of the principal fats, and the amounts of hydrated lime and caustic soda with which they are capable of reacting to form soaps.

Most manufacturers use very high grade tallows, lard oil and other fats in some products where lower grades of fat could be used if they could be secured from reliable sources and of uniform quality. Changes in color, per cent of unsaponifiable matter and melting point of fats are contributing factors to the production of greases of poor uniformity. The best brands of cup grease on the market are manufactured from horse fat, lard oil, tallow

Table 1—Alkali Required for Theoretical Saponification of Principal Fats and Oils Used in the Manufacture of Greases

Glyceride	Saponification Value mg. KOH/gr.	Per cent by weight Caustic Soda of 98 per cent NaOH (.728 × Sap. Value)	Per cent by weight Hydrated Lime of 72 per cent CaO (.694 × Sap. Value)
Rape seed oil	173	12.6	12.0
Castor oil	180	13.1	12.5
Lard oil	195	14.2	13.5
Tallow oil	193	14.0	13.4
Corn oil	190	13.8	13.2
Cottonseed oil	195	14.2	13.5
Soya bean oil	192	14.0	13.3
Beef tallow	195	14.2	13.5
Horse fat	196	14.3	13.6
Hydrogenated fish oil	193	14.0	13.4
Sperm oil	140	10 2	9.7
Beeswax	89	6.5	6.2
Degras	130	9.5	9.0
Spermaceti wax	130	9.5	9.0
Oleic acid (Red oil)	195	14.2	13.5
Stearic acid	215	15.7	14.9
Wool fatty acids	203	14.8	14.1
Rosin	175	12.7	12.2
Rosin oil	90	6.5	6.2

and tallow oil; other fats less frequently used are palm oil, cottonseed oil, whale oil, fish oil, soya bean oil, bone fat, garbage grease (reclaimed), castor oil, hydrogenated oils, naphthenic acids, degras, and rape seed oil.

It is agreed by most practical grease makers that horse fat gives cup greases of smoother texture and more unctuous consistency than can be obtained with other fats. Mixtures of lard oil and tallow, however, may be used in the production of lubricants which are believed to be in every way as satisfactory as those in which horse fat alone is employed as a soap stock. Those fats in which the unsaturated fatty material predominates—whale oil, soya bean oil, fish oil, etc.—produce greases of low melting point and slightly rubbery texture. It is not apparent that greases made from fatty acids are superior to those made with the whole fats. The fats yield on saponification about ten per cent of their weight of glycerin, which remains in the grease. If the soap is made before the addition of any mineral oil to the mixture it is possible to recover the glycerin by methods similar to those used in the soap industry. As yet the recovery of glycerin in grease manufacturing has not gained popularity. Many manufacturers use oleic acid alone or in combination with fats in making cup greases, particularly

when the reaction is carried out in an open kettle and speed of reaction is an important factor. The use of stearic acid is often resorted to for the production of greases of high melting point; considerable care must be used to prevent lumps of calcium stearate from forming.

Table 2—Principal Fats, with Their Chemical Formulas, Molecular Weight, and Other Data.

Name	Formula	Molecular weight	Per cent NaOH absorbed	Per cent Glycerol set free	Per cent Fatty acids set free	Natural source (See below)
Saturated fats of the acetic series:						
Formin	$C_3H_5(O.HCO)_3$	176	68.19	52.28	78.41	1
Acetin	$C_3H_5(O.CH_3CO)_3$	218	55.04	42.20	82.56	2
Propionin	$C_3H_5(O.C_2H_5CO)_3$	260	46.15	35.38	86.14	3
Butyrin	$C_3H_5(O.C_3H_7CO)_3$	302	39.74	30.47	87.42	..
Valerin	$C_3H_5(O.C_4H_9CO)_3$	344	34.88	26.74	88.94	4
Caproin	$C_3H_5(O.C_5H_{11}CO)_3$	386	31.09	23.83	90.16	5
Œnanthylin	$C_3H_5(O.C_6H_{13}CO)_3$	428	28.04	21.50	91.14	..
Caprylin	$C_3H_5(O.C_7H_{15}CO)_3$	470	25.54	19.57	91.91	6
Perlargonin	$C_3H_5(O.C_8H_{17}CO)_3$	512	23.44	17.97	92.58	7
Caprin	$C_3H_5(O.C_9H_{19}CO)_3$	554	21.66	16.61	93.13	8
Undecylin	$C_3H_5(O.C_{10}H_{21}CO)_3$	596	20.14	15.44	93.63	..
Laurin	$C_3H_5(O.C_{11}H_{23}CO)_3$	638	18.81	14.42	94.06	9
Tridecylin	$C_3H_5(O.C_{12}H_{25}CO)_3$	680	17.65	13.53	94.41	..
Myristin	$C_3H_5(O.C_{13}H_{27}CO)_3$	722	16.63	12.75	94.75	10
Pentadecatoin	$C_3H_5(O.C_{14}H_{29}CO)_3$	764	15.70	12.04	95.11	..
Palmitin	$C_3H_5(O.C_{15}H_{31}CO)_3$	806	14.89	11.41	95.30	11
Daturin	$C_3H_5(O.C_{16}H_{33}CO)_3$	848	14.16	10.85	95.52	12
Stearin	$C_3H_5(O.C_{17}H_{35}CO)_3$	890	13.48	10.34	95.72	13
Nondecylin	$C_3H_5(O.C_{18}H_{37}CO)_3$	932	12.87	9.87	95.92	..
Arachidin	$C_3H_5(O.C_{19}H_{39}CO)_3$	974	12.32	9.44	96.10	14
Medullin	$C_3H_5(O.C_{20}H_{41}CO)_3$	1016	11.81	9.06	96.27	15
Behenin	$C_3H_5(O.C_{21}H_{43}CO)_3$	1058	11.34	8.69	96.40	16
Unsaturated fats of the oleic series:						
Olein	$C_3H_5(O.C_{17}H_{33}CO)_3$	800	15.00	11.49	95.26	24
Unsaturated fats of the linolic series:						
Elaeomargarin	$C_3H_5(O.C_{16}H_{29}CO)_3$	638	14.35	11.01	95.45	26
Linolein	$C_3H_5(O.C_{17}H_{31}CO)_3$	878	13.67	10.48	95.68	27
Unsaturated fats of the linolenic series:						
Linolenin	$C_3H_5(O.C_{17}H_{29}CO)_3$	872	13.76	10.55	95.56	28
Unsaturated fats of the ricinoleic series:						
Ricinolein	$C_3H_5(O.C_{17}H_{32}OH.CO)_3$	932	12.87	9.87	95.92	29

(1) The acid in certain nettles, in the bodies of ants, and in the needles of certain species of pine trees. (Never used in the soap or grease industry; included in order to make the table complete.) (2) The chief acidic constituent of vinegar. The esters occur in the odoriferous oils of many plants. (3) Small quantities in butter fat. (4) Cows' butter. (5) Palm oil, cocoanut oil and butter fat. (6) Same as 5. (7) Said to occur in the leaves of the Pelargonium roseum. (8) Same sources as 5. (9) Same sources as 5. (10) Spermaceti, cocoanut oil and palm oil. (11) Together with olein and stearin in practically all of the animal and vegetable fats and oils. (12) Very seldom found in animal or vegetable oils and fats. (13) Occurs in large quantities in tallow, lard, and to some extent in nearly all animal and vegetable oils and fats. (14) One of the constituents of peanut oil. (15) Very seldom occurs naturally; said to be a combination of palmitic and stearic acids. (16) One of the constituents of ben oil. (24) One of the constituents of lard oil, tallow, and most of the animal and vegetable fats and oils. (26) Small quantities in the drying oils. (27) The drying oils such as linseed oil, tung oil, etc. (28) One of the chief constituents of linseed oil. (29) Castor oil.

The principal fats, with their chemical formulas, molecular weights, percent caustic soda required for theoretical saponification, percent glycerol set free by saponification, percent fatty acids obtainable from the whole fat, and natural sources are given in Table 2.

Table 3—Principal Fatty Acids, with Their Chemical Formulas, Molecular Weights, and Quantity of Caustic Soda Required for Neutralization

Name of acid	Formula	Molecular weight	Per cent NaOH absorbed
Saturated fatty acids of the acetic series, general formula $C_nH_{2n+1}COOH$			
Formic	H . COOH	46	86.96
Acetic	CH_3COOH	60	66.67
Propionic	C_2H_5COOH	74	54.06
Butyric	C_3H_7COOH	88	45.45
Valeric	C_4H_9COOH	102	39.22
Caproic	$C_5H_{11}COOH$	116	34.48
Œnanthylic	$C_6H_{13}COOH$	130	30.77
Caprylic	$C_7H_{15}COOH$	144	27.77
Perlargonic	$C_8H_{17}COOH$	158	25.31
Capric	$C_9H_{19}COOH$	172	23.26
Undecylic	$C_{10}H_{21}COOH$	186	21.51
Lauric	$C_{11}H_{23}COOH$	200	20.00
Tridecylic	$C_{12}H_{25}COOH$	214	18.27
Myristic	$C_{13}H_{27}COOH$	228	17.55
Pentadecylic	$C_{14}H_{29}COOH$	242	16.53
Palmitic	$C_{15}H_{31}COOH$	256	15.63
Daturic	$C_{16}H_{33}COOH$	270	14.81
Stearic	$C_{17}H_{35}COOH$	284	14.09
Nondecylic	$C_{18}H_{37}COOH$	298	13.43
Arachidic	$C_{19}H_{39}COOH$	312	12.82
Medullic	$C_{20}H_{41}COOH$	326	12.28
Behenic	$C_{21}H_{43}COOH$	340	11.77
Carnaubic	$C_{23}H_{47}COOH$	368	10.87
Hyaenic	$C_{24}H_{49}COOH$	382	10.47
Cerotic	$C_{26}H_{53}COOH$	410	9.76
Melissic	$C_{30}H_{61}COOH$	452	8.85
Unsaturated fatty acids of the oleic series, general formula $C_nH_{2n-1}COOH$			
Acrylic	C_2H_3COOH	72	55.55
Crotonic	C_3H_5COOH	86	46.51
Tiglic	C_4H_7COOH	100	40.00
Hypogaeic	$C_{15}H_{29}COOH$	254	15.75
Oleic	$C_{17}H_{33}COOH$	282	14.19
Unsaturated fatty acids of the linolic series, general formula $C_nH_{2n-3}COOH$			
Elaeomargaric	$C_{16}H_{29}COOH$	266	15.04
Linolic	$C_{17}H_{31}COOH$	280	14.29
Unsaturated fatty acids of the linolenic series, general formula $C_nH_{2n-5}COOH$			
Linolenic	$C_{17}H_{29}COOH$	278	14.40
Unsaturated fatty acids of the ricinoleic series, general formula $C_nH_{2n}O_3$			
Ricinoleic	$C_{17}H_{32}OH$. COOH	298	13.43

The principal fatty acids are listed in Table 3, with their chemical formulas, molecular weights, and the per cent of caustic soda required to neutralize them.

Soap Stocks' Statistics

A comparison of the quantities of saponifiable vegetable and animal oils and fats consumed by the lubricating grease industry with other oil and fat consuming industries indicates that grease making is indeed not a negligible factor. According to data compiled by the United States Tariff Commission,[4] for the period from 1912 to 1924, the total average yearly consumption of vegetable oils, animal fats and fish oils was 6,400,623,000 pounds. The fatty materials included in this survey were coconut, corn, cottonseed, peanut, and soya bean oils, vegetable stearin and hydrogenated oils, pork fat and lard, oleo stearin, oleo oil, greases and tallow. The range in consumption of these stocks by the most important industries, for the period 1912-1924, were given as follows:

Industry	Pounds Consumed Yearly
Lard-compound	876,927,000–1,173,446,000
Oleomargarine	66,537,000– 314,596,000
Lubricating greases *	37,500,000
Salad oil and mayonnaise	6,264,000– 11,420,000
Soap and detergent	775,156,000–1,188,776,000

* Based on annual production of 250,000,000 pounds of grease.

The above tabulation accounts for only about half the total yearly consumption of fatty materials amounting to about six and one-half billion pounds, the balance being spread among many small industries.

Examination of Soap Stocks

It is not intended to give detailed instructions for the chemical and physical testing of the saponifiable materials used in grease making; however, a few remarks relative to the interpretation of the results obtainable from such tests, particularly with regard to their significance in grease making, should not be out of place:

Sampling: It is difficult to obtain a representative sample of fat from incoming shipments when made in 50-gallon barrels. It has been suggested that the best procedure is to utilize some form of tryer or sampler and take samples from at least one of each three barrels received. A proportional composite sample may then be made and the sample melted and thoroughly mixed. Where the fatty materials are in a liquid condition at the temperature when received sampling by means of a thief as recommended by the A.S.T.M. is suggested. Those interested will find the precautions and procedures described in A.S.T.M. Standard,[5] Designation D270-33, most helpful and in line with the practices carried out by the oil industry. For tank car shipments an upper, middle and lower sample should be taken and composited. For cakes of stearic acid a borings sample, consisting of the chips obtained by the use of an auger, is suggested.

[4] "Certain Vegetable Oils" (Part 2, Economic Study of the Trade in and the Prices and Interchangeability of Oils and Fats.) 1926.

[5] A.S.T.M. Standards 1933, Part II, "Non-Metallic Materials," 846-867.

Samples should be carefully marked preferably with a pencil hard enough to dent the card board tag, noting material sampled, order number, date, name of sampler, number and kind of packages (tank car number) and laboratory tests required.

Sampling devices, bottles, corks, screw or slip top sample cans, and even the samplers hands, should be kept clean to avoid contamination of the samples with substances which would render the laboratory tests of little use.

Iodine Number: (Number of grams of iodine absorbed by 100 grams of fat or fatty oil) (Syn.: Iodine Value): The iodine number gives a measure of the chemical unsaturation of the oil or fat. High iodine numbers of 117 to 206, as in the case of vegetable drying oils, indicate the suitability of the oil in paint manufacture, but is ordinarily considered an objectionable feature of soap stocks for grease manufacture, for the reason that the chemically unsaturated fats are more subject to oxidation and color depreciation, even when converted to appropriate soaps used as lubricating grease bases. A low iodine number is characteristic of solid fats, the range for animal fats being 19 to 100. An item which affects the prices of grease making soap stocks is the fact that most edible oils and fats have iodine numbers in the same range as are considered desirable in soap and detergent manufacture as well as producing best yields in grease manufacturing.

The iodine number is also a direct measure of the amount of hydrogen an oil will combine with when subjected to the hardening process. Hydrogenation lowers the iodine number and increases the melting point. Hydrogenated fats are being used in increasing volume by grease makers, although in some cases a lower yield, peculiar odors, and the production of less transparent greases have prejudiced a few prospective users. The iodine number is probably the most important constant used for identifying fatty materials or fatty acids. Unlike the fatty acids or glycerides of the acetic or saturated series, the unsaturated acids and fats of the oleic and linoleic series absorb halogens to form chiefly addition products. As an example, oleic acid, $C_{17}H_{33}COOH$, reacts with two atoms of iodine to form the addition product, di-iodo stearic acid, $C_{17}H_{33}I_2COOH$. The test method originally proposed by Hübl and modified by Wijs makes use of iodine monochloride. More recently, Hanus proposed the use of iodine bromide as it was more readily prepared for use.

Melting Point: The melting point of fats and fatty oils indicates whether it will be a solid or a liquid and can be readily handled at usual atmospheric temperatures. Fats are usually mixtures of various triglycerides, consequently they do not have a sharp melting point, but on heating tend to shrink and then pass gradually into the liquid condition. The melting points of fatty acids may be determined with greater accuracy particularly if they are in a relatively pure state. Several methods have been proposed by different investigators and none appears to have yet been thoroughly standardized and universally adopted.

Capillary Tube Method. A small quantity of the melted fat is drawn into a tin walled capillary tube about 3 cm. long and then placed on ice.

After the fat has congealed for several hours, the tube is fastened by means of rubber bands to the bulb of an accurate thermometer. The thermometer is then mounted in a flask, or large test tube, of water and gradually heated at a rate of about 0.5° C. per minute until the fat is observed to melt, which is ordinarily when it becomes transparent.

The Pohl Method. The bulb of a thermometer is coated with the melted fat about 3 mm. thick. The thermometer is then mounted as in the capillary tube method in a flask of water which is slowly heated until melting is observed.

The Bouis Method. This is similar to the Capillary Tube Method, the modification consisting in arranging the tube, open at both ends, so that as melting occurs the liquefied fat is forced by the water to the level of the water. By some, this is termed the softening point as the fat plug which moves to the water surface may not be completely transparent.

The Wiley Method. Small thin disks of the fat are prepared by dropping the melted sample on a cake of ice. A two-layer system of alcohol and water is then prepared and cooled with ice. A disk of the fat, about 1 cm. in diameter, is then introduced into the alcohol mixture and sinks to the point where the density of the solution is equivalent to its own. Heat is then applied very slowly, about two minutes per degree Centigrade, and the temperature noted at which the disk becomes a sphere.

The Ubbelohde Method. The apparatus for making this test, consisting of a small glass cap and orifice attachable to the bulb of a special thermometer, is available in many petroleum laboratories, and for this reason the dropping point of fats obtained by this method is often reported to the grease plant. The sample is filled into the small glass orifice tube, attached to the thermometer bulb and both suspended in a test tube surrounded by a water bath. Heating is carried out at about 2° F. per minute and the temperature noted at which the first drop of melted fat falls to the bottom of the test tube.

Titer Test (Solidifying Point of Mixed Fatty Acids): Oils and fats of high titer test produce hard soaps and consequently give high yields of lubricating grease. The titer is much more characteristic than the melting point and has therefore been widely adopted by soap makers for the evaluation of fats. Briefly, the test is made by obtaining the mixed fatty acids from the fat by saponification, decomposition of the sodium soaps with sulphuric acid and washing out the glycerol set free, the sodium sulphate and the excess sulphuric acid, with boiling water. The melted fatty acids are then transferred to a glass bottle and stirred with a thermometer while cooling. When no change in temperature is noted for thirty seconds, stirring is discontinued, and a slight rise in the temperature will be noted. The highest temperature attained on this rise is the titer.

Valenta Test: The turbidity of the mixed fatty acids with respect to 90 per cent acetic acid is a very useful means of identifying fats as such or after they have been saponified and employed in grease making. The fatty acids must be very carefully isolated as impurities cause irregular results.

One tenth ml. of the melted fatty acids are introduced into a special glass tube 5 cm. long and 7 mm. in diameter. One end is closed and graduated at 0.1 and 0.2 ml. capacities. One tenth ml. of 90 per cent acetic acid is also introduced using a small dropping tube. A thermometer equipped with a water tight rubber stopper is then placed in the tube and the apparatus warmed in water until the mixture becomes clear and homogeneous. It is then removed from the water and gently agitated until the temperature is reached at which turbidity is noted. This temperature is ordinarily well defined and may be read to within a tolerance of 0.5° C.

TABLE 4.—Valenta Test

	(90 per cent by weight Acetic Acid Turbidity Temperatures ° C.)
Origin of Fatty Acids	
Rape oil	75
Oleic acid	64
Stearic acid	64
Nut oil (Arachis oil)	61
Sperm oil	57
Palm oil	55
Beef tallow	55
Seal oil (Fish oil)	54
Lard oil	53
Neatsfoot oil	52
Cottonseed oil	49
Wool wax	41
Cottonseed stearin	34
Palm kernel oil	30
Coconut oil	20
Castor oil	Soluble at 15
Blown Fatty Oils	
Blown rape oil (acids soluble in petroleum ether)	Above 50
Blown fish oil (acids soluble in petroleum ether)	35–45
Blown castor oil	Soluble at 15
Sulphonated Oils	
Sulphonated fish oil	Soluble at 15° C., but rendered insoluble on heating at 100° C., (often during isolation and drying).
Sulphonated castor oil	Soluble at 15° C. (Not affected by heating at 100° C.)

Acid Number (Neutralization Number or Neutralization Value):[6] This is the weight in milligrams of potassium hydroxide required to neutralize the fatty acids in one gram of fat or fatty oil. The per cent of free fatty acids may also be reported as percentage of oleic acid. The presence of free fatty acids in soap stocks may ordinarily be attributed to decomposition of the tryglycerides brought about by the chemicals used in refining them, or bacterial action which may be influenced by moisture, air, heat and

[6] See: "A.S.T.M. Tentative Standards, 1935," "Neutralization Number of Petroleum Products and Lubricants. A.S.T.M. Designation D188-27T," 732.

light. The acid number is not a reliable means of identifying fats and oils, as fats of the same class may vary greatly in the content of free fatty acids. Free fatty acids are, of course, objectionable in fats intended for food as they may be the materials which cause the fat to be "rancid." The presence of even very large amounts of free fatty acids in stocks intended for lubricating grease or soap manufacture does not make them unsuitable, but does tend to reduce the price which a soap maker can pay for them because the free fatty acids reduce the yield of glycerin obtainable, which is a valuable by-product. Of further interest is the fact that fats containing from 10 to 50 per cent of free fatty acids commonly have an objectionable odor and color. Both of these items are of importance in producing greases of high and uniform quality.

Saponification Number (Saponification Value, Koettstorfer Number): This is the number of milligrams of potassium hydroxide required to saponify one gram of the fatty material. The presence of free fatty acids tends to increase the saponification number. If the acid number is subtracted from the saponification number, the milligrams of KOH actually used in saponifying the glycerol esters is obtained and is known as the ESTER NUMBER. The saponification number is a fair means of identifying fatty material and may also be used for determining the amount of a fatty product blended with mineral oil. The Standard Method for Saponification Number, A.S.T.M. Designation D94-28, may be found on page 868 of the A.S.T.M. Standards for 1933.

Color: The color of grease making soap stocks is of considerable importance in that much of the color existing in the fat may appear in the final product. The purchase of lubricating greases is appreciably affected by the color and appearance of the product. Where dark and black greases are being made the color of the soap stock need not be considered, but the trouble of purchasing, the expense of storing additional grades of fats and the advantages of buying large quantities of one grade often prevent taking full advantage of the lower price of low grade fats.

Probably the best and most convenient method for determining the color of fats and fatty oils is the Tentative A.S.T.M. Method for Color of Petrolatum by Means of the Union Colorimeter. Products not darker than 8 color are heated to from 20 to 30° F. (11-17° C.) above their melting point, placed in a 4-ounce sample bottle and examined by means of the A.S.T.M. Union Colorimeter. For darker products dilution with 85 per cent by volume of water white kerosene is recommended. The A.S.T.M. Color numbers and the N.P.A. color names are:

1 —Lily White	4 —Orange Pale
1½—Cream White	4½—Pale
2 —Extra Pale	5 —Light Red
2½—Extra Lemon Pale	6 —Dark Red
3 —Lemon Pale	7 —Claret Red
3½—Extra Orange Pale	8 —Dark

Odor: Cheap soap stocks such as yellow grease, brown grease, and reclaimed fats may have an objectionable odor which will be imparted to

the lubricating grease in which it may be used. When fats are acted on by oxygen from the air, particularly in the presence of moisture and light, free fatty acids are formed which are subject to further changes, often producing a disagreeable odor and acrid taste. If the odor of the soap stock is not too objectionable it may be concealed by incorporating a suitable amount of an appropriate perfume, with the grease before drawing it from the kettle.

No exact method for making the organoleptic test for odor has been developed as the olfactory nerves and sensitivity of different operators varies greatly. A theory regarding the nature of odors has been advanced based on the assumption that odors arise from the unsatisfied affinity (residual affinity) of the molecule. Fats and fatty oils follow the general rule that unsaturated compounds have stronger odors than saturated bodies. In some cases age, degree of refinement, and purity cause exceptions to the rule.

Specific Gravity: Either the Westphal balance, pyknometers, or hydrometers may be used for determining the specific gravity of fatty materials. The tables for converting specific gravity to A.P.I. degrees and pounds per gallon, as given in the Tag Manual for Inspectors of Petroleum,[7] are quite useful in computing formulae and kettle charges; however, the tables for correcting gravities from observed temperatures to the standard temperature of 60° F. (15.5° C.) should not be used as they are applicable to petroleum products rather than fats.

If considerable precision is desirable the gravity should be calculated using the following formula:

$$\text{Sp. Gr.}\frac{15.5}{15.5} = \text{Sp. Gr.}_t + k(t - 15.5)$$

where Sp. Gr._t is the specific gravity at the observed temperature t in degrees C. The symbol k is the change in specific gravity for one-degree C. Values of k may be taken as:

	Value of k
Beef tallow	.000674
Lard oil	.000651
Stearic acid	.000750
Oleic acid	.000656

Acetyl Value: This is the number of milligrams of potassium hydroxide required to neutralize the acetic acid obtained by saponifying one gram of the acetylized oil or fat. It is of value in identifying fats containing the hydroxyl group for the reason that in this test acetic anhydride is heated together with the fat, the hydrogen of the hydroxyl group is replaced by the acetyl group which is later set free after saponification by means of hydrochloric acid. Castor oil in which the glyceride of rincinoleic acid predominates has unusually high acetyl values ranging from 146 to 151. Other soap stocks have the following values:

[7] Published by the C. J. Tagliabue Manufacturing Company, 88, 32nd Street, Brooklyn, N. Y.

Fat or Fatty Oil	Acetyl Value
Beef tallow	2.7–8.6
Bone fat	11.5
Corn oil	7.5–11.5
Cottonseed oil	21–25
Lard oil	3.5
Neatsfoot oil	7.5–9.5
Palm oil	11.5
Rapeseed oil	15
Sardine oil	20–22
Soya bean oil	5

Hehner Value (The percentage of water-insoluble fatty acids obtainable from a fat or fatty oil): This test is not frequently applied to fats used in lubricating grease manufacture.

Barium Value: The relation between the percentage of water soluble and insoluble barium soaps is of value in determining the presence of cocoanut oil in edible fats but is seldom used for fats used as lubricating grease bases.

Polenske Number: This is the number of milliliters of tenth normal potassium hydroxide required to neutralize the insoluble volatile fatty acids distilled from five grams of fat. It is of most interest in connection with butter analysis.

Reichert-Meissl Number: The number of milliliters of tenth normal sodium hydroxide needed to just neutralize the water soluble volatile fatty acids distilled from five grams of fat is called the Reichert-Meissl Number. It is of no great importance as applied to grease making fats.

Viscosity: The viscosity of stocks such as lard oil, tallow, oleic acid and rape seed oil is sometimes needed when making blends of these materials with mineral oil. In grease plants this value is most conveniently obtained by means of the Saybolt Universal Viscosimeter or the Saybolt Furol Viscosimeter in the case of blown rape seed oils. (Abbreviations S.U.V. and S.F.V.). For fats, the test is usually carried out at a bath temperature of 210° F. (98.9° C.). For complete details regarding the procedure for making viscosity tests: "A.S.T.M. Standards, Designation D88-33."

Percent Ash: This is obtained by igniting a small sample in a crucible. More than a very small trace of ash indicates the presence of soaps left from refining, or that the fat has been contaminated with dirt, iron scale, or other foreign material.

Percent Water: If an incoming shipment of fat is found to contain more than a mere trace of water, as indicated by the popping noise made when the fat is heated in a small pan over a flame, then the percentage of water should be accurately determined with a view to rejecting the shipment or correcting the purchase price in line with the purchase specifications and agreement with the supplier. The most convenient method for this determination at a grease plant laboratory is the "A.S.T.M. Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials, Designation D95-30." The sample dissolved in a special gaso-

line is heated in a glass flask and the water deposited in a graduated glass trap after being condensed. This trap is graduated in 0.10 ml. divisions and the difference in making duplicate determinations should not be more than 0.20 per cent for fifty gram samples.

Classification of Soap Stocks

Saponifiable fats and oils most frequently encountered in grease making may be classified as shown below:

I. Non Drying Vegetable Oils (Olive Oil Type)
- Olive Oil
- Peanut Oil

II Non Drying Vegetable Oils (Rape Oil Type)
- Rape Seed Oil

III. Non Drying Vegetable Oils (Castor Oil Type)
- Castor Oil

IV. Non Drying Animal Oils
- Lard Oil
- Neatsfoot Oil
- Tallow Oil

V. Vegetable Semi Drying Oils
- Corn Oil
- Cottonseed Oil
- Sesame Oil
- Soya Bean Oil

VI. Vegetable Drying Oils
- China Wood Oil
- Linseed Oil
- Tung Oil

VII. Vegetable Fats
- Chinese Vegetable Tallow
- Palm Oil

VIII. Animal Fats
- Beef Tallow
- Bone Fat
- Horse Fat
- Lard
- Mutton Tallow

IX. Sperm Oils
- Arctic Sperm Oil
- Sperm Oil

X. Animal Waxes
- Beeswax
- Degras
- Spermaceti

XI. Vegetable Waxes (Non-Glyceridic)
- Carnauba Wax
- Montan Wax

XII. Fish and Marine Animal Oils
- Herring Oil
- Menhaden Oil
- Sardine Oil
- Whale Oil

PROPERTIES OF SOAP STOCKS

Non Drying Vegetable Oils

Peanut Oil and Olive Oil. Peanut oil at 9½ to 13½c per pound and olive oil at 11 to 25c per pound are too expensive for use in lubricating greases. Olive oil foots at 10c a pound has been used in some specialty greases. Olive oil contains about 25 per cent solid and 75 per cent liquid triglycerides. Peanut oil contains from 4 to 6 per cent of arachidin and lignocerin, but as yet there is no published evidence that these substances are of particular merit in grease making.

Rape Seed Oil. This consists chiefly of the triglycerides of rapic and erucic acids, and the separated mixed fatty acids contain approximately 95 per cent of unsaturated acids. Raw rape seed oil is used for compounding car oils and sometimes in making gear greases where low pour points and channeling tests necessitated by extreme cold climates, are required. Suggested purchase specifications and typical characteristics of the raw oil are:

	Suggested Purchase Specifications	Typical Tests
Specific gravity	.910–.918	.913–.917
Pour point ° C.	0 Max.	−12 to −1
Iodine value	90–105	94–105
Acid number	6 Max.	.5–3.0
Percent ash	Trace	.003
Percent water	None	
Solid impurities	None	
Saponification value	170–175	168–179
A.S.T.M. color	2½ Max.	2–2½
Halphen test (test with sulphur and carbon bisulphide reagents for presence of cottonseed oil)	Negative	...
Titer test ° C.	11–14	11.5–13.7

The cost of rape seed oil may vary from 6½ to 8½c per pound, the blown oils ranging from 4 to 5c per pound higher.

Blown Rape Seed Oil. Typical tests on this product used for compounding lubricating oils for the production of Marine Engine Oils are:

Specific gravity	0.970
A.S.T.M. color	2½
S.U.V. at 210° F.	315
Halphen test	Negative
Solubility in petroleum ether	100 per cent

Vegetable Castor Oil. This is seldom used in grease manufacture except by a few specialty manufacturers who consider the soaps of castor oil are superior lubricants although they are difficult to work with and give low yield. It is obtainable in U.S.P., No. 1, No. 2, and No. 3 grades. Suggested purchase specifications for the No. 1 grade together with typical tests are:

	Suggested Purchase Specifications	Typical Tests
Specific gravity $\frac{15.5° \text{ C.}}{15.5° \text{ C.}}$	.960–.970	.952–.972
Pour point ° C.	−10 Max.	−18 to −15
Acid number	3 Max.	.1–2.0
Saponification value	179–183	175–183
Iodine Value	85 Max.	83–85
A.S.T.M. color	3 Max.	2–3

The cost of castor oil may vary from 9 to 15c per pound depending on demand and the grade. In addition to the above tests a solubility test is usually made consisting of mixing one part by volume of the oil with four volumes of 90 per cent ethyl alcohol at about 20° C. The castor oil should be completely soluble. Castor oil is of course not miscible with lubricating oils and even gasoline.

IV. Non Drying Animal Oils

Lard Oil. In the preparation of cup greases experience has shown that mixtures of Tallow and Lard Oil permit the production of products practically equivalent to those manufactured by the old school grease makers when using horse fat. Lard oil is produced by the cold pressing of lard ren-

dered by means of either the fire or steam process, from hog fat. It is obtainable in the United States in the following grades: Prime Winter Edible, Prime Winter Inedible, Off Prime, Extra No. 1 Winter Strained, No. 1, No. 2, and No. 3. Typical properties of pure lard oil are listed in the following table:

	Properties of Lard Oil
Specific gravity $\frac{15^\circ \text{ C.}}{15^\circ \text{ C.}}$	0.913–0.915
Solidifying temperature ° C.	−2 to 4 (28.4–39.2° F.)
Acid value	0.1–5.0
Saponification value	193–198
Iodine value	62.5–79
Hehner value	97
Unsaponifiables	0.6 per cent
Fatty acids:	
Melting point ° C.	33–38.4
Titer test ° C.	27–33

One large grease manufacturer purchases Lard oils on the basis of the following requirements:

Extra No. 1 Winter Strained Lard Oil. Must be pure lard oil of good quality. The color shall not be darker than No. 3 A.S.T.M. It must not solidify at temperatures above 45° F. It must contain not more than 2 per cent of free fatty acids calculated as oleic acid. It must contain no mineral oil or other adulterants and when saponified with alcoholic potassium hydroxide the resulting soap must be soluble in water showing no turbidity. When shaken with alcoholic silver nitrate solution it must not show the usual dark red color reaction for cottonseed oil.

Extra No. 1 Lard Oil. Shall not be admixed with other oils. Its color shall not be darker than No. 4½ A.S.T.M., must contain not more than 15 per cent of free fatty acid calculated as oleic acid, and when saponified with alcoholic potassium hydroxide the resulting soap must be soluble in water, showing no turbidity which would indicate the presence of mineral oil.

No. 1 Lard Oil. Shall not contain other fatty oils as adulterants, its color may be brown but must be lighter than No. 4½ A.S.T.M. and shall not contain more than 20 per cent free fatty acids calculated as oleic acid. It must pass the test for the presence of mineral oils and unsaponifiables as for the two previous specifications.

Purchase Specifications for No. 1 Winter Strained Lard Oil for Use in Grease Manufacture:

	Specifications	Range of Actual Tests
Specific gravity $\frac{15.5^\circ \text{ C.}}{15.5^\circ \text{ C.}}$	0.915–0.920	0.915–0.918
A.S.T.M. Pour point ° F.	45 Max.	35–45
Iodine value (Hanus)	65–80	70–78
Acid value (mg. KOH per gram)	10 Max.	6–10
Percent ash	Trace	.001–.01
Percent water	Trace only	none–0.20
Solid impurities	Trace only	Trace
Saponification value	190 Min.	193–198
A.S.T.M. color	3½ Max.	2–3½
Odor	Sweet	Satisfactory

The average cost of lard oil meeting the above specifications, delivered to California grease plants, was 9.5c per pound. The New York prices for lard of different grades have been (about) as follows:

	Cents per pound F.O.B., New York 1935	1936
Prime winter edible	9¾–20½	14 –16
Extra No. 1 winter strained	8½–11¾	9½–11½
No. 1	8½–11	8½– 9½
No. 2	10 –11	....

Lard oil may be shipped in railway tank cars equipped with steam heating coils or in wooden barrels of 375 to 400 pounds capacity.

Tallow Oil. The so-called acidless tallow oils are commonly used in compounding plants as an ingredient of steam cylinder oils. Suggested specifications include:

	Suggested Specifications	Typical Tests
Specific gravity $\frac{15.5° \text{ C.}}{15.5° \text{ C.}}$	.900–.925	.905–.920
Pour point ° C.	35 Max.	20–34
Iodine value	50 Max.	45–53
Acid value	2	.2–1.5
Percent ash	Trace Max.	.001
Percent water	0.5	Trace
Solid impurities	None	....
Saponification value	190 Min.	190–198
A.S.T.M. color	2 Max.	1–2
Titer test ° C.	38 Max.	33–37
Odor	Sweet	....

Neatsfoot Oil. This material used in compounding steam cylinder lubricants should meet the following requirements:

	Suggested Specifications
Specific gravity	.900–.917
Pour point ° C.	—7 to 0
Iodine value	65–91
Acid number	5 Max.
Percent ash	Trace
Saponification value	192–200
A.S.T.M. color	2 Max.
Titer test ° C.	18–30

V. Vegetable Semi Drying Oils

Corn Oil, Cottonseed Oil, and Soya Bean Oil. They have all been used in the commercial manufacture of lubricating greases as have their fatty acids. Typical tests on the oils are:

	Corn Oil	Cottonseed Oil	Soya Bean Oil
Specific gravity	.921–.928	.919–.920	.924–.927
Pour point ° C.	—10 to —20	15 to —14	—8 to —15
Acid value	1–3	.5–1.5	.3–3
Saponification value	187–193	194–196	189–194
Iodine value	111–128	103–112	121–139
Melting point of fatty acids ° C.	17–21	32–35	22–31
A.S.T.M. color	1–2	1–3	1–3

Corn oil consists of approximately 4.5 per cent saturated triglycerides, 44.8 per cent is olein, and linolin 48.2 per cent. Cottonseed oil is also chiefly composed of the glycerides of oleic and linolic acids. Soya bean oil contains about 88 per cent of unsaturated glycerides including those of oleic, linolic, linolenic, and lignoceric acid. These unsaturated, liquid fatty acids do not give as high yields of grease as the more consistent solid fats. Soya bean oil was used by one manufacturer to give his cup grease a slight greenish tint. The mixture of soap stocks used in making cup greases should not contain large quantities of the unsaturated fatty substances as oxidation and color depreciation is likely to occur.

VI. Vegetable Drying Oils

China Wood Oil, Tung Oil and Linseed Oil. They are practically never used in grease making as they are too expensive and even of greater importance is the fact that they would cause gumming of bearings or parts due to their affinity for oxygen. In some cases they have been suggested as ingredients for cable coatings and as lubricants for large exposed, slow moving gears, but even in these uses their high cost must be considered. As base stocks for the synthesis of new soap stocks to meet particular needs they appear to have some promise.

VII. Vegetable Fats

Palm Oil and Chinese Vegetable Tallow. They have been used in cup grease manufacture in the past to a considerable extent but their use has now been largely abandoned on account of their high price and lack of characteristics which warrant their use.

Lagos Palm Oil used in a well known brand of cup grease on the Pacific Coast was purchased on the basis of its free fatty acid content, the acid number ranging from 40 to 100.

VIII. Animal Fats

Tallow. From the standpoint of volume more tallow is used in grease making within the United States than any other soap stock. The reason for this may be found in the quality of the greases obtainable when using this fat and in its fairly low cost as compared with the value of tallow sold in other countries, where it is more often refined to produce edible grades.

Tallow is rendered from the suet or adipose tissue of cattle and sheep. Beef tallow is ordinarily preferred to mutton tallow, particularly when considering the yield of railroad journal greases prepared by the cold sett process, although the chemical constitution indicates no important differences. The suet may be rendered in an open kettle at low temperature or is more commonly subjected to steam under pressure in closed rendering vats. Commercial tallows often have a dirty brown or gray color, which may be improved by refining consisting of treatment of the remelted tallow with hot water sometimes with the addition of sodium carbonate, sodium chloride or alum. Fresh tallow should not have a strong odor. On being stored for long periods of time mutton tallow particularly develops a

peculiar sweetish odor which, according to some investigators, is due to the formation of a volatile fatty acid. Tallow ordinarily consists of about 61 per cent of stearin, 33 per cent olein and 6 per cent of palmitin, but may vary rather widely from these values depending on the nature of the feed consumed by the live stock from which it was obtained.

With regard to evaluating the grease making value and yield of grease from various grades of tallow, both the titer test, which is the solidifying point of the isolated mixed fatty acids, and the iodine value are of importance. The higher the percentage of stearin and saturated triglycerides the higher the titer and the lower the iodine number. The iodine number of tallow may vary from 35 to 46 and the titer test from 38 to 46° C.

Contaminants sometimes found in tallow consist of water, metal scale from shipping and storage containers, dirt, particles of skin, hair, and sinew. These may be determined by dissolving 10 to 20 grams of the tallow in a flask with 50 ml. of petroleum ether and pouring through a dried, tared filter. The filter should be carefully washed with the ether until all of the fat has been extracted, when the filter (preferably a Gooch) is dried and weighed, the increase being insoluble materials other than water which must be determined separately. Adulterants which the grease maker must also contend with are cheap fats such as garbage grease, bone fats, margarine refuse, cottonseed stearin, and sometimes distilled wool fat. Special technic must be employed for the detection of these materials, and the reader is referred to books dealing specifically with the analysis of fats and fatty oils for this information.[8]

Other uses of tallow than for grease making are: manufacture of candles, compounding lubricating oils, tanning and dressing leather, engraving and lithography and as food. The principal grades in descending order of merit are: "edible," "prime packers," "fancy," "prime renderers," "Prime No. 1," "Prime No. 2," and "B Country." Tallow is quoted and sold for delivery in railroad tank cars equipped with steam coils for heating, and in barrels (375 pounds) and tierces (50 pounds).

Properties of Beef and Mutton Tallow. The following ranges in properties apply generally to rendered beef and mutton tallows:

	Beef Tallow	Mutton Tallow
Specific gravity	$0.860–0.895 \frac{100° C.}{15.5}$	$0.858 \frac{100° C.}{15.5}$
Solidifying temperature ° C.	31–42.2 (87.8–108.0° F.)	44 (111.2° F.)
Acid value	0.10–5.00	0.20–5.00
Saponification value	193–198	195–196
Iodine value	35.4–46.3	48–61
Acetyl value	2.7–8.6	
Hehner value	96–96.5	
Fatty acids:		
Melting point ° C.	42.5–44	33.5–49
Titer test ° C.	37.9–46.2	40–48.5

[8] *See:* Lewkowitsch, J., "Chemical Technology and Analysis of Oils, Fats and Waxes," The Macmillan Company, New York.
Meerbott, P. B., and Stanislaus, I. V. S., "American Soap Makers Guide," The Norman W. Henley Publishing Co., New York.
Allen, A. H., "Commercial Organic Analysis," **II**, P. Blakiston's Son Co., Inc., Philadelphia.

Purchase Specifications for Beef Tallow for Grease Making. A Pacific Coast compounder has used the following purchase specifications for tallow used as soap stock in cup and fibre greases for many years and uniform material was nearly always supplied:

The tallow furnished must be of the grade known as Prime No. 2 (Crude Tallow) and shall be suitable for making greases containing a saponified base. It shall be clean, fresh and free from dirt and comply with the following requirements:

	Purchase Specifications	Range of Actual Tests
Specific gravity $\frac{15.5° \text{ C.}}{15.5° \text{ C.}}$	0.910–0.950	0.911–0.930
Ubbelohde melting point ° C.	36 Min.	40–42
Iodine value	50 Max.	36–49
Acid value (mg. KOH per gram fat)	10 Max.	4–10.0
Percent ash	.005 Max	.001–.004
Percent water	0.10 Max.	Trace
Solid impurities	None	None
Saponification value	193 Min.	193–199
A.S.T.M. color	No. 3 Max.	2–3
Odor	Not Offensive	....
Melting point of mixed fatty acids ° C.	40 Min.	41–44

Tallow Costs. Prime No. 1 Tallow on the New York market ranged from 4.75 to 6.75c per pound during 1936. At this time, tallow meeting the above requirements was obtainable at California points for 7.125c per pound. The relative values for different grades of tallow are (about) as follows, but these are subject to considerable variation on account of supply and demand: Edible 9¼ to 9½c per pound, Fancy 7¼ to 7½c, Prime Renderers' 6⅞ to 7c, Prime No. 1 6¼ to 6½c, B Country 4¾ to 5c.

Greases. The term grease is a generic one often applied to the lower grades of cattle, hog and sheep fats; particularly bone fats, "gut" greases and sometimes recovered garbage greases. The colors, odors and free fatty acids content vary greatly depending on the source and degree of refining. The following data may be of interest to grease makers in selecting low grade substitutes for Lard Oil and Tallow; however, the prospective users of the cheaper varieties of these greases should be warned that, in most cases, the odors which will exist in the finished greases will not ordinarily be acceptable to a discriminating trade, and that perfumes and deodorizers may be necessary in order to market lubricants made with them.

White Grease. Choice White grease sells for 7 to 9c per pound and is usually even more costly than No. 1 beef tallow. There are times, however, when demand lags, that it can be obtained for as little as 4c per pound. It is a satisfactory lubricating grease soap stock. Export Choice White grease usually sells at a slight premium over the domestic.

Pig's-Foot Grease. This stock can frequently be obtained for about 8½c per pound.

A. White Grease. This is a slightly lower grade than Choice White and sells for about 0.75c per pound less.

B. White Grease. Sells for ⅛ to ¼c per pound less than A. White Grease.

Yellow Grease. The price range for this product during 1935 was 5 to 6.75c per pound, and in 1936 has ranged from 4½ to 5½c per pound. As its name indicates it has a yellow color and its free fatty acid content will vary from 10 to 15 per cent.

Yellow Grease No. 2. A lower grade product, which will test from 16 to 20 per cent free fatty acids as oleic, and is obtainable at about ¼c per pound less than regular yellow grease. The odor is objectionable.

Brown Grease. A dark colored refuse fat having a free fatty acid content of 15 to 50 per cent and an unsatisfactory odor. Sells for 4½ to 5½c per pound.

Crackling Grease. This is similar in properties and value to yellow grease.

Bone Grease, Naphtha. May be obtained at about 5 to 6c per pound. This product is obtained by extracting animal bones with naphtha and then distilling off the naphtha. Melting points of bone grease vary from 68 to 83° F., and the mixed fatty acids melt at 86° F. The saponification number is 190 or higher, the iodine number 55.5 to 57.5. It usually contains dirt and moisture.

Stearins. The stearins are obtained by cold pressing various fats. By cold pressing tallows, oleostearin and tallow oil are obtained. Oleostearin is usually higher in price than the high grade tallows, but on account of the greater yield of grease possible with this stock, warrants greater attention from grease makers. It is, of course, obvious that if a grease plant attempts to stock too many fats, the cost of storage facilities and handling becomes an item which may force the adoption of a few staple fats obtainable in uniform quality from dependable sources. A few grease manufacturers, located near packing house centers, have, however, been able to capitalize effectively on the use of greases and stearins.

The following typical prices of stearins for the fall of 1935 indicate the range in values and the variety of soap stocks available: Prime Oleo Stearin, 11c to 12c per pound in barrels; Lard Stearin, 9½c to 10c; A. White Grease Stearin, 7c to 7½c; Yellow Grease Stearin 6c to 7c.

Herring Stearin and "Light Fish Foots" have been used in soap stock mixtures for lubricating greases.

Horse Fat. This fat has, for many years, been the choice of many grease makers; particularly, for cup grease manufacture. Its uncertain availability and variation in quality have ruled it out in the majority of grease plants in this country. Its typical characteristics are:

Specific gravity	.915–.933
Acid number	0.5–22
Saponification value	194–200
Iodine value	80–86
Melting point of mixed fatty acids ° C.	31–38

Some grease makers give preference to Horse Fat containing from 10 to 20 per cent of free fatty acids, this being desirable where the open kettle method of manufacture is in use as the fatty acids are more readily saponified.

Horse fat considered suitable for grease manufacture on the Pacific Coast had the following properties: Specific Gravity .915; Melting Point, 35° C.; Percent Water .25; A.S.T.M. Color 3½; Acid Number 15; Iodine Number 60; Saponification Value 196; Titer Test 36° C.

Hydrogenated Fats. Some grease makers apparently use hydrogenated fats with considerable success. Others consider that the yields, appearance of the grease, and texture are not equivalent to the use of normal mixtures of tallow, lard oil, or horse fat. The following tests were obtained

on a sample of "Sterogen": Specific Gravity .916; Ubbelohde Melting Point ° C. 39; Saponification Value 193; Acid Number 1.09; Percent Ash, .002; A.S.T.M. Color 2; Iodine Value 56.8 and Melting Point of the mixed fatty acids 37° C.

IX. Sperm Oils

Sperm oils have long been considered as being good lubricants, and their occasional use in lubricating greases is not so much with the intention of using them as a soap base, but on account of the increased oiliness and reduced coefficient of friction which they may produce in use. Ordinarily they yield from 30 to 40 per cent of unsaponifiable matter consisting of waxy alcohols soluble in alcohol.[9] Sperm oils differ from normal whale oils in that they consist of much larger amounts of these unsaponifiable waxes.

	Typical Tests
Specific gravity	.878–.884
Acid value	.3–.8
A.S.T.M. color	2–
Saponification value	120–143
Percent ash	.002
Iodine value	80–86
Pour point ° C.	13–18
Titer test ° C.	8–9

Sperm whale oil may be obtained as "Crude," "Natural" or "Bleached Winter" with regard to grade.

X. Animal Waxes

Beeswax. This material has been in use as an ingredient of various engine greases for many years. It is finding some application as a means for reducing the length of fiber in soda soap base greases intended for the lubrication of high speed ball and roller bearings. It may be obtained in a wide variety of grades: African, Chilean, Brazilian, Indian, U.S.P., Domestic, Crude, Refined, White, and Yellow. The cost of the imported wax varies from 21 to 30c per pound. The composition of beeswax is complex, investigators reporting that it contains about 85 per cent of myricin (or melissic palmitate $C_{46}H_{92}O_2$) and 12 per cent of cerotic acid, ($C_{25}H_{51}COOH$).

	Characteristic Tests
Specific gravity	.960–.969
Melting point ° C.	60–65
Acid number	16–21
Saponification value	82–96
Iodine value	8–11

Degras. In this country Degras is the term applied to Wool Grease, and Wool Fat. It is ordinarily dark brown in color and has a strong characteristic sheep-like odor. It is recovered from the solutions used to wash sheep's wool. Moellon Degras, recovered from the tanning of chamois is not often used in lubricants. Neutral hydrous wool wax, known as

[9] Toyama, Y., Analyst, III, 726.

lanolin, and the anhydrous wax, termed adeps lanae, are obtained by refining degras. Grades obtainable are: American, English, Neutral, and Raw. The domestic degras sells for 4½ to 7c per pound.

Suggested purchase specifications for Raw Degras suitable for nearly all needs in grease making, and oil compounding together with typical tests on this product are:

	Suggested Specifications	Range of Characteristics
Specific gravity $\frac{15.5°\text{ C.}}{15.5°\text{ C.}}$	.920–.950	.922–.945
Melting point ° C. Ubbelohde	35–40 Max.	32–38
Iodine value	20–40	15–36
Acid value	30–60	35–66
Percent ash	.200 Max.	.002–.150
Percent water	2 Max.	Trace–1.7
Solid impurities	None	....
Saponification value	100–150	85–142
Color	Brown	Brown
Titer test ° C.	40–50	41–55

Spermaceti Wax. This wax at 19 to 25c per pound is, ordinarily, too expensive for utilization in lubricating greases. However, it has been employed, in limited amounts, as a modifier in certain greases, namely, smooth soda soap base greases. It is obtained from the sperm whale and consists chiefly of cetyl palmitate ($C_{32}H_{64}O_2$). Its action in lubricating greases is probably due to the solvent properties of the cetyl alcohol ($C_{26}H_{53}OH$) set free on saponification.

	Range of Characteristics
Specific gravity $\frac{99°\text{ C.}}{15.5°\text{ C.}}$	.806–.945
Melting point ° C.	40–47
Acid value	.5–4.5
Saponification value	126–135

XI. Vegetable Waxes

The waxes given in the following table of properties are sometimes utilized in lubricants and will be referred to in the following chapters:

	Name of Wax			
	Carnauba	Montan	Jap	Candelilla
Specific gravity	.995–.999	....	.970–.980	.983
Melting point ° C.	84–86	80–90	40–54	67–68
Acid value	3–10	70–80	7–12	17
Saponification value	76–89	75	206–237	51–65
Iodine value	12–15	16	4.9–13	37
Color	Yellow-Green	Brown	Yellow	Yellow-Brown

XII. Fish Oils

On account of their objectionable odor fish oils are seldom used in lubricating greases although large quantities of these products in the raw condition or as deodorized oils are consumed in compounding car oils and various special machinery oils, apparently to increase the oiliness factor. The

term "fish oil" applies to mixtures of sardine, herring, menhaden, salmon and tuna oils. It will range in price from 4½ to 9c per pound. Purchase specifications and ranges in laboratory tests are given below:

	Suggested Purchase Specifications	Range of Characteristics
Specific gravity	.920–.940	.917–.939
Iodine value	130–195	102–192
Saponification value	185 Min.	170–200
Acid value	6 Max.	2.3–4.9
Percent water	0.5 Max.	....
A.S.T.M. color	3½ Max.	....
Odor	Not Offensive	....

Blown fish oils having specific gravities of .990-1.022, S.U. Viscosities of 600 to 1000 at 210° F., and a color of 6 A.S.T.M. are frequently used in compounding marine engine oils.

A mixture of fish and other oils termed "Compounders Oil" has recently been offered to grease makers, but its color was opaque brown-black, its ash value was .387 per cent, and its saponification value only 104.5.

Fatty Acids

At most grease plants it is not considered desirable to recover the glycerol split off from the fats or fatty oils during the saponification process. The chief reasons for this practice are, no doubt, the high initial cost of equipment for fat splitting, the special technique required for this process, the demands being made on grease makers for new products and the production of possibly a hundred or more varieties of greases in a few kettles and mixers, and the availability of oleic and stearic acids at reasonable cost direct from manufacturers who specialize in this sort of work. In some cases, which will be discussed later in connection with cup greases, it is desirable to leave the glycerol in the grease, and in others it is advisable to make use of fatty acids particularly where transparent, stable types of lime base greases are considered necessary, and the glycerol may be objectionable.

In the soap making industry, more than half of the total quantity of soap produced is made directly by the neutralization of fatty acids with caustic alkali or alkaline carbonates. The Twitchell process [10] makes use of fatty acid sulfonates, or a sulfo-fatty-aromatic acid, such as $C_6H_4(SO_3H)(C_{18}H_{35}O_2)$, as saponifiers for splitting whole fats. The sulfo fatty acids may be converted to the salts of calcium, barium, or aluminum, to form stable dry compounds which are used by dissolving them in sulfuric or hydrochloric acid, diluting with water and boiling in an open kettle in the desired proportions with the fat to be split. Petroff [11] developed the use of sulfonated naphthenic acids as catalysts for fat splitting and they are now extensively used.

A complete discussion of the production of stearic and oleic acids at the plant of the Emery Industries, Inc., Ivorydale, Ohio, has been prepared

[10] U. S. Patent 601,603 (1897).

[11] U. S. Patent 1,079,437.

by T. R. Olive.[12] The fats, tallows, and by-products from the Chicago district are first blended, and washed with acid to clean them. This is followed by a further treatment with 60° Bé. sulfuric acid and live steam. A charge of fats and an equivalent quantity of water are run into the copper lined splitting vats and about 2 per cent by weight of Twitchell's D.P. Saponifier together with .75 per cent of 60° Bé. sulfuric acid. The batch is boiled with live steam until about 90 per cent of free fatty acids are produced. The glycerin is separated, fresh sulfuric acid added, and the splitting process continued until about 97 per cent of the total acids available are produced. Separation of oleic and stearic acids is then carried out either by cold-pressing, or steam distillation.

Oleic Acid (Syn. Acid Elaidic, Oleinic Acid, Red Oil, Elaine), The chemical formula for oleic acid is $C_8H_{17}CHCH(CH_2)_7COOH$. Elaidic acid is a white, crystalline, stereoisomeric form of oleic acid, and may be produced by the action of nitrous acid on the red oil at 200° C. in the presence of sodium bisulphate. The commercial grades obtainable are: U.S.P.; Saponified; Distilled; and White. Either the saponified or distilled red oil is ordinarily obtainable at 8 to 12c per pound. Suggested purchase specifications and typical tests on saponified red oil suitable for grease making are:

	Suggested Purchase Specifications	Range of Characteristics
Specific gravity $\frac{15.5°\ C.}{15.5°\ C.}$	.890–.920	.891–.915
Pour point ° C.	20 Max.	0–40
Iodine value	80 Min.	63–96
Acid value	180 Min.	183–200
Percent ash	.05 Max.	.001–.21
Percent water	None	Trace–1.2
Solid impurities	None	....
A.S.T.M. color	5 Max.	4–8
Boiling point ° C.	....	286

An inferior and unsatisfactory oleic acid for grease making had the following properties: Specific Gravity .907; Color, black; Acid Value 181; Percent Ash .230; Iodine Value 83; Pour Point ° C. 16. The dark color of oleic acid is frequently due to the formation of iron soaps which form when the acid is stored for long periods of time in iron tanks. After storage in 50-gallon iron tanks for as long as a year the A.S.T.M. color should not increase more than from 5 to 8.

Stearic Acid (Syn.: Cetylacetic Acid, Stearinic Acid, Stearophanic Acid and Octodecylic-n Acid. Its chemical formula is $CH_3(CH_2)_{16}COOH$. The grades available in commercial quantities are: U.S.P.; Saponified; Distilled; Single, Double and Triple Pressed. As a grease making material it is usually shipped as cakes, 200 to 220 pounds to a burlap bag. The range in price for the double pressed, saponified stearic acid frequently used for preparing lubricating greases soap bases is 9 to 13c. Suggested purchase specifications and the range of Characteristics for Double Pressed stearic acid are:

[12] Olive, T. R., *Chem. Met. Eng.*, 721 (1929).

	Suggested Purchase Specifications	Characteristics
Specific gravity	....	.900
Acid value	195 Min.	200–208
Melting point ° C. Ubbelohde	52–60	52–58
Saponification value	....	215
Color	White	White-Yellow

Animal Fatty Acids. The mixed fatty acids derived from tallow and lard are frequently used in grease manufacture. They should contain not less than 95 per cent free fatty acids calculated as oleic, and should have a melting point of 95 to 110° F.

Wool Fat Fatty Acids.[13] These are obtained as a by-product in the manufacture of lanolin and adeps lanae. They are particularly desirable for reducing the length of fibre in soda soap base greases and as agents for stabilizing calcium soap base greases so that they may be heated to temperatures above 212° F. and on cooling return satisfactory grease conditions, rather than separate. In this latter utilization they are treated as normal soaps stocks in preparing the batch. Typical tests are:

Acid value	184.5
Saponification value	203.5
Percent unsaponifiable	1.5

Classification of Fats Based on Isolated Fatty Acids

The following classification of fats has been proposed by Sheely [13a] who has pointed out that mixed triglycerides are an important factor in considering the composition of soap stocks. In some cases his analytical data are not in agreement with the data given in the foregoing sections and this must be attributed to variations in sources of samples rather than difficulties in analysis.

Class I

	Per Cent		
	Coconut Oil	Palm Kernel Oil	Babassu Oil
Saturated Acids			
Caproic, $C_6H_{12}O_2$	Traces	Traces	0.1
Caprylic, $C_8H_{16}O_2$	9.5	3.0	6.5
Capric, $C_{10}H_{20}O_2$	4.5	3.0	2.7
Lauric, $C_{12}H_{24}O_2$	51.0	52.0	45.8
Myristic acid, $C_{14}H_{28}O_2$	18.5	15.0	19.9
Palmitic acid, $C_{16}H_{32}O_2$	7.5	7.5	6.9
Stearic acid, $C_{18}H_{36}O_2$	3.0	2.5	....
Unsaturated Acids			
Oleic acid, $C_{18}H_{34}O_2$	5.0	16.0	18.1
Linoleic acid, $C_{18}H_{32}O_2$	1.0	1.0	....
Titer	22–25	20– 25	23– 25
Iodine value	7.5–9.5	16– 20	12– 16
Saponification value	252–260	244–255	246–250

[13] See: Donath, *Chem. Ztg.*, **62**, (1899); also Rosengren, *Ibid.*, 382.

[13a] "Fatty Acids," paper presented before the 4th Annual Convention of the National Association of Lubricating Grease Manufacturers, Inc., Chicago, Oct. 12, 1936.

It will be noted that these oils are characterized by compositions of low molecular weight acids, lauric and myristic acids predominating, with smaller percentages of C_{16} and C_{18}, and also C_8 and C_{10} acids. The high saponification values, 244 to 260, and low iodine values, 7.5 to 20, the latter due to the low content of unsaturated acids, are characteristic of these oils.

Class II

	Per Cent		
	Palm Oil	Beef Tallow	Lard
Saturated Acids			
Myristic acid, $C_{14}H_{28}O_2$	1.0	2.0	....
Palmitic acid, $C_{16}H_{32}O_2$	35.5	32.5	24.6
Stearic acid, $C_{18}H_{36}O_2$	8.5	14.5	15.0
Unsaturated Acids			
Oleic acid, $C_{18}H_{34}O_2$	48.0	48.3	50.4
Linoleic acid, $C_{18}H_{32}O_2$	7.0	2.7	10.0
Titer	42– 46	41– 45	37– 39
Iodine value	50– 54	40– 45	53– 61
Saponification value	200–205	195–199	196–197

In this class are placed lard and beef tallow as well as palm oil, which is one of the few vegetable oils which simulates the animal fats. The composition of lard is roughly the same as the other fats from hogs. Note that palm oil contains a higher ratio of palmitic acid to stearic acid than tallow or lard, which incidentally accounts for its higher titer. The unsaturated acid content of all three fats is very similar, with the hog fat showing the highest linoleic acid value.

Class III

	Per Cent			
	Cottonseed Oil	Corn Oil	Peanut Oil	Soya Bean Oil
Saturated Acids				
Myristic acid, $C_{14}H_{28}O_2$	0.5	....	....	....
Palmitic acid, $C_{16}H_{32}O_2$	21.9	7.7	6.3	6.8
Stearic acid, $C_{18}H_{36}O_2$	1.9	3.5	4.9	4.4
Arachidic acid, $C_{20}H_{40}O_2$	0.1	0.4	3.3	.7
Lignoceric acid, $C_{24}H_{48}O_2$	....	0.2	2.6	.1
Unsaturated Acids				
Oleic acid, $C_{18}H_{34}O_2$	30.6	45.4	60.6	33.4
Linoleic acid, $C_{18}H_{32}O_2$	44.9	40.9	21.6	51.5
Linolenic acid, $C_{18}H_{30}O_2$	....	....	....	2.3
Titer	32– 36	13– 19	28– 30	20– 23
Iodine value	105–115	115–125	90– 95	131–136
Saponification value	191–195	189–193	187–188.5	192–194

In this class are included the most important so-called semi-drying vegetable oils, cottonseed, corn, peanut and soya bean. Note that they consist largely of the unsaturated acids, oleic and linoleic, with smaller percentages of palmitic, stearic, myristic, arachidic and lignoceric acids.

Class IV

	Per Cent— Menhaden Oil	Whale Oil
Saturated Acids		
Myristic acid, $C_{14}H_{28}O_2$	5.9	8.0
Palmitic acid, $C_{16}H_{32}O_2$	16.3	12.1
Stearic acid, $C_{18}H_{36}O_2$	0.6	2.3
Unsaturated Acids		
Myristoleic acid, $C_{14}H_{26}O_2$	....	1.5
Palmitoleic acid, $C_{16}H_{30}O_2$	15.5	15.0
Oleic acid, $C_{18}H_{34}O_2$	....	33.4
Linoleic acid, $C_{18}H_{32}O_2$	29.6	9.0
Linolenic acid, $C_{18}H_{30}O_2$	....	Traces
C_{20} acids, $C_{20}H_{40} - XO_2$	19.0	8.2
C_{22} acids, $C_{22}H_{44} - YO_2$	11.7	10.5
Titer	....	22–25
Iodine value	140–180	105–135
Saponification value	189–193	192–196

Class IV typifies the raw fish oils which are unique in that they contain relatively high percentages of high molecular weight unsaturated acids, not all identified as yet but known to comprise carbon chains having 20 or more carbon atoms. These oils, when hardened by the hydrogenation process, yield very hard products which have found many applications in the lubricating grease field as well as in soap making.

Rosin

Two types of rosin are available for grease manufacture: Wood Rosin (Colophony) obtained by extraction of the wood of the long leaf pine with solvents, and Gum Rosin (Colophony, Yellow Resin, Abietic Anhydride, Resina) produced as a residue from the distillation of crude turpentine. The various grades, colors, and price ranges are given in the table below:

	Color	N. Y. Price Range Cents per pound; 280 pound bbls Wood Rosin	Gum Rosin
A	Black Common	....	....
B	Common Strain	....	4.45–6.00
C	Strain	....	....
D	Good Strain	....	4.95–6.25
E	No. 2	....	5.15–6.65
F	Good No. 2	(FF) 4.05–6.35 Wrks.	5.20–6.90
G	Low No. 1		5.25–6.90
H	No. 1	....	5.25–6.90
I	Good No. 1	4.30–7.00	5.25–6.90
K	Low Pale	....	5.27–6.90
M	Pale	4.55–7.25	5.35–6.90
N	Extra Pale	4.95–7.75	5.70–7.10
W G	Window Glass	....	5.85–7.25
W W	Water White	....	5.90–7.75

Any of the rosins from grades F through N will be found suitable for blending with fatty soap stocks in the production of smooth soda soap greases. Rosin consists of three isomeric forms of abietic acid ($C_{20}H_{30}O_2$) 81 per cent, "resenes" about 10 per cent, essential oils and bitter substances $\frac{1}{2}$ per cent and impurities $\frac{1}{2}$ per cent. The sodium soaps when present in

lubricating greases in appreciable amounts, being quite hydroscopic tend to take up moisture from the air. Their sticky, gummy characteristics eliminate them from fine lubricants unless these properties are desired or they are needed to reduce the length of fibre.

Depending on the age, source, and grade, American lump rosins, will have characteristics according to the tabulation below:

	Range of Characteristics
Specific gravity	1.06–1.09
Melting point ° C.	100–140
Iodine value	55–184 (122 normal)
Acid value	131–182 (150–152 normal)
Percent ash	.008
Saponification number	170–180
Color	N–F
Percent solid impurities	.02–.10

Rosin Oils

Rosin oils are the distillates produced from various grades of rosin and are particularly valuable in sett greases or axle greases. A detailed discussion of rosin oils will be found in the chapter on Sett Greases. Grades obtainable are First, Second, and Third Run; "Kidney" and "Bloom." Kidney rosin oil of the following specifications has been found suitable for the manufacture of a line axle and sett greases:

	Suggested Purchase Specifications	Range of Characteristics
Specific gravity $\frac{15.5° \text{ C.}}{15.5° \text{ C.}}$	.990–1.030	1.008–1.022
Pour point ° C.	10–30	7.2–21
Iodine value	80–110	68.0–99.7
Acid value	75 Min.	80.0–85.8
Percent ash	0.050 Max.	.001–.038
Percent water	0.50 Max.	.40–.65
Solid impurities	None	....
Saponification value	75 Min.	81.3–95.1
S.U.V. at 210° F.	....	48–1187

Bloom rosin oils have acid numbers ranging from about 36 to 60 as compared with an extreme range of 65 to 95 for the Kidney grades.

Naphthenic Acids

These petroleum acids, used in grease making to obtain smooth soda soap base lubricants, are best derived from Gulf Coast or California gas oils of about 31° A.P.I. gravity. They can also be produced from lubricating oil distillates, but their recovery is more difficult. Fractional precipitation of the soaps by partial acid treatment, partial lye treatment of the gas oils while in the agitator, extraction of the oily material from sodium naphthenate solutions with solvents and finally distillation of the naphthenic acids under vacuum are some of the methods employed in producing naphthenic acids of high acid value (270 to 315 mg. KOH/gram).

Naphthenic acids of the following grades are available for grease making:

	Grade A	Grade B
A.P.I. gravity	11.5	10.7
Specific gravity	.9895	.9951
A.S.T.M. color	8	8
Flash ° F., O.C.	240	275
S.U.V. at 100° F.	58	800
Percent sulphur	.16	.79
Percent water	1.8	.5
Neutralization value	303	167
Percent ash	.333	.065

The prices of naphthenic acids vary from 9 to 12c per pound.

PERFUMING SUBSTANCES

In some grease plants there is a tendency to eliminate the use of perfumes as they are considered an indication of cheap soap making materials and petroleum products. It is true that the use of Oil of Mirbane, a standard odorant of the past, has been discontinued in most grease plants, and in its place essential oils and synthetic odorants have come to occupy a place of importance.

There are several reasons why the use of perfuming substances in lubricating greases is of value. The most important from a manufacturing standpoint is that the objectionable odors of such ingredients as tars and unrefined lubricating oils used in inexpensive greases are masked or altered. The addition of perfuming substances to greases masks the true odor of the fatty ingredients, whether of high or low grade, thus making it very difficult and in some cases impossible for even experienced analytical chemists to identify the fat used. The chief value of the added odors, to a customer, is that particular greases may be easily identified, and the user will know that he is obtaining the same product.

Certain lubricating greases, particularly those made in fire-heated kettles, at high temperatures, develop characteristic odors, which are valuable in the same sense as has been indicated above. In some cases the odor is characteristic of the process used, and it is impossible to duplicate such an odor by the addition of known synthetic substances. A few of the most important perfuming substances will now be described.

Nitrobenzene (*Oil of Mirbane*) $C_6H_5NO_2$. This compound is most generally used on account of its cheapness and strong odor, which is that of bitter almonds or benzaldehyde. It is prepared by the action of a mixture of nitric and sulfuric acids on benzene at a temperature of about 104° F. Its boiling point is 402.8° F.; the specific gravity is 1.205 (24.668° Bé.); refractive index at 68° F. is 1.550. Oil of mirbane is insoluble in water, soluble in alcohol, ether, fatty oils and benzene. Nitrobenzene is sometimes adulterated with alcohol. Great care must be taken in storing nitrobenzene, as it is highly inflammable. Great care should be exercised in using it, as twenty drops taken internally have been known to cause death; even the vapors when breathed for short periods of time cause poison symptoms.

Workmen should be cautioned not to allow nitrobenzene to come in contact with the skin.

Sassafras Oil. This material is used to a limited extent on account of its strong odor and low price. It is obtained by the distillation of the roots, and particularly the bark of the roots, of the *Sassafras officinale,* which is a native of North America. The refined oil has the following characteristics: Specific gravity at 60° F., 1.068 to 1.082 (9.232 to 10.865 Bé.); optical rotation + 3° to + 4°; refractive index 1.528 to 1.531; acid value, 0; ester value, 1 to 2. Sassafras oil is soluble in two volumes of 90 per cent alcohol. The composition of the oil has been reported as: Safrol, 80 per cent; pinene and phellandrene, 10 per cent; d-camphor, 7 per cent; cadinene, 2 to 5 per cent; eugenol, 0.5 per cent.

Camphor Oil. Has a characteristic odor and is sometimes used in lubricating oils and greases. It is a by-product obtained in the distillation of crude camphor. Chemically it is a mixture consisting of camphor in solution ($C_{10}H_{16}O$ or C_9H_6CO), pinene, cineol, and phellandrene. Price range 18 to 23c per pound.

Specific gravity	.870–1.040
Ref. index	1.465–1.481 at 20° C.

Citronella Oil. Is obtained from the grass *Andrapogon nardus.* It consists of a mixture of geraniol, citronellal ($C_{10}H_{18}O$) and methyl eugenol. Its specific gravity is .885 to .900, and refractive index 1.4811 to 1.4830 at 20° C. Its odor is somewhat similar to that of the Indian Lemon Grass Oils in place of which citronnella oil is sometimes used. The price range is 22 to 30c per pound.

Cedarleaf Oil. This natural oil has a savine-like odor, specific gravity .883 to .888, boiling point 173 to 180° C., and saponification value 8 to 14. Cedarleaf oil at 52 to 70c per pound is considered too costly for use in normal greases, although it has been used in some special products sold at a high price in limited quantities.

Lemongrass Oil. This scent at 50 to 70c per pound is infrequently used in lubricating greases. Its color may be as dark as 5 A.S.T.M. and its specific gravity .9036.

Pine Oil. This material has a characteristic pine odor, and is frequently added to wire rope lubricants, gear greases, and crankpin greases. It is obtained by either dry or steam distillation, or solvent extraction of pine wood. Specific gravity is .925 to .942, boiling point 185 to 225° C., and refractive index is 1.4820 to 1.4870 at 20° C.

Pine Tar. Is a black semi-solid material having the characteristic odor of terebene and dipentene. It is obtained by the destructive distillation of *Pinus palustris* and its specific gravity may vary from .950 to 1.150. It is also known as "Stockholm Tar."

Oil of Birch Tar. This material has a peculiar smokey odor and is sometimes used in cable greases, curve greases and axle greases.

Wintergreen Oil. (See methyl-salicylate)

Organic Esters. Many of the alkyl salts of organic and inorganic acids, known as esters, are quite suitable as odorants for lubricating greases.

Ethyl Acetate ($CH_3COOC_2H_5$). It has a boiling point of 77.15° C. and its specific gravity is 0.9003. This acetate is soluble in oils, water, alcohol and ether. It has a characteristic fragrant odor. The 85 per cent ester may be obtained at prices ranging from 6 to 10c per pound, the anhydrous ester will cost about 1½c per pound more, and either may be employed in greases. Ethyl acetate is the base for the so-called essence of pears.

Ethyl Benzoate ($C_6H_5COOC_2H_5$). This material has an aromatic odor and is frequently employed in perfumery.

Ethyl Butyrate ($CH_3(CH_2)_2COOC_2H_5$). This ester, also known as essence of pineapples, is a colorless liquid boiling at 120° C. and has a specific gravity of 0.8788.

Ethyl Nitrate ($C_2H_5NO_3$). This ester, also known as nitric ether, has a boiling point of 88.7° C. and its specific gravity is 1.105 to 1.116.

Amyl Acetate ($CH_3COOC_5H_{14}$). This oily liquid, having a specific gravity of 0.866 and boiling at 138 to 148° C. has a fruity, pear-like odor and is frequently used in lubricants and greases, particularly where it is necessary to mask the objectionable odors of pressure still residues, fuel oils, reclaimed crankcase oils and asphaltic residues in general. Amyl acetate is also known as banana oil.

Amyl Butyrate, Amyl Formate, Amyl Nitrite, Amyl Salicylate, and Amyl Valerate (Apple Oil). These all have definite odors which are of some value in masking objectionable odors in lubricants or imparting to them a means of identification due to odor.

Benzyl Acetate, Benzyl Benzoate, Benzyl Chloride, Butyl Acetate, Butyl Formate, Ethyl Salicylate, Geranyl Acetate, Glyceryl Acetate, Methyl Acetate, Methyl Salicylate, and Ethyl Valerate. They have all been given consideration as perfumes for lubricating greases and offer possibilities depending on cost of the ester and the nature of the grease to be scented.

E. Vellner [14] has suggested the use of various combinations of scents and colors for specific identification of lubricating greases. Two ounces of scent and the necessary dye are added to each hundred pounds of grease, Scents specified are oil of cloves, wintergreen, peppermint, terpineol, or lavender, and ionone, or violets; colors specified are yellow, green, red, blue, and orange aniline dyes.

DYES AND COLORING SUBSTANCES

Throughout the history of grease making it has been more or less necessary to add dyes and coloring materials to lubricating greases in order to meet the demands of particular customers. In some plants, the addition

[14] British Patent 359,590.

of very small amounts of yellow or orange dyes is practiced to produce greases of uniform color from soaps and oils subject to slight variations in color. An engineer has proposed that special automotive greases shall be colored bright hues in order that they may be readily identified and mistakes in applying them to the various parts of a car prevented.

Lubricating Oil Orange. This oil soluble coal tar dye is obtainable at about 50c per pound and is used extensively in dyeing greases. From .003 to .008 per cent by weight will ordinarily suffice.

Lubricating Oil Red (C). A bright red oil soluble dye which will cost from 65 to 95c per pound. Several greases are marketed in this country colored with red dye.

Oil Soluble Light Yellow. This dye at $2.75 per pound is sometimes used.

Sudan Yellow. This dye at $1.25 per pound is more frequently used.

Pyloil Special Violet. This is a brilliant violet dye for specialty products and will cost about $3.85 per pound.

Oil Soluble Blue Green. This dye at $7.75 per pound is infrequently used in lubricating greases.

Fluorescent Green HW. This is a green bloom dye which, when added to greases made with pale colored oils, produces the appearance of Pennsylvania Bright Stocks. A similar color may be produced by extracting the coloring matter from oil gas tar and using it as a dye in a manner similar to that commonly employed in dyeing lubricating oils.

Oil Soluble Blue. This dye is used in a few specialty greases.

INORGANIC FILLERS

Graphite. While graphite may be considered more of a lubricant than most inorganic fillers it will be discussed under this heading as a matter of convenience. Acheson colloidal graphite known as "Oildag" has been recommended for special graphited greases, joint compounds, and wire drawing compounds. Concentrated Oildag contains about 10 per cent by weight of the colloidal material, semi-concentrated 2 per cent and R.F.U. (ready-for-use) Oildag still less. The cost of these products is quite high and most grease manufacturers adopt some form of fine flake or foliated graphite rather than the true colloidal (dispersed) material.

In general, it may be considered that lubricating greases are improved by the addition of graphite, provided a small amount of the best grades of finely divided flake or amorphous graphite is used. Greases containing low viscosity oils are particularly improved. Exceptions to this generalization are that bearings with highly polished surfaces and minute clearances (high grade ball and roller bearings) should not be lubricated with normal graphite greases, and if graphite is considered necessary then only the colloidal grade should be employed. Typical graphite greases are: Graphite Cup Grease, Graphite Waterproof Grease, Graphite Axle Grease, Graphite Pipe

Joint Compound, Graphite-Petrolatum Compound, Brake Clyinder Lubricant, Triple Valve Grease, Center Plate Grease, Graphite Worm and Gear Grease, and Gold Dredger Grease.

Powdered Graphite. This material for use in ordinary grease manufacture may have an ash content of 14 to 16 per cent. Graphite of lower ash value is obtainable but the cost is greater. Not more than 12 per cent of this grade of graphite should be retained on a 200 mesh (per inch) screen. Acheson Graphite No. 39, obtainable at about 10c per pound, and in some cases having an ash content of 0.14 to .30, is quite acceptable. Graphite for use in greases should contain not more than a trace of grit. Graphites containing from 20 to 35 per cent of ash and considerable grit have been offered to grease makers but should be rejected as being dangerous.

Grades of graphite normally obtainable on the market are: Crude, Powdered, Flake, Domestic, East Indian, Madagascar and Ceylon.

Powdered Mica (Muscovite, Laminated Talc). This material is frequently found as an ingredient of axle greases. It is a native hydrous silicate having a specific gravity of 2.9. It decomposes on heating. The grade acceptable for grease making should pass a sieve test in which not more than 5 per cent by weight shall remain on a 40 mesh (per inch) screen. More finely divided grades of 100 and 140 mesh are commercially available at costs ranging from 1.7 to 3.1c per pound. Other grades, 16, 24 and 30 mesh are available, but the 40, 60 and 80 mesh sizes are most commonly used in axle greases.

Asbestos Floats. This is the finest grade obtainable with regard to state of division. Asbestos is of course a compound silicate and aluminate of magnesium, calcium and iron. The Canadian grades consist of Chrysotile ($3MgO,2SiO_2,2H_2O$) and Crocidolite ($NaFe(SiO_3)_2$, $FeSiO)_3,H_2O$). This material is, of course, a mild abrasive and sometimes a lapping agent. It should, therefore, be recommended with caution as a grease ingredient. However, in some industrial services, where natural dirt and grit must be contended with, the floats properly compounded with oil and soaps form a seal which prevents the entry of the more abrasive material and actually lengthens the life of bearings. Where extraneous abasives cannot gain entry asbestos floats should not be employed.

Talc (Talcum, soapstone, Steatite.). Natural hydrous magnesium silicate, $Si_4O_{12}Mg_3H_2$ of the commercial grade known as "air floated" has been frequently used as a filler of lubricating greases, either alone or in combination with graphite or asbestos floats.

Zinc Oxide (ZnO). This is often used as an ingredient of lubricants. It is a white amorphous powder having a specific gravity of 5.47. French process, American process, Leaded, Green, Red or White Seal grades are suitable for greases.

Powdered Lead. This is now available on the market and has been utilized in greases.

Powdered Copper. This material has been offered to grease makers.

ORGANIC FILLERS

Animal hair, wool waste and wool yarn are often incorporated with soda and lime base greases to meet certain severe dust and dirt conditions and to prevent excessive leakage from bearing housings which are not tight.

Wool Waste. This should contain not less than 50 per cent of wool fibers, not more than 5 per cent of fatty or mineral oils, and not more than 1 per cent of dirt. The balance of the waste may consist of vegetable fibers; cotton, hemp, jute, etc.

Wool Yarn. This is usually incorporated with soda soap grease or lime-soda grease for the production of wool yarn journal box greases. The strands of wool yarn should be about 20 inches long and should consist of not less than 70 per cent by weight of wool fiber and the balance cotton fibre.

Wool Yarn and Horse Hair. A line of "wool-yarn elastic" greases is manufactured by some grease plants, the horse hair being thoroughly mixed with the wool yarn before combining with the grease. The horse hair produces a more resilient grease preventing a heavy sodden condition in the journal box.

Goat Hair. Used in the past to produce greases having greater cohesion.

Ground Cork. Very seldom found in modern gear greases, are the quantities of ground cork which were common during the earlier years in the development of the automobile.

Sawdust. This material is very seldom found as an ingredient in modern greases.

Flake Naphthalene. This organic has been used in some greases as a deodorant and filler.

LIME

Lime is also known as Quick Lime, Calcium Oxide, Burnt Lime, and Calx. Its molecular weight is 56.07. Lime must be hydrated with water to form $Ca(OH)_2$ before it can be effectively utilized for the saponification of fats to produce soap bases for greases. While the older grease making technique included this step, the present general availability of satisfactory hydrated lime in commercial quantities has eliminated the necessity of "slaking" lime.

HYDRATED LIME

(Calcium Hydroxide, Slaked Lime. Formula: $Ca(OH)_2$; Mol. Wt. 74.0854)

The hydrated lime used in the manufacture of lubricating greases should be of the highest quality and should be uniform as to its available lime content. Variation in the strength of lime used may cause considerable variation in the consistency of the resulting cup greases. There are cases on record where the use of lime containing considerable quantities of magnesium have caused very irregular results. The silica content should be as low as possible, because cup greases are often used on bearings run-

ning with very small clearances and comparatively large quantities of silica in the lubricant might cause excessive wear. In most cases, for the lubrication of loose running bearings the amount of silica ordinarily found in hydrated lime is not objectionable. The chemical analysis of a lime suitable for grease manufacture follows:

Calcium hydroxide	98.47
Iron oxide and alumina	.20
Magnesia	.71
Silica	.17
Fineness, 200 mesh	99.95

Some large producers of grease prefer to buy a good grade of quick lime and hydrate this material themselves with a known quantity of water. Very often the lime used in the manufacture of grease is not thoroughly slaked and again it has an excess of water in it after slaking. Either of these conditions will vary the quality of the soap in which the lime is used as well as the quality of the finished grease. Variations in the water content of the hydrated lime used over a period of several months at one grease plant was as follows:

	Water Per cent
Hydrated lime No. 1	23.51
Hydrated lime No. 2	27.68
Hydrated lime No. 3	28.00
Hydrated lime No. 4	27.77

In this plant the quicklime after hydration was ground in a Raymond Pulverizer and sifted through a "Hummer" vibrating screen of 100 mesh. No complaints have ever been made on grit in the grease made at this plant.

The best plan for the control of the strength of lime used is to submit samples from each shipment of lime to the laboratory for determination of the available lime content, by the American Society of Testing Materials method, and make a correction for variations in strength in the quantity of the lime charged to the grease kettle. Causes for variation in the available lime content of powdered hydrated lime due to other reasons than variations in the moisture content are:

Irregular burning of the limestone, resulting in uneven decomposition of the calcium carbonate.

Impurities in the limestone itself.

Physical changes brought about during slaking. This may be a case of molecular aggregation whereby a portion of the lime is rendered physically unavailable, or a molecular change brought about by local burning during hydration which might change some of the lime into a less soluble form.

The state of moisture and temperature conditions during hydration cause chemical reactions between the constituents in the lime which produce small amounts of insoluble products.

The examination of the results of tests on batches of cup grease in which various amounts of lime were used showed that within certain limits

(from 3 per cent deficiency to 3 per cent excess over the theoretical amount of lime required) the yield and quality of the finished grease were not appreciably affected. By increasing the quantity of water added, it was possible to obtain satisfactory grease when even greater excesses of lime were used.

In both the United States and England it is now possible to purchase high grade powdered hydrated lime in 100-pound paper bags which is entirely satisfactory for grease making. The use of this prepared hydrated lime eliminates chances of the grease making any error in hydrating quick lime and also the possibilities of irregular hydration and consequent variations in the available $Ca(OH)_2$ content. Garlick recommends a maximum silica content of 0.5 per cent and a maximum iron oxide and aluminum content of 0.2 per cent. He gives the analysis of a satisfactory grease making hydrate as follows:

	True Analysis	Water and CO_2 Free Lime
Calcium oxide	75.2	98.2
Magnesium oxide	.40	.52
Al_2O_3	.19	.25
Fe_2O_3	.04	.05
SiO_2	.46	.60
So_3	.26	.34
Co_2	.44	...
H_2O	23.0	...

The following analytical data on hydrated lime for grease making covers different incoming shipments at one grease plant for a period of ten years (1926 to 1936) and is of interest in showing the variations which may be expected in commercial supplies of the hydrate.

	Per cent by weight			Per cent by weight	
	Available Calcium Oxide	Calcium Carbonate		Available Calcium Oxide	Calcium Carbonate
	73.50	1.5	.3 Silica	72.20	1.0
	73.40	1.5		72.00	2.0
	73.36	1.4		72.00	1.0
.003 Silica	73.10	1.0		72.00	1.0
	73.08	1.0		72.00	1.5
	72.81	1.5		71.90	2.5
	72.80	3.0		71.80	2.0
	72.80	1.5		71.65	.5
	72.60	1.5		71.50	2.5
	72.52	3.5		71.26	3.0
	72.50	1.0		71.12	2.0
	72.40	2.0		71.10	2.0
	72.24	4.0		70.56	3.5
	72.24	1.0		68.88	..
	72.20	2.0		67.79	3.5
				67.60	1.5
				59.36	2.5

While it is not likely that variations in available CaO ranging from 70 to 74 per cent would introduce very serious difficulties a slightly more narrow specification is desirable to allow a sufficient margin of safety and to take into consideration testing variables. When manufacturing grease to low free alkali specifications the range must be quite narrow.

Suggested Purchase Specifications:

	Per cent by weight
Available CaO	71.5–73.5
Silica	.45 Max.
MgO	.75 Max.
Fineness, 200 mesh/inch	95 Min.

This grade of hydrated lime can ordinarily be obtained for about 1c per pound.

CAUSTIC SODA

This material also known as Soda, Sodium Hydroxide, Lye, Sodium Hydrate, and White Caustic, has the chemical formula: NaOH; its molecular weight is 40.0047; melting point 318° C; and density 2.13. It may be purchased in Stick form, as a solution of about 45° Bé., Technical Solid, Ground, Flaked, and Granulated. It is usually obtainable and sold on the basis of its purity in terms of percent Na_2O; 60 per cent and 76 per cent Na_2O being the usual grades in the United States. Percentages of NaOH corresponding with percentages of Na_2O are:

Percent Na_2O	Percent NaOH
77½	100.00
77	99.35
76	98.06
74	95.48
72	92.90
70	90.32
60	77.42

If the production of greases warrants, it is most economical to purchase strong solutions of caustic soda in tank car lots; provided, of course, storage facilities can be arranged, and the grease plant is sufficiently near a dependable source, so that the freight rate is not prohibitive. Otherwise, it is best to buy drums of solid caustic and put it into solution with the aid of steam, or the granular, or flake caustic, which may be dissolved directly in water.

The following table giving the specific gravity at 15.5° C. (60° F.) and corresponding degrees Baumé and percentages of NaOH will be found useful in computing grease formulae and kettle charges:

Table 5—Sodium Hydroxide Solutions
(Caustic Soda)

By Lunge

Specific Gravity 60°-60° F.	Degrees Bé.	Degrees Twaddle*	Percent NaOH	Grams NaOH Percent	Specific Gravity 60°-60° F.	Degrees Bé.	Degrees Twaddle*	Percent NaOH	Grams NaOH Per Liter
1.007	1.0	1.4	0.61	6	1.220	26.1	44.0	19.58	239
1.014	2.0	2.8	1.20	12	1.231	27.2	46.2	20.59	253
1.022	3.1	4.4	2.00	21	1.241	28.2	48.2	21.42	266
1.029	4.1	5.8	2.70	28	1.252	29.2	50.4	22.64	283
1.036	5.1	7.2	3.35	35	1.263	30.2	52.6	23.67	299
1.045	6.2	9.0	4.00	42	1.274	31.2	54.8	24.81	316
1.052	7.2	10.4	4.64	49	1.285	32.2	57.0	25.80	332
1.060	8.2	12.0	5.29	56	1.297	33.2	59.4	26.83	348
1.067	9.1	13.4	5.87	63	1.308	34.1	61.6	27.80	364
1.075	10.1	15.0	6.55	70	1.320	35.2	64.0	28.83	381
1.083	11.1	16.6	7.31	79	1.332	36.1	66.4	29.93	399
1.091	12.1	18.2	8.00	87	1.345	37.2	69.0	31.22	420
1.100	13.2	20.0	8.68	95	1.357	38.1	71.4	32.47	441
1.108	14.1	21.6	9.42	104	1.370	39.2	74.0	33.69	462
1.116	15.1	23.2	10.06	112	1.383	40.2	76.6	34.96	483
1.125	16.1	25.0	10.97	123	1.397	41.2	79.4	36.25	506
1.134	17.1	26.8	11.84	134	1.410	42.2	82.0	37.47	528
1.142	18.0	28.4	12.64	144	1.424	43.2	84.8	38.80	553
1.152	19.1	30.4	13.55	156	1.438	44.2	87.6	39.99	575
1.162	20.2	32.4	14.37	167	1.453	45.2	90.6	41.41	602
1.171	21.2	24.2	15.13	177	1.468	46.2	93.6	42.83	629
1.180	22.1	36.0	15.91	188	1.483	47.2	96.6	44.38	658
1.190	23.1	38.0	16.77	200	1.498	48.2	99.6	46.15	691
1.200	24.2	40.0	17.67	212	1.514	49.2	102.8	47.60	721
1.210	25.2	42.0	18.58	225	1.530	50.2	106.0	49.02	750

*Twaddle values are derived from specific gravity by the following formula:

$$\text{Twaddle} = (200 \times \text{Sp. Gr.}) - 200$$

$$\text{Specific Gravity} = \frac{\text{Tw.} + 200}{200}$$

Caustic soda solution having the following typical tests is purchased in tank cars by one grease manufacturer and utilized with success:

Gravity, ° Bé. at 60° F.	49.6
Specific gravity at 60° F.	1.5178
Percent Na_2O	36.82
Percent NaOH	47.56
Percent Na_2CO_3	0.18
Pounds per gallon, 60° F.	12.64

GREASE CONTAINERS

Shipping containers for lubricating greases have been utilized in a wide variety of forms varying from small collapsible metal tubes to steel drums of 100 gallons capacity. The following suggested specifications for grease cans, pails, buckets, and steel drums should be of interest:

Table 6—Cans and Pails (Specifications)

Name	Plunger Type Grease Can 1 lb.	5 lb.	Lithographed Grease Pails 10 lb.	25 lb.	Bucket 25 lb.	50 lb.
Diameter, outside	$3\frac{7}{16}$"	$5\frac{7}{16}$"	$7\frac{5}{16}$"	10"	$11\frac{1}{4}$"	$11\frac{1}{4}$"
Height, outside	$4\frac{17}{32}$"	$8\frac{13}{32}$"	$8\frac{1}{4}$"	$11\frac{1}{4}$"	$9\frac{1}{4}$"	$17\frac{1}{4}$"
Gage	32	30	33	$7\frac{1}{4}$ friction lid	27	24

Table 7—Steel Grease Drums (Specifications)

Capacity—Gallons	15	15	30	30	50	50
Covers						
Cast iron flat hex plug	2"		2"		2"	
Chipboard gasket	2"		2"		2"	
Chipboard gasket	9"	12"	9"	15"	9"	15"
Detailed Data						
Inside diameter	$15\frac{3}{8}$"	$15\frac{3}{8}$"	$18\frac{1}{4}$"	$18\frac{1}{4}$"	$22\frac{13}{32}$"	$22\frac{13}{32}$"
Inside length	$18\frac{7}{8}$"	$18\frac{7}{8}$"	$26\frac{13}{16}$"	$26\frac{13}{16}$"	$19\frac{1}{4}$"	$29\frac{1}{4}$"
Exact vol. U. S. gallons	15.17	15.02	30.49	30.14	50.53	50.18
Depth of chime	$\frac{3}{4}$"	$\frac{3}{4}$"	$\frac{3}{4}$"	$\frac{1}{4}$"	$\frac{3}{4}$"	$\frac{3}{4}$"
Outside dia. of chime	$15\frac{3}{4}$"	$15\frac{3}{4}$"	$18\frac{5}{8}$"	$18\frac{5}{8}$"	$22\frac{13}{16}$"	$22\frac{13}{16}$"
Height of swedge	$\frac{5}{8}$"	$\frac{5}{8}$"	$\frac{5}{8}$"	$\frac{5}{8}$"	$\frac{7}{8}$"	$\frac{7}{8}$"
Center spacing of swedge	9"	9"	9"	9"	13"	13"
Outside dia. of swedge	$16\frac{11}{16}$"	$16\frac{11}{16}$"	$19\frac{3}{16}$"	$19\frac{3}{16}$"	$24\frac{1}{4}$"	$24\frac{1}{4}$"
Overall length of drum	$20\frac{1}{2}$"	$20\frac{1}{2}$"	$28\frac{3}{8}$"	$28\frac{3}{8}$"	$30\frac{3}{8}$"	$30\frac{3}{8}$"
Average weight with plug	20 lbs.	20 lbs.	32 lbs.	32 lbs.	49 lbs.	49 lbs.
General Data						
Nominal capacity U. S. gallons	15	15	30	30	50	50
Nominal capacity in lbs.	105	105	215	215	420	420
U. S. gage thickness of mat'l.	19	19	19	19	18	18
Size small head opening, "	2		2		2	
Size large head opening, "	9	12	9	15	9	15

Chapter III

Theory of Lubricating Grease Manufacture

Before discussing grease formulae in detail, and describing actual manufacturing procedure and equipment the basic theoretical principles underlying the processes for manufacturing calcium, sodium and aluminum base greases will be given. As was indicated in Chapter I, regarding the colloidal nature of lubricating greases, some of the theory relating to greases has not been fully established and in some cases is contradictory. However, greases are now being manufactured on a scientific basis and the manufacturer who has a complete knowledge of the existing theoretical principles will frequently be able to put into practice improvements and economies which would never occur to the old fashioned rule-of-thumb grease maker.

Disregarding those lubricants prepared by mixing together petroleum products with inorganic or organic materials other than soaps to produce substances having "yield value" as distinguished from liquids, we may, in general, regard lubricating greases as combinations of petroleum products and salts of fatty acids which are suitable for certain types of lubrication. Consequently, the first step in manufacturing lubricating grease is the preparation of the soap base, this being an appropriate salt of one or more fatty acids. The soaps used in grease making may be defined as the salts of any monobasic aliphatic acid containing ten or more carbon atoms.

SODIUM SOAPS

Chemists contend that when a fat or fatty oil is boiled together with a solution of sodium hydroxide the chemical change which takes place, termed saponification, occurs in three steps as indicated by the following equations:

1. $$\begin{array}{l} CH_2O(C_{18}H_{35}O) \\ | \\ CHO(C_{18}H_{35}O) \\ | \\ CH_2O(C_{18}H_{35}O) \\ \text{Tristearin (Tallow)} \end{array} + 3NaOH = \begin{array}{l} CH_2OH \\ | \\ CHO(C_{18}H_{35}O) \\ | \\ CH_2O(C_{18}H_{35}O) \\ \text{Distearin} \end{array} + \underset{\substack{\text{Sodium Stearate} \\ \textbf{(Soap)}}}{C_{18}H_{35}OONa} + 2NaOH$$

2. $$\begin{array}{l} CH_2OH \\ | \\ CHO(C_{18}H_{35}O) \\ | \\ CH_2O(C_{18}H_{35}O) \\ \text{Distearin} \end{array} + 2NaOH = \begin{array}{l} CH_2OH \\ | \\ CHOH \\ | \\ CH_2O(C_{18}H_{35}O) \\ \text{Mono-stearin} \end{array} + \underset{\substack{\text{Sodium} \\ \text{Stearate}}}{C_{18}H_{35}OONa} + NaOH$$

3. $$\begin{array}{l} CH_2OH \\ | \\ CHOH \\ | \\ CH_2O(C_{18}H_{35}O) \\ \text{Mono-stearin} \end{array} + NaOH = \begin{array}{l} CH_2OH \\ | \\ CHOH \\ | \\ CH_2OH \\ \text{Glycerol} \end{array} + \underset{\substack{\text{Sodium} \\ \text{Stearate}}}{C_{18}H_{35}OONa}$$

The condensed equation representing the saponification process is as follows, in which R indicates any of the fatty acid radicles having ten or more carbon atoms:

$$\underset{\text{Fat}}{C_3H_5(O\,.\,CO\,.\,R)_3} + \underset{\substack{\text{Caustic}\\ \text{soda}}}{3NaOH} = \underset{\text{Glycerol}}{C_3H_5(OH)_3} + \underset{\text{Soap}}{3R\,.\,COONa}$$

Another factor, which must be taken into consideration, is the structure of the glycerides which make up the fat. In most discussions of saponification, the fats are considered as being normal, simple triglycerides, that is, each glyceryl radical is combined with three oleic acid groups, three stearic acid groups, etc. This, however, is not strictly true, and many fats owe their distinguishing characteristics to quantities of mixed glycerides which are present. For instance, tallow will contain appreciable amounts of a complex glyceride in which the C_3H_5 group is linked to two stearic acid radicals and one oleic acid radical. Many such combinations are possible; for example, two oleic groups with one stearic; one each of oleic, stearic and palmitic; two stearic and one palmitic acid radical; two palmitic and one oleic acid radical, etc. It will be noted in the following chapters that fatty acids are frequently suggested as soap stock in the manufacture of grease and it may be pointed out that reasons other than the quality and service characteristics imparted by their use are: better control of the soap stock composition; a higher yield of soap than from triglycerides; ease and completeness of saponification.

In starting a batch of soda base or fibre grease, the grease maker must decide what relative porportions of fat, mineral oil, solid caustic soda and water will be charged. The concentration of these items will have a definite effect on the character of the soap base produced and the equilibria attained during the cooking process. Either an open kettle or an autoclave may be utilized, but consideration must be given to the selection of the most appropriate porportions of these ingredients. For the sake of convenience, we shall term the kettle contents at the start of the process, before the steam has been turned on, the "charge." The composition of the charge may vary greatly but will normally fall within the limits given in the following table:

	Per Cent	
Mineral oil	0 to 82	(May also consist of reworked or slop grease)
Fat (tallow)	14 to 78	
Water	2 to 53	(Seldom added as such, but as a part of the caustic soda solution which may contain from 10 to 50 per cent of NaOH)
NaOH	2 to 13	

Kettle charges, for either open cooking or the autoclave, in use by two grease plants are:

Per Cent	Plant 1	Plant 2
Mineral oil	42	26
Tallow	42	49
Water	10	18
NaOH	6	7

The laws of mass action will govern the rate of saponification in general, but the velocity will be influenced, to some extent, by impurities such as fatty acids, and the degree of emulsification prior to cooking, and particularly by the temperature and pressure conditions.

MASS ACTION THEORY

According to mass action theory the velocity of the saponification process is quite rapid at first, after which there is a gradual slowing up until equilibrium has been established. Lewkowitsch found that for open boiling with a theoretical amount of caustic soda, only 94 per cent of the tallow was saponifiable. With large excesses of sodium hydroxide, "salting out" occurs and the reaction velocity may be retarded; also, in the presence of large quantities of glycerol, a reversible reaction may take place in which the triglycerides may be reformed and NaOH produced from the soap. If it could be assumed that the saponification reaction proceeded to completion as follows according to G. Martin:[1]

$$\underset{\text{Tristearin}}{C_3H_5(OC_{17}H_{35}CO)_3} + 3\underset{\substack{\text{Sodium}\\\text{Hydroxide}}}{NaOH} \longrightarrow \underset{\text{Glycerol}}{C_3H_5(OH)_3} + 3\underset{\substack{\text{Sodium}\\\text{Stearate}}}{C_{17}H_{35}COONa}$$

where we represent the molar concentrations (gram-molecular weights per liter) as:

a—Original concentration of fat
b—Original concentration of caustic soda
x—The quantity of fat saponified after time "t"

then the reaction velocity, other considerations being equal, may be expressed as

$$\frac{dx}{dt} = k(a-x)(b-x)^3$$

in which k is the reaction constant.

It is probable that the reaction is not quadrimolecular (three molecules of NaOH and one molecule of tristearin) but is more likely of bimolecular type, in which one molecule of NaOH reacts with one molecule of tristearin, and proceeds in three steps as previously discussed. Since the analysis of mono and distearin is difficult, and the foregoing equation only roughly indicates the reaction velocity even though quadrimolecular, for practical purposes it may suffice to consider that the reaction rate for one of the steps in the saponification reaction is proportional to the average rate of the series of three reactions. The bimolecular reaction rate may be expressed by the equation:

$$\frac{dx}{dt} = k(a-b)(b-x)$$

The reaction constant, k, is then:

$$\text{(I)} \qquad k = \frac{1}{(a-b)t}.\log\frac{(a-x)b}{(b-x)a}$$

[1] "The Modern Soap and Detergent Industry," I., (1924).

Or, where b is greater than a and a is greater than x:

$$(II) \qquad k = \frac{1}{(b-a)t} \cdot \log \frac{(b-x)a}{(a-x)b}$$

The following data applies to open and closed kettle saponification in the presence of mineral oil as in grease making:

	Gram mols. per liter	
	Open Kettle	Autoclave
a, Molecular concentration of fat in charge	.425	.425
b, Molecular concentration of NaOH in charge	1.350	1.350
x, Quantity of fat saponified in time t (98% of a)	.416	.416
t, Time in hours	7	2

Substituting these values in Equation II we find that the reaction constant for open kettle saponification is 0.314, less than one half the computed value for the autoclave which is 0.786.

If the law of mass action applies to saponification in the presence of mineral oil, then the rate of soap formation will be proportional to the active masses of the fat and caustic soda present at any given time. In van't Hoff's kinetic derivation of the law of mass action it is assumed that the rate of chemical change is proportional to the number of collisions per unit of time between the molecules of the reacting substances, which in the case of saponification are fat and caustic soda. According to this theory then:

$$n_1A_1 + n_2A_2 + \ldots\ldots = n_1'A_1' + n_2'A_2' + \ldots\ldots$$

in which n_1 mols of A_1 (tristearin) and n_2 mols of A_2 (caustic soda) reacts to form n_1' mols of A_1' (glycerin) and n_2' mols of A_2' (soap). If the letter c is used to denote the molecular concentration (gram mols per liter) then the equilibrium constant K may be obtained from the equation:

$$K = \frac{c_{A_1'}^{n_1'} \cdot c_{A_2'}^{n_2'}}{c_{A'}^{n'} \cdot c_{A_2}^{n_2}}$$

or

$$K_{\text{(for sodium soap)}} = \frac{\text{(mols of tristearin in charge) (mols of soap at equilibrium)}}{\text{(mols of glycerin at equilibrium) (mols of caustic soda in charge)}}$$

Theoretically, if the value of K, the equilibrium constant, is known the equilibrium concentrations or yields may be computed from the known concentrations of the reacting materials. However, the law is only strictly applicable to very dilute solutions and it is not surprising that computations based on the above equation are often misleading, in view of the viscous reacting media and the presence of many other substances than those considered in the equation.

Taking typical kettle charges, one in which saponification is carried out under quite dilute conditions and another in which the concentrations are high, the effect on the equilibrium constant may be estimated as being quite small. In actual practice the formation of steam in the reacting masses, the

loss of moisture, and inefficient mixing which may cause localized spots of highly concentrated materials do much to produce a hetrogeneous condition not adaptable to accurate computations.

	Gram Mols per Liter		
	Case A	Case B	Case C
From Charge:			
Tallow (Tristearin)	.160	.160	.736
NaOH	.510	.538	2.370
By Analysis:			
Soap	.578	.578	2.650
Glycerin	.185	.185	.839
Equilibrium Constant K	0.980	.930	0.980

In cases *A* and *C* the same percentage of excess caustic soda has been used (approximately 6 per cent), whereas in case *B* it is considered that the yields of soap and glycerin are from a practical standpoint equivalent to those in case *A*, the excess of NaOH in case *B* being 12 per cent over theoretical. It is evident, from these figures, that normal changes in the relative quantities of the soap ingredients (ratio of alkali to fat remaining constant), oil and water, do not affect the Equilibrium Constant appreciably, but doubling the excess alkali lowers the constant.

The use of the autoclave for saponification reduces the cooking period by roughly two-thirds, normal open kettle saponification requiring from six to eight hours with a jacket pressure of 100 pounds per square inch.

In order to carry out the saponification process to as near completion as possible in the shortest period of time an adequate excess of caustic soda should be added. Some fats, and particularly fatty oils such as corn oil, cottonseed oil and castor oil, require greater excesses than others. Too great excesses should be avoided as salting out will take place, as previously mentioned, and there is some evidence that the free caustic soda in the presence of moisture which may accidently gain entry to a bearing may cause rapid rusting of highly polished steel anti-friction bearing parts. In Chapter I, Table 1, the theoretical amounts of alkali for saponifying various fats and fatty oils are given; however, it should be kept in mind that in actual practice these values must be increased by adding excess alkali to the extent of from one to twenty per cent over these figures. In autoclave cooking of the soap, only very slight excesses need be used; in open kettle work with low steam pressures in the jacket, the excesses must be greater. Naturally, a great many factors control the extent of the excess alkali added, the most important being the maximum free alkali content allowable in the finished grease, free fatty acids present in the soap stock mixture, the presence of alkali-absorbing material such as naphthenic acids and other material in the lubricating oil or petroleum residues to be blended with the soap base, and the time and temperature conditions of manufacture as well as the degree of mixing or agitation possible. Where grease formulae are given, in the following chapters, it may be assumed that adequate excesses are provided with due consideration being given to the character of the other ingredients used in the grease and the other factors just mentioned.

Velocity of Saponification as Affected by Temperature

It is a well established fact that the velocity of a chemical reaction is accelerated by increases in temperature. It has been shown as the result of a large number of experiments in connection with a wide variety of chemical reactions that, in general, the velocity of a reaction is doubled or trebled for an increase of temperature amounting to 10° C. Thus, in saponification in open kettles the temperature seldom rises to more than 105° C. (221° F.) on account of the water present. But, in pressure saponification with steam in the jacket at 100 pounds per square inch, the interior of the autoclave at 70 pounds per square inch and its contents at 148.9° C. (300° F.), it is not uncommon to find that the reaction velocity accelerated ten fold. van't Hoff has developed a formula dealing with this phenomenon, as follows:

$$k_1 = k_0 c^{\left(\frac{A(T_1 - T_0)}{T_0 T_1}\right)}$$

in which: k_0 and k_1 represent reaction velocity constants at the respective temperatures T_0 and T_1. "c" is the base of the Naperian system of logarithms and A is a constant.

SOLUBILITY AND COLLOID CHARACTERISTICS

The sodium salts of the saturated fatty acid series are easily soluble in water, but the solubility decreases as the molecular weight of the fatty acids increases. Thus, the alkali salts of palmitic and stearic acids are no longer soluble in cold water. They dissolve, however, if boiled with water. The following solubilities are by Krafft:

Table 1—Solubilities of Sodium Salts of Saturated Fatty Acids

Sodium salt of	Temperature of separation from aqueous solutions containing 25%	20%	15%	10%	1%	Melting point of Soap
Stearic acid		156° F.	154° F.	145° F.	140° F.	500° F.
Palmitic acid		144			113	518
Myristic acid		127			88	482
Lauric acid	113° F.	99			52	491
Elaidic (iso-oleic)		113			95	437
Oleic acid	57				0	450
Erucic acid		95			80	446

The neutral salts of the alkali metals, soaps, are not readily soluble in the organic solvents usually employed, such as ether, petroleum ether, benzene, etc., (alcohol is an exception). In the anhydrous state, the soaps are more soluble in ether and in benzene than in petroleum ether. Acid soaps, those in which there is an excess of fatty acids, behave differently. It is of considerable importance that the ratio of caustic soda to fatty ingredients and the degree of dehydration of the soap affect greatly the texture, melting point, and stability of the soda base greases.

Much of the theory developed by McBain on the formation of soda soaps in aqueous solution is applicable to the manufacture of soda base grease. For further information on the theory of soda soap formation and properties the original works of McBain should be consulted.

Aqueous sodium soap solutions have a high electrical conductivity, only a moderate osmotic activity, and a colloidal nature. Soaps are conceived as a type of a large class of substances which may be defined as colloidal electrolytes, which owe their properties to the presence of a highly hydrated and highly charged excellently conducting "ionic micelle." On cooling aqueous soap solutions there are obtained:

Typical transparent gels, consisting of strings of soap molecules, microns or even millimeters in length. The formation of these chains is explained by the phenomena of residual affinity.

Curds, which are felts of curd fibers enmeshing mother liquor. The micelle being colloidal sorbs (*i.e.*, adsorbs and absorbs) most of the available material around it.

FACTORS AFFECTING CHARACTER OF SODA BASE GREASES

When the soda soaps are formed in the presence of mineral lubricating oil and water by boiling fat and caustic soda together, a heavy spongy material is obtained. This sponge is affected very little by mechanical agitation. Samples of such sponge or fiber greases have been ground through a colloid mill the surfaces of which were only a thousandth of an inch apart and operated at very high speeds without breaking down the fibrous texture of the grease. When the sponge greases are first formed a large quantity of the mineral oil is sorbed by the soap structure. The exact nature of the resulting product is dependent upon a large number of factors, the most important of which may be listed as:

The Nature of the Fat or Fatty Acid. The relative quantities of saturated and unsaturated fatty material, liquid and solid fats and their purity have a very pronounced effect on the texture, melting point and appearance of the finished product.

The Characteristics of the Mineral Lubricating Oil. The soda soaps are capable of forming satisfactory greases with practically any mineral oil whether highly refined by treatment with sulfuric acid and filtration through clay or in a relatively crude state as a lubricating distillate. Large quantities of tar and petroleum residues are often incorporated in the fiber greases. The effect of the tar or asphalt is to produce a grease of smoother texture than could be obtained with highly refined lubricating oils. It has been found that the production of fiber greases using refined Pennsylvania cylinder stock is very difficult, small lumps of hard soap being formed when the grease is dehydrated and cooled. Rapid cooling and the use of slightly less caustic soda than is required for complete saponification of the fatty ingredients have a corrective effect. Greases using the heaviest California or Texas stocks are easily made. Where it is necessary to utilize Pennsylvania cylinder stock it is best to start the kettle charge with comparatively low viscosity Naphthenic Lubricating Oil, and blend in the

cylinder stock after saponification and formation of the sponge base have been completed, this being usually carried out at temperatures lower than the maximum attained at the completion of saponification.

The Temperature and Rate of Increase in Temperature During Saponification. These often affect the characteristics of the finished grease. It appears probable that the soap is formed in accordance with the teachings of the phase rule and that the soap formed is not a product which has a definite composition but is a variable depending on:

The temperature of boiling.
The exact nature of the fatty material.
The composition of the saponifying medium and nigre.
The pressure under which the boiling is carried out.

The Ratio of Caustic Soda to Saponifiable Material. This has a very important relation to the melting point and texture of the finished product. Excess caustic decreases the tendency of the grease to form heavy fibers and also produces a higher melting product. This is particularly true for asphaltic base oils.

The Extent of Dehydration. When soda base greases are heated to 350° or 400° F., depending on the composition of the product, they melt to a liquid. At these temperatures practically all of the water has been removed from the system but traces still remain. If this liquid is cooled rapidly a clear gel is formed which is readily broken by mechanical agitation. If stirred while cooling slowly a normal fibrous grease is obtained. Dehydration at 500° F. produces a product of a very smooth texture and very high melting point, provided a proper excess of caustic soda has been employed.

The Rate of Cooling. The rate at which melted soda base greases are cooled during the finishing process has a decided effect on the texture and appearance of the final product, rapid cooling producing a grease of less fibrous texture.

The soda base fiber greases differ from the lime base cup greases in that the internal phase of the grease is oil rather than water as for the cup greases. The soda base greases cannot be considered as emulsions since they may exist as practically dehydrated products. Those soda base greases, however, having appreciable quantities of water, carry the water in the external phase. From a colloidal viewpoint, it may be said that the sodium soap molecules have an excess of negative ions absorbed on them and, being readily wetted by water, yield oil-in-water emulsions. From a practical consideration they are mineral oils sorbed by a lattice structure soap, giving a plastic solid.

Soda soaps may be formed by the direct neutralization of free fatty acids with caustic soda. The reaction which takes place may be expressed as follows:

$$\underset{\text{Fatty acid}}{R.COOH} + \underset{\text{Caustic soda}}{NaOH} = \underset{\text{Soap}}{R.COONa} + \underset{\text{Water}}{2H_2O}$$

In these equations R represents the radicle of the fatty acid used. If sodium carbonate is used in place of the caustic soda, the reaction is expressed thus:

$$\underset{\text{Fatty acid}}{2R.COOH} + \underset{\substack{\text{Sodium}\\\text{Carbonate}}}{Na_2CO_3} = \underset{\text{Soap}}{2R.COONa} + \underset{\substack{\text{Carbon}\\\text{dioxide}}}{CO_2} + \underset{\text{Water}}{H_2O}$$

VARIOUS METHODS FOR MANUFACTURING SODIUM SOAPS

The following methods have already been mentioned:

Boiling together fats or fatty oils with hydroxide solution. The heat may be applied by any of the following means:

Steam jacketed open kettle.
Steam jacketed autoclave.
Fire heated open kettle (Coal, coke, oil or gas).
Fire heated still.
Closed or open kettle jacketed for indirect heating media such as hot oil, mercury vapor, triphenyl oxide, etc.
Electrical resistance applied to either open or autoclave type kettle.
Ignition of a part of the oil in an open kettle to produce heat for later dissolution of the soap.

Neutralization of fatty acids with caustic soda solution in an open kettle or still.

Reaction between sodium carbonate and fatty acids.

Other theoretical possibilities exist but are of little practical significance. For instance, metallic sodium, sodium peroxide, sodium bicarbonate, sodium percarbonate and sodamide (NH_2Na), usually in the presence of sufficient moisture to promote hydrolysis, have been employed experimentally.

Blending the Mineral Oil with the Soap Base

The principles underlying the preparation of the soap base have been discussed. The completeness of saponification may be judged by the following means:

Laboratory analysis. This is too slow for routine manufacture, but results from occasional samples from the commercial batches may be of considerable value to the grease maker in estimating when the soap is finished.

A quick determination of the per cent of free alkali present is often useful, particularly when the exact amount of the excess alkali used is definitely known.

A consistency test or determination of the A.S.T.M. Penetration on a quickly cooled sample from the kettle is frequently resorted to in determining the correct time at which to start the addition of the mineral oil.

Melting Point determinations are sometimes made on the soap base prior to mixing in the mineral oil, particularly when high melting point specifications must be complied with.

Determination of the water content of the soap base is frequently of importance if the maximum temperatures attainable in the grease kettle are not particularly high.

The general experience of the grease maker based on yields of grease for commercial batches, and occasional data as indicated in the items above is more often the method used for determining when the batch has been sufficiently saponified.

The following factors will govern the rate at which the mineral oil may be run into the kettle and intermixed with the soap base:

The Temperature of the Soap Base. At temperatures well above 250° F. the oil can be run into the kettle with the sodium soap at a quite rapid rate without causing the base to break up and "swim" about, forming lumps which can only later be incorporated with considerable difficulty as a smooth grease.

The Temperature of the Oil Being Added. If the oil is stored out of doors and is below about 50° F. some difficulty may be experienced in blending it with the soap base.

The Composition of the Soap Base. Excess free alkali increases the difficulties of oil blending, due to the fact that it exerts a salting out effect, thus causing the soap to resist ready dispersion in the oil. The presence of free fatty acids, fat, mono and distearins and mono and dioleates, glycerin, and other soap solvents accelerate dissolution of the soap.

The Composition of the Mineral Oil. The paraffinicity of the oil added affects the ease of blending in the oil, the more polar the oil the more readily may it be added and at a higher rate. Thus, naphthene base lubricating oils offer no difficulty.

The Efficiency of Agitation. Double motion scrapers are desirable in order to produce thorough mixture in the least possible time. Inefficient agitator paddles may permit the entire batch to rotate in the kettle, in such a manner that little free surface becomes available to sorb the oil being added.

The Degree of Hydration of the Soaps. The anhydrous soaps are sometimes very difficult to get into proper dispersion.

VARIOUS METHODS OF MANUFACTURING CALCIUM SOAPS

The calcium soap bases for various forms of cup greases may be prepared commercially by a wide variety of methods. The most important of these are:

Direct action of hydrated lime on fat, or fatty oil, in an open mixer heated by one of the following means:

Steam at 50 to 175 lbs. per square inch in jacket.
Electrical resistance.
Direct application of flame to kettle bottom and sides, due to combustion of wood, coke, oil, gas or coal.
Indirect application by means of some heat carrying fluid such as hot oil, etc.

By neutralization of fatty acids with hydrated lime, utilizing an open kettle and either a cold sett process or accelerating the reaction by cooking as in first method above.

By the action of hydrated lime on solutions of sodium soaps in an open kettle.

By the action of hydrated lime on fats, or fatty acids, in a steam jacketed (direct or indirectly heated autoclave) kettle.

In any of the above items the lime soaps may be completed, dried and stored for future blending with oil to produce grease. The more usual procedure, however, is to finish the grease in the open kettle, or, in the case of an autoclave, discharge the soap into an open kettle and add the appropriate amount of mineral oil at once.

CATALYSIS

The velocity of reactions may be greatly accelerated by the presence of small amounts of various substances which apparently do not participate in the reaction and remain essentially unchanged when the reaction is complete. A catalyst, when present in very small amounts, may greatly accelerate the reaction of relatively large masses, it does not initiate a reaction but promotes it, and equilibria do not appear to be disturbed by its presence. Very frequently small amounts of caustic soda are utilized in batches of lime soap, where it apparently serves as a catalyst. The catalytic action of

the sodium hydroxide in this case may be attributed to the hydroxyl ion, its effect being proportional to the concentration of the OH ion.

THE CHEMISTRY OF CALCIUM SOAP FORMATION

Double Decomposition

Relatively pure calcium soaps may be made as precipitates by adding a solution of some soluble lime salt, such as calcium chloride or calcium acetate, to a solution of sodium or potassium soap in water. The reactions involved are as follows, using tristearin, one of the principal constituents of tallow, as a typical example:

Formation of Sodium Soap

$$\underset{\text{Tristearin}}{C_3H_5(O\,.\,CO\,.\,C_{17}H_{35})_3} + \underset{\text{Caustic Soda}}{3NaOH} = \underset{\text{Glycerol}}{C_3H_5(OH)_3} + \underset{\text{Sodium Stearate}}{3C_{17}H_{35}COONa}$$

Conversion of the Sodium Stearate to a precipitate of water-insoluble lime soap

$$\underset{\substack{\text{Calcium}\\\text{Chloride}}}{CaCl_2} + \underset{\substack{\text{Sodium}\\\text{Stearate}}}{2C_{17}H_{35}COONa} = \underset{\substack{\text{Calcium}\\\text{Stearate}}}{(C_{17}H_{35}COO)_2Ca} + \underset{\substack{\text{Sodium}\\\text{Chloride}}}{2NaCl}$$

This procedure is used only where the recovery of glycerol is of importance, and where relatively pure soap is desired.

Direct Action of Hydrated Lime on Fat

The usual method followed in the manufacture of lime soaps for use in grease lubricants is the direct saponification of the whole fat or glyceride, as it is properly termed, with hydrated lime by boiling, thus:

$$\underset{\text{Tristearin}}{2C_3H_5(O.CO.C_{17}H_{35})_3} + \underset{\substack{\text{Hydrated}\\\text{Lime}}}{3Ca(OH)_2} = \underset{\text{Glycerol}}{2C_3H_5(OH)_3} + \underset{\substack{\text{Calcium}\\\text{Stearate}}}{3(C_{17}H_{35}COO)_2Ca}$$

In this process the glycerol remains in the finished lubricant.

Direct Neutralization of Fatty Acids with Hydrated Lime

This procedure is often followed in preparing calcium soaps suitable for use in greases. The chemical equation given below represents the reaction which takes place readily at a temperature of 100° to 150° F.

$$\underset{\text{Stearic Acid}}{2C_{17}H_{35}COOH} + \underset{\substack{\text{Hydrated}\\\text{Lime}}}{Ca(OH)_2} = \underset{\substack{\text{Calcium}\\\text{Stearate}}}{(C_{17}H_{35}COO)_2Ca} + \underset{\text{Water}}{2H_2O}$$

Effect of Lime[2]

In order to show the effect of using various quantities of hydrated lime, the writer has prepared a series of greases making use of the pressure method

[2] Klemgard, E. N., *Refiner Natural Gasoline Mfr.*, 68 (March, 1929).

of cooking the soap, in which all of the ingredients have been maintained as constant as possible with the exception of the lime. The amount of lime used was varied to include batches with an excess and also a deficiency over that required for theoretical saponification of the fat.

The saponification value of the mixture of 20 per cent of Tallow and 80 per cent Lard Oil was 195 mg. KOH per gram of fat. The amount of available calcium oxide, as determined by the American Society of Testing Materials method, in the hydrated lime was found to be 69.0 per cent; this is equivalent to 91.3 per cent. available calcium hydrate. The actual moisture content of the hydrated lime, as determined by heating a sample of the material, was found to be 23.51 per cent. The theoretical amount of water to hydrate pure calcium oxide is 24.43 per cent. Using these values, the amount of powdered hydrated lime required for complete theoretical saponification may be calculated as 14.08 pounds of lime per hundred pounds of the fat.

In making this series of greases, using the pressure kettle for saponification of the fat, the procedure as already given was followed, particular care being taken to give the batches identical treatment at such points as:

Time and temperature of soap formation.
Temperature to which soap was brought before the mineral oil was started in.
Temperature and relative time at which water was added.
Temperature of drawing grease and filling sample cans.

The dry soap base before starting to add the final oil is calculated to have the following formula:

74.6 per cent Fat (lard oil and tallow)
10.4 per cent Hydrated lime
15.0 per cent 180 Pale oil.

This soap base for each batch was analyzed in order to determine the completeness of saponification with results as shown below:

No. Batch	Parts Lime per 100 Fat	Free Fat Acids in Soap Base Per Cent.	Uncombined Fat in Base Per Cent	Combined Fat in Base Per Cent
1	12.17	3.62	5.56	65.88
2	12.47	2.42	4.58	69.98
3	12.75	3.04	3.94	70.10
4	13.04	.91	3.83	61.88
5	13.33	.50	5.14	72.23
6	13.67	.39	2.03	72.38
7	13.92	1.02	3.79	71.53
8	14.22	.00	2.16	66.60
9	14.51	.44	3.14	67.16
10	14.79	.00	2.26	69.05
11	15.09	.48	3.19	71.40
12	15.38	.00	2.49	65.60
13	15.67	.17	2.85	66.57

It is apparent that the resulting grease made with less than about 14 pounds of hydrated lime per 100 pounds of fat (14.08 required for theoretical complete saponification) will contain an appreciable amount of free fatty acids as well as considerable free fat. However, even with an excess of

lime the base shows from 2.16 to 3.19 per cent of free fat and up to about 0.50 per cent Free Fatty Acids. It may be concluded that saponification of the fat at 50 pounds pressure for at least 40 minutes is not sufficient to bring about complete reaction; however, former experimental work has indicated that with slight excesses of lime the reaction is about 95 per cent complete at this pressure in 20 minutes. Continuation of the reaction under these conditions for a period up to three hours increases the completeness of reaction by about 3 per cent, equilibrium apparently being established at about 98 per cent. Even though the reaction is not entirely complete, the speed of the reaction, as well as the extent of the reaction, are such a great improvement over saponification at atmospheric pressure, that the process is rapidly gaining favor. The use of one-half pound of caustic soda per 100 pounds of fat, together with the lime, has a tendency to increase both the speed and extent of the reaction.

The following table gives the formulae of the series of cup grease in which the amount of lime used has been varied from 2.03 per cent to 2.62 per cent.

	Per Cent				Parts Hyd. Lime per 100 Pts. Fat	Penetration at 77° F.	Melting Point ° F.
Batch	Water	Fat	Hydrated Lime	180 Vis. Pale Oil			
1	0.30	16.68	2.03	80.99	12.17	195	140
2	0.30	16.68	2.08	80.94	12.47	243.8	144
3	0.40	16.68	2.13	80.79	12.75	210	151
4	0.40	16.68	2.18	80.74	13.04	145.5	156
5	0.80	16.68	2.22	80.30	13.33	93.5	182
6	0.80	16.68	2.28	80.24	13.67	82.5	184
7	0.80	16.68	2.32	80.20	13.92	80	184
8	0.80	16.68	2.37	80.15	14.22	76.5	186
9	0.80	16.68	2.42	80.10	14.51	85	186
10	0.80	16.68	2.47	80.05	14.79	92	186
11	0.80	16.68	2.52	80.00	15.09	90	186
12	0.80	16.68	2.57	79.95	15.38	144	195
13	0.80	16.68	2.62	79.90	15.67	192	198

The results show conclusively that the highest yield of a satisfactory appearing grease is obtained when using slightly more lime than that required for theoretical complete saponification, *i.e.*, a maximum yield is obtained when 1 per cent excess lime is used. This occurs in batch 8 where 14.22 pounds of lime per hundred parts of fat was used; this is 0.14 pounds greater than the theoretical quantity and constitutes an excess of nearly one per cent.

It was observed that batches 1 to 4 were very soft and had a mucky, opaque appearance. Batches 12 and 13 also exhibited these characteristics, but gave very high melting points. It is of considerable interest that the melting points of the finished greases are closely related to the quantity of lime employed, low melting points characterizing a deficiency of lime and excess lime producing high melting points. These are factors of considerable importance to the grease maker attempting to meet melting point and penetration specifications, who in the past has used the rule-of-thumb method of arriving at a specific product. It should be understood that this work

deals only with changes in the characteristics of the grease brought about by use of various quantities of hydrated lime.

The function of the water added to the grease is to hydrate the calcium soaps and to cause them to become dispersed in the oil phase giving a product of marked gel properties. The loss of this water from the system causes separation of the oil and soap. When work was first started on this series of greases it was decided to keep that amount of water added constant at about 0.7 per cent; however, with the lower quantities of lime it was found that this water was not required. In fact, this amount of water caused the grease to be slimy and of poor consistency.

In contrast to this result, it was noted that when batches 11 and 12 were repeated adding 1.65 per cent water instead of the 0.8 per cent, satisfactory appearing greases having penetrations of 95 and 100 (about 190-275 A.S.T.M. unworked) respectively were obtained. It appears possible that the excess lime has a tendency to "seed out" or "salt out" the lime soaps and that additional water has a corrective effect.

It may be stated that the per cent available calcium oxide, as determined in the hydrated lime used for making cup grease by the pressure method, may vary over a total range of about 6 per cent without greatly affecting the consistency of the finished product.

NATURE OF REACTION BETWEEN HYDRATED LIME AND FATS

As in the case of the saponification of tristearin with caustic soda it is very likely that direct saponification of fats with calcium hydrate also proceeds in three steps as indicated in the following equations:

1) $$\underset{\text{Tristearin}}{\begin{matrix}CH_2O(C_{18}H_{35}O)\\ 2CHO(C_{18}H_{35}O)\\ CH_2O(C_{18}H_{35}O)\end{matrix}} + \underset{\substack{\text{Calcium}\\ \text{Hydrate}}}{3Ca(OH)_2} = \underset{\text{Distearin}}{\begin{matrix}CH_2OH\\ 2CHO(C_{18}H_{35}O)\\ CH_2O(C_{18}H_{35}O)\end{matrix}} + \underset{\substack{\text{Calcium}\\ \text{Stearate}}}{(C_{18}H_{35}OO)_2Ca} + \underset{\substack{\text{Calcium}\\ \text{Hydrate}}}{2Ca(OH)_2}$$

2) $$\underset{\text{Distearin}}{\begin{matrix}CH_2OH\\ 2CHO(C_{18}H_{35}O)\\ CH_2O(C_{18}H_{35}O)\end{matrix}} + \underset{\substack{\text{Calcium}\\ \text{Hydrate}}}{2Ca(OH)_2} = \underset{\text{Mono-stearin}}{\begin{matrix}CH_2OH\\ 2CHOH\\ CH_2O(C_{18}H_{35}O)\end{matrix}} + \underset{\substack{\text{Calcium}\\ \text{Stearate}}}{(C_{18}H_{35}OO)_2Ca} + \underset{\substack{\text{Calcium}\\ \text{Hydrate}}}{Ca(OH)_2}$$

3) $$\underset{\text{Mono-stearin}}{\begin{matrix}CH_2OH\\ 2CHOH\\ CH_2O(C_{18}H_{35}O)\end{matrix}} + \underset{\substack{\text{Calcium}\\ \text{Hydrate}}}{Ca(OH)_2} = \underset{\substack{\text{Glycerol}}}{\begin{matrix}CH_2OH\\ 2CHOH\\ CH_2OH\end{matrix}} + \underset{\substack{\text{Calcium}\\ \text{Stearate}}}{(C_{18}H_{35}OO)_2Ca}$$

It is well known that mono and distearin and mono and diolein are better emulgators than the whole fats. In the grease structure, it is possible that they act as bonding agents or intermediate molecules in hydrating the calcium soaps. They may, therefore, be responsible for the production of more stable types of cup greases, particularly when they may exist in the presence of no appreciable amounts of free alkali.

According to R. G. Pelly, of the Technical Research Works, Ltd.,[8] derivatives of polyhydric alcohols in which the hydroxyl groups are replaced

[8] British Patent 188,364 (July 7, 1921); *see* also: Pelly, R. G., U. S. Patent 1,463,092 (July 24, 1923).

by fatty or other acid radicles may be added to mineral oil, fats, and waxes to increase their lubricating and emulsifying properties and to produce a heavier consistency. While there is some practical experience which indicates that prolonged pressure saponification to produce calcium stearate and oleate results in slightly lower yields of finished grease than a shorter period in which mono and distearates and oleates may remain in the batch, further actual experimental evidence is needed to justify such a view.

Melting Point of Calcium Soaps

	Melting Point of Fatty Acid ° C.	Melting Point of Calcium Salt ° C.
Palmitic acid	62	153–156
Stearic acid	69	150–154

NATURE OF THE COMBINATION OF LIME SOAPS AND WATER IN CUP GREASES

It is evident that water held in a state of emulsification in the oil, as small particles visible in the microscope. is of little value in promoting proper dispersion of the calcium soaps. Since certain calcium soaps such as those of wool grease. and calcium mono and dioleates and stearates appear to hold water in a bound condition more firmly than normal calcium salts, and since they improve the heat stability of their greases, it is obvious that choice of soap stock and probably the extent of saponification are important factors in determining the stability of greases. Regarding the quantity of water necessary in cup greases, Kauffman [4] considers the following items are applicable:

If a relatively large amount of unsaponified fat is present in the finished grease, it is probable that the amount of water present during saponification, in the open kettle process, was smaller than in a grease containing the same amount of soap, but only a very small amount of free fat, manufacture otherwise being similar.

Lime soaps manufactured from the same fats, under similar conditions, should "fix" or combine in a bound condition with similar amounts of water.

The percentage of water required for proper dispersion of calcium soaps in cup greases increases with the percentage of soap present.

Calcium soaps incorporated with very viscous mineral oils require more water than those greases made with the same amount of soap, but containing low viscosity oils. There is also some evidence the degree of paraffinicity, treatment and presence of asphaltic constituents and polar bodies all have an influence on the quantity of water needed in the grease.

In cases where the soap is prepared separately before incorporation with mineral oil, the harder the soap, the more water will be required.

The quantity of water required will also vary with the facility with which the fats are saponified with calcium hydroxide, those soaps which are readily saponified at a high rate of speed requiring less water. It is likely that the polarity of the fat and its ability to form emulsions and combinations with water greatly influence the effectiveness of the water present, by increasing its state of division, dispersion and available surface.

Water requirements will also vary with the titer of the fat mixture used as soap stock, the higher the melting point of the mixed fatty acids, the more water will be needed. No doubt, the degree of unsaturation also influences the water requirements.

Depending on the foregoing factors and others, the amount of water in cup grease will vary from less than one tenth per cent to as much as six per cent.

[4] *Refiner Natural Gasoline Mfr.*, 96 (May, 1929).

CALCULATION OF THE SOAP MAKING VALUE OF FATS

In Chapter II, the significance of various laboratory tests, such as the Iodine Number, Saponification Value and Acid Number, was discussed. Their full importance in evaluating soaps stocks is seldom realized by most grease makers, the tests being ordinarily made on incoming shipments of fats, checked against purchase specifications and employed as a means of acceptance or rejection. Where various blends of fats are used in making soap for grease, the characteristics of the fats should be carefully studied by the grease plant chemist and used as a basis for determining the theoretical amount of alkali required to which the normal excess should be added. The effects of this excess, or various total amounts of alkali, with regard to yield have been referred to and it is evident that precise control is not an absolute essential. However, when such factors as texture, color, outertone, and transparency are taken into consideration, then close attention to all manufacturing details, including the variables in the soap stock characteristics and process factors, must be under close control.

Singer [5] has indicated the following method of arriving at the total soap making value of a fat from the per cent H_2O, per cent Ash, per cent Unsaponifiable, Saponification Number, and the Acid Number. As an example, a fat having the following characteristics may be considered:

	Per Cent		
H_2O	0.1	Saponification No.	253
Ash	0.2	Acid Number	13
Unsaponifiable	0.3		

The total non-fats (Water, Ash and Unsaponifiable) amount to 0.006 grams per 1 g. The Ester Number (Sap. No.—Acid No.) is 240.

The Molecular weight [6] of the fatty acids is:

$$\frac{56{,}100 - (56{,}100 \times 0.006) - (12.68 \times 240)}{253} = 208$$

in which 56,100 is 1000 times the molecular weight of KOH, and 12.68 is one third of the molecular weight of the glycerol residue (C_3H_2). (Mol. weight of C_3H_5 less 3H.)

The actual percentage of free fatty acids (x) may be found as follows:

$$x = \frac{208 \times 13}{56{,}100} = 0.0483 = 4.83 \text{ per cent.}$$

The combined fatty acids (y) may be found from the following proportional expression:

$$y : (1 - 0.006 - x) = m : (m + 12.68), \text{ then } y = 0.8914 = 89.14 \text{ per cent,}$$

in which m is 208, the average molecular weight of the fatty acids. The total amount of fatty acids available for making grease base would be: x plus y; which in this case is 4.83 per cent plus 89.14 per cent or 93.97 per cent.

[5] Singer, H., *Seifensieder-Ztg.*, **54**, 813-4 (1927).
[6] C. A., 1697 (1928).

From E. T. Webb's study of the fatty acids and their salts [7] the iodine number and the saponification number are of chief importance in considering the yield of soap obtainable. Iodine numbers above about 100 will ordinarily indicate fats of low yield characteristics. On account of the complex colloid factors involved it has not been possible to predict with certainty the yield of grease obtained from a fat of known chemical characteristics. However, it is possible to work out a reasonably accurate relation for a given process and formula system, between the yield of grease and the function $(S—I)$, in which S is the saponification number and I is the iodine value.

SOLUBILITY FACTOR

While lubricating greases may not be considered as true solutions of soaps in lubricating oils it is reasonable to expect that some definite relation exists between the solubility factors of soap stock mixtures and the yield of greases of a given soap content. Webb obtains the "relative solubility factor" by multiplying the per cent of fats in the mixture with iodine numbers of 30 to 70, by the numerical differences between the average iodine number and the average saponification number of all the fats. When several such fats are present the arithmetical products obtained are added and the sum divided by the numerical difference between the iodine number and the saponification number after multiplying by 100. The result is termed the Solubility Coefficient, and the higher its value the more soluble the soap, and most likely the lower the yield of grease. Webb gives the following example:

	% in Mixture	Iodine No.	Saponification Number
Palm oil	20	56	200
Suet	30	40	198
Cottonseed oil	22	109	194
Palm oil	15	15	250
Rosin	13	120	176
Average saponification number		202.4	
Average iodine number		65.0	
Difference		137.4	

The Solubility Coefficient is obtained from the following equation:

$$\text{Sol. Coef.} = \frac{137.5 \times 100}{20 \times (200 - 56) + 30 \times (198 - 40)} = 1.80.$$

Control of the Solubility Coefficient of fats used in making soaps will result in more uniform batches of lubricating grease. The relation between solubility coefficients and yields of grease offers a fruitful field for further investigation.

THE WET PROCESS FOR MANUFACTURING CUP GREASES

Generally speaking, the manufacture of cup greases in open steam jacketed kettles may be conducted by either of two methods; the wet process

[7] *Soap Gazette; Ind. sapon.* **27**, 181-3 (1927); C. A., 879 (1928).

and the dry process. In the former, the desired amount of fat, a small quantity of mineral oil (usually not more than the equivalent weight of the fat), and the appropriate quantity of hydrated lime and water are stirred in the kettle until the saponification reaction is complete. If the batch becomes too dry a further quantity of water may be added. In completing the batch, it is not only necessary to estimate when saponification is complete, but to judge when the moisture content is correct as indicated by the plasticity of a small quickly cooled sample. Considerable skill is necessary in using this method to produce batches which are uniform in appearance and consistency.

THE DRY PROCESS FOR MANUFACTURING CUP GREASES

This procedure takes its name from the system of dehydration employed followed by adding a definite amount of water to hydrate the lime soap. The hydrated lime, water, oil, and fat are cooked, while agitating, until equilibrium is established, adding some water if necessary. The batch is then nearly dehydrated, cooled by allowing oil to run in until a temperature of about 205 to 215° F. is reached when a definite amount of water to hydrate the lime soaps is slowly added. The balance of the mineral oil is then stirred in, the steam being off in the jacket. In another form of the dry process the fat is mixed with powdered hydrated lime in the kettle. Water is then added directly to the kettle as required.

Pressure saponification will be discussed in the following chapters.

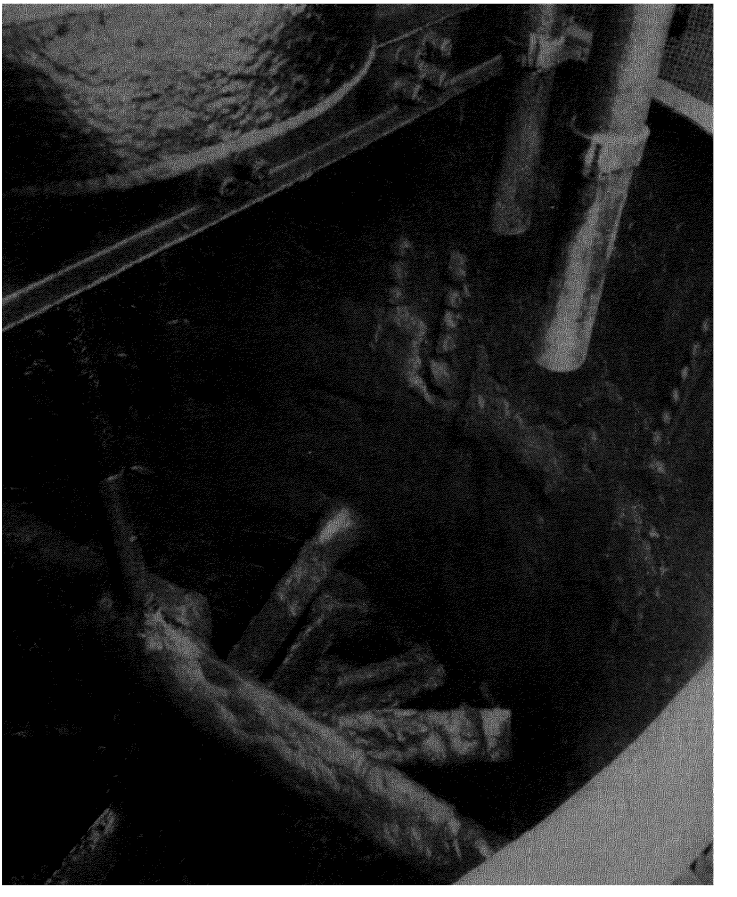
MECHANICAL AGITATION

Experience has indicated that many of the theories relative to the effects of mechanical agitation on rate of reaction and heat transfer are applicable to grease manufacture. Agitation may be secured by means of the rotation of various-shaped stirring devices in a vessel of the grease. Some of these agitators are:

Paddle arms fixed to a vertical shaft (the usual plan).
Two way agitation, paddle arms and paddles attached to a sweep.
Paddle arms and stationary baffles.
Spiral agitator which scrapes sides of kettle.
Relatively high speed propeller.
Spiral screw lifting kettle contents at center of vessel.
Combination of spiral screw sweep paddles.
Circulation by means of various gear pumps (very frequently used in grease plants). The pumps take suction from the bottom of the kettle and discharge back at the top.
Air and steam agitation. (Seldom used except for fluid materials.)
Grease mills and colloid mills.
Kneaders (seldom used except for a few special greases containing hair, wool yarn, etc.)
Spiral screws (as in grease stills and coolers).

There is one disadvantage from the use of kettles equipped with the usual revolving paddles and stationary arms in that material will stick to the kettle walls near these stationary bars. In cooking soap in open kettles of

this kind, as the level of the batch recedes, due to water being removed, a coating of hard soap and grease will remain on the kettle wall and escape mixture. If not scraped off manually it will dry and further harden and may later break off and fall into the batch to produce objectionable soap lumps. To prevent this action, scraper type paddles have been found to



Courtesy Sowers Mfg. Co.

PLATE I.—View of Batch of Grease During Manufacture.
Deposits of grease not removed by adequate scraping.

be very helpful. Close-fitting scrapers are not possible on account of the seams in riveted kettles. The deposits typical in this type of equipment are shown in Plate I. The effects of the scrapers are clearly indicated in Plate II. The small spring steel scrapers attached to the outer edges of the sweep, and kept in tension by means of adjusting screws, will of course

wear, their normal life being from one to three years. The contamination of the grease with metal from this source is however negligible, according to Huggins,[8] being less than one part in a million. Not only do mechanical scrapers reduce the dangers of producing lumpy grease, but greatly increase heat transfer and reduce the time for cooking greases. In one case in which aluminum oleate was being incorporated with mineral oil, a temperature of



Courtesy Sowers Mfg. Co.

PLATE II.—Experimental batch prepared with some of the spring steel scrapers removed. The kettle surface is scraped free of material where the scrapers are used.

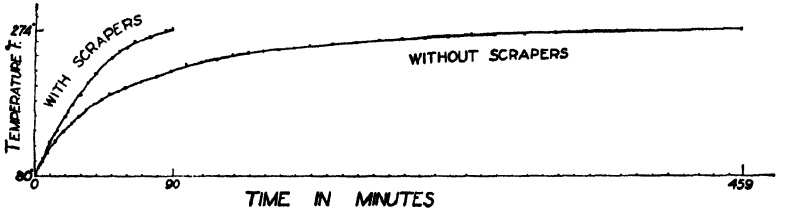
275° F. was attained in 90 minutes with scrapers and 459 minutes without scrapers. See Figure 1.

Huggins also reports that the time to cool a batch of No. 3 cup grease from 290° F. to 160° F. could be reduced from 97 minutes without scrapers to 37 minutes with scrapers, a time savings of 62 per cent. The data obtained in this test is given in Figure 2.

[8] Huggins, F. E., "Grease Manufacturing Equipment," presented before the Lubricating Grease Manufacturers Association, Oct. 14, 1935.

STEAM CONSUMPTION

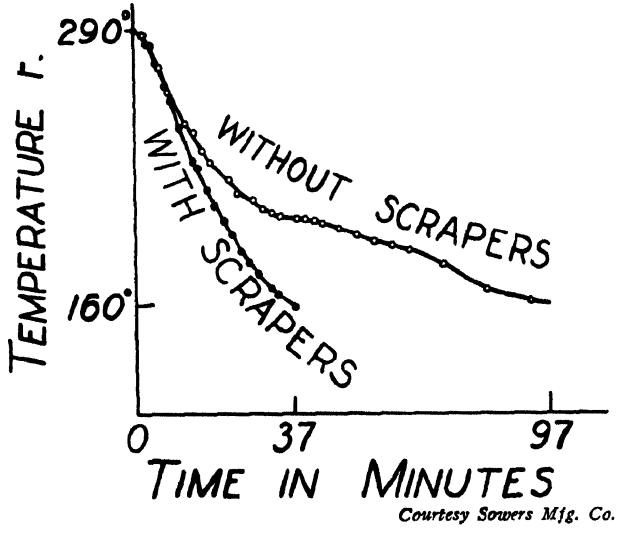
The results of extensive investigations of the total heat required for making various types of grease are not available. It is evident, however, that the steam consumption is quite variable, depending on the system of



Courtesy Sowers Mfg. Co.

FIGURE 1.—Time for heating to 274° F., with and without scrapers.

cooking the soap, nature of agitation employed, character of lagging on kettle and steam lines, thermal conductivity of the kettle walls, specific heat of the grease, and amount of water evaporated during the cooking process as well as the composition of the grease being made. Considered as an average for the normal grease plant, approximately 1,800 pounds of steam, at 100 pounds per square inch, will be required for each 1000 gallon batch

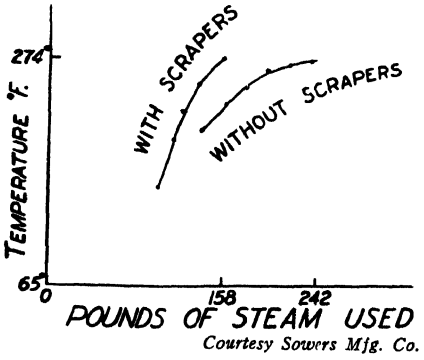


Courtesy Sowers Mfg. Co.

FIGURE 2.—Relative time for cooling batch of No. 3 Cup Grease with and without scrapers.

produced. At refineries where large quantities of steam are available its cost may vary from 15 to 20 cents per thousand pounds. At an average cost of 17.5c per thousand pounds 1,800 pounds of steam would cost $0.315, the cost of steam for a 1000-gallon batch of grease. This amounts to only 0.0041c per pound of grease produced, and is, therefore, not a very impor-

tant factor when compared with the total cost of the grease which may be about 3.0c per pound.



Courtesy Sowers Mfg. Co.

FIGURE 3.—Steam Consumption.

It is claimed by F. E. Huggins that when using kettles equipped with spring steel scrapers, steam consumption will be reduced by one-third. The data on which this claim is made is given for a small batch of aluminum base grease as Figure 3.

TABLE 2—Steam Data. Properties of Saturated Steam

Lbs. per sq. in. Absolute Pressure p	Inches of Mercury Vacuum* G	Temperature Fahrenheit t(F)	Temperature Centigrade t(C)	British Thermal Units above 32° F. Heat of the Liquid h	Latent Heat of Evaporation L	Total Heat of Steam H	Specific Volume Cubic feet per pound v
.0886	29.74	32.	0.0	0.00	1073.4	1073.4	3294.
.206	29.5	54.	12.2	22.1	1061.1	1083.2	1480.
.452	29.	76.6	24.8	44.6	1048.6	1093.2	708.
.698	28.5	90.1	32.3	58.1	1041.1	1099.2	468.
.943	28.	99.9	37.7	67.9	1035.7	1103.6	352.
1.18	27.5	107.3	41.8	75.2	1031.6	1106.8	286.
1.44	27.	114.2	45.7	82.1	1027.7	1109.8	237.
1.68	26.5	119.8	48.2	87.7	1024.5	1112.2	204.
1.93	26.	124.8	51.5	92.7	1021.8	1114.5	180.
2.17	25.5	129.2	54.0	97.1	1019.3	1116.4	160.
2.42	25.	133.3	56.3	101.2	1017.0	1118.2	145.
2.66	24.5	136.9	58.3	104.8	1014.8	1119.6	132.
2.91	24.	140.3	60.1	108.1	1013.0	1121.1	122.
3.40	23.	146.2	63.5	114.1	1009.4	1123.5	105.
3.89	22.	151.2	66.2	119.7	1006.1	1125.8	92.9
4.87	20.	161.2	71.8	129.0	1001.0	1130.0	75.2
5.86	18.	169.0	76.1	136.9	996.4	1133.3	63.3
6.84	16.	175.8	79.9	143.7	992.4	1136.1	54.7
7.82	14.	181.8	83.2	149.7	988.8	1138.5	48.3
8.80	12.	187.2	86.2	155.1	985.6	1140.7	43.3
9.78	10.	192.2	89.0	160.1	982.6	1142.7	39.2
12.2	5.	202.9	95.0	170.9	976.0	1146.9	31.8
14.7	0.	212.0	100.0	180.0	970.4	1150.4	26.8

*Refers to 29.92" Barometer

CONVERSION CENTIGRADE TO FAHRENHEIT

(°C. × 1.8) + 32 = °F.

Example

Change 20° C. to degrees Fahrenheit.
(20° C. × 1.8) + 32 = 68° F.

CONVERSION FAHRENHEIT TO CENTIGRADE

(°F. − 32) × $\frac{5}{9}$ = ° C.

Example

Change 68° F. to degrees Centigrade.
(68° F. − 32) × $\frac{5}{9}$ = 20° C.

TABLE 2.—(*Continued*)

Lbs. per sq. in. Absolute Pressure p	Lbs. per sq. in. Gauge Pressure G	Temperature Fahrenheit t(F)	Temperature Centigrade t(C)	British Thermal Units above 32° F. Heat of the Liquid h	Latent Heat of Evaporation L	Total Heat of Steam H	Specific Volume Cubic feet per pound v
19.7	5	227.2	108.4	195.3	960.5	1155.8	20.4
24.7	10	239.4	115.2	207.7	952.5	1160.2	16.5
29.7	15	249.7	120.9	218.2	945.5	1163.7	13.9
34.7	20	258.8	126.0	227.4	939.3	1166.7	12.0
39.7	25	266.8	130.4	235.6	933.6	1169.2	10.6
44.7	30	274.1	134.5	243.0	928.5	1171.5	9.45
49.7	35	280.6	138.1	249.7	923.8	1173.5	8.56
54.7	40	286.7	141.5	256.0	919.3	1175.3	7.82
59.7	45	292.4	144.7	261.8	915.1	1176.9	7.20
64.7	50	297.7	147.6	267.2	911.2	1178.4	6.68
69.7	55	302.6	150.3	272.3	907.4	1179.7	6.23
74.7	60	307.3	152.9	277.1	903.9	1181.0	5.83
79.7	65	311.8	155.4	281.8	900.5	1182.3	5.49
84.7	70	316.0	157.8	286.1	897.3	1183.3	5.18
89.7	75	320.1	160.1	290.3	894.1	1184.4	4.91
94.7	80	323.9	162.2	294.3	891.1	1185.3	4.66
99.7	85	327.6	164.2	298.1	888.2	1186.3	4.44
104.7	90	331.2	166.2	301.8	885.4	1187.1	4.24
109.7	95	334.6	168.1	305.3	882.6	1187.9	4.06
114.7	100	337.9	169.9	308.8	880.0	1188.8	3.89
124.7	110	344.2	173.4	315.3	874.9	1190.2	3.59
134.7	120	350.1	176.7	321.5	870.6	1192.1	3.34
139.7	125	352.9	178.3	324.4	867.8	1192.2	3.23
144.7	130	355.7	179.8	327.3	865.5	1192.8	3.12
154.7	140	360.9	182.7	332.8	861.1	1193.9	2.92
164.7	150	365.9	185.5	338.1	856.9	1195.0	2.76
189.7	175	377.5	191.9	350.3	847.0	1197.3	2.41
214.7	200	387.9	197.7	361.3	838.0	1199.2	2.14
239.7	225	397.3	202.9	371.3	829.6	1200.9	1.93
264.7	250	406.1	207.8	380.6	821.7	1202.3	1.75
289.7	275	414.3	212.4	389.3	814.3	1203.6	1.60
314.7	300	421.9	216.6	397.4	807.2	1204.7	1.48

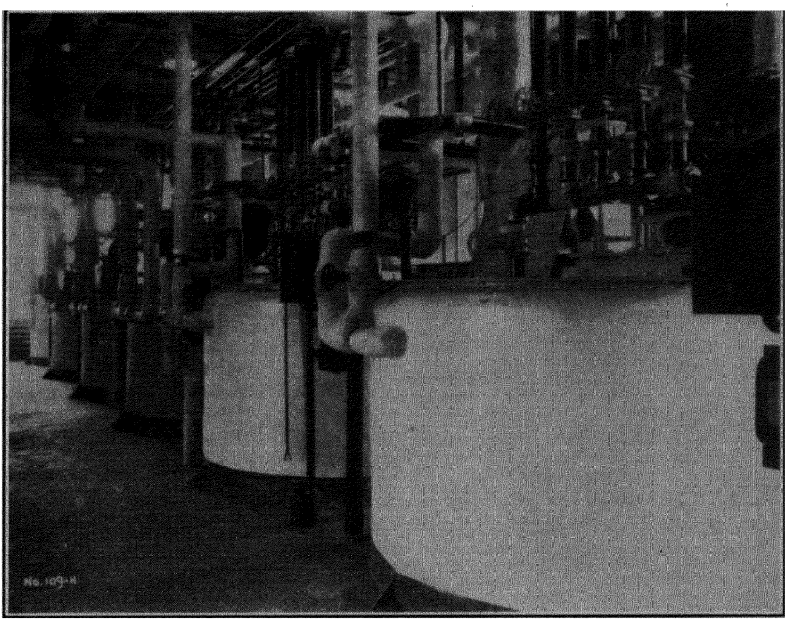
Expressed in another manner, the production of the soap and blending in the oil require approximately 2,100,000 Btu., for a one-thousand gallon batch.

It is evident that, on the basis of the following data from Demorest,[9] refinery produced steam at a cost of 1.5c per one hundred thousand Btu. is much cheaper than other forms of heat available to the independent grease makers:

Source of Heat	Cost per 100,000 Btu., Cents
Bituminous coal ($4.00 per ton)	1.6
City gas (75c per 1,000 cu. ft.)	14.0
Fuel oil (6c per gallon)	4.5
Steam (Refinery cost from gas and fuel oil)	1.5
Electricity (1.5c/kw. hr.)	44.0

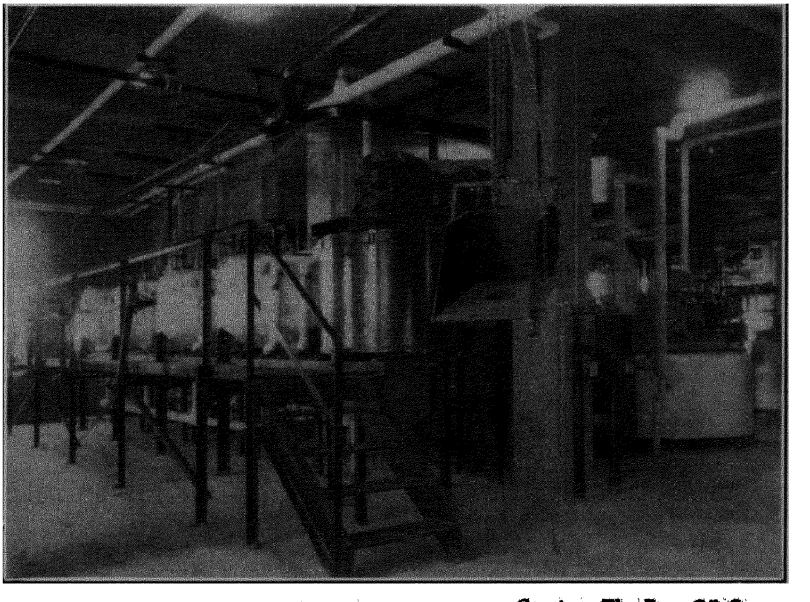
Three-inch pipe line for conveying steam in grease plants should be insulated with at least two inches of a good grade of pipe covering. The fact that the heat loss from one thousand square feet of exposed surface, at one

[9] Demorest, *Chem. Met. Eng.*, **32**, 233 (1925).



Courtesy The Pure Oil Co.

PLATE III.—View of modern grease kettle installation with adequate insulation. Note vapor removal system and pipe lines for five different varieties of lubricating oil.



Courtesy The Pure Oil Co

PLATE IV.—Operating floor of a modern grease plant.

hundred pounds per square inch steam pressure, represents over three hundred tons of coal annually is sufficient justification for seriously considering the installation of adequate pipe covering. The necessary investment in insulation will, as a rule, pay for itself in from one to two years. Properly insulated kettles and steam lines are shown in Plate III, which shows the operating floor of a modern American grease plant.

Each grease plant foreman or manager should issue a list of precautions regarding the conservation of steam. The following items should be of interest in this connection:

Do not open kettle jacket steam inlet valve before steam outlet valve has been opened.

Do not close steam outlet valve before steam inlet valve is closed.

Steam inlet valves should be throttled to the extent that the pressures shown by the jacket gauges should not rise above a predetermined maximum (30 to 150 pounds per square inch).

The steam relief valve should be set at the predetermined maximum allowable pressure, and if steam blows off through the relief valve, either the steam inlet valves are not properly throttled, or the relief valve is out of order and should be repaired.

Steam trap water gauge glasses should be inspected at least once each day, if full of water it is an indication that the automatic valve is stuck.

POWER CONSUMPTION

Quite frequently, the power provided for agitating grease batches is much more than normally used, to provide an ample margin of safety. This is considered good practice as it is difficult, if not impossible, to predict when it may become necessary to process very viscous products which could not be stirred with the power requirements for, say, a batch of ordinary No. 3 cup grease. When stirring very heavy greases, it is sometimes necessary to stop the paddles for taking samples, noting temperatures, or for other reasons, and it is quite likely that the power required for starting up again is at least 50 per cent greater than for normal continuous operation.

It might be assumed that two-way agitation would require an excessive amount of power as compared with one-way stirring. However, tests do not indicate that this is entirely the case. At one grease plant an open jacketed mixer provided with double agitation was equipped with a 20 H. P. motor. Measurements indicated that the maximum power required for the greases being manufactured was only 3.1 H. P. (2.3 kw.). Obviously, a 5 H. P. motor would be normally sufficient, but to provide for peak loads a safety factor of at least 4 was used. In England, a similar grease kettle was provided with a 25 H. P. motor.

A 300-gallon autoclave should normally be provided with a 5 H. P. motor and autoclaves of 500 to 1000 gallon capacity will ordinarily be provided with 10 H. P. motors, or less if no particularly viscous grease bases are to be made.

In order to secure sufficient turbulence and agitation with single motion paddles a speed of 25 to 45 R. P. M. is usually necessary, depending on the size of the kettle, whereas with two-way agitation it is customary to operate the sweep at 15 R. P. M. and the inner paddles at 25 R. P. M. At 40 R. P. M.

the speed of the outer end of the paddle will be about 754 feet per minute, whereas at 25 R.P.M. it is 471, and at 15 R.P.M. only 283 feet per minute. While the power requirements are not directly proportional to these speeds, they do vary with speed. In a test with two-way agitation in which the inner paddles were maintained at a speed of 16 R. P. M., the speed of the sweep was varied from 30 to 15 R. P. M. with a reduction of 80 per cent in the power requirements at the lower speed. With two-way agitation, the velocity of the paddles with respect to each other is additive. Thus, with double paddle speeds of 25 and 15 R. P. M., the velocity of the ends of the paddles will be 754 feet per minute with respect to each other, which is just equal to that of a similar single paddle operating at 40 R. P. M.

Excessive paddle velocities should be avoided as they tend to form aerogels, by occlusion of air in the grease, and may greatly impair the appearance of the finished product.

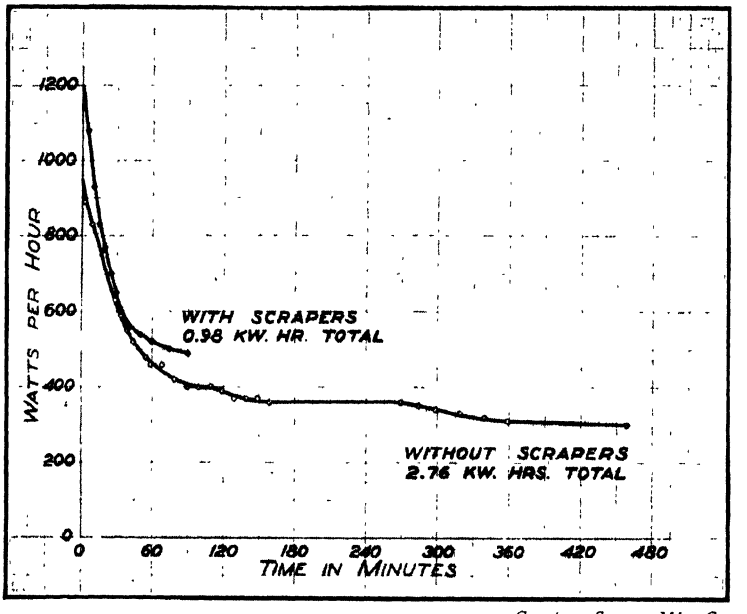
The power consumption of agitating systems built on the double motion principle may be appreciably conserved by staggering the inner paddles in such a manner that they pass the sweep paddles in succession rather than all at one time. In making motor installations it should be remembered that there is a limit to the power that may be safely transmitted by the driving gears and paddle mechanism. The application of 30 or 40 H. P. might cause considerable damage to equipment in attempting to stir some very heavy lubricants. Safe allowable H. P. ratings are given in Table 3 for "New England Type 37 Agitator" drives, these devices being combinations of worm gears and spur gears, mounted in Timken bearings, for one way agitation:

Table 3—Safe H.P. Ratings for Worm and Spur Gear Agitator Drives

(Vertical Shaft Speeds, Ratios and Horsepower Ratings)

Motor Ratios	1740 R.P.M.		1140 R.P.M.		855 R.P.M.	
	R.P.M.	H.P.	R.P.M.	H.P.	R.P.M.	H P.
100.	17.4	4.75	11.4	3.	8.55	2.2
90.5	19.2	5.20	12.6	3.5	9.45	2.5
82.3	21.2	5.6	13.9	3.75	10.4	2.75
75.	23.2	6.25	15.2	4.	11.4	3.
68.8	25.4	6.75	16.6	4.5	12.4	3.4
63.4	27.5	7.30	18.	4.75	13.5	3.70
58.2	29.9	7.9	19.6	5.25	14.7	3.8
54.	32.2	8.5	21.1	5.6	15.8	4.25
50.	34.8	9.25	22.8	6.0	17.1	4.70
46.5	37.5	10.25	24.5	6.5	18.4	5.
43.3	40.2	11.25	26.4	7.0	19.7	5.25
40.2	43.4	12.5	28.4	7.5	21.2	5.6
37.5	46.4	13.75	30.4	8.0	22.8	6.0
35.	49.8	14.8	32.6	8.6	24.4	6.4
32.5	53.5	16.2	35.1	9.5	26.3	7.0
30.5	57.	16.9	37.4	10.75	28.0	7.4
25.	69.6	19.4	40.7	11.5	34.2	9.2
22.6	77.	20.7	50.4	15.3	31.8	10.4
20.5	84.5	21.75	55.4	16.5	41.5	11.75
18.75	93.	22.75	60.8	17.5	45.5	13.5
17.20	101.	23.5	66.4	18.8	49.6	14.6
15.80	111.	24.5	72.2	19.8	54.	16.4
14.55	120.	25.	78.5	20.70	58.8	17.25

The scrapers sometimes attached to the sweeps of the two-way agitators cause power to be used at a higher rate, not only because work must be done to scrape material from the kettle walls, but also on account of the scrapers deflecting the grease in towards the center of the kettle, thus increasing the power absorption of the paddle system. This power increase is not great, being not more than about 25 per cent of that normally required for paddles not equipped with scrapers. Because of the appreciable savings in time required for heating and cooling, there is a net savings in total power consumption when the scrapers are used. Figure 4 shows the relative power consumption for a small batch of aluminum base grease, with and without the use of scrapers.



Courtesy Sowers Mfg. Co.

FIGURE 4.—Power curves obtained by operating mixer with and without scrapers. In both tests aluminum oleate base grease was heated from 80° to 274° F. Power saved by scrapers, 64%.

The run with scrapers required a total of .98 kw. hours, whereas that without scrapers took 2.76 kw. hours.

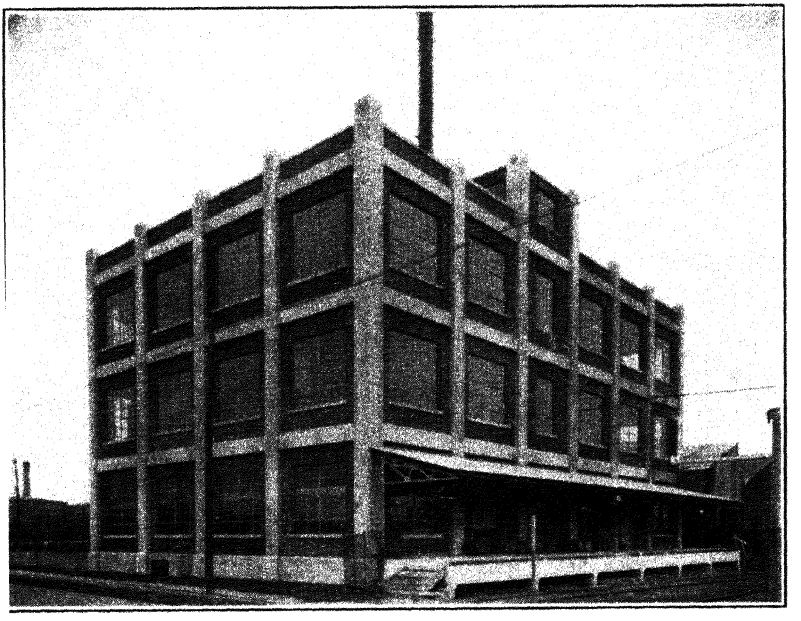
In one grease plant the agitators for five kettles were driven from a single line shaft operating at 120 R. P. M. through a belt to a large back-geared motor. This motor was considerably overloaded when all five kettles were in operation, and when only one or two kettles were used the greases picked up air. Reducing the shaft speed to 80 R. P. M. and the paddle speeds from 30 to 20 R. P. M. overcame both difficulties.

COOLING

Depending on the type of grease being manufactured, a maximum temperature will be attained at near the completion of saponification, or after a certain amount of oil has been blended with the soap. From the time of this maximum temperature on, the grease manufacturing process is one of cooling. The temperature of the batch is reduced to the filling temperature by one or a combination of the following factors:

Convection. A strong induced draft on the kettle vapor system will materially assist quick cooling of the batch.

Loss of heat through the kettle jacket by conduction. It is an advantage to be able to circulate cold water through the jacket for cooling, and thus increase the output of the kettle.



Courtesy The Pure Oil Co.

PLATE V.—Exterior view of a modern fireproof grease plant.

When the grease is circulated to a gear pump and back into the kettle again more rapid cooling is effected in view of the increased radiating and cooling surfaces of the system.

Increased agitation, or the use of spring steel scrapers which remove the congealed grease from the kettle walls, improve the rate of cooling. See Figure 2.

The grease may be drawn from the kettle and passed through chillers or wax coolers; or run into cooling pans to cool without agitation.

The addition of cold mineral oil.

The temperature at which the grease is packaged is quite important as it has an important bearing on the degree of sett attained and the amount of breakdown which may occur on subsequent working at normal temperatures.

Either suitable cast iron or welded kettles may be employed for alternate heating with steam and cooling with water in the jacket. One manufacturer, using a cast kettle heats its contents to 212° F. with steam at 70 pounds per square inch, then turns off the steam and circulates water at 50 pounds per square inch. Cooling is in this manner accomplished in about one-third the time required if no cooling water is used.

Another grease manufacturer has found that it is possible to cool a 600 gallon batch of grease from 300° F. to 190° F. in about one hour when the temperature of the cooling water ranges from 70 to 80° F. In winter with the cooling water at 40 to 45° F. equivalent cooling may be accomplished in about thirty minutes.

Chapter IV

Grease Manufacturing Equipment and Plant

The cost of a grease manufacturing plant will vary with its size (throughput), the nature of the buildings and the value of the apparatus installed. It is estimated that within the United States the total capital invested in lubricating grease manufacturing plants is roughly $5,000,000. The number of pounds of grease manufactured per year, per dollar invested, will vary from six to fifty. Ordinarily, the capital invested in a grease plant will range from $50,000 to $150,000. There are, of course, exceptional cases where cheap grease plants have been put into operation for the production of a limited variety of greases at a cost of less than $7,000, and others in which the capital invested has reached $450,000.

CHEMICAL ENGINEERING ECONOMICS OF THE LUBRICATING GREASE INDUSTRY

It is of interest that nearly all of the large lubricating grease plants have been constructed with major consideration given to the following factors:

Availability of fats and fatty oils at low cost.

Availability of low cost petroleum products. We, therefore, frequently find grease plants as one of the departments of large oil refineries, the managements of these companies obviously being convinced that an adequate market for lubricating greases exists, and that the costs of manufacture and sales insure a reasonable net profit.

Transportation of finished greases and location of plant with regard to large industries which consume grease.

Other factors such as labor, fuel, power, water supply, waste disposal, and public service are of less importance.

The design and construction of the grease plant must take into consideration the following factors:

Railroad spur and truck dock for receiving raw materials (fats and oils) and shipping finished greases. Marine loading facilities must be arranged for if export shipments are to be made.

Storage tanks for oils and fats, storage rooms for solid grease making ingredients, and adequate warehouse facilities for storage of finished greases.

Boiler plant for generation of steam, and availability of electrical power.

Selection of grease mixers and their location within a suitable building in such a manner that continuity of production will be assured.

Expansion of each department for future needs.

From the chemical engineering standpoint grease manufacture is a combination of the subjects known as "heat flow," "mixing" and "cooling," and these items are stressed in the discussions on grease making. The subjects of disintegration, mechanical separation, distillation, evaporation, etc., may all be of importance in connection with the raw materials used in grease

manufacture but must be omitted from this volume in the interests of brevity. The design and convenience of material handling equipment is of greatest importance in a grease plant.[1] Either reciprocating or centrifugal pumps should be employed for unloading incoming tank cars of fats and lubricating oils. The location of the storage tanks should be so chosen if possible that gravitation of their contents to the kettles will be possible.

The geographical location of the plant will dictate whether or not it is better to locate the storage tanks on the top floor of the compounding plant, or in the basement, or if they are to be placed in the open, and the necessity for lagging the transfer lines or providing them with steam jackets in order that fats and viscous oils will not congeal in them during cold seasons.

In the layout of a grease plant attention should be given to fire and explosion hazards. If fire heated kettles are included, it may be best to locate them in separate buildings away from the main plant or, in any case, provide adequate reinforced concrete fireproof rooms for such mixers. Grease plants are in operation having from one to five stories. A plant of three or four floors is considered best as advantage may be taken of gravity in transferring raw materials to the soap mixers or autoclaves, from the autoclaves to the open mixers, and from the mixers to the filling apparatus. In a grease plant of only one floor the kettle outlet may be very nearly level with the floor and a rotary pump must be used to fill barrels and can fillers. By locating the kettle on a mezzanine floor the kettle may be emptied by gravity. Since, in most cases, a grease filter is now employed, which is fed by a pump, the advantages of drawing the grease by gravity are not as great as might be expected.

Before discussing the individual pieces of equipment in the grease plant general descriptions of a few representative plants will be presented from which the reader may become familiar with typical installations:

English Grease Plant

This plant is located on a three-acre site and was designed for a throughput of four to five million pounds per year of all types of greases. Adequate provisions for extensions and expanded facilities were made. The storage shed is so located that one side adjoins the four storied reinforced concrete grease plant, and the other is contiguous to the railroad spur. A short roadway to the gates of the site and turning ground for motor vehicles is provided, also low ramps are placed at convenient locations around the plant and storage shed to ease the transfer of incoming supplies and shipment of greases. The building is broadly divided into three parts; a boiler house, manufacturing bay, and storage shed for full and empty packages. The three portions are all adjacent, with the manufacturing bay between the other two.

The boiler house is a single-story building, 30 feet by 45 feet long, containing one oil-heated Cochran boiler rated at 1000 pounds of steam per

[1] *Chem. Met. Eng.*, **32**, 494.

hour, with necessary feed pumps and auxiliaries. There is room for an additional boiler when required. An oil heater of the ordinary tubular type, having 900 square feet of heating surface, is provided for supplying indirect heat to a high temperature grease kettle and the autoclaves.

The manufacturing apparatus is placed in a single bay, 20 feet by 90 feet long, having four floors. The ground floor is used exclusively for filling packages, except for a 2000-gallon underground slop tank and the experimental laboratory facilities. On the first floor are five open grease mixers: two of 1000-gallons capacity, two 650-gallon and one 300-gallon tilting kettle. These are all fitted with jackets for steam heating or cooling with water. On the second floor (the floor next above) are two autoclaves arranged for oil heating in the jackets. These are of 500-gallon and 300-gallon capacity, feeding into the two 1000-gallon and two 650-gallon open mixers, respectively, placed on the floor below. Also on this floor (2nd) is a 30-foot by 9-foot storage tank for fuel oil employed for heating purposes and also as an ingredient of black greases. There are also five 1000-gallon tanks for refined lubricating oils used in grease making.

The third floor supports the steam-jacketed measuring tanks; two 300-gallon, and three 500-gallon, one of which is reserved for the measuring and mixing of black oils used in manufacturing dark greases. The other tanks are connected in pairs to the autoclaves, the 300-gallon vessels to the 300-gallon autoclaves, and the 500-gallon vessels to the 500-gallon autoclave. Just above each pair of measuring tanks is a small (50-gallon) hydrated lime mixing tank, and a 100-gallon caustic soda mixing vessel. Efficient mixing of the contents of these measuring and mixing vessels is insured by the use of portable electrically driven stirring devices.

A platform elevator is provided for transferring greases and ingredients from floor to floor. No bulk storage tanks for fats is provided for in this plant. Their normal storage place is the first floor, from which they are moved to the third floor, a steam coil inserted to melt the fats, and drum contents pumped to the measuring vessels. The autoclaves and open steam-jacketed kettles are each fitted with individual electric motor drives and stirring devices. The stirrers are of the double acting type, two sets of paddles rotating in opposite directions, the outer set being fitted with spring steel scrapers attached to the periphery in such a manner that they scrape the inside of the kettle, preventing the accumulation of soap lumps and assisting thorough mixture of the entire kettle contents.

The manufacturers of mixers of this kind (W. J. Fraser & Co., Ltd., Dagenham, Essex, England) claim that they are capable of larger outputs of more transparent grease, with greater accuracy of control and consistency. They are provided with large valves for quickly transferring the kettle contents and are specially riveted to enable the jackets to alternately take steam or cold water for cooling.

For filling the finished greases a portable rotary pump is coupled to the outlet of the kettle and the grease pumped through a large area screen filter. An automatic grease filling machine is used for filling one, five, seven and ten-pound cans. When the saponified soap base is discharged from the

autoclaves to the open mixers large volumes of steam are liberated. This is removed with the aid of a cowl and ventilating pipe, the suction being created by an electrically driven fan. The finished greases are moved to and stored in a shed, 40 feet by 120 feet long, which has a concrete floor and is equipped with steel racks for drums of various types of grease. Easy access is provided to this storage shed from the loading ramps through double leaf sliding doors. A 2000-gallon slop tank is situated underground, and is used for dumping any off-grade or deficient grease. When it is desired to rework the slop grease, a pump and line are used to transfer it to any of the measuring tanks on the top floor. The slops may also be pumped to the fuel oil storage, if desired, and from which it may be run to the boiler house and burned.

For experimental work, a 15-gallon autoclave is available on the first floor, also a 60-gallon steam-jacketed mixer and a 25-gallon gas-heated mixer. This equipment may, of course, be used for the preparation of very small commercial batches for special purposes. For extremely high melting point greases, which may be difficult to fill into packages on account of the high friction occurring in the filling pipes, a 300-gallon tilting kettle is used thus avoiding any trouble from this source. After the constituents of the required grease have been measured in one of the sets of measuring tanks, they are run directly to this mixer, saponified, finished and poured into trays or containers to cool.

Stothert & Pitt pumps are used throughout the plant for moving fluids. These are used for pumping incoming oils from railway car discharge points. All steam lines are 4 inches in diameter, water lines are 3 inch, oil lines are 3 inch, and the ventilating main is ten inches in diameter. Some American plants, making use of the autoclave, obtain somewhat greater flexibility by permitting the pressure vessel to serve from three to even eight open mixers, and increase the size of the autoclave to 750 gallons. It is plain that there is a greater opportunity for time saving when the soap for several batches of grease can be prepared at one time and that the investment involved per pound of grease produced would be less. However, where the soap base is split between several kettles, there is some loss of control unless a meter for accurately measuring the hot soap from the autoclave can be developed.

The autoclave and mixers described above have a capacity of 27,000 pounds of cup grease in four hours. A 750-gallon autoclave and five 1000-gallon open mixers will produce 45,000 pounds of similar grease in the same time. In the forgoing plant entire soda base greases are charged to the autoclave and cooked for about two hours at 170° C. (338° F.) and are drawn into an open mixer over a period of about one hour, where cooling proceeds rapidly at first, and is finally ready to fill after about six to ten hours.

Oklahoma Lubricating Grease Plant

Lincoln and Showell [2] have described a grease plant which may be considered representative of modern practice in the United States. This plant

[2] Lincoln, B. H., and Showell, P. S., *Oil and Gas J.*, 26 (March 24, 1932).

was designed to package and ship 12,000,000 pounds of grease per year working with ten men, one eight-hour shift per day. With two shifts per day the production of grease can be doubled (24,000,000 pounds per year). The plant is housed in a three-story building with full basement. The first floor provides 12,320 square feet of working space. Twenty tanks, ranging from 12,000 to 21,000 gallons, are adjacent to the plant and are used for the storage of all fats and lubricating oils used in the greases. The tanks are provided with closed steam coils for heating the ingredients to temperatures above atmospheric in order to prevent solidifi-

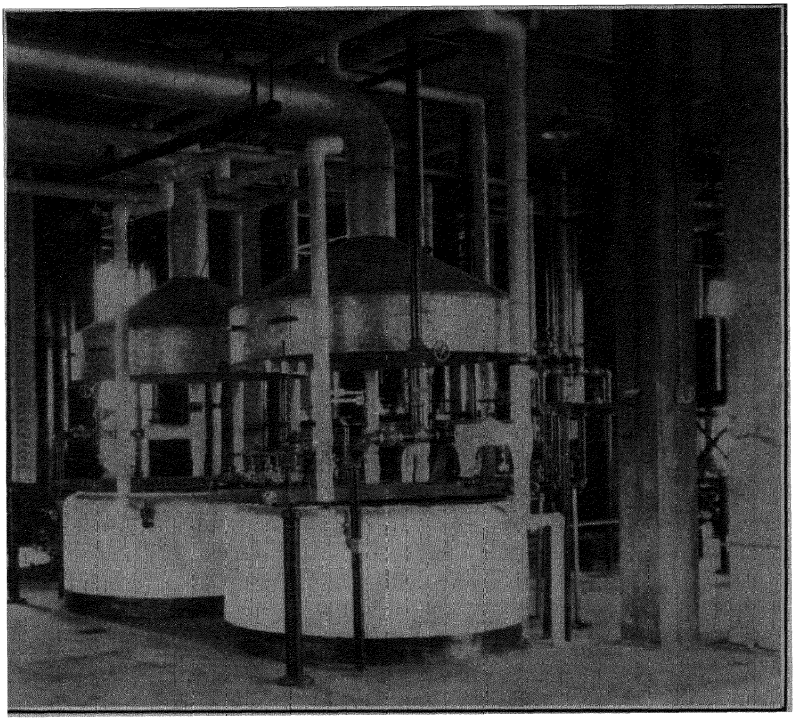


PLATE V1.—View of modern grease kettles, showing ventilating system and cowls for vapor and fume removal

cation. All transfer pumps are located in the basement and have individual motor drives with remote control from either the second or third floors. On the third floor are storage for cardboard cartons, an accurate scale for weighing all fats and fatty acids, and a large rectangular tank on trucks which is used in conjunction with the scales for measuring the fats. The autoclave is equipped with a swing pipe through which soap may be discharged to any one of three finishing kettles, on the second floor. Also, on the third floor are two kettles for mixing axle grease ingredients, which flow to a proportioning pump on the second floor where the ingredients are brought together and fed to an automatic can filling machine.

All of the mixers open on the second floor. They are of Dopp manufacture and equipped with double motion bridge type agitators (paddles) and adjustable spring steel scraper blades which sweep the entire inner surface of the kettle. The capacities of these kettles are:

One	1700-gallon, Jacketed, Steam Heated.
Two	1000-gallon, Jacketed, Steam Heated.
Two	1000-gallon, Single shell, Direct Fire Heated
One	200-gallon, Single shell, Direct Fire Heated.

In addition, there are three axle grease and gear compound kettles, a 300-gallon autoclave, and nine 850-gallon oil measuring tanks provided with gauge glasses calibrated in gallons. They discharge by gravity into any of the grease mixers. All of the finishing mixers are equipped with agitators, driven by individual electric motors through direct connected speed reducers, and double silent chains from individual jack shafts, all mounted on a mezzanine framework directly behind the kettles and on the first floor. The drive chains pass through slots in the floor to the agitator drive shafts. The two fire-heated kettles are provided with General Electric multiple-speed motors, permitting four speeds ahead and four in reverse. This is considered necessary in view of the widely different viscosities of the soda soap greases handled in high heat equipment.

Each of the mixers on the second floor is provided with a vapor cowl which feeds into a common vapor header. The pipe connecting each cowl with the header is provided with a close fitting damper for use when a kettle is not in operation. An electrical motor driven fan furnishes suction by means of which vapors and fumes from the kettles are drawn from the kettles, thus permitting a fume-free atmosphere in the grease plant. The main header is equipped with a trap for separating condensed oil, after which it discharges through a stack above the grease plant. An electric elevator is utilized for moving empty containers from their storage on the second floor to the filling machine on the first floor.

A six-point electric pyrometer indicates the furnace flue and kettle temperatures for the two gas-fired vessels. Another serves the five mixing kettles. On the control panels are located pressure gauges showing the steam pressure on all jacketed kettles.

The heating of the gas-fired kettles is controlled by pressure regulation on the gas feed to the burners as registered by manometers on the instrument board. The temperature of the stack gases are reduced to 600° F., or lower, by means of induced draft of cold air. On the second floor is a laboratory test table, such tests as can be made by the grease maker being carried out at this point, the finished products being sent to the control laboratory for final inspection. The grease research and experimental laboratory is located on the third floor, and is equipped with a 30-gallon gas-fired open kettle, a 15-gallon autoclave, and a 50-gallon steam-jacketed finishing kettle.

Each of the finishing kettle discharge lines is equipped with a 60- to 100-mesh per inch monel metal grease filtering screen. A portable, automatic can filling machine may be connected to any of the mixers. It automatically

fills four cans at one time, and caps them, after which they are moved to a casing machine. From the casing machine the cartons of grease are moved by overhead belt conveyor to storage or direct to a box car on the spur track. Portable automatic drum filling equipment may be rolled under the grease mixers when filling drums, and when not in use can be moved to give greater working space. At the rear of the kettles is a roller type conveyor on which the weighed drums are moved to a spiral conveyor and then to the basement for storage.

Grease Plant Layout Suggested by the Sowers Manufacturing Company

The layout shown in Plate VI has been developed for the inclusion of Dopp cast iron kettles.[3]

Lime, caustic, solid fats in drums, etc., are taken by the elevator to the second floor where they are stored until needed. Oils and liquid fats kept in large storage tanks can be pumped to calibrated measuring tanks located at a higher elevation than the grease kettles, so that they can be fed into them by gravity. Some plants prefer to weigh all materials and use a hopper suspended from a scale supported by a monorail going over the kettles. Other plants dispense with both measuring tanks and weighing tank, and pump direct from large outside storage tanks to the kettles. A meter may be put in the line near the kettle, or the kettle may be filled to a certain level.

The finished grease is drawn out of the kettles directly into containers or pumped through a filter before being packaged. It often pays to use a can filling machine.

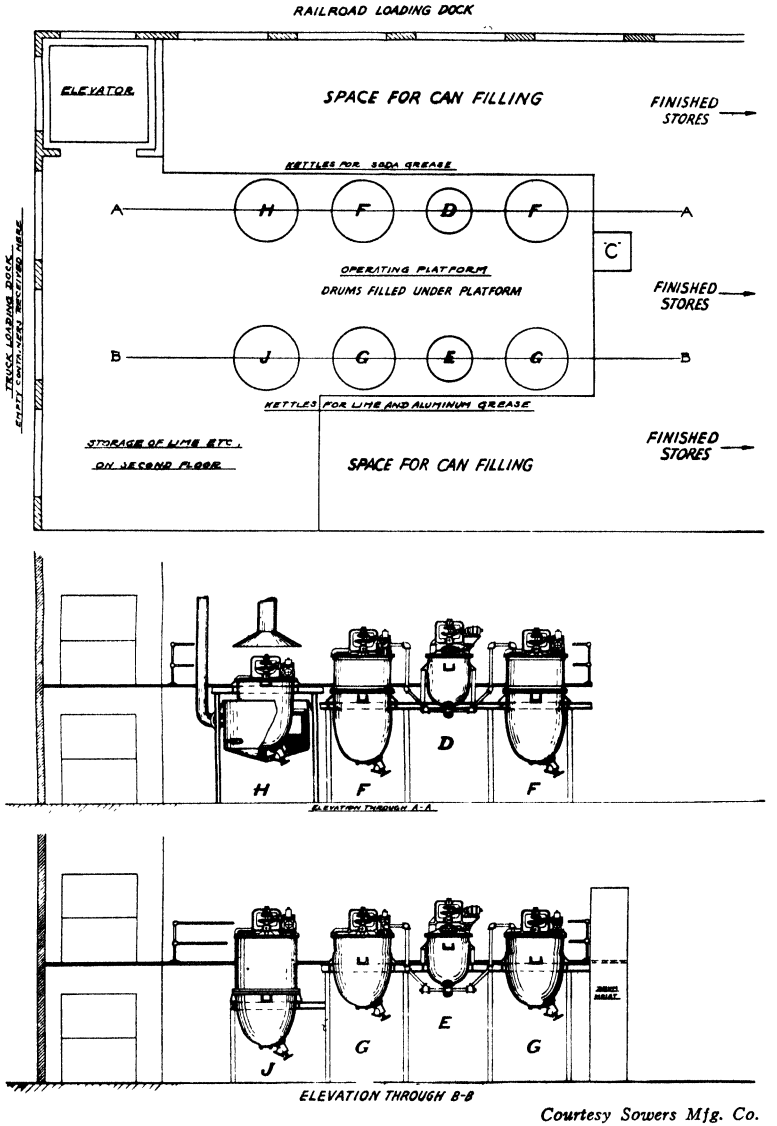
Empty containers are frequently brought to the filling floor on a belt conveyor, but may be delivered by truck. Cans should move in straight line flow to the filling floor and then continue in the same direction to storage or go direct to the railroad loading dock.

Materials are charged and kettles operated from the second floor. So that activities on both levels can easily be co-ordinated, there should be no second floor over the spaces used for can filling at each side of the rows of kettles. To facilitate going from one level to the other, it is well to provide a hoist platform, about 3 feet square, which the men can use as an elevator. This is indicated at "C," Plate VI.

If lime base greases and soda base greases of the types which can be finished in steam jacketed kettles are to be made in quantity, the equipment should include pressure mixers as described on Page 115 for supplying the completely saponified bases, and open kettles for finishing. Pressure mixers are shown at "D" and "E," while the open kettles which they supply with base are shown at "F" and "G." The base is transferred by pipe from the pressure mixer to whichever open kettle it is to be finished in.

Cast seamless kettles provided with agitators which positively scrape

[3] Data presented in this section taken from the article: "Grease Making in Dopp Positively-Scraped Kettles." Composite Catalog of Oil Refinery and Natural Gasoline Plant Equipment, 1936 Edition.



Courtesy Sowers Mfg. Co.

PLATE VI—Typical layout of American grease plant.

their steam jacketed surfaces enable greases to be heated to higher temperatures than could otherwise be obtained with steam. By using such equipment, some grease plants have entirely done away with fire-heated kettles, their greases which formerly were prepared in fire-heated kettles now being made with steam. However, some greases having exceptionally high melting points require temperatures higher than steam at practical working pressures will give, and are usually made in fire-heated kettles. Such a kettle,

gas-fired, is shown at "H." There are burners at four points inside a checkerwork of brick which surrounds the kettle. This gives even distribution of heat, avoiding hot spots such as there would be if the flame impinged on the kettle. The outlet for the finished grease, which closes flush with the inside of the kettle, passes through the setting and is insulated from it.

The temperature of grease made in either a steam-jacketed or a fire-heated kettle is best obtained by a thermocouple. For very heavy grease made in an open kettle the agitator is stopped while the thermocouple on the end of a rod is stuck into it. The thermocouple immediately gives the correct temperature whereas a thermometer would have to stay in the grease for a longer time until its bulb warmed up. For lighter greases the thermocouple is permanently installed through the bottom of the kettle and projects into the grease about 1½ inches, the bottom blade of the agitator being slotted to clear it. A special design of thermocouple enables the true temperature of the grease to be obtained regardless of the temperature of the steam-jacket or fire-box through which the thermocouple passes. With this arrangement there is no need to stop the agitator for taking the temperature. If desired, a continuous temperature recorder can be used.

Some greases are best made entirely under pressure. Pressure mixers "D" and "E" can be used for such greases, during which time it would be desirable to use the open kettles "F" and "G" for making greases which do not require pressure saponification. With these kettles used with steam and cold water alternately in their jackets and having positive-scraping agitators, some aluminum base greases can be cooled in the same kettles in which they are compounded. This saves a great deal of time and labor, as compared with cooling in shallow pans, and also saves the large floor space required for pan-cooling. If these greases are to be made in quantity, the plant should be provided with full-jacketed kettles as shown at "J," for such a kettle has more than twice as much jacketed surface as one of the same capacity kettles shown at "F." It is estimated that such a full-jacketed, positively-scraped mixer makes a batch of thin aluminum stearate grease and cools it in a little less than four hours, as compared with a matter of two to three days for pan-cooling of the same grease in summer.

Cast iron having low temperature-growth characteristics should be used for fire-heated kettles, because such kettles will not burn out like steel will. The same material, but with seamless jacket construction, is desirable for steam-heated kettles, as this cast-in-one-piece construction eliminates leaks and permits steam and cold water to be used alternately in the jacket, when the equipment is built for such a purpose and piped and operated according to instructions.

Double-motion agitation—two sets of mixing blades revolving in opposite directions—gives intimate contact between fat and alkali; enables oils to be added to grease bases in less time; and gives the milling action which some greases require.

The agitators actually scrape the wall of the kettle. Positive-scraping keeps material from baking on the wall of the kettle. Baked-on material, if not periodically removed by manual scraping, will fall into the batch

and make it lumpy. For high-temperature work the scraping prevents local overheating and carbonization. Positive-scraping also shortens the time required for heating or cooling because oils and fats are effective insulators if allowed to stay stagnant next to a heating or cooling surface. Greases are being heated and cooled with positive-scraping agitators in as little as one-fifth of the time required by an agitator clearing the heating or cooling surface by less than half an inch. Tests have also shown that positive-scraping makes steam savings of up to 50 per cent.

A complete and modern grease plant for manufacturing two and a quarter million pounds of grease per year may be constructed for approximately $75,000.00. No provision is made for high heat kettles nor for the manufacture of aluminum stearate base greases. The following products could be made: Pale Greases 280,000 pounds (Cup Greases, Fiber Greases etc.); Dark Green Greases 1,340,000 pounds (Journal Greases, Gun Greases, Gear Lubricants, Universal Joint Grease, and Wheel Bearing Grease); Black Greases 356,000 pounds; and Axle Grease 254,000 pounds; a total of 2,230,000 pounds per year.

	Estimated Cost
Item 1—One 650-gallon autoclave (Sowers Mfg. Co.)	$ 5,000.00
Pipe, fittings, etc.	300.00
Item 2—Three 1000-gallon Sowers mixers D-bridge agitators, C. I., steam jacketed Drive and fittings	15,000.00
Item 3—One No. 2 galena axle grease mixing machine	1,100.00
Item 4—Can fillers	2,000.00
Item 5—Miscellaneous oil and grease pumps	1,000.00
Item 6—One Toledo filling scales	600.00
Item 7—Four 20,000-gallon fat tanks @ $900.00	3,600.00
Item 8—Ten lubricating oil storage tanks (25,000) @ $950.00	9,500.00
Item 9—Twenty 2000-gallon oil tanks @ $150.00	3,000.00
Item 10—One reinforced concrete building (2½ stories), with elevator	25,000.00
Item 11—Grease screen	500.00
Item 12—Miscellaneous and contingencies	3,000.00
Item 13—Engineering and supervision	5,400.00
Total estimated cost of grease plant	$75,000.00

GREASE KETTLES

Single-Shell, Steel, Direct-Heated Kettles

These kettles are manufactured of suitable boiler plate (usually about ½ inch thick) in sizes ranging from 100 to 2,200 gallons in capacity. These kettles may be obtained in either riveted or welded construction, with single or double-motion paddles, and with flat or dished and concave bottoms. They are the least expensive kettles available and will cost roughly as follows:

Capacity—Gallons	Estimated Cost Range Dollars
100–150	275– 350
300 (47″ diameter, 42″ deep)	325– 450
500 (54″×60″ deep)	485– 600
1000 (72″×72″ deep)	725–1500
1500	1300–1800
2000	1600–1900

Usually these kettles are equipped with stationary baffles and the rotating paddles are set at about 45 degrees. With concave bottoms the price is about 10 per cent higher than for flat bottoms. Welded construction is preferable to riveted for which the above estimates apply. A typical 500-gallon riveted mixer, will ordinarily have $\frac{3}{8}$" steel bottom and a $\frac{5}{16}$" side wall. A 6" draw off may be located in the center of the bottom or at one side to eliminate coking in the draw off line. The vertical agitator shaft should be supported by a ball bearing carried by cross beams mounted at the top of the vessel. On the outside of the kettle should be riveted, or welded, large angle irons for mounting and supporting the kettle in brick work.

Depreciation on such kettles is high as the steel bottoms in contact with the fire are rapidly deteriorated and in many cases have failed, resulting in fires. When they are used in conjunction with wood, coal, or coke as fuel a draft fan should be provided to give better control of heating. Riveted kettles are manufactured by the Sun Shipbuilding & Dry Dock Company, Chester, Pa.; Charles Ross & Son Company, Brooklyn, New York; and P. F. Campbell, Philadelphia, Pa. Welded kettles with either flat or round bottoms are now available from many of the larger iron works throughout the country.

The use of fuel oil as a means of heating such kettles is common, there being many brands of fuel oil burners which are entirely satisfactory. Gas, however, is preferable and the burner developed by the Surface Combustion Company should prove satisfactory. The tangential type burner generally used for brass and lead melting furnaces may also be employed with gas. It consists of an industrial type gas burner mounted in the fire-clay wall of a circular furnace in such a way that the flame will strike the kettle tangentially near the bottom. In the usual construction two such ports are placed on opposite sides of the kettle. The disadvantage of this system is that not all of the available kettle heating surface is utilized and hence the system mentioned above is probably superior.

Plate XVI, page 135, shows the installation of a typical single-shell kettle, mounted in brick and fired with oil. Plate XV, page 134, indicates the general arrangement of a grease still found in some American grease plants.

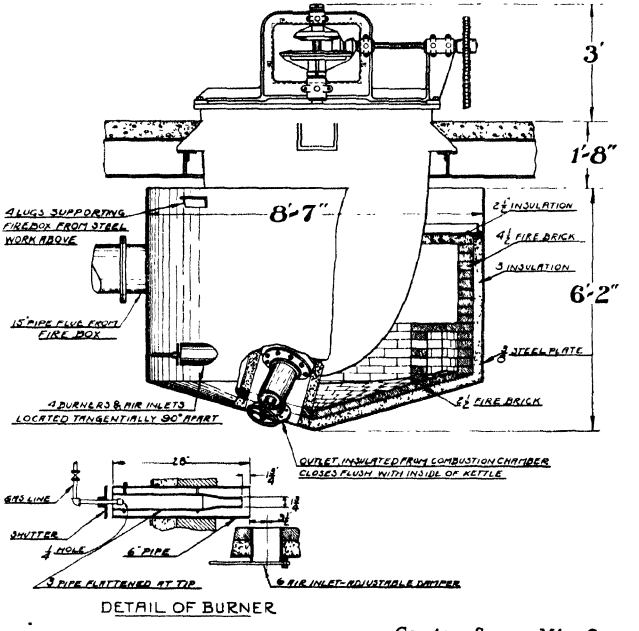
Cost. The estimated cost of a single 1000-gallon direct-fired kettle, with all auxiliaries erected in a building, 30 feet by 70 feet, is roughly $37,000.

	Labor	Material
Earthwork		
Moving scrap from site, cutting back bank and changing road	800	120
Excavation for building and retaining wall	250	
Concrete		
Building foundation and 110-ft. retaining wall	740	370
30′×70′ concrete floor, 6" slab	320	320
30′×30′ reinforced concrete second floor	550	220
Foundation for furnace for kettle	265	105

	Labor	Material	
Brickwork			
Floor drains and 180 ft. of 6″ drain to main 10″	170	110	
Brickwork for furnace and stack	365	460	
Main Equipment			
1 6′×6′ 1000-gal. mixer tank, with stirring mechanism for direct-firing	300	1,500	
Texrope drive for kettle	100	200	
1 15-H.P. 875 R.P.M. 440-volt motor and starter with base and Texrope pulley	50	375	
1 8″ steel draw-off valve with reducer for bottom outlet	25	350	
12 ft. flexible metallic hose for grease		75	
80 27″×33″×6″ grease pans, No. 10 black iron	20	520	
1 set of platform scales, portable		75	
Installation of 2 oil burners, heaters, and fuel pump	150	650	
2 6′×6′ 1200-gallon measuring tanks	300	700	
1 12′ lift, 1000-pound capacity, worm drive power stacker for lifting new material to second floor	25	725	
1 40-Bbl. fuel tank	50	175	
Equipment and steam coils for fuel tank	100	100	
1 30′×70′ steel and corrugated iron building, 12 ft. high, with 30′×30′ second floor 8 ft. high	300	6,000	
Unlisted small items	250	500	
Steelwork			
30 single pipe supports for 2″ steam line (steel hangers on 6′×6′ R. W. posts)	300	90	
Steelwork on furnace, doors, etc.	125	250	
16″ dia.×25 ft. high stack, No. 10 black iron, including erection and guying	125	120	
Sheet metal vapor vent from kettle	100	75	
Runway at furnace, supports for measuring tanks, etc.	100	200	
Support for grease pans	100	50	
Carpenter Work			
Scaffolding for brickwork and lines	150	75	
Electrical Work			
Connections to 440-volt line	125	200	
Lighting, including 10 KVA transformer	200	300	
Piping			
3″ and 2″ fuel piping	300	500	
2″ steam line from tank No. 26, 500 ft.	225	800	
700 ft. 3″ cylinder stock line	350	575	
650 ft. 3″ 1000-red oil line	325	550	
800 ft. 1½″ galv. fresh water line	210	175	
500 ft. 1½″ H.P. air line	100	125	
Internal piping and manifolding inside of building	250	500	
Insulation			
500 ft. of 2″ double 85% magnesia with weatherproof covering for steam line	175	250	
Painting			
Pipe lines, tanks, steelwork	130	45	
	8,520	18,530	27,050
20% Contingencies			5,400
15% Engineering, overhead and supervision			4,850
Estimated Cost			$37,300

Single-Shell, Cast Iron, Direct-Heated Kettles

The most generally used cast iron kettle, of open-top construction for direct-firing by gas or oil, is that manufactured under the "Dopp" brand by the Sowers Manufacturing Company. This manufacturer claims the use of an extremely dense close-grained iron having a tensile strength of 35,000 to 45,000 pounds per square inch. It is this manufacturer's further claim, substantiated by the author's experience, that iron of this quality is less subject to burning and deterioration than normal steel. The moulded Dopp iron kettle is preferable to a riveted-steel kettle in that its walls are smooth surfaces which permit the use of spring steel scrapers. The use



Courtesy Sowers Mfg. Co.

PLATE VII.—Method of Installation of Single Shell Cast Iron Kettle for Manufacture of Sodium Soap Greases by Direct Gas Fire.

of these scrapers has been shown to materially shorten the time for the kettle contents to reach a given temperature. They also assist in preventing local overheating and burning of material that would normally stick and scorch on the kettle wall were such scraping not employed. A typical mounting of the Dopp kettle arranged for gas firing is shown in Plate VII.

Supporting lugs [4] are located near the top of the kettle so that the part of the kettle in the zone of most intense heat is free to expand and contract. Typical data from the production of a 5000-pound batch of soda base grease in the 1000-gallon kettle shown in Plate VII is given below:

[4] Publication of the Sowers Manufacturing Co., "Discussion No. 4," Circular 73.

Time	Temperatures °F. Grease	Bottom of Furnace	Upper Part of Furnace	At Fan Suction	Agitator R.P.M. Sweep	Paddles	Total Power as kw.	Gas Cu. Ft.
6:30 A.M.					12	20		
7:06		115			12	20	1.1	
7:30	125	155	560	85	12	20	1.5	310
8:00	165	190	640	90	Down	Down	0.9	310
8:30	180	250	735	100	Down	Down	0.4	440
9:00	200	280	750	110	Down	Down	0.3	370
9:30	220	325	775	120	Down	Down	0.1	360
10:00	225	350	790	130	12	20	1.2	365
10:30	235	370	800	135	12	20	3.0	360
11:00	240	365	785	135	12	20	2.8	315
11:30	300	400	830	145	12	20	1.9	395
12:00	335	425	840	150	12	20	1.3	375
12:30 P.M.	355	435	830	150	12	20	0.7	360
1:00	360	450	830	150	6	10	0.6	340
1:30	365	450	835	150	6	10	0.7	
1:45	380	450	835	150	12	20	0.3	340
								170 cut fire
2:30	345	315	400	120	12	20	1.5	4800 total
3:00	340	285	330	110	12	20	0.9	
3:30	340	270	300	110	6	10	1.0	
4:00	335	260	270	105	12	20	0.9	
4:30	330	250	255	100	12	20	0.9	
							22.0 total	

The melted fat, caustic soda solution, and one-half of the final mineral oil content are run into the kettle, the agitator started and after a few minutes of preliminary mixing the heat is increased. Shortly after this the batch becomes too stiff for the agitator to run continuously so during this period the paddles are operated intermittently as sufficient melting takes place. After the required maximum temperature is reached (380° F.) and dehydration completed, the fire is drawn, the balance of the oil added and the completed grease drawn off. The apparently low furnace temperatures shown in the table are due to the use of a large excess of air with the gas, the average CO_2 content being only 3.6 per cent. While this method of firing may be inefficient from a thermal standpoint, it is not a serious matter as the consumption of gas is quite low, only 4800 cubic feet being burned for the above batch. For this plant natural gas having 1250 Btu. per cubic foot was available at only 8c per 1000 cubic feet.

Electrically-Heated Cast Iron Kettles

It is sometimes difficult to decide what form of direct-heat kettle to install as many factors enter in, such as availability of space in present grease plant, fire hazard and cost. The use of fuel-oil heating is sometimes irregular and produces localized hot spots. There is probably less danger from this source with gas-firing. Gas is much cheaper than electrical power for heating

purposes. If the Btu. value of gas at 7c per 1000 cubic feet is only realized to the extent of 50 per cent and electric power is considered as being 100 per cent effective, the seven cents worth of gas would be equivalent to $1.25 for the same effective heat from electricity.

Following is a cost estimate for a 500-gallon cast iron electrically-heated kettle, equipped with spiral-ribbon type agitator (15 R. P. M.) and involving the installation of thirty-six G. E. heating elements (3.8 kw. at 110 volts); total is $14,355.

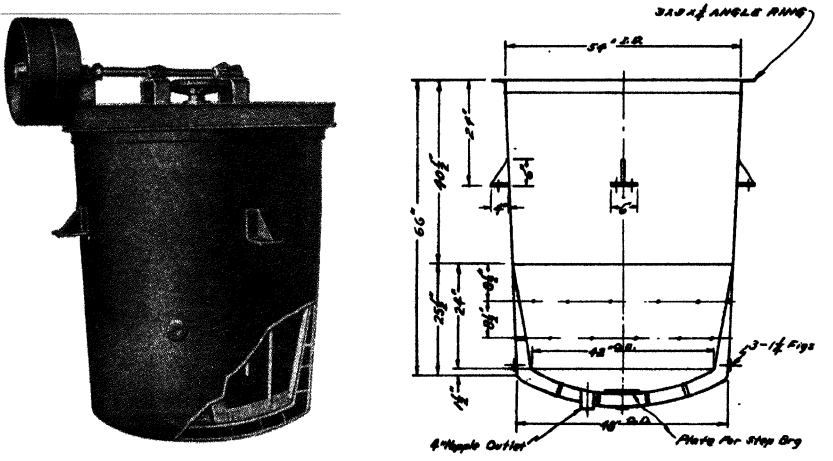
Concrete Work			
Assuming kettle is installed after bldg. floor is completed		$ 275	
Main Equipment			
500-gal. kettle with stirring mechanism and motor	@ $4,000		
G. E. heating elements	1,900		
Steel housing with insulation	600		
Pans, moulds and grease filling equipment	2,000		
		8,500	
Steel Work			
Supports for kettle and pipes		525	
Carpentry		50	
Electrical Work			
Connecting motor and heating elements		400	
Piping		600	
Painting		35	
Subtotal		10,405	
20 per cent Contingencies		2,080	
		12,485	
15 per cent engineering, supervision, overhead		1,870	
Total Estimated Cost			$14,355

Jacketed, Steel Kettles for Steam Heat (or indirect heating)

Steel kettles, jacketed for steam, are seldom standardized as to design, type of riveting, thickness of the boiler plate, shape of paddles, size of outlets, and maximum safe working pressure in the jacket. Since they are not available generally as standard stock apparatus they are usually built to order. Kettles of this type are manufactured by the Charles Ross & Son Company, Brooklyn, New York; P. F. Campbell, Philadelphia, Pennsylvania; and F. E. Martin & Company, Lake Bluff, Illinois. A Martin steam-jacketed grease mixer is illustrated in Plate VIII. It has a 4-inch draw-off near the center of the dished bottom, $\frac{5}{16}$-inch steel plate jacket, tested for 85 pounds of steam per square inch, sides of shell $\frac{1}{4}$ inch thick, agitator shaft $1\frac{5}{8}$ inches in diameter, drive pulley 24 inches in diameter, the weight being 2250 pounds. Kettles of this type are frequently employed by small compounders who manufacture a limited line of products. Vertical side kettles provided with dished bottoms, steam outlets, 4-inch draw-off con-

nections, extended-arm type paddles, and tight and loose pulleys suitable for the small compounder have the following dimensions:

Capacity gals.	Diameter in.	Height in.	Weight lbs.
200	36	48	850
300	42	48	1,150
400	48	48	1,450
500	54	48	1,600
600	60	48	1,775



Courtesy F. E. Martin, Lake Bluff, Ill.

PLATE VIII.—500-gallon steam jacketed grease kettle.

Ross manufactures a kettle, jacketed on sides and bottom, from ½-inch plate, riveted and caulked, stay-bolted and tested at 100 pounds per square inch. Single-motion, horizontal paddles set at an angle with steam fittings and tight and loose pulleys. The dimensions of these kettles are:

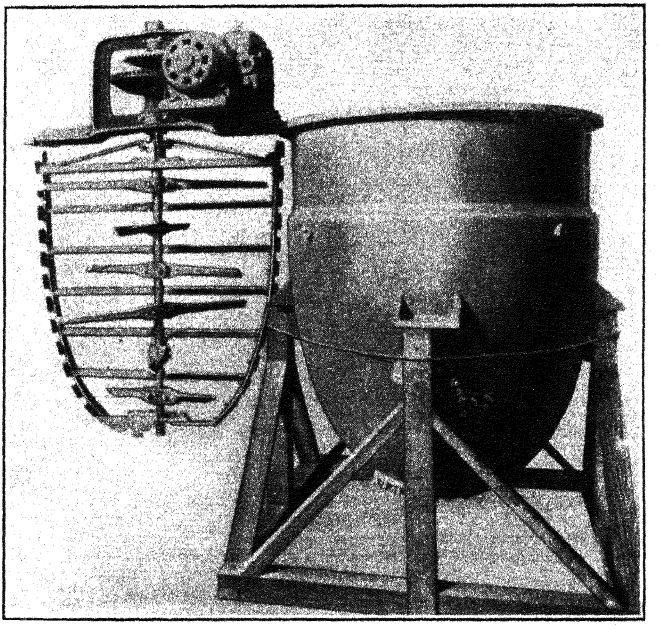
Sizes gals.	Floor Space Long in.	Wide in.	High in.	Weight in Pounds On Skids	Boxed
50	46	35	36	850	1,000
135	52	41	50	1,300	1,500
200	63	54	55	1,675	1,925
300	68	58	58	2,075	2,350
500	89	71	65	3,100	3,550
700	89	71	77	3,400	3,900
800	89	71	89	3,900	4,500

Open-Jacketed, Cast Iron Kettles for Steam Heat (or indirect heat)

Steam pressures of 100 to 150 pounds per square inch in kettle jackets are now common practice in many American grease plants. When making soda base greases, dehydration is often difficult if sufficient heat is not available and, for this reason, many grease makers have felt that fire-heated kettles were preferable to those heated by steam. If it is not necessary to

melt the sodium soap, as in the preparation of smooth soda base greases, sodium base fibre greases are just as well produced in steam-heated kettles as in fired kettles. Another misunderstanding has arisen from statements that fire-cooked lime base greases were superior to steam-cooked greases. Since the temperatures in each process are well within the limits of steam cooking the advantage of fire-saponified cup greases is not at all apparent.

Many grease companies have found that fibre greases can, in most cases, be made just as well, if not more uniformly, in steam-jacketed kettles. For the production of sodium base greases of high melting point and smooth texture hot oil circulated through the jacket will provide adequate temper-



Courtesy Sowers Mfg. Co.

PLATE IX.—Cast iron jacketed grease kettle equipped with double motion agitator.

atures. The most outstanding make of cast iron kettles is the Dopp brand, manufactured by the Sowers Manufacturing Company, Buffalo, N. Y. These kettles are usually provided with two separate sets of paddles which revolve in opposite directions. The outside sweep and the paddles, or cross pieces attached to the same, are driven by a bevel gear through a hollow shaft. Center paddles are attached to a central vertical shaft, which passes through a hollow shaft or sleeve, and are driven by a small bevel gear attached to the top of the shaft. This small gear is inverted with respect to the large bevel gear which drives the sweep, and is one-half the size of the large bevel gear; consequently, the two sets of paddles revolve in opposite directions, the central paddles at twice the speed of the sweep. The central

vertical shaft extends downward through the bottom of the sweep where a suitable step bearing is provided. Steel scrapers, adjustable for tension and wear by means of screws, alternately arranged, are attached to the outside sweep and completely scrape the jacketed inner surface of the kettle. This form of construction is clearly shown in Plate IX.

These kettles are cast in one piece, the inner-kettle walls, jackets, and reinforcing connections or "staybolts" between outer and inner walls are integral. The inner shell is as thin as possible in order to permit rapid transfer of heat. The cast iron kettles may be obtained in capacities of 10 to 1000 gallons, the 1000-gallon kettle costing approximately 5000 dollars. The following agitator speed data will be of interest:

Paddle Speed

Outside sweep	10 to 15 R.P.M.
Center paddles	20 to 30 R.P.M.

Gear Ratios

Capacity, Gallons	Outside Sweep	Center Paddles
10 to 50	2 to 1	1 to 1
65 to 400	3 to 1	1½ to 1
50 to 1000	5 to 1	3 to 1

Tight and Loose Drive Pulleys.

Capacity, Gallons	Pulley size, inches
10 to 40	14×2
50 to 100	14×3
125 to 200	14×4
250 to 300	16×5
350 to 400	20×5
500 to 1000	24×5

To find the size of pulley to use on lineshaft, multiply the desired R.P.M. of the mixer pulley by the diameter of the mixer pulley and divide by the R.P.M. of the lineshaft.

Example: Desired agitator speed is 20 R.P.M. Gear Ratio is 3 to 1, therefore mixer pulley speed should be $20 \times 3 = 60$ R.P.M. Pulley on mixer is 14" diameter. Lineshaft speed is 120 R.P.M. What diameter of pulley should be used on lineshaft?

$$\frac{60 \times 14}{120} = 7''$$ which is diameter of pulley to use on lineshaft.

If lineshaft or motor runs so fast that it would not be practical to belt direct from it to mixer, an intermediate countershaft must be used. To determine size of pulley (B) on countershaft which is to be driven by pulley (A) on lineshaft or motor, multiply R.P.M. of pulley (A) by square of diameter of pulley (A) and divide by the desired R.P.M. of the mixer pulley and by the diameter of the mixer pulley.

Example: Pulley on mixer is 14" diameter, desired speed is 60 R.P.M. Pulley (A) is 5" diameter, speed is 900 R.P.M. On the countershaft what is the diameter of pulley (B) that is to be driven by pulley (A)?

$$\frac{900 \times 5 \times 5}{60 \times 14} = 26.78''$$

With 26" pulley for (B), countershaft speed will be 173.46 R.P.M. For driving the mixer use pulley on the countershaft of the same diameter as (A), namely 5", which will make the pulley on the mixer run at 61.95 R.P.M.

Cast Iron Autoclaves (Steam-Jacketed)

Pressure saponification has passed through its experimental and trial stages and is now the accepted procedure not only in the largest grease plants making the bulk of the more common greases such as cup grease, but also in the smaller plants whose production is directed more to special greases.

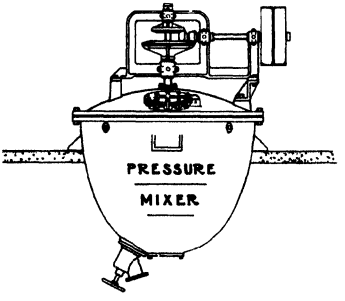


FIGURE 1

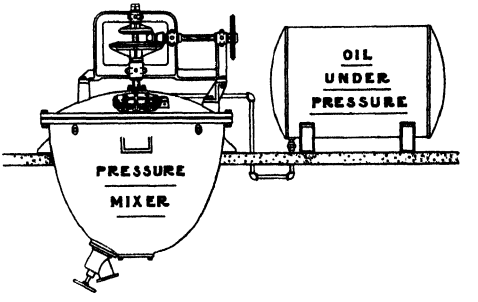


FIGURE 2

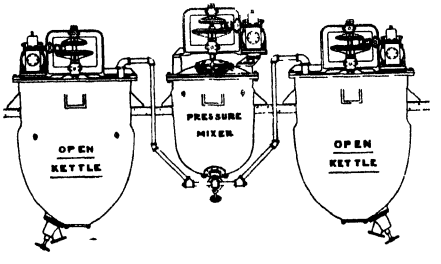


FIGURE 3

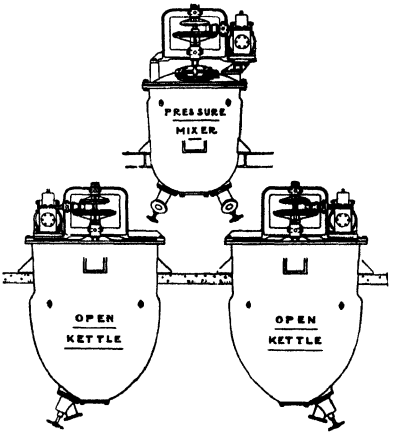


FIGURE 4

PLATE X.—Various arrangements of autoclaves for pressure saponification and Grease Making.

Figure 1, Plate X, shows the simplest installation of a pressure mixer, in which both saponification and finishing are taken care of in the one unit. When excess moisture is not produced by the reaction, and when the oil is such that it is not darkened by prolonged heating, everything necessary for the finished grease may be charged into the pressure mixer, the unit closed, steam turned into the jacket long enough to give the desired reaction and then the steam turned off and cold water circulated through the jacket until the grease is cool enough not to lose moisture, or if desired, until the grease is cool enough to be packaged.

With this procedure nothing is left to the judgment of the operator and with the same raw materials each batch is nearly identical. On the other hand this simple procedure is not generally followed because the large percentage of oil present makes the saponification take longer than when only the base is under pressure, and the daily output per dollar invested in equipment is very much less than with the set-ups shown by Figures 3 and 4, Plate X.

Sometimes only the base is cooked under pressure, and the oil worked in after saponification is complete. In this case the oil may be added after the internal pressure has been released. Certain special greases are made by adding the oil while the base is under pressure, using the arrangement shown by Figure 2. The latter is desirable when the moisture content of the grease must be very carefully controlled.

Figure 3 shows the most common arrangement of a pressure mixer and open kettles, which is ideal for a two-story grease plant. Here only the soap is made under pressure. The pressure mixer is charged with the fat, the saponifying agent (usually a solution or suspended in water or oil) and, in some cases, a little oil so that the base will not become too stiff. Then the unit is closed, the agitator started, and steam turned into the jacket. One large pressure mixer works satisfactorily with steam at only 80 pounds pressure, but most grease plants have steam at 100 to 150 pounds pressure which causes an internal pressure of 50 to 85 pounds to be developed. Under these conditions saponification takes only 15 to 20 minutes although the usual practice is to take off a 6-barrel batch of base every 1 to 2 hours, with larger batches requiring somewhat longer time.

Upon opening one of the flush-closing outlets the base is blown into one of the open kettles, much of the moisture in it flashing into steam as the pressure is released. Then, while oil is being added to the first batch of base, a second batch of base is saponified under pressure. The pressure mixer does not require cleaning, even when changing from lime to soda base or vice versa. The second batch of base goes into the second open kettle, and by the time that the third batch of base is saponified the first open kettle is again empty and ready to receive it.

When the finishing is done in kettles with double-motion, positive-scraping agitators only 1½ to 3 hours is required for adding oil to a lime base. In practice, equipment set up as shown in Plate X regularly turns out two batches of finished grease per open kettle per day, but when necessary produces three batches per open kettle per day, as compared with only one batch of the same grease per kettle per day when not using a pressure mixer.

In Plate X, Figure 4, the base remaining in the lines after the pressure mixer is empty drains by gravity to the open kettles, but it has proved entirely practical to install a pressure mixer on the same floor as the open kettles as shown by Plate X, Figure 3. The self-generated pressure developed by heating the base is sufficient to force it up and into the tops of the open kettles. Then while the pressure mixer is saponifying the next batch the operator removes two plugs and pushes a rod through the bottom horizontal length of pipe to remove the small amount of base which drains back

down to that point. This prevents the eventual clogging of the line with base.

The use of pressure saponification not only improves the uniformity of the grease, reduces the chance of lost batches and permits grease manufacture by less experienced operators, but also further reduces manufacturing cost by cutting down the "cost-per-pound-of-grease" that must go for equipment, maintenance, steam, power and labor.

Satisfactory cast iron autoclaves are available in sizes up to 1000 gallons from the Sowers Manufacturing Company. As in the case of the open mixers these are cast in one piece, the inner shell, jacket and staybolts being integral. They may be equipped with either single or two-way agitation, and normally have the adjustable steel scrapers which contact the inner surfaces of the kettle and promote heat transfer. The power for the agitation is best supplied by means of an individual electric motor drive, preferably through a short composition rope drive. The cast iron cover is bolted to the top of the kettle and the entire vessel will readily operate at internal pressures up to about 80 pounds per square inch, the normal hydrostatic test being made at 160 pounds per square inch. In the cast iron cover is a 14-inch by 18-inch manhole, a six-inch vapor outlet, and four service connections tapped for 1½ inch pipe.

Steel Autoclaves

Either riveted or welded steel autoclaves are available from various iron works in sizes ranging from 100 to 1500 gallons. It is now possible to secure welded round-bottom kettles with smooth inner surfaces free from the objections due to rivet heads and seams as in the case of riveted kettles. Such kettles can be manufactured to withstand extremely high jacket temperatures and internal pressures. In some cases they may be made with thinner walls than in the case of cast iron kettles, and consequently may be quicker to heat and cool.

If the riveted, steel kettles are made of very heavy plate they are not subject to rapid depreciation, but, due to dead spaces near the edges of seams and around rivet heads, are responsible for some contamination when alternately preparing sodium and calcium base greases.

Axle Grease Mixers

Any grease plant manufacturing over (about) 250,000 pounds of axle greases per year, from rosin oil and lime sett, will find it desirable to install an automatic proportioning system for bringing together the rosin oil mixture and the lime component. It is, of course, quite possible to mix axle greases by hand in a fifty-gallon barrel, but if small containers are to be filled, this means hand work and the automatic machine is most convenient. It ordinarily consists of a specially constructed can-filling machine by the use of which the lime part and rosin oil mixture are thoroughly and

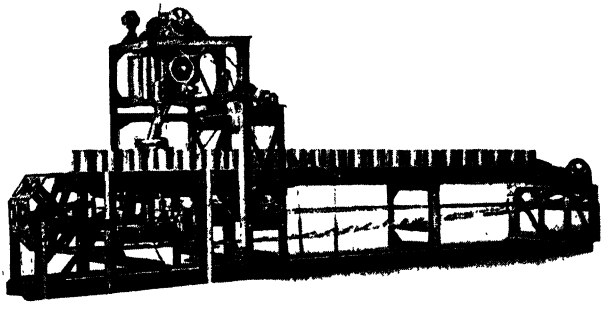
quickly mixed in proper proportions and then automatically filled into the containers.[5]

Tanks equipped with mechanical agitators of the single-motion type should be provided; one for the lime mixture (sett) and one for the rosin oil and mineral oil mixture. These mixers which do not necessarily require provisions for heating, act as feed tanks for the axle grease mixer.

The Galena Manufacturing Company, Galena, Ill., have for many years sold a very satisfactory line of axle grease mixers. These machines have the following capacities:

Machine No. 2. For filling axle grease in from 1- to 7-pound pails.
Machine No. 3. For liquids, semi-solids and axle grease filling in from 7- to 25-pound pails.
Machine No. 4. For filling axle grease in pails, barrels and half-barrels.
Machine No. 5.—For filling screw-top cans with axle grease.

The No. 2 Machine is automatic with regard to mixing, filling and weighing axle grease into various cans of from one to seven pounds inclusive. It is provided with three special rotary pumps, which may be purchased separately if desired, one for delivering continuously the correct proportion



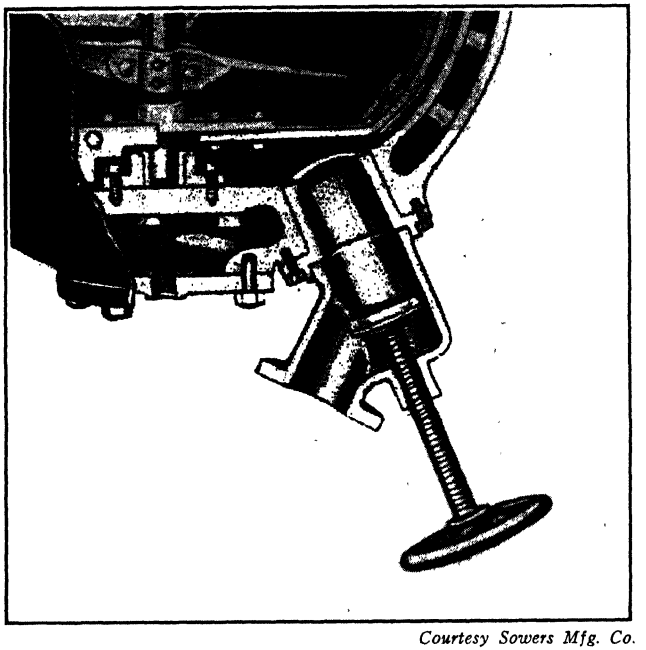
Courtesy Galena Mfg. Co.

PLATE XI.—Filling machine for mixing, filling and weighing liquids and semi-liquids and axle greases.

of rosin oil mixture (prepared oil), and one for delivering the prepared sett, into a small mixing vessel. The third rotary pump, called the grease pump, is located at the bottom of the mixing can for removal of the mixed grease. Its capacity is equal to the combined capacity of the other two pumps. By selection of suitable gears for regulating the speed of the pumps, the quantity of material in the mixing can, and the period it remains there for thorough mixing, may be controlled to meet the requirements of the formula being used. The grease pump delivers a continuous stream through a special cut-off to first one and then another of two filling points or containers. Different proportions of oil and sett may be combined by the use of different gear combinations.

[5] Kauffman, H. L., *Petroleum Engr.*, (Feb., 1931).

The machine is provided with two endless conveyors consisting of flat plates or links with a hole in the center of each. The conveyors travel intermittently and alternately move ahead one link at a time when released by the cut-off. They carry the empty cans to the filling point and the full cans to the point where the lids are put on. A special scale is placed below each conveyor at the filling point, the platform of the scale projecting through the hole in the conveyor and supporting the empty can while being filled. While the package on one conveyor is being filled, the other conveyor is carrying away a filled package and bringing an empty one into position. The upright post of the scale carries a horizontal rod projecting forward. Upon this rod is suspended the scale hook, just above a revolving rachet which then effects the cutting off of the mixed grease supply by power.



Courtesy Sowers Mfg. Co.

PLATE XII.—Flush opening kettle outlet valve.

The cans are filled with reasonable accuracy, and without smearing the outsides. The capacity of the machine ranges from fifteen hundred pounds per hour in 1-pound cans to forty-five hundred pounds in 7-pound cans. Two operators are required; one places the empty cans on the conveyor, and the other covers the full cans and cases them with one handling only. In order to prevent greases from slopping on the inside of the cover, a very quick reacting rosin oil and a suitable formula must be employed.

The machine may also be equipped with an independent hand operated cut-off which will allow it to be used for filling larger packages or barrels placed along the sides of the conveyors on the floor. When the hand

cut-off is used the packages are not filled by weight but are filled by the operator to the desired volume. When furnished for belt drive the tight and loose pulleys are 11 by 3 inches and should be operated at a speed of 28 to 30 R.P.M.

The capacity of the No. 3 machine ranges from forty-five hundred pounds per hour in 7-pound cans to nine thousand pounds in 25-pound pails.

Kettle Outlets

Most steel plate kettles are equipped with a gate valve (4 or 6 inch) in the bottom of the kettle. Where the sleeve for this valve passes through the kettle jacket a pocket of unmixed material remains. Usual practice with such outlets is to pack this space with grease from a previous batch. A more desirable system is the use of flush-opening kettle draw-off valves. These should be located off-center, and away from the agitator step bearing, in order to leave an unobstructed opening. Valves, as shown in Plate XII, have been found most satisfactory.

SCREENS AND FILTERS

In most grease plants, even where autoclave saponification is carried out, it is considered desirable to screen the grease to remove small hard lumps of soap base, gritty particles, and black specks of carbonaceous material which sometimes is found in the lime. Fine screens may be used for thin, soft greases and coarse screens must be used for heavy greases to prevent excessive back pressures. One grease plant uses 60-mesh per inch screen for all grades of cup grease except No. 5 and higher.

Disk or Plate Type Grease Screen. At one grease plant, screen of suitable mesh is fastened between two circular metal plates about thirty inches in diameter. Bolts around the edges of the plates and appropriate gaskets prevent leakage of the grease, which is drawn from the kettle to the suction side of a gear pump and forced through a lagged 3-inch line to the inlet of the screen, which is at the center of one of the plates. Plugs are provided on the inlet side of the screen which may be removed for occasional cleaning, but it is frequently necessary to unbolt the flanges of the plates and remove the screen for thorough cleaning.

From the screen the grease is led directly through a flexible metal hose to the hopper of a can filling machine.

Grease Filter. An elaboration of the single-plate type grease screen is the multiple-plate grease filter developed by the Oceco Division of the Johnston & Jennings Co., Cleveland, Ohio. This device is shown in Plate XIII and it is claimed that it is particularly valuable for removing lime specks, and soap particles from greases intended for high-pressure lubricating systems. The standard size machine is usually installed in the kettle outlet line directly ahead of the filling facilities, and will filter from 150 to 250 pounds of grease per minute. It requires a floor space of 30

inches by 6 feet 6 inches. The character of the material removed from grease by this filter is shown in Plate XIV.

Most of the pressure guns used for applying greases are equipped with fine wire screens and if there is foreign material in the grease these

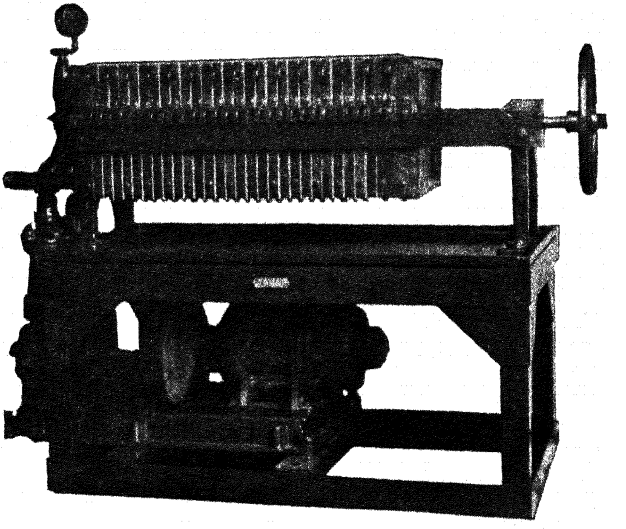


PLATE XIII—Grease filter press.

Courtesy Johnston & Jennings

screens will clog, and no grease will be fed to the force pump. The filter press is equipped with screens of different mesh and is capable of handling No. 4 Cup grease when using coarse screen.

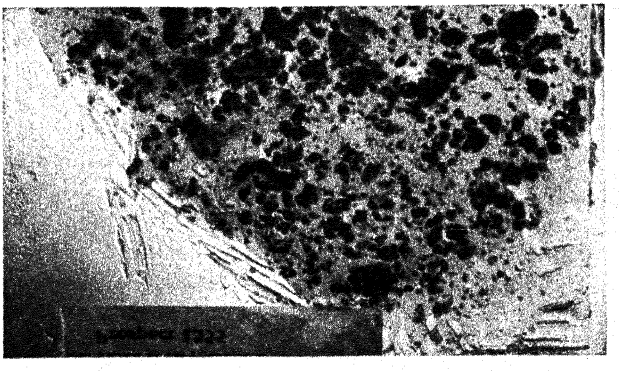


PLATE XIV. — Material removed from grease by filter.

Courtesy Johnston & Jennings

Cylindrical Screen (Non-scraping type). In some grease plants simple thimble-shaped screens are used. Usually two screens are used, the inner one being of coarser mesh. The screens are brazed to light metal frames and enclosed in a 6-inch pipe nipple provided with suitable flanges at the ends for inlet and outlet.

Portable Grease Screen. A combined portable grease pump and screen arranged for scraping has been designed by the engineers of a Pacific Coast grease plant and used for several years with considerable success. Nine-inch truck wheels are attached to a cast iron truck bed which is 4 feet 4 inches long and 3 feet 4 inches wide. On one corner of the bed is mounted a 7½-H. P. electric motor connected through reduction gears to a 45-G. P. M. (against 125 pounds) Northern Rotary Pump, the scraper drive being attached to the outboard end of the pump shaft which turns at 280 R. P. M. A 4-inch flexible metal tubing is attached to the kettle draw-off and the suction of the pump. The pump discharges into one end of the cylindrical screen vessel. The screen of 60-mesh is reinforced on the outside with ¼-inch mesh wire netting and is removable for cleaning or replacement with similar arrangements of different mesh. The inner surface of the screen is scraped with a spiral-ribbon type steel scraper, and is followed directly by another similar scraper the edge of which is about one inch from the screen. By this arrangement of scrapers, tailings, lumps of grease, solid matter and grit are worked to the center of the vessel and are forced out at the axis of the spiral screw scraper, at the opposite end of the cylinder. The filtered grease is forced through the screen and is taken off through an outside jacket and side connection to the point of filling drums, or through a flexible metal hose to a Cornell Grease meter by means of which various cans may be readily filled to accurate weight.

The internal spiral scraper is geared to rotate at 8.75 R. P. M. While the screening surface is only 4½ square feet in area, it is being continuously scraped to remove material which would clog it, and is therefore a very efficient piece of apparatus.

Permanent installations of this kind may be made directly under the kettles which are most continuously in use and will be found to give good service.

GREASE PUMPS

Suitable rotary pumps for handling lubricating greases may be obtained from many manufacturers. Those built by the Northern Pump Company, Minneapolis, and the G. and C. Jacketed Rotary Pump, sold by the Guyton and Cumfer Mfg. Co., Chicago, Ill., will be found satisfactory. Where reciprocating pumps are needed for oils the "Oil Well Supply Company" Duplex pumps are outstanding.

GREASE MILLS

Grease mills are used to give a worked consistency to grease so that it will not decrease in consistency on further working. In addition to this form of stabilization it is often required to mill aluminum stearate base greases and sodium base greases which have been cooked at high temperature and permitted to cool without appreciable agitation. In these cases the mills act as homogenizers, to give a uniform product without heavy or lumpy portions. The Buhr Stone Mills manufactured by the Charles

Ross & Son Company, Brooklyn, are no doubt the most popular make in this country, being in use in many of the outstanding grease plants. The Ross French Conical Buhr Stone Mills are composed of a top stationary stone, a conical running stone, a cast iron jacket and spout for delivering the ground grease, and a hopper of about one barrel capacity for feeding the mill from above. At the throat of the hopper is a spiral worm for assisting the feed of grease to the stones. They are equipped with bevel gear drives from below and tight and loose pulleys. These mills sell for $300 to $1800 depending on the size and condition. They should not be confused with colloid mills which are relatively high-speed equipment. The stone mills operate at a low speed, usually less than 100 R. P. M.

The Ross mills are made in three standard sizes and are particularly intended for smoothing out the congealed lumps in lubricating grease to produce smooth homogeneous lubricants particularly adapted for application in pin cups or other places where a high degree of uniformity is essential. The stone requires only infrequent dressing as there should be no abrasive material in lubricating greases. Grease milling is ordinarily considered as the simple process of smoothing out and obliterating the clots or lumps of heavy grease formed by chilling. The mills are also useful for incorporating graphite with cup grease and similar purposes.

Size.No.	Floor Space	Weight	Capacity, Pounds of Grease per 8 Hours
1	3′ 2″ × 1′ 6″	270	200 to 600
2	3′ 4″ × 1′ 6″	600	100 to 2000
4	4′ 8″ × 2′	1200	2000 to 4000

P. F. Campbell, Philadelphia, also manufactures stone buhr mills which are adaptable to grease making. The hoppers are however intended for dry materials rather than grease and require the installation of a proper feeding device. Stone buhr mills require from 2 to 12 H. P. for their operation, the power depending on the size of the mill and the nature of the grease being processed. The cost of milling grease will range from .30 to .75c per pound.

Types of grease which are particularly adaptable to milling are those having an aluminum stearate base, and the sodium soap base ball bearing lubricants, and automotive wheel bearing greases.

COLLOID MILLS FOR GREASES

A great deal of experimental work has been done with various types of colloid mills or homogenizers. The high disruptive forces in these mills do not appear to be beneficial to many types of grease. Aluminum base greases are heated by passage through the mill to such an extent that they become stringy and gelatinous. Fiber greases are smoothed somewhat but do not lose all of their fibrous characteristics. Normal cup greases are broken down and stable cup greases are smoothed to some extent but encounter great danger from aeration.

It is known, however, that at least one grease plant has successfully adapted a special form of colloid mill to removing the lumps from a sodium base grease cooked at high temperature and cooled in trays. This grease is made with a considerable proportion of petrolatum. After it is cooled it is charged to a large hydraulic grease ram which feeds the mill. Special precautions must be taken to prevent the entry of air to the grease being milled.

Spensley has developed the use of a high speed centrifugal type mill.[6] The mill is provided with rotating disks equipped with intercalating pins. This mill may be used for breaking up lumps in grease, smoothing the texture, or for incorporating powdered materials.

APPARATUS FOR COOLING GREASE

To complete the grease manufacturing process the product should be cooled to a suitable temperature before packaging. Some of the means for accomplishing this are:

In the grease mixer, with or without agitation. Circulation of water in the jacket greatly accelerates the cooling.

Circulation of the grease by means of rotary pumps.

Grease chillers.

Use of special cooling trays.

Cooling the grease directly in the shipping containers, which may vary in size from one pound to one hundred gallons.

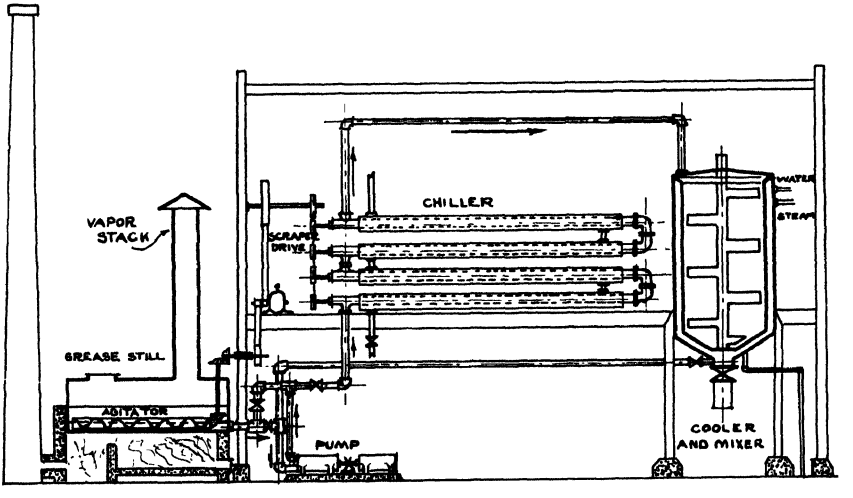


PLATE XV.—General arrangement of grease still and cooling equipment.

Under the heading of chilling machines may be mentioned the Bethlehem Chilling Machine which consists of five vertical sections of 12-inch pipe jacketed for cold brine or, in the case of grease cooling, cold water may be used. The inner surfaces of the pipes are freed from congealed grease by

[6] U. S. Patent 1,722,687.

spring-loaded steel scrapers attached to a shaft which rotates at 50 R. P. M. A 5-H. P. motor is needed to operate the machine operating on consistent greases.

These chillers are also made in a single unit 60 inches in diameter, also equipped with scrapers. Old-style horizontal, tubular type wax chillers, equipped with spiral scrapers and jacketed for cooling water, have been

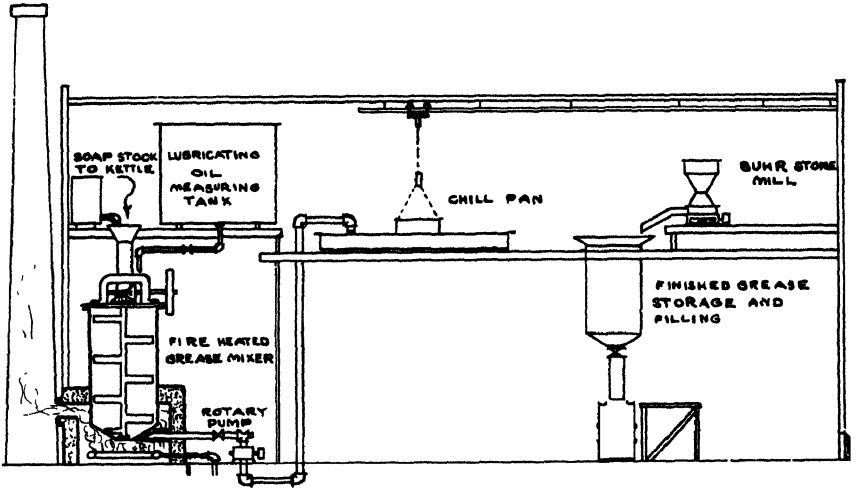


PLATE XVI.—General arrangement of plant for manufacturing greases by fire-cooking, chilling and milling.

pressed into service as grease coolers with considerable success. An installation of this sort is shown in Plate XV. Nill [7] has patented the use of the cylindrical device used by the packing industry for chilling lard, as a means of chilling wax-like lubricating greases.

CHILL PANS (COOLING TRAYS)

In the manufacture of smooth sodium and aluminum soap base greases open trays may be employed when other facilities are not available. A satisfactory design of chill pan for use where the throughput is small and the floor space limited, is to mount the pan in a 2-inch angle iron frame which may be 3 feet wide by 12 feet long. The pan may be made of No. 14 black iron with the edge turned at a height of 10 inches.

Where space is limited, portable racks may be constructed holding from ten to fifteen trays each about five inches high. Some difficulty in securing uniform cooling where the trays are superimposed, has been encountered and many grease makers prefer stationary trays, permanently mounted about two feet from the floor so that the grease may be readily removed by hand. A room which is subject to close temperature control is of con-

[7] U. S. Patent 1,937,463 (Nov. 28, 1933).

siderable assistance in the production of many types of chilled greases. A typical chill pan installation is shown in Plate XVI.

Twenty-inch channel irons set in a concrete floor, and provided with regularly spaced metal dams, have been used successfully for cooling locomotive journal grease. The bottoms of the channels may be fitted with water jackets, and the circulation and temperature of water so controlled as to produce journal grease cakes of quite smooth texture.

CAN FILLING MACHINES

Can filling machines of various capacities are available from the manufacturers, those built by the Filler Machine Company of Philadelphia being outstanding. A small automatic grease meter is used in some grease plants with considerable success. It is inexpensive compared with the usual fillers which are most convenient if a very large number of cans of different size are to be run each day.

Mead Cornell [8] has invented a grease meter which is quite satisfactory. It will fill in quantities of 5, 10, 25, and 50 pounds. It is operated by a gear-type grease pump of the type normally used in grease plants, which develops

Table 1—Typical Packages Necessary in a Grease Plant

	Cases—Lithographed Cans[3]						Cases Net Weight Lbs.			Steel Drums NRST			Barrels	
	24 1-lb.	48 1-lb.	12 5-lb.	6 10-lb.	2 25-lb.	2 5-gal.	Blocks	Sticks	Solid Pack	105-lb.	215-lb.	420-lb.	¾ Wood	Wood
Pressure Gun Lubricant	x	x	x	x	x					5	5	5		
Cup Grease 2	x	x	x	x	x					6	6	6		
Cup Grease 00, 0 and 1			x	x	x					5	5	5		
Cup Grease 3, 4 and 5			x	x	x					6	6	6		
Gear Grease 1 and 3			x	x	x					5	5	5		
Gear Grease 4 and 5			x	x	x					6	6	6		
Rocker Arm Lubricant 3, 4, 5			x	x	x									
Lime Base Gear Grease			x	x	x					5	5	5		
Winter L. B. Gear Grease										5	5	5		
Fiber Grease 00, 0 and 1			x	x	x					5	5	5		
Fiber Grease 2, 3, 4 and 5			x	x	x					6	6	6		
Liquid Grease						2				5	5	5		
Special Cup Grease 1					x					5	5	5		
Special Cup Grease 3					x					6	6	6		
Graphite Grease 3, 5			x	x	x					6	6	6		
Black Gear Greases A and C				x	x					5	5	5		
Black Gear Greases E and G				x	x					6	6	6		
Airbrake Cylinder Grease											5	5		
Journal Grease									90					
Rod Grease								65						
Wool Yarn Grease											6	6		
Curve Grease												6		
Axle Grease				x	x					6	6	6	2	2
Dark Grease				4	4									
P. M. Block Grease							75							
Tunnel Bearing Grease							75							
Transmission Greases					4									
Blk. Cup Grease (all grades)				x	x					6	6	6		

x—Standard packages, all Divisions.
2—Packed on SPECIAL ORDER only.
3—Lithographed cans are not to be re-labeled.
4—Special Department Brand only.
5—Non-returnable steel tanks for soft grease. Have 2″ and 9″ end openings.
6—Non-returnable steel tanks for hard grease. Have 15″ end opening only (105-lb. 12″ only).

[8] Cornell Machine Company, Cleveland, Ohio.

a pressure of 100 pounds per square inch or less, depending on the consistency of the grease at average filling temperature, which is normally 165 to 185° F. At this temperature it is possible to handle greases having an A. S. T. M. worked penetration as low as 200 at 77° F. The speed of operation will vary according to the consistency of the greases filled, and will normally operate about as follows:

Size of Container Pounds	Number of Containers per Hour
5	1000
10	1000
25	500
50	250

PACKAGING

Each grease plant should, of course, work out a standardized system for packaging their greases. Some products are required in relatively few sizes while others must be packed in all sizes from a 1-pound can to a 420-pound drum. The list in Table 1 suggests typical packages found necessary by one grease plant.

Chapter V
Calcium Base Greases

In many instances the independent grease plant manager is an extremely busy man. He must interview the salesmen of raw materials, containers, and transportation; he checks credit ratings, and supervises the operations of his plant particularly with regard to material consumption and operating costs. This places the responsibility for standardizing the grease making processes, development of new grease formulae and testing incoming raw products in the hands of the grease plant foreman (who may not be a technical man), the grease maker, and the plant chemist and his assistants. These men are constantly seeking new formulae, either as a means of improving their existing line of greases or to meet the needs for new lubricants at the demand of the sales force. Too often, their idea of a formula is a list of the soap making ingredients and oils, together with the proportions to be used, with little information as to the means of compounding, specifications of the raw materials and ranges in the specifications of the finished greases.

In selecting a new formula the possibilities presented by from ten to fifty formulae, either developed in the plant laboratory or obtained from the literature on lubricating greases, should be considered in accordance with the following items:

Estimate the cost per pound of each grease, using current costs of ingredients, including freight and handling charges.

Select at least two which may call for the fewest number of ingredients and can be made at lowest cost, at least one which may be high priced but offers possibilities of having superior properties, and compare these formulae with a complete analysis of the best product (one which has been found to be most satisfactory in service tests in the field) obtainable from other manufacturers.

Investigate the source of supply for new materials needed in any of these formulae, particularly with regard to reliability of the source, and also the various grades obtainable, range of market prices and tariff if an imported material.

Consider the possibilities of utilizing present storage and existing grease kettles in view of the production schedule on hand, and if a trial batch can be made.

Make at least a ten-pound batch giving particular attention to the conditions of temperature, agitation, and viscosity of the batch, to determine if these can be obtained in existing full scale plant kettles.

With a sample from either the trial batch or experimental batch available determine if the appearance, consistency, melting point, etc., are satisfactory and meet requirements, then investigate the factors relative to screening, packaging, and stability of product in storage. It is also well at this point, and before offering the product to the market, to conduct laboratory and field service tests in order to fortify the sales force with tangible information as to the serviceability of the new lubricant.

When the new grease has been put into production no needless formula changes should be made. The colors of oils and fats, consistency, texture and packaging must be uniform or complaints from customers will very likely follow.

In the following formulae many complex mixtures will be found as well as many relatively simple compositions. If not otherwise stated the formulae will be given in terms of parts by weight or per cent by weight. If it is necessary to know the number of gallons of an ingredient, multiply the specific gravity, obtainable from the plant laboratory or chemical handbooks, by 8.328 (the number of pounds one gallon of water weighs) to obtain the weight of one gallon of the ingredient. The number of pounds called for in the formula divided by the weight per gallon will be the number of gallons required.

In this chapter those lubricating greases which depend chiefly on the presence of the fatty acid salts of calcium (lime) for their consistency will be discussed with regard to their formulae, methods of manufacture and most important characteristics as shown by laboratory and service tests. Throughout this, and the following chapters greases will be classified with first consideration given to the predominating alkali or metal base (in the order: Calcium, Sodium, Aluminum, Lead, Zinc, and miscellaneous), the secondary means of classification being the specific uses for which they are intended and the presence of special compounding materials. While this may seem inconvenient to those who wish to secure quickly all the information on various greases for a given purpose, it is assumed that the majority of the readers will be chiefly interested in a manufacturing procedure and general information for any given class of lubricants. To avoid needless repetition and cross referencing it is considered desirable to follow the foregoing plan of classification based on type of soap. The general discussions on such subjects as Railroad Journal Lubrication, Steel Mill Greases, Paper Mill Lubricants, etc., will therefore fall in the chapters bearing on the most important and popular types of these greases.

INDUSTRIAL CUP GREASES

In general, the foregoing heading would include almost any kind of lime base cup grease. However this section is limited to include a discussion of early cup greases and modern processes for the manufacture of the more normal varieties of pale colored lime base greases intended for general service.

Early Calcium Base Grease Patents

In the introduction a discussion of greases in use up to about the year 1860 was given. In 1861 Earle[1] obtained a patent wherein a mixture produced by dissolving caustic lime in water was mixed in about equal proportions with sperm, olive, rape, lard, fish, or other animal oils to form a lubricant. Such a product would, of course, be much less consistent than normal cup greases. Johnson[2] obtained provisional protection for a semi-fluid lubricant made from olive, neat's foot, or colza oil heated together with lime

[1] British Patent 794 (March 30, 1861).
[2] British Patent 1,355 (May 29, 1863).

water. For greater consistency tallow or palm oil was added, and there is not much evidence that in either case an appreciable amount of lime soap was produced, except from the fatty acids present. Another provisional patent is on record in the name of Hutton regarding a lubricating grease made by heating and stirring together quick lime and various distillation products from coal tar. In 1864, Ward [3] obtained a patent covering solutions of earthy or metallic soaps in mineral oils, with or without the addition of tallow or vegetable fats. Ward employed the double decomposition method for preparing the lime soaps, using calcium chloride solution for treating an aqueous emulsion of white soda soap, with hydrocarbon oils. Ward also, in some cases, added aluminum soaps and tallow or palm oil to this composition.

In 1865, West [4] obtained provisional protection for his mixture of kaolin or mica, tallow, and coal tar, in which the fat was first saponified with an alkaline earth such as calcium oxide. That calcium naphthenate was known to early grease makers is indicated in the Lundy patent of 1867,[5] which mentions that the acid tars from petroleum may be neutralized with alkali or lime and combined with calcium cottonseed oil foots soap to produce a grease. Morgan [6] suggested mixing olive oil, rape seed oil, or palm oil with mineral oil and paraffin and treating the mixture in an agitator with lime water to obtain a soft grease for engine bearings. In 1872, Williams [7] obtained a U. S. patent for a compound of tallow, sulfur, lime water, borax, white and black lead, rosin, and mineral oil.

Thomas, in 1873,[8] produced lubricating greases by treating mixtures of heavy petroleum and oleic acid with slaked lime in the presence of other materials. The soaps of sodium, barium, strontium, and magnesium, were also mentioned but the calcium soaps were considered preferable. Draper's [9] plan for manufacture of lubricating grease was to add lime water to petroleum, while stirring, and then introduce any kind of animal fat in the ratio of one pound of fat to one gallon of the lime-oil mixture. Combination of the oil with the fat prior to addition of the lime water was also recommended. Draper points out that it was customary to boil such mixtures to promote reaction and evaporate water; however, he did not propose heating during the mixing process.

In carrying out the Hutchinson [10] process approximately one hundred pounds of tallow, or other animal fat, and mineral oil was melted together in a suitable vessel, and then by means of a heated iron bar the mixture was set on fire and allowed to burn the more volatile hydrocarbon portions. Five pounds of hydrated lime, two pounds of sulfur, two pounds of graphite, and two pounds of charcoal were added. The product was then

[3] British Patent 1,113 (May 3, 1864).
[4] British Patent 2,118 (August 16, 1865).
[5] British Patent 211 (January 26, 1867).
[6] British Patent 3,354 (Nov. 20, 1869).
[7] U. S. Patent 132,379 (Oct. 22, 1872).
[8] British Patent 3,603 (Nov. 5, 1873).
[9] U. S. Patent 171,508 (Dec. 28, 1875).
[10] U. S. Patent 134,890 (Jan. 14, 1873).

passed through a paint mill before packaging. It was pointed out by Hutchinson that when this compound was applied to a hot bearing, the temperature was soon reduced to a proper and normal condition. This was, no doubt, due to the presence of the sulfur and graphite which have extreme pressure properties.

Another instance in which lime base greases have been prepared by actually burning a portion of the batch is indicated in the patent issued to E. N. Hilbert,[11] Spokane, Washington. In the Hilbert process inexpensive cup or pressure greases are made from oil reclaimed from crankcase drainings and a soap base. The soap is prepared from 15 pounds of fat, 12 pounds of hydrated lime solution, and 3 pounds of caustic soda. The mass of oil and soap base are agitated in a heated kettle, and then ignited to burn off the more volatile oils from the crankcase drainings. This, of course, appreciably increases the temperature of the batch and facilitates dispersion of the soap base in the oil.

Lake's [12] British patent of 1876 is quite similar to that of Draper. No particular mention is made of heating the ingredients: animal fat, lime water, and petroleum. The addition of further amounts of mineral oil were recommended to produce softer greases when desired. Newton [13] boiled slaked lime, tar oil, and rosin oil together to produce a wagon and machinery grease. Irvine [14] patented a process for preparing cup greases consisting of the saponification of tallow, or other animal or vegetable fats, with alkalies or alkaline earths, dehydration of the soap by heating and combination of the soap with mineral oils including those derived from petroleum. A plastic substance or an oil of increased body was produced according to the proportion of soap employed. Broadbent's [15] grease consisted of animal or vegetable oil, mineral oil, milk of lime, chalk, sal soda, and water. Hadden [16] patented a grease consisting of lard oil, lime, "coal oil," chalk, and potassium carbonate.

Field [17] claimed a lubricating grease of high melting point prepared by dissolving stearates and oleates of calcium, magnesium, barium, lead, or zinc in heavy hydrocarbon lubricating oils or neutral animal or vegetable oils, using moderate heat. In 1886, Hutchinson [18] pointed out the desirability of improving the stability and other properties of soap base greases by the addition of small excesses of fatty acids or fat. A typical example of the grease which he patented had the following composition:

Parts by weight	
1	Hydrated lime
52	Mineral oil
14	Oleic, stearic, or margaric acid

[11] U. S. Patent 1,707,633 (Apr. 2, 1929).
[12] British Patent 108 (Jan. 10, 1876).
[13] British Patent 824 (Feb. 28, 1876).
[14] British Patent 3,116 (July 29, 1880).
[15] U. S. Patent 233,197 (1880).
[16] British Patent 1,883 (Apr. 28, 1881).
[17] British Patent 1,467 (March 27, 1882).
[18] British Patent 1,026 (Jan. 23, 1886).

The grease was prepared by boiling the foregoing materials together in a kettle. Fink [19] secured a patent covering a complicated mixture of animal, vegetable, and mineral oils together with chalk, and sulfur, the entire mixture being treated with lime water. A friction reducing journal-box grease was patented in 1886 by McKenzie [20] consisting of the following materials:

Per cent by weight	
5	Animal fat
75	Mineral oil residue
15	Slaked lime
5	Manganese oxide

These materials were mixed thoroughly together and heated to the boiling point of water. When cold, it had a pasty consistency. Ridsdale,[21] in 1891, obtained a patent for a lubricant in cake form for use on the roll necks of steel mills. The formula for one of his greases was:

Per cent by weight	
2	Lime
35	Rendered suet
14	Sulfur
19	Graphite
30	Heavy petroleum oil

In preparing the lubricant the lime was slaked, mixed with the suet and heated until all of the water was driven off and combination had taken place. The remainder of the ingredients were then added and the batch cooled and drawn into molds.

A variation of Hutchinson's [22] original patent was obtained in 1891 and consisted of adding to the normal grease consisting of calcium oleate and stearate in mineral oil a mixture containing sodium or potassium "pinate," apparently to improve the stability of the original product.

W. S. and W. F. Chenhall [23] obtained a patent for solidifying animal, vegetable, and mineral oils by heating them with the rosin soaps of calcium, magnesium, sodium, or potassium. The advantages of greases made from wool grease pitch and waxy vaseline combined with boiling lime water were first recognized by Peters [24] and the calcium base grease so obtained was employed for roll neck lubrication. Ekenberg [25] was first to prepare modern type lubricating greases from mineral oil and the lime soaps of wool fat.

To manufacture lubricating greases Plisson [26] developed a new type of grease kettle equipped with a special form of planetary agitator. A typical example of the grease produced is indicated in the following formula·

[19] U. S. Patent 335,443 (1886).
[20] U. S. Patent 351,195 (Oct. 19, 1886).
[21] British Patent 3,782 (Mar. 3, 1891); (U. S. Patent 432,313).
[22] British Patent 16,034 (Sept. 22, 1891).
[23] British Patent 952 (Jan. 18, 1892).
[24] British Patent 11,972 (1895).
[25] British Patent 16,541 (July 25, 1896).
[26] British Patent 1,659 (Mar. 1, 1890).

Per Cent	
4	Lime
2	Baryta
4	Water
10	Cocoanut oil
5	Colza oil
75	Russian petroleum

Kuess [27] has proposed the following formula for solidifying oils:

10 Kg.	Rosin
100 liters	Petroleum oil
10 Kg.	Animal fat
3 Kg.	CaOH
3 Kg.	Water
1 liter	HCl
7 Kg.	NaOH (added to 12 liters of water)

Le Maitre [28] has proposed a calcium base grease in which raw lanolin is treated at an elevated temperature with a mixture of sulfuric and nitric acids, neutralized, dried and combined with heavy lubricating oil. The mixture is then boiled together with calcium hydroxide to obtain a plastic grease.

Blass [29] also made use of wool grease in the preparation of a lime base grease. In one example, anthracene, wool grease fatty acids and calcium hydroxide were heated together and then allowed to cool to a consistent grease. In 1906, Ermen [30] obtained a patent for greases composed of cottonseed oil, castor oil, tallow, wool grease, and fish oil after treatment with sulfuric acid at temperatures above that at which sulfonated oils are obtained, neutralized and employed as the emulsifying agent. The solid grease was obtained by mixing together the treated fatty oil and mineral oil, and combining with slaked lime. Hadden,[31] in 1913, recognized the advantages of saponifying fatty acids with calcium hydroxide in the presence of mineral oil, using an autoclave to hasten the reaction. Hydrated lime appears as one of the ingredients in the grease patented by Pettet,[32] the other ingredients being petroleum oil, water, and bicarbonate of soda. Reynard and Edser [33] prepared lubricants by treating crude wool grease with calcium hydroxide in sufficient quantity to convert practically all of the free fatty acids to lime soaps, after which the mass was dehydrated by heating and incorporated with lubricating oil for the production of a grease. Greases made in this manner may contain from five to fifty per cent of the wool fat product, and since they are quite stable, are particularly suitable for ball bearing lubrication. For this purpose, seventy per cent of the wool fat product and twenty five per cent of paraffin oil has been recommended by the patentees.

[27] British Patent 7,481 (Nov. 6, 1904).
[28] British Patent 15,818 (Aug. 2, 1905).
[29] British Patent 6,601 (Mar. 19, 1906).
[30] British Patent 24,135 (Oct. 30, 1906).
[31] British Patent 16,480 (July 17, 1913).
[32] U. S. Patent 1,072,421 (Sept. 9, 1914).
[33] British Patent 164,107 (Feb. 28, 1920).

Modern Calcium Base Grease Patents

Byrnes [34] has patented a process in which the mineral oil vapor and air is passed through catalytic material at a regulated temperature. It is claimed that the condensed reaction products contain aldehyde fatty acids and waxes. These are thickened by treatment with lime to convert them to soaps suitable for use as lubricating grease bases. According to U. S. patent 1,700,055 (of January 22, 1929), the catalysts employed by Byrnes are the blue oxides of molybdenum held at temperatures of 230 to 500° C. From the treatment of Pennsylvania gas oil the following yields are claimed by the inventor:

Per Cent	
53	Total aldehyde fatty acids
18	Aldehydes
17.5	Alcohols and heavy hydrocarbons
11.5	Light products, mostly hydrocarbons distilling under 200° C.

Byrnes prepares greases from various combinations of the lime soaps of the aldehyde acids, water, and mineral oil, the soaps being added with, or without, removal of other substances of the catalytic reaction. The lubricant claimed is one containing a salt of an oxygenated organic acid and other lubricating material. Also claimed are the condensation products of aliphatic aldehydes, free acids obtained from the partial oxydation of mineral oil, and polymerized aldehyde fatty acids as ingredients of lubricants.

Spensley [35] has assigned to the Chemical Engineering Company his patent relative to the use of a high speed mill for producing lubricants. Chemical reactions that are usually brought about only by long heating, agitation, or both, are effected rapidly and at lower temperatures by intimately mixing the interacting substances, with or without others, by passing them through a high-speed centrifugal pin disc mill of the horizontal disc type, in which a continuous stream of the substances is introduced at the center and the mixture is discharged substantially all round the circumference. Stauffer and like greases are produced by saponifying fatty matter with the theoretical quantity of base and then running the soap and mineral oil through the mill, or by passing the base, fat and oil through the mill simultaneously. An animal fat such as bone grease, a vegetable fat such as palm oil, stearin, or olein, or a mixture, are run through the mill with the theoretical quantity of lime and about 2 per cent of water, or of caustic soda and about 5-7 per cent of water; 16-24 parts of the soap are mixed with 84-76 parts of mineral oil, passed through the mill, and then boiled for 15-30 minutes.

Marks [36] has assigned to the Merco Nordstrom Valve Company a patent covering a lubricating grease suitable for use on plug valves, or other parts subjected to high temperatures and pressures, comprising a

[34] British Patent 173,750 (Jan. 6, 1921).
[35] British Patent 201,968 (Apr. 4, 1922).
[36] British Patent 229,957 (Sept. 20, 1924).

mixture of a mineral oil and a soap made from a vegetable oil, vegetable wax, and quicklime. Flow points as high as 600° F. are claimed. In an example, 780 grams of castor oil and 220 grams of carnuaba wax are melted together and boiled for 30 minutes with 500 grams of quicklime in 7 liters of water. The water is poured off, and the soap is melted, cooled, mixed with $\frac{1}{6}$ its weight of flake graphite, and ground. It is then mixed cold with twice its weight of railroad valve oil, or other mineral oil of high flash point, and the mixture melted. Maximum process temperatures of 400-500° F. are mentioned. A similar lubricant for steam valves was issued to S. J. Nordstrom,[37] and referred specifically to a combination of mineral oil, the calcium soap of castor oil, carnauba wax and calcium oxide.

James McKee [38] has assigned to the Sun Oil Company a United States patent dealing with the preparation of a lime base grease in which two mixtures: one in which calcium hydroxide is mixed with mineral oil and another in which fatty acids are blended with a predominating quantity of mineral oil, are mixed together and heated to expel moisture and promote reaction. The batch may then be adjusted by adding small quantities of fatty acids and water. Further quantities of mineral oil may be added to obtain greases of the desired consistency and the batch heated to a maximum temperature of from 200 to 240° F.

J. Y. Johnson [39] has assigned to the I. G. Farbenindustrie Akt.-Ges., a patent for lubricating greases produced by heating together mineral oil, vegetable, animal, or fish oils, or mixtures of these with bleached montan wax. An aqueous solution of an alkali hydroxide is then added to neutralize the free acidity. In an example, mineral oil was mixed with rape and fish oil, and heated with montan wax which may be esterified or partly esterified and partly converted to a calcium salt. Sodium hydroxide solution may also be added while stirring to produce a consistent grease.

The Panoleum Company, Cleveland, Ohio, are the assignees of an invention by White [40] of greases containing an amide or anilide of a higher fatty acid to increase the viscosity of the oil content. White's lubricant consists of mineral lubricating oil, a soap which is added to increase the melting point, and an amide or anilide, the composition being free from water: from five to fifteen per cent of stearic, palmitic, oleic, or arachidic anilide and three to five per cent of calcium, sodium, or zinc soaps of animal or vegetable oils such as lard, tallow, cotton seed, or castor oil, or mixture of such soaps.

Rosenbaum [41] has patented a grease consisting of mineral oil, oil soluble castor oil which is apparently included to increase oiliness, and water insoluble soaps such as calcium soap. Sullivan and Arveson have assigned to the Standard Oil Company of Indiana a patent for a mixed calcium and

[37] U. S. Patent 1,514,096.
[38] U. S. Patent 1,619,352 (Mar. 1, 1927).
[39] British Patent 317,630 (Aug. 3, 1928).
[40] British Patent 347,574 (Oct. 25, 1929).
[41] U. S. Patent 1,860,622 (May 31, 1932).

sodium soap base grease. Its formula includes fatty acids, mineral oil, calcium hydroxide, caustic soda, water, and montan wax.

In order to stabilize lime base greases containing viscous mineral oils Hodson [42] has proposed incorporating oil miscible solvents such as the mono-ethyl or mono-butyl ether of ethylene glycol. It is sometimes difficult to produce a relatively stable grease from very viscous paraffinic oils and lime soaps of fatty acids of high molecular weight, and no doubt minor portions of such organic structure stabilizers are beneficial. It is likely that the mono or diglycerides of oleic, lauric or stearic acids, suggested by Pelly,[43] may also tend to stabilize such calcium base greases. The advantages of utilizing hydrogenated fats in cup grease manufacture have been pointed out by Hilliker.[44] In his process mineral oil is processed with a small amount of water and 2 to 35 per cent of a calcium soap of a hydrogenated fat or fatty acid, by heating to 260-300° F. and maintaining this temperature for about one and one half hours. The batch of grease is then cooled to about 240° F., and one per cent of water added at this temperature. Cooling is continued and further quantities of mineral oil may be added. The finished cup grease contains aproximately one per cent of water.

The addition of rubber, rubber hydrocarbons and latex have long been recognized as appropriate ingredients for increasing adhesion and cohesion of lubricants. A great number of patents have been issued claiming these substances. Their addition to soap base greases however, appears to be of recent adoption. The N. V. de Bataafsche Petroleum Maatschappij have obtained a French patent in which natural rubber from latex is incorporated with metallic or nonmetallic soaps to the extent of one to five per cent prior to mixing the soaps with mineral oil. In the Klemgard patents U. S. 1,975,582 (October 2, 1935) and U. S. 1,979,943 (November 6, 1935) uncoagulated rubber from latex is added to finished greases as a stabilizing agent and to increase adhesion.

Hilliker,[45] in 1932, applied for a patent dealing with the manufacture of calcium base cup greases from hydrogenated fatty material. In his process, a small amount of mineral oil, water, and hydrated lime are heated together with the hydrogenated fat for one hour at 260-300° F., after which the batch is cooled and stirred to a temperature of 240° F., when a small quantity of water is added. Mineral oil is run in while stirring and cooling to 220° F., which temperature is maintained until all of the desired oil has been worked into the batch. The grease is then stirred and cooled to 190-210° F., so that no further loss of consistency or break down will occur on aging.

Johnson [46] has found that the sulfonated urethanes, or the alkali metal salts of these compounds, are useful emulgators. It is likely that small quantities of these materials would serve to improve the stability of normal calcium base cup greases.

[42] U. S. Patent 1,982.662 (Dec. 4, 1935).
[43] Canadian Patent 354,964 (Dec. 24, 1935).
[44] French Patent 791,279 (Dec. 6, 1935).
[45] U. S. Patent 1,989,197 (Jan. 29, 1935).
[46] British Patent 381,204 (June 25, 1931).

PALE INDUSTRIAL CUP GREASES

Production in Open Steam Heated Kettles

The following formula has been used by a Pacific Coast grease manufacturer for the production of more than twenty million pounds of grease, and has been standardized to the extent that few manufacturing difficulties are encountered.

Cup Grease Formula "A" (25-Barrel Batch)

Lard oil, Extra No. 1	150 gallons
Inedible tallow	300 pounds
Hydrated lime	198 pounds
Western mineral lubricating oil	900 gallons
Water	75 gallons
Oil mirbane	6 pounds

QUALITY OF MATERIALS USED.

Mineral Oil. The viscosity of the oil should be 180 to 100° F. (Saybolt). The color of the oil should be between numbers 9 and 10½ Robinson. The oil used in this grease was of western origin and had a gravity of 19° to 21.5° Bé.

Extra No. 1 Lard Oil. This oil should be of a red or brown color and should not contain over seven per cent free fatty acids calculated as oleic.

Inedible Tallow. This fat should be clear, fresh and free from dirt. The color should not be darker than pale yellow, the melting point should not be less than 110° F. It should not contain more than 5 per cent of free fatty acids calculated as oleic.

Hydrated Lime. This material should be finely powdered and contain about 27 to 28 per cent water and 66.6 per cent available calcium oxide as determined by the American Society of Testing Materials method. (A.S.T.M. Tentative Standards, 1923, page 286.)

PROCEDURE.

Charge in an open steam jacketed kettle 50 gallons of the oil, slop cup grease, or tailings from the previous batch.

Charge into the kettle the lard oil and tallow in the quantities specified.

Mix in a separate receptacle in the order named: 198 pounds hydrated lime, 30 gallons mineral oil, 75 gallons water.

Add this mixture to the kettle and start the agitating paddles.

Turn on steam in jacket and bring the temperature of the contents of the kettle to 300° F. in from 2 to 4 hours.

Allow the lime soap thus made to stand in kettle over night with steam on but without agitating.

The soap is then tested in order to determine if the saponification process has been complete, and an estimate of its water content made. Analysis by chemical methods for the determination of uncombined fat or lime is usually considered too slow for practical purposes so that it falls on the grease maker to determine by experience and judgment, if the lime soap base is in the proper condition for mixing in the additional oil. A soft pasty consistency indicates incomplete saponification or an excess of water. A stringy, more or less transparent soap denotes excessive water evaporation, and indicates that more than the usual amount of water should be added later. The soap should be firm and break evenly but should not crumble too readily as this denotes an excess of lime. If the soap is unsatisfactory a correction should be made by adding from one to five gallons of 20° Bé. caustic soda solution or in the case of excess lime an appropriate amount of tallow should be added.

Run in mineral oil till the total quantity of oil in the kettle is 400 gallons.

The temperature at this period should be brought to 212° F.

Run in additional oil till the total volume of oil added is 750 gallons.

Scrape down the sides of the kettle and add slowly 4 to 10 gallons of water. The temperature should be 180° to 185° F.

Stir for ten minutes.
Add six pounds of oil of mirbane.
Run in the balance of the mineral oil which is 150 gallons.
Stir for 15 to 20 minutes.
Fill at a temperature of 160° to 170° F.

The grease produced by this process is of a medium consistency known to the trade as No. 3. Its melting point is 190° to 195° F. and consistency as taken by the A.S.T.M. penetrometer at 77° F. is 225, unworked.

Grades of Different Consistencies

In order to produce cup greases of different consistencies the following series of formulas may be used. The same general procedure as for Cup Grease "A" in which the open type of steam jacketed kettle is employed will be found satisfactory. It will be noted that these formulae call for appreciable quantities of caustic soda. It is very probable that when the caustic soda is used the saponification of the fats is more quickly and completely carried on. However, if a waterproof grease is desired hydrated lime may be substituted for the caustic soda and very satisfactory greases produced.

Formulae for Five Grades of Cup Grease (Formulae "A")

Grade	Per cent by weight				
	1	2	3	4	5
CaO	1.54	1.69	1.86	2.17	2.82
Caustic soda	.22	.25	.27	.31	.42
Water	1.06	1.16	1.28	1.49	1.98
Lard oil, No. 1	2.75	3.02	3.33	3.88	5.14
Tallow, No. 2	11.01	12.10	13.30	15.53	20.57
Mineral oil, 180 viscosity	83.41	81.77	79.85	76.41	67.76

The CaO referred to is quicklime which in order to be used must be hydrated with about 25 per cent of water. This hydrated lime may then be used with the same proportion of water as for Cup Grease No. 1. The caustic soda given above is dry caustic soda which has been dissolved in water to give a 35 per cent solution. The water indicated above is that which remains in the finished grease.

Babb's Formula. J. E. Babb gives the following formula system and process of manufacturing cup grease, in which paraffine base oil of 100 to 150 Saybolt viscosity at 100° F. is used.

Composition—Calcium Soap Greases

Grade	Per cent by weight		
	Hard	Medium	Soft
Tallow	30.00	22.00	12.00
Calcium oxide	3.30	2.44	1.32
Paraffine oil	65.20	74.45	86.08
Water	1.50	1.11	.60

The boiling or saponification of the fats is allowed to take place in a steam jacketed kettle having stirrers mechanically driven by motor and belt drive.

In making cup grease, a typical batch of thirty barrels, or 12,000 pounds, taking the formula for the hard grade, would be 3,600 pounds of tallow, 396 pounds of caustic lime, 7,824 pounds of paraffine oil and 180 pounds of water.

The tallow is first charged into the kettle and steam turned on. When the tallow is melted, the lime (preferably hydrated with 25 per cent of its weight of water) is added. Stirring is now commenced, to mix the lime and tallow. Water is added cautiously to prevent foaming, enough to equal about twice the weight of lime used. Steam pressure is increased gradually to insure normal boiling of the batch so as to prevent excessive boiling of the mass too high up in the kettle. With practice, the right amount of water and steam can be regulated so as to avoid danger of the soap boiling over. If the tallow contains about 12 per cent of free fatty acids, or more, the danger of boiling over is minimized and the saponification takes place more rapidly. Boiling and stirring of the mass is continued for five hours when a sample of the soap is taken out on a long stick or paddle and allowed to cool.

The soap, when detached from the stick, should break with a snap, similar to a thin piece of glass or a sheet of hard glue. The better procedure will be to refer the sample of soap to the laboratory where the completion of the saponification may be determined with certainty. With practice and the help of the laboratory, one acquires considerable skill in determining complete saponification of the tallow by observation of the soap in the kettle and the character of the soap sample.

When saponification is complete an amount of oil is added to the soap equal to the weight of tallow, adding the oil slowly so as not to chill the soap. When the above so-called first oil is added, steam is turned off and the agitation or stirring kept up until the temperature of the mass falls to about 221° to 230° F. The water is now added, all at one time, waiting two or three minutes for the mass to show signs of rising. The second oil is run in as fast as possible, to lower the temperature and prevent the mass from rising too high in the kettle, and also to avoid too much evaporation of the water. When the mass appears to get thick at the top shut off the oil until the grease has a smooth appearance and the oil is well worked in, which will be in about fifteen minutes. Add the balance of the oil in small doses, avoiding too rapid chilling of the mass at the top or lumps of hard grease may form. When all the oil is in, stirring should be continued until the temperature is down to about 158° to 176° F. before running the grease out of the kettle.

Some prefer to saponify the tallow with the lime in the presence of an equal amount of oil, and when saponification is complete the mass is stirred for two or three hours until the temperature of the mass falls to 220° to 230° F. The water of hydration is now added and then the balance of the oil, finishing as in the previous method. The difficulty with the second procedure is to be able to tell when the tallow is all saponified, which requires more skill and experience. It is best to submit a sample of the soap mixed with the oil to the laboratory until one acquires the necessary skill.

With the acquirement of skill, the second procedure is perhaps the best to follow as there is much less danger of hard lumps of soap being formed in the grease. The grease after finishing is screened through a 12 × 12-inch filter press using 30 to 40 mesh perforated metal screen. An ordinary rotary soap pump is used for pumping the grease. The very hard greases are not always screened and then only through a very coarse mesh.

Kauffman's Formulae. According to Kauffman,[47] nearly all cup greases are made with calcium soap bases obtained by saponifying fatty oils with slaked lime. However, a few greases marketed under the name of cup greases contain only the soaps of sodium. As previously pointed out, some cup greases may be produced from various mixtures of calcium and sodium soaps, the calcium normally predominating. Cup greases have also been referred to as "hot-sett" or "boiled greases." Cup greases should be smooth, homogeneous, and have a butter-like texture. They are normally graded in consistencies numbered from 00 to No. 6, and either the 00 or No. 6 may be the hardest or the most consistent, but it is the usual plan to designate the No. 00 as the softest product. A No. 000 grease and liquid greases are marketed by some manufacturers. The soap contents may vary from a fraction of 2 per cent to as much as fifty per cent by weight. Kauffman lists horse oil, beef tallow, mutton tallow, lard oil and acidless tallow as the animal oils most frequently used; while cottonseed oil, soya bean oil, corn oil, palm oil, coconut oil, and peanut oil are the vegetable oils most frequently utilized.

Kauffman considered the use of two kettles for the production of cup grease as permitting closer control with resulting uniformity of consistency. According to this writer, it is desirable to slake quicklime for use in greases. His procedure consists of slaking the lime in barrels, first covering the lime with water and then later adding further quantities as steam is produced. Hand stirring is carried out and sufficient water added to prevent the temperature from exceeding 160-170° F. The mixture of lime and water is then permitted to stand for about five hours, and the supernatant clear water containing soluble salts and some calcium hydroxide in solution is discarded. The lime paste which precipitates is screened and used in the saponification process. In another process seventy-five pounds of lime are slaked in a tub with seventy-five gallons of water. The water is first heated with live steam to about 110° F. and the quicklime added to the water. The addition of cold water may be necessary to prevent boil-overs. The slaked lime is run through a fine screen before entering the grease kettle. In some cases, live steam is employed to facilitate the hydration of CaO. Some manufacturers employ two screens, the first of coarse mesh to remove large lumps, and a second to filter out finely divided particles.

Cottonseed Oil-Lime Base Cup Grease (Kauffman). Since cottonseed oil alone is very difficult to saponify, small quantities of both No. 1 lard oil and cottonseed oil fatty acids are also added to the kettle. The cottonseed oil, 100 gallons, is added to the kettle late in the afternoon of the day

[47] *Refiner Natural Gasoline Mfr.*, 93-96, 135-138 (May, 1929); also 106-114 (Sept., 1929).

previous to the one on which the batch is to be made. To the kettle are also added at this time 10 gallons of No. 1 lard oil and five gallons of cottonseed oil fatty acids. The slaked lime, 140 pounds of calcium hydroxide (the equivalent of 106 pounds of quicklime), is then carefully screened into the kettle.

According to theory only about 10 per cent of CaO (13.5 per cent of $Ca(OH)_2$), based on the weight of cottonseed oil used, is required for theoretical saponification. However, experience has indicated that for open-kettle treatment of this fatty oil for grease making 13 per cent of CaO (17.2 per cent $Ca(OH)_2$) may be necessary to obtain satisfactory saponification. This represents an excess of calcium hydroxide amounting to about 27 per cent, much higher than is required for normal animal fats, and higher than is desirable for the production of greases of low free-alkali content.

After the foregoing charge has been added to the kettle, steam is turned on in the jacket and the kettle contents heated to approximately 130° F. The animal fat and cottonseed fatty acids react more quickly with the lime than does the cottonseed oil; thus, thorough emulsification of the soap ingredients soon takes place, and on account of this dispersion and the presentation of very large surface areas of the fatty oil, subsequent saponification of the cottonseed oil takes place at a fairly rapid rate.

After two hours of paddle agitation the steam is turned off, and the batch permitted to stand over night, much of the reaction between the lime and the fatty oil taking place during this 14-hour period.

In an alternate procedure 20 gallons of mineral oil may be introduced with the fatty materials at the beginning of the process.*

On the following morning steam is turned on in the kettle jacket, the paddles started, and the soap allowed to dry out as much as possible. For a period the soap will become a crumbly mass, and on the application of further heat, tends to melt and become smooth.

When the soap base becomes smooth and no lumps remain, the 140 S.U.V. mineral oil, warmed to prevent sudden chilling of the soap, is run in quite slowly, several hours being required. After about 20 per cent of the mineral oil required by the batch has been run in, its rate may be increased without danger. Two grease kettles are used in this process, it being claimed that by screening the soap base, containing about 80 per cent of the required mineral oil, from an upper to a lower kettle, better control and elimination of lumps is possible. The addition of water may be necessary in the final stages of the process.

Four ounces of oil soluble yellow dye, dissolved in the mineral oil, are then added to the batch.

After screening the batch into the lower kettle, ten pounds of a ten per cent solution of caustic soda is added to the batch while stirring. It is claimed that this soda stabilizes the soap dispersion, and tends to prevent the separation of oil in the finished grease. Probably, its addition at the beginning of saponification would be just as beneficial and would probably promote reaction without the necessity of using such high excesses of lime.

The final quantity of mineral oil to bring the grease to grade is then run in and the batch agitated at a temperature of 160 to 170° F. Just prior to this, however, samples are taken and cooled and if the grease has a rubbery texture, additional water is added.

The usual system of finishing the grease to a given consistency, regardless of the soap content, is followed in this process. While the grease is being finished in the lower kettle a sample may be taken, cooled, and its worked A.S.T.M. consistency determined. The estimated amount of oil to adjust the batch may then be added.

Continued agitation of the finished grease in the kettle tends to break it down and decrease yields. Before drawing the grease from the kettle five pounds of oil of

* A.P.I. Grav. 24.3; S.U.V. 140/100°F; P.P. 30°F.

myrbane may be added as a scent. It is claimed by some grease makers that the myrbane has a stabilizing effect on the grease. The greases produced by this procedure are drawn from the kettle at (about) 150° F. to 160° F., but not at temperatures above 170° F.

Formulae for four different grades of this grease are tabulated below:

Consistency No.	00	1	3	5
Lard oil, gallons	10	10	10	10
Cottonseed oil, gallons	100	100	100	100
Cottonseed oil fatty acids, gallons	5	5	5	5
Oil of myrbane, pounds	5	5	5	5
Oil soluble yellow dye, pounds	.3	.3	.3	.3
Hydrated lime, $Ca(OH)_2$, pounds	140	140	140	140
140 S.U.V./100° F. mineral oil, gallons	510	480	425	320
Approximate A.S.T.M. worked penetration	450	375	250	150

Horse Oil Cup Grease Formula. The following cup grease, made with horse oil, requires a 1,400 gallon open steam-jacketed kettle, for the production of approximately 10,300 pounds of No. 3 grease, as a single batch. Mineral oil of the following properties has been used successfully:

A.P.I. gravity	27.5
Flash point ° F.	380
Fire point ° F.	440
S.U.V./100° F.	150
A.S.T.M. color	4

The formula for the No. 3 grade is:

Horse oil, gallons	225
CaO, pounds	225
Water, gallons	200
150 S.U.V./100° F. mineral oil, gallons	1115

One hundred and twenty-five gallons of horse oil, 100 gallons of mineral oil and the lime slaked in the water are charged in the kettle, during the afternoon, and stirred and boiled for a period of about one hour.

Steam is then turned off and the batch allowed to stand until the following day. On the second day, the balance of 100 gallons of horse oil is run in, the batch warmed and stirred, and then cooked at full steam pressure for several hours, until examination of the soap base indicates satisfactory saponification. It is possible that, by splitting the horse oil during the saponification process, a larger portion of calcium mono and diglycerides may be produced.

Cooking and the addition of warm mineral oil are so controlled that it should not be necessary to add water to disperse the lime soap.

The batch is drawn at about 165° F. after thoroughly stirring.

The worked A.S.T.M. penetration of this grease is about 250, or a No. 3.

Kauffman gives the following directions for making No. 4 cup grease from a horse oil lime soap base:

28° Paraffine oil having the following properties is used:

A.P.I. gravity	28.5
Flash point ° F.	350
S.U.V./100° F.	102
A.S.T.M. color	3
Pour point	30

Seventy gallons of this oil is run into the grease mixer.

The equivalent of 208 pounds of slaked quicklime is then added to the mineral oil with an excess of water, and pouring the material through a fine screen.

Three-fourths of the total horse oil to be used is then added to the kettle contents, which in this case would amount to 150 gallons. Heat is turned on in the kettle jacket and frothing soon starts. The foam may reach the top of the kettle edge and when this occurs 20 gallons of cold mineral oil is run in. This boiling up process may take place four times, oil being added in each case, and finally 30 gallons of oil is added. The steam is then turned off, agitation stopped and the batch permitted to stand over night.

On the following day the remaining fourth of the horse oil (50 gallons) is added after turning on the steam and starting the agitators. If tallow is to be used with the horse oil, it is introduced at the beginning of the batch.

Forty gallons more of the mineral oil is run in, while agitating, and the batch cooked until practically all of the water has been evaporated. Normally, this will require 3 to 4 hours. Ten gallons of oil is added, the kettle walls scraped down by hand, and boiling at full steam pressure continued for one half hour to thoroughly mix and saponify the material scraped from the walls.

Before adding the final quantities of oil a sample is taken from the batch and examined for consistency, texture and moisture content. In most cases, a small amount of water is added at this stage. It is added slowly, so as to secure satisfactory distribution, so that it will not remain in the grease as free water. If too much of the water is lost by evaporation in these final phases of the process it may be difficult to secure satisfactory hydration of the lime soap, and lumps and separation will occur.

Two to ten pounds of oil of myrbane, oil of citronella, oil of sassafras, or oil of wintergreen, either alone or as a blend, may be mixed with about five gallons of mineral oil and added to the batch.

The final drawing temperature of 170° F., is considered most appropriate for greases of this sort. The formulae for Horse Oil Compression Cup Greases of different grades, which may be manufactured in accordance with the foregoing procedure, are tabulated below:

Grease No.	1	2	3	3	4	5	6
Approximate A.S.T.M. worked penetration	375	325	250	250	200	150	100
Cake tallow, pounds	...	...	660	...	...	...	...
28° Paffe oil, gals. S.U.V. 100/ 100° F.	1060	1010	880	960	905	880	860
Sassafras oil, pounds	...	...	2	...	...	...	...
Myrbane, pounds	10	10	...	10	10	10	10
Quicklime, CaO, pounds	140	160	200	180	200	215	230
Yield of grease, pounds	9050	8850	8250	8650	8400	8350	8330

No. 0 cup grease may be made from No. 1 cup grease, prepared as indicated in the formula above, by adding seven gallons of the oil to 350 pounds of the grease. Blending 350 pounds of No. 0 with 14 gallons of the mineral oil will produce a No. 00 grease.

Tallow-Soya Bean Fatty Acid Cup Grease. The following formula makes it possible to produce cup grease quite similar to those in which horse oil is used:

Beef tallow, pounds	1000
Soya bean oil fatty acids, pounds	600
Calcium hydroxide, pounds	290
Water, gallons	120

A 12-barrel kettle is used for cooking this grease, and it is mounted over a 40-barrel mixer, used for finishing the grease. About 65 per cent of the mineral oil is incorporated with the lime soap in the upper kettle, after which it screened while hot into the lower vessel and brought to grade with

the desired amount of mineral oil. The mineral oil used has the following properties:

A.P.I. gravity	27–29
Flash point ° F., Min.	365
S.U.V./70° F., Min.	360
S.U.V./100° F., Min.	140
Pour point ° F., Max.	30
A.S.T.M. color, Max.	4

One hundred gallons of the mineral oil, all the fats, and any slop grease available are mixed together in the upper kettle. The lime suspended in the water is then run in slowly, over a period of about one half hour, the steam pressure in the jacket being 15 to 20 pounds per square inch.

The batch is cooked for 4 to 5 hours until the proper amount of water remains, and oil added slowly.

The grease is then screened from the upper to the lower kettle at a temperature of 220 to 250° F., this depending on the consistency and soap content of the grease. The higher this temperature of transfer, the more water will need be added to the batch in the lower mixer.

The grease is finished in the lower kettle, the oil being run in while stirring. The first 100 gallons of oil may be added fairly rapidly over a period of 5 to 10 minutes. Thereafter, the mineral oil is added more slowly. When the desired amount of oil has been added, the grease is tested for consistency, and if satisfactory, water is turned on in the kettle jacket and the temperature of the grease brought to 167 to 185° F., at which temperature it is drawn.

Lard-Cottonseed Oil Lime Base Cup Grease Formulae. The following are various formulae of such greases:

Grease No.	00	1	3	5
Approximate A.S.T.M. worked penetration	...	375	250	150
Lard oil, pounds	76	76	76	76
Cottonseed oil, pounds	768	768	768	768
Quicklime, CaO, pounds	90	90	90	90
Water (finished grease), pounds	24	24	24	24
Oil of myrbane, pounds	5	5	5	5
Oil soluble yellow dye, ounces	4	4	4	4
25° Paraffin oil, pounds S.U.V. 150/100° F.	4994	3943	3313	2512
Yield of grease, pounds	5957	4906	4276	3475

In the above formula system 7 per cent excess CaO is used, and in some cases where saponification is difficult fatty acid additions may be required, or this excess increased.

Horse Grease-Tallow Lime Soap Base Cup Grease. The following formula system has been used with success by an Atlantic Coast manu-

Grade No.	1	2	3	3½	4	5
Approximate A.S.T.M. worked penetration	375	300	250	225	175	150
Percent horse grease	4.48	5.18	5.77	7.11	7.66	9.20
Percent country tallow	6.72	7.86	8.63	10.67	11.49	13.80
Percent CaO (Quicklime)	1.40	1.68	1.87	2.23	2.40	2.88
Percent 28° Paffe oil 100 S.U.V. /100° F.	85.00	83.60	81.73	77.76	76.02	71.22
Percent water	1.40	1.68	2.00	2.23	2.43	2.90
Total material cost, cents per lb.	1.948	2.093	2.187	2.433	2.532	2.811
Manufacturing cost, cents per lb.	.300	.300	.300	.300	.300	.300
Container cost, cents per lb.	.405	.405	.405	.405	.405	.405
Total grease cost, cents per lb.	2.653	2.798	2.892	3.138	3.237	3.561

facturer, using open kettle jacketed for steam at 150 pounds per square inch.

In making a batch of the No. 2 grease on the above formulae the following quantities were used:

	Pounds
Horse grease	400
Country tallow	600
Calcium oxide	130
28° pale oil	6453
Water	140
Oil soluble yellow No. 7463	.75

All of the fats and 20 gallons of oil are charged into the grease mixer. The total time for weighing and adding these ingredients is about two hours.

Sixty gallons of water is used to slake the lime; it is then screened and run into the kettle while agitating.

The batch is stirred for 20 minutes with no steam turned on.

Cook for 4 hours at full steam pressure, gradually adding oil until about 110 gallons has been run into the kettle.

After five hours, a total of 386 gallons of oil should be in the mixer.

After the sixth hour, a total of 512 gallons of oil should be in the mixer.

At this time, the temperature of the batch should fall to about 220° F., and 3 gallons of water in which is dissolved ½ pound of NaOH should be added; also, .75 pounds of Yellow Dye.

At the seventh hour, the total volume of oil added should be about 822 gallons.

At the eighth hour, the total volume of oil in the mixer should be 870 gallons. At a temperature of 180 to 190° F., the batch is drawn through a 20-mesh per inch brass screen.

Horse-Fat Cottonseed Fatty Acid Grease. A New Jersey grease plant used the following formula for many years with considerable success. The soap charge was:

	Per cent
50 per cent Horse fat, 50 per cent by weight cottonseed oil fatty acids	84.0
Hydrated lime (16.7 pounds per 100 pounds fat)	14.0
Water	2.0

Various grades were prepared on the following formulae system:

Grease No.	Per cent Soap	Per cent 100 S.U.V./100° F. Paffe. Oil
00	12	88
0	14	86
1	17	83
2	20	80
3	25	75
4	33	67
5	50	50

V Horse Fat Greases. The formulae systems given in this section were used for a great many years by an Atlantic seaboard oil company.

"V" Cup Greases

	Light		Medium		Heavy	
	Per cent	Pounds	Per cent	Pounds	Per cent	Pounds
Horse fat	9.46	357	14.06	500	17.02	567
Calcium oxide	1.38	52	1.88	67	2.28	76
Camphor oil	.16	5	.17	5	.18	5
150 S.U.V./100° F. Paffe oil	89.00	450 gals.	83.89	400 gals.	80.52	390 gals.
Pounds CaO per 100 pounds fat	14.6		13.25		13.3	

"T" Cup Greases (Percent Formulae)

Grade No.	2	3	3½	4	5	6
Horse fat	9.89	15.60	16.02	17.76	20.37	28.81
Calcium oxide	1.44	2.09	2.15	2.38	2.73	4.27
Camphor	.17	.19	.29	.19	.20	.20
Dye solution	.33	.37	.44	.38	.34	.40
Filtered spindle oil	88.17	75.00	81.10	92.29	76.36	66.32
Pounds calcium oxide per 100 pounds fat	14.56	13.4	13.4	13.4	13.4	14.8

"MK" Cup Grease (Weight Formulae)

Grade Number	1	2	3	4	
Selected horse fat	370	370	370	370	Pounds
Calcium oxide	40	40	40	40	Pounds
400 S.U.V./100° F. Paffe oil	2400	2200	1850	1580	Pounds
Water	60	60	60	60	Pounds

European Fatty Acid Greases. This formula has been in use by a well known grease plant in Hamburg, Germany. It is evident that the use of fatty acids for the manufacture of cup greases has been appreciated there to a greater extent than in America.

	Per cent by weight
Spindle oil mixture, S.U.V. 100/100° F.	83.0
Cottonseed oil fatty acids	9.0
Mixed vegetable oil fatty acids	5.0
Wool fat fatty acids or wool fat stearin	1.0
Calcium hydroxide	2.0–3.0
Zinc oxide	.15
(Dropping point 100° C.)	

"FA" Cup Grease Formula.

This formula is used in Germany by a reputable grease manufacturer:

	Per cent by weight
100 S.U.V./100° F. spindle oil	78.5
Train oil (whale oil fatty acids)	18.0
Calcium hydroxide	3.5
(Dropping point 85° C.)	

Wool Fat, Train Oil Fatty Acid, Cup Grease.

Both this and the foregoing grease may be compounded with 0.5 to 2 per cent of zinc oxide to produce an opaque color and to possibly increase their lubrication value to a slight extent. This product has been manufactured in Germany:

	Per cent by weight
Spindle oil, 100 S.U.V./100° F.	85.2
Whale oil fatty acids	10.7
Wool fat olein	1.0
Wool fat stearin	1.0
Hydrated lime	2.1

Production in Fire-Heated Open Kettles

The manufacture of a well known cup grease used in pressure lubricating systems is made by the following formula in open coke fire-heated kettles. The finished grease has a melting point of 190° to 200° F. It is of the commercial grade known as a "soft No. 3," and is known to give excellent service when used in pressure guns for the lubrication of automobile chassis bearings.

Formula for Cup Grease No. 3

	Pounds
Horse fat	1000
Powdered hydrated lime	150
Water	275
Texas red oil, 300 viscosity	6000

QUALITY OF MATERIALS USED.

Mineral Oil. The mineral oil used in this grease has the following properties:

Saybolt viscosity at 100° F.	300
Gravity	21.0° Bé.
Flash point	340° F.
Fire	390° F.

Horse Fat. This ingredient should have a free fatty acid content of not less than 25 per cent and not over 45 per cent. Since it is found that the per cent acid varies considerably in different shipments it is customary to submit the fat to the laboratory for determination of the acid content and add oleic acid in order to bring it to a uniform value of 40 per cent.

PROCEDURE.

Charge 1000 pounds of melted horse fat in the open fire-heated kettle.

Run in 100 gallons of the mineral oil.

Mix in a fifty gallon drum the following: 150 pounds hydrated lime, 225 pounds water. Add this milk of lime mixture to the contents of the kettle and start the paddles which should rotate at about 30 r.p.m.

After mixing for ten minutes the fire should be started under the kettle. By having a forced draft of air on the coke fire close regulation of the heating may be effected.

A lime soap of proper consistency should be obtained in from 1¼ to 2 hours. This soap must be judged by the grease maker according to the directions given for the manufacture of cup grease in an open steam-jacketed kettle.

The temperature at this time should be 220° to 240° F. Cold oil in small quantities are added to the batch if it has a tendency to foam up badly.

The agitation is then stopped and the kettle walls scraped free of soap. Fifty pounds of water are added to the kettle and the agitation started again. The temperature should be about 210° F.

Draw the fire under the kettle.

The oil is continuously run into the kettle and should all be in after about three hours.

The final drawing temperature of the grease should be 180° to 190° F. At this temperature the grease is removed from the kettle by means of a gear pump and

screened through a 40-mesh screen, which breaks up any soap lumps which may be formed and removes gritty particles.

The grease may then be worked further and stored in large hoppers over the can filling machines. Such a grease is said to have a worked consistency.

Fire-Cooked Cup Grease. While there appears to be no technical reason why a fire-cooked cup grease may be superior to those for which other means of heating is provided, sales claims to this effect have been made. The formulae used by one Pacific Coast grease plant for making fire-cooked cup greases meeting U. S. Federal specifications and also sold for general purposes were:

Grade	Soft	Medium	Hard
A.S.T.M. worked penetration	300–355	240–290	190–230
300 S.U.V./100° F. Naphthene red oil, per cent by weight	88.483	81.274	75.815
No. 1 lard oil, per cent	10.253	....	....
CaO, per cent	1.120	1.880	2.228
Oleic acid, per cent	.143	....	....
Acidless tallow oil	....	16.844	21.956

The above formulae are on a water free basis. Small amounts of free fatty acid, such as oleic acid, may be added to the grease in its final stages in order to meet the Government requirement of 0.10 per cent maximum uncombined CaO.

Production by Pressure Cooking

The advantages of the application of pressure for promoting saponification reactions were well recognized prior to 1850. In 1838, Arthur Dunn [48] obtained a British patent for improvements in the manufacture of soap, consisting of first saponifying at higher temperatures, and under pressure. In 1861, British Brooman [49] obtained a patent for preparing a lubricant from caoutchouc, and other materials, by heating under pressure in a closed vessel. By 1865, Brooman [50] had perfected a process for the pressure saponification of fats with lye. He employed a horizontal cylinder, heated by fire near its center, and provided it with a horizontal shaft and paddles for agitation. Fat and alkali were heated together while agitating, no more water being used in the batch than was required in the finished product. There appears to be no evidence, however, that Brooman completed greases in this apparatus, and it is more likely that it was used either for making soap bases or normal detergents.

The Kuess [51] patents were granted in 1906 and dealt with the manufacture of lubricating greases and solidified oils by melting together equal amounts of stearin and vegetable oil, such as coconut oil, with petroleum, and adding a solution of caustic potash. The product was heated under pressure in an autoclave at 105-115° C., and then treated with a solution of a

[48] British Patent 7,783 (1838); see also: *Newton's London Journal*, **14**, conjoined series, 100; and **18**, 32; also: *Mechanics Mag.*, **32**, 351; *Meeson and Welsby's Report*, **5**, 675; and *Billings on Patents*," 130-131.

[49] British Patent 3,121 (1861).

[50] British Patent 1,540 (1865).

[51] British Patents 14,567 and 14,568 (June 26, 1906).

salt of such elements as boron, aluminum, or magnesium. Water was then evaporated as the last stage of the process.

In his patent application (July 17, 1913) R. Haddan,[52] acting as the agent of Dr. Hulsberg of Germany, pointed out the economy of grease lubrication over that of oil application, and also stressed the difficulties due to boil-overs when saponifying fats with lime in open vessels. He further emphasized the facts that, in open kettle saponification, the excesses of lime employed to secure more complete saponification remained in the greases as slightly abrasive material, and that the open kettle processes were slow and tedious, particularly with regard to the control of the final moisture content needed to disperse the lime soaps. The Haddan process consisted of boiling a mixture containing fatty oil, fatty acids, mineral oil, and a metal oxide, or hydrate, such as aluminum or magnesium oxides, or calcium hydroxide, together in an autoclave at pressures appreciably above atmospheric. It was stated that the distinctive feature of the process was the addition of fatty acid. Stearic, palmitic and oleic acids were mentioned as being suitable for this purpose. Suint and wool grease were refined by treatment with sulfuric and nitric acids, and then mixed with calcium hydroxide and mineral oil to produce a grease, by boiling under pressure. Pressures of about 65 to 75 pounds per square inch were employed and the maximum temperatures attained were of the order of 150° C. Pressures of 10 atmospheres and temperatures of 400° C., were mentioned but not considered necessary by the inventor. Either live steam at 400° C. was injected directly into the autoclave or water was at first added to the steam jacketed autoclave as in modern practice. After saponification was carried out for several hours the steam was shut off and the batch allowed to stand and cool for 12 hours. Water, which was in excess of that required in the grease, separated and was drawn off from the bottom of the autoclave. A typical formula was as follows:

Kg.	
2500	American lubricating oil
210	Tallow
40	Wool fat stearin

This mixture was charged into the autoclave and heated to about 100° C. with live steam. To the autoclave was then charged:

Kg.	
400	Oleic acid
260	Calcium hydroxide

The batch was then heated to 150° C. at a pressure of 5 atmospheres until a cooled sample indicated a satisfactory consistency. It is obvious that the above formula includes very large amounts of excess lime and that accordingly Haddan probably did not achieve one of the most important objects of his patent, the elimination of uncombined lime.

S. E. Bowrey is responsible for the statement that the firm of grease manufacturers with which he is associated in England, designed and put

[52] British Patent 16,480 (Apr. 23, 1914).

into use an autoclave for pressure saponification of soaps for use as greases bases, in the year 1917.

Hulsberg [53] obtained an American patent for his pressure saponification process in 1916. It was essentially the same as the British issue, but included references to the use of talgol as a soap stock, which was defined as hydrogenated or hardened fatty oil. Other fats or waxes considered desirable in his lubricant were: beeswax, ceresin, paraffin, wool fat olein, palm oil, cottonseed oil, and cottonseed oil stearin.

On July 14, 1923, Walter Atkinson,[54] assigned to the Standard Oil Company of California, his application for a patent covering the pressure saponification process now used in many grease plants, after considerable laboratory experimental work had been completed by Loud and Klemgard. It was the object of the inventor to promote complete saponification, with the reduction of free lime and free fat in the finished greases. It was also proved that greater uniformity was possible with autoclave cooking than when the open kettles were in use. It is, of course, obvious that in the latter process, part of the water introduced with the lime is gradually evaporated during the saponification period. Thus, to produce satisfactory open kettle cup greases it is necessary to first estimate the amount of evaporation which takes place by examining the soap base, and then adding an excess of water to the base, so that after cooking has been completed the desired amount of water will remain in the soap. Atkinson considered that, by conducting the saponification process in a closed vessel, under pressure, where the loss of water by evaporation could be prevented, variation in the water content of the soap mass could be eliminated, and the saponification substantially completed without difficulties due to insufficient water being present to promote saponification of the lime and fat, and obviating the necessity for the grease maker to judge at what period the reaction was complete. Experiments indicated that when using the autoclave, and the higher temperatures which it permitted, equivalent saponification could be obtained in about one third the time required when the process was carried out at atmospheric pressure.

With completion of the saponification phase of the process, the pressure may be released, the temperature lowered, and the desired amount of mineral oil mixed with the soap base, better control and a more uniform product resulted. The amount of water present in the grease and the method of introducing it were found to have an important bearing on the color of the finished lubricant. Using the minimum amount of water, which may be closely controlled in the pressure method, results in greases which are more transparent as well as uniform in color. It was learned that by releasing the pressure on the soap base and then incorporating the bulk of the mineral oil with the base, water being retained for hydration of the soap from the start of saponification, the grease produced would be of clear, dark red color. When the water was added, after much of the mineral

[53] U. S. Patent 1,200,617 (Oct. 10, 1916).
[54] U. S. Patent 1,736,302 (Nov. 19, 1929).

had been incorporated with the soap, a light colored grease of more opaque appearance, no doubt due to free emulsified water, was produced.

An 8000-pound batch of grease may be produced by the Atkinson process following the procedure as outlined:

Charge to the autoclave 340 pounds of beef tallow and 1300 pounds of lard oil. At the same time a mixture of the following materials is added:

Pounds	
180	Hydrated lime
60	NaOH
180	Water
300	Mineral oil

The mineral oil is added to prevent the formation of soap lumps and bunching.

The autoclave is closed, paddle agitation started, and steam turned on in its jacket, at a pressure of 50 to 150 pounds per square inch. A pressure of 50 pounds per square inch and a temperature of 265° to 275° F. will prevail within the autoclave. Under these conditions, saponification should be complete in approximately one half hour.

The steam in the jacket is then shut off, and the pressure on the soap released, either by means of a bleed valve, or preferably by discharging the soap under its own pressure into another grease mixer having an open top. Fairly uniform reduction of water content takes place by flash evaporation, leaving on the average about 76 pounds of water in the soap mass.

Mineral oil is then run into the soap in the open kettle and mixed in as fast as the soap will absorb it, the total quantity being sufficient to bring the total weight of the batch to 8000 pounds.

As previously mentioned, water may be added either at the beginning, or near the end, of the final oil blending phase of the process. The average quantity used is 50 pounds.

The grease may be drawn and screened at a temperature of 170° F.

Cup Grease (Pressure Saponification Process)

Further details of the foregoing process are described in the following method and formulae which have been used successfully by a Pacific Coast grease manufacturer for many years:

The lime soap being cooked under pressure is quickly and completely formed, eliminating all guessing as to the extent of saponification.

Where large quantities of grease are manufactured one small pressure kettle for the saponification process will be sufficient to produce soap for several of the usual open type steam jacketed mixers. Thus, a large quantity of grease may be quickly made without heavy investment in many kettles.

The process is subject to standardization and it is possible for inexperienced workmen to handle a batch satisfactorily.

The quality of grease produced is remarkably uniform and the chances for loss of batch are practically eliminated.

Formula for Cup Grease

Extra No. 1 lard oil	150 gallons
Water	14 gallons
180 viscosity western pale oil	900 gallons
Powdered hydrated lime	198 pounds
No. 1 inedible tallow	300 pounds
Oil of mirbane	6 pounds

The quality of the materials used is the same as in Formula 2.

PROCEDURE.

Charge in a closed pressure cooker of 12-barrel capacity 30 to 50 gallons of oil or grease tailings from previous batches.

Run into the pressure kettle 150 gallons of Extra No. 1 lard oil and 300 pounds of melted tallow.

Mix the following materials in a separate container in the order named and add them to the contents of the pressure kettle.

30 gallons mineral oil.
198 pounds powdered hydrated lime.
10 gallons water.

Close and fasten the manhole or opening in the pressure kettle. Test the kettle for leaks by admitting compressed air to the closed kettle till a pressure of 15 pounds is indicated by the gage.

Release the pressure, then close the relief valve.

Turn on the steam in the jacket of the kettle. A pressure of about 100 pounds is required. Rotate the paddles at about 38 R.P.M.

Maintain the pressure kettle at a temperature of not less than 300° F. and 50 to 80 pounds pressure for a period of twenty minutes.

In an open steam-jacketed mixer of 30 barrels capacity bring about 50 gallons of slop grease to a temperature of 225° to 265° F.

Open the valve on the 12-barrel pressure kettle and allow the lime soap to be discharged under its own pressure into the large open mixer. The release of the soap to atmospheric pressure causes the rapid evaporation of the greater part of the water in the lime soap. The transfer line from the pressure kettle should lead into the open mixer about one third the distance from its top.

Start the paddles on the open kettle and start adding oil to the soap.

Add oil till the total volume of oil in the kettle is 400 gallons.

The temperature of the batch should now be brought to 212° F.

Oil should then be added till the total volume of oil in the kettle is 750 gallons.

Add 4 gallons of water. The temperature should be 180° to 185° F.

Stir for 10 minutes.

Add 6 pounds of oil of mirbane and the balance of the mineral oil.

Stir for 15 to 20 minutes.

Fill at 160° to 170° F.

The properties of this product are about the same as the grease produced by Cup Grease formula "A."

DIFFERENT GRADES AND CONSISTENCIES

Twenty-five-barrel batches of cup grease of grades 1 to 5 may be made by the following formula and process:

Tallow, No. 2	1032 pounds
Lard oil, Extra No. 1	258 pounds
Water	18 gallons
Oil of mirbane	12 pounds

Pale oil, 180 viscosity

	Gallons
for No. 5 grade	450
for No. 4 grade	660
for No. 3 grade	800
for No. 2 grade	900
for No. 1 grade	1015

The volume of oil as given above should be corrected as indicated in item 15 if slop grease is to be reworked.

Charge not more than 50 gallons of slop cup grease in the pressure soap cooker.

Run in 1032 pounds of No. 2 tallow (which should be melted at a temperature of about 125° F.) and the lard oil.

Mix together in the order named and add to the cooker:

Hydrated lime	180 pounds
180 viscosity pale oil	40 gallons
Water	15 gallons

Close the pressure mixer and test it at a pressure of about 15 pounds air pressure. Start the agitating paddles.

Full steam pressure of 100 pounds per square inch should be admitted to the jacket of the mixer over a period of about five minutes.

The soap is cooked for twenty minutes at 300° to 330° F. under a steam pressure of approximately 65 pounds per square inch.

Charge 100 gallons of cup grease from a previous batch (which is known as slop grease, or tailings) in the open thirty barrel mixer. This grease acts as a plug in the kettle draw off to prevent the soap from being deposited there. Heat the slop grease to a temperature of 255° to 265° F., in order that the soap when discharged into the kettle will not be chilled.

Allow the soap in the pressure kettle to be discharged through a 4-inch pipe line into the open mixer. More than 90 per cent of the water in the soap is rapidly evaporated by this sudden release of pressure.

Apply full steam pressure of 100 pounds per square inch to the jacket on the bottom of the open mixer, gradually over a period of five minutes.

The 180 viscosity oil is then allowed to run into the mixer by gravity from graduated overhead tanks. Stirring at a paddle speed of about 35 R.P.M. is continued throughout the process.

When a total of 350 gallons of 180 viscosity oil has been added to the mixer the temperature of the kettle contents should not be less than 200° F.

Heat the grease to 222° to 226° F. and turn off the steam in the mixer jacket.

Add additional oil at a rate to insure thorough mixing with the soap, until the total oil added is 100 gallons less than given in 1. If the slop grease or kettle tailings are of different consistency than the grade being made, a correction should be made as in 20.

The remaining 3 gallons of water is then added followed by 12 pounds of oil of mirbane (nitro-benzene) and 50 gallons of 180 viscosity pale oil.

Continue stirring for 10 to 15 minutes, then take out a one pound sample. The lid of a five pound can may be conveniently used for this. After the sample has cooled it is tested for consistency and inspected for lumps, air bubbles, etc.

If the test is satisfactory the balance of 50 gallons of 180 viscosity oil is added.

The grease is then drawn into barrels or into the hopper of a machine for filling small cans of various sizes at the following temperatures:

Grade No. 5	is filled at	190°	to	200°	F.
Grade No. 4	is filled at	180°	to	190°	F.
Grade No. 3	is filled at	170°	to	180°	F.
Grade No. 2	is filled at	160°	to	170°	F.
Grade No. 1	is filled at	150°	to	160°	F.

A change from one grade to a softer grade may be made within reasonable limits by adding to the quantity of the harder grade in the mixer a percentage by volume of 180 viscosity oil as follows:

Grade 5 to 4	33% of the No. 5 Grade
Grade 5 to 3	55% of the No. 5 Grade
Grade 5 to 2	71% of the No. 5 Grade
Grade 5 to 1	88% of the No. 5 Grade
Grade 4 to 3	17% of the No. 4 Grade
Grade 4 to 2	29% of the No. 4 Grade
Grade 4 to 1	42% of the No. 4 Grade
Grade 3 to 2	10% of the No. 3 Grade
Grade 3 to 2	21% of the No. 3 Grade
Grade 2 to 1	10% of the No. 2 Grade

Temperatures and operating data for batches of number three grease, made on the foregoing formulae, using the autoclave for mixing the grease following saponification, are given below:

Saponification	Pressure, lbs./sq. in.	Temp. °F.
Start	Atmospheric	70
10 min.	17–46	144–296
20 min.	27–62	278–310
30 min.	32–65	287–317
40 min.	35–66	296–320
50 min.	37–67	290–321
1 hour	38–67	290–321
Steam off in autoclave jacket		
70 min.	29–55	268–321
80 min.	17–44	256–310
90 min.	7–37	250–295
100 min.	Atmospheric 30	232–284
110 min.	" 26	222–268
2 hours	"	221–261
Oil started		
2 hours, 10 minutes	"	218–255
2 " 20 "	"	210–247
2 " 30 "	"	215–280
2 " 40 "	"	214–280
2 " 50 "	"	208–282
3 " (steam in jacket)	"	220–280
3 " 10 minutes	"	240–275
3 " 20 "	"	223–280
3 " 30 "	"	223–274
3 " 40 "	"	212–280
3 " 50 "	"	203–270
4 "	"	198–253
4 " 10 "	"	198–252
4 " 20 "	"	196–244
4 " 30 "	"	195–232
4 " 40 "	"	188–220
4 " 50 "	"	192–210
5 "	"	184–210
5 " 10 "	"	184–206
5 " 20 "	"	180–205
5 " 30 "	"	176–205
5 " 40 "	"	176–205
5 " 50 "	"	174–204
6 "	"	172–204
6 " 10 "	"	170–204
6 " 20 "	"	169–203
6 " 30 " (Water added)	"	168–200
6 " 40 "	"	167–198
6 " 50 "	"	166–182
7 "	"	165–180
7 " 10 "	"	165–180
7 " 20 "	"	165–178
7 " 30 "	"	163–176
7 " 40 "	"	158–176
7 " 50 "	"	158–176
8 " (Draw)	"	158–175

Medium Cup Grease (Pressure Method)

In 1929, Klemgard [55] published further details for the preparation of a medium grade cup grease, in accordance with the principles established by the work done in connection with the Atkinson patent. A formula and method was outlined as shown below:

Per cent by Weight Finished Grease	Material	Pounds for 6000-pound Batch
16.68	Soap Stock (20 per cent No. 1 crude tallow) (80 per cent No. 1 lard oil)	1000.8
2.34	Hydrated lime (23.5 per cent Water of hydration)	140.4
.50	Water with soap	125.0
		25.0
	Final	25.0
80.48	180 pale asphalt base oil	4828.8

PROCEDURE.

Measure the soap stock in a steam-bottom measuring tank at 125° F. Have the laboratory determine the exact weight per gallon of the soap stock at 125° F. and gage that volume which would be equivalent to 1000.8 pounds. Run the soap stock by gravity into the pressure soap kettle.

Weigh out in a tank mounted on the scales the following material:

	Pounds
Hydrated lime	140.4
180 pale oil	250.0
Tap water	125.0

Mix these materials well and run the mixture into the pressure kettle.

Close the pressure kettle, start agitation and turn on full steam in the jacket.

Cook the soap under a pressure of 50 to 70 pounds per square inch for 40 minutes.

Heat jacket of open grease mixer to 280° F. and open valve in line connecting pressure soap kettle and the 1000-gallon open mixer. It is a good plan to have about 300 pounds of hot oil or slop grease in the open mixer in order to prevent lumping and chilling of the soap base. As the soap is discharged into the open mixer about 90 per cent of the water is rapidly evaporated from the mixture.

The 180 Pale oil is then started into open mixer at a rate which permits thorough mixing with the soap. The advantage of using the double motion agitation with scrapers for the sides and bottom of the kettle is apparent at this stage of the process.

When all but about 800 pounds of the oil has been run in and the temperature is 200 to 210° F. add the final water.

Continue the addition of the oil until it has all been mixed in.

Draw the grease through a 40-mesh screen at a filling temperature of 165° F.

Pressure Saponification Process (General Discussion)

The Sowers Manufacturing Company,[56] makers of autoclave suitable for use in this process, have published several articles on the advantages of closed kettles in grease making. One of their discussions follows:

"The most simple equipment for carrying out the pressure saponification process is the use of a single autoclave. Into the closed Steam-jacketed kettle, equipped with a double-motion, scraping type agitator, are charged measured amounts of (a) the fat to be saponified; (b) the saponifying agent usually made into a cream with oil or water; (c) water if needed and if the fat is so free from fatty acids that insufficient

[55] *Refiner Natural Gasoline Mfr.*, 68 (March, 1929).

[56] Reprinted from The Process Number of *Refiner Natural Gasoline Mfr.*, (February, 1932).

water will be formed by the reaction; (d) all of the lubricating oil needed to make the finished grease.

"The pressure mixer is closed, agitator started and steam turned into the jacket. After a predetermined time steam is turned off and the jacket vented and slowly filled with cold water; then, while agitation continues, cooling water is circulated through the jacket until the grease has cooled sufficiently to be drawn off without losing its moisture. This cooling may be continued, if desired, until the grease is cool enough to be run directly into packages.

"Advantages of this method are that the uncertainty of the human element is removed since the entire process is standardized; using the same raw materials each batch of finished grease is exactly like each previous batch. The disadvantages are that each batch can be only as large as the capacity of the pressure mixer; that certain oils are darkened by prolonged heating; and that the presence of the large volume of oil needed for the finished grease slows up the reaction of saponification. In consequence, this method is used chiefly for hard-to-make specialities which bring good returns, such as lead base transmission lubricants, water pump greases, etc., rather than for low priced products made in large volume, such as cup greases.

"There is a modification of the method just described. The base is made under pressure without the oil being present, oil under pressure being later added and worked in before the pressure is released. This works well where saponification cannot be carried out properly when reacting agents are diluted by the oil, yet where the moisture content must be so carefully controlled that it is not practical to work the oil into the base in an open mixer.

"The most generally accepted method of installing equipment for making greases by the pressure method is to mount a relatively small autoclave over two or more open grease mixers. Here the pressure mixer makes only the base, finishing being done in open kettles. Since saponification and finishing are thus carried on separately, they may be carried on simultaneously, one pressure mixer being used to supply base alternately for two or more open kettles.

"The pressure mixer is charged only with the fat, the saponifying agent, a little water, and in some cases, a little oil so that the resulting base will not be too heavy or too hard to work oil into later on, in the open kettles.

"With steam in the pressure mixer jacket at 125 to 150 pounds an internal pressure of 50 to 85 pounds is developed. (One large pressure mixer is operating satisfactorily with steam in the jacket at 80 pounds.) Saponification is practically complete in 15 to 20 minutes, although usual practice is to hold the base under pressure for 30 to 60 minutes. One user of this method has reported that he has never found more than one-tenth of one per cent of unsaponified fat after holding under pressure for 30 minutes.

"The base is then discharged into one of the open kettles, losing much of its moisture by 'flash evaporation' in discharge. This gives a nearly dry base to which definite amounts of oil and water are added for finishing.

"Most installations are made with the pressure mixer mounted on the floor above the open mixers so that the soap drains by gravity out of the discharge lines. A few recent installations have the top of the pressure mixer level with the tops of the open mixers, using the pressure generated in the pressure mixer to force the soap up into the tops of the open kettles. The disadvantage of having to remove soap from the lines after each batch is blown is more than offset by the simplified installation and the convenience in operation resulting from having all mixers on one floor.

"Including time required for charging and discharging the pressure mixer and for bringing the batch up to temperature and pressure, a six-barrel batch of base is usually made in from one to two hours, with larger amounts requiring somewhat longer time. For finishing the grease in open kettles with double-motion, scraping type agitators, from 1½ to three hours are required. In practice, the combination of these operations results in two, and in some cases, three batches of grease from each open kettle per day; as compared with the older practice of making only one batch per kettle per day.

"The use of pressure saponification not only improves the uniformity of the grease, reduces the chance of lost batches and permits grease manufacture by less experienced operators, but also further reduces manufacturing cost by cutting down the 'cost-per-pound-of-grease' that must go for equipment, maintenance, steam, power and labor."

The Subkow Process

In carrying out the original Atkinson process, the soap base from the autoclave was discharged under its own pressure through a pipe to a point several feet above the bottom of an open grease mixer. In 1931, Subkow [57] applied for a patent covering the discharge of the hot soap into a body of relatively cold oil at a point below the surface of the oil. His object was to secure still better control of the water content than Atkinson. He recognized that an excess of water present in the finished grease caused it to become a yellowish paste-like material, and that with such small amounts of water required in the grease (.5 to 2.5 per cent by weight) its control was an important item in securing greases of uniform appearance. He called attention to the fact that in open kettle saponification the rate of water evaporation might be greater than anticipated by the operator, that excess water may be lost before saponification was completed and that the result of deficient hydrolysis was a grease containing both free fatty acids and free alkali.

Subkow pointed out that in pressure saponification processes such as the Hulsberg method, in which large amounts of mineral oil are present during the cooking period, the high dilution increases the time factor, and that such greases usually contain excessive moisture. In the Subkow method the fats and hydrated lime are cooked under pressure with a predetermined amount of water required to completely hydrolyze the fats but not constituting an excess over that necessary in the grease. No appreciable amount of water is allowed to escape, either during the saponification process or when the soap is discharged from the autoclave. If desired, additional amounts of water may be added to the soap during the period when it is being mixed with the mineral oil; however, Subkow prefers to regulate the water at the beginning in the autoclave. At the completion of saponification the autoclave contents may be cooled and further quantities of oil added, or preferably the batch discharged at full maximum saponification temperature into a body of oil, at a sufficiently slow rate to avoid splattering of the soap on the kettle sides. This reduces the tendency towards the formation of soap lumps in the finished grease, even when proper screening has been employed.

In carrying out the process one autoclave and two open mixers are employed. The soap making materials are first measured into a small mixing kettle, provided with both paddle and compressed air agitation. The fats used may be tallow, cottonseed oil, lard oil or olive oil. A mixture of tallow and cottonseed oil is preferred. To this mixture is added the proper amount of hydrated lime or caustic soda, or mixtures of alkali, depending on the type of grease being manufactured. Water is then added in a sufficient quantity to effect saponification, but not more than will be needed in the finished grease. A small amount of mineral oil is also added to prevent bunching and lump formation during the autoclave treatment. The soap ingredient mixture is pumped through a centrifugal pump to the autoclave,

[57] U. S. Patent 1,871,760 (August 28, 1934).

where it is cooked under pressure for a total period of about two hours. The steam pressure in the jacket is maintained at 90 to 120 pounds per square inch, the temperature of the autoclave contents being 220 to 350° F. The temperature will vary with the pressure prevailing within the autoclave, which may be 15 to 75 pounds per square inch, or higher, and may be increased by admitting compressed air. During saponification continuous paddle agitation is carried on. During the saponification, as carried out in the autoclave, no water should be permitted to escape. The charge to the autoclave should be so calculated that at the end of the reaction the water content of the mass will be the predetermined amount required to properly hydrate the lime soap. If fatty acids are present in the soap stock in appreciable amounts, then stoichimetric calculation should be made for the quantity of water produced by neutralization. In order to reduce the soap to desired consistency and to disperse the soap in a further quantity of oil, the soap mass is transferred through a pipe line and discharged into a body of the oil. Subkow mentions a viscosity 100 to 200 S.U.V. at 100° F., heated to a temperature of 140 to 160° F., as the suitable viscosity and temperature of the oil charged in the open mixer. Not all of the oil intended to be used in the grease should be placed in the open kettle at one time. Discharge of the soap base should be controlled at a slow rate in order to avoid blowing the soap from the open kettle. The agitators in the open kettle should be in operation during the transfer of the soap in order to promote dispersion as it is discharged into the body of oil. The discharge period should be about 20 to 30 minutes. After discharge, the temperature of the mass should be about 175° F., and preferably not over that at which evaporation of water would take place in substantial amounts. The use of a closed kettle for reducing the soap and introducing the soap and additional oil under pressure, and by cooling the mixture with water in the pressure kettle jacket, loss of water may be definitely avoided. After adding any additional amount of oil, stirring for 20 minutes, and adding oil of myrbane and dye if needed, the drawing temperature for the particular grade being made is reached and the batch pumped off into packages. Further details of a typical batch as suggested by Subkow are:

Charge in the autoclave after mixing in the open kettle agitator:

Pounds	Material	Per cent
220	Prime tallow	61.2
25	Cottonseed oil	6.9
35	Hydrated lime	9.7
75	100 S.U.V./100° F. mineral oil	20.8
5	Water	1.4

Close the autoclave, turn on agitating paddles, and cook with 100 pound steam in the jacket for 2 hours.

During this period, the maximum pressure within the autoclave will reach 35 pounds per square inch, and the maximum temperature will be about 285° F. The soap produced will weigh about 360 pounds, will have a water content of about 1.8 per cent, and will have very low contents of free lime or free fatty acids.

Charge in the open mixer 910 pounds of the mineral oil and heat it to 149° F.

Discharge the soap into the bottom of the open mixer over a period of 20 to 30 minutes. A temperature of 180° F. may be attained, just after discharge of the soap.

After stirring for 20 minutes a final temperature of 190 to 200° F. may be reached. Additional water to bring the total water content to 1.7 per cent by weight, may be added at this point.

Add 50 pounds of mineral oil, oil of myrbane and dye as desired.

Adjust the batch to a temperature of 190° F. and draw the grease from the kettle.

German Experience with the Pressure Saponification of Lime Base Greases

The Hulsberg process, in which live steam was injected directly into the autoclave, was in use by a well known German grease plant during the years following 1915. These so-called "Ossagol" greases were manufactured in an autoclave with direct steam saponification and ordinarily contained from 0.25 to 1.0 per cent by weight free fatty acids. Their manufacturers claimed that no complaints were received due to the presence of this amount of free fatty acids by customers, nor were complaints on account of bearing corrosion at all serious. The product was usually made with about 20 per cent by weight of fat and following the saponification with direct steam, was drawn off from the autoclave in a liquid condition into trays. After cooling, the heavy grease base was forced through a sieve and then the necessary amount of oil worked in to obtain greases of desired consistency. The manufacturers recognized the fact that such greases contained excess water due to the injection of live steam with little subsequent evaporation, and that they had a grainy structure on account of pressing the cold grease base through the screen. The heat stability of the early Ossagol greases was not appreciably better than greases normally prepared at atmospheric pressure with excess of lime. The saponification under pressure with live steam was, therefore, finally abandoned and an open still was adopted, which in this particular plant was found to produce grease of improved quality, with regard to yields, dropping points, clarity, and water content.

In 1931, however, and in view of the success attained by American grease makers with pressure saponification, experiments were again undertaken with the autoclave. In the German experiments a still of 1500-liters capacity was used. It was equipped with internal heating coils and provided with external direct-firing. The maximum pressure obtainable with it was only about 30 pounds per square inch, and the maximum temperature of the soap base reached only about 135° C. (266° F.), whereas in American practice the pressures are ordinarily 65 to 100 pounds, and temperatures range from 160 to 170° C. (320 to 338° F.).

In carrying out the German process the hydrated lime, and a very small amount of caustic soda solution, are mixed together with mineral oil and injected through a nozzle into the fat charged to the autoclave, the autoclave paddles being in motion. The autoclave is then closed, steam heat turned on in its coils and the direct fire below it is started. After about one hour, a temperature of 135° C. is attained and the pressure has reached two atmospheres or slightly more. Further heating may be necessary in order to bring the quantity of neutral fat in the soap base to a negligible amount, and its free fatty acid content to less than ½ per cent by weight. At higher

temperatures and pressures saponification could, of course, be completed in less than the two hours sometimes needed in these experiments. The autoclave is emptied through a line passing through its top in the manner of a siphon, but using the steam pressure existing within the vessel, and its contents transferred to an open grease mixer. The oil in the open mixer has been heated to an appropriate temperature to avoid chilling the soap, which consisted of about 30 per cent by weight of the autoclave charge.

It was concluded by the German investigators that the autoclaved greases could be made which were better in appearance than those obtained by the usual process in open stills, as they were generally more transparent and of smoother texture. It was also demonstrated that from one-third to one-fourth less time was required by autoclave saponification than by using open-fired stills. Nearly 50 per cent of the time ordinarily used for mixing the soap base with further quantities of oil was saved when using the pressure method. The dropping points of the pressure saponified greases were found to be 5 to 10° C. lower than those made in the open still process. However, since in the latter substantial amounts of excess lime were employed, and whereas in the autoclave experiments the theoretical amounts of lime were being employed, some differences in dropping points on this account may be expected; also, the system of adding large amounts of oil to the soap base while being saponified in the autoclave may have some influence.

No appreciable differences were observed in autoclaved greases made with fatty acids and those made with whole fats. The greases containing about 30 per cent of soap and taken directly from the autoclave had better stability on being heated for 105° C. (221° F.) for one hour than the same greases after being mixed with further quantities of oil in open mixers. Satisfactory heating test results were usually obtained by adding small amounts of free fatty acids, such as oleic acid, naphthenic acids, wool grease acids, or even wool grease, to the soap base in the autoclave or to the grease being finished in the open mixer. Satisfactory heat stability was obtained in all cases for greases containing from 12 to 15 per cent by weight of soap and ¼ to ½ per cent by weight of free added oleic acid. The heating tests were made by placing a small quantity of grease in a shallow tray in an oven for one hour at 105° C. After this period, the tray was taken out and cooled. No oil separation, nor precipitation of soap or formation of granular material should occur. A thin layer of foam caused by the evaporation of water may be disregarded.

In many cases lime base cup greases are objected to on the grounds that they separate when used in high speed ball or roller bearings, or may be subjected to high temperatures, and the use of wool grease has been common practice as a means of stabilizing such greases for special uses of this kind. In some cases, the wool fat greases have been saponified with too great excesses of lime and this is objectionable on account of the lapping effect the free lime has with respect to the fine finishes and close fits of antifriction bearings.

It is claimed that greases with poor heat stability, as indicated by the foregoing test, break down at temperatures below their dropping points,

and separate into a free oil portion and soap or thick grease. Greases having high heat stability characteristics after being heated to above their melting points and cooled again, are more likely to become smooth, homogeneous and satisfactory lubricants in service. Any increased corrosion due to the presence of free fatty acids must be overcome by including various anti-corrosive materials, or the batch so closely controlled with regard to the free lime and free fatty acid contents that corrosion is not an important factor.

Dropping Points of Pressure Saponified Greases

Experiments were made with a 1.5-liter copper autoclave. The charges consisted of 300 grams of the fats indicated below, and the theoretical quantity of lime to saponify the fat, plus 100 grams of spindle oil. The mixture was heated in the autoclave at temperatures of 170 to 175° C. (338 to 347° F.), and at pressures of 70 to 85 pounds per square inch, for a period of twenty minutes. After this saponification, the pressure was reduced to atmospheric, oil added to give a grease of about number three grade, and 0.3 per cent of forty ° Bé. NaOH added drop by drop. In cases where greases of very high gloss are desired 0.3 per cent by weight of forty ° Bé. KOH solution may be substituted for the caustic soda. It will be seen from the following table that soap stock consisting of a predominating quantity of tallow with lard results in maximum dropping points. The addition of substantial amounts of stearic acid to the following soap stocks will slightly increase the dropping point.

Soap Stock Mixture	Ubbelohde Dropping Point ° C.	° F.
Soya bean oil (12 per cent)	72	161.6
Horse fat (10 per cent by weight in grease)	90	194
Hog fat (8 per cent lard in grease)	92	197.6
Sheep tallow, 45 per cent Rape oil fatty acids, 32 per cent Vegetable fatty acids, 23 per cent } 10 per cent by weight	90	194
Lard, 20 per cent Sheep tallow, 80 per cent } 10 per cent by weight	94	201.2
Horse oil fatty acids (10 per cent by weight)	92	197.6

Two Russian investigators, Velikovskii and Varentzov,[58] have reported that slightly higher melting points were possible for lime base cup greases made by pressure saponification. They prepared a series of greases from 15 per cent by weight of vegetable oil, 80 per cent mineral lubricating oil, 3 per cent hydrated lime (an excess of about 38 per cent) and 2 to 5 per cent of water, the pressures in the autoclave being varied from 5 to 10 atmospheres (74 to 147 pounds per square inch). Pressure saponification and even pressure blending of the mineral oil appeared to improve the greases from the standpoint of being thinned out by heat. In addition to the improved viscosity-temperature ratio of the pressure saponified greases, it was noted by these investigators that the time factor was appreciably shortened for increased pressures.

[58] *Neftyanoe Khozyaistvo* (12), **26**, 60-3 (1934).

Standardized Cup Grease Formulae (Pressure Saponification Method)

The following formulae have been well standardized and are known to give consistent results. The "300 Pale Oil" referred to is the same as that for which the chief characteristics have been given in Chapter II, page 38, as "300 California Pale Oil." The tallow and lard oil used should meet the requirements as indicated in the suggested purchase specifications for these materials (See Chapter II).

Grease Cup No.	Average Weight of Batch, Pounds	Constituents	Gallons	Pounds Net	Per cent by Weight
0	6800	Tallow	45	343.22	5.04
		Lard oil	15	114.40	1.68
		30° Bé. caustic soda	¼	.62	.01
		Hydrated lime		63.00	.93
		Water		43.00	.64
		300 pale oil	820	6245.76	91.70
				6810.00	100.00
1	6600	Tallow	75	572.00	8.67
		Lard oil	25	190.70	2.89
		Hydrated lime		105.00	1.59
		30° Bé. caustic soda	0.5	1.24	.02
		Water		79.20	1.20
		300 pale oil	742	5651.86	85.63
				6600.00	100.00
2	5550	Tallow	75	572.00	10.29
		Lard oil	25	190.70	3.43
		Hydrated lime		105.00	1.88
		30° Bé. caustic soda	0.5	1.24	.02
		Water		83.40	1.50
		300 pale oil	605	4607.66	82.88
				5560.00	100.00
3	4650	Tallow	75	572.00	12.18
		Lard oil	25	190.70	4.06
		30° Bé. caustic soda	0.5	1.24	.03
		Hydrated lime		105.00	2.24
		Water		75.14	1.60
		300 pale oil	493	3751.92	79.89
				4696.00	100.00
4	4625	Tallow	90	686.50	14.64
		Lard oil	30	228.80	4.88
		30° Bé. caustic soda	½	1.24	.02
		Hydrated lime		125.00	2.67
		Water		93.80	2.00
		300 pale oil	467	3554.66	75.79
				4690.00	100.00
5	5200	Tallow	150	1144.05	21.62
		Lard oil	50	381.35	7.21
		30° Bé. caustic soda	1	2.49	.05
		Hydrated lime		210.00	3.97
		Water		142.83	2.70
		300 pale oil	448	3409.28	64.45
				5290.00	100.00

Cup Grease No.	Average Weight of Batch, Pounds	Constituents	Gallons	Pounds Net	Per Cent by Weight
6	4200	Tallow	150	1144.05	26.73
		Lard oil	150	381.35	8.91
		30° Bé. caustic soda	1	2.49	.06
		Hydrated lime		210.00	4.91
		Water		115.56	2.70
		300 pale oil	319	2426.55	56.69
				4280.00	100.00

	Per cent by Weight
Liquid grease	
Cup grease No. 3	6.05
300 S.U.V. pale oil	93.94
	100.00
Tallow	.74
Lard oil	.25
30° Bé. caustic soda	.00
Hydrated lime	.14
Water	.10
300 pale oil	98.77
	100.00

Autoclave Loading (Lime Mixture, Measurement of Fats)

Blow out autoclave fat and lime inlet lines with air before charging equipment to guard against frozen valves or lines.

Measure 40 gallons of 300 Pale Oil into the 80 gallon lime mixing tank.

Add 105 pounds of hydrated lime and stir for at least ten minutes with "Lightnin" mixer. (If double batch is being prepared the first mixture can be made with 100 pounds and the second with 110 pounds of lime.) Run in oil to make a total of 80 gallons.

Add 10 gallons of water for each mix, and continue stirring.

Measure into the autoclave measuring tank the following fats, making sure that they are warm enough to be in a completely fluid condition (not over 140° F.).

	Single batch	Double batch
Tallow	75 gallons	150 gallons
Lard oil	25 gallons	50 gallons

Run lime mixture into autoclave and then follow with ½ gallon of 30° Bé. caustic soda for each single batch. In no case add caustic soda direct to lime mix. Then run in 25 gallons of 300 Pale Oil to wash out the system.

After standing at least 10 minutes, check fat measuring tank with water finding paper. Run in the fat mixture, as measured.

Saponification

Close the charging valves (lime and fat inlet valves) to autoclave.

Start the agitating paddles.

Turn on steam in autoclave jacket and regulate at 75 pounds per square inch. Inspect trap to insure that it is functioning.

Time of cooking:

If starting with the autoclave cold, continue the agitation and cooking at a jacket pressure of 75 pounds per square inch for 1 to 1½ hours. (During cold weather 1½ hours, hot weather only one hour.)

If autoclave is still hot from previous batch, cooking shall be continued for 45 minutes (40 to 50 minutes allowed). The period of cooking starts at the time steam is turned on until batch is dropped.

The autoclave temperature shall be 240 to 270° F. at the time the soap is discharged. The internal pressure of the autoclave should be 15 to 45 pounds per square inch, at the completion of saponification.

Record time, temperatures and pressures in autoclave record book.

Reduction of the Soap

Prior to discharging the soap from the autoclave into the open mixers, 100 gallons of 300 Pale Oil, or about this quantity of slop cup grease, is placed in each open mixer and heated to about 190° F.

The soap is discharged directly into the open mixers, the agitator paddles not being in motion.

As soon as the soap has been cleared from the transfer line by blowing with compressed air, the kettle paddles are started and 300 Pale Oil run in at low rate until a "quick consistency" test of a sample of the grease indicates that on further cooling the A.S.T.M. Worked Penetration Test will meet the desired specifications. Small quantities of water may be added if the appearance of a cooled sample of the grease indicates a grainy condition.

Quick Consistency Test

A 1-pound sample of the grease is taken from the kettle when the grease maker estimates from the amount of oil added to the batch, and from a finger test of the grease, that it is near the desired consistency, but not softer than grade to be made. This 1-pound sample is stirred with a thermometer until the temperature is uniformly just 150° F. The stirring should be done slowly so as to avoid beating air into the grease. The surface of the grease is then carefully smoothed and the A.S.T.M. penetration test made but at a grease temperature of 150° F.

The foregoing greases should meet the following specifications for A.S.T.M. Worked Penetration at 77° F. It is, of course, true that aging will in some cases cause the grease to soften, and in others to harden, but it will be found that the quick test as suggested is quite useful in predicting the final worked penetration at 77° F. The following relations between the quick test at 150° F. and the A.S.T.M. Worked Penetration at 77° F., for the foregoing formulae are:

Cup Grease Grade Number	Specified A.S.T.M. Worked Penetration Range at 77° F. (A.S.T.M. D217-33T)	Worked Penetration Range at 150° F.
0	420–450	440–460
1	360–390	405–415
2	300–330	350–365
3	240–270	290–305
4	180–210	225–240
5	50– 80 (Unworked)	....

A graph of the relation between these two penetration tests is a valuable guide for the grease maker to follow.

Drawing Temperatures and Screening

The following temperatures of grease, which should be complied with when drawing the batch in order to avoid inconsistent grades, and the size of screen normally used, are listed below for each grade of the foregoing greases:

Cup Grease Grade No.	Drawing Temperature Range ° F.	Size of Wire Screen, Mesh per Inch
0	150–160	120
1	160–170	100
2	170–180	60
3	180–190	60
4	190–200	40
5	200–210	20
Liquid grease	100–150	120

FORMULAE.

Greases made on the foregoing formulae may also have computed formulae as follows in which the glycerol remaining in the greases is included with the soap:

Cup Grease Grade No.	Liquid	0	1	2	3	4	5	6
Composition, per cent by weight:								
Water	.10	.64	1.20	1.50	1.60	2.00	2.70	2.70
Soap	1.13	7.66	13.17	15.62	18.51	22.21	32.85	40.61
Mineral oil 300 S.U.V.	98.77	91.70	85.63	82.88	79.89	75.79	64.45	56.69
Soap Formulae, per cent by weight:								
Tallow	65.82	(same for all grades)						
Lard oil	21.93	(same for all grades)						
NaOH	0.15	(same for all grades)						
$Ca(OH)_2$	12.10	(same for all grades)						

Guaranteed Specifications

Cup greases made in accordance with the foregoing formulae should comply with specifications as indicated below:

Cup Grease Grade No.	Liquid	0	1	2	3	4	5	6
Worked penetration at 77° F. (A. S. T. M.-D217-33T)	...	420–450	360–390	300–330	240–270	180–210	135–165	50–80 (Unwrkd)
Average soap content (A.S.T.M.-D128-27)	3.0	6.0	9.0	12.0	16.0	20.0	25.0	30.0
Melting point ° F. (Ubbelohde) min.	...	...	175	180	185	190	195	200
Free alkali (percent NaOH) (A. S. T. M.-D128-27) max.	0.20	0.30	0.35	0.40	0.45	0.50	0.60	0.70
Free acid (percent oleic) (A. S. T. M. D128-27) max.	0.10	0.20	0.30	0.30	0.50	0.50	0.50	0.50
Ash, per cent by weight (A. S. T. M. D128-27) max.	0.60	1.60	1.80	2.40	2.60	3.00	3.50	4.00
Water by distillation, per cent by weight (A.S.T. M.) D95-30) max.	1.25	1.25	1.75	2.00	2.50	3.00	3.00	3.50
Corrosion (No. 530.4)	(None for all grades)							
Mineral oil, S.U.V./100° F.	(300–325 for all grades)							
A.S.T.M. color	(Maximum 3 for all grades)							

Relation Between A.S.T.M. Soap Content and Per Cent Ash

From the results of a large number of soap and ash determinations on greases made in accordance with a system similar to the Standardized Formulae, it is concluded that on the average, for each one per cent of ash, the cup grease will contain 8.51 per cent of calcium soap.

Grease	Sample No.	Worked Penetration	Per cent Soap A.S.T.M.	Per cent Ash	Ratio of Soap to Ash
Liquid grease	1	...	1.67	...	...
Liquid grease	2	...	2.04	0.19	...
Cup grease No. 00	1	411	6.7	0.80	8.37
Cup grease No. 00	2	444	8.5	0.95	8.95
Cup grease No. 00	3	401	6.2	1.15	5.39
Cup grease No. 00	4	411	8.5	1.01	8.42
Cup grease No. 00	5	406	6.4	0.76	8.42
Cup grease No. 00	6	382	8.5	0.95	8.95
Cup grease No. 0	1	360	9.1	1.03	8.84
Cup grease No. 0	2	402	6.7	0.75	8.93
Cup grease No. 0	3	371	8.6	0.98	8.78
Cup grease No. 0	4	360	8.5	1.00	8.50
Cup grease No. 0	5	340	11.4	1.31	8.70
Cup grease No. 1	1	344	11.1	1.36	8.16
Cup grease No. 1	2	326	11.3	1.34	8.43
Cup grease No. 1	3	338	12.0	1.31	9.16
Cup grease No. 1	4	328	10.5	1.29	8.14
Cup grease No. 2	1	298	14.8	1.77	8.36
Cup grease No. 2	2	298	14.6	1.68	8.69
Cup grease No. 2	3	292	13.6	1.44	9.45
Cup grease No. 3	1	250	15.3	1.82	8.41
Cup grease No. 3	2	266	14.4	1.66	8.67
Cup grease No. 3	3	254	15.7	1.73	9.07
Cup grease No. 3	4	270	16.6	1.87	8.88
Cup grease No. 3	5	273	15.7	1.77	8.87
Cup grease No. 3	6	271	16.2	1.88	8.62
Cup grease No. 4	1	208	19.2	2.12	9.06
Cup grease No. 4	2	227	18.8	2.09	9.00
Cup grease No. 4	3	214	20.0	2.26	8.85
Cup grease No. 5	1	117	31.9	3.79	8.42
Cup grease No. 5	2	118	30.6	3.53	8.67
Cup grease No. 5	3	178	25.9	4.88	5.31
Cup grease No. 5	4	107	31.4	3.59	8.75
Cup grease No. 5	5	96	31.4	3.56	8.82
Cup grease No. 5	6	102	31.4	3.51	8.95
				Average ratio	8.51

Relation Between Worked and Unworked A.S.T.M. Penetration

The lack of correlation between worked and unworked penetration values for greases made on the foregoing formulae is quite apparent in the following data:

Grade of Grease Sample	Worked Penetration	Unworked Penetration	Difference
	Company A		
4	224	225	−1
5	183	159	24
6	172	171	1

Grade of Grease Sample	Worked Penetration	Unworked Penetration	Difference
	Company B		
4	306	246	60
4	308	241	67
4	237	227	10
5	212	222	−10
5	215	207	+8
6	175	184	−9
6	167	146	21
6	175	153	22
7	157	143	14
	Company C		
4	220	172	48
4	216	178	38
4	293	218	75
5	195	151	44
5	169	120	49
5	197	128	69
5	199	171	28

Relation Between A.S.T.M. Worked Penetration and Soap Content

The results of a great number of A.S.T.M. soap analyses on various grades of cup grease made in accordance with the foregoing standarized formulae have shown that a definite relation exists with the worked penetration. The minimum and maximum soap contents for given A.S.T.M. worked penetrations at 77° F. are listed below:

A.S.T.M. Worked Penetration at 77° F.	A.S.T.M. Soap Content (by analysis)		Average Soap Content From Equation A
	Minimum	Maximum	
475	3.0	7.5	4.8
450	3.5	8.2	5.8
425	4.2	9.2	6.8
400	5.0	10.4	7.8
375	6.0	11.6	8.9
350	7.0	13.0	10.0
325	8.0	14.4	11.3
300	9.5	16.0	12.7
275	11.0	18.0	14.1
250	13.0	20.0	15.8
225	14.6	22.0	17.5
200	16.6	24.2	19.5
175	18.9	26.7	21.8
150	21.3	29.5	24.4
125	24.0	33.0	27.5
100	27.0	36.0	31.4

When all of the foregoing analyses were plotted on rectangular coordinate paper it was evident that the relation between penetration and soap content was a hyperbolic function, although the points were scattered over a fairly wide band as shown in the table. This empirical relation is expressed by the following general equation:

$$\log_{10} y = \log_{10} C - mx \log_{10} e, \text{ in which:}$$

y is the A.S.T.M. Worked Penetration at 77° F., between the limits of 475 and 100;
C is a constant;
m is the slope (when the data is plotted on semi-logarithmic paper);
e is the Napierian base, 2.718281 or $\log_{10} e$ is 0.43429;
x is the A.S.T.M. soap content.

It is, of course, obvious that the constants m and C may be greatly influenced by changes in soap stock, excess alkali, degree of hydration of the soap, completeness of saponification and the character of the oils used. However, it is believed that when grease making is finally placed on a thoroughly scientific basis all of these variables will be evaluated with reasonable accuracy and that such theories will be extended and become quite useful as a practical aid in predetermining yields and manufacturing conditions.

In simplified form the above general equation for the average yield of grease becomes:

Equation A $\log_{10} y = 2.80149 - 0.02563\, x$

The maximum yields which may be expected from the Standardized Cup Grease formulae (minimum soap contents) may be represented by the following equation:

Equation B $\log_{10} y = 2.75766 - 0.03112\, x$

The minimum yields are shown by the following relationship:

Equation C $\log_{10} y = 2.860948 - 0.02382\, x$

Similar equations may be worked out by any grease maker to cover the formula system, materials and process he uses.

Relation Between Soap Content and Apparent Viscosity, Shear Rate and Oil Viscosity

While it has been possible to express the empirical relationship between A.S.T.M. worked penetration and soap content, Arveson [59] has not been able to express the relationship between the soap content and apparent viscosity determined at constant shear rates as a simple equation. It is felt, however, that there should be some general relationship governing the flow of different grades of greases made with the same materials and by similar manufacturing methods. Arveson found that when plotting V_g/V_o as a function of V_oS on logarithmic paper his data fell in a definite narrow band. In these functions:

V_g is the apparent viscosity of a grease sample;
V_o is the apparent viscosity of the oil content of the grease;
S is the shear rate of the grease sample in reciprocal seconds. (See Appendix.)

Arveson also found that better correlation was obtainable when a soap factor, termed K, was introduced. He demonstrated that K was a function

[59] Ind. Eng. Chem., (6), 26, 628-634 (June 1934).

of the concentration of the soap in the lubricating grease and that it varied with the nature of the soap and the temperature at which viscosity determinations were made. V_g/V_o is dimensionless and the expression V_oS includes dimensions that are those of stress in the c-g-s system. Arveson concluded that if the absolute values of K were known they must have stress dimensions since only two dimensionless groups occur.

For a series of lime-fatty acid greases the following soap factors were reported:

Soap Factor K (Temperature 77° F.)	A.S.T.M. Soap Content Per Cent
0.1	1.8
1.0	3.0
10.	6.
100.	9.5
1000.	17.
10,000.	29.

The soap factor K is probably related to the elastic properties of the grease and its value is, no doubt, related to the theoretical "yield value." Arveson's work resulted in the following generalizations:

V_g/V_o	V_oS/K
100,000	0.0001
10,000	0.002
1,000	0.04
100	0.5
10	10.
5	100.
3	1000.
2	10,000.
1	100,000.

The curve represented by the above figures approaches a line having the slope of -1. On this basis as V_oS/K approaches zero, the required stress approaches the value KC, in which C would be a factor for conversion of K units to the C.G.S. system.

In other words, when $V_oS/K = O$, the stress in dynes per square centimeter is $G = KC$, and KC is the yield value depending on the soap concentration and is apparently independent of the oil viscosity.

Operating and Manufacturing Costs (Pressure Saponification)

While no comparable data is available it is probable that the operating costs for a grease plant using autoclave for pressure saponification of the fats is lower than those in which steam jacketed open kettles are used for this purpose. Where labor and coal are quite cheap it appears that direct firing may be even less costly than either of the foregoing methods. In one grease plant equipped with a single autoclave and several open steam-jacketed mixers for the manufacture of the "Standardized Cup Greases" just discussed, the total monthly production of greases amounted to about 900,000

pounds. The average monthly costs, exclusive of compounding materials, were approximately as indicated in the table:

(Grease Compounding)	$ per Month	
Direct supervision	198.30	
Operating labor	212.34	
Operating material	3.19	
Maintenance labor	31.57	
Maintenance material	8.49	
Cleaning expense, labor	53.97	
Cleaning expense, material	30.39	
Plant changes, labor	2.23	
Plant changes, material	.90	
Sub-Total		541.38
Utilities:		
Steam	33.12	
Water	2.55	
Electricity	14.76	
Total direct expenses		591.81
Total overhead expenses		1,007.19
Total direct and overhead expenses		1,599.00
Direct depreciation		729.01
Indirect depreciation		140.38
Total expense		2.468.39
Total expense cents per barrel of grease		117.7
Total expense cents per gallon of grease		2.14
Total expense cents per pound of grease		.276

Total Cost of Pressure Saponified Greases

A series of cup greases are costed in the tables below, each computation being indicated to show the usual method employed. The "A" cost is the direct out-of-pocket cost, "B" includes the out-of-pocket and also depreciation and overhead, and "C" is the out-of-pocket plus overhead only.

Formulae		Unit Cost					
	Gallons	A	B	C	A	B	C
		Cup Grease No. 0					
Tallow	7.50 (56.9 lbs.)	5.0	6.0	5.5	$ 2.85	3.41	3.13
Lard oil	2.50 (19.0 lbs.)	10.0	12.0	11.0	1.90	2.28	2.09
Hydrated lime	(10.5 lbs.)	0.79	0.95	0.87	.08	.10	.09
30° Bé. caustic soda	0.05 (.124 lbs.)	2.77	3.32	3.05	...	.01	...
Water	1.46	0.025	0.03	0.0275	...	...	...
300 pale oil	147.60	2.90	4.35	3.40	4.28	6.42	5.02
Total material cost	159.11 Gals.				$ 9.11	12.22	10.33
Total material cost	1 Gal.	(Expressed in cents)			5.73	7.68	6.49
Grease comp. cost	1 Gal.	(Expressed in cents)			1.22	2.14	1.53
Cost 1 gallon		(Expressed in cents)			6.95	9.82	8.02
Cost 1 lb. cup grease No. 0 (7.70 lbs. per Gal.)		(Expressed in cents)			0.90	1.28	1.04

Formulae	Gallons	A	B	C	Unit Cost A	B	C
		Cup Grease No. 1					
Tallow	7.50 (56.9 lbs.)	5.0	6.0	5.5	$ 2.85	3.41	3.13
Lard oil	2.50 (19.0 lbs.)	10.0	12.0	11.0	1.90	2.28	2.09
Hydrated lime	(10.5 lbs.)	0.79	0.95	0.87	.08	.10	.09
30° Bé. caustic soda	0.05 (.124 lbs.)	2.77	3.32	3.05	...	.01	...
Water	1.26	0.025	0.03	0.0275	...	...	...
300 pale oil	101.60	2.90	4.35	3.40	2.95	4.42	3.45
Total material cost	112.91 Gals.				$ 7.78	10.22	8.76
Total material cost	1 Gal.	(Expressed in cents)			6.89	9.05	7.76
Grease comp. cost	1 Gal.	(Expressed in cents)			1.22	2.14	1.53
Cost 1 gallon		(Expressed in cents)			8.11	11.19	9.29
Cost 1 lb. cup grease No. 1 (7.73 lbs. per Gal)		(Expressed in cents)			1.05	1.45	1.20
		Cup Grease No. 2					
Tallow	7.50 (56.9 lbs.)	5.0	6.0	5.5	$ 2.85	3.41	3.13
Lard oil	2.50 (19.0 lbs.)	10.0	12.0	11.0	1.90	2.28	2.09
Hydrated lime	(10.5 lbs.)	0.79	0.95	0.87	.08	.10	.09
30° Bé. caustic soda	0.05 (.124 lbs.)	2.77	3.32	3.05	...	.01	...
Water	1.04	0.025	0.03	0.0275	...	...	...
300 pale oil	72.40	2.90	4.35	3.40	2.10	3.15	2.46
Total material cost	83.49 Gals.				$ 6.93	8.95	7.77
Total material cost	1 Gal.	(Expressed in cents)			8.30	10.72	9.31
Grease comp. cost	1 Gal.	(Expressed in cents)			1.22	2.14	1.53
Cost 1 gallon		(Expressed in cents)			9.52	12.86	10.84
Cost 1 lb. cup grease No. 2 (7.75 lbs. per Gal.)		(Expressed in cents)			1.23	1.66	1.40
		Cup Grease No. 3					
Tallow	7.5 (56.9 lbs.)	5.0	6.0	5.5	$ 2.85	3.41	3.13
Lard oil	2.5 (19.0 lbs.)	10.0	12.0	11.0	1.90	2.28	2.09
Hydrated lime	(10.5 lbs.)	0.79	0.95	0.87	.08	.10	.09
30° Bé. caustic soda	0.05 (.124 lbs.)	2.77	3.32	3.05	...	.01	...
Water	0.97	0.025	0.03	0.0275	...	...	...
300 pale oil	51.50	2.90	4.35	3.40	1.49	2.24	1.75
Total material cost	62.52 Gals.				$ 6.32	8.04	7.06
Total material cost	1 Gal.	(Expressed in cents)			10.11	12.86	11.29
Grease comp. cost	1 Gal.	(Expressed in cents)			1.22	2.14	1.53
Cost 1 gallon		(Expressed in cents)			11.33	15.00	12.82
Cost 1 lb. cup grease No. 3 (7.81 lbs. per Gal.)		(Expressed in cents)			1.45	1.92	1.64
		Cup Grease No. 4					
Tallow	7.50 (56.9 lbs.)	5.0	6.0	5.5	$ 2.85	3.41	3.13
Lard oil	2.50 (19.0 lbs.)	10.0	12.0	11.0	1.90	2.28	2.09
Hydrated lime	(10.5 lbs.)	0.79	0.95	0.87	.08	.10	.09
30° Bé. caustic soda	0.05 (.124 lbs.)	2.77	3.32	3.05	...	.01	...
Water	0.90	0.025	0.03	0.0275	...	...	...
300 pale oil	36.90	2.90	4.35	3.40	1.07	1.61	1.25
Total material cost	47.85 Gals.				$ 5.90	7.41	6.56
Total material cost	1 Gal.	(Expressed in cents)			12.33	15.49	13.71
Grease comp. cost	1 Gal.	(Expressed in cents)			1.22	2.14	1.53
Cost 1 gallon		(Expressed in cents)			13.55	17.63	15.24
Cost 1 lb. cup grease No. 4 (7.86 lbs. per Gal.)		(Expressed in cents)			1.72	2.24	1.94

	Formulae Gallons	A	B	C	Unit Cost A	B	C
		Cup Grease No. 5					
Tallow	7.50 (56.9 lbs.)	5.0	6.0	5.5	$ 2.85	3.41	3.13
Lard oil	2.50 (19.0 lbs.)	10.0	12.0	11.0	1.90	2.28	2.09
Hydrated lime	(10.5 lbs.)	0.79	0.95	0.87	.08	.10	.09
30° Bé. caustic soda	0.05 (.124 lbs.)	2.77	3.32	3.05	...	.01	...
Water	0.86	0.025	0.03	0.0275	...	...	...
300 pale oil	28.70	2.90	4.35	3.40	.83	1.25	.98
Total material cost	39.61 Gals.				$ 5.66	7.05	6.29
Total material cost	1 Gal.	(Expressed in cents)			14.29	17.80	15.88
Grease comp. cost	1 Gal.	(Expressed in cents)			1.22	2.14	1.53
Cost 1 gallon		(Expressed in cents)			15.51	19.94	17.41
Cost 1 lb. cup grease No. 5 (7.90 lbs. per Gal.)		(Expressed in cents)			1.96	2.52	2.20
		Cup Grease No. 6					
Tallow	15.0 (113.8 lbs.)	5.0	6.0	5.5	$ 5.69	6.83	6.26
Lard oil	5.0 (38.0 lbs.)	10.0	12.0	11.0	3.80	4.56	4.18
Hydrated lime	(21.0 lbs.)	0.79	0.95	0.87	.17	.20	.18
30° Bé. caustic soda	0.10 (.249 lbs.)	2.77	3.32	3.05	.01	.01	.01
Water	1.46	0.025	0.30	0.0275	...	...	...
300 pale oil	45.10	2.90	4.35	3.40	1.31	1.96	1.53
Graphite	....	...	...	...	...	...	...
Total material cost	66.66 Gals.				$10.98	13.56	12.16
Total material cost	1 Gal.	(Expressed in cents)			16.47	20.34	18.24
Grease comp. cost	1 Gal.	(Expressed in cents)			1.22	2.14	1.53
Cost 1 gallon		(Expressed in cents)			17.69	22.48	19.77
Cost 1 lb. cup grease No. 6 (7.97 lbs. per Gal.)		(Expressed in cents)			2.22	2.82	2.48

No. 7 S.C. Cup Grease

A very dense grade of cup grease has been successfully developed and marketed by a Southern California grease plant, for use in compression cups and for packing mill bearings. Its analysis is:

Physical Tests

Color	Light yellow
Odor	Oil of myrbane
Penetration (unworked) at 77° F.	78
Dropping point, ° C.	102

Composition, per cent by weight

Soap (as calcium stearate)	34.3
Water	5.8
Free alkali (as $Ca(CH)_2$)	0.1
Petroleum oil	56.6
Free fat	None
Glycerin	3.2
Total	100.0

Ash, per cent by weight	3.77
Ash analysis, per cent by weight	
Insol. in HCl	Trace
Calcium oxide	99.0
Undetermined	1.0
Total	100.0
Fatty acids from soap	
Acid value, mgs. KCH/gm.	205
Iodine value (Hanus)	57
Melting point ° C.	41
Color	Light brown
Extracted Petroleum Oil	
Color, N.P.A.	4.5
Gravity, A.P.I.	21.4
S.U.V./100° F.	242

Quick Control Test With Abraham Consistometer

Merrill,[60] in 1925, before the A.S.T.M. penetration test was fully standardized, developed a quick test for the control of consistency in cup grease manufacture, making use of the Abraham consistometer. With this instrument a plunger with a flat circular head and shank of reduced cross section is forced into a sample of a plastic solid at a uniform velocity. The force required to cause penetration is applied by means of a hand wheel and screw and is transmitted to the screw through a spiral spring. The elongation of this spring is indicated by a pointer on a scale and gives the force directly in metric units. The rate of penetration is indicated by a hand which makes one revolution for one centimeter of travel and hence causes the plunger to penetrate at the standard rate of one centimeter per minute. The force is applied at such a rate that the hand turns at the same speed as the second hand of a watch. Plungers having 0.01, 0.1, 1 and 10 square centimeters and springs of different size are used. The results are reported as the cube roots of the pressures and are known as the Abraham hardness numbers or Abraham consistencies.

The advantages of the Abraham instruments are:

The consistency of very small samples may be determined.

A very wide range of consistencies may be determined, from semifluid transmission greases to dense railroad greases.

The results are expressed on a single scale of numbers of convenient size, with large numbers representing the harder greases.

It is possible to allow for hard spots and air pockets in the sample of grease being tested.

The disadvantages of the instrument are:

The apparatus is expensive.

Great skill is required by the operator to obtain satisfactory results.

Samples in large containers cannot be tested.

[60] *Ind. Eng. Chem.*, (10), 17, 1068.

Merrill gives the following consistencies determined by the Abraham method for a series of cup greases made from calcium tallow soap base and Western mineral oil:

Grade	Approximate normal consistency (unworked at 77° F.)	Approximate soap content
0	2.0	10.5
1	3.3	11.5
2	4.5	14.5
3	6.5	17.5
4	8.5	20.0
5	11.0	22.5
6	16.5	29.0

U. S. Government Cup Greases

(Federal Standard Stock Catalog, Section IV, Part 5, Grease; Lubricating, Mineral. VV-G-681, Oct. 2, 1934).

According to the above Federal Specification, mineral lubricating greases shall be of three grades, designated as soft, medium, and hard. Cup greases made on the formulae for grades two, three and four in the preceding section will comply with the Federal Specifications for soft, medium and hard greases, respectively. In the case of the No. 2 grease, however, the grease should be made with slightly more oil than indicated in the formula in order to comply with the minimum oil content prescribed at 85 per cent by weight. The pressure saponification procedure should be followed, and if analysis indicates the presence of more than 0.10 per cent of uncombined CaO, then suitable adjustments with oleic acid should be made to just comply with this requirement. It is considered good practice to have the grease checked for all of the following government requirements before drawing it from the kettle. While this may mean reserving a kettle for government grease for a longer period than necessary for ordinary batches, it is probably the safest course to follow even if more costly from the standpoint of time and labor.

D-1 Mineral lubricating grease shall be a smooth, homogeneous mixture of mineral oil and pure odorless lime soap made from animal or vegetable fats or oils or fatty acids, completely saponified with hydrated lime, and shall be suitable for the lubrication of such parts of motor equipment and other machinery as are lubricated by means of compression cups, and for use in ball and roller bearings.

D-2. Mineral lubricating grease shall contain no fillers such as rosin, rosin oils, talc, wax, powdered mica, sulfur, clay, asbestos, or other undesirable or deleterious impurities.

D-3. Mineral lubricating grease shall possess only a slight odor of mineral oil, and may be rejected if it has any other distinct odor.

E. Detail Requirements

E-1. See section F.

F. Methods of Sampling, Inspection, and Tests

F-1. Any requirements of the individual departments are noted under section H.

F-2. *Sampling.*—Unless otherwise specified, samples shall be taken according to the procedure described in section F of Federal Specification VV-L-791 of the issue in effect on date of invitation for bids.

F-3. *Inspection and tests.*—Unless otherwise specified herein, all tests shall be made according to the methods for testing contained in section F of Federal Specification VV-L-791 of the issue in effect on date of invitation for bids.

F-4. *Specification limits.*—Mineral lubricating grease shall comply with the following specifications:

Test	Soft	Medium	Hard
Grease:			
Penetration	300–355	240–290	190–230
Mineral oil content, per cent (minimum)	85	80	75
Water content, per cent (maximum)	1.5	2.0	2.0
Ash as sulfates, per cent (maximum)	5.0	6.0	8.5
Uncombined CaO, per cent (maximum)	0.10	0.10	0.10
Insoluble matter, per cent (maximum)	0.10	0.10	0.10
Corrosion	nil	nil	nil
Mineral-oil constituent:			
Viscosity at 130° F. Saybolt universal seconds, (minimum)	125	125	125
Flash point, ° F. (minimum)	340	340	340
Fire point, ° F. (minimum)	370	370	370

Methods of test

Test	Federal Specifications Board	American Society for Testing Materials
Penetration	31.11	D217–33T
Mineral oil content	541.2	D128–27
Water content	300.13	D 95–30
Ash as sulfates	541.2	D128–27
Uncombined CaO	541.2	D128–27
Insoluble matter	541.2	D128–27
Corrosion	530.4	.....
Separation of mineral oil constituent	541.2	D128–27
Viscosity	30.43	D 88–33
Flash point	110.33	D 92–33
Fire point	110.33	D 92–33

G. Packaging, Packing, and Marking

G-1. Any requirements applicable to the individual departments are noted under section H.

G-2. *Packaging.*—Delivery as specified by the purchaser.

G-3. *Packing.*—Unless otherwise specified, the subject commodity shall be delivered in standard commercial containers, so constructed as to insure acceptance by common or other carriers for safe transportation, at the lowest rate, to the point of delivery.

G-4. *Marking.*—Unless otherwise specified, shipping containers shall be marked with the name of the material, type, and the quantity contained therein, as defined by the contract or order under which the shipment is made, the name of the contractor, and the number of the contract or order.

H. Requirements Applicable to Individual Departments

H-1. The following departmental specifications of the issue in effect on date of invitation for bids shall, in so far as applicable, form a part of this specification, and bidders and contractors should provide themselves with the necessary copies when submitting bids to the respective departments:

H-1a. Navy: General Specifications for Inspection of Material, copies of which may be obtained without cost upon application to the Bureau of Supplies and Accounts, Navy Department, Washington, D. C.

H-1b. Army: United States Army Specification for Inspection of Material.

I. Notes

I-1. This specification covers the grades of mineral lubricating cup grease intended for use in the lubrication of such parts of motor equipment and other machinery as are lubricated by means of compression cups, and for use in ball and roller bearings.

I-2. The *soft grade* of mineral lubricating grease is intended for use in ball and roller bearings and in compression cups for bearings of machinery operating under light loads and at high speeds.

I-3. The *medium grade* of mineral lubricating grease is intended for use in compression cups for bearings of machinery operating under medium loads and at medium speeds.

I-4. The *hard grade* of mineral lubricating grease is intended for use in compression cups for bearings of machinery operating under heavy loads and at low speeds, or for high speeds in hot climates.

I-5. For graphite lubricating grease, see Federal Specification VV-G-671.

I-6. An alphabetical index of Federal specifications may be obtained upon application, accompanied by money order or coupon, or cash, to the Superintendent of Documents, Government Printing Office, Washington, D. C., price to be obtained from the Superintendent of Documents.

I-7. Copies of Federal Specification VV-L-791 (referred to in par. A-1) may be obtained from the Superintendent of Documents, Government Printing Office, Washington, D. C.

There is some doubt that the 0.10 per cent uncombined CaO specification prescribed by the government is really significant of high quality. It is quite likely that greases which, on analysis by the A.S.T.M. method, are found to contain appreciable free alkali may also contain at the same time free oleic acid, so that the result of the analysis is an indication of the excess CaO in the grease over that which reacts with any free fatty acids present in the presence of the petroleum ether and fifty per cent alcohol solution required in the A.S.T.M. analysis. Experience has shown, that in order to meet the 0.10 per cent CaO maximum requirement, that for a 6000-pound batch of the medium grade grease, ten gallons of oleic acid may be required. This acid, at 48.66c per gallon, increases the cost of the grease by 0.081c per pound. To this extra cost should be added the additional expense of 0.02c per pound for making several free alkali determinations, and 0.30c per pound for the time the compounding kettle is held inactive pending the results of laboratory analyses, it being assumed that the kettle would be in use for other greases if not reserved for this purpose. It thus appears that the extra cost of meeting the government requirements may amount to as much as 0.40c per pound of grease, above the normal cost.

Tentative (American Society of Lubrication Engineers) Standards for Cup Greases

(Published by F. C. Otto, "Correct Lubrication" Vol. III, No. 114, Mar. 11, 1936). These are tentative standards, published for the purpose of eliciting criticism and suggestions, and as such are subject to annual revision.

Lubricating Greases covered by these standards shall be smooth and homogeneous mixtures of mineral oils and pure odorless lime soap made from animal or vegetable fats or oils and/or fatty acids completely saponified with hydrated lime.

These greases shall not contain filler such as clay, talc, whiting, mica, asbestos, graphite, zinc oxide, wood fiber, rosin, rosin oil, wax, sulfur or other undesirable or deleterious impurities.

A.S.L.E. Standard Number:	Penetration A.S.T.M. worked min.	Penetration A.S.T.M. worked max.	Basic Soap Calcium % min.		Basic Soap Calcium % max.	Mineral Oil % min.	Viscosity S.U. of Oil at min.	Viscosity S.U. of Oil at max.	Viscosity S.U. of Oil at F.	Water % max.	Melting Point ° F. Mercury
11	...	...	5		6	93	70	110	100	0.80	165
12	...	370	11		13	85	70	110	100	1.50	190
13	...	310	15		16	81	70	110	100	1.80	205
14	...	275	16		18	79	70	110	100	2.00	205
15	...	250	18		20	77	70	110	100	2.30	205
16	...	230	20		22	75	70	110	100	2.50	205
17	...	210	22		24	72	70	110	100	2.50	205
18	250	310	12	h	14	..	75	...	100	1.50	...
19	180	240	23	h	27	..	200	...	100	2.50	...
20	165	225	20	h	25	..	...	...	...	...	210
31	...	...	1½		2	97	300	...	100	0.15	...
32	...	...	3		4	95	300	...	100	0.30	...
33	...	360	10		12	86	300	...	100	1.50	200
34	...	350	12		14	83	300	...	100	1.50	200
35	...	310	15		16	81	300	...	100	1.80	205
36	...	285	16		17	80	300	...	100	2.00	205
37	...	250	18		20	77	300	...	100	2.30	205
38	...	230	22		24	72	300	...	100	2.50	210
39	i	...	.75	h	1	..	300	325	100	...	...
40	300	350	5	h	8	..	275	325	100	1.00	...
41	300	350	8	h	11	..	300	500	100	1.00	...
42	270	320	9	h	12	..	475	525	100	1.00	...
43	300	355	..		..	85 p	125	...	130	1.50	...
44	240	290	..		..	80 p	125	...	130	2.00	...
45	190	230	.		..	75 p	125	...	130	2.00	...
61	...	...	5		6	93	500	...	100	0.80	160
71	350	400	4	h	6	.. r	140	...	210	1.00	...

Notes: i)—Viscosity of grease 645-765 seconds S.U. at 100° F. and 225-255 seconds S.U. at 130° F.
h)—Lime shall not contain more than 7 per cent of magnesium oxide and not more than 2 per cent of gritty non-soap forming substances. A small amount of caustic soda is permissible, if desired, to help complete saponification, provided the sodium soap does not exceed 7 per cent of total soap.
p)—Mineral oil must have a minimum flash point of 340° F. and minimum fire point of 370° F.
r)—Maximum viscosity permissible 4000 seconds at 100° F.

S. P. Railway Cup Greases

Appropriate grades of the foregoing Standardized Cup Greases comply with the corresponding S. P. Railway purchase specifications as shown in the following tabulation:

Standardized Cup Grease No.	S. P. Railway Specification Number	
3	101-D	No. 3
4	101-D	No. 4
5	101-D	No. 5

Shipments meeting the following S.P. specifications, revised as of February 15th, 1926, should be checked individually for soap content and ash, as in some cases batches of the standard greases will not comply.

Material and Uses:

Three greases are covered:
No. 3 grease
No. 4 grease
No. 5 grease

To be used for lubrication of such parts of motor equipment and other machinery as are lubricated by means of compression cups. (May be used also on light gears.)

No. 3 grease shall be furnished in all cases unless No. 4 or No. 5 grease is specifically ordered. No. 4 grease is shown in specification to take care of the few cases where a hard grease is desired. No. 5 grease is for use with tie tampers.

General Requirements:

To be compounded of calcium soap made from a high grade animal or vegetable oil or fatty acid, and a highly refined mineral oil.

The mineral oil used in reducing the soap shall be a straight well refined mineral oil with a viscosity at 100° F. of not less than 100 Saybolt Universal.

Shall be a boiled grease containing not less than 1 per cent or more than 3 per cent of water when finished.

A clean copper plate shall not be discolored when submerged in the grease for 24 hours at room temperature.

Shall contain no fillers, such as rosin, resinous oils, soapstone, wax, talc, powdered mica, sulfur, clay or asbestos.

Requirements:

No. 3 Grease

Shall contain approximately 17 per cent to 20 per cent of calcium soap. The ash shall not be greater than 2.25 per cent.

No. 4 Grease

Shall contain approximately 24 per cent to 27 per cent of calcium soap. The ash shall not be greater than 3 per cent.

No. 5 Grease

Shall contain approximately 29 per cent to 34 per cent of calcium soap. The ash shall not be greater than 3.5 per cent.

Remarks:

All specifications for cup grease previously issued are hereby cancelled.

Dyed Export Cup Greases

Custom has dictated that many cup greases imported by Japan and China should have a brilliant yellow or red tint. This has no doubt come about by the fact that these countries pay very low prices for their greases, and some compounders rather than considering it necessary to develop a low-cost, special product for this trade have found it most convenient to dye one of their regular lines of greases and offer it at a competitive price.

By modifying the Standardized cup grease formulae previously discussed acceptable greases for trade in the Orient have been manufactured:

Constituents	Gallons	Pounds Net	Per Cent by Weight
Yellow Cup Grease No. 1 (Export Japan)			
Cup grease No. 2 (Code 202)		4969.23	99.385
300 pale oil	4	30.47	.609
Oil orange		.30	.006
		5000.00	100.000
Yellow Cup Grease No. 2 (Export Japan)			
Cup grease No. 2 (Code 1012)		4969.23	99.385
300 pale oil	4	30.47	.609
Oil orange		.30	.006
		5000.00	100.000

Constituents	Gallons	Pounds Net	Per Cent by Weight
Yellow Cup Grease No. 3 (Export Japan)			
Cup grease No. 3 (Code 203)		4969.23	99.385
300 pale oil	4	30.47	.609
Oil orange		.30	.006
		5000.00	100.000
Yellow Cup Grease No. 4 (Export Japan)			
Cup grease No. 4 (Code 204)		4969.23	99.385
300 pale oil	4	30.47	.609
Oil orange		.30	.006
		5000.00	100.000
Yellow Cup Grease No. 5 (Export Japan)			
Cup grease No. 6 (Code 206)		4969.23	99.385
300 pale oil	4	30.47	.609
Oil orange		.30	.006
		5000.00	100.000

Specifications of the yellow cup greases intended for export to Japan and manufactured in accordance with the foregoing formulae, are listed below:

Export Cup Grease Grade Number	1	2	3	4	5
Worked penetration at 77° F. (A.S.T.M. D217-33T)	315–355	275–315	240–275	190–230	50–90 (Unworked)
Water by distillation, per cent by weight (A.S.T.M. D95-30), max.	2.0	3.0	3.0	3.0	3.5

Color, Yellow. (Must comply with color of standard samples.)

The range in quantities of dye used in No. 3 Cup Greases are given below, in most cases 0.0068 per cent by weight is the maximum quantity needed to give a strong coloration:

Dye	Dye Manufacturer	Per cent by weight in No. 3 Standardized Cup Grease
Graysol Yellow 2-G	Geigy Co., Inc., N. Y.	.0017
Graysol Yellow 2-G	Geigy Co., Inc., N. Y.	.0034
Graysol Yellow 2-G	Geigy Co., Inc., N. Y.	.0068
Oil Yellow No. 1	Amer. Aniline Products Co., N. Y.	.0017–.0034–.0068
Oil Yellow No. 2	Amer. Aniline Products Co., N. Y.	.0017–.0034–.0068
Methyl Orange	Braun-Knecht-Heiman, S. F.	.0018
Butter Yellow OB	Cassella, L., Germany	.0017–.0034–.0068

Cup Greases for Australia and New Zealand

Both dyed greases and those having a natural color are sold in Australasia; the consistencies, however, must be carefully controlled to meet local conditions. These requirements are met by grease made in accordance with the formulae listed below:

Cup Grease No. 1 (Australia and New Zealand)

Same formula as Standardized Cup Grease No. 2

Cup Grease No. 2 (Australia and New Zealand)

	Gallons	Pounds Net	Per Cent by Weight
Tallow	90	686.50	10.91
Lard oil	30	228.80	3.64
30° Bé. caustic soda	½	1.24	.02
Hydrated lime		125.00	1.99
Water	676	100.64	1.60
300 pale oil		5147.82	81.84
		6290.00	100.00

Cup Grease No. 3 (Australia and New Zealand)

	Gallons	Pounds Net	Per Cent by Weight
Tallow	90	686.50	13.76
Lard oil	30	228.80	4.59
30° Bé. caustic soda	½	1.24	.02
Hydrated lime		125.00	2.50
Water	507	84.83	1.70
Cut 300		3863.63	77.43
		4990.00	100.00

Cup Grease No. 4 (Australia and New Zealand)

Same formula as Standardized Cup Grease No. 4

Yellow Cup Grease No. 1 (Australia and New Zealand)

Same formula as Yellow Export Cup Grease No. 1

Yellow Cup Grease No. 2 (Australia and New Zealand)

	Gallons	Pounds Net	Per Cent by Weight
Standardized cup grease No. 2		4969.23	99.385
300 pale oil	4	30.47	.609
Oil orange		.30	.006
		5000.00	100.000

Yellow Cup Grease No. 3 (Australia and New Zealand)

	Gallons	Pounds Net	Per Cent by Weight
Standardized cup grease No. 3		4969.23	99.385
300 pale oil	4	30.47	.609
Oil orange		.30	.006
		5000.00	100.000

Yellow Cup Grease No. 4 (Australia and New Zealand)

Same formula as Yellow Cup Grease No. 4

Yellow Cup Grease No. 5 (Australia and New Zealand)

Same formula as Yellow Export Cup Grease No. 5

Cup greases made on these formulae will comply with the following specifications:

Australian Cup Grease No.	A.S.T.M. Worked Penetration Range	Water Per Cent by Weight (Maximum)
1	295–335	2.0
2	255–295	3.0
3	220–255	3.0
4	170–210	3.0

Australian Yellow Cup Grease Number	A.S.T.M. Worked Penetration Range	Water Per Cent by Weight (Maximum)
1	295–335	2.0
2	255–295	3.0
3	220–255	3.0
4	170–210	3.0
5	45– 70 (Unworked)	3.5

Australian Heavy Duty Cup Grease

For use in certain locomotive driving journal boxes, and for packing the bearings of heavy jaw type rock crushers a non-separating grease is required in Australia and may be made by following the formula given here:

Constituents	Gallons	Pounds Net	Per Cent by Weight
Oleic acid	128	958.00	21.29
Stearic acid	32	240.00	5.33
Hydrated lime	...	170.00	3.78
30° Bé. caustic soda	½	1.24	.03
Water		45.00	1.00
300 pale oil	400	3085.76	68.57
		4500.00	100.00

This grease which costs 2.24c per pound to manufacture has the following characteristics:

A.S.T.M. unworked penetration at 77° F.	78–111
Ubbelohde dropping point	95° C. (203° F.)
Water, per cent by weight	1.0–2.0
Free $Ca(OH)_2$, per cent by weight	.22–.28

It is known that small amounts of free fatty acids, or wool grease fatty acids and their lime soaps, greatly effect improved heat stability in cup greases when heated to above their melting points. It appears also, that with increased soap contents the tendency to separate free oil is reduced. The following heat tests on greases discussed in this section have been made in conjunction with the development of heavy duty cup greases for the Australian trade:

Heat Tests

Test No.	Name of Grease	Character of grease after heating to 250° F. for 5 minutes and cooling while stirring
1	Standardized Cup Grease No. 5 (Percent free alkali 0.68) (Percent free fat 0.05)	Was a firm cake of grease when cooled; on stirring a small amount of oil separated.
2	Item 1 plus ½ per cent lard oil	About same result as for test 1.
3	Standardized Cup Grease No. 5 (Check on above test 1)	Small amount of separated oil. Soap cake granular.
4	Standardized Cup Grease No. 4	Separation of large proportion of oil content.
5	Item 4 plus ½ per cent lard oil	Excessive separation, soap cake softer.
6	Standardized Cup Grease No. 1	Large amount of separated oil; soap residue hard and granular.
7	350 Penetration Fatty Acid Cup Grease	Some separation, pasty.
8	Australian Heavy Duty Cup Grease	Pasty, little oil separation.

English Cup Greases

A reputable English grease manufacturer has for many years, marketed cup greases having the following characteristics:

Grease	1	2	2a	3	4	White Cup
Color	Yellow	Yellow	Brown	Yellow	Green Brown	White
Texture	Buttery	Med. Cup	Hard Cup	Hard Cup	Med. Cup	Hard Cup
Mineral oil, per cent by weight	78.4	76.25	74.0	74.0	79.8	
Nature of mineral oil	Mexican Spindle	Mexican Spindle	Mexican Spindle	Mexican Spindle	500 Venezuela	Water White Transformer
Fatty matter, per cent by weight	16	18	20	20	17	20
Nature of fatty matter	Tallow Fatty Acids	Same	Same	Same	Cotton and Wool Stearin	White Tallow Fatty Acids
Per cent by weight, water, max.	2	2	2	2	1	1
Percent $Ca(OH)_2$	3.6	3.75	4.0	4.0	2.2	7.0
A.S.T.M. penetration at 77° F.						
worked	350	315	248	239	222	165
unworked	314	218	180	175	195	114
Ubbelohde dropping point °F.	200	210	220	220	180	325

All of the above greases have high excesses of free lime, with the exception of No. 4, which is intended for roller and ball bearing lubrication. The effect of the excess lime on the melting points of the greases is quite apparent.

English vs. German Cup Greases

In 1935, a German investigator reported the results of his investigations of the stability of a large number of English cup greases made by the autoclave process, with those of similar greases made in open stills at atmospheric temperature in Germany. He concluded that from the standpoint of stability alone pressure saponified greases were not superior to those produced in open vessels. It was further evident that stability towards high temperatures was more a matter of the grease constituents and the formula than the process used.

Analytical methods employed were:

Neutralization Number, Method G. This test was made by first dissolving 5 grams of grease in 60c.c. of benzol-alcohol (2:1) and heating. After complete solution and further dilution with 60c.c. of the benzol-alcohol solution the sample is titrated with standardized KOH solution against alkaline blue as an indicator.

Neutralization Number, Method A. According to this method a 10-gram sample of the grease is dissolved in 100c.c. of benzol, and filtered. The filtrate obtained is diluted with 100c.c. of 70 per cent ethyl alcohol and titrated with standarized KOH solution against alkaline blue as an indicator.

Determination of Free Lime. A 10-gram sample of the grease is thoroughly extracted in Soxhlet apparatus with benzol. The residue of free lime left in the extraction thimble is titrated separately with standardizd HCl solution and calculated to CaO.

Heating Test "G." This test is a modification of an older method worked out by Timken engineers. About 25 grams of grease are placed in a Cleveland open flashpoint cup and heated on a hotplate, while stirring with a thermometer, until a temperature of 200° C. (392° F.) is attained. The melted lubricant is then poured into a large clock glass and allowed to cool without stirring. Satisfactory heat stable greases should show no indications of oil separation after twenty four hours. The grease should remain homogeneous and plastic.

Heat Test IA. Twenty-five grams of the grease is heated for two hours at 110° C. (230° F.) in a small glass beaker. The grease is then allowed to stand for twenty-four hours and observed for oil separation.

Heat Test IIA. The greases are heated in a small glass beaker for ten minutes at 140° C. (284° F.) while stirring with a thermometer and then poured into another glass beaker which is at room temperature.

The results of the tests on commercial and experimental English made greases as compared with German commercial lubricants are shown in the tables, pages 194, 195, 196.

The following observations are of interest with regard to the foregoing data tables. The German Grease No. 1, according to neutralization number test "G," is highly alkaline whereas the English greases 2, 3, 4, 5, and 6, made in the autoclave, vary from slightly acid to moderately alkaline. By method "A," the German grease is only slightly alkaline and all the English greases (2 to 6) are slightly acid. The free-lime content of the German Grease No. 1 is considerably higher than the corresponding English No. 1 greases made by pressure methods. By heating test "G," all of the No. 1 greases are unsatisfactory, this also being the case for normal American Cup Greases. English greases 1, 2, 3, and 4 contain a small amount of ZnO, whereas greases 5 and 6 are made with no coloring material added. The non-colored greases appear to be somewhat more stable than those containing the ZnO, this material probably being responsible for the formation of hard cores and causing a pronounced non-homogeneous condition in the cooled samples.

By method "A," all of the No. 2 greases (7, 8, 9, 10, 11, and 12) have an acid reaction; greases 11 and 12 containing no ZnO are somewhat superior to the others in heating test "G," but all fail to pass this test. For greases 13 to 18 inclusive, which are of No. 3 grade, the German Grease No. 3 is highly alkaline, whereas with the exception of No. 16, they are acid as tested by method "G." This difference is due to formulae and pressure saponification. As to the free-lime content and the heating tests, the results on the No. 3 greases are quite similar to those on the No. 1 and No. 2 greases.

The dyed greases are colored with oil soluble yellow aniline dyes. With regard to heat stability they appear to be much in line with the foregoing products.

Grade	1 German No. 1	2 English No. 1	3 English No. 1	4 English No. 1	5 English No. 1	6 Experimental English No. 1	7 German No. 2	8 English No. 2	9 English No. 2	10 English No. 2
Flow point — dropping point, ° C.	99/103	95/99	95/99	94/97	92/96	95/99	99/103	99/103	96/100	100/104
Fat content, per cent	14.5	13.0	14.7	14.6	14.0	13.8	14.5	18.3	18.6	18.0
Ash, percent CaO	2.5	2.1	3.0	3.0	1.4	1.4	2.5	4.0	2.8	4.0
Neutralization value, method "G"	2.3 alk.	0.9 alk.	0.12 acid	0.15 acid	0	0	1.6 alk.	1.3 alk.	1.1 alk.	1.8 alk.
Neutralization value, method "A"	0.06 alk.	0.14 acid	0.34 acid	0.45 acid	0.34 acid	0.39 acid	0.08 acid	0.17 acid	0.11 acid	0.06 acid
Free lime, percent CaO	0.57	0.06	0.01	0.006	0.	0.02	0.59	0.06	0.06	0.1
Heating test "G"										
Separation of oil	much	much	much	much	little	little	much	much	much	much
Core	hard	hard	hard	hard	not very hard	not very hard	hard	hard	hard	hard
Total result	unsatisfactory	very unsatisfactory	very unsatisfactory	very unsatisfactory	unsatisfactory, superior to col. grease	unsatisfactory, superior to col. grease	unsatisfactory	unsatisfactory	unsatisfactory	unsatisfactory
Heating test IA										
Separation of oil	much, 7 c.c.m.	much, 8 c.c.m.	none	none	none	much, 6 c.c.m.	little	no	no	no
Formation of crust	yes	thin	no	no	no	no	yes	yes	yes	yes
Core	a little hard, homogeneous	homogeneous, plastic	soft homogeneous	soft homogeneous	soft homogeneous	soft homogeneous	plastic, homogeneous	plastic, homogeneous	plastic, homogeneous	plastic, homogeneous
Heating test IIA										
Separation of oil	much	much	very little	very little	none	none	much	much	little	rather much
Formation of crust	no	no	yes	no	no	no	no	no	no	no
Core	hard, slightly brittle	hard, brittle	soft, not homogeneous	hard, brittle	soft, homogeneous	soft, homogeneous	hard, brittle	hard, brittle	hard	hard, brittle

Grade	11 English No. 2	12 Experimental No. 2	13 German No. 3	14 English No. 3	15 English No. 3	16 English Export No. 3	17 Experimental No. 3	18 Experimental No. 3	19 German dyed grease	20 Experimental dyed grease
Flow point — dropping point, ° C.	95/99	95/99	99/103	98/102	98/102	98/102	95/99	97/101	100/105	98/102
Fat content, per cent	18.4	17.5	14.5	22.5	22.0	19.8	22.0	18.8	19.5	25.4
Ash, percent CaO	2.0	2.0	2.5	3.8	3.7	3.3	2.5	2.5	2.9	2.9
Neutralization values method "G"	0.3 acid	neutral	2.0 alk.	0.2 acid	0.2 acid	1.1 alk.	0.6 acid	0.15 acid	2.2 alk.	0.3 acid
Neutralization values method "A"	0.34 acid	0.28 acid	0	0.34 acid	0.5 acid	0.17 acid	0.34 acid	0.5 acid	0.3 acid	0.25 acid
Free lime, percent CaO	0.01	0.01	0.64	0.0	0.02	0.06	0.01	0.01	0.75	0.06
Heating test "G"										
Separation of oil	little	little	much	much	much	much	little	little	much	little
Core	not very hard	not very hard	hard	hard	hard	hard	less hard than col. grease	less hard than col. grease	hard	hard
Total result	unsatisfactory, superior to col. grease	unsatisfactory, superior to col. grease	unsatisfactory	unsatisfactory	unsatisfactory	unsatisfactory	just satisfactory	rather unsatisfactory	unsatisfactory	unsatisfactory
Heating test IA										
Separation of oil	no	no	much, 7 c.c.	none	none	none	none	none	little	none
Formation of crust	yes	yes	yes	thin	thin	thin	thin	thin	yes	no
Core	plastic, homogeneous	plastic, homogeneous	rather hard, homo.	hard, homogeneous	hard, homogeneous	hard, homogeneous	rather hard, homo.	rather hard, homo.	soft, homogeneous	more solid, homo.
Heating test IIA										
Separation of oil	none	none	very much	rather much	rather much	rather much	none	none	little	none
Formation of crust	no froth	no froth	no	no	no	no	froth	froth	no	no
Core	rather hard, not homo.	rather hard, not homo.	hard, brittle	hard, brittle	hard, brittle	hard, brittle	soft, homogeneous	soft, homogeneous	hard, brittle	solid, homogeneous

Grade	21 English dyed grease 3	22 German dyed grease 1	23 English dyed grease I	24 English dyed grease 1	25 English dyed grease 1	26 German roller bearing	27 English R B.	28 German R. B	29 English R. B.	30 Experimental R. B.
Flow point — dropping point, ° C.	96/100	99/103	98/102	95/99	99/103	98/102	98/102	98/102	99/103	91/95
Fat content, per cent	25.6	13.5	11.7	12.3	10.6	18.	19.8	18.	19.9	15.3
Ash, percent CaO	2.9	1.5	1.3	1.6	1.3	1.7	2.1	1.7	0	0
Neutralization value method "G"	0.3 acid	1.9 acid	0	0.4 alk.	0.7 alk.	2.1 acid	1.7 acid	2.1 acid	1.9 acid	0
Neutralization value method "A"	0.2 acid	1.1 acid	0.06 acid	0	0.06 alk.	2.5 acid	1.8 acid	2.5 acid	1.5 acid	0
Free lime, percent CaO	0	0	0.02	0.02	0.04	0	0	0	0	0
Heating test "G"										
Separation of oil	much	little	much	little	much	no	no	no	no	very much
Core	hard	nearly soft	not very hard	soft	hard	soft		soft		not very hard
Total result	unsatisfactory	satisfactory	unsatisfactory	satisfactory	unsatisfactory	very satisfactory	satisfactory	very satisfactory	satisfactory	unsatisfactory
Heating test IA										
Separation of oil	none	none	very much 10 c.c.m.	little 1.5 c.c.m.	very much 11 c.c.m.	none	none	none	none	very much 11 c.c.m.
Formation of crust	no	no	yes	no	no	no	no	no	froth	no
Core	rather solid, homogeneous	soft at top, brittle at bottom	solid, brittle	soft, homogeneous	hard, brittle	soft, homogeneous	soft, homogeneous	soft, homogeneous	soft, homogeneous	soft, homogeneous
Heating test IIA										
Separation of oil	none	none	much	none	very much	none	none	none	none	none
Formation of crust	no	no	no	no	no	no	no	no	no	thin
Core	more soft, homo.	very soft, homo.	solid, brittle	soft, homogeneous	solid, brittle	soft, homogeneous	soft, homogeneous	soft, homogeneous	soft, homogeneous	soft, homogeneous

THE USE OF CUP GREASE

Correct lubrication is an operating problem of great industrial importance, and it is one that gets less thought than its importance warrants. From a technical standpoint, it is receiving more and more attention. Considerable effort is now being made by the large users of lubricants as well as the manufacturers to determine the exact actions and reactions that take place in the lubricant itself under the effects of heat, speed and pressure. Operating engineers and industrial executives interested in reducing plant overhead are coming to realize the importance of the problem, as speeds, temperatures and weights of bearings change in accordance with advances in design and metallurgy.

Automotive machinery, marine machinery units, steel rolling mills, paper mill machinery, cranes and hoists, excavating machinery, wood working machinery, road machinery, textile machinery, ball and roller bearings and thrusts wherever they may be found, are examples in which the application of grease lubrication has been of great value. There are many industrial bearings that cannot employ fluids, yet have great necessity for correct and ample lubrication.

Grease Compared with Oil

The matter of comparing grease with oil for lubrication purposes of necessity involves the consideration of the mechanical details of the bearing and journal, and the relative adaptability of each lubricant for the service intended. The question of where and when to use grease lubrication requires a thorough knowledge of machinery operation and the technology of lubrication. There are certain definite characteristics of grease lubricants which give them decided advantages over oil in many cases. It is generally assumed that grease lubrication is best suited to low speed service. By virtue of the fact that a grease film is normally thicker than an oil film due to its heavier "body," the rate of distortion in the film should be less. It is not known that this condition would hold under high shaft speeds, since the questions of bearing pressure, bearing area, relative bearing dimensions, the nature of the load on the bearing, type of bearing metal, nature of the feed (constant or intermittent), operating conditions and means of applying the lubricant would by their effects render each case individual in its operation.

Another advantage grease lubricant presents is the usual freedom from splashed or dripped used lubricant. In certain types of machinery leakage of the lubricant to the product being handled will result in its being ruined. Examples of this may be found in the paper, textile and baking industries. Means of lubrication on much of the machinery used in these industries renders cleanliness of the bearings and other wearing parts practically impossible were oil to be used. For this reason grease is resorted to, being applied by compression cups, gravity type funnel cups or pin cups, and pressure lubricators.

"Grease is grease" and nothing more to most people, so far as its suitability to meet specific conditions of lubrication is concerned; but it should take its place, in technical importance, alongside the oil of which it is substantially the substitute. It is believed that it is the oil content of cup greases that performs the major portion of the lubrication and that the grease should be made from an oil suited to the conditions of temperature, speed and pressure.

It is generally believed that the use of grease on bearings operating at high speed is accompanied by certain disadvantages. Greases are chiefly of advantage in lubricating slower speed high-pressure units where a relatively thick film of lubricant is desirable. There has been extensive effort in a few technical articles and advertising bulletins to promote the use of grease by extravagant statements as to its qualifications. Such statements would in many cases lead the ordinary user to believe that grease is a cure-all wherever lubricant is required. The use of grease while it has a broad and varied field is however clearly limited to the lubrication only of such points as are favorable to its application.

One of the disadvantages of grease which is sometimes cited is its lack of stability. Grease, if not of the highest quality, will on being stored for long periods of time show a tendency towards lumpy separation of the oil and soap constituents. Another disadvantage is that cup greases are not uniform in character. The grades of different manufacturers often differ markedly in regard to consistency, melting point, et cetera. Some producers have numbering systems of their own for grading greases which often confuse the trade.

Cases where Grease is Preferred

In the practice of lubrication engineering certain manufacturing and operating conditions will frequently be encountered which are not adaptable to oil lubrication and it is often found desirable and in some cases imperative to resort to lubricating greases. The most important of these conditions may be listed as follows:

Those cases where there is liability of the oil dripping or splashing where it would do damage to materials being handled by the machine. The leakage of oil to break bands, clutches and leather belts may often be overcome by the use of a suitable grade of grease.

Where oil cannot be retained in the journal due to high pressures on the bearing surfaces, slow rubbing speeds and high bearing temperatures which cause the oil to be excessively squeezed from the bearing.

In cases where it is necessary to insure positive lubrication using a comparatively small amount of lubricant, and yet having a sufficient reserve to enable the bearing to operate with little or no attention for long periods of time. This applies to inaccessible bearings where gravity feed grease cups or pin cups are used and where the use of oil would entail shutting down the machinery for its application.

Where the bearing may be subject to repeated shocks or suddenly reversed bearing pressures as found on motor car chassis, crank pins, etc.

When relatively high operating temperatures are encountered.

In those cases where it is desirable that the lubricant retain its position on the journal or "stay put," whether the machine is in motion or idle, such as in locomotive driving journal lubrication.

In all cases of relatively high speed, or wherever the oil can be effectively applied and retained by the bearing, grease lubrication is not desirable unless other exceptional conditions warrant it, since there would be an increase in power consumption and bearing temperatures. Grease should not be used where it may come in contact with steam and in particular where the exhaust steam is condensed and returned to the boiler feed.

Grease-Feeding Equipment

The type of equipment available and in use for feeding greases to the parts to be lubricated is often an important factor in determining when to use grease and in selecting the most desirable grease. Those methods most frequently encountered for applying grease to the surfaces to be lubricated may be listed as follows:

Packing the grease on and around the journal at required intervals.
Applying the grease to the wearing surfaces by means of a swab, a paddle or a brush.
Plain hand compression grease cups.
Pin cups, gravity or vibration feed; funnel cups.
Automatic compression cups (spring or air types).
Compression cups with automatic feed.
Screw feed, marine type, hand operated cups.
Pressure system, using some form of grease gun for forcing the grease to the bearing at pressures of from 100 to 5000 pounds per square inch (Alemite, Zerk, Thuro (Larkin), etc.).

Grease Cups. The usual type of grease cup is that known as the plain hand compression cup. Its general use is due to its cheapness and the fact that it can be made in very small, compact sizes. This type of cup is sometimes fitted with a spring ratchet which prevents the cap from being lost when used under conditions where the machine is subject to heavy vibration. These cups are sometimes equipped with a ball check valve when used on water circulating pumps and other places where water might back up into the cup.

In the general type of compression grease cup the bonnet or cap is filled with the grease. When the top is turned down, the grease is forced into the bearing. The chief fault of this procedure is that the grease is fed unevenly, more grease than is required for satisfactory lubrication being forced into the bearing when the cap is screwed down. Grease is often forced out of the bearing and wasted. Not only is this type of cup wasteful but it requires frequent attention. The advantages of the cup are apparent in those places where its size, simplicity and low cost are of importance. The metals used in the construction of these cups may be pressed steel, cast iron or brass. The capacities vary from one-quarter ounce to eight ounces.

The gravity type of grease cup is particularly adapted to line shaft lubrication. It is also largely used on hoisting machinery and cranes where it can be installed where the vibrating rod can reach the rotating shaft. This type of cup is provided with a soft copper rod, cut to the correct length to permit its end to rest lightly on the revolving shaft.

Table 1—Industrial Uses of Cup Grease

Machine	Name of grease	Application	Grade	A.S.T.M Worked penetration	Ubbelohde Melting point ° F	Viscosity of mineral oil at 100° F.
Airplanes	Pressure	Pressure or cups	1	375	175	450
Air brakes, railway	Graphite	Cups	3	250	185	110
Air compressors	Cups	Cup	3–4	255–195	190–200	100
Automatic stokers	Pressure	Pressure	1	375	175	300
Automobiles:						
Chassis	Pressure	Pressure or cups	1	375	175	300
Wheel bearings (See wheel bearing greases)	Pressure	Pressure or pack	1	375	175	300
Water pump (See water pump greases)	Cup	Cups	3–4	255–195	190–200	180
Distributor	Cup	Pressure or cups	3–4	255–195	190–200	180
Suspension springs (See spring grease)	Graphite	Pack	3	255	185	110
Speedometer shaft	Graphite	Pack	3	255	185	180
Steering-gear (See steering gear lubricant)	Pressure	Pressure or cups	1	350	175	300
Axles, wagon	Axle	Paddle	3	255	185	400
Baking machinery:						
Dough dividers	Pressure	Pressure	1½	350	180	300
Ball bearings:						
Low temperatures	Petrolatum	.....	....	....	....	....
Medium temperatures	Cup	Pack	1	375	175	300
Boiler plant machinery:						
Water and oil pumps:						
Recip., External Bearings	Pressure	Pressure	1	375	175	300
" " "	Pin cup	Pin cups	1	375	175	225
Centrifugal pumps	Pressure	Pressure	2	315	180	300
" "	Pin cup	Pin cups	1	375	175	225
Coal conveyor rolls	Cup	Cups	3	255	185	180
Ash gate roll bearing	Pressure	Pressure	1½	350	180	300
Draft fans, ball and roller bearings	Cup	Pack	3	255	185	180
Scraper chain sheaves	Cup	Cups	1	375	175	180
Blowers and fans	Pressure	Pressure	1½	350	180	300
" " "	Pin cup	Pin cups	1	375	175	225

Brick plant machinery:						
Skip hoists, cars, roller bearings	Cup	Pack	1–2	375–315	175–180	180
Brick and tile cutters	Pressure	Pressure	1	375	175	300
Bottling plants:						
Bottling machine bearings	Pressure	Pressure	1	375	175	300
Belt conveyors bearings	Pressure	Pressure	1	375	175	300
Cable car pulley and reel bearings	Cup	Compression cups	3	255	175	180
Car loaders	Pressure	Pressure	1	375	175	300
Cement mills:						
Ball mills or granulators	Cup	Compression cups	3	255	185	300
Cold rolls	Neck	Pack	4	195	195	300
Conveyors, belt	Pressure	Pressure	1	375	175	300
Cranes	Cup or pressure	Cups	1	375	175	300
Crushers, jaw: Pitman bearing	Cup	Cups	3	255	185	400
Double seamer	Cup	.....	5	150	200	400
Drawing, wire	Drawing	Pack	3	255	185	100
Drills, rock: chucks	Cup	Pack or pressure	3	255	185	300
Elevator bearings	Cup (graphite)	Cups	1½	350	180	300
Engines, steam: crank pins	Crank pin	Cups	5	150	200	400
Electric motors: ball bearing	Cup	Pack	1	375	175	300
Electric street railways:						
Ball and roller bearings	Cup	Pack	1½	350	180	400
Door engines, slides	Liquid	.....		500 plus	120	300
Air brake cylinders	Liquid	.....		500 plus	120	300
Controller contacts, trips, fingers, drums, etc.	Liquid	.....		500 plus	120	300
Motormen's valves, inspec.	Liquid	.....		500 plus	120	300
Trolley wheels, hollow	Liquid	.....		500 plus	120	300
Trolley bases	Semifluid	.....	00	470	120	300
Signals, interlocking: cylinders	Semifluid	.....	00	470	120	300
Flour mills:						
Cleaning screens	Cup	.....	0	430	160	250
Scourers	Cup	.....	0	430	160	250
Sifters	Cup	.....	0	430	160	250
Purifiers	Cup	.....	0	430	160	250
Reels	Cup	.....	0	430	160	250
Flour dressers	Cup	... .	0	430	160	250

Table 1—Industrial Uses of Cup Grease—(*Continued*)

Machine	Name of grease	Application	Grade	A.S.T.M. Worked penetration	Ubbelohde Melting point ° F.	Viscosity of mineral oil at 100° F.
Flour mills:						
Bran dusters	Cup	.....	0	430	160	250
Bagging machines	Cup	.....	0	430	160	250
Line shafting	Cup	.....	0	430	160	250
Wheat rolls: fixed collar bearing	Cup	.....	0	430	160	250
Fans and blowers: ball bearing	Cup	Pack	0	430	160	250
Fence looms	Pressure	Pressure	1	375	175	300
Freight elevators	Pressure	Pressure	1	375	175	300
Farm implements:						
Combined harvesters	Pressure	Pressure	1	375	175	300
Disc harrows	Cup	Cups	2	315	180	400
Plows: wheel bearings	Cup	Cups	2	315	180	400
Gas engines, crank shaft	Cup	Cups	1½	350	180	300
Gear sets (enclosed)	Gear	Pack	00	470	140	450
Gins, cotton	Pressure	Pressure	1	375	175	300
Grain elevators	Pressure	Pressure	1	375	175	300
Grain harvesters	Pressure	Pressure	1	375	175	300
Hyatt roller bearings	Liquid—3	Pack	3	255	...	300
Line shafts	Pin cup	Gravity cups	0	430	160	240
Laundry machinery	Pressure	Pressure	1	375	175	300
Lathes	Cup	Cups	3	255	185	180
Marine engines	Cup	Cups	3	255	185	180
Mechanical stokers	Pressure	Pressure	1½	350	180	300
Mining equipment:						
Mine cars, plain	Pit car	Pressure	2	315	180	300
" " Hyatt (see Hyatt roller bearings)	Mine car	Pressure	2	315	180	300
" " Timken	Mine car	Pressure	3	255	185	350
Rock drills: chuck fittings	Cup	Pack	3	255	185	180
Air hoists	Cup	Cups	3	255	185	180

Mine pumps: plunger poles	Graphite	Hand	4	195	195	300
Newspaper plants: presses, cams	Cup	Cups	1	375	175	180
Paper mills:						
Paper box plants:						
Cornering machines	Cup	Cups	1	375	175	180
Scouring machines	Cup	Cups	1	375	175	180
Die presses	Cup	Cups	1	375	175	180
Paper slitters	Cup	Cups	0	430	140	180
Auto box machine	Cup	Cups	0	430	140	180
Logging operations:						
Donkey engines, crank pins	Crank pin	Cups	5	150	200	700
Miscellaneous slides	Liquid	Hand	...	...	...	300
Trucks, chassis bearings	Pressure	Pressure	1	375	175	300
Pulp mill:						
Tumbling drums	Cup	Cups	3	255	185	180
Splitters	Cup	Cups	3	255	185	180
Chip screens	Cup	Cups	3	255	185	180
Pulp screens	Cup	Cups	3	255	185	180
Fourdrinier machines:						
Miscellaneous felt rolls	Pressure	Pressure	1½	350	180	300
Rotary cutters, folders, layboys, trimmers, rewinders, and roll wrapping machines	Cup	Cups	3	255	185	180
Paper coating and saturating machines	Cup	Cups	3	255	185	180
Creping and corrugating machines	Cup	Cups	3	255	185	180
Bag and box machinery	Cup	Cups	3	255	185	180
Envelope, tube, cup and cap machinery	Cup	Cups	3	255	185	180
Plush and tapestry mills	Cup	Cups	0	430	160	300
Pneumatic tools:						
Drills, riveting hammers, chipping and caulking tools, concrete breakers, wood and stone working tools, operating on dry air.						
Valves, pistons, and other moving parts	Cup	Pack or pressure	00	470	140	180

Table 1—Industrial Uses of Cup Grease—(*Continued*)

Machine	Name of grease	Application	Grade	A.S.T.M. Worked penetration	Ubbelohde Melting point ° F.	Viscosity of mineral oil at 100° F.
Pneumatic tools:						
Rock drills, stoppers, augers, hand hammers, drifters, and other tools operating on a mixture of air and water.						
Valves, pistons, and other moving parts	Cup	Pack or pressure	0	430	140	180
Chuck fittings	Cup	Hand	3	255	185	180
Air hoists: motor bearings	Cup	Pack	2	315	180	180
Pumps, reciprocating (steam driven)						
External bearings (compression)	Cup	Cups	1 or	375	175	180
			3	255	185	180
" "	Pin cup	Pin cups	0 or	430	160	225
			1	375	175	...
Pumps, electrically driven:						
External bearings	Compression cup	Cups	1	375	175	180
" "	Pin cup	Pin cups	1	375	175	225
Pumps, rotary and centrifugal:						
Bearings: compression cups	Cup	Cups	1 or	375	175	180
			3	255	185	180
External bearings	Pin cup	Pin cups	1	375	175	225
Ball and roller bearings	Cup	Cups	1	375	175	180
Quilt mills:						
Carding machines, garnetting machines, sewers and lapers	Cup	Cups	3	255	185	180
Railways:						
Air brakes	Liquid	Pack	...	...	...	300
General shop lubrication	Cup	Cups	3	255	185	180
Shipyards:						
Plate and angle shops:						
Pneumatic tools	Cup	Pack or pressure	00	470	140	180
Shipways:						
Cranes	Cup	Cups	3	255	185	180
Launching	Launching	Hand	4	195	195	100

Shoe factories:						
Clicking machine	Cup	Cups	0	430	160	250
Channeling machine	Cup	Cups	0	430	160	180
Rounding machine	Cup	Cups	1	375	175	180
Insole tackers	Cup	Cups	0	430	160	250
Jack roller	Cup	Cups	0	430	160	250
Large splitting machine	Cup	Cups	3	255	185	180
Stapling machine	Cup	Cups	0	430	160	250
Inking machine	Cup	Cups	0	430	160	250
Burnishing machine	Cup	Cups	0	430	160	250
Brushing machine	Cup	Cups	00	470	140	180
Buffing machine	Cup	Cups	00	470	140	180
Gang brushing machine	Cup	Cups	00	470	140	180
Counter moulder	Cup	Cups	0	430	160	250
Butting machine	Cup	Cups	0	430	160	250
Stamping machine	Cup	Cups	0	430	160	250
Slugging machine	Cup	Cups	0	430	160	250
Tree machine	Cup	Cups	0	430	160	250
Tip-scouring machine	Cup	Cups	0	430	160	250
Heel seat nailer	Cup	Cups	0	430	160	250
Soling machine	Cup	Cups	0	430	160	250
Inseam trimmer	Cup	Cups	0	430	160	250
Inseam welt stitcher	Cup	Cups	0	430	160	250
Insole and heel seat trimmer	Cup	Cups	00	470	140	180
Marking machine, toe trimmer, pull overs	Cup	Cups	0	430	160	250
Nigger head	Cup	Cups	0	430	160	250
Sole leveling machine	Cup	Cups	0	430	160	350
Heel trimmer	Cup	Cups	00	470	140	180
Sanders	Cup	Cups	00	470	140	180
Heel breast scourer	Cup	Cups	00	470	140	180
Crimping machine	Cup	Cups	0	430	160	250
Outsole stitcher, heeling machine, heel-slicing machine, sole cutters, skivers, rollers, sole grading machine	Cup	Cups	0	430	190	250

Table 1—Industrial Uses of Cup Grease—(*Continued*)

Machine	Name of grease	Application	Grade	A.S.T.M. Worked penetration	Ubbelohde Melting point ° F.	Viscosity of mineral oil at 100° F.
Steel mills:						
Blast furnace:						
Trolley bearings and line shafting (Winter)	Cup	Cups	1	375	175	180
(Summer)	Cup	Cups	3	255	185	180
Open hearth furnace:						
Furnaces, door plungers	Graphite	Hand	4	195	195	300
Cold rolls	Cold neck	Pack	5	150	200	400
Charging machines and ladle cranes	Cup	Cups	3	255	185	180
Sugar mill machinery:						
Syrup, juice, and circulating pumps (see recommendations for centrifugal pumps, and reciprocating pumps).						
Tanneries:						
Shaving machine						
(Summer)	Cup	Cups	4	195	195	180
(Winter)	Cup	Cups	3	255	185	180
Splitter						
(Summer)	Cup	Cups	4	195	195	300
(Winter)	Cup	Cups	3	255	185	300
Setting machine	Cup	Cups	3	255	185	300
Coloring machine	Cup	Cups	3	255	185	180
Jack rollers	Cup	Cups	3	255	185	180
Textile mills:						
Scouring machinery:						
Washers and dryers bearings	Cup	Cups	3	255	185	180
Cams	Cup	Cups	3	255	185	180

Carding machinery:						
Comb boxes	Comb box	Pack	00	470	140	225
Ball or roller bearings	Cup	Pack	0	430	160	300
Cleansing equipment:						
Plain bearings	Cup	Pack	3	255	185	180
Ball or roller bearings	Cup	Pack	0	430	160	300
Gills and backwashers:						
Fallers, screws, slides, etc.	Cup	Hand	1	375	175	180
Gears, etc.	Cup	Pack	2	315	180	180
Combing machines:						
Roll bearings	Cup	Pack	00	470	140	180
Cam motions	Cup	Hand	3	255	185	180
Drawing and spinning machinery:						
Roll bearings	Cup	Pack	00	470	140	180
Miscellaneous bearings, gears, chains, etc.	Cup	Hand	3	255	185	180
Weaving equipment:						
Roll bearings	Cup	Pack	00	470	140	180
Cams, harness motions, gears and chains	Cup	Hand	3	255	185	180
Finishing machinery:						
Fulling machines, washers, raising, napping, cropping and pressing equipment, plain bearings	Cup	Pack	3	255	185	180
Tinplate mills:						
General lubrication and line shafting	Cup	Cups	3	255	185	180
Tractors:						
Fan bearings, axle bearings, steering mechanism joints and connections, link pins and rollers	Pressure	Pressure or cups	1½	350	185	300
Wire drawing	Drawing	Pack	3	255	185	180

Gravity, the tendency to create a slight vacuum in the bearing, the vibration of the copper rod, and the transfer of heat from the journal up the rod all tend to cause the grease to flow down the shaft. This type of cup may be classed as a semi-automatic grease cup. It feeds only when the shaft is in motion and requires no adjustment until the cup needs refilling. For lubrication of ordinary shafting one filling of the usual four ounce cup will last for a month and in some cases two or three times this long.

The automatic compression cup is particularly adapted for moving bearings such as crank-pins, cross-heads, slides, eccentrics, etc. The pressure for forcing the grease into the bearings is supplied by coil springs or compresed air. For this reason the feed is semi-automatic.

The object of the Uniflow type of cup is to control and maintain uniform the flow of grease from the cup, by proportioning the area of the exit orifice against the varying spring pressure acting on the grease, as the piston moves from full compression to the end of its stroke. This feature is realized by a tapered orifice at the bottom of the cup, within which a controlling plunger plug moves. When the cup is filled with grease and the cover is in place, the piston is pushed up, carrying the regulating plunger plug to the small end of the tapered orifice, and, as the spring pressure, due to full compression, is highest at that time, the plug restricts the outlet to its minimum. As the piston moves down and the spring pressure is reduced, the area of the outlet exit is enlarged proportionately by the movement of the plug, which is carried by the piston and which thus moves down in the tapered orifice, thus offering a larger opening for the lubricant to pass through. It is possible to vary the flow of the grease according to the taper and set of the orifice to meet any specified feed for proper lubrication. It is obvious that no grease will be fed to the bearing when the journal is at rest, which prevents flooding and waste. The amount of grease in the cup at any time is indicated by the position of a rod which protrudes through the cap of the cup and moves with the piston. It is said that this type of cup gives a steady dependable flow of grease.

The Marine type of cup is threaded on the inside and the plunger on its outer surface. It is used on marine engines or other places where it is necessary to force grease some distance. It is also found on mining machinery. It may be obtained in $\frac{1}{8}$, 1, $1\frac{1}{2}$, 3, 6, and 10-ounce sizes.

Pressure System. The pressure system of applying lubricating greases consists of the following three units:

A grease gun or compressor which may be hand or power operated. Some of these compressors develop a pressure of 5000 pounds and in some cases even higher.

A conduit, rigid or flexible, provided with a quickly detachable coupling, through which the grease is forced to the bearings.

Fittings, which are check valve nipples screwed into the bearing housings and remain there as permanent fixtures.

General Applications for Cup Greases

Table 1 indicates the general applications for which normal cup greases are adaptable. In many cases, for best results, specialized lubricants should

TABLE 2—Analyses of Commercial Cup Greases

Per cent by weight

Type	Free Fat	Combined Fat	Free Fatty Acids	Free CaO	Combined CaO	Water	Oil	Viscosity (100° F.)	Melting Point (° F.)	A.S.T.M. Penetration
No. 1 Cup Grease	2.61	9.49	.18	.40	.95	.23	86.14	105	157	303
No. 2 Cup Grease	4.13	11.62	.38	.35	1.16	Trace	82.36	105	159	233
No. 3 Cup Grease A	2.82	10.90	.48	...	2.47	1.86	81.47	90	220	...
No. 3 Cup Grease B	.78	8.54	.60	.24	.85	.50	88.69	350	177	350
No. 3 Cup Grease C	1.91	15.25	.50	.72	1.76	1.62	78.22	...	200	...
No. 3 Cup Grease D	...	13.85	...	...	2.08*	.98	83.08	100	195	...
No. 3 Cup Grease E	3.92	16.78	...	...	...	...	....	105	166	185
No. 3 Cup Grease F	4.00	16.77	.07	.16	1.68	.40	76.92	105	192	180
No. 3 Cup Grease G	2.53	16.22	.09	.88	1.89	2.03	76.36	...	164	...
No. 4 Cup Grease A	2.78	25.05	.07	2.63	2.79	15.44	51.26	...	208	...
No. 4 Cup Grease B	6.50	20.23	Trace	.24	2.03	.30	70.70	105	174	114
No. 5 Cup Grease	4.19	25.48	...	.42	2.55	1.62	65.74	105	180	78
High Pressure Grease A	.41	12.63	Trace	.27	1.26	1.50	83.90	236	...	...
High Pressure Grease B	.30	8.71	.87	.09	.87	1.00	88.16	...	189	...
High Pressure Grease C	.50	13.35	...	...	1.45	1.00	83.70	300	205	325
No. 3 Pin Cup Grease A	1.70	7.96	1.04	.22	.79	1.25	86.94	241	181	410
No. 3 Pin Cup Grease B	1.76	7.70	.76	.08	...	.95	87.98	224	180	425
No. 3 Black Cup Grease	.24	16.12	Trace	.55	1.61	2.66	78.82	132	195	...
No. 2 Tallow Compound	1.70	14.70	.40	.90	1.50	1.30	79.50	...	208	200
No. 5 Tallow Compound	1.80	27.20	.20	1.30	2.70	2.80	64.00	...	242	40

* Hydrate.

be applied. In the case of No. 0 and No. 00 Cup Grease, the worked penetrations are given, beyond the accurate range of penetrometers, as MacMichael viscometers are not available at all grease plants.

Pale Automotive Cup Greases

There is no great distinction in manufacturing methods between automotive cup greases and pressure gun lubricants intended for general chassis lubrication. During the period when lime base greases containing heavy mineral oil and special compounding materials were being developed as special gun greases, normal No. 2 cup greases were marketed by many grease manufacturers as auto cup greases. Heavy grades of automotive cup greases were marketed prior to the advent of pressure lubrication systems when the automobile carried a great number of cups as compared with the modern vehicle. The consistency of auto cup greases has now been varied to meet the requirements of cup and pressure lubrication systems and they are sometimes sold as a lower priced chassis or pressure gun grease. Where modern cars carry cups (excepting the water pump) the auto cup greases may be used, as they are usually slightly more consistent than pressure gun greases.

A survey of auto cup greases and pressure gun lubricants made in 1934 indicated several outstanding differences which may be of some importance from the standpoint of lubrication technique, and are of interest in connection with marketing policies of the various manufacturers. In the majority of cases the auto cup greases were made with mineral oil having viscosities less than 300 S.U.V./100° F., whereas most of the pressure gun greases examined had viscosities above this value and as high as 1200 S.U.V./100° F. The cup greases are usually yellow as compared with green or red green for pressure lubricants. Their average consistency is about 250 (worked A.S.T.M. Penetration) as compared with 340 for gun greases.

American Auto Cup Greases (1934)

The characteristics of these greases are shown in the table on page 211.

The following specifications cover an automotive cup grease which has been marketed in the Mississippi valley for several years:

Grade	No. 3
A.S.T.M. worked penetration	240–270
Ubbelohde dropping point ° F., min.	185
Per cent by weight free alkali, max.	0.50
Per cent by weight free acid (as oleic), max.	0.50
Percent water by distillation, max.	2.50
Percent ash, max.	2.60
Corrosion (530.4)	Neg.
A.S.T.M. soap content	14–20
Kind of soap	Calcium
Mineral oil:	
S.U.V./100° F.	300–325
A.S.T.M. color, max.	3

	1	2	3	4	5	6	7	8
Per cent by weight soap	18.0	12–13	15–16	15–20	16.6	18.1	18.0	15.0
Kind of soap	Ca	Ca	Ca	Ca	Ca	Ca	Ca	Ca
Per cent water (max.)	1	0.7	0.9	...	0.65	0.7	2.0	.5–1.5
Per cent by weight mineral oil	80	87–88	84–85	...	82.25	81.2	80	...
Grav. A.P.I. of mineral oil	24–25	28	28	...	24	25	...	...
Flash ° F.	400	360	360	...	...	...	420	...
Fire ° F.	450	400	400	...	...	...	475	...
S.U.V./100° F. of mineral oil	480–500	100	100	95	300	110	75–150	...
A.S.T.M. color of mineral oil	7	4	3	3	6	3	3	...
Pour point of mineral oil ° F.	0	25	25	0	0	0	30	...
Color of grease	Lt. Brown	Yellow	Yellow	Yellow	Yellow	Lt. Yellow	Lt. Yellow	Red
A.S.T.M. penetration at 77° F. worked unworked	185–215	285–315	200–270	225–275 150–200	250	250	255–265	220–240
Percent free alkali as $Ca(OH)_2$	0.20	0.20	...	...	...	...	0.25	...
Dropping point ° F.	200–205	...	...	194–203	190	190	180–190	190
Percent ash as sulfates	5	...	...	...	1.47	1.68	6	2–4
Corrosion (copper strip)	Neg.	Neg.	Neg.	Neg.	Neg.	Neg.	Neg.	Neg.
Odor	Mirbane	...	...	...	...	...	Mirbane	Mirbane

In 1935, eight of the largest grease manufacturers in America were prepared to meet all of the above requirements with the exception of soap contents and ash which they specified as follows:

Company Number:	1	2	3	4	5	6	7	8
A.S.T.M. soap content, per cent by weight:								
Min.	16	12	17	17	18	17	20	16
Max.	..	15	..	18	20	18	23	..
A.S.T.M. ash, percent max.	2.6	2.5	2.6	2.0	2.6	1.75	6.5	1.6

Purchase Specifications: Motor Company C, No. 2½ Winter Grade Cup Grease (MS 48-1930).

This specification covers a grade of Cup Grease used during the cold season from November 1st to March 31st of each year.

This grease shall be a well manufactured, homogeneous product, composed of 12 per cent to 14 per cent calcium soap, made from high grade animal or vegetable oils or fatty acids, and a highly refined mineral oil with a minimum viscosity of 100 seconds Saybolt at 100° F.

The grease shall contain no fillers such as resin, resinous oils, soapstone, wax, talc, powdered mica, graphite, sulphur, clay, etc.

Ash Content—Maximum 2 per cent.

Water—Maximum 1.5 per cent.

Corrosion—A clean copper plate shall not be discolored when submerged in the grease for 24 hours at room temperature.

Odor—There shall be no disagreeable odor or rancidity.

Melting Point—Minimum 175° F. (See Par. No. 6).

Stiffness—50-70 MacMichael degrees obtained with a MacMichael Viscosimeter, using No. 22 wire at 20 R.P.M. and at 90° F.

Cold Test—The grease shall be comparatively easy to stir at 0° F.

Separation—The soap and oil shall not separate and shall not harden objectionably with age or exposure.

All tests shall be made according to the methods for testing lubricants adopted by the American Society for Testing Materials, unless otherwise specified.

Tests for Melting Point shall be made as follows:

Apparatus: Glass tube, 8 cm. long by 0.4 cm. bore, open at both ends; 600 cc. low form beaker; thermometer and burner (Bunsen).

Procedure: One end of the tube is dipped in the grease so that a column 1 cm. long is used. The tube is attached to a thermometer by means of a rubber band, so that the bottom of the grease column is opposite the thermometer bulb, and is placed in a water or air bath. The heat is applied at the rate of 5 to 7° F. per minute. The temperature is read when the grease column moves in the tube. This is the melting point. The test shall be run in duplicate.

Purchase Specifications: Motor Company C, No. 2½ Winter Grade Cup Grease. (MS 49-1930).

This specification covers a grade of cup grease used during the warm season from April 1st to October 31st of each year.

This grease shall be a well manufactured, homogeneous product, composed of 14 to 16 per cent calcium soap, made from high grade animal or vegetable oils or fatty acids, and a highly refined mineral oil with a minimum viscosity of 100 seconds Saybolt at 100° F.

The grease shall contain no fillers such as resin, resinous oils, soapstone, wax, talc, powdered mica, graphite, sulphur, clay, etc.

Ash Content—Maximum 2.4 per cent.

Water—Maximum 1.5 per cent.

Corrosion—A clean copper plate shall not be discolored when submerged in the grease for 24 hours at room temperature.

Odor—There shall be no disagreeable odor or rancidity.
Melting Point—Minimum 190° F. (See Par. No. 6).
Stiffness—75-95 MacMichael degrees, obtained with the MacMichael Viscosimeter, using No. 22 wire at 20 R.P.M. and 90° F.
Separation—The soap and oil shall not separate and shall not harden objectionably with age or exposure.

All tests shall be made according to the methods for testing lubricants adopted by the American Society for Testing Materials, unless otherwise specified.

Tests for Melting Point shall be made as follows:

Apparatus: Glass tube, 8 cm. long by 0.4 cm. bore, open at both ends; 600 cc. low form beaker; thermometer and burner (Bunsen).

Procedure: One end of the tube is dipped in the grease so that a column 1 cm. long is used. The tube is attached to a thermometer by means of a rubber band, so that the bottom of the grease column is opposite the thermometer bulb, and is placed in a water or air bath. The heat is applied at the rate of 5 to 7° F. per minute. The temperature is read when the grease column moves in the tube. This is the melting point. The test shall be run in duplicate.

Purchase Specifications: G.M. Cup Greases (1933).

General: Greases shall be homogeneous combinations of refined mineral oil with calcium or sodium soap as specified.

Filler: Greases shall not contain filler such as clay, talc, whiting, asbestos, wood fibre, etc. Graphite when added at purchaser's request to any non-graphite grease shall comply with the sand and silicate requirement of this specification.

Consistency: Consistency of distributor grease shall accord with standards approved by the purchaser.

Odor: Greases shall not be perfumed and shall be free from disagreeable odor or rancidity.

Permanence: The soap and oil shall not separate at reasonable temperatures within a reasonable time. Greases shall not harden objectionably with age or exposure.

Corrosion: Greases shall not corrode metal.

Methods of Test: Tests will be made in accordance with methods described in G.M. Standards, Vol II, Page D-6.

G.M. No.	Grade	Consistency Worked m.m. Min	Consistency Worked m.m. Max.	Viscosity Saybolt Universal of Mineral Oil 100° F. Min.	100° F. Max.	210° F. Min.	210° F. Max.
4532-M	Shop Grease, G-1	30	35	275	325		
4533-M	Shop Grease, G-2	27	32	475	525		
4534-M	Cup Grease, G-2½	25	31	75			

G.M. No.	Grade	Soap	Free Fatty Acids, as Oleic Max.	Uncombined Lime Max.	Sand Silicates or Gritty Substances Max.	Water Max.	Graphite
4532-M	Shop Grease, G-1	5 to 8%*	0.2%	0.10%	0.02%	1.0%	
4533-M	Shop Grease, G-2	9 to 12%*	0.2%	0.10%	0.02%	1.0%	
4534-M	Cup Grease, G-2½	12 to 14%*	0.3%	0.15%	0.03%	1.5%	

* The lime used in preparing the soap shall not contain more than 7 per cent of magnesium oxide and not more than 2 per cent of gritty non-soap forming substances. Not less than 93 per cent of the soap shall be calcium (and magnesium) soap. A small amount of caustic soda may be used, if desired, for completing the saponification, but the sodium soap must not exceed 7 per cent of the total soap.

Black Industrial Cup Greases

Under this heading dark-colored or black lime base cup greases intended for general industrial purposes are discussed. The color of these products may be due to the natural appearance of dark lubricating distillates, the presence of tarry matter, or the use of dyed oils. The dark color ordinarily indicates mineral lubricating material of appreciably higher viscosity than

the pale spindle oils frequently used in the pale industrial cup greases. The black cup greases are particularly suitable for heavy pressures and slow speeds, and the presence of asphaltic matter often makes them of extraordinary value where bearings must be lubricated in the presence of water. For conical ball, pebble or rod mills, the bearing caps should be filled once per day with No. 4 or No. 5 Black Cup Grease. For logging locomotive pin bearings, and donkey engine crank pin bearings the heavy grades of black cup greases made with very viscous oils have found extensive use. The heavy black cup greases may, in some cases, be found useful as lubricants of large exposed slow speed gears, especially where heat resistance is essential.

Many of the early cup greases previously mentioned in connection with various patents, were prepared with crude oil and consequently were black in color. In 1900, Hudnall obtained a patent [61] for a black grease containing lime soap. It was prepared by making an emulsion of light mineral oil with slaked lime, adding black oil and then heating this mixture. As the final stage of the process the mixture of the foregoing materials was stirred together with a hot sodium soap solution, and during this period the lime soaps formed in accordance with the double decomposition reaction. Crude oils have been [62] treated with calcium chloride to thicken them as substitutes for vaseline. In 1916, Tutleman [63] obtained a patent for a lime base grease made with black oil. For a consistency equivalent to an ordinary No. 3 cup grease she recommended the following formula:

13 pounds	Lime (CaO)
3½ gallons	Water
7½ pounds	Black mineral oil
2½ gallons	Commercial oleic acid
2½ gallons	Sodium carbonate
1 gallon	Yellow ocher

It was claimed that one of the outstanding features of this product was its tendency to reduce the temperature of bearings which were running at a high temperature. The lime was slaked in the water, the soda ash added, and the mixture strained. The black oil and fatty acid (red oil) were combined and mixed with the alkali solution to permit saponification to take place. The process was carried out in the cold and the proportions were apparently so selected that no difficulty in dispersing the lime soap and stabilizing it were in evidence.

Heavy black oils have been found to be particularly useful in automotive wheel bearing greases.

Standardized Black Cup Grease Formulae (Pressure Saponification Method)

The following formulae and process has been in use for several years on a commercial scale, and the products have been well received on the Pacific Coast.

[61] U. S. Patent 670,783.
[62] British Patent 24,801 (Nov. 15, 1902).
[63] U. S. Patent 1,204,830 (Nov. 14, 1916).

Constituents	Gallons	Pounds Net	Per Cent by Weight
Black Cup Grease No. 2			
Tallow	80	610.16	10.00
Hydrated lime		100.00	1.64
30° Bé. caustic soda	0.5	1.24	.02
300 pale oil	533	3699.00	60.64
Heavy vac. dist.	98	793.00	13.00
100–120 penn. asph.	94	793.00	13.00
Water		103.60	1.70
		6100.00	100.00
Black Cup Grease No. 3			
Tallow	100	762.70	11.74
30° Bé. caustic soda	0.5	1.24	.02
Hydrated lime	...	120.00	1.85
100–200 penn. asph.	110	920.34	14.17
Heavy vac. dist.	123	993.72	15.30
300 pale oil	466	3547.61	54.62
Water		149.39	2.30
		6495.00	100.00
Black Cup Grease No. 4			
Tallow	100	762.70	16.24
Hydrated lime		120.00	2.56
30° Bé. caustic soda	0.5	1.24	.03
300 pale oil	297	2261.71	48.17
Heavy vac. dist.	94	704.25	15.00
100–120 penn. asph.	87	732.42	15.60
Water		112.68	2.40
		4695.00	100.00
Black Cup Grease No. 5			
Tallow	100	762.70	24.60
Hydrated lime	...	120.00	3.87
30° Bé. caustic soda	0.5	1.24	.04
300 pale oil	225	1712.01	55.23
100–120 penn. asph.	49	411.05	13.26
Water		93.00	3.00
		3100.00	100.00
Black Cup Grease No. 6			
Tallow	100	762.70	28.14
Hydrated lime		120.00	4.43
30° Bé. caustic soda	0.5	1.24	.04
300 pale oil	180	1372.46	50.65
100–120 penn. asph.	44	369.60	13.64
Water	...	84.00	3.10
		2710.00	100.00

The procedure for loading the autoclave and saponifying the fat is the same as described for the Standardized Pale Industrial Cup Greases.

Reduction of the Soap

Fifty gallons of black Cup Grease slop, or 300 S.U.V./100° F. Pale Oil, is charged into an open grease mixer of 1000-gallons capacity, and

heated to 200 to 220° F. The completed soap from the autoclave is then discharged into the mixer, and the agitating paddles started. The 300 Pale oil is then allowed to flow slowly into the kettle, and adjusted at a rate which will not cause serious flooding and consequent chilling of the grease. In an overhead kettle, the required amount of 100 to 120 Penetration Asphalt (Air Blown) is heated to about 150° F. After the required amount of 300 Pale oil and Heavy California Vacuum Lubricating Distillate (87 S.U.V./210° F.) has been blended with the soap base the melted asphalt is slowly added while agitating.

Particular care must be exerted to insure the presence of an adequate amount of water for dispersing the lime soap in such a viscous grease. Usually from 5 to 10 gallons of water must be added, preferably just before running in the melted asphalt. Water contents which are too low result in brittle grease, and produce non-homogeneity.

When the grease has been reduced to approximately the desired consistency, agitation is discontinued and a sample taken for consistency test, the quick test method previously discussed being employed.

When laboratory approval of the grease in the kettle has been received it is brought to the desired drawing temperature as indicated below, and then filled at once into steel drums or cans.

Black Cup Grease No	2	3	4	5	6
Drawing temp. ° F.	170–175	175-180	180–190	185–195	195–205
Mesh of screen used	60	60	20	20	20
A.S.T.M. unworked penetration at 77° F.	310–360	230–290	190–230	80–120	55– 80
Ubbelohde dropping point ° F.	200–205	200–210	200–215	205–220	205–220
Percent free alkali ($Ca(OH)_2$)			.40–.60		

Applications

The foregoing Black Cup Greases have been used as crank pin greases, as general heavy machinery lubricants, and as drive shaft bearing greases on gold dredgers. They may be subjected in service to temperatures over 200° F., and while they melt at from 200 to 220° F., they can not be considered as non-separating greases. On heating to above their melting points and then cooling, free oil will normally appear and the soap tends to separate. This separation is not so evident in the No. 4 grade as for the softer and heavier grades. They contain from 0.20 per cent to 0.60 per cent free alkali, and if sufficient free fatty acid is incorporated with the finished grease in the cold, to produce a product which on analysis is neutral or slightly acid, the stability with respect to separation is improved. The addition of from ½ to 1 per cent of aluminum stearate still further increases their stability, and increases the viscosity of the melted grease. While these greases separate at 210° F., when stabilized they do not separate in three hours at 250° F. However, the dropping point has not been raised; therefore, these greases cannot be recommended for use at very high temperatures.

The problem of stabilizing a calcium grease against separation, when melted, appears to be a problem of stabilizing the colloidal solution of soap in oil so as to inhibit the formation of soap crystals. The loss of water may be a factor in causing the separation of the soap from the oil, but tests made on Black Cup Grease No. 2 indicate that complete separation can occur when the amount of water lost by evaporation is too small to be measured by the A.S.T.M. method. The presence of free alkali or large amounts of free acid appear to lessen the stability of these greases to heat. Excess free alkali causes separation of the soap from the oil as a hard granular mass. Excess free acid causes the structure of the grease to be completely destroyed upon heating so that the cooled grease resembles a heavy oil. Stearic acid may be used instead of oleic acid to produce a neutral grease but appears to be slightly less effective in stabilizing the grease. It is thought that basic calcium oleate and stearate are responsible for the separation of the soap in greases containing free alkali and that the presence of an excess of acid results in the soap being more completely dissolved in the oil than it is in the colloidal solution existing in a properly made grease. The aluminum stearate probably hinders crystal formation by giving a mixed soap and by aiding in building up the proper charge on the soap particles. It produces a softer grease upon cooling after melting than is obtained with oleic acid alone.

During an investigation blends of Black Cup Greases Nos. 2, 3, 4, 5 and 6 with various amounts of oleic acid, stearic acid, tallow, glycerol and aluminum stearate were heated in an oven at temperatures of 220° F. and 250° F. The greases were warmed while adding the ingredients and great care was taken to insure uniform mixing. The softer grades were mixed with a spatula, the harder grades with an A.S.T.M. worker.

In preliminary experiments the separation of the soap from the oil was tested by means of the filter press method given in the A.S.T.M. Standards, September, 1933. This test would probably be very effective in predicting oil separation in storage, but is not suitable for testing the separation of the soap from the oil of a melted grease. Attempts were made to express separation numerically by determining the per cent of oil which will drain from the separated soap. This was found to be unsatisfactory, since some greases may be completely broken down without oil appearing on the surface in sufficient quantities to be drained off. However, if the hot grease is allowed to completely cool, it is easy to detect separation by comparing the worked and unworked structure with that of the original grease. Although such a test is purely qualitative and includes a personal factor, it is quite exact since the granular texture of "alkaline" separation and the thinning of "acid" separation become so obvious upon working with a spatula that there can be no doubt as to whether or not a grease retains its original structure. Since most greases in the field are judged by their "feel" and "appearance" when rubbed between the thumb and finger, it is probable that greases which pass the above unworked and worked structure test will be found to be satisfactory.

It was thought that glycerol might be effective in stabilizing the soap

by helping to reduce the evaporation of water and by furnishing OH groups which might serve as stabilizing agents as well as water itself. Glycerol appears to have a very slight stabilizing effect, but not enough to justify its use. Tallow was also found to be ineffective, and stearic acid appears to be slightly less effective than oleic acid as a stabilizing agent and is more expensive. As previously mentioned neutral wool fat and wool grease fatty acids are effective as stabilizing agents but they were not included in this investigation.

The greatest portion of the experiments was made in an effort to determine the quantity of oleic acid and aluminum stearate that produces the most stable blend. The following tables give the appearance of various

TABLE 3—Condition of Black Cup Greases After 16 Hours at 220° F.

Grade No.	Per Cent Oleic Acid	Per Cent Al. Stearate Gun Grease *	Free Acid or Alkali	Oil Separation	Texture
2	0	0	Alkali	Layer	Very granular
2	1	0	Alkali	Trace	Works up nearly like original
2	2	0	Neutral	None	Works up thinner than original
2	3	0	Acid	None	Works up very fluid
2	5	0	Acid	None	Liquid grease
2	0	1	Alkali	Layer	Very granular
2	0	3	Alkali	None	Slightly granular upon stirring
2	2	2	Neutral	None	Works up like original grease
2	2	5	Neutral	None	Works up like original grease
2	1	1	Alkali	None	Slightly thinner than 2-2-2
2	1	Glycerin	Alkali	Layer	Like No. 2 alone
2	2	Glycerin	Alkali	Layer	Thinner than No. 2 alone
2	2	Tallow	Alkali	Layer	Like No. 2 alone
2	1	Stearic Acid	Alkali	Trace	Works up slightly granular
2	2	Stearic Acid	Neutral	None	Works up very slightly granular
3	0	0	Alkali	Layer	Very granular
3	2	0	Neutral	None	Works up nearly like original
3	2	5	Neutral	None	Like original
3	2	10	Neutral	None	Thinner
3	2	Stearic Acid	Neutral	Trace Oil next to can**	Thin and granular
4	0	0	Alkali	None	Slightly granular
4	2	0	Neutral	None	Like original
4	2	5	Neutral	None	Like original
4	2	10	Neutral	None	Like original
5	0	0	Alkali	Some	Granular
5	3	0	Neutral	None	Slightly granular
5	3	5	Neutral	None	Slightly granular
5	3	10	Neutral	None	Like original
6	0	0	Alkali	Some	Granular
6	10	0	Acid	None	Very thin. Complete breakdown
6	5	0	Acid	None	Not granular
6	4	0	Neutral	None	Not granular
6	4	5	Neutral	None	Not granular
6	4	10	Neutral	None	Like original

* Aluminum Stearate Gun Grease—5 per cent is equivalent to 0.7 per cent aluminum stearate.

** In the harder grades oil never separates on the surface but sometimes collects next to the can, causing the grease to be free from the container.

blends of black cup grease when heated to 220° for 16 hours and to 250° for 3 hours.

It is concluded that the heat stability of these greases could be greatly improved by the addition of 0.7 per cent aluminum stearate and sufficient oleic acid to produce a neutral grease (.05 per cent alkali to 0.3 per cent acid).

It is considered best to add the aluminum stearate after saponification is complete. Slop grease and oil are added to the kettle into which the autoclave is emptied. If the aluminum stearate was added to this oil and slop grease, aluminum stearate grease may be formed in the kettle before the autoclave was emptied into it. Aluminum stearate readily forms a grease with 300 Pale Oil at temperatures as low as 212° F. within time intervals as short as thirty minutes.

TABLE 4—Condition of Black Cup Greases After 3 Hours at 250° F.

(All Samples Approximately Neutral)

Grade No.	Per Cent Oleic Acid	Per Cent Al. Stearate Grease	Oil Separated	Texture
2	2	0	Some on surface	Works up well, very slightly granular
2	2	2	None	Like original
2	2	5	None	Like original
2	2	10	None	Thinned
3	2	0	None	Thinned
3	2	5	None	Like original
3	2	10	None	Thinned
4	0	0	None	Slightly thinned
4	2	0	None	Nearly like original
4	2	5	None	Slightly thinned
4	2	10	None	Slightly thinned
5	0	0	None	Completely broken down
5	3	0	None	Very hard and crumbly. Works up slightly granular
5	3	5	None	Good texture, works up smooth but soft like a No. 4
5	3	10	None	Good texture, works up smooth but soft like a No. 4
6	0	0	None	Completely broken down
6	4	0	None	Very hard, works up nearly like original
6	5	0	None	Very hard, works up like original
6	4	5	None	Good unworked texture, works up very smooth but softer, like a No. 4
6	4	10	None	Good unworked texture, works up very smooth but softer, like a No. 4

The dropping points of the different grades of Black cup greases fall within a narrow range and are not shifted appreciably by the addition of oleic acid and aluminum stearate.

Grade No.	Per Cent Oleic Acid	Per Cent Al. Stearate Gun Grease	Dropping Point Degrees Centigrade
2	0	0	95
2	1	0	94
2	2	0	95
2	2	2	98
2	2	5	98
4	0	0	97
6	0	0	95
6	1.5	0	99
6	4	0	95
6	4	10	95

Ball and Roller Bearing Greases

(Calcium Base)

Many of the pale and dark industrial cup greases discussed in the foregoing sections are adequate for many ball and roller bearing applications. However, the tendency is towards more stable types of lime base greases and, furthermore, where high temperature conditions exist and grease is used, special short fiber sodium base greases are preferred. In the following general discussions both calcium and sodium base ball bearing greases will be discussed, although the data on sodium greases will be limited here and later amplified in the chapter on this class of greases.

In 1929, the Fafnir Bearing Company issued a list of recommended greases for ball bearings operating under normal conditions of service, which were considered as being:

Bearing Temperatures—30° F. to 100° F.
Speeds—up to 4000 R.P.M.
Bearing Sizes, No. 200 to No. 414.

Of the approved products, only 11 per cent were manufactured with a sodium base, the balance being lime soap greases. This is in contrast with more modern developments. This approved list is quoted below:

The Alemite Mfg. Co., —Alemite Lubricant
(Recommended only where housings are oil tight, as slight separation is sometimes present after service.)
Atlantic Refining Co., —No. 1 Excelsior
Economy Lubricating Co., —No. 2 Cup Grease
Fisk Bros. No. 3 Extra "K"
The Gulf Refining Co., —"High Pressure" Grease —(Satisfactory up to 180° F.)
E. F. Houghton, F18 Absorbed Oil
Lubriko Co., —M-6
Marland Refining Co., —Triangle Cup Grease Nos. 2, 3 —(No. 3 Preferred)
N. Y. & N. J. Lubricant Co., —F No. 3 Non-Fluid
Pennsylvania Lubricating Co., —Cup Grease No. 2
Refiners Oil Co., —No. 2 Cup Grease
Sinclair Refining Co.,
—No. 3 Cup Grease and Opaline Pressure System —(No. 3 preferred)

The Standard Oil Co. of Calif., —Calol grease No. 2, Calol No. 3 Cup
The Standard Oil Co. of Indiana, —Superla 57
The Standard Oil Co. of N. J., —Arctic Cup Grease No. 2
The Standard Oil Co. of N. Y., —Special C and E —(Special E preferred)
The Texas Co. —Texaco Star No. 1

These Greases have been Especially Selected because:

They are neutral.
They have no affinity for water.
They do not separate at normal temperatures.
They are stiff enough not to churn and heat up.
They are soft enough to successfully lubricate the bearings.
Their use will insure clean housings and infrequent renewals of lubricant.
They will not dry out.

Periods of lubrication depend somewhat on local conditions—loads, speeds, temperatures, etc. In average service lubrication is necessary not more than four times a year; for unusual conditions of speed or dirt this company should be consulted.

Also, in 1929, the Timken Roller Bearing Company published the following specifications for roller bearing grease and an approved list of approximately 100 acceptable greases; 14 per cent being of aluminum stearate base with practically all of the balance being manufactured with calcium soap.

APPROVED LIST OF LUBRICANTS

for Low and Medium Speed Industrial Application

The grease shall be a smooth, well-manufactured product, composed of a high grade soap and a refined, clean mineral oil. It shall be free from corrosive matter, grit, rosin, waxes, talc, mica, graphite, clay or other fillers of any kind.

Local conditions at times require that deviation be made from the normal consistency of grease used. However, the following specification and list of approved greases will serve as a guide for general lubricating conditions.

The grease shall conform to the following:

Consistency (Karns-Maag Method)	15.0 MM to 30.0 MM @ 75° F.
Ash	Maximum 2%

Corrosion—A bright copper plate *shall show no discoloration submerged in the grease for 24 hours.*

Moisture	Maximum 2%

Resistance to water; It shall not emulsify readily with water.

The oil from which the grease is compounded shall conform to the following tests:

Flash	Minimum	340° F.
Fire	Minimum	380° F.
Viscosity @ 100° F.	Saybolt Universal	200 min.
Cold Test (Pour)	Maximum	+ 30° F.

Alloil Lube Corp., Los Angeles, Calif., Alloil BHE-7, Alloil AL-E, Alloil AAL-2
Adam Cooks' Son's, Inc., New York, Albany High, Pressure Grease.
Ajax Grease Co., Chester, Pa., Ajax XX Grease, Ajax XXX Grease.
American Lubricant Inc., Buffalo, N. Y., No. 520 Grease, No. 530 Grease.
American Oil Corp., Jackson, Mich., Special Ball and Roller Bearing Grease No. 812 and No. 814.
Atlantic Refining Co., Philadelphia, Pa., Excelsior No. 1, Excelsior No. 2.
Associated Oil Co., San Francisco, Calif., Firex No. 3 Grease.
Bassick Manufacturing Co., Chicago, Ill., Alemite.
Brooks Oil Co., Cleveland, Ohio, Ajax Roller Bearing Grease.

Canfield Oil Co., Cleveland, Ohio, No. 302A Grease, No. 303A Grease.
Damacus Manufacturing Co., Cleveland, Ohio, No. 27-B Special.
Duckham & Co., Ltd., Alexander, London, England, Special Bearing Grease H.B.B.
Empire Refineries Inc., Tulsa, Okla., Roller Bearing Grease.
Enterprise Oil Co., Buffalo, N. Y., Duplex Gear Grease.
Erie Lubricant Co., Cleveland, Ohio, Elco No. 200 Gr., Elco No. 250 Gr.
Fiske Brothers Refining Co., Toledo, Ohio, Fiske Ex. K No. 3, Fiske Ex. K No. 2½.
Franklin Refining Co., Pittsburgh, Pa., Franklin No. 7, Mine Car Grease.
Galena Signal Oil Co., Franklin, Pa., Perfection Grease, No. 0 and No. 1.
Great Eastern Refining Co., Huntington, W. Va., No. 2 K Roller Bearing Grease.
Gulf Refining Co., Pittsburgh, Pa., Gulf Anti-Friction Grease, Gulf Pressure Grease, Gulf Precision Grease.
Harris & Co., A. W., Providence, R. I., No. 2 High Pressure Grease.
Houghton & Co., E. F., Philadelphia, Pa., F-18 Absorbed Oil, L-36 Absorbed Oil.
Hodson Corp., The, Chicago, Ill., Plastic Oil No. 1.
Hulburt Oil & Grease Co., Philadelphia, Pa., No. 2 Hulburt.
Hollingshead Co., R. M., Cleveland, Ohio, Whiz High Pressure Lubricant.
Industrial Oil & Grease Co., Warren, Pa., Reliance No. 3 Cup.
Imperial Oil Co., Ltd., Toronto, Ont., Can., Polarine Grease L.
Jado Laboratories, Cleveland, Ohio, Jado Grease.
Kentucky Independent Oil Co., Covington, Ky., Goldenrod Lubricator Grease.
Keystone Lubricating Co., Philadelphia, Pa., Keystone No. 2, Keystone No. 602.
Lindsay McMillan Co., Milwaukee, Wisc., Delco Industrial Grease No. 2, Delco Industrial Grease No. 3, Delco Cup Grease No. 361.
Miller Co., Frank, New York, N. Y., High Pressure Chassis Lubricant.
Miller & Son Co., A. D., NS Pittsburgh, Pa., No. 60 Mine Car Grease.
Modern Lubricating Oil Co., Columbiana, Ohio, G. and B No. 42.
National Refining Co., Cleveland, Ohio, Roller Bearing Grease No. 1, Roller Bearing Grease No. 2.
New York Lub. Oil Co., New York, N. Y., Monogram Jellux Grease.
New York & New Jersey Lub. Co., New York, N. Y., F-2 Non Fluid Oil.
Oil Kraft Inc., Cincinnati, Ohio, Medium Pressure Grease, Heavy Pressure Grease.
Ohio Grease Co., The, Loudonville, Ohio, No. 1 Cup Grease.
Panhard Oil Corp., New York, N. Y., Panhard Jellux Grease.
Panoleum Products Co., Cleveland, Ohio, P-C 50.
Pennsylvania Lub. Co., Pittsburgh, Pa., Penola No. 7.
Pennsylvania Lub. Co., Pittsburgh, Pa., Castroleum Grease A, Castroleum Grease B.
Pennzoil Company, Oil City, Pa., Pennzoil Press Lubricant.
Pheonix Oil Co., Cleveland, Ohio, Roller Bearing Grease No. 164.
Perry Refining & Mfg. Co., Chicago, Ill., No. 503 R. B. Grease.
Philadelphia Grease Mfg. Co., Philadelphia, Pa., No. 1 Roller Bearing Grease, No. 2 Roller Bearing Grease.
Pierce Petroleum Corp., St. Louis, Mo., Pennant Grease No. 4.
Pure Oil Co., Cincinnati, Ohio, Tiolene Lubricator Grease, Tiolene Super-Service Cup Grease.
Robinson & Co., Wm. C., Baltimore, Md., Autoline Roller Bearing Grease.
Roxana Petroleum Corp., St. Louis, Mo., Maximus Grease No. 23.
Sinclair Refining Co., Chicago, Ill., Opaline Pressure System Grease, Sinclair Bearing Grease "C."
Standard Oil Co., of Calif., San Francisco, Cal., Matchless Grease No. 1, Matchless Grease No. 2.
Standard Oil Co., of La., New Orleans, La., Standard Roller Bearing Grease.
Standard Oil Co., of N. J., New York, N. Y., Standard Roller Bearing Grease.
Standard Oil Co., of Ind., Chicago, Ill., Superla No. 57, Superla, No. 63.
Standard Oil Co., of Ohio, Cleveland, Ohio, No. 200 V Cup Grease.
Stevens Oil & Grease Co., A. J., Kansas City, Mo., Lucid Cup Grease No. 2, Lucid Cup Grease No. 3.
Sun Oil Co., Philadelphia, Pa., Special Roller Bearing Grease.
Swan & Finch Co., New York, N. Y., Cataract Cupese No. 52.
Sonneborn Sons, Inc., L., New York, N. Y., Amalie High Pressure Lub.
Texas Co., The, Pittsburgh, Pa., Texaco Star Grease No. 1.
Tidewater Oil Co., New York, N. Y., High Pressure Lub. Veedol Light Cup Grease.
Troco Lub. Co., The, Philadelphia, Pa., Troco No. 1.
Tiona Petroleum Co., Philadelphia, Pa., Timsin H. P. Chassis Lub.

Utah Refining Co., Salt Lake City, Utah, Visco N. C. No. 2 Cup Grease.
United Oil Mfg. Co., Erie, Pa., No. 2½ Hi-Heat Grease.
Vacuum Oil Co., New York, N. Y., Voco Grease AA No. 2.
Viscosity Oil Co., Chicago, Ill., Graigrene No. 2, Graigrene No. 3.
Wakefield & Co., C. C., New York, N. Y., No. 1 Roller Bearing Grease.
Waverly Oil Works, Pittsburgh, Pa., No. 93-H.
Warren Refining & Chemical Co., Cleveland, Ohio, Sylva Solidified Oil.

English Calcium Base Ball Bearing Greases

A suitable grease may be made by following the formula given below. Either an autoclave or an open steam jacketed kettle is required for carrying out the process:

Per Cent by Weight	
17.30	Distilled fatty acids*
2.48	Calcium hydrate
.02	NaOH
80.20	600 S.U.V./100° F. red mineral oil.

* Acid Value, 201; Iodine Value, 45; Dropping Point, 44° F., contains wool fatty acids.

The grease made on the following formula will ordinarily contain .05 per cent of free lime. After stirring and cooling to 70 to 80° C., sufficient free fatty acid is added so that by analysis about ½ per cent will be found in the finished grease. The water content is not greater than 0.7 per cent by weight and the A.S.T.M. Unworked Penetration is about 275.

Wool-Fat Alcohol, Calcium Soap Base Greases (French patent application: 376,507, Nov. 13, 1934).

This invention relates to a process for the preparation of suspensions of metal soaps in mineral oils. It consists in maintaining the metal soaps in suspension by means of small quantities of alcohols of relatively high molecular weight, such as wool fat alcohols, oleyl alcohol, and similar alcohols.

It is well known that the use of small quantities of water in the preparation of consistent greases, tends to stabilize the suspensions of the calcium soaps. However, the use of water has the disadvantage that said water evaporates easily, for example while the grease is in use, so that the state of suspension is broken.

Moreover, the use of water as a stabilizer in the preparation of suspensions having a low percentage of metal soaps in oils, such as for example the oils called "non-fluid oils," has the disadvantage that sufficiently stable suspensions are not always obtained.

It was found possible to make stable suspensions of metal soaps in oils, which stand up during use, by utilizing as stabilizer an alcohol of high molecular weight such as wool fat alcohol, oleyl alcohol and similar alcohols. By this means it is possible to completely avoid the use of water.

An example will serve to illustrate the procedure:

EXAMPLE FOR THE PREPARATION OF A "NON-FLUID OIL"

Heat in an autoclave, while stirring for half an hour and at a temperature of 170° C., a mixture composed of:

	Grams
Commercial stearic acid	100
Oleic acid	100
Mineral oil E. 50=4.5	400
Lime (70 per cent CaO)	25
Water	50 cc.

Then 50 gr. of the reaction mixture are mixed into 1.050 Kg. of said mineral oil, after which the mass is stirred until it has become homogeneous. To this product add 20 gr. of wool fat alcohols. There is then obtained a product having a viscosity E. 20 = about 70, which is very stable and which no longer leaves a soap deposit after heating.

EXAMPLE FOR MAKING A CONSISTENT GREASE WITH A CALCIUM STEARATE BASE

In this process the stabilizing effect of wool fat alcohols is indicated by the fact that it is possible to make a calcium base grease which is entirely anhydrous.

Saponify:	Grams
Stearic acid	200
Mineral oil E. 50=4.5	200
Lime (70 per cent CaO)	35
Water	50

This mixture is heated to about 170° C. in an autoclave for half an hour. Then to 50 grams of the reaction mixture is added 100 gr. of the mineral oil and 15 gr. of wool fat alcohols, after which the whole is kept at 160° C. until a homogeneous mass is obtained.

After cooling, a homogeneous grease having a melting point of 105° C. forms.

English Roller Bearing Grease

An analysis of this grease gave the following results:

Physical Tests

Appearance	Smooth, dark cup grease
Odor	Petroleum oil
Dropping point ° C.	99
Penetration	300 A.S.T.M.

Composition (Per cent by weight)

Soap	18.67
Oil	81.08
Water	None
Free alkali (as $Ca(OH)_2$)	None
Free fat	None
Free fatty acids	0.42
Gain in analysis	0.17
Total	100.00

Ash (Per cent by weight)		1.77
Calcium oxide (CaO)		96.6
Undetermined		3.4
	Total	100.00
Corrosion Tests		
Copper strip 3 hrs. at 212° F.		OK
Steel strip 3 hrs. at 212° F.		OK
Copper strip 24 hrs. at 80° F.		OK
Heat Test		
Heated 2 hrs. at 110° C.		Slight separation
Heated 10 minutes at 140° C.		No separation

Stable Ball Bearing Grease

A lime base grease which returns very nearly to its original consistency after melting at 110° C. and which shows no separation of free oil when cooled, after heating for ten minutes at 140° C., may be manufactured on the following formula:

	Per Cent by Weight
500 S.U.V. mineral lubricating oil	80.00
Wool grease distillate	17.00
Hydrated lime	1.90
Water	1.10

D'Orimont Grease

In 1912, D'Orimont obtained a British patent [64] for a process of making consistent lubricating greases with the alkaline or alkaline earth soaps of vegetable or animal oils, mineral oil, and fatty alcohols. The fatty alcohols were defined as cerylic alcohol, or cholesterin, or other alcohols which occur in wool grease. It was recognized by the inventor that raw wool grease contains considerable amounts of free fatty acids and that when a mixture of mineral oil, suet and tallow were boiled together with lime, that not only were the fats saponified but the wool grease fatty acids were also neutralized. The inventor also made use of wool grease alcohols obtained by the treatment of raw wool fat with caustic soda, in a blend of mineral oil and lime soap together with vegetable or animal oils. In carrying out the process the following proportions were preferred:

	Parts
Wool fat alcohols	5
Tallow	5
Lime soap	10
(This mixture was heated to 130° C., washed, decanted, and filtered.)	
Mineral oil	105

Hulsberg Process

The Hulsberg process has already been discussed in the section on lime base cup greases. It will be recalled that this inventor preferred to utilize

[64] British Patent 2,382 (Jan. 30, 1912).

wool fat stearin in combination with lime tallow soap and mineral oil to obtain a stable lubricant.

Ekenberg Process

In 1899, Ekenberg obtained an American patent [65] for his method of manufacturing an anhydrous, transparent lubricating grease from mineral oil and wool fat soaps. Ekenberg was quite aware of the necessity of hydrating the lime soaps normally used in lime base cup greases. In this process fatty acids from wool fat were dissolved in mineral oil and the solution heated to a temperature between 120 and 200° C., when the required amount of lime or magnesia was added to neutralize the fatty acids. Ekenberg pointed out that if glycerides were present that an inferior product from the standpoint of transparency would be obtained. To produce a grease of medium consistency a formula calling for fifteen per cent by weight was recommended.

To produce the fatty acids from wool fat, Ekenberg saponified the raw fat in an autoclave at temperatures of 200 to 300° C. with strong caustic soda or potash. He then added sulfuric acid to spilt the soaps and liberate the fatty acids.

Fatty Acid Ball Bearing Grease

As the result of work completed in 1925, the author published in 1927 [66] the formula for a grease made with a mixture of stearic and oleic acids, in which the oleic acid predominated, and a slight deficiency (2 per cent less than the theoretical quantity required for neutralization equivalent to 0.18 per cent free fatty acid) of alkali to obtain a grease which would not separate on dehydration.

In 1928, Lange and Mason [67] applied for a patent for fatty acid greases having a free fatty acid content not greater than about 0.5 per cent. These inventors found that by making a calcium soap of fatty acids having molecular weights greater than that of stearic acid (284) and dispersing such soap in mineral oil that a transparent grease could be obtained, and further mineral oil added to produce any desired consistency. The inventors employed a quantity of lime insufficient to completely neutralize the fatty acids in order to manufacture a stable grease. They preferred to maintain the free fatty acid content in the neighborhood of 0.1 per cent as they considered that the lower the free acid content, the higher the melting point of the grease. Stearic acid, and fatty acids from wool wax, carnauba wax, and Chinese vegetable tallow were recommended for use, either alone or in combination with animal and vegetable fatty acids containing appreciable quantities of oleic acid. In carrying out the process, the fatty acids are mixed with a small quantity of mineral oil and heated to a temperature of about 200° F., when either powdered hydrated lime or milk of lime

[65] U. S. Patent 620,899 (Mar. 14, 1899).

[66] Klemgard, E. N., "Lubricating Greases," 1st. Ed., 36, The Chemical Catalog Co., Inc. (Reinhold Publishing Corp.), 1927.

[67] U. S. Patent 1,920,202 (Aug. 1, 1933).

may be added and heating continued until practically all of the water is evaporated from the batch, and neutralization of the fatty acids effected, leaving a small amount of free fatty acids as previously referred to. Since the finished grease contains no water, it is claimed that it may be heated to temperatures of 400 to 500° F., and when cooled returns to its original condition.

It is possible that heat stable greases may also be obtained by manufacturing a grease with slight excesses of lime and later adding wool fat, wool grease fatty acids, or other fatty acids.

Hodson [68] has carried out experiments with oil-miscible soap solvents which appear to improve the heat stability of cup greases made with the lime soaps of high molecular weight fatty acids.

Experimental Heat Stable Ball Bearing Grease

Formula

	Per Cent
Sardine oil	10.0
Stearic acid	2.0
Hydrated lime	2.0
Caustic soda	1.0
Aluminum base pressure gun grease	10.0
Lead oleate	5.0
Carnauba wax	5.0
Degras	4.0
300 western neutral	51.0
	100.0

Method of Preparation

The sardine oil and stearic acid may be saponified by heating with hydrated lime and water in an open vessel. The 300 Western neutral oil is added to the lime soap. The Aluminum base gun grease, lead oleate, carnauba wax, and degras are then added. A fluid product results. It seems probable that the free fatty acids from the carnauba wax and degras are responsible for the fluid condition of the product. 45° Bé. caustic soda is therefore added to neutralize the fatty acids. Some difficulty with foaming may be experienced at this point. The final product is heated to 250° F. and allowed to cool.

Tests on Grease

Ubbelohde, dropping point, ° C.	76
Water, per cent	0.6
Unworked penetration	278
Worked penetration	311
Copper strip corrosion test, 24 hours, laboratory temperature	OK

General Properties

Texture. Similar to cup grease.

Heat Stability. The product can be melted by heating to 250° F. When cooled, the grease will return to its original consistency with no

[68] U. S. Patent 1,982,682 (Dec. 4, 1934).

separation of oil. Under similar conditions, cup greases will show extensive separation of oil.

Water Resistance. If a thin layer of this grease is placed in a shallow dish, and sufficient water poured over the grease to cover the surface, it will be found at the end of twenty-four hours that the surface of the grease will be clear on portions from which the water has evaporated.

Structure Changes. Unlike many sodium and aluminum greases, the product does not assume a rubbery texture at high temperatures.

DISADVANTAGES OF THIS GREASE

These are as follows:

Faintly granular appearance.

Dark color detracts from the appearance of the grease. This difficulty could probably be avoided by using more highly refined raw materials, particularly carnauba wax, degras and oleic acid (used in the preparation of lead oleate).

The dropping point is slightly lower than for cup grease of the same consistency.

It is possible that some increased tendency to gum in service might result from the use of fish oil soap, lead oleate and degras. The use of more saturated calcium soaps would tend to reduce gumming. However, the more saturated soaps apparently yield granular products which break down excessively upon working.

It is possible that some increase in wear might result from the use of lead soap. This disadvantage would be more or less counterbalanced by an improved resistance to scoring due to an improved film strength.

High cost as compared with cup grease. The carnauba wax would in itself contribute to an increased cost of about 1.5c per pound.

One large distributor of lubricating greases has published the chart on page 229, from which it is plain where lime base and sodium base greases may best be used as well as oil. The reasons for these selections will be apparent on reading the general discussion of the status of ball and roller bearing lubrication.

General Discussion of Ball Bearing Lubrication

The function of all bearings is to support rotating shafts and to preserve their proper alignment; at the same time they must allow the shafts freedom to revolve. Means are effected, through lubrication, to protect the bearings and moving parts against wear and to reduce the frictional forces produced by the relative motion between metal surfaces in contact.

The ordinary bearing consists of a supporting surface usually of plain cylindrical form which encloses or partly encloses the revolving shaft or journal. The journal rubs against this surface causing metallic contact unless a film of oil is maintained between it and the journal. The interposing and maintenance of such a film is the function of lubrication and it should result in the substitution of metallic friction by fluid friction.

In the case of ball and roller bearings, or so-called "anti-friction bearings," the journal is separated from the bearing surface by balls or rollers which roll round the journal when the latter rotates. These balls or rollers are enclosed between two cylindrical surfaces called "races" (one being fixed rigidly to the journal, and the other being fixed similarly to the bearing housing) and they are held in position and separated from one

TABLE 5.—Ball Bearing Lubricant Recommendations

Speed R.P.M.	Bearing Series	S.A.E. Bearing Bore Size — Grease	S.A.E. Bearing Bore Size — Oil	Operating Temperature — Below 32° F.		32° to 150° F. — Moisture Present	32° to 150° F. — No Moisture	150° to 200° F. — Moisture Present	150° to 200° F. — No Moisture
Up to 500	Light	0 to 22	All Sizes	Oil or Special Grease	Oil	Pale Oil	Pale Oil	Pale Oil	Pale Oil
	Med.	0 to 22			Grease	Cup Grease No. 0	Fiber Grease* No. 1		Fiber Grease No. 2
500 to 2000	Light	0 to 18			Oil	Pale Oil	Pale Oil	Pale Oil	Pale Oil
	Med.	0 to 16			Grease	Cup Grease No. 0	Fiber Grease No. 1		Fiber Grease No. 2
2000 to 5000	Light	0 to 10			Oil	Pale Oil	Pale Oil	Pale Oil	Pale Oil
	Med.	0 to 9			Grease	Cup Grease No. 0	Fiber Grease No. 1		Fiber Grease No. 2
5000 to 10,000	Light	0 to 6			Oil	Pale Oil	Pale Oil	Pale Oil	Pale Oil
	Med.	0 to 5			Grease		Fiber Grease No. 1		Fiber Grease No. 2
Above 10,000	Light	0 to 4			Oil	Pale Oil	Pale Oil	Pale Oil	Pale Oil
	Med.	0 to 3			Grease		Fiber Grease No. 1		Fiber Grease No. 2

* All "Fiber Greases" in above table refer to "short fiber" or relatively smooth sodium soap greases.

another by a suitable cage which is free to revolve with them round the shaft.

The ball and roller bearing operates on a fundamentally different principle from the plain bearing; in the latter, there is sliding motion between the journal and the bearing, whilst in the former there is rolling contact between the balls or rollers and the races, one race revolving with the journal and the other being stationary. Theoretically, ball bearings involve point contact only and roller bearings involve line contact only, whilst in the case of plain bearings, relatively large surfaces are in sliding contact all the time. In practice, of course, where lubrication is adequate, the sliding contact between metallic surfaces is replaced by sliding contact between fluid surfaces. Apart from this, however, it is evident that the frictional forces in the two types of bearing are quite different in character and vary considerably in extent. Generally speaking, work expended in overcoming friction is very much reduced by the use of ball or roller bearings.

The frictional resistance in the case of a plain bearing is several times greater at the start than after a few revolutions when the oil film is established, whereas in ball and roller bearings the starting frictional resistance is only very little more than the running friction. Moreover, the co-efficient of friction is less than in the case of plain bearings and it varies to a less extent with changes of load and speed.

Roller Bearings. Rollers may be cylindrical, barrel-shaped or conical, the latter are generally referred to as tapered rollers. They are generally solid but there is a hollow type made by The Hyatt Roller Bearing Co. consisting of hollow cylinders of helically wound strips of steel. The cage prevents the rollers from rubbing against each other but there is relative motion between the cage and the rollers by virtue of the fact that the latter also rotate round their axes. The relative motion gives rise to frictional forces at points where the rollers and cage touch each other. Moreover, in order to keep the rollers in proper alignment with the races, the latter are sometimes provided with suitable flanges against which the ends of the rollers slide; thus, forces are set up at the ends of the rollers as well as over the rolling surfaces.

In the case of non-cylindrical rollers, end-thrust is generally set up during operation. This may tend to force the rollers against one end of the cage and in turn to force the cage against the end of the bearing housing, causing additional rubbing surfaces. The same effect is often created in cylindrical roller bearings due to the rollers not being perfectly cylindrical or not being in perfect alignment with the journal. End-thrust may be considerable and the resulting friction may contribute largely towards the total friction.

It should be noted that aggravated end-thrust is often the cause of excessive wear, hot bearings and vibration or rumbling in the bearing housing. Modern roller bearings are generally so designed that end-thrust is small.

Generally speaking, the starting effort is only slightly greater than the

running effort, but it may be twice as great where there is excessive end-thrust. That the starting effort is actually somewhat greater than the running effort will be apparent when it is realized that, generally speaking, there is no lubricating film between the rollers and the races when the journal is at rest. This point will be clearer when the lubrication of roller bearings is discussed.

Ball Bearings. As in the case of roller bearings, the balls and cage rub against each other at different relative speeds giving rise to frictional forces. This effect is only appreciable, however, at those parts of the cage which separate the balls from one another, for the balls touch the sides of the cage at the poles of their axes of rotation and there is no relative motion at these points, whilst in the case of roller bearings, the ends of the rollers touch the sides of the cage in areas of appreciable size. Moreover, the balls touch the cage at points only, whereas rollers touch the cage along lines. The areas over which the frictional forces act are, therefore, greater in the case of roller bearings.

Apart from the consideration of end-thrust, it is apparent from the above that more energy will be dissipated due to frictional forces in roller bearings than in ball bearings.

End-thrust cannot exist in ball bearings, except to a very small extent in some self-aligning types where the races are not flat, or in types adapted for heavy stresses where the races are grooved and more than two-point contact is involved. In the latter case a certain amount of grinding occurs between the balls and races during operation.

It is this absence of end-thrust which gives ball bearings such a great advantage over roller bearings, as the resultant frictional resistance is, as a rule, very much less and the bearings are consequently less inclined to heat.

On the other hand, roller bearings will withstand much greater pressures than ball bearings and it is in the field of heavy machinery where roller bearings find their widest application.

Lubrication of Ball and Roller Bearings. It is sometimes said that from the point of view of reducing friction, ball and roller bearings need no lubrication, and it has certainly been shown in certain ideal cases with new bearings that frictional resistance, and therefore power loss, remains very much the same whether a lubricant is used or not. However, in all machinery involving relative motion between metal surfaces in contact, it is absolutely essential to interpose a suitable lubricating medium between these surfaces in order to preserve them, to prevent wear and to ensure smooth running. Experience has shown that ball and roller bearings are no exception to this generalization; if a lubricant is not employed, such bearings invariably run noisily and are subject to rapid deterioration and wear. The reasons for this are as follows:

In practice, pure rolling motion and point or line contact are not realized. The moving elements and races are elastic to a certain extent and in consequence they are slightly deformed at the points of contact due to the temporary stresses to which they are subject. The result is that point and line contact are substituted by surface

contact. Frictional forces are set up as these surfaces move against one another and to reduce these forces a lubricating film must be interposed between the surfaces.

The moving elements must be allowed a certain amount of play with the result that when the journal is rotating the moving elements rebound at high speed against the cage and races. If a lubricant is not employed to provide a cushioning effect these impacts give rise to a continuous ringing sound.

There is relative motion between the rolling elements and the cage and between the cage and the housing. This involves surfaces moving against one another and consequent friction. A lubricating film must be interposed between these surfaces to prevent actual contact.

In cases where there is end-thrust a lubricant is obviously necessary.

The surfaces of the moving elements and races are very liable to become corroded by the atmosphere unless they are protected by a suitable rust preventing medium. A very slight amount of corrosion gives rise to pitting and once started it develops rapidly throughout. Pitted surfaces, by reason of the continued impact, give rise to serious wear and disintegration of the rolling elements and cage. It is, therefore, essential to protect the surfaces from rust and to maintain them in a highly polished condition.

No matter how tight the housing is made, a certain amount of abrasive dust may enter the bearing and give rise to scoring. The presence of a lubricant helps to improve the seal and to prevent the entry of foreign matter.

Either greases or oils are used for the lubrication of ball and roller bearings. Generally speaking, oils are considered preferable to greases because they are more mobile and consequently they do not offer so much resistance to the motion of the moving parts. Friction is therefore, in general, greater when a grease is used. Oils should also be given preference in cases of abnormally high surrounding temperatures as they are less susceptible than greases to deterioration by heat. In certain cases, however, it is not practicable to use oils. This may be due to any of the following reasons:

Inaccessibility of the bearings.

Dusty or dirty surroundings. Grease provides a most effective seal against the entry of foreign bodies by forming a fillet at each end of the bearing and in this respect is much superior to oil. Oil can only be used in such circumstances when very efficient dust guards are fitted.

Housings not oil-tight. Oil tends to be thrown out of the housings by centrifugal force; in some cases it is impracticable to use oil-tight housings and a grease must therefore be used.

Space limitations. If there is not enough space available to allow of a housing with ample closures and adequate oil reservoir, grease should be used.

Position of bearings—grease should be given preference if the bearing housing is in an inclined position.

It should be pointed out that at temperatures above 300° F. an oil must generally be used as it is not possible to manufacture normal lime base greases which do not separate into their constituents at these temperatures. A stiff sponge grease such as a No. 4 may be used in some cases up to temperatures as high as 400° F., where it is not possible to use an oil because of the housings not being oil-tight.

It is also advisable to recommend a light, low cold-test oil when surrounding temperatures are consistently low.

Oil Lubrication

There is one important difference between plain bearings and ball or roller bearings as regards oil lubrication. In a plain bearing, there is

invariably appreciable friction due to the shearing of the lubricating film; viscous oils build specifically thicker films than thin oils, and the resistance to shear of a thick film is less than that of a thin film. Furthermore, the viscosity of the oil must be sufficient to prevent it from leaking away from the bearing faster than it is supplied.

These factors govern the limits of viscosity of oils for plain bearings. In ball or roller bearings, friction due to shearing of the film only exists to a very slight extent as the principal motion is rolling instead of sliding. Moreover, the oil leakage is governed only by the tightness of the housing. Hence, generally speaking, lighter oils may be recommended than for plain bearings involving the same journal speeds and loads.

Grease Lubrication

As indicated above, in humid and dirty surroundings, grease is to be preferred to oil and also in cases where oil is inclined to leak out of the bearing. Some manufacturers stipulate that oil should be used in all cases where speeds are above 1000 R.P.M., whilst others show a general preference for grease even up to speeds of 20,000 R.P.M. It is, therefore, impossible to give a ruling on the subject. Generally speaking, oils should be given preference where it is practicable to use them because of the less resistance which they offer to the movement of the rolling elements. It may be borne in mind, however, that in some bearings operating at very high speeds, it may not be practicable to provide the bearing with adequate sealing devices and in such cases a grease must be used.

Grease should never be used where operating temperatures are above 400° F., and not generally above 300° F., as mentioned previously.

Ball and roller bearing greases must not contain any chemical constituents likely to corrode the moving elements causing pitting or roughening of the surfaces. They must also be free from filling matter which would exert an abrasive action on such surfaces. So-called non-abrasive fillers, such as mica or graphite, should also be avoided as the filling matter tends to separate from the grease and build up layers which eventually interfere with the movement of the rolling elements. Moreover, such material cannot flow from beneath the balls or rollers with the result that the latter skid and excessive wear takes place. Water content should be as small as possible so as not to encourage the formation of rust—the small traces of water necessary for the formation of a grease structure are of course permissible. Further, they must not tend to separate into their constituents during use or give rise to hard deposits through caking.

The most suitable type of grease to employ depends on the temperature, speed and load.

A lime base grease of a soft consistency and relatively low melting point is recommended where temperatures, speeds and pressure conditions are not excessive, *e. g.,* ordinary line shafting bearings. Grease filled bearings operating at normal speeds (up to 2000 R.P.M.) and under normal loads usually run at 30 to 35° F. above the atmospheric temperature, although the form of the housing has a decided influence on the ultimate running

temperature as it is a controlling factor in the amount of heat radiated from the bearing. Generally speaking, normal lime base greases of a consistency suitable for ball or roller bearings should not be used above 120° F., as at higher temperatures they become too soft to provide an adequate seal. In some cases, of course, where very efficient seals are fitted it will be possible to use this type of grease up to somewhat higher temperatures without any trouble through leakage. Where loads and pressures are small and temperatures normal as for example in bicycles, laboratory instruments

Recommendations

Service	Temperature	Grade	Examples of Application
Normal loads and speeds (not above 2000 R.P.M.)	Up to 120° F.	(See English Cup Grease No. 4)	Axle bearings of automobiles, tractors, road machinery, agricultural machinery, industrial trucks, mine cars, etc. Conveying and handling equipment. Cranes. Electric motors (small types only). Grinding mills (Small, roll type). Hoisting drums. Line shafting. Pillow blocks. Pulley wheels. Pulverizers. Sheave wheels. Trolley wheels. Winches.
Light loads and high speeds.	Up to 170° F.	Short Fiber Grease	Small electric motors. Small fans. Vacuum cleaners. Vertical spindles of high speed drilling machines. Woodworking machinery.
Normal loads and high speeds	Up to 200° F.	Soda Soap Grease	Artificial silk centrifugal separators. Axle bearings of tramcars, trains, industrial trucks, etc.
Heavy loads and all speeds.			Armature bearings of electric generators, stationary electric motors and electric traction motors. Blowers. Boot and shoe making machinery. Conveying and handling equipment. Disintegrators. Exhaust fans. Grinding mills (roll type). Line shafting. Machine tools. Mixing rolls. Paper machinery. Printing machinery. Pulverizers. Rotary blowers. Sawing machinery. Woodworking machinery.
Normal loads and high speeds. Heavy loads and all speeds. Initially heated bearings.	Up to 300° F.	Heavy Soda Grease	Same applications as above, where a stiffer grease is required to give an efficient seal. Also for initially heated bearings, *e. g.*, paper machines.

and sewing machines, a pure petrolatum is recommended by some firms, but a light grease is always to be preferred. Petrolatum should not be offered unless specifically demanded, as it is poor in lubricating properties and has an exceedingly low melting point (*ca.* 120° F.).

Where temperatures, loads or speeds are high, special greases with high melting points are necessary. Such greases must have the property of retaining their physical structure when in the neighborhood of their

melting points or after successive heating and cooling and in spite of the violent agitation to which they are subjected at high speeds. They must also have the property of not "creeping" at such temperatures, *i.e.*, their consistency must be such that they will not ooze out of the bearing housing. Armature bearings of electric traction motors and large stationary motors and railway axle roller bearings are examples of cases where these special greases are required. The mechanical stresses experienced in these particular cases tend to set up considerable heating effects and, unless the correct type of grease is used, very considerable wear will take place.

Inspection and Refilling. Regarding frequency of inspection and refilling, it is difficult to generalize as this depends on the service, the working conditions and the size of the oil or grease reservoirs provided in the housing. It is recommended, however, that bearings running under normal conditions should be inspected and refilled every three months. More frequent inspection is advisable if housings are leaky or if bearings operate under high temperature conditions, as in such cases, consumption is likely to be greater than under normal conditions. The effect of heat will also tend to cause the oil or grease to deteriorate. The same advice applies to cases where the bearings are exposed to dust, dirt or moisture; in such cases frequent renewal of the lubricant is an obvious precaution.

It is always advisable to clean out the bearings when the lubricant is changed. It is usual to flush the bearings with a thin hot oil. When changing from a lime base grease to a soda base grease, or vice versa, it is particularly important to clean the bearing thoroughly so as to remove all traces of the old grease; the reason for this is that the presence of a small quantity of grease of a different base tends to disturb the chemical equilibrium of the grease in use, often resulting in decomposition. In the case of new bearings, this flushing is extremely important as it is necessary to remove metallic particles which may have been produced during the running-in period.

Regarding the filling of oil lubricated bearings, some authorities consider that the bearings should be filled so that the lowest ball or roller is just covered, whereas others consider that the oil level should be below the center of the lowest rolling element. In any case the level should not be higher than the top of the lowest rolling element. Too much oil will lead to high temperatures due to churning. Oil is not used as a cooling medium as in the case of plain bearings and hence a minimum quantity can be used.

In the case of grease lubricated bearings it is usual to fill the space initially half full with grease. It should be emphasised that too much grease should not be forced into the bearing. Overfilling is a common cause of hot bearings. When bearings run hot it is a common mistake to force more grease into the bearing; instead of reducing the temperature, this tends to aggravate the trouble, the extra quantity of grease merely causing greater resistance to the movement of the rolling elements, increasing the frictional forces and consequently leading to still higher bearing temperatures. If the temperature runs high, it will be frequently found that reducing the amount of grease in the bearing will cause the temperature to return to normal.

Investigation of Bearing Condition. An exact opinion as to bearing conditions can only be obtained by dismantling the bearing. It is often possible to diagnose trouble by studying the noise of the bearing during running. The following points will give a rough idea of defects which give rise to abnormal noises:

A regular or irregular scraping noise points to the presence of foreign bodies such as metal chips, sand or dirt.

A regular grinding noise indicates that the balls or rollers are cracked or so jammed that free movement about their axes is prevented. An irregular grinding noise usually indicates that the cage is grazing other parts of the bearing.

A jolting noise indicates crumbling of the surfaces of the races or rolling bodies or untrueness of the latter. It may also be due to hard resinous material adhering to the rolling bodies if a poor quality of lubricant has been used.

A regular clear metallic ringing sometimes verging on whistling indicates dryness of the moving bodies and races, hence deficient lubrication.

Rattling, alternately strong and weak, indicates a ball or roller too loose or that the cage has too much play.

A regular humming sound indicates that the bearing is running in order.

Ball Bearing Grease Requirements. Greases contain, as we have seen, more or less combined water. If certain temperatures are met with, this condition will cause the soap and hydrocarbon oil permanently to separate. The soap is valueless for purposes of ball bearing lubrication. It will clog up part of the surfaces of the races and balls and actually prohibit their lubrication by the free oil, providing the latter has not already run out of the bearing housing, which usually is none too oil tight if designed for grease. This separation of the grease of a cheaper quality often takes place at 115° to 125° F. For ordinary conditions of bearing lubrication, a grease should not separate at a temperature lower than 175° or 200° F.

Experience shows that the general requirements for a suitable grease for ball bearing lubrication are, according to one well known bearing manufacturer:

Chemical neutrality.
Absence of corrosive or abrasive matter.
Unchanged consistency for the speed and temperature conditions of the installation.
Low internal friction.
Long life, that is, the original properties of the grease should be maintained for a long period of time and the grease should not harden or thicken in service.

Specifications for a suitable ball bearing lubricating grease should meet the following requirements as given by a large ball bearing manufacturer:

Free acid (calculated as oleic) maximum 0.10 per cent;
Free alkali (calculated as sodium hydroxide) maximum 0.10 per cent;
Free lime (calculated as calcium hydroxide) maximum 0.50 per cent;
Neutral saponifiable oil, maximum 1.10 per cent;
Viscosity of mineral oil (minimum 200 sec. Saybolt Universal 100° F.) and abrasive particles, sand, etc., absent.

Unfortunately, the appearance of a grease does not permit any definite conclusion to be drawn as to its quality, and it should be carefully tested

for its lubricating qualities and presence of free alkali, in addition to testing it for acid.

An easy way of testing grease for the presence of acid or free alkali is to cover a highly polished steel surface with the grease and expose it to the sunlight for about two weeks. If the grease contains either one of these injurious elements in sufficient quantity the surface of the steel will show etchings or pit marks.

Graphite as an Aid to Ball Bearing Lubrication. Graphite, if very finely deflocculated and added in proper proportion to a grease or oil, is an excellent lubricant in that it forms a tough and enduring film over the metallic surfaces. For this reason, it is highly recommended for the lubrication of gears and some forms of sliding bearings, as it fills in the interstices in the bearing surfaces and allows the true lubricant to operate efficiently. As applied to ball bearing lubrication, however, its value is at least doubtful and quite often decidedly detrimental results have been obtained. In spite of its unctuous qualities, it cannot be considered as a beneficial lubricant for ball bearings, and some graphite greases and compounds marketed today for the purpose of combined gear and ball bearing lubrication are far from giving uniformly satisfactory results. The principal difficulty seems to be that of obtaining pure graphite, free from foreign matter and abrasives, and of dividing it into fine enough particles and then of blending it in the right proportion with oil or grease. Because of the difference in the action between a ball bearing and plain bearing, as between rolling and sliding, graphite has a tendency to pack between the balls and retainer and in the grooves. Moreover, graphite is a lapping compound, and tends to wear the ball and groove surfaces.

Ball bearing manufacturers have made repeated tests with graphite lubricants, and results have been far from uniform. Some found that the graphite seems to act as an abrasive, causing wear of the ball races. This may be due to inherent abrasive impurities contained in the graphite. Others have found it to be quite satisfactory, or at least that it has no detrimental effects.

In view of these varying experiences, it does not seem advisable to use graphite as an aid in ball bearing lubrication, much as it is to be desired that ball bearings should benefit from its peculiar action in the same manner as do gears. Its use in ball bearing-equipped machinery is advisable only if the increased efficiency and life of the other wearing parts subjected to graphite lubrication offset the possible harmful effects of such lubrication on the ball bearings.

Where the economical benefits, resulting from graphitic lubrication of machinery in conjunction with which ball bearings are used, dictates the use of such lubricant, the following specifications for such lubricants should be rigidly adhered to:

> Lubricant specified should contain not more than 4 per cent, by weight, of lubricating mined flake graphite having a carbon content of not less than 98 per cent and passing through a No. 170 mesh screen, such graphite mixed in the proper manner with suitable base lubricant insuring correct proportion and even distribution of the graphite.

S.K.F. Requirements for Ball Bearing Greases. Several years ago the S.K.F. Industries, Inc., published the following list of specifications for ball bearing grease, and they have been considered as standard requirements for some time. However, in view of recent developments, other requirements are now insisted on by bearing manufacturers, as will be discussed in the remainder of the section on ball bearing greases.

They must not separate on standing or on heating below the melting point.
They must not gum or become sticky.
They must not harden or decompose.
They must not corrode any type of bearing.
There must not be any resin, mineral salts, or abrasives, such as sand, free lime, etc.
The consistency should be about No. 2 or No. 3.
Free fatty acid per 1 gram grease not to exceed the equivalent of 0.3 mg. KOH, per 1 gram grease.
The same for free alkali.
Free saponifiable oil not to exceed ½ per cent.

Marlin-Rockwell Requirements for Ball Bearing Greases.[69] The following requirements of ball bearing greases have been summarized by Ahlberg:

They should be free and clean from all injurious foreign matter, abrasives and fillers of any kind.
They should be free from acid or corrosive matter.
Each lot should be of uniform consistency, and consistency should be uniform from one lot to another.
There should be no free oil.
They should be non-separating during standing or while in operation.
There should be no leakage and minimum thinning during operation.
Minimum volume increase during service.
They should be non-oxidizing with no tendency to dry or gum.
They should be of a butter or vaseline consistency.
Non-hydroscopic.
They should have low internal friction.
They should be low in water content.
They should be of stable character.

Agitation Test

The Marlin-Rockwell practice on receiving a sample of grease for examination is first, to obtain a melting point; secondly, determine the kind of soap, and whether or not a filler is present. If no fillers are present, they then conduct an agitation test. The advantage of this test lies in the fact that in a very short time, results are obtained which indicate what may be expected of a grease over a longer period of actual service. It brings to light characteristics in certain lubricants which may be considered objectionable. From the results of this test they are able to tell whether or not there is a separation of oil from the soap base, either permanent or temporary, whether there are changes in consistency when cooled after testing, whether or not there are any changes in structure, and in some instances, changes of color due to airification. Airification generally produces an increase in the volume of greases.

[69] Ahlberg, C. R., "Ball Bearing Lubrication and the Investigation of the Decomposition of Ball Bearing Greases," presented at: Third Convention of the Lubricating Grease Manufacturers Association, (Oct. 14, 1935).

If the grease passes the agitation test, the following analyses are then made: Ash, per cent of Soaps, Viscosity of Mineral Oil, per cent of Free Acid or Alkali, and Fillers, etc.

Should the analysis of the grease meet their approval, they then conduct an oxidation test. This test consists of filling a bearing approximately one-half full of grease, wrapping in wax paper and placing in a cardboard carton. It is then examined every three (3) months for signs of hardening and gumming. A grease must show no signs of change for at least a period of one year and six months to meet with their requirements.

Investigation of Decomposition of Greases in Ball Bearings. Many theories have been used to explain the different stages in the decomposition of greases and unfortunately these theories are by no means in harmony. The following quotations are the answers to questionnaires sent to several leading lubricant engineers and chemists by the Marlin-Rockwell Company:

Explanation of Reaction causing Decomposition of Greases during Storage.

A. "We know of no definite research on the stability of greases which will answer this question. Our own laboratory observations, however, lead us to believe that the problem is largely one of oxidation with the resulting formation of products of a sticky or gumming nature, which cause trouble in packed ball bearings. The reaction is probably in many ways similar to that of the hardening of drying oils commonly used in paints, although oils of the drying type are far too unstable to be used in greases, especially those intended for ball bearings."

B. "Very little is known concerning the nature of the reaction that takes place during the decomposition of greases when packed in ball bearings and placed in storage. It appears to be clear, however, that the reaction results in the production of organic acids which are corrosive, and in a change in the state of dispersion of the soap in the grease, the latter effect doubtless being occasioned by the former."

C. "Our explanation of the reaction which takes place when greases are spread in thin films on highly polished steel bearings in storage for long periods of time is that grease in this condition favors oxidation due to the relatively large area exposed to the air. Both the saponified fat or soap and the mineral oil are subject to such oxidation. Naturally, soap stocks of a highly unsaturated nature are more subject to this oxidation than stearates or saturated fatty acid radicals. Most all lubricating greases contain small amounts of free fat which have escaped sponification even though the grease may contain appreciable quantities of free alkali. This free fat is also subject to decomposition and consequent formation of free fatty acid. The amount and distribution of the free alkali in the grease is an important factor in preventing fatty acid formation. In some greases the free alkali exists in small highly concentrated particles or nuclei and is not available for reaction with free fatty acids, as they are formed by decomposition."

Effect of Bacteria, Enzymes and Oxygen.

A. "Of these three possibilities, we are inclined to believe, as stated under (1), that oxygen is the most important factor concerned with the decomposition of greases. It is doubtful whether bacteria or enzymes play any role in such decomposition, although we must admit that we have no definite proof."

B. "It is definitely known that oxygen causes this decomposition. It is probable that bacteria and enzymes also effect the decomposition but the evidence for this is slight."

C. "We believe that oxygen and bacteria are important factors effecting the deterioration of lubricating greases. We are not at all certain that enzymes are the activators which promote appreciable oxidation as it is our impression that this class of product ordinarily associated with living animal and vegetable matters is quite sensitive to changes in environment, and that their presence in a grease would render them inactive. Since as yet enzymes have not been isolated, we hesitate to blame them for any particular difficulties encountered with lubricating greases." (Klemgard)

Hydrolysis of Greases.

A. "It is to be doubted whether hydrolysis of the grease has any significance as far as stability against decomposition is concerned. This is especially true where the grease contains a slight excess of alkali which would tend to decrease the tendency towards hydrolysis that might be encountered with a neutral or slightly acid product."

B. "In the decomposition of grease the hydrolysis of the soap causes a change in the state of dispersion of the soap. However, this can hardly be regarded as a fundamental effect but is rather the consequence of the formation of acids."

C. "We do not think that hydrolysis is an important factor in grease decomposition. It is, of course, admitted that certain types of soda base greases, particularly those containing sodium or potassium resinate, are definitely hydroscopic in that they will take up appreciable quantities of moisture from the air. This process can probably proceed far enough so that such moisture will be brought into contact with the metal surface and thus promote rusting and deterioration of the metal surface. We do not, however, have definite and tangible proof of this latter point." (Klemgard)

There is experimental proof that oxidation of oils begins by the direct attachment of oxygen molecules to oil molecules. The additional compounds in this case are most probably peroxides. Since these peroxides are chemically active, they act as catalyst and promote oxidation of substances at ordinary temperatures which would otherwise require high temperatures for their decomposition. Therefore, it is obvious that an improperly finished oil containing even a small amount of oxidizable substance, becomes a suitable catalyst for the decomposition of greases even at low temperatures.

As an illustration of this fact a case is cited in which experiment was made with a light naphthenic coastal oil for washing and cleaning bearings. These bearings were wrapped in waxed paper with some of this light naphthenic oil remaining on them, and packed in cardboard cartons for a period of approximately three (3) months with no other slushing compound being used. After a period of three months the bearings were found in a semi-locked condition. Apparently, the oil used for washing and cleaning these bearings collected at the foci of the ball with the races due to capillary action and oxidized, forming a gummy, tenacious product which acted as an adhesive that caused the bearings to run rough and in some cases become locked.

Perhaps electrolysis and hydrolysis may not be among the important factors in the decomposition of greases. Nevertheless, there are cases in which oxidized greases contained large amounts of iron, and it is a well known fact that iron is a good catalyst. Since the total acidity in some cases of the badly oxidized greases was relatively low, and not active enough to cause corrosion or etching to the bearing members, the presence of iron can only be explained by the action of electrolysis which was favored by the presence of water or moisture, or as an impurity in the raw grease making materials.

It is known that recently experiments have been performed with antioxidants in the retarding of the oxidation of fats, but we have as yet seen no information on inhibitors used for retarding oxidation of mineral oils. Some of the inhibitors now being used for fatty oils, etc., are:

"Hydroquinone, Alpha-Naphthol, Arcinol, Thymohydroquinone, Guai-

aco, Resorcinol, and Fractions of the unsaponifiable liquids, of wheat germ oil and palm oil."*

There is no question but that this decomposition of grease is materially reduced by using a stable mineral oil and a soap of good quality fat which has been properly blended, with due attention being given to the heating, cooling, and mixing of the ingredients, so as to form the most stable equilibrium of the emulsion. Inhibitors no doubt tend to prevent decomposition, but not a sufficient length of time has passed to correctly judge their efficiency.

In the selection of a fat used in making soaps, the simpler the composition, provided it is a fat that contains all saturated compounds, the less trouble it is liable to cause in making a high grade grease. Therefore, fats of the stearic and palmatic acid groups form the most stable soaps, provided they are of good quality and well saponified. It goes without saying that a low grade impure tallow which consists mostly of palmatic and stearic acid glycerides does not make a satisfactory soap, although it contains saturated fatty acids, because of the small amount of oxidizable impurities which these low grade tallows contain.

Experiments were made on the bacteria and enzymes content of both sodium and calcium soap greases. These greases contained from 15.0 to 20.0 per cent of soap and were compounded with an oil of a viscosity of approximately 300" S.U. at 100° F. These new greases, which had been exposed to atmospheric conditions at room temperature for approximately three weeks, were found to contain no indications of the presence of enzymes. The method used for the determination of enzymes was that described in U. S. Department of Agriculture, Bureau of Soils—Bulletin No. 36 (1909). The bacteria were determined by smearing a small amount of the sample of greases on a standard gelatin culture media and incubating at 20° C. for a period of a week. Another method for determining bacteria was also tried. In this method a stab was made in a test tube containing standard gelatin media and incubated for a period of one week at 20° C. No signs of bacterial growth was noted in any of these tests.

Acidity in Greases

That good ball bearing greases should have not more than .1 per cent F.F.A., expressed as oleic, nor should they contain more than .25 per cent of free alkalinity, calculated as NaOH, are the opinions of several bearing manufacturers.

It is common knowledge that greases increase in acidity after use. The difference of opinion as to the harm that this increased acidity causes to ball bearings is confusing. Experience does not seem to help much either, for there are cases in which much trouble has been caused by greases which had a relatively lower F.F.A. content than other greases, which one would expect to cause trouble due to the unusually high F.F.A., referring to the F.F.A. in used greases.

It is admitted that the F.F.A. determination does not tell us much more

* *Ind. Eng. Chem.*, **27**, 724 (1935).

than the fact that acids are present in the grease. The method does not differentiate between acids which are inactive and those which are active.

By inactive acids, we have in mind the fatty acids which are insoluble in water and which have more than ten (10) carbon atoms in it's molecule. Stearic, palmetic, and oleic acids are the common fatty acids of this group. All fatty acids which contain less than ten (10) carbon atoms in their molecules are soluble, some more than others. These acids are volatile and are mostly liquids at ordinary temperatures. Acetic, butyric and proprionic acids are the common acids of the volatile active group.

Since the active acids are volatile it was attempted to separate them from the greases by first, cracking the grease with phosphoric acid and then steam distilling the mixture. The hydrogen ion concentration (pH) of the distillate was determined and finally it was titrated with a Leeds and Northrup quinhydrone potentiometer.

	Initial pH	pH Tenfold Dilution	Difference
Grease A	3.55	4.25	0.7
Grease B	3.70	4.30	0.6
F.F.A. as Oleic			
A—	3.42 per cent		
B—	1.70 per cent		

A. Total volatile soluble acids calculated as acetic acid .46 per cent.
B. Total volatile soluble acids calculated as acetic acid .28 per cent.

In the data marked "A," the active acidity is present in quantities sufficient to cause corrosion to bearings, basing our opinion on the results obtained by R. F. Innes in his investigation of strong acids in vegetable tanned leathers—J.I.S.L.T.G., **15**, 203 (1931).

The data in "B," shows the absence of strong acids. These acids are formed by the oxidation of the mineral oils which first oxidize, to peroxide and finally to the fatty acids. However, it is believed that the decomposition takes place wholly in the mineral oil, for the total volatile acids, due to tallow, is very low (1.3 mgs. KOH per gram of tallow). This is equivalent to .028 per cent acetic acid in grease.

It is realized that this method used for the determination of the active acidity was not developed exclusively for lubricating greases. The principle, however, is sound and was used by I. M. Kolthoff in detecting strong acids in dilute solutions. This method is the only one available on the problem of the determination of active acidity. There is no doubt that further investigations on the method will improve the determination of active acidity.

After a grease has once passed the Marlin-Rockwell tests and met with their approval as a suitable ball bearing lubricant, they have, on several occasions, experienced great difficulties in obtaining from the manufacturer uniform products comparable to the samples originally examined. One of their greatest problems in grease lubrication is due to this lack of uniformity of greases. They feel that a great deal can be accomplished by the grease manufacturer in standardizing greases.

With regard to the methods used for checking the Marlin-Rockwell requirements, they first make a chemical analysis of each grease as it is received in their laboratory for test. This analysis includes determination of melting point and kind of soap with which the grease is made. For these chemical analyses they use the A.S.T.M. methods wherever possible.

Following the chemical analyses, they make the following physical tests on greases, namely, agitation test, 100 hour running test and oxidization test. These tests are described as follows:

Agitation Test

The apparatus consists of a motor driven vertical shaft supported by two pillow blocks. A one piece cup and a cap are fastened to the bottom pillow block. The cap has a section cut out in such a manner that the grease can be observed at all times. A No. 204-F type bearing is mounted on the shaft and the cup screwed into place. The unit is then immersed in an oil bath to a point $\frac{1}{8}$ inch to $\frac{3}{16}$ inch above the top edge of the bearing. An electric hot plate is used to heat the oil bath. A speed of 3400 R.P.M. is maintained during the test.

The lubricant is then introduced into the bearing through the opening in the cap and the unit brought to a temperature 15° F. to 25° F. below the melting point of the grease. While the grease is under agitation, it is closely observed to note the action and to ascertain the temperature at which any change of structure or separation takes place. At the conclusion of the test, the bearing and lubricant are removed from the apparatus and cooled to room temperature. The grease is examined for separation, change of structure and change of consistency. These are the three important factors in the acceptance or rejection of a grease on the basis of the agitation test.

The advantage of this test lies in the fact that in a very short time results are obtained which indicate what may be expected of a lubricant over a longer period of actual service.

It brings to light characteristics in certain lubricants which may be considered objectionable.

Separation of oil from the soap base, either permanent or temporary, changes in consistency when cooled after testing, changes in structure, channeling, and in some instances changes of color due to aerifying, can be classified as objectionable features and should be taken into consideration when recommending a lubricant for any particular application.

Separation—Permanent. When this occurs, it is possible to see free oil after the grease has been cooled to room temperature. This condition hardly needs any comment, since it is obvious that if this separation continues over an indefinite period of time the soap will form a hard residue which will be detrimental to the efficient operation of the bearing even at the point of causing bearing failure.

Separation—Temporary. This condition occurs when evidence of free oil can be seen while the grease is under agitation and on cooling does not remain free but carries enough soap with it to solidify. It has been observed that a grease showing temporary separation generally does not return to its original consistency but remains a little softer when cooled after testing.

Changes in Consistency. Some greases become harder and some become softer when cooled to room temperature after testing. When a grease becomes softer there is the danger of leakage to be considered, while a grease that becomes harder carries with it the liability of heating and increased starting torque.

Changes in Structure. This condition is usually accompanied by a change in consistency and next to permanent separation it is the most undesirable characteristic a lubricant can possess. In the case of some fibre greases, the change is so drastic and takes place so suddenly as to be somewhat startling to the observer. This is particularly true of most greases classified as short fibre in structure. At a certain point in the test this type of grease will suddenly turn to a longer fibre and assume the consistency of soft wax, balling up around the shaft leaving the bearing as dry as though no grease had ever been applied. Buttery greases often change to fibre greases, and vice versa, under the combined action of heat and agitation.

Channeling. This is a characteristic of nearly all calcium base greases and a few sodium soap base greases. When the lubricant is applied to the bearing, the ball separator cuts a channel, the grease retaining the shape cut out by the separator and no further lubrication of the ball grooves occurs until sufficient heat has been applied to lower the consistency of the grease to a point where it will be drawn into the ball grooves by the action of the bearing.

Changes in Color. When this is the only effect of agitation, the opinion is that it is not objectionable. In many instances, however, it is an indication of one of the foregoing objectionable characteristics since it is seldom found alone.

100 Hour Running Test

After passing the agitation test, the grease is returned to the Chemical Laboratory to complete the chemical analyses. If found to be OK by the Chemical Laboratory it is again returned to the Physical Laboratory for a final test in a bearing under normal loads for a period of 100 hours.

The apparatus used in this test consists of a No. 307 bearing in an oil tight housing placed between two ball bearing equipped pedestals. The housing is so designed as to allow a load of 1350 pounds to be applied to the bearing. The apparatus is belt driven at a speed of 1600 R.P.M.

Both sides of the bearing are filled one-half full of the lubricant placed in the apparatus. Records of the temperature rise and the room temperature are made at frequent intervals over the 100 hour period. At the conclusion of the test, the bearing and lubricant are removed from the housing and allowed to cool to room temperature. The lubricant is then closely examined for separation, change of structure, and change of consistency.

This test constitutes a check on the agitation test. Sometimes the rapid agitation and elevated temperature failed to bring to light a slight separation or change of structure that is disclosed by the slower agitation over a much longer period. Any separation or change in structure in this test is sufficient cause for the rejection of the grease. It is well to mention at this point that the only greases submitted to 100 hour tests in this laboratory were those which have successfully passed the agitation test.

Oxidization and Gumming Test

A bearing is filled about one-half full of the grease to be tested, wrapped and placed in storage. It is examined every three months for oxidization and gumming. Incidentally, we have found that sodium soap greases are much less susceptible to oxidization than calcium soap greases, and for this reason do not recommend the latter unless moisture is present.

A grease containing a filler, such as asbestos, clay, graphite, zinc or oxide, is submitted to an additional hundred hour wear test in addition to the above tests. Any grease containing a filler is generally automatically thrown out.

Timken Requirements. The Timken Company require greases for steel mills, electric motors, and mine cars to comply with the requirements indicated in the columns marked "A," below. The tests on Standardized Cup Grease are indicated in the columns marked with "B":

Service	A Steel Mills	A Low and Med. Speed Indust.	B Cup No. 4	A Mine Cars	B Cup No. 3	A Mine Cars	A Electric Motors
Date of Spec.	May 1, '29	Sept. 1, '29		Mar. 1, '30		May 1, '29	
Karns-Maag							
Consist 75° F.	30–45	15–30	13.6	25–40	21.5	25–40	20–35
Unworked penetration 77° F.	264–315	197–264	186	245–300	240	245–300	225–282
Worked penetration 77° F.			216 (Spec. 190–230)		263 (Spec. 240–280)		
Ash, percent max.	2	2	2.31	2	1.66	2.5	2
Corrosion test 24 hrs room temperature	O.K.	O.K.	O.K.	O.K.	O.K.	O.K.	O.K.
Moisture, percent max.	2	2	2.0	2	1.6	2	2
Mineral Oil							
Flash, min., ° F.	400	340	370	340	360	340	340
Fire, min., ° F.	450	380	415	380	410	380	380
S.U. v. 210° F. min.	75						
Cold test max., ° F.	40	30	·	30	–0	30	30
S.U. v. 100° F. min.		200	328	200	300	200	200
Resistance to water	...	O.K.	O.K.	O.K.	O.K.	O.K.	...

TABLE 6—Heavy Duty Applications

Operating Temperature	Moisture Present		No Moisture	
	Oil	Grease	Oil	Grease
Below 32° F.			Extreme Pressure Oil Viscosity 75 sec. to 100 sec. @ 210° F. Saybolt Universal. Load capacity minimum 33 lbs. lever load. Timken Lubricant Tester.	Extreme Pressure Grease; Lime or Soda Base. Consistency Karns-Maag Method 30.0 mm. to 45.0 mm. @ 75° F. Load capacity minimum 33 lbs. lever load. Timken Lubricant Tester.
32° F. to 125° F.	Extreme Pressure Oil Viscosity 100 sec. to 175 sec. @ 210° F. Saybolt Universal. Load capacity minimum 33 lbs. lever load. Timken Lubricant Tester.	Extreme Pressure, Water Resistant Grease. Consistency Karns-Maag Method 20.0 mm. to 30.0 mm. @ 75° F. Load capacity minimum 33 lbs. lever load. Timken Lubricant Tester.	Extreme Pressure Oil Viscosity 100 sec. to 175 sec. @ 210° F. Saybolt Universal. Load capacity minimum 33 lbs. lever load. Timken Lubricant Tester.	Extreme Pressure Grease; Lime or Soda Base. Consistency Karns-Maag Method 20.0 mm. to 30.0 mm. @ 75° F. Load capacity minimum 33 lbs. lever load. Timken Lubricant Tester.
125° F. to 180° F.	Extreme Pressure Oil Viscosity 100 sec. to 250 sec. @ 210° F. Saybolt Universal. Load capacity minimum 33 lbs. lever load. Timken Lubricant Tester.	Extreme Pressure, Water Resistant Grease. Consistency Karns-Maag Method 15.0 mm. to 25.0 mm. @ 75° F. Load capacity minimum 33 lbs. lever load. Timken Lubricant Tester.	Extreme Pressure Oil Viscosity 100 sec. to 250 sec. @ 210° F. Saybolt Universal. Load capacity minimum 33 lbs. lever load. Timken Lubricant Tester.	Extreme Pressure Grease; Lime or Soda Base. Consistency Karns-Maag Method 15.0 mm. to 25.0 mm. @ 75° F. Load capacity minimum 33 lbs. lever load. Timken Lubricant Tester.
Above 180° F.	Extreme Pressure Oil Viscosity 175 sec. to 250 sec. @ 210° F. Saybolt Universal. Load capacity minimum 33 lbs. lever load. Timken Lubricant Tester.	Not Recommended.	Extreme Pressure Oil Viscosity 175 sec. to 250 sec. @ 210° F. Saybolt Universal. Load capacity minimum 33 lbs. lever load. Timken Lubricant Tester.	Not Recommended.

Timken Lubricant Requirements

O. L. Maag [70] has listed the Timken requirements for roller bearing lubricants, as shown in the tables below. Both mineral oil requirements and greases of both sodium and calcium base are included.

General Machinery Lubrication

Water Resistance: Lime base greases are recommended for conditions where moisture is present.

Corrosion: A bright copper or steel plate shall show no marked discoloration after being submerged in the grease for twenty-four hours.

Water Content: 1 per cent maximum.

Ash: 2 per cent maximum.

Separation: Grease shall show no bleeding or separation either in storage or in use.

Properties of Oil Content of Roller Bearing Grease

Flash: Minimum, 340° F.

Fire: Minimum, 380° F.

S.U.V./100° F. 200 minimum.

Heavy Duty Lubrication

Corrosion: A bright copper or steel plate shall show no marked discoloration after being submerged in the grease for fifty hours at normal bearing temperatures.

Water Resistance: It must function satisfactorily under all water conditions encountered.

Moisture: Maximum, 1 per cent.

The mineral oil from which the grease is compounded shall conform to the following tests:

Flash: Minimum, 375° F.

Fire: Minimum, 425° F.

Viscosity: Saybolt Universal, 75 seconds minimum at 210° F.

Cold Test, Pour: Maximum, plus 40° F.

The lubricant, when of the extreme pressure class, must prevent scoring on the Timken Lubricant Tester with a minimum load of 33 pounds on the lever arm at a minimum rubbing speed of 400 feet per minute.

Air Brake Greases

In the past, railway air brake cylinders were provided with pistons bearing leather packings. To keep these pliable, and to promote a better seal, lubricants which would have no deteriorating effect on leather were considered necessary. The air brake is subject to temperatures ranging from 40 degrees below zero to 110 degrees above, and under these conditions the lubricant used should not freeze and stick the piston, form a deposit in the bottom of cylinder, nor be picked up by the air stream and be carried into the triple valve to interfere with its delicate operation. The lubricant should prevent rusting as the air in the cylinder frequently carries considerable moisture, and the grease should not be washed away too readily.

In view of these requirements it is not surprising that many of the earlier air brake cylinder greases were soft graphite cup greases made with very low viscosity, low cold test oils. It was considered best to use fats and leave the glycerin in the grease after saponification with lime, as it is likely that the glycerin favorably affects the low temperature operation of the grease under wet conditions.

[70] *Iron Steel Eng.* (Dec. 1935).

TABLE 7—Type and Viscosity of Lubricants Recommended for Timken Bearings in General Machinery Applications.

Speed—Up to 500 R.P.M.

Operating Temperature	Bearing Size	Moisture Present		No Moisture	
		Oil	Grease	Oil	Grease
Below 32° F.	Up to 6″ O.D.			Neutral Mineral; Viscosity 75 sec. to 500 sec. @ 100° F. Saybolt Universal.	Lime or Soda Base; Consistency 20.0 mm. to 50.0 mm. @ 75° F. Karns-Maag Method.
	6″ O.D. and above			Neutral Mineral; Viscosity 75 sec. to 500 sec. @ 100° F. Saybolt Universal.	Lime or Soda Base; Consistency 20.0 mm. to 50.0 mm. @ 75° F. Karns-Maag Method.
32° F. to 125° F.	Up to 6″ O.D.	Neutral Mineral; Viscosity 100 sec. to 700 sec. @ 100° F. Saybolt Universal.	Lime Base; Medium Consistency 20.0 mm. to 40.0 mm. @ 75° F. Karns-Maag Method.	Neutral Mineral; Viscosity 100 sec. to 700 sec. @ 100° F. Saybolt Universal.	Lime or Soda Base; Medium Consistency 20.0 mm. to 40.0 mm. @ 75° F. Karns-Maag Method.
	6″ O.D. and above	Neutral Mineral; Viscosity 100 sec. to 700 sec. @ 100° F. Saybolt Universal.	Lime Base; Medium Consistency 20.0 mm. to 40.0 mm. @ 75° F. Karns-Maag Method.	Neutral Mineral; Viscosity 100 sec. to 700 sec. @ 100° F. Saybolt Universal.	Lime or Soda Base; Medium Consistency 20.0 mm. to 40.0 mm. @ 75° F. Karns-Maag Method.
125° F. to 180° F.	Up to 6″ O.D.	Neutral Mineral; Viscosity 60 sec. to 100 sec. @ 210° F. Saybolt Universal.	Lime Base; Medium Consistency 15.0 mm. to 30.0 mm. @ 75° F. Karns-Maag Method.	Neutral Mineral; Viscosity 60 sec. to 100 sec. @ 210° F. Saybolt Universal.	Soda Base; Medium Consistency 15.0 mm. to 30.0 mm. @ 75° F. Karns-Maag Method.
	6″ O.D. and above	Neutral Mineral; Viscosity 60 sec. to 100 sec. @ 210° F. Saybolt Universal.	Lime Base; Medium Consistency 15.0 mm. to 30.0 mm. @ 75° F. Karns-Maag Method.	Neutral Mineral; Viscosity 60 sec. to 100 sec. @ 210° F. Saybolt Universal.	Soda Base; Medium Consistency 15.0 mm. to 30.0 mm. @ 75° F. Karns-Maag Method.
Above 180°	Up to 6″ O.D.	*Neutral Mineral; Viscosity 100 sec. and up @ 210° F. Saybolt Universal.	Not Recommended.	*Neutral Mineral; Viscosity 100 sec. and up @ 210° F. Saybolt Universal.	Not Recommended.
	6″ O.D. and above	*Neutral Mineral; Viscosity 100 sec. and up @ 210° F. Saybolt Universal.	Not Recommended.	*Neutral Mineral; Viscosity 100 sec. and up @ 210° F. Saybolt Universal.	Not Recommended.

* Refined cylinder oil meeting these specifications may be obtained from most companies.

TABLE 8—Type and Viscosity of Lubricants Recommended for Timken Bearings in General Machinery Applications.

Speed—500 to 1000 R.P.M.

Operating Temperature	Bearing Size	Moisture Present		No Moisture	
		Oil	Grease	Oil	Grease
Below 32° F.	Up to 6" O.D.			Neutral Mineral; Viscosity 75 sec. to 500 sec. @ 100° F. Saybolt Universal.	Lime or Soda Base; Consistency 20.0 mm. to 50.0 mm. @ 75° F. Karns-Maag Method.
	6" O.D. and above			Neutral Mineral; Viscosity 75 sec. to 500 sec. @ 100° F. Saybolt Universal.	Not Recommended.
32° F. to 125° F.	Up to 6" O.D.	Neutral Mineral; Viscosity 100 sec. to 700 sec. @ 100° F. Saybolt Universal.	Lime Base; Medium Consistency 20.0 mm. to 40.0 mm. @ 75° F. Karns-Maag Method.	Neutral Mineral; Viscosity 100 sec. to 700 sec. @ 100° F. Saybolt Universal.	Lime or Soda Base; Medium Consistency 20.0 mm. to 40.0 mm. @ 75° F. Karns-Maag Method.
	6" O.D. and above	Neutral Mineral; Viscosity 100 sec. to 700 sec. @ 100° F. Saybolt Universal.	Not Recommended.	Neutral Mineral; Viscosity 100 sec. to 700 sec. @ 100° F. Saybolt Universal.	Not Recommended.
125° F. to 180° F.	Up to 6" O.D.	Neutral Mineral; Viscosity 60 sec. to 100 sec. @ 210° F. Saybolt Universal.	Lime Base; Medium Consistency 15.0 mm. to 30.0 mm. @ 75° F. Karns-Maag Method.	Neutral Mineral; Viscosity 60 sec. to 100 sec. @ 210° F. Saybolt Universal.	Soda Base; Medium Consistency 15.0 mm. to 30.0 mm. @ 75° F. Karns-Maag Method.
	6" O.D. and above	Neutral Mineral; Viscosity 60 sec. to 100 sec. @ 210° F. Saybolt Universal.	Not Recommended.	Neutral Mineral; Viscosity 60 sec. to 100 sec. @ 210° F. Saybolt Universal.	Not Recommended.
Above 180°	Up to 6" O.D.	*Neutral Mineral; Viscosity 100 sec. and up @ 210° F. Saybolt Universal.	Not Recommended	*Neutral Mineral; Viscosity 100 sec. and up @ 210° F. Saybolt Universal.	Not Recommended.
	6" O.D. and above	*Neutral Mineral; Viscosity 100 sec. and up @ 210° F. Saybolt Universal.	Not Recommended.	*Neutral Mineral; Viscosity 100 sec. and up @ 210° F. Saybolt Universal.	Not Recommended.

* Refined cylinder oil meeting these specifications may be obtained from most companies.

Over 1000 R.P.M.

Below 32° F.	Up to 6" O.D.			Neutral Mineral; Viscosity 75 sec. to 300 sec. @ 100° F. Saybolt Universal. z	Soda Base; short fiber, Consistency 30.0 mm. to 50.0 mm. @ 75° F. Karns-Maag Method. o
32° F. to 125° F.	6" O.D. and above			Neutral Mineral; Viscosity 75 sec. to 300 sec. @ 100° F. Saybolt Universal. z	Not Recommended.
125° F. to 180° F.	Up to 6" O.D.	Neutral Mineral; Viscosity 100 sec. to 700 sec. @ 100° F. Saybolt Universal. b	Lime Base; Medium Consistency 20.0 mm. to 40.0 mm. @ 75° F. Karns-Maag Method. p	Neutral Mineral; Viscosity 100 sec. to 700 sec. @ 100° F. Saybolt Universal. b	Soda Base; Medium Consistency 20.0 mm. to 40.0 mm. @ 75° F. Karns-Maag Method. r
	6" O.D. and above	Neutral Mineral; Viscosity 100 sec. to 700 sec. @ 100° F. Saybolt Universal. b	Not Recommended.	Neutral Mineral; Viscosity 100 sec. to 700 sec. @ 100° F. Saybolt Universal. b	Not Recommended.
Above 180°	Up to 6" O.D.	Neutral Mineral; Viscosity 60 sec. to 100 sec. @ 210° F. Saybolt Universal. c	Lime Base; Medium Consistency 15.0 mm. to 30.0 mm. @ 75° F. Karns-Maag Method. s	Neutral Mineral; Viscosity 60 sec. to 100 sec. @ 210° F. Saybolt Universal. c	Soda Base; Medium Consistency 15.0 mm. to 30.0 mm. @ 75° F. Karns-Maag Method. t
	6" O.D. and above	Neutral Mineral; Viscosity 60 sec. to 100 sec. @ 210° F. Saybolt Universal. c	Not Recommended.	Neutral Mineral; Viscosity 60 sec. to 100 sec. @ 210° F. Saybolt Universal. c	Not Recommended.
	Up to 6" O.D.	*Neutral Mineral; Viscosity 100 sec. and up @ 210° F. Saybolt Universal. d	Not Recommended.	*Neutral Mineral; Viscosity 100 sec. and up @ 210° F. Saybolt Universal. d	Not Recommended.
	6" O.D. and above	*Neutral Mineral; Viscosity 100 sec. and up @ 210° F. Saybolt Universal. d	Not Recommended.	*Neutral Mineral; Viscosity 100 sec. and up @ 210° F. Saybolt Universal. d	Not Recommended.

* Refined cylinder oil meeting these specifications may be obtained from most companies.

Lime Base Air Brake Cylinder Grease

Formula

	Per Cent by Weight
No. 1 Lard oil	15.00
Calcium hydrate	2.15
Water	1.30
100 S.U.V./100° F. Texas Neutral	81.55

In some cases railroads preferred the above grease compounded with 6 per cent by weight of fine flake graphite. With the general adoption of fabric composition type disks to take the place of the leathers referred to above, the tendency has been away from the use of lime base greases and towards the application of light grades of sodium base fiber greases.

Automotive Greases (Calcium Base)

According to Webster's dictionary, lubricating greases are preparations, thicker than oil, which when applied to a surface, render it smooth or slippery. The lubricating greases which will be discussed in this section have these general properties, but more specifically are dispersions of soap in mineral oil and are intended to effectively prevent wear of automobile bearing surfaces. Greases, as defined by Webster, may have been adequate for wagon axles of a century ago, but with the development of the horseless carriage into our modern high speed automobile, "specialized" lubrication has become commonplace. A few years ago, special greases for water pumps, wheel bearings, steering gears and universal joints were not considered necessary, but in order to lengthen the useful life of motor cars, reduce expensive repair bills and particularly in view of more severe service due to sustained high speed driving and the demand for greater dependability and smooth handling, at least five special lubricating greases are now considered essential to the proper maintenance of your car's chassis.

Grease manufacturers in the United States produce annually nearly 250,000,000 pounds of lubricating grease, which is sold for approximately $18,000,000. Of this total amount, about 50 per cent, or 125,000,000 pounds, of special grease are consumed in automotive units. As in other industries, grease manufacturers are intently alert to new developments in mechanical engineering, particularly in the automotive field. This is only natural, since roughly one-half of the total amount of grease produced is consumed by the motorist. Special service tests for automobile lubricating oils and gasoline have been more or less standardized, but, as will be pointed out in this discussion, the service testing of lubricating greases has been largely a matter of special technic developed as occasion necessitated by the grease manufacturer, the automobile manufacturer, institution or consumer. Because casual observation of service results in equipment on the road often leads to erroneous results and comparisons, laboratory service tests are frequently resorted to in order to secure more precise data from which definite conclusions may be drawn. In some cases, a series of greases differing only slightly in their usual properties must be studied to

determine their relative service value. Road tests for thousands of miles might be needed to reveal practical differences, but accelerated laboratory service tests can be devised which enable definite comparisons to be made in a few hours.

In the following sections of this chapter, formulae, control and purchase specifications, manufacturing procedure, as well as the results of many laboratory service tests, will be presented for calcium base automotive greases. In some comparisons, results of tests on aluminum and sodium soap greases will be included to complete the discussions.

Pressure Gun Grease

Pressure gun grease is intended for the lubrication of miscellaneous chassis bearings, namely, spring shackles, king pins and steering knuckles. The adoption of the rubber bushed type of spring eye and the less popular centralized systems of lubricating various chassis bearings with lubricating oil, indicates that in some instances the application of various types of chassis grease has not by any means been considered ideal by automotive engineers. The development of the threaded spring shackle has introduced a further testing complication in that the retention of lubricant in this type of shackle is definitely better than in normal plain bushing shackles. The Tryon shackle, which depends on the feeding of the grease from a reservoir to the tapered bearing ends where clearances are quite small, necessitates a lubricant particularly resistant to separation of its soap base. Unstable greases, which permit the deposition of residues of soap containing relatively little oil, interfere with the free flow of lubricant to the bearing surfaces.

Many of the cars on the road, however, are equipped with some form of plain bushing shackle. In some cases, needle bearings have been fitted into the king pins, and of course the development of "knee-action" front wheels has brought about modifications in steering mechanism bearings. However, the chief problem regarding the quality of pressure gun greases is that of their ability to "stay in place." It is for this reason that service tests for determining the retention characteristics of such greases have been developed by grease manufacturers and are of interest.

The terms: pressure gun grease, pressure gun lubricant, high pressure grease, high pressure gun lubricant, chassis grease and chassis lubricant are synonymous. The present tendency is to use the name, pressure gun lubricant, the term high pressure grease being quite common ten years ago.

The formula for Cup Grease "A," already given, is a good example of an ordinary high pressure lubricant. The only objection to this product is that it is prone to leave a deposit of hard lime soap in the oil channels of shackle bolts and other openings, particularly when long intervals occur between greasing. The best high pressure lubricants for automobile and general industrial use should be made with an oil of 250 to 550 Saybolt viscosity at 100° F., as such greases are more resistant to pressure between the bearing surfaces, by reason of the fact that they give a more viscous lubricating film.

There appeared on the market in 1923, a line of greases for this field which were of high melting point and did not leave the residues just referred to. These may be made by the following processes.

High Pressure Grease, Soft

Formula

	Per Cent by Weight
Commercial stearic acid	2.43
Oleic acid	6.57
Oil of mirbane	.05
Hydrated lime	1.23
Red dye (National Aniline Co. Oil Red O.)	.002
320 Western red oil	88.818
Water	.800

PROCEDURE

The acids are first melted in a steam heated kettle and a mixture of the lime, about 10 per cent of the oil and 1.5 per cent of water is added. The steam is then turned on, agitation being continued throughout.

When a temperature of 250° F. is attained the steam is turned off and the mineral oil started in from an overhead tank.

When 27 to 30 per cent of the oil is in, the 0.8 per cent of water is added slowly while stirring. The temperature at this period should be 220° to 230° F.

The balance of the oil is then run in while stirring and as fast as possible without chilling or causing the grease to lump. The grease is drawn at about 140° F.

CHARACTERISTICS OF PRODUCT

A.S.T.M. penetration	450
Melting point	181° F.

High Pressure Grease, Heavy Grade

Formula

	Per Cent by Weight
Stearic acid	2.98
Oleic acid	8.04
Hydrated lime	1.59
Water	.62
Western pale oil 100 Vis./100° F.	12.25
Oil of mirbane	.06
Western red oil 400 Vis./100° F.	74.46

PROCEDURE

The same process as for No. 1 is used.

CHARACTERISTICS OF PRODUCT

A.S.T.M. penetration	208
Melting point	201° F.

This grease on loss of the water of hydration does not separate but remains as a stable gel.

Standardized Pressure Gun Lubricant

The lubricant made on the following formula has been marketed successfully for several years. The viscosity of its oil content is 450 S.U.V./100° F., the grease is colored with a lubricating oil of about 300 S.U.V./100° F.,

containing approximately 5 per cent by weight of color bodies extracted from the tar derived from the process of making illuminating gas from light petroleum fractions. Fluorescent green coal tar dyes may be substituted for this coloring essence with equal effect.

Constituents	Gallons	Pounds Net	Per Cent by Weight
Oleic acid	64	479.10	7.94
Stearic acid	16	120.00	1.99
Hydrated lime		85.00	1.41
30° Bé. caustic soda	¼	.62	.02
Water		18.10	.30
300 pale oil	198	1510.60	25.03
600 red oil	450	3454.65	57.23
Dye essence	47	366.93	6.08
		6035.00	100.00

AUTOCLAVE LOADING

Measure 40 gallons of 300 S.U.V./100° F. Pale Oil into lime mixing tank.

Add 85 pounds of hydrated lime for single batch. (If double batch is being made one lime mixture may be made with 100 pounds of lime and an additional mixture with 70 pounds.) Stir well with a Lightnin Mixer. Add oil to bring mixture to 80 gallons.

Pump into the fat measuring tank in the order named:

	Single Batch Gallons	Double Batch Gallons
Stearic acid (melted)	16	32
Oleic acid	64	128

Run the lime mixture into autoclave, followed by ¼ gallon of 30° Bé. caustic soda. (A total of ½ gallon for the double charge.)

Run in 3 gallons of water (4 gallons for double batch).

Measure out 45 gallons of 300 Pale Oil and run it into the autoclave. This serves to flush the system free of lime and caustic soda. The total amount of 300 Pale Oil should be 1½ times the volume of fatty acids used, 120 gallons for the single charge. For the double charge (160 gallons of fatty acids) a total of 240 gallons of 300 Pale Oil should be used. In the case of the single charge the total volume of ingredients will be about 225 gallons, while the double batch will be 450 gallons.

SAPONIFICATION

Same procedure as for Standardized Cup Grease, except that the autoclave pressure will range from 5 to 25 pounds per square inch and temperature 220 to 245° F.

REDUCTION OF SOAP

Fifty gallons of 300 Pale Oil and 50 gallons of slop High Pressure Grease are charged in each of the open mixers and heated to about 200° F. to 220° F. The soap is then discharged from the autoclave into the two open mixers. During this procedure much of the water formed by the neutralization reaction between the lime and fatty acids, as well as some of that added to the autoclave is lost by "flash" evaporation.

The fatty acid grease is handled somewhat differently in the mixer than when making cup grease with fats (triglycerides). The general appearance of the grease in its earlier stages is similar to cup grease at the same period, but the soap is worked up much drier than is possible for cup grease. After about two thirds of the 300 Pale oil has been mixed in and the temperature

of the batches has been considerably reduced, it begins to assume the smooth appearance of a normal lime base grease.

Mixing in the oil while the soap base is relatively hot and in a fluid condition assists in preventing the occlusion of air, and permits the production of grease of a high degree of transparency. The prevention of excess alkali and the reduction of the water content to a minimum are also responsible to a certain extent for the production of bright, transparent grease.

Finally the dye stock is run in which gives the product a red-green outertone.

When, in the compounder's judgment, the grease is of the required grade, it is pumped through a sixty mesh screen to the containers and a sample sent to the laboratory for approval.

The lubricant made in accordance with the preceding formula and process should comply with the following manufacturing specifications:

A.S.T.M. worked penetration at 77° F.	340–370
Min., Ubbelohde dropping point ° F.	185
Max., percent ash (A.S.T.M., D-128-27)	1.50
Max., percent free alkali (A.S.T.M. D-128-27)	0.35
Average A.S.T.M. soap content	10.0
Max., percent free acid (A.S.T.M. D-128-27)	0.30
Max., percent water by distillation (A.S.T.M. D-95-30)	1.00
S.U.V. of mineral oil at 100° F.	400–500
A.S.T.M. color of mineral oil, max.	5
Corrosion test (530.4)	Neg.

Pressure Gun Grease Purchase Specifications

The results of a survey of the guaranteed specifications for chassis lubricants marketed by sixteen manufacturers in the Eastern part of the United States, indicated that six preferred to use aluminum stearate as their base and the balance were manufactured with lime soaps of various sorts.

Company Number	Percent Soap	Base	Percent Water	Percent Min. Oil	Grav. of Oil (A.P.I.)	S.U.V./ 100° F. of Oil	A.S.T.M. Color of Oil	Pour Point of Oil	Color of Grease
1	14	Ca	1	84	23–24	300–310	5	0	Red, Trans.
2	7	Al	Tr	92.8	24.9	54/210	..	20	Dk. Green
3	7	Al	Tr	92.6	21.6	115/210	..	25	Dk. Green
4	11–12	Ca	.75	88–89	25.7	285	..	25	Dk. Yellow
5	9	Ca	1	89.9	25	800–810	6	25	Red
6	12	Ca	1	87	25	800–810	6	25	Red
7	9–12	Ca	..	...	...	250	..	..	...
8	10	Ca	.5	89.5	24	300	6	0	Red
9	9	Ca	.5	90.5	23.5	750	7	15	Grn. Cast
10	10	Al	0	90	20.0	300	..	..	Yel. Trans.
11	9	Al	Tr	91	22–23	110/210	5	10	Clear Red
12	8	Al	Tr	92	23–24	85/210	5	5	Clear Red
13	8	Ca	Tr	92	20.5	310	5½	−10	Pale
14	4	Al	0	96	20	750	..	..	Green
15	8–12	Ca	½–1	...	...	200–700	..	..	Red
16	6–8	Ca	½	...	...	700–1200	..	..	Dk. Green

Company Number	A.S.T.M. Worked Penetration at 77° F.	Percent Free Alkali	Percent Free Acid	Odor	Dropping Point ° F.	Percent Ash as Sulfates	Corrosion
1	315–345	.15	0.1	Myrbane	190	4	Neg.
2	too soft	..	..	...	...	...	Neg.
3	too soft	..	..	...	...	...	Neg.
4	330–360	..	..	...	...	...	Neg.
5	340–360	.25	..	Citronella	200	3	Neg.
6	290–310	.25	..	Citronella	205	3.75	Neg.
7	300–350	..	..	...	153	...	Neg.
8	325	..	..	...	170	.91 CaO	Neg.
9	325	..	..	...	170	.85 CaO	Neg.
10	...	..	..	...	...	...	Neg.
11	340–370	..	..	...	...	3½	Neg.
12	..	..	..	...	...	3½	Neg.
13	360–370	.15	0.10	...	200	3.2	Neg.
14	..	..	..	...	...	...	Neg.
15	345–390	..	..	Myrbane	190–230	.5	Neg.
16	390–450	..	..	...	...	1–1.5	Neg.

Abridged analyses of ten pressure gun greases marketed recently on the Pacific Coast are tabulated below:

Name	A.S.T.M. Penetration Unworked	Worked	Dropping Point ° C.	Kind of Soap	Percent Ash	S.U.V./ 100° F. of Min. Oil	Pressure S.U.V. 5 pounds per square inch (grms./min.) 35° F.	77° F.	100° F.
1—Un	360	357	89	Al	...	301	.56	9.7	...
2—Pen	362	375	97	Ca	1.04	319			
3—Vd	347	340	90	Ca	1.06	310			
4—Al	375	370	90	Ca	...	350			
5—Hv	315	314	96	Ca	1.61	868			
6—Zn	323	336	95	Ca	...	335	0.36	2.05	15.1
7—Sl	320	318	94	Ca	1.34	450	0.72	11.0	25.2
8—Se	342	345	97	Ca	1.70	311	1.46	19.8	...
9—Sp	354	354	92	Ca	1.72	312	2.06	22.8	...
10—Sp	325	335	96	Al	...	295	0.30	7.90	37.0

Chassis Lubricants Purchase Specifications

G. M. STANDARDS—CHASSIS LUBRICANT—G. M. 4612-M—GRADE G-11

General: Grease covered by this specification shall be a homogeneous combination of mineral oil and calcium soap.

Filler: Shall not contain filler such as clay, talc, whiting, asbestos, wood fiber, etc.

Consistency: The worked consistency (A.S.T.M.) shall be from 350 minimum to 400 maximum.

Soap: The total soap content shall be from 4 per cent to 6 per cent. The lime used in preparing the soap shall not contain more than 7 per cent of magnesium oxide and not more than 2 per cent of gritty non-soap forming substances. Not less than 93 per cent of the soap shall be calcium and magnesium soap. A small amount of caustic soda may be used if desired for completing saponification, but the sodium soap must not exceed 7 per cent of the total soap.

Viscosity: Saybolt Universal of mineral oil at 100° F. shall not exceed 4000 seconds; at 210° F. shall not be less than 140 seconds.

Acidity: Free fatty acids as oleic	Maximum 0.2 per cent
Uncombined Lime:	Maximum 0.10 per cent
Sand Silicates or Gritty Substances:	Maximum 0.03 per cent
Water:	Maximum 1.0 per cent

Notes: This lubricant is intended for use with chassis lubrication fittings.

G. M. STANDARDS—CHASSIS LUBRICANT—G. M. 4640-M

General: Grease covered by this specification shall be a homogeneous combination of mineral oil and calcium soap.

Filler: Shall not contain filler such as clay, talc, whiting, asbestos, wood fiber, etc.

Consistency: The worked consistency (A.S.T.M.) shall be from 300 minimum to 350 maximum.

Soap: The total soap content shall be from 8 per cent to 11 per cent. The lime used in preparing the soap shall not contain more than 7 per cent of magnesium oxide and not more than 2 per cent of gritty non-soap forming substances. Not less than 93 per cent of the soap shall be calcium and magnesium soap. A small amount of caustic soda may be used if desired for completing saponification, but the sodium soap must not exceed 7 per cent of the total soap.

Viscosity: Saybolt Universal of mineral oil at 100° F. shall be between 300 and 500 seconds.

Acidity: Free fatty acids as oleic	Maximum 0.2 per cent
Uncombined Lime:	Maximum 0.10 per cent
Sand Silicates or Gritty Substances:	Maximum .02 per cent
Water:	Maximum 1.0 per cent

Odor: Grease shall not be perfumed and shall be free from disagreeable odor or rancidity.

Permanence: The soap and oil shall not separate at reasonable temperatures within a reasonable time. Greases shall not harden objectionably with age or exposure.

Corrosion: Grease shall not corrode any metal used for machine construction.

Methods of Test: Tests shall be made in accordance with methods described in G. M. Standards.

Notes: This lubricant is intended for use with chassis lubrication fittings.

PRESSURE GUN GREASE. "F" MOTOR COMPANY SPECIFICATIONS M-538

The material desired under this specification is a well manufactured homogeneous product of calcium soap, made from high grade animal oils or fatty acids, and a refined mineral oil suitable for use in pressure lubricators such as Alemite Zerks.

A special product is not the aim of this specification, simply good practice in production, freedom from deleterious substances and uniformity in the material.

The grease should have the following chemical constants:

Calcium soap	8½–10%
Free fatty acid (calc. as oleic)	0.2% max.
Free lime	0.1% max.
Unsaponified saponifiable matter	1.5% max.
Moisture	1.0% max.
Mineral oil	Balance

Viscosity of Mineral oil at 100° F. to be 275 to 300 (Saybolt Universal) and to have nil acidity.

Penetration on a worked sample to be 320 to 350 tenths of a millimeter at 77° F. using 150 gram load for 5 seconds (A.S.T.M. D217-26T Method).

Shall contain no filler.

Shall be non-corrosive.

Shall contain no grit.

Shall be non-rancid and shall not bleed even on standing for a period of time.

Melting point to be 194–212° F.

As specified in the Purchase Order.

A checking slip bearing complete information written with waterproof ink and enclosed in a waterproof envelope, shall accompany all shipments.

In carload lots these envelopes shall be tacked inside of car, near door.

Material will be received subject to chemical and physical examination.

Material not conforming to the above requirements will be subject to rejection for full credit or replacement, at our option.

General Discussion of Pressure Gun Greases

Examples of points requiring chassis lubrication are shackle bolts, knuckle joints of the steering gear mechanism, king pins, saddles attached to the rear springs, and brake and clutch pedal shafts. The same lubricant is generally used at all these points requiring lubrication. This is partly a matter of convenience. However, the lubrication problems at these points are similar, so that a lubricant adapted to one point is usually fairly well adapted to others. For example, the following points of similarity may be noted:

There is no question of high speed bearings involved. Movements of parts, when they may be considered rapid at all, are intermittent. Therefore, there is no problem of high temperatures being built up at bearing surfaces.

Many of the parts are in position where exposure to water is possible under normal conditions of operation. At points which are more or less protected from water, a grease which is resistant to water is not necessarily at a disadvantage.

Loads at these various points are usually not excessive. Exceptions are due to differences in the design of parts made by different automobile manufacturers. In exceptional cases, then, special lubricants may be required.

These various parts are ordinarily lubricated by forcing the lubricant through pressure fittings into parts requiring lubrication. A more or less fluid type of lubricant is therefore required, since otherwise, difficulty may be encountered in getting the lubricant to flow under pressure. This difficulty is intensified at low temperatures due to the increase in the viscosity of the lubricants.

Efficiency of lubrication is largely a matter of retention of the lubricant. The lubricant gains entrance to the moving parts only at the moment of application of the grease. After the lubricant has worked out of these parts, no further protection is available until another application of lubricant is made unless a small amount of lubricant feeds in by capillary action. Retention problems at these different parts are somewhat similar. Very fluid types of lubricants will work out of these parts more rapidly than less fluid types. There will, of course, be some differences in the efficiency with which these parts hold a fluid lubricant. However, a product which is superior from the standpoint of retention in one part should in most cases prove superior in other parts.

The problem of shackle lubrication has been selected as typical, and experimental work has been carried out with special attention being given to the plain pin shackle and the screw type shackle. A detailed examination of the relationships between mechanical design and lubrication should prove of interest. This is especially true where mechanical designs are such that no lubricant at all is required, as for example with the use of rubber bushings in shackles, or where mechanical designs are such that specialized lubricants are required (*e. g.*, Bijur system, Tryon shackles, wick oilers, etc.).

The similarities in the lubrication problems involved at various parts requiring lubrication have suggested a few desirable properties for a chassis lubricant. In general, a chassis lubricant suitable for manufacture must have, in addition to purely technical qualifications for lubrication, certain requirements from the standpoint of manufacture and sales; for example, ease of manufacture, low cost, and excellent appearance. The desirable properties and attributes of a chassis lubricant are considered in outline below under two headings. First, taking into consideration the purely technical matters of lubrication, and second, taking into consideration manufacture and sales aspects.

PROPERTIES OF IDEAL CHASSIS LUBRICANT—TECHNICAL

Retention. As pointed out above, good retention is one of the important characteristics of an ideal chassis lubricant. Correlation of retention with the measureable physical properties is, of course, a matter of research. There are two types of chassis lubricants in common use, high viscosity oils, and greases. The greases, of course, are the much more complex products, since they are plastics, and therefore have a resistance to deformation not met with in oils. This leads to problems in connection with resistance to deformation, texture, etc., in addition to questions of viscosity, such as would be met with in the consideration of oils.

Oils cannot be expected to have the qualities of retention of greases since they will flow readily under mere pressures such as might be set up by gravity or the inertia resulting from moving parts. In general, the harder the grease the more difficult it will be to displace it from the part being lubricated. The harder greases will, therefore, be effective as a lubricant over a longer period of time. The only difficulty with using a hard product is that the greases are applied by passing the greases under pressure through pressure fittings into the parts to be lubricated. If a grease is too hard, extremely high pressure will be required to force the grease through the proper channels.

Fluidity. Since these chassis lubricants must flow under pressure, a certain amount of fluidity is necessary for a chassis lubricant.

Mechanisms at service stations for pumping the greases through to the fittings are variable. Two examples may be cited:

The grease is kept in a tank under pressure. From the tank the grease is forced through a small pipe (which may be of considerable length) to a small booster pump which supplies the pressure necessary to force the grease through the pressure fittings into the bearing surfaces.

In another arrangement the grease must flow from a container under the force of a partial vacuum into a piston type pump. Under this arrangement, it will be evident that the grease must be fluid enough to flow readily under pressures of 10 to 15 pounds per square inch, even though low temperatures are encountered as a result of cold weather.

In either of the cases above a relatively high fluidity is required, so that the first requirement of an ideal chassis lubricant is that it should be hard so that it will be retained well, and that secondly it should be sufficiently soft and fluid so that it may be readily pumped around. In practice, there is no way of realizing a grease which is ideal in both respects, since the qualities are contradictory. From the practical point of view, and other than devising proper guns, there are two ways to handle the situation. One way is to compromise so that a fairly good product (but not an ideal one) is obtained both as regards to retention and fluidity. The other method of handling the situation is to retain one quality at the expense of the other. This would logically lead to winter and summer grades of lubricants, a fluid lubricant to flow well in winter, and a heavier lubricant for improving retention during the summer time. While this policy of winter and summer

grades of lubricant might prove satisfactory from a technical point of view, from a marketing point of view it is not particularly desirable, since it leads to an unduly large number of products to be kept at distribution points.

In connection with the problem of fluidity the question of temperature susceptibilities of the products used in chassis lubrication should not be overlooked. From the standpoint of theory the decreased rate of flow of a grease at low temperatures is due to two factors: an increase in the yield point of the grease, and an increase in the viscosity of the grease, the viscosity of a grease being a measure of the increased pressure necessary to produce a unit increase in the rate of flow of a grease at pressures higher than the yield point. Though both yield point and viscosity are important in decreasing the rate of flow, at low temperatures the viscosity of a grease is extremely important. Where soap contents of two greases are quite similar, the viscosity of the grease will depend a good deal upon the oil viscosity. For this reason, a grease containing a highly viscous oil will be very susceptible to temperature as regards to rate of flow, since in a viscous oil the absolute increases in viscosity as the temperature is decreased is large in comparison with the increase in viscosity of a light oil.

Water Resistance. Since chassis lubricants in service may sometimes come in contact with water, the question of water resistance is important. Water, for example, coming in contact with a soda base grease (not resistant to water) may have the following effects.

The grease may be washed out of the point to be lubricated.
The water may be partially emulsified with the grease, leaving lubrication fairly intact, but the water content will result in an increased tendency to corrosion.
Water remaining in the grease may freeze in cold weather causing the grease to become brittle. This is not, of course, desirable from the standpoint of lubrication.

Resistance to Corrosion. Resistance to corrosion may probably be improved through the following policies regarding chassis lubricants.

Water content should be kept at a minimum value which is still consistent with the quality of the product and ease of manufacture.
Alkalinity should be kept as low as possible.
Only water resistant greases should be manufactured.
Special anti-rust and chemical compounds might be added to reduce corrosion. However, this should probably not be necessary, so long as water resistant greases only are manufactured.

High Film Strength. Theoretically a grease having a high film strength should prove more versatile as regards the number of conditions under which it will operate successfully. However, in the majority of situations in which chassis lubricants are used the loads are not particularly high and shear rates are not particularly high. An "extreme pressure" product may, therefore, not be necessary since it is only at high loads and high shear rates that the E.P. type of lubricant becomes necessary. In this connection it should also be noticed that the majority of chassis lubricants now manufactured contain light oils which may not be expected to carry any great loads on extreme pressure testers. Apparently no great amount of difficulty has been met in the use of these lubricants containing such oils. In view of these considerations it is likely that an E.P. type chassis lubricant

is not necessary for general use. In special cases such as with Tryon shackles, or screw shackles, where an E.P. type of lubricant may be necessary, an E.P. base may be incorporated.

Resistance to Dripping. Resistance to dripping is probably of secondary importance to other considerations. At service stations it has been customary to use an excess of grease at each point where pressure lubrication is required. The excess lubricant hangs from the fittings until after the grease operations are over. The excess grease is then removed by rags. If the lubricant applied is too fluid the lubricant may drop from the fittings onto the floor. Modern equipment, however, has been devised which prevents over-lubrication. Another remedy is to decrease the fluidity of the grease, either by increasing the soap content, or by adding materials such as latex which give the grease a stringy texture. Either remedy is likely to lead to difficulties at low temperatures where it is essential that the products should flow readily in spite of the low temperature.

Stability. The important desirable qualities of stability for a lubricant grease are:

Stability in Storage. The product should not separate, bleed or undergo other change in storage.

In the case of a lime base grease, stability to bleeding may be increased by increasing the unsaturation of the soap stocks, reducing the alkalinity of the grease, and increasing the hydration of the lime soap. Unfortunately, increasing the unsaturation of the soap stocks also increases the tendency to oxidize and gum, while increases in the water content of the grease make the grease less transparent.

Stability Towards Working. From the standpoint of retention it is essential that a grease should not break down too easily on working so as to run out of the bearing. Stability to working will depend to a certain extent upon the soap stocks. Aluminum soaps frequently break down excessively on working.

Calcium and sodium base greases can be improved in this respect by continuing agitation of the grease to lower temperatures before pumping the grease into containers.

Milling a grease may also improve the stability of a grease to working.

Stability to Heat. While stability to heat is a desirable characteristic of a grease it is not especially important in chassis lubrication since high temperatures are seldom encountered.

Sodium and aluminum base greases are more stable to heat than calcium base greases. A calcium base grease prepared from selected fatty acid stocks will prove more stable to heat than an ordinary cup grease.

DESIRABLE PROPERTIES OF CHASSIS LUBRICANT-MANUFACTURE AND SALES

Low cost of materials.

Low cost of manufacture. A product for which the anticipated volume is small should not require large outlays for plant equipment.

Excellent appearance.

Transparency.—A transparent grease will have a good appearance provided other qualities are satisfactory.

A grease to be transparent must be made from light oils which are quite transparent. The water content must also be low. For example, a soda base grease will be transparent only if produced by a high heat process so as to produce a dehydrated product. The grease must also be free of fillers.

Fluorescence. Fluorescence contributes to the appearance of a grease. This fluorescence can be obtained through the use of fluorescent dyes. For example, a fluorescent grease (yellow and green) may be obtained by adding an oil containing a mixture of oil soluble coal tar dyes (fluorescent green and blue green) to a fatty acid-calcium base grease containing 300 pale oil. The results, however, are usually

not quite so satisfactory as from the use of dyes as those obtained through blending with Pennsylvania Bright Stock which lends fluorescence to a grease.

Color. The question of color is closely related to that of fluorescence. In general, black or muddy looking greases will not have the sales appeal of greases having lighter colors. If certain colors are avoided which are obviously unsuitable, there remains a large range of colors, any one of which should prove satisfactory from the standpoint of sales.

Stringy Texture. A stringy texture seems to have an appeal from the standpoint of sales. This has been especially apparent in recent times as shown by greases containing rubber and poly-iso-butylene.

This stringy texture may be obtained by adding a few hundredths of a per cent of latex. The disadvantage is that appreciable amounts of latex decrease the fluidity of the grease at low temperatures.

Freedom from unpleasant odors. The addition of materials to a lubricant which have unpleasant odors is not desirable. Examples of materials which fall in this classification are fish oil, blown rape oil, sulfurized fatty oils, Stockholm Pine Tar (added to cover up less desirable odors), degras, etc.

There is also the possibility of adding materials of aromatic odor (*e.g.*, nitrobenzene, organic esters, etc.) to a grease, which lend a pleasant rather than unpleasant odor. The advantage of this sort of policy is that it may help to identify a product in the mind of the consumer.

Commercial Types of Lubricants and Modifications Which Might be used for Chassis Lubricants—Advantages and Disadvantages

A heavy fiber grease might be used very satisfactorily in a hot, dry section of country if resort were made to handle the lubricant with suitable grease guns. The grease, however, would prove entirely impractical for use at some service stations under severe winter conditions, because it could not be pumped about. If the climate were particularly subject to wet weather, complaints might be heard to the effect that the fiber grease was ruined by water. In view of the multiplicity of types of lubricants which deserve some consideration, these different types have been tabulated below. The advantages and disadvantages have been included in the table so that comparisons may easily be made. It is assumed, in every case, that the lubricant is sufficiently fluid that it is convenient for use as a chassis lubricant.

Lime Base Greases.

Type of Lubricant	Advantages	Disadvantages
1. Cup greases, prepared from fat. Low oil viscosity.	Water resistant. Temperature suspectibility, fair.	Not transparent. Not resistant to heat.
2. Fatty acid grease. Low oil viscosity.	Water resistant. Low temperature susceptibility. Transparency. More resistance to heat than cup grease.	
3. Lime base grease. High oil viscosity.	Water resistant.	High temperature susceptibility.
4. Graphite greases.	Advantages of cup greases.	Probably some increase in wear due to lapping action of graphite.
5. Lime base grease with mica filler, low viscosity oil.	Advantages of cup grease.	Increase in wear due to lapping action of mica.
6. Cold Sett greases.		High water content with tendency to become brittle at low temperature. No transparency, oxidize and gum badly.

Type of Lubricant	Advantages	Disadvantages
Soda Base Greases.		
1. Fiber grease prepared from light oil.	Heat resistant. Low temperature susceptbiility.	Fibrous texture, lack of transparency, contribute to poor appearance. Not water resistant.
2. Fiber grease prepared from heavy oil.	Heat resistant.	Fibrous texture, lack of transparency, contribute to poor appearance. Better water resistance.
3. High heat, milled grease, light oil.	Heat resistant. Low temperature susceptibility.	Poor water resistance.
4. High heat, heavy oil.	Transparency.	Poor water resistance.
Aluminum Base Greases.		
1. Aluminum stearate. Light oil.	Unexcelled transparency. Water resistant. Heat stability.	Tendency to break down on working. Change of pressure viscosity with temperature not quite so favorable as with calcium base greases.
2. Aluminum stearate. Heavy oil.	Water resistance. Heat stability.	Break down on working. High temperature susceptibility.
3. Aluminum Oleate.		
Lubricants Prepared With Filler.		
1. Asbestos floats with heavy oil.	Some degree of water resistance. Heat resistance.	High temperature susceptibility. Increased wear due to abrasive qualities of asbestos.
2. Asbestos floats with light oil.		Poor appearance. Grey paste obtained with low cohesion. Poor wear qualities.
Simple and Compounded Oils.		
High viscosity cylinder oil (temperature properties depend upon the oil).	Water resistance. Heat resistance.	Poor retention.
1. Cylinder oil compounded with Blown Rape oil.	Fairly low temperature susceptibility.	Emulsifies with water. Poor retention. A product heavy enough to be retained would have a high temperature susceptibility.*
2. Oil-asphalt blends.		
3. Oil and wax blends.		High temperature susceptibility.

* Tendency to emulsify would hold water in contact with metal, promoting corrosion. In case of cold weather, ice would be formed in the oil.

MATERIALS AVAILABLE FOR USE IN CHASSIS LUBRICANTS—Advantages and disadvantages of each.

The desirable properties for chassis lubricant have been discussed in some detail in the foregoing sections. The realization of these properties depends upon the proper selection of materials, control of the proportioning of these materials, and proper control of manufacturing operations. In view of the importance of the selection of materials, an outline has been made, of materials which could conceivably be used in a chassis lubricant.

Soaps.—The selection of soap stocks for preparation of a lubricant is very important since the questions of water resistance, heat stability, and resistance to bleeding are closely connected with the types of soap stocks used in making the greases. An outline of the characteristics of important soaps available for grease manufacture follows.

Base of soap	Properties imparted to grease by selection of particular soap base	Fatty component of soap	Property imparted to grease	Manufacturing Process	Important properties dependent on manuf. process
Na	Lack of water resistance due to tendency of soaps to emulsify. Heat resistance (High melting point, low tendency to separation. Tendency toward fibrous texture).	Non-drying fats. Example: tallow, lard oil.	Resistance to oxidation. Fibrous texture.	Open kettle, steam jacketed, using fats or acid and base.	Fibrous non-transparent grease.
		Drying fats. Example: fish oil.	Susceptible to oxidation.	Fire pot, high heat.	Anhydrous, transparent products. Fiber depends on fatty component.
		Non-drying fatty acids. Examples: oleic and stearic acids.	Resistance to oxidation decreased. Tendency to fiber formation in comparison with fats.	Cold set.	Smooth grease. High water content.
Ca	Lack of heat resistance. Since stability depends on hydration of soap in most cases. Water resistant. Smooth texture.	Non-drying fats. Example: tallow, lard oil.	Resistant to oxidation. Opaque grease (cup grease). Increased tendency to discolor. Oxidize. Decreased yield. Greater resistance to bleeding.	Autoclave method. Lime with fatty material finishing grease in open kettle. (Water essential).	
		Drying fats. Example: fish oil.		Open kettle. (Water essential). Cold sett. (Water essential).	High alkalinity. High water content. Resistance to boiling water in heavy grease.
		Non-drying fatty acids. Example: oleic and stearic acid.	Improved heat resistance.		
			Improved transparency due to lower water and glycerol content.		
Al	Stability to heat, though melting point is not particularly high. Water resistant. High tendency to break down upon working.	Stearic acid.	Smooth texture, exceptionally transparent.	Commercial soaps heated with oil.	Homogeneity dependent on regularity of cooling.
		Oleic acid.	Stringy texture.	Steam jacketed kettle.	

Method of Standardizing Pressure Gun Greases on Basis of Appearance

The evaluation of greases according to their appearance is an arbitrary evaluation, based upon personal opinions. As a guide in evaluation of the appearance of the lubricants, appearance may be considered as consisting of (1) color: reflected and transmitted light; (2) transparency; (3) texture. The following table lists these properties for the greases examined and includes a numerical rating in which (1) indicates the highest quality and (6) the lowest. The colors have been rated in the order in which they approach the rich, clean colors associated with highly refined mineral oils, highly transparent red in transmitted light and a rich green in reflected light. The transparency is taken as referring to absence of cloudiness rather than to depth of color. In texture good greases should be smooth and "buttery" and should not be so sticky as to indicate the excessive addition of rubber.

Pressure Gun Lubricants—Evaluated as to Appearance

Product	Color		Transparency	Texture	Appearance Av. Rating
	Reflected	Transmitted			
Aluminum stearate base lubricating glease	1 Green	1 Red	1 Clear	1 Very Smooth Sticky	1
Standardized pressure gun lube plus 38 per cent bright stock	1 Green	1 Red	2 Clear	1 Very Smooth Very Sticky	1.25
Standardized pressure gun lube plus 15 per cent bright stock, present formula	1 Green	1 Red	2 Clear	2 Smooth, Sticky	1.5
Standardized pressure gun lube plus 10 per cent bright stock	2 Green	2 Red	2 Cloudy	3 Smooth, Sticky	2.25
Experimental cup grease and 15 per cent bright stock	2 Green	2 Red	2 Cloudy	4 Smooth Not Sticky	2.50
Experimental dyed pressure gun lube	1 Green	1 Red	2 Cloudy	2 Smooth, Sticky	1.5
Experimental fluorescent green H. W. dyed cup grease	3 Green	3 Red	3 Cloudy	3 Smooth, Sticky	3.00
Experimental light cracked tar dyed cup grease	3 Green	4 Brown	3 Cloudy	3 Smooth, Sticky	3.25
Commercial pressure gun lube No. 5002	3 Green	3 Red	2 Cloudy	3 Smooth, Sticky	2.75
Commercial auto gun lubricant	3 Green	3 Red	3 Cloudy	3 Smooth, Sticky	2.75
Eastern pressure gun lube	3 Green	3 Red	3 Cloudy	2 Sticky, Smooth	2.75
U.O.C. Eastern pressure gun lube	3 Green	3 Red	3 Cloudy	4 Smooth Not Sticky	3.25
High pressure lube	5 Gray Brown	5 Same	4 Cloudy	2 Smooth, Sticky	4.00
S.C. grease chassilubricant	4 Light Brown	4 Same	3 Cloudy	5 Smooth Not Sticky	4.00
S.G. Hi-pressure	4 Dull Orange	4 Same	3 Cloudy	5 Smooth, Sticky	4.00
Q.S. pressure gun lube, superfine quality	5 Brown Black	5 Same	5 Cloudy	3 Smooth Not Sticky	4.50
P. No. 310	5 Brown Black	5 Same	5 Cloudy	3 Smooth Not Sticky	4.50
T. chassis grease	6 Muddy Yellow	6 Same	6 Very Cloudy	6 Not Smooth Very Sticky and Stringy	6.00

Oils

The oils used in a lubricant are important since they are largely responsible for the lubricating qualities. Soaps, fillers, etc., are carriers of the lubricating oil. The characteristics of an oil in service may, of course, be altered a great deal by the addition of E. P. bases, fatty materials or other compounds.

The important characteristics that an oil imparts to a lubricating grease are summarized in the following table.

Characteristic of oil	Characteristic imparted to grease
Low viscosity	Relatively low change of pressure viscosity with temperature.
High viscosity	Relatively high change of pressure viscosity with temperature.
High viscosity index	Relatively low change of pressure viscosity with temperature.
Low viscosity index	High change of pressure viscosity with temperature.
Transparent oil	Tends toward transparent grease.
Fluorescent oil	Fluorescent grease.
Good E. P. qualities	Good E. P. properties. However, grease may have good E.P. qualities in spite of rather poor E. P. qualities of the oil if lead soap or certain fillers are added.
Color	The color of the oil is usually imparted to the grease since the soaps are usually colorless. However, soaps prepared from dark fats or fatty acids may discolor a grease.
Susceptibility to oxidation	The grease will also tend to oxidize unless temperatures of operation are low.

Fillers

Fillers constitute a third class of materials which might be put to use in a chassis lubricant. While some of them may have certain lubricating qualities, most of them have mild abrasive qualities which increase wear.

Examples of available fillers, with comment on them, are given below.

Filler	Comment on the use of the filler
Powdered mica	Lapping action, increases wear.
Powdered graphite	Probably some lapping action. Some improvement in E. P. qualities are obtained, however.
Asbestos floats	Lapping action, increases wear.
Cellulose	Difficult to disperse, and the cellulose is extremely sensitive to water.
Zinc oxide	There is little information available as to the effects of zinc oxides or other oxides as fillers. Probably some increase in wear would result.

Miscellaneous Materials Which Might be Used in Chassis Lubricants

The following miscellaneous materials are available for compounding chassis lubricants, though in most cases they have properties which render them objectionable. Comments on the materials are contained in the table below.

Material	Comment on suitability for use as chassis lubricant
Blown oils	May improve V. I. of the oil. However, the blown oils are expensive, and the odors are usually objectionable.
Asphalt	Black color not particularly desirable for chassis lubricant.
Petrolatum	Tendency to harden lubricant at low temperatures is objectionable.

Perfumes	Use of perfumes is largely a matter of sales policy. The present policy of some manufacturers is to avoid their use.
Fats	Fatty materials may possibly contribute to oiliness. However, oiliness is a factor only in high speed bearings. Since no question of high speed bearings is involved in chassis lubrication the use of expensive fatty material does not seem justified.
Gums	Typical gums are usually not soluble in oil. Even if it were possible to get them into solution it is probable that excessive oxidation would take place.
Glue	The emulsification of a glue of protein character in a grease contributes to excessive water content, as somewhat emulsified appearance and an unpleasant odor. No particular advantage in the way of changed texture seems to be apparent.
Latex	A few hundredths of a per cent of latex in a grease results in a stringy texture for a grease. This seems to be advantageous from the standpoint of sales. However, the latex content reduces the fluidity of a lubricant.
Polymerized butylenes	These are now available in commercial quantities at prices which encourage their being used in chassis lubricants.
Sulfurized fatty oils	Has advantage of a flat viscosity temperature curve. However, the extreme pressure properties imparted are not necessary for most chassis lubrication and this E. P. base is expensive.
E. P. bases chlorinated compounds	Some conditions apply as for sulfurized E. P. bases except that advantages with regard to temperature susceptibilities are considerably smaller.
Waxes	Contribute to hardening of lubricant at low temperatures.
Fatty acids	It is probable that fatty acids, in high concentration, contribute to corrosion of metal parts, especially of certain types of alloys.
Free alkali	Excess of free alkali seems to contribute to corrosion.

Testing

The results of retention tests on spring shackles should be interpreted, keeping in mind that they are subject to a considerable degree of experimental error. The tests as a whole are very difficult to reproduce. This is due to a number of factors.

Air bubbles are apt to get in the grease. This factor in general will not be reproducible.

Temperatures were not always the same. An effort was made to keep the temperatures as close to 75° F. as possible. However, this was not always possible. The laboratory was not equipped to serve as a constant temperature room.

In the assembly of the apparatus slight differences in the relative position of parts may prove a factor in the retention of the grease.

Texture does not seem to be connected with the kind of soap (exclusive of the base) used in the grease, since results of analysis of typical stringy grease show no particular differences from those for an ordinary lime base grease. The stringy texture is, in most cases, probably due to the addition of a very small percentage of materials either identical with or similar to latex. It is difficult to show the presence of latex by analysis in the concentration of a few hundreths of a per cent. This is partly due to difficulty of avoiding some slight emulsification with water in the breaking down of the grease so that subsequent observations in turbidity tests are not very reliable.

Although it is difficult to show the presence of latex in stringy high pressure lubricants by analysis, the physical properties produced by the stringy texture and decreased rate of flow of the grease can readily be duplicated through the addition of latex to the ordinary smooth-texture calcium base grease.

The flow data in the table should be considered in the light of their relationship to the difficulties met in the use of the greases in cold weather at service stations. Sufficient data has not been obtained in the table above to differentiate between yield values and viscosity of the greases as factors in the rate of flow. However, the data illustrate several points with regard to the flow of the greases:

When greases of similar oil viscosities are compounded, flow rates at low pressures correlate well with the penetrations of the greases.

These flow rates decrease rapidly with decreased temperature. Though the rate of decrease of flow is roughly parallel the aluminum greases seem to have a steeper slope than the typical calcium base grease.

The addition of latex or similar material to the grease materially reduces flow rates at low pressures.

A further point with regard to flow rate at low temperatures is evident if high viscosity oils are used so that high grease viscosities are obtained at low temperatures. However, most commercial chassis lubricants have avoided the difficulty by using only comparatively low viscosity oils.

Assuming a fairly constant oil viscosity, the flow data suggests that a practical lower limit for the penetration of grease is a necessary consequence of the rapid decrease in flow rate of these greases with decrease in penetration. The lower limit of penetration will be determined by the lower temperature at which the grease is to be used. For fairly low temperatures, probably the penetration should not be below 320, since complaints have been received on standardized pressure Gun Lubricant C, where batches of grease have apparently "set-up" in storage and penetrations barely below 320 were realized.

In connection with study of commercial chassis lubricants the following table of results on the properties of commercial pressure gun lubricants is of special interest. It will be noted that all of the greases are either calcium, sodium or aluminum base greases. Oil viscosities range from 35 to 3100 S.U. seconds at 100° F.

[71]As with most lubricating grease problems, the viscosity of the mineral oil content, the consistency of the grease while being applied and while performing within the bearing, and the quality and amount of soap may all influence results obtained in service. For this reason, precise service tests for establishing the value of these items are very difficult. That much research remains to be done can be inferred from the fact that one finds on the market different gun greases manufactured with calcium, aluminum, and sodium soaps. Thus, the "experts," who in this particular case must be the grease manufacturers, do not agree. Perhaps a condition exists which is similar to that occurring in the food and clothing industries: that

[71] Reprinted from the article: "Laboratory Service Testing of Automotive Lubricating Greases," by E. N. Klemgard, *Ind. Eng. Chem.*, **8**, 83 (Mar. 15, 1936).

which pleases one is rejected by another for psychological reasons. Technically it should be possible to determine which of two greases is best adapted to service in a given chassis bearing under given service conditions. However, the term "given service conditions" is a limitation which greatly restricts our general problem, and atmospheric temperatures, rain, dust, ice, wear, and design are variables which stand in the way of simplified service testing. The following method of testing has been worked out with a view to including the more normal types of operating conditions and thus forms the basis of opinions on retentivity as related to oil viscosity, consistency, type of soap base, and quantity of soap.

Testing Pressure-Gun Greases for Retentivity

Either plain pin or screw-type shackle bolts have been adopted for the testing of pressure-gun greases in the apparatus shown in Plates XVII and XVIII because they present the most important current shackle bolt lubri-



Plate XVII.—Modified apparatus for determining retention properties. Produces more rapid vibration than device shown in Plate XVIII.

cation problems. A model A Ford front spring with shackles and a Buick series 33-50 rear spring shackle, representing characteristic plain pin and screw-type shackle bolts, respectively, make it possible to study retentivity.

The actuating cam shown in Plate XVIII was driven by a 3-horsepower electric motor through a 30 to 1 reduction gear. This mechanism imparts a 2.5-cm. (1-inch) deflection to the spring 60 times per minute. The cam is so designed that after maximum lift it leaves the roller follower and the spring returns freely to normal position. The deflection may also be obtained by means of a suitably designed cam as in Plate XVII.

Before each test the spring shackles were completely disassembled and washed thoroughly with benzene, care being taken to remove all the old grease from the grease ducts and fittings. After thorough drying, the shackles and bolts were assembled, making certain that all interchangeable parts were replaced in the positions from which they were taken.

A small screw-type grease gun was filled with the grease to be tested and weighed on a balance to the nearest 0.05 gram. Each shackle was charged with grease until it showed signs of being forced out at the ends of the shackle bolt bushings. The filling gun was then reweighed and a small pad of cotton was used to wipe off the

TABLE 9—Physical and Chemical Tests on Commercial Pressure-Gun Lubricants.

Lubricant Designation	Penetration at 77° F. Unworked	Worked	Dropping Point Ubbelohde	Kind of Soap	Ash %	S. U. Viscosity of Mineral Oil Content at 100° F.	Texture	Flow of Grease through Saybolt Universal Viscosity Tube 35° F. G./min.	77° F. G./min.	100° F. G./min.
355 Al 400	...	385	89	Aluminum	..	400	Smooth	0.5	9.7	...
375 Ca 301	362	375	97	Calcium	1.04	301	Smooth	...	...	...
340 Ca 319	347	340	90	Calcium	1.06	319	Smooth	...	...	...
370 Ca 350	375	370	90	Calcium	..	350	Smooth	...	...	...
314 Ca 868	315	314	96	Calcium	1 61	868	Smooth	...	...	...
336 Ca 335	323	336	...	Calcium	..	335	Stringy	0 3	2.0	15.1
318 Ca 460	320	318	...	Calcium	1.34	460	Smooth	0.7	11.0	25.2
345 Ca 310	342	345	...	Calcium	1.7 max.	310	Smooth	1.4	19.8	...
354 Ca 315	354	354	...	Calcium	1.7 max.	315	Smooth	2.0	22.8	...
335 Al 290	325	335	...	Aluminum	..	290	Smooth	0.3	7.9	37.0
321 Ca 3000 (blown saponifiable oil)	316	321	90	Calcium	..	3000	Stringy	...	1.1	...
319 Ca 35 (latex)	309	319	88	Calcium	..	35	Stringy	...	0.2	...
227 Ca 300	207	227	92	Calcium	..	300	....	...	...	...
318 Ca 500 (polyisobutylene)	...	318	95	Calcium	..	500	....	...	...	...
305 Ca 310 (milled)	305	305	91	Calcium	..	310	....	0.5	4.4	...
285 Ca 500 (latex)	...	285	95	Calcium	..	500	....	0.2	2.2	...
282 Ca 425	...	282	...	Calcium	..	425	....	0.3	5.0	...
350 Na 3100	...	350	135	Sodium	..	3100	....	Trace	1.1	...
450 Na 2500	...	450	...	Sodium	..	2500	....	...	...	...
390 Al 3000	...	390	101	Aluminum	..	3000	....	Trace	...	...
400 Al 1000 (app.)	...	400	...	Aluminum	..	1000	....	...	...	...
350 Al 1000 (app.)	...	350	...	Aluminum	..	1000	....	...	...	...
Oil 2000/100° F.	...	...	...		..	2000	....	...	...	...

excess grease. The increase in weight of the cotton, subtracted from the loss of weight of the gun, gave the weight of grease charged to the shackle. The clearance between shackle and bushing was maintained at approximately 0.02 cm. (0.008 inch).

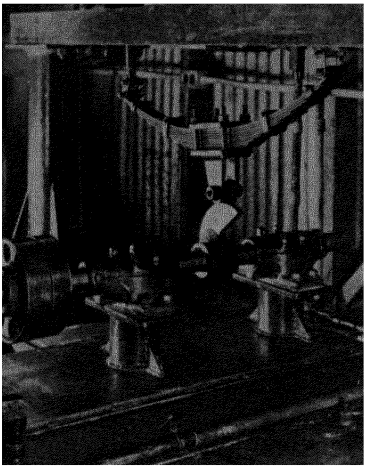


PLATE XVIII.—Apparatus for determining retention properties of pressure-gun greases in spring shackles.

One form of grease-retention test consisted of taking a set of six readings during a 2-hour test period, the first four being taken at the end of 15-minute periods and the last two at the end of 0.5-hour periods. The readings were obtained by shutting the motor down and wiping the edges of the shackle bolts and bushings thoroughly with a weighed wad of cotton, and then measuring the increase of weight of the cotton.

An examination of Figures 1, 2, and 3 reveals that neither the kind of soap base, the viscosity of the oil content, the penetration value, nor slight variations in soap content alone make one product better than another. Probably the combination of these factors and the modifying agents present which influence adhesion, aëration, and stability of the grease while in the shackles are the important items in determining the retentivity of a pressure-gun grease.

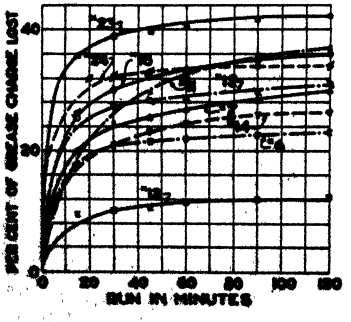


FIGURE 1.—Grease Retention Tests. Front spring with plain bushing shackles on chassis lubricant tester. Charge, 2.80 to 2.90 grams of grease.

Grease 305 Ca 310 (Figure 2) shows a loss of 22 per cent in the 2-hour retention test (first number indicates worked penetration; second, soap base symbol; third, Saybolt Universal viscosity of oil content at 37.78° C..

100° F.). Grease 354 Ca 315 is similar with regard to penetration, type of soap, and mineral oil viscosity, but shows a loss of 28.8 per cent. A possible explanation of this difference is that the first grease has been milled to

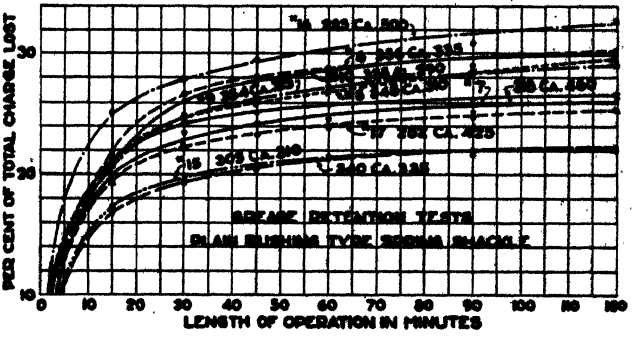


FIGURE 2.

stabilize it, thus increasing its retentivity. It therefore may be concluded that the usual tests for penetration, soap base within certain limits, and oil viscosity do not accurately indicate the degree of retention which may be expected from a given pressure-gun grease. It also follows that further research towards establishing a correlation between retention, pressure viscosity, and stability of the grease film within the bearing are greatly needed.

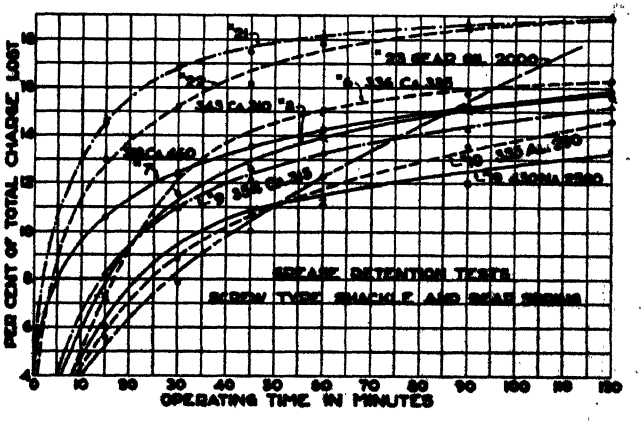


FIGURE 3.

RETENTIVITY OF PRESSURE-GUN GREASES IN PRESENCE OF WATER

In order to obtain information as to whether or not an aluminum stearate-base grease was more resistant to the action of rain water than a lime-base grease, the retention tester discussed above and shown in Plate XVIII was equipped with a water-spray apparatus.

Alterations made in equipment consisted in the addition of a device for spraying water upon the shackles, and the building of a small tank about the apparatus to collect the water sprayed upon the shackles. The spray apparatus consists of two atomizers dipping into a tray of water. The two atomizers are operated by air under pressure, the intensity of the air jet being controlled through the use of an air valve and a pressure gage. The spray apparatus was placed in position so that a fine spray of water could be thrown up on the shackles.

Modifications of this device have undoubtedly been devised by others for splashing, dripping, or spraying water onto the shackles. The device as used is a simple method of obtaining a gentle washing action analogous to what might be met in service in stormy weather. The rebound of spray from the side of the tank and other parts of the apparatus results in water contacting the shackles from every direction from which water might come in actual service. The test conditions can also be fairly well reproduced by controlling the air pressure and the level of the water in the tray.

TABLE 10—Retention of Grease

(Lubricant tester as shown in Plates XVII and XVIII plain shackles, 2-hour period, fine spray of water)

Kind of soap	Grease 335 Al 283 Aluminum	Grease 354 Ca 327 Calcium	
Per cent of soap	10.2	7.9	
Worked penetration	335	354	
Oil viscosity, S. U. at 100° F.	283	327	
Charge of grease, grams	2.81	2.81	2.88
Total water spray on shackles, liters	12.50	12.15	12.20
Water wiped from bearing surfaces and grooves, grams	0.141	0.235	0.302
Grease wiped from bearing surfaces and grooves, grams	0.160	0.149	0.179
Grease obtained by pressure-benzene treatment	1.241	1.320	1.302
Total grease retained, grams	1.401	1.469	1.481
Percent retention	50	52	51

The spring, shackle bolts, and the crossbar from which the spring and shackles were suspended were assembled outside the tank which surrounds the lift cam. An excess of grease was forced through the Zerk fittings of the apparatus and measurements made as previously described.

A 2-hour test period was selected. This was consistent with previous tests carried out in the absence of water, in which the length of the test period was 2 hours. Except for the spray playing on the shackles, the conditions of the test were identical with those of tests carried out on a dry basis. It was not possible to carry out observations on the amount of grease leaving the shackles at 15-minute or 0.5-hour intervals so as to obtain a retention-time curve.

The problem of recovering the grease from the water sprayed on the shackles was given consideration. However, this seemed impractical since the surfaces of the interior of the tank were large and the surfaces of the springs were irregular and likely to retain a certain amount of grease in crevices. The removal and measurement of the grease retained on the

bearing was resorted to since the surface areas were smaller and easier to work with.

The results shown in Table 10 were obtained in the test.

The results on the two greases are in fairly close agreement, except in the amount of water removed from the shackles. However, these water figures are not very reliable, since losses are bound to occur through evaporation of water and probably also the loss of droplets of water from the shackles in removing shackle bolts. Neither grease showed any evidence of emulsification in the shackles as the result of the entrance of water.

TABLE 11—Chemical Analyses and Laboratory Tests; Miscellaneous Commercial Pressure Gun Greases

	So—No. 0	Al	St—Cal H. P.
Physical Tests:			
Appearance	Semi-solid dark Cup No. 0	Semi-solid dark Cup No. 0	Semi-solid dark Cup No. 0
Dropping point	94° C.	89° C.	91° C.
Penetration at 77° F. (A.S.T.M. worked)	335	350	360
Odor	Not offensive	Not offensive	Not offensive
Composition (Per cent by weight)			
Soap	10.70	8.0	9.10
Moisture	1.10	.80	.80
Oil (by difference)	87.92	91.14	90.07
Free alkali	.28	.06	.03
	100.00	100.00	100.00
Ash:			
Percent ash	1.56	.92	1.06
Calcium (CaO)	95.0	90.5	90.0
Iron and alumina (R_2O_3)		3.0	1.8
Undetermined	5.0	6.5	8.2
	100.00	100.00	100.00
Fatty Acid from Soap:			
Acid value (Hgms. KOH)	197	194.0	193.5
Iodine value	75.2	67.0	73.0
Color	Brown	Brown	Brown
Melting point	36° C.	35° C.	33° C.
Mineral Oil:			
S.U.V. at 100° F.	496 approx.	563	549
Gravity ° A.P.I.	22.0	20.7	21.0

The comparison between these two greases seems to be a fair basis for comparison of calcium- and aluminum-base chassis lubricants under wet conditions, since the oil viscosities, consistencies, and soap contents of the two greases were similar.

A complete investigation of the effect of water on shackle lubrication would of course include an investigation of screw-type shackles as well as plain shackles, and also the effect of different methods of bringing water in contact with the shackles. The effect of high-pressure washing should be of special interest in this regard. The effect of oil viscosity, soap content, and glycerol content on retention under wet conditions might also be

investigated. However, since the results so far obtained show so little difference in the behavior of calcium- and aluminum-base greases in the presence of water, it is doubtful if much information of value would be obtained by further work on a comparison of aluminum- and calcium-base greases, although a comparison with sodium-base grease would no doubt be of interest.

Ordinarily, it is considered that temperature conditions met with in pressure-gun grease service are so low that high melting point soda-base greases are unnecessary. Since speeds and load are low, extreme pressure agents in most cases appear to be unnecessary.

		ZN.—H.P.
Physical Tests:		
Appearance		Light greenish brown
Odor		Fatty
Penetration at 77° F. (A.S.T.M. unworked)		371
Penetration at 77° F. (A.S.T.M. worked)		347
Dropping point ° C.		85
Composition (Per cent by weight):		
Soap		11.60
Oil		86.81
Moisture		0.75
Free alkali		0.07
Free fat		0.29
Insoluble (fillers)		0.01
Gain or loss		0.47
	Total	100.00
Mineral Oil Extracted:		
Color		Opaque
Gravity ° A.P.I.		20.7
S.U.V. at 100° F.		335
Ash, Total Per cent on Original:		*1.24*
Acid insoluble		Trace
Iron and aluminum as (R_2O_3)		1.41
Calcium oxide as (CaO)		91.00
Undetermined		7.59
	Total	100.00
Fatty Acids from Soap:		
Iodine value (Hanus)		52
Acid value, (mg. KOH)		207
Melting point, ° C.		40
Color		Light Brown

Automotive Waterproof Greases

Formula for Standardized Waterproof Grease

Constituents	Gallons	Pounds Net	Per Cent by Weight
Stearic acid	20	150.00	15.20
Oleic acid	20	149.72	15.17
Hydrated lime		67.00	6.79
Water	6	49.97	5.06
1000 pale oil	74	569.88	57.77
Oil of orange		.10	.01
		986.67	100.00

PROCEDURE

Prepare the lime mixture in a 50-gallon open head tank using Lightnin Mixer:

1000 Pale oil (temperature 90-100° F.)	34 gallons
Hydrated lime	67 pounds
Water	6 gallons
Oil orange, dye solution (contains 0.9 ounces of oil orange per gallon of 1000 pale oil)	2½ gallons

Mix the following in an open 100-gallon tank:

Stearic acid should be as hot as possible, 140-160° F., in order to prevent separation of stearic acid crystals.)

Stearic acid	20 gallons
Oleic acid	20 gallons
1000 Pale oil	40 gallons

Charge into grease kettle.

Run the lime mixture (Item No. 1) into the grease kettle equipped with agitating paddles, quickly and completely, while stirring (paddles running).

SAPONIFICATION

Continue agitation for one half to one minute. After this period of stirring the grease will have become quite thick, and must be run at once into 50-gallon grease tanks, where the saponification continues without further stirring. The grease on standing may attain a temperature of 175° F., or slightly higher. After standing for 24 hours, and the temperature has subsided, a pound sample is sent to the laboratory for test, and if found to meet specifications the grease is packed by hand into the shipping containers, one and five pound cans.

PROPERTIES

Waterproof grease is a heavy, plastic, yellow-orange colored grease intended to seal and lubricate automobile waterpump bearings and packing glands. It does not melt or dissolve in hot water.

SPECIFICATIONS

Waterproof grease must conform to the following requirements:

Percent free alkali, $(Ca(OH)_2)$	0.50–1.75
Percent water (W. B. D.)	5 to 7
Penetration, worked, A.S.T.M.-D217-27T	160–210
Boiling water test*	OK

* A cube of the grease approximately one inch square shall not be appreciably deformed, melted, or dissolved after being boiled with tap water for twenty minutes.

EXPERIMENTAL FORMULAE

Table 12 shows results for experimental batches made with different proportions of lime, oleic and stearic acid indicate clearly the need for using an appreciable amount of stearic acid and a large excess of lime.

According to Matthews,[72] a serviceable automotive water pump grease should comply with the following broad requirements:

Percent calcium soap base	24.0 max.
Percent water	1.0 max.
A.S.T.M. worked penetration	160–190
Dropping point ° F.	210 min.
S.U.V. of oil content at 100° F.	100 min.
Color of oil, A.S.T.M.	3

[72] *Proc. Am. Petroleum Inst.*, **III, 16,** 65 (Nov., 1935).

TABLE 12.—Formulae and Test Results, Experimental Waterproof Greases

Batch No.	No. 1	No. 2	No. 3	No. 4-A	No. 5	No. 6	No. 7	No. 8	No. 9	No. 10	No. 11	Stand. Waterproof Grease	G. M. Specifications
Unworked penetration at 77° F.	185	170	180	190	190	227	240	220	157	182	168	167	After 5 min. and 1 min. stirring 16.5
Worked penetration at 77° F.	268	258	260	260	265	328	335	330	326	221	346	200	22.5 min.
Percent filler	Trace	Trace	Trace	Trace	Trace					.01		Trace	None
Percent free alkali as $Ca(OH)_2$	.015	.09	.13	.15	.5					0.37		.81	0.40% max.
Percent water	6.0	6.0	6.0	6.4	6.2					6.0		5.8	...
Percent grit	None	None	None	None	None					None		None	0.05% wax
Corrosion (cu. strip)	OK	OK	OK	OK	OK					OK		OK	OK
Dropping point ° C.	114	100	100	106	102					86		110	210° F. min.
Boiling water test	OK	OK	OK	OK	OK					Not OK		OK	...
Percent soap													20.0-25.0%
Odor				OK						OK		OK	Neut.
Permanence	No Sep.	No Sep.	No Sep.	No Sep.	No Sep.					OK		OK	No sep. or hardening in 3 months.
Stearic acid, per cent	10.5	10.5	10.5	10.5	10.5	13.0	15.0	17.0	21.0	...	21.0	15.49	
Oleic acid, per cent	10.5	10.5	10.5	10.5	10.5	8.0	6.0	4.0	...	21.0	...	15.48	
Powdered lime, per cent	2.9	3.15	3.36	3.57	3.8	3.6	3.6	3.6	3.6	3.6	3.9	6.30	
Water, per cent	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.00	
1000 pale oil, per cent	71.1	70.85	70.64	70.43	70.2	70.4	70.4	70.4	70.4	70.4	70.4	57.72	
Oil of orange												0.01	

A grease made on the following formula will meet these requirements:

	Per Cent by Weight
Beef tallow	20
Hydrated lime	3.2
Water	1.0
150 Texas neutral	75.8

(If a 210° F. minimum dropping point is not obtained with the beef tallow available it may be necessary to substitute an appropriate amount of stearic acid for an equivalent weight of the tallow.)

Waterproof Grease Purchase Specifications

The following specifications have been proposed by five different manufacturers:

	1	2	3	4	5
Percent soap	24	24/26	20/25	35.44	24
Kind of soap	Ca	Ca	Ca	Lime	Ca
Percent moisture, max.	1	1.6	...	...	1.5
Percent mineral oil	74	74.4/72.2	...	45.82	74½
Gravity A.P.I.	27/28	25.7	...	26.0	26/33½
Flash	375	410	...	...	350–370
Fire	420	470	...	...	410/430
S.U.V. at 100° F.	125/135	285	85	185	100/105
S.U.V. at 130° F.	...	...	...	95	...
S.U.V. at 210° F.	42/44	...	...	45	...
Special vis.	...	...	...	...	...
Color	3	...	...	3	2
Pour	plus 10	plus 25	...	23	30–35
Color of lub.	Med. Yellow	Dk. Yellow	...	Yellow	Yellow
Penetration, worked	160/190	...	165/185 75/80 (unworked)	...	250/265
Free alkali	0.3	...	...	...	.25
Free acid	0.1	...	...	...	...
Filler	...	...	...	...	...
Dropping point	208/210	...	...	...	215
Percent ash as sulfates	6	...	...	11.14	...
Corrosion	Neg.	...	...	...	...
Odor	Myrbane	...	...	...	...

G. M. Purchase Specifications for Water Pump Grease (GM-4614-M)

General: Grease covered by this specification shall be a homogeneous combination of refined mineral oil and calcium soap.

Filler: Shall not contain filler such as clay, talc, whiting, asbestos, wood fiber, etc.

Consistency: The Worked Consistency (A.S.T.M.) after five minutes and one minute stirring shall be minimum 16.5 mm., maximum 22.5 mm. (165–225).

Soap: The total soap content shall be from 20.0 to 25.0%. Not less than 93% of the soap shall be calcium (and magnesium) soap. A small amount of caustic soda may be used if desired for completing saponification, but the sodium soap must not exceed 7% of the total soap.

Alkalinity: ($Ca(OH)_2$) Maximum 0.40%.

Uncombined Lime: Maximum .25%. The lime used in preparing the soap shall not contain more than 7% of magnesium oxide and not more than 2% of gritty non-soap forming substances.

Melting Point: (Over Mercury) Minimum 210° F.

Odor: This grease shall not be perfumed and shall be free from disagreeable odor or rancidity.

Permanence: The soap and oil shall not separate nor shall the grease harden at reasonable temperatures within a period of three months.

Corrosion: This grease shall not corrode any metal used for machine construction.
Methods of Test: Tests shall be made in accordance with methods described in G. M. Standards.
Notes: This grease is intended and recommended for lubrication of water pumps.

Five Commercial Automotive Water Pump Greases

An analysis of these greases showed the following results:

Physical Properties:				
Appearance	Granular Orange	Smooth Yellow	Sl. Grainy Brown	Smooth Lt. Brown
Odor	Soapy	like nitrobenzene	Soapy	Soapy
Dropping point (° C.)				
Ubbelohde	106	102	91	85
Gillette	100	100	91	98
Penetration at 77° F.				
unworked	105	125	148	141
worked	165	173	206	198
Corrosion (Cu. strip 3 hrs. at 212° F.)	OK	OK	OK	OK
Boiling water test	OK	OK	Not OK	Not OK
Separation test (3 hrs. at 110° C.)	None	None	None	None
Percent increase in vol. at 210° F.	6.4	4.6	12.0	9.0
Composition (Per cent by weight):				
Soap	32.6	26.8	28.3	26.2
Oil	60.0	68.5	62.5	51.0
Moisture	5.8	2.7	5.6	13.2
Free acid (as oleic)	...	0.56	0.84	...
Free alkali (as $Ca(OH)_2$)	2.2	...	...	4.2
Ash:				
Per cent	5.01	2.92	4.94	8.39
Analysis CaO	98.9	97.3	61.8	63.0
P_2O_5	0.5	1.6	2.5	6.4
MgO			34.4	
Mineral Oil:				
Color	Opaque	7—	Opaque	4½
S.U.V. at 100° F.	910	156	99	94
S.U.V. at 210° F.	66.8	42.0	32.6	32.5
Gravity	21.9	24.7	29.2	32.1

Physical Tests:	
Appearance	Yellow
Odor	Nitrobenzene
Penetration, A.S.T.M., unworked @ 77° F.	108
Penetration, A.S.T.M. worked @ 77° F.	175
Dropping Point ° C.	103
Boiling water test	OK
Composition, (per cent by weight):	
Soap	26.02
Oil	67.15
Moisture	3.00
Free alkali, (as $Ca(OH)_2$)	0.11
Free fat	1.60
Insoluble impurities	0.02
Loss in analysis	2.10
Total	100.00

Oil Extracted:

Color, N.P.A.	4
Gravity ° A.P.I.	24.0
S.U.V. at 100° F.	160
S.U.V. at 130° F.	84
Flash, Cleveland Open Cup, ° F.	380
Fire, Cleveland Open Cup, ° F.	430

Ash, Total Per Cent on Original: 2.90

Acid insoluble	0.69
Iron and aluminum, as (P_2O_3)	1.17
Sodium carbonate	None
Calcium oxide	96.60
Calcium sulfate	None
Undetermined	1.54
Total	100.00

Fatty Acids, from Soap:

Iodine value (Hanus)	45
Acid value, mg. KOH	195
Melting point ° C.	42
Color, N.P.A.	4½

Five Commercial Water Pump Greases

The characteristics of these greases are as follows:

Date	10/15/29	4/25/31	6/1/31	6/8/31	6/5/31
Penetration unworked	——	93	117	120	132
Penetration worked	170	272	143	167	200
Dropping point ° C.	90	110	94	91	110
Percent soap	24.5	28.6	34.0	35.7	33.4
Percent oil	62.3	62.4	46.3	44.0	57.69
Percent water	7.0	2.5	14.0	5.2	6.0
Percent free alkali	0.5	4.2	0.4	14.8	1.39
S.U.V. at 100° F.	——	364	106	110	1,000
Boiling water test (20 minutes)	Not OK	OK	OK	Not OK	OK

Marine and Automotive Waterproof Grease

The outstanding feature of the analysis given here, is the presence of free magnesia and magnesium soaps. This product has, however, given good service for more than twenty-five years.

Physical Tests

Appearance	Lt. Yellow Solid Lub.
Melting point ° C.	90
Penetration at 77° F. (A.S.T.M. worked)	170
Odor	Soapy

Composition (per cent by weight)

Soap	24.5
Water (W.B.D.)	7.0
Free fat	5.7
Free alkali (CaO)	.5
Oil (by difference)	62.3
	100.0

Ash	5.4
Magnesium (MgO)	38.2
Calcium (CaO)	54.5
Iron	Trace
Undetermined	7.3
	100.0%
Fatty acids from soap	
Acid value (mgms. KOH)	190
Iodine value (Hanus)	56.4
Melting point	32° C.
Mineral Oil	
Gravity (° A.P.I.)	29.8
Melting point	30° C.

Gummy Deposits in Automobile Radiators

During the past few years, since grease cups at the water pump were replaced in many makes of cars by high-pressure grease fittings, there has been much talk of a "brown gummy substance" which appears to clog radiators. Anti-freezes have been blamed for the formation of this brown gum, and many radiator specialists will insist that anti-freeze causes this trouble.

A laboratory which has worked on cooling systems for several years, recently completed an investigation of this problem. It found that the common anti-freezes, such as alcohol, glycerin and ethylene glycol, have nothing to do with the trouble, if they play any part at all, they help to prevent the trouble.

It was found that:

Overlubrication of the water pump and the use of the wrong type of grease, causing introduction of large amounts of grease into the cooling system followed by the action of this grease on rubber hose connections in the presence of heat, rust, and water, is the primary cause of this peculiar type of scale formation.

The rubbery formation was found during the test work in both water and the common anti-freeze mixtures.

Some of the factors influencing the rate of this formation are: Temperature, degree of aeration of coolant, type and condition of hose, impurities in coolant and amount of rust or sediment present.

The formation of such gummy accumulations can be prevented by exercising proper care in lubricating the water pumps on motor cars, and by the use of a "waterproof" type of grease.

On the basis of the test work done on this subject, it appears that the accumulations of the rubbery scale sometimes found in radiators are caused by frequent over-greasing of the water circulating pump, resulting in the introduction of large amounts of grease into the cooling system. This grease in the presence of rubber hose, heat, rust, and water or anti-freeze solutions is then converted to the rubbery, spongy scale found. The factors noted above are listed in the apparent order of their importance in causing the scale formation.

In the test work samples of the scale were analyzed and found to consist roughly of about 80 per cent organic matter and 20 per cent ash. The ash

consisted chiefly of rust, an appreciable amount of calcium salts and traces of carbon, magnesia and silica. The relatively large amounts of calcium were probably from the grease.

The first of a series of tests was made by subjecting samples of a light grade of grease, commonly used for chassis lubrication, to the actions of various coolants in the presence of heat, aeration, cast iron, radiator metals, and rust. Distilled water, tap water containing large amounts of salt impurities, and anti-freeze solutions of 50 per cent concentration were the coolants tested. In these tests no rubber hose connections were present. The tests were run for a total of 300 hours, about 250 hours at 190° F. and 50 hours at room temperature. During the entire period the grease showed no appreciable change. While the test solutions were hot, the grease was fluid and oily and floated on the surface of the liquids. When cold, the grease returned to about the same consistency as originally. In view of the fact that the grease, under these test conditions, showed no indications whatsoever of forming the rubbery scale, the tests were discontinued. The conclusion to be drawn was that heat, aeration, rust, hard or soft waters, anti-freeze, or impurities such as may be present in cooling systems, cannot alone cause the transformation of grease or oil to the gum-like scale.

A second series of tests was made by heating mixtures of 20 per cent iron oxide and 80 per cent grease in a stream of hot air. Two such mixtures were maintained at 220° F., and two at 250° F., in a strong current of air for 150 hours. At the end of this time all of the mixtures had darkened considerably and had become appreciably thicker or more viscous. However, all retained their greasiness and when heated in water become fairly fluid. These tests indicated that the greases contain no readily oxidizable oils, and that heat and aeration alone will not cause gum formation.

The final tests were made to determine the action of the grease on rubber hose connections. It is a known fact that excessive amounts of certain oils and greases in coolants have marked solvent actions on hose connections, causing the rubber to become soft and spongy.

The best means of preventing the occurrence of this condition in cooling systems is by exercising more care in lubricating the water pumps and by the use of a suitable grease. Pump bearings can only accommodate a small amount of lubricant, any excess usually being forced through the bearing into the coolant, and soft greases that are non-resistant to hot water may be drawn into the cooling system by pump vacuum. If circulating pumps are properly aligned and if the shafts, bearings and packing glands are in good condition, so that the excessive tightening of the packing nuts is not necessary, only a small amount of grease should be needed. This is, of course, contingent on the use of the proper grease, which is the waterproof type. Among the various makes of waterproof greases available on the market, the graphite-base type using electric furnace abrasive-free amorphous graphite is one of the most effective, as the graphite content is not affected by hot water or anti-freeze solutions and remains in the bearing after ordinary greases are gone.

A laboratory service testing device, making use of Chevrolet or other

makes of water pumps, is shown in Plate XIX. By selection of a motor pulley of suitable size, pumpshaft speeds may be attained of 500 to 2000 R.P.M. Water at temperatures up to 100° C. (212° F.) or various anti-freeze mixtures which may have a detrimental effect on the lubricating film are placed in the rectangular metal boxes on the sides of which the pumps are mounted. Practical tests and comparisons of the abilities of greases to resist the displacing action of the various solutions can be made.

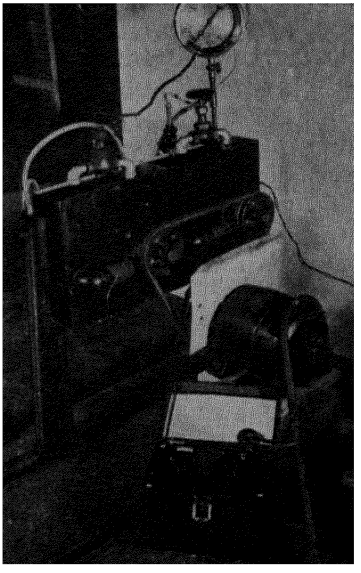


PLATE XIX. — Laboratory Apparatus for Determining Service Characteristics of Automotive Water Pump Greases.

Prolonged tests with various makes of pump packing, in which the packing gland is kept abnormally tight, yield results bearing on the characteristics of grease and packing most desirable for preventing shaft scoring. With this device it is also possible to gain information as to the quantity of grease which may find its way into the cooling system and retard heat transfer.

CALCIUM BASE GEAR GREASES

(Automotive, Transmission and Differential, Industrial)

While the number of lime base gear greases appearing on the market during the last ten years has been many, it is doubtful if their quality is such as to warrant their popularity. Practically all of these lime base greases break down to a granular mass or a thin oil which has little resistance to leakage. However this type of grease finds particular favor in cold districts where ease of shifting gears is an important factor. For further information on the stability of transmission greases the chapter on Soda Base greases should be consulted.

The pour point of a gear lubricant is of little or no importance in determining whether the lubricant will feed satisfactorily to the ring gear and pinion and not channel at low temperature.

In the case of fluid greases the viscosity numbers by which the lubricants are classified shall be determined by the viscosity of the oil without the soap or other compounding ingredients.

Wherever the viscosity numbers are used on fluid greases the container labels should show in some suitable manner that the number applies to the oil without the soap or other compounding ingredients.

Lubricants designed to carry pressures much greater than those which can be carried by viscosity alone may be marked with the letters E.P. (extreme pressure) in addition to the S.A.E. viscosity number.

General Information

Transmission and rear-axle lubricants are made from mineral oil with or without the addition of animal or vegetable oils, soaps, etc. For certain purposes such as decreasing the tendency of the lubricant to leak from housings, small amounts of soaps or other materials are sometimes added. Although these additions may increase the apparent viscosity of the oil, they have little or no effect on the load-carrying property or the tendency of the lubricant to prevent easy gear shifting.

The selection of a transmission or rear-axle lubricant depends upon the conditions to which the lubricant will be subjected. In general, for high temperature operating conditions lubricants of viscosity No. 160 will be more suitable than those of No. 110 or No. 90. For lower temperatures the lubricants of lower viscosity numbers may be preferable. Where neither high nor low extremes of temperature are to be encountered a lubricant of viscosity No. 110 may be satisfactory. For extremely low temperatures, or where lubricants of the lower viscosity numbers cannot be obtained, it may be desirable to thin the lubricant with a low viscosity, low pour-test crankcase oil.

For average use in transmissions and rear-axles of passenger cars the temperature of the lubricant is not high enough to make the resistance of the lubricant to oxidation of much importance. However, for use under severe conditions where higher temperatures are more common, lubricants that are more resistant to oxidation are always preferable and may be necessary.

For lubrication under conditions where extreme pressures are encountered, special "extreme pressure" (E.P.) lubricants can be supplied by a number of manufacturers. These lubricants are intended for use where pressures are too great to permit maintenance of "fluid-film" lubrication.

Lubricants containing solid materials in suspension are undesirable for ball or roller bearing lubrication.

On account of the many different conditions under which these lubricants are used and on account of the different methods of manufacture, a single detailed specification is not applicable to all transmission and rear-axle lubricants and as a result the car owner must depend upon the oil company for the quality of the product, using the S.A.E. viscosity numbers as a means of obtaining lubricants having the desired viscosity or body.

Formulae for Standardized (Calcium Base) Auto Gear Greases

The formulae for two such greases are as follows:

S.A.E. 80

Constituents	Gallons	Pounds Net	Per Cent by Weight
Tallow	25	190.68	2.99
30° Bé. caustic soda	.25	.62	.01
Hydrated lime	—	30.00	.47
Water	—	57.42	.90
100 red oil	441	3314.50	51.95
162/210° F. lub. dist.	344	2786.78	43.68
		6380.00	100.00

The pour point of a gear lubricant is of little or no importance in determining whether the lubricant will feed satisfactorily to the ring gear and pinion and not channel at low temperature.

In the case of fluid greases the viscosity numbers by which the lubricants are classified shall be determined by the viscosity of the oil without the soap or other compounding ingredients.

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S.A.E. 80

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Tallow	25	190.68	2.99
30° Bé. caustic soda	.25	.62	.01
Hydrated lime	—	30.00	.47
Water	—	57.42	.90
100 red oil	441	3314.50	51.95
162/210° F. lub. dist.	344	2786.78	43.68
		6380.00	100.00

S.A.E. 90

Tallow	50	381.35	4.30
Hydrated lime	—	60.00	.68
30° Bé. caustic soda	0.5	1.24	.01
Water	365	115.31	1.30
100 red oil	688	2741.70	30.91
162/210° F. lub. distillate		5570.40	62.80
		8870.00	100.00

LOADING THE AUTOCLAVE

100 Red Oil, water and the powdered hydrated lime are mixed together in an eighty gallon tank, by means of a "Lightnin" mixer. Considerable care must be used to prepare a good emulsion with these materials, as it has been found that much better results may be had, saponification is probably more rapid, and the finished grease more attractive in appearance, if the powdered lime is well dispersed in the liquid medium. The lime mixture is prepared as follows:

Mix together:

25 gallons	100 red oil
12.5 gallons	Water
60 pounds	Hydrated lime

An additional quantity of 40 gallons of 100 Red Oil is then mixed in while the lime is being agitated to produce a smooth emulsion, the 50 gallons of tallow is measured into a 600-gallon autoclave or pressure cooker. The lime mixture is then also run into the autoclave after which the 0.25 gal. of 30° Bé. caustic soda and 85 gallons of 100 Red Oil are added.

SAPONIFICATION

All openings to the autoclave are then closed, the two-way agitating paddles started, and steam at 100 pounds per square inch is admitted to the jacket of the mixer. Heating and agitation are continued for 1¼ to 1½ hours. At the end of this period a pressure of 40 to 55 pounds per square inch and a temperature of 260 to 280° F. are attained. Under these conditions the reaction between the lime and fat is considered as sufficiently complete and the partially reduced soap is discharged under its own pressure through a steam jacketed line to open mixer No. 31.

REDUCTION OF THE SOAP

Prior to the discharge of the soap from the autoclave, the open mixer is loaded with about 50 gallons of slop grease and 100 gallons of 100 Red Oil or deficient oil. This mixture is heated to approximately 180° F., at the time the soap is run in. It is essential that the contents of the kettle should be at about this temperature so that the slop grease may be well dissolved in the oil and so that the hot soap base will not be chilled with consequent lump formation in the finished grease. At kettle temperatures in considerable excess of 220° F. too great a portion of the water content of the soap base will be lost by flash evaporation during the discharge of the soap base and it would be necessary to add to the batch a sufficient amount of water to hydrate the lime soap and bring it into the required colloidal condition for satisfactory grease formation. With proper control of the pressure and temperature of the autoclave and the temperature of the open mixer it is usually unnecessary to make any additions of water to the batch.

After it has been ascertained by the compounder that the partially reduced soap in the mixer has a satisfactory consistency and the correct degree of plasticity (*i. e.*, contains about 3.5 to 5 per cent of water and shows no great tendency to bleed excessively when a small sample is cooled) the balance of the 100 Red Oil is slowly added in a continuous stream without the application of further heat to the kettle. However, if there is a tendency for the grease to form lumps due to cold weather conditions or the too rapid addition of cold oil, then intermittent application of steam to the kettle may be necessary.

When the addition of the 100 Red Oil has been completed and the grease has a

satisfactory buttery appearance, the Lub. Distillate (150-165 seconds Saybolt at 210° F.; color, green-black) is mixed in until a product of the required MacMichael viscosity, as determined by the laboratory, has been arrived at.

DRAWING

From five to eight hours is usually required to complete the batch, and at the end of this period the temperature of the grease is 120 to 150° F. If the consistency and appearance of the product have been approved by the laboratory the grease is pumped from the kettle through a cylindrical screen of 60 mesh per inch. This screen removes bits of unreduced lime soap, hard clots of grease, etc., which often prove objectionable to customers making use of such a lubricant in high pressure greasing systems equipped with fine mesh strainers.

PROPERTIES

This gear grease shall be smooth and lustrous. Its color is dark green. The soap content is less than about 10.0 per cent by weight. In consistency, No. 90 is comparable with a soft No. 0 cup grease.

SPECIFICATIONS

These greases must conform to the following requirements:

Percent Water (W.B.D.)	1 to 3
MacMichael Viscosity (No. 26 Wire, 60 R. P. M.)	110 to 145 at 80° F., (S.A.E. 90); 20 to 52 at 80° F., (S.A.E. 80).
Percent ash	1.3 Maximum

TEMPERATURE SUSCEPTIBILITY OF S.A.E. 90 STANDARDIZED AUTO GEAR GREASE

The effect of change of temperature on the MacMichael Viscosity and Spike Penetration of the S.A.E. 90 Gear Grease is tabulated below:

Temperature ° F.	MacMichael Viscosity No. 26 Wire 60 R.P.M. 1.0 cm. bob.	Spike Penetration 1/10 mm. Time—5 sec. Total Weight—5 oz. Spike—$\frac{1}{2}$" Head $\frac{1}{16}$" Shank
200	5	...
180	20	...
160	35	...
140	50	...
120	70	...
100	95	...
80	130	...
60	200	...
40	295	...
30	340	500 plus
20	400 (Approx. S.U.V. 67,000)	500 plus
10	...	550
0	...	520
−10	...	340
−20	...	170
−30	...	142
−40	...	138

It is evident from this data that the No. 90 Grease would not channe in service at temperatures down to zero and would cause no great difficulty at a temperature of minus 20° F.

MECHANICAL STABILITY OF S.A.E. No. 90 STANDARDIZED GEAR GREASE

It has been mentioned that lime base gear greases were not ideal from the standpoint of stability and change in consistency due to mechanical agitation. The data below shows that there is a great decrease in viscosity of this grease for about 25 hours of operation, after which equilibrium appears to have been reached. A 1928 Chrysler gear box, operating in high speed with the input shaft turning at 1750 R.P.M. was used to obtain the data listed below:

Time (Hours)	MacMichael Viscosity 77° F., No. 26 Wire, 60 R.P.M.
0 (Original Grease)	130
5	104
10	77
15	55
20	36
25	27
30	25
35	24
40	23
45	23
50	23

Black Calcium Base Gear Greases

Automotive gear greases which are black in color will ordinarily contain appreciable amounts of asphalt either as such or as normal constituents of fuel oils or residuals.

The objection to the use of asphalt in gear greases are based on the assumption that the asphalt is non-lubricating, that the asphalt may come out of solution, dry or oxidize and thus forms a very poor, hard, lubricant which in some cases results in lubrication failure. In contrast to these serious objections, the following advantages of the use of asphalt are listed:

The residuum stocks containing asphalt are very low in cost, and thus the greater proportion of them that are incorporated in the greases, the lower the cost of the grease. The yields of lubricating oils of high viscosity from asphaltic crudes are low, and their value consequently high. The use of asphalt and light lubricating oil mixtures is for this reason favored by some refiners, where a cheap oil of high viscosity is required for making greases. It should be noted, however, that frequently it is required to melt 50-60 penetration asphalt from 55 gallon drums which is a rather slow and expensive procedure. Often there is no provision for handling viscous black oils and any savings that could be shown in cost of materials would be reduced considerably by the cost of additional equipment for handling the black oil mixtures.

The black color of a grease is considered an advantage by many manufacturers and users, although the value in this direction is not obvious, and the writer's preference is decidedly for a dark green product. It is often stated that the black grease is more desirable in that it does not change color in use, as is the case with lighter colored greases which change in tint due to the agitation of the gears and air occlusion.

Another advantage of asphalt-oil mixtures as a grease ingredient, which is of more practical value, is the fact that the low temperature characteristics are considerably improved and that the temperature-viscosity curve is flatter than for lubricating fractions alone. Thus:

	50 Per Cent Transmission Oil 50 Per Cent 1000 Pale Oil	25 Per Cent 35 Pen. Asphalt 75 Per Cent 100 Pale Oil
S.U.V. at 100° F.	614	505
S.U.V. at 210° F.	58	61

In some cases the residues are rich in natural sulfur compounds which may act as E.P. agents.

The asphaltenes and resinous material in asphalt have been found to be very adhesive lubricants and appear to form more pressure resistant films than some highly refined lubricating oils.

DARK GEAR GREASE No. 3

No. 3 Standardized cup grease	29.7 per cent
55 Penetration asphalt	18.7 per cent
300 pale oil	51.6 per cent
McMichael viscosity (No. 26 wire)	39 at 83° F.
	40 at 79° F.
	46 at 76° F.
	48 at 72° F.
Dropping point—73° C.	

DARK GEAR GREASE No. 4

No. 2 Standardized cup grease	54.2 per cent
55 Penetration asphalt	29.2 per cent
300 pale oil	8.3 per cent
Transmission oil	8.3 per cent
McMichael viscosity (No. 22 wire)	45 at 85° F.
	47 at 83° F.
	50 at 77° F.
	52 at 72° F.
Dropping point—85° C.	

PROCEDURE

The Cup Grease is warmed to about 110 to 130° F. and the asphalt melted at a temperature of 150° F. is mixed in. The batch is then allowed to cool and the balance of the oil run in to give a grease of the correct consistency. The temperature of the batch at no time should go above 150 to 160° F., and preferably should be kept at as low a point as possible and still obtain uniform mixture of the materials being processed.

COSTS OF DARK GEAR GREASES

Estimated costs of these greases are as follows:

No. 3 Per Cent by Weight			
29.7	No. 3 Standardized cup grease	at $ .0323	$ .00959
18.7	50–60 Penetration asphalt	at .0100	.00187
51.6	300 pale (A. T.)	at .01275	.00671
Cost of materials per pound of grease			.01617
Cost of compounding			.01120
Total cost per pound ex kettle			$ .02937

No. 4 Per cent by Weight			
54.2	No. 2 Standardized cup grease	at $ .0323	$ .017420
29.2	50–60 Penetration asphalt	at .0100	.002920
8.3	300 pale oil (A. T.)	at .01275	.001058
8.3	Transmission oil	at .0056	.000465
Cost of materials per pound of grease			$ .021863
Cost of compounding			.011200
Total cost per pound ex kettle			$ .032063

American Commercial Lime Base Auto Gear Greases

A survey of a great number of gear greases offered to the American motorist in 1935 indicated that sodium soap was the most popular choice. The relative proportions were:

Type of Soap in Gear Grease	Per Cent
Sodium	43
Calcium	22
Mixed sodium and calcium	17
Aluminum	14
Lead	4

Lime Base Gear Grease Purchase Specifications

Five Eastern grease manufacturers offer lime base gear greases complying with the following requirements:

Number	1	2	3	4	5
Percent calcium soap	6	7.5	4.5	8–12	12–14
Percent water, max.	1	2	2	1½	1½
Percent mineral oil	93	92.5	95.5	...	84.5–86.5
Tests on Mineral Oil:					
A.P.I. gravity	22–23	18	20	...	...
S.U.V.	75–95/210	156 at 210	70 at 210	100 at 210	3000-6000 at 100
Color	Black	Black	Black	...	Drk. Green
Pour point ° F.	—30	45	0	...	...
Consistency:					
Worked penetration		360–380	...	345–390	345–365
MacMichael viscosity	25–35				
Percent free alkali	0.05	0.15	0.15	...	.25
Percent free acid	0.10	Nil	Nil	...	.25
Corrosion test	Pos.	Neg	Neg.	Neg.	Neg.
Odor	No perfume	none	none	Myrbane	Myrbane

Lime Base Gear Grease (1933)

Analysis of this grease gave the following results:

	No. 3½ Gear Lubricant
Physical Tests:	
Penetration A.S.T.M. (unwk'd. at 32° F.)	360+ 30
Penetration A.S.T.M. (worked at 32° F.)	
Penetration A.S.T.M. (unwk'd. at 77° F.)	360+130
Penetration A.S.T.M. (worked at 77° F.)	
Dropping point ° C.	84
Gravity ° A.P.I.	
S.U.V. at 210° F.	
Flash ° F.	
Pour point ° F.	

No. 3½ Gear Lubricant—(*Continued*)

Composition (per cent by weight):

Soap	4.96 (Calcium base)
Oil	93.00
Moisture	0.60
Free fat	
Asphaltic matter	1.97
Sulfur	
Saponification value (mg. KOH)	
Gain or loss in analysis	0.52
Total	100.00

Oil Extracted:

Color, N.P.A.	Black
Gravity, A.P.I.	18.3
S.U.V. at 100° F.	1155
S.U.V. at 210° F.	83

Lime Base Gear Grease with Filler

A lime base gear grease containing sawdust, the sort of grease we seldom see in modern practice, was proposed by Turner [73] in 1863. It was made in accordance with the following formula:

	Gallons
Spindle oil	10
Oleic acid	2
Lime water	6
Sawdust	45

Analyses of Some Commercial Lime Base Transmission Greases

Analyses of several lime base transmission greases have been made with the results shown in Table 13.

TABLE 13—Analyses of Commercial Lime Base Transmission Greases

	Per cent by weight				
	Transmission grease	Transmission grease	Transmission grease, light	Transmission grease, medium	Transmission grease, heavy
Mineral oil	82.25	91.45	91.04	83.46	78.85
Free fat	1.78	.47	.41	1.75	1.55
Combined fat	13.18	5.70	4.70	11.94	15.22
Free fatty acids	.54	.62	1.26	Trace	Trace
Free CaO	.43	.04	.13	.16	.36
Combined CaO	1.32	.57	.47	1.19	1.52
Water	.50	1.15	1.04	1.50	2.50
Melting point, ° F.	....	145	....	145	203
	Transmission grease	Transmission grease	Gear grease	Gear compound	Gear compound, light
Mineral oil	91.60	80.67	78.63	96.43	89.66
Free fat	.88	6.99	9.01	.75	5.32
Combined fat	6.10	9.17	9.14	1.29	4.40
Free fatty acids	.35	1.42	.73	1.39	.18
Free CaO	.21	.07	....	....	....
Combined CaO	.61	.98	1.48	.14	.45
Water	.25	.70	1.01	Trace	Trace
Melting point, ° F.	197	....	144	90	100

[73] U. S. Patent 39,185 (July 7, 1863).

Notes on Care and Maintenance of Transmissions, Differentials and Clutch Bearings

The necessity for proper lubrication of the transmission and differential cannot be over-emphasized. The same lubricant is used for the gears of these units as for the bearings which support the shafts. The gears are frequently made of nickel steel hardened to increase wear resistance. Annular ball bearings, roller bearings and spiral type roller bearings are used to support the gear shafting. Oil of suitable viscosity is preferable for automotive gear lubrication provided the gear housings are sufficiently tight to prevent leakage, otherwise a suitable lubricating grease may be found desirable.

The selection of the correct grade and character of lubricant for transmission use is a problem requiring careful study by competent engineers familiar with the design and construction of these models as well as the performance of lubricants under the various conditions of service to be encountered.

In order to prevent wear, each working part must be protected by a film of lubricant adequate to withstand the severe pressures developed. The supply of lubricant must be free from foreign matter of any kind which would pit or cut highly polished gear teeth and ball bearing surfaces. The enclosure of the gears and bearings in the transmission case permits them to run in a bath of lubricant which is carried to the working parts by the turning of the gears. In some cases and under certain service conditions the unit pressures may become excessive and extreme pressure lubricants will be needed. A sufficient amount of sulfurized lard oil or other sulfurized fatty oil may be incorporated with the Standardized Gear Greases to meet such conditions. From 5 to 15 per cent by weight of these E.P. agents should suffice for most cases.

The lubricant must follow, coat, and cushion the gear teeth during service, and be well distributed to all bearings as well. For winter operation at low temperatures the use of the S.A.E. 80 and 90 lubricants should be insisted on. For summer, S.A.E. 110, 160 or 250 are the lubricants normally used.

Transmissions and differential are normally provided with drain plugs and combination filling and level testing plugs. The lubricant should be checked at regular intervals. Some oil companies say each 1000 miles, others every 500 miles, particularly in heavy duty interurban work. When checking, if the level is low, the gear case should be replenished with the same type of lubricant as is being used. Mixtures of calcium base greases containing water with sodium base greases are responsible for frothing and leakage difficulties. These types of greases with certain E.P. gear oils and lead base greases have, in some cases, given rise to difficulties due to aeration and frothing.

The gear cases should be drained each 5000 miles of vehicle operation to reduce, as far as possible, excessive wear to gears and bearings due to the entry of dust when checking the levels and during operation, and the

slow accumulation of metal particles, which gradually contaminate the lubricant. The charts prepared by the automobile companies and service companies should be consulted for the grades of lubricants for each make of car and the capacities of the gear cases.

To drain, first clean all dirt from around the filler plug. Then remove the drain plug at the bottom of the transmission case and allow all the oil to run out. Then flush the case and gears thoroughly with kerosene. When this has drained out, replace drain plug and refill with recommended lubricant to proper level, which is the height of the filling plug.

If the lubricant is filled above this level, leakage and waste may occur. Excess lubricant due to the churning of the gears may be forced out through the mainshaft bearing cover.

If the lubricant falls greatly below the proper level, the gear teeth may become dry causing excessive wear with resultant noisy operation; the transmission shaft bearing and shifting mechanism will also suffer from lack of lubrication.

General Discussion of Gear Grease Service Characteristics [74]

Factors having a bearing on the consistency of gear greases and the nature of the differences between transmission grease and transmission oil will be pointed out. Information relative to the consistency of a transmission grease for best service behavior will be given. Data on the stability or change in consistency of transmission greases while under service conditions will also be given, which will include investigations of lime, soda and mixed base greases.

Consistency is often defined as the "degree of solidity" or the "degree of fluidity" of a grease. The "consistency" will determine the resistance a grease will offer to motion, under any given set of conditions. Oils under normal pressures behave as true fluids, and their consistency in the above sense may be defined completely by the term viscosity.

Assuming stream line flow, the flow rate for a given viscosity will be doubled; or if the pressure is reduced the flow rate will be proportionately reduced. No matter how small the pressure some flow will take place, at a rate proportional to pressure. Because of this proportionality between pressure and flow, a viscosity determination can be made under any suitable pressure; for instance, under the gravity head of the liquid, as in commercial viscometers of the Saybolt type. This single determination will permit predicting what the flow rate will be in the same apparatus under any other pressure.

This simple relationship between pressure and flow rate is not true for greases. It is possible for a certain grease under gravity head in an ordinary orifice type viscometer, that no flow might take place.

The conclusion might be that it has an infinite viscosity; that is, gives an enormous resistance to motion. Yet, practical tests show that in an automobile transmission, such a grease might offer less resistance to the motion of the gears than certain oils, which flow freely through the viscometer under the same gravity head which permitted no flow to take place with the grease.

If the pressure is gradually increased, a point will be reached where

[74] Klemgard, E. N., *Petroleum Age*, (May, 1929; and June, 1929).

the grease begins to flow, and increased pressure causes an increased flow rate. To obtain the relation between pressure applied and flow rate for a lubricating grease it is evident that tests will have to be made at more than one pressure; a determination at a single pressure as by an ordinary viscometer, for oils, would not permit predicting flow rates at other pressures.

This fundamental difference between oils and greases, may be stated as follows:

An oil is a viscous liquid; that is, a body in which the smallest stress (shearing force) will cause a constantly increasing change of form. The ratio between stress and flow is measured by viscosity.

A grease is a plastic solid; that is, a body in which no continuous alteration of form is produced by stresses up to a certain minimum value. This minimum value has been termed by Bingham "Internal Friction" and by others "Yield Value." If the stress exceeds the above minimum value, flow will take place at a rate which is proportional to the excess of the shearing force above the minimum required to start flow. That is, if we consider the excess of force only, we have the relationship between pressure and flow as is obtained with an oil. This statement has been found to be untrue for certain greases at pressures slightly above the internal friction value and may be due to a different type of flow which is not flow of "telescoping layers" or stream line flow.

This relation between pressure and rate of flow for an oil is defined by its viscosity, or the reciprocal of it which is known as "fluidity." With plastic solids, the similar relation between excess pressure and flow may be defined as mobility. Mobility may also be defined as the area, over which a unit of tangential force in excess of the yield value when applied will cause a relative velocity of one centimeter per second between two parallel layers of grease one centimeter apart, the intervening space being filled with the grease. "Ultimate Viscosity" is also a term to indicate mobility and may be defined as the viscosity which the grease would have if its friction or yield value were zero.

The value of friction (internal friction) is generally low, so that for relatively high pressures, it may be neglected, in which case the grease behaves for all practical purposes like an oil which has the same viscosity as the ultimate viscosity of the grease. It should be noted, however, that this statement is true only if the pressures are relatively high, or what amounts to the same thing, if the velocities involved are relatively high; theoretically, they should be of an infinite nature, an idea which the word ultimate is supposed to convey.

From what has been said it is apparent that a grease combines two valuable properties:

At low pressures, it behaves like an extremely viscous oil, it will stay in place. For instance, when the differential and transmission gears are standing still, oil will in time drain off the parts practically completely, no matter how viscous, while grease will drain off only to a point where the shearing stress in the grease film produced by its weight is equal to the

internal friction; that is, an appreciable film of grease will be retained indefinitely. Yet, when the gears are in motion grease will offer a relatively low resistance to motion.

While it has not been completely investigated, there is evidence that a grease consisting of metallic soap and lubricating oil has the ability to prevent noisy operation of the gears, particularly at high velocities. The yield value or internal friction may be that property of the grease which promotes silent operation and cushioning of the gears.

Elasticity is another property of grease which has a bearing on consistency. Elasticity is the ability of the grease to absorb energy and to give it back elastically. This property of the grease in recovering from deformation produced by force may also be a contributing factor to silent gear operation. It is of note that oils compounded with aluminum soaps (oleate, stearate, and palmitate), to give a gel, have considerable elasticity.

Texture of the grease is determined by the presence or absence of a cellular or fibrous structure, and the relative size and structure of same. Sodium and aluminum salts of the higher fatty acids produce a structure in the oil which is apparently a lattice, or network of very fine threads, or parallel bundles. This lattice is sometimes termed a micelle.

The stability of these internal structures is of vital importance in the service behavior of transmission greases. The relative stability of the greases will in turn depend on the persistency of these internal structures. The investigation of the gels produced by such soap structures, and the technic required for making stable gels which satisfy all of the large number of other requirements of transmission greases as satisfactory lubricants, are problems confronting the grease chemist.

The production of the most stable grease will depend not only on the selection of the metal or metals which, when combined with a fatty acid will give molecules which will orient themselves in such a way as to produce the most stable micelle, but also on the choice of that fatty acid, or mixture of fatty acids, and possibly other organic acids.

It should be pointed out that under conditions of heavy service, viscosity alone does not explain the lubricating qualities even of oils. Another property known as "oiliness" is of great importance. There is little doubt that the property of oiliness applies to greases as well as to oils. Oiliness is not of great importance for the relatively light service that a lubricant has had to give in automobile transmissions or differentials in the past, but new designs demand a high degree of oiliness and film strength.

There is a case on record where a gear set was run on a testing block for forty-eight hours under very heavy load conditions, absolutely dry, without damage to the gears. The use of free sulfur or sulfur loosely combined with hydrocarbon material has the remarkable effect of causing the gears to take on a very high polish and is advocated by some manufacturers of grease. Lead oleate and other lead soaps are employed by other manufacturers to increase the oiliness of their transmission and differential greases.

Apparatus for Determining Consistency of Greases; the Plastometer or Pressure Viscometer

It has been pointed out that the consistency of a grease is defined by at least two properties; its internal friction and its ultimate viscosity. It was also shown that a method of measuring consistency involves an apparatus which permits the determination of flow rates at various pressures. An instrument, resembling in general the usual orifice type oil viscometer may be used. It consists essentially of a container for the grease or oil to be tested, closed air tight at the top by means of a cover. At the bottom is an exchangeable nozzle, or orifice, 0.111 inches internal diameter by one inch long. A needle valve, passing through a stuffing box in the cover, is used for closing the orifice. Paddles are attached to a stem of the needle valve for stirring the grease in bringing it to uniform temperature. A small needle valve, mounted on a spring, may be used for quickly closing the outer end of the orifice.

The grease container is surrounded by an outer jacket containing a constant temperature bath. Water is circulated to and from the thermostat by means of a pump and hose. An air line serves to put any desired pressure on the grease in the container, the air pressure being measured by means of a U-tube manometer. The pressure is regulated by an adjustable water column, consisting of a glass vessel which has a vertical adjustment, and is connected to the air vessel by means of a rubber tube. This arrangement holds the pressure constant within very close limits.

Method of Operating Pressure Viscometer

The viscometer is filled with the grease to be tested, the cover is screwed on, the orifice closed by the lower needle valve, and the thermostat set for the desired temperature. The pressure is then adjusted to the desired value. The instrument is left standing until the thermometers in the bath and in the grease show the same temperature; the equalizing of temperatures may be accelerated by stirring. The needle valve is then opened and after regular flow has set in, the grease discharged during a suitable time interval, timed by a stop watch, is gathered in a crucible and then weighed on the analytical balances. From this data, the flow rate at a given pressure and temperature may be readily computed.

The procedure is then repeated at different pressures, but at the same temperature. Thus, the relation between pressure and flow rate at one temperature may be established. This relation is true only for the particular instrument and can be reduced to absolute units, independent of the instrument by the following equations:

$$\frac{V}{t} = \frac{3.1416\, m\, R^3\, (F-f)}{4} \tag{1}$$

$$F = \frac{RP}{2l} \tag{2}$$

$$f = \frac{RP}{2l} \tag{2a}$$

In which

V = Volume in c.c. discharged in time t.
t = Time, seconds.
m = Mobility, square cm./dyne seconds.
R = Radius of capillary, cm.
l = Length of capillary, cm.
P = Pressure used to overcome internal friction, dynes/square cm.
$F, f,$ = Shear, dynes/sq. cm.

The above equation may be made more convenient for use by expressing it in the following terms:

G = Weight of grease discharged in grams per 100 seconds.
$= \frac{100\,Vs}{t}$
s = Specific gravity.
D = Diameter of orifice in inches.
n = Absolute Viscosity—dyne sec./sq. cm.
H = Pressure, inches of water corresponding to G.
h = Pressure at which flow starts, inches of water.
F = Internal Friction, lbs./sq. ft.

Using these expressions, equations (1) and (2) may be written:

$$G = \frac{100{,}000\ D\ 4\ s}{Ln}(H-h) \tag{3}$$

$$F = 1.3\frac{D}{L}h \tag{4}$$

Viscometer Constants:

D = 0.11 inches
L = 1.0 inches

If these values are introduced, equations (3) and (4) become:

$$G = \frac{15\ s\ (H-h)}{n} \tag{5}$$

$$\frac{n}{s} = \frac{15\ (H-h)}{G} \tag{6}$$

$$F = 0.14h \tag{7}$$

Equation (5) for the relation between G and H is that of a straight line and holds only when the material throughout the tube flows in telescoping layers. This condition is not fulfilled with the smaller flow rates. The value h should, therefore, be determined by using the straight part of the curve only projecting it down to the intersection with the H-axis, and assuming that the intercept represents h, the pressure at which flow would begin, if at small flow rates the same law of flow would apply as at higher rates.

The kinematic viscosity from equation (6) may be converted to Saybolt seconds by dividing it by 0.0022. The final equations are:

$$\text{Ultimate Saybolt Viscosity} = \frac{6800\ (H-h)}{G} \text{ seconds}$$

$$\text{Internal Friction} = 0.14h \text{ pounds per sq. ft.}$$

STABILITY OF TRANSMISSION GREASES; STIRRING APPARATUS

In the case of the greases made with sodium soaps, the soap base is compounded with a suitable mineral oil at a temperature of 250 to 400° F. At about 350° F., the mixture assumes the characteristics of a liquid; on cooling and stirring, a form of "oriented coagulation" takes place and we have formed a colloid material in which there is an internal structure. It is probable that this colloid structure, which is responsible for the consistency of the grease, consists of a lattice or network of very fine threads made up of oriented soap molecules. The persistency of the polar soap molecules to form the lattice structure or micelle and remain in this oriented condition is responsible for the degree of stability of the grease. Other factors in the manufacture of grease which have a bearing on the stability of the micelle are the nature of the mineral oil, rate of cooling, degree of agitation during cooling, amount and relative time of adding mineral oil to the mixture. In the case of saturated hydrocarbon oils apparently consisting of symmetrical molecules having little polarity, coagulation of satisfactory threads does not take place readily, as is the case with the highly unsaturated oils. Rapid cooling without agitation produces a gel in which the lattice structure has evidently not had the opportunity to form bundles of threads or fibers. The amount of water present has an important effect on the colloid structure, complete dehydration and heat treatments having a destructive effect on bundle formation and excess water dissolves the sodium soaps and gives a product of poor yield value.

The calcium soaps behave differently and require an appreciable amount of water present to hydrate the soaps in order to produce a gel structure. In general, this type of gel, even when the soap is properly hydrated, is subject to considerable change in consistency when mechanically agitated. Loss of the water and agitation bring about separation of the soaps and oil.

In order to study the effect of heat and agitation on the various transmission greases, a motor driven stirring device was employed. It consists of a can holding about one pound of grease, which rests in a brass cup or holder, provided with a lid. On the outside is an electric heating element, by which the temperature of the grease can be regulated. The grease was stirred by a small propeller, driven by a vertical motor.

Four or five samples of each grease studied were tested for consistency; Sample A, original grease; Sample B, stirred for two hours with external heat; Sample C, stirred for two hours at 150° F.; and Sample D, stirred for sixteen to nineteen hours at 150° F. Another sample was run for twenty hours in the differential of a model T Ford car under normal operating conditions.

RESULTS OF CONSISTENCY TESTS ON TRANSMISSION GREASES

A comparison of the consistency of the samples, as determined by the pressure viscometer, gives a practical indication of the stability of the grease. In order that the nature of the greases may be fully understood, chemical analyses were made by the A.S.T.M. method with the following results:

Grease No. 1 is manufactured in Ohio and sold in practically every state in this country. The quantity sold is in excess of five million pounds per year. Grease No. 2 is manufactured in New Jersey and also has nation wide distribution. Greases 4 and 5 are manufactured in California and sold in the Pacific Coast area. Grease No. 6 is manufactured in New Jersey and has national sales. Consistency tests on these greases determined by the pressure viscometer are given below:

Per Cent	1	2	3	4	5	6
Mineral oil	80.69	78.63	89.08	91.58	82.25	89.66
Water	0.70	1.01	.30	Trace	0.50	Trace
Free fat	6.99	9.01	4.26	3.18	1.78	5.32
Combined fat	9.17	9.14	5.61	4.66	13.18	4.40
Free fatty acids	1.42	0.73	0.00	0.13	0.54	0.18
Comb. CaO	0.98	1.48	....	0.12	1.32	0.45
Free CaO	0.07	....	....	....	0.43	....
Comb. NaOH	0	0.	0.75	0.33	0	0
Free NaOH	....	....	Trace	....	....	....
S.U.V. of Mineral oil, 210° F.	66.	70.	120.	80.	71.	55.
Melting point ° F.	150.	144.	120.	117.	145.	100.

Consistency of Greases at 125° F. (Before "A"; and after "B"; 20 hours run in differential)

Grease	Internal Friction (Lbs./sq. ft.) A	Internal Friction (Lbs./sq. ft.) B	Saybolt Viscosity Sec. A	Saybolt Viscosity Sec. B
No. 1	4.58	1.10	31,000	1200
No. 2	4.2	1.00	29,000	2200
No. 3	1.8	0.70	93,000	3300
No. 4	1.6	0.45	82,000	1700
No. 5	1.3	1.30	19,700	1230
No. 6	1.0	0.45	4,050	690

Results of Ford Rear Axle Tests on Transmission Greases:

Grease	Nature of Lubrication: Gears	Left Bearing	Right Bearing	Leakage
No. 1	Good	Good	None	None
No. 2	Poor	....	....	None
No. 3	Good	Poor	Poor	None
No. 4	Good	Good	Good	15 grams
No. 5	Good	Good	Good	5 grams
No. 6	Good	Good	Good	10 grams

Note: All tests made on 1924 axle, axle level, charge 1.25 lbs.
Good lubrication; parts well covered with lubricant.
Poor lubrication; practically dry, in same condition as assembled.
All runs made for twenty hours, average grease temperature 125° F.

In considering the tests on the service behavior of the greases the following items are of importance:

Readiness to flow to parts to be lubricated

At the end of each twenty-hour run in the Ford differential the axle was taken down and all parts carefully inspected for lubrication. The amount of lubricant that had leaked from the differential past the roller bearings into the axle housings was weighed and reported as leakage.

There is a little doubt that almost any grease will be able to give satisfactory lubrication in the differential or transmission, provided it is able to reach the parts which have to be lubricated. The service tests indicated that with some greases, even after a twenty-hour run, the differential gears and the roller bearings remained in the same practically dry condition in which they were assembled at the beginning of the test, although a normal charge of lubricant was present in the differential housing. Other greases gave scant lubrication of some parts. This is not very objectionable, because very small amounts of lubricant are required for the lubrication of the hardened surfaces. It is also reasonable to assume that in a longer test more of the lubricant would have reached these parts. Form a service viewpoint the difference between good and scant lubrication is therefore not serious, but is valuable as an index as to what properties a grease must have to flow readily to the parts to be lubricated.

Some automobiles are now designed so that the universal joint is lubricated by the grease in the differential housing, which runs through a small hole in the propeller shaft housing and flows to the universal joint. For this type of construction a consistent grease will not flow through the hole provided and the universal joint will be scantily lubricated.

According to theory, flow will take place at a constant rate under any given conditions, if the following relationship is satisfied:

Ultimate Viscosity = A (B—Internal Friction).

A and *B* being constants, this is the equation of a straight line. This line is the boundary between satisfactory and unsatisfactory greases and its equation being from actual tests.

Ultimate Saybolt Viscosity = 3,000 (1.75—Internal Friction).

It is of note that such a relationship may be expected to hold only with reservations. Different greases will stir down differently under repeated tests; the sample taken from the differential at the end of a twenty-hour run is really a mixture of unstirred grease clinging to the walls and portions which have thinned down considerably; temperatures will also vary under different service conditions. In view of these conditions, the correlation shown in the foregoing equation, relating consistency with service behavior, is remarkably good and sufficiently accurate for all practical purposes.

It may be concluded that the lubrication of the differential, and also the transmission, depends on the consistency which the grease attains when stirred at the temperature existing in the housing. The original consistency of greases, as purchased, gives at best a poor indication of their suitability, because different greases thin down differently and after being thinned behave differently with changes of temperature.

The behavior of the grease in service in differentials and transmissions may be predicted with reasonable accuracy by stirring a sample with a motor at 150° F., for about twenty hours and then determining its consistency at 125° F. The results indicate that the most desirable consistency should be about that given by the above equation; that is, if its ultimate

viscosity is low its should have a value for internal friction of less than 1.75 pounds per square foot at 125° F.

Leakage

Leakage of course should be kept at a minimum. As leakage is a phenomenon of flow this demand is to a certain extent in contradiction to the requirements that the lubricant should flow to parts freely. In the Ford rear axle, for instance, the roller bearings in the differential housing must receive lubrication; that is, the lubricant must leak by the thrust washers to the bearings. However, if it is able to flow through the small clearances between the thrust washers, to the bearings, it will certainly flow out through the large openings between the rollers and the axle housing; that is, it will leak.

Leakage can, therefore, be reduced only so far as is consistent with the requirements of satisfactory and sufficient lubrication. It is desirable, therefore, to keep the consistency of the lubricant as close as possible to the boundary between good and poor lubricants already discussed. The viscosity should be high, if the friction is low, and vice versa. The question often arises, which is preferable, an oil of high viscosity or a grease of relatively low viscosity? As regards leaking from the differential into axle housing it would make little difference, but after the lubricant has reached the axle housing an oil will keep on flowing; while a grease, on account of its internal friction, will tend to pile up on the walls of the housing and flow only as more and more accumulates toward the outer end of the housing, and from there finally past the felt washers to the wheels and possibly to the brake drums. This flow along the axle housing will be slower still because the temperature there will be considerably lower than that in the differential compartment. As the internal friction of the grease increases with the drop in temperature, the friction at low temperatures could be very high and yet remain at 125° F., within the limits given above. As regards leakage from the axle housing to the wheels, a grease is therefore superior to an oil.

It should be pointed out, however, that leakage should be prevented by mechanical means primarily and not by the consistency of the lubricant. This could be readily accomplished by giving a reasonable amount of attention to the design of packing, felt washers, etc. The mechanical condition of the axle, wear and design of the parts exerts another influence on leakage. It has been shown that the flow through circular orifices, under any given set of conditions, varies with the fourth power of the diameter. An orifice $\frac{1}{16}$ inch in diameter will give a flow sixteen times as large as an orifice $\frac{1}{32}$ inch in diameter. When the clearances are of a magnitude of a few thousandths of an inch, as in the case of the thrust washers of the differential, a change amounting to a few thousands only will exert an influence on leakage much larger than can be readily produced by varying the consistency of the lubricant. It is also of note that leakage is generally higher if a large amount of lubricant is in the differential compartment,

and for this reason care should be taken that not more than the correct amount is present.

Resistance of grease to gear motion and gear shifting

The resistance of the gear to the turning of the gear should be small in order to give low loss of power and, what is more noticeable, will prevent sluggish action in gear shifting. This is of particular importance in the winter operation of automobiles.

GENERAL DISCUSSION OF GEAR SHIFTING EFFORT

The physical characteristics of transmission lubricants which govern their shifting characteristics are discussed below. The types of lubricants can be classified in four general classes:

Low Cold Test Oils
High Cold Test Oils
Fluid Greases
Semi-Fluid Greases

Lubricants having different viscosity or body representing each class were used.

Variations in transmission design were studied by using three types of transmission, representing modern practice in present day automobiles:

Conventional three speed spur gear type
Three speed Syncro-Mesh type
Four speed internal gear type

Test Procedure

In testing the various lubricants the transmissions were filled to the proper level and then were motor driven for a time, sufficiently long to insure thorough splashing of the lubricant over the entire portion of the transmission assembly such as would occur during regular operation of an automobile. After the above procedure, the transmissions were placed in the cold room and allowed to remain until the entire assembly had reached the desired temperature. The shifting effort was then measured by pulling the shifting lever through from second to high gear, at a uniform rate. In the four speed transmission, however, the shift was made from third to high gear. The effort necessary to shift through the above cycle was determined by measuring the pull in pounds at the end of the shift lever.

The various products tested are shown in the table. All except lubricants A and B are fluid and their viscosities at three temperatures and cold tests are indicated. Lubricants A and B are semi-fluid greases and their consistency at 70° F. is indicated, and the viscosities shown apply to the oils used in the greases.

The results of shifting tests are tabulated below. The kinematic viscosity in poises shown in this table was obtained by extrapolation on the Herschel temperature viscosity chart. The values secured for the semi-fluid greases were obtained by assuming that the viscosity of the oil in the greases determines their effective viscosities at any temperature. Previous work has indicated that the viscosities indicated here would be obtained if they were run in a pressure viscosimeter.

Conclusions

The shifting effort in pounds may be plotted against Kinematic viscosity and shows fairly good correlation. The precision of the shifting effort tests does not warrant a distinction between kinematic and absolute

TABLE 16—Description of Lubricants

Saybolt Viscosity 210° F.	Saybolt Viscosity 130° F.	Saybolt Viscosity 100° F.	A.S.T.M. Solid Test	Description
120	800	2300	+5° F.	Highly refined Mineral Oil
200	1250	3500	−5° F.	Highly refined Mineral Oil
210	1700	4700	+75° F.	Steam refined Cylinder stock
168	1100	3200	...	Special Lead Soap
129	800	2100	...	Special Lead Soap
90	500	1350	0° F.	Winter Transmission Oil
110	650	1800*	...	"A" Semi-fluid Grease, Stormer Viscosity 30–70 Seconds, 700 grams at 70° F.
75	340	850*	...	"B" Semi-fluid Grease, Stormer Viscosity, 30–70 Seconds, 400 grams at 70° F.

* Saybolt Viscosity applies to the viscosity of the oil in the grease.

TABLE 17.—Transmission Shifting Effort Compared with Various Lubricants

Product	Temperature of Test, ° F.	Kinematic Visc. Poises at Temp. of Test	Transmission Shifting Effort, in Pounds: Spur Gear Transmission	Syncro-Mesh Transmission	Internal Gear Transmission
Highly refined	plus 30	350	2	2	2
Mineral Oil	plus 15	1,500	6	5	2½
60 at 210° F.	plus 2	6,000	8	5	4
	minus 14	50,000	10	6	5
Highly refined	plus 32	275	2	1½	1½
Mineral Oil	plus 15	1,000	4	3	3
200 at 210° F.	plus 5	2,000	5	4	4
	minus 10	10,000	14	7	7
Steam Refined	plus 32	1,000	2	2	1½
Cylinder Stock	plus 9	13,000	4½	3	3
	minus 12	200,000	50	45	45
Special Lead	plus 32	300	2½	2	2
Soap	plus 18	900	4	3	3
168 at 210° F.	plus 8	2,200	5	5	7
	minus 14	20,000	25	8	..
Special Lead	plus 30	250	1	1	1
Soap	plus 14	1,000	2½	2½	2½
129 at 210° F.	plus 5	2,200	4½	4½	3½
	minus 10	13,000	13	7	6
Winter Transmission	plus 32	125	1	1	½
Oil	plus 17	400	1½	1½	1
	plus 5	1,300	3	3	2
	minus 12	10,000	4	4	3½
Semi-Fluid Grease	plus 30	200	1½	1	1
Oil Visc. 100 at	plus 10	1,300	3	2	2
210° F.	minus 12	15,000	15	6	8
Semi-Fluid Grease	plus 30	100	1	1	1
Oil Visc. 75 at	plus 18	250	1½	1½	1½
210° F.	plus 8	600	3	2	2
	minus 8	4,000	8	6	4

viscosities. This shows that the shifting effort is proportional to oil viscosity and independent of cold test of the oil or body resulting from soap over the range of products tested.

The slope of the lines with different types of transmissions probably has little significance and may vary for different designs of the same type of transmission.

When the viscosity of the lubricant becomes 200,000 Saybolt seconds (200,000/460 = 435 poises) the shifting effort is about 2 pounds, making gear shifting quite possible. But at 500,000 seconds the effort will exceed 3 pounds and shifting becomes difficult.

TABLE 18—Effect of Different Lubricants at Low Temperatures on Power Required to Turn Shift Gear Transmission (50 R.P.M.)

Lubricant	Grease 1	Oil C	Grease 5	Oil A
S.U. Viscosity of Lubricant at 75° F.	21,000	8550	13,500	24,500
Temperature, ° F.	Torque in pounds at 21" to turn shaft at 50 R.P.M			
30	5.5	16	30	35
40	3.0	8	14	21
50	1.6	5	8	12
60	1.0	3	5	8
70	0.8	2	3	5
80	0.6	1.2	1.8	3

The above table presents the results of dynamometer tests. The indications are that the ultimate viscosity of the lubricant after use is a satisfactory criterion of the resistance to gear motion and gear shifting. Grease No. 1 appears to be an exception to this statement. It has a high internal friction and yet gave in the test series at 50 R.P.M. a much lower resistance to motion than its ultimate viscosity would indicate. This is attributed to the fact that, on account of the high internal friction, large amounts of the grease were able to cling to the wall of the housing without being stirred down. The sample taken from the transmission is, therefore, a mixture of the original heavy grease with portions which had thinned down considerably. Further, the high internal friction permits the grease to pile up on the walls, with considerable channeling around the gears, which in turn reduces the resistance to motion.

Channeling will be of little help, however, when the gears are shifted; that is, when a gear is forced from one location where it has dug itself more or less free into another location and a fresh mass of grease. The conclusion is apparently justified that the resistance to motion, both for turning and shifting, is determined by the ultimate viscosity of the lubricant rather than the internal friction. This is in agreement with theory because the velocities involved are sufficiently high to make internal friction negligible.

The ultimate viscosity of 8,000 to 12,000 seconds at 75° F. is considered satisfactory. Lower values are of some advantage at extremely low temperatures, but higher values will give progressively higher resistance to motion and gear shifting.

STABILITY

A satisfactory transmission grease should not permanently change its consistency if exposed to heat or agitation in storage and handling. After being put into service it should not change through too wide a range. The tests on the six commercial greases show that they all change considerably both under agitating and heating. The first thinning down from the consistency of the fresh grease is common to all and not objectionable, provided it is not progressive and does not go beyond the values determined for satisfactory service behavior.

Such greases, *e.g.,* No. 5 and No. 6, thin down permanently to fairly light oils when exposed to temperatures approaching 150° F. for any length of time. This is very objectionable. If they are used in a Ford rear axle they are too thin and will leak readily. In a shift gear transmission they have too low viscosity and are likely to produce noisy operation.

Other greases, like No. 1 and No. 2, become lumpy or granular masses if agitated for some length of time at a temperature of about 150° F. This is objectionable on account of the poor appearance and also because there is a tendency for it to gum the roller bearings and not flow readily to the parts to be lubricated.

In any case, the grease should neither under agitation nor under the influence of temperatures up to 150° F., thin down permanently to a consistency below that for best service behavior. In general, temperatures not higher than about 130° F. may be expected in the service for which automobile transmission greases are intended. A temperature as high as 150° F. is unlikely, but such a temperature is possible under certain conditions.

It will be noted that of the greases tested only one, No. 4, is considered satisfactory. But, even this grease is slightly too light for Ford rear axles and too heavy for shift gear transmission. The best grease studied would be Grease No. 1, provided it was a stable product. The fact that it breaks down to give a lumpy mass might not be a serious disadvantage as the tests in that regard are not conclusive. However, the probability exists that this lumpy mass of lime soap might gum certain of the wearing parts. Grease No. 5 is satisfactory in every respect as long as it is not exposed to temperatures approaching 150° F.

The fact that all of the greases tested appear to have large sales and give some sort of satisfaction is explained by the fact that the usual differential will be well lubricated even if the lubricant is too thin. The axle will show a tendency to leak, but this need not be serious if the felt washers are kept in good condition, or other mechanical means of preventing leakage are provided. It is also essential that no excess of lubricant is used. Objectionable greases are those which are entirely too heavy even under service conditions.

All of the greases tested will give apparent satisfaction in shift gear transmissions under ordinary conditions. The disadvantage of hard gear shifting will be noticeable in very cold weather. The disadvantage of a relatively high resistance to motion under normal operating conditions

cannot be noticed by the driver, but would be reflected in a slightly higher gasoline consumption if sharp enough observations could be made, which would be possible only by dynamometer tests. By the use of very low viscosity oils of subzero cold test and making use of stable soaps, greases may be made which give good service at 30° below 0° F.

In general, from the results given herewith, and also from a large number of tests not reported, it is concluded that greases made with sodium soaps are much more stable to heat and agitation than the lime soap greases. It has been pointed out that soda greases have the serious disadvantage of losing their grease characteristics when brought into accidental or unavoidable contact with water. The lead and aluminum base greases combine the characteristics of good stability and insolubility in water.

It is of interest that almost any kind of transmission lubricant is likely to give fair results provided extremes in operating conditions are avoided. None of the greases tested and reported in the foregoing sections are thoroughly satisfactory for both differentials and shift gear transmissions. All of the greases studied are unstable in some degree, but wide variations in the magnitude of the stability are noted. Processing greases in colloid mills has been shown to be of some value in the production of lubricants which show little change in service.

While many of the disadvantages of greases as compared with oils have been indicated, it should be pointed out that there are many satisfactory greases on the market which are satisfactory with respect to stability and all service requirements. It is of considerable interest, however, that the majority of greases sold throughout the country do have serious objectionable features.

Steering Gear Grease (Calcium Base)

The use of a calcium soap in a steering gear lubricant is not desirable since calcium soap greases ordinarily are not heat stable but tend to separate as shown by much analytical data. This separation of calcium soap lubricants upon heating has been noted by Ross, Gemmer and Saginaw Steering Gear Companies. The Ross Co., closes the list of Approved Steering Gear Lubricants with the statements: "Avoid use of *Cup Grease,* graphite, white lead or heavy solidified oil." Gemmer uses exactly the same phrase at the close of their approved list of 1931, and in their specifications of 1930 state: "Soaps: Lime soap content is not recommended, due to separations, grit from uncombined lime and magnesia and added water." Saginaw Steering Gear Division, General Motors Corp., after completing a series of tests on Floyd's Steering Gear Lubricant manufactured to General Motors specifications 4567-M (Saginaw Steering Gear Company's SSG-06), make this statement: "With respect to efficiency and corrosion this lubricant is suitable for steering gear use, but under no condition should it be used for this purpose on account of the extreme separation."

The attitude of General Motors toward the use of calcium soap in steering gear lubricants has been somewhat clouded by the fact that the

above condemnation of a calcium soap lubricant is confirmed by their specification, G.M. No. 4608-M, but contradicted by SSG-06, which specifies the use of 5 to 6 per cent calcium soap. Furthermore, R. K. Floyd, in recommending a lubricant for General Motors, states: "It does not matter whether the soap be calcium or soda."

While no instance has been noted in which the major companies condemned the use of aluminum soap, the analytical separation tests, as well as Timken Wear and Film Strength tests, prove that upon heating and cooling aluminum soap lubricants become solid and regain their original consistency only upon working. Since such solid lubricants will not flow to the gears, and since it cannot be worked to its former semi-fluid state until it does reach the gears, there is some danger that gears may become dry and insufficiently lubricated when such a lubricant is used. Obviously, this solidifying property of aluminum soap makes it an ideal component of a lubricant from the viewpoint of lubricant "retention" but lubrication should not be sacrificed for "retention." However, the worst quality of aluminum soap lubricants is that they show very low efficiency at low temperature.

Sodium soap greases, unlike calcium and aluminum soap greases, undergo no change upon repeated heating and cooling. This is confirmed by analytical data, by the Timken Wear Tests, and by the fact that no record is available indicating that any of the major steering gear manufacturing companies condemn the use of sodium soap lubricants.

Tests upon a dynamic efficiency machine confirm that it is possible to blend a sodium soap lubricant which is superior to either calcium or aluminum soap lubricants in both efficiency and retention. Since a sodium soap lubricant possesses heat stability, as well as all the desirable qualities of the calcium and aluminum soap lubricants, there appears to be no justification for the use of these non-heat stable soap lubricants.

Tentative G.M.C. Specification for Steering Gear Lubricant SSG-06 Compared with Experimental Lubricant

General: Steering gear lubricant purchased under this specification shall be a uniform blend of refined mineral oil, sulfur, saponifiable extreme pressure base and a calcium soap-free from excessive water and fillers such as clay, talc, whiting asbestos, wood fiber, etc.

Composition: The composition shall be as follows:

		Experimental Grease
Refined mineral oil	74-76 per cent	74 per cent
Sulfur saponifiable E.P. base	19-21	20
Calcium soap	5-6	5.5 Calcium Soap

The sulfur saponifiable E.P. Base shall contain:

Saponifiable min.	90 per cent	90 per cent
Sulfur, min.	8 per cent	8 per cent

The liquid oil portion, composed of the refined mineral oil blended with sulfur saponifiable E.P. Base, shall have the following properties:

		Experimental Grease
Flash, min.	325° F.	375
Fire	370° F.	425
Viscosity @ 0° F., maximum	18,000	16,000
Viscosity @ 100° F.	240-310	280
Viscosity @ 210° F.	51-55	
Color, maximum	6 NPA 75 per cent kerosene	6 +
Pour, maximum	—20° F.	below — 20
Sulfur, per cent	1.75-2.75	1.86
Saponifiable, per cent	17.5-19.5	18.0

Corrosion: Shall not corrode any metal used for machine construction in the absence or presence of water. OK

Non-Combustible Sediment: Maximum 0.03 per cent OK

Bleeding Test: Shall not develop excessive bleeding or precipitable change in consistency or smoothness when approximately 40 c.c. of the steering gear lubricant is heated in a 50 c.c. Grifin low form beaker for 1 hour at 175° F. OK

Graphite Spring Grease (Calcium Base)

The following formula has been found satisfactory for the manufacture of an automotive spring stabilizing compound containing a minimum of 40 per cent by weight of fine flake graphite.

Constituents	Gallons	Pounds	Percent Net
Tallow	70	533.89	7.49
Hydrated lime		74.00	1.04
30° Bé. caustic soda	½	1.24	.02
Water		71.30	1 00
Graphite		2941.00	41.25
300 pale oil	103	780 73	10.95
60 pale oil	368	2727.84	38.25
		7130.00	100.00

A.S.T.M. worked penetration at 77° F. for the above grease is 288.

Following are the formulae for five graphite spring stabilizing compounds:

No. Formula	1	2	3	4	5
Graphite: Source	Acheson 39	Acheson 39	Acheson 39	Dixon 0572	Dixon 6572
Percentage	45	45	43	43	43
Cup Grease: Grade	Cup No. 0	Cup No. 0	Cup No. 5	Cup No. 5	Cup No. 4
Percentage	55	54	17	23	37
Mineral oil added	None	None	60 Pale 40 per cent	60 Pale 34 per cent	1000 Red 20 per cent
Other materials added	..	Glycerol 1 per cent	...	...	...
Worked penetration	210	289	300	294	270
Water	0.4 per cent	0.4 per cent	0.2 per cent	...	...
Ash	...	...	0.75 per cent	...	...
Mineral oil, S.U.V. at 100° F.	300-330	300-330	80-100 est.	90-110 est.	480-510 est.
Soap, per cent by weight (A.S.T.M.)	est. 5.0 per cent	5.0 per cent est.	5.24 per cent	7.0 per cent est.	7.0 per cent est.
Kind of soap	Calcium	Calcium	Calcium	Calcium	Calcium

Recommendations of Spring Manufacturer "A" for Graphite Spring Stabilizing Compounds

This product shall consist of 40 to 50 per cent by weight of flake graphite and 50 to 60 per cent by weight of a base material or vehicle for the

graphite. The graphite in this compound must be of crystalline variety, not amorphous graphite, all of it passing through a No. 200 mesh screen and at least 75 per cent through a No. 325 mesh screen, and 90 to 92 per cent Graphite Carbon Content is specified and not simple Carbon Content. A grade No. 0572, manufactured by the Joseph Dixon Crucible Company, is entirely satisfactory. The base material shall maintain a uniform consistency in the temperature range from zero to 150° F. and shall not emulsify with water.

The analysis of a base material which they found satisfactory was:

Water		.40 per cent
Alcohol		.82
Total volatile at 100° C.		1.22
Unsaponifiable oil		87.96
Sp. Gr. at 15.5° C.	.9157 = 23° Bé.	
Viscosity at 100° F. (Saybolt)	480 Sec.	
Viscosity at 210° F. (Saybolt)	54 Sec.	
Flash	406° F.	
Fire	473° F.	
Saponifiable matter		9.91
Saponification value	200	
Iodine value (Wiji)	65.8	
Titre	35.3° C.	
Ash		0.91
Ash consists of lime (CaO)	.86 per cent	
Oxides of iron and aluminum	.02	
Magnesium oxide (MgO)	.02	

GRAPHITE SPRING STABILIZING COMPOUND

The following is the analysis of this Compound:

Physical Tests:	
Appearance	Black
Odor	Slightly soapy
Penetration, A.S.T.M. worked @ 77° F.	305
Penetration, A.S.T.M. unworked @ 77° F.	308
Composition (per cent by weight)	
Soap	6.9
Moisture	0.4
Graphite	42.5
Oil	48.9
Free alkali as $(Ca(OH)_2)$	0.3
Unaccounted for	1.0
Total	100.0
Mineral Oil, Extracted:	
Gravity ° A.P.I.	24.5
Color, N.P.A.	7–
S.U.V. at 100° F.	229
Flash, ° F.	375
Fire, ° F.	445

Ash (per cent by weight):	2.25
Analysis, per cent by weight:	
Acid insoluble (silica and silicates)	50.4
Iron and aluminum (mainly iron as Fe_2O_3)	21.4
Calcium as (CaO)	20.9
Unaccounted for (sodium present)	7.3
Total	100.0
Fatty Acid from Soap:	
Acid value (mgs. KOH/gr.)	203
Iodine value (Hanus)	52
Color	Light brown
Dropping point ° C.	44
F. Graphite:	
Graphitic carbon, per cent by weight	92.6

NATIONAL GRAPHITE SPRING GREASE

The results of an analysis of this grease are as follows:

Physical Tests:	
Appearance	black
Dropping point ° C.	99
Odor	soapy
Penetration at 77° F.	
Composition:	
Oil, per cent by weight	74.4
Soap, per cent by weight	9.6
Filler (graphite), per cent by weight	12.9
Moisture, per cent by weight	2.2
Free fat, per cent by weight	2.1
Free alkali ($Ca(OH)_2$), per cent by weight	0.06
Gain in analysis	−1.26
	100.00
Filler:	
Ash, per cent by weight	0.51
Per cent through 200 mesh screen	97.8
Ash, per cent by weight:	1.25
Analysis:	
Calcium CaO, per cent by weight	93.2
Acid insoluble, per cent by weight	3.4
Not analyzed, per cent by weight	3.4
	100.00
Fatty Acids from Soap:	
Acid value (mg. KOH/gm.)	202
Iodine value (Hanus)	45
Color	light brown
Melting point	42
Mineral Oil:	
Color, N.P.A.	7+
S.U.V. at 100	204
S.U.V. at 210	46
Gravity, ° A.P.I.	21.3

TABLE 19—Flow Characteristics of Seven Graphite Spring Stabilizing Compounds (Temperatures near zero ° F.)*

Run No.	Pressure lbs./sq. in.	Bath temp., ° F.	Grease temp., ° F.	Time, seconds	Weight of grease, gms.	Rate of flow, gms./min.
			45% Graphite + 55% Cup No. 0			
1	100	−2°	−1°	60	2.411	2.411
2	100	−1°	−3½°	60	1.324	1.324
3	100	−4°	−2°	60	2.278	2.278
4	100	−2°	+1°	60	2.951	2.951
5	100	−6°	−2°	60	2.026	2.026
6	100	0°	+2°	60	3.267	3.267
			99% (45% Graphite + 55% Cup No. 0) + 1% Glycerol			
1	100		−2°	60	3.303	3.303
2	100		0°	30	1.293	2.586
3	100		0°	60	2.891	2.891
4	100		2°	60	3.439	3.439
5	100		6°	60	4.934	4.934
6	100		12°	60	6.751	6.751
			17% Cup No. 5 + 43% Graphite + 40% 60 Pale Oil			
1	100	−9°	−1°	30	18.935	37.870
2	100	−18°	−6°	30	13.180	26.360
3	100	−4°	2½°	20	16.468	49.404
4	100	−7°	0°	30	20.200	40.400
5	100	−4°	−2°	30	17.436	34.872
6						
			23% Cup No. 5 + 34% 60 Pale Oil + 43% Dixon's 0572 Graphite			
1	100	−6°	0°	30	10.336	20.672
2	100	−4°	+1½°	30	13.143	26.286
			37% Cup No. 4 + 20% 1000 Red Oil + 43% Dixon's 0572 Graphite			
1	100	−13°	−2°	120	2.572	1.296
2	100	−2°	−2°	90	1.713	1.142
3	100	0°	+1½°	90	2.490	1.660
4	100	6°	5°	90	3.107	2.071
5	100	8°	8°	90	3.845	2.563
			Lubropak			
1	100	−8°	−4½°	30	4.779	9.558
2	100	−1°	−1½°	30	7.647	13.294
3	100	0°	+1½°	30	10.338	20.676
4	100	+2°	3½°	20	7.941	23.843
5	100	4°	5°	20	9.521	28.563
6	100	7°	7½°	20	10.925	31.875
			Graphite Grease Sold by Anderson Mfg. Company			
1	100	−8°	−2°	60	8.584	8.584
2	100	−10°	−3°	45	4.300	5.740
3	100	3°	0°	30	7.617	15.200
4	100	−7°	−1½°	30	5.520	11.040
5	100	−2°	−½°	30	6.583	13.166
6	100	+2°	+2½°	30	8.700	17.400
7	100	−7°	−2½°	30	5.021	10.042
8	100	−3°	−1½°	30	5.822	11.644
9	100	−1°	0°	30	7.372	14.744

* Tests were made with Saybolt Furol viscosimeter tube.

Service tests have shown that there is no great distinction between lubrication efficiency when applying the spring stabilizing compound with the weight of the car either on or off the springs. Where the springs are not equipped with covers it is, of course, necessary to apply the grease directly to the spring leaves which have been forced apart with a special chisel. The grease is best applied through a small nozzle attached to the hose of the usual high pressure greasing system.

Bergen [75] has recommended the following compound for leaf springs:

	Parts by Weight
No. 4 cup grease (heavy cup grease)	2
Whiting	1
Perfume.	

Russell's [75a] Spring Grease has the following formula:

	Per Cent by Weight
White lead	83.1
Powdered graphite	5.2
No. 3 cup grease	10.4
Glycerin	1.3
	100.0

INDUSTRIAL GREASES

Crusher Greases

The lubricating grease requirements for rock crushers and auxiliary equipment are not essentially different than for many other forms of machinery and it is not considered that special greases are necessary; however, in many cases the oil viscosity should be higher than normal. Where Pitman bearings found in jaw crushers are equipped with grease cups, Standardized Cup Greases No. 3 or No. 5 should be used, depending on the season. These same greases may be used on eccentrics and other wearing parts. No. 3 cup grease may also be used on roll crusher, and ball and tube mill shaft bearings, rotary screen bearings, vibrating screen bearings and guides, and rock conveyer roll bearings.

Where a non-separating grease is desired, that having the formula for Australian Heavy Duty Cup Grease made with the lime soaps of oleic and stearic acids, and discussed in a previous section will be found particularly useful.

Asbestos Cup Grease

Jackson [76] has proposed the following formula for a consistent lubricating grease:

	Per Cent by Weight
Cup grease	39.0–91.5
Asbestos floats	5–50
Ground soapstone	1–10
Ground borax	1–2.5

[75] U. S. Patent 1,486,563 (Mar. 11, 1924).
[75a] British Patent 414,948 (Dec. 7, 1933).
[76] U. S. Patent 1,097,549 (Jan. 22, 1914).

Curve Greases

For a discussion of the general requirements of curve greases reference should be made to Chapter VI, as these products are normally made with the lime soaps of rosin, or rosin oil, in combination with heavy petroleum distillates. Curve greases are applied by hand to the sides of street railway tracks at points where there is appreciable curvature, in order to reduce friction and wear on car wheel flanges.

Where, for purposes of standardization, the use of a rosin sett grease has been decided against, a black cup grease such as Standardized Black Cup Grease No. 2, having an A.S.T.M. Unworked Penetration value of about 360 at 77° F. may be employed. Curve greases are also used for the lubrication of switch points, frogs, etc.

Matsunawa [77] has prepared greases which are emulsions of lubricating oil and water stabilized by petrolene. It is understood that petrolene consists of asphaltenes and maltenes derived from petroleum asphaltic residua. Such products could be utilized as curve greases providing the petrolene can be obtained at a sufficiently low cost.

Crank Pin Greases

A special heavy bodied, dense cup grease made with very viscous oil is required for the satisfactory lubrication of crank pins and wrist pins found on the donkey engines used for construction work and in the logging industry. Heavy black oils having a viscosity as high as 250° at 210° F. have been found in grease used in this service. The first three greases are for light service.

Dark Crank Pin Grease, Soft

Formula

	Per Cent by Weight
Cup grease (Formula No. A, Grade 2)	95.00
16° Bé. fuel oil	5.00
Melting point	155° F.

Dark Crank Pin Grease, Medium

Formula

	Per Cent by Weight
Cup grease (Formula No. A, Grade 4)	95.00
16° Bé. fuel oil	5.00
Melting point	165° F.

Dark Crank Pin Grease, Hard

Formula

	Per Cent by Weight
Cup grease (Formula No. A, Grade 5)	95.00
16° Bé. fuel oil	5.00

[77] British Patent 388,466 (Oct. 3, 1931).

Heavy Duty Crank Pin Grease, Soft

Formula

	Per Cent by Weight
No. 1 lard oil	15.00
Hydrated lime	2.46
100 pale oil	56.60
Water	1.40
Oil of mirbane	.20
California residuum	23.34

PROCEDURE

The usual method of manufacturing cup grease in an open steam jacketed kettle is satisfactory for the production of this grease.

CHARACTERISTICS OF PRODUCT

A.S.T.M. penetration	154
Melting point	184° F.

Heavy Duty Crank Pin Grease, Medium

Formula

	Per Cent by Weight
No. 1 lard oil	20.50
Hydrated lime	3.37
100 pale oil	51.08
Water	2.75
Oil of mirbane	.30
California residuum	22.00

CHARACTERISTICS OF PRODUCT

A.S.T.M. penetration	128
Melting point	201° F.

Heavy Duty Crank Pin Grease, Hard

Formula

	Per Cent by Weight
No. 1 lard oil	23.50
Hydrated lime	3.86
100 pale oil	48.70
Water	2.75
Oil of mirbane	.30
California residuum	20.89

CHARACTERISTICS OF PRODUCT

A.S.T.M. penetration	85
Melting point	205° F.

Special Grade

An extra hard grade of this grease is often used under severe conditions. It is made on the same formula but with about 30 per cent of fat. It should have a penetration of 60.

The lubricant termed Australian Heavy Duty Cup Grease, the formula and characteristics of which were given previously will be found to give satisfactory service for many crank pin applications.

Analysis of Soft Crank Pin Grease "K"

Physical Properties:	
Worked penetration at 77° F.	325
Dropping point, ° C.	81
Composition, per cent by weight:	
Free alkali, as NaOH	0.018
Water	1.3
Filler	0.5
Soap (as calcium oleate)	8.8
Petroleum oil	90
Properties of Extracted Oil:	
Sayb. Furol Visc. at 100° F.	189
Flash, o.c. ° F.	430
Gravity, ° A.P.I.	18.5

Double Decomposition Greases

Double decomposition calcium base greases are seldom encountered in modern lubrication practice. Hudnall and Calvert [78] have proposed a grease made by stirring air slaked lime with signal oil, adding black oil, heating, adding a hot solution of sodium soap, stirring and allowing to cool.

In another process rape seed oil is boiled together with a mixture of calcium hydroxide and caustic soda to produce a predominance of calcium soap. This product is then incorporated with mineral oil to produce a grease.

Drawing Compounds

Petrolatum and Standardized Cup Greases No. 2 and No. 3 have been utilized in wire drawing operations.

Formula for Special Wire Drawing Compound

	Per Cent by Weight
Beef tallow	15
Hydrated lime	2.25
Water	2.0
100 S.U.V./100° F. western neutral	80.75

Cup Greases to which have been added various extreme pressure agents, such as sulfurized fatty oils, chlorinated naphthalene, and organic phosphates, appear to improve the efficiency of the wire drawing operation. Lutz [79] has patented the process of wire drawing in which the lubricant is controlled with respect to its pH value. The lubricant this inventor prescribes consists of a mixture of soap, fat and water. In British Patent 12,358 (May 26, 1913) wire drawing lubricants containing sulfur, selenium

[78] British Patent 22,867 (Dec. 14, 1900).

[79] U. S. Patent 2,040,321.

or tellurium are suggested as suitable extreme pressure bases. No doubt these materials could be incorporated with suitable grades of cup grease in order to improve their efficiency. Wood [80] has found that carbon tetrachloride, in combination with finely divided graphite, is a suitable dye lubricant for magnesium base alloys. It is, of course, well known that both graphite and carbon tetrachloride have definite extreme pressure properties.

Elevator Guide or Slide Greases

Greases under this heading are applied, usually by hand, to elevator guides and slides. They may also be applied to the plunger which serves to lift the elevator platform in the hydraulic type elevators. In this latter case the grease should be waterproof and should contain no harmful acids or abrasives that would injure the rod or attack the packings.

The following lime base graphite grease will be found suitable for most general applications to elevator slides and guides:

	Per Cent
Beef tallow	15.00
Hydrated lime	2.07
Caustic soda	.02
Fine flake graphite	5.50
100 S.U.V./100 western pale neutral oil	77.41

This grease should have an A.S.T.M. worked penetration value of 240 to 270 at 77° F., an Ubbelohde dropping point of not less than 185° F., and a maximum ash content of 3.2 per cent by weight. The free acid and free alkali contents should not exceed 0.40 per cent.

Extreme Pressure Cup Greases

There is need for the further development of greases of this type. The normal cup greases which have been discussed in the foregoing sections may be compounded with various organic chlorides, tellurides, phosphates, sulfides, and sulfurized fatty oils to improve their film strength properties. The literature of the past gives many instances where investigators attempted to improve their lubricants by incorporating with them materials which would act as cooling agents when they are applied to hot running bearings. In recent times, the fear of incorporating abrasive agents which would increase the wear of anti-friction bearings has prevented any extensive use of many of the substances which were once common.

It is, of course, obvious that Peters' [81] lubricant for instance, consisting of hard tallow, hydrated lime, black oil, wood ashes, salt, sulfur, and graphite could not be used in competition with modern cup greases, but at the same time, certain bearings running hot due to overloads might be cooled to a considerable extent by the application of such a concoction, even though wear was greatly increased. Perhaps some modern investigator may find the germ of an idea for developing a satisfactory lubricant from

[80] U. S. Patent 1,946,121.

[81] U. S. Patent 174,738 (Feb. 21, 1876).

the formula developed by Knowles,[82] which lists among other materials: ammonium hypochlorite, potassium cyanide, whiting, lime, potassium nitrate, lard oil, and silver nitrate.

Adams [83] has developed the following formula:

	Per Cent by Weight
Mica	44.75
High viscosity mineral oil	50.00
Cup grease	5.00
Powdered sulfur	.25

Montgomery [84] has proposed a soft lubricant containing the calcium soap of olive oil and tallow, together with powdered sulfur.

Since many extreme pressure agents depend on acid forming constituents for their effectiveness, it is not surprising that excesses of free alkali may impair their efficiency.

Graphite Cup Greases

These greases may be made by the addition of from one-tenth to ten per cent of powdered, flake or colloidal graphite to any of the cup greases already described. These greases find a wide variety of uses and are of special value where the lubricant is liable to be washed away, as on marine equipment, elevator slides, water pumps, etc.

The graphite may be worked into the grease while near its drawing temperature or incorporated with the cold grease in a grease mill.

Graphite Cup Grease

Formula

	Per cent by weight
Cup grease (Stand. cup grease No. 3)	93.00
Medium ground graphite	2.00
American talc	5.00

Graphite Lubricant

Formula

	Per cent by weight
Cup grease (Stand. cup grease No. 4)	86.29
Steam refined cylinder stock	6.80
Powdered plumbago (graphite)	6.91

Marine Graphite Grease

Formula

	Per cent by weight
Cup grease (Stand. cup grease No. 3)	92.00
Fine ground graphite	8.00

Special Graphite Grease

Formula

	Per cent by weight
Hard tallow	10.00
Dark petrolatum	80.00
Fine graphite	10.00

[82] U. S. Patent 176,011 (Feb. 8, 1876).
[83] U. S. Patent 1,066,971 (July 8, 1913).
[84] British Patent 2,154 (May 8, 1882).

Formulae for Standardized Graphite Cup Greases

Formulae for four greases are as follows:

Constituents	Gallons	Pounds Net	Per Cent by Weight
Graphite Grease No. 1			
Tallow	37	282.20	7.43
Hydrated lime		39.00	1.03
30° Bé. caustic soda	¼	.62	.02
Water		30.40	.80
Graphite		190.00	5.00
300 pale oil	428	3257.78	85.72
		3800.00	100.00
Graphite Grease No. 2			
Tallow	78	594.90	10.44
Hydrated lime		82.00	1.44
30° Bé. caustic soda	½	1.50	.03
Water		45.60	.80
Graphite		285.00	5.00
300 pale oil	616	4691.00	82 29
		5700.00	100.00
Graphite Grease No. 3			
Tallow	100	762.70	14 28
Hydrated lime		105.00	1.97
30° Bé. caustic soda	½	1.24	.02
Water		96.12	1.80
Graphite		300.00	5.62
300 pale oil	535	4074.94	76.31
		5340.00	100.00
Graphite Grease No. 6			
Tallow	200	1525.40	31.83
Hydrated lime		210.00	4.38
30° Bé. caustic soda	1	2.49	.05
Water		143.70	3.00
Graphite		250.00	5.22
300 pale oil	349	2658.41	55.52
		4790.00	100.00

Graphite cup greases made on the foregoing formulae will comply with the following requirements:

Grade	1	2	3	6	
A.S.T.M. worked penetration at 77° F.	360-390	300-330	240-270	50-80	Unworked
Ubbelohde dropping point, ° F., Min.	175	180	185	200	
Percent ash, A.S.T.M., max.	2.4	2.6	3.2	4.8	
Percent free alkali, A.S.T.M., max.	.35	.40	.45	.70	
Average calcium soap content, A.S.T.M.	9	12	16	30	
Percent free acid, A.S.T.M., max.	.30	.30	.50	.50	
Percent water by distillation, A.S.T.M., max.	1.75	2.00	2.50	3.50	

The manufacturing procedure followed in preparing the Standardized Graphite Cup Greases is quite similar to that for making the similar grades of normal cup grease. When the quick consistency test made on a sample of the cup grease at 150° F. indicates the desired consistency, the fine powdered graphite, dispersed in some of the mineral oil is run into the grease mixer while the agitating paddles are in operation.

The Standardized Graphite Cup Greases may be used in accordance with the following suggestions:

Application by means of grease cups (also air cushion cups, Alemite fittings, Zerk or Industrial Dot fittings) to bearings operating with normal loads, and clearances where excessive heating is not encountered:

Grade	Bearing Surface Speed (feet/min.)
1	High, over 300
2	Medium High, 200-300
3	Medium, 100-200
6	Slow, 0-100

U. S. Government Graphite Greases

The Government requirements are in brief:

Grade	Soft	Medium	Hard
Percent Water, max.	1.5	2.0	2.0
A.S.T.M. worked penetration	300-355	240-290	190-230
Mineral oil content, Percent, min.	85	80	75
S.U.V./130° F. min.	125	125	125
Flash ° F. min.	340	340	340
Fire ° F. min.	370	370	370
Graphitic carbon content of graphite used, percent min.	82	82	82
Percent graphite in grease	2-3	2-3	2-3
Fillers; percent free CaO, max.	0.1	0.1	0.1
Corrosion test (530.4)	Neg	Neg	Neg

The formulae of the Standardized Graphite Cup Greases may be readily adjusted with regard to graphite content and oil content to meet these requirements.

English Graphite Cup Grease

Formula

	Per cent by weight
100 S.U.V./100° F. Spindle oil	69.40
White tallow fatty acids	20.00
Calcium hydrate	3.10
Water (in finished grease)	1.50
"Colloidal Silver Graphite"	7.00

This lubricant will conform to the requirements listed below:

A.S.T.M. penetration at 77° F.
Worked 220-260
Unworked 160-180
Ubbelohde dropping point 220° F.

Graphite Chain and Gear Grease

Formula

	Per cent by weight
Oleic acid	17.6
600 Pa. steam refined cylinder oil	36.0
Horse fat or tallow	7.2
Powdered graphite	8.0

The above materials are well mixed together and brought to a temperature of 130 to 150° F. In a separate mixer prepare a special sett mixture of:

	Per cent by weight
Hydrated lime powder	5.5
Water	4.5
300 S.U.V. pale oil	21.2

A graphite waterproof grease of this type is also useful for the lubrication of wire ropes, chains, exposed gears, cranes, derricks, dredges, steam shovels, pile drivers, pump plungers, hoisting engines, quarry machinery, mining machinery, elevator plungers, elevator guides, fire hydrant valves, and cold roll necks.

Graphite Cold Neck Greases [85]

Formulae

Grade No.	2	3	3½	4	5
Tallow	8	10	10	12	18
Lard oil	4	7	5	6	9.25
Calcium oxide	1.6	2.2	1.7	2.3	3.35
Flake graphite	10.0	4.0	10.0	10.0	4.0
100 S.U.V./100 pale oil	10.0	36.77	9.0	10.0	32.7
100 S.U.V. at 210° F. black oil	66.37	40.00	64.27	59.67	32.67
Melting point ° F.	207	210	202	210	215
A.S.T.M. penetration:					
worked	310	260	230	200	....
unworked	200	150	130	100	70

Graphite Block Grease

In practically all graphite greases the quantity of graphite is less than 50 per cent by weight. Williamson [86] has, however, developed a block grease consisting of a cake of dense artificial graphite free from non-graphitic bonds, and impregnated with lubricants such as cup grease.

General Discussion of Colloidal Graphite Cup Greases

It will be recalled that under the discussions on ball bearing lubrication the use of flake graphite and graphite containing high quantities of ash, was warned against. For rough bearings, however, fine flake and powdered graphite is frequently used in combination with calcium soap greases as indicated in the foregoing formulae. Colloidal graphite (size .001 mm. to .006 mm.) is, of course, less subject to complaints of excess wear as com-

[85] *Correct Lubrication, Bull.:* 71, 1935.
[86] U. S. Patent 1,100,335 (1916).

pared with those often made against flake graphite, particularly when used in anti-friction bearings.[87] When true colloidal graphite is used in grease making, only small proportions are included, as the small size of the graphite improves its effectiveness and the high cost of colloidal graphite tends to prohibit excesses.

Colloidal Graphite Cup Grease Formulae

Formulae for several greases of this kind are as follows:

Grade No.	Fluid	1	1½	2	2½	3	5
Lard oil	5	10.0	12.0	14.0	15.0	16.0	28.0
Hydrated lime	.41	1.38	1.65	1.93	2.07	22.0	3.86
Water	.70	1.20	1.30	1.50	1.60	1.70	2.00
Caustic soda	.01	.02	.02	.03	.03	.04	.05
100 S.U.V./100° F. pale oil	91.88	85.40	83.03	...	...	...	...
300 S.U.V./100° F. pale oil	...	...	...	79.54	78.30	57.26	63.09
Colloidal graphite	2.00	2.00	2.00	3.00	3.00	3.00	3.00
A.S.T.M. worked penetration at 77° F.	*	380	320	310	280	260	140

* Saybolt Furol Viscosity at 122° F.; 40 seconds.

Uses of Colloidal Graphite Cup Greases

These are summarized as follows:

Fluid. This product may be used for ring oiled bearings and slow flow oil cups. If the colloidal graphite tends to settle the lubricant should be stirred prior to use. Some of its applications are: eccentric collar oiled bearings, mine car wheels, sight feed oil cups, and small high-speed bearings in general.

No. 1. This product is semi-fluid and is intended in general for pneumatic tools, small high-speed bearings and high-speed gears. Some of its uses are: air hammers, drills, grinders, clippers, light enclosed gear work, cups where such a product is needed, liquid tight ball and roller bearings, high speed light duty gears.

No. 1½. This is an extra soft cup grease for grooved bearings operating at medium pressures, particularly when pin cups are used for application. Specific uses are: air brake pistons, pressure lubricating systems, elevator cups, engine governors, roller bearing mine car wheels, shop shafting, street car ball and roller bearings, and pin cups, funnel cups, or copper rod cups.

No. 2. For bearings operating at normal pressures, usual clearances, and at surface speeds of 200-300 feet per minute this product may be used in any form of applicator which will handle soft cup grease. Specific uses will include: air brake pistons, pressure lubricating systems and air compression type grease cups in general, and belt conveyor roll cups, blower fan bearing cups, conveyor bearings, cranes, crusher rolls, elevators, griffin mills, power shovels, press bearings, punch presses, pumps, railway automatic signal bearings, Raymond mill roller arms, scales, shears, spring compression cups, and trolley wheels.

No. 3. For surface speeds of 100 to 200 feet per minute and for medium pressures and temperatures, this grease is suggested for use in compression cups. Specific uses include: air compressor cups, blower fan bearings, cement mill bearings, cranes, crusher rolls, engine bearings, flexible couplings, heavy conveyors, mine hoists, paper mills, power shovels, press bearings, pumps, punch presses, shears, and shovels.

No. 5. This grease is intended for use in bearings subjected to heavy duty at surface speeds of 0-100 feet per minute. Some of its applications are: low temperature cement mill bearings, exposed crane bearings, dock machinery, heavy conveyors, heavy pumps, mine hoists, power shovels and tube drawing.

[87] "Memorandum on Solid Lubricants," *Bull. 4, Dept. Sci. Ind. Research, Advisory Council,* (1920).

ANALYSIS OF No. 2 COLLOIDAL GRAPHITE GREASE

A No. 2 colloidal graphite grease was found to have the following analysis:

Physical Tests:	
Appearance	Grey
Melting point ° C.	88°
Penetration at 77 A.S.T.M. Worked	280
Odor	Not offensive
Composition:	Per cent
Soap	14.5
Water	2.0
Free fat	.3
Mineral oil (by difference)	79.5
Alkalinity (CaO)	.6
Graphite	3.1
Total	100.00
Ash, 1.53 per cent:	Per cent
Calcium (CaO)	94.80
Aluminum (Al_2O_3)	1.91
Sulphates (SO_3)	1.97
Undetermined	1.32
Total	100.00
Fatty Acids from Soap:	
Acid value (Mgms. KOH)	205
Iodine value	47.5
Color	Brown
Melting point	42° C.
Mineral Oil:	
S.U.V. at 100° F.	100
Gravity at 60° F.	23.3

Graphite Pantograph Grease

For the lubrication of bearing surfaces on pantographs used on electric locomotives which receive their power through an overhead wire, a special grease containing a high percentage of graphite is necessary. The formula for one such grease is:

Constituents	Gallons	Pounds Net	Per Cent by Weight
Tallow	150	1138.05	17.50
Lard oil	50	380.35	5.86
30° Bé. caustic soda	1	2.49	.04
Hydrated lime		210.00	3.23
300 pale oil	320	2461.61	37.87
Water		162.50	2.50
Graphite		2145.00	33.00
		6,500.00	100.00

This product which will cost approximately 7.8c per pound to manufacture will fulfill the following requirements:

Soap content	23-27 per cent
Free acid, max.	0.50 per cent
Free lime, max.	0.25 per cent
Water, max.	2.25 per cent
Grit	Less than 1 per cent by weight of sand, silicates, etc., including ash from graphite. The use of Dixon's No. 0607 graphite will permit this specification.

Graphite Gold Dredge Greases

The lubrication of the underwater tumbler bearings of gold dredges constitutes a severe lubrication problem, as these bearings operate in a slurry of water, earth and grit. Side stresses tend to open the bearing, and if it is worn it acts as a suction pump drawing the abrasive slurry onto the lubricated bearing surfaces. These bearings are more than a foot in diameter and are normally babbit lined.

Formula for Graphite Dredger Grease

	Per Cent by Weight		Per Cent by Weight
Acheson No. 39 graphite	25	Tallow	6.60
Asbestos floats	5	Lard	2.20
Cup grease No. 3	54	NaOH	02
300 pale oil	16	Water	.87
	—	Lime	1.21
	100	300 pale oil	59.10
		Graphite (Acheson No. 39)	25.00
		Asbestos floats	5.00
			100.00

Analysis of Typical Commercial Gold Dredger Grease

Unworked penetration at 77° F.	228
Worked penetration at 77° F.	238
Composition, per cent by weight:	
Filler (graphite)	34.3
Soap (calcium stearate)	12.9
Water	0.6
Free acid as oleic	0.7
Mineral oil	51.3
Unaccounted for	0.2
Total	100.0
Extracted Mineral Oil:	
S.U.V. at 100° F.	306
Gravity, ° A.P.I.	20.2
Graphite:	
Graphitic carbon, per cent by weight	14.8
Grade	medium fine
Dirt	present
Ash, per cent by weight:	4.00

Ash analysis, per cent by weight:	
Acid insoluble	43.3
Iron and aluminum oxides, (Mostly Fe_2O_3)	19.9
Calcium oxide as (CaO)	32.7
Unaccounted for	4.1
Total	100.0
Fatty Acid from Soap:	
Acid value, mg. KOH/gr.	196
Iodine value, (Hanus)	48
Dropping point ° C.	42

ANALYSIS OF VA LOWER TUMBLER GREASE

This analysis showed the following results:

Worked penetration, at 77° F.	263
Dropping point, ° C.	88
Color	Black
Graphite, per cent by weight	29.9
Asbestos, per cent by weight	5.2
Free acid as Oleic, per cent by weight	1.13
Water, per cent	1.7
Ash, per cent by weight	7.21
Ash analysis, per cent by weight:	
Insol. in acid	48.5
Iron and aluminum as H_2O_3	29.0
Calcium as CaO	19.0
Magnesium as MgO	1.1
Undetermined	4.4
Extracted mineral oil:	
S.U.V. at 100° F.	172
Gravity, ° A.P.I.	22.1

Bentonite Grease

Olsson [88] has proposed the use of colloidal bentonite as a substitute for graphite in lubricating greases. A formula in accordance with his views would be:

	Per Cent by Weight
No. 3 Cup grease	90
Bentonite	5
Water	5
	100

Hair and Wool Greases

Many combinations of various types of cup greases or graphite cup greases with goat hair, wool, horse hair, and the like are possible. The object of incorporating these fibers with the lime base grease is to produce a compound which will hold the grease in contact with a rotating journal, yet will not permit the lubricant to be consumed excessively. Such lubricants are obviously makeshifts, yet they may be called for from time to time. Both sodium base and lime greases may be mixed with animal hair. For

[88] U. S. Patent 1,517,577 (Dec. 2, 1924).

low temperatures, where water is present, the calcium base grease mixture are to be preferred.

Medium Wool Waste Grease

Formula

	Per Cent by Weight
Wool waste	10
Standardized No. 3 cup grease	90
	100

These ingredients must be well worked together, either in a special kneading machine or by hand with rakes, the cup grease and wool waste being weighed out into a suitable trough, or vessel, if the latter mixing method is used. In view of the objectionable odors which develop in greases of this sort it is desirable to add small amounts of preservatives (phenol, formaldehyde, etc.) and antioxidants.

Heavy Wool Waste Grease

Formula

	Per Cent by Weight
Wool waste	12
Standardized No. 5 cup grease	88
	100

Medium Hair Grease (Black)

Formula

	Per Cent by Weight
Goat hair	9
Standardized black cup grease No. 3	91
	100

Wool Yarn Elastic Grease

This product is intended for packing open bearing boxes, where journal speeds are quite high and the yarn greases must not become heavy or sodden. Such bearings are in many instances exposed to dirt, water and abrasives. The object of the yarn and hair is to prevent their entry.

Formula

	Per Cent by Weight
Wool yarn elastic (a mixture of wool yarn with horse hair. The horse hair may be either curled or straight)	8
Standardized cup grease No. 0	92
	100

Frantz has assigned to the Texas Company a patent [89] dealing with the design and operation of a device for incorporating hair, wool waste and wool yarn with lubricating greases. The materials to be mixed, are measured into a horizontal barrel, which is mounted on girth gears, and the barrel put in motion. The animal fibers are subjected to a combined shredding and combing action due to the action of fingers attached to the inside of the barrel.

[89] U. S. Patent 1,564,306 (Dec. 8, 1925).

Jackhammer Lubricants

These are semi-fluid calcium base greases made with refined mineral oils having viscosities ranging from 300 to 1000 S.U.V./100° F. The following grease is made by adding 600 S.U.V./100° F. Texas Red Oil to a lime base gear grease:

Formula

	Per Cent by Weight
Tallow	1.08
Hydrated lime	.17
30° Bé. caustic soda	.01
Water	.33
100 S.U.V./100° F. pale oil	15.22
162 S.U.V./210° F. lub. dist.	15.70
600 S.U.V./100° F. Texas red oil	67.49

SPECIFICATIONS:

Saybolt Viscosity, Furol at 122° F.	70-80

Launching Grease

The chief requirements of launching greases are that they should not be readily washed away from the planks over which the ship must pass and that they should be of low cost. We, therefore, find the cheapest cup greases of about No. 4 consistency being used in this service. It usually is not profitable for a grease plant to manufacture a special grease for this purpose unless unusually large orders are received. A grease having worked penetration value of 195 to 250 and made with low viscosity oils, either pales or black, will usually be found satisfactory. In some cases calcium-rosin soap base greases are used. (See Chapter VI, Skid Greases).

In the past, tallow alone was used for launching ships, but the lower price of cup greases suitable for this purpose has ended this practice. The grease, as mentioned above, should be soft enough to smear on the shipways in any weather without resorting to heat for softening the grease. A failure of the ship to launch readily may be attributed to an unsatisfactory grease, but more often it may be explained by the design and construction of the ways.

Calcium Base Liquid Greases

Calcium base liquid greases are mineral lubricating oils thickened with small quantities of calcium soap. They are generally manufactured by working a large quantity of mineral oil of suitable grade into a soft grade of cup grease. Many different brands and consistencies of liquid greases are found on the market, made of varying amounts of lime soap, free fat, and mineral oil of both asphalt and paraffine base, with a wide range of viscosity.

Liquid greases to some extent replace engine and general machinery oils as well as air compressor oils. It is believed that such soap-thickened oils have lower coefficients of friction than mineral lubricating oils having

equal viscosity, under the same conditions of service. In line with this view, one large manufacturer of lubricants is marketing a soap-thickened oil for the lubrication of automobile engines and other internal combustion engines. Both lead and soda soaps are being used for this service. (See chapter on lead base greases.) The lead soap has small or no tendency to thicken the oil, but as little as 0.1 to 0.2 per cent of anhydrous sodium soap causes an increase of from 100 to 200 in the Saybolt viscosity of an oil of 300 viscosity at 100° F.

Bearings of the ring and chain oil type are often lubricated satisfactorily with liquid greases, the advantage being that the lubricant has little tendency to creep over the shaft or to be thrown onto belts, floors, adjacent woodwork and material in the process of manufacture. In general, liquid greases are more satisfactory lubricants at low temperatures than oils of equal viscosity at room temperature of around 80° F.

Satisfactory liquid greases should contain just enough water to prevent separation of the calcium soaps but not a large excess which would cause the resulting product to be cloudy. Lime in excess of the theoretical amount required for the quantity of fat present in the grease should not be employed, as it has a tendency to cause precipitation of the lime soap. Such a grease is also more susceptible to soap separation when a portion of the moisture may become evaporated from the product in service or in storage in warm places. Heat stable liquid greases are made by some manufacturers.

Medium Grade Liquid Grease

Formula	Per Cent by Weight
Calcium soap (Cup grease formula A)	4.5
Asphalt base pale oil 180 vis. at 100° F.	28.8
Asphalt base pale oil 450 vis. at 100° F.	66.6

The analysis of a liquid grease of low viscosity for light conditions, and for use in air drill lubrication follows:

	Per Cent by Weight Analysis
Combined fat	1.94
Free fatty acids	.14
Free fat	.40
Combined CaO	.22
Free CaO	Trace
Water	Trace
Oil (20° Bé., 220 vis. at 100° F.)	97.30

Standardized Liquid Grease

Formula	Per Cent by Weight
Standardized cup grease No. 2	18
300 pale oil	82
	100

Composition

	Per Cent by Weight
Tallow	1.85
Lard oil	.62
Hydrated lime	.34
30° Bé. caustic soda	...
Water	.27
300 pale oil	96.92
	100.00

This product meets the following specifications:

	Per Cent
Free alkali, max.	0.20
Free acid, max.	0.10
Ash, max.	0.60
Water, max.	0.75
Corrosion (530.4)	Neg.
Saybolt furol viscosity at 122° F.	35–45 seconds

Heat Stable Commercial Liquid Grease (No. 96)

Analysis

Viscosity, Saybolt Furol at 122° F. on the grease	325
	%
Oil	93.0
Soap	4.1
Saponifiable matter	1.1
Undetermined	1.8
Total	100.0
Tests on Mineral Oil Extracted:	
Gravity, ° A.P.I.	19.5
Viscosity, Saybolt Universal at 100° F.	629
Ash, per cent	0.41
Ash Analysis:	
	%
Acid insoluble	1.5
Calcium as (CaO)	96.1
Undetermined	2.4
Total	100.0

Heavy Liquid Grease

Formula

	Per Cent by Weight
Tallow	1.860
Lard oil	.620
Hydrated lime	.340
Water	.270
300 pale oil	96.91
	100.000
Saybolt Furol Viscosity at 122° F.	63 seconds

G-M Semi-Fluid Grease Specifications (4615-M)

General: Grease shall be a semi-fluid homogeneous combination of refined mineral oil and calcium soap.

Filler: Shall not contain filler such as clay, talc, whiting, asbestos, wood fiber, etc.

Viscosity: (Saybolt Universal).

Grease: at 100° F.	645–765 seconds
Grease: at 130° F.	225–255 seconds
Mineral Oil: at 100° F.	300–325 seconds
Mineral Oil: at 130° F.	135–150 seconds

Soap: The total soap content shall be 0.75–1.00%. Not less than 93% of the soap shall be calcium (and magnesium) soap. A small amount of caustic soda permissible providing sodium soap does not exceed 7% of the total soap.

Uncombined Lime: ..... Maximum 0.02%

Permanence: The soap and oil shall not separate nor shall the grease harden at reasonable temperatures within a period of three months.

Corrosion: This grease shall not corrode any metal used for machine construction.

Acidity:	Maximum 0.04%
Sand Silicates or Gritty Substances:	Maximum 0.02%

Odor: Neutral.

Methods of Test: Tests shall be made in accordance with method described in G. M. Standards.

Note: This grease is intended as an abrasive vehicle for gear lapping.

Australian Ario Liquid Grease

Analysis

Physical Properties:	
Color	Cloudy Brown
Odor	Petroleum Oil
Sayb. Furol Visc. at 122° F.	39
Appearance	Liquid Grease
Corrosion	None
Composition (Per cent by weight):	
Petroleum oil	96.60
Soap (as calcium oleate)	2.51
Water	0.10
Free alkali (as NaOH)	0.07
Undetermined	0.72
	100.00
Ash, per cent by weight	0.403
Analysis of Ash, per cent by weight:	
Iron and aluminum oxides (R_2O_3)	5.80
Calcium as calcium oxide (CaO)	54.10
Sodium as sodium sulphate (Na_2SO_4)	25.22
Zinc as zinc oxide (ZnO)	14.49
Undetermined	0.39
	100.00
Properties of Petroleum Oil:	
S.U.V. at 100° F.	240
Gravity, ° A.P.I.	22.6
Color, N.P.A.	3½

Loom and Card Grease

Card Grease or Comb Box Grease is the lubricant applied to bearings of textile carding machinery and in particular the "doffer comb" journal. Usually antifriction bearings are employed but where plain babbited bearings are used a semifluid grease is desirable, which by itself will flow, but will not creep out of the box to an objectionable extent. Leakage of the lubricant damages the "card clothing" and it is preferable to use very light colored greases whenever possible. This, of course, means utilizing pale colored lubricating oils and soap stocks.

Typical Formulae for Textile Greases

The following formulae are for three typical greases:

	No. 00 Anti-friction Card Grease	No. 0 Anti-friction Card Grease	No. 3 Card Cylinder Bearing Grease
White acidless tallow	3.00	4.00	17.00
Hydrated lime	.42	.56	2.37
Caustic soda	.03	.04	.16
100 Pale neutral oil (max. color 2, A.S.T.M.)	96.05	94.75	78.97
Water	.50	.65	1.50
A.S.T.M. worked penetration	...	...	250–300
MacMichael viscosity (80° F.—No. 26 Wire)	15–35	30–60	...

Calcium Naphthenates

The calcium salts of naphthenic acid are not very effective as bases for lubricating greases. They appear to go into solution and produce poor yields as compared with animal and vegetable oil lime soaps. It is possible that, since they are quite soluble in mineral oil, they may tend to stabilize dispersions of other less soluble metallic salts of oleic and stearic acid in mineral lubricating oils. Parker [91] has proposed the use of 0.05 to 2.0 per cent of calcium naphthenate in motor oil in order to improve its stability with regard to oxidation and carbon deposition.

Plug Valve Lubricants

Brunstrum [92] has developed the following formula for a waterproof grease intended for the lubrication of plug valves:

	Parts by Weight
Powdered graphite	2
Petroleum residue (M.P. 85–100° F.)	2
Anhydrous calcium soap	1

The Merco Nordstrom grease made with the calcium soaps of castor oil and carnauba wax, referred to in the first sections of this chapter has also been used as a plug valve lubricant.

[91] U. S. Patent 2,001,108 (May 14, 1935).
[92] U. S. Patent 1,982,199 (Nov. 27, 1934).

Mine Car Grease

In old style mine cars, equipped with plain bearings, the lubricant is usually a fairly heavy black oil. With the advent of anti-friction bearings and their adoption by mine car makers, grease lubrication has become a necessity. To prevent the entry and harmful effects of water and grit, it is probably best to use a fairly heavy grade of lime base cup grease made with an oil of viscosity greater than 200 S.U.V./100° F. Either the No. 2 or No. 3 Standardized Cup Greases will be found acceptable.

Purchase specifications for a suitable mine car grease should be about as follows:

A.S.T.M. worked penetration	240–280
Percent ash, max.	2.0
Percent water, max.	2.0
Corrosion	Negative
Mineral Oil Content:	
Flash, min.	340° F.
Fire, min.	380° F.
Pour point ° F., max.	200
S.U.V./100° F., min.	30

Must be resistant to the action of water.

Further data on mine car greases will be found in the section on ball and roller bearing lubrication in this chapter.

While black oils were suitable for the lubrication of old style mine cars equipped with plain bearings, anti-friction bearings now in use require a suitable lubricant. The grease should be of medium consistency and should effectively seal the bearing against entry of dirt and water. Heavy bodied greases are more effective in this respect than soft lubricants, but may be objected to by mine operators in view of the fact that they may be difficult to apply through the grease guns now commonly used for this purpose. While lime base greases are preferable for general purposes, in some dry mines there may be long hauls above ground or down long grades which necessitate the application of brakes with a consequent increase of the temperature of the mine car wheel bearings. In such cases, a high melting point soda soap base grease, made with a viscous oil, has been used with success.

Metal Containing Cup Greases

Ralph Frizell[90] has proposed the addition of finely divided metallic particles to various grades of cup grease to improve their lubricating properties under heavy load conditions. Copper, lead and zinc is suspended in the grease as "comminuted spherical particles."

Pin Cup Greases

The pin or gravity type grease cup is particularly adapted to the lubrication of line shafting and machinery in general where the cup can be attached

[90] U. S. Patent 1,758,598 (May 13, 1930).

directly in a vertical position to the upper half of the bearing. The cups are provided with a soft copper rod which passes through the center of a funnel and rests in a vertical position on the revolving shaft. The service rendered by these cups is of a semi-automatic nature.

An analysis of grease which has given satisfactory service in pin cups for several years is as follows:

	Per Cent by Weight
Free fat	1.7
Free fatty acids	1.04
Combined fat	7.96
Free CaO	.22
Combined CaO	.79
Water	1.25
Mineral oil	86.94
Gravity	25.1
Vis./100 ° F.	241
Cold test	30–35° F.
Melting point	180° F.
Penetration A.S.T.M.	415

Mixed Base Pin Cup Grease

Formula

	Per Cent by Weight
No. 1 lard oil	10.000
Hydrated lime	1.345
Solid caustic soda	.048
Mineral oil (with lime)	2.500
Mineral oil	86.106

Quality of Oil Used

The oil used in this product is a blend of Eastern and asphaltic base lubricating oils:

Paraffine base oil 100 Vis./100° F.	50 per cent (By volume)
Asphalt base oil 650 Vis./100° F.	37 per cent
Asphalt base oil 1743 Vis./100° F.	13 per cent

This oil has the following physical tests:

Viscosity at 100° F.	223
Robinson color	2¾ Plus
Gravity	24.2° Bé.

Procedure

Charge in a pressure cooker the lard oil and the following mixture:

	Per Cent of Total
Hydrated lime	1.345
Caustic soda (dissolved in water to 40° Bé.)	.048
Water	1.400
Mineral oil	2.500

Heat under a pressure of 50 pounds per square inch for one hour.

Discharge soap under pressure to an open mixer and heat to a temperature of 280° F.

Mix oil into the soap until the temperature has reached 200° F. and about 20 per cent of the oil is in.

Add 1.1 per cent of water after scraping down the kettle.
Mix in the balance of the oil while agitating, steam turned off.
Continue agitation for about thirty minutes after all of the oil is mixed in. This done to obtain a worked consistency.
Draw the grease at a temperature of 150° F.

CHARACTERISTICS OF PRODUCT

This grease made with various amounts of soap has the following characteristics:

Soap, per cent	30	25	20	15	11.39
Melting point	197	196	195	183	175
Penetration at 77° F.	45	57	130	245	380

Pin Cup Grease, Steam Cooked

The following product is made with 12 per cent excess hydrated lime over that required for complete saponification of the lard oil.

Formula

	Per Cent by Weight
Extra No. 1 lard oil	12.00
Hydrated lime	1.91
320 Vis./100° F. asphalt base red oil	84.59
Water	1.50

PROCEDURE

The temperature of drawing this grease is 150° F. The water is added at a temperature of 220° F. in order to insure proper hydration of the lime soap. The process is practically the same as for the pin cup grease No. 1.

CHARACTERISTICS OF PRODUCT

The A.S.T.M. penetration of this lubricant is 250 and the melting point 197° F. Only 0.02 gram per hour was consumed in a pin cup mounted on a bearing running at 140° F. At 200° F. 1.28 grams were consumed per hour.

No. 2 Grade "K" Pin Cup Grease

Analysis

Physical Tests:	
Appearance, smooth, dark colored semi-solid grease	
Melting point	92° C.
Penetration at 77° F. (A.S.T.M. worked)	290
Odor	Fatty
Composition (Per cent by weight):	
Soap	11.80
Water	1.20
Free fat	3.35
Oil (by difference)	83.62
Alkalinity (CaO)	.03
Glycerin (present)	
	100.00
Ash:	1.14 per cent
Iron and alumna (R_2O_3)	4.47
Calcium (CaO)	90.00
Undetermined	5.53
	100.00

Fatty Acids from Soap:	
Acid value (Mgms. KOH)	200
Iodine value (Hanus)	73
Color	Brown
Melting point ° C.	35
Mineral Oil:	
S.U.V. at 100° F.	244
Gravity ° A.P.I.	25.3

Discussion of Feed Rates of Pin Cup Greases

Certain grease manufacturers have, in the past, strongly urged the adoption of the pin or gravity type grease cup, and at the same time marketed greases for these devices which fed at a uniform, very slow rate not attainable with ordinary cup greases. These cups may be installed on shafts of from one inch to six inches in diameter, which operate at 50 to 5000 R.P.M. It will be noted from the following data, that some greases, having equivalent dropping points and consistencies, feed at much slower rates through the funnel cup orifice and along the copper rod fitted into this orifice.

	A.S.T.M. Worked Penetration	Melting Point ° F.	Consumption grams/hour at 140° F.
Pin cup grease K	290	198	3.0
Normal cup grease	297	196	24.0

From a study of the analyses of these greases it was evident that the "K" grease contained appreciable quantities of both free fat and free lime which, at the temperature of the cup, either combine to form a more consistent grease, or cause a form of syneresis which leaves a more consistent product that reduces the flow of grease through the funnel cup. This is made more clear by the following table of straight face cone penetrations made on these two greases at temperatures of from 40 to 180° F. It is possible that during the manufacture of greases such as the "K" product that free fat is milled together with the finished grease in the cold.

	Cone Penetration	
Temperature of Grease	Grease K	Normal Cup Grease No. 3
50	310	225
60	350	240
80	405	375
100	410	320
120	395	395
140	352	510
160	375	...
180	420	...

Pneumatic Tool Greases

The following analysis of three greases highly recommended by a well known manufacturer of pneumatic tools, indicates that both sodium and calcium greases find acceptance:

Worked penetration at 77° F.	270	267	227
Unworked penetration at 77° F.	250	258	205
Dropping point, ° C.	152	92	100
Water, per cent	0.55	1.50	0.85
Free alkali, per cent by weight	0.01% NaOH	0.03% $Ca(OH)_2$	Neutral
Soap, per cent by weight	13.0	13.6	13.5
Ash, per cent	2.45	1.55	1.46
Analysis, per cent by weight:			
Acid insol.	...	3.2 (iron and acid insol.)	
Sodium carbonate	93.9	4.4	19.0
Calcium oxide	...	88.3	78.0
Iron oxide	...	...	2.0
Corrosion	OK	OK	OK
S.U.V. at 100° F.	476	382	316
S.U.V. at 210° F.	57.3	48.0	47.1
Gravity of oil, A.P.I.	27.0	23.1	26.3

Standardized Cup Greases No. 3 and No. 4 will give fair service as pneumatic tool lubricants, but will be found inferior to certain soda soap greases which will be discussed later.

General Discussion of Penumatic Tool Greases

Correct lubrication of pneumatic tools can be attained by the use of proper lubricants and the proper method of applying them. The lubrication of all pneumatic tools is indeed a serious problem because this equipment is subjected to very rough treatment, both by the operator and owner. Operators forget to oil the tools during the shift, allowing them to run dry and the owners will allow them to remain "dry" during idle periods. The correct types of lubricants are not always chosen and improper means of applying may be used.

Compressed air lines and storage tanks always contain much water, both in the precipitated and vapor form, which is brought into the compressor by the air and precipitated when the compressed air cools in the lines and tanks. Most of the air line water blows out through the exhaust, yet some passes down the cylinder walls by the pistons, and into the crankcase and reduction gear case. If the grease has emulsifying tendencies towards the formation of oil-in-water types, it will not be readily held on the cylinder walls, thus causing a lack of lubrication.

The effect of the water on the grease in the crankcase is such that its fibrous texture is destroyed. The grease swells, thus decreasing its lubrication value and forces itself out through the stuffing boxes, breathers, and exhaust.

Air motors operate under very steep temperature gradients. The crankcase temperatures reach 200° F. or more under extreme conditions, while it is common for chips of ice to blow out through the exhaust. Also, the temperature of the surroundings, where motors are operating, varies greatly both climatically and with the type of work being done. The steep temperature gradient makes it very necessary for the grease to lubricate well over a wide temperature range. It must reach the valve surfaces and tops of pistons where the temperature is below 32° F. and yet must possess the

requisite body to form a tenacious oil film at temperatures in the neighborhood of 200° F.

Dust, grit, sand, and many other minute abrasive particles are drawn in by the intake of the compressor; these, coupled with the rust, scale and particles of metal from the compressor, tank and pipe lines, are carried into the air tool, thus contaminating the grease. This contamination not only increases wear, due to its abrasiveness, but it adversely affects the formation of oil films. If a good grade of air cleaner is installed at the compressor intake and the air lines thoroughly "blown out" before attaching them to the motor, the harmful effect of these foreign substances as well as water will be greatly reduced.

Channelling of the grease will take place in the crankcase of a reciprocating motor or in the reduction gears if the grease is too hard. If the grease is too soft it will cause excessive leakage or will not cling to the high speed moving parts. The base for the grease must be carefully selected for different reasons.

Since water is always present in pneumatic tools it is considered by some that a soda soap grease is undesirable because of its tendency to form an oil-in-water emulsion. Lime soap greases are more resistant to the action of water upon them, yet they are affected by the severe agitation and the temperature which causes them to separate into their original oil and base phases. Aluminum base greases have not been investigated but from past experience with these greases it is believed that they would become "rubbery" under the agitation and temperature causing an excessive power absorption.

Velocities of moving parts in air motors are quite high for the type and size of machine. A Thor motor, for instance, has a crankshaft speed of 1332 r.p.m. and an average piston rubbing speed of 390 feet per minute for a chuck speed of 200 r.p.m. Other types of pneumatic tools have much higher speeds.

It would appear from the preceding discussion that a host of various greases would be necessary for the proper lubrication of air motors and other pneumatic tools; however, Mr. James I. Clower of the Virginia Polytechnic Institute recommends the following greases and oils for the lubrication of all types of pneumatic tools.

Lubricant	Saybolt Viscosity 104° F.	Saybolt Viscosity 140° F.	Saybolt Viscosity 210° F.	Pour Point ° F.	Flash ° F.	Fire ° F.	Specific Gravity	Compounding*
Oil A	960	350	90	10	360	400	0.940	17%
Oil B	500	190	60	10	350	385	0.930	16%

Grease C A water resistant lime soap grease of soft consistency (No. 2) is recommended by this authority. It should contain no excess free fatty acid or alkali.

* Compounding is the percent of animal or vegetable oil added to the mineral oil. Mr. Clower considers blown rapeseed oil to be excellent for compounding in pneumatic tool oils.

Recommendations

Oil A is recommended for lubrication of heavy mounted machines such as drifters operating under severe conditions.

Oil B is to be used in small hand held tools such as paving breakers, backfill tampers, etc., and light mounted tools operating under all conditions.

Grease C is to be used in motors with reciprocating pistons both in the crankcase and reduction gear housing. In vane type motors this grease is recommended for use in the reduction gear housings.

The selection of a grease for the motors is partly a matter of compromise between the channelling effect and excessive throw-off. Consequently, in order to obtain satisfactory products fibrous texture greases should be considered. This fibrous texture can be found in soda base greases but is not present in mixed or lime base greases.

Calcium Phenyl-Stearate Greases

Gilfoil [93] has reported that calcium phenyl stearate is capable of being used to produce normal cup greases. Such lubricants do not appear to have any particular advantages over calcium oleate or stearate greases. In preparing the phenyl-stearic acid the method of Schmidt [94] was used in which technical oleic acid, c.p. benzol and anhydrous aluminum chloride are the reacting materials. Sodium and lead phenyl-stearate correspond to normal oleates when dispersed in lubricating oils. The increased cost of the phenyl-stearates is, of course, a factor which will limit their use in greases, unless some particular virtue can be discovered for them. As ingredients of liquid greases used for Diesel engine lubrication they have been found effective in reducing piston ring sticking.

Recuperator Greases

The lubricating grease used by the U. S. Government and the army for the recoil mechanisms of 75 and 155 mm. gun carriages has been termed Recuperator Grease. Its requirements are:

A.S.T.M. penetration, worked	240–290
Percent water	1.0–3.0
Corrosion test (copper strip, 3 hours at 212° F.)	Negative
Percent ash, max.	2.3
Percent soap content, approximately	18
S.U.V. of mineral oil content, min.	180

	Formulae	
	I	II
Tallow	15.50	12.00
Lard oil	...	4.00
Hydrated lime	2.20	2.30
Caustic soda	.03	.03
200 pale oil	81.77	...
300 pale oil	...	80.17
Water	1.50	1.50
	100.00	100.00

[93] Notes on Lubricating Greases Made from the Soaps of Phenyl-Stearic Acid," *Ind. Eng. Chem.*, (5), **22**, 487 (May, 1930).

[94] *J. Am. Chem. Soc.*, **52**, 1172 (1930).

Rubber Cup Greases

Latex may be added to any cup grease, either while it is in the process of manufacture and at temperatures of 150 to 300° F., or after they have been cooled to normal temperatures. For complete details the Klemgard [95] patent should be consulted. Ordinarily, not more than about 0.50 per cent of the rubber hydrocarbon should remain in the grease to produce a high degree of adhesion. A stringy texture may also be given to cup greases by adding from .1 to 2.0 per cent of polymerized isobutylene.

Tunnel Bearing Grease

While tunnel bearing greases are no longer in great demand, it is of interest to note that they, in general, were made with soda soap bases in order to withstand the high temperatures encountered and to produce a cake of grease of good wearing qualities. Lime base greases may be used if some arrangement is made to hold them in contact with the bearing. Engel has proposed the saturation of sponges, or sponge-like material such as waste, with a mixture of tallow, rape seed oil, and calcium soap. The material is heated above its melting point and the waste is saturated with the liquid lubricant and allowed to cool.

Twister Ring Greases

The lime base greases used for twister ring lubrication in the textile industry, will vary from a number two to a number four and one-half in consistency, depending on whether wet or dry twisting is being done, and whether the grease is applied by means of cups or by hand. Tallow compounds alone, without soaps, are sometimes used but, if properly manufactured, special cup greases will prove satisfactory. 100 S.U.V./100° F., or even 75 S.U.V./100° F., pale oils should be used in order to reduce friction as much as possible; the soap stocks should be free of fats which tend to oxidize and, in some cases, it is desirable to increase the oiliness of the lubricants by adding free fat of a non-oxidizing character, such as acidless tallow oil.

Roll Neck Greases

These greases will be considered in greater detail in the chapters on rosin greases and mechanical mixtures. The following analyses are of interest, however, in showing the extent calcium soaps are being used in neck greases in Europe.

[95] U. S. Patent 1,975,582 (Oct. 2, 1934).

	VOC Plate Mill Grease No. 1	*H & J Grade Z–48*
Appearance.	Fairly soft, yellow grease.	Fairly soft, yellow block grease.
Melting point (Ubbelohde)	149° C.	124° C.
Ash	4.3 per cent weight	5.8 per cent weight
Nature of ash	Essentially Sodium Carbonate Na_2CO_3 content = 92 per cent weight. (Calculated on the ash.)	Essentially Sodium Carbonate Na_2CO_3 content = 97 per cent weight. (Calculated on the ash.)
Water content	0.3 per cent weight	0.18 per cent weight.
Acid value equivalent to	0.9 Mgm. KOH/1 gm.	0.1 Mgm. KOH/1 gm.
Total free and combined fatty acids	23 per cent weight	29.6 per cent weight
Nature of fatty acids	Rosin acids and animal fatty acids probably derived from Lard Oil.	Rosin acids and animal fatty acids probably derived from Lard Oil.
Analysis of Fatty acids.		
Rosin acids (calc. on total fatty acids)	29.8 per cent weight	35.0 per cent weight
Fatty acids of animal origin (by difference)	70.2 per cent weight	65.0 per cent weight
Properties of fatty acids of animal origin.		
Melting point (capillary tube)	43° C.	36.5° C.
Iodine value	72	86
Valenta test	66° C.	33° C.
Unsaponifiable matter	73.4 per cent weight	65.2 per cent weight
Nature of unsaponifiable matter.	Mineral oil.	Mineral oil.
Physical properties of unsaponifiable matter.		
Specific gravity at 15°/4° (48/29S)	0.905	0.900
Viscosity Red. I. at 140° F.	120 Secs. approx.	75 Secs. Approx.

From the above figures, the probable constitution of these greases is as follows:

Caustic Soda (NaOH)	3.2 per cent weight	4.4 per cent weight
Lump Rosin (Colophony)	7.0 per cent weight	10.0 per cent weight
Auimal fat	16.0 per cent weight (Probably lard)	20.0 per cent weight (Probably lard oil)
Mineral oil	73.3 per cent weight	64.6 per cent weight
Water	0.5 per cent weight	1.0 per cent weight

	H & J Grade AB—1 Soft	*F Miller Grade T—150*
Appearance.	Soft, brownish-yellow block grease.	Soft, brownish-yellow grease.
Melting point (Ubbelohde)	142° C. (with decomposition).	75° C.
Ash	9.5 per cent weight	8.0 per cent weight
Nature of ash	Essentially Calcium Carbonate.	Essentially Calcium Carbonate.
Water content	0.6 per cent weight	1.6 per cent weight
Alkalinity equivalent to	0.5 Mgm. NaOH/1 gm.	1.2 Mgms. NaOH/1 gm.
Total free and combined fatty acids	14.8 per cent weight	28.4 per cent weight
Nature of fatty acids	Essentially Rosin Acids.	Rosin acids, and animal and vegetable fatty acids derived probably from a mixture of Wool Grease and Neat Oil.
Analysis of Fatty Acids.		
Rosin acids (calc. on fatty acids).	92.5 per cent weight	38 per cent weight
Fatty acids of animal and vegetable origin (by difference).		62 per cent weight
Properties of Fatty Acids of animal and vegetable origin.		
Melting point (capillary tube)		41° C.
Iodine value		88 (Confirmed)
Valenta test		80 (Confirmed)
Unsaponifiable matter	77.6 per cent weight	65 per cent weight
Nature of unsaponifiable matter.	Mineral oil.	Mineral oil.
Physical properties of unsaponifiable matter. Specific gravity at 15°/4° C.	0.908	0.906
Viscosity Red. I. at 140° F.	50 Secs. Approx.	50 Secs. approx.
Cholesterol reaction on unsaponifiable matter.		Positive

The probable constitution of these greases is as follows:

Calcium hydroxide ($Ca(OH)_2$)	7 per cent weight	6.0 per cent weight
Lump rosin (colophony)	15 per cent weight	10.0 per cent weight
Animal and vegetable fat.		17.5 per cent weight (Probably Wool Grease and Nut Oil).
Mineral oil.	77.5 per cent weight	65.0 per cent weight
Water.	0.5 per cent weight	1.5 per cent weight

	VOC Duplex Block Grease	*Glycoline Block Grease*
Appearance.	Black, fairly hard and greasy feel.	Hard, dull black, slightly greasy.
Melting point (Ubbelohde)	98° C.	91° C.
Ash	3.5 per cent weight	17.8 per cent weight
Nature of ash	Sodium Carbonate and Calcium Carbonate together, with small amounts of Silica, Ferric Oxide and Magnesia.	Essentially Limestone (Silica, Alumina and Ferric Oxide, Lime and Magnesia) together with a small quantity of Sodium Carbonate.
Alkali content of ash as NaOH	18.1 per cent weight (Calcd. on the Ash).	1.1 per cent weight (Calcd. on the Ash).
Water content	0.8 per cent weight	0.1 per cent weight
Acid value equivalent to	9.1 Mgms. KOH/1 gm.	0.33 Mgm. KOH/1 gm.
Total free and combined fatty acids	32 per cent weight	34 per cent weight
Nature of fatty acids	Probably derived from Wool Grease.	Probably derived from Wool Grease.
Properties of Fatty Acids.		
Melting point (capillary tube)	47° C. (approx.)	42-45° C.
Iodine value of fatty acids	32	22
Unsaponifiable matter	63.5 per cent weight (by difference)	48.2 per cent weight (by difference)
Nature of unsaponifiable matter.	Black viscous material.	Black viscous material.
Cholesterol test of unsaponifiable matter	Positive	Positive
Test for vanadium of ash of unsaponifiable matter.	Absent	Absent

Formulae for Calcium Base Roll Neck Greases

The following formulae are for three such greases:

Roll Neck Grease C

	Pounds
Beef tallow	150
Hydrated lime powder	20
Water	25
Talc	25
Flake graphite	60
Fine powdered graphite	20
Pennsylvania cylinder stock	200
25° Bé. caustic potash	3 (gallons)

The manufacturing process is similar to that of making cup greases.

Cold Neck Grease C

Oleic acid	25 gallons
Hydrated lime powder	141 pounds
Water	30 gallons
1000 S.U.V./100° F. black oil	30 gallons

The process used is that of making cold sett cup greases. See the section on preparing Waterproof Greases.

Roll Neck Grease

	Per Cent by Weight
Oleic acid	20
Water	24
Hydrated lime	16
200 S.U.V./100° F. Texas red oil	20
1000 S.U.V./100° black oil	20

A steel mill roll neck grease of considerable interest was patented by J. Batson [96] in 1884. It consisted of brown Yorkshire wool grease, wool oil grease, graphite, quicklime, suet and tallow with or without curriers grease or "dubbin." These materials were melted together to make a grease, or the suet was formed into small bars and embedded in a matrix formed by melting the remaining materials together.

Schmidt's [97] steel mill roll neck grease has the following formula:

	Parts by Weight
Paraffine wax	95
Black lubricating oil	11
Rosin	25
Air slaked lime	35
Wool grease (degras)	20
Soapstone	50
Ammonium chloride	9

[96] British Patent 1,252 (Jan. 11, 1884).

[97] U. S. Patent 1,780,315 (Nov. 4, 1930).

Chapter VI

Sett Greases

The sett greases are mixtures of the calcium soaps of rosin acids and various grades of mineral lubricating oils. They constitute the cheaper grades of semi-solid lubricants and are important in that they are satisfactory for the lubrication of rough, heavy bearings operating at slow speeds, and in the logging industry where a cheap grease is required for the lubrication of skidways. They find extensive use in gear and street railway curve lubrication. Sett greases are almost invariably made by mixing while cold or at temperatures below 150° F., mineral lubricating oil, rosin oil, and a lime mixture known as the sett. The sett is an emulsion consisting of mineral oil of light viscosity, water, hydrated lime, and a small quantity of the rosin oil which reacts with the lime to produce a small quantity of rosin soap which promotes emulsification. If the lime is not held in a fine state of division in the emulsion, the reaction with the major portion of the rosin oil in the final mix will be uneven and slow, resulting in a lumpy grease and one which is prone to separate.

The final mixture will normally solidify in a few minutes and in some cases less than a minute, and the resultant product will have a satisfactory consistency and be free from lumps or separation of the mineral oil from the lime soap, provided that proper precautions are observed in the selection of the various stocks, that the ingredients are mixed in the correct proportions, and that the mixing has been carried out thoroughly.

In general, sett greases are considered more easily made than cup greases, but due to variations in the rosin oils obtained commercially it has been found difficult to produce sett greases of uniform consistency from day to day.

It should be noted that some manufacturers use small quantities of caustic soda in the manufacture of their sett greases, resulting in the production of greases which have greater stability to mechanical agitation, and which are of particular value where a grease is required to adhere well to wet metallic surfaces such as street railway track curves.

GENERAL PROPERTIES OF ROSIN OILS USED IN SETT GREASES

It is a well known fact that the rosin oils used in the manufacture of axle greases, curve greases, and skid greases are the source of considerable variations in the consistencies of the finished greases. Just what constituents of the rosin oil are responsible for this variation will be discussed later.

It appears that, in general, if the Saybolt viscosity at 210° F., of the rosin oil used is greater than about 250, the time of setting and the consistency and quality of the resulting greases is satisfactory. Rosin oils of high abietic acid content are usually, although not invariably, of greater value as to grease setting properties. In purchasing stocks of rosin oil, it is customary for the rosin oil manufacturer to submit a sample of the product he intends to ship. This sample may be tried out in the laboratory and its grease setting properties determined by making small experimental batches. The chemical laboratory may also make the following inspections of the rosin oil sample:

Gravity—° Bé.
Viscosity—Saybolt at 210° F.
Acidity, in terms of oleic acid.

On the arrival of the stocks ordered it is possible that the inspections of the oil may be practically indentical with those of the preliminary sample found to be satisfactory by the laboratory trial, and yet be of low quality as a grease sett. The reason for this variation is not obvious but is believed to be revealed in the results of the following research on this subject.

Rosin oils are manufactured by the process of distilling rosin obtained from: gum or yellow "dip" obtained directly from pine trees, or from pine wood chips, by means of various organic solvents.

The distillation process may be any of the following: (a) distillation by fire in the usual type of rosin still; (b) distillation by fire in the presence of superheated steam; (c) distillation in vacuo.

Hawley [1] states: "If rosin is carefully distilled in a vacuo a very large proportion can be obtained as solid undecomposed rosin; if distilled slowly at atmospheric pressure a considerably smaller proportion of unchanged rosin is obtained; and if distilled rapidly a still smaller proportion. In practice frequently the unchanged rosin is not wanted and the 'first run' oil is redistilled in order to decompose it further; the 'second run,' 'third run,' etc., oils resulting are thinner and contain less 'rosin acids.' Distillation under pressure might cause the same results as repeated distillations at atmospheric pressure."

"Kidney rosin" or "first run" rosin oil is most usually used as a grease sett. It is made by the rapid destructive distillation of rosin at a temperature of from 400° to 720° F. About 60 to 65 per cent of the rosin is distilled over and recovered as rosin oil suitable for grease manufacture.

LABORATORY TESTS ON ROSIN OILS

In order to determine the effect of the process of manufacture of the rosin oils on their grease-setting properties, lump rosin was distilled under various conditions and the oils were used in making laboratory scale batches of sett greases.

[1] Hawley, L. F., "Wood Distillation," page 111, The Chemical Catalog Co., Inc. (Reinhold Publishing Corp.), New York (1923).

Preparation of the Oils

The rosin oils were prepared by distilling regular grade F rosin of 83.4 to 83.9 per cent acid calculated as oleic.

RUN 1.—Vacuum Distillation of Rosin.

Time	Vacuum, cm. of hg.	Temperature Overhead, ° F.
Start	6.0	153
15 min.	7.0	160
30 min.	7.0	370
60 min.	7.0	540

Yields

	Per Cent by Weight
Distillate	87.2
Loss (gas, etc.)	8.8
Residue	4.0

RUN 2.—Fire Distillation at Atmospheric Pressure.

Time	Temperature of Overhead, ° F.	Temperature of Liquid Rosin, ° F.
Start	190	400
1 hour	565	700
2 hours	580	720

Yield of rosin oil ..... 87 per cent by weight.

RUN 3.—Slow Vacuum Distillation.

Time	Vacuum cm. of hg.	Temperature of Overhead, ° F.	Temperature of Liquid Rosin, ° F.
Start	6.0	180	320
1 hour	6.0	475	580
2 hours	6.0	532	598
3 hours	7.0	360	644
4 hours	5.0	120	585
5 hours	7.0	325	658

Yields

	Per Cent by Weight
Distillate	67.7
Residue	13.5
Loss	28.8

RUN 4.—Vacuum Distillation (In Two Cuts).

Cut C ..... Start to 600° F. (Still temperature)
Cut D ..... 600° to 640° F.

RUN 5.—Vacuum Distillation (Slow, 5 hours).

Temperature ..... 545° to 740° F.
Vacuum ..... 9 to 7 cm. of mercury

Yields

	Per Cent by Weight
Distillate	75.33
Loss	20.33
Residue	4.33

Preparation of the Sett

A batch of standard "light sett" was made on the following formula and used in preparing experimental samples:

Fifty Pound Batch Light Sett.

Water	6 pounds 4 oz.
Kidney rosin oil	10 oz.
Asphalt base pale oil, 180 vis./100° F.	32 pounds 10 oz.
Pale oil	7 pounds
Powdered hydrated lime	3 pounds 14¾ oz.

The seven pounds of oil was warmed to 120° to 130° F. with the Kidney rosin oil. The water was then mixed with the lime separately and added to the rosin oil mixture and stirred at about 120° F. until considerable thickening was noted. The remainder of the oil was then run in. This light sett or lime part as it is sometimes called is stirred with another mixture of rosin oil and mineral oil known as the prepared oil, in the manufacture of sett greases.

The procedure followed in making laboratory batches of sett grease was to mix the prepared oil at 100° F. and pour into it the sett; stirring at a uniform rate (about 60 R.P.M.) with an eighteen-inch spoon for one and a half minutes. The grease was then poured from the mixer into one-pound cans and a stop watch started at the same time. Every fifteen seconds the can was tilted slightly until the grease had taken a set and would not flow. This is known as the time of setting, or time of sett.

Consistencies of the grease were determined at 77° F. by the A.S.T.M. method at 77° F., and were made twenty minutes after mixing and after standing 24 hours, 3 days, and 7 days.

Results of Tests

Three parts by volume of the mineral oil was mixed with one volume of each of the rosin oils prepared above to make a series of prepared oils. Then two parts by volume of each of the prepared oils was mixed with one part of the light sett according to the standard procedure given. Table 1 gives the results of the experimental batches.

TABLE 1.—Grease-Setting Properties of Laboratory Prepared Rosin Oils.

Rosin Oil	Vis. Saybolt at 210° F.	Per Cent Acids as Oleic	Time of Sett Min.	A.S.T.M. Penetration After 20 Min.	1 Day	3 Days
Purchased K Rosin oil	265	41.35	5	268	134	131
R–1	734	63.28	2½	160	65	57
R–2	60	20.18	Did not sett			
R–3	261	52.65	2½	161	120	80
R–4–C	131	46.63	3	202	98	92
R–4–D	365	56.80	3	178	70	55
R–5	197	51.25	3	137	77	62
R–5–10	197	51.25	3	205	128	130

The prepared oil for grease R-5-10 was made with one part of R-5 rosin oil and four parts of mineral oil instead of three parts showing that by using a rosin oil properly manufactured the rosin oil in the grease may be reduced by 20 per cent.

An oil such as R-5 is also satisfactory from a time-of-setting standpoint, it being desirable that the grease sett quickly in order that the cans of grease may be packed at once in cases without splashing the grease on the covers.

An experimental batch of grease was made using the untreated grade F rosin. It was found impossible to use this material in place of rosin oil as it had no setting properties, and what soap was formed separated from the oil.

From the results of this work it appears that the nature of the abietic acid has been altered by distillation, the most satisfactory oils being prepared by vacuum distillation.

CHEMISTRY OF ABIETIC ACID

The structure and molecular weight of abietic acid occurring in rosin has been the subject of many elaborate investigations. Ruzicka and Meyer [1a] give abietic acid as found in American rosin the formula of $C_{20}H_{30}O_2$. Some insight into the structure has been gained by its dehydration to retene $C_{18}H_{18}$, by Vesterberg [1b] and the identification of the latter as 1-methyl-7-isopropylphenanthrene by the work of Bamberger and Hooker,[1c] and Bucher.[1d] There remains, however, to be fixed the position of the double bonds and of the methyl and carboxyl groups which may be eliminated by dehydrogenation.

In the light of present knowledge abietic acid is a decahydroretenecarboxylic acid and belongs to the diterpene series. In the extraction of the abietic acid from rosin different methods have yielded the product with widely different melting points.

The International Critical Tables give the formula for Abietic Acid as $C_{19}H_{28}O_2$ with a molecular weight of 288.22 and melting point of 161° C. Spaght, Parks and Barton [1e] have reported that commercial rosin and abietic acid have temperature density susceptibilities closely in accord with the following linear equations:

FF Rosin	$d = 1.094 - 0.0006\,t$
I Rosin	$d = 1.075 - 0.0006\,t$
Abietic acid	$d = 1.077 - 0.0006\,t$

in which

t is the temperature of the rosin in ° C.
d is density, grams/cm^3.

[1a] Ruzicka, L., and Meyer, J., *Helvetica Chim. Octa.*, **5**, 315-44 (1922); *C. A.*, **15**, 3476.
[1b] Vesterberg, *Ber.*, **36**, 4200 (1903).
[1c] Bamberger and Hooker, *Ann.* **229**, 102.
[1d] Bucher, J. E., *J. Am. Chem. Soc.*, **32**, 374 (1910).
[1e] "Viscosity Data for Commercial Rosin and Abietic Acid," *An. Ed., Ind. Eng. Chem.*, (2), **7**, 115 (Mar. 15, 1935).

These investigators have reported viscosity data by the falling ball method as follows:

Temperature, ° C.	Viscosity, $\log_{10} V$		
	FF Rosin	I Rosin	Abietic Acid
30	10.73	9.73	10.35
40	9.00	8.11	8.71
50	7.38	6.60	7.07
100	1.93	1.54	1.78
130	0.52	0.27	0.31

V = Viscosity in poises

A typical analysis of "I" Wood Rosin is as follows:

Melting point	175.50° F.
Acid number	163.40
Saponification number	171.10
Unsaponifiable matter	7.30 per cent
Petroleum ether insoluble	0.15 per cent
Gasoline insoluble	0.06 per cent
Specific rotation	4.30°
Color	80 Amber 1.00 Red (Lovibond)

Very little information has been found in the literature as to the action of alkalies and lime in particular on abietic acid. Archbutt and Deeley [1f] state that crude rosin oil invariably contains a considerable percentage of rosin acids (undecomposed colophony) which have distilled over with the hydrocarbons, and it is the combination of these acids with the lime, forming a soap when the rosin oil and lime are stirred together, which is the real cause of the formation of rosin grease. Refined rosin oil which has been freed of rosin acids is incapable of forming grease; on the other hand, the larger the percentage of rosin acids contained in the crude rosin oil, the stiffer the grease which can be formed.

It may be pointed out that if the undecomposed rosin acids are desirable as grease setting material, lump rosin containing large quantities of these acids would be still more satisfactory. Such has not been found true in the laboratory.

A more logical explanation of the grease-setting properties of rosin oil is presented in the patent of the Pensacola Tar and Turpentine Co.[1g] This patent covers the manufacture of a lubricating grease consisting of mineral oil sett by milk of lime or other alkali and the acid body obtained when rosin is distilled under reduced pressure or with superheated steam. The acid is said to be an isomer of abietic acid, and its proportion in the acid body varies from 68 to 93 per cent according to the conditions of the distillation, which may vary from a five inch vacuum and a temperature of 280° to 345° C. to a vacuum of 28 inches and a temperature of 270° to 305° C.

It is very probable that all rosin acids contain a hydrogenized retene nucleus and that a distillation under vacuum or a steam distillation brings about a change in the relative positions of the methyl and isopropyl side groups. That the acids found in lump rosin must be volatilized and con-

[1f] Archbutt, L., and Deeley, R. M., "Lubrication and Lubricants," (1927).
[1g] U. S. Patent 1,109,298 (1914); British Patent 18,235 (1914).

densed to be satisfactory as a grease sett has been shown by the results of experimental batches. Simply heating lump rosin at its boiling point for several hours did not give a product of satisfactory grease-setting properties.

It is obvious that it would be very difficult if not impossible to work out a simple routine method for the determination of the particular grease-setting acids to be used as a purchase specification. The making of a small test batch of grease with the sample of rosin oil in question is probably the best procedure.

In 1867, Pain and Corry [2] proposed distilling rosin oils over Fe_2O_3 in order to improve them with regard to their lubricating and soap making properties.

RELATION BETWEEN YIELD OF GREASE AND EXCESS LIME IN SETT GREASES

The activity of the lime in the sett grease process is appreciably affected by the degree of its dispersion (which is favorably influenced by incorporating rosin soaps with the sett), the viscosity of the oil used in the grease, and the presence of resins, asphaltenes, and saponifiable matter in the oil, as well as the temperature at which the ingredients are combined. The degree of agitation is also important. Keeping as many of these factors constant, as was possible, the following data was obtained, indicating that there is not much point in using more than (about) fourteen times the theoretical quantity required for neutralization of the abietic acid present.

Cone Penetration at 77° F. after Standing 20 Minutes from Time Mixed	Cone Penetration at 77° F. after Standing One Day	Factor N*
500	500	8
475	400	10
440	300	12
425	275	14
414	260	16
410	242	18
408	238	20
406	237	30

$$* N = \frac{\text{Weight of CaO used in sett grease}}{\text{Theoretical weight of CaO needed for neutralization of abietic acid in sett grease.}}$$

ANALYSES OF COMMERCIAL ROSIN OILS

The analyses of several rosin oils used for grease manufacture are presented in Table 2.

COLD SETT CURVE GREASES

Curve or track greases are used on railway track curves to reduce the friction of the train and reduce wear on the track. In some curve greases an excess of hydrated lime is employed, which, in the presence of moisture,

[2] British Patent 344 (Feb. 7, 1867).

TABLE 2—Analyses of Rosin Oil

Sample No.	Mg. of KOH Per Gram of Oil	Per Cent Acid as Abietic (Mol. Wt. 346)	Penetration of Standard Sample of Grease	Time to Sett in Minutes	Viscosity at 210 ° F.
1	30.8	18.95	212	3	48
2	38.8	23.85	353	100 plus	60
3	75.5	46.5	120	1½	128
4	76.8	47.2	133	2	96
5	77.9	47.9	119	2½	200
6	78.0	48.0	101	½	79
7	79.3	48.7	109	2½	91
8	82.3	50.6	119	3	265
9	84.0	51.6	127	2¼	265
10	85.0	52.2	115	1½	192
11	86.5	53.1	117	2½	345
12	87.0	53.5	126	6	149
13	86.0	52.9	101	2½	382
14	90.8	55.9	119	3½	343
15	91.0	55.95	94	3	346
16	99.0	60.08	88	3	800
17	99.0	60.08	82	6	1187
Lump rosin, Grade F	165.9	102.0	300 plus	100 plus	....

acts as a bond between the grease and steel track. These greases are applied with a brush or a paddle to the railway track curves. Satisfactory greases for this service are easily applied at a temperature range of zero to 32° F. The usual track oils, and many of the greases, are too stiff at this temperature to be easily applied.

After application of the grease to the curves the passage of the first car over the rails should smooth the grease out along the rail. A grease of buttery texture lends itself to easy application and spreading on the rail curves. In certain cities in the northern part of the United States the tracks are wet during a large part of the year. Satisfactory products used during this season are very adherent to the curves under these conditions. The large quantity of free lime often found in these greases and the adhesive properties of the mineral lubricating stock are responsible for this action. The lime is believed to absorb the moisture on the rail, forming an emulsion which bonds the lubricant to the metal rail.

In some cities dust conditions are particularly bad. Viscous, tarry lubricants collect this dust and become dry and powdery and of poor lubricating quality. Certain curve greases collect in tarry masses between the rails and the guard rail. On warm days, this used grease is often picked up and thrown onto the streets, where, in extreme cases, it is a considerable nuisance.

Satisfactory curve greases should not contain lumps of free lime. The consistency and quality of the grease should not be variable in different shipments. Certain curve greases have been known to separate and precipitates of the lime soaps have been found in the bottoms of the barrels.

Winter Grade Curve Grease

Formula

	Gallons
Prepared oil	3.0
Asphalt base fuel oil, 16° Bé.	3.5
Kidney rosin oil	1.0
Dark sett	5.5

The formula of the finished grease is:

	Per Cent by Weight
Fuel oil	78.17
Kidney rosin oil	7.8
Calcium oxide	5.61
Water	8.42

This grease has an A.S.T.M. penetration of approximately 200; the melting point is 250° F.

Summer Grade Curve Grease

Formula

	Gallons
Prepared oil	3.0
Asphalt base fuel oil, 16° Bé.	3.5
Kidney rosin oil	1.0
Dark sett	5.0

The formula of the finished grease is:

	Per Cent by Weight
Fuel oil	78.13
Kidney rosin oil	8.3
Calcium oxide	5.43
Water	8.14

Hard Curve Grease

Formula

	Gallons
Prepared oil	3.0
Asphalt base fuel oil, 16° Bé.	3.5
Kidney rosin oil	1.0
Dark sett	3.5

The formula of the finished grease is:

	Per Cent by Weight
Fuel oil	77.99
Kidney rosin oil	10.27
Calcium oxide	4.70
Water	7.04

Curve Grease

The analysis of a curve grease which has given satisfactory results is as follows:

Analysis

	Per Cent by Weight
Saponifiable rosin oil	5.25
Water	1.00
Filler (CaO)	15.42
Mineral oil	78.33

Color	Black
Flash	295° F.
Viscosity at 100° F.	215
Cold test	5° F.
Gravity	19.0° Bé.
Melting point	198° F.
Penetration at 77° F.	290

The chief characteristics of this product, which are responsible for its satisfactory service, are:

The water content of this grease is only 1 per cent. Most greases for this service contain from 4 to 10 per cent of water.

A dark colored mineral oil of 215 viscosity at 100° F., containing very little tar, is used.

A large percentage of uncombined lime is present in this product, the value of which has already been discussed.

The soft consistency of this grease and a slightly tacky texture are characteristics which are responsible for the easy application of this product to the rails by means of a paddle.

Formula

	Per Cent by Weight
Kidney rosin oil	8.00
Dark mineral oil	76.00
Powdered hydrated lime	15.00
Water	1.00

The dark mineral oil is a blend of 20 per cent by volume 16 gravity asphalt base fuel oil and 80 per cent of 100 viscosity at 100° F., Western pale oil; the viscosity of the mixture is 269 at 100° F.

A mixture of 80 parts of kidney rosin oil and 233.7 parts of the mineral oil is made in a steam jacketed kettle, and the materials heated to a temperature of 100° to 125° F., depending upon the setting properties of the rosin oil. If the setting properties of the rosin oil are relatively poor, a higher temperature is employed.

A "sett" is made by mixing 10 parts of water and 150 parts of powdered hydrated lime, and the mixture allowed to stand over night. This material is then made into a paste with 526.3 parts of the dark mineral oil, in another regular grease mixer.

Two parts by volume of the "sett" are mixed with one part of the rosin oil mixture and stirred for one minute. This mixing process may be carried out by hand in barrels or smaller containers, but is most satisfactorily accomplished by means of an automatic "sett" grease machine.

After mixing, the grease is allowed to stand in the containers for from five to twenty minutes, when it should be of such a consistency that it will not be splashed on the lids of the containers.

Track Grease (Summer)

A satisfactory grease for summer use may be made on the following formula by a process similar to that given in No. 4.

Formula	Per Cent by Weight
Fuel oil, 16 gravity	85.00
Kidney rosin oil	13.00
Lump lime	1.20
Water	.80

Tram Curve Grease

FORMULA:

A curve grease which will have an A.S.T.M. Unworked Penetration of 160 to 250 at 77° F. may be manufactured on the following formula:

Constituents	Gallons	Lbs. Net	Per Cent by Weight
Rosin oil	1	8	.04
Water	50	416	1.90
Lime		500	2.28
Light vac. dist.	550	4,317	19.68
Rosin oil	400	3,320	15.14
Fuel oil	1700	13,372	60.96
		21,933	100.00

Curve Greases are also termed tram greases and may be referred to as "floating grease," or "corfe grease."

COLD SETT AXLE GREASES

Greases satisfactory for the lubrication of wagon axles have a consistency similar to that of Cup Grease No. 4. These greases are often mixed with powdered mica or graphite, and inasmuch as they are used for the lubrication of journals operating at slow speeds and low bearing pressures, often in poor mechanical condition, such additions are warranted. They may also be used for the lubrication of diamond drill rods and coarse gears as found in sawmills. Such greases give satisfactory lubrication on bearings coming in contact with water, such as those found on dredges.

Cold Sett Axle Grease

Formula	Pounds	Per Cent by Weight
Red oil, 450 vis. at 100° F.	2545	23.11
E. pale paraffine oil, 100 vis.	5089	46.23
Kidney rosin oil	1541	14.00
Bloom rosin oil	960	8.72
Powdered mica	180	1.64
Water	469	4.26
Powdered lime	225	2.04

PREPARATION OF OIL:

A prepared oil is made by mixing together the following materials:

Red oil, 180 vis. at 100° F.	600 gallons
Kidney rosin oil	180 gallons
Bloom rosin oil	120 gallons
Powdered mica	180 pounds

The 180 viscosity oil is made by mixing the 450 viscosity oil and the pale oil together in the proportion of one to two.

PREPARATION OF SETT:

The sett is made on the following formula:

Water	75 gallons
Kidney rosin oil	7½ gallons
180 Viscosity oil	510 gallons
Powdered hydrated lime	300 pounds

PROCEDURE:

The lime is first charged in a grease mixer and the mineral oil, in which has been dissolved the rosin oil, is run in slowly while mixing. The water is then added.

Two parts by volume of the prepared oil are mixed with one part of the sett, either by hand or by means of the automatic sett grease machine.

This grease has a penetration of about 170 A.S.T.M. and a melting point of 250° F.

MICA

Mica is the name applied to a group of minerals characterized by the facility with which they split into thin lamina which are flexible and more or less elastic. The micas are complex silicates containing aluminum and potassium generally associated with magnesium but rarely with calcium. Water is always present and often fluorine is found to be present. Mica is prepared for the market by splitting the blocks of rough mica into plates which are cut into any desired pattern by means of shears. The refuse mica when finely ground constitutes the material used for lubricating purposes. The small particles of mica still retain their thin laminated structure.

Mica has long been used in this country as an ingredient of axle greases. In 1895, Lovett [3] obtained a patent relating to the manufacture of a grease in which suitable proportions of mica, and dry slaked lime, were mixed with mineral oil and heated to temperatures above 100° F., when a suitable amount of rosin oil was added and the mass allowed to cool.

Dark Axle Grease

Axle grease may also be made on the following formula:

	Pounds
Hydrated lime	33
American talc	63
Water	65
100 Pale oil	40
Lump rosin, grade F	10
100 Pale oil	19
Heavy Ohio black oil	150
Kidney rosin oil	24

[3] British Patent 19,890 (Oct. 22, 1895).

Rosin-Rosin Oil Axle Greases

Kauffman [4] has reported a series of axle grease formulae in which a mixture of lump rosin and rosin oil is combined with lime in the formation of sett greases.

SETT FORMULAE:

	A	B	C	D	E	F
Hydrated lime, lbs.	300	450	300	450	420	154
Rosin oil, gals.	2.5	3	2.5	3	2	2
100 S.U.V./100° F., pale neutral, gals.	75	80	65	70	...	...
Fuel oil, gals.	...	...	...	...	70	65
Water, to make a total volume of 158 gals. approx.	50	30	60	40	44	58
Approx. weight of sett, lbs./gal.	8.5	9.0	8.5	8.7	8.7	8.6

Kauffman gives directions for slaking quick lime for use in these greases but there is no good reason why a good grade of commercial hydrate may not be used. To prepare the sett the hydrate may be mixed with the water, and 2 to 3 gallons of rosin oil dissolved in the mineral oil is stirred in. The sett should have the following approximate analysis:

Calcium hydroxide	10.5 per cent
Water	8.5 per cent
Calcium abietate	1.5 per cent
Mineral oil and unsaponifiable	79.5 per cent
	100.0 per cent

PREPARED OIL FORMULAE:

Prepared oils are made by simply mixing together the following materials in a suitable agitator:

Formula:	A	B	C	D
100 S.U.V./100° F., pale neutral, gals.	2,172	....	....	2,526
Summer black oil, gals.	....	2,540	2,560	....
Kidney rosin oil, gals.	528	460	440	440
Lump rosin mixture, gals.	160	160	160	194
250 S.U.V./100° F., red oil, gals.	300	....	....	....

The use of lump rosin greatly cheapens the grease without appreciably affecting its quality. The formula for this lump rosin mixture as used in the above table is:

	Gallons	Pounds	Per Cent by Weight
Common lump rosin	185	1,720	41.77
Kidney rosin oil	90	747	18.14
100 S.U.V./100° F., pale neutral	225	1,651	40.09

In a steam jacketed mixer the rosin oil and Neutral are measured and heated to 275° F. The lump rosin is then added and the kettle contents heated to 300° F. to effect solution.

[4] *Petroleum Engr.*, 72-78 (February, 1931).

To prepare the axle greases various proportions of the setts and prepared oils are run through the special axle grease mixing machine, or mixed by hand in barrels. The ratios used may be as follows:

Sett (Volumes)	Prepared Oil (Volumes)
1	2
2	3
2	1
1	1

Medium Black Axle Grease

A black axle grease having an Unworked A.S.T.M. Penetration of 100 to 200 at 77° F., and which has also been used with success as a street car motor gear grease, may be satisfactorily manufactured on the following formula:

FORMULA:

Sett or Lime Part; 1 Volume.

	Per Cent by Weight
Rosin oil	.05
Water	2.80
Hydrated lime	3.36
Light vacuum distillate	29.05

Prepared Oil or Rosin Oil Part; 2 Volumes.

	Per Cent by Weight
Rosin oil	22.36
Fuel oil (black oil)	42.38

LIME SETT:

The sett is made on the following formula:

Powdered hydrated lime	500 pounds
Light vacuum distillate	550 gallons
Water	50 gallons
Rosin oil	1 gallon

DARK PREPARED OIL:

A prepared oil is made by mixing together:

Rosin oil	200 gallons
Fuel oil	400 gallons

PROCEDURE:

Sett. From 75 to 100 gallons of the Light Vacuum Distillate is run into the mixer, which is followed by the one gallon of rosin oil. The hydrated lime is then added and the mixture stirred, until a smooth paste is formed, when the balance of the oil is mixed in. The 50 gallons of water is then measured in, and the mixture stirred for some time, which completes the preparation of the Lime Sett.

Dark Prepared Oil. The rosin oil and fuel oil are measured into the kettle and agitated together for 15 minutes without the application of heat.

Mixing. From the kettles the Lime Sett and Dark Prepared Oil are run through pipes to the two proportioning gear pumps attached to the automatic axle grease mixing and filling machine on the filling floor. The proportioning device is set so that two parts by volume of the Dark Prepared Oil and one of the Sett are brought into contact with each other in the mixing chamber.

From the mixer the grease may be directed into cans and filled into larger containers, where the grease sets within a short time to a consistent product.

H.O. Dark Axle Greases

An Atlantic Coast grease plant made axle grease in accordance with the following formula, using 25 parts of powdered lime per hundred parts by weight of rosin oil:

Formula

	Per Cent by Weight
Summer black oil	80
Kidney rosin oil	16
Powdered hydrated lime	4

A.O. Dark Axle Grease

This product made on the Pacific Coast had the following constituents:

Formula

	Per Cent by Weight
Medium naphthenic vacuum distillate	65.59
Water	5.23
Rosin oil	18.76
Powdered hydrated lime	10.42

COLD SETT SKID GREASES

Dark Skid Grease

Formula

	Per Cent by Weight
Asphalt base fuel oil	77.80
Kidney rosin oil	13.43
Calcium oxide	3.51
Water	5.26

PROCEDURE:

A prepared oil is first made by mixing together 3.5 gallons of 16° Bé. asphalt base fuel oil and one gallon of kidney rosin oil. The sett is a mixture of the hydrated lime, the balance of the fuel oil and the water. Three parts by volume of the prepared oil are mixed with two parts of the sett by the same process as in making axle grease.

CHARACTERISTICS OF PRODUCT:

The A.S.T.M. penetration of the dark skid grease is 184 and the melting point is 235° F.

Light Skid Grease (Medium Grade)

Formula

	Per Cent by Weight
Red oil, 180 vis. at 100° F.	76.97
Kidney rosin oil	13.49
Lump lime (Calcium oxide)	3.09
Water	6.45

PROCEDURE:

A prepared oil is first made by mixing 3 volumes of 180 viscosity asphalt base oil with 1 volume of kidney rosin oil. This is then mixed with an equal volume of sett made by mixing 75 gallons of water, 7.5 gallons kidney rosin oil, 510 gallons 180 viscosity fuel oil and 300 pounds hydrated lime.

Light Skid Grease

Formula

	Per Cent by Weight
180 vis. oil	78.38
Kidney rosin oil	12.07
Calcium oxide	3.10
Water	6.45

Light Skid Grease No. 2

Formula

	Per Cent by Weight
180 vis. oil	75.92
Kidney rosin oil	17.68
Calcium oxide	2.08
Water	4.32

GRAPHITE SETT GREASES

Either flake graphite or fine ground graphite may be added in any desirable amount to the foregoing axle greases.

SETT GREASE PATENT SURVEY

In 1873, Love [5] developed a grease consisting of petroleum, petroleum products, rosin, and lime water, with or without, fats, flour or gums. Williams described the preparation of a boiled axle grease in 1872, using a maximum temperature of 250° F. in his process. His formula was:

	Parts by Weight
Borax	5
Water	15
Tallow	45
Powdered sulfur	5
Lime	5
Graphite	10
White lead	4
Rosin	6
Mineral oil	5

A fire heated kettle was employed, to attain the temperature of 250° F., and stirring was continued in order to assist cooling, after the fire was withdrawn.

Fitzgerald's [6] process, in use in 1874, is quite similar to the modern methods of making sett greases. His formula was:

[5] U. S. Patent 143,362 (Sept. 30, 1873).
[6] U. S. Patent 151,214 (May 26, 1874).

		Gallons
Prepared Oil:		
	25° Bé. Paraffine oil	189
	First run rosin oil	45
Sett:		
	Hydrated lime putty (CaO and water)	18
	Rosin varnish	3

The sett was prepared in the usual form of grease mixer. From five to ten gallons of the prepared oil was then run in and well mixed by means of agitating paddles. This served to emulsify the lime and disperse it in a manner similar to that achieved in modern sett mixtures. The balance of the prepared oil was finally added and the batch mixed to thoroughly incorporate the ingredients and promote the reaction, after which it was drawn at once from the kettle.

An axle grease containing both calcium, potassium and sodium resinates was patented by Pappin.[7] It was composed of rosin, lime, caustic soda, potash, fat, tar, and chalk.

Frazer [8] realized that different fractions collected during the distillation of rosin varied with respect to their grease setting properties. His formula was:

Ammonium chloride	0.5 pound
Water	3 pints

Mix with ½ gallon of No. 1 Rosin Oil, and 2½ gallons of No. 4 Rosin Oil. Then add while stirring:

Hydrated lime powder	10 pounds
No. 1 rosin oil	1 gallon
No. 2 rosin oil	½ "
No. 3 rosin oil	⅔ "

Kirkpatrick [9] claims a lubricating compound of the following formula:

Black oil	6 gallons
Rosin	½ gallon
Lime water	5 gallons
Melted tallow	1 gallon
Talc	6 pounds
Spanish whiting	6 "
Spirits of niter	1 quart

Pettet [10] has assigned to the Pacific Lubricating Co. a gear or wheel lubricant of the following formula:

	Per Cent by Weight
Crude petroleum	40
Water	40
Unslaked lime (which may contain 2 per cent of sodium carbonate)	10
Rosin dissolved in petroleum distillate	10

[7] U. S. Patent 188,479 (Jan. 27, 1877).
[8] U. S. Patent 219,455 (Sept. 9, 1879).
[9] U. S. Patent 652,393 (Mar. 28, 1899).
[10] U. S. Patent 1,081,059.

Pettet [11] has also patented a lubricant consisting of:

	Parts by Weight
Liquid petroleum	42
Paraffine wax	24
Graphite	$2\frac{3}{4}$
Lime water	41
Rosin	14

The Ridsdale [12] patents cover fuels and lubricants made in accordance with the following formula:

	Parts by Weight
Paraffine oil	65–70
Lime	20–25
Rosin	8–9

A mixed calcium-sodium grease was also developed by Ridsdale:[13]

	Parts by Weight
Petroleum	108
Rosin	15
Sodium hydroxide	3
Lime	41
Brown wool grease	10

[11] U. S. Patent 1,143,724 (June 22, 1915).
[12] British Patent 3,782 (1891).
[13] British Patent 14,383 (Aug. 9, 1892).

Chapter VII

Sodium Base Greases

The manufacturing equipment and processes for making sodium soap greases are in general, quite similar to those in use for the manufacture of calcium base greases. The majority of sodium soap greases have a definite sponge or fiber like texture which distinguish them from calcium cup greases. The cohesive properties of the sodium greases are greater than for corresponding grades of cup grease, as are their dropping points. Sodium soap greases may readily be recognized by working a small sample of the grease in the hand, or on a glass plate, with water. The appearance of a definite milky coloration is characteristic of sodium soaps which are disintegrated by the water. The reason sodium greases are not more extensively used is based on the fact that this soap is not resistant to the action of water. If it were not for this inability of most sodium greases to resist the action of water, these higher melting point greases of greater mechanical stability, would probably be used in place of much calcium and aluminum grease now sold. This suggests two research problems:

The development of waterproofing substances which may be added to sodium soap greases without decreasing their dropping points, or stability, and preferably would give them a smooth texture.

The development of new synthetic soap bases (hydrocarbon polymers, new waterproof, high melting point soaps, etc.) to replace calcium, sodium and aluminum oleates and stearates which have been in use so long with, in many cases, unsatisfactory results.

Thompson and McGivern [1] have proposed the improvement of sodium soap greases with respect to their water-proof properties by incorporating certain organic substances. They first prepare a soap by saponifying olein, stearin, rosin, rosin oil, fish or vegetable oils, or fatty acids, with sodium or potassium hydroxides, or triethanolamine. The soap may be formed by mixing together the fat, mineral oil, and alkali, and treating the mixture in a colloid mill. Alternatively, the soap may be mixed with mineral oil and heated to effect solution. Finally, the water-proof properties of the grease are claimed to be improved by the addition of such substances as glue, varnish, linseed oil, casein, gums, shellac, rubber, silicates, or mixtures of these substances.

EARLY SODIUM BASE GREASE PATENTS

It is evident from the following survey of soda soap grease patents that the sodium salts of the fatty acids have for more than eighty years been employed as one of the most important means of increasing the consistency

[1] British Patent 338,176 (July 9, 1929).

of oils in order to produce lubricating greases. In 1855, Pallier [2] produced lubricating greases from oils and fats saponified with alkali by heating and boiling and then permitting the mixture to cool to 180° F. At this temperature flour, starch, and water was added to obtain the desired consistency. To produce soft lubricants saponification was only partly completed. A lubricating grease for carriage axles and heavy shafting was made by de la Rue [3] consisting of Burma petroleum refined by treatment with nitric and hydrochloric acids, followed by neutralization with NaOH, mixed with tallow, palm oil, coco-nut oil, and sodium and potassium soaps. These materials were melted together and agitated until nearly cold. Barry [4] made a black tarry soda base grease by mixing asphaltic residues with ten to fifteen per cent of strong caustic soda solution and mixing for three hours at 30° C. Hill [5] proposed a lubricant prepared by dissolving common yellow soap, tallow, candle wax, palm oil, pulverized steatite, soda or borax, and sometimes lard, in a jelly prepared from water and "Caragheen" moss.

Moreau and Ragon [6] pointed out that the difficulty of solidifying mineral oils could be overcome by saponification in the presence of fatty materials. For this purpose, they suggested one hundred parts each of white tallow, fish oil or animal fat, and two hundred parts of caustic soda should be saponified by boiling, adding one hundred parts of water as the reaction proceeded. To this soap two hundred parts of heavy mineral oil was gradually added and a fiber grease was produced by boiling and stirring together these materials. Caunter [7] produced lubricants from tar, crude mineral oil, or the residue left from the distillation of peat, by saponifying these materials with caustic soda. The product thus obtained was then mixed with vegetable or animal oils (palm oil, tallow, or rosin oil) and the residue left from the distillation of hydrocarbon oils. Petroleum or mineral oil was mixed with soaps and grease, recovered from soap-suds or greasy water obtained from scouring operations, to make a lubricant according to an early patent granted to Teall, Lepaige, and Simpson.[8] Lassiel [9] proposed a grease consisting of soap, soda ash, Irish moss, and water.

In 1865, an important patent was granted to Brooman [10] covering the pressure saponification of fats with caustic soda. His autoclave consisted of a horizontal cylinder heated by an external fire and was provided with safety valves. The process was continuous, the fat and lye being pumped into one end of the autoclave and the soap leaving through a valve at the other end.

Ward [11] boiled peat with sodium or potassium hydroxide to obtain a

[2] British Patent 1,372 (June, 1855).
[3] British Patent 2,002 (Sept. 4, 1855).
[4] British Patent 2,107 (Sept. 18, 1855).
[5] British Patent 109 (Jan. 14, 1862).
[6] British Patent 2,233 (Aug. 9, 1862).
[7] British Patent 3,290 (Dec. 29, 1863).
[8] British Patent 966 (Apr. 5, 1865).
[9] U. S. Patent 50,015 (1865).
[10] British Patent 1,540 (June 5, 1865).
[11] British Patent 484 (Feb. 15, 1866).

lubricant. A fluid, soda soap base liquid grease was developed by Bradford [12] for the lubrication of sewing machinery. One-half pound of olive oil Castile soap was dissolved in one gallon of water, and when cold was thoroughly mixed with one gallon of pure sperm oil. After settling, water was drawn off and discarded. Betts' [13] grease was made by saponifying fat with caustic soda solution and then combining the cold soap with mineral oil. Weston [14] found that a solution of Castile soap in water was a suitable lubricant for photographic films. Bernhard [15] made a lubricant from the following materials:

	Parts by Weight
Sodium soap	100
Gall	100
Starch	100

Black viscous greases were prepared by Crane [16] from petroleum residue which had been treated with strong sulfuric acid and then combined with caustic soda.

Irvine's [17] soda base greases were quite similar to modern dehydrated greases. His process was to dissolve, by the action of heat, alkali soaps in mineral oil, the soaps being the alkali salts of fatty acids and substantially free from water when added to the mineral oil. In one example Irvine dissolved about 15 per cent of potassium soap in mineral oil by heating to 400° F. Semi-fluid greases were made by dissolving from 0.5 to 2.5 per cent by weight of alkali soap in mineral oil.

Groth [18] proposed a solution of soap in water for lubricating glass parts.

Dickson and Mills [19] obtained a patent for lubricating grease consisting of lard oil, or tallow liquified by heat if necessary, and mixed with caustic soda solution. It was essentially a solution of soda soap and water. An aqueous lubricant was prepared in 1882 by Scharr [20] consisting of soda soap dissolved in water, to which was added sodium carbonate, borax, isinglass, salicylic acid and mineral oil. In the preparation of lubricants Henninger [21] proposed combining animal or vegetable fats with ammonium and sodium carbonates, together with mineral oil. In 1906, Samuel [22] developed a continuous method for manufacturing soaps in which the heated fatty material was injected into a perforated chamber filled with hot alkali solution. For grease making Samuel used this process for the preparation of olive oil, or whale oil soda soap to be mixed with petroleum oil. In 1906, Samuel [23] assigned to the Karsam Soap Co. the patent covering his

[12] U. S. Patent 150,128 (Apr. 28, 1874).
[13] U. S. Patent 161,468 (Feb. 18, 1875).
[14] U. S. Patent 164,625 (1875).
[15] U. S. Patent 185,886 (Dec. 16, 1876).
[16] British Patent 146 (Jan. 13, 1879).
[17] U. S. Patent 242,828 (July 29, 1880).
[18] British Patent 115 (1881).
[19] British Patent 1,259 (1881).
[20] U. S. Patent 268,547 (Dec. 5, 1882).
[21] U. S. Patent 288,054 (Nov. 6, 1883).
[22] British Patent 7,380 (Mar. 27, 1906).
[23] British Patent 24,822 (Nov. 5, 1906).

mixing apparatus as used in the preparation of lubricating greases. Mineral oils were incorporated with soaps by injecting them in a finely divided condition into a soap containing mixture at temperatures below the boiling point of water. He also suggested a process in which the temperatures were sufficiently high to volatilize the mineral oils used. In an example, 2 parts of tallow were saponified with caustic soda and 5 parts of lubricating oil were injected into the hot soap.

Bayliss and Brownsdon [24] prepared lubricants by mixing together petrolatum, caustic soda, and sodium amalgam. Gerling [25] proposed a lubricant consisting of 80 per cent sulfite cellulose solution and 20 per cent of sodium hydroxide solution. Rurata [26] developed the following formula for a lubricating grease:

	Parts by Weight
Mineral oil	50
Water	150
Soya bean albumen	40
Phenol preservative	2
Sodium carbonate	4

Recent Sodium Soap Grease Patents

James [27] has suggested the preparation of the sodium salts of mineral oil oxygenated organic acids as suitable bases for lubricating greases. Brunstrum [28] has developed a lubricant consisting of the sodium salt of alginic acid incorporated with a polyhydric alcohol. The investigations of Burwell [29] resulted in the development of saturated aliphatic acids derived from petroleum, which, when neutralized with caustic soda, were considered to be suitable as bases for greases.

FIBER STRUCTURE

The fiber structure of sodium base greases has been discussed at length in Chapter I, and the microscopic methods proposed by Farrington for measuring fiber length were reported. The principal theories and fundamentals pertaining to sodium soap production were presented in Chapter III; this chapter will, therefore, be confined to a presentation sodium soap grease formulae and a discussion of the physical, chemical and service characteristics of the sodium base greases.

COEFFICIENT OF FRICTION

Rhodes and Allen [30] made experiments with sodium base greases containing Pennsylvania lubricating oil of 445 S.U.V./100° F. and first found what many practical grease makers had learned by experience; that the

[24] British Patent 17,910 (Aug. 7, 1907).
[25] British Patent 114,312 (Dec. 13, 1915).
[26] U. S. Patent 1,466,022 (1923).
[27] U. S. Patent 1,700,056 (Jan. 22, 1929).
[28] U. S. Patent 1,970,902 (Aug. 21, 1934).
[29] U. S. Patent 2,012,252 (1935).
[30] *Ind. Eng. Chem.*, **25**, 1275 (Nov., 1933).

dispersion of sodium oleate, and particularly sodium stearate, in oils of low polarity is a difficult task and requires the presence of some third agent or phase. With naphthenic oils these difficulties are not nearly so apparent, except for very high viscosities. With the glycerol normally present from the saponification of tri-glycerides suitable fiber greases may be made with Pennsylvania oils. These investigators found that when the Pennsylvania oil was saturated with about 0.1 per cent of sodium oleate and the normal amount of glycerin, at 100° C., further increases in the percentage of soap did not decrease the static coefficient of friction as determined with an inclined plain apparatus.

Sodium Base Liquid Greases

Grease No.	1	2	3	4
	Formula			
445 S.U.V./100° F. Pennsylvania oil	100	99.09	99.05	99.90
Percent sodium oleate	0	.01	.05	.10
	Coefficient of Static Friction			
Temperature ° C.				
20	.134	.122	.112	.100
60	.135	.123	.112	.100
80	.142	.127	.118	.108

When attempting to interpret data of this kind in terms of what takes place in a bearing, it should be remembered that there is some evidence that temperatures in the shear plain of the grease are, no doubt, much greater than 100° C. when the bearing surface speeds are high. It is evident, however, that increases in the soap content, up to the point of saturation, cause progressive decreases in the coefficient and permit higher critical temperatures at which the coefficient begins to increase appreciably with temperature.

In explanation we quote from Rhodes and Allen:

"The formation of an absorbed film of the type met with in lubrication may be considered as analogous, in some respects at least, to the crystallization of a solid from a liquid. In both cases, we have to deal with the equilibrium between the irregularly oriented molecules of the liquid phase and the regularly arranged molecules in a solid or adsorbed phase. In both cases, a rise in temperature tends to shift the equilibrium in the direction that will make the liquid phase the more stable one. As long as the interatomic, or intermolecular, forces that hold the component units together in the space lattice are stronger than those that tend to disintegrate the crystal, or the adsorbed layer, the material remains in the solid form; as the temperature is increased, the disruptive forces become stronger and a point is reached at which liquefaction occurs. In case of a true crystal the internal forces that hold together the components units of the space lattice are uniform throughout, and, therefore, the temperature at which the solid phase ceases to be stable is sharply defined and the material shows a definite melting point. With the oriented adsorbed films which are encountered in lubrication, the internal forces are not symmetrical. The molecules in the inner layer next to the metal are firmly bound, but as we pass toward the liquid side the intermolecular forces become weaker and weaker and the orientation in the successive layers become more and more irregular. At no one interface is there an abrupt change from the regular orientation of the film to the disorganized structure of the liquid. Consequently, there is no sharp point at which the film suddenly disappears as the temperature is increased. The film simply becomes thinner and

thinner as the more weakly oriented layers remote from the solid surface are liquefied. The critical temperature at which the adsorbed film begins to show marked reduction in thickness and the coefficient of static friction begins to rise sharply is not, therefore, as well defined as is the melting point of a solid. The exact position of the break in the curve will depend upon several factors,—the nature of the adsorbed material, the firmness with which the material is adsorbed on the solid surface of the bearing, the thickness of the adsorbed film, etc. The introduction of even a very small amount of soap (0.01 per cent) into the oil raises the critical temperature, since the soap film is more firmly adsorbed than is the film from the original oil. With increasing amounts of soap the critical temperature falls again, since the adsorbed films are thicker and the outer layers are less subject to the orienting action of the metal surface and are, therefore, more susceptible to the disintegrating action of the higher temperatures. When only a very small amount of soap is present and, therefore, the film is very thin, even a slight disintegration of the film causes a rapid increase in the coefficient of static friction. Consequently, the break in the curve is a sharp one. With large amounts of soap the films are thicker and the disintegration of the outer layers has relatively less effect, so that the breaks in the curve are more gradual."

When the soap solutions were tested in undried air the coefficients were higher (about .180) and at a test temperature of 80° C. dropped off rapidly, reaching at 100° C. values on the order of 0.050.

Effect of Glycerol on Static Coefficient of Grease

Rhodes and Allen prepared greases containing 5 per cent by weight of sodium oleate, with the normal amount of glycerin present, and with four and five times this quantity; the static coefficients were determined:

Grease No.	1	2	3	4	5	6
Glycerin (× normal)	5	5	1	3	3	2
Tests in wet or dry air	wet	dry	wet	wet	wet	dry
	Coefficient of Friction at ° F.					
20	.160	.120	.128	.095	.010	.060
60	.160	.120	.120	.092	.083	.055
80	.170	.142	.080	.090	.075	.045
100	.190	.178	.060	.005	.040	.005

It is evident that at 100° F. the presence of five times the normal amount of glycerin will greatly increase the friction; but that, with two and three times the theoretical quantity of glycerin, the coefficient will be reduced to appreciably low values.

SODA BASE GEAR GREASES—REQUIREMENTS AND PROPERTIES

It is of interest that in the past the manufacturers of greases have given out very little information to dealers and salesmen about their transmission and differential gear lubricants—particularly information of technical value.

The chief requirements of a satisfactory differential and shift gear transmission lubricant are:

Readiness to flow to parts to be lubricated.
Leakage from differential to axle housing; should be as small as possible.

The resistance to turning of the gears should be small. This will give a low loss of power and, what is more noticeable, will prevent sluggish action in shifting. From the latter viewpoint especially the behavior at low temperatures is of interest.

Stability. The lubricant should not permanently change its consistency, that is, become permanently thin or thick, or separate out soap, if exposed to heat or agitation in storage and in handling. After being put in service, the consistency should not change, or if it does, as with most greases, not through too wide a range.

There is little doubt that almost any grease will be able to give satisfactory lubrication in a differential or transmission provided it is able to reach the parts which have to be lubricated and does not contain objectionable materials such as wood fiber, asbestos, etc. In some practical tests on a commercial transmission lubricant after a twenty-hour run the differential gears and the roller bearings remained in the same practically dry condition in which they were assembled at the beginning of the test, although a normal charge of lubricant was present in the differential housing. This is a serious condition. Excessively heavy lubricants should be avoided except in those cases where the conditions of wear are such that normal lubricants cannot be retained. Tests on a large number of commercial gear lubricants have shown that they behave quite differently under service conditions, some being much more unstable than others. The consistency of a fresh grease should never be taken as a criterion of its service behavior.

The scientific study of the stability of transmission greases is facilitated by means of a pressure viscometer. Samples of greases as shipped and those obtained by stirring the grease by means of an electric motor may be tested satisfactorily for consistency by means of the pressure viscometer. Actual used samples from the rear axle housings of automobiles are also tested with very interesting results.

According to theory flow will take place at a constant rate under any given conditions, if the following relation is fulfilled:

$$\text{Viscosity} = A \ (B - \text{Internal Friction}),$$

A and B being constants, this is the equation of a straight line. As the result of many tests the equation for this line has been found to be:

$$\text{Viscosity} = 3000 \ (1.75 - \text{Internal Friction})$$

It should be remembered that such a relation may be expected to hold only with reservations. Different greases will stir down differently in repeated tests; the samples taken from the rear axles at the end of a test are mixtures of unstirred grease clinging to the walls and portions which have thinned down considerably. Temperatures will vary with service conditions, type of grease, et cetera. In view of these conditions, the correlation between consistency and service behavior is remarkably good and sufficiently accurate for all practical purposes.

The lubrication of a rear axle depends upon the consistency which the grease attains under stirring at the temperature reached in the axle housing under service conditions. The original consistency of the grease as shipped gives at best a poor indication as to how it will behave in service, because different greases thin down differently, and after being thinned down behave differently with changes of temperature.

The behavior of a grease in the gear case may be predicted with reasonable accuracy by stirring a sample artifically for some time, for instance, twenty hours, and then determining its consistency at 125° F. This consistency for Ford "T" cars should be about that given by the equation, that is:

The grease should have after stirring, if its viscosity is low, an internal friction (*see* Chapter V) less than 1.75 pounds per square foot at 125° F. If it is an oil (friction value of zero) a viscosity even as high as 5,000 Saybolt would be permissible for Ford "T" rear axles.

Fraunhofen [31] has obtained data for the compounding of lubricants for modern gears, and made experiments with a testing machine consisting of two pairs of gears and two long shafts. One of the shafts was divided, and by twisting both parts against each other the two pairs were put under an artificial load. The apparatus was driven by an electric motor, and the frictional loss determined by the torque of the motor. The lubricants tested were placed in one of the two boxes which contained a hardened and an unhardened gear. It was found that the load to which some extreme pressure lubricants can be subjected is increased considerably by addition of chemically active substances, such as sulfur, or carbon tetrachloride. The favorable effect of free oleic acid was also confirmed. The addition of sodium soaps was found to greatly reduce the noise and leakage loss, but also the lubricating power was impaired. Calcium, lead and aluminum soaps reduced the noise less than sodium soaps. Rubber latex was found to be satisfactory in reducing noise. Colloidal graphite improved the lubricating efficiency.

Low-Temperature Consistency of Gear Lubricants

Knopf [32] has presented the following most interesting discussion of low temperature consistency tests:

"The pour tests of a motor oil is not a measure of its starting quality in low temperature service, and at best is only roughly indicative of the temperature at which the oil fails to pass through the screen and into the circulating-oil pump. The pour test of a gear lubricant, likewise, is of very little value in determining whether the lubricant will feed satisfactorily to the ring-gear and pinion, or whether channeling will take place at 0° F., for example.

"One gear lubricant with a 15° F. pour test may channel and fail to lubricate properly at 25° F., whereas another with a 25° F. pour may function satisfactorily at 0° F. Some lubricants—for example, those high in petroleum or soap content—when allowed to remain undisturbed for a considerable period of time at low temperatures, will appear solid, but when agitated the consistency is greatly reduced although the temperature is practically unchanged.

"Normally the lubricant in a rear-axle housing fills the tooth spaces and thoroughly coats the teeth submerged in the lubricant bath. Assuming that the temperature of the lubricant has dropped to 0° F. over night, the torque, or drag, on starting, will depend on the nature of the lubricant. Light-consistency lubricants generally offer only a small resistance to the passage of an object through them; others, though seemingly heavy in consistency, still permit relatively easy rotation of ring-gear; other lubricants cause excessively high starting torques.

[31] *Automobiltech Z.*, **37**, 503-7 (1934).

[32] *An. Ed. Ind. Eng. Chem.*, **5**, 128 (March 15, 1934).

"After a few revolutions of the ring-gear, a certain amount of the lubricant, depending on the nature, will have been picked up by the ring-gear and carried to the pinion. Channeling will take place between the ring-gear face and the lubricant, unless the lubricant is mobile enough to flow in and fill the gap. Again, the lubricant might flow in but refuse to adhere to the teeth, in which case the pinion will be lubricated only by the film originally left on the teeth, and for certain lubricants this film is insufficient for proper lubrication.

"The performance of various lubricants can be studied satisfactorily by means of a rear-axle test set, provided with temperature control and a means for loading with normal operating tooth pressures. However, a much more accurate method has been developed, which expresses these various characteristics numerically and enables one to classify gear lubricants accurately.

"The viscidometer was developed primarily to determine the viscidity of plastic or semi-fluid lubricants by measuring the resistance offered by the passage of an object through the body of the lubricant, which is indicative of the drag resistance, as well as the non-channel characteristics of the product when employed as a rear-axle lubricant at low temperatures. It also measures the consistency of plastic lubricants at ordinary temperatures when a large plunger is used.

"An added feature of this test throws further light on the non-channel property of a lubricant and indicates accurately the adhesiveness as related to the pick-up that may be expected on a ring-gear. Both viscosity and viscidity may be defined as the resistance offered by a fluid to the relative motion of its particles. This definition implies chiefly internal friction or cohesiveness. Orifice-type viscometers, like the Saybolt, measure viscosity in terms of both adhesiveness and cohesiveness, the former property being more pronounced the smaller the diameter and the longer the outlet tube of the instrument. The MacMichael, on the other hand, might be said to measure consistency or firmness, which is mostly a function of cohesiveness rather than adhesiveness. A gear lubricant, however, should also possess a certain amount of adhesiveness or the bob will not take hold, resulting in an erroneous reading.

"Viscidity may also be defined as the quality possessed by a heavy stick product, of being viscid and having a ropy glutinous consistency; hence, the name viscidometer given to the instrument. As the plunger used in this test resembles a 60-penny spike, the test is commonly called the spike test."

These tests are fully described in the appendix.

Processes for the Manufacture of Soda Base Gear Greases

Non-Fibrous Soda Base Transmission Grease

EQUIPMENT REQUIRED

The equipment required for the manufacture of this lubricant is the usual type of grease mixer having steam jacket on bottom and sides and should also be equipped with water for rapid cooling of the kettle contents. If the kettles are available only in large sizes, twenty-barrel capacity or larger, a chilling device similar to a wax chiller but being water-jacketed, should be used for finishing the grease.

The kettle should be capable of maintaining a temperature of at least 325° F. It should be equipped with efficient stirring mechanism which can be operated at about 40 r.p.m.

Formula

	Per Cent by Weight
Vegetable castor oil	1.564
Lard oil Extra No. 1	1.044
Lump rosin Grade F	.696
Caustic soda	.696
Asphalt base black oil 70 vis./210° F.	96.000

PROCEDURE

The soap stocks together with the heavy black oil representing 2.4 per cent of the finished grease are melted together at 180° to 200° F. in the open steam-jacketed grease mixer.

The caustic soda as 40° Bé. lye is then run into the kettle, agitation being carried on throughout the entire process unless otherwise stated.

The soap is cooked for one to two hours, till practically all of the water has been evaporated. The final temperature should be 300° to 325° F.

The mineral oil is then run in at a rate of about twenty per cent per hour, the temperature being maintained at about 300° F.

After all of the oil has been mixed in, heating may be continued to drive off water which may have been accidentally present in the oil.

The steam is then turned off and the cooling water turned into the jacket. Cooling should be carried out as rapidly as possible in order to produce a grease of smooth texture.

Cooling should be continued, agitating intermittently to avoid excessive occlusion of air, until the temperature drops to about 90° F. or less, which completes the process.

CHARACTERISTICS OF PRODUCT

The cost of the materials for manufacturing this product will run about one cent per pound. This product, properly manufactured, has all the requirements of a satisfactory gear lubricant such as discussed in the first part of this chapter. It will be noted that the soap stock is a mixture of liquid fats and rosin, and that there is a large excess of caustic soda; this combination is responsible for the smooth texture and stable properties of the resulting grease. The grease has a very glossy texture and is comparable in this respect to those greases in which naphthenic acids are used.

Fire-Cooked Soda Base Transmission or Gear Grease

EQUIPMENT REQUIRED

The equipment used for the manufacture of this type of grease consists of an open twenty-five barrel fire-heated kettle, having a single shell. The source of heat may be either gas, fuel oil or coke in connection with an air blast. Rapid cooling is not essential. Efficient agitation throughout the process will result in products of greater stability.

Formula

	Pounds
Prime tallow	100
Flake caustic soda	15
Water	22
Ohio black oil 100 vis./210° F.	4500

PROCEDURE

The tallow and 10 per cent of the black oil or an equivalent quantity of tailings from a previous batch are melted together in the fire-heated kettle to about 150° F.

The caustic soda and water are then mixed together in a separate tank and run into the melted fat in the kettle, agitation being on during the entire process.

In from one to two hours the temperature is raised to about 400° F., when the black oil is started in. Mixing off is continued until about one-half of the oil is in, and the temperature is about 200° F. Mixing off may be continued in this kettle until a cooled sample indicates that the proper grade of grease has been obtained. If maximum production is required the grease at this stage may be pumped to an unheated open mixer and the balance of the black oil run in.

After stirring for eight to twelve hours the grease may be drawn at 100° to 120° F.

CHARACTERISTICS OF PRODUCT

Mixing in all of the oil at a temperature of 350° to 400° F. will produce a grease more resistant to being broken down by gear action. This product is of medium grade and is suitable for general use as a transmission and differential lubricant.

Steam-Cooked Soda Base Gear Grease

EQUIPMENT REQUIRED

The production of this grease may be carried out in a kettle similar to that used for the manufacture of Soda Base Grease No. 1, or the kettle may be steam-jacketed up the sides for only six or eight inches.

Formula

	Per Cent by Weight
Caustic soda solid	.57
Tallow No. 1	2.86
Stearic acid	.57
Asphalt base lubricating distillate 90 vis./210° F.	96.00

Formula

	Pounds
Tallow, No. 1	525
Stearic acid	105
Caustic soda solution, 30° Bé.	420
Distillate, 90 vis./210° F.	2100–1950

PROCEDURE

Charge in a twenty-barrel mixer 50 to 150 gallons of the distillate or tailings from previous batches.

Charge the tallow and stearic acid in the kettle.

Turn on the agitation and continue agitation till batch is completed, unless otherwise noted.

Turn on the steam in the jacket and heat to 150° to 157° F.

Add the caustic soda solution and stir for ten minutes.

After thickening starts turn on the full head of steam which should be about one hundred pounds per square inch.

When a fibrous soap has formed add 50 gallons of oil.

Permit the batch to stand over night with full steam pressure on but without agitating.

On the following morning start agitation and add 200 gallons of oil, if the batch does not boil up add 200 gallons of oil or enough to bring batch up to 1100 gallons.

Run in the balance of the oil.

Turn off the steam and turn on cooling water in the jacket of the kettle.

A sample of the grease should then be taken and cooled and tested on the consistometer or compared with a standard sample.

Add more oil if required to give the exact standard consistency.

Fill at a temperature of 150° to 160° F.

CHARACTERISTICS OF PRODUCT

This product is of rather heavy consistency and is suitable for use in badly worn gear sets.

Steam-Cooked Soda Base Gear Grease

This product may be made by the same procedure as given for No. 3, a heavy-bodied, durable grease being produced.

Formula

	Per Cent by Weight
Animal fatty acids	6.064
Caustic soda	.850
Texas Bright Stock 604	90.398
Crude tallow, No. 1	2.688

Medium Transmission Lubricant, Light

Practically the same process as for No. 3 may be used for this grease.

Formula

	Per Cent by Weight
Caustic soda, solid	1.02
Water	.50
Western pale oil, 100 vis./100° F.	56.68
Western red oil, 450 vis./100° F.	34.50
Crude tallow, No. 2	7.30

Heavy Transmission Lubricant

Formula

	Per Cent by Weight
Crude tallow, No. 2	10.00
Caustic soda, solid	1.50
Water	.75
Western pale oil, 100 vis./100° F.	54.15
Western red oil, 450 vis./100° F.	33.60

Medium Transmission Lubricant, Dark

Same process as No. 3.

Formula

	Per Cent by Weight
Crude tallow, No. 2	6.52
Caustic soda, solid	1.03
Western black oil, 410 vis./100° F.	60.10
Western steam refined stock, 146 vis./210° F.	32.35

Heavy Transmission Lubricant, Dark

Same process as No. 3.

Formula

	Per Cent by Weight
Crude tallow, No. 2	10.57
Caustic soda, solid	1.67
Western black oil, 410 vis./100° F.	57.04
Western steam refined stock, 146 vis./210° F.	30.72

Medium and Heavy Dark Transmission Lubricants, Steam-Cooked

EQUIPMENT REQUIRED

A thirty-five barrel grease mixer jacketed on the bottom only is employed for the production of the following product which is very similar to Formulas No. 7 and No. 8.

Formula

Crude tallow, No. 2	800 pounds
Caustic soda, 40° Bé.	360 pounds
Asphalt base black oil, 71 vis./210° F. (Heavy Grade)	825 to 1000 gallons
Asphalt base black oil, 71 vis./210° F. (Medium Grade)	1300 to 1500 gallons

PROCEDURE

The black oil should be heated to a temperature of 100° to 120° F. before adding to the mixer. This heating can be carried out most conveniently in a tank placed over the mixers, equipped with steam coils, from which the oil can flow by gravity into the mixer.

Not more than 50 gallons of tailings from the previous batch should be charged to the grease mixer.

Eight hundred pounds of No. 2 tallow is then melted and charged in the kettle followed by 360 pounds of 40° Bé. caustic soda solution.

The paddles are then started and agitation continued unless otherwise stated till the batch is completed.

After twenty minutes the steam is turned on and increased to a full head of 100 pounds per square inch in five minutes.

When the steam is started 100 gallons of the black oil is run into the kettle.

When the batch has attained the proper sponge 50 gallons of the black oil is run in at a rate of approximately ten gallons per minute, and the batch stirred for five minutes.

Fifty gallons of oil is run in and the batch stirred for five minutes.

One hundred gallons of oil is added to the batch and stirring continued for five minutes.

Add a second one hundred gallons in the same manner.

Add a third one hundred gallons in the same manner.

The steam is turned off and the batch is allowed to stand over night without agitation.

The batch is heated and agitated until no further agitation takes place at a temperature of 300° F.

The batch is allowed to stand with steam on for a second night if required.

The batch is stirred for twenty minutes.

A sample is taken and cooled. A test on the consistometer is of value at this point.

The remainder of the oil is run in if required.

Draw the heavy grade at 140° to 170° F.

Draw the medium grade at 120° to 150° F.

Series of Experimental Gear Greases

The series of experimental greases (Table 1, page 372) was made to show the effects of various ratios of caustic soda to fatty ingredients. They were all made by the usual steam kettle method. It was found that a more stable grease was produced and also that a grease of smoother texture was produced when the larger excesses of caustic soda were employed.

The series in Table 1 shows the large number of possibilities of combining only a very few materials. All of the experimental products varied considerably in their properties, although any of them would give satisfactory lubrication. The oil used in these greases was Western black oil of the Saybolt viscosity indicated.

Knopf [33] Gear Grease

One of the most difficult lubrication problems presented in the operation of automobiles is that of transmissions and differential gears. In the

[33] U. S. Patent 1,789,614 (Jan. 20, 1931).

Table 1—Analyses of Experimental Gear Greases

Mineral Oil	Combined fat	Free fat	Total NaOH	Per cent NaOH for Fat	Org. acids in mineral oil	Excess	Viscosity of mineral oil, 210° F.	Soap stock
Per Cent								
93.50	4.49		.61	.606	...	.004	71	No. 2 tallow
97.00	2.49		.495	.336	.111	.048	71	No. 2 tallow
95.00	3.84		1.140	.518	.109	.513	71	No. 2 tallow
95.33	3.86		.810	.520	.110	.180	71	No. 2 tallow
95.16	3.85		.989	.519	.109	.361	71	No. 2 tallow
95.65	3.72		.634	.528	.111	.005	71	Animal fatty acid
96.50	2.99		.600	.424	.111	.035	71	Animal fatty acid
95.18	3.69		1.123	.524	.109	.490	71	Animal fatty acid
96.50	2.67		.822	.490	.111	.221	71	Animal fatty acid
90.26	7.86		1.882	1.060	.091	.731	51	No. 2 tallow
92.50	6.05		1.443	.815	.093	.534	51	No. 2 tallow
80.95	7.84		2.218	1.508	.090	1.070	51	No. 2 tallow
92.51	5.84		1.648	.788	.093	.767	51	No. 2 tallow
86.82	7.57	3.79	1.818	1.020	.088	.710	51	No. 2 tallow
80.55	7.01	10.75	1.685	.945	.081	.659	51	No. 2 tallow

early days of the automobile, it was not expected that it would be operated for twelve months of the year; automotive engineering had not progressed this far, the closed body had not been developed to its present stage and, with but few exceptions, highways were not passable for the automobile but for a limited season. As a consequence, this problem of lubrication was then largely confined to meeting conditions of moderate and hot weather. As mechanical and body designs progressed, making the automobile more and more useful through all the twelve months of the year,

Table 2—Analyses of Commercial Soda Base Gear Greases
Per cent by Weight

	Transmission lubricant	Transmission grease	Transmission grease	Gear compound	Transmission grease
Mineral oil	97.58	95.97	95.95	96.63	95.53
Free fat	.25	.43	.73	1.15	1.25
Combined fat	.93	2.90	2.75	1.83	2.39
Free fatty acids	.24	.28	.18	.13	.49
Combined NaOH	.20	.42	.39	.26	.34
Water	.80	Trace	Trace	Trace	Trace
Melting point, ° F.	...	...	137	90	...

	Transmission grease, medium	Transmission lubricant	Transmission grease	Gear grease
Mineral oil	95.01	89.11	89.08	84.23
Free fat	1.29	.79	4.26	5.28
Combined fat	2.58	8.73	5.61	8.88
Free fatty acids	.76	.13	...	.96
Combined NaOH	.36	1.25	.75	.45
Water	Trace	Trace	.30	.20
Melting point, ° F.	95	...	120	176

a number of different lubricants were employed for the lubrication of transmissions and differentials to use the increasing range of temperatures encountered in automobile operation, but, in the past, the best solution of the problem has been to use one type of lubricant in hot weather and another in cold weather. The disadvantages of two lubricants complicate manufacture and distribution. Knopf has attempted to overcome this difficulty by developing an all-year lubricant herewith described.

For operation throughout the year a gear lubricant must essentially retain its lubricating characteristics at temperatures as high as 110° F., and at temperatures of −10° F., and throughout the intervening range, and must also satisfy several other less fundamental but nevertheless important requirements. For example, conditions in differentials are usually higher than in transmissions, which would simplify the problem with respect to transmissions, except that reasonable ease of shifting transmission gear must also be maintained throughout the range of operating temperatures. Knopf's gear lubricant consists essentially of an oxidized blend, substantially free from water, of light lubricating oil, heavy flux oil and sodium soaps of petroleum acids, the latter particularly in the form of caustic bottoms from the distillation of lubricating oils from Gulf Coast Type Stocks over caustic soda; this blend, in a particularly advantageous form, having a MacMichael viscosity of 55 to 100 or higher, 70 to 85 poises at 100° F., a Knopf viscidity not exceeding 12 ounces at 0° F., and a Knopf adhesiveness not exceeding 75 ounces at 0° F. The MacMichael viscosity and the Knopf viscidity are more important than the Knopf adhesiveness. Knopf's lubricant generally consists of about one-half of light lubricating oil, about one-third of heavy flux oil, and about one-sixth of caustic bottoms; the blend being oxidized by air blowing. It may comprise a blend of 65 to 88 per cent, or 83 to 85 per cent, of a mixture of light lubricating oil and heavy flux oil, in proportions such that the composite has a Saybolt viscosity of 55 to 62/210° F., and 12 to 35 per cent, or 15 to 17 per cent, of caustic bottoms of the type referred to above having a Knopf viscidity of 15 to 35 ounces, or 15 to 25 ounces, at 100° F.; the blend being blown with air at elevated temperature, 250 to 300° F., until the MacMichael viscosity at 100° F., of the resulting composite blend, approximates 55 to 100, or 75 to 85 poises, and the Knopf viscidity 4 to 12 ounces at 0° F.

This lubricant has a viscidity low enough to permit easy shifting of transmission gears, even at temperatures as low as 0° F., or lower. It is sufficiently adhesive to feed properly in gear sets and maintain adequate lubricating films, particularly in differentials, even at high temperatures, up to 120° F. It has a low drag resistance which involves a minimum of power loss and permits easy starting. It does not channel, permitting gears to run dry, even at temperatures as low as 0° F. The grease is non-corrosive and has sufficient body to minimize any leakage from gear housings, even at high temperatures.

For the determination of viscosities at temperatures in the range of 100° F., for this type of lubricant, the standard MacMichael torsion viscosimeter is satisfactory. But, for the determination of viscidity and adhesiveness at low temperatures, the usual types of instruments are not entirely satisfactory. The Knopf Viscidometer and the Knopf Adherometer are sufficiently accurate and have been described in Appendix A.

A standard automobile transmission and differential, with openings cut in the gear housings to permit observations, arranged within a thermostat chamber, and connected with an electric motor and prony brake, to be driven at appropriate speeds and loads, were used in the development of this grease. This equipment allowed direct observation of the action of the lubricants studied over a wide range of temperatures. A stroboscope was also used in connection with this apparatus to permit the study of formation and maintenance of lubricant films in the gear sets by direct observation. Tested in this equipment, the deficiencies of many normal lubricating greases were more apparent than in actual use. Many normal lubricants, which were satisfactory at high temperatures, were found to be deficient at low temperatures, and vice versa; for example, lubricants which maintained satisfactory lubricating films and were of good body at high temperatures became so viscous at low temperatures as to channel or to make the shifting of transmission gears difficult, if not impossible; while those sufficiently fluid at low temperatures failed to provide proper lubrication at high temperatures on account of lack of viscosity and adhesiveness to feed well on the gears.

Tests in this laboratory gear set, checked by tests in actual service, were found by Knopf to lead to the following requirements for satisfactory service over wide temperature differentials:

(a) MacMichael viscosity of 55 to 100 (or better 70 to 85) poises at 100° F.

(b) Knopf viscidity of 4 to 12 ounces (or not exceeding 12 ounces) at 0° F.

(c) Preferably, a Knopf adhesiveness of 20 to 75 ounces (or not exceeding 75 ounces) at 0° F.

PROCEDURE

Light Gulf Coast lubricating oil, having a S.U.V./100° F., of 100 seconds, is thoroughly blended with a heavy Gulf Coast flux oil, having a viscosity of 1000 to 5000 S.U.V./210° F., in proportions to produce a base blend, having 55 to 62 S.U.V./210° F. This base blend is then heated to a temperature at which it is freely fluid (250 to 275° F.), and caustic bottoms from the re-distillation of Gulf Coast lubricating oils over caustic soda are preheated to a similar temperature and then are gradually blended with the base blend. Thorough mixing is essential. Using caustic bottoms having Knopf viscidity of 20 ounces at 100° F., about 20 per cent of caustic bottoms on the base blend is ordinarily required. The composite blend is then heated to about 300° F. and blown with air until the resulting product has a MacMichael viscosity of 85 to 90 poises at 100° F., and a Knopf viscidity of about 10 ounces at

0° F. The temperature during blowing is controlled to a maximum of 300° F., to prevent excessive oxidation. The period of oxidations is usually about 4 to 8 hours. The air applied during this operation should be previously dried by passage through a calcium chloride dryer. If water is present in the base blend prior to admixture of the caustic bottoms, it should be blown with dry air to dehydrate it prior to admixture of the caustic bottoms. The finished product is then run to storage. Cooling should be carried out at a slow rate.

Experiments have shown that similar products from California crudes may be employed to obtain gear greases of similar characteristics.

In 1931, Knopf and White [34] obtained further protection for their system of making gear greases; in this case they specified that their blend of light lubricating oil, heavy flux oil, and sodium soaps of petroleum acids, should have a MacMichael viscosity of 85 to 95 poises at 100° F., and a Knopf viscidity not exceeding the range of 12 to 13 ounces at 0° F.

Maverick [35] has developed an unctuous hydrocarbon condensation product, having a molecular weight greater than 170, and having not less than 13.5 parts of hydrogen per 100 parts of carbon, which is saponified to produce a metallic soap. When added to lubricating oil or greases it substantially depresses the channeling point.

Herman [36] has proposed a sodium soap gear grease, having improved temperature—viscosity susceptibility, which consists of mineral oil of 200 to 350 S.U.V./100° F., containing dispersed partially, or wholly, saponified oxidized rape seed oil. Such a lubricant would also have a greater degree of adhesiveness than greases made with normal stocks.

S.A.E. Fibrous Transmission and Differential Greases

Formula for S.A.E. 110 Gear Grease

Constituents	Gallons	Pounds Net	Per Cent by Weight
Tallow	40	305.08	4.04
45° Bé. caustic soda	9	45.19	.60
600 Calif. red oil	285	2185.00	28.94
Naphthenic bright stock	643	5014.73	66.42
		7550.00	100.00

Formula for S.A.E. 160 Gear Grease

Constituents	Gallons	Pounds Net	Per Cent by Weight
Tallow	50	381.35	4.33
45° Bé. caustic soda	11	55.23	.63
1000 Calif. red oil	65	497.20	5.65
Naphthenic bright stock	146	1135.20	12.90
Mid-Continent S. R. stock	885	6731.02	76.49
		8800.00	100.00

METHOD

The melted tallow is gauged in the fat measuring tank and then run by gravity to the proper mixing kettle below.

[34] U. S. Patent 1,831,614 (Nov. 10, 1931).
[35] U. S. Patent 1,963,239 (June 19, 1934).
[36] U. S. Patent 2,008,419 (July 16, 1935).

The caustic soda solution, made up to just 45° Bé., is measured in 5-gallon containers and charged through the chute to the mixer.

The tallow and caustic soda are stirred together for several minutes in order to form a good emulsion; after which the full steam pressure is turned on in the kettle jacket.

Heating is continued for three to five hours, during which time the 1000-Red Oil may be run in, to soften the soap and permit more rapid evaporation of the water in the soap. When a tough, dry, fibrous soap base has been prepared Naphthenic Bright stock may be mixed in.

Considerable care must be exercised in order to produce a sufficiently dry soap base that is in a satisfactory plastic condition to be readily mixed off with the heavy mineral oil. In some cases, where the steam is turned on at once, or where there may be present a considerable amount of the grease from previous batches containing heavy oil, there is a tendency for the soap to boil up in the kettle and form small hard seeds. These lodge on the sides of the kettle and the upper portions of the agitator paddles. On continued heating the soap base subsides and the soap seeds "fit" together to form a homogeneous soap base. When "seed" formation takes place a considerably longer time is required to bring the soap base to the desired condition for mixing off.

The time required for completing a batch of 110 or 160 is from 8 to 12 hours.

When the grease has been brought to what appears to be the satisfactory grade a sample is submitted to the laboratory for a MacMichael viscosity determination. If not conforming to specifications the grease is made to do so; more oil being added when too low in viscosity. No gear grease is approved for filling shipping containers until it is brought within specified viscosity limits.

After the gear greases have been reduced to the required viscosity in the kettles, a portable pump is then employed to pump the grease from the kettles through screens. The object of screening these greases is to remove any improperly reduced soap or foreign material. Both the suction and discharge of the portable pump are connected, by means of flexible metal hose, to the kettle and the pumping arrangement is so designed that it may be employed to circulate the grease, discharging it back into the top of the mixer, or to force the grease through the cylindrical type screens to the shipping containers. The screen cylinder is scraped free from soap slots and lumps by means of a rotating "ribbon" scraper. The pressure required to force the grease through the screen will vary with the fluidity of the grease, and the amount of solid material within the screen chamber, but will ordinarily remain below 50 pounds per square inch.

The size of the screens employed are:

	Mesh per Inch
S.A.E. 110	60
S.A.E. 160	20
S.A.E. 250	20

From the screens the grease may either be metered into 5-, 10-, and 25-pound cans or may be run directly to 15-, 30-, or 50-gallon steel shipping tanks.

Properties

The 110 grease has a dark green color and is semi-fluid in consistency, but flows readily at normal temperatures. It is semi-fibrous in texture.

The 160 grease is dark olive green in color, is of heavier consistency than No. 110, but has about the same texture and fiber structure.

These gear greases will meet the following specifications:

	S.A.E. 110	S.A.E. 160
MacMichael viscosity (60 R.P.M., No. 26 Wire, 80° F.)	72–87	100–140
Minimum Ubbelohde dropping point	..	104° F.

Mechanical Stability of S.A.E. Gear Greases

Service tests in a Chrysler gear box for 5000 miles, at a speed equivalent to 45 M.P.H. on the road, shows these greases are fairly stable, although they appear to aerate somewhat during the first 1000 miles:

Sample taken after Miles:	MacMichael Viscosity (60 R.P.M. No. 26 Wire, 80° F.) S.A.E. 110	S.A.E. 160
0	87	140
500	105	176
1000	90	188
2000	58	152
3000	56	112
4000	55	104
5000	53	99

Temperature-Viscosity Susceptibility of S.A.E. Gear Greases

Temperature ° F.	MacMichael Viscosity (in Terms of the No. 26 Wire) S.A.E. 110	S.A.E. 160
10	975	....
20	800	1600 plus
30	560	1200
40	380	925
60	145	270
80	80	135
120	35	54
200	5	7

Channel Tests, S.A.E. Gear Greases

According to S.A.E. requirements greases 110 and 160 must not channel in service at temperatures above 10° F. and 35° F. respectively. As with dropping points, such greases do not have definite channeling temperatures, there being a gradual transition from a plastic non-channeling grease to a hard product which is cut by the gears. A study of the following data for

spike tests, made over a wide temperature range, indicates the difficulty in arriving at precise channel points:

Temperature of Grease ° F.	Spike Penetration $\frac{mm}{10}$ (5 sec., 5 oz. wt.) 110	160
40	390	480
35	280	320
30	195	170
20	125	79
10	76	44
0	41	18
−10	14	3

Analysis of Two Transmission and Differential Greases

These analyses gave the following results:

Physical Tests:	110	160
Color and appearance	black and fibrous	
Dropping point ° C.	89	145
MacMichael visc. at 80° F. with No. 24 wire	67	...
MacMichael visc. at 80° F. with No. 26 wire	165	...
Worked penetration A.S.T.M. at 77° F.	...	322
Composition, per cent by weight:		
Filler (asphaltic material)	1.0	0.7
Soap (as sodium oleate)	5.7	7.5
Free fat	1.1	0.4
Water	0.2	0.2
Free acid (as oleic)	0.6	0.4
Petroleum oil	94.6	92.0
Gain in analysis	3.2	1.2
Total	100.0	100.0
Extracted Petroleum Oil:		
Color	Black	Black
Gravity, ° A.P.I.	15.1	16.4
Flash, Cleveland, open cup, ° F.	370	360
Fire, Cleveland, open cup, ° F.	405	400
Viscosity, Sayb. Univ. at 210° F.	118	86
Ash, per cent by weight:	0.524	1.005
Ash Analysis, per cent by weight:		
Iron and aluminum oxides (Fe_2O_3)	10.5	21.8
Sodium sulfate (as Na_2SO_4)	52.3	12.0
Sodium carbonate (as Na_2CO_3)	38.0	60.8
Gain in analysis	0.8	...
Unaccounted for	...	5.4
Total	100.0	100.0
Fatty Acids from Soap:		
Acid value, mg. KOH/gm.	177	187
Iodine value (Hanus)	40	47
Dropping point ° C.	56	...

ANALYSES OF THREE ATLANTIC COAST AUTO GEAR GREASES

These analyses gave the following results:

MacMichael Visc. at 70° F.	Lub. No. 110	Lub. No. 160	Lub. No. 250
No. 26 Wire	15	70	380 (worked penn. at 77° F.)
20 R.P.M.			
60 R.P.M.	28	156	....
No 22 Wire			
20 R.P.M.	....	11	45
60 R.P.M.	....	25	25
Water, per cent by weight	0.1	0.1	0.1
Free acid, per cent as oleic	0.06	....	....
Free Alkalinity, per cent as NaOH	....	0.10	0.08
S.U.V. of extracted oil at 100° F.	1040	3200	4214
S.U.V. of extracted oil at 200° F.	91.0	146	158
Ash, per cent by weight	0.28	0.92	1.92
Ash Analysis, per cent by weight			
Sodium (as Na_2SO_4)	67.7	45.6	26.7
Sodium (as Na_2CO_3)	2.8	47.1	70.3
Calcium (as $CaSO_4$)	10.1	1.6	1.1
Magnesium (as $MgSO_4$)	3.0	Trace	None
Iron and Aluminum (as R_2O_3)	1.5	0.8	0.8
Acid insoluble	1.2	0.6	0.2
Undetermined	13.7	4.3	0.9
Total	100.0	100.0	100.0

English Gear Shield Grease

Formula	Per Cent by Weight
100/100° F. Mexican spindle oil	37.0
100 Penetration asphalt	37.0
Cottonseed oil stearin	20.0
Water	3.4
Sodium hydroxide	2.6

This grease is a black, hard material which melts at about 210° F.

FIBER OR SPONGE GREASES

The term fiber grease or sponge grease is usually applied to soda base greases. The theory explaining the fibrous texture of these greases has been presented. From a general consideration the sponge greases have relatively high melting points and are often used where bearings run at elevated temperatures due to heat radiated or conducted to the bearing from external sources. They do not separate readily from the oil but give very stable and probably very thick, tenacious, lubricating films. It is obvious that these products can not be used where they may come in contact with any quantity of water as they are water-soluble and would be readily washed away. These greases are almost invariably boiled or at least semi-boiled products.

Heavy Sponge Grease, Steam Cooked

In the manufacture of soda base greases, much smaller batches of grease than usual must be made on account of the large amount of expansion that takes place. In the following product the charge should be for approximately one third of the capacity of the kettle. The ordinary steam-jacketed kettle is used.

Formula

	Pounds
Inedible beef tallow	1200
Caustic soda, solid	168
Paraffine oil, 100 vis./100° F.	2620

PROCEDURE

The caustic soda is made up to a 25 per cent solution with water. The tallow is added to the kettle and just melted, at about 125° F., when the caustic solution may be run in, stirring throughout.

The steam is turned on in the jacket and in a short time it is noted that saponification has started. Agitation is continued until it is noted that the soap is completely formed and begins to bunch.

Oil in small quantities up to a total quantity equal to the bulk of the tallow used may be added, as the batch is allowed to sponge.

Heating is continued till practically all of the water has been evaporated from the batch, which may be accomplished at a temperature of 300° F.

The balance of the oil is added slowly so as to avoid breaking up the grease or allowing it to swim in the oil.

When the oil is all in, stirring is continued until the temperature falls to 195° F. The batch may then be drawn into containers.

VARIATION OF GRADE

In making softer grades of fibre grease the steam may be turned off after the first quantity of oil has been added in order to give lower drawing temperatures. The above process may be used for the production of the following three grades of fibre grease:

	Heavy	Medium	Soft
	Per Cent		
Inedible beef tallow	30.00	24.00	18.00
Caustic soda, solid	4.49	3.51	2.63
Paraffine oil, 100 vis./100° F.	65.51	72.49	79.37

Fiber Grease, Fire Cooked

Formula

	Pounds
Prime tallow	150
Caustic soda, solid	22½
Water	33
Paraffine oil, 130 vis./100° F.	600

PROCEDURE

The tallow and an equal weight of the paraffine oil are charged in a fire-heated kettle and the temperature brought to 100° F.

The caustic soda dissolved in the water is then added to the mixture in the kettle and the temperature brought to 250° to 300° F. in from one to two hours.

The balance of the oil is run in and the temperature brought to 400° F.

The fire is then drawn and the batch cooled to 200° F. while stirring. It then may be filled into containers.

Heavy Fiber Grease, Fire Cooked

Use same process as for No. 2.

Formula

	Pounds
Prime tallow	150
Caustic soda, solid	22½
Water	33
Paraffine oil, 130 vis./100° F.	450

Fiber Grease, Steam Cooked

Formula

	Pounds	Per Cent by Weight
Tallow, No. 1	975	26.16
Caustic soda, 30° Bé.	700	4.45
Western pale oil, 180 vis./100° F.	2549	68.38
Water	....	1.01

The process used for the manufacture of the above product is the same as for No. 1. It is of note that the caustic-fat ratio is higher for this grease in which the Western asphalt base oil is used; this allowance is made for the organic acidity of the mineral oil. The melting point of this product is 250° F.

Soft Fiber Grease, Steam Cooked, Western Oil

Formula

	Per Cent by Weight
Caustic soda, solid	3.13
Water	.99
Tallow, No. 1	9.90
Tallow, No. 2	9.90
Asphalt base pale oil, 100 vis./100° F.	76.08

Process same as for No. 1.

Analyses of Fiber Greases

Fiber Grease

	Per Cent by Weight
Free fat	.90
Combined fat	13.37
Free fatty acids	.10
Combined NaOH	1.88
Water	Trace
Mineral oil	83.75
Viscosity of mineral oil at 100° F.	212.

Paper Mill Grease

	Per Cent by Weight
Combined fat	24.4
Free fat	2.0
Free fatty acids	.3
Combined NaOH	3.4
Free NaOH	None
Water	Trace
Mineral oil	69.9
Gravity	27.1° Bé.
Vis./100° F.	165
Flash	380° F.
Cold test	30–35° F.
A.S.T.M. penetration	142

Fibrous Ball and Roller Bearing Greases

An Oklahoma grease plant utilized the following formulae for many years, although it is obvious that the greases produced contained considerable excess caustic soda:

	Heavy Grade Pounds	Medium Grade Pounds
Mid-Continent S. R. stock	90	..
500 S.U.V./100° F. Texas red oil	..	53
Beef tallow	22	12
Caustic soda (NaOH)	6	3
Water	6	3

In order to make a smooth, soft ball bearing grease two parts by weight of the Medium Grade product was milled with one part of the 500 S.U.V./100° F. lubricating oil.

English Fiber Greases

The formulae for these are:

	Hard	Medium	Soft
Tallow fatty acids, per cent by weight	20	16.3	10.5
NaOH	3	2.2	1.5
100/100° F. spindle oil	77	65.5	48.0
Steam refined cylinder stock	..	...	40.0

Standardized Fiber Greases

The formulae for a series of standardized greases are:

Fiber Grease	Constituents	Gallons	Pounds Net	Per Cent by Weight
No. 0	Tallow	60	457.62	6.20
	45° Bé. caustic soda	13.25	66.50	.90
	300 pale oil	900	6855.88	92.90
			7380.00	100.00
No. 1	Tallow	60	457.62	8.25
	45° Bé. caustic soda	13.25	66.50	1.20
	300 pale oil	660	5025.88	90.55
			5550.00	100.00
No. 2	Tallow	60	457.62	11.59
	45° Bé. caustic soda	13.25	66.50	1.68
	300 pale oil	450	3425.88	86.73
			3940.00	100.00
No. 3	Tallow	100	762.70	17.10
	45° Bé. caustic soda	22	100.45	2.48
	300 pale oil	471	3586.85	80.42
			4460.00	100.00
No. 4	Tallow	100	762.70	21.10
	45° Bé. caustic soda	22	110.45	3.04
	300 pale oil	362	2756.85	75.95
			3630.00	100.00
No. 5	Tallow	100	762.70	23.69
	45° Bé. caustic soda	22	110.45	3.43
	300 pale oil	308	2346.85	72.88
			3220.00	100.00

Method

Loading. The tallow, which is kept in 2,000-gallon steam-heated tanks on the loading floor, is measured in the fat-measuring tank, used in conjunction with the autoclave. The melted tallow is then run through the empty autoclave into any one of the grease mixers.

The 45° Bé. caustic soda solution is then measured by hand into 5-gallon measures and run through a vertical galvanized iron loading chute to the kettle containing the melted fat. Paddle agitation is started at once.

Saponification. After the melted tallow and caustic soda have been stirred together for several minutes and a creamy emulsion formed, the steam is turned on in the jacket of the grease kettle, and the saponification carried out as rapidly as possible. Within three to five hours a heavy plastic soap is formed. If, at the end of this time, the grease appears to be sufficiently dry, a small amount (30 to 50 gallons) of 300 Pale Oil is mixed with the soap to soften it and to promote further evaporation of the remaining water. Boiling of the heavy soap base is continued, a small stream of the 300 Pale Oil being run in if, according to the compounders judgement, the base is sufficiently dry. At this stage the grease should become fairly translucent and a tough fiber developed. If the grease is too moist the grease will have a short fiber and a dull, opaque appearance. If too wet, further heating may be required.

After a satisfactory soap base has been made, the desired amount of 300 Pale Oil is added in a small stream, and at a rate which permits it to be taken up by the soap base.

The steam is then turned off and the desired amount of oil run in to bring the grease to proper grade.

A sample of the grease is then sent to the laboratory.

If the grease is too heavy in consistency a further quantity of 300 Pale is added and the consistency again checked. When the grease has been finally approved by the laboratory it is turned over to the fillers and drawn into the required containers.

The softer grades of fiber grease are drawn at a temperature of 120 to 160° F.; the Nos. 3 and 4 grades at 170° to 240° F. No. 5 fiber grease is drawn at the maximum temperature usually obtainable in the kettles, which is 240° to 250° F.

The kettle drains are equipped with screens, through which the grease flows under the force of gravity. These screens are of the mesh indicated below for the various grades of grease being manufactured:

Grade of Grease No.	Size of Screen
00	16 mesh to inch
0	16 mesh to inch
1	¼ inch mesh
2	¼ inch mesh
3	¼ inch mesh
4	None
5	None

SPECIFICATIONS

The specifications for Standardized Fiber Greases are:

Grade	0	1	2	3	4	5	6
A.S.T.M. worked penetration at 77° F.	420–450	360–390	300–330	240–270	180–210	135–165	50–80 (Unworked)
Average A.S.T.M. soap content	4	7	11	16	22	28	34
Min. Ubbelohde dropping point, ° F.	140	185	230	255	285	310	330
Min. Ubbelohde dropping point, ° C.	60	85	110	124	141	154	116
Max. percent free alkali	.50	.50	.50	.50	.50	.50	.50
Max. percent free acid	.30	.30	.40	.40	.40	.40	.50
Max. percent water	.75	.75	1.00	1.25	1.50	1.75	2.50
Max. percent ash	1.6	2.8	3.4	4.5	6.0	6.5	7.0

Free Alkali Content of Fiber Greases

The question is frequently asked: What are the harmful effects of free alkali and free organic acids? Ball bearing manufacturers in many cases

consider that 0.10 per cent is the maximum amount of both alkali and acid which should be allowed. It seems reasonable, however, that free alkali contents up to 0.50 per cent should cause no particular difficulty; particularly if the alkali is dissolved in moisture which may be present. Free fatty acids, on the other hand, should be kept at as low a value as possible without involving excess costs in manufacturing.

Some experiments have been made by adding various amounts of free NaOH and oleic acid to a No. 5 Fiber Grease and then determining the corrosive nature of these greases with respect to copper and steel. The results indicate that free oleic acid in appreciable amounts is more dangerous than free alkali, although neither have any detrimental effect on steel for short periods of time. It should, of course, be kept in mind that commercial greases may contain both free alkali and free acid, simultaneously.

Unworked Penetration at 77° F.	Per Cent Water (W.B.D.) in Grease	Per Cent Free NaOH in Grease A.S.T.M.	Per Cent NaOH added to Grease (by wt.)	Dropping Point Ubb. ° C.	Corrosion Test 48 Hrs. Copper	Steel
170	1.8	.06	0	151	O.K.	O.K.
170	1.4	.06	0	151	O.K.	O.K.
145	1.1	.14	.20	157	O.K.	O.K.
141	1.2	.40	.40	162	O.K.	O.K.
133	1.3	.74	.80	164	O.K.	O.K.
150	..	..	.10 oleic acid	...	O.K.	O.K.
175	..	..	1.00 oleic acid	...	Green Tarn.	O.K.
165	1.0	.10	1.41	155	O.K.	O.K.

Relation Between Soap Content and Worked Penetration for Standardized Fiber Greases

As the result of a great number of A.S.T.M. soap content determinations made on various samples of the Standardized Fiber Greases it was learned that, for a given A.S.T.M. worked penetration, the soap content would normally fall within the limits shown below. For comparison the soap content ranges, as calculated from equations I and II, are also listed:

A.S.T.M. Worked Penetration at 77° F.	A.S.T.M. Soap Content		Soap Content from Equations I and II	
	Max.	Min.	Max.	Min.
475	5	2	4.7	1.9
400	9	3	8.4	4.9
350	12	6	11.3	7.7
300	16	9	14.6	10.0
250	20	13	18.6	13.2
200	25	18	23.4	17.2
150	30	22	...	22.3
100	..	29	...	29.5
75	..	35	...	34.5

The soap content-penetration curves take the form:

$$\log_{10} y = \log_{10} C - mx,$$

in which:

y is the worked penetration,
x is the A.S.T.M. soap content.

and

m is the slope.

Equation I (Maximum Soap Contents) is:

$$\log_{10} y = 2.7705 - 0.020\,x$$

Equation II (Minimum Soap Contents) is:

$$\log_{10} y = 2.7235 - 0.02453\,x$$

Relation Between Water Content and Soap Content (Standardized Fiber Greases)

The average water content is directly proportional to the soap content. The relation is, of course, affected in degree by the nature of the manufacturing process and particularly the maximum temperatures attained during saponification and mixing. The average of a large number of samples of greases made on the foregoing formulae shows the following relationship:

A.S.T.M. Soap Content	Average Water Content by A.S.T.M. Analysis	Water content* from Percent Water = 0.0633 times A.S.T.M. Soap Content
1	...	.06
2	...	.12
4	.25	.25
6	.37	.38
8	.55	.51
10	.76	.64
12	.77	.76
14	.82	.88
16	1.01	1.02
18	1.24	1.14
20	1.26	1.27
25	1.49	1.58
30	1.76	1.90
35	2.20	2.22

* The relation between soap content and water content is linear and takes the general form $y=mx$, in which y is the water content and x is the soap content. The value of m (slope) is of the order of 0.004 or less for dehydrated greases, and for the Standardized Fiber Greases is 0.0633.

Relation Between Soap Content and Ubbelohde Dropping Point (Standardized Fiber Greases)

The average dropping point obtained from a large number of tests on sample of each grade of fiber grease has been found to correlate with the A.S.T.M. soap content as shown in the following table:

A.S.T.M. Soap Content	Average Ubbelohde Dropping Point ° C.
1	50
2	64
4	80
6	95
8	107
10	118
15	138
20	155
25	163
30	177
35	182

Relation between Worked and Unworked Penetration

From the following data for heavy grades of Standardized Fiber Greases it is evident that no fixed correlation of worked with unworked A.S.T.M. penetration is possible.

Grade of Grease Sample	Worked Penetration	Unworked Penetration	Difference
4	224	225	— 1
5	183	159	24
6	172	171	1
4	220	172	48
4	216	178	38
4	293	218	75
5	195	151	44
5	169	120	49
5	197	128	69
5	199	171	28
4	306	246	60
4	308	241	67
4	237	227	10
5	212	222	—10
5	215	207	8
6	175	184	— 9
6	167	146	21
6	175	153	22
7	157	143	14

Relation of Soap Content to Ash

For the Standardized Fiber Greases the ash value multiplied by the average factor, 5.03, will equal the A.S.T.M. soap content.

Standardized Fiber Greases

Grease	Sample	Worked Penetration	Percent Soap A.S.T.M.	Percent Ash	Ratio of Soap to Ash
Fiber Grease No. 00	1	395	3.3	0.66	5.00
" " "	2	442	4.3	0.85	5.06
Fiber Grease No. 1	1	395	3.3	0.66	5.00
" " "	2	341	11.2	2.34	4.79
" " "	3	358	10.9	2.10	5.19
" " "	4	409	8.5	1.57	5.41
" " "	5	395	9.2	1.80	5.11
" " "	6	408	5.2	1.06	4.90
Fiber Grease No. 2	1	340	9.5	1.97	4.82
" " "	2	331	10.7	1.95	5.49
" " "	3	341	14.0	2.54	5.51
Fiber Grease No. 3	1	253	9.5	2.69	3.53
" " "	2	270	16.6	3.25	5.11
" " "	3	302	14.5	3.13	4.63
" " "	4	297	16.9	3.18	5.31
" " "	5	333	12.8	2.85	4.49
Fiber Grease No. 4	1	300	16.6	3.27	5.08
" " "	2	304	17.6	3.50	5.03
" " "	3	274	17.1	3.25	5.26
Fiber Grease No. 5	1	177	30.7	5.61	5.47
" " "	2	176	22.2	4.50	4.93
" " "	3	183	25.5	4.69	5.44
" " "	4	188	23.5	4.85	4.85
" " "	5	219	22.9	4.20	5.45
" " "	6	190	22.3	4.55	4.90
				Average Ratio	5.03

Series P Fiber Greases

Grease	Sample	Worked Penetration	Percent Soap A.S.T.M.	Percent Ash	Ratio of Soap to Ash
Fiber Grease No. 2	1	353	6.0	1.15	5.22
" " "	2	350	5.9	1.23	4.80
" " "	3	330	8.3	1.51	5.50
Fiber Grease No. 4	1	306	14.3	3.26	4.39
" " "	2	237	14.9	3.00	4.97
" " "	3	308	14.8	3.51	4.22
" " "	4	220	16.2	...	...
Fiber Grease No. 5	1	212	26.4	...	...
Fiber Grease No. 6	1	176	22.6	4.55	4.97
" " "	2	165	25.0	5.00	5.00
Fiber Grease No. 7	1	163	24.7	5.01	4.93
				Average Ratio	4.89

English Fiber Grease (F-4 Sponge Grease)

This product has the following characteristics:

	Per Cent by Weight
100 S.U.V./100° F. Mexican spindle oil	77.2
Tallow fatty acids	20.0
Caustic soda	2.8
Water	Trace
A.S.T.M. Penetration	
Worked	232
Unworked	220
Melting point, ° F.	300

Fiber Grease Patents

In 1871, Lake [37] obtained a British patent for a process of manufacturing a lubricating grease. A solution of caustic soda was mixed with tallow and petroleum and the mixture was heated until it ceased to foam. It was then placed in bags and subjected to pressure by which a portion of the oil or liquid was separated. Both the liquid and solid portions were used as lubricants. Animal hair was mixed with the solid portion, which was then used in lubricating-boxes without packing. The Hollis [38] fiber grease was made by processing tallow with soda, caustic potash, and silicate of soda solutions. Castor oil, mineral oil, and either rape, olive, peanut, or cottonseed oils were mixed with the foregoing soaps. Green [39] heated together, in a still mineral oil, alcoholic solutions of caustic soda and potash, petrolatum, and fat. The soap thus obtained was then mixed with further quantities of lubricating oil to produce greases of desired consistency. Green [40] also proposed the production of lubricating greases composed of the soda soaps of oxidized mineral oils and paraffin wax. The mineral oil was blown with hot air and then placed in a still or vacuum pan, equipped with a reflux condenser, and caustic soda added, together with castor oil, vegetable oil, or rosin, and the mixture brought to the boiling point of water. Alcoholic solutions of alkalies were also employed. In a patent

[37] British Patent 1,715 (July 1, 1871).
[38] British Patent 3,855 (Sept. 5, 1881).
[39] British Patent 4,226 (Sept. 5, 1882).
[40] British Patent 5,954 (Dec. 31, 1883).

obtained by Johnson [41] oxidized oleic acid, or chlorinated fats, were saponified with alkali, or alkaline earth oxides, in the presence of mineral or rosin oil. The mixtures were heated to temperatures of about 160° C.

Apparently Markel [42] was first to recognize the value of hydrogenated fats for producing soaps for use in lubricating greases. According to Otto,[43] castor oil for use as a lubricant was heated with about 5 per cent by weight of a metallic salt of stearic or other higher fatty acids, the sodium soap being preferred. The process was also carried out by heating castor oil with an insufficient amount of caustic soda to effect complete saponification. Travis and Glabau [44] passed the following mixture through a colloid mill to obtain a lubricant:

	Parts by Weight
Mineral oil	40
Water	59.5
Sodium oleate	0.5

Their lubricants also consisted of a predominating quantity of mineral oil with as much as 1.5 per cent of sodium oleate, linoleate, stearate, or palmitate. Beckmann [45] manufactured a consistent grease by mixing blubber oil fatty acids, split oil or fat other than castor oil, with mineral oil and alkali. In an example, the following proportions were given:

	Kg
Split blubber oil (about 55 percent fatty acids)	5
Castor oil	5
Tallow (75 percent split)	150
Mineral oil	2000
47° Bé. caustic soda solution	60

Various Analyses

TXC Sponge Grease

This product was intended for general industrial use and particularly for ball bearing lubrication:

Physical Tests:	
Appearance	Fibrous, light color
Odor	Not offensive
Melting point	152° C.
Composition (per cent by weight:)	
Soap	14.47
Oil	83.78
Fillers	0.03
Water	0.80
Alkalinity (NaOH)	0.04
Undetermined	0.88
Total	100.00

[41] British Patent 449 (Jan. 11, 1886).
[42] British Patent 13,042 (June 5, 1907).
[43] British Patent 193,099 (Oct. 19, 1922).
[44] British Patent 195,033 (Mar. 17, 1922).
[45] British Patent 406,136 (Mar. 23, 1933).

Ash:

Na_2CO_3	97.8
Undetermined	2.2
Total	100.00

Fatty Acids from Soap:

Iodine value	49.1
Acid value	203
Melting point	47° C.
Probable composition	Tallow

Mineral Oil:

Gravity ° A.P.I.	26.1
Viscosity at 100° F.	176

A.S. Uba Compound

This fiber grease has been marketed for several years by one of the larger western manufacturers, with considerable success:

Physical Tests:

Appearance	Dark Green, Fibrous
Odor	Caustic
Penetration, A.S.T.M. (unworked at 77° F.)	370
Penetration, A.S.T.M. (worked at 77° F.)	400
Dropping point ° C.	101

Composition (per cent by weight:)

Soap	5.60
Oil	90.10
Moisture	0.80
Free alkali, as sodium hydroxide (NaOH)	0.02
Free fat	0.90
Asphaltic material	1.14
Undetermined	1.44
Total	100.00

Oil Extracted:

Gravity ° A.P.I.	15.6
Viscosity, Saybolt Univ. at 100° F.	2976
Viscosity, Saybolt Univ. at 77° F.	93

Ash, total per cent on original: 0.75

Insoluble in acid	2.3
Sodium carbonate (as Na_2CO_3)	92.5
Calcium	None
Aluminum	None

Fatty Acid from Soap:

Iodine value (Hanus)	44
Acid value (mg. of KOH)	126
Melting point ° C.	61
Color	Very dark brown

"Z" Fiber Grease

This product has been used with success by cement mills, mines, and gypsum plants:

Physical Tests:

Appearance	Fibrous
Color	Light Yellow
Odor	Alkaline
Penetration, A.S.T.M. (unworked) at 77° F.	170
Dropping point ° C.	155

Composition (per cent by weight:)

Soap	23.12
Oil	75.75
Moisture	1.40
Free alkali as sodium hydroxide (NaOH)	0.50
Filler	0.02
Gain in analysis	0.79
Total	100.00

Oil Extracted:

Color, N.P.A.	Approximately 4+
Gravity, ° A.P.I.	21.4
S.U.V. at 100° F.	190
Flash ° F.	339
Fire ° F.	375
Pour point ° F.	—0

Ash, total per cent on original: 4.09

Sodium carbonate (as Na_2CO_3)	99.20
Undetermined	0.80
Total	100.00

Fatty Acids from Soap:

Iodine value (Hanus)	31
Acid value (mg. KOH)	171
Melting point ° C.	42
Color	Light Brown

Probable Composition, per cent by weight:

190 Pale oil	75.6
Soda soap (mixture of stearate and oleate)	23.0
Water	1.4

FIREPROOF GREASES CONTAINING SODIUM SOAPS

In 1876, Bartell [46] proposed a complicated lubricant which was claimed to be fireproof. It consisted of copper sulfate, potassium carbonate, calcium chloride, soapstone, strong sodium hydroxide solution, lard oil, lubricating oil, salt, tartaric acid and water. Straup [47] has developed a compound for

[46] U. S. Patent 183,106 (Sept. 16, 1876).

[47] British Patent 979 (Jan. 14, 1902).

rendering lubricating greases and oils non-explosive and fireproof. The abrasive character of wood ashes, which was one of its ingredients, probably explains its claimed property of causing bearings to run cool. To manufacture this lubricant the inventor suggested that tallow be stirred into a hot solution of wood-ashes, ammonium chloride and quinine.

SODA BASE SOLIDIFIED OILS

Solidified oils or greases are usually made in fire-heated single walled kettles. Variations in this type of grease are due mostly to changing the composition of the soaps, which are usually combinations of tallow, rosin and degras stearins. The object in the selection of the soap stocks is to produce a sodium soap which is more soluble in the mineral oil or one in which the residual affinity of the soap molecules is such that a coarse, fibrous texture is not produced. The process used, the choice of mineral stock and the ratio of caustic soda to fatty ingredients are very important factors in the production of these smooth unctuous soda base greases.

Saponification is carried on in much the same way as performed in making fiber greases, with the exception that after saponification is complete and all the oil is in, the temperature of the batch is run up to 400° to 450° F. The heat is then removed and the temperature allowed to drop until the melting point of the grease is almost reached, which should be about 350° F. depending on the amount of caustic soda used and the titer or melting point of the fatty constituents. The grease is then run into shallow pans having a depth of five inches. If heavy greases are being run, it is allowed to cool over night and is then cut into squares or blocks, wrapped in heavy oiled paper or waxed paper and boxed. If a smooth buttery grease is desired the cooled grease may be run through a mill or if the equipment is available the hot melted grease may be run through a chilling machine to produce the texture desired.

This type of grease is used for general lubrication of bearings, slides, gears, etc. The soft grades may be applied by the usual methods in pressure systems or compression cups, applied to gears directly or pumped into gear cases. The hard grades are used to pack open bearings and are used in the form of bricks or bars.

Soft Solidified Oil

This product is made in a single walled kettle heated by means of a gas or coke fire.

Formula

	Pounds
Vegetable castor oil, No. 3	180
Lard oil, No. 2	120
Lump rosin, Grade F	80
Caustic soda, solid	80
Mid-Continent pale oil, 100 vis./100° F.	5300

The first part of the process is carried out the same as for the production of normal fiber grease by the fire-cooking process.

When a temperature of 400° F. is attained the grease is pumped into shallow pans about ten feet square and five inches deep, where it is allowed to cool over night. The grease is then milled and is ready for filling. The Ross conical buhr stone mills have been found suitable for this milling process.

McKee Process for Manufacturing Solidified Oil [47a]

McKee has patented the process of making a lubricating grease from fat, naphthenic acids derived from petroleum, caustic soda, and mineral lubricating oil. While a typical formula and process are given below, the writer has obtained better results by using a mixture of castor oil and a small amount of rosin in place of the tallow. It is believed that the purpose of the naphthenic acids, which when made into grease are converted to sodium naphthenate, is to give a product with a low melting point which is readily dehydrated, thus resulting in a grease of smooth texture rather than a fiber grease. Cooling the grease in the liquid state to ordinary temperature, at a rapid rate and out of contact with air, is important in securing a satisfactory appearing product.

FORMULA

A typical formula for making this type of grease is as follows:

	Per Cent by Weight
Refined mineral lubricating oil	83.40
Maitland fatty acids, 50 saponification	9.00
Tallow	6.00
Caustic soda, sufficient for complete neutralization and saponification	1.60

The fatty acids referred to are prepared by distilling from Gulf Coast crude petroleum a relatively heavy lubricating distillate, treating the distillate with sulfuric acid and separating out the precipitated sludge with a large part of the acid, treating the remaining body of the acid-containing oil with an alkali such as caustic soda, separating out the precipitated soap and alkaline salt water, decomposing the soap with a relatively weak solution of a mineral acid and eliminating most of the salt water. These acids are undoubtedly naphthenic acids but are probably of a different series than the naphthenic acids obtained by a similar treatment of California crude oil.

In manufacturing this product or greases of a similar character, the fats and mineral oil are charged in a horizontal, cylindrical, fire-heated grease still, in the bottom of which is a spiral screw for agitating and drawing the still contents. The temperature of the still is slowly raised to slightly over 212° F., at which temperature the fire is held in check and the moisture

[47a] U. S. Patent 1,530,386 (Mar. 17, 1925).

resulting from the addition of the caustic soda as a 25 per cent solution is slowly driven off. While the period during which the water is being boiled off and the reaction of the alkali and the saponifiable material is taking place may be shortened to the actual time required to boil off the water without causing the still to boil over, it is advisable to hold the still at the temperature specified for at least four hours in order to insure complete combination of the ingredients.

After this first reaction and driving off of all of the water, the mixture is raised rapidly to about 340° F., after which the burners are turned off and the mixture pumped out of the still into and through the cooler and agitator. Here the mixture is cooled and stirred, in equipment very similar to a wax chiller. The still fluid grease is then discharged into a grease mixer where the process is completed.

It is claimed that grease made by this process has low cold test properties, does not separate or leave soapy deposits or residues, and is capable of being melted and cooled without altering the properties of the lubricant. It is of particular value in the lubrication of spring shackle bolts and other elements that are greased only periodically and then left exposed to the air for a prolonged time.

The following analysis is of a smooth soda base grease:

	Per Cent by Weight
Combined fat	10.25
Free fat	.73
Free fatty acids	Trace
Combined NaOH	1.46
Water	None
Oil	87.56

Oklahoma Solidified Oil

A special bearing grease was manufactured by an Oklahoma grease plant, with the following formula:

	Per Cent by Weight
100 S.U.V./100° F. Lubricating distillate	4.00
15° Bé. caustic soda solution	22.22
Neutral degras	18.22
100 S.U.V./100° F. Texas pale oil	55.56

Kaufman's [48] Ball and Roller Bearing Grease

The formula for a smooth sodium soap base grease has been developed by Kaufman and Puryear, which is as follows:

	Per Cent by Weight
Sodium naphthenate	6.9
Sodium stearate	14.5
Medium lubricating oil	77.89
Water	.71

[48] U. S. Patent 2,055,796 (Sept. 29, 1936).

Formulae for Two Smooth Soda Soap Greases

These formulae are as follows:

	Tallow-Swedish Rosin Oil Blend	Dry Fiber Grease
Swedish rosin oil, per cent	12.50	...
Tallow, per cent	12.50	22.80
Caustic soda, 45° Bé., per cent	8.00	7.80
100 pale oil, per cent	67.00	69.40
	Prepare as a fiber grease	Dry and work 48 hrs. at 290° F.
Tests		
Unworked penetration	195	215
Worked penetration	215	216
Melting point, ° C.	150	200+
Free alkali, per cent	...	0.3
Water, per cent	...	0.05

Solidified Oil Patents

In the following extracts from the patent literature it will be noted that many materials have been selected by inventors to decrease the length of fiber which normally forms in ordinary sodium soap base greases. Some of the materials are rosin, wool fat, waxes, naphthenic acids, gums and oxidized substances. Billington [49] preferred to make use of the soda soaps of rosin oil in order to make a smooth grease suitable for wagon axles, street cars, etc. In his process 200 pounds of tallow was placed in an iron kettle and heated to about 200° F. by injecting live steam. Sufficient 20° Bé. caustic soda solution was then added to saponify the tallow and, while hot, salt was added to cause separation of the soap. The sodium was cooled and cut into bars. The soap was then placed in an autoclave and heated together with rosin oil, 50 pounds of soap being mixed with 100 pounds of rosin oil. While still hot, 50 pounds of mineral oil is mixed in and the grease run into packages where is solidified to form a dense cake of lubricant. In 1884, a patent was granted to Little [50] for a lubricating grease containing potash soaps of rosin oil dissolved in further quantities of rosin oil or mineral oil. Kingzett [51] treated such materials as turpentine, rosin oil, eucalyptus oil, camphor, tallow, etc., with an excess of caustic soda in order to produce a consistent lubricating grease. In 1886, Brough [51a] described a grease cup for use in connection with a lubricant consisting of the sodium soaps of vegetable oils, together with arrowroot or flour. Smith and Pearson [51b] made use of a steam jacketed still, equipped with agitating paddles, for incorporating 3 to 5 per cent of sodium soap with electrically refined lubricating oil, at temperatures of about 350° F.

Templeman [52] developed the process in which any free alkali existing in the soap was neutralized by adding oleic acid or rosin. His soap was

[49] U. S. Patent 179,256 (June 6, 1876).
[50] British Patent 275 (Jan. 2, 1884).
[51] British Patent 2,210 (1885).
[51a] British Patent 4,851 (April 7, 1886).
[51b] British Patent 3,044 (Feb. 20, 1889).
[52] British Patent 17,440 (Oct. 13, 1891).

prepared by combining ground, anhydrous caustic soda with animal or vegetable oils and fats at temperatures above 210° F. The reaction was preferably under pressure and other substances such as clay, rosin, silicate of soda, were incorporated and the grease cake formed in a mould. Pommerhanz [53] developed a process for manufacturing lubricating greases from mineral or fatty oil and fats, without the application of heat. To a quantity of mineral oil, suitable amounts of melted wool fat, caustic soda, and concentrated salt solution were added. After stirring the ingredients combined to form a stiff yellow mass. Some of the materials used were rape oil, olive oil, tallow, cocoa butter, stearin, paraffin, wax, and rosin. The basic patent of Ekenberg [54] covered greases made with both the wool fatty acid salts of calcium and sodium. His process was to dissolve in petroleum the fatty acids derived from wool fat (free from fatty acids found in normal glycerides) and heat to temperatures of 120 to 200° C. when caustic soda was added. Pommerhanz and Ristic [55] prepared a lubricant from Iceland moss soaked in water and boiled to obtain a consistent product. Raw and neutral wool fat, and mineral oil were then stirred in while heating. Caustic soda solution was then added and the mass stirred and allowed to stand to form a consistent cake of grease. It appears that Jarocki and Kickzorowski [56] were first to realize the importance of sodium naphthenates obtained from Caucasian petroleum, as grease bases.

A solidified oil was proposed by Biedermann [57] which was manufactured by treating animal, vegetable, and mineral oils, together with a suitable amount of melted wool fat with caustic soda solution. The entire mass was heated until saponification was substantially complete and a pigment added if desired. Jewett [58] mixed soda soap with fat and added melted rosin to the mixture at a temperature of 225° F. and then incorporated this mixture with peanut oil. Another solidified oil made with soda soap of wool fat was patented by Hutchinson [59] in 1900. The soap was dissolved in benzene, naphtha, and water, to obtain a lubricant for application to wool fibers. Kuess [60] developed a process for solidifying oils in which petroleum was mixed with an equal quantity of animal fat and saponified with a solution of caustic potash, or with a mixture of caustic soda and caustic potash. The autoclave was employed for heating these materials together under pressure at temperatures of 110 to 115° C. Solutions of borax, and copra were also added; no doubt in order to smooth the fibrous texture of the grease. A further disclosure of the use of sodium naphthenate as a grease base was made by Breda,[61] in 1906. He isolated naphthenic acids from the alkali sludge produced from Russian petroleum and treated them with oxidizing agents such as potassium permanganate and then distilled. The

[53] British Patent 9,882 (May 17, 1893).
[54] U. S. Patent 620,899 (Aug. 15, 1896).
[55] British Patent 3,585 (Feb. 12, 1898).
[56] British Patent 13,881 (June 22, 1898).
[57] British Patent 1,029 (Jan. 16, 1899).
[58] U. S. Patent 647,838 (Oct. 20, 1899).
[59] British Patent 15,241 (Aug. 27, 1900).
[60] British Patent 14,568 (June 26, 1906).
[61] British Patent 19,477 (Aug. 31, 1906).

soda soaps of the acids thus obtained were suggested for use as lubricating grease bases. A smooth soda soap grease was developed by LeCocq [62] in 1912. It was prepared by combining cholesterin and other fatty alcohols derived from wool grease, with sodium soaps and mineral oil.

A solidified oil for use as a fuel or lubricant was prepared by Minuto and Porte [63] from paraffine oil and soap powder. Other substances such as camphor, sodium silicate, naphthalene, or beeswax were added. Akker [64] solidified kerosene to produce fuels and lubricants by mixing together at temperatures not over 35° C. the following materials:

	Pounds
Kerosene	90
Stearine	7.5
68° Bé. caustic soda solution	1.33
85 per cent Alcohol	1.16

Triggs [65] was one of the first investigators to recognize the importance of high temperatures in producing solidified oils (soda base greases of smooth texture). His process consisted of adding 6 to 9 per cent of soda soap to mineral oil and heating to a temperature above 350° F., but not over 500° F. In an example, 1.5 pounds of soft soap made from castor, rape seed, or cotton seed oil, was stirred into 20 pounds of mineral oil previously heated to 212 to 250° F., and the temperature increased to 450° F. until frothing ceased. On cooling, without agitation, a stiff jelly was obtained. Triggs [66] also added 7 to 9 per cent of the sodium soaps of the foregoing vegetable oils to heat treated castor oil, to produce a lubricating grease. Maverick [67] has assigned to the Standard Oil Development Company a patent for gear grease composed of mineral oil and synthetic products together with suitable soaps. In general, it comprises a heavy hydroformed or other hydrocarbon oil having a Saybolt viscosity above 95 seconds at 210° F., and a minor proportion of heavy, unctuous synthetic product prepared by condensing with themselves, or with other substances, aliphatic hydrocarbons, of molecular weight 170, or higher, and containing 13.5 to 15.0 parts of hydrogen for each 100 parts of carbon. Less than 1.0 to more than 20 per cent of condensation product may be used, and the oils may contain metallic soaps, for example lead, sodium, or aluminum stearates or oleates. Suitable synthetics are obtained by condensing naphthalene or benzol, or aromatic gas oil, chlorinated paraffine wax, or polymerizing dehydrogenated wax in the presence of aluminum chloride.

Ornfeldt [68] solidifies mineral oils by mixing together soap and natural or artificial beeswax, or essential constituents,—cerotic acid and beeswax alcohols. The product contains 1 to 5 per cent of beeswax and 2 to 8 per cent of soap. Alcohol or other solvent may be mixed with the soap and paraffine,

[62] British Patent 2,382 (Jan. 31, 1912).
[63] British Patent 110,926 (Nov. 4, 1916).
[64] British Patent 166,274 (Oct. 9, 1919).
[65] British Patent 351,041 (Mar. 18, 1930).
[66] British Patent 351,042 (Mar. 18, 1930).
[67] British Patent 396,147 (Mar. 11, 1932).
[68] British Patent 416,927 (Mar. 15, 1933).

ceresin, or other wax with the beeswax. To the products may be added scents, oil of eucalyptus, lemon, thyme, myrbane, or camomile. The jelly-like products may be rendered pulverent by adding magnesium carbonate, talcum or other suitable solids. The products may be used as lubricants.

Soda Soap Greases for Ball Bearing Lubrication

The chief problem in connection with adapting fiber greases for use as ball bearing lubricants is that of reducing their fiber length and altering their texture in such a manner that they will not cling to rotating shafts and be drawn from bearing housings. It is, therefore, not surprising that many inventors have applied for patents relative to smooth soda soap greases (solidified oils) and that many modifiers such as naphthenic acids, wool grease, abietic acid, and other materials have been suggested. McKee and Eckert [69] have developed the formula for a smooth soda soap grease consisting of 85 per cent mineral oil, 9 per cent "Maitland fatty acids" (50 per cent saponifiable naphthenic acid), 6 per cent tallow, and sufficient 30 Bé. caustic soda solution to completely neutralize and saponify these soap stocks. McKee and Eckert [70] have given directions for making a smooth soda soap grease from mineral oil, vegetable oil, naphthenic acids, and caustic soda, heated to above 210° F. to effect dehydration, and then cooling the grease thus formed in a series of pipes equipped with spiral scrapers. Saponification is carried out in a grease still equipped with an agitator. Hughes [71] has developed methods for treating heavy naphthenic lubricating oil fractions with sulfuric acids, neutralization of the oil with caustic soda solution, separation of the emulsion formed, and purification of the naphthenic soaps thus produced. These soaps, or the acids derived from splitting them, have been found suitable for making smooth soda soap greases.

Pungs [72] has noted the effects of various waxes in decreasing fiber length in soda soap greases. He has developed a grease consisting of mineral oil, bleached montan wax, and sufficient soda to neutralize the free acids in the wax. Heat is applied to carry out the reaction. Becker [73] has proposed a lubricant consisting of the sodium soaps of fatty acid oil soluble hydrocarbon sulfonates.

Various Analyses

BT H.M.P. Solidified Oil

This smooth sodium soap grease probably contains salts of naphthenic acid, abietic acid, and possibly degras derivatives. It has been processed at a high temperature (in excess of 375° F.) and cooled quickly to avoid fiber formation.

[69] U. S. Patent 1,529,658.
[70] U. S. Patent 1,530,386 (Mar. 17, 1925).
[71] U. S. Patent 1,577,723 (Mar. 23, 1926).
[72] U. S. Patent 1,776,193 (Sept. 16, 1930).
[73] U. S. Patent 1,888,974 (Nov. 29, 1932).

Physical Tests:

Appearance	Smooth
Odor	Slightly soapy
Color	Transparent yellow-brown
Dropping point ° C.	145

Composition, per cent by weight:

Moisture	.05
Soap (as sodium stearate)	7.0
Free fat	0.1
Petroleum oil	91.5
Free alkali (as NaOH)	0.12
Unaccounted for	1.23
Total	100.00

Petroleum Oil:

Gravity ° A.P.I.	22.0
Color, N.P.A.	2½+
Flash, Cleveland open cup, ° F.	355
S.U.V. at 100° F.	201

Fatty Acids from Soap:

Color	Dark Brown
Odor	Resembling Stearic Acid
Iodine value (Hanus)	65
Acid value (mg. KOH/gram)	176

Ash, per cent by weight: 1.32

Sodium carbonate (Na_2CO_3)	95.7
Aluminum and iron (mostly Fe_2O_3)	1.1
Calcium oxide (CaO)	0.9
Unaccounted for	2.3
Total	100.00

KSN Solidified Oil No. 748

It is likely that this product has been manufactured by heat treating at high temperatures. It is obvious that the soap stocks have been selected most ingeniously. The exact nature and source of the fatty acids is, of course, not revealed by the normal routine analysis:

Physical Tests:

Appearance	Smooth
Odor	Soapy
Color	Green brown
Dropping point ° C.	145
A.S.T.M. penetration at 77° F. (unworked)	273
A.S.T.M. penetration at 77° F. (worked)	298

Composition, per cent by weight:

Free alkali (as NaOH)	0.3
Moisture	0.1
Soap (as sodium stearate)	15.9
Free fat	0.5
Mineral oil	38.8
Gain in analysis	0.6
Total	100.00

Mineral Oil:	
Gravity ° A.P.I.	19.7
Color, N.P.A.	8–
Flash, Cleveland open cup ° F.	380
S.U.V. at 100° F.	406
Fatty Acids from Soap:	
Color	Brown
Odor	Resembles Stearic Acid
Neutralization value (mg. KOH/gr.)	200
Iodine value (Hanus)	8.0
Dropping point ° C.	51
Ash, per cent by weight:	2.73
Iron and aluminum (mainly Fe_2O_3)	0.4
Sodium carbonate (Na_2CO_3)	96.4
*Unaccounted for	3.2
Total	100.00

* Calcium, magnesium and lead absent.

HPH No. 465 Non-Leak Grease

This dark colored, smooth texture sodium soap base grease has excellent non-leak and non-drip characteristics, and is suitable for industrial and automotive gear lubrication for temperatures as low as 30° F.

Gravity ° A.P.I.	12.5
S.U.V. at 210° F.	335
S.U.V. at 100° F.	3,050
Channel test at 35° F.	O.K.
Channel test at 30° F.	Not O.K.
Flash, Cleveland open cup ° F.	405
Fire ° F.	455
Corrosion, copper strip 3 hrs. at 212° F.	O.K.
Alkalinity, mgs. KOH per gram	2.20
Saponifiable, per cent	5.10
Asphaltenes, Holdé method, per cent	3.85
Benzol insoluble, per cent	1.86
Ash, per cent	5.26
Ash Analysis:	
Acid insoluble (mostly silica as SiO_2)	6.5
Iron (as Fe_2O_3)	16.0
Calcium sulfate (as $CaSO_4$)	40.6
*Sodium (as sodium sulfate Na_2SO_4)	37.2
Magnesium (small amount present)	
Gain in analysis	0.3
Total	100.00

Timken Film Strength: O.K. at 17,160 pounds
" " " Not O.K. at 20,000 pounds

Timken Wear Test: (375 feet per minute, 17,160 pounds per square inch)

Loss in cup	6 mgs.
Loss in block	4 mgs.
Total loss in weight	10 mgs.

* The presence of sodium was identified. The value for sodium sulfate was obtained by calculation from the sulfates actually determined.

Requirements of Sodium Soap Base Ball and Roller Bearing Greases

In Chapter V and the Appendix of this volume sections have been devoted to general discussions of calcium base ball bearing greases. In this section purchase specifications, typical formulae and manufacturing processes for various sodium soap ball bearing greases will be presented. It may be noted that various agents for increasing film strength (organic sulfur compounds, phosphates, chlorides, tellurides, etc.) are in some cases added to the normal sodium soap greases; also, that various anti-oxidants, which will be listed in Chapter X, are becoming popular additions to the normal greases, in order to produce lubricants which are less subject to oxidation and which may be left in the bearing housings for longer periods of time without renewal.

Various Specifications

In 1928 a large western railroad company issued the following specifications governing the purchase of greases for their use. These specifications have been in effect for nine years and it is reasonable to conclude that the greases purchased in accordance with their requirements have given satisfaction.

Uses:

For lubricating ball or roller bearings on industrial electric motors.

Material:

Two types of lubricant, grease and oil, are covered by this specification.

Selection of Lubricant:

The kind of lubricant used will be governed by condition of speed and bearing temperature as shown in the following table:

Shaft Revolutions per Minute			Bearing Temperature	
Shafts, 1¾" and under	Shafts, 2" to 3½"	Shafts, 4" and over	32° to 150° F.	Over 150° F.
Below 400	Below 200	Below 100	Medium Oil or Grease	Heavy Oil
400 to 2000	200 to 1000	100 to 500	Medium Oil or Grease	Heavy Oil
2000 to 6000	1000 to 3000	500 to 1500	Light Oil, Medium Oil or Grease	Medium Oil
Over 6000	Over 3000		Light Oil	Medium Oil

General Characteristics of Grease and Oils:

To be free as possible from acid forming tendencies, to insure adequate protection of the polished metal surfaces. There must be no tendency for the grease to cause corrosion or pitting of balls, roller or raceways. A bright copper plate shall show no discoloration after being submerged in the lubricant for three hours at 212° F. temperature.

Grease

Material:

Shall consist of a high grade soda soap and a refined clean mineral oil, having a minimum viscosity of 200 sec. Saybolt Universal at 100° F.

Physical Characteristics:

Must not separate on standing or on heating below its melting point.
Must not gum or become sticky.
Must not harden or decompose.

Must not contain resin, mineral salts, sand, free lime, graphite or fillers of any kind, nor over 1 per cent water.

Shall have consistency of No. 2 grease.

Free fatty acid per 1 gram grease must not exceed the equivalent of 0.3 mg. KOH, per 1 gram grease.

Free alkali per 1 gram grease must not exceed the equivalent of 0.3 mg. KOH, per 1 gram grease.

Free saponifiable oil not to exceed ½ per cent.

Methods for testing as given in Bureau of Mines Technical Paper 323 B to be followed.

It is generally conceded that properly made sodium soap greases, even though they may be effected by the presence of water, are more stable in service and are less affected by temperature changes than calcium base

* Speed R.P.M.	Bearing Series	Bearing Bore Size		OPERATING TEMPERATURE					
				Below 32° F.	32° to 150° F.		150° to 200° F.		Above 200° F.
		Grease	Oil		Moisture Present	No Moisture	Moisture Present	No Moisture	
Up to 500	Light	0 to 22	ALL SIZES	Consult Engineering Dept.—New Departure Mfg. Co.	Oil: 400-500 Sec. Visc. @ 100° F. †	400-500 Sec. Visc. @ 100° F.	500-600 Sec. Visc. @ 100° F. †	500-600 Sec. Visc. @ 100° F.	Consult Engineering Dept.—New Departure Mfg. Co.
	Med.	0 to 22			Grease: CALCIUM 12-14% Soap Oil as Above	SODIUM 12-14% Soap Oil as Above		SODIUM 18-20% Soap Oil as Above	
500 to 2000	Light	0 to 18			Oil: 400-500 Sec. Visc. @ 100° F. †	400-500 Sec. Visc. @ 100° F.	500-600 Sec. Visc. @ 100° F. †	500-600 Sec. Visc. @ 100° F.	
	Med.	0 to 16			Grease: CALCIUM 12-14% Soap Oil as Above	SODIUM 12-14% Soap Oil as Above		SODIUM 18-20% Soap Oil as Above	
2000 to 5000	Light	0 to 10			Oil: 300-400 Sec. Visc. @ 100° F. †	300-400 Sec. Visc. @ 100° F.	400-500 Sec. Visc. @ 100° F. †	400-500 Sec. Visc. @ 100° F.	
	Med.	0 to 9			Grease: CALCIUM 12-14% Soap Oil as Above	SODIUM 12-14% Soap Oil as Above		SODIUM 18-20% Soap Oil as Above	
5000 to 10,000	Light	0 to 6			Oil: 200-300 Sec. Visc. @ 100° F. †	200-300 Sec. Visc. @ 100° F.	300-400 Sec. Visc. @ 100° F. †	300-400 Sec. Visc. @ 100° F.	
	Med.	0 to 5			Grease:	SODIUM 12-14% Soap Oil as Above		SODIUM 18-20% Soap Oil as Above	
** Above 10,000	Light	0 to 4			Oil: 100-150 Sec. Visc. @ 100° F. †	100-150 Sec. Visc. @ 100° F	200-300 Sec. Visc. @ 100° F. †	200-300 Sec. Visc @ 100° F.	
	Med.	0 to 3			Grease:	SODIUM 12-14% Soap Oil as Above		SODIUM 18-20% Soap Oil as Above	

* Limiting bearing speed based on ball surface speed of approximately 3000 feet per minute.

† Oils used should not readily emulsify with water.

Grease must be of smooth texture—not of fibrous nature.

** It is recommended that high speed applications, particularly above 15,000 r.p.m., be referred to the New Departure Engineering Department for approval.

Note: Specifications in the above table are for general guidance. Commercial lubricants, which vary slightly from these specifications, should give satisfactory results.

Potassium base greases of similar specifications may be used where sodium grease is specified above.

Radax bearings, Types 20,000 and 30,000, should be lubricated with oil for all speeds and temperatures.

greases. In 1932, the New Departure Manufacturing Company [74] issued the general recommendations tabulated on page 401, for lubricants for use under various service conditions and it will be noted that where moisture is not encountered, the sodium greases are preferred.

The general requirements of this company prior to 1932 were for a sodium grease containing 18 to 20 per cent of soap, as indicated below, and no restrictions were placed on the oil viscosity as indicated in the foregoing table.

Soap	18 to 20 per cent
Kind of soap	Sodium
Water	Not over .1 per cent
Acid (oleic)	Not over .2 per cent
Dripping point	380 to 400°

The lubricant must be free from foreign matter such as silica, etc., and also have no staining effect on brass.

Various Analyses

Sodium Soap Base Ball Bearing Greases

The following analyses are for commercial greases which have been approved by one or more ball bearing manufacturers during the past three years, and represent fairly well the qualities being currently demanded by bearing manufacturers.

GE Ball Bearing Grease

Physical Properties:

Appearance	Green-brown, granular
Odor	Pungent, soapy
Penetration at 77° F.:	
Unworked	155
Worked	270
Dropping point (Ubbelohde) ° C.	133

Composition: (per cent by weight)

Soap	16.8
Oil	81.5
Water	0.1
Acidity (as oleic)	0.56

Ash:

Per cent		3.20
Analysis (percent of ash)	Na_2CO_3	93.6
	Fe_2O_3	0.8
	CaO	0.6
	Na_2SO_4	trace
	Acid insoluble	trace
	Undetermined	5.0
	Total	100.0

[74] "Lubrication of New Departure Ball Bearings." (1932).

Mineral Oil:

Color	Opaque
S.U.V. at 100° F.	166
S.U.V. at 210° F.	44.2
Gravity, ° A.P.I.	27.3
Flash, o. c. ° F.	365
Fire o. c. ° F.	420

LKO-M Ball and Roller Bearing Grease

This grease gave the following results:

Physical Tests:

Appearance	Smooth
Odor	Slightly Soapy
Color	yellow
Dropping point ° C.	157
A.S.T.M. penetration at 77° F. (unworked)	275
A.S.T.M. penetration at 77° F. (worked)	309

Composition, per cent by weight:

Free alkali (as NaOH)	0.9
Moisture	0.2
Soap (as sodium stearate)	18.3
Free fat	0.4
Mineral oil	80.0
Loss in analysis	0.2
Total	100 0

Mineral Oil:

Gravity ° A.P.I.	26.2
Color, N.P.A.	5+
Flash, Cleveland, open cup	385
S.U.V. at 100° F.	247

Fatty Acids from Soap:

Color	Light brown
Odor	Resembling stearic acid
Neutralization value (mg. KOH/gr.)	180
Iodine value (Hanus)	40.2
Dropping point ° C.	44

Ash, per cent by weight: 3.47

Iron and aluminum (mainly Fe_2O_3)	0.4
Calcium oxide (CaO)	9.4
Sodium carbonate (Na_2CO_3)	87.8
Unaccounted for	2.4
Total	100.00

F-B-M Ball Bearing Grease

This grease gave the following results:

Physical Tests:	
Appearance	Smooth, stringy
Color	Golden brown
Odor	Similar to Oil of Mirbane
Penetration, A.S.T.M. (unworked) at 77° F.	241
Penetration, A.S.T.M. (worked) at 77° F.	292
Dropping point ° C.	150
Composition, per cent by weight:	
Oil	85.9
Filler	None
Moisture	0.7
Free acid (calculated as oleic acid)	0.2
Soap	12.3
Loss	0.9
Total	100.0
Oil Extracted:	
Color	Green red
Gravity, ° A.P.I.	27.2
S.U.V. at 100° F.	469
Ash, total per cent on original	2.23
Acid, insoluble	None
Iron and aluminum (as R_2O_3)	1.01
Calcium oxide (as CaO)	0.02
Magnesium oxide (as MgO)	0.01
Sulfur trioxide (as Na_2SO_4)	3.07
Balance as ash was considered as sodium carbonate (Na_2CO_3) by difference	95.89
Total	100.00

Comments

The product is a soda base grease.

The grease is prepared from an eastern oil of low viscosity.

The water content, 0.7 per cent, indicates that the grease was not prepared by a high heat process.

The excellent appearance, especially as regards the color, of the grease is probably due in part to a certain content of eastern bright stock.

The grease is slightly acid, rather than alkaline, which is probably responsible for some of the reduction in the amount of fiber found in normal fiber grease.

There was no evidence of any large concentration of petrolatum in the grease.

The high degree of transparency and the absence of a decided fibrous texture are very unusual in a soda base grease not prepared by a high heat process.

This reduction in fiber was probably accomplished through an ingenious selection of soap stock, and possibly also through the use of a grease mill.

The analysis is of interest in that it indicates the possibility of preparing a quite smooth, transparent soda base grease without resort to high heat.

UN-RL Ball and Roller Bearing Grease

This grease gave the following results:

Physical Tests:	
Appearance	Stringy
Odor	Soapy
Color	Brown
Dropping point ° C.	199
A.S.T.M. penetration at 77° F. (unworked)	169
A.S.T.M. penetration at 77° F. (worked)	199
Composition, per cent by weight:	
Asphaltic material (P. E. insoluble)	0.2
Free alkali (as NaOH)	0.2
Moisture	0.1
Soap (as sodium stearate)	20.2
Lead soap (as lead stearate)	1.0
Free fat	0.7
Mineral oil and unsaponifiables	78.5
Gain in analysis	0.9
Total	100.0
Mineral Oil:	
Gravity, ° A.P.I.	26.3
Color	6 dil
Flash, Cleveland, open cup	440
S.U.V. at 210° F.	83
Pour point ° F.	110
Fatty Acids from Soaps:	
Color	Brown
Odor	Resembles both stearic and oleic acids
Neutralization value (mg. KOH/gr.)	178
Iodine value (Hanus)	50.7
Dropping point ° C.	40
Ash, per cent by weight:	2.65
Iron and aluminum (mostly aluminum)	13.9
Calcium oxide (CaO)	0.8
Sodium carbonate (by difference)	85.3
Total	100.00

Calipsol Fiber Grease [75]

This showed the following results:

	Analysis
Penetration at 77° F. (A.S.T.M.)	188
Ubbelohde dropping point ° C.	120
Percent water	0.40
Percent free fatty acid (as oleic)	0.25
Percent mineral oil	82.08
S.U.V./100° F. of mineral oil	114
Percent ash as sodium carbonate	3.19

[75] British Patent 6,640 (Feb. 22, 1912). This grease mentioned as a composition having a melting point in excess of 100° C.

BT High Melting Point Ball Bearing Grease

This grease gave the following results:

Physical Tests:

Color	Dark green
Odor	Slightly soapy
Appearance	Slightly stringy
Dropping point ° C.	167
Penetration at 77° F. (unworked)	338
Penetration at 77° F. (worked)	375

Composition, per cent by weight:

Moisture	Trace
Soap (as sodium stearate)	3.6
Free fat	0.3
Petroleum oil	96.2
Free alkali (as NaOH)	0.1
Gain analysis	0.2
Total	100.0

Petroleum Oil:

Gravity, ° A.P.I.	20.4
Color	Green red
Flash, Cleveland open cup	450
S.U.V. at 100° F.	1965
S.U.V. at 210° F.	96

Fatty Acids from Soaps:

Color	Dark brown
Odor	Resembling stearic and oleic acids
Iodine value (Hanus)	68
Neutralization value (mg. KOH/gr.)	187

Ash, per cent by weight: 0.65

Sodium carbonate	94.6
Iron and aluminum (Mostly Fe_2O_3)	2.7
Undetermined (CaO and MgO absent)	2.7
Total	100.0

Visco Grease for Motor Bearings

This grease gave the following results:

Physical Tests:

Appearance	Fibrous
Odor	Slightly soapy
Dropping point ° C.	177
Penetration A.S.T.M., worked at 77° F.	245
Penetration A.S.T.M., unworked at 77° F.	234

Composition, per cent by weight:

Asphaltic material (P. E. insoluble)	0.1
Soap	16.6
Free fat	0.4
Free alkali (as NaOH)	0.1
Oil (includes petroleum or wax)	81.5
Moisture	0.1
Unaccounted for	1.2
Total	100.0

Mineral Oil:

Gravity, ° A.P.I.	23.9
Pour point ° F.	105
S.U.V. at 130° F.	401
S.U.V. at 210° F.	86
Color	Green black

Fatty Acids:

Acid value (mgs. KOH/gr.)	200
Iodine value (Hanus)	49
Dropping point ° C.	43
Color	Dark brown

Ash, per cent by weight:	2.23
Sodium carbonate (as Na_2CO_3)	100.4
Gain in analysis	0.4
Total	100.00

KSN Ball Bearing Grease

This grease gave the following results:

Physical Tests:

Appearance	Smooth
Odor	Soapy
Color	Green brown
Dropping point ° C.	145
A.S.T.M. penetration at 77° F. (unworked)	273
A.S.T.M. penetration at 77° F. (worked)	298

Composition, per cent by weight:

Free alkali (as NaOH)	0.3
Moisture	0.1
Soap (as sodium stearate)	15.9
Free fat	0.5
Mineral oil	83.8
Gain in analysis	0.6
Total	100.0

Mineral Oil:

Gravity ° A.P.I.	19.7
Color, N.P.A.	8–
Flash, Cleveland open cup ° F.	380
S.U.V. at 100° F.	406

Fatty Acids from Soap:	
Color	Brown
Odor	Resembles stearic acid
Neutralization value (mg. KOH/gr.)	200
Iodine value (Hanus)	8.0
Dropping point ° C.	51
Ash, per cent by weight:	2.73
Iron and aluminum (mainly Fe_2O_3)	0.4
Sodium carbonate (Na_2CO_3)	96.4
Unaccounted for	3.2
Total	100.00

High Temperature Magneto Grease

This grease gave the following results:

Physical Tests:	
Appearance	Soft smooth cup grease
Melting point	175 Drop
Odor	Not offensive
Composition, per cent net:	
Soap	26.6
Moisture	Trace
Free fat	1.02
Oil	72.12
Free Na_2O	.26
Ash:	
Na_2CO_3	5.25
Sodium oxide	95.4
Undetermined	4.6
Mineral Oil:	
Color	6 plus
S.U.V.	115/100
Grav. at 60° C. A.P.I.	29.9

TXM No. 3 Ball Bearing Grease

This grease gave the following results:

Physical Tests:	
Appearance	Dark, sticky, smooth
Melting point ° C.	130
A.S.T.M. penetration	180
Odor	Soapy
Composition, per cent net:	
Soap	13.1%
Moisture	1.0
Free fat	.5
Oil	85.2
Free Na_2O	.2

Ash:

Na_2CO_3	2.3
Sodium oxide	94.8
Sulfates	1.1
Undetermined	4.1

Fatty Acid:

Acid value	204
Iodine value	52

Mineral Oil:

S.U.V./116 at 210° F.	
Gravity at 60° C.	26.8

ANTI-CORROSIVE BALL AND ROLLER BEARING GREASE

The following analysis covers a lubricant which has been found to give very satisfactory service when applied to the bearings on which battleship gun turrets turn. It appears to have the ability to prevent excessive corrosion and rusting of the antifriction bearings which in some cases come in contact with sea water. It appears to contain iron soaps or other organic iron compounds:

Physical Tests:

Appearance	Greenish brown
Odor	Somewhat similar to lard oil
Viscosity, MacMichael at 80° F.	35
Dropping point ° C.	27

Composition (per cent by weight):

Soap	0.27%
Oil	99.10
Moisture	0.45
Free alkali	Trace
Insoluble	0.03
Gain or loss	0.15
Total	100.00

Mineral Oil:

Color	Greenish brown
Gravity ° A.P.I.	27.1
S.U.V. at 100° F.	2640
S.U.V. at 210° F.	142
Dropping point ° C.	17

Ash, Total per cent on original: 0.19%

Sodium carbonate (as Na_2CO_3)	26.00%
Calcium carbonate (as $CaCO_3$)	Trace
Undetermined (principally iron)	74.00
Total	100.00

Various Formulae

Two Ball Bearing Greases (No. 2 Short Fiber and No. 5 Long Fiber)

These formulae are as follows:

	No. 2 Short Fiber	No. 5 Long Fiber
Oleic acid	6.11	....
Stearic acid	6.28	....
Tallow	....	16.78
Caustic soda, 45° Bé.	1.85	2.43
1000 pale oil	58.43	....
1000 red oil	....	8.48
150 S.U.V./210° Br. Stk.	11.33	....
Dark green petrolatum	16.00	72.31 Mid-Cont S. R. Stock
	100.00	100.00

Typical Tests

Appearance	Standard Sample	...
Unworked penetration	200–250	250–300
Worked penetration	340*, 310*	325*
Water, per cent by weight	0.1*	1.5
Melting point, ° C.	180	100, 126*
Free alkali, per cent	0.25	0.2*
Free acid, per cent	None*	None*
Fillers	None*	None*
Corrosion	None*	None*
Mineral oil vis. at 210° F.	78*	153*
Kind of soap	Sodium	Sodium

Specifications

Appearance	Standard Sample	Standard Sampl
Worked penetration	300–330	300–330
Water, per cent by weight, max.	1.5	1.5
Melting point	180	120
Free alkali, per cent max.	0.5	0.5
Free acid, per cent max.	0.3	0.3
Fillers	None	None
Corrosion	None	None
Mineral oil vis. at 210° F.	60–90	150–190 (2000–3000 at 100° F.)
Kind of soap	Sodium	Sodium

A practical test in an electric motor developing 20 H.P. and operating at 3600 R.P.M. has been made with the No. 5 Long Fiber Ball Bearing Grease. It shows that this grease is retained well and does not develop excessive bearing temperatures. The results are given below:

* By actual test

5 p. m. at Start	Short End		Pulley End		Grease Running Out	
	Temp.	Rise	Temp.	Rise	Short End	Pulley End
5:10	28	5			None	None
5:20	31	8	26.5	3.5	None	None
5:30	34	11	27.5	4.5	None	None
5:40	37	14	28	6	None	None
5:50	39	15	28.5	6.5	None	None
6:00	41	18	28.5	6.5	None	None
6:20	42	19	29	6	None	None
6:40	44	21	29	6	None	None
7:00	44.5	21.5	28	5	None	None
7:30	40	17	27.5	4.5	None	None
8:00	35.5	13.5	28	5	None	None
8:30	28	6	27	5	None	None
9:00	28	6	27	5	None	None
5 P. M.	27	6	26	5	None	None

Soft Ball Bearing Grease

A short fiber ball bearing grease, having a melting point of about 155° C. and an unworked A.S.T.M. penetration of 380, may be made on the following formula:

	Per Cent by Weight
Oleic acid	2.95
Stearic acid	3.04
NaOH	.89
Petrolatum	6.79
Heavy naphthenic bright stock	4.37
1000 pale oil	22.81
300 pale oil	59.15
	100.00

Specifications

Worked penetration at 77° F.	360–390
Melting point (Ubbelohde Method) ° F., Min.	300
Free alkali, percent NaOH, Max.	0.25
Corrosion	OK
Mineral oil S.U.V. at 100° F.	400–600
Water by distillation, per cent by weight, Max.	0.75
Must conform to standard sample Miscellaneous specification	Fillers, None

No. 4 Ball Bearing Grease

A heavy, smooth texture, sodium soap ball bearing grease may be made on the following formula:

	Per Cent by Weight
Stearic acid	10.42
Oleic acid	10.12
Caustic soda	3.07
Petrolatum	16.20
Motor 70	9.82
1000 Pale	50.37
	100.00

Tests

Penetration—A.S.T.M. at 77° F. worked	210
Dropping point—Ubbelohde ° C.	200
Free alkali, per cent	0.45
Ash, per cent	4.95
Water, per cent	0.05
Soap, per cent by titration	23.10
Viscosity of oil extracted—S.U.V. at 210° F.	78

Estimated Cost

The following table gives an estimate of the cost of No. 4 Ball Bearing Grease. "A," indicates the out of pocket cost; "B," the out of pocket, plus overhead and depreciation of plant; and "C," is the out of pocket cost to which the overhead only has been added.

Description of Item	Quantity	Unit Cost, Cents			Total Cost, Dollars		
		A	B	C	A	B	C
Stearic acid	104.2 lbs.	9.37	9.37	9.37	9.76	9.76	9.76
Oleic acid	101.2 lbs.	9.25	9.25	9.25	9.36	9.36	9.36
Caustic soda	30.7 lbs.	2.82	2.82	2.82	.87	.87	.87
Petrolatum No. 8700	162.0 lbs.	2.91	2.91	2 91	4.71	4.71	4.71
140/210 Motor oil (98.2 lbs.)	12.6 gals.	4.99	7.65	6.03	.63	.96	.76
1000 Pale oil (503.7 lbs.)	65.4 gals.	4.15	6.20	4.95	2.71	4.05	3.24
Compounding cost	1000 lbs.	.08	.18	.12	.80	1.80	1.20
Cost ball bearing grease No. 4, ex kettle	1000 lbs.	2.88	3.15	2.99	28.84	31.51	29.90

BALL BEARING GREASES OF VASELINE TEXTURE

The following formulae were investigated in order to develop greases of Vaseline texture, which would be non-separating while in storage and in operation, and which would not become excessively fibrous due to the agitation and heat developed in bearings.

Grease No.	Caustic Soda	Beeswax	Degras	Tallow	Sperma-ceti	Castor Oil	Palm Oil	Stearic Acid	Coco-nut Oil	300 Pale Oil	Al Stearate Grease
1	2.0	12.0	...	8.0	...	...	...	...	...	78.0	...
2	2.1	6 8	...	...	5.5	6.8	...	...	...	78.8	...
3	2.3	...	...	...	...	...	7.0	8.0	...	82.7	...
4	3.0	...	...	...	...	...	...	10.0	5.0	82.0	...
5	2.2	...	20.0	...	...	...	...	...	...	77.8	...
6	1.7	...	...	...	...	...	...	12.0	...	71.3	15.0

These greases were all treated at temperatures of 350° F., or higher, and allowed to cool without appreciable agitation. Equal amounts of water were used to dissolve the caustic soda making a 50 per cent solution.

The characteristics of these greases as compared with seven commercial greases are shown in the following table:

Melting Test

Grease	Temperature of Pronounced Structure Change ° F.	App. Temp. of Complete Solution of Soap in Oil	Extent of Separ. of Free Oil After Cooling Per Cent*	Ubbel. Dropping Point ° C.	Worked Penetration
EL	320	370	None	...	...
KSN	300	450	0.2	145	298
LKO-M	Very slight change at 260°	370	None	...	...
UN-RL	No sudden consistency change noted	470	None	199	199
BT-HMP	240	360	11	145	...
BT-L	No sudden consistency change noted	400	None	167	375
SH	No sudden consistency change noted	470	None	Approx. 200	225–350
1	310	360	None	154	172
2	260	330	Trace	...	238
3	320	420	Trace	180	...
4	300	430	32	171	225
5	230	310	None	136	248
6	290	430	None	...	...
7	350	470	Very extensive	...	...

Grease	Oil S.U.V. at 100° F.	Type of Soap	Texture	Separation in Storage
EL	2200	Soda-Lime	Smooth	.....
KSN	406	Soda	Smooth	Trace
LKO-M	....	Soda-Lime	Smooth	Trace
UN-RL	83 at 210° F.	Soda-Aluminum Lead	Fibrous	None
BT-HMP	201	Soda	Smooth	Trace
BT-L	1965	Soda	Smooth	Extensive
SH	....	Soda	Fibrous	None
1	300–330	Soda	Smooth	None
2	300–330	Soda	Smooth	None
3	300–330	Soda	Smooth	None
4	300–330	Soda	Smooth	None
5	300–330	Soda	Smooth	None
6	300–400	Soda-Aluminum	Smooth	None
7	1000	Soda	Smooth	.....

* 500 grams of grease were melted in a 1500 c.c. beaker and the grease was allowed to cool spontaneously. Separation was noted after approximately 24 hours.

English Ball Bearing Greases

Formula

Four English Ball Bearing greases are compounded as follows:

Designation	R-B-4 Per Cent by Weight	F-D-4 Per Cent by Weight	No. 45 Per Cent by Weight	Fiber Grease No. 4 Per Cent by Weight
110 S.U.V./100° F. Mexican spindle oil	84.0	48.0	65.5	77.0
Tallow	14.0			
Sodium hydroxide	2.0	1.5	2.2	3.0
Naphthenic cylinder stock	...	40.0	16.3	...
Tallow fatty acids	...	10.5	16.0	20.0

Analyses

Typical analyses of these greases have been made with the following results:

	F-D-4	Fiber Grease No. 4	No. 45
Water (W.B.D.), per cent	0.8	1.2	0.8
Percent ash	2.43	4.17	3.33
Soap base	Sodium	Sodium	Sodium
Melting point, ° C.	134	146	146
Free alkali	0.61 (NaOH)	0.52 (NaOH)	0.24 (NaOH)
Penetration at 77° F. (unworked)	324	227	283

German Tests on Roller Bearing Greases

A device was constructed for conducting a series of tests. It consisted of a 55 cm. ball bearing with grease retainer washer mounted at the end of a shaft and coupled to a roller bearing. The two bearings were mounted in the same housing which was provided with a hook for suspending any desired weight. The roller bearing had a diameter of about 130 cm. and was provided with a thermometer for indicating the temperature increases within. In making the tests the grease was filled to within 1 cm. of the top rim and the bearings run at a shaft speed of 2000 R.P.M. for 12 days. This was considered equivalent to 100,000 kilometers. Tests were also made at 3000 R.P.M. with a suspended load of 670 kilograms. The greases tested were as follows:

Grease No.	60 Per Cent by Weight	83 Per Cent by Weight	2 Per Cent by Weight
T.G. wax	7	4	4
Montan wax Nova	...	4	4
Montan tax T.V.	...	3	3
Tallow	3	...	...
200 S.U.V. at 100° F. spindle oil	89	88	...
100 S.U.V. at 100° F. spindle oil	...	...	88
40° Bé. sodium hydroxide solution	3.3	2.1	2.1

Other greases tested were the English Fiber Grease F-4; the English ball bearing grease R-B-4; V.O.C. 1200; and Calypsol grease. No. 60 darkened during all tests, became dry on the surface and somewhat brittle. The grease was rather sticky at the start and caused a high starting resistance. After a short while it became worked to a micaceous condition by the rollers and the parts were not well lubricated. No. 83 was satisfactory in all tests. It retained its color, became only slightly firmer and dryer, without losing its greasy effect. The rollers were well greased throughout.

No. 2 was also satisfactory in all tests. It retained its color, was slightly dry on the surface and was sufficiently firm at high temperatures. V.O.C. 1200 failed in all tests. This grease appeared to be too soft from the beginning and was consequently too much consumed. This led to increased temperatures. It separated oil and formed lumps. Calypsol grease also failed, because it became very dry, micaceous, and formed lumps. At higher temperatures it was very firm and rubber-like. This was due to a pro-

nounced fibrous structure. English grease F-4, which also had a fibrous structure, leaked badly due to its soft consistency and consequently produced a high increase in temperature. It became soft and black. The rollers were heavily interspersed with this soft black grease.

No. 2 and No. 83 were therefore considered the best products.

Behavior in the Cold

A study was made of the consistency of the various greases at temperatures of − 20° and − 30° C. with the following results:

Grease	1½ hours at −20° C.	1½ hours at −30° C.
No. 60	firm	hard
No. 83	hard	hard
No. 2	firm	hard
No. 45	firm	firm
R-B-4	soft	medium
F-D-4	soft	firm
Calypsol	firm	very firm
V.O.C. 1200	very soft	soft
V.O.C. 1200-H	soft	medium soft

Condition of Bearings

Grease	Creeping After 24 Hours	Condition of Bearings After 24 Hours
R-B-4	Little; at higher temperature, very much	Rollers fairly dry; at higher temperatures bearings strongly smeared (grey-black oily mass).
No. 80	Little.	Rollers running in dry, micaceous grease.
No. 83	Little	Rollers running in plenty of soft grease.
No. 2	Grease creeps excessively.	Rollers slightly greased with fresh grease.
V.O.C. 1200	Very heavy creeping.	Bearings strongly smeared (greyish-black oily mass).
Calypsol	Medium.	Bearings hardly greased.
F-D-4	Very heavy loss.	Rollers run in plentiful black grease.

Condition of Grease

Grease	After 5 to 10 min.	Appearance, color, consistency, structure, oil separation after 24 hours. At Normal Temperatures 60 to 80° C.	At Increased Temperatures 100 to 140° C.
R-B-4	Grease very soft and very pale.	Grease hardly altered; only somewhat firmer	Plenty oil separation; grease soft, thin and black.
No. 80	Grease somewhat paler and somewhat softer.	Grease somewhat darkened on surface, dry, and micaceous; separated from edge. Pressed in grease also dry and micaceous.	Some oil separated; color blackish-green; grease looks dry, brittle, gelatin-like and firm.
No. 83	Grease somewhat paler and softer.	Grease firm and dry; separation from edge; by pressing grease nicely soft.	Some oil separation.

Condition of Grease—(Continued)

Grease	After 5 to 10 min.	Appearance, color, consistency, structure, oil separation after 24 hours. At Normal Temperatures 60 to 80° C.	At Increased Temperatures 100 to 140° C.
No. 2	Grease somewhat paler and softer.	Grease retains color; somewhat dryer on surface; below remained soft.	Small oil separation; somewhat firmer.
V.O.C. 1200	Grease very pale and very soft.	Whole packing pulled and most part exuded. Grease black, very soft and interspersed with small lumps. Heavy oil separation.	Grease somewhat firmer, sticky, rubber-like.
Calypsol	Grease somewhat paler and softer.	Grease dry, micaceous and crumbly, some lumps.	Color dark brown; very firm, rubber-like, crumbly.
F-D-4	Grease somewhat softer; color unchanged.		Slight oil separation; grease greyish-black and soft.

Bearing Temperatures, ° C.

Greases	Normally Filled Without Pressure from	to	With Pressure from	to	Full Bearing on Both Sides — Without Pressure from	to	With Pressure from	to
R-B-4	24	72.5	79	135.5	20	121	116	126.5
No. 60	24	81.5	75.5	84	22	129	111.5	123
No. 83	25	74	65	87	23	110	110	114
No. 2	24	75	67	88	24	81	74	95
V.O.C. 1200	24.5	105	99	109	19	96.5	90	99
Calypsol	24	102	65	109	21	118	115	123
F-D-4	14	126	118	125	18	129	125	125.5

Mean Watt Consumption Less Free Running Value of Testing Shaft

Greases	Normally filled	Fully filled
R-B-4	120	400
No. 60	175	430
No. 83	140	205
No. 2	110	155
V.O.C. 1200	250	260
Calypsol	200	440
F-D-4	400	400

U. S. Army Air Corps Specification No. 3560-B (May 1, 1933)

This specification covers four greases having melting points over 270° F. and is intended for the lubrication of bearings which operate at high temperatures, particularly electric equipment such as motors, generators, magnetos and the like. The general requirements are:

The grease shall be a smooth, homogeneous mixture, free from crusts, lumps, or separated material.

Water-Method 300.12.—The water content shall not exceed 1.0 per cent.

Insolubles-Method 541.2.—The insolubles shall not exceed 0.2 per cent.

The grease shall pass the corrosion and stability test described in Section VI.

DETAIL REQUIREMENTS

Penetration-Method 31.1.—The penetration of the worked grease shall be within the following limits:

Grade	Penetration
375	350–400
295	270–320
210	185–235
115	90–140

Mineral Oil Content-Method 541.2.—The mineral oil content shall be not less than as follows:

Grade	Mineral Oil Per Cent
375	84
295	77
210	69
115	60

Alkali and Acid Content-Method 541.2.—Free acid calculated as oleic acid shall not exceed 0.10 per cent for all grades. Free alkali calculated as sodium hydroxide shall not exceed 0.10 per cent for grades 375 and 295, and shall not exceed 0.20 per cent for grades 210 and 115.

Properties of Mineral Oil Content-Method 541.2.—The mineral oil content shall conform to the requirements specified in Table 1.

TABLE 1

Grade	Viscosity S.U.V. at 130 deg. F.	Pour point (A.S.T.M.) maximum deg. F.	Specific Gravity 60 deg. F. maximum	Color (A.S.T.M.) maximum
375	100 to 140	15	0.890	7
295	130 (min.)	..	....	7
210	150 (min.)	..	....	7
115	150 (min.)	..	....	7

Melting Point.—The melting point determined as hereinafter described shall not be below 270 deg. F. for Grade 375; 300 deg. F. for Grade 295; and 320 deg. F. for Grades 210 and 115.

METHOD OF INSPECTION AND TEST

The grease shall be subject to inspection by the Procuring Agency at such place or places as specified in the order. When inspection is conducted at the place of manufacture, the Inspector shall be furnished all necessary facilities to determine compliance with this specification.

Acceptance or approval of material in course of manufacture, shall in no case be construed as guarantee of the acceptance of the finished product.

Sampling.—Samples shall be taken according to the procedure described in Part 5, Bureau of Mines Technical Paper No. 323. Whenever practicable, the Inspector shall forward an original unopened container to the proper laboratory for test.

As far as applicable all tests will be made according to the latest issue of Bureau of Mines Technical Paper No. 323 to which the method numbers given for the various requirements refer.

Melting Point.—The materials required for this test are as follows:

An accurate thermometer of suitable range graduated to 1 deg. F. or 0.3 deg. C. or closer. An A.S.T.M. High Softening Point Asphalt thermometer is convenient.

A test tube about 7 inches long and $\frac{7}{8}$ inch inside diameter with flared top.

A 1-liter tall form beaker.

A steel tube $\frac{5}{16}$ inch inside diameter (0.185 to 0.190) and $\frac{3}{4}$ inch long.

Means of filling the steel tube with the grease to be tested, attaching the steel tube to the thermometer, a cork for holding the thermometer in the test tube, a metal plate and a suitable transparent, high-boiling point liquid for a heating bath.

The method of test shall be as follows: Fill the steel tube with the grease to be tested and attach it to the thermometer so that it is parallel to the stem and the mid-point of the tube corresponds to the mid-point of the thermometer bulb. Fit the thermometer vertically through a vented cork in the test tube so that the lower end of the steel tube is about 1½ inches from the bottom of the test tube. Suspend the test tube by the flared top through a hole in a metal plate which rests on and covers the beaker. The beaker shall be filled with liquid so that the surface is within about one inch of the top with the test tube in place. Apply heat so that after the initial time required to warm the bath and obtain a uniform rate, the temperature recorded on the thermometer rises at a rate of 4.5 deg. F. plus or minus 0.5 deg. F. per minute. Record the temperature at which the grease melts and a drop falls from the tube or the bulk of the grease slides out of the tube as the melting point.

In order to obtain concordant results the following precautions must be observed. The inside of the steel tube must be smoothly reamed and bright and it must be clean and dry before filling with the grease. Air pockets must not be present in the grease in the tube. The rate of temperature rise after the preliminary warming to not over 120 deg. F. must be closely observed.

Corrosion and Stability Test.—An A.S.T.M. lubricating oil pour test tube of a similar container shall be filled with the grease and a clean, bright, mechanically polished copper strip immersed therein. The tube shall then be placed in an air oven at 95 to 100 deg. C. (203 to 212 deg. F.) for 18 hours, after which it shall be allowed to cool to room temperature. The grease shall show no crusts, lumps, or separated material and shall be of typical greasy texture and consistency similar to its original condition. There shall be no green coloration of the grease nor the copper strip and no brown nor black stain on the copper.

PACKING AND MARKING

This grease shall be packed in commercial, friction top containers of the size specified in circular proposals.

The containers shall be marked as follows:

Grease, High-Melting Point
Grade No. .....
Specification No. 3560-B
Order No. .....
(Manufacturer's Name or Trade Mark)

Domestic Shipment.—The containers shall be packed in boxes acceptable to the freight agent in compliance with the current issue of the Consolidated Freight Classification.

Export Shipment.—When export shipment is specified, the containers shall be packed in accordance with Specification No. 2-22.

Shipments shall be marked in accordance with Specification No. 100-2.

NOTES

The type of grease described by this specification is sometimes commercially designated as "Fiber Grease."

This grease is intended for use in engine accessory equipment operating at high temperature.

In general the various grades should be used as follows: Grades No. 375 and 295 for ball bearing; Grades No. 293 and 210 for plain bearings; Grade No. 295 for gears; Grade No. 375 for saturating lubricating wastes or washers. Grade No. 115 is to be used when a high degree of lubrication is not required and it is essential that the grease stay in place for long periods of time. Grade No. 375 is also intended for use in equipment which must operate at low temperature.

ASBESTOS-SODIUM SOAP GREASES

Lubrication engineers have developed many varieties of sodium soap base greases to which are added graphite, talc, mica or asbestos fibers, in

any suitable proportion. In the following analysis of KSN-288A Grease 6.5 per cent of fibrous asbestos has been added to improve the wearing properties of the grease pack, and impart cooling and extreme pressure characteristics.

Physical Test:

Appearance	Fibrous
Color	Slate gray
Odor	Alkaline
Penetration, A.S.T.M. (unworked at 77° F.)	62
Dropping point ° C.	165

Composition, per cent by weight:

Soap	34.9
Oil	56.6
Moisture	1.5
Free alkali (as sodium hydroxide, NaOH)	0.2
Filler	6.5
Undetermined	0.3
Total	100.0

Oil Extracted:

Color, N.P.A.	4
Gravity, ° A.P.I.	21.7
S.U.V. at 100° F.	132
Flash ° F.	310
Fire ° F.	350

Ash, total per cent on original	16.1
Insoluble in acid (probably mostly asbestos)	6.2
Iron (as Fe_2O_3)	29.5
Sodium carbonate (as Na_2CO_3)	32.8
Calcium	Trace
Copper	Trace
Magnesium (as MgO)	31.3
Undetermined	0.2
Total	100.0

Fatty Acid from Soap:

Iodine value (Hanus)	44
Acid value (Mg. of KOH)	196
Melting point ° C.	51
Color	Light Brown

COLD MADE SODA BASE GREASES

Those tallow compounds and semi-fiber grease, railroad greases and soda base greases in general, made by the action of caustic soda on fats or fatty acids in the cold or substantially under the boiling temperature of water, are known as cold made soda base greases. Due to the fact that they are not boiled the fiber formation is not pronounced and they have a texture intermediate between cup greases and the usual fiber grease. They are water-soluble and should not be used in the presence of water.

Cold Made Ball Bearing Grease (Non-cooked)

If a hot 65 per cent solution of caustic soda is mixed with asphalt base cylinder stock and combined with melted tallow a smooth semi-fibrous grease is obtained.

Formula

		Per Cent by Weight
Mix No. 1.	Crude tallow, No. 2	15.00
	Asphalt base cylinder stock	25.00
Mix No. 2.	Caustic soda, solid	2.175
	Water	1.175
Mix No. 3.	Cylinder stock	20.00
Mix No. 4.	Cylinder stock	36.65

PROCEDURE

Mix No. 1 is first made by heating the fat and oil together until melted, and bringing the temperature to 100° to 110° F.

The caustic soda is dissolved in water, heating if required. This solution must be kept hot, as at temperatures below 150° F. it will be below its critical crystallization point. A temperature of 170° to 200° F. has been found satisfactory.

The hot caustic soda solution is then gradually stirred into the 20 per cent of the cylinder stock, the resulting temperature being about 120° F. Under these conditions upon reducing the temperature the caustic soda does not separate as might be expected but remains suspended in the oil.

The caustic mixture is then added to the fat mixture No. 1 while stirring, and the temperature raised gradually to 150° F., which requires only a short time. Local heating in the kettle jacket to above 212° F. will result in lumps and heavy fiber formation.

As the batch is being heated the remainder of the oil is run in and the temperature allowed to come to 150° to 170° F.

The batch is then stirred and cooled, by turning on the water in the jacket of the kettle, to a temperature of 110° to 120° F., when it may be drawn.

CHARACTERISTICS OF PRODUCT

It is of note that this grease increases in consistency for several weeks after manufacture so that shipments should not be made until the grease has been properly aged. A grease of this type has found considerable usage in the lubrication of enclosed ball and roller bearings, particularly those operating in a vertical position. The melting point of the product is about 250° F. and it can be made in any consistency by varying the amount of soap used.

DRAWING COMPOUNDS

A complete discussion of the problem of drawing wire and metal shapes is beyond the scope of this book. A few selected formulae for drawing lubricants containing sodium soap will be found below:

Lauster [76] has developed the following lubricant for drawing iron wire:

	Per Cent by Weight
Beef tallow	30
Sulfuric acid	6
Powdered sodium soap	5
Powdered talc	10
Sodium carbonate	5
Green soap	5
Rye flour	20
Water	19

[76] U. S. Patent 1,529,314.

Behr [77] has proposed a mixture of sodium soap, mineral lubricating oil, rosin oil, and lithopone as a wire drawing lubricant. Richards [78] has investigated the possibilities of using soluble oil emulsions as drawing lubricants. He has proposed a compound consisting of about 30 per cent of sodium soap of fat, having a titer test of at least 40, 20 per cent palm oil, 20 per cent of refined lubricating oil, having a minimum S.U.V. of 200/38° F., the balance being water. Aqueous dispersions appear to have been used extensively in wire drawing operations. In 1929, Esping [79] offered the formula containing a potassium soap:

	Parts
Potassium cottonseed oil soap	5
Suet	.5
Slippery elm bark	.5
Water	26

These materials were beaten together and incorporated with sufficient air to give them a foamy consistency.

Rutherford [80] proposed the following lubricant for wire drawing:

	Parts
Soluble alginate	1
Tallow	4
Sodium soap	2
Water	195

For similar service Stamberg [81] developed the following formula for the Standard Oil Company of Indiana:

	Per Cent
Green acid soap (sodium naphthenates)	6
Spindle oil	22
Wilkinite clay	25
Tallow	15
Mahogany soaps (sodium sulfonates)	10
Water	22

In addition to wire drawing this lubricant was proposed for die-pressing. A somewhat similar lubricant was suggested by Adams [82] consisting of lubricating oil, colloidal clay, water, and the alkali metal salts of oil soluble petroleum sulfonates. Wilkin's [83] wire drawing emulsion was prepared with water, animal fat, and the oil soluble sodium sulfonates (soaps) from petroleum and mineral oil.

For drawing electrical wire through conduits, and around sharp bends, Bedlovitz proposed the following potassium base lubricants:

	Per Cent by Weight
Water	88.83
Cottonseed oil	3.00
Potassium hydroxide	5.84
Soluble elm bark extract	2.33

[77] U. S. Patent 1,491,127 (April 22, 1924).
[78] U. S. Patent 1,732,065 (Oct. 15, 1929).
[79] U. S. Patent 1,732,882 (Oct. 22, 1929).
[80] U. S. Patent 1,944,273 (Jan. 22, 1934).
[81] U. S. Patent 1,781,607 (Nov. 11, 1930).
[82] U. S. Patent 1,871,939 (Aug. 16, 1932).
[83] U. S. Patent 1,907,920 (May 9, 1933).

Effect of drawing die, lubricant and drawing velocity on the power consumption in drawing of fine wires of steel, especially in multiple drawing machines, has been investigated by Anton Pomp and H. Heckel.[83a]

Tests with dies of Widia metal (tungsten carbide) showed that consumption was highest with rape-oil lubrication. The most favorable angle for the die was 6 to 9 degrees for soap and 9 to 15 degrees for rape oil and drawing liquid. The influence of both lubricant and die angle becomes less important with increasing section of the wire and the efficiency improves. With diamond dies, drawing liquid proved to be the best (compn. 100 liters water, 6 kg. rye flour, 2.5 kg. beef tallow, 2.5 kg. soft soap). Compared with Widia dies, a reduction up to 36 per cent in power consumption was found, also with rape-oil lubrication; soap required higher power. The most favorable angle is smaller than for Widia dies. Multiple drawing arrangement resulted in a slight reduction of the drawing effort due to the back pull in the wire inherent in this drawing process.

ENGINE GREASES

Engine greases are seldom employed for lubrication of modern machinery. Most frequently they were mixtures of potash soaps with waxes and oils, the process being carried out at normal temperatures (see Cold Sett Sodium Base Greases). They were intended for use in grease cups and for packing bearings on steam engines and the like. In view of their high free fat contents they often had a high oiliness value.

Engine Tallow Grease Formula

The formula for this product is:

	Pounds
Tallow	56
600 Pennsylvania S.R. stock	50
Yellow ceresine wax	5.25
Sodium hydroxide (dissolved in a minimum quantity of water)	.75

Bridgit[84] Grease

This was a combination of mineral oil, beeswax, tallow and caustic soda solution. Potassium hydroxide was also employed.

Adam Cook's Engine Grease[85]

The formula for this product is:

	Pounds
Tallow	100
Lard	20
Lubricating oil	50
15° Bé. caustic soda solution	20
Gelatin	2
Rosin	1
Gum tragacanth	.25
Gum arabic	.25
Curriers grease (contains tannin)	10

[83a] *Mitt. Kaiser-Wilhelm Inst. Eisenforsch. Dusseldorf*, **17**, 107-26 (1935).

[84] U. S. Patent 126,282 (Apr. 30, 1872).

[85] U. S. Patent 149,996 (Apr. 21, 1874).

The fats were melted together with the rosin, the gelatin and gums dissolved in water together with the lye, and added to the warm fats. After stirring the mass was left to stand and form a consistent grease.

Newton's [86] grease was made by heating a mixture of fat and wax with caustic soda or potash solutions. French's [87] engine bearing grease was more complicated and consisted of mineral oil, tallow, caustic soda, spermaceti, bay-wax or beeswax, chalk and graphite. McLay's [88] grease was composed of mineral oil and various waxes, in which sodium soap was dispersed and water added. Purvis [89] proposed the following formula:

	Parts
Japan tallow	200
Tallow	300
Lard	108
Castor oil	108
Sodium olive oil soap	200
Calcium carbonate	10
Sodium carbonate	10

The fats, soap and oil were heated to above 210° F. and the fillers added. Hummy [90] developed the following formula, which includes sodium resinates: spermaceti, paraffin wax, crude petroleum, pine pitch, sawdust, caustic soda, and 10 per cent of water. In the Donati [91] process the following materials were heated together at 248° F.:

	Parts
Lard	1000
Yellow ceresin wax	50
Sodium carbonate	100
Martius yellow	5
Graphite	2

Thompson [92] developed a water soluble compound consisting of oxidized mineral oil, ceresin and paraffine waxes, 1 to 2 per cent of caustic soda, sodium soap, water and gelatin.

EXTREME PRESSURE TYPE SODIUM SOAP GREASES

Many of the early sodium soap base greases contained mild abrasives, which no doubt acted as extreme pressure agents, and tended to smooth bearings with rough surfaces and caused them to operate at lower temperatures. Many of the substances referred to in the following paragraphs have been further investigated in recent times, particularly with a view to improving them as film strength agents.

[86] British Patent 1,144 (May 6, 1861).
[87] British Patent 2,249 (June 29, 1874).
[88] British Patent 1,501 (Jan. 28, 1889).
[89] British Patent 13,936 (Sept. 4, 1890).
[90] British Patent 5,791 (Mar. 16, 1896).
[91] British Patent 19,291 (Sept. 10, 1898).
[92] British Patent 13,437 (June 16, 1903).

Bays [93] lubricant for thrust blocks consisted of:

Hard sodium soap	0.5 pounds
Sodium carbonate	.25 "
Tallow	6 ounces
Lubricating oil	1 pint
Sulfur	1 ounce

Anderson's [94] axle grease doubtlessly had cooling properties and extreme pressure properties. It was manufactured in accordance with the formula:

Hard sodium soap	4.5 pounds
Water	1.5 gallons
Sulfur	.5 pounds
Fine coal dust	3.0 "

The mixture was boiled together and poured into molds to cool. Mendosa's [95] lubricant was prepared with tallow, caustic soda, water, borax, clay, sulfur, graphite, and sodium chloride. Keirby [96] invented a lubricant for impregnating hemp stuffing box packing, consisting of mineral oil, tallow, lead ore (lead sulfide), sodium soap and sodium carbonate.

A grease suitable for steel mill rolling necks was proposed by Fitch [97] in 1880, which contained sodium soap, ammonium chloride, ammonium carbonate, red lead oxide, petroleum and water. A grease formulated by Norris [98] consisted of mica, charcoal, ammonium chloride, lubricating oil, asphaltic residue, beef tallow, strong caustic soda and water. Chard's [99] grease was composed of talc, tallow, animal oil, alkali, rubber and asbestos. Wass [100] suggested as a lubricant, a mixture of the following: asphalt, fatty oil, glycerin, lead oxide (PbO), finely divided lead and zinc, and sufficient caustic soda to saponify the fatty oil. Efros [101] considered that a complex mixture of magnesium silicate, sodium phosphate and creosote, together with glucose, flour, sodium soap, tallow and mineral oil was suitable for bearing lubrication. Stewart's [102] grease was made with the sodium soap of tallow, graphite, lead carbonate, camphor, rosin, and sodium chloride. In 1893, Riedel [103] heated fatty oils and fatty acids of the unsaturated series (oleic and linoleic acids), or abietic acid, with sulfur at a temperature of 248 to 320° F., at which temperature the sulfur was combined with the fatty substances. These "thio-fats" or "thio-acids" were then saponified with caustic soda and employed as lubricants.

Callahan's [104] grease contained a definite abradant, and the following materials:

[93] British Patent 322 (1914)
[94] U. S. Patent 184,492 (May 20, 1876).
[95] U. S. Patent 170,581.
[96] British Patent 19 (Jan. 4, 1865).
[97] U. S. 236,150 (Aug. 7, 1880).
[98] U. S. Patent 267,921 (Nov. 21, 1882).
[99] U. S. Patent 344,079 (1886).
[100] British Patent 3,832 (Mar. 14, 1887).
[101] U. S. Patent 557,568.
[102] U. S. Patent 594,576.
[103] British Patent 10,707 (May 31, 1893).
[104] U. S. Patent 1,003,983 (Mar., 1908).

	Pounds
Fine emery dust	6
Graphite	24
Sodium soap	35
Tallow	35

GASOLINE INSOLUBLE GREASES (PUMP GREASES)

Arveson [105] has developed the formula for a lubricating grease which is claimed to adequately lubricate pumps handling gasoline, or petroleum solvents. This formula is:

	Parts by Weight
Free fatty acids	2.5
Sodium fatty acid soap	17.5
Glycerin	37.0
Mineral lubricating oil	35.0

Kaufman [106] has also investigated the possibilities for preparing lubricating greases for application in the presence of hydrocarbon solvents, and certain chemicals. He found that mixtures of castor oil soap and castor oil were suitable for many applications, particularly to lubricate and seal the bearings of pumps, which may be used for handling sulfur dioxide or petroleum solvents. In such cases, the lubricant must serve as a seal to prevent loss of the volatile solvents. Normal cup greases have been employed but have not been satisfactory, as the high mineral oil content is soon washed away. Another plan is to circulate a small portion of the fluid handled by the pump, through the packing glands surrounding the bearing surfaces. While this has been found fairly satisfactory for fluids which are not very volatile, it cannot be adopted for materials of high vapor pressure. Castor oil is essentially insoluble in mineral oils and is also little affected by sulfur dioxide. The castor oil used in such greases is essentially tri-ricinolein, and the presence of small amounts of tri-stearin, stearic acid, or ricinoleic acid has not been found objectionable.

In making Kaufman's grease the castor oil is charged to the grease kettle and sufficient sodium hydroxide solution to saponify approximately one-half of it is run in. The caustic soda solution should contain about 49 per cent of NaOH in order to keep the amount of water to be evaporated at a minimum. The mixture is agitated with paddles and heated to a temperature of about 400° F. The melted product is drawn off in pans to cool and solidify. Made with the foregoing quantities the grease should have a melting point of 292° F. and an A.S.T.M. unworked penetration of approximately 26. The product made in this way is, of course, substantially anhydrous. Other soaps of castor oil may be employed, such as aluminum or calcium ricinoleate.

Nordstrom's [107] lubricating grease, which was insoluble in gasoline

[105] U. S. Patent 1,900,759 (Mar. 7, 1933).
[106] U. S. Patent 1,952,012 (Mar. 20, 1934).
[107] U. S. Patent 1,514,095.

and intended for use in plug valves handling petroleum solvents, was manufactured on the following formula:

	Per Cent by Weight
Hard sodium soap	32
Soft potassium soap	52
Powdered graphite	4
Glycerin	12

Various mixtures of glycerin and graphite have been tried for the lubrication of centrifugal pumps handling gasoline but the results have not been entirely satisfactory. A sodium base grease such as normally used for lubrication of paper mill roll bearing lubrication, while better than normal cup greases, is rapidly consumed and does not seal adequately Petrolatum is useless. With mixtures of castor oil and graphite, the castor oil appears to be washed away leaving much of the relatively dry graphite behind. Greases prepared with very viscous steam refined cylinder oils (200 to 400 S. U. V. at 210° F.), and from 5 to 40 per cent of sodium or potassium oleates or stearates, have been found to be promising (1), where the bearing speeds are not so high as to prevent the use of a lubricant having a high viscosity, even when diluted with solvent being pumped; and (2), where the dissolution of such material in the solvent is not objectionable. The addition of asphalts, pitches, and gums to such greases seems to have a beneficial effect. Greases made with from 8 to 50 per cent by weight of aluminum stearate and engine oils, or steam refined cylinder stocks, have been found useful for gasoline pump bearing lubrication.

Beyer [108] has developed a process of manufacturing gasoline insoluble greases in which castor oil is added to a boiling solution of sodium hydroxide, or potassium hydroxide, heating being continued until a temperature of 150° F. is attained. The grease is obtained by filtering while the reaction mixture is allowed to cool.

Gasoline Insoluble Carburetor Valve Grease (U. S. Air Corps)

The following specifications cover a grease made with sodium soap and castor, the product containing no mineral oil.

Air Corps Specification, No. 3564, February 17, 1933.

GREASE, LUBRICATING, GASOLINE RESISTANT

GENERAL SPECIFICATIONS:

The current issue of the following specifications in effect on date of issuance of proposals, forms a part of this specification:

2-8 Castor Oil for Aircraft Engine Lubrication
2-22 Boxes for Oils and Greases for Export Shipment
100-2 Standard Specifications for Marking Shipments

The current issue of the Bureau of Mines Technical Paper No. 323 "Method of Testing Lubricants and Liquid Fuels" also forms a part of this specification.

[108] French Patent 799,718 (June 18, 1936).

GRADES:

This specification covers one grade of grease for the lubrication of carburetor mixture control valve stems of aircraft engines and shall be suitable therefor.

MATERIAL AND WORKMANSHIP:

This grease shall be composed of a mixture of raw castor oil conforming to Specification 2-8 and a commercially pure sodium soap of animal fat.

The grease shall contain no fillers or other substances not naturally present in raw castor oil or sodium soap.

The workmanship shall be such that the finished product meets all requirements hereinafter specified.

GENERAL REQUIREMENTS:

There are no general requirements applicable to this specification.

DETAIL REQUIREMENTS:

The water content shall not exceed 0.5 per cent.

The A.S.T.M. penetration shall not be less than 190 nor more than 250 worked.

Material insoluble in hot, 95 per cent ethyl alcohol shall not exceed 0.5 per cent.

The grease shall meet the requirements of the corrosion and stability test hereinafter described.

Deliveries containing less than 15 per cent or more than 35 per cent of soap may be subjected to special tests to determine their suitability for the intended use.

METHOD OF INSPECTION AND TESTS:

The grease shall be subject to inspection by authorized Government Inspectors who shall be given all necessary facilities to determine compliance with this specification.

Acceptance or approval of material in course of manufacture, shall in no case be construed as a guarantee of the acceptance of the finished product.

Samples shall be taken according to the procedure described in Bureau of Mines Technical Paper No. 323, Part 3.

As far as applicable, all tests shall be conducted according to the procedures described in Bureau of Mines Technical Paper No. 323, Part 2. The method numbers applicable are as follows:

Water Content—Method 300.12.
Penetration—Method 31.1.

Corrosion and Stability Test.—An A.S.T.M. lubricating oil pour test tube or similar container shall be filled with the grease and a clean, bright, mechanically polished copper strip immersed therein. The tube shall then be placed in an air oven at 95° C. to 100° C. (203° F. to 212° F.) for five hours, after which it shall be removed and allowed to cool to room temperature. After cooling the grease shall show no crusts, lumps, or separated material and shall be of typical greasy texture and consistency similar to its original condition. There shall be no green coloring of the grease or the copper strip and no brown or black stain on the copper.

Insoluble in Alcohol.—Treat an accurately weighed sample of approximately 15 grams with about 200 ml. of hot neutralized 95 per cent ethyl alcohol in a 400 ml. beaker, stirring until the grease is completely disintegrated. Filter through a dried and weighed Gooch crucible, wash the beaker and crucible thoroughly with hot alcohol, dry and weigh. Calculate increase in weight to per cent insoluble. Reserve the filtrate.

Castor Oil.—The saponification number on an accurately weighed sample of about 4 grams as described in standard chemical texts or Technical Paper No. 323. Calculate the per cent castor oil, using 183 as the saponification number, but first correcting the caustic used for any free acid or free alkali found in the filtrate from insoluble in alcohol determination.

Soap Content.—Ash an accurately weighed sample of approximately eight grams in a well glazed porcelain crucible, first burning off the oily constituents slowly and finally igniting at dull red heat until practically all of the carbonaceous material is

removed. Treat the crucible in a 400 ml. beaker with 25 ml. of 0.5 N HCl diluted with sufficient water to cover the crucible. Stir until the ash is dissolved or thoroughly disintegrated. Titrate the excess acid with O.5 N caustic, using methyl orange as an indicator, and calculate the acid consumed to soap, first correcting for any free alkali found in the filtrate from alcohol insoluble. One ml. 0.5 N HCl equals 0.153 grams soap.

Free Acid or Free Alkali.—Titrate the filtrate from determination of alcohol insoluble with 0.5 N acid or caustic as required, using phenolphthalein as an indicator. Use any free acid or alkali found to correct the saponification and any free alkali to correct the soap titration as directed above.

PACKING AND MARKING:

Gasoline-resistant grease shall be furnished in one-half pound collapsible tubes.

Domestic Shipment.—Domestic shipments shall be packed in boxes acceptable to the freight agent in compliance with the current issue of the Consolidated Freight Classification.

Export Shipment.—When export shipment is specified, the tubes containing the grease shall be packed in boxes in accordance with Specification No. 2-22.

Shipments shall be marked in accordance with Specification No. 100-2.

NOTES:

Gasoline-resistant grease is intended for use in the carburetor mixture control valve stems of aircraft engines.

The method numbers referred to in Section VI of this specification refer to the method numbers as given in the Bureau of Mines Technical Paper No. 323-B. The latest method should be used. Method No. 300.12 is a revision of 300.11. Copies of this paper may be obtained from the Superintendent of Public Documents, Government Printing Office, Washington, D. C.

Notice.—When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

PATENTS CONCERNING SODA SOAP GREASES CONTAINING GRAPHITE

Numerous patents have been granted covering mixtures of soda soap greases with graphite (black lead, or plumbago). In 1872, French[109] was confronted with the problem of maintaining graphite in a state of suspension in mineral oil. His plan for accomplishing this purpose was to employ a mixture of beeswax and soda-tallow soap to solidify the oil and prevent settling of the graphite. An example of his process is shown in the following formula:

Petroleum lubricating oil	1 gallon
Graphite	14 ounces
Beeswax	5 "
Mutton tallow	1.5 "
Caustic soda	0.5 ounce

[109] U. S. Patent 126,282 (Apr. 30, 1872).

Roat's [110] formula was more complicated and consisted of:

Tallow (lard or other grease)	48 pounds
Beeswax	3 ounces
Water	1 gallon
Ammonium chloride	1 pound
Borax	1 ounce
Sodium tartrate	1 "
Ammonium carbonate	1 "
Graphite	0.5 pound

McMillan [111] considered that beeswax was too expensive to use for this purpose, so in 1885, he developed the following formula, utilizing rosin oil as a substitute for beeswax:

Petroleum lubricating oil	1 gallon
Rosin oil	6 ounces
Beef tallow	4 "
Graphite	4 "
Sodium bicarbonate	0.5 ounce

Munger [112] developed the following formula for a semi-fluid lubricant:

Petroleum oil	1 gallon
Graphite	6 ounces
Japan wax	3 "
Tallow	4 "
Caustic soda	1 ounce

These materials were heated and stirred together at a temperature of about 180° F. In another patent Munger [113] proposed a similar formula in which the Japan wax and tallow in the foregoing lubricant was replaced with 4 ounces of palm oil. In order to overcome the obstacles incident to suspending graphite in mineral oil, Johnson [114] proposed the following formula in 1878:

Petroleum oil	1 gallon
Graphite	8 ounces
African palm wax	8 "
Caustic soda (2 ounces sodium bicarbonate)	1 ounce

Various dispersions of graphite in soap, with no mineral oil present, have been patented. According to one inventor [115] a mixture of alcohol, Castile soap, and graphite was recommended. Comstock's [116] grease consisted of 8 parts of graphite dispersed in one part of soft sodium soap. Traun [117] dispersed graphite in water, using soaps, tannin, and phenolsulphonic acids as dispersion agents.

A very early patent was granted to Leroy [118] in 1861. He kneaded

[110] U. S. Patent 136,868 (Mar. 18, 1873).
[111] U. S. Patent 311,347 (Jan. 27, 1885).
[112] U. S. Patent 198,664 (Sept. 15, 1877).
[113] U. S. Patent 205,124 (Nov. 7, 1877).
[114] U. S. Patent 199,913 (Jan. 15, 1878).
[115] U. S. Patent 519,096.
[116] U. S. Patent 804,455 (Feb. 28, 1905).
[117] British Patent 155,836 (Feb. 8, 1919).
[118] British Patent 58 (Jan. 9, 1861).

together tallow, lubricating oil, and a solution of sodium and potassium hydroxide. Graphite was then added to the grease thus produced. Williamson [119] melted together tallow, mineral oil, caustic soda, talc, and graphite in the preparation of a lubricant for machinery. The distinguishing characteristic of Barbason's [120] grease was the utilization of magnesium carbonate. Other ingredients of his product were crude petroleum, vegetable or animal oils, graphite, and sodium bicarbonate. Berry's [121] grease may be described as follows: "A lubricant, particularly adapted for the use in marine engines and other condensing-engines, is made from finely-powdered graphite, steatite, etc., and a substance wholly soluble in water, such as a neutral or alkaline soap. Curd soap, or marine soap made from coconut oil, with the addition of soda, may be used.

Several patents involve the use of sodium and ammonium soaps, mineral oil, and graphite. For instance in 1909, Watkins [122] obtained a patent for a mixture of soap, graphite, mineral oil, tallow, sodium chloride, and water. His formula was:

Castile soap	1.5 pounds
Flake graphite	.25 "
Ammonia	1.0 gill
Heavy lubricating oil	2.0 quarts
Tallow	0.5 pounds
Sodium chloride	1.0 pound
Water	3 pints

Another invention [123] included the use of soap, flake graphite, ammonia, mineral oil, tallow, and water, but no salt.

SODIUM SOAP BASE LIQUID GREASES

As in the case of calcium soap base liquid greases, many soda soap liquid greases have been found in service. It has now become fairly commonplace to lubricate automobile internal combustion engines with lubricating oils containing small amounts of dissolved soaps to increase their oiliness and film strength, to decrease their pour points and reduce their tendency to stick piston rings and form objectionable carbon deposits. While such oils will not normally pass rigid emulsion test requirements this fact appears to be overlooked in view of other advantages their presence imparts. Liquid greases have also found extensive use in a wide variety of other applications some of which will be mentioned in the following patent review.

Jones and Roberts,[124] in 1887, made a fluid lubricant based on the following formula:

[119] U. S. Patent 278,067 (May 22, 1883).
[120] U. S. Patent 359,418 (1887).
[121] British Patent 9,796 (May 11, 1901).
[122] U. S. Patent 905,646 (Mar. 9, 1909).
[123] U. S. Patent 939,646.
[124] British Patent 14,129 (Oct. 18, 1887).

	Parts
Sodium carbonate	10–20
Sodium soap	10–20
Water	40–60
Animal, vegetable, or mineral oil	20–30

Burch [125] assigned to the Crew-Levick Company a patent dealing with the process of making an emulsified lubricant by mixing oleic acid with borax solution, and then adding mineral oil, and an alcoholic solution of caustic soda, and agitating the mixture until a clear bright fluid oil is obtained. This product is claimed to emulsify well with water. de Cew [126] atomized fatty oils and mineral oils in warm alkaline solution and discharged the mixture into water at lower temperatures in order to produce an emulsion suitable for lubrication purposes.

Gruse [127] developed a fluid sodium soap base grease in which mineral oil and a minimal amount of sodium soap dissolved in a miscible thinner of lower viscosity, were combined.

An anhydrous soda soap liquid grease has been developed by Edmiston [128] which is made by blending mineral oil and fatty acids, and then mixing in caustic soda dissolved in glycerin. Kaufman [129] has proposed the process for making a semi-fluid grease heating mineral oil and fat together and then adding caustic soda solution. The product is then cooled and stirred to obtain a lubricant having a viscosity about 20 per cent greater than the mineral oil content.

Improved crankcase oils have been suggested in the following patents. In 1928, Penniman [130] dissolved sodium and metallic soaps together with polar bodies, such as the higher fatty acids, in mineral oil containing a small amount of an antioxidant. Yacco's [131] motor oil consisted of mineral oil mixed with a solution of a metallic soap, such as copper oleate, in a vegetable oil, such as olive oil, peanut or sesame oil. Other soaps which his patent application covered were the oleates of sodium, aluminum, lead or zinc, and finely divided metals. Becker [132] has obtained three patents covering the use of 0.15 to 0.50 per cent of sodium oleate in motor oil. In one example, he heats ½ per cent of alkali metal fatty acid soap with mineral oil at temperatures above 350° F., but below the boiling point of the lubricating oil. Rudigier [133] dissolved sufficient sodium naphthenate in mineral oil, having a viscosity of only 65 to 200 S.U.V. at 38° F., to produce a finished motor oil having a viscosity of 135 to 140 S. U. V. at 100° F. and a maximum pour point of 37° F.

[125] U. S. Patent 1,246,869.
[126] U. S. Patent 1,317,617.
[127] U. S. Patent 1,808,853.
[128] U. S. Patent 1,810,555 (June 16, 1931).
[129] U. S. Patent 2,002,819 (May 28, 1935).
[130] British Patent 317,406 (Aug. 15, 1928).
[131] British Patent 371,643 (July 18, 1930).
[132] British Patent 260,602 (Oct. 29, 1925).
[133] U. S. Patent 1,628,646 (May 17, 1927).
[133] U. S. Patent 1,628,647 (May 17, 1927).
[133] U. S. Patent 1,750,134.

Becker and Sloane [134] worked out the process in which not more than 5 per cent of sodium soap, dissolved in a volatile petroleum solvent, was blended in a still with mineral lubricating oil. The solvent was distilled off and the liquid grease passed through cooling coils to rapidly cool the material through the temperature range in which gell formation would otherwise take place. The soap was first compounded in the same still which was provided with a propeller type agitator.

Story [135] has observed that by adding from 0.05 to 0.30 per cent of sodium soap to motor oils containing wax, and heating to 300 to 350° F., the pour point of the treated material was appreciably reduced.

Becker [136] has discovered that small amounts of alkali earth metal salts of oleic, stearic and palmitic acids, dissolved in lubricating oils improve their lubricating value and tend to reduce friction. He dissolved one-eighth of one per cent of a mixture of sodium oleate and stearate in lubricating oil having an S.U.V. of 285 at 100° F., and that when this liquid grease was fed to the bearing of an Olsen friction testing machine, at the rate of 40 drops per minute, that the friction was 5 to 6 pounds when the bearing pressures were varied from 25 to 175 pounds per square inch, the speeds being varied from 300 to 710 R.P.M. Bearing temperatures were 86 to 119° F. Under identical conditions, but when feeding the oil containing no soap, the friction was 8 to 11.5 pounds, and the temperatures varied from 88 to 108° F. The addition of the small amount of soap increased the viscosity of oil from 285 to 340 S.U.V. seconds at 100° F. Temperatures of 450° F. were employed for dissolving the soap.

Brick Oil

The investigations of Sandwith and Rayner [137] resulted in the formulation of two lubricants suitable for use in mould lubricating in brick manufacture:

Potassium oleate	20 pounds
Water	30 gallons
Shale or mineral oil	10–20 "

Also:

Potassium oleate	15 pounds
Sodium oleate and stearate	5 "
Water	30 gallons
Lubricating oil	10–20 "

Cutting Oil

Wilson [138] has proposed the use of a mixture of sodium sulpho-carbolate, together with 20 per cent of phenol and 4 per cent of cresol as a cutting lubricant.

[134] U. S. Patent 1,943,806 (Jan. 16, 1934).
[135] U. S. Patent 1,896,343 (Feb. 7, 1933).
[136] U. S. Patent 1.628,646 (May 17, 1927).
[137] British Patent 16,125 (July 21, 1903).
[138] British Patent 24,433 (Nov. 5, 1907).

Anti-Corrosive Oil (Gun Oil)

Klever [139] has developed a solvent for removing the residue of nitric powders from fire arms, which was made by mixing mineral oil with sodium oleate and an alcohol such as glycerin.

Gas Holder Seal

Wagner [140] has suggested the use of sodium soap dissolved in mineral oil as a means of sealing large gas holders.

Airtool Liquid Grease

The analysis of a soda base Liquid Grease is given below. This product is said to give satisfactory lubrication for air tools operating under conditions where the air contains much water.

Analysis

	Per Cent by Weight
Combined fat	4.05
Free fatty acids	.19
Free fat	.40
Combined caustic soda	.60
Water	Trace
Mineral oil	94.56
Gravity	20° Bé.
Vis. at 100° F.	207
Melting point of grease	70° F.

PAPER MILL GREASES

Formulas and processes for the manufacture of mixed soda-lime base roll grease for use on the dryer rolls of paper machines, calendar roll bearings on ironing machines, etc., are given in the next chapter. The following pertains to soda base roll grease only. These greases may be considered as heavy fiber greases which have been dehydrated completely by heating to 450° to 500° F.

Soda Base Roll Grease, Western Oil

Formula

	Per Cent by Weight
Crude tallow, No. 1	24.91
Stearic acid	2.76
Caustic soda, solid	4.40
Western pale oil, 180 vis./100° F.	67.93

PROCEDURE

The tallow and about an equal quantity of the oil are charged in a fire-heated kettle and melted together; the caustic soda solution as 40° Bé. lye is then run in.

After saponification is completed the balance of the oil is run in, agitation being carried on at all times.

[139] U. S. Patent 919,884 (Dec. 22, 1905); see also British Patent 27,254 (Jan. 4, 1905).
[140] British Patent 303,316 (Apr. 13, 1928).

The temperature is then raised to 495° to 500° F. which will require several hours.

At this temperature the molten grease is run into iron pans of such size that after cooling blocks of grease that will fit the grease caps on dryer roll bearings may be cut from them without waste.

Dark Soda Base Roll Grease

Formula

	Per Cent by Weight
Lump rosin, grade F.	7.5
Caustic soda, solid	2.1
Lard oil, No. 1	7.5
Cylinder stock, asphalt base, steam refined	82.9

The method of production is the same as above except that the maximum temperature reached is 400° F. The melting point of this grease is 340° F., penetration 27.

Dark Soda Base Roll Grease

Formula

	Per Cent by Weight
Lump rosin, grade F.	7.5
Stearic acid	7.5
Caustic soda, solid	2.1
Asphalt base cylinder stock	82.9

Process same as above. The melting point of this product is 365° F.; the penetration is 51.

Soda-Lime Base Roll Grease, Steam and Fire Cooked

The following grease making use of Western oil is known to give satisfactory results.

Formula

	Pounds
Tallow No. 1	720
Stearic acid	80
Caustic soda, 40° Bé. solution	387
Powdered hydrated lime	4
Western pale oil, 180 vis./100° F.	255 gallons

PROCEDURE.

Charge 25 gallons of slop grease from previous batches.

Charge the tallow, stearic acid, lime and caustic soda in a steam-heated kettle Start to heat gradually with agitation on.

Take batch to a temperature of 290° F. and allow to cool over night, or draw at once into barrels for storage.

The grease is finished in a fire-heated kettle which may be of small capacity. Charge 600 pounds of the above product in a fire-heated kettle and add 5 pounds of 40° lye and three pounds of hydrated lime.

Heat to 495° to 500° F. in 4½ to 5 hours.

Draw the molten grease into small pans for cooling.

CHARACTERISTIC OF PRODUCT

The melting point of this product is 380° F.

Soda-Lime Base Roll Grease

Formula

	Per Cent by Weight
Crude tallow, No. 1	24.00
Stearic acid	3.00
Caustic soda, solid	4.05
Hydrated lime	.50
Pennsylvania base pale oil, 100 vis./100° F.	10.00
Caustic soda, solid	.62
Asphalt base pale oil, 100 vis./100° F.	57.83

PROCEDURE.

The fats and 10 per cent of the oil are melted in a fire-heated kettle and the first caustic added as 40° Bé. lye.

These materials are sponged together and heated to about 350° F.

After cooling to about 280° F. the second caustic is added and the remainder of the oil run in while heating and stirring.

After all of the oil is in heat to 500° F. and maintain at this temperature for 10 minutes, then draw into cooling pans.

Analysis of a Commercial Soda-Lime Roll Grease

Formula

	Per Cent by Weight
100 vis. Pale oil	71.00
Combined fat	24.62
Free fat	1.78
Combined NaOH	3.55
Combined CaO	.499
Glycerine	.421
Free fatty acids	.98
A.S.T.M. penetration	47.5
Melting point	375° F.

Dark Soda Base Roll Grease

Formula

	Per Cent by Weight
Lump rosin, grade F	7.5
Stearic acid	7.5
Caustic soda, solid	2.6
Asphalt base cylinder stock	82.4

The procedure for this product is the same as for No. 2. The finished grease has a melting point of 375° F. and a penetration of 50.

Heavy Dark Soda Base Roll Grease

	Per Cent by Weight
Lump rosin, grade F	7.5
Caustic soda, solid	2.6
Lard oil, No. 1	7.5
Asphalt base cylinder stock	82.4

The same process may be used for this product as given on page 434; its melting point is 380° F. and the penetration is 50.

Analyses of Soda Base Roll Greases

In Table 3 are presented the analyses of five commercial soda base roll greases.

TABLE 3.—Analyses of Commercial Soda Base Roll Greases.
Per cent by weight

	Roll Grease	Block Grease	Block Grease	Block Grease	Roll Grease
Mineral oil	83.77	29.33	55.21	82.39	65.81
Free fat	2.68	5.86	8.91	1.40	...
Combined fat	11.62	55.58	30.83	12.74	29.75
Free fatty acids	.24	1.29	.65	...	...
Combined NaOH	1.69	7.94	4.40	1.83	4.44
Water	Trace	...	...	...	...
Melting point, ° F.	375	365	336	303	388
A.S.T.M. penetration	35	...	...	...	...
Type of oil used	Petrolatum	Cylinder stock	Cylinder stock	Cylinder stock	Pale oil

Arveson's Mill Grease [141]

The inventor of this lubricant has intended it for use in cement mills, and paper mills where heavy machinery may be equipped with open bearings for application of bricks of lubricating grease. Its formula is:

	Parts
Rosin oil (This may be prepared by heating 30 parts of K grade rosin with 70 parts of spindle oil, for 42 hours at 500° F.)	26.2
Tallow	12.2
500 S.U.V./210° F. mineral oil	59.0
Sodium hydroxide	3.2

All of the tallow is mixed with one half of the oil and all of the sodium hydroxide (which may be dissolved in a small amount of water) in a conventional kettle provided with heating and agitating means. This mixture is heated, with agitation, to a temperature of about 375° to 450° F., preferably about 400° F., until the tallow has been completely saponified. The rosin oil is next added, the heating and agitation being continued, and the temperature is again raised to about 400° F., to effect a reaction between the excess alkali and the rosin acids. Finally, with continued heating and agitation, the other half of the oil is added and the temperature is again brought to 400° F. The mixture should be neutral or slightly alkaline at this point and if the reaction is acid a calculated amount of sodium hydroxide should be added. The grease is then ready to be poured into moulds. The mill grease prepared by this process is claimed by its inventor to be different from, and superior to, greases formed by the normal processes, which consist of saponifying a mixture of fatty acid and rosin. Instead of obtaining a grainy, soft, low melting composition, a smooth, clear, hard, elastic, high melting point mill grease is obtained that has good wear resistance.

[141] U. S. Patent 1,882,721 (Oct. 18, 1932).

Tests with different rosin-tallow mixtures disclose a peak in the consistency curve. If rosin oil is used, the inventor claims that this peak occurs when the amount is slightly more than twice the amount of tallow (rosin acid ratio to tallow is slightly greater than 1 : 1). If lump rosin is used, the consistency peak occurs with a rosin : tallow ratio of 1 : 1. The grease at peak consistency is hard and smooth and has a higher melting point than the other mixtures. At peak consistencies the consumption of this mill grease reaches a minimum, as indicated by experiments at 250 to 260° F. The melting point of these greases decreases from 370° F., at 20 per cent rosin, to 360° F., at 60 per cent rosin. Greases made with rosin show substantially the same characteristics as those made with rosin oil; the latter, however, at peak consistency, have a lower consumption, at 250 to 260° F., and a better top bearing temperature (the temperature at which the last trace of grease leaves the bearing cup). The top bearing temperature for a 50 per cent rosin grease was 330° F. while that of the corresponding rosin oil grease was about 360° F. (within 5° F. of its melting point). It is considered important that the tallow be completely saponified prior to the addition of the rosin, the rosin being used to take up the excess sodium hydroxide and consequently regulates the free alkali content.

McKee's High Melting Point Grease [142]

McKee has made a study of the effect of high cooking temperatures on the melting point of the greases produced. He preferred to use hard cocoanut oil soap or Castile soap chips, free from the soaps of hydrogenated fats which apparently gave lower melting points. His greases were made with from 5 to 50 per cent of the sodium soap and 95 to 50 per cent of paraffine base lubricating oil, treating the grease at temperatures of about 450° F. The soap and mineral oil are mixed in a grease kettle and heated to temperatures of 450 to 490° F., the optimum treating temperature depending on the soap content, the higher the soap content, the higher the treating temperaure. The maximum temperatures were considered to be 500 to 600° F. McKee considered that normal dehydrated greases had melting points of the order of 300° F., whereas it is well known that many paper mill greases have been marketed for many years having melting points of 375 to 410° F., and being processed at temperatures of 400 to 500° F. McKee observed, however, that for greases having a relatively low soap content, the melting point was raised by heating the mixture of soap and oil to about 500° F., but the grease melting points did not rise proportionately to the temperature to which the composition was heated. When heating a mixture containing 15 to 25 per cent of soap to about 450° F., it gave a grease having a melting point of about 410 to 420° F.; while heating the same mixture to about 500° F. produced a grease which had a melting point of 450 to 455° F. McKee considered that the lag of melting point behind the heating point seemed to slightly increase with the increase of the heating point up to

[142] U. S. Patent 1,637,703 (Aug. 2, 1927).

about 500° F. The lower the soap content, the more pronounced seemed to be the lag.

Bunstrum-Schmidt Paper Mill Grease [143]

These investigators noted that a satisfactory dryer roll grease of adequate melting point, and having good wear resisting properties, could be obtained by utilizing the formula:

	Per Cent by Weight
Glycerin	45–80
Sodium soap of hydrogenated oil	10–30
Sodium abietate	10–25

Kaufman's Mill Grease [144]

This product is a dehydrated mixture of about 50 per cent of the sodium soap of neatsfoot oil and mineral lubricating oil.

English Callender Roll Grease

Formula

	Per Cent by Weight
100 S.U.V./100° F. Mexican spindle oil	18.0
Tallow	35.3
Rosin	15.0
Blown rape seed oil	15.0
Water (in finished grease)	2.0
Sodium hydroxide	4.7

In making this grease the tallow is first saponified, after which the other ingredients are added at a lower temperature. The melting point of the finished grease is about 190° F.

Patents Concerning Mill Greases made with Sodium Soaps

In 1871, Johnson [145] patented a mill and railway axle grease consisting of sodium soap, tallow, palm oil, water and sodium carbonate. As fillers, he suggested the use of potassium silicate, which he considered was "precipitated in a gelatinous form and blended with the grease to form one of less fusibility." The Cornelius [146] formula involved the use of sperm or fish oil, and tallow saponified with caustic soda. Jeyes [147] proposed a lubricating grease composed of rosin, creosote, naphthalene, and caustic soda. Lake's [148] grease, patented in 1878, consisted of petroleum, palm wax, graphite, paraffine wax, and sodium bicarbonate. He also used in another mixture, petroleum, graphite, paraffine wax, and caustic soda. Green [149] obtained protection for his mill grease formula which included a mixture of

[143] U. S. Patent 1,982,198 (Nov. 27, 1934).
[144] U. S. Patent 2,024,990 (Dec. 17, 1935).
[145] British Patent 1,378 (May 23, 1871).
[146] British Patent 3,036 (Sept. 16, 1873).
[147] British Patent 4,636 (Dec. 7, 1877).
[148] British Patent 975 (Mar. 11, 1878).
[149] British Patent 2,682 (June 18, 1881).

petroleum, tallow, rosin, and caustic soda. Wade [150] manufactured a solidified brick of lubricating grease composed of:

	Parts
Tallow	10
Castor oil	11
Beeswax	15
Potassium oleate	15
Petrolatum	10
Sperm oil	30
Glycerin	10

Pitt's [151] car, mill, and axle grease consisted of the following:

Petroleum residue	42 gallons
Animal grease (tallow)	50 pounds
Rosin	60 pounds
Strong caustic soda solution	2.5 gallons
Sodium chloride	5 pounds

With the exception of the caustic soda solution the above materials were mixed and heated to 250° F., when the caustic soda solution was added.

A solid grease was prepared by Chenall [152] in 1892. Its formula was:

	Pounds
Petroleum oil	650
Sodium carbonate (and caustic soda)	250
Rosin	90

Rainbow [153] dissolved soda soap in fat and added mineral oil and phenol to obtain a lubricant. Ridsdale and Jones [154] developed a formula for a very hard lubricating grease having a melting point appreciably over 210° F. It contained hard alkaline earth soap, normal sodium soap, stearin, and fat. Sulfur was added to improve the lubricant as a coolant. Sonneborn [155] manufactured a consistent grease in brick form from cotton fibers, lubricating oil, petrolatum, and sodium soaps of animal fats, molded under pressure. Calypsol [156] greases were patented in 1911 and consisted of mineral oil and soda soaps of palm oil, intimately mixed with wool waste, and utilized for mill and journal box bearing lubrication. Bennecke [157] proposed the use of Varrentrap's reaction for converting oleic acid to palmitic acid by treatment at high temperatures with heavy excesses of caustic soda.

Various Analyses

Five Commercial Paper Mill Greases

These products gave, on analysis, the following results:

[150] British Patent 3,774 (Apr. 21, 1888).
[151] U. S. Patent 193,280 (July 17, 1877).
[152] British Patent 952 (Jan. 14, 1893).
[153] British Patent 16,329 (Nov. 28, 1887).
[154] U. S. Patent 486,196 (Nov. 15, 1892).
[155] British Patent 3,718 (1902).
[156] British Patent 6,640 (Mar. 16, 1911).
[157] British Patent 141,720 (May 6, 1914).

	High Temperature Block Grease	Nova Block Grease	Lubro Block Grease M-5
Physical Tests:			
Appearance	Gray block	Light yellow block	Light yellow smooth texture
Penetration at 77° F. worked	....	....	288
Penetration at 77° F. unworked	61	65	240
Dropping point ° C.	184	187	164
Composition (Per cent by weight):			
Soap	36.2	28.8	17.9
Oil	70.8	70.0	82.0
Moisture	0.5	0.5	0.5
Free alkali	0.2	0.2	0.2
Filler	Trace	Trace	Trace
Gain in analysis	1.7	0.5	0.6
Oil Extracted:			
Color, N.P.A.	4	5	6
Gravity, ° A.P.I.	20.9	25.8	26.6
S.U.V. at 100° F.	223	223	259
Flash ° F.	345	390	390
Fire ° F.	400	445	445
Ash, Total per cent on original	6.01	5.44	3.45
Acid insoluble	Trace	Trace	Trace
Sodium carbonate (as Na_2CO_3)	97.5	94.0	87.8
Calcium oxide (as CaO)	....	....	8.5
Undetermined	2.5	6.0	3.7

	KSN Block Grease No. 200 Extra Light	TX Block Grease
Physical Tests:		
Melting point (cube method) ° F.	280	455
Penetration at 77° F.	130	70
Breakdown test	O. K.	O. K.
Color	Light brown	Dark brown
Composition (Per cent by weight):		
Soap	21.7	46.3
Oil	75.0	45.8
Water	0.9	1.0
Acidity (as oleic acid)	2.2	0.1
Asphaltic material	None	2.6
Undetermined	0.2	4.2
Total	100.0	100.0
Fatty Acids from Soap:		
Neutralization (mgms. KOH)	182	191
Melting point ° C.	45.0	36.0
Iodine value (Hanus)	75.0	58.0
Mineral Oil:		
S.U.V. at 210° F.	64	138
Flash o. c., ° F.	445	400
Fire ° F.	495	600
Gravity ° A.P.I.	27.1	21.0
Appearance	Petrolatum	Hvy. oil

	KSN Block Grease No. 200 Extra Light	TX Block Grease
Ash	4.08	9.10
Sodium (Na_2CO_3)	97.6	95.0
Undetermined	2.4	5.0
Total	100.0	100.0

TRC PHILADELPHIA MILL GREASES

These greases gave the following results when analyzed:

	For Cement Mills	For Raymond Mills	For Paper Mill Lubrications
Physical Tests:			
Penetration at 77° F.	75	70	77
Melting ° C.	173	175	171
Odor	Sl. Soapy	Sl. Soapy	Sl. Soapy
Appearance	Lt. brown Block grease	Lt. brown Block grease	Lt. brown Block grease
Composition (Per cent by weight)			
Soap	23.7	24.0	21.0
Mineral oil	74.5	74.4	74.9
Free fat	0.1	0.1	0.8
Alkalinity (per cent NaOH)	0.1	0.1 (% oleic)	0.4
Water	0.2	0.2	0.2
Undetermined	1.4	1.2	2.7
	100.0%	100.0%	100.0%
Mineral oil:			
Gravity ° A.P.I.	28.1	28.5	26.6
S.U.V. at 100° F.	116	99	195
Ash, per cent	5.25	5.03	4.81
Calcium (CaO)	6.3	5.5	6.6
Sodium (Na_2CO_3)	88.0	85.2	92.5
Iron and alumina (R_2O_3)	1.2	1.3	...
Magnesium (MgO)	Trace	3.9	...
Undetermined	4.5	4.1	0.9
	100.0%	100.0	100.0
Fatty Acids from Soap:			
Acid value (Mgms.)	190	194	191
Iodine value (Hanus)	40	43	45
Melting point ° C.	43	46	48
Probable composition	Tallow	Tallow	Tallow

Note:—These greases have nearly the same chemical composition, and differ but slightly in their external appearance.

General Recommendations of Lubricants for Paper Mills

That many lime base greases are necessary, in addition to the usual high melting point soda soap base dryer roll greases, is definitely indicated

by the following recommendations released by one of the large manufacturers of greases.[157a]

Section of Machine	Part to be Lubricated	Type of Bearing	Method of Lubrication	Recommended Viscosity Saybolt Universal
Wet End Cylinder Section	Cylinder Mold Couch Rolls Felt Return Rolls Wet Felt Whipper Rolls	Ball or Roller Bearings	Grease Fitting	Lime Soap Grease, No. 2 Consistency Oil Content 750–770″ @ 100° F.
Wet End Fourdrinier Section	Breast Rolls Table Rolls Wire Carrying Rolls Plain Couch Rolls	Ball or Roller Bearings	Grease Fitting	Lime Soap Grease, No. 2 Consistency Oil Content 750–770″ @ 100° F.
	Suction Couch Rolls	Internal and External Bearings	Grease Fitting	Lime Soap Grease, No. 2 Consistency Oil Content 750–770″ @ 100° F.
	Shake Motion and Minor Parts	Shake Crank Pin Roller Bearing and Connecting Rods	Wick Feed Grease Fitting Grease Fitting	300–310″ @ 100° F. Lime Soap Grease, No. 2 Consistency Oil Content 750–770″ @ 100° F.
	Main Shake Shaft	Plain	Oil	83–85″ @ 210° F. 98–100″ @ 210° F.
	Lift Worm		Oil	140–145″ @ 210° F.
Wet End Press Section	Press Rolls Squeeze Rolls Wet Felt Rolls, Paper Rolls, Etc.	Roller Bearings	Grease Fittings	Lime Soap Grease, No. 2 Consistency Oil Content 750–770″ @ 100° F.
	Suction Press Rolls	Internal and External Bearings	Grease Fittings	Lime Soap Grease, No. 2 Consistency Oil Content 750–770″ @ 100° F.
	Broke Conveyor Witham Head	Roller Bearings	Grease Fittings	Lime Soap Grease, No. 2 Consistency Oil Content 750–770″ @ 100° F.
Dryer Section	Dryers and Felt Rolls	Plain and Roller Bearings	Wick Feed Ring Oiled and Continuous Circulation	83–85″ @ 210° F. 98–100″ @ 210° F.
		Roller Bearings	Wet Sump not Continuous Circulation	115–150″ @ 210° F.
			Grease Fitting	Soda Soap Grease, No. 2 Consistency Oil Content 400–410″ @ 100° F.
	Chain Drives	Chain Sprocket and Shaft Bearing	Bath Grease Fitting	400–410″ @ 100° F. Lime Soap Grease, No. 2 Consistency Oil Content 750–770″ @ 100° F.
Finishing Room	Calenders	Plain Bearings	Collar Oiling and Continuous Circulation	83–85″ @ 210° F. 98–100″ @ 210° F.
		Roller Bearings	Grease Fittings	Lime Soap Grease, No. 2 Consistency Oil Content 750–770″ @ 100° F.
			Oil—Wet Sump or Continuous	115–150″ @ 210° F.
	Reel, Slitters, Winder, Cutters	Roller Bearings	Oil	83–85″ @ 210° F. 98–100″ @ 210° F.
			Grease Fitting	Lime Soap Grease, No. 2 Consistency Oil Content 750–770″ @ 100° F.
		Winder Roll Dump	Hydraulic Fluid Medium	300–310″ @ 100° F.
Power Transmission	Line Shafting	Roller Bearings	Grease	Lime Soap Grease, No. 2 Consistency Oil Content 750–770″ @ 100° F.
			Oil	83–85″ @ 210° F. 98–100″ @ 210° F.
	Gear Units	Spiral Bevel	Splash	Extreme Pressure Lubricant of S.A.E. Viscosity No. 90 rating. Non-corrosive. Free from Sulphur and other deleterious substances. Channel—0° F. Max.
		Hypoid	Splash	Extreme Pressure Lubricant of S.A.E. Viscosity No. 90 rating. Non-corrosive. Free from Sulphur and other deleterious substances. Channel—0° F. Max.

[157a] The above recommendations apply particularly to Beloit paper making machinery. *Work Factor News*, 5th Ed. (1934).

MUSICAL SCREW POST LUBRICANT

Adler has carried out investigations of lubricants suitable for application to violin screw posts and the like. He has concluded that a composition of petrolatum, chalk, vegetable oil, powdered graphite, and sodium hydroxide for saponifying the vegetable oil, was satisfactory.

PLUG VALVE LUBRICANTS

Calcium base plug valve lubricants have been discussed in Chapter V. For high temperature applications sodium soaps are sometimes used. In most cases, the plug valve greases are cast into small sticks which may conveniently be placed in the cavity of the plug valve stem. The grease is forced to the valve surfaces and causes the plug to lift off its seat, when a screw fitted into the grease cavity is turned up. Plug valve grease manufacturers offer a variety of products for different industrial purposes, depending on whether the temperature of the valve reaches maxima of 300° F., 400° F., 525° F., or 250° F., and according to the nature of the fluid being handled, *i. e.,* steam, gasoline, crude oil, gas, etc.

Sodium Soap-Mica Plug Valve Lubricant

Base greases frequently used for mixing with petrolatum, waxes, graphite, etc., in the preparation of valve lubricants, have properties as shown below:

	A*	B
Color	Pasty gray	Yellow brown
Penetration A.S.T.M.	127	116 (135 after working in mica)
Dropping point	165	182
Odor	Alkaline	Alkaline
Appearance	Fibrous	Fibrous
Free alkali	0.20	0.34
Percent water by dist.	1.00	0.70

Composition (Per cent by weight):

	(By Analysis)	(Per Cent by Weight)	
Soap	22.89	1. Tallow	= 16.06
Oil	69.75	2. Stearic acid	= 9.58
Moisture	1.00	3. Caustic soda	= 3.78
Free alkali	0.20	4. 150 Pale oil	= 32.50
Filler (mica)	6.54	5. 300 Pale oil	= 30.58
Gain in analysis	0.38	6. Water	= 0.70
		7. Mica	= 6.50

* "A" is the base grease for "B," and contains no mica.

Water Solubility Tests

It is sometimes required that valve lubricants pass a water solubility test. This may be carried out as follows. The results of the test, as applied to several greases, are also given.

TEST CONDITIONS

Ball of grease, diameter approximately 7 mm., to be coated with mica, and placed in a test tube half-full of water.

RESULTS

The following results were obtained:

Grease No.	Formula, Per Cent		No. of days required to bring about separation of oil in sufficient amount to float to the surface of the water
1.	Tallow	33.0	1 Day
	Caustic soda	4.7	
	Mica	6.5	
	Sodium chloride	3.0	
	200 pale oil	52.8	
		100.0	
2.	Tallow	33.0	13 Days
	Caustic soda	4.7	
	Mica	6.5	
	Sodium oxalate	5.0	
	200 pale oil	50.8	
		100.0	
3.	Stearic acid	33.0	10 Days
	Caustic soda	4.7	
	Triethanolamine	2.0	
	Mica	6.5	
	200 pale oil	53.8	
		100.0	
4.	Stearic acid	33.0	Grease still intact at 10 days
	Caustic soda	4.7	
	Mica	8.0	
	Rubber latex	0.3	
	200 pale oil	54.0	
		100.0	
5.	Grease "A"		Approx. 4 days (almost complete disintegration in 8 days)

Gasoline Solubility Tests

TEST CONDITIONS

Ball of grease, approximately 7 mm. in diameter, to be coated with mica, and placed in a test tube half-full of gasoline.

RESULTS

The following results were obtained:

Grease "A."—At the end of twenty-four hours the grease had cracked in a number of places, and flattened out somewhat, losing its spherical shape.

Grease "4."—Cracking of the grease was more pronounced than with "A" grease. However, a spherical shape was retained better.

COMMENTS

In the water solubility tests above, it will be noted that the greases prepared from stearic acid give the best results. In this respect the water solubility tests correlate with the emulsification tests. Grease No. 2, containing

sodium oxalate, proved exceptional in that it gave an excellent water solubility test in spite of a poor emulsification test.

The full extent of the superiority of stearic acid greases in the water solubility tests is not evident in the data above. A grease similar to "4" (0.2 per cent latex instead of 0.3) has stood for over a month with very little disintegration and with a separation of only a few droplets of oil. Grease "A" was badly disintegrated at the end of a week.

Conclusions

The susceptibility of a grease to emulsification depends primarily upon the soap stocks. The presence of salts, triethanolamine, glycerol, or other materials is usually of secondary importance.

Greases prepared from saturated soda soaps of molecular weights approximating those of sodium stearate are unusually resistant to emulsification.

Greases prepared from saturated soaps of lower molecular weight, for example soaps prepared from palm kernel oil, are unusually susceptible to emulsification. This behavior is not surprising in view of the current practice in soap manufacture of using palm kernel oil, or coconut oil, to make soap chips which dissolve readily.

The presence of sodium oleate in a grease renders it especially susceptible to emulsification regardless of whether the oleate is formed by saponification of oleic acid or tallow.

Sodium linolenate, the principle soap formed in the saponification of linseed oil, when used in conjunction with sodium stearate, yields greases which are much less susceptible to emulsification than those containing sodium oleate. (Exceptional resistance is obtained when care is taken to dehydrate the grease.)

The apparent anomaly found in the ready emulsification of greases prepared from degras is probably due to the presence of hydroxylated soaps. Soaps prepared from degras have a high molecular weight and a low unsaturation (therefore a low oleate content) and might, therefore, be expected to be resistant to emulsification.

Laboratory Tests and Suggested Specifications

Typical laboratory tests and suggested specifications for sodium soap, plug valve greases are given below:

	A	1	2	3	Suggested Specifications
A.S.T.M. penetration at 77° F.					
Unworked	127	126	132	110	.....
Worked	165	157	155	148+	135–165
Dropping point ° F.	329	333	309	392+	310 min.
Soap, per cent	22.9	24.0	27.3	35	.....
Mica, per cent	6.54	5.4	6.6	8	8–9
Free alkali	0.2		0.13	0.56	0.5 max.
Percent moisture	1.00		1.60	2.3	3 max.

ANALYSIS OF FIBER GREASE FOR PLUG VALVES

This grease gave the following results on analysis:

Physical Tests:

Color	Pasty gray
Penetration, A.S.T.M. (unworked) at 77° F.	127
Dropping point ° C.	165
Odor	Alkaline
Appearance	Fibrous

Composition, per cent by weight:

Soap	22.89
Oil	69.75
Moisture	1.00
Free alkali	0.20
Free fat	0.00
Filler (mica)	6.54
Gain in analysis	0.38
Total	100.00

Oil Extracted:

Color, N.P.A.	Approximately 2½
Gravity, ° A.P.I.	19.8
S.U.V. at 100° F.	216
Flash, ° F.	350
Fire, ° F.	395

Ash, total per cent on original:

Sodium carbonate (as Na_2CO_3)	35.08
Mica, white	61.40
Undetermined, probably mostly iron	3.52
Total	100.00

Fatty Acids from Soap:

Iodine value (Hanus)	46
Acid value (mg. KOH)	201
Melting point ° C.	47
Color	Light brown
Probable composition	Stearic and oleic acid

Probable composition, per cent by weight:

200 pale oil	70.0
Soda soap (60 per cent stearate and 40 per cent oleate)	22.5
Water	1.0
White mica	6.5
Total	100.0

Pressure Viscosity of Plug Valve Grease

Since plug valve grease must be readily applicable over a wide range of temperatures, pressure viscosity data is of interest. The following data is for a grease made on the formula as shown below, the greases meeting the specifications indicated. A Saybolt Universal Viscosity tube was employed.

Formula

	Per Cent by Weight
Tallow	16.06
Stearic acid	9.88
Caustic soda	3.78
150 pale oil	32.50
300 pale oil	30.58
Water	0.70
Mica	6.50

Specifications

Penetration, unworked at 77° F.	100–150
Melting point ° C., Ubbelohde (min.)	150
Free alkali, percent NaOH (max.)	0.50

$$\eta = \frac{PT}{Q_T}\left(\frac{\pi R^4}{8L}\right) = \frac{PT}{Q_T} \times \text{a constant}$$

$$\frac{1}{\eta} = \frac{Q_T}{PT} \times \text{a constant}$$

Unworked Grease and Constant Pressure

Temperature	Pressure	Grams per Minute	Average Grams per Minute	$1/\eta$	η
71.6	100	6.58	6.49	.0649	15.4
		6.40			
99	100	39.36	38.52	.3852	2.6
		37.68			
131	100	140.6	140.6	1.406	.71
156	100	399.6	405.6	4.056	.24
		411.6			

Unworked Grease and Approximately Constant Flow Rate

71.6	100	6.58	6.49	.0649	15.4
		6.40			
131	40	10.26	9.84	.246	4.07
		9.42			
154	20	6.76	6.56	.328	3.05
		6.36			

Worked Grease and Approximately Constant Flow Rate

46	79	7.86	8.01	.1014	9.89
		8.16			
66	50	9.44	9.51	.190	5.28
		9.58			
98	24	8.98	8.67	.361	2.76
		8.36			
98	20	6.58	6.83	.341	2.93
		7.08			
120	15	9.36	9.82	.65	1.52
		10.28			
120	15	11.08	10.51	.70	1.42
		9.94			
135	15	6.58	6.76	.52	1.92
		6.94			
144	17	5.80	5.78	.34	2.95
		5.76			
146	20	6.08	6.08	.304	3.29
154	20	7.04	7.04	.352	2.84

PNEUMATIC TOOL GREASES

Experiments with Thor Air Motor

The following general discussion of pneumatic tool lubrication is based on some experiments made with a Thor Size 13, Model 976 air motor fitted with a small pony brake for measuring horsepower output. The following conclusions were reached:

A satisfactory air motor grease is a matter of compromise as to consistency. It must be heavy enough to cling to fast moving parts yet it must be thin enough to keep from channeling.

The power absorbed in agitating the crankcase grease and frictional horespower is a considerable percentage of the input horsepower to the motor and must be seriously considered when selecting a satisfactory grease.

An air motor grease must have sufficient cohesion and adhesion to remain in the motor and not be lost out through stuffing boxes and exhausts.

It is doubted whether an air motor grease must be of a lime soap base. Commercial product, F-9, is a straight soda base grease and performed much better than either of two factory recommended greases which are lime soap and soda soap mixed base greases.

All possible measures must be taken to reduce to a minimum the water and abrasives in the air supply. Therefore, the air lines should be thoroughly blown out before attaching to the motor.

"Standardized Fiber Grease No. 3" is one of the most satisfactory products that was tested for the lubrication of reciprocating type air motors. It is a soda base grease, yet its performance during the test was entirely satisfactory.

An acceptable air motor lubricant could also be made from a blend of Dark Fiber Grease No. 4 and Universal Joint Grease. The blend would have a lower horsepower output than the Fibre No. 3, but its retention characteristics would probably be about the same.

Contrary to the opinions of some authorities, it is found that the importance of the property of resistance to moisture of the lime soap base grease is not as great as believed.

The following data applies to the Thor air motor used in the tests which will be discussed in this section:

Size	13
Model	976
Stroke	$1\frac{3}{4}$ inches
Bore	$1\frac{5}{8}$ inches
Firing order	R1, L1, R2, L2
Reduction gear ratio	80:12

Possibly, reciprocating air motors should be more correctly called air engines, since they are small reciprocating steam engines, using compressed air instead of steam. At present, air motors are being made in both reciprocating and multi-vane types, the reciprocating air motors having either two, three or four cylinders. The two-cylinder type is used for specialty jobs, such as "close quarter" and track wrenches; the three-cylinder type is made with the piston extending radially and equally spaced from a one-throw crankshaft; the four-cylinder type is either made in a 90° "V" such as air drills, or spaced radially as found in air hoists. Since the 90° "V", four-cylinder motors are the most commonly used and subjected to the most severe operating conditions, it was considered advisable to use this type in

the test. The Ingersoll-Rand type of drill uses one master rotary valve, rotating at one-half the crankshaft speed. The Thor size No. 3 air motor, such as used in these tests, is of the 90° type, using two valves, one for each cylinder bank, being operated in an oscillatory motion by a small "dog" connected to a crankshaft eccentric. Since each piston has a working stroke every crankshaft revolution, the eccentric is driven at crankshaft speed so that the valve will make one complete oscillation per revolution of the crankshaft.

The opposing pistons are interconnected, using the same throw on the crankshaft by means of a forked type connecting rod, using steel on steel bearings, while the two main bearings of the crankshaft are roller bearings operating on steel journals in bronze bushings. A straight toothed spur gear is used to drive the reduction gear which is attached integrally with the Morse No. 3 taper socket. All the axial thrust on the drill socket is taken by a thrust ball bearing in steel races, while the lateral thrust is taken by steel journals operating in plain bronze and steel bearings.

The motor was fitted with a rope brake so as to provide a means of adjusting the applied load, the brake drum was fixed to the motor by a Morse No. 3 taper with a tanged end. One end of a one-half inch manila rope, wrapped three times (in the direction of drum rotation) around the brake drum, was fastened to a spring balance, and the other end was passed over a pulley to an adjustable weight "w." The spring balance and pulley were suspended from a davit which was supported on the test block. Lubrication of the friction surface of the brake was necessary so an oil bath was installed by placing a five-gallon oil can around the brake drum and filling it with lard oil until the lower one-eighth of the drum was covered.

Since the problem was primarily one of grease retention it was necessary to devise a means of collecting the lost grease. It was found that the grease was lost from the motor at four places: (1), the crankcase pressure release valve or breather; (2), the packing gland on the socket shaft; (3), the packing gland at the feed screw; (4), air exhaust. The air exhaust contained the grease that had leaked between the pistons and the cylinder walls and will be referred to hereafter as "blowby" grease. A circular collector was used to obtain the loss through the packing gland at the socket, while simple drip pans sufficed for the breather and packing gland at the feed screw. The blowby grease came out in small particles along with the exhaust air so a separating device, arranged to cause the air coming out of the exhaust to change direction twice before exhausting to the atmosphere, was installed. This change in direction caused the exhaust air to deposit all of its grease particles on the surfaces of the ducts or collectors.

The crankcase temperature was measured by means of a 20 to 230° F. mercurial thermometer, placed into the crankcase so that the bulb was as near as possible to the moving parts.

Water content of the air from the compressors available was no doubt similar to the air found at other air motor installations, but it was desired to simulate the worst possible conditions, so an air saturator was installed. Air from a one-inch line was passed through a globe valve and into a

steel tank, to within two inches of the bottom, and well below the surface of the water in the tank. Large drops of water were broken up in the water separator, which consisted of a 3-inch by 6-inch pipe nipple closed at each end with ¼-inch screen and filled with aluminum raschig rings. A three-quarter-inch leader was used to conduct the air from here to the throttle of the air motor. Air pressure was adjusted by the globe valve and determined by the pressure gage placed at the top of the air saturator tank.

Test Procedure

Preceding each new test, the air motor was completely disassembled and thoroughly washed with benzol. When reassembling the motor only a thin film of grease was placed on all the moving parts, allowing the bulk (454 grams, or 1 pound.) of grease to be placed in the crankcase, thus affording an equal test for all greases. After the motor was strapped on the test block and all accessories, such as the rope brake and grease collectors, were put in place, five inches of water was placed in the air saturator and the three-quarter-inch leader was connected between the throttle valve and the motor.

The tests were started on the hour, being run the first hour with full air line pressure attached to the "wide open" throttle. The time between the first and second hour of operations was used to determine the lubricating efficiency of the grease. This was accomplished by adjusting the globe valve for pressures by increments of 10 pounds per square inch between 30 pounds and the maximum available. At each pressure the torque on the air motor was adjusted to approximately 16.25 foot pounds and the speed taken with a hand tachometer was recorded. The order in which the data was taken, starting with the 30 pounds per square inch and working upward to the maximum pressure by the increment stated, was to keep as high a crankcase temperature as possible for the particular grease under test. From this data, it was possible to determine the required air pressure, acting at "wide open" throttle, to obtain a torque of 16.25 foot pounds and 200 R.P.M. These values of pressure, torques and speed were maintained throughout the remainder of the test. They were selected because 65 to 70 pounds per square inch was the maximum dependable air pressure obtainable from the line.

Data for the retention characteristics of the grease were obtained by wiping, with an accurately weighed pledget of cotton, from the motor the lost grease at intervals of two hours of continuous running or oftener if necessary. The wiped up blowby grease was kept separate from that obtained from the other parts of the motor so as to increase the value of the data. Since much water was absorbed by the cotton while wiping off the blowby, it was necessary to dehydrate the cotton before each weighing.

Readings were taken every hour during the eight-hour test and recorded. They consisted of the following: Time, throttled air pressure, reading of spring balance, the variable weight, "w," the R.P.M. and crank-case temperature. If the pressure had changed during the preceding hour, due to air line fluctuation, it was readjusted by means of the globe valve after

the readings had been taken. Also, if the spring balance did not read about 63 pounds (so as to give 16.25 foot pounds of torque) the variable weight, "w," was adjusted until this was corrected. It was necessary to add water in the air saturator about twice during each run in order to maintain the water level in the tank between five and three inches above the bottom.

At the end of each run the motor was carefully dismantled and each part thoroughly examined in order to ascertain whether or not good distribution of the lubricant had been maintained. The used grease was then collected, placed in a container, and set aside for further tests.

A means of testing a grease's ability to withstand agitation was considered and the following test was devised: New samples of greases were placed in Saybolt Universal Viscometer tube, fitted with a gasketed screw cap. Twenty-five pounds per square inch pressure was supplied on the top of the grease in the viscometer tube by means of a nitrogen cylinder and pressure regulator. Flow was determined through this tube by means of weighing the amount of flow in a definite time measured with a stop watch. These rates of flow were taken at various temperatures between 32° F. and 100° F. and plotted as logarithms against temperature. Duplicate samples of grease were placed in a gear pump that was driven by a one-half horsepower, 1700 R.P.M., electric motor. These samples were pumped through the pump continuously for eight hours. The suction side of the pump was directly connected by a $\frac{1}{4}$-inch pipe to the discharge side so that complete circulation was assured. After the eight hours the grease was removed and a pressure viscosity curve was determined as above. The No. 52 grease became very lumpy making it impossible to determine this curve because flow through the tube was interrupted by the lumps.

Grease Retention Tests for the Thor Model No. 976 Air Motor, Using Various Lubricating Greases

The results of these tests are shown in the following tables:

Test No.	Name of Grease	Length of Run Hours	Air Press. Pounds per Square Inch	Resultant Scale Force Pounds	R.P.M.	B.H.P.	Crank-case Temp. ° F.
1	F-9	0	75	57.8	215	0.642	...
		1	60	56.5	196	0.570	118
		2	60	58.7	204	0.618	105
		3	60	59.5	195	0.599	106
		4	62	59.5	200	0.614	109
		5	67	62.6	224	0.722	112
2	No. 52	0	62	...	...	....	...
		1	62½	60.0	198	0.611	120
		2	63	59.9	200	0.617	123
		3	62½	59.7	203	0.625	123
		4	61½	59.7	206	0.634	116
		5	63	61.5	202	0.640	114
		6	61½	61.5	201	0.637	105
		7	62	59.2	205	0.625	103
		8	63	61.1	205	0.646	101

Test No.	Name of Grease	Length of Run Hours	Air Press Pounds per Square Inch	Resultant Scale Force Pounds	R.P.M.	B.H.P.	Crank-case Temp. ° F.
3	No. 57	0	..	...	...	....	...
		1	69	57.3	219	0.646	125
		2	64	59.4	212	0.648	117
		3	67	58.8	205	0.622	116
		4	65	53.9	216	0.600	114
		5	64	62.4	210	0.675	103
		6	62½	57.4	220	0.650	95
4	Stand-	0	70				
	ardized	1	72	38.0	196	0.382	138
	Wheel	2	72	38.7	202	0.403	140
	Bearing	3	Not	Not	Not	Not	Not
	Grease		Det.	Det.	Det.	Det.	Det.
		4	75	59.2	195	0.604	138
		5	75	60.8	196	0.615	134
		6	75	56.8	190	0.556	118
		7	73	55.8	198	0.570	125
		8	75	53.8	208	0.577	128
5	Gear	0	87	...	...	....	...
	Grease	1	70	57.1	172	0.636	138
	S.A.E.-160	2	73	47.8	192	0.506	139
		3	75	59.9	200	0.473	146
		4	73	58.9	196	0.595	142
		5	73	58.9	202	0.613	145
		6	72	56.9	200	0.586	145
		7	72	56.9	200	0.586	145
		8	76	54.9	220	0.622	148
6	Dark	0	..	...	...	....	...
	Fiber	¾	85	47.8	219	0.540	147
	Grease	1½	75	56.7	205	0.600	143
	No. 4	2½	75	59.5	185	0.567	145
		3½	75	58.5	193	0.572	140
		4½	80	58.5	215	0.650	145
		5½	75	56.5	195	0.568	144
		6½	75	58.5	207	0.625	145
		7½	75	57.5	220	0.652	146
		8½	75	56.2	230	0.667	144
7	Universal	0	75	...	...	....	...
	Joint	1	75	45.8	240	0.566	165
	Grease	2	75	59.5	200	0.613	162
		3	73	59.5	194	0.595	152
		4	75	60.5	192	0.599	153
		5	75	60.5	204	0.636	150
		6	75	60.5	210	0.655	147
		7	75	73.4	212	0.800	153
		8	75	74.4	200	0.766	151
8	F-9	0	..	...	...	....	...
		1	74	59.7	238	0.732	125
		2	68	62.9	222	0.720	120
		3	67	63.9	216	0.710	116
		4	65	63.9	216	0.710	118
		5	66	63.9	200	0.657	108
		6	66	63.9	204	0.670	108
		7	66	66.4	208	0.711	111
		8	66	65.4	208	0.700	110

Test No.	Name of Grease	Length of Run Hours	Air Press. Pounds per Square Inch	Resultant Scale Force Pounds	B.H.P.	R.P.M.	Crank-case Temp. ° F.
9	Stand.	0	..	...	...	....	...
	Fiber	1	75	52.3	236	0.637	120
	Grease	2	70	59.9	206	0.635	114
	No. 3	3	67	57.9	222	0.660	108
		4	67	60.7	230	0.720	105
		5	63	59.7	213	0.656	105
		6	65	59.7	220	0.676	105
		7	55	58.7	196	0.594	108
		8	60	59.7	200	0.615	107
10	Stand.	0	75	...	...	....	...
	Fiber	1	82	48.7	254	0.639	156
	Grease	2	68	57.4	195	0.576	138
	No. 2	3	70	62.4	212	0.680	137
		4	70	63.4	208	0.680	137
		5	69	65.4	200	0.673	122
		6	69	63.4	198	0.646	117
		7	68	60.4	200	0.622	111
		8	68	63.4	195	0.636	110

Test No.	Name of Grease	Length of Run Hours	Charge Grams	Weight Wiped Off (Grams)	Weight Lost Through Exhaust (Gms.)	Cumulative Total Wt. of Grease Lost (Grams)	Cumulative Total Per Cent of Charge Lost
1	F-9	0	454	...	...	...	...
		1		5.001–¼ hours	0	5.001–¼ hours	1.101–¼ hours
		2		0.9	4.4	10.3	2.3
		3		...	...	...	...
		4		0.6	...	10.9	2.4
		5		0.7	2.4	14.0	3.1
2	No. 52	0	454	...	...	...	...
		1		...	...	...	...
		2		6.0	13.5	19.5	4.3
		3		10.2	0	29.7	6.6
		4		18.1	11.5	59.3	13.1
		5		15.5	0	74.8	16.5
		6		11.2	8.6	94.6	20.9
		7		2.2	0	96.8	21.4
		8		0.3	6.3	103.4	22.8
3	No. 57	0	454	...	...	...	...
		1		0	40.3	40.3	8.9
		2		...	...	...	...
		3		0.7	25.3	66.3	14.7
		4		0	4.2	70.5	15.6
		5		0	1.6	72.1	15.9
		6		2.5	1.2	75.8	16.7
4	Stand.	0	454	...	...	...	...
	Wheel	1		3.1	3.4	6.5	1.4
	Bearing	2		...	...	...	...
	Grease	3		Not Det.	Not Det.	Not Det.	...
		4		3.3	0	9.8	2.2
		5		...	...	...	...
		6		1.5	1.8	13.1	2.9
		7		...	...	...	...
		8		0.5	0.3	13.9	3.1

Test No.	Name of Grease	Length of Run Hours	Charge Grams	Weight Wiped Off (Gms.)	Weight Lost Through Exhaust (Gms.)	Cumulative Total Wt. of Grease Lost (Grams)	Cumulative Total Per Cent of Charge Lost
5	Gear Grease S.A.E.-160	0	454	...	...	...	...
		1		27.1	4.8	31.9	7.1
		2		...	...	...	...
		3		23.9	5.9	61.7	13.6
		4		...	...	...	...
		5		16.5	6.0	84.5	18.7
		6		...	...	...	...
		7		...	...	...	...
		8		25.1	22.5	132.1	29.2
6	Dark Fiber Grease No. 4	0	454	...	...	...	...
		¾		27.3	0	27.3	6.0
		1½		...	...	...	...
		2½		33.5	6.2	67.0	14.7
		3½		...	...	...	...
		4½		18.9	11.1	97.0	21.4
		5½		...	...	...	...
		6½		15.9	3.3	116.2	25.6
		7½		...	...	...	...
		8½		10.7	3.2	130.1	28.7
7	Universal Joint Grease	0	454	...	...	...	...
		1		...	...	...	...
		2		4.5	1.5	6.0	1.3
		3		...	...	...	...
		4		1.5	1.1	8.6	0.9
		5		...	...	...	...
		6		0.9	0.8	10.3	2.3
		7		...	...	...	...
		8		0.1	1.2	11.6	2.6
8	F-9	0	454	...	...	...	...
		1		...	...	...	...
		2		2.4	2.4	4.8	1.1
		3		...	...	...	...
		4		1.6	1.3	7.7	1.7
		5		...	...	...	...
		6		0.3	0.5	8.5	1.9
		7		...	...	...	...
		8		0.1	1.6	10.2	2.3
9	Stand. Fiber Grease No. 2	0	454	...	...	...	...
		1		21.0	9.2	30.2	6.7
		2		...	...	...	...
		3		28.1	3.4	61.7	11.4
		4		...	...	...	...
		5		11.7	1.8	75.2	16.6
		6		...	...	...	...
		7		...	...	...	...
		8		11.1	0.9	87.2	19.2
10	Stand. Fiber Grease No. 3	0	454	...	...	...	...
		1		...	...	...	...
		2		0	9.8	9.8	2.2
		3		...	...	...	...
		4		0	6.6	16.4	3.6
		5		...	...	...	...
		6		9.2	5.2	30.8	6.8
		7		...	...	...	...
		8		9.7	7.9	48.4	10.7

Performance of the Thor Model No. 976 Air Motor Using Various Lubricating Greases

The results of these tests are shown in the following table:

Test No.	Name of Grease	Air Pressure Pounds per Square Inch	Resultant Scale Force Pounds	R.P.M.	B.H P.
1	F-9	29	60.4	55	0.171
		40	59.2	116	.353
		52	59.3	173	.529
		62	59.6	210	.646
		74	59.8	240	.740
2	Pneumatic Tool Grease No. 52	31	60.2	58	0.180
		41	60.6	106	.332
		48	60.0	154	.476
		60	60.1	189	.586
		68	60.3	218	.679
		76	59.7	252	.775
3	Pneumatic Tool Grease No. 57	38	59.5	68	0.208
		42	59.3	123	.376
		51	59.1	148	.452
		58	59.5	182	.559
		70	59.6	216	.665
		79	59.9	229	.706
		89	60.3	245	.762
4	Wheel Bearing Grease	42½	60.5	67	0.209
		48	59.7	92	.284
		55	59.7	125	.384
		60	60.3	133	.413
		67½	60.6	156	.487
		75	60.5	181	.565
5	Grease S.A.E.-160	32	60.5	25	0.078
		42	60.1	38	.115
		52	60.9	86	.270
		60	60.0	119	.368
		67½	60.1	145	.450
		75	61.0	178	.560
6	Dark Fiber Grease	38	61.5	31	0.096
		47	59.3	77	.240
		57	60.2	114	.321
		65	59.9	146	.453
		73	60.7	178	.510
		85	60.0	202	.636
7	Universal Joint Grease	35	59.7	45	0.139
		44	63.1	87	.283
		55½	59.5	133	.407
		64	60.5	145	.452
		67	60.5	173	.539
		73	59.5	196	.600
8	F-9	32	60.4	69	0.215
		43	60.4	133	.414
		53	59.1	180	.549
		61	61.1	205	.647
		70	61.0	227	.714
		73	59.3	239	.730

Test No.	Name of Grease	Air Pressure Pounds per Square Inch	Resultant Scale Force Pounds	R.P.M.	B.H.P.
8-A	Motor Oil S.A.E.-10	26	60.4	44	0.137
		38	59.2	122	.372
		50	60.6	180	.563
		60	62.5	211	.680
		70	60.4	242	.752
		74	60.3	255	.793
9	Fiber Grease No. 2	30	59.7	39	0.120
		40	60.1	96	.298
		50	61.2	150	.472
		60	61.7	186	.590
		70	62.7	215	.694
10	Fiber Grease No. 3	30	58.7	31	0.094
		40	57.8	90	.268
		50	58.1	138	.413
		60	59.2	175	.534
		69	60.1	201	.623
		74	60.2	218	.676
		86	59.7	238	.732

Values of Horsepower Absorbed by Excess Friction Plus Agitation of Grease for an Air Pressure of 65 Pounds Per Square Inch

The results of these tests are shown in the following table:

Grease	IHP-FHP Min.	B.H.P.	GAHP* + Excess FHP**	Per Cent of Max. HP Lost $\frac{\text{GAHP*+Excess FHP**}}{\text{IHP+FHP Min.}} \times 100$
F-9		0.672	0.053	7.30
	0.725	0.678	0.047	6.48
No. 52	0.725	0.650	0.075	7.49
No. 57	0.725	0.622	0.103	14.20
Gear Grease S.A.E.-160	0.725	0.434	0.290	40.0
Dark Fiber Grease No. 4	0.725	0.442	0.283	39.0
Universal Joint Grease	0.725	0.518	0.207	28.5
Wheel Bearing Grease	0.725	0.466	0.249	34.3
Fiber Grease No. 2	0.725	0.648	0.077	10.60
Fiber Grease No. 3	0.725	0.591	0.134	18.49

* GAHP is the horsepower absorbed by grease agitation.

** Excess FHP is the amount of power absorbed by friction, when using a given grease, in excess of the power absorbed by friction when using Standardized Motor Oil S.A.E.-10.

Temperature—Rate of Flow Data

Flow was through a Saybolt Universal Viscometer Tube under twenty-five pounds per square inch pressure. The results are shown in the following table:

Run No.	Name of Grease	Grease Temp. ° F.	Pressure Pounds per Square Inch	Time Seconds	Flow of Grease Grams	Rate of Flow of Grease gms/min.
1	F-9	38	35	120	3.631	1.816
2	(Before agitation)	51½	35	90	5.416	3.611
3		75	35	60	14.100	14.100
4		75	35	60	16.723	16.723
5		98	35	30	19.365	38.730

Run No	Name of Grease	Grease Temp. ° F.	Pressure Pounds per Square Inch	Time Seconds	Flow of Grease Grams	Rate of Flow of Grease gms./min.
1	F-9	32½	25	60	9.636	9.736
2	(After agitation)	46½	25	43	23.977	29.950
3		70	25	20	36.977	109.770
4		82	25	10	30.903	185.418
1	Pneumatic Tool	39½	25	60	3.489	3.489
2	Grease No. 57	73	25	30	20.932	41.864
3	(Before agitation)	73	25	20	13.966	41.898
4		81	25	20	25.272	75.816
5		97	25	14	36.400	156.000
1	Pneumatic Tool	46	25	10	10.370	62.220
2	Grease No. 57	61	25	5	11.121	133.452
3	(After agitation)	88	25	5	31.310	375.720
1	Pneumatic Tool	38	25	30	5.800	11.600
2	Grease No. 52	66½	25	30	26.879	53.758
3	(Before agitation)	84	25	5	10.931	131.172
4		98	25	5	14.238	170.856

Properties of Pneumatic Tool Greases Tested

The properties of the various greases tested are shown in the following table:

Name of Grease	Unworked Penetration	Worked Penetration	MacMichael Viscosity	% Free Alkali NaOH	Drop Point ° C.
Standardized Gear Grease S.A.E.-160	...	...	160 at 80 No. 26 Wire	0.07	44
Dark Fiber Grease No. 4	...	...	80 at 95 No. 24 Wire	0.08	82
Fiber Grease No. 2	...	...	81 at 80 No. 22 Wire	0.03	149
Fiber Grease No. 3	175	255	.....	0.16	161
Universal Joint Grease	285	350	.....	0.25	124
Wheel Bearing Grease	222	300	.....	Neutral	192

	F-9	52	57
Penetration at 77° F.			
Worked	270	267	227
Unworked	250	258	205
Dropping point, ° C.	152	92	100
Water, per cent	0.55	1.50	0.85
Free alkali, per cent by weight	0.01 Percent NaOH	0.3 Percent $Ca(OH)_2$	Neutral
Soap, per cent by weight	13.0	13.6	13.5
Ash analysis, per cent by weight	2.45	1.53	1.46
Acid insoluble	....	3.2 (Iron and acid insol.)	....
Sodium carbonate	93.9	4.4	19.0
Calcium oxide	....	86.3	78.0
Iron oxide	....	....	2.0
Corrosion	O.K.	O.K.	O.K.
S.U.V. at 100° F.	476	382	316
S.U.V. at 210° F.	57.3	48.0	47.1
Gravity of oil, A.P.I.	27.0	23.1	26.3

Conditions of the Motor When Dismantling After Tests

The following conditions were observed:

F-9.

Crankcase inspection showed that the grease was sticking to the crankshaft and connecting rods very well, wrapped around as taffy might be. A very satisfactory film of oil was present on the crankshaft bearings and cylinder walls. Oil passage holes were filled showing that their bearings were getting grease and this was confirmed by inspection of the bearings themselves. The reduction gear teeth as well as the valve eccentric bearing showed that excellent lubrication had existed.

No. 52 and No. 57.

Both of these greases showed similar lubrication characteristics. Channelling existed in both greases but was more pronounced for the No. 57. The grease showed a tendency to separate, depositing small puddles of oil at various points in the grease. No particles of grease appear on the crankshaft, but the shaft bearings as well as cylinder walls had an oil film. All grease passage holes except those to the valve parts themselves were filled with grease, showing a proper flow of grease to those parts. The general condition of the motor throughout showed rather satisfactory lubrication.

Wheel Bearing Grease and Universal Joint Grease.

Both of these greases displayed a definite lack of proper lubrication of the motor's moving parts. The wheel bearing grease showed a very bad condition of channelling both in the reduction gear case and crankcase. Very few of the grease passages were filled with grease, leaving the bearings that they fed in a dry condition. In fact, the only parts lubricated were the reduction gear teeth and the crankshaft bearings. This condition definitely determined that neither of these greases could be satisfactorily used as an air motor lubricant.

Gear Grease S.A.E.-160 and Dark Fiber Grease No. 4.

These two greases showed no signs of channelling in either the crankcase or reduction gear case and showed that satisfactory lubrication had taken place throughout. All grease passage holes were filled and the bearings to which they led had nice oil films.

Standardized Fiber Grease No. 2 and No. 3.

The condition of the crankcase after the eight-hour run with these greases resembled closely the condition found with F-9 Grease. All moving parts and bearings had excellent oil films on them. The cylinder walls and reduction gear teeth as well as its bearings showed that very good lubrication had taken place. All grease passage holes were filled and the bearings to which they led were well greased.

Sample Calculations

The brake horsepower output of the air motor is computed by use of the following derived expression:

Diameter of brake drum: 6.50 inches.
½-inch manila rope is used.

The actual rope brake formula assumes no flattening of the rope where it is in contact with the drum.

$$\text{B.H.P.} = \frac{d \times RPM \times F\left(\frac{d+a}{d}\right)}{33{,}000}$$

where

d = diameter of brake drum in feet;
a = diameter of rope in feet;
F = resultant scale force in pounds, spring scale reading minus weight "W."

Since the rope actually flattens in contact with the drum, thus decreasing the value of a, we may, without serious error, consider the term

$$\frac{d+a}{d}=1$$

The above equation then becomes:

$$\text{B.H.P.}=\frac{d\times RPM\times F}{33{,}000}$$

with the value for d put in, the equation in usable form is:

$$\text{B.H.P.}=\frac{\left(\frac{6.50}{12}\right)RPM\times F}{33{,}000}$$

Reference to test No. 8, using Grease F-9 and the fourth hour data, the computation of B.H.P. is shown below:

$$F=63.9 \text{ pounds}$$
$$RPM=216$$
$$\text{B.H.P.}=(.516\times 10^{-4})\ 63.9\times 216=0.710$$

The computations for the grease retention tests are very simple, being merely addition. Test No. 8 is shown computated below:

Weight Wiped off Grams	Weight Lost Through Exhaust (Grams)	Cumulative Total Weight of Grease Lost (Grams)	Cumulative Total Per Cent of Charge Lost
2.4	2.4	$2.4+2.4=4.8$	$\frac{4.8}{454}\times 100=1.1$
1.6	1.3	$2.4+2.4+1.6+1.3=7.7$	$\frac{7.7}{454}\times 100=1.7$
0.3	0.5	$2.4+2.4+1.6+1.3+0.3+0.5=8.5$	$\frac{8.5}{454}\times 100=1.9$
0.1	1.6	$2.4+2.4+1.6+1.3+0.3+0.5+0.1+1.6=10.2$	$\frac{10.2}{454}\times 100=2.3$

The columns of weight wiped off and weight loss through exhaust are obtained from weighings.

The column "Cumulative Total Per cent of Grease Lost" is computed for any hour by adding together all the values of weights of grease found in the two columns mentioned before, preceding and including that hour.

Cumulative total per cent of charge lost is found by dividing the values in the latter column by 454 (the original charge) and multiplying by 100.

Correct lubrication of all pneumatic tools can be attained only by the use of proper lubricants and the proper method of applying them. Since it is definitely our problem to supply grease and not oiling or greasing devices we have investigated only the lubricating characteristics of greases. The lubrication of all pneumatic tools is indeed a serious problem because this equipment is subject to very rough treatment, both by the operator and owner. Operators forget to oil the tools during the shift, allowing them to run dry and the owners will allow them to remain "dry" during idle periods. The correct types of lubricants are not always chosen and improper means of applying may be used.

Compressed air lines and storage tanks always contain much water, both in the precipitated and vapor form, that is brought into the compressor by the air and precipitated when the compressed air cools in the lines and tanks. Most of the air line water blows out through the exhaust, yet some passes down the cylinder walls by the pistons, and into the crankcase and reduction gear case. If the grease has emulsifying tendencies toward the formation of oil-in-water types, it will not be readily held on the cylinder walls, thus causing a lack of lubrication.

The effect of the water on the grease in the crankcase is such that its fibrous texture is destroyed. The grease swells, thus decreasing its lubrication value and forces itself out through the stuffing boxes and exhaust.

Air motors operate under very steep temperature gradients. The crankcase temperatures reach 200° F., or more under extreme conditions, while it is common for chips of ice to blow out through the exhaust. Also, the temperature of the surroundings where motors are operating varies greatly both climatically and with the type of work being done. The steep temperature gradient makes it very necessary for the grease to lubricate well over a wide temperature range. It must reach the valve surfaces and tops of pistons where the temperature is below 32° F. and yet must possess the requisite body to form a tenacious oil film at temperatures in the neighborhood of 200° F.

Dust, grit, sand and many other minute abrasive particles are drawn in by the intake of the compressor; these, coupled with the rust, scale and particles of metal from the compressor, tank and pipe lines, are carried into the air tool, thus contaminating the grease. This contamination not only increases wear due to its abrasiveness, but it adversely affects the formation of oil films. If a good grade of air cleaner is installed at the compressor intake and the air lines thoroughly "blown out" before attaching them to the motor, the harmful effect of these foreign substances as well as water will be greatly reduced.

Channelling of the grease will take place in the crankcase of a reciprocating motor or in the reduction gears if the grease is too hard. If the grease is too soft it will cause excessive leakage or will not cling to the high speed moving parts. The base for the grease must be carefully selected for different reasons.

Since water is always present in pneumatic tools it is considered by some that a soda soap grease is undesirable because of its tendency to form an oil-in-water emulsion. Lime soap greases are more resistant to the action of water upon them, yet they are affected by the severe agitation and the temperature which causes them to separate into their original oil and base phases. Aluminum base greases were not experimented with but from past experience with these greases it is believed that they would become "rubbery" under the agitation and temperature causing an excessive power absorption.

Velocities or moving parts in air motors are quite high for the type and size of machine. The motor used in this experiment has a crankshaft speed of 1332 R.P.M. and an average piston rubbing speed of 390 feet per minute for a chuck speed of 200 R.P.M. Other types of pneumatic tools have much higher speeds.

Reference to the data shows a very wide variation in the horsepower output of the air motor for a given pressure using different greases. The brake horsepower of the motor is equal to the indicated horsepower minus the friction horsepower and the power absorbed in agitating the grease.

$$\text{B.H.P.} = \text{I.H.P.} - (\text{F.H.P.} + \text{grease agitation H.P.})$$

By placing a very small amount of Motor Oil S.A.E.-10 in the crankcase, we reduce the power absorbed by grease agitation to very nearly zero. Because of the good oil films obtained with the motor oil it can be assumed that the F.H.P. reached a value as low as possible in this motor.

The lubrication afforded the air motor by each grease tested will be either as good as, or inferior to, the lubrication of motor oil, meaning that the power absorbed by friction will be either equal to, or greater than, that absorbed by the motor oil. The amount of power absorbed by increased friction over the minimum F.H.P. is referred to as excess friction horsepower, or F.H.P. excess. The power absorbed by agitating the grease in the crankcase is referred to as G.A.H.P. Therefore, it is obvious that the loss in power noted by the use of different greases is caused by that used in (F.H.P. excess) + G.H.P. The determination is made by finding the difference in ordinate of the curve for the grease under question, and the Motor Oil S.A.E.-10 curve, these values being shown in the tables. An inspection of these values will show how serious a problem this can be when selecting a grease. A gasoline engine usually shows a mechanical efficiency of about 85 per cent. From this, as a basis, a grease showing a loss of greater than 15 per cent to 20 per cent should not be considered as an acceptable lubricant.

The question of depreciation of the motor during the tests was considered: therefore, tests Nos. 1 and 8 were both run on grease F-9. No appreciable change in the motor was noted in either efficiency or retention data; this close duplication increased the value of the experimental results by proving the accuracy of the tests. This motor was purposely selected because of its poor mechanical condition presenting the worst condition for the grease to cope with.

A summary as to the benefits gained by correct lubrication are many; they may be tabulated as follows:

Higher output of the tool per pound of air pressure. This is caused by decreasing the frictional horsepower of the machine.

Reduced air consumption by providing an adequate oil seal between piston and cylinder. Also, the air consumption will be decreased as shown in the preceding paragraph.

Decreased wear on all moving parts, thus lessening the expense of replacements and operation.

Fewer "dry machines" during operating periods, and less rusting during idle periods.

The following information deals directly with the lubrication of pneumatic tools of various types and is considered of interest.

Normally, high pressure air for pneumatic tool use is compressed to about 100 pounds per square inch. This pressure cannot be considered high. However, the line pressure cannot be used as a criterion of the maximum pressures existing, because the pistons of air motors and other pneumatic tools are usually "air cushioned" so as to assist the deceleration of them at the end of the stroke. This high pressure not only has a tendency to break down the grease film by pressure alone, but an accompanying temperature rise is present. Under conditions of extreme "over-stroke," caused by grinding of a drill steel shank, or the small end of a piston rod due to unevenness, flames may shoot from the exhaust due to the extreme pressures created. Consequently, the most efficient lubricant is one that will adhere to the metal rubbing surfaces under the high pressures and temperatures.

U. S. Government Requirements for Pneumatic Tool Grease Stock Number L (14-G-640)

The following comparison of the properties of a typical sample of F-9 Grease, Standardized Fiber Grease No. 4, and the Navy requirements, indicate that neither comply in all respects. These requirements are quite peculiar in that they specify a grease containing from 23 to 29 per cent of soap, yet it must flow at 52 to 60° F.

	F-9 Smooth Soda Soap Grease	Stand. Fiber Grease No. 4	Navy Specifications
Mineral oil, per cent by weight	84.3	81.0	70 to 75
Soap, per cent	12.5	17.14	23 to 29
Ash, per cent	2.32	3.17	2.2 to 2.4
Loss on heating at 105° C.	*0.39	*0.52	0.5 to 1.8
Dropping point, Ubbelohde °C.	148	160	.....
Flow point	....	....	52 to 60
Penetration at 77° F., worked	246	....	.....
Penetration at 77° F., unworked	222	225	.....

* Results obtained when heating 10 grams of grease in a drying oven for five hours at 105° C

RAILROAD LUBRICANTS

Air Brake Cylinder Lubricants

Emery [158] has investigated the requirements of air brake cylinder lubrication and in 1906 developed a formula including the ingredients: water, sodium chloride, sodium soap, graphite, and slippery elm bark. In 1912, he [159] proposed a semi solid grease for air machinery, having a moisture-soluble surface and adaptable to being carried by air currents to the parts to be lubricated. It consisted of water, sodium soaps, glycerin, mineral oil and graphite. This product was, of course, quite similar to his invention, assigned to the Emery Pneumatic Lubricator Company [160] covering a lubricant consisting of an emulsion of mineral oil and graphite in a menstrum of soap and glycerin. Any water soluble soap was considered satisfactory but potassium soap was specifically referred to.

Fiber Grease, Air Brake Cylinder Lubricant

In many cases a normal sodium soap base fiber grease will be found entirely satisfactory for air brake cylinder lubrication. Such a grease should consist of 4.87 per cent sodium soap and 95.13 per cent of mineral oil, the formula being:

Soap	4.87
Tallow	87.27
Sodium hydroxide	12.73
150 S.U.V. at 100° F. pale lubricating oil	95.13

This grease may be made by any of the usual procedures for manufacturing fiber greases, and should comply with the following S.P. specifications:

SEMI-FLUID AIR BRAKE CYLINDER GREASE

USES:

For use in air brake cylinders.

MATERIAL:

Two grades of grease are covered by this specification.
(a) for steam line trains.
(b) for electric line trains.

REQUIREMENTS:

To be compounded of mineral oil and soda soap.

Mineral oil base to have a viscosity between 40 and 45 deg. Saybolt Universal at 210 degrees Fahrenheit for grade (a) and not less than 75 Saybolt Universal at 210 degrees Fahrenheit for grade (b). The grease must separate readily in a Sharples centrifuge when heated to approximately 150 degrees F.

Saponifiable fat base must be a pure animal oil.

The grease must just flow at 50 to 60 degrees Fahrenheit. Determinations to be made in manner similar to Cold Test, A.S.T.M. Method D97-22T.

The consistency must be such that the constituents will not separate after heating the grease to 250 degrees Fahrenheit and cooling to 60 degrees Fahrenheit.

[158] British Patent 14,263 (1906).
[159] U. S. Patent 1,020,893 (Mar. 19, 1912).
[160] U. S. Patent 1,035,868.

The grease shall contain no tar, asphalt, resin, resinous oils, soapstone, wax, talc, powdered mica, sulphur, clay, asbestos or similar filler.

A sheet of polished copper when covered with the grease must show no sign of attack in 48 hours.

Grade (b) must be black or pale yellow in color and stringy in consistency.

REMARKS:

All specifications for Semi-fluid Air Brake Cylinder Grease previously issued are hereby cancelled.

A useful air brake cylinder grease may be made on the following formula:

Ingredients	Per Cent by Weight
S.U.V. at 100° F. mineral oil	55.20
Aluminum stearate	2.50
Potassium oleate	7.50
Glycerin	19.50
Graphite	6.60
Water	9.00
	100.00

Analysis of UP Brake Cylinder Grease

This grease gave the following results:

Physical Tests:

Appearance	Semi-fluid smooth grease
Odor	Oil of mirbane
Color	Grayish black

Composition (per cent by weight):

Soap	9.74
Oil	54.80
Water	9.00
Free alkali	0.04
Glycerin	19.20
Graphite	6.61
Undetermined	0.61
	100.00

Ash (per cent by weight)	2.88
Acid insoluble (probably mostly silica)	3.52
Iron and alumina (as oxides)	25.33
Potassium (as potassium carbonate, K_2CO_3)	59.85
Potassium sulfate calculated from SO_3 determined (K_2SO_4)	10.76
Undetermined	0.54

Mineral Oil:

Gravity ° A.P.I.	23.9
S.U.V. at 100° F.	150
Color	Black
Flash, ° F.	345
Fire, ° F.	400
Pour point ° F.	Below −10

Fatty Acids:

Acid value (mg. KOH)	199
Iodine value (Hanus)	119
Dropping point, ° C.	48
Appearance	Dark brown

Locomotive Air Pump Lubricants

To obtain compressed air for actuating the air brakes on railway trains, most locomotives are equipped with air pumps, the cylinders and piston rods of which require adequate lubrication. Steam refined cylinder stocks have been frequently used for this purpose but in some cases a special grease is demanded. Such a product may be made on the following formula:

Locomotive Air Pump Lubricant No. 2

Formula

	Per Cent by Weight
Lard oil	9.0
Caustic soda	1.3
Heavy No. 130 Oil (S.U.V. 131 at 210° F.)	15.0
1000 Pale oil	0.6
Water	74.1
	100.0

Analysis

Water	74 per cent
Free alkali	.03 per cent

Conformity to Specifications

	Specification	Value for Lubricant No. 2
Water, per cent	80 max.	74
Sodium soap, per cent	12 max.	9.7 (approx.)
Oil, per cent	6 max.	15.6 (approx.)
Free caustic soda, per cent	1.0 max.	.03
Mineral oil, S.U.V. at 210° F.	125–130	131

Proposed Purchase Specifications for Locomotive Air Pump Greases (S. P. Company Sept. 18, 1934)

USES:

For lubrication of air cylinders of locomotive air pumps. It will be used in a cup.

REQUIREMENTS:

To be compounded of mineral oil and soda soap base.

Mineral oil to have a viscosity of between 125 and 130 seconds, Saybolt Universal at 210° F.

Soap base must be made of pure animal oil and caustic soda.

Grease to have the following approximate composition:

Moisture	Not more than 80 per cent
Sodium soap	Not more than 12 per cent
Oil	Not less than 6 per cent

A sheet of polished copper when covered with the grease must show no sign of attack in 48 hours.

Free caustic—not over 1.0 per cent.

Must not break down or show sign of separation while in storage.

Hot Box Coolants

Railway lubrication engineers have for many years known that when car journals ran hot and the waste packing smouldered or caught on fire, that the application of powdered sulfur, graphite, sulfurized fatty oils, and more recently sulfurized lead soaps, organic phosphates, chlorides and tellurides, would tend to make them run cool, no doubt due to their extreme pressure lubrication properties, and their ability to produce a smooth bearing surface by reason of their corrosive action or mild abrasive action.

In 1920, Gill [161] developed a cooling compound for hot journal boxes which consisted of 35.5 per cent corn oil, 0.89 per cent castor oil, 6.82 per cent caustic soda, 0.54 per cent lamp black, 0.89 per cent methyl salicylate and 55 per cent by weight water. Howe [162] has proposed the following cooling compound:

	Per Cent by Weight
Soft fine mica	50
Mineral oil	100
Sodium soap	30

Stevens [163] prepared an Anti Hot Box Oil consisting of mineral lubricating oil treated with and containing wood ashes.

A Texas petroleum residue, high in sulfur content, was marketed for some time as a hot box coolant. It had the following tests:

Percent ash	.086
Melting point (cube method)	290° F.
Melting point (ball and ring)	297° F.

Soda Base Dehydrated Railroad Greases

A complete discussion of grease lubrication of locomotive driving journals is given in the following sections. While in most cases in the past a cold made soda base grease has been preferred for this service, recently there have appeared on the market several dehydrated or boiled soda base greases which are giving satisfactory results for several of the largest railroads in this country.

The cold made journal compounds and rod cup greases often contain 7 per cent of water and in some cases as much as 15 per cent. Since these greases are subjected to temperatures in excess of 210° F. they sometimes steam and puff in such a way that the grease is excessively consumed. In some cases the water is evaporated from the grease leaving a hard, dry, crumbling material of poor lubricating value. This hard grease will not feed onto the bearing properly, with the result that an increase in the temperature of the bearing takes place and finally carbonization of the grease occurs.

The chief merit of the boiled railroad greases is that they do not contain

[161] U. S. Patent 1,423,103 (Aug. 2, 1920).
[162] U. S. Patent 1,586,087 (May 25, 1926).
[163] U. S. Patent 1,796,310 (Mar. 17, 1930).

water and are less subject to the action just described. They have high melting points and give satisfactory lubrication over a wide range of temperatures. Their chief disadvantage is that they are not readily molded into cakes in which form they are applied to the journals.

These greases are considered as having the largest proportion of soda soap in conjunction with the most viscous mineral oils. They do not find extended use outside of driving journal lubrication.

Dehydrated Driving Journal Compound

	Per Cent by Weight
Crude tallow, No. 2	38.00
Caustic soda, solid (17.37% 40° Bé. lye)	6.08
Asphalt base lubricating distillate, 80 vis./210° F.	16.92
California residuum (450 flash tar)	10.00
Steam refined stock, 600 Pennsylvania	29.00

PROCEDURE

The cold tallow is charged in a fire-heated kettle and the caustic soda solution run in at once.

The temperature is raised to 300° F. as rapidly as possible without boiling over the kettle, stirring continuously.

The mixture of tar and lubricating stocks is then started into the batch, very slowly at first.

After about two hours the temperature should be 500° F. and all of the oil mixture incorporated with the soap.

A temperature of 500° to 530° F. is maintained for a period of five hours, agitating throughout.

The grease is then drawn in shallow iron pans to a depth of about three inches. Rapid cooling gives a product of fine structure with a minimum of oil separation.

CHARACTERISTICS OF PRODUCTS

This product will maintain its shape at a temperature of 380° F. and is not completely melted at 450° F. The consistency is about 55 A.S.T.M.

Dehydrated Driving Journal Compound

Formula	Per Cent by Weight
Tallow, No. 2	38.12
Caustic soda, solid	6.48
Heavy asphaltic residuum	25.32
Cylinder stock, 600 Pennsylvania	25.32
Asphalt base cylinder stock	4.76

The same process as for No. 1 may be used. The melting point of this grease is 425° F. and it has a consistency of 40 to 45 A.S.T.M.

Van Gundy and Scanlin Journal Compound [163a]

Formula	Pounds	Per Cent by Weight
Flake caustic soda	432	7.21
Hard tallow	2680	44.66
Steam refined cylinder stock	2888	48.13

[163a] U. S. Patent 1,599,854 (Sept. 14, 1926).

The finished grease is said to analyze as follows:

	Per Cent by Weight
Soda soap	46.00
Free NaOH	.40
Glycerol	4.76
Steam refined cylinder stock	48.14
Water	.40

PROCEDURE

The caustic soda is dissolved in water and added to the tallow in a fire-heated kettle.

The soap is heated for several hours until the major portion of the water has been removed.

The cylinder stock is added and the temperature brought to 450° to 500° F. or higher and maintained for several hours. Intermittent stirring is carried on.

The grease is drawn into shallow pans.

L. A. Walker Journal Compound [163b]

Formula

	Per Cent by Weight
Paraffine base cylinder stock	49.00
Stearic acid	39.25
Caustic soda, solid	7.00
Water	4.75

The analysis of the finished material is given as:

	Per Cent by Weight
Cylinder stock	52.5
Sodium stearate	46.0
Free alkali	.5
Water	1.0

EQUIPMENT REQUIRED

These ingredients are compounded and mingled in an open top steam jacketed kettle having a capacity of not less than four times the bulk of the original batch of the ingredients so that the mass may not overflow the kettle due to saponification during the interval of reaction.

PROCEDURE

The stearic acid is first placed in the kettle and a suitable degree of heat is applied to melt it, but the temperature should not exceed 200° F. The cylinder stock is next added, at which interval the degree of heat remains the same; agitation is employed throughout.

The alkali in solution with the water is added last and a vigorous reaction of neutralization is set up; this causes an autogenous generation of heat whereby the temperature of the mass is raised to approximately 230° F. Throughout this interval of reaction water is expelled from the mass in dense clouds of steam. Reaction is further indicated and attended by an intense foaming and an increase in the bulk of the mass.

[163b] U. S. Patent 1,598,225 (Aug. 31, 1926).

The pressure of the steam in the jacket is brought to 110 pounds and the kettle contents to a temperature of 345° F. Cooking is continued until the batch indicates a tendency to draw away from the sides of the kettle, to ball up on the agitator which indicates that the process is completed. No method for removing the material from the kettle is indicated in the patent specifications.

Dehydrated Driving Journal Compound

Formula

	Per Cent by Weight
Lard oil, No. 1	45.00
Caustic soda, solid	7.88
Western lubricating distillate, 80 vis./210° F.	8.00
California residuum	16.00
Steam refined stock, 600 Pennsylvania	23.12

PROCEDURE

As a temperature of 500° to 530° F. is required to completely dehydrate this product it is found necessary to make use of a fire-heated kettle equipped with paddle agitation. The lard oil, distillate and caustic soda as 40° Bé. lye are charged in the kettle and heated to 300° F. while agitating. About one hour is required for this part of the process.

A mixture of the cylinder stock and tar is then run into the kettle over a period of about one hour, the temperature at the same time being raised to 400° F.

Heating and stirring is continued for one hour when the temperature should be 500° F.

A temperature of 500° to 530° F. is maintained on the batch for a period of three hours.

The batch is then cooled to 500° F. and is drawn into molds to a depth of about three inches in order to insure rapid cooling.

Medium Driving Journal Compound

Formula

	Per Cent by Weight
Hard tallow	39.60
Caustic soda, solid	8.30
Water	8.30
Paraffine base cylinder stock	43.80

The process for manufacturing this grease is the same as for No. 4.

Kaufman's Dehydrated Journal Greases

Kaufman found that by continued heating of sodium soap greases at high temperatures, the melting point was not increased, but the yields were appreciably better, thus permitting a higher oil content for a given consistency. Quoting from Kaufman: "The hydrocarbon or mineral lubricating oil and fatty ingredients are mixed in suitable proportion, depending on the quantity of these ingredients desired in the finished product, and are heated to a desired digestion or conversion temperature. During this heat treatment the oils are preferably continuously stirred. This temperature of treatment may range from approximately the melting point of the resulting

grease, as for example 400° F., or even lower, to upwards of 600° F. The heated mixture of oils is then maintained substantially constant at the desired temperature for a predetermined time, or until the mixture attains a desired degree of digestion or conversion, preferably determined by a progressive increase in the viscosity of the mixture. The digestion mixture is then saponified in the usual manner by the addition thereto of a suitable alkaline material such as caustic soda, lime or the like, either with or without prior cooling of the digested mixture."

Kaufman preferred to subject the mineral and fatty oil ingredients to heat treatment after mixing. He pointed out that it was also feasible that these ingredients could be separately subjected to heating, a digestion with subsequent mixing, while hot and prior to saponification. While Kaufman has not published his views as to whether polymerization, oxidation, or splitting off of water, has occurred during his process, he considered that new compounds were formed which increased the hardness of the finished grease. It was pointed out in the first edition of "Lubricating Greases," page 192, that the possibilities for producing greases of improved lubricating quality were quite promising when utilizing hydrocarbon stocks which had been appropriately oxidized at high temperatures and combining them with alkali.[164] Kaufman reports that it is likely that by heat treatment some oxidation of the hot mixture results from the contact with air, or the mechanical stirring of air into the mixture while heating. He found that a considerable increase in viscosity, and a resulting increase in the hardness of the finished grease, was accelerated by actually introducing air into the hot mixture, apparently by blowing. With the mixture at a temperature of about 485° F. the saponifying agent must, of course, be added very slowly with continued agitation of the kettle contents in order to avoid objectionable frothing and foaming over the kettle top.

The following examples show the formulae and process conditions for Kaufman's process as applied to the manufacture of Locomotive Journal Grease:

Grease Number:	1	2	3
S.U.V. at 210° F. of cylinder stock	151	163	220 (Distillate)
Percent cylinder stock	37.37	39.78	59.20
Percent paraffine distillate 372 S.U.V./100° F.	22.90	24.36	....
Percent hard tallow	29.20	26.10	31.88
Percent stearic acid	3.25	2.90	....
Percent flake caustic soda (98 per cent)	3.64	3.42	4.46
Percent water	3.64	3.43	4.46
Manufacture:			
Digestion period, hours to reach 485° F.	5	5	2
Maintained at 485° F., hours	7	11	14
S.U.V. at 210° F. of mixture before heating	70	73	105
S.U.V. at 210° F. after digestion	104	111	155
Hours to add caustic soda solution	4.75	5.25	5.25

[164] See also: Klemgard, E. N., "Lubricating Greases," 1st Ed., p. 176, regarding Hot Neck greases made from oxidized stocks.

Grease Number:	1	2	3
Analysis of Finished Grease:			
Free alkali	acid	acid	0.28
Water	0	0	0
Sodium soap	30.0	24.85	30.60
Mineral oil	59.95	66.22	62.54
Free fatty acid (oleic)	.42	.42	.14
Free neutral fat	.65	none	none
Undetermined	8.8	8.51	6.44
Melting point ° F.	370	376	368
A.S.T.M. unworked penetration	33	29	30
Modified A.S.T.M. unworked penetration (475 gram additional weight)	81	75	75

Kaufman [165] has reported other examples of his process for heat treating mixtures of fats and mineral oils prior to saponification in order to increase the yields of dehydrated grease. The following are typical:

Formula:	A	B	C
S.U.V. at 210° F. of cylinder stock	176	176	176
Cylinder stock	60.89	61.03	61.18
Hard tallow	32.07	32.15	32.22
Flake caustic soda	3.52	3.41	3.30
Water	3.52	3.41	3.30
Time of Heating Oil and Fat Prior to Saponification:			
Hours to heat to 485° F.	3	4	2½
Hours at 485° F.	31	26½	21¾
S.U.V. at 210° F. of Oil and Fat Mixture:			
Before heat treatment	99	100	100
After heat treatment	165	154	140
Properties of Final Grease:			
Melting point ° F.	348	366	337
A.S.T.M. penetration (unworked)	41	47	84
Modified A.S.T.M. penetration (475 grams)	86	105	176

ment the yields of grease are progressively greater (penetration less for essentially equivalent formulae). The following examples show in another way the results indicated above; that by heating the mixture of soap and oil for greater periods of time, finished greases of similar penetration, but of lower soap contents, may be manufactured:

	D	E	F
S.U.V. at 210° F. of cylinder stock	176	176	176
Per Cent by Weight			
Cylinder stock	59.03	67.89	71.86
Hard tallow	32.67	25.51	23.00
Flake caustic soda	4.15	3.30	2.57
Water	4.15	3.30	2.27
Time of Heating Oil and Fat Prior to Saponification:			
Hours to heat to 485° F.	3	5	3½
Hours at 485° F.	22	30	37½

[165] U. S. Patent 1,971,750 (Aug. 28, 1934).

S.U.V. at 210° F. of Oil and Fat Mixture:

	D	E	F
Before heat treatment	95	109	110
After heat treatment	133	187	210

Properties of Final Grease:

Melting point ° F.	384	386	381
A.S.T.M. unworked penetration	37	37	40
Modified A.S.T.M. penetration (475 grams)	89	86	89

Miller-Karns Journal Grease [165a]

These inventors produced a railroad journal grease in a steam jacketed kettle, from sodium stearate and cylinder stock, and containing less than 2 per cent of water. Their process was assigned to the Valvoline Oil Company.

Formula

Summer Grade

	Per Cent by Weight
Mineral oil cylinder stock (paraffine base)	52.18
Stearic acid	39.23
Granular sodium hydroxide	6.87
Water	1.72

Winter Grade

	Per Cent by Weight
Mineral oil	54.12
Stearic acid	37.64
Sodium hydroxide	6.59
Water	1.64

In making this grease an open top steam jacketed kettle was employed, having sufficient capacity to prevent the grease from overflowing. The stearic acid was first charged into the kettle, the oil run in and the temperature of the mixture brought to 200° F. The caustic soda is heated with the water to form a paste, and it is added while agitating the acid and oil mixture. According to the inventors: "Shortly after the addition of the alkaline paste, a very violent reaction takes place with a boiling and rising of vapors, and an autogenous generation of heat, the temperature of the mass being raised to approximately 230° F. During this reaction water, in the form of steam, is expelled from the mass. The heat is, preferably, turned off just prior to the addition of the alkaline paste. The agitation, however, is continued during the reaction of saponification. The mass of material greatly increases in bulk and rises toward the top of the kettle giving off, as previously mentioned, water in the form of steam. As soon as the reaction is completed, the bulk of the material begins to decrease and descend in the kettle and pull away from the sides. At this time the material is a relatively dry, spongy, or gummy mass. Heat with agitation is now applied,

[165a] U. S. Patent 1,973,537 (Sept. 11, 1934).

a temperature of approximately 350° F. being preferred, for the purpose of softening the compound to bring it into a smooth doughy form or homogeneous mass, in which form it may be caused to flow and be drawn from the kettle. Agitation is preferably continued through this period until the compound is removed from the kettle."

The average analysis of the Miller-Karns grease is:

	Per Cent by Weight
Sodium stearate	46.97
Free caustic soda	.44
Mineral oil	51.79
Water and undetermined	.80
Melting point	450° F.

Anhydrous, California Bright Stock Journal Grease

The following materials are heated together to a temperature of 380° F., and then poured into a mold to cool, pressure being applied to smooth the top of the grease cakes and to produce a more dense grease structure, with less tendency for oil separation.

Per Cent by Weight	Material		Cents
39.000	Stearic acid	39 pounds at 15.966c per pound	622.674
6.200	Flake caustic soda	6.2 pounds at 6.4c per pound	39.680
54.787	140 Calif. brt. stock	54.787 pounds or 6.97 gallons at 15c per gallon	104.550
.013	Lampblack	0.013 pounds at 25c per pound	.325
	Cost of materials for	100.000 pounds of grease	767.229
	Cost of materials for	One pound of grease	7.672
	Cost of compounding		1.000
	Total cost per pound of grease (expressed as cents)		8.672

Specifications	Typical Tests	
Percent oil (55½ to 56½)		55.6
Percent free alkali (not over 1 per cent)	0.20	0.05
Percent water (not over 1.25 per cent)	0.40	.20
Penetration at 77° F. (A.S.T.M. D5-25) (55 to 75)	75	61
Penetration after pressing	95	73
S.P. breakdown test (min. 320° F.)	OK	OK
Melting point (cube method; 470 to 485° F.)	475	475
Mineral Oil Tests:		
S.U.V. at 210° F.	126	138
Flash, o. c. ° F.	430	495
Fire, o. c. ° F.	530	590
Gravity ° A.P.I.	20.9	19.0

The following A.S.T.M. cone penetration data and needle penetrations show that the structure of a grease of this kind is considerably changed by pressing into sticks and cakes:

Cone Penetrations

Temperature, ° F.	Original Sample	Cake	Sticks
77	26	27	37
150	38		
212	37	60	81
250	70	128	195
300	199		
350	293	295	335
400	360+19	360+20	360+50
	Needle Penetrations		
77	62	80	93
150	143		
212	200	360+	360+
220	360+		

English Dehydrated Rod Cup and Locomotive Journal Grease (1932)

Formula	Per Cent by Weight
Tallow	42.5
Caustic soda (solid)	7.5
170 S.U.V. at 210° F. Naphthenic S. R. stock	50.0

The method of manufacture is the same for both greases, a normal size batch being 600 pounds.

The tallow and caustic soda are placed in an iron pan in the cold, the caustic soda being in the form of an aqueous solution containing about 40 per cent soda. The mixture is then heated by means of direct fire to 200° F; the fire is now turned out and the soap allowed to stand with occasional stirring for a couple of hours. The soap is now gradually heated to about 250° F. to 300° F., and maintained at that temperature until thoroughly dry, as evidenced by the powdery nature of the product. If the soap is heated to about 300° F., it tends to char and to melt, so the product should be closely watched at this stage. Mechanical agitation is preferable, but if not available, hand stirring may be resorted to. It is important that the soap should be thoroughly dried because the less moisture there is in the soap at this stage, the shorter will be the time required for the later stages. It takes approximately 6 hours to thoroughly dry the soap.

Having dried the soap thoroughly, about $\frac{1}{3}$ to $\frac{1}{2}$ of the total weight of oil required is added to the pan and the temperature is slowly raised to 500° to 530° F. with continuous stirring. Frothing occurs at this stage, but if the above method is followed closely the frothing will not present any difficulties. The capacity of the pan should be at least three times the capacity of the batch being manufactured.

After frothing has ceased, the remainder of the oil is added and thoroughly stirred in. At this stage, the mixture is heated to 530° F., the addition of the oil brings the temperature down to 490° F. (approx.), which is a satisfactory temperature for pouring. This operation takes about 15 minutes.

The finished product is now filled off at a temperature between 450° F. and 500° F. into shallow iron trays about 4 inches deep and allowed to cool, after which it is cut up into blocks of any desired size. After cooling, it should be allowed to stand for at least 48 hours before use. Four iron trays are used per batch, two having a capacity of 300 pounds each and two having a capacity of 200 pounds each.

The grease is filled direct from the pan into trays, the control valve being operated from the center of the pan. The first two trays may be filled at the same time by means of a two-way delivery pipe. The remaining two trays are filled separately from an extension arm fitted on one outlet of the two-way delivery pipe, the other outlet being stopped with a screw bung.

The preparation of the soap to the dry stage takes up Friday afternoon and Saturday morning. This includes the cleaning of the iron pot, etc. The first batch of oil is usually added about 10:30 a. m. on Monday morning, and this stage is not completed until 3 p. m. the same afternoon. The remaining oil is now added and the product ready to pour in 15 to 30 minutes. The times quoted are for a 600-pound batch and are, of course, approximate.

If the temperature exceeds 540° F., the glycerin formed during the saponification will boil. This alters the appearance of the froth on the surface of the molten grease, the individual bubbles composing the froth being very much larger than when the temperature is below 540° F. and the frothing is due to water alone.

Various Analyses

"PERLA" DRIVING JOURNAL AND ROD CUP GREASE

This grease gave the following results:

Physical Tests:	
Appearance	Black, semi-fibrous cake
A.S.T.M. unworked penetration	30
Dropping point ° C.	205
Odor	Fatty
Composition (per cent by weight):	
Soap	49.50
Mineral oil	43.00
Free fat	3.50
Free fatty acid (as oleic)	2.90
Fillers	None
Tarry matter (insoluble in P.E.)	.30
Water	.20
Loss and undetermined	.60
Fatty Acids from Soap:	
Neutralization value	208
Iodine value (Hanus)	18
Melting point ° C.	54
Color	Dark brown

Mineral Oil:

Color	Black
A.P.I. gravity	20.9
S.U.V. at 210° F.	170

Ash:

Total percent of original grease	8.55
Percent sodium carbonate	95.50
Percent calcium oxide	Trace
Loss and undetermined	4.50

Penetration tests at various temperatures on this grease show that above about 220° F. it becomes quite soft and might be consumed excessively in a hot journal box.

	77° F.	150° F.	212° F.	250° F.	300° F.	350° F.	400° F.
Cone penetration	26	32	37	70	199	293	379
Needle penetration	82	143	200	at 230° F. 360+			

ST ANHYDROUS JOURNAL GREASE

This product, while having a very high melting point was found to not feed properly in a test in a hot locomotive journal box.

Physical Tests:

Melting point (cube method) ° F.	475
Penetration at 77° F.	64
Breakdown test	OK

Composition:

Oil	50.8
Soap	49.5
Alkalinity (NaOH)	.4
	100.7

Fatty Acids from Soap:

Melting point ° C.	52.3
Neutralization value (mgms. KOH)	204.0
Iodine value	17.5
Probable composition	Stearic acid

Mineral Oil:

Gravity ° A.P.I.	20.2
Viscosity at 210° F.	119.0
Flash, o. c. ° F.	510.0
Fire, ° F.	590.0

Ash (Sodium carbonate 97.10 per cent) 9.73

DEHYDRATED PZ JOURNAL GREASE (1930)

This grease gave the following results:

Physical Tests:

Melting point (cube method) ° F.	460
Penetration at 77° F.	72
Breakdown test	OK

Composition (per cent by weight):

Soap	46.0
Oil	53.0
Water	1.0
Alkalinity (NaOH)	.5
	100.5

Fatty Acids from Soap:

Neutralization value	206
Melting point ° C.	54.4
Iodine value (Hanus)	11.0
Probable composition	Stearic acid

Mineral Oil:

S.U.V. at 210° F.	171
Flash, o. c. ° F.	520
Fire, ° F.	590
Gravity, ° A.P.I.	17.5

Ash	9.33
Sodium (Na_2CO_3)	93.1
Undetermined	6.9
	100.0

AS California Bright Stock Driving Journal Grease (1930)

This grease gave the following results:

Physical Tests:

Melting point (cube method)	455° F.
Breakdown test	OK
Penetration at 77° F.	71

Composition (per cent by weight):

Soap (sodium)	43.18
Oil	56.09
Water	00.22
Alkalinity (NaOH)	0.12
Fillers	0.50
	100.11

Mineral Oil:

Gravity ° A.P.I.	15.1
S.U.V. at 210° F.	176
Flash, o. c. ° F.	425
Fire, o. c. ° F.	500

Fatty Acids from Soap:

Iodine value (Hanus)	18.0
Acid value (mgms. KOH)	204
Melting point ° C.	60
Probable composition	Stearic acid

SNR JOURNAL GREASE (1931)

This product is known to be quite satisfactory in service.

Physical Tests:

Appearance	Brown grease
Odor	Soapy
Melting point, cube ° F.	475
Breakdown test	OK
Penetration (A.S.T.M. D-217-27T., cone at 77° F.)	43
Penetration (needle at 77° F.)	118

Composition (per cent by weight):

Insoluble matter	0.40
Soap	44.90
Oil	53.50
Water	0.50
Free alkali (as sodium hydroxide, NaOH)	0.28
Free fat (as olein)	0.80
Gain in analysis	0.38
Total	100.00

Ash (per cent by weight) 8.08

Sodium (as sodium carbonate, Na_2CO_3)	97.70
Undetermined	2.30
Total	100.00

Mineral Oil:

Gravity ° A.P.I.	23.5
S.U.V. at 210° F.	182
Color	Brown blk.

Fatty Acids:

Acid value (mg. KOH)	207
Iodine value (Hanus)	7
Dropping point ° C.	55
Color	Light brown
Probable composition	Stearic acid

S. P. Purchase Specifications for Driving Journal Compound (1925)

MATERIAL AND USES:

This grease to be used for locomotive driving box journals.

REQUIREMENTS:

The grease must be well manufactured and suitable in every way for the use intended. Under service conditions it must properly lubricate without excessive heating.

The grease shall be composed of soda soap (made from a good grade of tallow) combined with a well refined cylinder stock petroleum oil.

It shall be smooth and of uniform composition.

Soap Content: The soap content shall not be less than 40 per cent.

Grease shall not contain more than 1 per cent free alkali, calculated as NaOH.

The total moisture content shall not exceed 1.25 per cent.

The grease shall be properly aged by the manufacturer.

PHYSICAL TESTS:

A breakdown test of the grease shall be made by heating it in a closed tube to a temperature of 320° F. Closed tube shall be heated by immersing in an oil bath in which the temperature is raised at the rate of 10 deg. F. per minute. Grease must not show a marked puffing and must not fall off the suspension hook before the above temperature (320° F.) has been reached.

REJECTION:

Grease not meeting any of the above requirements or tests will be rejected.

All specifications previously issued for driving journal compound are hereby cancelled.

Cold Made Soda Base Locomotive Driving Journal Grease

The bearing pressure on locomotive axle boxes ranges from 100 to 300 pounds per square inch of projected bearing area. While this in itself is not a serious condition, the reversed thrust on journal boxes has a strong tendency to rupture the lubricating film, thus requiring that a very viscous lubricant be employed. It is estimated that ninety-eight per cent of the locomotives in America are using grease lubrication. The popular argument in favor of the use of grease on driving journals is that the bearings require less care and attention than when oil lubricated. To get the best results from grease the following points are of interest:

The brasses should be made from good bearing metal, homogeneous and free from all imperfections incident to poor foundry work. They should be bored to proper diameter and give a good fit.

The perforated screen grease retarder of the grease cellar must be made to conform exactly to the journal diameter and have full bearing against and over its entire surface. Otherwise the grease does not feed properly and becomes carbonized, resulting in a hot box.

The grease cake should be machine pressed to fit the cellar and not allowed to get too thin before renewal.

The driving journals of locomotives are responsible for a large amount of friction. Tests have shown that it is a relatively easy matter to supply a lubricant that will reduce this friction as shown by bearing temperatures, but it has also been demonstrated that such lubricants will not stand up when the driving journals are subjected to unusual loads, such as unequalized spring gear or wedges sticking, et cetera, so that it is necessary to pay some penalty in friction to insure against mechanical defects which occur frequently. It is said that railroads pay approximately three times as much for driving box and pin grease as for lubricating oil used.

The grease is sometimes blamed for hot boxes when the real cause is a slipping driving box lining.

It is difficult to get a grease that is just right. The grease must not get too soft when hot or it melts away, resulting in excessive consumption of grease and possible delay due to lack of grease and resultant hot boxes. On the other hand it must not be so hard as to carbonize and glaze on the surface as this means poor lubrication and results in a hot box. A happy medium is required.

The following grease has been used exclusively by one of the largest railroads in this country with very few journal failures.

Rod Cup and Driving Box Compound

Formula

	Pounds	Per Cent by Weight
Hard beef tallow, No. 1	800	42.75
Pennsylvania cylinder stock, 145 vis./210° F.	750	40.10
Flake caustic soda	160	8.55
Water	160	8.55
Lampblack	1	.05

PROCEDURE

The tallow, cylinder stock and lampblack are put in a steam jacketed kettle and brought to a temperature of 100° to 110° F.

The caustic soda and water are mixed together in a separate vat and cooled to 100° to 110° F.

The caustic soda solution is slowly run into the fat mixer, agitating continuously.

Agitation should be continued for five to twenty minutes depending on the acidity of the tallow which should be approximately 2 per cent free fatty acids calculated as oleic.

When the grease begins to thicken slightly it should be drawn out of the kettle into wooden barrels.

Driving Journal Compounds

Formula

	Per Cent by Weight
Tallow, No. 1	38.00
Flake caustic soda	7.60
Water	7.60
Lampblack	.02
Western cylinder stock, 140 vis./210° F.	46.76

PROCEDURE

The fat, oil and lampblack are thoroughly mixed in the kettle by melting the tallow and running in the cylinder stock. This mixture is then cooled to 90° to 95° F. and the caustic solution at this temperature is run in continuously for a period of about thirty minutes.

Cooling water is run through the jacket of the mixer during the entire process.

After the caustic has been added, slow agitation is continued for fifteen minutes, the temperature increases to about 120° F. and the batch is drawn.

While standing in the containers the temperature may increase to as much as 170° F.

Driving Journal Compounds

Formula

	Per Cent by Weight
Tallow, No. 1	40.00
Flake caustic soda	8.00
Water	8.00
Steam refined stock, 650 Pennsylvania	37.40
Pale oil, 100 Pennsylvania	6.60

PROCEDURE

The same procedure is used as for No. 2.

Greases Nos. 1, 2 and 3 have an A.S.T.M. penetration of 30 to 40 at 77° F. and a melting point of 450° F.

Heavy Driving Journal Compound

Formula

	Pounds	Per Cent by Weight
Acidless tallow oil	784	49.00
Western cylinder stock	511	32.00
Caustic soda, 50° Bé. solution	304	....
Water	...	9.5
Flake caustic soda	...	9.5

Stir the tallow and cylinder stock together for ten minutes at 95° F.

Add the caustic soda solution and draw at once 75 to 100 pounds of the mixture and pour it back into the kettle.

When the grease begins to thicken draw into wooden barrels.

Van Gundy Sodium-Lead Journal Grease [165b]

The formula for this product is:

	Per Cent by Weight
Tallow	40.00
Sodium hydroxide	8.00
Lead soap	10.00
Mineral oil (cylinder stock or asphaltic flux)	32.75
Water	9.25

Manley's Locomotive Driving Journal Box Grease [165c]

Manley has assigned to the Texas Company his patent for the following formula:

	Per Cent by Weight
Tallow	49.00
Sodium hydroxide	9.04
Water	9.04
Penn. cylinder stock	16.26
Asphaltic base residue	16.26
Graphite	.40

Sheasley Journal Box Grease [165d]

Prior to 1916 it had been the practice to ship solid journal compounds in large masses packed in barrels or other containers. When it was desired for use, it was taken cold and forced into a suitable mold for giving it the desired shape, or it was remelted and cast into a mold with the proper form to fit a locomotive driving journal. Both of these methods were open to serious objection, and grease presses were developed to form the grease cakes. In the former methods, the structure of the grease was altered and its lubricating properties impaired. Sheasley considered that greases for locomotive journal lubrication should contain as much mineral oil as possible. It, of course, must have sufficient consistency to be durable and resist excessive consumption. In normally manufactured cold sett journal greases water was frequently forced from the grease during pressing opera-

[165b] U. S. Patent 1,477,611 (Dec. 18, 1923).

[165c] U. S. Patent 1,550,912.

[165d] U. S. Patent 1,180,547 (Apr. 25, 1916).

tions. This water, carrying sodium hydroxide, caused a residue to form on the surface of the cake which, in some instances, interfered with proper lubrication. Sheasley objected to cold molding processes. He developed a process in which mineral oil was mixed with hard soap stock, such as tallow or the derivatives thereof, capable of being converted into hard soap, and the mixture was then treated with caustic soda for saponifying the soap stock, the alkali being in excess. The mixture was then cast into a suitable mold before the reaction of saponification was completed. There resulted a hard, tough, durable, homogeneous molded cake of lubricant possessing suitable wear resisting characteristics.

Sheasley mixed mineral oil with tallow at 115 to 125° F. and after stirring cooled the mixture to 105° F., when the caustic soda solution was sprayed into the fat mixture to effect saponification. The quantity of sodium hydroxide employed by Sheasley was approximately 50 per cent greater than theoretical requirements of the soap stock. His formula was:

	Pounds
Tallow	14.0
Mineral oil	9.7
Caustic soda	3.0
Water	3.0

The mixed grease was then poured into individual molds where saponification was completed. The temperature in the grease cakes may reach a value 175 to 250° F. The molds are preferably of pasteboard, or fiber, since such a mold is readily removed from the cake.

English No. 5 Yellow Locomotive Grease

Formula

	Per Cent by Weight
200 S.U.V. at 100° F. Mexican spindle oil	20.0
Tallow fatty acids	17.0
Palm oil	10.0
Sodium hydroxide	2.0
Water	51.0
Unworked A.S.T.M. penetration	158
Melting point ° F.	110

Aging of Locomotive Rod Cup Greases

Most all wet type rod cup greases become progressively harder over a period of about two weeks. Beyond this period the interior portions of the grease do not change markedly due to continuation of the saponification process. But, the surfaces may turn white due to the formation of sodium carbonate and other salts left by the evaporation of water and solutions from the mass of the grease. The following asphalt needle penetration tests (100 grams, 77° F., 5 seconds) for two typical rod greases over a period of two weeks, indicate the extent of change in consistency which takes place:

Formulas			
Per Cent by Weight		Per Cent by Weight	
40	Tallow	40	Tallow
43	S.R. stock	28	S.R. stock
		15	Dark petrolatum
8.08	Caustic soda	8.08	Caustic soda
8.92	Water	8.92	Water
Days	Penetration Tests		
1	169		174
2	159		163
3	147		152
4	140		144
5	136		140
8	132		134
10	130		133
12	129		131
14	129		130

Various Analyses

ST WET JOURNAL GREASE

This grease gave the following results:

Physical Tests:

Penetration at 77° F.	360+
Breakdown test	Puffing, not O.K
Melting point (cube method)	480° F.

Composition (per cent by weight):

Oil	42.4
Soap	41.0
Water	10.2
Free alkali (NaOH)	2.8
Filler and insoluble matter	.5
Undetermined	3.1
	100.0

Fatty Acids from Soap:

Neutralization value	204
Iodine value	48.6
Melting point	53.8
Probable composition	Tallow

Mineral Oil:

Gravity	25.3
Flash	58.0
Fire	665
S.U.V. at 210° F.	214

Ash, per cent 12.3

Sodium (Na_2CO_3)	95.2
Undetermined	4.8
	100.0

AS Rod Cup Grease (1929)

An unusual feature of this wet grease is its petrolatum content.

Physical Tests:		
Appearance		Brown, solid lubricant
Odor		Soapy
Melting point ° C.		Not possible
Composition (per cent by weight):		
Soap (sodium)		51.16
Oil		34.50
Water		9.70
Free alkali (Na_2O)		1.12
Fillers		1.25
Undetermined		2.27
	Total	100.00
Ash, per cent		11.19
Sodium (Na_2CO_3)		96.3
Undetermined		3.7
	Total	100.0
Mineral Oil:		
Gravity ° A.P.I.		20.7
Melting point ° C.		50
Appearance		Dark soft petrolatum
Fatty Acids from Soap:		
Neutralization value		208
Iodine value (Hanus)		58.2
Melting point ° C.		46.0

PZ Wet Rod Cup Grease (1931)

This grease gave the following results:

Physical Tests:		
Appearance		Dark green, solid lubricant
Odor		Soda soap
Melting point, cube ° F.		470
Penetration (A.S.T.M.) at 77° F.		34
Composition (per cent by weight):		
Soap		49.8
Oil		36.7
Water		10.5
Free alkali (NaOH)		1.14
Fillers		0.1
Free fat		0.28
Undetermined		1.48
	Total	100.00
Ash, per cent		10.4
Sodium (as Na_2CO_3)		98.6
Undetermined		1.4
		100.0

Mineral Oil:

Gravity ° A.P.I.	21.5
S.U.V. at 210° F.	119
Melting point (petrolatum M.P. method) ° F.	93
Dropping point (Ubbelohde method) ° F.	34
Appearance	Dark green, black
Point point ° F.	90

Fatty Acids from Soap:

Neutralization value (mg. KOH)	205
Iodine value (Hanus)	51
Dropping point ° C.	44
Color	Dark brown
Probable composition	Mixture of stearic and oleic

GSO Railroad Greases (1931)

These greases have the following characteristics:

	Electric Gear Grease	Driving Journal Compound	Rod Cup Grease
Physical Tests:			
Appearance	Black, grainy solid	Brown grease	Brown grease
Odor	Asphaltic	Soapy	Soapy
Melting point, cube ° F.	.....	475	490
Breakdown test	.....	O.K.	O.K.
Composition (per cent by weight):			
Insoluble matter	15.0	0.2	0.2
Soap	11.0 as (Ca resinate)	45.0 as (Na oleate)	57.0 as (Na oleate)
Oil	67.0	52.0	29.0
Water	0.6	1.0	7.8
Free alkali	1.4 as ($Ca(OH)_2$)	0.7 as (NaOH)	1.1 as (NaOH)
Free fat (as olein)	0.0	0.0	0.5
Undetermined	5.0	.....	4.4
Gain in analysis	.....	1.9	.....
Total	100.0	100.0	100.0
Ash (per cent by weight):	2.74	8.95	12.56
Sodium (as Na_2CO_3)	.....	96.6	97.2
Calcium (as CaO)	76.7	.....	.....
Sulfur (as sulfur trioxide, SO_3)	14.5	.....	.....
Iron and aluminum (as oxides, R_2O_3)	3.0	.....	.....
Undetermined	5.8	3.4	2.8
	100.0	100.0	100.0
Mineral Oil:			
Gravity ° A.P.I.	16.0	23.5	28.0
S.U.V. at 210° F.	290	200	110
Color	Brown black	Brown black	Brown black

	Electric Gear Grease	Driving Journal Compound	Rod Cup Grease
Fatty Acids:			
Acid value (mg. KOH)	166	205	205
Iodine value (Hanus)	.....	11	49
Dropping point ° C.	.....	55	44
Color	Transparent Light brown	Dark brown	Light brown
Probable composition	Rosin acid	Stearic acid	Oleic and stearic acid

S.P. Purchase Specifications for Rod Cup Compound (1925)

MATERIAL AND USES:

This grease to be used for locomotive rod cups.

REQUIREMENTS:

The grease must be well manufactured and suitable in every way for the use intended. Under service conditions it must properly lubricate without excessive heating.

The grease shall be composed of soda soap (made from a good grade of tallow) combined with a well refined cylinder stock petroleum oil.

It shall be smooth, uniform and must not crumble under pressure.

Soap content: The soap content shall not be less than 45 per cent.

Grease shall contain not less than 0.5 per cent nor more than 2.5 per cent free alkali, calculated as NaOH.

The total water, glycerine and impurities present shall not exceed one-third of the total dry soap content.

The grease shall be properly aged by the manufacturer.

All specifications previously issued for rod cup compound are hereby cancelled.

Sodium Soap Railroad Journal Grease Patents

The following patent reviews are listed in chronological order, beginning with one granted in 1872 to Robinson.[166] He found that his sodium soap grease was consumed in railway journals to only one-fourth the extent of the normal tallow compounds which were in general use at that time. His formula was:

	Pounds
Palm oil	15
Mineral lubricating oil (neutral paraffine oil). Mixed well in grease kettle.	30
Tallow	40
Caustic soda. Heat and stir to 360° F.	5
Fine powdered asbestos. Heat to 470° F.	10

The grease was then cooled, and pressed to remove about 15 per cent of the oil and mixed with hair, waste, or jute, for packing car journals. Pitt's[167] journal compound was composed of crude petroleum, tallow, rosin, salt, and caustic soda solution. All of the ingredients, with the exception of the caustic soda, were mixed together and heated to 250° F. When cold, the caustic soda solution was stirred in.

[166] U. S. Patent 126,987 (May 21, 1872).
[167] U. S. Patent 193,280 (Apr. 12, 1877).
[168] U. S. Patent 923,304 (July 11, 1907).

Simons' [168] car journal grease was composed of the following ingredients:

	Pounds
Powdered graphite	50
White lead	50
Tallow	37
Sodium hydroxide	9

Durham and Wyatt,[169] in 1855, made lubricating greases for railway axles, which consisted of tallow, sodium soap, rosin, and water. Webster's [170] journal grease was prepared with tallow, palm oil, oleic and stearic acids, the entire mixture being incompletely saponified with sodium hydroxide solution. Bethell [171] boiled palm oil, powdered steatite, and sodium hydroxide solution together, and then added Iceland moss. Lubricating oil, fat, sodium soap, and water were the ingredients used by Brooman [172] in making railroad journal grease. Spencer,[173] in view of difficulties experienced with very hard cakes of grease giving inadequate lubrication, proposed the novel idea of boring a number of holes in the hard grease cake and filling them with a softer lubricant. The hard cake was composed of rosin oil, hard sodium soap, and stearin pitch. The soft lubricant used for filling the recesses in the hard grease cake, consisted of suet, fat, or other soft locomotive journal greases. Lubricating oil, graphite, beeswax, and tallow were boiled together with sodium hydroxide solution to produce a railroad journal grease in accordance with the process patented by French.[174] In one of Hornby's [175] processes fat, such as tallow, and pitch were heated to 330° F. to manufacture a journal grease. In another formula animal oil, vegetable oil, and caustic soda solution were mixed together at a temperature of 200° F.

Newton's [176] railroad grease was made with mineral lubricating oil, fat, such as tallow, and sodium hydroxide solution, its distinguishing characteristic being the addition of a certain proportion of palm oil pitch. In the Alcock and Johnson [177] railroad journal grease, sodium carbonate was preferred for the saponification of tallow rather than sodium hydroxide. Their mixture was heated in order to promote saponification, but it is doubtful that much soap was formed other than by neutralization of any fatty acid present. Woods [178] reclaimed fish oil and converted it to a sodium soap which served as a railroad lubricant. Datte [179] made grease consisting of 67 per cent mineral lubricating oil, 30 per cent oleic acid, and 3 per cent of sodium hydroxide. This grease was used to impregnate hair rope which

[169] British Patent 1,812 (Aug. 10, 1855).
[170] British Patent 1,703 (July 4, 1861).
[171] British Patent 2,031 (Aug. 15, 1861).
[172] British Patent 1,373 (June 2, 1864).
[173] British Patent 3,145 (Oct. 29, 1869).
[174] British Patent 1,415 (May 9, 1872).
[175] British Patent 3,445 (Nov. 19, 1872).
[176] British Patent 1,007 (Mar. 19, 1873).
[177] British Patent 168 (Jan. 12, 1882).
[178] British Patent 4,434 (Mar. 24, 1887).
[179] British Patent 18,295 (Aug. 8, 1912).

was employed as journal box packing. In 1906, Walker [180] suggested a locomotive journal grease composed of a hard cake with many small holes filled with a mixture of soft grease and graphite. Kaufman [181] has patented a journal grease consisting of heavy mineral oil and fatty oil, which are heated together at 400 to 700° F. to cause oxidation and other reactions which increase the yield of grease obtained by saponifying the mixture with sodium hydroxide. His grease still is provided with a condenser for recovery of volatile oils and fatty acids. Kaufman [182] has also patented a dehydrated locomotive journal grease having an A.S.T.M. unworked penetration value of less than 100 and a melting point of 350 to 400° F. It consists of 20 to 35 per cent of the sodium soaps of the high molecular weight fatty acids.

Hilliker [183] has assigned to the Standard Oil Company of Indiana a patent for a locomotive journal grease based on the following formula:

	Per Cent by Weight
Heavy black oil	5.9
Heavy steam refined cylinder stock (flash point 530° F.)	34.4
Hydrogenated fatty acids	40.5
48° Bé. sodium hydroxide	13.1
Lard oil	6.1

A loosely bound mass consisting of about 50 per cent by weight steam refined cylinder stock and 50 per cent dehydrated sodium soap, the mixture having a free fatty acid content of approximately 5 per cent, has been patented by Miller and Smith.[184] Taylor [185] has proposed a locomotive journal compound containing sodium, calcium, and aluminum soaps. Its formula is:

	Per Cent by Weight
Sodium soap	30 to 45
Calcium soap	3 to 15
Aluminum soap	3 to 15
Viscous naphthenic oil having a viscosity index of 0–30 (Dean and Davis)	25 to 65

Taylor [186] also patented a journal grease composition consisting of 95 per cent of oil and soap, and 5 per cent of glycerin, water and impurities. The ratio of soap to oil was 1:1, the soap content being about 47.5 per cent by weight.

STEAM CYLINDER COMPOUNDS

Acidless tallow oils, dissolved in properly refined steam cylinder oils, are ordinarily considered the best type of lubricant for steam cylinders. Other materials to reduce the drop size in hydrostatic lubricators (alkali or metallic soaps, triethanolamine oleate, etc.), or to increase film strength,

[180] British Patent 17,252 (July 31, 1906).
[181] U. S. Patent 1,971,750 (Aug. 28, 1934).
[182] U. S. Patent 1,966,821 (July 17, 1934).
[183] U. S. Patent 1,989,196 (Jan. 29, 1935).
[184] U. S. Patent 2,005,553 (June 18, 1935).
[185] U. S. Patent 2,038,689.
[186] U. S. Patent 2,038,688.

may be added in small proportions. Graphite and tallow mixtures were once considered the ideal lubricant for marine and industrial steam engine cylinders, but, with increased steam pressures and temperatures, the more normal steam cylinder oils have been generally adopted. It is not believed that the steam cylinder greases give any better lubrication than properly blended steam cylinder oils; however, since they were once important lubricants, and, in many cases, were found to contain sodium soaps, brief mention will be made of them for the sake of completeness:

In 1876, Spence [187] proposed a steam cylinder grease consisting of sodium soap, starch or flour, oxgall and glue or gums. Hunter [188] considered that a mixture of sodium carbonate, Irish moss and catechu, boiled together in water, was a suitable lubricant for the steam engine cylinders of his day. Parson [189] published a formula which included coal tar, vegetable oils, mineral oils, paraffine wax, free sodium hydroxide, sodium soap, and free tallow. In 1903, Schutz [190] favored the lubrication of carbon dioxide compressors and steam cylinders with simply a sodium hydroxide solution. It is certain there would be no corrosion due to free fatty acids, when using such a lubricant.

Page's [191] steam cylinder compound probably contained both sodium and aluminum soaps. It consisted of tallow, sodium carbonate, sodium chloride, aluminum sulfate, water, cocoanut oil, and camphor. Chapman [192] fed a grease to steam cylinders, which was prepared from graphite, sodium hydroxide, glycerol, starch, and water, previously heated together at 250° F. Rolyat's [193] boiler lubricant was composed of castor oil, paraffine wax and sodium carbonate.

Southcombe and Wells [194] developed a cooling and lubricating composition for steam engine cylinders. It was an aqueous emulsion containing 90 to 95 per cent of water together with the following base:

	Parts
Mineral lubricating oil	85
Oleic, stearic or palmitic acid	5
Rosin or abietic acid	3
Sufficient sodium hydroxide, potash, or ammonium hydroxide to render the above mixture alkaline.	

H Engine Tallow

The following is a typical formula:

	Pounds
Tallow	56
600 Penn. steam cylinder oil	50
Yellow ceresin wax	5.25
Caustic soda	0.75
Water	0.75

[187] British Patent 4,410 (Nov. 14, 1876).
[188] British Patent 2,855 (July 26, 1877).
[189] British Patent 8,583 (May 23, 1889).
[190] British Patent 22,066 (Oct. 13, 1903).
[191] U. S. Patent 790,308 (Sept. 22, 1904).
[192] British Patent 2,284 (Jan. 29, 1907).
[193] British Patent 20,252 (Aug. 30, 1910).
[194] British Patent 123,409 (Feb. 22, 1918).

Petrolatum Type Steam Cylinder Grease

The following analysis of a steam cylinder tallow compound, containing no soap, appears to be a mixture of about 6 per cent of tallow with petrolatum, the mixture being scented with oil of myrbane. This product has been marketed since 1932 with considerable success, although a special device is needed for feeding it.

Physical Tests:	
Appearance	Smooth, stringy
Color	Greenish red
Odor	Similar to nitro benzol
Dropping point °C.	48
Melting point, A.S.T.M. method for petrolatum, ° F.	126
Composition (per cent by weight):	
Oil and petrolatum	93.50
Saponifiable matter	6.00
Ash	0.03
Free alkali	None
Soap	None
Moisture	None
Undetermined	0.47
Total	100.00
Oil Extracted:	
Gravity ° A.P.I.	27.0
Color	Greenish red
Flash ° F.	470
Fire ° F.	545
S.U.V. at 130° F.	310
S.U.V. at 210° F.	75
Point point ° F.	125
Ash (per cent on original)	0.03
Iron (as Fe_2O_3)	100

SHOE LUBRICANT

A lubricant suitable [195] for application to shoe leather may consist of the following materials:

	Parts
Graphite	12
Chalk	13
Tallow	3
Soap	4
Beeswax	13
Essential oil	0.1
Water	4

TEXTILE LUBRICANTS CONTAINING SODA SOAP

It is not our purpose to present a complete discussion of textile lubricants; however, the following data pertaining to various oils and greases used by the textile trades will be found of interest. Leith [196] was able to

[195] U. S. Patent 215,018.

prepare a lubricant for wool fiber which was essentially an emulsion of lubricating oil, rosin, fatty acids, and caustic soda. It was used in spinning and carding wool fiber. Hutchinson [197] assigned to the Swan and Finch company his patent dealing with a compound of sodium soap, and wool fat refined to remove free fatty acids and impurities, dissolved in water. Cella [198] made use of a solution of sodium-castor oil soap in lard oil for oiling textile fibers. In some cases he added water and glycerin to the foregoing mixture to produce an emulsion for textile use. Holden's [199] wool oil was made by mixing together turpentine, quilla, sodium carbonate, oleic acid, lard oil, cotton seed oil, palm oil, and olive oil, and treating this mixture with caustic soda. Raabe's [200] textile oil contained olive, rape, and castor oils together with olein and Iceland moss, this mixture being treated with caustic soda solution. By incorporating water with the foregoing oil Raabe [201] produced emulsions for use in spinning and carding. In 1900, Cella [202] suggested the use of the soda soaps of castor oil dissolved in mineral oil as a textile lubricant.

A patent was granted to Stockhausen,[203] in 1904, for a textile lubricant containing the sodium soap of Turkey Red Oil. This material later became a very popular ingredient of textile oils. Stockhausen's lubricant contained, in addition to the soda soap of sulphonated castor oil, fatty oils, mineral oils, fatty acids and carbon tetrachloride. In 1904, Boleg [204] prepared water soluble vaseline by incorporating with petrolatum 10 to 15 per cent of water soluble mineral oil. Ploeg [205] prepared soluble oils by partially saponifying fatty oil with caustic soda and then adding ammonia to maintain the desired alkalinity. Riep and Bauer [206] favored the use of solutions of sodium castor oil soaps as textile lubricants. Koster [207] made emulsifying oils by heating a derivative of an organic compound and a sodium salt of a higher fatty acid with water and mineral oil. He also used amides of higher fatty acids, glycerin, and stearamid in particular. Textile oils were also prepared by dissolving the sodium soaps of unsaturated fatty acids which had been heated at high temperatures to alter their structure, in mineral oil.[208] Koster [209] produced stable emulsions of oils and fats by heating them with an acidyl of an aromatic base and a salt of a higher fatty acid. In some cases, he added sodium salts of fatty acids and glycerol. Stockhausen [210] made soluble oils by mixing hydrocarbon oil

[197] U. S. Patent 131,345.
[198] U. S. Patent 661,473.
[199] U. S. Patent 704,939 (Apr. 24, 1901).
[200] U. S. Patent 720,922 (Nov. 20, 1902).
[201] British Patent 21,072 (Nov. 21, 1900).
[202] British Patent 21,073 (Nov. 21, 1900).
[203] British Patent 7,491 (Oct. 2, 1900).
[204] British Patent 13,983 (1904).
[205] U. S. Patent 805,433 (Mar. 8, 1904).
[206] U. S. Patent 884,878 (Apr. 27, 1905).
[207] British Patent 7,231 (Apr. 5, 1905).
[208] U. S. Patent 867,141 (May 2, 1906).
[209] British Patent 9,766 (1907).
[210] U. S. Patent 875,665 (Aug. 30, 1907).
[211] U. S. Patent 931,520 (Oct. 1, 1908).

chlorides with Turkey Red Oil or other sulphonated oils. He also saponified the sulphonated oils with 6 per cent of caustic soda in some cases and mixed them with the foregoing chlorides.

Dath [211] proposed a compound for dressing textile stocks consisting of 10 per cent of sodium soap, 15 per cent oil, 10 per cent glycerol, 2 per cent ammonium hydroxide, and 63 per cent water. Melamid [212] investigated the use of resinous bodies derived from coal tar, and treated them with sodium hydroxide, as ingredients of soluble oils intended for textile lubrication. Becker [213] has assigned to the Standard Development company a patent dealing with a lubricant suitable for use on artificial silk fibers and consisting of mineral lubricating oil containing a small proportion of a water soluble soap such as sodium stearate or sodium oleate. Fuchs' [214] textile oil consisted of an oil-water emulsion containing alkylated cellulose, and an alkali metal salt of an aromatic sulphonic acid containing several hydrocarbon residues as side chains. British Celanese [215] have patented mixtures of fatty oils, mineral oils, sodium acetate, and ethylene glycol. Pressel [216] has patented the following composition:

	Per Cent by Weight
Mineral oil	60
Fatty oil	33
Soap of sulphonated castor oil	5
Glycerin	2

The above compound is mixed with an equal volume of water to obtain a lubricant for textile fibers.

British Celanese [217] have developed the use of di-, or polyhydric alcohol esters in which a part of the hydroxy group is esterified with a higher fatty acid. Examples include glycerol mono oleate in conjunction with sodium, potassium, or triethanolamine soaps of castor, cocoanut and neatsfoot oils together with lecithin. In preparing emulsions for fiber lubrication, dispersion agents for soaps may be obtained by esterifying unsaturated aliphatic alcohols, having at least eight but not more than twenty carbon atoms, with sulfuric acid and then halogenating.[218] Becker [219] has proposed a lubricant for textile machine bearings consisting of lubricating oil of 50 to 250 S.U.V. at 100° F. containing 0.15 to 1.5 of sodium or other alkali metal soap. The sodium salts of dodecyl alcohol sulphuric acid esters have been proposed as ingredients of textile lubricants.[220] Kenney [221] has developed the formula for a textile oil which requires the use of low viscosity mineral oil, oleic acid, and the alkali (sodium) soap of oil soluble sulphonic acids

[211] U. S. Patent 941,361 (Apr. 7, 1909).
[212] British Patent 134,223 (Oct. 31, 1917).
[213] British Patent 277,637 (Sept. 14, 1926).
[214] U. S. Patent 1,728,118 (Sept. 10, 1929).
[215] British Patent 365,621 (Oct. 15, 1930).
[216] U. S. Patent 1,771,347 (July 22, 1930).
[217] British Patent 380,042 (Mar. 30, 1931).
[218] British Patent 400,986 (May 18, 1931).
[219] U. S. Patent 1,789,331 (Jan. 20, 1931).
[220] British Patent 392,027 (Aug. 4, 1932).
[221] U. S. Patent 1,871,889 (Aug. 16, 1932).

derived by the acid treatment of mineral oils. It is claimed that the product is particularly suitable for rayon oiling. In a new textile lubricant sodium soap is dissolved in a solvent such as the monoethyl ether of diethylene glycol.[222]

SOLUBLE OILS

Soluble oils are also known as emulsifying oils, since they are normally bright clear oils which, when mixed with water, produce milky emulsions. In some recent soluble oils the emulsion is so fine that instead of milky solutions in water, amber colored transparent solutions are formed. The soluble oils, when mixed in appropriate proportions with water, may be used as metal cutting lubricants, textile lubricants, and metal boring lubricants. In 1934, Butts [223] developed the following formula for a soluble oil intended for cutting operations:

	Parts
Lubricating oil	71–74
Castor oil	8¼–9½
Rapeseed oil	8–11
Sodium carbonate	0.6–1.25
Water as required	

In a boring oil proposed by Burak [224] sodium soap was dissolved in cyclohexanol and olein. Frieg [225] found that for shearing rubber the following mixture was useful:

	Per Cent by Weight
Tallow (containing 1 per cent NaOH and ¼ per cent of H_2SO_4 solution)	15
Vegetable oil (plus NaOH and H_2SO_4)	15
Water	70

Gay [226] has assigned to the Standard Oil Company (Calif.) a patent for a mixture of mineral oil and 10 to 95 per cent of an oil soluble alkali (sodium) salt of petroleum sulfonic acids. The emulsifying oil developed by Adams,[227] in 1932, consisted of lubricating oil, free liquid fatty acids, and an alkali metal soap of the acids derived from oxidized mineral oil. Previously, Adams and Kittrell [228] considered that for certain purposes the free fatty acids were not needed in this formula. Johnson [229] found that the alkali salts of water soluble green sulphonates from petroleum acid sludges, together with water and fatty oil, produced good emulsifying oils.

Sullivan's [230] soluble oil consisted of mineral lubricating oil and water, with the sodium soap of oxidized paraffine wax as the emulgator. A soluble oil consisting of mineral oil, a sodium soap, and a blending agent, such as the ether derivative of a polyhydroxy alcohol which retains a free hydroxyl

[222] U. S. Patent 2,025,435.
[223] U. S. Patent 1,979,250 (Nov. 6, 1934).
[224] British Patent 357,128 (June 5, 1930).
[225] British Patent 391,484 (Oct. 26, 1931).
[226] U. S. Patent 2,036,470.
[227] U. S. Patent 1,871,940 (Aug. 16, 1932).
[228] U. S. Patent 1,817,599 (Aug. 4, 1931).
[229] U. S. Patent 1,795,491 (Mar. 10, 1931).
[230] U. S. Patent 1,773,123 (Aug. 19, 1930).

group (the derivative being soluble in both oil and water), has been proposed by Merrill.[231] Merrill [232] also developed the use of mixtures of aliphatic alcohols, mineral oils, and sodium naphthenic acid soaps as soluble oils. Bennett [233] has claimed the process for making emulsions of water and carnauba wax in which sodium and/or potassium stearate is employed as the emulgator. Dimmig [234] has conceived of the idea of dispersing sodium resinate in lubricating oils using phenol as a mutual solvent. Wade's [235] soluble oil consisted of mineral oil, and the alkali metal soaps of sulphonated fatty oils together with an antioxidant. Frizell and Stagner [236] proposed the use of the alkali metal naphthenates; the naphthenic acids being derived from petroleum distillates and were considered by these investigators to be the oxidation products of hydrocarbons of the naphthene series, C_nH_{2n}. As an example, they gave hexahydrobenzoic acid, $C_6H_{11}COOH$. Their soluble oil formula was:

	Volume
Cresol	1
Sodium naphthenate	4
Mineral oil as required.	

Klever [237] observed the beneficial effects of the alcohols of high boiling point in improving the emulsifying characteristics of soluble oils. In his products the sodium soaps of castor oil fatty acids, hemp seed, palm, rape, cottonseed and olive oils, abietic acid, were employed in conjunction with the higher boiling alcohols, such as glycerin, and mineral oils. van der Ploeg [238] patented an oil-in-water emulsion stabilized with a mixture of an alkaline base (quinolin or pyridiene) with caustic soda. Thompson [239] oxidized a mixture of mineral oils and crude rosin oil, treated the oxidized mixture with sodium hydroxide, and further oxidized it with air at 105° C. and then combined it with ceresin wax, paraffine wax, and in some cases other materials, utilizing a mill to effect dispersion. Boleg's [240] soluble cutting oils were composed of mineral lubricating oil, in which was dispersed the sodium soap of rosin oil. Dyson and Gaskell [241] prepared a water soluble emulsifying oil by mixing petroleum with a fatty acid or a triglyceride, such as oleic acid, olein, stearic acid, or stearin, sodium or potassium hydroxide, and heating. Lean's [242] emulsified oil, consisting of sodium soap, mineral oil and water, was limited with respect to the free alkali content which was permitted to reach a "trace" as a maximum. Blackie's [243] aqueous emulsion contained mineral lubricating oil, isinglass

[231] U. S. Patent 1,739,686 (Dec. 17, 1929).
[232] U. S. Patent 1,695,197 (Dec. 11, 1928).
[233] U. S. Patent 1,637,475 (Aug. 2, 1927).
[234] U. S. Patent 1,621,483 (Mar. 15, 1927).
[235] British Patent 303,841 (July 6, 1927).
[236] U. S. Patent 1,582,257 (Apr. 1, 1922).
[237] British Patent 204,906 (1908).
[238] British Patent 7,699 (Apr. 11, 1905).
[239] British Patent 13,473 (June 16, 1903).
[240] British Patent 12,349 (June 13, 1899).
[241] British Patent 23,539 (Nov. 25, 1899).
[242] British Patent 1,698 (Jan. 23, 1909).
[243] British Patent 3,466 (Mar. 11, 1886).

solution, sodium soap, and paraffine wax. Lundy [244] mixed olive and rosin oils with sodium hydroxide, and added sodium tungstate, silicate, phosphate and magnesium chloride.

Burak [245] has given a discussion of cutting oils, pointing out that large amounts of free oleic acid in metal cutting and boring oils which are emulsified with water often causes iron parts to rust. He observed that if olein were added to soaps, or mixtures of soap substitutes and alcohols of high molecular weight, such as cyclohexanol, or homologues of cyclohexanol, that the emulsifying and fat dissolving action of such combinations was greatly increased, in accordance with the results of the following experiments:

1. Thirty parts of potassium oleate will dissolve 3 parts of mineral oil; but, 30 parts of potassium oleate and 7 parts of olein will dissolve 20 parts of mineral oil. Thirty parts of potash soap and 2 parts of cyclohexanol will dissolve 5 parts of oil. Furthermore, Burak found that 30 parts of potash soap, 7 parts of olein, and 2 parts of cyclohexanol would dissolve 32 parts of mineral oil to form a clear mixture, which represents an excess of 52 per cent compared with the numerical, additive effect of these two constituents.

2. As previously stated, 30 parts of soap will dissolve 3 parts of mineral oil. Burak noted that 30 parts of soap and 5 parts of olein would dissolve 10 parts of mineral oil. Thirty parts of soap and 2 parts of cyclohexanol will dissolve 5 parts of oil; however, 30 parts of soap, 2 parts of cyclohexanol, and 5 parts of olein will dissolve 20 parts of mineral oil—a gain of 90 per cent over the anticipated effect of these agents. This investigator pointed out that in order to emulsify or dissolve a certain amount of fatty acids, hydrocarbons, chlorinated hydrocarbons, carbon disulfide, pyridine, decahydronaphthalene, oil of turpentine, or mineral oils, a much smaller quantity of alcohol and olein is required if cyclohexanol is present.

As examples of Burak's formulae, the following are quoted: "Thirty-five parts of commercial soft soap are dissolved, as usual, in water or spirit and under constant stirring; mixed with 30 parts of spindle oil, 10 parts of decahydronaphthalene, and 50 parts of turpentine; whereupon, 8 parts of olein, and 1 part of higher alcohols, such as cyclohexanol, are added. The resulting emulsion is absolutely clear and can be mixed with any proportion of water to a *transparent* solution."

"Thirty-two parts of soft soap are mixed with 60 per cent of organic compounds insoluble in water, such as oil of turpentine, heavy petrol, spindle oil, trichlorethylene, carbon disulfide, pyridin, fish oil, and the like; whereupon, 6 parts of olein, and 2 parts of higher alcohols, such as methylhexalin, are added. If this emulsion is mixed with water it results in a transparent solution."

"Twenty parts of olein are mixed with 62 parts of insoluble organic compounds of the kind mentioned in the foregoing example, and 2 parts

[244] British Patent 3,108 (Sept. 23, 1873).
[245] U. S. Patent 1,938,804 (Dec. 12, 1933).

of cyclohexanol, and 1 part of methylcyclohexanol, are added. The mixture is saponified with a sufficient amount of potassium hydroxide, wherein 5 parts of a hydroaromatic sulphonate are dissolved, so as to leave an excess of 6 parts of free olein. If this emulsion is mixed with water it results in a transparent solution."

It is agreed that of the liquids available for cutting lubricants water has the highest heat-absorbing qualities. It flows readily into contact with the tool-cutting edge and the surface being operated on, but due to its low viscosity it does not form a very satisfactory lubricating film where the chips grind over the lip of the tool. The fact that water, when used alone, often leads to rusting of the metal being operated on makes it unsuitable as a cutting lubricant.

In the class of lubricants known as soluble cutting oils a small amount of mineral oil is held in emulsion in water by means of potassium or sodium soaps or sulfonated oils. The amounts of water used to make emulsions with the soluble oils vary with the character of the work being done.

Potash Base Soluble Oil

Formula

	Per Cent by Weight
Blown rape seed oil	7.6
Prepared degras	6.6
Caustic potash, 40° Bé.	3.0
Lump rosin, grade W.W.	4.8
Red paraffine oil, 210 vis. at 100° F.	78.0

Soda Base Soluble Oil

PROCEDURE.

Oleic acid is neutralized with a 32° to 40° Bé. solution of caustic soda, and cooked until a heavy soap forms, when it may be clarified with wood alcohol. Mineral oil is then added to the soap thus formed. The purpose of the alcohol is to keep the oil clear and bright and to aid in producing a perfect emulsion when the oil is mixed with water. The finished oil should contain from 20 to 30 per cent of the soda soap. The mineral oil generally used in this type of product has a viscosity of from 100 to 180 at 100° F. In some cases this type of oil has a tendency to evaporate off a portion of the alcohol, which permits separation of the oil and water.

Borax Base Soluble Oil

Formula

Kidney rosin oil	1 gallon
Borax (dissolved in a sufficient quantity of water)	2 pounds
Red paraffine oil, 28° Bé.	12 pints

PROCEDURE.

The rosin oil is saponified with the borax solution and cooked until the water is evaporated. This type of soluble oil should have a slightly alkaline reaction and sometimes ammonia or potash are added in small quantities to maintain this condition. In some cases where hard waters are used containing calcium salts, curds of lime soap may be formed when the emulsion is being made. The addition of a small amount of soda ash (1½ to 1 pound to 50 gallons of water) is often of value.

Sulfonated Base Soluble Oil

Sulfonated oils are often used in the manufacture of soluble cutting oils. These oils are vegetable or animal oils which have been treated with strong sulfuric acid, and the excess of acid washed out and the oil neutralized with a small amount of alkali or ammonia. Castor oils are most satisfactorily sulfonated.

Formula

	Per Cent by Weight
Sulfonated oil (castor and corn oils)	13.5
Paraffine oil, 80 vis. at 100° F.	17.0
Caustic soda, 10° Bé. solution	4.5
Sulfonated castor oil	25.0
Water	32.0

PROCEDURE

The mineral oil and the sulfonated castor oil are stirred together in a kettle.

The caustic soda solution is then added and the temperature brought to 210° F., when the batch may be allowed to stand over night.

The sulfonated castor and corn oils are then mixed in. A sample may then be tested to determine whether or not the oil is perfectly soluble. If not, more caustic may be added.

If a sample on chilling does not show cloudiness or separation, the final water may be mixed in, completing the process.

Soluble Oil

Formula

	Parts by Weight	Per Cent by Volume
Soluble oil base		16.66
Lump rosin, grade N	110	
Oleic acid	120	
Water	50	
Caustic soda, solid	15	
Denatured alcohol	45 to 50	
Pale oil, 180 vis. at 100° F.		83.34

PROCEDURE

The rosin and fatty acid are heated together until complete solution takes place and then cooled to 100° F.

The caustic soda is then mixed with the water, cooled to 100° F. and added at once to the rosin mixture. This completes the process for making the soluble oil base, which should have a free fatty acid content of 28 to 33 per cent. This base may then be mixed with any suitable pale oil in the proportions of 1 part of base to 5 of oil.

Soluble Oil

Formula

	Parts by Weight
28 to 30° Paraffine oil	250
Rosin	22
Oleic acid	22
Caustic soda	3
Water	10
Alcohol	7

PROCEDURE

The procedure for the manufacture of this product is the same as for Oil No. 5 above.

Soluble Wool Oil

Inasmuch as the formulas for textile oils are very similar to those for soluble base cutting oils, the formula and process of manufacture of a few wool oils will be given.

Formula

	Parts by Weight
Paraffine oil, 28° to 30° Bé.	250
Oleic acid	40
Lard oil, No. 1	15
Caustic soda	4
Water	12
Alcohol	10

Soluble Wool Oil

Formula

	Parts by Weight
Pale paraffine oil, 100 vis.	300
Lump rosin, grade N	25
Prepared degras	25
Caustic soda, 35° Bé.	12
Water	4

Soluble Wool Oil

Formula

Base

	Per Cent by Weight	Per Cent by Volume
Vegetable castor oil	36.36	20.00
Caustic potash, lump	8.20	
Water	7.72	
Oleic acid	36.36	
Dynamite glycerin	11.36	
Asphalt base red oil, 235 vis. at 100° F.		30.00
Asphalt base red oil, 95 vis. at 100° F.		50.00

Special Wool Oil

Formula

	Per Cent by Weight
Vegetable castor oil	7.27
Caustic potash, solid	1.64
Water	1.55
Oleic acid	7.27
Crude glycerol	2.27
Asphalt base pale oil, 100 vis. at 100° F.	80.00

Fat Liquor Oil

Formula

	Per Cent by Weight
Sulfonated cod oil	35.00
Asphalt base red oil, 100 to 200 vis. at 100° F.	45.00
Soy bean oil (or other animal or vegetable oil)	20.00

Paste Cutting Compound

Formula

	Per Cent by Weight
Paraffine oil, 25° Bé.	20
Lard oil	5
Caustic soda, solid	2½
Water	33½

The lard oil and half of the paraffine oil are mixed together in a kettle at about 110° F.; the caustic soda solution at about 90° F. is mixed in gradually, and followed by the remainder of the paraffine oil. Water up to 30 parts may be further added to make a cheaper product.

Cutting pastes are, in general, used where the work in hand is large, and are of particular value when portable tools are employed. Under these conditions compounded cutting oils or other fluids are not utilized on account of high cost and difficulty of application.

TUNNEL BEARING GREASE

A very hard grease for the lubrication of shaft and tunnel bearings of ships where grease is used may be made by the following formula. This grease resists the high operating temperatures and washing effects of the water which may come in contact with it.

Formula	Pounds	Per Cent by Weight
Oleostearin	20	5.14
Raw degras	30	7.71
Grade F lump rosin	20	5.14
Caustic soda 30° Bé.	50	3.09
Kidney rosin oil	100	25.71
Red oil 250 vis.	200	51.41
Water	...	1.80

English Tunnel Bearing Grease

Formula	Per Cent by Weight
100 S.U.V. at 100° F. Mexican spindle oil	72.8
Tallow fatty acids	14.0
Rosin oil	10.0
Sodium hydroxide	3.2

This grease is a very hard, brown, cake, suitable for application to ship propeller bearings. It melts at about 200° F.

SODA BASE WOOL YARN GREASES

These greases are used for packing open type boxes and may be employed as carriers for No. 2 or No. 3 cup grease. In using them a layer of the wool yarn grease should be placed in the bottom of the box against the revolving shaft and a layer of the correct grade of cup grease placed on top of the yarn. As grease is used it may be replenished without disturbing the wool yarn packing.

The formula below gives a grease of about the consistency of No. 3½ Cup Grease; it is mixed with short strands of wool yarn or wool waste.

Formula	Per Cent by Weight
Caustic soda	3.21
Water	3.99
Tallow, No. 1	25.82
Pale oil, 100 vis.	60.00
Wool waste	5.50
American talc	1.48

The soda base fiber grease is first made by any convenient method and drawn while in a semi-fluid condition into a large flat metal vat, in which is placed the wool waste. The waste is mixed by hand with the grease and the resulting product allowed to cool.

Formulas for two other wool yarn greases follow:

Formula	Per Cent by Weight
Calcium oxide	2.02
Caustic soda	.64
Water	1.43
Tallow, No. 1	4.00
Lard oil, Extra No. 1	15.00
Pale oil, 180 vis. at 100° F.	72.02
Wool yarn	4.64
Oil of mirbane	.08

Formula	Per Cent by Weight
Caustic soda	2.72
Water	1.30
Tallow, No. 2	18.90
Pale oil, 180 vis. at 100° F.	67.08
Wool yarn	10.00

Simpson Wool Yarn Grease [246]

This complicated, mild abrasive form of lubricant consisted of:

	Pounds
Cocoanut oil	25
Lard	25
Tallow	25
Graphite	25
Tin crystals	2
Sodium carbonate	15
Sodium chloride	10
Yarn waste	10

RECLAMATION OF SOAPS FROM JOURNAL GREASES

Frasch [247] has developed a process for reclaiming mineral oil containing oleates. He decomposes the soaps with sulfuric acid, and permits the lubricating oil, with oleic acid in solution, to separate from the oil insoluble material. He then saponifies the fatty acid-oil mixture with sodium hydroxide at temperatures of 50 to 60° C., obtaining a grease relatively free from oil insoluble material.

S.A.E. CHASSIS LUBRICANTS CLASSIFICATION

In January 1934, the S.A.E. Lubricants Division suggested a classification for chassis lubricants, the No. 300, No. 400, and No. 500 greases being made with sodium soaps, the others being water resisting greases. This

[246] British Patent 12,967 (Sept. 24, 1887).
[247] U. S. Patent 1,651,688 (Dec. 6, 1927).

classification has not as yet been officially adopted and is reproduced below as indicating the trend towards chassis lubricating grease standardization.

Proposed S.A.E. No.	Special Property Required of the Soap and Oil Combination	Worked Penetration Min.	Worked Penetration Max.	Vis. of Mineral Oil Used Min.	Vis. of Mineral Oil Used Max.
100	Water resistant.	300	400	S.A.E. 20	S.A.E. 30
200	Water resistant.	300	400	S.A.E. 40	...
300	Resistant to separation, especially at elevated temperatures.	200	300	S.A.E. 20	S.A.E. 30
400	Short fiber grease, resistant to separation, especially at elevated temperatures.	150	300	S.A.E. 40	...
500	Long fiber grease, resistant to separation, especially at elevated temperatures.	150	300	S.A.E. 40	...
600	Resistant to hot water.	60[1]	150[1]	...	...

[1] Penetration numbers of 150 or less are specified in terms of unworked penetration instead of worked penetration.

Applications

Proposed S.A.E. No.	
100	For use as a general chassis lubricant, especially when application conditions interfere with the use of a lubricant made with an oil of higher viscosity.
200	For use as a general chassis lubricant wherever application conditions are suitable for the use of a lubricant made with an oil of high viscosity.
300 & 400	Some types of these greases may be used for lubrication of both wheel bearings and grease lubricated universal joints.
500	For use in grease lubricated universal joints which require a grease of the long fiber type to provide best lubrication.
600	For water pumps on automobile cooling systems, for use where the lubricant comes into contact with hot cooling liquid.

It is noted that the proposed Nos. 400 and 500 are identical in the properties specified in the above table except for the length of fiber in the grease; the difference between them being that the No. 500 is a special universal joint lubricant which is for use with universal joints that cannot be service lubricated with lubricant having the properties of No. 400.

It was suggested that particular attention be given to the classification Nos. 300, 400 and 500 for two general uses, namely wheel bearings and universal joints, with the idea of eliminating all of them if possible, thus making not over two classifications for the two uses, wheel bearings and universal joints.

It was also suggested that the classifications Nos. 100 and 200 be studied carefully to determine whether or not there is any real necessity for lubricants that have a softer consistency (higher worked penetration). If lubricants with a higher worked penetration are considered desirable, the question is raised whether they should be covered by a separate classification, or whether either, or both, can be modified to include these lubricants of softer consistency.

It is time for the refiners and lubricant manufacturers who are making automobile chassis lubricants suitable for use in present day cars to step out, tell the world what good chassis lubricants are and sell their products on

that basis. If there are any companies whose greases or semi-liquid lubricants do not altogether measure up to the requirements of the present, they should revise their line so that they too can sell their greases on the basis of quality.

This would take the play away from certain companies who buy their grease from established manufacturers in the oil business and then sell it to the public through advertising campaigns warning the motorist against every grease except their own.

Undoubtedly there is room for improvement in some of the chassis greases being offered to the motorist. However, there are many companies manufacturing excellent lubricants for the purpose.

STEERING GEAR GREASES

The fundamental ideal characteristics of steering gear greases are:

"Efficiency"—Must permit gears to operate at maximum efficiency at all driving temperatures.
"Wear"—Must protect gears from wear.
"Stability"—Must not be affected by any temperature ever occurring in a steering gear.
"Retention"—Must not leak excessively from the gear housing.

As the mechanical construction of the gear determines the type of lubricant which will be most satisfactory, a complete study should be made of the construction of different types of gears, of the number of each type in use, of the lubricant specifications of gear manufacturers and of automobile manufacturers, of the lists of lubricants approved by gear manufacturers and the temperatures existing in a gear. Special test equipment using gears and test methods have in some cases been developed for determining the "efficiency" and "retention" of lubricants over a temperature range from — 40° to 180° F. "Wear" and film strength tests have been made. Complete analytical data from tests on lubricants have been obtained. In addition to a discussion of this data, the following topics will be discussed: Lubrication Problems of the Ross Gear; The Value of an E.P. Base in a Steering Gear Lubricant; and The Evaluation of Steering Gear Lubricants.

Within the last few years important improvements have been made in steering gear construction, improvements both in mechanical efficiency and in the lubricant retention of the gear housing. It is only natural that both the manufacturer and the user of these improved gears should desire a lubricant which does not detract from their efficiency. An examination of steering gear lubricants being marketed at the present time reveals that some oil companies are now trying to meet the new gear construction with a new lighter lubricant, while some have made no change. Steering gear manufacturing companies are trying to hasten the change by supplying their customers with lists of lubricants which meet their approval, but as yet automobile companies hesitate to be arbitrary in the matter of lubrication recommendations.

Testing Methods and Apparatus

Description of Test Apparatus and the Methods of Testing—"Efficiency" and "Retention"

The construction of an apparatus used in determining the effect of lubricants upon the mechanical efficiency and grease retention of antifriction and sector type steering gears, is shown in the accompanying Plate XX. The apparatus, which was designed and constructed especially for this investigation, consisted of two steering gears mounted in a cork lined temperature-controled cabinet, 12 inches × 12 inches × 32 inches high, rigidly built from iron plate and welded upon an angle iron base. The front and left side of the cabinet were removable to make the gears accessible.

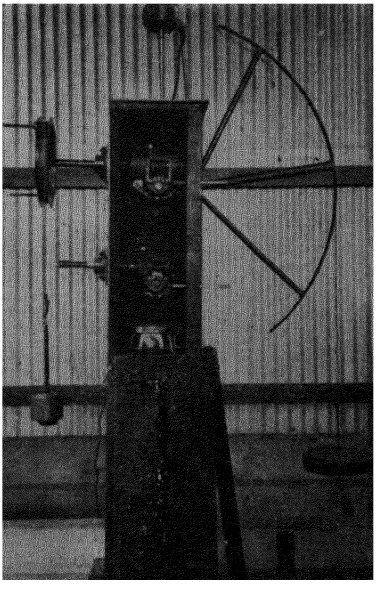


PLATE XX.—Apparatus for Determining Static and Dynamic Efficiencies of Steering Gears. Front Removed to Show Arrangement of Gears.

Two new steering gear assemblies were bolted to the iron plate on the right side of the cabinet, just as they would be bolted to the frame of a car, except that the steering shaft was placed in a horizontal position to permit the mounting of a pulley for measuring the input moment by means of weights. The steering shafts extended through the back of the cabinet, being cut off at 8 inches beyond the cabinet and supported by Hyatt roller bearings to permit loading the input torque wheel without cramping the gears; while the sector or roller shafts extended out the right side of the cabinet to permit the mounting of an output torque quadrant upon the splines in place of the steering arm. This quadrant was built of pipe welded together to form a grooved 120° sector of a wheel of 25-inch radius, which carried a ¼-inch cable supporting a 50-pound output torque weight. This quadrant carried

pointers mounted radially so that its position could be read directly from a scale to within one degree.

The steering wheel was replaced by a 5-inch radius pulley mounted on the stub shaft to furnish a means of applying the input moment. The input moment, like the output moment was furnished by weights suspended from the pulley by means of a $\frac{1}{2}$-inch steel cable wrapped about the wheel. The efficiency was calculated by the use of the following equation:

$$E = \frac{\text{Moment due to quadrant and weight}}{\text{Moment due to weight on pulley} \times \text{Ratio of gear}}$$

In selecting gears to be used in testing lubricants, a study was made of the types of gears, from the standpoints of popularity and mechanical construction.

From the data available it is evident that all gears for 1932, 1933 and 1934 may be grouped under four types:

Steering types and per cent of each based upon total car sales:

Ross cam and lever Ross cam and roller lever	About 8
Saginaw, Gemmer and Plymouth Worm and Roller	About 30
Saginaw, Gemmer and Own Worm and Sector	About 62
	100

From this, it is evident that the Ross gears of both the cam and lever and cam and roller type are used on less than 10 per cent of the cars manufactured in the last three years, and an examination of the table will reveal that the percentage of cars using this type of gear is decreasing, while the worm and roller and worm and sector types are becoming more popular.

For this reason, worm and roller and worm and sector types of gears were selected for the mechanical efficiency tests. The Saginaw worm and roller gear, made for the Buick under Gemmer patents, was selected as representative of the antifriction type gear. The Plymouth is equipped with a worm and roller type of steering gear of their own manufacture, and of the same essential design as the Saginaw and the Gemmer. Therefore, the Saginaw gear may be considered to present lubrication problems almost identical to those of the Gemmer and the Plymouth, representing in all about 30 per cent of the steering gears being manufactured. The worm and sector type made by Saginaw, Gemmer, and Chevrolet are so nearly the same in construction as to present almost identical lubrication problems. The Gemmer gear of the type used on the Ford was selected to represent the worm and sector type of gears in the tests.

It was originally planned to test all lubricants on both types of gear, but, as it was found in practice that data obtained on the worm and sector type gear paralleled that obtained on the worm and roller gear, except that it was less precise due to the lower efficiency of the gear, tests on various lubricants were confined to the Buick worm and roller gear.

The following brief description of the gears may be of interest. The Gemmer gear is a worm and of the three-tooth sector type, having a 14.7

to 1 gear ratio. The copper plated steel worm is carried at each end by roller bearings having an automatic spring adjustment worm and sector mesh, and manual centralizing adjustments. Lubricant leakage occurred around these adjusting devices. The hardened steel sector is carried by a steel shaft which turns in a plain bushing and end play is controlled by a hardened screw bearing against the sector shaft.

The Saginaw (Buick) worm and roller type gear has an 18.75 to 1 gear ratio. The hardened steel worm is carried by roller bearings. All adjustments on the gear, including end play of the roller shaft, adjustment for proper mesh of the roller in the worm, and centralization of tooth contact, are manual. In this gear the sliding sector is replaced by a grooved roller mounted on an antifriction bearing.

The casings of these gears were opened to give large inspection holes for convenience in changing lubricants without changing gear adjustment. These holes were closed with plates and in no way affected the action of the gears. Thermometers were inserted directly into the lubricant by means of packing nuts. The gears were adjusted to factory specifications at the beginning of the test and were not subsequently altered. Some leakage in both types of housings occurred around loosely fitting cork packing inserted between the shaft and the column which was cut off at the gear housing. These cork packings were not fitted tightly because it was desired to reduce friction on the shaft to a minimum. Therefore, there was some oil leakage at this point but this condition was not altered during the series of tests.

If this Saginaw gear was mounted in a car no leakage would occur, even if thin oil is used as a lubricant, but even a new Gemmer worm and sector type gear such as was used on these tests would not retain a very thin lubricant under road conditions, due to the leakage around the worm sector contact adjusting device.

Temperatures from room temperature to $-40°$ F. were secured by means of carbon dioxide snow condensed in a chamois skin bag formed around the gear housing; temperatures from room temperature to 180° F. were secured by means of a hot plate and a fan for circulating the warm air in the cabinet.

The following test method was used on both types of gears and may be conveniently described in steps:

Gears were cleaned thoroughly by removing inspection plates, cleaning with a gasoline spray and drying with an air jet.

Gear case was charged with 340 grams of lubricant for the Saginaw and 150 grams for the Gemmer.

The gear was set to the extreme position with the sector at the end of the worm and the quadrant placed upon the proper splines of the sector shaft to bring the pointers to 0° on the scale.

A 50-pound weight was added to the quadrant to give the proper output moment and the quadrant was then turned to 45° to place the roller or sector in the center of the worm.

The temperature of the lubricant in the case was then brought to $-40°$ F. and held there for 30 minutes to permit gears and lubricant to become set.

Weights were added slowly (0.1 pound at a time) to the input pulley until the

quadrant was moved, and the first weight causing movement was recorded for calculating input moment for determining "Static Efficiency" or "Yield Point."

The quadrant was moved slowly one full oscillation in the following manner: Down to zero (front wheels at extreme right), up to 90° (front wheels extreme left), and back to 45° position (front wheels straight ahead).

Enough weight was put on at this point to move the pointer 10° in five seconds. Before adding the weights the quadrant was moved as follows: Down 5°, then up 10°, then back to the test point and stop. Trial weights were added before releasing and timing the movement. The weight on the pulley required to move the quadrant 10° in five seconds in this manner was used for computing the "Dynamic Efficiency."

The Dynamic Efficiencies were then determined in the same manner at both the 30° and 60° positions.

The temperature of the lubricant was then raised to about −20° F. and the process repeated. Further determinations were made at 0°, 30°, 60°, 100°, 140°, and 180° F.

At approximately 60° F. the chamois bag was removed and the outside of the gear case was wiped clean. A suitable container was then installed to catch all the leakage. The retention test was started at this point and continued to 180° F.

After completing tests at 180° F. the gear case was opened at this temperature, and notes taken on the condition of the oil or grease film on the gears after the lubricant had drained.

This method was developed after making a few preliminary experiments to determine the most desirable conditions for the tests. These preliminary experiments revealed the following facts:

The sensitivity of gears to changes of temperature and lubricant is less under excessive loads (moments as high as 6250 inch pounds were tested) than under loads equivalent to normal hard steering (about 1350 inch pounds). This load was used in all subsequent tests.

The preliminary tests were made at quadrant positions of 10°, 20°, 30°, 40°, 50°, 60°, 70°, and 80°, but it was found that the average of these was equal to the average of positions 30°, 45°, and 60° positions which were used in subsequent tests.

The mechanical error was determined to be less than 3 per cent for "dynamic efficiency" and 4 per cent for "static efficiency" for temperatures above 0° F. At lower temperatures the error was slightly greater.

It was found that a curve for mechanical efficiency, plotted against pounds placed upon the input pulley at constant output moment, was a great aid in computing mechanical efficiency.

During preliminary tests it was found that at low temperatures the input moment required to just start motion might be considerably less than the moment required to cause a motion as rapid as that occurring in steering. This lead to tests being made under both conditions, the former being called "static" and the latter "dynamic"; that is, "static efficiency" was calculated from the moment required to start movement and "dynamic efficiency" was calculated from the moment required to produce a velocity of 2° per second in the quadrant. This velocity corresponds to about 37° per second on the steering wheel and therefore to moderately rapid turning. At the same time, it is low enough to reduce error due to accelerating the 50-pound output weight. Under the conditions of the tests the dynamic and static efficiencies are nearly equal at all temperatures where the lubricant was fluid; but, at low temperatures, the dynamic efficiency was much lower than the static.

The accompanying tables show the weight which must be applied to the input wheel to move the steering gear when it is operating with different degrees of efficiency on a constant output moment of 1350 inch pounds. Obviously, no particular output moment can be considered as typical for cars under all driving conditions since this moment varies widely with the speed of the car, the type of road, the inflation of the tires, and the load on the tires. The moment 1350 inch pounds corresponds to moderately hard steering, such as is encountered in parking in a crowded street, and curves showing pull on steering wheel plotted against steering gear efficiency, give a means of interpreting "efficiency" in terms of the pull the driver must exert on the steering wheel under the above conditions.

Timken Tests for "Wear" and Film Strength

The Timken Machine for making Film Strength and wear tests is a standard machine completely described by the Timken Manufacturing Co. and described in some detail in the "Appendix." The details of construction will not be repeated here, but for convenience the following explanation is made. The machine consists of three essential parts:

A ring of uniform known hardness is mounted firmly on a shaft which is rotated at a known uniform rate by appropriate driving devices.

A block of the same uniform hardness as the ring is mounted on an appropriate lever arm so that it may be pressed against the ring with a known force or pressure.

A device for circulating the lubricant to the point of contact between the block and the ring.

The film strength tests were made in the usual manner, an oil film being reported as O.K. for a given test when it protected the block and cup from welding, and therefore scoring, for a ten-minute period under the conditions (of surface rotational speed of the ring and pressure of the block on the ring) specified by the test. The standard test requires a surface speed of 400 feet per minute. A second set of tests was made at 100 feet per minute. A steering gear is a very low speed machine and therefore low speed data is probably more significant than high speed data. The pressures are obtained by the aid of a table prepared by the Timken Company from the weights applied to the lever arm. Tests were repeated until the maximum pressure at which the oil protected the steel was found. The maximum load which could be safely used on the machine corresponded

Analytical Test Methods

Test	Explanation
Appearance	Observation only.
Odor	Observation only.
Dropping point, Ubbelohde	Holde, "Hydrocarbon Oils and Saponification of Fats and Waxes," page 20.
Dropping point, Gillette	J. R. Battle's "Industrial Oil Engineering," page 220.
Pour point	A.S.T.M. D97-27 F.
Channel test	A modified Knopf Spike Test.
MacMichael vis.	Wire size and temperature given 1 cm. bob 60 R.P.M.
Corrosion	530.31.
Bleeding test	1 hour at 175° F. Cool and observe for evidence of oil "bleeding" from grease. G-M specifications.
Separation	Heat to 220° F., cool and observe evidence of separation of oil and soap or of solidification.

Soap, per cent	A.S.T.M. D128-27
Moisture, per cent	A.S.T.M. D95-27
Oil, per cent	A.S.T.M. D128-27
Free alkali or fatty acid	A.S.T.M. D128-27
Asphaltic material	A.S.T.M. D128-27
Sulfur, per cent	A.S.T.M. D129-33
Ash, per cent	A.S.T.M. D128-27
Ash analysis	A.S.T.M. D128-27
Mineral oil color	A.S.T.M. D155-32T
Mineral oil viscosity at 100° F.	S.U. A.S.T.M. D88-26
Mineral oil viscosity at 210° F.	S.U. A.S.T.M. D88-26
Mineral oil gravity	A.P.I. A.S.T.M. D287-32T
Mineral oil flash	A.P.I. A.S.T.M. D92-24

TABLE 4.—Pounds on Pulley and Mechanical Efficiency Relation—Data Used to Form Curve Sheets.

Buick Gear

Gear ratio								18.75 to 1					
Total movement of quadrant, degrees								90					
Radius of quadrant, inches								25					
Moment on quadrant, inch-pounds								1250					
Correction moment on quadrant at 45° position, inch-pounds								100					
Correction moment on quadrant at 30° and 60° positions approx., inch-pounds								100					
Total moment on quadrant, inch-pounds								1350					
Weight on pulley, pounds	10	15	20	25	30	40	50	60	70	80	90	100	110
Radius of pulley, inches							5						
Moment on pulley, inch-pounds	50	75	100	125	150	200	250	300	350	400	450	500	550
Correction moment on pulley, inch-pounds							7						
Total moment on pulley, inch-pounds	57	82	107	132	157	207	257	307	357	407	457	507	557
Mechanical efficiency = $\frac{\text{No. 8}}{\text{No. 13} \times \text{No. 2}}$	126.2		67.3		45.8	34.8	28.0	23.45	20.2	17.68	15.75	14.19	12.95

Ford Gear

Gear ratio								14.70 to 1					
Total movement of quadrant, degrees								90					
Radius of quadrant, inches								25					
Moment on quadrant, inch-pounds								1250					
Correction moment on quadrant at 45° position, inch-pounds								100					
Correction moment on quadrant at 30° and 60° positions approx., inch-pounds								100					
Total moment on quadrant, inch-pounds								1350					
Weight on pulley, pounds	20	30	35	40	45	50	55	60	70	80	90	100	110
Radius of pulley, inches							5						
Moment on pulley, inch-pounds	100	150	175	200	225	250	275	300	350	400	450	500	550
Correction moment on pulley, inch-pounds							7						
Total moment on pulley, inch-pounds	107	157	182	207	232	257	282	307	357	407	457	507	557
Mechanical efficiency = $\frac{\text{No. 8}}{\text{No. 13} \times \text{No. 2}}$	85.8	58.45	50.6	44.3	39.6	35.7	33.0	29.9	25.7	22.55	20.1	18.1	16.5

to a pressure of 57,140 pounds per square inch and as many lubricants were found to be O.K. at that pressure at a speed of 100 feet per minute it was not possible to determine their maximum strength at this speed. However, it is probable that any oil which gives protection to the block and cup at 57,000 pounds pressure per square inch and 100 feet per minute surface speed will give sufficient protection to steering gear parts, at least as regards E.P. properties.

Analytical Tests

The apparatus and methods used in making the analytical tests were either well known apparatus and methods or so simple as to be easily described in a single phrase; therefore, the information is given in a table on page 507.

The following summarizes the significant performance characteristics of the most important of the lubricants tested:

TABLE 5

No. of Lube	Mechanical Efficiency — Buick Gear — −40° F. Stat.	−40° F. Dyn.	% Efficient — 150° F. Stat.	150° F. Dyn.	Timken Wear 6 hours at 100° per minute, 40,000 pounds per square inch	Timken Film Strength 400 feet per minute, pounds per square inch O.K.	Failed	Heat Stability Separation at 220° F.	Retention Per Cent of charge lost
1	24.0%	13.0%	75.0%	77.0%	34.4 mg.	8575	11430	Jells	4.8
2	31.0	16.0	73.5	76.0	24.9	7140	10000	Jells	3.0
3	0 Solid	0	80.0	79.5	31.2	14280	17140	Becomes hard	1.15
4	71.0	55.0	76.5	79 7	...	14280	17140	O.K.	9.2
5	27.0	8.0	73.5	77.5	9.8	22860	25700	O.K.	8.9
6	62.0	36 0	76.2	78.0	8.2	17140	18570	O.K.	3.5
7	68.0	62.0	70.0	76.5	...	14280	17140	Oil separates	5.65
8	68.0	15.0	73.5	76.0	...	2900	5714	Oil separates	3.8

Lubricant No. 6 is seen to be exceeded in dynamic efficiency at low temperatures only by No. 7 which is unsatisfactory from the point of view of heat stability and by No. 4 which is considerably inferior from the point of view of retention. Its wear characteristics were good and its retention was only exceeded by Lubricant No. 3 which gave a poor efficiency at low temperatures and unsatisfactory heat stability.

The following control specifications are offered as showing the properties which a good lubricant should have:

Appearance	Similar to sample furnished
Dropping point, Ubbelohde, min.	230° F.
Pour point, max.	15° F.
MacMichael viscosity, No. 26 wire at 80° F., 1 cm. bob 60 R.P.M.	50–60
Corrosion	None
Separation test, 220° F.	Lubricant shall not separate nor become hard upon being heated to 220° F. and cooled to room temperature.
Fillers	None
Free alkali, max.	0.15 per cent
Free acid, max.	0.10 per cent
Water, max.	0.2 per cent

Film strength, Timken	100 feet per minute, 50,000 pounds min. (400 feet per minute, 15,000 pounds min.)
Mineral oil plus saponifiable E.P. base:	
Approx. S.U.V. at 100° F.	667
Approx. S.U.V. at 210° F.	75
Approx. S.U.V. at 0° F. (by extrapolation from A.S.T.M. chart)	80,000
Minimum Dean & Davis viscosity index	90

The static and dynamic efficiency curves are shown in Figures 1 and 2. It will be seen that the static and dynamic curves meet at the point of solidifying, while at 20° F. above the solidifying point the curves show their maximum difference.

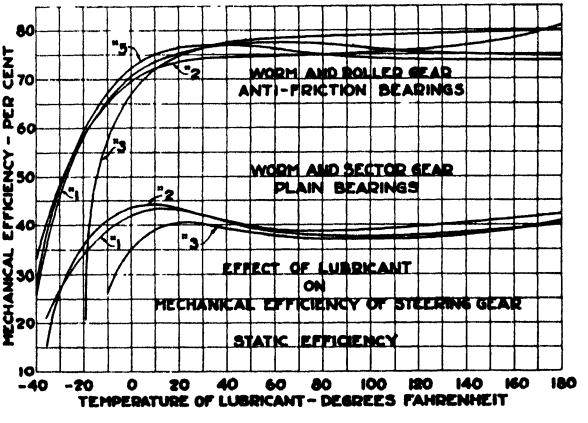


FIGURE 1

This comparison is only of minor importance from a practical viewpoint; the important question at issue is the effort required to turn a car. Therefore, the Dynamic Efficiency is of the major importance. The difference in Static and Dynamic Efficiency may be important for an academic study regarding grease consistency, but is considered of minor importance.

The leakage of lubricant from the Buick Saginaw Gear was due to the manner in which it was mounted in the "Efficiency" machine. Therefore, retention data for this gear are purely qualitative and not quantitative In a good new gear of this type no leakage would occur, even if a light oil were used. In an old worn gear some lubricants would be retained better than others, and it is probable that those lubricants which show the lowest percentage of charge lost would be best retained by a leaky gear housing. Greases No. 3 and No. 2, and Lubricant No. 6 all show a loss of less than 4 per cent. Of these, the first is so stiff as to permit only very low efficiency at low temperatures. No. 8 and No. 6 show about equal retention (3.5 per cent) and both permit high efficiency at low temperatures, as well

as high, but No. 8 has poor film strength and heat stability, while Lubricant No. 6 has excellent film strength and heat stability.

A Discussion of Wear and Film Strength Data

The Timken Film Strength tests were made at two different speeds—the usual speed of 400 feet per minute and a much lower speed of 100 feet per minute. The standard test gives values for comparison with other E.P. lubricants. The low speed tests seemed to be desirable, since a steering gear is a very low speed machine and the low speed data is therefore more significant. The design of the Timken machine prevented the use of speeds below 100 feet per minute or loads in excess of 57,140 pounds per square inch. It is probable that lubricants which for the 10-minute test period protect the Timken test block and ring from abrasion, when pressed together

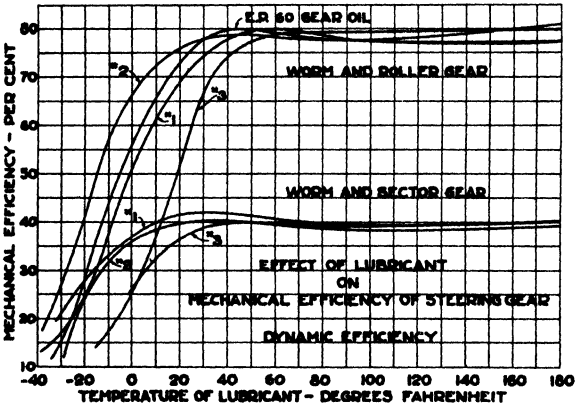


FIGURE 2

under a pressure of 57,000 pounds per square inch at a surface speed of 100 feet per minute, have sufficient film strength to protect the moving parts of any steering gear. This leads to the assumption that Lubricant No. 6, No. 7, and No. 3 possess sufficient film strength. It must be noted, however, that No. 7 was completely broken down to the oil during the test and never recombined to form a grease. No. 3 became as solid as a heavy axle grease and returned to its original consistency only upon standing and being vigorously worked. Most of the above group contained more than 1 per cent sulfur and it is probable that the others were fortified by some other type of E.P. base.

Wear tests made indicate that when the test is made under a load much below the film strength of the lubricant the wear is extremely small, unless the lubricant contains some solid filler such as asbestos. Since none of the lubricants contain fillers, it is probable that the difference in the wear occurring when many of the above lubricants were used would be very small,—so small as to be obscured by experimental error. Therefore, as wear tests are very time-consuming, these tests were limited to the products

listed. Tests were made at 40,000 pounds per square inch, 100 feet per minute, over a six-hour period, since it was desired to approach the slow speeds of the gear and to depend upon high loading to secure wear. It at first appears that such tests would be impossible, since products have film strengths lower than 40,000 pounds per square inch. It must be remembered, however, that this pressure is calculated upon the original narrow contact between a new block and a new ring. Within a 30 minute break-in period, during which the weight on the loading beam was slowly increased, this bearing surface is greatly increased and the unit pressure correspondingly decreased. The unit pressure is given in these wear tests in terms of the tables furnished by the Timken Company, but it is obvious that the actual unit pressure for a given weight on the loading beam is an inverse function of contact area and therefore decreases as the block wears to fit the ring. All lubricants were subjected to exactly the same treatment during the wear tests,—a thirty minute break-in period, during which the load on the beam was gradually increased until it was equal to the weight given in the tables as equivalent to 40,000 pounds per square inch, followed by a six-hour wear period with that weight on the beam.

As noted above, wear tests are not extremely accurate, but give a qualitative rather than quantitative measure of the wear which would result from the use of the various lubricants in a steering gear under conditions of friction at great pressure. No great significance can be attached to the differences in weight lost. The 34.4 mg. loss during the test on No. 1 may be attributed to the low film strength of this product. The 31.2 mg. loss during test on No. 3 may be attributed to two conditions of the test: The lubricant stiffened rapidly during the test so that at the end of two hours it was impossible to circulate the lubricant except manually and the test was stopped, the block and cup weighed and the loss in weight multiplied by three to give an equivalent of a six-hour test. (1) It is probable that wear is more rapid during the first portion of a test, since for reasons given above the unit pressures are greater. (2) It is possible that as the lubricant became extremely viscous and rubbery the protecting film did not furnish as good protection as when the lubricant was sufficiently fluid to flow freely into the contact area. Apparently No. 2 is more effective than either of the No. 1 or No. 3 in preventing wear, the loss being 24.9 mg. Lubricant No. 6 (loss of 8.2 mg.) gives the greater protection to be expected from a good E.P. lubricant being tested at loads far below its film strength. It will be noted that if these tests give a measure of the relative wear which would occur in a gear when lubricated by the above lubricants, then the lubricant No. 6 should save car owners from one-half to three-quarters of their present steering gear adjustment costs.

Analytical Data and Heat Stability

The tables of analytical data give two distinct types of information—information concerning composition and information concerning the chemical and physical properties of the lubricants. The composition of a lubricant

is of interest to the chemist in determining which substances are being used by competitors in preparing the lubricants, and in correlating composition with performance as an aid in preparing new products, but the composition of a lubricant does not give a measure of its qualifications for a particular lubrication condition. For this reason, no comment will be made upon the composition of the various lubricants other than to point out that there is a marked correlation between type of soap and heat stability. There is obviously a correlation between percent soap and viscosity, but in the present investigation this is obscured by the fact that the different products contain different types of soap.

Corrosion is only one chemical test which determines the fitness of a lubricant to be used for lubrication, and in this investigation no lubricant was found to be corrosive except one.

Of the physical tests, the separation test offers the more effective means of judging the various lubricants. It will be noted that upon heating the aluminum soap lubricants to 200° F., they either jell or solidify. The calcium soap lubricants separate and remain separated, and sodium soap lubricants are heat stable.

The remaining physical tests, such as pour point, channel test, MacMichael viscosity, dropping point, etc., are of interest in estimating the probable effect of a lubricant upon the mechanical efficiency and retention of a given steering gear assembly. However, since we have secured data on efficiency and retention by use of the much more exact Efficiency Machine, this data becomes of little value except as a means of correlating these routine tests with actual efficiency and retention.

Of these analytical data, that portion essential to the judging of the qualifications of the various products as steering gear lubricants may be summarized in two sentences:

One product only, was found to be corrosive. Eight products were found to be unstable to heat, leaving the six products classified as desirable lubricants when judged only by corrosion and heat stability.

Pressure Viscosity Data

The pressure viscosity data was obtained by means of a very simple constant pressure viscometer, consisting of a Saybolt Universal tube equipped with a screw cap so that pressure could be applied by means of a cylinder of nitrogen. Temperature was controlled by a bath surrounding the tube, and measured by means of a thermometer in the lubricant and a thermometer in the temperature bath. The apparatus was rather inconvenient to fill and slow in responding to temperature changes, but results obtained were reproducible at any given temperature to within 10 per cent. As 10 per cent corresponds to about 2° F. it is possible that most of the error was due to the poor temperature controlling devices. The pressure was kept at ten pounds per square inch except in the cases where the viscosity at low temperature was so high that it was necessary to use sixty

pounds per square inch. In such cases results were calculated back to ten pounds per square inch by means of the approximate equation:

$$\frac{P_1}{\text{Rate of flow 1}} = \frac{P_2}{\text{Rate of flow 2}}$$

The weight of the lubricant flowing from the viscometer in a given time was determined at each temperature and calculated to grams of lubricant flowing per minute. The log of the grams per minute were plotted against temperature.

In a rough general way the grams of lubricant flowing from an orifice per minute under a given set of conditions is proportional to the reciprocal of the viscosity and when plotted the curves on a pressure viscosity chart should rise with an increase in temperature. Such curves represent the gram rate of flow under a pressure of ten pounds at various temperatures and therefore are not a simple function of true viscosity as that term is usually defined. This study was made for the purpose of checking by a less expensive method on the data obtained by the use of the actual steering gears, and no attempt was made to obtain data in terms of actual viscosity units for the following reasons:

Most steering gear lubricants are not pure mineral oils but mixtures of oil and colloidally dispersed soap. They therefore are colloidal systems with a "limit of elasticity" or "shear stress" or "yield value" as well as a "true viscosity." Equations such as Bingham's equation

$$\frac{1}{u}\frac{4V/t}{\pi R^3} = (F-f)$$

have been used for calculating the "true viscosity" of colloidal systems in general and greases in particular from the measured or "apparent viscosity" obtained at two or three different rates of shear. Arveson[247a] found that this equation did not fit the data obtained by his extensive investigation of cup greases. On the contrary, he found that the measured or "apparent viscosity" may decrease as much as 13,000 fold with a 1,000,000 fold increase in shear rate and that the viscosity of the mineral oil is closely approached at extremely high rates of shear. It is to be hoped that other laboratories will soon be equipped to study viscosity under conditions similar to those reported in these instructive reports by Arveson; but obviously such an investigation should be complete in itself and was not undertaken as a part of this study of steering gear lubricants.

A steering gear is a low speed machine whose efficiency at low temperature is determined by the resistance which the lubricant offers to deformation by the gear. The studies reported were made to see if it were possible to correlate the rate of flow of the lubricant through an orifice with the efficiency of the steering gear. A flow rate of less than 0.2 grams per minute was found to occur for some of the lubricants at the same temperature at which the efficiency of gears using these lubricants

[247a] *Ind. Eng. Chem.*, **24**, 71 (1932); **26**, 634 (1934).

began to drop rapidly. Thus, by tracing across the graphs at this level it was seen that the temperature at which these lubricants would cause hard steering corresponds to low flow notes.

The pressure viscosity data on steering gear lubricants indicate that there is a very good correlation between high viscosity, low rate of flow, and low efficiency. Therefore, it may appear desirable to substitute the cheap, rapid viscosity measurements for the more expensive time-consuming efficiency measurements in determining whether or not certain lubricants were worthy of further investigation. Pressure viscosity data is not yet sufficiently accurate to permit an accurate estimation of the efficiency of a lubricant at high driving temperatures, but it does furnish a means of judging the temperature at which the grease will "freeze" the gears. Since all temperature "efficiency" is the most important quality of a steering gear lubricant, it does not seem desirable to spend much time in investigating lubricants which "freeze" at comparatively high temperatures.

Results of Pressure Viscosity Tests

Steering Gear Lubricant Temperature ° F.	1 Flow in Grams per Minute, S.U.V. Tube, 10 lbs./sq. in	2	3	5
0	0.1	0.1	...	0.2
20	0.7	0.5	.07	0.9
40	3.0	2.5	0.5	4.0
60	7.5	8.2	2.5	14.0
80	20.0	25.0	8.0	45.0
100	49.5	72.4	18.5	108.0
120	124.0	166.0	44.7	241.0
140	330.0	330.0	99.8	462.0

Discussion of Steering Gears and Lubricants

The Temperature Range of Steering Gears

Tests made on three different cars on hot summer days (about 100° F.) gave the temperature of the lubricant in the steering gear to be between 140° F. and 150° F. The temperature was measured after the cars were thoroughly warmed up by fast driving, in one case being measured by means of a thermocouple while the car was being driven; in the other two cases being measured by a thermometer inserted into the lubricant immediately after stopping the car. It appears that the draft of air from the fan is so heavy that the temperature of the air after passing through the radiator determines the temperature of the steering gear. The three tests indicate that steering gears seldom become warmer than 160° F. Heat stability tests were made at 220° F., not because a steering gear ever becames that warm, but because in all testing it is desirable to make use of extreme conditions to compensate for the short duration of the tests as compared to the service period of a lubricant. Expressed in another way, lubricants were tested at 220° F. to insure that they would always be stable at 160° F. If a lubricant is tested for a short time at 160° F. it may be reported as heat stable at that temperature when under weeks of use in a car in summer it would prove to be unstable. However, when tested at 220° F. and found to be stable, there is no doubt concerning the stability of the lubricant.

The lowest temperature existing in a gear is atmospheric temperature on a cold winter morning. In many parts of the United States this temperature will be below freezing, in more restricted areas below 0° F., while −40° F. has been reported. Since, as stated above, the steering gear is heated only by air it will heat up much more slowly than the motor, probably not more than 5 degrees by the time the driver is ready to start the car. Under the best possible conditions the steering wheel is apt to be hard to turn for the steering knuckles may be stiff with chilled lubricant and perhaps coated with ice. It is, therefore, very desirable that steering gear lubricant be of such a viscosity at low temperatures that the driver will not be burdened with prying loose a stiff gear as well as stiff knuckles. For this reason the investigation of the effect of temperature upon the efficiency with which various lubricants permitted gears to operate was carried down to temperatures of −40° F. It must be emphasized that the steering gear is often the greatest source of low temperature friction in the series of bearings between the steering wheel and the front wheels, since most chassis lubricants used in winter have good low temperature viscosities, and that six of the lubricants tested "freeze" the bearings absolutely solid at −40° F.

Steering Gear Types and Lubrication Problems

Data indicates that over 90 per cent of the cars built in 1932, 1933 and 1934 are equipped with either a worm and roller or a worm and sector type of steering gear. As previously mentioned, all of the worm and roller gears are so similar that their lubrication problems are almost identical. The same is true of the worm and sector type gear; in either case the only difference in the lubrication problems of the different makes of gears of these types are slight differences in housing design which may lead to slight differences in lubricant retention. This book deals primarily with these two types of gears—the Saginaw steering gear built for Buick, representing the worm and roller type and the Gemmer steering gear built for Ford, representing the worm and sector type.

The Ross gears are of a different type and present different lubrication problems which will be discussed on the following page.

The Jacox gear, used on early Buicks is no longer used on cars, but judging from the extensive area of sliding parts it must have become very inefficient at low temperatures with viscous lubricants. The Lavine Steering Gear is not yet reported as adopted for any make of car, but it does present a different type of steering gear, the "Ball Drive." The Lavine Gear Company, Milwaukee, Wisconsin, claim that their gear possesses several distinct advantages, one being that its construction is such that the use of any good gear lubricant insures complete lubrication at all times.

Lubrication Problems of the Ross Gear

While less than 10 per cent of the passenger cars built in the last three years use the Ross Gear, it is used on a number of trucks and busses and therefore the investigation of steering gear lubrication would not be com-

plete without a study of the particular lubrication problems of this steering gear. Ross Service Bulletins, Forms 272 and 210-A, point out that the lubricant should prevent "noise" and "rattle" as well as wear. "Use a heavy transmission grease of fibrous quality; it must possess cushioning as well as lubricating qualities. Do not use universal joint greases."

Lubricant No. 6 contains 24 per cent of Fiber Grease No. 1 and therefore has a "fibrous quality," but it is not so easy to determine the "cushioning" and "rattle" prevention qualities. A Ross steering gear was taken from a car in running condition, and without adjusting the gear, it was mounted upon a frame so that the steering arm could be conveniently vibrated and lubricants could be easily changed. The change in noise as the lubricant was added to the vibrating clean dry gears was slight in all tests. In order to differentiate between lubricants it was necessary to shut down all machinery in the vicinity and to depend upon the sound impressions received by listening people who did not see the lubricant poured upon the gears and therefore were not confused by sight impressions. The average votes of four listeners listed the three lubricants tested in the following order of decreasing efficiency in deadening noise:

1. No. 3. Approved by Ross.
2. No. 4a { Fiber No. 1 —66 per cent. / Floyd's Base—34 per cent.
3. No. 6.

From this rather crude test on several greases, several conclusions may be drawn:

No correlation can be made between soap type and noise deadening:

Type of Soap	Rating
Aluminum soap	1 and 5
Sodium soap	3 and 4
No metallic soap	2

The correlation between MacMichael viscosity and noise deadening is not good:

MacMichael Viscosity	Rating
126 at 80° F. 24 wire	1
100 at 80° F. 26 wire	2
56 at 80° F. 26 wire	4
180 at 80° F. 26 wire	5

The noise deadening under the conditions of the test was not marked and the distinction between lubricants was slight.

Judged by this crude test, Lubricant No. 6 is at least as efficient in deadening noise as other greases recommended by Ross.

Exact measurements of "cushioning" or "noise deadening" qualities of a lubricant cannot be made without the use of apparatus which is independent of the human ear as a measuring device. The cost of such apparatus would not be justified by the market for steering gear grease.

A Discussion of the Value of an E.P. Base in a Steering Gear Lubricant

In practice only about one-half or one-third of the tooth is in contact and, therefore, the pressure calculated from theoretical considerations should be multiplied by two or three to give the actual pressures existing in the

TABLE 6.—Analytical Data: Steering Gear Lubricants

Sample No.	1	2	3	5
Physical Tests:				
Appearance	Gr. Brown Stringy	Black Vis. Liquid	Black Semi-Solid	Black Liquid
Odor	Pine Tar	Sharp—Soapy	Soapy	Pungent
Dropping point:				
a. Gillette, ° C.	36	Room Temp.	76	Liquid
b. Ubbelohde, ° C.	48	78	92	Liquid
Pour point, ° F.	50	60	Above 120	−5
Channel test, ° F.	0	10	10	−10
MacMichael, 1 cm. bob, 60 R P M.	No. 26 wire 140 at 75° F.	No. 26 wire 65 at 70° F.	No. 24 wire 126 at 80° F.	No. 26 wire 33 at 76° F.
Corrosion, cu. 3 hr. 212° F	OK	OK	OK	OK
Bleeding Test, 175° F., 1 hr	OK	OK	OK	OK
Separation test, 220° F.	Jells	Jells	Becomes hard like axle grse	No Separation
Composition, Per Cent by Weight:				
Soap	10.2	7.3	8.7	4.6
Moisture	0.1	0.1	0.2	0.15
Oil	85.9	92.2	79.8	93.5
Free alkali or fatty acid	4.1 oleic	3.6 oleic	11.2 oleic	Pb present
Asphaltic material	2.0	0.3	2.0	0.32
Sulfur	0.33	0.50	0.83	2.00
Ash:				
Ash, per cent	3.08	0.77	1.78	3.3
Analysis, per cent	11.0 Na_2CO_3 69.3 PbO 13.8 $PbSO_4$	77.2 Al_2O_3 21.6 Na_2CO_3	80.1 Al_2O_3 7.3 Al_2SO_4 6.9 Na_2CO_3	90.0 PbO 0.9 Acid Ins
Mineral Oil:				
Color	Green Black	Black	Black	...
S.U.V. at 100° F.	1667	2000	3270	2640
S.U.V. at 210° F.	89.4	118	164	135
Gravity, ° A.P.I.	20.5	21.9	19.7	16.8
Flash, ° F.	405	285	390	345

Sample No.	6	7	8	9
Physical Tests:				
Appearance	Br. Stringy Semi-Solid	Light Brown Semi-Solid	Brown Semi-Solid	Dark Green Brown
Odor	E.P. Base	E.P. Base—Soapy	Fatty	Sharp—Soapy
Dropping point:				
a. Gillette, ° C.	Room Temp	42	50	Liquid
b. Ubbelohde, ° C.	126	78	81	Liquid
Pour point, ° F.	5	Too Heavy	Too Heavy	30
Channel test, ° F.	Below 0	0	0	10
MacMichael, 1 cm. bob, 60 R.P.M.	No. 26 wire 56 at 80° F.	No. 26 wire 55 at 70° F.	No. 26 wire 143 at 70° F.	No. 26 wire 93 at 80° F.
Corrosion, cu. 3 hr. 212° F	OK	OK	OK	OK
Bleeding Test, 175° F., 1 hr.	OK	OK	Separates	...
Separation test, 220° F.	No Separation	Grease breaks down	Separates	Slight Separation
Composition, Per Cent by Weight:				
Soap	2.9	5.6	7.1	8.0
Moisture	0.1	0.4	0.2	0.6
Oil	97.1	92.5	90.0	91.6
Free alkali or fatty acid	.01 NaOH	0.14 oleic	0.21 oleic	0.7 oleic
Asphaltic material	Trace	...	...	0.01
Sulfur	1.86	2.47	0.40	0.45
Ash:				
Ash, per cent	0.35	0.77	0.86	0.89
Analysis, per cent	90.0 Na_2CO_3	62.2 CaO 9.8 $Al_2(SO_4)_3$	91.6 CaO 6.4 Na_2CO_3 2.8 Fe_2O_3	80.5 CaO 19.6 $CaSO_4$ 3.7 Fe_2O_3
Mineral Oil:				
Color	3— Red	Red, Blue	8—	Brown-Black
S.U.V. at 100° F.	314	548	379	2810
S.U.V. at 210° F.	49	53.9	51.4	128
Gravity, ° A.P.I.	23.2	20.2	22.5	19.0
Flash, ° F.	375	320	370	405

TABLE 6—(*Continued*)

Sample No.	10	11	12	13
Physical Tests:				
Appearance	Green Brown Liquid	Black Liquid	Black Green	Black Brown
Odor	Sweet Soapy	Phenol-like	Sour	Faintly Soapy
Dropping point:				
a. Gillette, ° C.	Liquid	Room Temp	Liquid	Liquid
b. Ubbelohde, ° C.	Liquid	Liquid	Liquid	Liquid
Pour point, ° F.	30	50	60	−5
Channel test, ° F.	15	...	40	−10
MacMichael, 1 cm bob, 60 R.P.M.	No. 26 wire 180 at 80° F.	No. 26 wire 50 at 70° F.	No 26 wire 100 at 80° F	No. 26 wire 15 at 80° F.
Corrosion, cu. 3 hr. 212° F.	...	OK	OK	Not OK
Bleeding Test, 175° F., 1 hr.	...	OK	...	...
Separation test, 220° F.	Solidifies	No. Separ	Solidifies	No Separ.
Composition, Per Cent by Weight:				
Soap	6 6	1 2	6.3	3.5
Moisture	0 2	0 05	0 2	0.1
Oil	92 2	98 3	90.5	95.5
Free alkali or fatty acid	Al_2O_3 pres.	0 6 oleic	0.56 oleic	lead present
Asphaltic material	0 03	0.1	0.2	0.5
Sulfur	0 28	0.95	0.99	2.33
Ash:				
Ash, per cent	0 86	0 04	0.08	0.37
Analysis, per cent	85.6 Al_2O_3 8.4 $Al_2(SO_4)_3$	No Analysis	No Analysis	34.2 PbO 51.3 $PbSO_4$
Mineral Oil:				
Color	Green Red	Brown Black	Green	Black
S.U.V. at 100° F.	2298	4520	4560	1267
S.U.V. at 210° F.	120	230	218	92
Gravity, ° A.P.I.	21 8	21.6	23.7	18 6
Flash, ° F.	420	390	530	390

Sample No.	14	15	16	17
Physical Tests:				
Appearance	Green Semi-Solid	Dark Green Brown	Black	Green Semi-Solid
Odor	Slightly Soapy	Pine Tar	Nitro-benzene	Sweet
Dropping point:				
a. Gillette, ° C.	54	Liquid	Liquid	Room Temp
b. Ubbelohde, ° C.	84	Liquid	Liquid	64
Pour point, ° F.	100	15	−0	20
Channel test, ° F.	25	5	−5	0
MacMichael, 1 cm. bob, 60 R.P.M.	No 26 wire 154 at 70° F.	No. 26 wire 14 at 80° F.	No. 26 wire 14 at 80° F.	No. 26 wire 113 at 70° F.
Corrosion, cu 3 hr. 212° F.	OK	OK	OK	OK
Bleeding Test, 175° F., 1 hr.	OK	...	...	OK
Separation test, 220° F.	No Separ.	No Separ.	No Separ.	No Separ
Composition, Per Cent by Weight:				
Soap	5 5	3.7	3.1	5.2
Moisture	0.2	None	Trace	0.1
Oil	84 0	95.6	97.0	92.0
Free alkali or fatty acid	.03 NaOH	0 35 oleic	0.21 oleic	1.1 oleic
Asphaltic material	...	0.2	0 1	...
Sulfur	.37	2 37	5.76	2.47
Ash:				
Ash, per cent	1.02	0.03	0.005	0.97
Analysis, per cent	80.0 Na_2CO_3 9.6 Na_2SO_4 2.5 Fe_2O_3	No Analysis	No Analysis	59.6 Na_2CO_3 37.4 Na_2SO_4 2.2 Fe_2O_3
Mineral Oil:				
Color	Green Black	Green Black	...	Dark Green
S.U.V. at 100° F.	3930	1305	...	1097
S.U.V. at 210° F.	177	87	...	65.4
Gravity, ° A.P.I.	23.2	20.7	...	17.5
Flash, ° F.	490	415	...	410

gear. Thus, it is seen that pressures up to 20,000 pounds per square inch may exist in even the worm sector steering gear of a light car. This figure is based upon the assumption of a moment of 125 foot pounds (or 1500 inch pounds) being delivered by the front wheels to steering gear, a force which must be greatly exceeded in cases of "shimmy" and striking a curb or obstruc-

TABLE 7.—Summary of Retention Qualities of Steering Gear Lubricants

Lubricant Number	Gear	Grams Charge	Grams Loss	Per Cent of Charge Lost
1	Antifriction	...	..	...
1	Plain	...	..	...
2	Antifriction	330	10	3.0
2	Plain	150	9	6.0
3	Antifriction	348	4	1.15
3	Plain	151	0	0.0
4	Antifriction	336	31	9.2
5	Antifriction	337	30	8.9
6	Antifriction	345	12	3.5
6	Plain	146	12	8.2
7	Antifriction	335	19	5.65
8	Antifriction	340	12	3.5
9	Antifriction	358	11	3.1
10	Antifriction	350	15	4.3
11	Antifriction	331	21	6.35
12	Antifriction	345	18	5.2
13	Antifriction	318	25	7.9
14	Antifriction	339	5	1.47
15	Antifriction	326	54	16.5
17	Antifriction	342	20	5.85

tion at an angle. This is in the range where E.P. bases are desirable in lubricants, even though the motion is slow. In heavier cars and trucks pressures are higher. While the antifriction or ball and roller bearing type of gear reduces sliding friction to a minimum, areas of contact are reduced and unit pressures are correspondingly increased. The highest pressures existing in a steering gear result from "pounding" caused by shocks to the front wheels and not from the "rubbing" pressure delivered by the steering wheel. Therefore, it is possible that steering gears may present film strength

TABLE 8.—Wear Tests on Steering Gear Lubricants

Lubricant Number	Loss in Weight, Ring	Loss in Weight, Block	Total
1	29.5 mg.	4.9 mg.	34.4 mg.
2	18.4 mg.	6.5 mg.	24.9 mg.
3	19.2 mg.	12.0 mg.	31.2 mg.
5	5.4 mg.	4.4 mg.	9.8 mg.
6	5.5 mg.	2.7 mg.	8.2 mg.

and wear problems which cannot be interpreted in terms of Timken tests. However, there is also "pounding" in gears and Timken Test data have been helpful in measuring the protective quality of gear lubricants. Steering gear parts do become scored just as ring gear teeth do, so that it is probable that in spite of the low speeds a steering gear requires an E.P. lubricant.

The use of an extreme pressure base in steering gear lubricants has been

recommended by General Motors and by Gemmer. While no definite statement concerning the attitude of the Ross Gear and Tool Company toward the use of E.P. lubricants is available, it may be assumed that they approve since such lubricants as Gear Lubricant 160 E.P. appear on their approved list.

The wear tests made during this investigation indicate that the use of an E.P. base would probably reduce wear to about one-half or one-third that existing when some present Steering Gear Lubricants are used. In the

TABLE 9.—Timken Film Strength Tests on Steering Gear Lubricants

Lubricant Number	Pressure Lbs /Sq. In.	Speed Ft./Min.		Remarks
1	8575	400	OK	This lubricant gives a good film at all times. Returns to original consistency upon cooling.
	11430	400	Failed	
	35700	100	OK	
	38570	100	Failed	
2	7140	400	OK	This lubricant stiffens like No. 3 but to a lesser extent.
	10000	400	Failed	
	31400	100	OK	
	34280	100	Failed	
3	14280	400	OK	Lubricant becomes very stiff.
	17140	400	Failed	Lubricant becomes very stiff
	57140	100	OK	Lubricant completely solidified on cooling.
4	14280	400	OK	Slightly poorer film than No 6.
	17140	400	Failed	
	57140	100	OK	
6	17140	400	OK	Good film in all tests. Original consistency upon cooling.
	18570	400	Failed	
	57140	100	OK	
7	57140	100	OK	Lubricant was completely broken down to oil and soap. Unfit for further tests.
8	2900	400	OK	Badly separated by tests.
	5714	400	Failed	
	25700	100	OK	
9	8575	400	OK	Good film. Slightly thinner after tests.
	10000	400	Failed	
	57140	100	OK	
10	12860	400	OK	Turned dark and stiffened during test.
	15720	400	Failed	
	57140	100	OK	
11	14280	400	OK	Very viscous oil-like consistency throughout tests.
	17140	400	Failed	
	57140	100	OK	
13	57140	100	OK	Good E.P. Gear Oil. Sample too small for further tests.
	24280	400	OK	
14	8575	400	OK	Very heavy film of lubricant on ring at all times.
	11430	400	Failed	
	57140	100	OK	
15	24280	400	OK	Only small sample available. Further tests not possible. Good E.P. gear oil.
	57140	100	OK	
17	47100	100	OK	Fair film.
	51330	100	Failed	

present investigation no attempt has been made to determine either the minimum nor the most effective percent of E.P. base to be used in blending a steering gear lubricant. The 20 per cent blend meets the narrow range (19 per cent to 21 per cent) permitted by General Motors Company specifications and has been found to be very effective. The cost of preparing this 20 per cent blend (Lubricant No. 6) will be low and it is doubtful if any saving made by reducing the E.P. base content below 20 per cent would

compensate for possible loss of trade due to a failure to meet G.M specifications.

There will be loosely fitting gear housings in service for years to come. However, in Lubricant No. 6 a high degree of retention has been attained without greatly sacrificing efficiency. Since stability can be obtained without sacrificing any other quality it should be required from any recommended lubricant. For those lubricants investigated, wear is primarily related to film strength, since fillers are not used in any of them and only one was found to be corrosive. Since the use of a good E.P. base can give a lubricant sufficient film strength to reduce wear to a minimum without in any way reducing the other three qualities of the lubricant; no lubricant should be recommended unless it has a high film strength. Efficiency is, of course, the most important qualification; in fact, the other three are important only as they might ultimately affect the efficiency. It is recommended, that in judging a lubricant high efficiency over the entire temperature range be considered as the most essential qualification; that wear, stability, and retention be considered as less essential in the order mentioned.

Steering Gear Lubricant No. 6 (See previous sections for discussion of service characteristics).

	Per Cent by Weight
Standardized fiber grease No. 1	24.0
300 S.U.V./100° F. Gulf coast pale oil	56.0
Sulfurized lard or other fatty oil (Floyds E.P. base)	20.0

The Fiber Grease is heated to about 200° F. and a small amount of sodium hydroxide solution added, sufficient to neutralize any fatty acids in the sulfurized fatty. Following this, the 300 Pale Oil is run in and then the sulfurized fatty oil, the latter being added at a temperature of about 150° F.

Sodium Soap-Lead Soap Base Steering Gear Grease

	Pounds
Dark sodium base gear grease S.A.E. 110	6000
Fuel oil (black oil)	375
Stockholm pine tar	375
Lead fish oil soap	750

About 800 gallons of the dark gear grease is measured into a steam-jacketed grease kettle, and brought to a temperature of about 145° F. The fuel oil and Stockholm Tar are then measured in and the batch stirred for 15 minutes.

The lead soap is heated in a separate vat equipped with steam coils, to a temperature of 140 to 180° F., and, in a fluid condition, is measured into the mixer. No heat should be applied to the jacket of the mixer at this stage of the process as this tends to form clots and give the grease a very unattractive appearance. The final temperature of the batch should not exceed about 145° F. A product of this kind is particularly suited for Steering Gears found to leak badly when lubricated with fluid oils. This

grease should have a MacMichael viscosity of 40 to 60 at a temperature of 80° F. (No. 26 Wire, 60 R.P.M.). It will not channel in service at temperatures above 10° F.

UNIVERSAL JOINT GREASES

Some of the special universal joint greases now on the market will not lubricate as well as others. Furthermore, certain greases have been applied by service stations which are entirely unsatisfactory. Some greases which may be satisfactory if properly applied are so stiff that they will not handle well in grease guns available at the service stations. Universal joint greases must, of course, in order to be satisfactory, have a high degree of both adhesion and cohesion, to resist being torn apart and being worked out of the bearings. From the results of the service tests which are presented in the following sections it will be observed how much difference exists in the

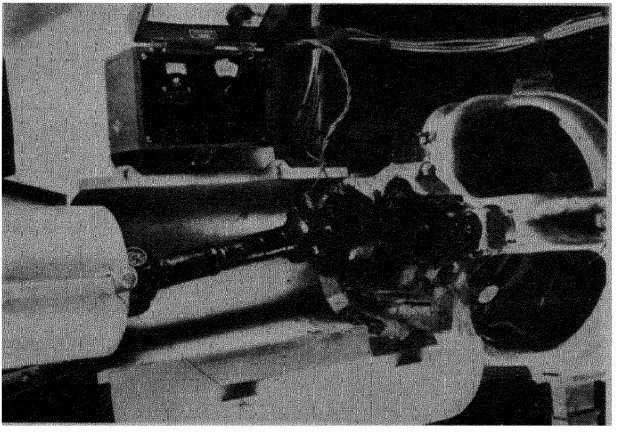


PLATE XXI.—Universal Joint Grease Service Tester, Viewed from Above.

Electric dynamometer at right for absorbing power transmitted. Note method for determining temperatures within joints by means of thermocouples and shield for accumulating grease thrown from joint.

retention properties of different types of greases. While road tests are needed to demonstrate conclusively the relative value of universal joint greases, laboratory service tests are quite useful as indications of what may be expected in the field, and have the advantage of being much quicker to carry out, more convenient and less expensive.

Plate XXI shows a view of an apparatus used for service testing of Universal Joint Greases and it will be noted that it consists essentially of two joints connected by an intermediate shaft, the entire assembly being driven by a 30 horsepower induction motor. Tests may be carried out at a speed of 3590 R.P.M. which corresponds to an approximate road speed of 80 M.P.H. The load transmitted was 20 horsepower and was absorbed by means of a 35 horsepower Sprague dynamometer. Conditions con-

ducive to accelerated wear were provided for by operating the joints at an angle of 10 degrees. Temperatures within the bearings were obtained by means of thermocouples and retention of the grease was studied and measured by collecting the grease thrown from the joints.

TABLE 10.—Summary of Results of Service Tests Made With a Spicer Universal Joint

No. 1 (Type G—Plain Bearing Joint)

	Max. Op. Temp. ° F. Above Housing	Fe_2O_3 Total Gms. in U. J. After Test	Throwoff Total Gms. 30 Hours 100 g. Init. Chg.	Pres. Vis. Gms./Min. at 30 Lb. /Sq. In. and 140° F.	Vis. of Oil Saybolt Sec. at 100° F.	Percent Soap	Pen. Cone at 77° F. Unworked	Mac-Michael No. 24 Wire at 95° F. Unworked
1.	30.[1]	.157[6]	65.0[6]	869.[5]	657.[7]	14.1	299	180
2.	31.[2]	.198[6]	62.5[5]	949.[6]	1062.[6]	11.4	345	100
3.	34.[3]	.133[3]	60.0[3]	544.[3]	247.[3] Fur.	8.2	304	160
4.	35.[4]	.245[7]	67.0[7]	602.[4]	745.[4] Fur.	14.2	204	Not det.
5.	38.[5]	Tr.[1]	58.8[2]	342.[2]	1986.[5]	7.8	310	Not det.
6.	50.[6]	.117[2]	54.5[1]	230.[1]	295[2]	18.5	275	Not det
7.	50.[7]	.143[4]	61.8[4]	602.[4]	2072.[4]	7.8	325	Not det.

NOTE. - Exponents indicate relative standing.

Penetration

Table 1 shows that very little relation exists between penetration and the temperatures attained within universal joints. Sample No. 4 has a fairly low unworked penetration value of 205, and the temperature rise within the universal joint was 35° F. Universal Joint Grease No. 7 was much softer, having an unworked penetration value of 325, yet the temperature rise was 50° F. Universal Joint Grease No. 6 is somewhat heavier than No. 7, but the lower penetration value of 275 apparently did not cause any significant difference in the bearing operating temperature which remained at 50 degrees above atmospheric. It is therefore concluded that from the standpoint of service, the penetration of a universal joint grease is not of paramount importance. It is, of course, fully realized that consistency and viscosity must be taken into consideration in dispensing greases at low temperatures. A range of 300 to 330 worked penetration is recommended. This penetration range should be satisfactory for purposes of manufacturing control.

Percent Water

Most commercial universal joint greases contain less than 1 per cent of water, and while it appears that the moisture content has no direct bearing on efficacy of lubrication, it is felt that a good grease should not contain more than 1.5 per cent of water. At very low temperatures, greases containing no glycerin and much water might be found objectionable.

Melting Point

Inasmuch as the service tests indicate no definite advantages for Nos. 5 and 7 higher melting point greases, particularly from the standpoint of bearing temperatures and wear, it is felt that the point is not especially

TABLE 11.—Analytical Data—Commercial Universal Joint Greases

Grease No.	1	2	3	4	5	6	7	8	9
Physical Properties:									
Appearance	Gran. & Dk. Gr.	Stgy. & Dk. Gr	Stgy. & Dk. Gr.	Stgy. & Dk. Gr.	Stgy. & Dk. Gr.	Stgy & Dk. Gr	Stgy & Dk. Gr.	Stgy. & Dk. Gr	Dk. Green
Odor	Sweet	Soapy	Soapy	Soapy	Soapy	Soapy	Soapy	Soapy	Brown Soapy
Melting point, ° C., Gillette	72	59	49	86	50	80	62	66	Liquid
Melting point, ° C., Ubbelohde	130	82	151	167	143	126	153	143	Liquid
Channel test, ° F.	at 25	at 15	at 30	at 80	at 15	at 30	at 20	at 25	10
Penetration at 77° F., unworked	...	...	...	205	310	275	325	310	...
Penetration at 77° F., worked	...	...	...	...	320	325	330	...	..
MacMichael vis. No. 24 wire at 95° F.	180	100	160	...	...	...	...	...	...
Corrosion, cu. strip, 3 hrs. at 212° F	OK	OK	OK	OK	OK	OK	OK	OK	OK
Bleeding test, 1 hr. at 175° F.	No Sep.	No Sep.	No Sep.	No Sep.	No Sep	No Sep	No Sep.	No Sep	...
Separation test at 220° F.	No Sep.	No Sep	No Sep.	No Sep.	...	...	...	No Sep.	...
Separation test at 250° F.	...	...	...	...	No Sep.	No Sep.	No Sep.	...	Slight
Composition, Per Cent by Weight.									
Soap	14.1	11.4	8.2	14.2	7 8	18 5	7.8	7.9	8 0
Moisture	0.70	0.65	0.1	0.60	0.7	1.5	0.55	0 45	0.6
Oil	85.0	86.0	91.0	86.0	90.7	80.3	91.0	92.5	91.6
Free alkali (or acid)				Neutral					
(oleic acid)	...	...	0.35	...	0 42	...	...	0.42	0.7
(NaOH)	03	.01	...	...	...	0.2	0.09	...	...
Asphaltic material	0.14	0 37	0.26	0.06	0.24	0.04	0 07	0.43	0.01
Sulfur	0.49	0 82	0.96	0 34	0.89	0.32	0 36	0.64	0.45
Ash:									
Per cent by weight	2.61	2.09	1.50	2.59	1 44	3.68	1.61	1.47	0.89
Analysis, percent of ash Na_2CO_3	95 4	92 2	87.2	87.4	82.1	95.2	82.8	87 6	...
Na_2SO_4	4.3	5.5	11.5	9.0	12.2	4.1	8.6	10.9	...
Fe_2O_3	...	...	...	...	1.0	1.1	...	...	...
Al_2O_3	...	...	...	...	...	...	2.9	...	...
CaO	0.4	...	0.5	...	2.0	0 2	2.3	0 9	95.0
Acid ins & Fe_2O_3	0.9	1.1	0 8	4 0	...	...	...	0 9	...
Mineral Oil:									
Viscosity, S.U. at 100° F.	657	1062	247 Fur.	745 Fur.	1986	295 Fur.	2072	1060	2810
Viscosity, S.U. at 210° F.	65	66	109 6	156	102	153	111	66.0	128
Gravity, ° A.P.I.	22.4	17 4	19.7	20.9	20.3	23.7	20.8	17.4	19.0
Flash, ° F.	400	395	415	465	405	515	335	365	405

important if service characteristics alone are taken into consideration. If a high melting point were indicative of good grease retention, we should expect Nos. 5 and 7 to be considerably less subject to leakage. An examination of Table No. 1 will indicate that the opposite of this is true. We therefore conclude that from the standpoint of service, melting point specifications, providing the greases are well made products having soda soap bases, are not essential.

Free Alkali

Our analysis indicates that Grease No. 5 has a free acid content of 0.42 per cent. However, the corrosion test (copper strip, 3 hours at 212° F.) was satisfactory and since wear in the service test was exceedingly low for this product, it must be concluded that the free acid content of even this amount is not a serious disadvantage. While these service tests have not been carried out with the particular view of ascertaining the practical significance of free alkali and free acid, the results obtained are such that no particular service disadvantages could be predicted on the basis of these qualities. It is therefore concluded that the free alkali and free acid specifications appear to have no very useful purpose with regard to service.

Fillers

It is the writer's belief that no grease recommended for universal joint lubrication should contain any appreciable quantity of filler as inert solid materials would greatly accelerate wear.

Ash

The ash content, of course, is some indication as to the percent of soap present in the grease. Since the soap content does not correlate at all well with retention properties, it cannot alone be taken as an indication of serviceability. It will be observed from the above table of results that Universal Joint Grease No. 5, containing only 7.8 per cent of soap, lost 58.8 per cent in 30 hours of operation, as sompared with 54.5 per cent for No. 6, containing 18.5 per cent of soap. This difference in retention properties is not proportional to the difference in soap percentages. Again, Universal Joint Grease No. 1, a nationally advertised product which was found to be lost from the joint to a greater extent than any of the other products tested, contains only 4.4 per cent less soap than No. 6 which had the best retention properties. Therefore, the necessity for specifying the ash or soap content is not at all apparent when considering service results. It is obvious that in order to conform to definite penetration ranges an adequate soap content must be utilized by the grease manufacturer. The fact that the ash content may vary within fairly wide limits does not appear to seriously affect service results.

Corrosion

None of the competitive universal joint greases investigated failed to meet the copper strip corrosion test.

Mineral Oil, Per Cent by Weight and Viscosity

In general, it is felt that the viscosity of the mineral oil should have a range of 2000 to 3000 seconds S.U. at 100° F. It is of particular interest to note that Grease No. 4, containing oil having a viscosity of 7450 at 100° F. is not particularly satisfactory from the standpoint of retention and has been found by these service tests to produce wear practically twice as great as greases containing oils having viscosities falling within the range 2000 to 3000. Two other greases examined have what would be considered as exceptionally low viscosity oil,—No. 1, containing oil having a viscosity of 647 at 100° F.; and No. 2, prepared with an oil having a viscosity of 1062. Both of these products were found to have high throwoff values and the wear data obtained place them as being definitely inferior. Operating temperatures for these two greases containing low viscosity oil were found to be slightly lower, however, than for other greases.

What has already been said above with regard to soap content can also be applied to the percent of mineral oil used for compounding universal joint lubricants. As pointed out above, slight variations in soap content, and consequently oil content, are not reflected in the service results.

The color of the mineral oil is chiefly responsible for the color of the finished grease. It is well known that many customers are overcautious in selecting greases on an appearance and color basis.

The above recommendations and conclusions have been reached after a study was made utilizing the Spicer plain bearing type joint. It is contended that the problems relating to the lubrication of antifriction needle bearing type universal joints are much less complex, due to their superior retention, than those presented by the usual trunnion type joint.

Tests with Needle Bearing Universal Joints

Gear oils and semi-fluid gear greases of the types recommended by the manufacturers of the needle bearing trunnion universal joints are rejected at a rate some ten times greater than that of a grease such as No. 6, while the operating temperatures are substantially the same in each case. At the end of 32 hours' testing, with each of the lubricants used, the rejection curve showed that little further rejection was probable, and that ample lubricant remained in the bearings.

Lubrication failure for needle joints appears possible only through careless assembly or damage to the cork seals, regardless of mileage run.

During the test period, which exceeded 10,000 miles, the wear on the bearings was negligible. The fine lapped finish of the bearing pins did not acquire a full polish during this time.

From the results of the study and testing of the anti-friction type universal joints, the following conclusions may be drawn:

The mechanical excellence of the needle bearing trunnion type universal joints is such that the lubrication problem is reduced to a small item.

The needle bearing trunnion type of universal joint will be used on practically all of the passenger cars using the Hotchkiss drive in the future.

The lubricant consumption of the needle bearing type trunnion universal joint may be from 7.5 to 15 grams during the following periods, as recommended by the manufacturers:

Spicer Types KR and KRL. In the passenger car sizes, no added lubrication is required or recommended. In the large truck and bus sizes, lubrication "as a precaution" is suggested each 3000 to 4000 miles.

Universal Products Company Needle Bearing Trunnion Type. No periodic lubrication is required.

Universal Products Company Needle Bearing Ball Type. (Probably Obsolescent.) Use heavy fiber grease, one ounce per 10,000 miles.

Mechanics Needle Bearing Universal Joints. Disassemble and pack with semi-fluid lubricant every 20,000 to 30,000 miles.

The following graph sheet, Figure 5, presents the performance of the greases tested in a clear form, showing especially the low throwoff of Universal Joint Grease No. 6 with its satisfactory operating temperature.

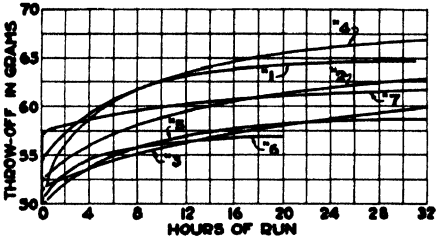


FIGURE 3.—Rejection of Lubricant by Spicer Plain Bearing Universal Joint, Type G.

Speed 3590 R.P.M., 10° angularity between faces. Original charge 100 grams of lubricant.

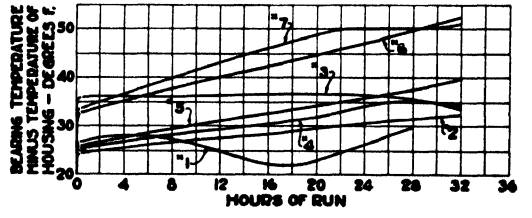


FIGURE 4. — Temperature Increase in Spicer Type G Universal Joint.

Speed 3590 R.P.M., 20 H.P., 10° angularity.

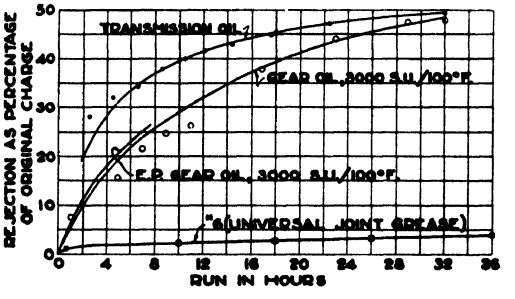


FIGURE 5.—Rejection of Lubricant from Spicer Needle-Bearing Universal Joint, Type KRL.

Speed 3590 R.P.M., load 20 H.P. Universal joint charged with lubricant to 7.5 grams.

Due to the fact that the performance of Universal Joint Grease No. 6 was so satisfactory in service tests, it is concluded that a grease is preferable to an oil.

Mechanical Features of the Needle Bearing Trunnion Universal Joints

Many of the needle bearing trunnion type universal joints produced at present follow the general design which is exemplified by the Spicer type KR. The cork seals are at the inner ends of the trunnion bearings, and are therefore relieved of hydraulic pressure, due to rotation when enough lubri-

cant has been rejected to empty the small passages in the cross member as far as the plane of the inner ends of the pin bearings. This feature assures excellent lubricant retention while running. The only possibility of loss of lubricant appears to be that of damaging the seals while assembling, or of excessive wear of the seals due to dusty conditions, in which case the lubricant, if an oil, could escape while standing.

The second valuable feature common to all makes of this type of universal joint is the provision of thrust surfaces at the outer ends of the pins only, in a position where the last trace of lubricant is effective.

Results of Tests of Needle Bearing Universal Joints with Four Lubricants

The study of the lubrication of the needle bearing type of universal joint was directed along the following lines:

A survey of the number and types of the devices in use.

A study of the construction of each type with attention to provision for lubrication, recommended frequency, type of lubricant and capacity provided for lubricant.

Service testing at high speed (3590 R.P.M.) and high angularity (10°) with a moderate loading of 20 h.p. The observations made were:

a). operating temperature;
b). throwoff of the lubricant;
c). wear of the bearings.

It was found that the gear oils and semi-fluid gear greases of the types recommended by the manufacturers of the needle bearing trunnion universal joints were rejected at a rate some ten times greater than that of "Standardized" Universal Joint Grease, while the operating temperatures were sub-

Make and Model of Car	Make of Universals	Type of Universals
Auburn—All models	Mechanics	Needle bearing trunnion
Buick—All models	Own	Plain bearing trunnion
Cadillac—All models	Mechanics	Needle bearing trunnion
Chrysler—All models	Universal Products	Needle bearing trunnion
Chevrolet	Own	Plain bearing trunnion
DeSoto	Spicer	Plain bearing trunnion
Dodge	Universal Products	Needle bearing ball
Essex-Terraplane	Spicer	Needle bearing trunnion
Ford 4 and 8 cylinder	Own	Plain bearing trunnion
Franklin—All models	Mechanics	Plain bearing trunnion
Graham—All models	Spicer	Needle bearing trunnion
Hudson	Spicer	Needle bearing trunnion
Hupmobile—All models	Universal Products	Needle bearing trunnion
LaSalle	Spicer	Needle bearing trunnion
Lincoln—All models	Spicer	Plain bearing trunnion
Marmon 16	Spicer	Plain bearing trunnion
Oldsmobile—All models	Spicer	Needle bearing trunnion
Packard	Spicer	Needle bearing trunnion
Pierce-Arrow 840A 1240A	Universal Products	Needle bearing trunnion
Pierce-Arrow 1248A	Spicer	Needle bearing trunnion
Plymouth	Universal Products	Needle bearing ball
Pontiac	Own	Plain bearing trunnion
Reo—All models	Universal Products	Plain bearing ball
Studebaker	Mechanics	Needle bearing trunnion
Willys	Universal Products	Needle bearing ball

TABLE 12.—Application of the Most Important Types of Universal Joints, U. S. A.

Make of Universal Joint	Type of Universal Joint	Total No. 1932 Cars Using	Per Cent of Total, 1932	Total No. 1933 Cars Using	Per Cent of Total, 1933	Per Cent of Total Increase or Decrease 1932 to 1933	Remarks
Univ. Prod. Co.	Needle bearing trunnion	11,655	1.1	37,345	2.5	+ 1.4	Auburn changed to all Mechanics needle bearing trunnion. Hupmobile put Univ. Prods. needle bearing trunnion on all models.
	Plain bearing ball	190,775	17.4	6,730	0.5	−16.9	
	Needle bearing ball	0	0	372,305	24.9	+24.9	
Spicer	Plain bearing trunnion	159,740	14.6	105,240	7.0	− 7.6	Cadillac changed to Mechanics needle bearing trunnion. Terraplane, Graham, Hudson, LaSalle, Oldsmobile, Packard and Pierce-Arrow all changed from Spicer plain to needle bearing trunnion.
	Needle bearing trunnion	0	0.0	355	0.0	+ 0.0	
Mechanics	Plain bearing trunnion	28,025	2.6	23,020	1.6	− 1.0	Cadillac changed to Mechanics needle bearing trunnion. Studebaker changed all models to needle bearing trunnion.
	Needle bearing trunnion	0	0.0	0	0.0	...	
Own, enclosed, used with torque tube	Plain bearing trunnion	679,420	61.9	914,760	61.3	− 0.6	No changes. These universal joints are used with torque tube drive in Buick, Chevrolet, Ford, Pontiac.

All other makes	All other types	26,795	2.4	34,050	2.2	— 0.2
Total, all makes	All models	1,096,410	100.0	1,493,805	100.0	...
Total, Univ. Prods.		202,430	18.5	316,380	27.4	+ 8.9
Total, Spicer		159,740	14.6	105,595	7.0	— 7.6
Total, Mechanics		28,025	2.6	23,020	1.6	— 1.0
Total, Enclosed (torque tube)		679,420	61.9	914,760	61.3	— 0.6
Total, Other makes		26,795	2.4	34,050	2.2	— 0.2

Type of Universal Joint	Total No. 1932 Cars Using	Per Cent of Total, 1932	Total No. 1933 Cars Using	Per Cent of Total, 1933	Per Cent of Total Increase or Decrease 1932 to 1933
Total Plain bearing trunnion	867,185	79.0	1,043,020	69.9	— 9.9
Total Needle bearing trunnion	11,655	1.1	37,700	2.5	+ 1.4
Total Plain bearing ball	190,775	17.4	6,730	0.4	—17.0
Total Needle bearing ball	0	0.0	372,305	24.9	+24.9
Total All others	26,795	2.5	34,050	2.3	— 0.2

TABLE 13.—Lubrication Recommendations by Universal Joint Manufacturers

Maker	Type	Lubrication	Cars	Year Models	Estimated Total Number for 1932 and 1933
Universal Products Company	Plain bearing ball without spline 200, 400, 500, 3500 Series	1 to 2 ounces of heavy fiber grease made for Universal Joints, every 3000 miles. No spline lubrication.	Auburn	Before 1932	
			Dodge	Before 1932 and 1932	
			Chrysler	Before 1932 and 1932	
			De Soto	Before 1932 and 1932	
			Graham-Paige	Before 1932 and 1932	
			Hupp.	Before 1932 and 1932	
			Cord	Before 1932	
			Oldsmobile	Before 1932	
			Reo	Before 1932 and 1932	
			Stutz	Before 1932	
			Plymouth	Before 1932 and 1932	
			Total cars, including 1932 Very few after 1932		181,000 cars
Universal Products Company	Needle bearing trunnion with spline 600, 700, 5200 Series	No periodic lubrication of Universal Joint. 1 ounce of Transmission Lubricant in spline every 5000 miles.	Auburn	1933	
			Hupp.	1932 and 1933	
			Chrysler	1932 and 1933	
			Studebaker	Before 1932	
			Pierce-Arrow	Before 1932	
			Reo	1932 and 1933	
			1932–1933 Total		56,150 cars
Universal Products Company	Needle bearing ball without spline 4200—Series	Heavy Fiber Grease made for Universal Joints. 1 ounce every 10,000 miles.	Plymouth	 1933	
			De Soto	 1933	
			Dodge	 1933	
			1932–1933 Total		356,990 cars
Mechanics Universal Joint Co.	Plain bearing trunnion	Heavy Oil, enough to fill.	Franklin	 1933	
			Studebaker	 1933	
			Rockne	1932	
			Hupp.	1932	
			Packard	1932	
			Total 1932–1933		62,180 cars

Mechanics Universal Joint Co.	Needle bearing trunnion	Lubricate after 20,000 miles by disassembling joint and packing in a high grade semi-fluid lubricant. Same type of lubricant in spline every 2500 miles.	Auburn	1932 and 1933	
			Studebaker	1933	
			Cadillac	1933	
			Busses	1933	
			Trucks	1933	
			Total		3,000 cars
Spicer	Plain bearing trunnion Closed type I-G	See list of greases approved by Spicer (These are all heavy soda-base products).	Cadillac	1932 and 1933	
			Dodge	1932	
			Essex	1932 and 1933	
			Franklin	1932	
			Graham	1932 and 1933	
			Hudson	1932 and 1933	
			La Salle	1932 and 1933	
			Hupmobile	1933	
			Lincoln	1932 and 1933	
			Marmon	1932 and 1933	
			Oldsmobile	1932 and 1933	
			Packard	1933	
			Total for 1932–1933		265,000 cars
Spicer	Needle bearing trunnion	See list of greases approved by Spicer.	Willys	1933	
			Total		355 cars
			But the 1934 tests indicate that the needle bearing trunnion is rapidly replacing the plain bearing trunnion.		
Own—Buick	Plain bearing trunnion stationary housing	Lubricated automatically from the transmission.	Buick	in 1932 and 1933	93,000 cars
Own—Chevrolet	Plain bearing trunnion stationary housing	Lubricated automatically from the transmission.	Chevrolet	in 1932 and 1933	797,000 cars
Own—Ford	Plain bearing trunnion	Universal Joint Grease. Soda Soap Grease.	Ford	in 1932 and 1933	570,000 cars
Own—Pontiac	Plain bearing trunnion	Lubricated automatically from the transmission.	Pontiac	in 1932 and 1933	133,000 cars

TABLE 14.—Relation Between Worked and Unworked Penetration for Standardized Universal Joint Greases.

Batch No.	Unworked Penetration	Worked Penetration	Breakdown
163	299	315	16
...	290	293	3
2372	278	293	15
2088	295	358	63
2132	287	312	25
Batch of 11-23-34	280	295	15
		Average Breakdown	22.8

stantially the same in each case. At the end of 32 hours testing, with each of the lubricants used, the rejection curve showed that little further rejection was probable, and that ample lubricant remained in the bearings.

A lubrication failure appeared possible only through careless assembly, or damage to the cork seals, regardless of mileage run.

TABLE 15.—Spicer Needle Type Joint Test; Results of a Typical Test

Grams of Lubricant Charged to Joint No. 1, 5.4 + (2.0 at end 5 hours) = 7.4.
Grams of Lubricant Charged to Joint No. 2, 5.9 + (0.7 at end 5 hours) = 6.6.
Lubricant Used: "Standardized" Universal Joint Grease.

		Bearing Temperature ° F.						Lubricant Throw off				
				Above Housing Temp.		Average No. 1 and No. 2	Above housing Temp.	Grams				
No. Hours Run	Housing Temp. ° F.	No. 1	No. 2	No. 1	No. 2			No. 1	No. 2	Average	Speed R.P.M.	Load B.H.P.
0	88	88	88	0	0	88	0	...	...	...	3590	20
½	100	112	105	12	5	108½	8½	0.1	0.1	0.1	"	"
1	100	110	110	10	10	110	10	...			"	"
2	100	106	112	6	12	109	9	...		...	"	"
3	101	120	116	19	15	118	17	...		..	"	"
4	100	...	...	..	..	...	...	...	.	...	"	"
5	98	120	112	22	14	116	18	...	.	...	"	"
6	98	116	111	18	13	113½	15½	...	.	...	"	"
7	96	110	105	14	9	107½	11½	...	.	..	"	"
8	96	110	107	20	17	108½	18½	...	.	...	"	"
9	87	108	102	21	15	105	18	. .	...	...	"	"
10	90	105	100	15	10	102½	12½	0.2	0.3	0.25	"	"
11	82	97	93	15	11	95	13	. .		...	"	"
12	82	100	95	18	13	97½	15½	..	. .	..	"	"
13	83	100*	98	17	15	99	16	...	.	...	"	"
14	90	100	100	10	10	100	10	...	. .	...	"	"
15	92	103	101	11	9	102	10	. .	...	...	"	"
16	96	105	107	9	11	106	10	...	...	...	"	"
17	103	114	111	11	8	112½	9½	0.2	0.3	0.25	"	"
19	110	126	123	16	13	124½	14½	...	...	...	"	"
20	107	122	121	15	14	121½	14½	...	...	...	"	"
21	102	120	119	18	17	119½	17½	...	...	...	"	"
22	100	118	116	18	16	117	17	...	...	...	"	"
23	99	114	112	15	13	113	14	...	...	...	"	"
24	98	110	110	12	12	110	12	...	...	...	"	"
25	90	107	105	17	15	106	16½	...	...	...	"	"
26	91	106	105	15	14	105½	14½	0.24	0.35	0.30	"	"
27	83	103	99	20	16	101	18	...	...	...	"	"
28	90	103	102	13	12	102½	12½	...	...	...	"	"
29	95	105	102	10	7	103½	8½	...	...	...	"	"
30	98	108	105	10	7	106½	8½	...	...	...	"	"
31	101	114	113	13	12	113½	12½	...	...	...	"	"
32	110	130	130	20	20	130	20	0.31	0.42	0.37	"	"

* Indicates one of thermocouples replaced.

During the test period, which exceeded 10,000 miles, the wear on the bearings was negligible. The fine lapped finish of the bearing pins did not acquire a full polish during this time.

The results of the study and testing of the antifriction type universal joints are shown graphically in Figure 5.

Universal Joints Used on 1934 Model Cars

The table on page 529 describes the universal joints installed on cars in 1934.

Standardized Universal Joint Greases

The formulae for greases suitable for summer and winter operation are given below. The product having a worked penetration value of 300 to 330 is probably best suited as a year round lubricant. The viscosity of the oil in both greases is approximately 2,730 S.U.V. at 100° F.

	A.S.T.M. Worked Penetration 250-300			300-330		
	Gallons	Pounds	Per Cent by Weight	Gallons	Pounds	Per Cent by Weight
Tallow	100	760	16.68	100	760	15.50
45° Bé. caustic soda	22	112	2.46	22	112	2.28
1000 red oil	143	1105	24.26	157	1210	24.67
Mid-Continent S.R. stock	339	2579	56.60	371	2821	57.55
		4556	100.00		4903	100.00

TABLE 16.—Spicer Needle Type Joint Test

Grams of Lubricant Charged to Joint No. 1, 7.2. Joint No. 2, 7.5.
Lubricant Used: Mid-Continent S.R. Stock.

No. Hours Run	Housing Temp. ° F.	Bearing Temperature ° F. No. 1	No. 2	Above Housing Temp. No. 1	No. 2	Average No. 1 and No. 2	Above housing Temp.	Lubricant Throw off Grams No. 1	No. 2	Speed R.P.M.	Load B.H.P.
0	84	90	90	6	6	90	6	...		3590	20
½	90	103	123	13	33	113	23	0.21		"	"
Down ½ hour										"	"
1	90	102	111	12	21	106½	16½	0.55		"	"
2	89	104	110	15	21	107	18	...		"	"
3	91	104	112	13	21	108	17	1.09		"	"
4	90	98	101	8	11	99½	9½	...		"	"
5	92	105	109	13	17	107	15	1.32		"	"
6	91	108	113	17	22	110½	19½	...		"	"
7	93	112	119	19	26	114½	23½	1.54		"	"
8	95	114	121	19	26	117½	22½	...		"	"
9	100	118	123	18	23	120½	20½	1.78		"	"
10	10	119	128	19	28	123½	23½	...		"	"
11	102	119	134	17	32	126½	24½	1.91		"	"
12	99	125	128	26	29	126½	27½	...		"	"
13	95	111	118	16	23	114½	19½	2.13		"	"
14	93	112	120	19	27	116	23	...		"	"
15	89	112	117	23	28	114½	24½	2.60		"	"
16	88	119	116	31	28	117½	29½	...		"	"
17	93	105	112	13	19	108½	25½	2.75		"	"
18	83	108	112	25	19	110	27	...		"	"
19	90	105	111	15	21	108	18	2.87		"	"
20	115	93	98	−22	−17	95½	−19½	...		"	"
21	87	101	106	14	19	103½	16½	3.10		"	"
22	90	109	114	19	24	111½	21½	...		"	"
23	93	106	114	13	21	110	17	3.20		"	"
24	95	109	115	14	20	112	17	...		"	"
25	100	110	115	10	15	112½	12½	3.28		"	"
26	100	113	121	13	21	117	17	...		"	"
27	102	118	124	16	22	121	19	3.35		"	"
28	105	117	127	12	22	122	17	...		"	"
29	111	119	121	8	10	120	9	3.45		"	"
30	100	114	122	14	22	118	18	...		"	"
31	98	112	120	14	22	116	18	...		"	"
32	93	111	16	18	23	113½	20½	3.47		"	"

Contaminated by throwoff from spline

Service Tests on Standardized Universal Joint Grease and Mid-Continent S.R. Stock

The results of the tests tabulated in Tables 15 and 16 indicate clearly the advantages of using a suitable grease rather than oil, particularly from the standpoint of consumption, in a Spicer Needle type joint.

Suggested Specifications for Standardized Universal Joint Grease

The following specifications are considered as suitable for use in control of the manufacture of universal joint grease.

Appearance	Standard sample
Worked penetration at 77° F. (A.S.T.M. D217-27T)	300 to 330
Water, per cent by weight (A.S.T.M. D95-30)	1.5 Max.
Melting point	Unnecessary
Free alkali, per cent (A.S.T.M. D128-27)	0.5 Max.*
Free acid, per cent (A.S.T.M. D128-27)	0.3 Max.*
Fillers, per cent (A.S.T.M. D128-27)	None
Ash, per cent (A.S.T.M. D128-27)	Unnecessary
Corrosion (U.S.B.M. 530.5)	None*
Mineral oil, per cent by weight (A.S.T.M. D128-27)	Unnecessary
S.U.V. at 100° F.	2000 to 3000
Color (A.S.T.M.)	Unnecessary
Soap, per cent by weight (D128-27)	Unnecessary
Kind of soap	Sodium

* Not considered essential.

Estimated Cost of Standardized Universal Joint Grease

TABLE 17.—Formula and Estimated Cost for No. 5 Dark Fiber Grease (Universal Joint Grease)

Estimated formula for grease of unworked penetration of 250 to 300.

	Quantity				Unit Cost			Total Cost		
Description of Item	Per Cent by Weight	Gals.	Lbs.	Unit of Cost	A c	B c	C c	A $	B $	C $
Tallow	16.68	100	760	Lb.	6.25	7.50	6 88	47.50	57.00	52.29
Caustic soda	2.46	...	112	Lb.	2.85	3.42	3.14	3 19	3.83	3.52
1000 red oil (Gulf Coast)	24.26	143	1105	Gal.	3.84	6.00	4.68	5.49	8.58	6.69
M-C S.R. stock	56.60	339	2579	Gal.	11.71	14.05	12.88	39.70	47.63	43.66
Compounding cost	13.56	...	4556	Bbl. of 336 lb.	30.20	52.90	37.80	4.10	7.17	5.13
Cost—Ex Kettle			4556	Lb.	2.19	2.73	2.44	99.98	124.21	111.29

Formula for grease meeting specifications of worked penetration of 300 to 330.

Tallow	15.50	100	760	Lb.	6.25	7 20	6.88	47.80	57.00	52.29
Caustic soda	2.28	...	112	Lb.	2.85	3.42	3.14	3.19	3.83	3 52
1000 red oil (Gulf Coast)	24.67	157	1210	Gal.	3.84	6.00	4.65	6.03	9.42	7.35
M-C S.R. stock	57.55	371	2821	Gal.	11.71	14.05	12.88	43.44	52.13	47.78
Compounding cost	14.59	...	4903	Bbl. of 336 lb.	30.20	52.90	37.80*	4.41	7.72	5.52
Cost—Ex Kettle			4903	Lb.	2.13	2.65	2.38	104.57	130.10	116.46

English Service Tests on Universal Joint Lubricants

Table 18 is a tabulation of results obtained for a series of greases tested in a Hardy-Spicer Joint No. 100, operating at 2,500 R.P.M. at 5.7 B.H.P. (12 pounds—feet) and at an angle of 7 degrees.

TABLE 18.—Results of Grease Tests in a Hardy-Spicer Joint No. 100

A signifies the universal joint nearer to the motor and B the joint nearer the dynamometer.

Test No.	Period of Test, Hours	Grease	Grease Initially Put in, Gms.	Grease Remaining at End of Test, Gms.	Condition of Grease at End of Test
1	10	As received from makers.	A.	31.0	Good.
			B.	35.5	Good.
2	20	Ambroleum.	119-A.	24.5	Resembled salad cream.
		V.2745.W.	68-B.	35.0	Good.
3	20	No. 2 Fiber Grease.	89-A.	28.1	Some dry soapy material had formed on the ring.
		No. 4 Fiber Grease.	74-B.	38.7	Good.
4	20	Emulsion Grease.	101-A.	15.0	Shows free water.
		"Oiline."	148-B.	30.0	Good.
5	5	Venez. Cyl. Oil.	51.5-A.	10–15	.....
		Penna. Br. Stk.	73-B.	10–15	.....
6	20	Colyear Univ. Compd.	72-A.	23.0	Streaky yellow patches in the dark grease.
		Dixon's Grease No. 672.	57-B.	31.0	Good.
7	20	Keystone Velox No. 1.	64-A.	26.5	Rather softer than the original grease.
		Havoline Univ. Joint.	80-B.	12.5	Very soft.
8	20	Sonneborn S.U.J.	60-A.	15	Worked into a cream. Did not stay well on working parts.
8	20	Keystone Velox No. 2.	93-B.	6.5	Worked into a more oily condition; kept well to working parts.
9	20	Polarine Fiber Grease.	92-A.	12.5	Formed yellow emulsion.
9	20	Standard Universal Grease (N. J.)	66.5-B.	27.5	Kept in good condition.
10	20	V.2745W.	81-A.	21.5	O.K. on collar, but oily on knuckle (had the consistency of a bright stock).
10	20	Wadham's Tempered U. J. Grease.	71-B.	32	Slightly soft, but O.K.
11	20	New V.2745W.	76.1-A.	22.8	Soft oily mass having lost all "sponginess."
11	20	V.2745W (as used in test No. 2).	77.2-B.	34.2	O.K.
12	20	Petroleum Jelly.	66.9-A.	17.5	Appearance unchanged.
12	20	V.2745W (German).	92.9-B.	29.3	Worked into a creamy emulsion.
13	20	No. 4 Fiber Grease (German).	96.3-A.	29.0	Worked into a white emulsion resembling paint.
13	20	German Fiber.	76.9-B.	20.5	
14	20	Ambroleum.	101.2-A.	20.40	As with test 2 resembled salad cream.
14	20	V.40932.	102-B.	10.5	Considerable amount flung out and mixture went very oily. Both above adhered fairly well to all parts.

TABLE 17.—(*Continued*)

A signifies the universal joint nearer to the motor and B the joint nearer the dynamo.

Test No.	Period of Test, Hours	Grease	Grease Initially Put in, Gms.	Grease Remaining at End of Test, Gms.	Condition of Grease at End of Test
15	20	V.40820.	81.5-A.	22.8	Both greases became yellowish emulsions, but the "Tackiness" of 40821 kept it on the working parts better than 40820.
15	20	V.40821.	98-B.	28.8	
16	20	Oiline.	51.0-A.	26.2	O.K. No change.
16	20	V.40822.	55.7-B.	27.8	Gone to emulsion as with 40821 and 40820.
17	20	V.2745W.	99.7-A.	26.0	Grease had consistency of Bright Stock, flowing easily.
17	20	V.2745W.	112.2-B.	26.0	
18	20	"Castrol" Unijoynt.	83-A.	29.5	Very good.
18	20	"Castrol" Unijoynt.	86.8-B.	33	Appears to be the best grease so far tested.
19	20	Texas "Marfak 1."	100-A.	25	Thinned out slightly.
19	20	Texas "Marfak 3."	94-B.	28	Thinned out slightly.
20	20	Skefko (Swedish Sample).	99-A.	21	Good.
20	20	Skefko (Japanese Sample).	100-B.	27	Good.
21	20	German Fiber Grease.	86.5-A.	21	Became much softer in use.
21	20	R.B. Grease.	82-B.	29	Became much softer in use.
22	20	V.2745W.	100.5-A.	29.8	Very good.
22	20	V.41106 (German).	105.5-B.	25.2	Oily and sloppy.
23	20	Dark Fiber Grease No. 4.	91.8-A.	26	Became very oily.
23	20	V.41049.	134.3-B.	34	Remained unchanged. Very tenacious and efficient. It was not possible completely to remove all the grease in the rig for weighing. This is the best grease tested.

Black Universal Joint Grease

The following formula has been used with success for manufacturing a dark adhesive universal Joint Grease. For heavy duty service, in trucks and road building machinery, it is sometimes mixed with about 50 per cent by weight of good quality wool yarn.

No. 2 tallow	800 pounds
40° Bé. caustic soda	360 "
Western black oil	1000 gallons

Viscosity 71 at 210° F. (Contains 20 per cent soft asphaltic residuum)

Analyses of Commercial Universal Joint Greases

The following tabulations of analyses indicate clearly the composition and characteristics of Universal Joint Greases sold in the United States during the past five years:

Designation	UN	T-M No. 3	AL U-Joint	ZN	SP
Physical Properties:					
Appearance	Stringy and Dark Green	Stringy and Dark Green	Granular and Dark Green	Stringy and Dark Green	Stringy and Dark Green
Odor	Soapy	Soapy	Sweet	Soapy	Soapy
Melting point ° C. Ubbelohde	143	167	130	82	151
Melting point ° C. Gillette	66	86	72	59	49
Pour point ° F.	Too heavy	Too heavy	Too heavy	Too heavy	Too heavy
Channel test ° F.	at 25	at 80	at 25	at 15	at 30
Cone penetration at 77° F.	310	205	....	....	....
MacMichael viscosity, No. 24 wire at 95° F.	*	*	180	100	160
Corrosion Cu. strip, 3 hrs. at 212° F.	O.K.	O.K.	O.K.	O.K.	O.K.
Bleeding test, 1 hr. at 175° F.	No sep.	No sep.	No sep.	No sep.	No sep.
Separation test at 220° F.	No sep.	No sep.	No sep.	No sep.	No sep.
Composition, per cent by weight:					
Soap	7.9	14.2	14 1	11.4	8.2
Moisture	0.45	0.60	0.70	0.65	0.1
Oil	92.5	86.0	85 0	86.0	91.0
Free alkali or acid	Oleic Acid 0.42	Neutral	NaOH .03	NaOH .01	Oleic Acid 0.35
Asphaltic material	0.43	0.06	0.14	0.37	0.26
Sulfur	0.64	0.34	0.49	0.82	0 96
Ash, per cent	1.47	2 59	2.61	2.09	1 50
Analysis (percent of Ash)					
Na_2CO_3	87 6	87.4	95.4	92.2	87.2
Na_2SO_4	10.9	9.0	4.3	5 5	11.5
CaO	.9		0.4		0.5
Acid Insoluble + Fe_2O_3	.9 (Fe_2O_3)	4.0	0.9	1.1	0.8
Mineral Oil:					
S.U.V. at 100° F.	1060	745 (Furol)	657	1062	247 (Furol)
S.U.V. at 210° F.	66 0	156	65	66	109.6
Gravity ° A.P.I.	17.4	20.9	22.4	17.4	19.7
Flash, ° F.	365	465	400	395	415

	S-E	S-P	S-C
Physical Properties:			
Appearance	Dark Green Stringy Soapy	Dark Green Stringy Soapy	Dark Green Stringy Soapy
Odor			
Melting point ° C.			
a. Gillette	50	62	80
b. Ubbelohde	145	153	126
Channel test ° F.	at 15	at 20	at 30
Penetration at 77° F.			
Unworked	310	325	275
Worked	320	330	325
Corrosion, Cu. strip, 3 hrs. at 212° F.	O.K.	O.K.	O.K.
Bleeding test** 1 hr. at 175° F.	None	None	None
Separation test** at 250° F.	None	None	None
Composition, per cent by weight:			
Soap	7.8	7.8	18.5
Moisture	0.7	0.55	1.5
Oil	90.7	91.0	80.3
Free alkali, (or acid)			
(oleic acid)	0.42		
(NaOH)		0.09	0.2
Asphaltic material	0.24	0.07	0.04
Sulfur	0.89	0.36	0.32
Ash:			
Per cent by weight	1.44	1.61	3.68
Analysis (per cent of ash) Na_2CO_3	82.1	82.8	93.2
Na_2SO_4	12.2	8.6	4.1
R_2O_3	1.0		1.1
Al_2O_3		2.9	
CaO	2.0	2.3	0.2
Mineral Oil:			
S.U.V. at 100° F.	1986	2072	295 Furol
S.U.V. at 210° F.	102	111	153
Gravity ° A.P.I.	20.3	20.8	23.7
Flash, ° F.	405	335	515

* Greases too heavy for pour point and MacMichael viscosity

** "None" denotes no separation.

WHEEL BEARING GREASES

General Discussion of Automotive Wheel Bearing Greases

Considerable trouble has been entailed as regards the performance in service of wheel-bearing greases. Such trouble has generally manifasted itself in one or more of the following ways, namely: (1), emulsification of the grease; (2), lack of lubrication of the rolls; (3), corrosion of the various parts of the wheel-bearing assembly; and (4), excessive throwoff of the grease into the brake drums.

As regards the first point, emulsification of the grease, such has often been the case with front wheel bearings using a soda base grease for lubrication. This results from the absorption, into the grease, of water from external sources and from the condensation of humidity from the air resultant from changes in external temperatures. Such emulsification results in: (1), loss of consistency, increasing the danger of throwoff the grease into brake drums; (2), corrosion of the metallic surfaces.

Considering the second point, inadequate lubrication of the rolls, the observation has been quite general that rolls of wheel spindle bearings are dry in service, even with greases bordering on those of the fluid type. In regard to the manner in which the grease distributes itself around the rolls, it appears that instead of the grease itself being in contact with the rolls, it

is apparently displaced from this contact by a thin layer of air which is carried with the roll, leaving the surface of the latter, as well as the race, quite dry. However, in spite of the insufficiency of lubrication, the deterioration of the rolls observed has not been excessive as a general rule, although cases are not uncommon where heavy scoring and abrasion has been observed. This latter condition is encountered more on trucks under heavy loads.

Corrosion of the various parts of the wheel-bearing assembly has also been quite prevalent, and, while it is believed that emulsification of the grease is a contributing factor, this corrosion is encountered with greases of all types of soap base. This corrosion exists chiefly under the inner race, even when extreme care is taken to have the latter well coated with the grease at the initiation of a run. Also, this corrosion cannot be attributed to the grease since lengthy tests in the laboratory have failed to indicate any corrosive action of the latter. It, therefore, appears that the corroded condition is the normal one, and is resultant primarily from the oxidation of suspended metallic particles coming from wear, as well as the oxidation of surfaces from which any protective grease film has been removed by the concentrated loads entailed.

Two factors, somewhat inter-related, are responsible for excessive throw-off of a wheel-bearing grease onto the brake drum. Either the grease may be of too low a penetration, or its melting point may be exceeded by the operating temperature of the wheel bearing parts. In addition, the consistency may have undergone a change in service resultant from emulsification, or possibly by being worked. The mal-effect of throwoff of grease for any of these reasons, is not only loss of grease, but the possibility of complete failure of braking effect on the front wheels.

Comparison of Sodium, Calcium and Aluminum Base Wheel Bearing Greases

The points of superiority for each type of grease are summarized:

With respect to emulsification with water, the lime base greases are quite resistant. The soda base greases are, of course, easily emulsified, but this may be prevented by keeping water out of the bearings.

With respect to lubrication of the rolls, no consistent wheel-bearing lubricant has been found which will effect this in an entirely satisfactory manner.

As regards prevention of corrosion, service conditions have revealed corrosion attendant with the use of greases of all three types of bases, and it does not appear as though the grease is responsible since laboratory corrosion tests failed to indicate any corrosive action; it does not appear possible to retain a grease film to protect against corrosion, at least with the high unit loads encountered.

As regards the temperatures at which greases of the various types will be thrown-off, in the worst condition (bearings packed full) it appears that fiber greases will be lost at about 400° F., aluminum stearate greases at 250° F., and lime base greases at about 200° F.; the amount of grease packed into the space between the bearings has a large effect on the throwoff temperature, however.

It has been the object of many investigations to determine the most suitable type of grease for the lubrication of wheel spindle bearings, from the above considerations regarding: (1), reduction of the tendency towards

emulsification of the grease; (2), lubrication of the rolls; (3), prevention of corrosion of the wheel-bearing surfaces; and (4), reduction of the tendency of the grease to throwoff from the bearings.

A summary of several wheel bearing grease investigations in which various laboratory service testing devices were employed will be included in this section.

In one series of tests a practical wheel bearing tester was utilized as shown in Plate XXII. Greases were tested with it at speeds equivalent to 40 M.P.H. and for a duration of one thousand miles. The tester consisted essentially of two automotive front wheel assemblies mounted on a 4-inch H-beam, the wheels being driven by the rotating drum of a chassis dynamometer. As shown in the illustration, the H-beam on which the wheels are mounted, is tied in at a point approximately two feet from the center

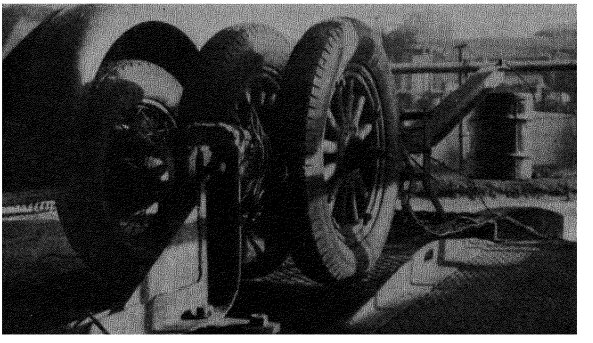


PLATE XXII.—Chassis Dynamometer Attachment for Making Wheel Bearing Grease Tests in Loaded Front Wheels.

Heat supplied by electric resistance coils to simulate conditions of braking.

line of the wheels; the beam has a total length of approximately 12 feet, loading being applied at the end farthest from the wheels. The lever ratio is 6.40 to 1, giving a total effective load of 1312 pounds on the two wheel spindles with the application of a 100-pound load at the free end of the H-beam; this includes, of course, the weight of the beam itself. The wheel-bearing assembly is the customary type used in automotive work, consisting of two spindles with two Timken roller bearings on each spindle. Operation of the wheel bearing tester is, of course, only possible during the operation of a car on the Chassis Dynamometer. Heating was accomplished by means of five heaters, one of which passed directly through a $\frac{3}{8}$-inch hole in the spindles, being insulated by means of porcelain tubing, the heating capacity being 525 watts. In addition a 1000 watt strip heater was mounted on each side and on the top and bottom flanges of the 4-inch H-beam. These four heaters were covered with asbestos board and molded asbestos for a distance of 20 inches above and below the wheel spindles in order to minimize heat dissipation. Thermal wells were located in each spindle, and the recorded

temperatures are therefore spindle temperatures. By means of this heating system, it was possible to obtain temperatures as high as 600° F., although this was not generally done in view of possible deleterious effects on the grain structure of the spindle steel when held for any length of time at this temperature. It was possible to obtain any desired rate of temperature rise by proper operation of a 10-ohm rheostat on the central heating unit, as well as by shutting on or off one or more strip heaters. In general, an effort was made to obtain a constant temperature gradient, although some deviations from such were encountered. At the initiation of each run the amount of grease packed in the bearing was weighed, and this weight recorded. At the conclusion of each run the amount of grease remaining on each bearing was also determined by weight, the difference between the initial and final weight being the approximate grease consumption. In all cases an effort was made to pack the bearings with the same amount of grease from run to run. There being two spindles available, two greases were submitted to test on each run.

Two series of runs of this type were made, at the equilibrium temperature, and at 200° F. After obtaining no sensible grease leakage on the first series of runs, it was decided to conduct the latter series at the higher tempeatures, with similar negative results on all but the experimental greases.

In addition to these observations, the condition of the rolls was carefully watched. Also, the rolls were wiped with tissue paper in an effort to observe for any possible trace of oil film, with negative results. One exception apparently exists, however, but it is believed that, since the lubricant used was simply a Mid-Continent residual stock with 1.5 per cent rubber latex, the oil was thrown onto the rolls after the instantaneous passing of the load onto each roll; in other words, it is improbable that the oil film actually remained on the roll while the latter was under load.

As regards calculation of the 650 pound load on each spindle resultant from the 100-pound weight at the end of the 12-foot H-beam, plus the weight of the beam, this was accomplished by finding the center of gravity of the beam and applying the general moment equation.

For the determination of the temperatures of throwoff, the following method was used. Constant weights of grease were packed onto the bearings, with the space between bearings packed full, half-full, and empty; the wheel spindle nut was adjusted for a trace of end play; load and speed conditions were identical with those above. The temperature gradient was held sensibly constant, readings being taken every ten minutes. Also, at these intervals, the condition of the grease was noted insofar as was possible by external observations. No felt washers were used in any case. The grease was observed for throwoff at both inboard and outboard bearings by watching for spots on a clean piece of cloth or paper held near to these bearings.

In all cases, the equipment was carefully cleaned with gasoline during the interim between tests in order to prevent any contamination from affecting subsequent results.

TABLE 19.—Tests for General Performance and Lubrication

No grease packed between inboard and outboard bearings; speed, 40 miles per hour; duration of run, 1000 miles; felt retainers used; load, 656 pounds per spindle; Timken Roller Bearings.

Grease	Average Oper. Temp. ° F.	Unworked Pen. at 77° F.	Dropping Point ° C. Before	Dropping Point ° C. After	Roll Lubrication	Grease Lost from Bearings to Inside of Hub—Grams Inboard Bearing	Grease Lost from Bearings to Inside of Hub—Grams Outboard Bearing
Wheel-Bearing Grease (soda base)	91; 94	193	200+	...	None	15; 16	7; 12
Wheel-Bearing Grease	200; 173; 176				None	29; 32; 8	21; 14; 9
Locomotive Gun Summer Heavy (Aluminum Stearate base)	85; 85	267	114	97	None	17; 23	10; 10
Cup No. 4 (lime base)		193 (Wkd.)	101	...			
Ballroll Medium (soda base)	85	214	210	200+	None	11	3
No. 5 (soda base)	86	282	124	134	None	7	13
Ballroll Medium	170	...	...	...	None	0	4
Wheel-Bearing (soda base) grease R	91	205	153	143	None	15	8
Wheel-Bearing (soda base) grease R	207	...	...	...	None	18	9
Wheel-Bearing (soda base) grease P	81	175	156	...	None	13	6
Wheel-Bearing (soda base) grease P	213	...	...	...	None	16	11
Wheel-Bearing (soda base) grease Z	83	230	168	...	None	21	10
Wheel-Bearing (soda base) grease Z	215	...	...	...	None	10	4
Wheel-Bearing (soda base) CY grease	83	233	175	161	None	11	5
Wheel-Bearing (soda base) CY grease	207	...	...	...	None	23	14
77 per cent Brt. Stk; 9.5 per cent oleic acid; 9 5 per cent stearic acid; 4.0 per cent 48 °Bé. caustic soda	213	290	107	...	None	22	12
88 per cent Brt. Stk; 10 per cent oleic acid; 2 per cent 48 °Bé. caustic soda	209	390	80	...	None	21	13
93.5 per cent X-101 Stk; 0 5 per cent latex; 5 per cent oleic acid; 1 per cent 48 °Bé. caustic soda	209	...	48	...	None	31	10
67 per cent Stand. W.B. Grease; 33 per cent No. 4 Cup Grease	208	260	114	...	None	35	15
50 per cent Petrolatum; 3 per cent degras; 20 per cent X-101 Stk.	208	(Fluid)	...	...	None	(All grease thrown out)	
38.5 per cent Asphalt; 38.5 per cent X-101 Stk; 23 per cent No. 4 Cup	208	165	90	...	None	(All grease thrown out)	
67 per cent Stand. W.B. Grease; 33 per cent 642 S.R. Stk.	208	380	...	...	None	(All grease thrown out)	
80 per cent Stand. W.B. Grease; 20 per cent 642 S.R. Stk.	208	335 (Wkd)	200+	...	None	27	7
85 per cent Stand. W.B. Grease; 15 per cent No. 4 Cup.	206	...			None	28	12

Discussion

As regards effecting lubrication of the rolls of the wheel bearing, such was not the case with any grease developed for this express purpose, nor with any competitive grease tested, nor with any Shell product. It does not appear, however, that such lubrication is necessary when loads are not excessive, in view of the absence of any appreciable deterioration of the rolls of the wheel-bearing tester, although adverse conditions of operation were frequent during its 14,785 miles of service. In this regard, it appears that instantaneous loads on the rolls are too great to permit establishment of an oil film, that it is only necessary to lubricate the roll carriers since sliding friction is only entailed here, and that with the pure rolling friction of the rolls it may be only necessary for the lubricant present to effect dissipation of the heat resultant from the internal friction of deformation of the metal.

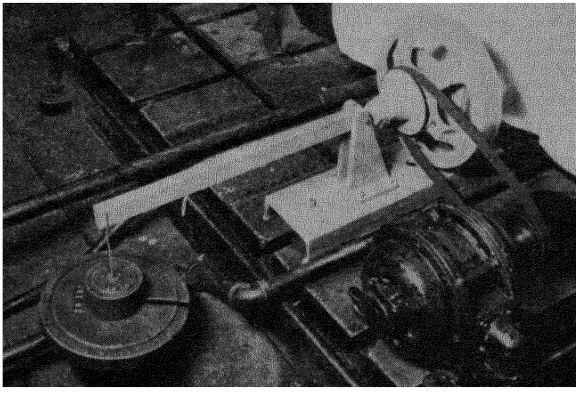


PLATE XXIII.—General Arrangement of Laboratory Apparatus for Making Service Tests on Wheel Bearing Greases.

No throwoff was observed for Standardized Wheel Bearing Grease at temperatures even as high as 400° F., at 40 miles per hour. In regard to this, Aluminum Base Locomotive Gun Grease (Summer Heavy) began to throwoff, with the bearings and space between them packed full, at a temperature of 250° F. Under similar conditions Cup Grease No. 4 was lost at about 200° F. There appears to be no precise correlation between dropping point, as determined in the laboratory and the temperature at which a grease is thrown off from a wheel-bearing, since it appears that the consistency is also involved. Also, the temperature of throwoff is also affected greatly by the amount of grease between the inboard and outboard bearings, this temperature being 50 to 100 degrees greater with this space packed half-full, than with it full or empty.

The results of a series of tests indicated that products of low melting point were lost to an objectionable extent, that visible films of lubricant on

TABLE 20.—Determination of Relative Temperature of Throwoff

Speed, 40 miles per hour; Load, 656 pounds per spindle; no felt retainers; Timken Roller Bearings.

Grease	Soap Base	Grease Between Bearings	Deg./Min. Temp. Grad. 100° Before Throwout	Max. Temp. Reached ° C.	Temp. of Throwoff—Inside Bearing	Temp. of Throwoff—Outside Bearing	A.S.T.M. Unworked Consistency	Dropping Point ° C.
Locomotive Gun	Alum. Stearate	None	2.50	572	200	266	Worked 309	99
Locomotive Gun	Alum. Stearate	None	2.20	332	280	No Loss	" 309	99
Locomotive Gun	Alum. Stearate	½ Full	1.97	489	380	400	" 309	99
Locomotive Gun	Alum. Stearate	Full	2.10	440	250	250	" 309	99
Cup No. 4	Lime	None	2.59	478	No Loss	206	" 193	101
Cup No. 4	Lime	None	2.20	336	No Loss	214	" 193	101
Cup No. 4	Lime	½ Full	2.24	500	350	215	" 193	101
Cup No. 4	Lime	Full	2.24	513	200	200	" 193	101
Stand. Wheel Bearing	Soda	None	2.50	600	No Loss	No Loss	160	219
Stand. Wheel Bearing	Soda	½ Full	1.97	489	No Loss	No Loss	160	219
Stand. Wheel Bearing	Soda	Full	2.10	436	430	No Loss	160	219
W.B. No. 5005	Soda	None	2.16	424	396	No Loss	169	148
W.B. No. 5006	Lime	None	2.16	418	276	220	158	101
B.R.B. "Cal-Oil"	Soda	None	2.59	500	No Loss	No Loss	240	149

the rollers were not formed with any of the lubricants although the molecular layers were no doubt sufficient for adequate lubrication, and in most cases the dropping point of the grease after use in the bearings was decreased.

Another service testing device [248] which has been utilized successfully consisted of a front wheel hub and spindle assembly from a model A Ford car. The arrangement of this spindle and the mechanism employed for loading the bearings in an axial direction are shown completely in Plate XXIII. An axial load of 1336 pounds is produced by a weight of 30 pounds at the end of the lever arm. This is equivalent to producing a total radial load of 7580 pounds on the bearing. Obviously, this load of 7580 pounds is greatly in excess of the normal load which a Ford front axle is required to carry, although shock loads may be even higher. The hub is operated by means of a one-half horsepower motor producing an axle speed of 860 R.P.M.. This

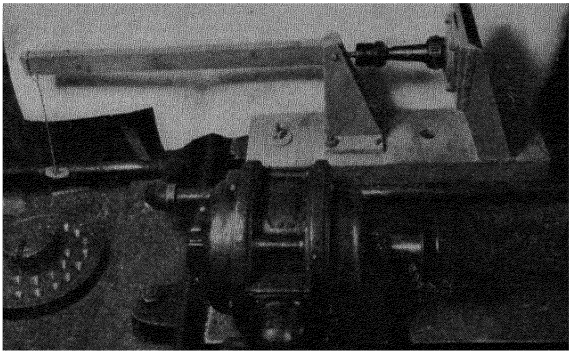


PLATE XXIV.—Wheel Bearing Grease Tester Shown in Figure 10.

Disassembled to show method of applying axial load by means of the weight and lever system. Note thermocouple leads to bearing.

is equivalent to a road speed of approximately 80 miles per hour. Both the loading and speed are considerably in excess of normal conditions for a car operating on the road. This, however, was considered necessary in order to produce accelerated tests.

Thermocouples were placed under the inner race of both spindle bearings and enabled fairly accurate temperatures to be obtained with the bearings in service. The entire wheel bearing grease tester was enclosed in a thin metal housing in order to avoid sudden fluctuations in temperature due to drafts. New outer bearing cones and races were used for each test run. After thoroughly cleaning the bearings with aviation gasoline, they were carefully packed with ten grams of the sample of grease to be tested, the grease being kneaded into the spaces between the Timken tapered rollers and their separator. Later tests indicate that one of the patent bearing packers would accomplish accurate results.

[248] See also Klemgard, E. N., *Ind. Eng. Chem. An. Ed.*, **8**, 83 (Mar. 15, 1936).

TABLE 21.—Analytical Results on Wheel Bearing Greases.

Grease No.	1	2	3	4	5	6	7	8	9	10
Physical Tests:										
Appearance	Bright black Spongy	Brown Slt. Stringy	Green black Stringy	Green brown Stringy	Light brown Stringy	Bright green	Gray brown	Dark green Granular Stringy	Dirty brown Soft Stringy	Brown Granular Stringy
Odor	E. P. base	Fatty	Soapy	Soapy	Soapy	Soapy	Soapy	Soapy	Soapy and like nitro-benzene	Sweet-pungent
Dropping point, ° C.										
Gillette	...	...	100+	80	100+	...	97	...	...	...
Ubbelohde	145	180	162	200+	133	162	109	138	129	139
Unworked penetration at 77° F.	...	...	164	204	199	198	159	155	240	189
Worked penetration at 77° F.	232	236	...	225	280	236	197	...	316	310
Separation test at 250° F.	None	...	None	None	None	None	None	None	None	None
Corrosion, 3 hrs. at 210° F.	O.K.	O.K.	O.K.	O.K.	O.K.	O.K.	O.K.	O.K.	O.K.	O.K.
Composition, per cent by weight:										
Water	1.4	...	1.4	0.1	1.75	0.45	1.8	1.0	1.8	1.1
Oil	...	...	80.3	83.5	79.0	78.5	72.0	79.5	81.0	73.0
Soap	...	...	19.9	16.4	19.0	18.8	23.5	18.4	17.2	22.0
Free alkali as NaOH	0.04	Neutral	0.19	0.12	0.02	0.18	0.05 as $Ca(OH)_2$	0.13	...	0.11
Free acid as oleic	...	...	...	...	...	...	...	...	0.07	...
Ash:										
Per cent by weight	2.86	...	4.14	3.20	3.41	4.02	2.60	3 90	2.95	4.30
Analysis, per cent of ash:										
Na_2CO_3	...	...	89.8	93.3	97.2	89.6	...	95 7	96.9	94.1
Na_2SO_4	...	...	7.0	3.4	2.0	8.5	...	2.8	3.4 Ca_2SO_4	3.9 Na_2SO_4
R_2O_3	..	...	2.2	1.0	0.2	1.5	1.6	1.3	...	...
CaO	...	...	1.0	0.2	0.4	0.3	96.2	...	...	...
Mineral Oil:										
S.U.V. at 100° F.	...	...	348 Fur.	1254	330	282 at 130°	355	803	1867	1440
S.U.V. at 210° F.	179 app.	...	66.1	76 0	50.3	72.2	52.1	63.7	90.4	81.8
Gravity, ° A.P.I.	...	...	22.2	21.1	23 9	25.4	22.1	20.9	19.6	20.2

Temperature readings for the bearings were made hourly and at the same time the housing and atmospheric temperatures were recorded. Following these hourly temperature readings, the driving motor was stopped and the apparatus disassembled for inspection. Care was taken to disturb the grease as little as possible. Observations of the oil films and the color of the surfaces of the rollers and races were made at this time, and particular attention was paid to any changes in the character of the grease. The tendency of the grease to leak from the bearing was also noted.

Results of Tests

The relative ratings of the wheel bearing greases tested are summarized in the following table. The best product tested is given an arbitrary value of 1 and that which is considered most unsatisfactory has been assigned a value of 7.

The temperature-time relationship obtained for the several greases tested is shown more clearly in the graph sheet, Figure 6, than is indicated in

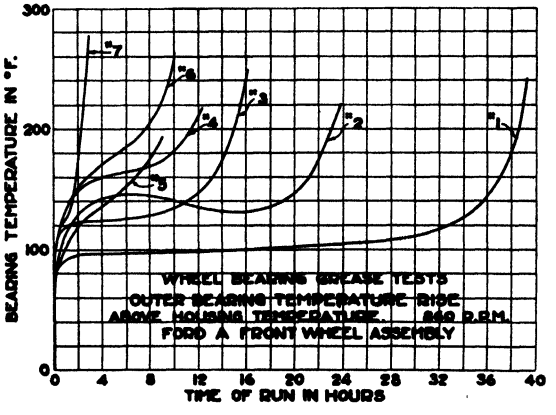


FIGURE 6.—Wheel Bearing Temperature Increases.
Load, 7580 pounds

the table above where the time to reach an objectionably high temperature is variable as well as the rise above the atmospheric temperature. It will be noted that Grease No. 1 was found to be definitely superior in all the qualities observed.

The analyses of the wheel bearing greases tested are shown in Table 21. The following brief comments deal with the laboratory tests made on the greases investigated and discuss the formulae and usual chemical laboratory tests.

PENETRATION

Grease manufacturers are not in general agreement as to whether worked or unworked penetration values should be established. Table 21 of analyses shows wheel bearing greases having variations in unworked pene-

trations ranging from 155 for Grease No. 8 to 240 for Grease No. 9. The worked penetration values range from 197 to 316. Information from other sources indicates that the consistency of competitive wheel bearing greases may vary from a worked penetration value of 170 to values as high as 300. It is, of course, realized that worked penetration values are more significant from the standpoint of service than corresponding unworked penetrations. For a satisfactory grease there should be no objection to establishing a worked penetration range of 210 to 240.

Percent Water

In general, it may be said that the water content of wheel bearing grease at present used throughout the country will vary from a trace to as much as 1.8 per cent. The effect of these variations in water content has not been conclusively demonstrated in the service tests which the author has conducted. It is felt that the presence of appreciable quantities of water is of little significance and it has been found that the experimental grease, containing as much as 1.1 per cent of water, produces no objectionable corrosion or discoloration of used grease or bearing surfaces. It may be possible that a high water content may tend to increase corrosion and wear, although this has not been definitely proven. In order to reduce the tendencies in this direction, one-tenth of one per cent of a material to inhibit corrosion has been added to Grease No. 1. Practical tests have shown that in the presence of this ingredient the water content may reach rather high percentages without detrimental effects to the bearings. A maximum water content of 1.5 per cent is a good working value.

Melting Point

It will be observed, from Table 21, that Ubbelohde dropping points vary from 100° C. for Grease 7 to 200+° C. for Grease No. 4. A study of dropping points in connection with service results and retention in wheel bearings has not conclusively demonstrated that a minimum specification as high as 180° C. is definitely necessary. In view of the fact that field and laboratory service tests have not resulted in noticeable leakage when operating on Wheel Bearing Grease No. 1, having an Ubbelohde dropping point of 145° C., a minimum Ubbelohde dropping point of 135° C. (275° F.) appears to be justifiable.

Percent Free Alkali and Percent Free Acid

There should be no objection to a maximum free alkali value of 0.6 per cent and a maximum free acid value of 0.3 per cent, but many bearing manufacturers specify much closer ranges. It is felt, however, that there should be no difficulty in maintaining wheel bearing greases at a point much nearer to neutral than is indicated by these rather wide ranges. From the standpoint of service, however, there are no indications that products having extreme values within these ranges will be objectionable under actual service conditions.

Percent Fillers

It is generally agreed that any good wheel bearing grease should contain no appreciable quantities of inert fillers, as these materials will act as lapping compounds and cause increased wear.

Percent Ash

Reference to the table of analysis (Table 21) reveals that the soap contents of the wheel bearing greases investigated vary from 16.4 to 23.5 per cent. The best service results were obtained with Grease No. 1, having a soap content of 19.5 per cent. In general, the ash values are proportional to the percentage of soap. However, appreciable deviations in the ash content are not considered as having any particular influence on the serviceability of the grease. It is, therefore, apparent that a maximum ash content of 5 per cent could be readily met by most grease manufacturers and there is no need for meeting rigid specifications established at appreciably lower values. From the standpoint of control and uniformity, however, it is felt that complying with the above suggested worked penetration ranges would serve in a better manner than making ash tests on all batches of wheel bearing grease.

Table 22.—Test of Wheel Bearing Greases

Grease	Time to Reach High Temperatures	Temperature Rise	Appearance of Roller Bearing	Retention of Grease	Final Rating
1	1 (39 hrs.)	1 (250° F.)	1	1	1
2	2 (24 hrs.)	2 (220° F.)	3	2	2
3	3 (16 hrs.)	3 (250° F.)	1	3	3
4	4 (12 hrs.)	5 (220° F.)	6	4	4
5	5 (9 hrs.)	4 (200° F.)	3	5	5
6	6 (10 hrs.)	6 (260° F.)	3	6	6
7	7 (3 hrs.)	7 (275° F.)	7	7	7

Corrosion

None of the greases investigated were found to be unsatisfactory when subjected to the three-hour copper strip corrosion test carried out at 212° F. While it might appear that there is no necessity for including a specification of this kind, in view of the general high quality of greases in this respect, it is felt that since wheel bearing greases may contain sulfurized materials and other E.P. bases, the corrosion test is of importance. There is some evidence from the field that soda soap base wheel bearing greases are corrosive when a small amount of water gains entrance to a bearing. For this reason, the addition to soda soap greases of traces of compounds which inhibit corrosion seems justified.

Mineral Oil Viscosity

With the exception of Wheel Bearing Grease No. 1, none of the products investigated contained oils having a viscosity greater than 100 S.U. at 210° F. In view of field and laboratory experience, an oil viscosity range the values of 160 to 190 seconds S.U. at 210° F. is suggested, this range being met by Grease No. 1.

Soap

The question of soap content has already been discussed under the heading of "Percent Ash" and need not be further commented on at this point. It has been observed that Wheel Bearing Grease No. 7 has been prepared with a calcium base and that in service tests this product has been given a rating of 7, and from all considerations was the most objectionable product investigated. Not emphasized in the following table, is the fact that leakage for Grease No. 7 was many times that of any of the other greases tested. It is significant that all of the other greases were manufactured with soda soap bases.

Film Strength

The necessity for adequate film strength for lubrication of the sliding surfaces along the cone rib of the roller bearing type wheel bearings will now be discussed. The relationship of unusual operating conditions (for example, too tight an adjustment of the bearings) to high unit loads will now be pointed out in connection with the consideration of the importance of film strength in wheel bearing lubrication.

The value of a large program of wheel bearing grease investigations in laboratory wheel bearing machines, for operating periods corresponding to 20,000 or 25,000 miles, is questioned on the grounds that the mild operating conditions necessarily involved would probably fail to show up basic differences in the greases.

While the exclusion of foreign material from a bearing is an important function of a grease, it does not seem probable that any material improvement could be made in this respect by using a special machine to study the exclusion of dust. Retention and consistency are probably the important factors involved so far as the grease is concerned, and these factors are fairly well covered by field experience and laboratory experiments.

In connection with the question of ball bearing type wheel bearings, it may be pointed out that ball bearing type wheel bearings are essentially a simpler problem of lubrication since sliding friction along the cone rib of roller bearing type wheel bearings is eliminated. Although unit loads at rolling contacts are somewhat higher with the ball bearing type, it is considered probable that Experimental Grease E.P.B., (also indicated as Grease No. 1) having an adequate film strength, will prove a superior grease for handling the high loads involved. Its formula is:

	Per Cent by Weight
Tallow	17.3
Caustic soda	2.5
Red oil, 1000 S.U.V. at 100° F.	18.8
Mid-continent steam refined stock, 185 S.U.V. at 210° F.	47.0
120 Penetration steam refined asphalt	8.9
Floyd's extreme pressure base	4.4
Potassium bichromate	0.1
Water	1.0
Total	100.0
Worked penetration	232
Ubbelohde dropping point, ° C.	145

The formula of Experimental Grease P.A.M. 70-B is:

	Per Cent by Weight
Tallow	21.50
Caustic soda	3.15
Motor 70	13.30
120 Penetration steam refined asphalt	18.90
650 Pennsylvania steam refined stock	43.05
Potassium bichromate	.10
Total	100.00
Worked penetration	236
Ubbelhode melting point, ° C.	

ASPHALT CONTENT

With regard to the question of the inclusion of asphalt in Grease E.P.B., it should be noted that the inclusion of asphalt in greases in general is not without precedent. For example, some cup greases contain a certain amount of asphalt. It is the writer's impression that these greases are popular in the field. Greases of this type are also marketed successfully by many manufacturers. Crankpin Lubricant, a jet black grease which is very popular for lubrication under quite a number of conditions, is met with in the lumber industry. It is known that the black color of this grease is due to a certain amount of asphalt.

A dark or black color of a petroleum product may usually be taken as an indication of a certain content of asphalt-like material. A review of many grease products will show that quite a number of them are either dark or black in color. If there were certain fundamental objections to an asphalt content in any and all lubricants, it would seem probable that their manufacturers would be receiving a much larger number of complaints on dark products than on light products. However, such is not the case.

In connection with the development of a superior wheel bearing grease the possibility of realizing an improvement through an increase in oil viscosity has been considered. At the outset it is known that the direct preparation of a grease from high viscosity eastern cylinder stocks would probably offer some difficulty. There is a tendency for fiber greases prepared from eastern cylinder stocks to separate, especially if the water content is reduced too much.

In the development of a grease of a high viscosity oil content, the following points must be taken into consideration.

It is advisable to carry through the first stages of manufacture of the soda base grease using only a relatively light cut of western stock. General experience has been that soda base greases of better texture and better stability were obtained when the first stages of manufacture were carried through with light oil.

Having used light oil in the initial stage of manufacture, a considerable increase in oil viscosity may be obtained through the addition of Midcontinent Cylinder Stock (Midcontinent S.R. Stock, 185 S.U.V. at 210° F.).

Regardless of the use of the Midcontinent stock in the grease, a final high oil viscosity of approximately 170 at 210° F., or higher, is not possible without resort to residual stocks of some sort which would be essentially asphaltic in nature.

The considerations above were the basis for the use of asphalt in Experimental Grease E.P.B. The expectations of superior performance on grease of high oil viscosity were fully realized in tests on the wheel bearing machine.

Direct use of asphalt could have been avoided through a compromise on the light oil content of the grease. This would have involved the reduction of the 1000 Red Oil from 18.8 per cent to approximately 12 per cent and the substitution of 650 Pennsylvania Steam Refined Stock (S.U.V. at 210° F. = 200) for the Midcontinent Steam Refined Stock. The use of the Eastern Cylinder Stock would not have eliminated the presence of all asphaltic materials in the grease, however, since the Eastern Cylinder Stocks contain asphaltic materials which may be recovered by distillation and extraction. The substitution of the dark-colored Eastern Cylinder Stock for the lighter-colored Midcontinent Stock used in the grease would of itself probably involve an increase in the asphalt content. This increase, of course, would be compensated through elimination of the direct addition of asphalt. The net result would seem to be a small reduction in the asphaltic materials present, a change in the character of the asphaltic materials present (due to difference between "eastern" and "western" asphalt) and a general shift in the direction of more saturated hydrocarbons due to the eastern stocks (with resulting increase in the A.P.I. gravity of the oil). Non-purchased stocks would be reduced from 35 per cent of the oil content to 15 per cent of the oil content. This would involve a considerable increase in the cost of the grease.

The question of asphalt content suggests the possibility of use of high viscosity bright stocks instead of cylinder stocks. This of course depends on the availability of any bright stocks of approximately 200 to 225 S.U. viscosity at 210° F. Usually, no bright stocks of this viscosity are available. Furthermore, it would seem to be a matter of doubtful economy to provide special storage facilities for an oil to be used in very small volume for wheel bearing greases alone.

Blend I

	Per Cent by Weight
1000 Red oil	27
Floyd's E.P. base	6
642 Midcontinent stock	67
	100

Timken film strength: 11430 O.K.
12860 Failed

Blend II

	Per Cent by Weight
1000 Red oil	24.0
Floyd's E.P. base	5.5
642 Midcontinent stock	59.0
120 Penetration asphalt	11.5
	100.0

Timken film strength: 11430 O.K.
12860 Failed

Although the thought in mind in preparing a grease of the composition of E.P.B. was the preparation of a grease of high oil viscosity, it is probable that the improved performance of the grease is due largely to the improved film strength of the more viscous oil. This film strength was further improved through the addition of a small percentage of extreme pressure base. This film strength could still be retained even though the asphalt were climinated. Data on the oil blends with and without asphalt are given on the preceding page.

Irrespective of the question of film strengths, the asphalt has certain definite advantages outlined below which are dependent on the oil viscosity and color of the grease:

The asphalt content in Experimental Grease E.P.B. makes it possible to reach an oil viscosity in the neighborhood of 170 to 175 S.U.V./210° F.

The dark color of Experimental Grease E.P.B. (this color is contingent upon the asphalt content) improves the appearance of the grease which has been emulsified with a little water. Lighter greases take on a much more "sloppy" appearance.

The dark color of the grease makes for a minimum evidence of discoloration of the grease under severe operating conditions. Therefore, there would be less likelihood of complaint that the grease would not stand up. Lighter greases show the discoloration very readily.

The dark color of the grease is more effective in concealing the presence of iron oxide which may be formed as the result of wear due to the bearings turning or vibrating on the spindle under practical field conditions. Therefore, there would be less likelihood of a complaint that the product was corrosive due to the iron oxide content.

It is possible that the dark color contributes to the visibility of the oil film on the rolls and on the races. Reports of field tests have indicated that there was a visible film of lubricant on the rolls and races from Experimental Grease E.P.B., while Wheel Bearing Grease No. 4 yielded little or no visible film of lubricant. This difference may be due to a difference in the "availability" of the oil in the grease; that is, there may be a greater tendency for the grease to leave a film of oil on the bearing. However, the asphalt content may have contributed to the visibility of the film. This would tend to reduce the likelihood of a complaint that the bearings were left "dry" by the grease.

Aside from the practical considerations, it is possible that the asphalt may have contributed to the "oiliness" of the wheel bearing grease; that is, the asphalt may have a tendency to reduce the coefficient of friction where lubrication is due to extremely thin films of material. Fatty materials are polar compounds which are known to have this property.

It is possible that asphalt, which is also a polar material, may have a similar effect.

The presence of Floyd's E.P. Base in a lubricant for antifriction bearings cannot be a very objectionable matter since gear lubricants have been used satisfactorily for a number of years for the lubrication of transmissions and differentials. Many oil companies have marketed lubricants containing E.P. bases, for use in transmissions and differentials. Both differentials and transmissions contain antifriction bearings. There seems to be no logical basis for considering the use of these E.P. gear oils for the lubrication of differentials any different in principle from the use of an E.P. base in a grease for lubrication of antifriction bearings.

The Timken Roller Bearing Company has had a tremendous amount of experience with the manufacture and application of roller bearings of

the type used on wheel bearings. It is significant that the Timken Company realizes the limitations of the Timken products to the extent that the necessity for extreme pressure lubricants for lubrication of Timken bearings under certain operating conditions has been admitted. The following quotation is taken from the Timken Engineering Journal for 1933:

"While marked progress has been made in the development of these lubricants (E.P. lubricants) from the standpoint of load carrying ability, some are still inclined to be abrasive and corrosive. Obviously, only these lubricants which have proved satisfactory in every way can be approved for use with Timken Roller Bearings. For this reason, definite recommendations should be obtained from the Timken Engineering Department to meet abnormal operating conditions, extreme pressures or problems involving special lubricants."

The necessity for a lubricant of adequate film strength for severe operating conditions has been confirmed by the experiments carried out with Experimental Grease E.P.B., containing the Floyd's E.P. Base, in which it was found superior to the other greases tested.

The necessity for a lubricant of adequate film strength may readily be seen from the mechanical construction of the bearing. Much of the thrust load on the bearing is carried at the contact surfaces between the ends of the rollers and the cone rib. The total contact area along the cone rib is small. Under abnormal loads due to shocks, excessive loads on the wheel or improper adjustment of the bearing, the unit loads along the cone rib are necessarily exceedingly high as a result of the small contact area. There is also a problem of lubrication under conditions involving sliding friction as compared with rolling friction met with at other surfaces of the bearing. With conditions involving sliding friction and high unit loads, it is obvious that a lubricant of adequate film strength is required if a bearing failure is to be averted.

While the Floyd's E.P. Base, sulfurized fatty oil, undoubtedly contributed to a superior film strength for Experimental Grease E.P.B., it is possible that Floyd's E.P. Base may have made other contributions to the performance of the experimental grease. The following ideas with regard to the "availability" of the oil in the grease are offered as suggestions which may be of interest. While there is no definite proof that these ideas are correct, the ideas are consistent with the experimental results obtained.

Syneresis

The "availability" of an oil in a grease might be defined in terms of a tendency for the grease to "bleed" at a given temperature and furnish a portion of its oil for lubrication. It is possible that an actual measure of this tendency to bleed at various temperatures might be obtained by centrifuging greases at various temperatures and noting the amount of separation. It is probable, too, that this tendency for the grease to bleed is closely associated with the "melting point" of the grease. With high melting point greases the oil is more firmly bound, and relatively high temperatures are required to bring about a tendency for the grease to separate.

Experimental Grease E.P.B. seems to be a grease in which the oil "availability" is high. The low Ubbelohde melting point of the grease, 145° C., (low in comparison with other soda base greases) strongly suggests this possibility. The mechanism through which superior lubrication is obtained with greases of high oil availability is best described by comparison with greases of high melting point and low oil availability.

If the grease is high in melting point and has little tendency to bleed, the grease squeezes out of the bearing surfaces at low temperatures leaving only a very scanty film of lubricant which affords only temporary protection. A slow rise in temperature occurs during which the scanty film of lubricant is oxidized and affords no further protection. A more rapid rise in temperature occurs due to poor lubrication. No oil is immediately available because of the high melting point of the grease. By the time the oil in the high melting point grease is available the bearing is well on the road to failure. The oil running on the hot bearing is rapidly consumed by oxidation * at the high local temperatures in the bearing and a bearing failure soon occurs.

In contrast with the situation with the high melting point grease, the grease of high oil availability yields its oil at a lower temperature. Adequate lubrication is obtained at temperatures which are not sufficiently high to cause rapid oxidation and consumption of the greases. A bearing failure is postponed or averted because the oil in the grease is available for lubrication. Since chemical activity is approximately doubled for every 10° C.

TABLE 23

Ranking and Number of Grease	Grease	Hours to High Temp.	Ubbel. Drop. Point ° C.	Timken Film Strength	Comments
1	Exp. Grease E.P.B.	39	145	8575 O.K.* 10000 Failed.	Good film strength, low melting point.
2	Exp. Grease PAM 70B	24	180	Est. a little lower than E.P.B.	Film strength satisfactory, but melting point a little high.
3	W.B. Grease No. 5005	16	162	5714 Failed.	Poor film strength. Some compensation through lower melting point than PAM 70B.
4	Stand. W.B. Grease	12	200	5714 O. K. 8575 Failed.	Melting point too high.
5	W.B. Grease No. 7009	9	145	5714 Failed.	Film strength low.
6	"Z" W.B. Grease	10	162	5714 Failed.	Film strength too low.
7	W.B. Grease No. 5006	3	100	.....	Lime base grease ran out of bearing.

* Oil in grease O.K. 11430, Failed 12860.

* There was evidence of oxidation and carbonization during some of the tests as indicated by discoloration of the greases of lighter colors. Evidences of oxidation have also been met with in field tests on Standardized Wheel Bearing Grease. (No. 4.)

rise in temperature, the importance of maintaining a low temperature as a safeguard against oxidation will be appreciated.

A high melting soda base grease usually has a tough fiber and a high alkalinity. The low melting point and reduced fiber of Experimental Grease E.P.B. is probably due to the reduction of the free alkali through the addition of Floyd's E.P. Base. Any excess of free alkali is removed by saponification of a portion of the Floyd's Base.

The question of resistance to oxidation suggests the possibility that Floyd's Base and asphaltic materials might be eliminated in order to obtain greases more resistant to oxidation. Best results in reducing the tendency to oxidation would be obtained through the exclusive use of eastern bright stocks in the grease. However, under severe service conditions the grease containing the Floyd's Base and asphalt has proved very effective in keeping operating temperatures at a minimum (due to the high film strengths imparted). These temperatures are probably more important factors in the oxidation of the grease than the presence of a small percentage of materials which are rather susceptible to oxidation.

The correlation between the ideas above and the results obtained may be seen in Table 23.

It is probable that the lubrication of wheel bearings is a more complex problem than one involving only film strengths, melting points and resistance to oxidation. However, a consideration of these points at least serves as a basis for speculation as to why some greases perform more satisfactorily than others.

Presence of Potassium Bichromate in Experimental Grease E.P.B.

It is understood that the advisability of adding potassium bichromate to a grease to prevent rusting was questioned by some lubricating engineers on the ground that potassium bichromate was a strong oxidizing agent and therefore not likely to prove stable in the presence of various materials in the grease. There undoubtedly is reason for additional work on the stability of potassium bichromate in grease. The analytical problem is complicated by the extremely small amount of bichromate present. It is possible that some valuable information on stability might be obtained through the analysis of greases containing higher percentages of potassium bichromate.

The only evidences at present with regard to the stability of potassium bichromate are indirect. That is, the stability may be inferred from the results of corrosion tests. In a test in which potassium bichromate was added in aqueous solution in the cold to Stand. Wheel Bearing Grease, a very remarkable reduction in corrosion has been obtained in a test which has lasted almost a year. This would seem to indicate that the bichromate was relatively stable in the cold in a grease for a period of at least a year, or that if slow deterioration of the bichromate were occurring the residual traces of bichromate were still very efficient in reducing corrosion.

In a corrosion test on steel strips on Experimental Grease E.P.B., to which 10 per cent of water had been added, the results seem to be a little

superior to those obtained with Stand. Wheel Bearing Grease (water added) to which no bichromate had been added. The corrosion results for Experimental Grease E.P.B. are not as remarkable at this time as the results which have been obtained on Stand. Wheel Bearing Grease with bichromate added. At present it is not possible to say whether or not some reduction in the efficiency of the bichromate has occurred due to the heat treatment in the manufacture of the grease. The corrosion test on Experimental Grease E.P.B. lasted only three months and the visible corrosion is localized in comparison with the general corrosion with Stand. Wheel Bearing Grease containing no bichromate. It is possible that as time goes on Experimental Grease E.P.B. will show itself to better advantage in comparison with the non-bichromate type greases.

It is advisable to carry out additional experiments with regard to the heat stability of potassium bichromate, the extent to which E.P. bases may be antagonistic to the elimination of corrosion through the use of bichromate and other points of interest in connection with the use of special types of materials to reduce corrosion.

The mechanism through which the bichromate accomplishes the reduction in corrosion is of theoretical interest. The bichromate added to the grease is converted to the chromate by the free alkali present in the grease. The following equation represents the chemical change which occurs:

$$K_2Cr_2O_7 + 2NaOH = K_2CrO_4 + Na_2CrO_4 + H_2O$$

The chromate formed is a mild oxidizing agent which yields its oxygen in accordance with the following equation:

$$2K_2CrO_4 + 2H_2O = Cr_2O_3 + 4KOH + 3O$$

The potassium (or sodium) chromate probably reacts with the surface of the steel to form a protective film of oxide which prevents further chemical changes at the surface of the metal. The phenomenon is probably similar to, or identical with, the chemical inactivity of a metal, brought about by certain oxidizing conditions, which is known as "passivity." A discussion of passivity may be found in *Transactions of the Faraday Society* (1923), U. R. Evans' "Influence of Obstructive Films on Anodic Processes."

A good general discussion of the factors involved in corrosion may be seen in *Industrial & Engineering Chemistry,* **17,** 339 (April, 1925), "A Study of Corrosion Factors and the Electrochemical Theory." This paper is of special interest in its interpretation of localization of corrosion as due to oxygen concentration cells.

Although potassium bichromate was used in the preparation of Experimental Grease E.P.B., sodium bichromate should be used in the commercial preparation of the product, since sodium bichromate costs only approximately 4½c per pound as compared with 8c per pound for potassium bichromate. There would be no objection to the use of sodium chromate if it could be obtained at a price comparable with sodium bichromate. The cost of the bichromate of course is of rather minor importance since only 0.1 per cent is used in the grease.

A number of limitations of tests under mild operating conditions are outlined below:

Operating temperatures may be too low to test the tendency of the grease to melt and run out of the bearing.

The bearings are loaded so lightly that no particular film strength is necessary for good lubrication along the cone rib.

Temperatures are so low that no severe test is made of the tendency of components of the grease to oxidize or volatilize.

No severe test is made of the capacity of the grease to maintain low operating temperatures through high oil availability.

The question as to whether severe operating conditions are ever met in service should be considered. If mild operating conditions only were met in service then film strength, melting point, resistance to oxidation and oil availability would all be of no consequence. The answer to this question is that wheel bearing failures occur under actual service conditions. These wheel bearing failures result in complaints with regard to the quality of the wheel bearing lubricant. Temperatures of operation under these conditions of operation are high as shown by bearings taken from service which are blue in appearance as the result of heat. In extreme cases the bearing may even be welded to the axle.

These bearing failures are probably due largely to improper adjustment of the bearings. Too tight an adjustment of the bearing brings about a very high thrust load with very high unit loads throughout the bearing. When the bearings are too loose vibration and pounding of the bearing may occur, resulting in failure of the bearing. In either case, local conditions of high unit loads are realized. If a reduction in the number of bearing failures through improper bearing adjustment can be realized through progress in lubrication, a real contribution to wheel bearing lubrication will have been made. If severe operating conditions can be met, the mild operating conditions will take care of themselves.

The attitude might be taken that oil companies should not be held responsible for bearing failures due to improper adjustment. The question might also be raised as to whether it were worthwhile to postpone bearing failure from improper adjustments for a few thousand miles, if the bearing failure cannot be postponed indefinitely or at least to a distance of say 20,000 miles. In this connection it should be noted that many companies operating trucks under heavy loads carry out a policy of inspecting and repacking wheel bearings after five or ten thousand miles of operation. If bearing failure can be postponed for this length of time readjustment of the bearing at the end of this period may restore normal mild operating conditions in the bearing, and a bearing failure will have been averted.

Addition of Water to Wheel Bearing Greases

Another test would be that of adding one-half per cent of water to a partially used grease which would then be subjected to the desired length test run, thus determining the action of the grease in the presence of moisture.

The question of water content is important in connection with the problem of corrosion. However, the length of time necessary to get significant results from corrosion is considerably greater than the ten or twelve day period which would be taken in a 20,000 mile test run in the laboratory. In order to carry out tests on corrosion under operating conditions, tests covering periods of the order of two months or longer should be considered. However, to expedite tests of such long duration in the laboratory, several wheel bearing machines would be desirable. As an alternative it is suggested that the question of the effect of water on wheel bearing lubrication might well be investigated as a part of the program of field tests which should be necessary before any product can be placed in the field.

Investigation of the Function of a Wheel Bearing Grease as a Medium for Excluding Foreign Matter

One of the functions of an antifriction bearing is to seal the bearing against the entrance of foreign matter, hence this phase should be given consideration.

The question of excluding dust and other materials from the bearing is of course important. However, it should be recognized that the exclusion of dust depends upon a number of factors other than the composition of the grease. The condition of the felt, which has as its purpose the retention of grease and the exclusion of foreign matter, is undoubtedly important. The details of packing the grease about the bearing may prove more important in cases where the felt is not in perfect condition, than the composition of the grease.

It is probable that if any improvement based on the composition of the grease could be made, it would be realized through the use of a heavier grease having a greater mechanical resistance to displacement and a higher melting point so as to be less susceptible to heat. However, the consistency of wheel bearing greases is more or less restricted to a worked penetration of 175 to 275 by public demand. Too heavy a grease may lead to the complaint that the grease is heavy and dry and does not lubricate properly. The question of melting point is especially important from the point of view of retention. If retention were poor the bearing would prove especially vulnerable to the entrance of foreign material. However, any grease to be given serious consideration as a wheel bearing lubricant must be retained well. Experiments already completed have covered the question of retention thoroughly.

The possibility of preparing an exceptionally high melting point grease, so as to improve retention and therefore offer more of a barrier to entrance of foreign material, does not seem promising since the high melting point greases tend toward poor lubrication.

Laboratory Study of Ball Bearings

Some authorities are of the opinion that roller and ball bearings subject to thrust loads should not be considered as presenting identical lubrication

problems, hence the necessity of an additional hub assembly containing ball bearings for use as wheel bearing grease testers.

The following points with regard to the ball bearing and roller bearing type wheel bearings should be noted:

The ball bearings are essentially a simpler problem in lubrication since the sliding friction along the cone rib of the roller bearing type is eliminated.

Both type of bearings present a problem in rolling friction, the ball bearings presenting a curved line of contact and the roller bearings a straight line contact. The ilnes of contact in the roller bearings, however, are considerably shorter.

The important point with regard to the comparison of the loads on the two types of bearings should be the question of tlie unit loads involved at the rolling contacts. The capacity to withstand high unit loads in either case will probably depend upon the film strength of the lubricant. Linear velocities are approximately the same for both types of bearings.

Assuming that unit loads are somewhat higher for ball bearings than for roller bearings, the logical point of attack in improving a product to withstand the more severe conditions in ball bearings would be to improve the film strength. This would mean that the film strength of the oil content of Experimental Grease E.P.B. would of necessity be increased from an O.K. 11,000 pounds per square inch Timken to, say O.K. 20,000 pounds per square inch, making the product definitely an extreme pressure product. Experience does not confirm the necessity of such a product though there can be little doubt but that it would prove satisfactory if corrosion could be avoided. Many companies having non-E.P. type wheel bearing greases have had no complaints on ball bearing type wheel bearings. It does not seem probable that complaints of failures will become more frequent using a product which is superior in film strength, and is therefore better adapted to withstand heavy loads.

The question of ball bearing lubrication should undoubtedly be given consideration. However, it is suggested that field tests should prove a sufficient confirmation of the suitability of the product.

Possibilities for Improvement of Wheel Bearing Greases

A number of the more promising possibilities for the improvement of wheel bearing greases are outlined below:

Improved film strength.
Improved stability of film strength.
Improved oiliness.
Improved oil "availability."
Improved resistance to oxidation.
Decreased corrosive qualities.
Improved retention.
Improved water resistance.
Improved appearance, both as regards new and used greases.

The first, second, third, and fifth points will depend a good deal upon progress in extreme pressure lubricants. For example, certain organic chloro bodies commercially available would improve both the film strength and resistance to oxidation. However, oiliness, the permanence of the pro-

tection given by the extreme pressure base and oil availability might be sacrificed. Improvement in extreme pressure bases may be slow. For that reason it may be wise to capitalize upon the progress already made in wheel bearing lubricants rather than to rely upon a slow and problematical improvement in extreme pressure bases.

Effect of Lubricants on Brinelling of Wheel Bearings

In the following sections, the very interesting experiments carried out by Almen [249] are reviewed, and in many instances quotations from his report are given.

In spite of the rough usage of high speed, overloads and infrequent lubrication, ball and roller bearings in automobiles and trucks give very little trouble in service. Automobiles will travel tens of thousands of miles without measurable wear of their anti-friction bearings, yet these same bearings are often seriously damaged while the automobile is stationary during shipment in freight cars. The bearing damage occurring in shipment consists of indentations in the bearing raceways opposite each of the balls or rollers on the loaded side of the bearings. These indentations have the appearance of having been produced by extremely high pressures and, therefore, the bearings are erroneously said to be "brinelled."

Bearing damage of the type described is by no means confined to automobile road wheel bearings in freight car shipments. It has been troublesome in many anti-friction bearings in service installations where the bearings are normally at rest and are used mainly to avoid static friction or where the bearings move through small angles. Among such installations are automobile spring shackles, king pin bearings and other steering gear parts. In aircraft engines, the valve rocker arm bearings have, until recently, been subject to this form of damage. The bearings in variable pitch propellers and control parts have, at times, been so badly damaged as to become inoperative and even the landing wheel bearings are damaged during flight. In fact, it is possible to ruin an assembled bearing by merely placing it on a vibrating plate. The following illustrations are typical examples of bearing indentations found in service.

Wheel bearing races taken from automobiles that have been shipped in freight cars show the indentations which are the result of small amplitude oscillations of the wheels during shipment due to vibrations of the freight car. For both front wheel ball bearings and front wheel roller bearings the actual indentations are small, the width of the marks being more a measure of the amplitude of the wheel oscillations than a measure of their depth.

The inner and outer races of an aircraft engine valve rocker bearing may also be seen to have a larger number of indentations than correspond to the number of balls in the bearing due to shifting of the ball cage relative to the races. These deep indentations are formed during the periods of rest between the valve opening and closing vents when the bearing carried

[249] Almen, J. O., "Effect of Lubricants on So-called Brinelling of Ball and Roller Bearings," 4th Annual Convention, National Assn. of Lubrication and Grease Manufacturers (Oct. 12, 1936).

practically no load but was severely vibrated by the engine. This trouble has been practically eliminated by substituting oil circulation for the grease lubrication previously used.

In one case a needle bearing automobile universal joint was badly indented while operating in a laboratory test fixture. This specimen was run for a period of 200 hours transmitting 45 horsepower at 1000 R.P.M. The shafts were in direct alignment and, therefore, the movements at the needle bearings were limited to errors in the set up and to deflections of the shafts and supports under load. Another test was made with the propeller shaft out of alignment 1.5 inches in which case the bearings were found to be only slightly indented. In a third test, with the shaft 2.5 inches out of alignment, three of the bearings were undamaged and the fourth very slightly marked. These results are in agreement with experience in service for propeller shafts having various degrees of alignment.

In an effort to determine the cause and to find a practical remedy for the so-called "brinelling" of ball and roller bearings, particularly in freight car and truck shipments of automobiles, the General Motors Research Laboratory conducted the investigations described herewith.

In all the cases of bearing indentation enumerated above, note that the bearings are at rest or have periods of rest and also that vibration is present. It is not necessary that the bearings be heavily loaded to produce indentations although, in general, the greater the load the greater the damage. However, it is possible under certain experimental conditions to show decreasing damage with increasing load. Characteristics of indented bearings is an accumulation of rust in the vicinity of the damaged area. In practice, this rust is not often observed because rotation of the bearing after a period of vibration quickly mixes the rust with the oil or grease with which the bearing is lubricated.

Since this so-called "brinelling" of ball and roller bearings may occur under conditions of load much too low to cause true brinelling, it is clearly not a case of pressure indentation but comes under the general classification of wear. Many experimenters have noticed wear of this type. it being mentioned by Eden, Rose, and Cunningham [250] as early as 1911. Various explanations of its cause have been suggested from time to time. Several of the theories and supporting data presented by earlier investigators are of interest as pertaining to the special case of wear with which we are here concerned.

Tomlinson,[251] experimenting with the friction of small balls on steel plates, produced oxidation similar to that which occurs in ball and roller bearings. He concluded, after considering such possbile effects as electrolysis, absorbed moisture and local heating, that the damage was caused by cohesive forces acting in a tangential direction lifting out particles of metal so small that they oxidize instantly. In support of this theory of molecular cohesion, he offers an experiment which may be very easily performed. Carefully clean a piece of plate glass and the surface of a fused

[250] *Proc. Inst. Mech. Eng.*, 875 (1911).
[251] *Proc. Roy. Soc. (London)*, **115**, (1927).

glass bead which is attached to a light rod. If the rod be lightly poised in the fingers and allowed to stroke the plate there is felt to be a series of snatches as the bead "welds" and breaks away from the plate. (Note particularly his use of the word "weld.") Examination of this plate by means of a lens will reveal scored dotted lines with even the lightest touch of the bead to the glass plate. Continuing this same experiment using hardened steel surfaces the sensation of snatching as the "weld" is made is much less marked. Again examination with a lens will reveal the path by the presence of a thin track of reddish *brown oxide*. According to Tomlinson, steel, glass, stellite and agate all produce the same effect on steel.

Dr. Max Fink,[252] experimenting with an Amsler wear testing machine, reached the conclusion that wear of the type under discussion is the result of the formation and rubbing off of successive oxide films. The Amsler machine used in these tests consisted of two circular discs geared together to rotate at the same angular velocity making contact on their peripheries. By making the discs of different diameters, relative slip of one per cent was obtained. The discs used by Fink were 10 mm. wide, 40 mm. diameter, running at 250 R.P.M. under a load of 110 pounds. In one case, after running in air for 50,000 revolutions, it was found that the loss in weight (one disc) was 0.1802 grams. When this test was repeated in an atmosphere of nitrogen, no wear occurred. He also noted that the rubbing friction was only one third as great when the test specimens were run in oxygen free atmosphere as when run in air. He observed that the test disc surface when run in an oxygen free atmosphere became smooth and bright in contrast to the specimens that were tested in air which were discolored with oxide films. As a result of these experiments, Fink concludes that oxidation is not a secondary effect, as concluded by Tomlinson, but is an essential component of wear. According to Fink, lubricants do not protect the rubbing surfaces against wear-oxidation, because the lubricants contain dissolved oxygen in sufficient amount to produce this form of wear.

During 1934, S. J. Rosenberg and L. Jordan,[253] of the National Bureau of Standards, attempted to duplicate the experiments of Fink using a similar machine and similar test conditions. They conducted tests in air and in atmospheres of nitrogen and hydrogen but, used relative slip between their test specimens of ten per cent instead of one per cent as used by Fink. The results obtained from these tests were not in agreement with the Fink tests. They found no appreciable difference in the amount of wear in air and in an oxygen free atmosphere of nitrogen or hydrogen on steel specimens having metallurgical characteristics similar to the Fink specimens.

In a discussion of Rosenberg and Jordan tests, Fink suggests that:

"The tests of Rosenberg and Jordan cannot be said to duplicate the results of Dr. Fink since the occurrence of oxidation in their tests is an indication that they did not have an oxygen free atmosphere in the test cell.

"The use of 10 per cent slip is too great for the softer materials, since in this case the component of the mechanical removal of metallic particles predominates. It

[252] Abstract—*Trans. Am. Soc. Steel Treating*, **18**, 1026.

[253] *Trans. Am. Soc. Metals*, **23**, 577.

may be mentioned that in the case of wheels running on rails, a maximum slip of about 5 per cent occurs.

"The field of friction oxidation is concerned with rolling friction with proportionately large surface pressures and small degrees of slip (50 to 150 kilograms pressure and 0 to 4 per cent slip). With sliding friction, the field of friction oxidation is concerned with 100 per cent slip and low surface pressures (approximately 0.5 kilograms per square centimeter) thus presupposing unhardened material.

"Oil does not protect against friction oxidation since oxygen is dissolved in the oil....."

It is probable that the conclusions reached by all the above investigators were justified within the limitations of their tests. The apparent contradictions of test results are no greater than have occurred in many other lines of research and can be explained by the differences in test conditions. A similar situation occurred a few years ago in regard to tests of E. P. lubricants. In this case, S. A. McKee, E. A. Harrington and T. R. McKee [254] of the Bureau of Standards, conducted comparative tests on four test machines and obtained widely different results for each of the machines. They, therefore, built a fifth machine from which a fifth set of results were obtained. The differences between the five machines were the relationship of such variables as rubbing speed, heat dissipation, specimen hardness, specimen finish and rate of load application. Since the lubricants under test were primarily intended to lubricate automobile rear axle gears, the important point was to determine the relative merit of lubricants in rear axles in which the relationship of the variables was not the same as in any of the laboratory test machines. The real problem was to determine which of the machines most nearly graded the lubricants in the order that these lubricants were graded by automobile rear axles in service.

These tests are mentioned merely to emphasize the difficulty of interpreting laboratory test data. The conditions prevailing in practice can rarely be reproduced or even approximated in the laboratory and it is, therefore, of utmost importance in industrial work to first determine what happens in service and then to vary the laboratory test procedure or equipment until results are obtained that are similar to service results.

When the results obtained by Tomlinson, Fink and the Bureau of Standards are compared with the so-called "brinelling" of ball and roller bearings in service, it appears that Fink's theory of wear oxidation meets all requirements. This does not mean, however, that wear of other machine parts in service can be explained by this theory. In fact, the wear destruction of gear teeth by scoring in service seems not to agree with the Fink theory but does agree with the weld theory of Tomlinson. Likewise, cases are known where an oxide film on rubbing parts is beneficial.

In preparation for the study of ball and roller bearing indentation, all available examples of this form of damage were examined. It was found that automobiles shipped to the Pacific coast suffered more damage than the shorter hauls and also that the damage was more severe in winter than in summer. The manner in which the automobiles were loaded in the freight cars had a large influence on the amount of damage that was done. Cars

[254] *S.A.E. Journal*, 32, 217.

loaded so as to carry the weight on the wheels were damaged more than cars that were blocked up.

With this background, a laboratory test machine was designed which was sufficiently versatile to permit wide variation in load, speed of operation, amplitude of vibration and temperature, with the hope that the machine could be made to produce damage of the type occurring in service. The test specimens used in this machine were commercial ball thrust bearings, the races of which are caused to rotate relative to one another through a small angle while loaded by a calibrated spring. A framework supports a central shaft on two ball bearings. A tubular structure is carried by the shaft on a second pair of ball bearings, the axis of which are slightly eccentric to the axis of rotation of the shaft. The test specimens, two commercial ball thrust bearings, are carried by the tubular structure being loaded by a pair of calibrated springs. The load may be carried by the threaded end of the tubular structure which bears against the calibrated disc springs. The tubular structure carries a slotted arm which engages the guide pin. As the shaft is rotated the tubular member assembly will be carried around by the eccentrically mounted bearings in the same manner as the big end of an engine connecting rod is carried around by the crankshaft. The pitch diameter of the ball groove is 1.672 inches, from which it follows that the movement of the balls on the races may be varied from 0 to 0.014 inches (0−2°). The spring load may be varied from 10 pounds per ball to 400 pounds per ball, corresponding to unit pressures, calculated by the Hertz equation, of 97,500 pounds per square inch to 332,800 pounds per square inch. The machine is driven by a variable speed DC motor.

In operation it was found that bearing indentations closely resembling indentations found in practice could be produced by this machine. The region of damage was found to be surrounded by rust just as occurs in practice. Indentations resulted at low loads as well as at high loads, requiring, however, a greater number of oscillations at low loads to produce an equal amount of damage.

The damage was independent of the speed of the operating motor but was roughly proportional to the total number of oscillations. It was also found that the damage was less at large amplitude than at intermediate amplitudes. The appearance of the damaged areas on the test specimen were somewhat rougher than the damaged areas of ball and roller bearings taken from practice, probably due to the fact that in practice these areas are usually smoothed by rotation of the bearing after the indentations are formed.

Among the preliminary tests made with the machine were two series of runs designed as a check on service observations on the effect of load and the effect of the amplitude of oscillation. Examination of the contact areas clearly show the effect of total loads on the three balls of 100, 200, 400 and 600 pounds each operated 25,000 cycles at oscillation amplitudes of the balls to the races of 0.006 inch. It was found that the severity of the markings increase with load.

The effect of varying the amplitudes of oscillation was also investigated

by Almen. Tests were run at a total load of 600 pounds for 100,000 cycles using grease "E" as a lubricant. The amplitudes, were 0.000 inch, 0.004 inch, 0.008 inch and 0.012 inch. It was seen that the damage to the race was greater at the intermediate amplitudes of 0.004 inch and 0.008 inch than 0.012 inch amplitude. This was in agreement with the automobile universal joint test and with service observations.

A series of tests was run on a large variety of oils and greases. These lubricants are listed in Table 24 in the order of their ability to reduce the severity of race damage. In all cases, the machine was run at a total load of 600 pounds, amplitude 0.006 inch, for 100,000 cycles. The only property of these lubricants that appeared to determine their rating was the viscosity at atmospheric pressure and at the operating temperature. Because of their superior performance in highly loaded gears, it was thought that E.P. lubricants would be more effective in reducing bearing damage than normal lubricants but all such lubricants also placed in the order of their viscosities. The same was true for saponifiable oils. Several oils were tested at freezing temperature and at 0° F. with the result that their rating fell in proportion to their change in viscosity. This would seem to account for the increased damage occurring to automobile wheel bearings in automobiles shipped during the winter months.

It was interesting to note that the worst of these lubricants, from the standpoint of test specimen damage, is the type of grease commonly used for automobile wheel bearings. The reason for this is that the viscosity must be high enough to prevent leakage.

A preloaded pinion bearing, filled at the factory with lubricant "A" gave considerable trouble by so-called "brinelling" during shipment to the West coast. This trouble was greatly reduced when lubricant "E" was substituted for lubricant "A."

These tests, and other observations, indicated that, wherever possible, low viscosity lubricants should be used in bearings subject to indentation. Following Fink's theory of oxidation wear, it appears that when heavy greases are used in such bearings the grease is pushed aside by the balls or rollers and air is admitted to the area of contact resulting in rapid oxidation. Lighter lubricants are held to the balls and races and provide a certain amount of protection of the contact area against air, hence reduced oxidation. At low temperatures, oils and greases that are sufficiently fluid to provide protection for the bearings at room temperature may become too viscous to adhere to the balls or rollers. A lubricant for this purpose should, therefore, be one that adheres strongly to the balls or rollers and the races under all conditions of service and under the highest and lowest temperature that will be encountered. It should not drain off during long periods of storage but should be present in sufficient quantity that the bearing may be run, as when the automobile is being loaded on the car, and still insure coverage for the contact region.

An attempt was made to meet these specifications, using an S.A.E. 10 engine oil with sufficient aluminum soap to give a viscosity approximately equal to S.A.E. 30 oil. It was thought that the rubbery consistency of this

lubricant would provide a cover and would resist draining. A service test was arranged in which twenty-five preloaded pinion bearings were filled and assembled in axles. The axle assemblies were set aside at room temperature in a position to drain for a period of two weeks after which they were assembled in cars and shipped to the Pacific coast during the late winter months. An inspection reported two bearings of the lot to be slightly

TABLE 24.—Corrosion Indentation Tests

The lubricants are listed in the order of their ability to reduce corrosion indentation based on microscopic examination of the test specimens. Test conditions: total load, 600 pounds; amplitude, 0.006 inch, 100,000 cycles.

Rating	Identification Symbol	Temp. ° F.	Lubricant
1	K	70	"IOW" engine oil (asphalt base).
2		70	DuPont GD-153 E.P. base (straight).
3		70	Oleic acid (straight).
4		70	Lard oil.
5		70	"20W" engine oil asphalt base plus 1½ per cent DuPont GD-162 E.P. base.
6		70	Castor oil.
7	G	70	"20W" engine oil (asphalt base).
9		70	Raw linseed oil.
9		70	No. 2½ cup grease plus 1 per cent oleic acid.
10		70	Experimental castor oil grease plus 5 per cent DuPont GD-153 base.
11		70	Steering gear grease (SSG 06).
12	F	70	Experimental lard oil grease (no mineral oil).
13		70	Experimental castor oil grease (no mineral oil).
14		70	Commercial wheel bearing grease plus 5 per cent pine oil.
15	E	70	Sodium soap grease plus 70 per cent commercial winter E.P. transmission lubricant.
16		70	Sodium soap grease plus 70 per cent commercial winter E.P. transmission lubricant plus oleic acid.
17		70	Commercial E.P. wheel bearing grease.
18		70	Experimental grease with 4.7 per cent zinc oxide.
19		70	Glycerin.
20		70	"Crisco" (shortening).
21	D	70	Commercial grease containing zinc oxide.
22		70	Black lubricating oil 90 vis. at 210° F.
23	C	70	Commercial sodium soap chassis lubricant.
24		0	"20W" engine oil (asphalt base).
25		30	Experimental lard oil grease (no mineral oil).
26		70	"20W" engine oil (Mid-continent).
27	B	70	Commercial slushing oil.
28		70	P and VM Naphtha (immersed).
29		70	Water (immersed).
30		32	No. 2½ cup grease plus 1 per cent oleic acid.
31		30	Sodium soap grease plus 70 per cent commercial winter E.P. transmission lubricant.
32		29	Sulfur saponifiable E.P. base.
33		0	Experimental lard oil grease (no mineral oil).
34		0	Sodium soap grease plus 70 per cent commercial winter E.P. transmission lubricant.
35		70	Sodium soap grease plus 30 per cent DuPont GD-162.
36		0	Experimental castor oil grease (no mineral oil).
37		70	Sodium soap grease plus 50 per cent sulfur base.
38	A	70	Sodium soap grease.
39		70	Sodium soap grease plus 45 per cent sulfur base plus 10 per cent lead soap.
40		70	Dry.

noisy. Although not entirely satisfactory, this experiment seems to justify the theory on which it was based and perhaps indicate the characteristics that a better lubricant should have.

As a further test of the oxidation wear theory, a number of laboratory tests were run in which protection against air was attempted by other means than oils or greases. It was found possible to compare the damaged areas after 100,000 cycles under 600 pounds total load at 0.006 inches

amplitude for: (1), dry; (2), lubricated with water; (3), submerged in mercury; (4), tinplated balls and races; (5), copper-plated balls and races; (6), tin-plate and mercury; and (7), copper-plate and mercury. In all cases of plated races, the plating was removed before observations were made.

Severe corrosion frequently is found on machine parts that are attached to one another in a manner to apparently prevent relative movement such as keyed taper shafts and hubs, press fitted wheels and axles, and the like. Where such corrosion occurs, it will be found that the attached members are subjected to fluctuating loads as from vibrations or from rotating loads.

In one of these an automobile front wheel spindle showed a damaged region where the pressed-on inner race of a roller bearing had rested. In a second a hardened automobile king pin corroded where it was attached to the axle. In such cases, the corrosion is probably produced in the same manner in which indentations are produced in ball and roller bearings. The slight movement that seems to be necessary for oxidation is present due to load variations and deformation of under stress. These cases are mentioned to call attention to the fact that oxidation wear is not peculiar to ball and roller bearings, but occurs on steel parts whenever high load and low slip are present.

Almen concluded that:

"The so-called 'brinelling' of ball and roller bearings is not brinelling. The indentations that are observed are the result of a form of wear dependent in the presence of oxygen.

"The indentation of ball and roller bearings cannot occur if air is excluded from the region of contact between the races and the balls or rollers.

"Oxidation wear can be greatly reduced but cannot be eliminated by the use of low viscosity lubricants.

"The use of low viscosity lubricants for many installations of ball and roller bearings is impractical due to inadequate oil seals.

"There is no general commercial solution now in use. The remedy that is applied varies according to circumstances."

Various Analyses

Ford Wheel Bearing Grease

The following analysis was made on a sample of grease taken from front wheel bearing of a new 1934 Ford Car:

Dropping point ° C.	133
Free alkali as NaOH, per cent by weight	0.07
Soap as sodium oleate (from ash), per cent by weight	19.5
Ash, per cent by weight	3.40
Ash Analysis, per cent by weight:	
Acid insoluble	0.4
Sodium carbonate (Na_2CO_3)	94.9
Sodium sulfate (Na_2SO_4)	1.4
Iron oxide (Fe_2O_3)	1.5
Viscosity of Oil:	
S.U.V. at 100° F.	269
S.U.V. at 210° F.	49.8
Gravity of oil ° A.P.I.	25.8

UN Wheel Bearing Grease Light (1934)

This grease had the following characteristics:

Physical Tests:

Appearance	Gray black, stringy, soft
Odor	Sharp, soapy
Dropping point ° C.	86
Penetration—Worked at 77° F.	365
Penetration—Unworked at 77° F.	355

Composition:

Soap, per cent by weight	11.7
Oil, per cent by weight	84.7
Water, per cent	0.40
Alkalinity (NaOH), per cent by weight	.02
Rubber hydrocarbons	None
Undetermined	3.18
Total	100.00

Ash:

Per cent by weight	1.27
Analysis, per cent by weight	
Acid insoluble	.02
Iron oxide	5.0
Calcium oxide	90.8
Calcium sulfate	4.0
Total	100.00

Mineral Oil:

Color	Red black
S.U.V. at 100° F.	332
S.U.V. at 210° F.	49.0
Gravity ° A.P.I.	21.1
Flash, ° F.	355
Fire, ° F.	405

Ford Wheel Bearing Grease M-544 (1934)

This grease gave the following results:

Physical Properties:

Color	Bluish brown
Odor	Soapy
Penetration at 77° F., unworked	161
Penetration at 77° F., worked	289
Dropping point ° C.	147

Composition, per cent by weight:

Oil	81.5
Soap	18.5
Water	0.6
Free fat	0.4
Free alkali	0.02
Insolubles	0.04
Gain in analysis	0.86

Ash Analysis, per cent by weight:

Sodium carbonate, Na_2CO_3	95.8
Sodium sulfate, Na_2CO_4	3.1
Iron oxide, Fe_2O_3	0.9
Acid insoluble	None
Calcium oxide, CaO	None
Undetermined	0.2
Total	100.0

Properties of Extracted Oil:

Gravity ° A.P.I.	25.6
Color, N.P.A.	4 dilute
S.U.V. at 100° F. (Micro)	267
S.U.V. at 130° F. (Micro)	126
Flash, ° F.	395
Fire, ° F.	465

Properties of Recovered Fatty Acids:

Iodine value (Hanus)	8
Dropping point ° C.	52
Acid value (mg KOH)	195

UN Ball and Roll Grease

The Pacific Coast grease manufacturer which marketed these greases ha considered that three different consistencies are needed in order to meet th requirements of all types of automotive equipment and seasonal tempera tures.

Physical Tests:	Light	Medium	Heavy
Color	Brown	Dark brown	Black
Odor	Alkali	Alkali	Alkali
Texture	Long fiber	Med. fiber	Short fiber
Dropping point ° C.	177	182	176
Penetration at 77° F., unworked	234	168	143
Penetration at 77° F., worked	230	195	190
Composition (per cent by weight)			
Soap	14.50	20.90	27.75
Mineral oil	84.75	77.88	71.26
Free alkali	0.02	0.04	0.02
Free fat	0.00	0.00	0.00
Water	0.10	0.10	0.10
Undetermined	.63	1.08	0.87
Total	100.00	100.00	100.00
Ash (per cent by weight)	2.56	3.73	4.49
Ash Analysis (per cent by weight):			
Sodium carbonate	99.60	99.60	99.80
Undetermined	0.40	0.40	0.20
Total	100.00	100.00	100.00

	Wheel Bearing Grease Designation							
	"Z"	PZ 350	MBG No. 5	RL	Stand.	SP 5006	SP 5005	SE Light
Physical Tests:								
Appearance	Br. Gr. stringy	Dk. Gr. granular stringy	Dirty brown soft stringy	Brown granular stringy	Brown green stringy	Gray brown smooth	Green black stringy	Light brown stringy
Odor	Soapy	Soapy	Soapy and like nitro-benzene	Sweet pungent	Soapy	Soapy	Soapy	Soapy
Dropping point ° C.								
Gillette	....	....	....	....	80	97	100+	100+
Ubbelohde	162	138	129	139	200+	100	162	133
Unworked penetration (77° F.)	198	155	240	189	204	159	164	199
Worked penetration (77° F.)	236	....	316	310	225	197	....	280
Separation test at 250° F.	None	None	None	None	None	None	None	None
Corrosion (3 hr. at 212° F.)	O.K.	O.K.	O.K.	O.K.	O.K.	O.K.	O.K.	O.K.
Composition (per cent by weight):								
Water	0.45	1.0	1.8	1.1	0.1	1.8	1.4	1.75
Oil	78.5	79.5	81.0	73.0	83.5	72.0	80.3	79.0
Soap	18.8	18.4	17.2	22.0	16.4	23.5	19.9	19.0
Free alkali (as NaOH)	0.18	0.13	....	0.11	0.12	0.05 as $Ca(OH)_2$	0.19	0.02
Free acid (as oleic acid)	....	....	.07	....	....	....	....	....
Ash:								
Per cent by weight	4.02	3.90	2.95	4.30	3.20	2.60	4.14	3.41
Analysis (per cent of ash)								
Na_2CO_3	89.6	95.7	96.9	94.1	93.3	....	89.8	97.2
Na_2SO_4	8.5	2.8	3.4 Ca_2SO_4	3.9 Na_2SO_4	3.4	....	7.0	2.0
R_2O_3	1.5	1.3			1.0	1.6	2.2	0.2
CaO	0.3				0.2	96.2	1.0	0.4
Mineral Oil:	at 130° F.						Furol	
S.U.V. at 100° F.	282	803	1867	1440	1254	355	348	330
S.U.V. at 210° F.	72.2	63.7	90.4	81.8	76.0	52.1	66.1	50.3
Gravity ° A.P.I.	25.4	20.9	19.6	20.2	21.1	22.1	22.2	23.9

	Light	Medium	Heavy
Recovered Fatty Acids:			
Neutralization number (mg. KOH)	203	200	199
Iodine value (Hanus)	55	49	50
Dropping point ° C.	42	44	44
Color	Dark brown and white crystals	Dark brown and white crystals	Dark brown and white crystals
Extracted Petroleum Oil:*			
S.U.V. at 130° F.	446	327	955
S.U.V. at 210° F.	92	78	160
Color	Cr. red	Cr. red	Black
Pour point, ° F.	80	80	85

Eight Commercial Wheel Bearing Greases

These greases are representative of those sold by different manufacturers throughout the United States in 1935.

Cal-Oil Ball and Roller Bearing Grease (1935)

This product has been marketed with considerable success as an automotive wheel bearing grease.

Physical Tests:	
Appearance	Color—Green-brown Structure—Fibrous
Odor	Caustic
Penetration at 77° F. (cone-unworked)	240
Dropping point ° C.	149
Composition (per cent by weight):	
Soap	14.50
Moisture	.35
Free fat	.60
Oil	81.90
Free alkali (per cent NaOH)	.26
Undetermined	2.39
	100.00
Mineral Oil:	
Color	Green-red
S.U.V. at 100° F.	476
S.U.V. at 210° F.	53
Gravity ° A.P.I. at 60° F.	19.5
Fatty Acid from Soap:	
Acid value (Mgs. KOH/gm. 0.1)	204
Iodine value (Hanus)	64
Melting point	35° C.
Color	Light brown
Ash (per cent by weight):	2.84
Ash analysis (per cent by weight)	
Acid insoluble	2.48
Iron as Fe_2O_3	2.98
Sodium carbonate	81.90
Sodium sulfate	11.20
Undetermined	1.44
	100.00

EASTERN WHEEL BEARING GREASES

A survey of a large number of wheel bearing greases marketed on the Atlantic Coast in 1934, indicated that nearly all were made with sodium soaps. The viscosity of the oil content varied greatly, from 185 to over 5000 S.U.V./100° F. These manufacturers considered worked penetration values of 170 to 300 were desirable for wheel bearing greases. Typical greases had the following specifications:

	1	2	3	4	5	6
Percent Sodium soap	18.5	15–20	12.7	16–22	15–16	22.47
Percent Water, max.	1	....	.75	....	trace	....
Percent Mineral oil	79.5	....	87.3	....	84–85	77–83
Grav. A.P.I.	23–24	....	22.3	....	25–26	26
Flash, ° F.	400	....	550	....	485–495	....
Fire, ° F.	450	....	630	....	560–570	....
S.U.V. at 100° F.	....	300	....	600/1800	4100/5000	185
S.U.V. at 210° F.	145–155	....	175	65–90	170–175	45
Color	Olive green	....	....	....	S.R. Green	3 A.S.T.M.
Pour point ° F.	20	....	65–90	....	....	25
Color (grease)	Olive green	....	Dark green	Green	....	Yellow
Worked (penetration)	170–180	....	300	195–280	....	....
Unworked penetration	....	....	250 min.	....	300–315	....
Percent Free alkali	.30	....	....	....	.25	....
Percent Free acid	.10	....	....	....	....	....
Dropping point ° F.	380–395	....	....	315–375	295–300	335
Percent Ash as sulfates	4.0	....	....	1.5–3.0	3.47	2.8
Corrosion test	Neg.	....	....	Neg.	....	....
Odor	Not perfumed	....	....	....	....	....

Standardized Wheel Bearing Grease

Formula

Oleic acid	73 gallons
Stearic acid	75 "
48° Bé. Caustic soda	29 "
Petrolatum	960 pounds
Prepared oil	550 gallons

The Prepared Oil may be blended according to either of the following formulae.

	I	II
1000 Pale oil	75	84
135 S.U.V. at 210° F. Calif. bright stock	25	..
150 S.U.V. at 210° F. Calif. bright stock	..	16

The Prepared Oil has the following characteristics:

S.U.V. at 210° F.	75 to 80
A.S.T.M. color (dilute)	2 to 3

The Petrolatum should have a melting point of 110 to 130° F., A.S.T.M. color of 3.5 to 4.5 dilute and an S.U. Viscosity of 100 to 115 at 210° F.

SAPONIFICATION.

Measure into a steam-jacketed kettle, 75 gallons of prepared oil, 73 gallons of oleic acid and 75 gallons of stearic acid. The draw off valve should be plugged with wheel bearing grease from a previous batch in order to prevent the caustic soda solution from draining into it and escaping reaction.

With the oleic acid and mineral oil at room temperature, and the stearic acid melted at a temperature of 150 to 180° F., the resultant temperature of the blend in the kettle should be 100 to 140° F., depending on atmospheric and initial kettle temperatures.

Measure out the 29 gallons of 48° Bé. caustic soda and charge it into the kettle while agitating paddles are operating.

After about ten minutes stirring turn on steam in the kettle jacket and maintain a pressure of 90 to 100 pounds per square inch. Make certain that the steam trap is operating and that steam is circulating through the kettle jacket.

As soon as the batch has started to boil (10 to 20 minutes after turning on steam) take a small sample and test it with phenolphthalein solution. If not alkaline, add at once 48° Bé. caustic soda solution in ½ gallon quantities until a definite pink is obtained.

After the batch has been heated and stirred for about three hours, it passes from a granular condition into a heavy plastic mass. At this stage more oil may be necessary in order to facilitate mixing as the grease base may become so heavy as to follow the paddles without maceration. The addition of about 25 gallons of oil is sufficient to avoid this difficulty.

Continuous heating and stirring should be carried out for five hours. During this period the draft fan should be in operation in order to free the kettle from steam as much as possible and facilitate the dehydration of the soap base. After the five-hour period of cooking has elapsed, the steam is turned off, and the exhaust valve also closed in order to prevent either live steam or exhaust steam from entering the jacket.

Stirring is continued for two hours, after which the batch is allowed to stand, preferably overnight. A sample of the base should be sent to the laboratory in order to determine its water content.

REDUCTION OF THE SOAP

After standing for at least ten hours the soap base should be cooled to about 250° F. With no steam on the kettle jacket, agitation is started and about 350 gallons of prepared oil is worked into the base. The temperature of the grease will drop to about 175° F. Excessive stirring should be avoided.

A sample of the grease should then be taken and cooled as rapidly as possible to 77° F. in order to check its consistency. (Unworked penetration A.S.T.M.) The dropping point of this sample is also determined, using the Ubbelohde method.

If the consistency and dropping point are satisfactory, the petrolatum is added.

A final check for consistency and dropping point is made and if found to meet specifications, the compounder approves the batch for withdrawal into shipping containers.

PROPERTIES

Wheel bearing grease is a fairly heavy short fiber plastic grease of brownish green color. It is intended for special service in automobile wheel bearings where high temperatures sometimes exist, and inferior greases may melt and come in contact with brake linings.

SPECIFICATIONS

Worked penetration at 77° F. (A.S.T.M. D217-27T)	175 to 225
Dropping point ° C. Ubbelohde method	Minimum 180
Free alkali (percent NaOH)	0.25

Must conform in texture and appearance with standard sample.

Chrysler Specifications

Chrysler Rear Wheel Bearing Grease Specifications (M.S. 1267-1936)

REAR WHEEL BEARING GREASE—
EXTREME PRESSURE TYPE—(PART NO. 657155)

This specification covers a grade of grease to be used for the lubrication of rear wheel bearings only, during the entire year.

This lubricant shall be a smooth homogeneous product, manufactured so as to produce a grease of the short fiber type.

This lubricant shall not contain any ingredients such as resin, resinous oils, abrasive fillers, or materials that are foreign to soap and mineral oil or that will corrode the bearings in storage or in service.

The lubricant shall be free from disagreeable odor or rancidity and shall not develop any objectionable odor or rancidity in storage or in normal use.

It shall not show any separation of the oil from the soap on standing or in normal use.

Composition:

This product shall be a uniform mixture of sodium soap, calcium soap, mineral oil, and a sulfur saponifiable extreme pressure base. This base shall be added to the composition in such a manner that it will not separate and form a "jelly," either in storage or in normal service, or as a result of any of the tests which may be outlined in this specification.

Sodium soap	10 to 13%
Calcium soap	3 to 4%
Sulfur—saponifiable base	10 to 12%
Moisture	0.3% maximum
Mineral oil	Balance

PHYSICAL PROPERTIES OF THE MINERAL OIL USED

Viscosity at 100° F. (Saybolt)	275 to 325 seconds
Pour point (A.S.T.M.)	plus 10° maximum
Flash point	350° F. minimum
Color	No. 6 maximum N.P.A.

Date changed: 12-26-35—Paragraph No. 6 revised. Page F-167.

The sulfur saponifiable base shall contain a minimum of 10% of added sulfur. The sulfur shall be incorporated in the saponifiable oil in such a manner that a polished copper strip will not be corroded when immersed in the finished grease for 3 hours at 250° F., but shall become corroded by the sulfur when immersed in the finished grease for 3 hours at 350° F.

Consistency:

The penetration of a worked sample, determined according to the A.S.T.M. method No. D-217, shall be 290 to 320 at 77° F.

Dropping Point:

The dropping point of the finished product shall be between 240 and 280° F. This test shall be conducted by the following tentative method:—

A copper wire mesh thimble is filled with the grease. The thimble is suspended inside of a large test tube by means of a copper wire attached to a cork in the top of the tube. Two thermometers are used, the bulb of one being on the outside of but adjoining the thimble, and the bulb of the other being immersed in the grease in the thimble with the bulb touching the bottom.

The tube is placed in a suitable bath and the temperature uniformly raised at the rate of 4 to 5° F. per minute. The average temperature of the two thermometers at which the first drop of lubricant leaves the thimble and drops to the bottom of the tube is the dropping point of the grease.

Top diameter of thimble	1.5 cm. (approx.)
Bottom diameter of thimble	.9 cm. "
Height of thimble	1.5 cm. "
Capacity of thimble	1.7 cc. "
Screen wire mesh	60

Note: These thimbles may be obtained from the Chrysler Engineering Laboratory.

Fiber:

The fiber structure of this grease shall be of the short type. Since there is no standardized or any satisfactory test to determine the length of fiber in grease the control of this property is to be made by examination and by comparison with retained standard samples which have been approved by the Engineering Department.

All tests shall be conducted according to the methods for testing lubricants adopted by the American Society for Testing Materials, unless otherwise specified.

Material covered by these specifications shall be purchased only from the sources which appear on the Engineering Approved Source List.

Chrysler Specifications for Wheel Bearing and Clutch Release Bearing Grease (M.S. 531-1936)

WHEEL, CLUTCH RELEASE, AND CLUTCH PILOT BEARINGS GREASE (PART NO. 307818)

This specification covers a grade of grease to be used for the lubrication of wheel bearings and the clutch release bearing during the entire year.

This lubricant shall be a well manufactured, homogeneous product, composed only of soaps and mineral oil, to produce a smooth grease of the short fiber type.

This lubricant shall not contain any ingredients such as resin, resinous oils, abrasive fillers, or materials that are foreign to soap and mineral oil or that will corrode the bearings in storage or in service.

The lubricant shall be free from disagreeable odor or rancidity, and shall not develop any objectionable odor or rancidity in storage or in normal use.

It shall not show any separation of the oil from the soap on standing or in normal use.

The specifications in Paragraph No. 7 are given as a recommendation when the lubricant is furnished in the clutch release bearings by the bearing vendor. In such cases, the vendor will be required to furnish samples to the Chrysler Engineering Department before purchase is made. When the lubricant is purchased directly by the Chrysler Corporation, it shall be purchased in accordance with these specifications.

Composition:

Sodium soap	11.0 to 13.0%
Calcium soap	3.0 to 4.0%
Moisture	maximum
Mineral oil	balance

The soap shall be made from a saponifiable oil that will not produce a rancid product.

Properties of the Mineral Oil Used:

Viscosity at 100° G. (Saybolt)	275 to 325 seconds
Pour point (A.S.T.M.)	plus 10° F. maximum
Flash point	350° F. minimum
Color	No. 6 maximum, N.P.A.

Consistency:

The penetrometer reading of both the unworked and worked samples of grease, determined according to the A.S.T.M. Method No. D-217 at 77° F., shall be agreed upon by each vendor and the Chrysler Engineering Laboratory. The permissible range shall not be greater than plus or minus 15 points from the established value.

Dropping Point:

The dropping point of the finished product shall be not less than 240° F. on an average of three samples tested. This test shall be conducted by the following tentative method:—

A copper wire mesh thimble is filled with the grease. The thimble is suspended inside of a large test tube by means of a copper wire attached to a

cork in the top of the tube. Two thermometers are used, the bulb of one being on the outside of but adjoining the thimble, and the bulb of the other being immersed in the grease in the thimble with the bulb touching the bottom.

The tube is placed in a suitable bath and the temperature uniformly raised at the rate of 4 to 5° F. per minute. The average temperature of the two thermometers at which the first drop of lubricant leaves the thimble and drops to the bottom of the tube is the dropping point of the grease.

Top diameter of thimble	1.5 cm.	(approx.)
Bottom diameter of thimble	.9 cm.	"
Height of thimble	1.5 cm.	"
Capacity of thimble	1.7 cc.	"
Screen wire mesh	60	

Fiber:

The fiber structure of this grease shall be of the short type. Since there is no standardized or any satisfactory test to determine the length of fiber in grease, the control of this property is to be made by examination and by comparison with retained standard samples which have been approved by the Engineering Department.

All tests shall be conducted according to the methods for testing lubricants adopted by the American Society for Testing Materials, unless otherwise specified.

Material covered by these specifications shall be purchased only from the sources which appear on the Engineering Approved Source List.

POTASSIUM SOAP GREASES

Lubricants containing potassium salts of fatty acids are not considered of any great importance in comparison with the relatively widespread use of sodium, calcium and aluminum soaps. In the nineteenth century many engine greases were made with potash soaps, as it was considered that they produced a smooth texture and an unctuous condition not possible with sodium soaps. The potassium soaps are often termed "soft soap" as they do not have the firm character of the sodium soaps. They are more soluble in water than the corresponding sodium soaps and are more soluble in mineral lubricating oils. It is not surprising that they do not give the yields of grease that are possible with sodium soaps. The addition of salt, frequently mentioned in earlier grease patents, converts potassium soaps to sodium soaps in accordance with the following equation:

$$\underset{\text{Potassium Stearate}}{C_{17}H_{35}COOK} + \underset{\text{Sodium Chloride}}{NaCl} = \underset{\text{Sodium Stearate}}{C_{17}H_{35}COONa} + \underset{\text{Potassium Chloride}}{KCl}$$

Greases made with potassium soaps, particularly those of unsaturated liquid fatty acids (recinoleic, oleic, linoleic acids), have better channel tests and are more fluid at low temperatures than similar greases made with sodium soaps. Aqueous solutions of potassium soaps solidify at much lower temperatures, and tend to form viscous liquids and gels to a greater extent than do corresponding sodium soaps. It is for this reason that they tend to produce a longer fiber texture than sodium soaps.

In order to produce high melting point greases from oleic acid as a soap stock, it is possible to fuse such unsaturated fatty acids with potassium

hydroxide, heating at temperatures in excess of 400° F., to produce palmitic acid and its salts which impart the higher melting point. Caustic soda may also be used for this purpose.

Potash Engine Greases

The following formulae are typical representatives of the engine greases which were most popular during the nineteenth century:

New York Engine Compound

A New York manufacturer marketed a series of greases having the following base:

	Per Cent by Weight
Tallow	15.0
KOH	2.2
Water	9.4
Tallow	73.4

The 15 per cent of tallow was first put in the kettle and heated to about 150° F., and the KOH solution added while stirring. Then, without delay, the balance (73.4 per cent) of tallow was mixed in. To make a No. 3 grease, 10 gallons of 100 S.U.V./100° F. mineral lubricating oil were mixed with 100 pounds of the foregoing soap base. A No. 2 grease was prepared with 15 gallons of this oil, and a No. 1 grease was obtained when using 25 gallons of mineral oil. For very heavy greases less than 10 gallons of oil was employed and beef tallow and beeswax were substituted for the mineral oil deleted.

10C Engine Grease Formulae

The data for seven grades are as follows:

	Parts by Weight						
Grade No.	1	2	3	0	X	XX	XXX
Yellow ceresin	10	10	12	7	15	20	25
Beef tallow	34	30	25	30	25	24	20
180 S.U.V./100° F. spindle oil	40	34	27	47	20	10	7
15 per cent solution of potassium hydroxide	18	16	16	16	16	16	16
Hard tallow	0	10	20	0	24	30	32

The ceresin wax and beef tallow are heated to 145° F. and the potash solution, also at this temperature, is added while stirring. The soap base is then heated to 208° F., while stirring, and cooled to 150° F., when the lubricating oil, hard tallow and oil of myrbane, if desired, is added.

No. O New York Grease

Ceresin wax	28 pounds
Tallow	135 "
25° Bé. paraffine oil	28 gallons
10° Bé. KOH solution	9 "

No. X New York Grease

Tallow	150 pounds
Crude paraffine scale wax	55 "
Paraffine wax	20 "
Ceresin wax	60 "
25° Bé. paraffine oil	8 gallons
10° Bé. caustic potash solution	7 "

No. XX Albany Grease

Tallow	140 pounds
Crude scale wax	70 "
Ceresin wax	40 "
25° Bé. paraffine oil	6 gallons
10° Bé. KOH solution	6 "

No. XXX Albany Grease

Tallow	150 pounds
Ceresin wax	55 "
Beeswax	50 "
135° F. M.P paraffine wax	110 "
FFF Pennsylvania bright stock	3 gallons
10° Bé. KOH solution	6 "

The process for combining the above ingredients was quite similar to that for the foregoing engine greases.

HA Engine Grease No. 1

	Per Cent by Weight
Tallow	50.0
Ceresin wax	5.0
100 S.U.V./100° F. Pennsylvania pale oil	25.0
8° Bé. caustic potash solution	20.0

HA Engine Grease No. 2

	Per Cent by Weight
Tallow	50.0
Ceresin wax	8.75
100 S.U.V./100° F. Pennsylvania pale oil	20.0
8° Bé. caustic potash solution	21.25

HA Engine Grease No. 3

	Per Cent by Weight
Tallow	44.00
Ceresin wax	12.20
100 S.U.V./100° F. Pennsylvania pale oil	16.35
8° Bé. caustic potash solution	17.45

Betts' Engine Cup Grease [255]

This was formulated as follows:

Lard	40 pounds
Caustic potash	1 pound
Water	3 gallons
Lard oil	5 "
Powdered asbestos	48 ounces

[255] U. S. Patent 161,469 (Mar. 30, 1875); see also: U. S. Patent 161,468.

The lard oil was first saponified with the potash solution after which the lard oil was mixed in. This oil had been previously ground together with the asbestos or paper pulp, or 24 ounces of each. The batch was cooked to eliminate substantially all of the water.

In 1863, Crosland [256] developed a grease which he claimed was suitable for propeller bearings and spinning machine bearings. It consisted of a mixture of potash soap, tallow, graphite and water. Johnson's [257] patent of this same year was much broader in its scope. His lubricant was made by mixing soap and water with an oil or fat, preferably a liquid oil of animal origin. These materials were boiled together at temperatures up to 212° F. The water was kept at a minimum, being only sufficient to carry the alkali used for saponification. Potassium soaps were preferred by the inventor, but he was aware that the oleates and stearates of other alkali material were also useful.

Mutton tallow, hydrated basic potassium oleate, butter fat, and small quantities of glycerol and sodium carbonate solution were the ingredients of Desmontils' [258] lubricating grease. The object of the glycerol was to prevent freezing at low temperatures. Gerdon and Schindler,[259] of Albany, New York, proposed an engine grease formula based on the use of tallow or fatty oil, wax, rosin, india rubber and caustic potash solution. Williams and Lamb [260] preferred to utilize sperm, rape, or other oils, mixed with alkalies, or with soft soap, or soap and water. A small amount of tallow was sometimes added to obtain the desired consistency. Live steam was employed for heating and promoting the blending of the materials. It was claimed by the inventors that the compound did not become dry or gummy in use, and that it was particularly suitable for the lubrication of steam and air cylinders.

Seeger's [261] grease was made by dissolving potassium soap in hot water and alcohol, and combining the solution with melted tallow, turpentine and cod-liver oil. After cooling the mass, rapeseed oil was added in any desired amount. Annab [262] prepared a lubricant for heavy bearings by mixing (about) one gallon of water with one half gallon of mineral oil and a quarter of a pound of soft soap. Stock,[263] of Watertown, New York, combined the following ingredients in the manufacture of a lubricating grease: beef tallow, paraffine lubricating oil, potassium hydroxide solution, pulverized talc, vermillion red, and oil of myrbane.

Graphite-Potassium Soap Lubricant

Traun [264] has proposed a method of dispersing graphite for purposes of lubrication, using a colloid mill the surfaces of which operated at not less

[256] British Patent 2,043 (Aug. 17, 1863).
[257] British Patent 2,677 (Oct. 29, 1863).
[258] British Patent 354 (1865).
[259] U. S. Patent 80,471 (1868).
[260] British Patent 2,976 (Nov. 12, 1870).
[261] British Patent 13,512 (Oct. 13, 1884); see also: U. S. Patent 339,483 (1886).
[262] British Patent 5,028 (Mar. 22, 1889).
[263] U. S. Patent 430,810 (Jan. 24, 1890).
[264] British Patent 155,836.

than 1000 meters per minute. To assist the dispersion of the graphite potassium oleate was found to be quite useful.

English Engine Grease

In 1920 Crawford [265] proposed the following formula, which is, of course, quite similar to many of the early American engine greases:

	Pounds
Beef tallow	300
Beeswax	20
15° Bé. potassium hydroxide solution	48
Water	50

The tallow and beeswax are melted together and strained. At a temperature of 150° F. the caustic potash solution is added and the mixture stirred intermittantly until saponification is substantially complete. A suitable oil soluble dye and one pound of oil of myrbane are added before the grease sets to a firm product.

English Potassium Soap Journal Grease

Hervieux and Bedard [266] developed a grease having the formula:

	Parts by Weight
Cod-fish oil	24
Beef tallow	16
Soft soap	2
Rosin	1
Cotton waste as required.	

Potassium Soap Cutting Compounds

In 1921 a New Jersey manufacturer marketed an emulsifiable cutting compound, in paste form, which had the following formula:

	Parts by Weight
Water	800
Caustic potash, 29° Bé.	270
Oleic acid	394
Paraffine oil, 28° Bé.	560

The potash solution is made by mixing 70 parts of caustic potash with 200 parts of water. The 800 parts of water, the oleic acid and the mineral oil are agitated in a mixer and the caustic potash solution run in.

In 1930, Lant,[267] of Vienna, proposed a composition consisting of hydrocarbon mineral oils, with or without neutral or neutralized animal or vegetable oils, intimately mixed with potassium oleate, and glycerin in the presence of not substantially more than 30 per cent by weight of water. The composition was of jelly form and was miscible with water to form a stable emulsion capable of being dried without altering the emulsifiable characteristics of the compound. Emulsions of this compound were recom-

[265] British Patent 170,705 (Aug. 12, 1920).
[266] British Patent 4,190 (Mar. 9, 1889).
[267] British Patent 372,261 (Oct. 27, 1930).

mended for cutting and boring operations and as textile lubricants. The proportions of glycerin and potassium oleate preferred were within the ratios 2:1 and 10:1, the maximum quantity of mineral oil permissible for stability was considered to be ten times and twenty-two times the respective quantities of potassium oleate. Homogenization was recommended before adding the paste to water. A typical formula was:

	Parts
98 per cent glycerin	4
Oleic acid	1
Potassium hydroxide	0.15
Water	.08
Spindle oil	10.0

These materials were mixed together in the order named at 70° C., and homogenized to obtain a clear jelly.

Potassium Soap Cooling Compounds

Braeman [268] developed the following formula for compound suitable for cooling hot journal boxes:

	Pounds
Potassium vegetable oil soap (containing 60 per cent of water)	100
Water	30
Neatsfoot oil	3
Graphite	3
Cocoanut oil	3
Sodium stearate	6¼

Potassium Soap-Latex Grease

Asprey [269] has suggested a lubricating composition consisting of mineral lubricating oil, in which rubber latex is stabilized by the addition of potassium soaps. The compound may be further thickened by the addition of paraffine wax or tallow. Asprey's [270] formula has been reported as being:

	Per Cent by Weight
Rubber latex (dry basis)	12.5
Potassium soap	2.6
Mineral lubricating oil	84.9

MIXED SODA-LIME BASE GREASES

There are several brands of grease on the market in which the thickening agent or base is a mixture of sodium and calcium soaps. These products have the smooth buttery texture of the usual lime base greases but are also characterized by the remarkable properties of stability imparted by the soda soap. The use of the soda soap also gives the product a much higher melting point than can be obtained in the usual cup greases. While these greases are usually employed for gear and transmission lubrication they may also be

[268] U. S. Patent 1,784,220 (Dec. 9, 1930).
[269] British Patent 449,779 (July 3, 1936).
[270] Australian Patent 16,889/34 (Apr. 4, 1935).

employed in pressure lubricating systems. When used for gear lubrication they thin down very little even at high operating temperatures. Continued use over long periods of time has little effect on the consistency. They have little tendency to form objectionable soap deposits, or discolor unduly in use. It is believed that these greases when used for journal lubrication give a very stable lubricating film which resists being squeezed from between the bearing surfaces to a greater extent than the usual lime base greases.

It is of interest that the asphalt base oils do not lend themselves readily to being made into this type of lubricant. While mixed base greases may be made by the usual methods for soda soap greases they retain the fibrous texture of the normal soda base grease. Texas oils are also difficult to use in these products. Mid-Continent and in particular Pennsylvania pale oils of 100 to 300 Saybolt viscosity at 100° F. are however most easily adapted. These greases may be made in any consistency from very soft greases of 400 or 500 penetration to heavy, high melting point block greases of 40 penetration or even less. The soap content may vary from a very small amount to as much as 35 or 40 per cent. The ratio of calcium to soda soap is usually about one to three.

The processes used for the manufacture of these greases are as follows:

Manufacture of soda soap and calcium soap in separate kettles, mixing the oil with the soaps separately, then bringing the two greases thus formed together in a regular grease mixer. The usual type of open grease mixer may be employed, having a steam jacket and capable of maintaining the kettle contents at a temperature of 300° F.

Manufacture of the soaps simultaneously or by double decomposition reactions in a fire-heated kettle and mixing in the oil at temperatures up to 400° F. The batch is then cooled to 200° to 220° F. when a small quantity of water may be added.

The formula for a typical grease of this type may be as given below. The oil used is a Pennsylvania base oil having a Saybolt viscosity of 400 at 100° F. and a gravity of twenty-seven has been found to be satisfactory in this product. While the fat indicated in the formula is inedible beef tallow other fats or mixtures of fats, fatty oils and fatty acids may be employed by making suitable modifications in the formula.

Medium Gear Grease

	Per Cent by Weight
Free fat	1.37
Combined fat	4.37
Free fatty acids	.33
Ash	.88
CaO	.11
Fe_2O_3 and Al_2O_3	.03
NaOH	.74
Water	3.30
Black oil	89.76

Light Metallic Gear Grease

	Per Cent by Weight
Mineral oil	91.58
Water	Trace
Free fat	3.18
Combined fat	4.66
Free fatty acids	.13
Combined CaO	.12
Combined NaOH	.33
Melting point	117° F.

Special Grease for Raymond Pulverizers

A special grease which has been found to give exceptionally good service for the lubrication of Raymond pulverizers is made on the following formula:

	Per Cent by Weight
Medium fiber grease	60.00
Cup grease, grade No. 2	40.00

Formula for Mixed Soda-Lime Base Grease No. 1
(Using fire-heated grease mixer)

	Per Cent by Weight
Inedible beef tallow	13.500
Hydrated lime	.525
Caustic soda (to be mixed with 1.5 per cent water)	1.500
Water	.200
400 pale oil	84.275

PROCEDURE.

The fat and about ten per cent of the oil are charged in the fire-heated kettle, the fat melted and mixed thoroughly with the oil.

A mixture of the alkalies and water as indicated above is then added to the kettle.

The fire is then started and saponification carried on until practically all of the water has been evaporated from the reaction mixture.

When a temperature of about 400° F. has been reached oil is run in slowly, the temperature being maintained at above 350° F. until all of the oil is in.

The batch is then stirred and cooled and the water added at 220° F.

The grease may be drawn at a temperature of about 200° F., when it will be in a plastic condition.

The manufacture of this type of grease in separate kettles is simply a combination of the open kettle methods for making lime and soda base greases, and will not be explained in detail. The procedure for manufacturing this type of grease for the lubrication of the dryer roll bearings found in paper mills has been discussed in the chapter on soda base greases. Such a grease contains about 30 per cent of soap.

Analyses of Commercial Soda-Lime Base Greases

The analyses of several mixed base greases have been made with the following results:

Lubricant

	Per Cent by Weight
Combined fat	23.81
Free fat	2.22
Free caustic soda	.08
Combined CaO	Trace
Free CaO	.41
Combined NaOH	3.77
Water	Trace
Mineral oil	69.71

Automobile Grease

	Per Cent by Weight
Free fat	1.26
Free Fatty acids	.02
Combined fat	14.88
Combined CaO	.34
Combined NaOH	.66
Water	.69
Mineral oil	82.10
Melting point	295° F.
Gravity of oil	26.9
Viscosity of oil at 100° F.	371

Soda-Lime Base Roll Greases

The use of brick grease or roll grease has found considerable favor for the lubrication of dryer roll bearings on paper machines and calendar roll bearings in laundries.

A study of the conditions in a large number of paper mills is summarized in Table 25.

TABLE 25.—Data on Dryer Roll Bearings in Nine Paper Mills.

Mill No.	Number of Dryer Roll Bearings	Size of Bearings (Inches)	Journal Surface Speed Feet per Minute	Max. Bearing Temp. ° F.	Max. Steam Pressure Pounds per Square Inch
1	68	9½ × 14	117	242	35
2	68	5 × 6	89	230	30
3	68	8 × 14	132	210	25
4	85	10 × 15	165	220	25
5	148	6 × 9	23.5	220	27
6	148	8 × 10	14.6	200	7.5
7	544	8 × 14	94	220	25
8	544	8 × 14	109	220	25
9	68	8 × 14	105	230	37

The bearing pressures for dryer rolls are not high, as the bearings are in most cases made in large sizes. The requirements of a satisfactory roll grease are:

It should not be excessively consumed under operating conditions. Some mills making very heavy paper require that one bar of grease weighing about two pounds should last for over six months without renewing. In other mills such as No. 4 above it has not been possible to obtain or develop a product which will last longer than one month.

The grease should feed sufficiently to give proper lburication. It should adhere well to the journal or it will be readily wiped off and consumption will be high.

It should not become dry, hard, carbonized or charred under operating conditions. The wearing surface of the bar should remain soft and oily under prolonged use.

The greases which best meet these requirements are soda-lime base greases in which light viscosity pale oils are used. It is believed that paraffine base oils are superior to asphalt base for use in these greases as they are not so apt to char.

Soda-Lime Base Roll Grease, Western Oil

Formula

	Pounds
Lime tallow soap (made separately)	12.5
Crude tallow, No. 1	22.4
Caustic soda, 40° Bé. solution	13.0
Western pale oil, 180 vis./100° F.	70.0

The composition of the grease is:

	Per Cent by Weight
Western pale oil	65.75
Crude tallow, No. 1	29.22
Caustic soda, solid	4.13
Calcium oxide (lump lime)	.89

PROCEDURE.

The tallow is charged in the fire-heated grease mixer together with 10 parts of the caustic soda solution. The temperature of the soap is then brought to about 270° F., when practically all of the moisture should be evaporated.

The lime soap is then added and the oil started into the batch while stirring.

When a temperature of about 250° F. is reached the remainder of the caustic soda solution is added very cautiously. The reason for this procedure is that by keeping the amount of caustic considerably under the theoretical amount required for saponification of the tallow at the start, the formation of a tight sponge is prevented, which it appears affects the texture and wearing qualities of the finished grease.

Heating is continued until the temperature reaches 400° F., which should take about one hour.

The grease is then heated as rapidly as possible to 500° F. and the heat turned off at once.

The grease is then drawn at this temperature into shallow pans to cool, after which it may be cut into bricks of appropriate size.

CHARACTERISTIC OF PRODUCT

The product made in this way has a melting point of 380° F.

Sodium-Calcium Ball Bearing Grease

Formula

	Per Cent by Weight
Oleic acid	8.0
Stearic acid	4.0
No. 5 Standardized cup grease (contains approximately 30 per cent Ca soap)	14.0
Sodium hydroxide (used as 45° Bé. solution)	1.76
S.A.E.-20 Pennsylvania motor oil	37.0
300 S.U.V. at 100° F. naphthenic pale oil	35.24

The fatty acids are first neutralized with the caustic soda, the cup grease then added and finally the lubricating oils, when the entire mass is heated to 450° F. The grease is then poured into pans to cool after which it is milled to smooth out a slightly grainy texture which forms. After milling, however, it has a smooth texture and is fairly transparent. The grease has the following characteristics:

A.S.T.M. worked penetration	275
Percent water	0.10
Alkalinity as percent NaOH	0.11
Ubbelohde dropping point ° C.	151
Viscosity of mineral oil content S.U.V. at 100° F.	325

Finlay's Mixed Sodium-Calcium Soap Base Universal Joint Grease [271]

The formula for this product is given below. The materials are charged in a fire heated grease kettle. The temperature is brought to 300° F., or higher, to saponify the fat (beef tallow), and melt together the heavy tar, calcium and sodium soaps formed, with the mineral oil. These make a homogeneous mass which is cooled to produce a dark brown, soft sticky grease having a melting point of about 400° F.

	Per Cent by Weight
Tallow	16.5
Caustic soda	2.5
Hydrated lime	2.5
2000 S.U.V. at 210° F. petroleum tar	5.0
150 to 200 S.U.V. at 210° F. cylinder stock	73.5

[271] U. S. Patent 1,699,961 (Jan. 22, 1929).

It is claimed that the calcium soap tends to shorten the fibers formed by the sodium soap, and also to soften the grease. The tar imparts adhesive properties and tackiness to the grease.

Mixed Sodium-Calcium Soap Base Grease Containing Poly-Isobutylene

Johnson [272] has proposed the addition of polymerized isobutylene of about 1000 molecular weight to the lubricating oil used in preparing a lubricating grease having as its base the sodium and calcium salts of oleic acid. Such polymers reduce the cold test of greases in which they are used and greatly increase the viscosity, adhesion and film strength of the oil content. Polymers of this kind may be added to aluminum, sodium, or calcium soap base grease to improve their adhesive properties. More recently U. S. Patents have been issued covering the use of poly-isobutylene having molecular weights of 20,000 to 40,000.

Miscellaneous Sodium-Calcium Greases

In 1877, Pappin [273] suggested treating greases and tallow with caustic soda, potassium hydroxide, and hydrated lime, and then adding lubricating oil, tar, rosin and chalk to produce a lubricant. This formula reveals the use of a combination of the sodium, potassium, and calcium salts of oleic, stearic and abietic acid, and possibly naphthenic acids, in a complex lubricating composition.

Lamon's [274] mixed base grease consisted of lard or tallow saponified with hydrated lime, caustic soda or other alkaline base and salt, together with lubricating oil, rosin, and paraffine wax.

[272] British Patent 399,527 (Mar. 4, 1932).
[273] U. S. Patent 188,479 (Jan. 27, 1877).
[274] U. S. Patent 282,648 (Aug. 7, 1883).

Chapter VIII

Aluminum Base Greases

ALUMINUM OLEATE COMPOUNDS

Aluminum soap is gaining favor in the manufacture of solid and semi-solid lubricating specialties. In general, it may be said that the procedure followed in the manufacture of aluminum soaps is that of double decomposition, in which a solution of water-soluble sodium or potassium soap is converted to aluminum soap by treatment with a solution of aluminum sulfate or alum.

Aluminum Oleate $[Al(C_{18}H_{33}O_2)_3]$, properly termed aluminum tri-oleate, is a yellowish, viscous mass, soluble in all proportions with mineral oil. Mixtures of aluminum mono-, di-, or tri-oleate with asphalt base mineral lubricating oils have been found to be very irregular in their stability, particularly in regard to viscosity. This soap is best prepared by the action of aluminum sulfate solution on a dilute solution of sodium oleate. It may also be prepared by the action of gelatinous aluminum hydroxide on oleic acid. Oils containing high percentages of aluminum oleate are not very satisfactory as greases, inasmuch as they are of a tough, livery texture.

Faragher [1] has assigned to the Gulf Refining Co. a patent claiming the use of aluminum di-oleate, $Al(C_{17}H_{33}COO)_2OH$, which is said to be more stable when dissolved in mineral oils than aluminum tri-oleate. He makes use of the reaction between aluminum hydroxide and oleic acid for the preparation of aluminum soap.

Aluminum soaps containing a small amount of mineral oil which is used to coagulate them during manufacture are termed "oil pulp," and minerals oils thickened by the addition of oil pulp are known as mineral castor oils. The mineral castor oils shown in Table 1 have been prepared, making use of 100 vis. at 100° F. asphalt base pale oil:

TABLE 1.—Properties of Mineral Castor Oil

Kind of Soap (aluminum oleate)	Per Cent of Soap	Furol Vis. at 100° F.	Furol Vis. After 7 Days	Furol Vis. After 14 Days	Sediment
Di-oleate	1.0	49	38	38	Fair
Mono-oleate	1.0	70	42	39	Bad
Tri-oleate	1.0	44	40	37	Very bad
Di-oleate	2.0	62	44	38	Fair
Mono-oleate	2.0	142	112	95	Very bad
Tri-oleate	2.0	59	42	38	Bad

After the soaps had been made, washed and dried, they were compounded with the mineral oil in the percentage indicated, at a temperature of 260° to 280° F.

[1] Faragher, W. F., U. S. Patent 1,550,608 (July 17, 1924).

Aluminum Oleate Pulp Stock

Formula	Parts by Weight
Pale oil, 100 vis. at 100° F.	10
Oleic acid	100
Water	200
Caustic soda, solid	14.2
Water	26.3
Aluminum sulfate	39
Water	250

PROCEDURE:

The oils and 200 parts of water are charged in a steam-heated kettle and taken to 180° F.

The caustic soda dissolved in the 26.3 parts of water is then added while agitating. The reaction which takes place here is the formation of sodium oleate:

$$\underset{\text{Oleic acid}}{C_{17}H_{33}COOH} + \underset{\text{Caustic Soda}}{NaOH} = \underset{\text{Sodium Oleate}}{C_{17}H_{33}COONa} + \underset{\text{Water}}{H_2O}$$

After mixing at this temperature for thirty minutes the aluminum sulfate solution is run in, and the sodium oleate is converted to aluminum tri-oleate in accordance with the following chemical equations:

$$\underset{\text{Sodium Oleate}}{6C_{17}H_{33}COONa} + \underset{\text{Aluminum Sulfate}}{Al_2(SO_4)_3 . 18H_2O} =$$

$$\underset{\text{Aluminum Tri-Oleate}}{2Al(C_{17}H_{33}COO)_3} + \underset{\text{Sodium Sulfate}}{3Na_2SO_4} + \underset{\text{Water}}{18H_2O}$$

A thick liver-like mass rises in the kettle and floats on the liquor; on heating to about 190° it coagulates.

After washing twice with 200 per cent of water the steam is turned on and the aluminum soap dried out, additional mineral oil being run in while the soaps are dehydrating. The proportion of mineral oil added depends on the viscosity of the desired mineral castor oil.

Aluminum Base Compounded Ford Oil

Formula	
Red oil, 300 vis. at 100° F.	1000 gallons
Aluminum stearate, Mallinckrodt's	90 pounds
Acidless tallow	17 gallons

PROCEDURE:

The acidless tallow and red oil are heated in a kettle to a temperature of about 215° to 250° F.

The aluminum stearate is then added under strong mechanical agitation by means of paddles, as blowers do not seem to give satisfactory mixture.

Agitation is continued while heating until complete solution is effected. After this is accomplished the stirring may be discontinued and the mixture allowed to cool slowly to room temperature.

Early Uses of Aluminum Oleate as a Base for Lubricants

Emil Dieterichs was one of the first to recognize the utility of oleates in lubricants and obtained patents on their use in 1874, 1875 and 1876. In 1882,[1a] he proposed combining fatty oils, fat, fatty matter or fatty acids

[1a] U. S. Patent 260,670 (July 4, 1882).

with aluminum salts. His process consisted of first saponifying the fatty material with any suitable alkali. The soap thus formed was dissolved in further quantities of water and a solution of alum, aluminum sulfate, or any soluble aluminum salt was added to precipitate the insoluble pulp. The resulting pasty mass was then placed on a filter and washed. Fish oil, neatsfoot oil, or any oxyhydrocarbon, or hydrocarbon oil that would combine uniformly without being objectionably discolored by the heat necessary to cause glutinous material to be precipitated, was used for dissolving the aluminum soap. One pound of alum was recommended for the conversion of one gallon of fat after saponification with alkali. Heat and agitation were employed for effecting solution of the soaps in the lubricating or fatty oils.

In 1885, William and Henry Marriott [2] proposed the use of aluminum oleate and stearate as a suitable means for increasing the viscosity of mineral or fatty lubricating oils. For normal lubricants they recommended the addition of about 5 per cent by weight of the aluminum soap. Rather than use tallow or lard oil for the source of their fatty material they preferred, on account of its cheapness, to use wool grease or brown grease from worsted or woolen washings, vegetable oil foots. The fatty acids were obtained from such fats by distillation and were then treated with about 11 parts of caustic soda for each 100 parts of fatty acids. 400 parts of water are used during the saponification with soda. For each 100 parts of fat 50 parts of alum dissolved in water was used to convert the sodium soaps to an aluminum pulp. After addition of the alum solution the batch is heated by injecting live steam. The liquor was then tested with ammonium hydroxide to indicate if an excess of alum had been added, and if not, more was added. The soap was then washed several times with hot water, until the test with ammonia indicated no appreciable quantities of aluminum salts in solution.

The aluminum soaps were dried by the application of superheated steam (300° F.) and then lubricating oil to the extent of four times the volume of soaps was added and mixed with soaps. This "viscom" base was then drawn off and stored. To prepare suitable lubricating oils it was blended with mineral or other oils at 212° F.

Aluminum Oleate-Chemical Theory

Aluminum tri-oleate is usually marketed as the technical hydrated product having the emperical formula: $Al(C_{18}H_{33}O_2)_3 + 6H_2O$. McBain and McClatchie [3] have reported that there is considerable doubt that predominating amounts of aluminum tri-oleate are formed when following the usual method of preparation consisting of treating an aqueous solution of sodium or potassium oleate with a water soluble aluminum salt. That predominating quantities of the tri-oleate are not formed under normal conditions, would be predicted by a consideration of the theory applicable to

[2] British Patent 11,726 (October 2, 1885).
[3] McBain and McClatchie, *J. Am. Chem. Soc.*, **54**, 3266 (1932).

the hydrolysis of aluminum salts. Aluminum is, of course, a very weak base and aluminum hydroxide is essentially insoluble; furthermore, there is a strong tendency towards hydrolysis for salts of weak fatty acids and weak bases. McBain added aluminum sulfate solution in excess to aqueous solutions of sodium and potassium oleate and found that the pH of the solution changed during precipitation from about 8.5 to 4.5. The oleates obtained after washing with water, alcohol and ether, were found to contain 5 to 6.2 per cent of Al from ash. This indicated an average composition more in line with the mono-oleate than the di-oleate or aluminum tri-oleate.

Henry and Streeter [4] considered that normal castor machine oils were made by incorporating aluminum tri-oleate with suitable mineral oils, and pointed out that the apparent viscosity of such products was subject to considerable change, being less after aging than when freshly made. These investigators described the preparation of castor machine oils of greater stability in which basic aluminum oleates were used in lieu of the tri-oleate. Aluminum forms three oleates as previously mentioned, and these may be represented by using the symbol R to indicate the oleic acid radical as follows:

I. Aluminum tri-oleate	AlR_3
II. Aluminum di-oleate	$Al(OH)R_2$
III. Aluminum mono-oleate	$Al(OH)_2R$

Henry pointed out that relatively stable castor machine oils were possible when prepared in such a way that the ratio between R and Al was 1.7 : 1 or 1.8:1. Faragher, Henry and Gruse [5] found that the drop in viscosity occurring with castor machine oils made with commercial aluminum oleate was dependent upon the fact that the ratio of oleic acid to aluminum was higher than indicated above; and that as high viscosities could be obtained with 3 per cent of their soaps as with 7 per cent of commercial aluminum oleates. They reported that for commercial oleates the ratio of oleic acid to Al was greater than 2:1 (di-oleate), and that sometimes the ratio was as high as 2.6:1. Preparations having a ratio less than 2:1 gave better results and the ratio can be dropped to as low as 1 : 1 (mono-oleate). They reported that for the mono-oleate the increase in viscosity for a given addition to lubricating oil was very much greater than with commercial oleates, and that for the proportions necessary to give the usual viscosity increases desired, no appreciable decrease occurred on aging the thickened oil. With the more concentrated solutions approaching the strength of those ordinarily made with tri-oleates, the viscosity instead of decreasing with age was found by the foregoing investigators to increase somewhat with time. By using soaps intermediate between the mono-oleate and di-oleate, the two tendencies, the tendency to decrease in viscosity characteristics of the di-oleates, and the tendency to increase in viscosity characteristic of the mono-oleates balance. The investigators were not able to determine definitely the point where the mixed oleates in strong solutions give thickened oils not decreasing in viscosity with age, but considered it to be somewhere just

[4] U. S. Patent 1,691,654 (Nov. 13, 1928).
[5] U. S. Patent 1,550,608.

below the 2:1 ratio required for the di-oleate. They reported that wher making aluminum oleate by the reaction between sodium oleate and aluminum sulfate a product with a low oleic acid content (lower than di-oleate) could not be made. This does not appear to be consistent with the views of McBain.

Faragher, et. al., considered that better preparations from the standpoint of stability could be made by substituting aluminum acetate or aluminum chloride for the aluminum sulfate of the standard method, but advanced no theory to account for their results. They found that still better preparations could be made by directly combining oleic acid with precipitated gelatinous aluminum hydroxide or hydrated alumina. Directly after precipitation alumina combines readily with oleic acid; but its activity decreases with time. The foregoing inventors recommended the method of precipitating a solution of aluminum sulfate of 10 per cent strength with sodium hydroxide or caustic soda, using the amount which will give a solution substantially neutral to phenolphthalein. Without troubling to wash the precipitate to get rid of mother liquor and salines (sodium sulfate), which would involve a delay and alteration of the properties of the alumina, they directly incorporated with it the calculated amount of oleic acid; no matter whether this ratio was the 1:1 ratio of mono-oleate or the somewhat higher oleic acid ratio of preparations intermediate between the mono-oleate and di-oleate. The oleic acid is added to the preparation with continuous stirring. Reaction takes place between the hydrated alumina and the oleic acid, as evidenced by a change in consistency of the mixture which becomes considerably more viscous. Toward the end of this change, the mixture is warmed somewhat to separate the oleate from the mother liquor in any suitable way. As the material agglomerates, more or less, the mother liquor can be conveniently removed by simple drainage. After removing the liquor, the soap is washed with water to remove nearly all sulfate which may be present. The wet, washed aluminum soap is then dried. A convenient and advantageous way is to suspend it in petroleum naphtha, or any other convenient oil, and subject the mixture to heat stirring, distilling off the water and naphtha together. Drying in the presence of volatile naphtha was found to give a better product. After the bulk of the water has been removed in this way, drying can be completed in vacuo or in a stream of inert gas, such as nitrogen or hydrocarbon gas. In general, it was found desirable to leave a little water in the oleate, say 0.3 to 0.4 per cent. After drying, the oleate may be broken up or pulped in any mineral oil to give a standard castor machine oil.

Henry and Streeter [5a] state their best results have been obtained with ratios between OH and R ranging between $OH_{1.55}$ $R_{1.45}$ and $OH_{1.45}$ $R_{1.55}$. Cottonseed and corn oil or other fats containing tri-glycerides are saponified by these investigators with sodium hydroxide; it is then dissolved in water and emulsified with mineral oil. They then separately prepare a basic aluminum salt solution by adding alkali to a solution of an aluminum salt, in the presence of a protective agent such as sodium citrate, in quantity

[5a] U. S. Patent 1,691,654.

sufficient to prevent the precipitation of aluminum hydroxide; the two solutions are then mixed in proportion such as to precipitate the water soluble soap in the form of a pulp of basic aluminum soap, which is then washed with hot water to free it of salts, dried and sufficient mineral oil added to produce a mineral castor oil of the desired viscosity.

Commercial oleic acid, or red oil, is less satisfactory because of its usual content of iron as an impurity. Rape oil may be used but is not as satisfactory as other oils previously mentioned. The basic solution of an aluminum salt is prepared by adding caustic soda solution to a solution of aluminum sulfate or chloride. The concentration of the aluminum salt solution may be 0.5 to 1 pound per gallon; the caustic soda solution may be 5° Bé. to 10° Bé. Basic aluminum sulfate solutions are suitable for preparing aluminum soaps of a composition approaching the di-oleate; while basic aluminum chloride is more suitable for preparing the lower ratio soaps; that is, those whose compositions approach $AlR_{1.5}(OH)_{1.5}$. In either case, according to Henry and Streeter, it is advantageous to add some protective agent such as citric acid or a citrate, tartaric acid or a tartrate, etc., to prevent precipitation of alumina. About 0.1 per cent of disodium citrate in the solution of the aluminum salt will suffice to prevent precipitation of alumina. The neutral sodium soap solution is emulsified with a quantity of lubricating oil to form a pulp, the emulsion is heated to boiling and the basic aluminum salt solution is added to it. To prepare a soap of the composition $AlR_{1.5}(OH)_{1.5}$, the amount of caustic soda added to aluminum chloride solution is sufficient to give the ratio $2AlCl_3$: 3NaOH, and the amount of this basic solution added to the soda soap solution is slightly in excess of enough to give the ratio $2AlCl_3$: 3NaOH : 3NaR. This leaves the liquor slightly acid to the usual indicators. The amount of lubricating oil added may be from one to two times the volume of the fatty oil originally used in making the soap. Within limits, the slower the addition of the aluminum salt solution the better. With a charge of ordinary size, it is recommended that the addition be spread over a period of about two hours. It is further recommended by the inventors that boiling and stirring should be continued for a period of about an hour after the addition of the aluminum salt. At this point the reaction of the solution in contact with the pulp should be slightly acid; pH 4.5 is advantageous. Besides the solution of aluminum soap in the oil, the batch at this stage contains a water solution of sodium sulfate which must be removed. This may be accomplished by washing the pulpy mass with boiling water for about thirty minutes, drawing off the water and digesting with a fresh charge of boiling water. This process is repeated until the wash water shows substantial freedom from sulfates. Three or four washings will usually suffice. The washed material is then dried. This may be accomplished by heating in a steam jacketed grease kettle while stirring. For products of high quality the drying operation must be carefully controlled.

Henry and Streeter found that complete dehydration gave poor yields of finished castor machine oil, whereas excessive quantities of water tended to cause separation and settling of the soap on storage. Satisfactory prod-

ucts must have a suitable degree of hydration intermediate between these extremes. The correct degree of hydration may be established by special methods for determining small amounts of water by chemical methods or, as is usually done, estimated by an experienced grease maker who is familiar with clarity and texture of the best products. Finally, the pulp is diluted by stirring in mineral lubricating oil until the viscosity of the desired commercial grade of castor machine oil is attained.

Henry and Streeter recommended the following method for preparing aluminum di-oleate. A fatty oil is saponified with the calculated amount of caustic soda by any of the ordinary soap making processes. A solution of this soap is emulsified with a body of lubricating oil, and to this emulsion is added further amounts of caustic soda. The amount of mineral oil used in pulping the mixture may be one to two times the volume of the fatty oil originally used. The amount of added alkali is about enough to give a ratio of 2NaR : NaOH. The alkaline mixture is heated to boiling and aluminum sulfate (or chloride) solution is added slowly. The amount of alum added is enough to give the ratio of $Al_2(SO_4)_3$: 4NaR : 2NaOH. The concentration of the alum solution may be 0.5 to 1.0 pound per gallon. The addition of alum solution should be spread over about two hours, and the boiling and stirring should be continued for about an hour thereafter. After the addition, the reaction of the solution in contact with the pulp should be slightly acid; pH 4.5 is advantageous. The pulp is washed, dried and diluted with mineral oil as described above. By this method, these investigators have found it possible to prepare aluminum di-oleate.

Henry [6] has assigned to the Gulf Refining Company a patent covering the method of making castor machine oils in which basic aluminum soaps and less basic aluminum soaps are incorporated with mineral lubricating oil to produce a desired fatty acid to aluminum ratio. This process comprises the emulsification of lubricating oil by an aqueous solution of neutral sodium soap; precipitating aluminum tri-oleate by the addition of a solution of an aluminum salt; then, adding sufficient caustic soda solution to correspond to two-thirds of the fatty acid in the tri-acid aluminum soap; thereby forming a solution of alkali soap. A solution of an aluminum salt is added to reproduce aluminum soap at the expense of said alkali soap, and in this way forming a mixture of soaps of different basicities, washing the mixture, drying the mixture by heating until it is nearly clear in appearance, and adding further lubricating oil to produce the viscosity desired.

Castor Machine Oils

Heavy Castor Machine Oil (Red)

	I	II
No. 1 Beef tallow	.....	37½ gallons
No. 1 Lard oil	.....	37½ "
Soya bean oil	100 gallons	.....
30° Bé. caustic soda solution	35 "	35 "
100 S.U.V. at 100° F. red mineral oil	1300 "	1300 "
Potassium alum	300 pounds	300 pounds

[6] U. S. Patent 1,691,882 (Nov. 13, 1928).

In formula I it will be noted that the caustic soda is insufficient to completely saponify the soya bean oil, and that some free fatty oil will no doubt remain in the finished pulp.

The fats and caustic soda are screened and run into a steam jacketed grease kettle, together with sufficient water, about 40 gallons, to dilute the caustic soda solution to about 20° Bé. This mixture is boiled and stirred until the fatty oils are as completely saponified as possible in three hours time. The aqueous solution of the soap should be fairly clear. In the meantime the alum used for precipitating the aluminum soap is dissolved in as little water as possible, about 100 gallons. This may be done by utilizing a 250-gallon wooden tank and injecting live steam to promote the solution.

The sodium soap solution is diluted with 200 gallons of water to facilitate precipitation, and the hot alum solution is run through cheese cloth into the kettle contents. The aqueous layer formed is solution of sodium and potassium sulfate, and the aluminum soaps rise to the top as a tough, soft greyish brown mass. The aqueous layer is drained off through the kettle draw-off valve, and the aluminum soaps are washed repeatedly with cold water, until no trace of sulfates remain. After the washing is completed, the aluminum soaps are heated, while continuously stirring, until nearly all of the water is evaporated and clear brown soap is obtained. Thirteen hundred gallons of the 100 Red Oil are then added very slowly under continuous agitation, to complete the batch.

The finished oil should have a color of No. 5 A.S.T.M. Various grades of castor machine oils made on these formulae should have viscosities as indicated below:

Grade	Saybolt Furol Viscosity at 122° F.
Extra heavy	900–1100
Heavy	600– 700
Medium	400– 500
Light	175 max.

Cottonseed Oil Fatty Acid Castor Machine Oils

Formula

	Parts by Weight
Cottonseed oil fatty acids	500
Sodium hydroxide	72.5
Water	5000
Potassium alum	204
Wash water	2000

The results of mixing the above pulp with 60 Pale Oil are indicated in the following table:

Per Cent Aluminum Cottonseed Oil Soap	Per Cent 60 Pale Oil	Furol Viscosity at 122° F. Fresh	After 14 Days
9.9	90.1	...	...
8.1	91.9	1085	240
7.4	92.6	682	177
6.3	93.7	475	101
5.7	94.3	180	44

Aluminum Ricinoleate Motor Oil

Rosenbaum has developed a motor oil of improved temperature-viscosity characteristics in which the aluminum soap of oil soluble castor oil is dissolved in a suitable mineral oil. About 90 per cent by volume of castor oil is first mixed with 10 per cent of 100 S.U.V. at 100° F. mineral oil and heated for 6 to 8 hours in a vacuum still to 650° F., to remove albuminous material and aldehydes which render the castor oil insoluble in mineral oil. Rosenbaum then saponified this oil soluble castor oil with caustic soda and diluted it with water to make a 20 per cent solution. This solution was then treated with aluminum sulfate solution and the insoluble aluminum pulp produced. The soap is then washed to free it of sulfates, dried and dissolved in lubricating oil to produce the final product.

Aluminum Oleate as a Means of Suspending Fillers in Oils

Many investigators have found that aluminum oleate, or other soaps, is quite useful in increasing the viscosity of lubricating oils when it is desired to prevent the settling, or separation, of fillers or solid lubricants such as sulfur, lead sulfide, graphite, clay and earths, colloidal lead, copper, zinc and aluminum or finely divided metals of super colloidal size. A particle of any of these substances in suspension in mineral oil is acted upon by the force of gravity, "G," the buoyant forces due to the fields of force of the oil molecules, and also the resistance offered by the viscosity of the oil. Since the viscosity of the oil may be greatly increased by the addition of suitable quantities of aluminum oleate, or other soaps, settling of finely divided solids may be greatly retarded by this means. The force exerted by the viscosity of the oil may be indicated by Stokes' law:

$$F = 6\pi nav$$

In this expression:

v is the velocity of the settling particle.
a is the radius of the falling particle.
n is the viscosity of the soap oil mixture.

Since G (the net gravitational force acting on a particle) is a constant, F varies with the velocity of the particle a constant limiting velocity will be reached when $F = G$; this limiting velocity v_o, is given by the following equation:

$$V_o = \frac{2}{9}\frac{(d_1 - d_2)\, g}{n} \cdot a^2$$

in which: d_1 is the density of the particle,

and: d_2 is the density of the lubricating grease.

Roy Cross Graphite-Bentonite Cutting Oil

Cross [7] has pointed out that the addition of metallic soaps, such as aluminum oleate, gives a gelatinuous condition to a lubricating oil, at

[7] U. S. Patent 1,739,631 (Dec. 17, 1929).

high temperatures, so that graphite, mica, or other solid lubricants do not settle out. At the same time the low cold test of about —20° F. for the oil he preferred to use prevented the oil from becoming stiff or solid at lower temperatures. Thus, when such a grease is used in a transmission no objectionable resistance is encountered in shifting gears or in transmitting power. For certain lubricating purposes, such as the cutting of threads, it was considered desirable by Cross to utilize water on account of its high specific heat. In such cases, an excellent emulsified jelly was produced by the use of low cold test, high viscosity lubricating, metallic soap, a special hydrous silicate of alumina (known as Bentonite) and distilled water. Graphite was, or was not, added. A typical mixture recommended by Cross was:

	Per Cent by Weight
Lubricating oil ..... S.U.V. 1835 at 70° F. Pour point — 20° F.	50
Aluminum oleate	3
Flake graphite (or mica)	3
Bentonite	4
Distilled water	40

This product was made by first heating the lubricating oil to about 400° F., then slowly adding the aluminum oleate, while heating and stirring, and continuing the heating until the oleate was completely dissolved, as indicated by the absence of sediment and the clarity of the solution. The graphite was then added to the batch, and stirring continued until the mixture was cooled. The Bentonite was then mixed with the water in the proportion of one part to ten parts. For this purpose a good grade of Bentonite, or one capable of gelling readily, was used. The one obtained from the deposit near Ardmore, South Dakota, is preferred. The Bentonite should have, according to Cross, an ability to absorb water and form a gel with water in the proportion of one part of Bentonite to twenty parts of water. The Bentonite gel was then mixed with the solution of aluminum oleate in lubricating oil. When stirred together a viscous grease was obtained suitable as a thread cutting lubricant and coolant. For gear lubrication, the same proportion of lubricating oil, aluminum oleate and graphite was recommended, while the proportion of Bentonite relative to the water content was greatly increased. Any high viscosity oil of low cold test was considered satisfactory by Cross.

Burke's Graphite Lubricant

In this development [8] graphite is permanently suspended in oil by means of a stabilizing agent such as aluminum soap. Burke found that 0.1 to 1 per cent by weight of aluminum oleate was quite effective in suspending 1 per cent of graphite in mineral lubricating oil. The amount of stabilizing agent used, according to Burke, in stabilizing the graphite suspension depends upon many factors. For example, upon the chemical composition

[8] U. S. Patent 1,732,221 (Oct. 22, 1929).

of the oil; that is, whether it is an oil of naphthenic or paraffinic base, and also upon the physical properties of the oil, its boiling range and particularly its viscosity. Also, the amount of stabilizer required depends upon the physical properties of the graphite and the amount of impurities and ash in the graphite, and the state of division.

The size of the graphite particles, and the viscosity of the soap oil mixture have, of course, been discussed in connection with the application of Stokes' law to this problem. The amount of stabilizing agent which must be used in order to make a permanent suspension of graphite in oil depends upon the percentage of graphite (by weight) which is to be worked with. Thus, the amount of stabilizing soap which may be used, will vary from 0.1 per cent to 1 per cent by weight, when the amount of suspended graphite is 0.1 per cent to 5 per cent of the total compound. Although aluminum soaps, and particularly aluminum oleate and stearate, have been found by Burke to be particularly effective as stabilizing agents, he found that other agents were effective in making stabilized suspensions of graphite in oil. Polyvalent soaps of suitable metals were considered as being satisfactory, particularly iron stearate, iron oleate, and the oleates and stearates of copper, chromium, nickel, cobalt, calcium, and magnesium. Very commonly, the polyvalent metal soaps are composed of mixtures of salts of the fatty acids (such as stearic, oleic, and palmitic acids) and these soaps were found by Burke to be very effective stabilizing agents for providing permanent suspensions of graphite in lubricating oils. He found the soaps which are more soluble in, or colloidally soluble in, and easily wetted by lubricating oils in which the graphite was to be suspended, were more effective in stabilizing the suspension. Burke's method consisted of grinding the soap and graphite into a plastic mass. To this plastic mass, he then slowly added a lubricating oil preferably heated to a temperature not exceeding about 100° C. The mixture was thoroughly stirred in order to dissolve the soap in the oil. After the soap was completely dissolved, the mass was diluted with more lubricating oil at ordinary temperatures to such an amount that the desired percentage of graphite remains in the oil.

Burke preferred the mixture of graphite and soap to be heated to about 100° C., while the grinding was carried on, in order to more thoroughly incorporate the graphite in the soap. At the higher temperatures the soaps, particularly iron and aluminum soaps, are in a fluid state and the mixing operation can be readily effected.

Asbestos-Aluminum Oleate Grease

Almost any grade of grease may be made by making appropriate mixtures of mineral oils, aluminum oleate and pulverized asbestos. Such products may have extreme pressure properties, but tend to be abrasive due to the lapping action of the solid particles of asbestos. The oleate, of course, greatly stabilizes the mixtures and prevents settling of the finely divided asbestos, particularly in the lighter grades of such lubricants. Hodson [9]

[9] U. S. Patent 1,764,837 (June 17, 1930).

has patented such a lubricant in the form of an elastic gel. It consists of mineral lubricating oil together with a gelling metallic soap (such as aluminum oleate) and finely powdered asbestos.

Aluminum Lanolate Greases

This soap is particularly useful as a grease base in view of its adhesive characteristics and ability to impart stability to greases made with other soaps, such as the sodium and calcium, as well as the aluminum salts, of oleic and stearic acids. A simple aluminum lanolate base grease was suggested by Krause.[10] It was prepared by saponifying wool grease, and by mixing an aqueous solution of the soap with a solution of alum or any salt of aluminum. The brown precipitate was termed aluminum lanolate. The finished greases were prepared by dissolving the dried lanolate in mineral oils to obtain the desired consistency.

Aluminum Oleate-Calcium Soap Motor Oil

Willock, Caplan, and Babb[11] have developed a motor oil of improved temperature-viscosity characteristics and, while it contains aluminum oleate, is free from the long stringy texture commonly associated with castor machine oils. Their lubricants consist of petroleum oils compounded with: a metallic soap such as aluminum oleate; a fatty acid such as red oil, or a fat such as corn oil; and a small percentage of another soap, in addition to the metallic soap. This additional soap, which is employed in very small quantities, may be a soap derived from corn oil, or any other vegetable oil or fat; or from lard oil, or any other animal oil or fat; or any fatty substance or material capable of forming soap with any of the alkalies; or alkaline earths, such as soda or calcium oxide; or with any of the metals, their oxides or salts such as lead, or zinc; or with any of the organic bases such as aniline.

These investigators found as being satisfactory a lime soap made from about 69 per cent of corn oil, 10 per cent caustic soda and 21 per cent of calcium chloride (purity 53 per cent), the resulting yield being about 70 per cent by weight of the total ingredients. Before this product was taken from the kettle, it was mixed with about one and one half times its weight of petroleum oil, so that it was more easily handled in subsequent use and contained 40 per cent of soap. Aluminum oleate, prepared by conventional methods, was mixed with about one and one-half times its weight of petroleum oil for the same reason. The following formulae are typical:

	Medium Motor Oil Per Cent	Tractor Oil Per Cent	Mine Car Lubricant Per Cent
200 S.U.V. at 100° F. Pennsylvania lubricating oil	90.80	85.75	71.00
40 per cent Aluminum oleate	8.00	13.00	27.00
40 per cent Calcium soap	.20	.25	1.00
Oleic acid	1.00	1.00	1.00

[10] British Patent 5,226 (Apr. 3, 1890).
[11] U. S. Patent 1,625,969 (Apr. 26, 1927).

Miscellaneous Use of Aluminum Oleate

In 1887, Hartung and Gallagher [12] proposed that mineral oils and greases be mixed with an aluminum soap which could be an oleate, stearate or palmitate obtained from the waste oleic acid from the manufacture of candles. Aluminum soaps have been found to be effective pour point reducers as they appear to prevent crystalline formation of wax in oils. Davis [13] has suggested the use of aluminum or lead oleate, rosin, gasoline gum, and gum damar and copal as suitable means of reducing the pour point of waxy lubricating oils. Lederer [14] has patented a lubricating grease composed of steam refined cylinder stock (150 S.U.V. at 210° F.) and 10 per cent by weight of aluminum oleate and stearate, the compound being solid at 212° F.

ALUMINUM STEARATE LUBRICANTS

Gardner [15] has noted that aluminum stearates available on the market may vary greatly with regard to their ability to form viscous gels with mineral oils. Free stearic acid was visible under the microscope when some commercial samples were examined. The finely divided stearates were, in general, better for increasing the viscosity of oils than those which were coarse or contained large agglomerates.

Gardner's Method for Examining Aluminum Stearate

Gardner has recommended the following methods for examining aluminum stearate:

Chemical Examination.

Moisture.—Different lots of stearate vary in the amount of moisture which they contain.

It may be determined in the usual manner—that is, by heating about 1 gram of the sample in an oven for 3 hours at about 105° C. A method used by Johnson gives somewhat more concordant results, and is probably more accurate in the case of low melting point stearates. The latter method is carried out as follows:

Carefully desiccate a quantity of oleic acid by drying in a moisture oven for several hours. Weigh into a small portion of the desiccated acid about one gram of the sample. Heat at 105° C. for 3 hours and determine the loss in weight. Several experiments were made to determine the effect on the gelling property of water soluble compounds that may be present. Small quantities of water, sodium sulfate solution, and sodium hydroxide solution were added to separate 10-gram portions of No. 1 stearate. To each, 99 cc. paraffin oil was added and the mixtures gelled as described in the jelly test. The samples to which pure water and sodium sulfate solution were added yielded gels apparently as firm as when nothing was added. The sample to which sodium hydroxide (a fraction of one per cent) was added, remained very thin throughout the entire heating, and when cold was very soft. The appearance was that of a highly refined Petrolatum Jelly (vaseline), for which it might serve in some arts. *Apparently caustic alkalies have a very marked effect upon the jelling of Aluminum Stearate.*

[12] British Patent 15,054 (Nov. 4, 1887).

[13] British Patent 401,341 (May 7, 1932).

[14] U. S. Patent 1,936,632 (Nov. 28, 1933).

[15] Gardner, Henry A., "Physical and Chemical Examination of Paints, Varnishes, Lacquers, and Colors," 884-896.

Results of Examination of Samples of Aluminum Stearate *

No.	Percentage—Moisture	Ignition Residue	Water Soluble	Al_2O_3, and Fe_2O_3	Appearance of Solution of 2 Grams of Stearic in 50 Gms. of Xylol	Viscosity of Xylol Solutions. Gardner-Holdt Standards
1	.52	7.85	.35	7.5	Colorless. Contains small amount of suspended material.	J
2	.54	9.4	.4	9.0	Milky. Very cloudy.	T
3	.68	10.82	1.6	9.22	Same as No. 2.	L
4	1.37	11.54	2.2	9.34	Pale yellow. Almost clear. Small amount of suspended matter.	C
5	.93	11.4	2.1	9.3	Same as No. 4.	Q
6	1.03	9.55	5.1	4.45	Faintly bluish fluorescence. Practically clear.	Less than A
7	3.63	9.5	3.8	5.7	Colorless. Slightly cloudy.	Less than A
8	1.9	9.6	.393	9.21	Very yellow. Clear.	Less than A
9	1.9	10.06	4.0	6.06	Colorless. Somewhat cloudy.	C
10	1.8	10.2	4.3	5.9	Colorless. Clear.	Less than A
11	2.8	10.5	1.64	8.86	Very yellow. Clear.	D
12	0.57	8.16	.8	7.36	Colorless. Clear.	Much more than T

* Gardner's results.

Total Ash, Aluminum Oxide, and Water Soluble.—The determination of these constituents is an important part of the chemical examination. They are easily and quickly determined, and are valuable indications of quality. Of the samples under investigation, those which have the lowest viscosity induction values have a high percentage of water soluble, while in those yielding the most viscous solutions, the water soluble content is very low. These determinations were carried out as follows:

About five grams of the sample was weighed into a silica crucible and carefully ignited over a Bunsen flame. The residue was reported as total ash. The crucible was then placed in a 400 cc. beaker and treated with boiling water to dissolve the water soluble portion. The solution was filtered through a quantitative paper and the paper burned in the same crucible. The second ashing represents Al_2O_3 with the traces of iron and other impurities that may be present and the difference between the two weighings represents the water soluble.

Iron.—The presence of iron is readily detected by the yellowish red color which it imparts to the stearate solutions. The proportion is usually very low, less than .1 per cent. In such small amounts it would have no effect on quality, except in so far as color is concerned. Traces were found in both good and poor samples, hence it is believed that a quantitative determination is unnecessary except possibly when the material is to be used in a white or very light-colored product. Even in the latter case observations of the color of solution alone would probably be sufficient..

Suggested Tentative Specifications for Aluminum Stearate

The aluminum stearate must be finely divided and satisfactory for use in the grease industry. It must be white in color, and contain not over 0.1 per cent iron as Fe_2O_3.

Moisture.—It shall contain not more than 1.5 per cent of moisture.

Water Soluble.—It shall contain not more than 2.0 per cent of water soluble material.

Water Insoluble Ash.—The ash from the water insoluble material shall be not less than 6.0 per cent.

Appearance of Solution.—When 2 grams of aluminum stearate is dissolved in 50 grams of xylol, a practically colorless and fairly clear solution shall be obtained.

Jelly Test for Aluminum Stearate

The following jelly test for aluminum stearate is given, by which it is possible to compare the qualities of various aluminum soaps from different sources: Heat 10 grams of aluminum stearate with 90 cc. of mineral oil in a 200 cc. glass beaker to a temperature of 215° to 250° F. This mixture should be stirred vigorously and constantly while being heated. After

complete solution is effected, allow the material to cool to room temperature slowly. In this test a thick jelly is formed which varies with different makes of soap, from a soft oily mass to a hard jelly. A clear solid jelly is preferred.

Licata's Method for Preparing Stearate Grease Samples [16]

Ten grams of the aluminum stearate and 90 grams of the mineral oil to be used are thoroughly mixed in the cold and then the mixture is heated to 275 to 300° F. The grease is then poured into ointment tins and placed in a cooling chamber which will cool the grease to 70° F. in eight hours. An electric drying oven with an initial temperature of 105° C. and with the current turned off was found satisfactory by Licata.

General Discussion of Aluminum Stearate Base Greases

Advantages of the greases made with an aluminum stearate base may be listed as follows:

A high degree of transparency and clarity which implies exceptional sales appeal.

Melting points are intermediate between those of calcium and sodium soap greases.

They are waterproof and are not broken down by water as is the case with sodium soap greases.

They become more adhesive and cohesive as temperature is increased to about the melting point. For most pressure gun applications this is advantageous, but for high speed ball bearing lubrication the gelatinous condition may be considered conducive to leakage and development of high bearing temperatures.

They are heat stable. They can be heated to above their melting points and on cooling heturn to a consistent grease. This is not true of most calcium greases.

Disadvantages of aluminum stearate base greases are chiefly of a manufacturing nature rather than concerned with service characteristics:

Cost of materials for aluminum stearate grease is greater than for normal calcium and sodium base greases.

The manufacturing labor cost and equipment required for the aluminum greases is greater in view of the necessity for using fairly high temperatures (250° to 300° F.) and chill pans for cooling the grease. Considerable hand labor is necessary in handling the grease.

Control of consistencies is difficult and some batches may require reworking.

There are many factors which may greatly affect the consistency, stability, color and yield of greases made with aluminum stearate:

Aluminum stearate, as marketed, does not appear to be a definite chemical compound. The moisture content, presence of free stearic acid, inorganic salts and aluminum soap content all affect the character of the finished grease. Some stearate manufacturers have modified their stearate by incorporating with it waxes such as beeswax, rubber latex, small amounts of aluminum oleate, and aluminum palmitate. The object of these inclusions is to produce some definite result with regard to stability, yield, adhesive-

[16] *Natl. Petroleum News*, **25**, 23 (1933).

ness, etc. This would indicate that straight aluminum stearate greases were by no means as yet considered as being ideal lubricants.

McBain and McClatchie [17] have investigated the composition of various aluminum stearates on the market and conclude that they are mechanical mixtures from which an appreciable amount of free fatty acid may be readily extracted with ether, leaving a monobasic or at most a dibasic soap. The stearates, which they prepared in the laboratory, on ashing yielded 5.0 to 6.2 per cent of aluminum. Gardner has reported that commercial stearates may yield as much as 4.92 per cent and as little as 2.35 per cent of aluminum; this, of course, without extracting any free stearic acid which may have been present. This question is, of course, quite controversial and it may be possible that the high ash values are due to a combination of the effects of inorganic aluminum salts (possibly alumina) left in the stearate and the presence of mono- and di-stearates. A low ash may indicate a relatively high percentage of the tri-stearate or the presence of considerable free stearic acid. Pure aluminum palmitate should, on careful analysis, be found to contain 3.4 per cent of aluminum; however, the palmitates prepared by McBain yielded from 4.43 to 5.38 per cent aluminum indicating mono- and di-stearates.

Craig [18] has proposed the normal basic double stearates, palmitates, oleates, etc., as ingredients of lubricating greases. They may be produced by causing a solution of alkali and a metal oxide soluble in the alkali solution to act on the desired fatty acids. As metal oxides suitable for this purpose zinc oxide and aluminum oxide were suggested. In an example, the normal compound $2(Na_2O) \cdot Al_2O_3 \cdot 10(C_{18}H_{35}O_2)$ is produced by mixing at 80° C. 687.11 pounds of stearic acid and 300 pounds of sodium aluminate solution containing 8.22 per cent of Al_2O_3 and 9.99 per cent of Na_2O; heated until dry. An excess of stearic acid may also be used.

The effect of the mineral oil on the nature of the finished grease has been investigated by Licata. He found that, for a given stearate and using oils having an S.U.V. of 300 at 100° F., a Mid-Continent Oil gave the highest yield. Pennsylvania Oil was next and Naphthenic Oil gave the softest grease. Oils having a viscosity less than about 300 S.U.V. at 100° F. were found to be of short texture, of a granular nature, and tended to bleed and be brittle. High viscosity oils gave a long feather and relative freedom from bleeding.

Cooling rate is probably the most important factor in controling the character of aluminum base greases during their manufacture. McKee [19] has demonstrated that the rule of cooling will affect the Worked A.S.T.M. Penetration from 193 to 267 1/10 mm.; or, in other words, a difference of 74 in the penetration is possible for greases otherwise made in exactly the same way, but cooled at different rates. Quick cooling gives a high yield, but also increases instability, bleeding, and brittleness. McKee reported as follows:

[17] *J. Amer. Chem. Soc.*, **58**, 3266 (1932).
[18] British Patent 338,919 (May 21, 1929).
[19] *Natl. Petroleum News*, **27**, 24-E (1935).

Time of Cooling Hours	A.S.T.M. Worked Penetration	Remarks
2	193	Granular, excess bleeding
12	225	Smooth, no syneresis
18	238	Smooth, no syneresis
25	267	Smooth, no syneresis

The effect of moisture is most important, particularly when preparing stearate grease with oils of low viscosity. McKee made greases with 310 S.U.V. at 100° F. Naphthenic Oil, heating them to 302° F. (150° C.) for various lengths of time and obtained results as follows:

Hours at 150° C.	A.S.T.M. Worked Penetration	Percent Water in Grease
0	275	0.15
1	240	0.12
3	225	0.10
6	222	0.05

It is obvious that with increased dehydration the yield is increased.

Corrosion accelerated by the action of lubricating greases is an important matter and it is of note that, in some instances, aluminum stearate greases have appeared to increase this objectionable action. It is not surprising since some stearates are known to contain appreciable quantities of stearic acid. Thus, greases made with them might, in the presence of moisture and air, cause appreciably more corrosion than other neutral greases. A grease was made with 95 per cent of 1100 S.U.V. at 100° F. Naphthenic Red Oil, and 5 per cent of a commercial aluminum stearate. This grease was heated at 100 to 105° F. for a prolonged period with strips of copper, mild steel, and brass. While the test at 100° C. indicated appreciable corrosion would occur, similar tests made at room temperatures indicated no objectionable corrosion.

Condition of Metals in Test at 100 to 105° C.

	Copper	Mild Steel	Brass
After 24 days	Staining on portion of strip exposed to the air. That in contact with grease unaffected. Grease broken down and darkened.	Unaffected. Grease broken down and darkened.	Corrosion had occurred on parts of the immersed strip. Corrosion most marked at air-grease interface. Grease broken down and darkened.
After 27 days	External staining noted at 24 days slightly aggravated. No corrosion by the grease had occurred.	Unaffected.	Condition noted at 24 days slightly aggravated.
After 41 days	Extensive external staining and uniform etching of the metal immersed in the grease.	Unaffected.	No appreciable change in condition; *i.e.*, corrosion on parts of immersed metal.

Conditions of Metals in Test at Room Temperature

	Copper	Mild Steel	Brass
After 27 days		No staining or corrosion.	
After 41 days		No staining or corrosion.	

Film strength and oiliness do not appear to be greatly affected by the presence of aluminum stearate, although those greases containing appreciable amounts of free stearic acid, from the original soap, have been found to have noticeable characteristics in this respect. Wilch [20] has observed that greases made with aluminum stearate have a low cohesive character when made with low viscosity oils, as indicated by their short shear and buttery texture. These greases, also known as "solidified oils," are frequently used for the lubrication of Tryon shackles as they do not produce objectionable residues and, when properly compounded with oleate or latex, are excellent lubricants from the standpoint of freedom from dripping.

The yield and melting points of various mixtures of a commercial aluminum stearate, manufactured on the Atlantic coast, with 300 S.U.V. at 100° F. Naphthenic Pale Oil have been investigated by the author. The greases were heated to 300° F. for ten minutes and then cooled in three-inch layers at room temperature of 75° F. The results were:

Per Cent by Weight Aluminum Stearate	A.S.T.M. Penetration		Ubbelohde Dropping Point, ° C.
	Worked	Unworked	
6	430	420	78
7	392	367	82
9	360	295	83
10	344	278	83
12	320	243	86
14	298	212	92
16	280	185	108

It is of interest to note that as the percentage of aluminum stearate is decreased the difference between worked and unworked penetration values becomes progressively less, so that at a soap content of about 5 per cent the differences disappear. The dropping points increase with increased soap content as would be expected.

Syneresis (bleeding) and hysteresis on alternate heating and cooling have been investigated by the author. It was found the Pennsylvania oils, compounded at high temperatures, show little separation; when made at low temperatures they separate badly. In 1927, Klemgard [21] recognized this phenomenon and proposed the formula consisting of:

	Per Cent by Weight
Technical grade aluminum stearate	7.5
300 S.U.V. at 100° F. paraffine oil (Pennsylvania or Mid-Continent)	92.5

This grease was processed at a temperature of 425° F. to secure maximum transparency; 335° F. being considered the lowest temperature for obtaining maximum yields and best transparency. Naphthenic base oils compounded at high temperatures tend to separate but, when compounded at low temperatures, are softer and do not separate badly. The data on which these conclusions were based is given below:

[20] *Natl. Petroleum News*, **26**, 29 (1934).

[21] U. S. Patent 1,749,251 (Mar. 4, 1930).

Nature of Oil	Max. Temp. During Mfg. ° F.	Cooled to ° F.	A.S.T.M. Unworked Pen.	After 3 Months: Per Cent Oil Separated	After 3 Months: A.S.T.M. Unworked Pen. 3 Months	After 3 Months: A.S.T.M. Unworked Pen. Change
Pa. and Calif.	300	Room	289	.20	215	74 minus
300 Naph.	350	"	280	2.44	251	29 "
" "	250	"	263	.56	285	22 plus
" "	275	"	292	.77	248	44 minus
" "	300	"	278	.44	312	34 plus
140 Pa.	250	"	320	6.26	317	3 minus
" "	300	"	288	3.40	313	25 plus
" "	370	"	280	.78	235	45 minus
" "	250	"	346	soft	400 plus	.....
" "	250	32	315	2.96	275	40 minus
" "	370	Room	300	.75	290	10 minus
" "	370	32	224	.20	239	15 plus
300 Naph.	250	Room	293	soft	400 plus	.....
" "	250	32	198	5.50	227	29 plus
" "	370	Room	309	soft	268	41 minus
" "	370	32	207	5.75	177	30 minus

All of the above greases were made with 10 per cent by weight of the same commercial aluminum stearate.

As to the stability of the aluminum stearate base grease, it has been pointed out from time to time that they are unstable even on storage. That they break down on mechanical mixing is shown by the following tests on typical samples:

	Experimental I	Experimental II	RHF H.P.	SP MX No. 21
A.S.T.M. unworked penetration	367	350	354	344
A.S.T.M. worked penetration	400 plus	400 plus	400 plus	400 plus

MacMichael viscosities were determined on samples of grease composed of 7.5 per cent of aluminum stearate and 92.5 per cent of 150 S.U.V. at

MacMichael Viscosities (Degrees)		Temperature of Grease ° F.
	Sample I	
12		290
32		275
64		260
84		250
116		240
140		215
170		200
193		185
	Sample II	
185		125
145		130
100		145
100		155
90		165
70		175
65		185
	Sample III	
32		280
64		265
84		250
116		225
140		200
170		185
190		170

210° F. Naphthenic Bright Stock. The viscosities were made with the No. 26 wire, a 1 cm. bob, and at a speed of 60 R.P.M.

Since the cooling rates are not equivalent and possibly due to the evaporation of moisture from the greases under test, as well as the effects of agitation during cooling rather than quiescent cooling, considerable hysteresis or failure to return to normal viscosities of the original greases may be observed in the above data. Incidently, this data shows that aluminum stearate greases have considerable viscosity at temperatures above 212° F. Calcium base greases will be found to be quite thin at temperatures much over this value.

Sales Demonstrations of Aluminum Stearate Greases

Salesmen frequently mix their aluminum stearate base greases with gasoline and ignite the mixture before a prospective customer. The mixture burns leaving practically no ash as the alumina is very light and is carried off. Similar tests on sodium or calcium greases leave a hard charred mass. The inference is that the aluminum grease is "pure 100 per cent solidified oil" and therefore, in view of its "high oil content," must be a superior lubricant. No lubrication technologist would be misled by such a demonstration. Stearate greases, compounded with rubber hydrocarbons, may be placed on a smooth surface, such as an anvil, and struck with a hammer without causing splattering as such greases are very cohesive. Less cohesive greases, such as normal aluminum, calcium or sodium greases, may splatter badly.

Analysis of Commercial (Grease Making) Aluminum Stearate (MS—532, —1930)

The following analysis was made on the stearate manufactured by an Atlantic Coast company and offered for use particularly as a grease base:

Composition, per cent by weight:	
Fatty acid (combined)	90.48
Ash	7.20
Sulfates (SO_3)	1.06
Fatty Acid:	
Acid value (mgs. KOH)	159
Iodine value (Hanus)	22
Melting point ° C.	50
Color	White
Odor	Beeswax
Probable source may be a mixture of stearic acid and beeswax	
Ash:	
Aluminum oxide (Al_2O_3)	97.60
Potassium carbonate (K_2CO_3)	1.04
Loss and undetermined	1.36
Total	100.00

Probable Composition, per cent by weight:	
Aluminum soap (probably stearate and wax)	97.7
Potassium sulfate (K_2SO_4) impurity in manufacturing process used	2.3

Grease Making with MS-532

The above manufacturer recommended the following process for making greases with their stearate, which is quoted below:

"Introduce one-half of the necessary oil in the kettle. Add the base and mix thoroughly without any heat. After the two have been thoroughly mixed in the cold so that the base is well dispersed, add remainder of oil and turn on heat. Raise the temperature, while mixing to 260 to 280° F. As the temperature is increased the base will slowly dissolve so that at (about) 180° F., the mixture is very tough and rubbery. This should not cause any alarm because on further heating the mixture will thin out. When the maximum temperature desired has been reached, *i.e.*, between 260 to 280° F., continue mixing at this temperature for two hours.

"The rate of cooling from this maximum temperature to room temperature resolves itself into two parts. The first part takes place from the maximum temperature; *i.e.*, 260 to 280° F., to the setting temperature, or gel point. There is no particular difficulty during this stage, and for expediency the cooling should take place in the grease kettle with agitation to insure uniform heat loss.

"Cooling may proceed rather rapidly and can be accomplished by circulating cold water in the steam jacket of the kettle. When the temperature has been lowered to about 200° F., it is well to cool somewhat slower. As soon as the mass of grease shows the slightest tendency to lose its fluidity and tends to agglomerate in large masses, agitation is stopped.

"We are now approaching the critical stage of grease making which is the gel point of the grease. It is of utmost importance that the grease be discharged from kettle before this point is reached. Here, we need the assistance of some skill for what we want is to approach this gel point as near as possible before discharging the grease into containers. For your guidance we will say that this point lies roughly between 160 to 180° F.

"The consistency of the finished grease depends on the rate of cooling in these containers. The grease maker decides what he wants and cools accordingly. For example, for an average grease cooling in drums, the volume-surface relation at a certain room temperature and discharge temperature should be such that it will require about three days to cool to 90° F. For harder greases, where cooling rate desired is somewhat faster, pans are more desirable. We emphasize the fact that the grease in containers must not be disturbed and care must be exercised not to cool too rapidly.

"Two general rules that should be observed are as follows:

"Wherever exposed surfaces of grease in containers tend to cool too fast, producing a danger of top granular grease layer, radiation losses may be minimized by placing a piece of insulating material such as wrapping paper, cardboard or fiber board on the surface.

"Mixing paddles in kettle should, if possible, be kept below surface of grease to prevent entrainment of air."

Analysis of Five Commercial Aluminum Stearates

The analyses of five commercial stearates is given below; 1 was manufactured in New York, 2 in New Jersey, 3 in Buffalo, 4 unknown, and 5 in Cleveland:

Sample No.	Ash Per Cent	Moisture Per Cent	Percent of Ash Water Soluble	Percent of Ash Water Insoluble Mostly (Al_2O_3) (by Difference)	Pene. on Grease* Unworked at 77° F.	Pene. on Grease* Worked at 77° F.
1	8.3	2.5	9.04	90.96	216	360
2	9.90	2.5	6.51	93.49	181	295
3	9.24	2.5	4.07	95.93	226	372
4	8.24	...	1.45	98.55	156	260
5	10.32	...	11.30	88.70	185	290

* Samples of grease were made using 10 per cent of the samples of aluminum stearate and 90 per cent of Motor Oil 50, heating to 270° F., agitating for five minutes and cooling.

Early Uses of Aluminum Soaps as Lubricating Grease Bases

In 1855, Johnson [22] prepared a grease by first melting together potassium carbonate, aluminum nitrate and aluminum sulfate with rosin. Fats were also incorporated with the composition and the finished product contained resinates, oleates, stearates, and probably palmitates of aluminum.

Gerdon and Schindler,[23] of Albany, N. Y., in 1868 prepared the following grease:

	Parts
Tallow	100
Potassium hydroxide	3
Borax	5
Alum	3

The chemicals were dissolved in water and boiled together with the tallow to promote reaction and evaporate the water. The final grease had melting points of 100 to 110° F.

Dieterichs,[24] of Cleveland, prepared aluminum soaps in 1882 and dissolved them in oxyhydrocarbon or hydrocarbon oil to produce lubricating greases. A grease manufactured by Martinez,[25] in New Orleans in 1885, had the following composition:

Mineral oil	50 gallons
Scrap rubber (caoutchouc)	1 pound
Alum	1 "
Graphite	1 "
Rosin soap	1 "

The above materials were mixed with ten gallons of the oil and heated to 200 to 350° F.

Aluminum Stearate Base Chassis Greases (Lucid Compounds)

According to Kopp,[26] aluminum stearate greases when used in automotive gear cases, where they may become mixed with other lubricants, may become badly emulsified with any water present and also tend to pick up air and increase in volume. No data is available to indicate whether the aluminum soaps are worse offenders in this regard than other types of greases. Furthermore, the nature of the oil in the grease is also of importance in overcoming frothing difficulties of gear lubricants. Lucid lubricants, so called, are aluminum stearate base greases made in such a manner as to have a long stringy texture, and thus tend to resist dripping and loss when used in shackle bearing on automotive units. According to Kopp, the stringiness of lucid compounds may be obtained by the addition of aluminum oleate in sufficient quantity or by adding 100 S.U.V. at 100° F. pale oil containing dissolved rubber. While, strictly speaking, rubber may not be a lubricant, the lubricating qualities of many of the soaps used in greases

[22] British Patent 1,116 (May 17, 1855).
[23] U. S. Patent 73,089 (Jan. 7, 1868).
[24] U. S. Patent 260,670 (July 4, 1882).
[25] U. S. Patent 326,311 (Sept. 15, 1885).
[26] *Petroleum Engr.*, 4, 84 (1932).

may also be questioned. The preparation of the gum rubber solution in light pale oil is not convenient or simple. Kopp recommended setting aside drums containing the necessary quantities of crepe rubber and the pale oil in a warm room at about 100 to 140° F. The mixture is stirred occasionally to facilitate solution. The adhesiveness of the grease is, of course, due to the stringiness imparted by the rubber or aluminum oleate.

As previously mentioned, the rate of cooling is very important in making aluminum stearate base greases, and for the manufacture of lucid greases the general rules apply.

The grease should be drawn off from the kettle into rectangular pans, about eight inches deep, which are placed in rooms maintained at 70 to 80° F. This, of course, means that during winter the room must be heated and during summer artificial cooling should be resorted to. Kopp gives the following two formulae and points out that for a high degree of clarity the soap content should be kept at a minimum as well as the amount of material used to secure a stringy texture.

Formula I

	Pounds	Per Cent by Weight
Aluminum stearate	70	5.8
750 S.U.V. at 100° F. motor oil	1083	89.8
5 per cent by weight rubber solution	50	4.0
Stearic acid	6	0.5

This grease will contain 0.2 per cent by weight of rubber hydrocarbons. The aluminum stearate is thoroughly mixed in the cold with the Motor Oil. After adding one-quarter of the weight of the oil to the kettle, to it then add all of the aluminum stearate and stir thoroughly. When the stearate is intimately mixed the remainder of the oil may be run in, stirring continuously, and the batch heated gradually to 300° F. The batch is then allowed to cool to 230° F. in the kettle. The rubber mixture and stearic acid is then added and the grease drawn off into the pans to a depth of about six inches. The pans are about 4 feet wide and 8 feet long. The pans are placed in a room at 75° F. After about 12 hours, the grease will lose much of its rubbery character and sett at about 160 to 200° F.

Formula II

	Per Cent by Weight
Aluminum stearate	5.0
Aluminum oleate	3.0
750 S.U.V. at 100° F. motor oil	92.0

The aluminum stearate is mixed with the oil in substantially the same manner as in Formula I. When all the oil has been added the aluminum oleate pulp, containing about 20 per cent by weight of aluminum oleate, is thoroughly stirred in. The batch is heated gradually, as in the above case, to 300° F. and drawn off and cooled as before.

Rubber Latex-Aluminum Stearate (Lucid Grease)

Klemgard [27] has developed a method of preparing lucid greases in which much of the inconvenience of preparing gum rubber solutions may be avoided. Rubber latex or natural rubber is employed which consists of very minute rubber globules dispersed in a watery fluid. The latex often contains, besides water and rubber, various constituents as impurities, such as proteins and resinous materials, as well as some anti-coagulant such as ammonia. Latex containing from 30 to 50 per cent by weight of rubber hydrocarbons is recommended by Klemgard.

Formula

	Per Cent by Weight
Technical aluminum stearate	9.5
Rubber latex	.5
300 S.U.V. at 100° F. naphthenic pale oil	90.0

This grease will contain about 0.25 per cent by weight of rubber hydrocarbons. In manufacturing this grease, the usual type of steam jacketed compounding kettle may be used with success, although it is possible to dissolve the materials in the oil in a still, or other heated receptacle, without the means of strong mechanical agitation. About one-third of the oil is placed in the compounding kettle, the aluminum stearate added and the mixture stirred to obtain a suspension of the stearate in the oil. The rubber latex, in liquid form, is then added to the contents of the kettle and steam turned on. Heating is continued until substantially all of the water present in the latex and the stearate is evaporated, which will ordinarily require temperatures of 150 to 250° F. when naphthene base oil is used. Where the percentage of aluminum stearate is above (about) 15 per cent of the mixture, or when making use of oils of paraffine base, higher temperatures ranging from 250 to 500° F. may be required in order to dissolve the soap and to obtain a satisfactory yield of a transparent homogenous material.

The grease is drawn off into shallow iron pans to cool, the depth of the grease layer being from 2 to 8 inches depending on the atmospheric temperature.

It is now possible to purchase stearate already blended with the correct amount of latex, introduced during the manufacture of the aluminum stearate. Klemgard worked out the details of this process in 1934.[28]

The addition of rubber to aluminum soap greases is not particularly new. For instance, in 1885,[29] a grease was prepared consisting of mineral oil, scrap rubber, or caoutchouc, rosin soap, graphite and alum.

Rubber latex is, of course, readily dispersed in water and when blended with (about) 70 per cent of water may be sprayed into a closed tank containing dry powdered aluminum stearate. The colloidal rubber solution is desposited evenly on the stearate particles if proper air agitation is provided. The mixture may then be dried by heating at 212° F. and when

[27] U. S. Patent 1,975,582 (July 28, 1930).
[28] U. S. Patent 1,979,943 (Nov. 6, 1934).
[29] U. S. Patent 326,311 (1885).

dry shows little tendency to pack or agglomerate unless the amount of rubber is great.

In the manufacture of aluminum stearate it will be found convenient to mix a desired amount of latex with the sodium stearate solution. When an aqueous solution of alum is added to such a mixture of latex and sodium stearate solution, aluminum stearate bearing the rubber latex will be precipitated. Rubber bearing stearates of this kind are readily worked up into lucid greases. Another method of incorporating rubber with aluminum stearate greases is to either cut or shred it into very fine particles. These may be mixed dry with the stearate or dissolved separately in hot oil. From 3 to 10 hours, however, may be required to dissolve the gum rubber.

Horwitz [30] has developed formulae for aluminum stearate greases of definitely stringy character, the stringiness being considered desirable as a means of preventing dripping and loss of lubricant, therefore providing more efficient lubrication. In one case Horwitz adds about 5 per cent by weight of pure glycerin to 95 per cent of a metallic soap, such as aluminum stearate, to produce a grease base. From one-half to 3 per cent by weight of this base may be incorporated with mineral oil for the production of a lucid, stringy lubricant. In another example, Horwitz combines the glycerin with (about) 20 per cent of water to increase the volume to facilitate the initial introduction of a greater amount of the aluminum stearate; for instance, about 50 per cent of stearate, and also effects quicker mixing of the stearate with the glycerin. After thoroughly mixing the resultant mass may be subjected to temperatures of 180° to 190° F. or slightly higher to evaporate the moisture.

Another stringy base may be made by mixing together 5 per cent of glycerin, 12 per cent sperm oil and 8 per cent of castor oil with 75 per cent of aluminum stearate. These materials are thoroughly stirred together to produce a product of granular or powdered character having slightly greasy or oily characteristics. About 3 per cent by weight of either of these bases is mixed with one-third of the mineral oil required, in a steam jacketed grease mixer. The batch is heated to about 220° F. when a clear product should be obtained. The remaining two-thirds of the oil is then added, thoroughly mixed in, and the lubricant cooled and drawn from the kettle.

Gebhardt [31] has prepared consistent greases by mixing 95 to 50 pounds of mineral oil and 5 to 50 pounds of an aluminum soap of a saturated fatty acid, containing 15 or more carbon atoms per molecule, heating slowly to 160 to 260° F., and cooling rapidly enough to prevent reversion to a thin product. He preferred to use 85 to 55 pounds of cylinder stock oil with 15 to 45 pounds of aluminum stearate or palmitate, heating them to 260° F. in 4 to 7 hours. He then cooled the grease at atmospheric temperature in layers 2 to 6 inches in thickness, and suggested the use of artificial cooling at 0° F. Other substances, especially such as to render the grease stringy or feathery, were added if desired.

[30] U. S. Patent 1,939,170 (Dec. 12, 1933).

[31] British Patent 406,399 (Sept. 29, 1932); see also: U. S. Patent 1,902,635 (March 21, 1933).

Horwitz [32] has also patented a grease base composed of aluminum stearate, Japan wax, and castor bean oil.

Use of Rubber in Lubricating Greases

There is evidence, as shown in the following table, that rubber has been used in lubricants as long ago as 1855:

H. Hyde, British Patent 2,668 (Nov. 27, 1855).
A lubricant containing a small amount of an elastic hydrocarbon, such as East Indian or Para caoutchouc, dissolved in hydrocarbon oil (at 80 to 150° C.).

C. Schiele, British Patent 1,317 (May 29, 1860).
Caoutchouc dissolved in fatty oils such as olive oil. Low heat is used.

R. A. Brooman, British Patent 3,121 (Dec. 19, 1861).
Caoutchouc heated at 125 to 230° C. to form "hevéone" which is blended with olive or other oils to render them more viscous.

W. E. Newton, British Patent 2,176 (July 31, 1862).
Caoutchouc dissolved in crude coal oil or rock oil at about 200° C.

J. Wilkins, British Patent 2,012 (June 2, 1875).
Petroleum is mixed with dense substances such as india-rubber solution, tallow, sult, etc., to form lubricating compositions.

E. Poole, British Patent 2,381 (June 12, 1880).
Raw india-rubber is placed in benzene, or olein oil, till it has softened; the mixture is then heated till the whole has dissolved to a liquid paste, which is then mixed with other oils, previously heated.

W. T. Hicks and J. Elliott, British Patent 9,477 (June 29, 1888).
Rolling mill lubricant consisting of clay, animal fat, blacklead, and india rubber.

Krause,[33] in 1891, prepared a lubricant from lanolin and rubber by the following process:

Wool grease was treated with the required amount of caustic soda. The sodium soap was then dissolved in water and filtered. A solution of alum was prepared and the two solutions were brought together. A brown compound was precipitated which the inventor called "aluminum lanolate." For the production of lubricants, this product was dried and dissolved by heating in fluid mineral oil. In other examples of Krause's compounds the aluminum lanolate was incorporated with India rubber in one of three ways:

The compounds were melted together.
By pulverizing the lanolate and grinding it with the rubber.
By dissolving each of the substances and mixing them while in a plastic state.

Transparent Aluminum Stearate Base High Pressure Grease

The following comments relative to the manufacture and appearance of this product will be of interest:

[32] U. S. Patent 1,881,591 (Oct. 11, 1932).
[33] British Patent 5,226 (Feb. 7, 1891).

Color:

The color of these greases, as with nearly all soap base lubricants, is chiefly dependent on the color of the oil content. If a red color with green outercast is desired then FFF Pennsylvania stock may be blended with suitable pale oils, or fluorescent green dyes derived from coal tar or petroleum residues should be used. Greases having A.S.T.M. colors of 1, 2, 3, and 4 are quite possible when using aluminum stearate. The temperature, to which the grease is heated during manufacture, has an influence on the color of the finished grease, the higher temperatures causing considerable darkening, particularly with naphthenic oils of low viscosity index.

Transparency:

The method of preparing samples of aluminum base grease, has a very important bearing on transparency. In some cases the greases were poured into the small glass jars, while in a liquid state. In this manner, all air bubbles are eliminated and a very transparent product obtained; providing, of course, the color of oil employed is sufficiently light.

The trick of preparing samples by pouring the melted grease into small jars, where it cools rapidly to a transparent product, has been used by several manufacturers, and is open to objections. The grease prepared in this way takes on a false "sett" which is not comparable with the product produced on a commercial scale. In the commercial production, it is customary to transfer the grease from the chill pans directly to the containers in order to maintain the transparency of the grease. Working of the grease in this way causes a slight decrease in consistency, occlusion of air and in general there is a tendency for the grease to become more opaque. From a commercial standpoint, it is possible to pour the hot grease into one-pound tins where the grease cools to give a product which is identical to that formed in the glass jars. Cooling the grease in larger containers results in products of irregular consistency and soft "cores" in the center of the cans.

Observations and formulas for several products made by pouring the hot grease into small jars are given below:

No. 1: This grease has the formula:

	Per Cent
Aluminum stearate	9.00
300 pale oil	50.00
3 F stock	8.00
140 Eastern pale	33.00

It was noted that the grease is so dark, due to the color of the 3 F stock and other oils used, that a card immersed in it can hardly be read. Air pockets are retained by this grease at a temperature of 275° F., while being poured into the jars.

No. 2: Is slightly lighter in shade than No. 1, and has the formula:

	Per Cent
Aluminum stearate	9.00
3 F stock	10.00
300 pale oil	81.00

This sample also retains air pockets due to the high viscosity of the grease at a pouring temperature of 275° F. It showed some oil separation.

No. 3:

	Per Cent
Aluminum stearate	9.00
300 pale oil	91.00

Poured at 275° F. into jar. Made with No. 4 color, 300 Pale.

No. 3A: Same formula as 3, butt poured at 325° F. which is a higher temperature than can be attained with equipment at most compounding plants. However, for purposes of advertising, the samples could be prepared by the laboratory at any desired temperature but would, of course, not be representatives of commercial production. Is slightly turbid.

No. 4:

	Per Cent
Aluminum stearate	9.00
140 Pennsylvania pale oil	91.00

Poured at 275° F. Is bright, but oil stock quite light in viscosity.

No. 5:

	Per Cent
Aluminum stearate	9.00
300 pale oil	46.00
140 Pennsylvania pale oil	45.00

Poured at 275° F. No turbidity.

It was noted that the color of these greases as well as their degree of transparency was dependent on the characteristics of the oil used in compounding them. This sample shows that even with about 50 per cent of eastern stock a bright product may be made.

No. 6:

	Per Cent
Aluminum stearate	9.0
100 pale oil (No. 2 color)	91.0

This sample was a very pale grease, produced with 100 Pale oil, and it had only a very slight turbidity; the oil, however, was considered too light in viscosity for a satisfactory H.P. lubricant, but was included in order to show the results of using a very pale western oil.

No. 7: Same as No. 3, but made with No. 3 color 300 Pale oil. The tendency towards turbidity was quite marked, and it appeared that this was characteristic of the greases made with the heavier western stocks of low A.P.I. gravity.

No. 8:

	Per Cent
Aluminum stearate	9.0
Turbine oil B. (No. 3 color) 300 vis. at 100°.....	91.0

Very transparent and bright.

It has already been pointed out that the most translucent or transparent aluminum base greases are made at high temperatures and with eastern pale oils. The Pennsylvania grade oils give the brightest and most transparent greases; Mid-Continent oils may be used to produce fairly bright products and are considered satisfactory, as are also mixtures of about equal parts of Eastern and Western oils.

It has not, however, been possible to produce a product equal in appearance to those made with light Pennsylvania oil, making use of Naphthenic 300 Pale Oil. While a definite, scientific explanation of the cause of the turbidity in greases made with heavy Naphthenic oils is lacking, it may be possible that the transparency is a function of the index of refraction of the oil. It is known that the refractive properties of similar products from various crude oils are found to be parallel to their specific gravities. Saturated hydrocarbons have smaller refractive indices than those of similar boiling point, but of naphthene base, and these again have lower indices than the corresponding aromatic ones. In a series of distillates from the same crude, the refractive index rises with the boiling temperature. Thus, it is possible to make a fairly bright grease with 100 Pale; but 300 Pale, having a higher specific gravity and refractive index, gives turbid products. The eastern oils of low specific gravity, have correspondingly low refractive indices, and result in bright, aluminum base grease products.

No. 9:

The Turbine Oil used in the following grease had an S.U.V. at 100° F. of about 200 and was refined from naphthenic crude to have a viscosity index of about 75 (Dean and Davis).

Per Cent		Pounds	Gallons	Cost*	Cents
88	Turbine oil B	4000	526 at	12.5c per gal.	6575.000
3	FFF stock	135	18 at	66.0c per gal.	1188.000
9	Malinckrodt's aluminum stearate	409	... at	28.606c per lb.	11699.854
	Cost of materials for 4544 lbs. of grease				19462.854
	Cost of materials for one lb. of grease				4.283
	Cost of compounding for one lb. of grease				1.000
	Total cost of one lb. of grease ex kettle				5.283

A brief "log" of the batch is given below:

10:00 A.M. Start charging oil stocks to kettle.
10:45 " Start charging aluminum stearate.
11:20 " Charging of all materials to kettle completed, and paddle agitation on.

		Temperature of Grease, ° F.	Steam Pressure in Jacket
1:00 P.M.	Steam on	95	80 pounds per square inch
2:15 "		180	85 " " " "
2:30 "		190	95 " " " "
3:30 "		230	98 " " " "
3:45 "		230 to 240	98 " " " "
4:00 "	Grease drawn at temperature of 230° F. into the following containers:		

Method of Cooling	Penetration at 77° F. (after 24 hrs.)
1 pound can	282
5 pound can	278
3" layer in 3' × 12' cooling tray	310–335
25 pound can	341
10 pound can	380
15 gallon tank	360 plus
30 gallon tank	360 "
50 gallon tank	360 "
Grease kettle (very soft)	400 "

Room temperature 60 to 80° F.

All of the samples from the above containers were clear and transparent, but the consistencies varied considerably according to the method of cooling. It is obvious that the chill pan (or cooling tray) is most satisfactory for the manufacture of the aluminum stearate base greases.

It will be noted that the grease poured while hot, into the small tin containers, has taken a very pronounced "sett," and is slightly too heavy for a satisfactory pressure grease. Very slow cooling in large containers results in soft grease.

Inspections of the grease made by cooling in the tray to a depth of three inches are given below:

A.S.T.M. penetration at 77° F.	310–335
Dropping point, ° F.	203
Corrosion test (copper)	O.K.
Percent water	0.10
Percent ash	0.987

The effect of temperature on the A.S.T.M. Unworked Penetration is shown as follows:

Temperature ° F.	Unworked Penetration at Temperature Indicated
77	318
100	362
120	398
130	400

Analyses of Commercial Aluminum Stearate Base Pressure Gun Greases

N High Pressure Grease

Physical Tests:	
M.P. ° C.	86
Composition (per cent net):	
Soap	8.1
Oil	91.9
Ash, per cent Al_2O_3	69

Mineral Oil:

S.U.V. at 210° F.	150
Gravity ° A.P.I.	19.7

"All Oil" Chassis Grease

An analysis of this grease showed the following results:

Physical Tests:

Color	Dark green
Penetration (A.S.T.M.) core method (unworked at 77° F.)	350
Dropping point ° C.	103
Odor	Aluminum stearate

Composition (per cent by weight):

Soap	13.60
Mineral oil	85.80
Free fat (as olein)	0.46
Moisture	0.40
Gain in analysis	0.26
Total	100.00

Mineral Oil:

Color	Greenish black
Gravity ° A.P.I.	19.8
S.U.V. at 210° F.	163
Flash (o.c. ° F.)	525
Fire (o.c. ° F.)	620

Ash, total per cent on original 1.21

Aluminum oxide (Al_2O_3)	95.30
Undetermined	4.70
Total	100.00

Fatty Acids from Soap:

Iodine value (Hanus)	11.33
Acid value (mg. KOH)	203.50
Dropping point ° C.	55
Color	Light brown
Probable composition	Stearic acid

GP Chassis Grease 3-A (1934)

An analysis of this grease showed the following results:

Physical Properties:

Penetration at 77° F.	
Worked	288
Unworked	182
Dropping point ° C.	118

Composition (per cent by weight):

Alkali	Aluminum soap
Water	0.2
Filler	trace
Soap	13.5
Oil	86.5

Ash:	
Percent	1.52
Analysis	
Al_2O_3	69.5
$Al_2(SO_4)_3$	14.9
CaO	1.0
Mineral Oil:	
Viscosity at 100° F. Furol	418
Viscosity at 210° F. Sayb. Uni.	195
Gravity, ° A.P.I.	23.5

Roller and High Pressure Grease (1935)

An analysis of this grease showed the following results:

MacMichael viscosity at 80° F. using 26 wire	49
Moisture, per cent	0.20
Filler, per cent	0.09
Soap, calculated as aluminum stearate	3.95
Mineral oil	93.10
Undetermined	2.66
Total	100.0
Viscosity, Sayb. Univ. at 210 of oil extracted	65
Ash, per cent	0.50
Ash Analysis:	
Aluminum as Al_2O_3	86.2
Lead as PbO	14.5
Gain in analysis	0.7
Total	100.0

Low Cold Test Aluminum Stearate Base Grease

Bennett and Story [34] have assigned to the Mid-Continent Petroleum Company their patent for a grease composed of the following ingredients:

	Per Cent by Weight
100 to 1000 S.U.V. at 100° F. mineral oil	85 to 75
Low boiling point hydrocarbon oil	5 to 15
Aluminum stearate	10 max.

These inventors have also proposed [35] the use of 0.05 to 0.30 per cent of aluminum stearate to reduce the pour point of waxy motor oils to less than 20° F.

Aluminum Stearate-Naphthene Base Oil Pressure Gun Grease

Southard [36] has patented a lubricating grease consisting of lubricating oil having a flash point within the range 300 to 650° F., and having 33 per cent of unsaturated and aromatic hydrocarbon material, compounded with from 5 to 20 per cent of commercial aluminum stearate.

[34] U. S. Patent 1,942,636 (Jan. 9, 1934).
[35] U. S. Patent 1,806,735.
[36] U. S. Patent 1,804,124 (May 5, 1931).

Aluminum Stearate-Lead Oleate Base Greases

Horwitz [37] has developed a lubricating grease consisting of the following substances:

	Parts by Weight
Lead oleate	5
Castor bean oil	15
Aluminum stearate	75

Aluminum Stearate as a Means of Suspending Graphite in Oils

As in the case of aluminum oleate, the stearate is frequently used as a means for preventing the precipitation of graphite from lubricating oils. The following are typical formulae which have been employed:

	Per Cent by Weight Aluminum Stearate	Per Cent by Weight Graphite	Per Cent by Weight Mineral Oil	S.U.V. of Oil at 100° F.
Light	3½	4	92½	200
Medium	3	8	89	300
Heavy	3	10	87	500

DeHart [38] has patented a process for producing a lubricant by mixing together ingredients consisting of Jefferisite, aluminum stearate and a hydrocarbon lubricating oil; then heating the mixture to near (but below) the flash point of the oil; and then permitting the compound to slowly cool. In carrying out the process, a portion of Jefferisite and, preferably, an equal portion of aluminum stearate are mixed together in their powdered form and later thoroughly mixed with lubricating oil in the cold. Then, the oily mixture is slowly heated to nearly its flash point. This was considered necessary to bring the aluminum stearate to near its melting point, where it nearly all passes into solution in the oil. The quantities recommended by DeHart were about 2 ounces of the 50 per cent mixture of Jefferisite and aluminum stearate with each gallon of oil. Oils having S.U.V. at 210° F. of 140, 155, 190 and 200 and flash points of 550, 540, 595 and 550° F. respectively were considered satisfactory by the inventor.

Aluminum Stearate Base Motor Oils

Bennett and Story [39] have investigated the possibilities for utilizing aluminum stearate in low cold test motor oils. In diluted oils they found that the presence of the aluminum stearate helped the oil to retain its lubricating value. A diluted oil containing soap, they believed, had better film forming capacity, superior adhesion to bearings, tended to decrease oil consumption, and decrease the coefficient of friction. They considered that the soap in a colloidal state caused the diluted lubricant to adhere better as a film to metal surfaces. They proposed the use of a mineral oil having a viscosity of 100 to 1000 S.U.V. at 100° F. and containing 0.1 to 10 per cent of soap, such as aluminum stearate, and 5 to 15 per cent naphtha, which

[37] U. S. Patent 1,882,664 (Oct. 18, 1932).
[38] U. S. Patent 1,709,311 (Apr. 16, 1929).
[39] U. S. Patent 1,942,636 (Jan. 9, 1934).

was a low boiling point hydrocarbon oil (boiling between 200 and 500° F.). A typical formula for winter use was:

	Per Cent by Weight
200 S.U.V. at 100° F. pale oil	89
Aluminum stearate	1
300 to 500° F. B. P. naphtha	10

Bennett, Story and Beckley [40] also developed a motor oil of improved oiliness consisting of:

	Per Cent by Weight
Neatsfoot oil	4.0
Aluminum stearate	.1
Lubricating oil	95.9

Aluminum stearate has been proposed as an ingredient of motor oils for increasing viscosity, and improving the temperature-viscosity characteristics, and possibly for improving oiliness and freedom from ring-sticking.

Aluminum Soap Motor Oil:

	Per Cent by Weight
Aluminum stearate	1.5
S.A.E. 20, 30, or 40 motor oil	98.5

Aluminum Stearate Base Gasoline Proof Grease

Yonge [41] has obtained greases resistant to water, gasoline, and kerosene by blending castor oil, aluminum ricinoleate and an aluminum soap of another fatty acid. For example, 60 pounds of castor oil, 20 pounds of aluminum ricinoleate and 20 pounds of aluminum stearate are stirred together at 150° C. The ricinoleate is obtained by boiling castor oil with a slight excess of strong caustic potash, or other alkali solution, preferably neutralizing the soap with hydrochloric or other acid, and then adding it to hot saturated potash-soda or other alum.

Aluminum Stearate Base Mine Car Greases

Winter Grade Mine Car Grease:

	Per Cent by Weight
Aluminum stearate	5
70 S.U.V. at 100° F. naphthene red oil (maximum pour point 5° F.)	95

Medium Grade Mine Car Grease:

Formula

	Per Cent by Weight
Aluminum stearate	8
70 S.U.V. at 100° F. naphthene red oil	92

[40] U. S. Patent 1,806,734 (May 26, 1931).
[41] British Patent 376,310 (Apr., 1931).

Summer Grade Mine Car Grease:

Formula

	Per Cent by Weight
Aluminum stearate	12
70 S.U.V. at 100° F. naphthene red oil (maximum pour point 35° F.)	88

Aluminum Stearate Gear Greases

On account of their tendency to emulsify and froth aluminum stearate greases for automotive gears have not become very popular. Where the proper selection of lubricating oils and development of manufacturing processes have overcome the difficulties due to excessive frothing, the additional costs appear to retard their rapid acceptance by the trade.

Aluminum Base Light Gear Grease:

	Per Cent by Weight
Aluminum stearate	5
600 S.U.V. at 100° F. naphthenic red oil	95

Heavy Aluminum Base Gear Grease:

	Per Cent by Weight
Aluminum stearate	9
200 S.U.V. at 210° F. naphthene S. R. stock	91

Locomotive Gun Greases

Within the past five years the use of portable high pressure greasing systems have been adopted for applying lubricating greases through the usual types of fittings to the valve gear and many other bearing surfaces found on locomotives. Sodium and calcium greases made with viscous oils have been found satisfactory for this service, but some railroads are definitely in favor of grease made with an aluminum stearate base. Whether or not sufficient field service tests have been made to prove the necessity for using the stearate greases is questionable. It appears likely, however, since many of the locomotive parts are subjected to high temperatures due to radiated and conducted heat from the boiler, that the stearate greases, which become more adhesive with moderate increases in temperature, are more serviceable in view of their better retention ability.

Locomotive Gun Greases

The following formulae cover yearly requirements:

	Summer Heavy Per Cent by Weight	Summer Per Cent by Weight	Winter Per Cent by Weight
Aluminum stearate	14.5	13.0	8.15
80 S.U.V. at 210° F. naphthenic red oil	85.5	87.0	....
500 S.U.V. at 100° F. naphthenic red oil	....	....	91.85
Tests			
A.S.T.M. worked penetration	235–265	265–300	350–390
Ubbelohde dropping point ° F. min.	194	194	185

PROCEDURE:

Batches are based on the use of 1000 pounds of technical grade aluminum stearate. Measure into a steam jacketed grease mixer, 200 gallons of the desired red oil, depending on the grade to be made.

Dump into the kettle through the loading chute, twenty 50-pound packages of aluminum stearate (1000 pounds).

Start the agitating paddles and stir for one-half hour. If at the end of the period the stearate has not been thoroughly dispersed to form a smooth homogeneous paste, additional oil should be run in, and stirring continued.

When the stearate is well dispersed, additional oil is allowed to run in while stirring, until the total required amount of oil has been added. At the same time full steam pressure (100 pounds per square inch) is turned on in the kettle jacket.

Heating and stirring are continued for 4 to 8 hours or until a temperature of 260 to 270° F. has been attained.

At this temperature the steam is turned off, paddles stopped, and one chill pan of grease drawn off to cool.

After the grease in this pan has cooled to room temperature a sample is tested for worked penetration and must meet the specifications given above.

On the second day, and after the sample has been approved, the batch is again heated to 260 to 270° F.

At this temperature and while stirring add 0.02 to 0.05 per cent by weight of water, depending on the period and temperatures for which the grease batch has been heated.

Pump the grease at once through a 20-mesh screen to the chill pans, which should be filled to a level of about 3½ inches.

Allow the grease to cool to room temperature without disturbing it. This will require about 12 hours.

Samples from the chill pans are again tested for worked penetration.

If the samples are approved by the laboratory, the grease is transferred by hand with shovels from the chill pans or trays to 55-gallon drums, making certain to discard any crusts, or brittle grease which may have formed at the sides of the pans or on the surface of the grease, due to rapid chilling at those points.

Refrigerator Car Door Grease

Railways, and in particular fruit express companies, have found it very desirable to make use of a soft, white aluminum stearate base grease. It seals the doors of their cars and prevents entry of rain water or warm air; which, of course, increase ice consumption. It also prolongs the life of the canvas seals which are attached to the edges of the doors. A low pour point is desirable (50 to 125° F.) as the grease is frequently applied by means of a brush and a thick grease would involve difficulties in application. Also, at very low temperatures, a thick grease might stick the doors and impair the resiliency of the canvas seals.

UN Refrigerator Car Door Grease (1931)

The analysis of a satisfactory Refrigerator Car Door Grease is given below:

Appearance	Nearly white
Consistency	Liquid jelly
Ash, per cent by weight	0.48
(Ash consisted principally of alumina)	

Analysis of Oil Separated from Grease

Color, N.P.A.	1+
Flash, ° F.	290
S.U.V. at 100° F.	60
Pour point, ° F.	—0

Formulae, Tests, and Estimated Cost of Five Refrigerator Car Door Greases

GREASES A. AND B.:

Composition (per cent by weight):	A	B
Aluminum stearate	4.0	4.0
Union crystal oil medium	96.0	.....
Union white neutral oil	.....	96.0
Tests on Greases:		
MacMichael viscosity		
No. 26 wire at 77° F.	120	110
Pour point, ° F.	120	125
Tests on Oils Used in Greases:	Crystal oil	White neutral oil
Gravity, ° A.P.I.	29.7	30.7
Color (Saybolt)	30+	20
S.U.V. at 100° F.	104	65
Pour point, ° F.	−0	−0
Flash, Cleveland ° C.	305	285
Unsulfonated residue	100.0	97.5

	Cents	Cents
Costs, Grease "A":		
4.0 pounds Aluminum stearate at	28.606	114.424
96 pounds Crystal oil medium, 13.14 gallons at	39½	519.030
100 pounds of grease		633.454
1 pound of grease		6.334c
Compounding 1 pound of grease		0.500
1 pound of grease ex chill pan		6.834c
or say		6.8c per pound
Costs, Grease "B":		
4 pounds of Aluminum stearate at	28.606	114.424
96 pounds of White neutral oil, 13.2 gallons at	29	382.800
100 pounds of grease		497.224
1 pound of grease		4.972
Compounding		0.500
1 pound of grease ex chill pan		5.472
or say		5.5c per pound

Grease "C":	
Composition (per cent by weight):	
No. 1 White oil	96.0
Aluminum stearate	4.0
Tests on Grease:	
MacMichael viscosity	
No. 30 wire at 76° F.	....
No. 26 wire at 77° F.	66
Pour point, ° F.	115
Tests on No. 1 White Oil:	
Gravity, ° A.P.I.	31.4
Viscosity, Sayb. at 100° F.	72
Color	30+
Flash, Cleveland, o.c. ° F.	290
Pour point, ° F.	−0
Unsulfonated residue	99.8

Costs, Grease "C":

	Cents	Cents
4 pounds of Aluminum stearate at	28.606	114.424
96 pounds of No. 1 White oil, 13.28 gallons at	34.5	458.160
100 pounds of grease		572.584c
1 pound of grease		5.725
Compounding		.500
1 pound of grease ex chill pan		6.225
or say		6.25c per pound

Refrigerator Car Door Grease D

Composition:

97.0 per cent Blend of 54 per cent Spray Oil No. 0 and 46 per cent Spray Oil No. 2
3.0 per cent Aluminum stearate

Tests:

MacMichael viscosity 76° F. No. 30 wire	231
Pour point ° F.	65

Tests on Spray Oil Blend Used:

Gravity ° A.P.I.	28.1
Color N.P.A.	1+
Viscosity, Sayb. at 100° F.	75

Refrigerator Car Door Grease E

Composition:

98.0 per cent Blend Spray Oils No. 0 and No. 2 to 75 Viscosity at 100° F.*
2.0 per cent Aluminum stearate

100.0 per cent

MacMichael viscosity at 79° F. No. 30 wire 205

54 per cent Spray oil No. 0
46 per cent Spray oil No. 2

Standardized Refrigerator Car Door Grease

Formula

Technical grade aluminum stearate	65 pounds
No. 1 white oil	129 gallons

Procedure:

The No. 1 white oil should comply with the following requirements:

S.U.V. at 100° F.	65 to 75
Saybolt color	25 min.

The white oil is measured into a suitable steam jacketed grease kettle.

The stearate is then weighed and dumped into the kettle containing the white oil.

The agitating paddles are then started and stirring is continued for two hours or until the stearate is thoroughly dispersed in the white oil.

Steam at 90 to 100 pounds per square inch is then admitted to the jacket and the batch heated as rapidly as possible to 250° F.

A cooling tray is then filled to a depth of a 4 inches with the melted grease, and permitted to stand without disturbance overnight. A sample is then taken for the approval of the laboratory.

If the test sample is found satisfactory the grease is again heated to 250° F. and from 0.2 to 0.5 pounds of water added to the batch.

After stirring for ten minutes at this temperature the grease is drawn through a 60-mesh screen to the chill pans.

After cooling for 18 hours a sample of the grease is tested and if it meets all requirements is transferred to shipping containers.

PROPERTIES:

This grease is intended primarily for waterproofing the canvas sealing pads in use on refrigerator car doors. It is semi-fluid, and white-translucent in appearance.

SPECIFICATIONS:

MacMichael viscosity (No. 30 wire, 77°., 60 R.P.M. 1 cm. bob, after 5 minutes)	250–325
Pour point ° F.	80–120

Must conform in color to standard sample.

Rocker Arm Lubricants

Due to the fact that systems are being perfected for the satisfactory lubrication of rocker arms found on radial type aviation motors, by the direct application of suitable oils, it is possible that now less attention need be paid to this phase of grease lubrication than in the past. Probably, the best excuse for including a discussion of this subject in the chapter on aluminum stearate greases, is that the U. S. Navy and Army have been strongly in favor of greases of this type for use on rocker arms. Recent tests, as well as service data obtained in the past, do not indicate that an aluminum base grease is necessarily superior to some made with, say, a sodium soap base. In 1929, the Wright Aeronautical Corp. had approved three aluminum base greases and two sodium soap base greases. By 1931, the Continental Aircraft Engine Company had approved four aluminum greases and three sodium greases. The Pratt and Whitney Aircraft Company, in their Service Bulletin, Serial No. 22, of 1929, recommended a commercial grease made with an aluminum base or an equivalent product.

Zublin [42] has carried out extensive service tests on rocker arm lubricants with a laboratory tester which he developed. As a result of his work he concluded that no relation was evident between the physical and chemical properties of the grease and the service life of the lubricant. Different aluminum base greases behaved differently and it was not possible to determine what properties made certain greases last longer than others. In any case, it was not demonstrated that aluminum base greases were definitely superior to other types.

Zublin reported in brief the following results:

[42] *Ind. Eng. Chem.*, **23**, 1434 (1931).

Soap Base	Percent Soap	S.U.V. at 210° F. of Mineral Oil and Type	Time in Rocker Arm Until Dry (Test at 300° F.) Hours	Remarks on Field Tests
Sodium	19	200 S.R.	50	Good on push rods Too stiff for rockers
Sodium	10	150 S.R.	45	Too stiff for rockers
Aluminum	10	200 600 S.R.	28	Satisfactory in dry climate
Aluminum	10	200 600 S.R.	27	Same as above
Aluminum	10	200 600 S.R.	26	Same as above
Na and Al	15	200 S.R.	21	.....
(Hard Petrolatum)	50	150 S.R.	10	.....

U. S. Army and Navy Specifications for Rocker Arm Lubricants

In 1929 the following requirements were published:

NAVY STANDARD FOR ROCKER ARM LUBRICANT

Specifications for lubricant for aviation engine rocker arms have been issued by the Bureau of Aeronautics of the Navy Department under the designation E-100. Arrangements are being made for the testing (at the expense of seller) of specimens of this lubricant at the Engineering Experiment Station, Annapolis, Md., looking toward the institution of an acceptable list of vendors.

The new specifications are as follows:—

GENERAL SPECIFICATIONS

1. General specifications for inspection of material together with appendix VI (lubricants and liquid fuel), issued by the Navy Department, in effect at date of opening of bids, shall form part of these specifications.

GRADES

2. Rocker arm lubricant shall be furnished in but one grade.

MATERIAL AND WORKMANSHIP

3. Rocker arm lubricant shall be a smooth, homogeneous mixture of neutral mineral oil and pure, odorless aluminum soap, either aluminum stearate or palmitate.

GENERAL REQUIREMENTS

4. Lubricant shall contain no fillers, such as rosin, rosin oil, talc, wax, powdered mica, sulphur, clay, asbestos, or other undesirable or deleterious impurities.

5. It shall possess only a slight odor of mineral oil; any other distinct odor shall be sufficient cause for rejection.

6. It shall contain not more than 0.10 per cent of insoluble matter.

7. The lubricating oil used in the manufacture of the lubricant shall be a well-refined neutral mineral oil of the following physical properties:

(a) The viscosity, Saybolt, Universal, at 130° F., shall not be less than 125 seconds.

(b) The flash and fire points, open cups, shall be not less than 340° F. and 370° F., respectively.

8. Lubricant shall show no green color in the material on the copper or a brown stain remaining on the copper after washing with benzene (C_6H_6) which passes the corrosion test by method No. 36.

9. Rocker arm lubricant shall conform to the following requirements:—

	Maximum percent
Ash, as sulphates	4.50
Moisture	0.5
Mineral oil content	85.0

Penetration (A.S.T.M., worked), 300-360.

Navy Tentative Specifications, Grease, Aluminum Soap—for Aircraft Use. No. M-304, March 17, 1936 (Superseding E-100 of Nov. 25, 1929)

A. APPLICABLE SPECIFICATIONS

A–1. The following listed specifications, of the issue in effect on the date of invitation for bids, form a part of this specification and bidders and contractors should provide themselves with the necessary copies.

(a) Navy Department General Specifications for Inspection of Material.
(b) Federal Specification VV-L-791, Lubricants and Liquid Fuels; General Specifications (Methods for Sampling and Testing).

B. GRADES

B–1. Aluminum-Soap Grease shall be furnished in the following grades, as specified:

Grade A, Soft
Grade B, Medium

C. MATERIAL AND WORKMANSHIP

C–1. Aluminum soap grease shall be a smooth, homogeneous mixture of mineral oil and pure, odorless, aluminum soap.

D. GENERAL REQUIREMENTS

D–1. The grease shall contain no fillers such as rosin, rosin oils, talc, wax, powdered mica, sulfur, clay, asbestos, graphite, or other undesirable or deleterious impurities.

D–2. It shall possess only a slight odor of mineral oil; any odor of perfume or other distinct odor shall be sufficient cause for rejection.

D–3. It shall contain not more than 0.10 per cent of material insoluble in A.S.T.M. precipitation naphtha and hydrochloric acid.

D–4. It shall contain not more than 0.30 per cent of water.

D–5. It shall show no green color in the material on the copper nor a brown stain remaining on the copper after washing with benzene (C_6H_6) which passes the corrosion test by Method 530.1.

D–6. The lubricating oil used in the manufacture of the grease shall be a well-refined mineral oil having the following properties:

D–6a. The Viscosity Index (Dean and Davis) shall be at least 95.

D–6b. The pour point shall not be higher than 30 degrees Fahrenheit.

D–6c. The color shall not be darker than Number 8.

E. DETAIL REQUIREMENTS

E–1. Aluminum-soap grease shall conform to the following specifications:

	Grade A, Soft	Grade B, Medium
Oil:		
Per cent	92, minimum	86, minimum
S.U.V., 210° F.	140–180	180–220
Flash point, ° F.	450, minimum	500, minimum
Fire point, ° F.	500, minimum	550, minimum
Ash, as Al_2O_3, per cent	1.5, maximum	2.0, maximum
Penetration	400, minimum	325–375
Melting point, ° F.	200, minimum	250, minimum

F. METHODS OF SAMPLING, INSPECTION AND TEST

F–1. INSPECTION TESTS

F–1. (a) One sample of at least 5 pounds shall be taken at random from each lot of each grade of grease under inspection. When delivery is made in containers holding 10 pounds or less, the sample shall be taken in the original container without

disturbing the contents. When the shipping container holds a greater quantity than 10 pounds, sample as follows: punch several holes in the bottom of a 5-pound grease tin, or other tin with a sharp edge, and force it, top down, into the grease until it is full; cut off the grease at the top of the can with a curved knife; remove and cut off level with the top of the can with a straight knife.

F–1. (b) Samples shall be forwarded to the Engineering Experiment Station, Annapolis, Maryland, for inspection tests.

F–2. METHODS OF TESTS

All tests shall be made by the following methods, as described in Federal Specification VV-L-791, referred to in Section A:

	Method Fed. Spec. Bd. No.
(1) Water	300.13
(2) Corrosion	530.4
(3) Insolubles	541.2
(4) Mineral oil content	541.2
(5) Flash and fire points	110.33
(6) Viscosity	30.43
(7) Color	10.22
(8) Ash	341.2
(9) Penetration	31.11
(10) Melting point (see note)	140.1

NOTE:—Melting point shall be determined according to Method 140.1 except that (1) glycerin shall be substituted for water in the heating bath, (2) worked grease shall be applied to the thermometer bulb to a thickness of 0.05 inch instead of being melted and then congealed on the cold bulb, and (3) the top and bottom of the thermometer bulb each shall be free of grease for approximately 0.05 inch to avoid any insulation of the bulb by the layer of grease.

F–3. TYPE APPROVAL TESTS

F–3. (a) Type approval tests are for the purpose of establishing the ability of a manufacturer to supply acceptable material in accordance with the specifications. The right is reserved to reject any bids on aluminum-soap greases which have not been subjected to the required Type Approval Tests and found satisfactory. The attention of manufacturers is called to this requirement, and they are urged to forward samples of the material which they propose to offer to the Navy in the future, in order that tests may be made. These tests will be conducted at the expense of the manufacturers. It is to be understood that the manufacturer shall pay all transportation charges to and from the point where tests are made. In the case of the failure of the sample or samples submitted to prove satisfactory, consideration will be given to the requests of manufacturers for additional tests only after it has been clearly shown that changes have been made in the product with reference to the method of manufacture, etc., which the Bureau concerned considers sufficient to warrant conducting such additional tests. Attention is invited to Section F–3 (b).

(Sections F-3b, F-3c, and G omitted).

H. NOTES

H–1. Requests and requisitions should specify the grade desired.

H–2. Aluminum-soap grease is intended for use in high temperature, high-speed locations where oil is inapplicable, such as in rocker-arm bearings, and controllable-pitch propellers, and other uses, in accordance with service instructions issued by the Bureau of Aeronautics.

H–3. Copies of Bureau of Aeronautics, Navy Department specifications, and other Navy Department specifications forming a part thereof, may be obtained upon application to the Bureau of Supplies and Accounts, Navy Department, Washington, D. C.

H–4. Copies of Federal Specification VV-L-791, Lubricants and Liquid Fuels; General Specifications (Methods for Sampling and Testing) may be obtained from the Superintendents of Documents, Washington, D. C.—price 15 cents each.

U. S. Army Specification No. Y-3558, Dec. 20, 1930

GREASE, LUBRICATING, ROCKER ARM
(For Aircraft Engines)

I. GENERAL SPECIFICATIONS

1. The current issues of the following specifications in effect on date of issuance of proposals, form part of this specification:

100–2 —Standard Specifications for Marking Shipments.
2–22—Boxes for Oil and Grease for Export Shipment.

The current issue of the Bureau of Mines Technical Paper No. 323—"Method of Testing Lubricants and Liquid Fuels" also forms a part of this specification.

II. GRADES

1. This specification covers the grade of grease used by the Air Corps, for the lubrication of rocker arms and rocker arm bearings.

III. MATERIAL AND WORKMANSHIP

1. The grease shall be such as will meet the requirements specified herein.

IV. GENERAL REQUIREMENTS

1. There are no general requirements applicable to this specification.

V. DETAIL REQUIREMENTS

1. Rocker arm grease shall be a smooth, homogeneous mixture of mineral oil and pure odorless aluminum soap.

2. The grease shall contain no fillers such as rosin, rosin oils, talc, wax, powdered mica, sulphur, clay, asbestos, or other undesirable or deleterious impurities. It shall possess only a slight odor of mineral oil. The grease may be rejected if it has the odor of any objectionable material.

VI. METHOD OF INSPECTION AND TESTS

1. *Sampling.*—Samples shall be taken according to the procedure described in Part 3, Bureau of Mines Technical Paper No. 323.

2. *Water—Method 300.12.*—The water content shall not exceed 0.5 per cent.

3. *Penetration—Method 31.1.*—The penetration (worked) shall be 300–400.

4. *Corrosion—Method 530.4.*—Grease under this specification shall show no green color in the material on the copper or a brown stain remaining on the copper after washing with benezene (C_6H_6) which passes the corrosion test (Method 530.22).

5. *Analysis of Grease—Method 541.2:*
   a. *Fillers.*—Grease under this specification shall not contain more than 0.10 per cent of insoluble matter.
   b. *Mineral Oil Content.*—The mineral oil content shall not be less than 88 per cent.
   c. *Properties of the Mineral Oil Constituent.*—After the mineral oil has been separated, the oil shall conform to the following requirements: Viscosity, Method 30.41—The viscosity at 210 degrees Fahrenheit (98.89 degrees Centigrade) shall be not less than 180 seconds nor more than 220 seconds, S.U.V. The Pour Point shall not be above 25 degrees Fahrenheit (—3.89 degrees Centigrade).

6. All tests shall be made according to the methods for testing contained in Part 2 of the latest issue of the Bureau of Mines Technical paper No. 323.

Laboratory Tests on Rocker Arm Lubricants

The author has developed an accelerated life test for rocker arm lubricants which is described in the following sections. The apparatus was

made from parts from both Pratt and Whitney "Wasp" engines and Wright "Whirlwind J-6" engines. No commercial lubricant tested had a life of more than six hours in the former tester or more than four hours in the latter.

APPARATUS:

The actual cylinders, valves, and valve actuating mechanism of the Wright J-6 and the Pratt & Whitney Wasp engines were used. These cylinders were mounted by means of built-up (welded) steel brackets at the side of an automobile crankcase in such a manner that the pushrods may be operated in the normal fashion by the cam followers. The crankcase-and-camshaft assembly chosen was that of a 1927 model Buick Six, as this assembly was separable from the cylinder block and embodied an oil pump driven from the camshaft. The crankshaft was left in place but disconnected by removing the large timing gear. The lubricating-oil piping was rearranged so as to flood the camshaft and its bearings with oil. The cam-followers, which are of the roller type, were modified slightly at the upper end to take the ball-ended pushrods of the airplane engine mechanism. The camshaft at the exposed timing gear end was directly coupled through a leather disc coupling to a ½ H.P. induction motor of 1200 r.p.m. Those valve followers not used in the test apparatus were removed, the holes being stoppered, and the top surface of the crankcase was finally made oil-tight by a cover plate. In this way all apparatus except the actual rocker arm mechanism was made self-contained and self-oiling, requiring no attention throughout the test period.

The cylinders were housed in sheet-metal jackets to protect them from all air currents, which, because of the large and effective radiating area of the cylinders, would disturb the desired temperature equilibrium.

Heat was applied directly to the inside of the cylinder barrels by open Bunsen flames, this being considered the best simulation of the actual condition of operation. Temperature (or flame) control was wholly manual as it was considered that the test did not justify the use of costly thermostatic or gas pressure control apparatus. Due to widely fluctuating gas pressure, the temperature at the rocker arm pins was held to the desired limit of $\pm 5°$ F. with some difficulty. Iron-constantan thermocouples and a potentiometer were used. A third Bunsen burner arranged as a bleed, was of some assistance in keeping the flames steady.

METHOD OF TEST:

Before each test run the rocker arm mechanism was disassembled, inspected and washed. Special care was taken to insure that the drilled grease passages of the Wasp engine were open. Assembled and adjusted for valve clearance, as nearly as possible 10 grams of the desired grease were injected through the pressure lubrication fitting. Stiff greases were warmed. The roller and ball ends of the Wright rocker arms were coated with the grease. The temperature was then brought up to the desired value in not over 30 minutes, and the apparatus started. At hourly intervals the

covers were removed and both ends of each rocker arm inspected. It was found that incipient dryness could be detected visually as well as by working the parts together with the fingers, the latter causing a distinct squeak. Discoloration, polishing, abrasion, or actual oxidation of the parts, notably the ball and cup of the Wright assembly, occurred during the hour following incipient dryness. Time of failure was estimated as lying between these two events, according to the observed condition of the parts. It is believed that any test can be reproduced to ½ hour, several check runs supporting this assertion.

It was hoped that failure would be indicated by squeaks as reported by Zublin, but the din caused by the valves seating at 1200 r.p.m. in open-bottomed cylinders prevailed above all other sounds in the vicinity.

RESULTS:

The life of the lubricants under test is condensed in the following table together with certain physical properties of the greases. Further comment on the greases follows in the order of the tabulation:

Summary of Rocker Arm Lubricant Data

		Viscosity, Seconds				Life, Hours		
	Name	Furol 300° F.	Furol 400° F.	MacMichael	Pen.	Wasp 350° F.	Wright J-6 375° F.	Drop Point
1.	S.A.E. 160 A	....	10.9	135 at 80° F.	...	2½	1	59
2.	S.A.E. 160 B	17	11.5	105 at 80° F.	...	3.0	1	45
3.	Dark Fiber 4 A	....	11.0	88-114 at 95° F.	330–370	6–	3–	121
4.	Dark Fiber 4 B	28.5	11.1	No. 24 wire	330–370	6	2+	90
5.	Dark Fiber 5 A	197	10.6	.....	295	4–	40	165
6.	Dark Fiber 5 B	Won't run	14.3	.....	277	3	2½	104
7.	Rocker 15	Very high	84	.....	370–410	2½	3	...
8.	St. 4	43	11	.....	...	4½	Breakdown	...
9.	M.F. 2	23.4	10.6	.....	...	3	3	...
10.	M.F. 3	540	10.3	.....	...	4+	3	...
11.	Experiment 1	34	11.2	133 at 95° F. No. 24 wire	350	4½	1½	81
12.	Experiment 2	34	....	116 at 95° F. No. 24 wire	380	4½	1½	65
13.	Experiment 3	83	....	150 at 95° F. No. 24 wire	320	4	3	97
14.	Experiment 4	....	....	.....	...	5	3	173
15.	Experiment 5	....	....	.....	294	4	6+	145
16.	Experiment 6	....	....	.....	345	3	3	119
17.	Experiment 6A	....	....	.....	390	3½	...	55
18.	Experiment 7	....	....	.....	283	...	2–	149
19.	Experiment 8	....	....	.....	283	...	2½–	...
20.	Experiment 9	....	....	.....	247	...	3	...

1, 2 S.A.E. Lubricants 160, A and B, were samples chosen for high and low dropping points respectively. Both greases are too thin at high temperatures, the short life resulting from this characteristic.

3, 4 Dark Fiber 4, Samples A and B, chosen for high and low dropping points respectively. This grease proved to be the most reliable lubricant for the Wasp rocker arms, but lacks adhesiveness at high temperatures, failing therefore to give good service in the Wright J-6.

5, 6 Dark Fiber 5. Samples A and B, selected for high and low dropping points respectively. Sample B showed signs of separation and poor texture; the dropping point was 61° below that of Sample A. This grease must be warmed before charging due to its heavy body, and therefore might be rejected as a rocker arm lubricant by operators. The test indicates that properly selected batches should serve very satisfactory in Wright J-6 engines where adhesiveness at high temperature is the prime requirement.

7. Rocker Arm Lubricant 15 is an aluminum stearate base grease prepared to meet Air Corps specification Y-3558. This type of lubricant is used exclusively by the Army and Navy with apparent success. The results of this test indicate

that this grease is not as serviceable under extreme conditions as Dark Fiber 4. There have been operator's statements to the same effect.

8. Stanavo Rocker Arm Lubricant 4 is a grease of mixed soda and aluminum base, of attractive appearance. At the operating temperature of 350° F. it fed too freely from the Wasp rocker arm. It was not run on the Wright J-6 but is obviously too light to give good service in that application.
9. M. F. Grease No. 2 is a dark soda base fiber grease similar to Dark Fiber 4, but of indifferent performance, due probably to excessive fluidity at operating temperature.
10. M. F. Grease No. 3 is similar to Dark Fiber 5. Gummy residues formed in the grease passages of the Wasp rocker arm.
11. Experimental Grease No. 1 was made to the following formula:

Tallow	4 liters
45° Bé. caustic soda	880 cc.
650 Pennsylvania steam refined stock	19 liters

It is a dark short fiber grease, which does not adhere well at high temperatures. Signs of oil separation are apparent.

12. Experimental Grease No. 2 consists of 85 per cent Experimental Grease No. 1 and 15 per cent Floyd's Base. The performance was exactly the same as that of Experimental Grease No. 1.
13. Experimental Grease No. 3 consists of Experimental Grease No. 1 with 0.3 per cent latex. An unsightly gummy residue was formed. The addition of latex apparently increased the life of the grease in the Wright J-6 application over that of Experimental Grease No. 1.
14. Experimental Grease No. 4 is a mixture of 1500 grams Dark Fiber 4, 200 grams castor oil and 20 grams latex. The performance of this grease was inferior to Dark Fiber 4 in the Wasp engine, but a little better than Dark Fiber 4 in the Wright J-6.
15. Experimental Grease No. 5 consisted of the following:

	cc.
Tallow	200
45° Bé. caustic soda	44
1000 Red oil	100
650 Pennsylvania steam refined stock	1000
45 Penetration steam refined asphalt	200

This grease proved to be the best of the group tested in the J-6 engine. The asphalt rapidly oxidized to a glossy black film on the working surfaces of the push-rod, ball and cup, this film remaining intact long after the parts were apparently dry. Failure occurred when this film finally broke down. The tendency to oxidize and gum eliminates this grease as a lubricant for Pratt & Whitney rocker arms, although in this single test it gave good service.

16. Experimental Grease No. 6 contained twice as much 650 S. R. Stock and Asphalt as No. 5. It proved to be very fluid at working temperature.
17. Experimental Grease No. 6-A represents an attempt to prepare a soft grease for Pratt & Whitney engines, which would still have good endurance. The formula is:

	cc.
Tallow	200
45° Bé. caustic soda	44
1000 Red oil	100
650 Pennsylvania steam refined stock	2000

The penetration was 390; the 3½ hour endurance is above that of S.A.E. 160, and indicates that this might be worth further study.

18. Experimental Grease No. 7 was prepared for the Wright engine rocker arms. A very heavy grease, high in asphalt, the short life cannot be explained.
19. Experimental Grease No. 8 is a heavy (247 Pen.) grease of the following composition:

Tallow	200 cc.
45° Bé. caustic soda	44 "
1000 Red oil	500 "
Edeleanu lube oil extract	500 grams

In common with the preceding grease No. 7, this grease gave indifferent performance in the Wright J-6, indicating that there is perhaps a limiting body or stiffness for a grease intended for this service. In the Wasp the residue of this grease plugged the oil holes, as was expected.

20. Experimental Grease No. 9 is primarily a wheel bearing grease, containing Floyd's base and potassium dichromate. It is too heavy for the Pratt & Whitney rocker arms, but gives average service in the Wright.

Conditions of Test

To obtain a good simulation of service conditions, and to put the tests on a systematic basis, the following points were observed, and conditions studied:

Mechanical features of the valve gear in aircraft engine service.
Temperatures to which the lubricant is subjected in service.
Speeds of operation in service.
Service complaints.
Previous work on the subject.

Mechanical Features

The first point is of great importance in that it is shown that the grease is expected to accomplish the impossible in some cases due to the exigencies of design forced upon the valve gear of the two most widely used aircraft engines—the Wright and the Pratt & Whitney radial series.

Engines of these two manufacturers are universally used in this country for commercial applications where 400 H.P. per unit or over is required. This field embraces all scheduled air lines, all Navy, and the bulk of the Army Air Corps requirements. In addition, Wright engines are made in 5, 7, and 9 cylinder units of a little over 30 H.P. per cylinder, the range of engine sizes so produced being wide enough to give them perhaps first rank in popularity in the lower priced and sporting fields. The Pratt & Whitney "Wasp Junior" of 300 H.P. is also widely used.

In view of the above, the Wright and Pratt & Whitney problems were given special study and the apparatus for testing Rocker Arm Lubricants was built with the requirements of these typical engines in view. In passing, it may be said that all other valve gear layouts for radial air-cooled aircraft engines built in this country were made with an eye to the two leading designs and are subject in a greater or less degree to the same lubrication problems.

The valve gear design and lubrication problems arise from the radial arrangement of the cylinders, which places the valves in the most remote position with respect to the other working parts. To lubricate these valves with the engine oil under pressure would require extensive plumbing installations, a line to each valve, thus adding complication and chance for failure due to pipe breakage to an engine type which owes much of its success to its simplicity and reliability in these respects. Air cooling also takes toll of lubricants due to the high cylinder head (and therefore rocker arm) temperatures, and the extreme expansion of metal parts which renders the construction of an oil-tight rocker housing impracticable. Recourse is therefore had to the use of grease in the lubrication of the rocker arm bearing

and the two bearing areas at the ends of the rocker arm engaging the push-rod and the valve stem, respectively.

For both Wright and Pratt & Whitney valve actuating mechanisms, it will be noted that the rocker arm rotates on two single-row ball bearings which are subject to a large thrust component due to the angularity of the push-rod. Grease is supplied to the annular space between the bearings by suitable passages drilled in the stationary pin; the Alemite or Zerk fitting projects from the rocker arm box. The Wright bearings are provided with felt grease seals; the Pratt & Whitney uses the self-sealing type of bearing. The passages in which the thermocouples are shown were drilled especially for test purposes, and do not communicate with the grease holes.

For the Pratt & Whitney rocker arm, it will be found that holes are drilled connecting the annulus between the ball bearings with both ends of the rocker arm. The push-rod end comprises a hardened spherically-ground cup which works with the ball on the end of the push-rod. The tappet adjusting screw is contained in the opposite end of the rocker arm, and the flattened ball on the lower end of the adjusting screw makes contact with the valve stem. It is seen that the rocker arm grease passage registers with an annular groove in the tappet screw, a radial and an axial passage in the tappet screw completing the grease path from the central supply to the spherical bearing surface in the tappet screw. This passage offers high resistance to the flow of the grease, precluding the use of a product of high apparent viscosity.

The angular acceleration of the rocker arm causes the grease to travel outward, emptying the central chamber. The problem is one of metering. Thus, with a viscous grease the tappet ball will be starved due to the high resistance of the passage, while the push-rod ball will be adequately lubricated. If a thin grease is used it will be thrown to the push-rod ball in excessive amounts, soon exhausting the small available supply. These passages are easily clogged, and greases forming a gummy residue under service conditions must be avoided.

The highest temperature is encountered at the valve stem. There is a linear motion of the tappet over the valve stem, but this is comparatively small. The angular movement of the tappet ball in its socket is more important and it is in this bearing and at the push-rod ball joint that lubrication failure results in squeaks. In some cases "rocker arm birds" have been traced to dry valve stems; for this condition the rocker arm lubricant cannot be blamed.

The problem in the case of the Wright rocker arms is of a different type. A heat-resistant adhesive grease is indicated since lubrication fails when the applied film of lubricant breaks down. There is no provision for a replenishing feed. The case of the valve-end of the rocker arm is better, however, as the roller was found to be adequately lubricated in all cases after the push-rod ball film had failed. There are no small grease passages to be guarded against gum deposits, so that the consistency of the grease to be used is governed by the requirements of the ball bearings. In fact, it

would be desirable to use a light grease for the ball bearings, since they are sealed, and a special heat-resisting viscous grease for the push-rod ball and socket joint and the valve stem roller follower.

To sum up, the problem in lubricating the Pratt & Whitney rocker arm is to select a grease which will flow through the passages at the proper rate, and without clogging. The Wright rocker arms require an adhesive grease as there is no provision for replenishing the film on the push-rod ball joint.

Temperatures

The appearance of several Wright push-rod sockets obtained in the field indicates that the operating temperatures are high, as these parts are covered with red oxide and show signs of abrasion. Reports from the southern part of the state show that the push-rod ball and socket joints of the top (No. 1) cylinder of the Wright J-6 engines run dry in less than one hour in some cases.

Thermocouples were installed in the rocker arm pins of Pratt & Whitney Wasp and Wright J-5 engines, and the temperatures of these parts observed while the airplanes were engaged in maneuvers. The thermocouples were installed by removing the pressure lubrication fittings and pushing the couples to the full depth of the axial grease hole in the rocker pin. It is seen that the position is very close to that of the couples as used in the experimental work. The data observed were:

Engine	Airplane	Cowling	Service	Temperature		
				Atmos.	Oil	Rocker Pin
P. & W. Wasp	Helldiver	Curtiss anti-drag ring	Tactical, cruising throttle	50° F.	45° C.	211° F.
Wright J-5	Fledgeling	None	Tactical, mostly full throttle	50	40	125

The low observed temperatures were probably due to the following conditions:

The atmospheric temperature was low.

The airspeed of these airplanes is reasonably high, resulting in good cooling.

The Wright J-5, although having the same type of valve gear as the J-6, is known as a cooler-running engine.

The Wright J-5 was not cowled, and while the Wasp engine had a ring cowl, it was of a type that provides excellent cooling of the cylinder heads.

It is known that in summer the oil temperatures for these engines reach a value of double that observed during this test, and it is reasonable to expect somewhat greater cylinder head and rocker arm temperatures to correspond. Zublin assumed a maximum temperature of 300° F. for the atmosphere of the Wasp rocker arm box. Preliminary tests showed that the best available greases failed in less than 6 hours at 350° F. rocker pin temperature in the Wasp. On the basis of service reports which ascribed higher temperatures to the J-6, 375° F. rocker pin temperature was chosen for tests on the J-6.

SPEEDS

The Wasp and J-6 engine crankshafts rated R.P.M. vary from 1800 to 2100 according to the type of service. These engines are generally run at speeds below 1800 R.P.M., and the maximum R.P.M. in military service (such as dive-bombing) is always less than 3000. The speed of operation undoubtedly has a great influence upon the feed of grease through the Wasp rocker arm, and also is the controlling factor in the bearing loads which are due to acceleration. For test purposes a camshaft speed of 1200 R.P.M. corresponding to 2400 R.P.M. crankshaft speed was selected as representing extremely rigorous service.

SERVICE COMPLAINTS

Soda base greases such as Dark Fiber Grease No. 3, No. 4, and No. 5 have been widely used with good results. The record of the aluminum base greases such as Rocker Arm 15, made to U. S. Army spec. Y-3558 is generally poor. Complaints of "throwing-out" excessively, and of failure to lubricate the J-6 for more than a few minutes have been received.

The fact that the Army and the Navy have success with this grease is due to the careful lubrication and valve clearance check every 10 hours, and the use of very few J-6 engines. Rocker arm squeaks, however, frequently occur in Naval aircraft in spite of the frequency of the lubrication and inspection.

In formulating a test method cognizance was taken of the relative frequency of complaints from J-6 operators, and high temperatures were therefore employed in the test apparatus.

Formulae for Greases Meeting U. S. Army Specification Y-3558

	A (15)	B (15)	Specifications
Composition (per cent by weight):			
Aluminum stearate	11	11	....
E Bright stock	89	....	....
F Bright stock	....	89	
Mineral oil	....	....	88 min.
Tests:			
Percent water	0.2	0.1	0.5 max.
A.S.T.M. worked penetration	322	380	300–400
Copper corrosion 530.4	Neg.	Slt. tarnish	Neg.
Percent insoluble fillers	.023	.041	0.10 max.
Mineral oil			
S.U.V. at 210° F.	211	198	180–220
Pour point ° F.	25	10	25 max.

PROCEDURE:

The oil and aluminum stearate are mixed in a grease kettle and heated to 300° F. The batch is then cooled to 200° F. in the kettle.

The grease is then cooled slowly to the gel point which should be about 160 to 180° F.

At 160 to 180° F. the grease is screened through 20 mesh screen into cooling trays to a depth of six inches.

With a room temperature of about 90° F., nearly two days will be required to cool the grease, test it and shovel it into packages.

Analysis of Rocker Arm Lubricant No. 15 (1935)

This analysis showed the following results:

Physical Tests:

Appearance	Smooth, slightly stringy
Color	Dark green
Dropping point ° C.	109

Composition (per cent by weight):

Soap (as aluminum stearate)	9.4
Free fat	0.2
Moisture	0.1
Petroleum oil	90.1
Unaccounted for	0.2
Total	100.0

Petroleum Oil:

Gravity ° A.P.I.	23.4
Pour point ° F.	10
Color	5 + dilute
Flash, Cleveland, o.c. ° F.	550
S.U.V. at 210° F.	192

Fatty Acids from Soap:

Color	Light brown
Odor	Resembling stearic acid
Neutralization value (mg. KOH/gr.)	197
Iodine value (Hanus)	12.6
Fatty acids are very likely stearic acid	

Ash (per cent by weight) 1.07

Aluminum (as Al_2O_3)	87.2
Unaccounted for (calcium absent)	12.0
Total	100.0

Suggested Specification for Rocker Arm Lubricant No. 15

The following is suggested as a satisfactory specification for this purpose:

Worked consistency at 77° F.	Min. 340—max. 390
Mineral oil, S.U.V. at 210° F.	Min. 200—max. 210
Pour point ° F.	Max. 25
Melting point ° F., Gillette method	185
Soap content (A.S.T.M.—D-128-27)	Aluminum—8.5 per cent min. (B.W.)
Fillers	None
Ash (A.S.T.M.—D-128-27)	Per cent by weight—max. 1.2
Water	None
Corrosion (U.S.B.M. method)	530.4 (negative)
Mineral oil content, per cent by weight	88 min.

The grease shall have practically no odor.

Analysis of BT High Melting Point Sodium Base Rocker Arm Grease (1935)

This analysis showed the following results:

Physical Tests:

Appearance	Stringy
Color	Dark green
Dropping point ° C.	193

Composition (per cent by weight):

Asphaltic material (P.E. insoluble)	0.1
Soap (as sodium oleate)	14.9
Free fat	0.3
Moisture	0.1
Petroleum oil and unsaponifiable	84.1
Free alkali	trace
Unaccounted for	0.5
Total	100.0

Petroleum Oil:

Gravity ° A.P.I.	24.0
Pour point ° F.	5
Color	8+
Flash, Cleveland o.c. ° F.	545
S.U.V. at 210° F.	187

Fatty Acids from Soap:

Color	Brown
Odor	Resembling oleic and stearic acids
Neutralization value (mgs. KOH/gm.)	189
Iodine value (Hanus)	71.2
Fatty acids are probably principally Oleic with some Stearic acid.	

Ash (per cent by weight)	3.28
Sodium carbonate	76.9
Iron and aluminum (mainly iron as Fe_2O_3)	1.0
Unaccounted for	22.1
Total	100.0

Analysis of "All Oil" Aluminum Base Rocker Arm Lubricant

This analysis showed the following results:

Soap, per cent by weight	9.1
Ash, per cent by weight	1.23
Ash analysis, per cent by weight, Aluminum oxide (Al_2O_3)	90.0

Extracted Mineral Oil:

S.U.V. at 210° F.	144
Gravity ° A.P.I.	18.3
Rubber*	Negative
Dropping point ° C.	91

*Test is doubtful for samples of less than one per cent of rubber.

Analysis of ST Rocker Arm Lubricant

This analysis showed the following results:

Penetration, A.S.T.M., at 77° F., unworked	400
Penetration, A.S.T.M., at 77° F., worked	417
Ash, per cent by weight	0.963

Ash Analysis:

Aluminum as (Al_2O_3)	66.1
Sodium carbonate as (Na_2CO_3)	33.5
Undetermined	0.4
Total	100.0
Aluminum soap, (calculated from ash) per cent	7.29
Sodium soap, (calculated from ash)	1.86

Analysis of TP Aero Rocker Arm Grease

This analysis showed the following results:

Physical Tests:

Appearance	Smooth transparent semi-solid grease
Melting point ° F.	104
Odor	Not offensive
Penetration at 77° F.	360+

Composition (per cent by weight):

Soap	9.90
Moisture	0.10
Free fat	.20
Oil by difference	89.80
Total	100.00

Ash Analysis (per cent by weight): .943

Aluminum (Al_2O_3)	88.0
Sulfates	8.5
Undetermined	3.5
Total	100.0

Fatty Acids from Soap:

Acid value (mgs. KOH)	205
Iodine value	14.5

Mineral Oil:

Viscosity at 210° F. (approximate)	250
Gravity at 60° F. A.P.I.	24.5

Analysis of Dark Fiber Greases

This analysis showed the following results:

	No. 3	No. 4
MacMichael viscosity	136 at 80° F. (No. 26 wire, 60 R.P.M.)	96 at 95° F. (No. 24 wire, 60 R.P.M.)
Penetration at 77° F. (worked)	....	385
Melting point ° F.	145	149
Alkalinity, per cent NaOH	.17	.08
Ash, per cent	.82	1.80
Soap, per cent	3.6	10.6
Oil, per cent	95.8	88.4
Tests on Extracted Oil:		
S.U.V. at 210° F.	132	140

Analysis of TX-M.K. Rocker Grease

This analysis showed the following results:

Physical Tests:		
Appearance		Dark, sticky, smooth lubricant
Melting point ° C.		130
Penetration (Shell)		180
Odor		Soapy
Composition (per cent by weight):		
Soap		13.1
Moisture		1.0
Free fat		.5
Oil (by difference)		85.2
Free alkali (Na_2O)		.2
	Total	100.0
Ash (per cent by weight):		2.30
Sodium (Na_2CO_3)		94.8
Sulfates		1.1
Undetermined		4.1
	Total	100.0
Fatty Acids from Soap:		
Acid value (mgms. KOH)		204
Iodine value		52
Mineral Oil:		
S.U.V. at 210° F. (approx.)		116
Gravity at 60° F. A.P.I.		26.8

Analysis of a Sodium Soap Base Rocker Arm Grease

This analysis showed the following results:

Constituents:	
Mineral oil, per cent by weight	80.17
Soap content, per cent by weight	20.15
Water, per cent by weight	0.32
Glycerin	Trace
Free alkali, per cent by weight (NaOH)	0.18
Free fatty acids	None
Free fatty acids separated from soap, per cent by weight	15.16
Ash, per cent by weight	3.01

Melting point { Gillette ° F.	223
Melting point { Ubb. Drip Pt. ° C.	128
Melting point { Ubb. Flow Pt. ° C.	133
Worked consistency	351
American airways heat test	O.K.

Analysis of Ash:	Per cent based on Ash	Per cent based on Grease
SiO_2, per cent by weight (silica)	0.89	0.027
R_2O_3 (iron and aluminum oxides)	0.22	0.007
$CaCO_3$	1.08	0.033
Na_2SO_4	4.49	0.135
Na_2CO_3	87.29	2.627
K_2CO_3	5.71	0.172
Fe, Al, Cl	Slight trace	
Fillers and other metals	None detected	
Unaccounted for	0.32	0.009
Total	100.00	3.010

Tests on Mineral Oil:	
Gravity ° A.P.I.	23.3
Pour point ° F.	−5
Flash ° F.	470
S.U.V. at 130° F.	829
S.U.V. at 210° F.	128

Tests on Fatty Acids from Soap:	
Iodine value	25.7
Saponification value	124
Titer test ° C. (indefinite—approx.)	30.6 to 37.6
Appearance and color	Dark brown, viscous, semi-solid
Odor	Oleic and degras resemblance
Acid No.	108.4
Stearic, palmitic and resin oil	None detected
Color reaction*	Indicates degras
Unsaponifiable, per cent by weight	19.53

* Burchardt-Liebermann test using acetic anhydride and sulfuric acid.

Analysis: Aluminum Stearate Base Stainless Grease

This product has been used with success in food factories and textile plants where a white grease is desirable to prevent excessive damage to goods handled.

Physical Tests:	
Appearance	White, smooth grease
Odor	Slight; not soapy
Worked penetration, A.S.T.M. at 77° F.	344

Composition (per cent by weight):	
Soap, as aluminum stearate	10.1
Mineral oil	89.8
Unaccounted for	0.1
Total	100.0

Mineral Oil: S.U.V. at 100° F.	281
Fatty Acids from Soap: Apparently stearic acid	
Ash (per cent by weight) Apparently aluminum oxide	0.83

Aluminum Stearate Steam Cylinder Oils for Corliss Steam Engines

One of the most difficult industrial lubrication problems encountered is to properly lubricate the valves of a Corliss Valve steam engine to get the maximum reduction of friction and wear with a minimum oil consumption.

This problem is complicated because Corliss valves are heavy and essentially unbalanced; they oscillate instead of rotate so the lubricant cannot be carried to all the rubbing surfaces in the usual manner. The rubbing speeds are high and for the area covered the lubricant must be spread over great length and then travel a long distance to adequately lubricate the bearing ends. These factors are common to all four valves, but in addition, the exhaust valves, being located at the bottom of the cylinder, are subjected at times, particularly when starting the engine, to the washing action of water. Further, when an engine is operated condensing the force of the vacuum tends to remove the lubricant from the exhaust valves.

The proper solution to the problem is secured only by a careful study of many factors, the most important being:

The character of the steam
The character of the oil
The point and method of oil introduction.

Character of the Steam

Without doubt, the character of the steam has a decided effect on lubrication. Although wet steam provides easier atomization of oil than dry steam, particularly superheated steam, satisfactory atomization and distribution, and therefore lubrication, must be obtained under all conditions. If the lubricant does not meet the requirements of the particular steam conditions encountered, unsatisfactory engine performance and high friction losses will be experienced.

Character of the Oil

Properly selected petroleum stocks, compounded with the correct fixed oil, will produce a cylinder oil with the characteristics necessary to give good lubrication. Undoubtedly the most important single requirement of the lubricant is that it have the ability to replace any water present and to resist removal by water. If the cylinder oil does not have this ability the proper oil film can be neither established nor maintained, so scoring will result.

It is obvious that certain parts of the valves and valve seats can be lubricated only by the oil that is mixed with the steam. Therefore, it

is imperative that the oil atomize quickly and completely and have the proper dispersion to give the uniform coverage necessary to prevent dragging valves or metal to metal contact at certain points.

The oil also must keep to a minimum the carbon-like and gummy deposits sometimes encountered even though such deposits are principally caused by impurities in the steam. Unless such deposits are kept to a minimum, by using the proper oil, serious gumming and poorly operating valves will result.

Finally the oil must separate satisfactorily from the exhaust to prevent difficulty in process work, in heating systems, or in the boilers.

Method of Application

After securing the oil which has the proper characteristics for the steam conditions, certain precautions must be observed in introducing it. It is desirable to introduce the oil at least six feet ahead of the throttle for the best results. Since it cannot be installed ahead of the separator this is often the limiting distance for the oil inlet. If sufficient distance is not allowed improper atomization and distribution will be experienced.

Many ingenious devices have been used as quills in the steam line to improve atomization, nevertheless the most satisfactory method of introducing the oil consistent with freedom from plugged quills is the use of a straight $\frac{3}{8}$-inch pipe extended to the center of the steam line.

A careful consideration of these factors will go a long way toward solving the problem of lubricating a Corliss engine. However, it is important that the edges of the valve lips are not so sharp that the oil film is scraped off, and that the valves do not bind.

Corliss valves are sensitive and in some instances drastic measures must be taken to secure proper lubrication. For example, it may be necessary to have an oil line supply oil directly to the valve seat so the oil will be wiped on the rubbing surfaces by the movement of the valve.

MacLaren and Rogers [43] have assigned to the Standard Oil Company of Indiana a patent for a steam cylinder oil which will readily form a stable water-in-oil emulsion, and which will maintain a continuous film between piston ring and cylinder surfaces of Corliss and Allis Chalmers engines. Their formula contains a heavy lubricating oil, an asphaltic material, a water-in-oil emulsifying agent, and a compounding substance such as lard oil. A typical formula is:

	Per Cent by Weight
Heavy steam refined oil	89.2
Aluminum stearate	.3
Petroleum cracked residue (steam reduced)	2.0
No. 1 Lard oil	8.5

The steam refined oil may be of a Mid-Continent type and may be characterized by a viscosity of about 210 S.U.V. at 210° F., and A.P.I. gravity of 21.1 and a flash point of 530° F. Any equivalent lubricating oil

[43] U. S. Patent 1,924,211 (Aug. 29, 1933).

may be used for this purpose. For instance, these inventors have used a steam refined paraffine base Pennsylvania lubricating oil having a viscosity of about 217 S.U.V. at 210° F., and A.P.I. gravity of 25.1 and a flash point of 580° F. A paraffine base oil is preferred for high temperatures and, in any case, the lubricating oil should be free of gum forming constituents. Instead of aluminum stearate other soaps may be used such as calcium stearate, iron stearate and the like. Oleates may be used, but were considered objectionable from the standpoint of gum formation. The soap should be definitely an emulsifying agent of the water in oil type.

A steam reduced asphalt produced from a cracked petroleum residuum is preferred because asphalts of this type are characterized by their fluidity at high temperatures. This asphalt may have a melting point by the Ball and Ring Method of 135 to 145° F., and may have a needle penetration of a wide range, 5 to 20 at 77° F. being considered satisfactory. The asphalt was considered necessary in conjunction with the metallic soap to give the oil proper emulsifying characteristics. The lard oil is a compounding agent which increases oiliness, and should be refined to remove as much gum forming material as possible. The inventors found that the temperature of mixing was quite important, and that 380 to 500° F. was desirable depending on the particular soap and asphalt material used. At temperatures below these values the aluminum stearate and asphalt could not be incorporated in a stable and uniform state. One method of preparation was to mix all the ingredients together at about 400° F. In another method, the lard oil, aluminum stearate and asphaltic material were mixed together with 10 per cent of the steam refined lubricating oil in a grease mixer; (1) continuously stirring and heating to 400° F., (2) then cooling to 260° F. and adding the balance of the lubricating oil.

MacLaren has also assigned to the Standard Oil Company of Indiana a patent [44] covering the use of 1 to 10 per cent of hydrogenated fat, 0.5 to 2.0 per cent free fatty acid, steam refined cylinder stock and small amounts of aluminum stearate to decrease the oil drop size in the hydrostatic lubricator.

Miscellaneous Uses of Aluminum in Lubricating Compounds

In 1899, Harris [45] made a mixture of lubricating oil with suitable proportions of Stockholm tar, white lead, borax, sodium hydroxide, alum, lime, tar, balsam and water. Armstrong [46] made use of a composition consisting of powdered aluminum suspended in a solution of nitro-cellulose, as a metal drawing compound. Cara and Frank [47] have developed an inorganic lubricant consisting of colloidal silicic acid or aluminum hydroxide in the form of a brine or gel as a lubricant for nitric acid pumps. Perucca [48] heated hydrated aluminum silicate with castor oil at temperatures above

[44] U. S. Patent 1,998,767 (Apr. 23, 1935).
[45] British Patent 4,352 (Feb. 28, 1899).
[46] British Patent 367,198 (Mar. 6, 1931).
[47] British Patent 405,450.
[48] British Patent 412,188 (Dec. 19, 1932).

270° F., in the preparation of a lubricating oil. Spalding [49] has proposed the use of powdered metal salts of fatty acids as dry lubricants for the bobbins used in the spinning industry. Aluminum stearate was considered suitable for this purpose.

Aluminum Naphthenate Castor Machine Oils

Rebber [50] has assigned to the Union Oil Company a patent covering the use of various proportions of aluminum naphthenate dispersed in the lubrication oils for the production of castor machine oils. Frizell [51] has also assigned to the Union Oil Company his patent for a castor machine oil comprising an aluminum naphthenate soap, together with 11 per cent of its volume of alkali metal naphthenate (sodium naphthenate), with or without water up to 2 per cent of the total blend with lubricating oil. The object of this formula was to improve the stability of the viscosity of aluminum naphthenate blends which normally greatly decrease in viscosity with age.

[49] U. S. Patent 1,694,148.
[50] U. S. Patent 1,582,227 (Apr. 27, 1926).
[51] U. S. Patent 1,582,258 (Apr. 27, 1926).

Chapter IX

Lead Base Greases

Lead soaps, such as lead linoleate, are often used as driers in the manufacture of boiled oils and varnishes. They are best prepared by precipitating soluble soap solutions with lead acetate. Lead oleate, $Pb(C_{18}H_{33}O_2)_2$, known as lead plaster, is much used in pharmacy. It may be prepared by warming together 100 parts of oleic acid and 16 parts of sodium hydroxide dissolved in 90 parts of water. This soda soap is then dissolved in 2000 parts of water and into it is poured, while stirring, a solution of 67 parts of lead acetate in about 800 parts of water. The resulting precipitate is washed twice with water to free it from sodium salts and may then be dehydrated by heating at about 220° F.

If the sodium or potassium soaps of any fat or fatty acid such as whale oil, stearic acid, castor oil, linseed oil, degras, beeswax, rosin, or naphthenic acids are made, the lead soaps may be prepared by similar procedure. Lead soaps are sometimes used in compounding transmission greases, railroad greases and oils. It is claimed that they are instrumental in producing more stable lubricating films and reducing friction.

Lead oleate is a sticky, brown, ointment-like mass, soluble in mineral oil. It is of interest that the lead salts of the higher saturated fatty acids are nearly insoluble in ether, whereas lead oleate, lead linoleate, lead ricinoleate, and, in short, the lead salts of the most frequently occurring unsaturated acids, are soluble. The lead salt of erucic acid is but sparingly soluble in ether; the lead salt of iso-oleic acid is said to be insoluble or more insoluble than the lead salt of oleic acid. The lead salt of ricinoleic acid is insoluble in low-boiling petroleum ether.

Table 1 gives the melting point and solubility of the most important lead soaps:

TABLE 1.—Melting Point and Solubility of Important Lead Soaps.

Lead Salt of	Melting Point ° F.	Solubility in Petroleum Ether — Grams per 100 cc. at 68° F.	Solubility in Petroleum Ether — Grams per 100 cc. at 140° F.
Caproic acid	163–165		.0608
Heptylic "	194–196	.02	.0528
Capryic "	182–184	Practically insoluble	.0384
Nonylic "	201–203	" "	.0450
Capric "	210	" "	.0170
Lauric "	217–219	" "	Practically insoluble
Myristic "	224–225	" "	.0210
Palmitic "	233.6	" "	Practically insoluble
Stearic "	257	" "	.0170
Oleic "	113–122	Readily soluble	Readily soluble

For some greases, particularly those used in automobile transmissions and automobile worm gears, where a very unctuous, free-flowing grease is required, lead is used in place of calcium and sodium soaps. Greases of this character are said to resist the scraping, squeezing action of the worm against the gear wheel. While lead soap greases do not emulsify or dissolve with water, when in dilute solution in mineral oils moisture has a tendency to precipitate the lead soap.

LEAD OLEATE—MINERAL OIL COMPOUNDS

Normal or basic lead oleate may be prepared in open steam jacketed grease kettles by allowing lead oxide (PbO) to react on commercial oleic acid derived by either saponification or distillation. There is evidence, however, that the reactions are not complete and that pressure saponification is desirable particularly when preparing lead soaps from triglycerides such as fish oil, rape oil, etc.

FORMULA FOR NORMAL LEAD OLEATE:

	Pounds	Per Cent by Weight
Oleic acid (2 pound mols)	564.72	52.8
100 S.U.V. at 100° F. pale oil	282.36	26.4
Lead oxide (PbO)	223.20	20.8

The reaction which takes place is represented below in which R indicates the oleic acid radicle:

$$2RCOOH + PbO = (RCOO)_2Pb + H_2O$$

According to Diggs and Campbell the quantity of mineral oil shown in the formula is essential for the production of satisfactory normal soaps. One half of the oil is measured into a grease kettle and the lead oxide added and stirred to obtain a smooth paste. The oleic acid is then mixed with the remainder of the pale oil and the mixture run into the kettle. A few small particles of the lead oxide will escape reaction and may not be broken up by the agitation provided by the paddles.

The batch is heated to 275° F. for three hours. After about fifteen minutes considerable foaming will take place. Then the batch becomes clear and darkens.

FORMULA FOR BASIC LEAD OLEATE:

	Pounds	Per Cent by Weight
Oleic acid	847.08	47.1
Lead oxide	669.60	37.2
100 S.U.V. at 100° F. pale oil	282.36	15.7

The reaction which takes place is:

$$2RCOOH + 2PbO = RCOO_2Pb \cdot PbO + H_2O$$

The preparation is carried out as for normal lead oleate, but heating to 300° F. for three hours. In the kettle the mixture does not become very clear but retains a grey, opaque appearance. In some cases, a very small

amount of black material, consisting chiefly of lead, may be formed. No appreciable sediment should remain on the bottom of the kettle after the batch has been drawn.

Samples were prepared with the foregoing normal and basic lead oleates and it was found that the normal soap had little effect on decreasing the pour point and tended to reduce the viscosity at 210° F. The basic soap, however, increases the viscosity at 210° F. and is effective in reducing the pour point:

	Normal				Basic			
Percent of Lead Soap	0	3	6	9	0	3	6	9
A.P.I. gravity	19.4	15.2	18.3	16.5	21.1	19.6	18.2	15.9
S.U.V. at 210° F.	161	141	151	141	170	183	226	271
Pour point ° F.	60	65	60	60	70	35	45	40

FORMULA FOR LEAD OLEATE—PRESSURE METHOD:

		Per Cent by Weight
Litharge (PbO)	1500 pounds	30
Oleic acid	500 gallons	70

These materials are run into an autoclave and cooked for four hours with the kettle closed, using steam in the jacket at 100 pounds per square inch. On drawing the batch from the autoclave no appreciable amount of uncombined lead oxide should be noticeable.

The product obtained should have the following properties:

Specific gravity at 60° F.	1.14–1.40
Percent acid as oleic (by extraction with ethyl alcohol and titration in the absence of the lead soap)	25 max.
Percent lead as Pb	20 min.
Percent impurities insoluble in P.E.	3.0 max.
Percent water	1.0 max.

Actual tests made on lead oleate prepared as above were: Lead as PbO, 28.7 per cent: Free Oleic Acid, 19 per cent; and matter insoluble in petroleum ether, 2.86 per cent. With oleic acid at 7.152c per pound and litharge at 5.5c per pound the cost of this product, including 1c per pound for compounding, is 7.65c per pound.

Van Gundy and Dimmig [1] prepared what was considered to be normal lead oleate by heating oleic acid and lead oxide together at temperatures between 520 and 525° F. They believed that lead oleate formed at lower temperatures was basic and more or less insoluble in mineral oil. Also, that it tended to increase the pour point of lubricating oil, although the soap prepared by the author at 275° F. was found to have little effect on pour point. Van Gundy's normal lead oleate was claimed to be readily soluble in mineral oil and to decrease the pour point of lubricating oils by preventing the crystallization of wax.

In 1929, Dimmig [2] reported that he considered the use of basic lead oleates as unsuitable for lubricants. He claimed they were sticky and

[1] U. S. Patent 1,594,762 (Aug. 3, 1926).
[2] U. S. Patent 1,729,823 (Oct. 1, 1929).

stringy in character, were not permanently soluble in mineral oil, and did not reduce the viscosity of the oils at low temperatures. He considered the formula of basic lead oleate to be 2 $Pb(C_{17}H_{33}COO)_2 \cdot PbO$. Dimmig preferred the normal lead oleate having the formula $Pb(C_{17}H_{33}COO)_2$, which is oily in character, permanently soluble in oil and tends to reduce the viscosity of mineral oils at low temperatures. He prepared his normal lead oleate by the reaction between lead oxide and oleic acid in the presence of CO_2. He introduced the carbon dioxide into the mixing kettle while heating and stirring at temperatures of 350 to 500° F. The reactions which took place were considered to be:

I. $3PbO + 4C_{17}H_{33}COOH = 2Pb(C_{17}H_{33}COO)_2 \cdot PbO + 2H_2O$

II. $2Pb(C_{17}H_{33}COOH)_2 \cdot PbO + CO_2 = 2Pb(C_{17}H_{33}COO)_2 + PbCO_3$

III. $PbCO_3 + 2C_{17}H_{33}COOH = Pb(C_{17}H_{33}COO)_2 + H_2O + CO_2$

It is probable that the lead oxide and oleic acid react immediately to form basic lead oleate. Carbon dioxide then reacts with the basic salt to form the normal oleate and lead carbonate, and the oleic acid reacts with the lead carbonate, yielding normal lead oleate, water and carbon dioxide. Dimmig blended from 2 to 10 per cent of the normal lead oleate with mineral oils at 200° F. to obtain lubricants suitable for use at low temperatures.

Otto [3] has discussed various methods of preparing both normal and basic lead oleate for use in lubricants. He has noted that some lead base lubricants are unstable. In one case a precipitate, six inches deep, formed in a drum of the lubricant after standing for three months. The lead soap, in this case, was produced by double decomposition methods. It was not stated whether, or not, free sulfur or sulfurized fats were present in this lubricant. Otto gives the following formula for a lead base gear lubricant. In an open fire heated kettle is placed 11.8 per cent of whale oil and its temperature increased to 500° F. While stirring, 4.2 per cent of PbO is sifted in and the temperature maintained at 500° F. for two hours. At a temperature about 20° F. lower than its flash point, 84 per cent of mineral oil is added. A good grade of steam refined cylinder stock of 150 S.U.V. at 210° F. or a black oil of similar viscosity having a pour point not over 20° F. was recommended by Otto. After the lubricant has cooled to 150° F. it is drawn into shipping containers.

During the past fifteen years a wide variety of lead base lubricants have been made by incorporating from 12 to 18 per cent of normal lead oleate with lubricating oils of 90 to 100, 140 to 160, and 180 to 200 S.U.V. at 210° F. One of the most important requirements of greases of this kind is the maximum amount of free lead oxide left in the finished lubricant. Lead oxide is a lapping agent and will greatly increase wear of anti-friction bearings. To obtain as nearly complete saponification as possible, some manufacturers prepare a soap from menhaden fish oil by treating it under pressure in an autoclave with PbO. In some plants the reaction is substantially completed in about thirty minutes when using the pressure method.

[3] *Petroleum Engr.*, 75 (June, 1931).

LEAD BASE TRANSMISSION OIL

Formula

	Pounds
Whale oil	550
Lead oxide (PbO)	209
Black oil, 60 vis. at 210° F.	4000

PROCEDURE:

The whale oil is first charged in a fire-heated grease kettle and brought to a temperature of 475° to 525° F.

At this temperature the litharge (PbO) is slowly sifted in, while agitating.

Agitation and heating are continued for from 1½ to 2 hours.

A sample of the product may then be taken for consistency determination, and if satisfactory the batch is cooled to about 150° F., when it may be drawn into containers.

It is claimed by some manufacturers of lead base lubricants that the use of a lead soap such as lead oleate, made by high-temperature process, imparts a low cold test to the resulting lubricant.

ANALYSIS OF A COMMERCIAL LEAD BASE TRANSMISSION LUBRICANT

The analysis of a well-known lead base transmission lubricant is given below:

	Per Cent by Weight
Combined fat	5.98
Free fat	0.71
PbO	2.85
Water	0.55
Black oil (Asphalt 5.77)	84.24

Lead Base Compounded Ford Oils

It has been known for many years that the use of motor oils compounded with various soaps had a tendency to reduce chattering of the transmission bands of Model T Ford cars. Oils compounded with aluminum oleate or stearate are marketed by many concerns. While the aluminum soap base oils are effective in reducing or eliminating chatter, it has been found that they show marked changes in viscosity when stored and are considered unsatisfactory for this reason. The result of much practical and scientific work has indicated that lead oleate is particularly adaptable to the compounding of chatter-free oils.

Lead Oleate Base Ford Oil

Formula

	Per Cent by Weight
Oleic acid (commercial)	8.03
Water	29.22
Caustic soda	1.14
Water	19.70
Lead acetate (technical)	5.41
Water	36.50

PROCEDURE:

The method of preparing this product has already been indicated above. In brief, it consists of converting an aqueous solution of sodium oleate to lead oleate by treatment with a lead acetate solution.

The commercial oleic acid (Emery Elaine red oil) and water are first charged in the kettle. The caustic soda solution is run in while agitating at room temperature. The steam is then turned on in the kettle jacket and the temperature brought to 210° F.

The sodium oleate solution thus formed is cooled to 180° F., when the lead acetate solution is run in over a period of about five minutes, to convert the lead soap.

The steam is turned on, to coagulate the soap mass. The temperature reaches about 170° to 180° F. The agitation, which has been carried on throughout to this point, is discontinued, and the soap allowed to sink to the bottom of the kettle. The supernatant sodium acetate solution is then siphoned off and either discarded or treated for the recovery of the sodium acetate.

The lead soap is then washed with 200 per cent of water at 170° F. The batch is then cooled about 100° F. and the washed water siphoned off.

The lead soap is then dehydrated by heating to 300° F., over a period of about two hours. It may then be mixed off with a light grade of motor oil in any proportion desired, a three-quarter per cent solution by weight having been found to be very satisfactory.

Lead Oleo-Naphthenate Base Ford Oil

Lead oleate, stearate, palmitate, linoleate, etc., may be used singly or in any combination with mineral lubricating oil to give products of chatter-preventing properties, but are of poor stability, particularly in the presence of moisture.

The production of lead soaps by double decomposition (action of a soluble lead salt on an alkali oleate) has already been discussed. Lead oleate may also be prepared by neutralization of oleic acid with oxides of lead or lead carbonate, or by heating triolein with lead oxides.

It is possible to produce from petroleum a saponifiable material known as naphthenic acid. This material is most conveniently derived from California petroleum, but may also be obtained from the crudes produced in Texas or Mexico. It is believed that the constitution of these derived naphthenic acids is different from those obtained from Russian petroleum. Since it is possible to produce water-soluble sodium soaps from the naphthenic acids, lead salts of the naphthenic acids may be easily produced by treating such a solution with lead acetate.

A mineral oil compounded with a small percentage of lead naphthenate is stable in the presence of water, but has little effect in reducing chatter of the transmission bearings. Lead soaps plus lead naphthenate, made separately, when dissolved in oil, prevent chatter when the lead soap is present in excess of the naphthenate. Such a compounded oil is not stable to moisture.

Lead salts of fatty and petroleum acids (naphthenic acids), precipitated simultaneously from a solution of the alkali salts of the higher fatty acids (true soaps) and alkali naphthenate by a water-soluble lead salt, prevent chattering and are stable to moisture, particularly when the fatty acid used is oleic acid.

If the oleic acid in the mixture of oleic and petroleum acids predominates, stability is not assured, although chatter-preventing qualities are maintained. When the petroleum acids predominate in the mixture, a stable oil will be

produced, but the chatter-preventing qualities will be impaired. The mixture consisting of nearly equal quantities of oleic and petroleum acids is preferred. Three quarters of one per cent of the lead compound (lead oleonaphthenate) dissolved in a suitable motor oil eliminates chatter, smaller amounts preventing chatter to a lesser extent. Larger quantities of the lead compound are of no material value in preventing chatter, and add considerably to the cost of the product.

The petroleum acids which give the best results are produced from the waste alkali washes obtained in the refining of light lubricating distillates from the sources indicated above and have the type formula $C_nH_{2n-2}O_2$. Acids containing from 7 to 20 carbon atoms of this type may be present in the mixture termed naphthenic acids. While it is known that organic acids of this type predominate, it is probable that other hydrocarbons and organic acids are present.

A mixture of lead compound prepared with mineral lubricating oil as described is very stable, considerable quantities of water being absorbed without precipitation of the lead compound. The lead salts of the higher saturated fatty acids are nearly insoluble, whereas lead oleate, lead linoleate, lead ricinoleate, in short the lead salts of the most frequently occurring unsaturated acids are soluble and adaptable to the production of chatter-preventing oils. While it is most convenient and economical to make use of oleic acid in manufacture of the lead compound, it is possible that triglycerides (whole fats or fatty oils) such as triolein, linseed oil, castor oil, etc., could be made use of for the production of satisfactory products.

Calcium, magnesium, zinc, copper, or other metallic soaps plus metallic naphthenates, precipitated simultaneously from a solution of alkali soap naphthenate, are capable of reducing chatter in various degrees, but are not stable to moisture.

Magnesium, calcium, copper, zinc, tin and iron oleates or other fatty acid salts reduce chatter to a greater or less extent, but are characteristically unstable when present in small quantities in mineral oil. Traces of moisture turn them cloudy, precipitation occurring after standing from one hour to several days.

Sorenson [3a] claims the use of soap in mineral oil to prevent chattering. The patent covers the process of treating the brake bands "with mineral oil having a soap mingled herewith." He also claims [3b] the "lubricating compound" consisting of mineral oil and ammonia soaps. It is not apparent that Sorenson had in mind any soaps other than the alkali soaps of the fatty acids.

The use of any metallic naphthenate made separately and added to a metallic compound such as lead oleate will not produce a stable compound. Frizell [3c] claims a castor machine oil in which aluminum fatty acid salts or aluminum naphthenate is stabilized by the addition of sodium naphthenate or other alkali metal naphthenate. It is claimed that small amounts of water

[3a] U. S. Patent 1,414,311.
[3b] U. S. Patent 1,414,227.
[3c] U. S. Patent 1,582,258.

do not result in precipitation of the soaps. Cresols have been used in some cases for producing stable mixtures of alkali metal salts in mineral oil. Aluminum naphthenate alone in mineral oil, besides being unstable, has poor chatter-preventing qualities.

In the preparation of lead oleo-naphthenate satisfactory for compounding non-chatter oils, it is imperative that the lead oleate and naphthenate be precipitated simultaneously from the same solution, if a stable base is to be produced. There is cause to believe that complex lead salt of the oleic and naphthenic acid is formed.

The reactions involved in the preparation of the lead oleo-naphthenate are represented in the following chemical equations, in which $C_{13}H_{25}COOH$ is used as a typical naphthenic acid:

(1) Removal of naphthenic acid from gas oil with lye—

$$C_{13}H_{25}COOH + NaOH = C_{13}H_{25}COONa + H_2O$$

(2) Liberation of naphthenic acid from wash lye with acid—

$$2C_{13}H_{25}COONa + H_2SO_4 = 2C_{13}H_{25}COOH + Na_2SO_4$$

(3) Formation of soda soap of oleic and naphthenic acid simultaneously—

$$C_{17}H_{33}COOH + C_{13}H_{25}COOH + 2NaOH = C_{17}H_{33}COONa + C_{13}H_{25}COONa + 2H_2O$$

(4) Conversion of sodium salts to lead salts—

$$Pb(C_2H_3O_2)_2 + C_{17}H_{33}COONa + C_{13}H_{25}COONa = \begin{matrix} C_{13}H_{25}COO \\ C_{17}H_{25}COO \end{matrix} > Pb + 2NaC_2H_3O_2$$

Process for Manufacture of Lead-oleo-naphthenate Base Ford Oil

MATERIALS REQUIRED:

Commercial oleic acid or Emery Elaine oil, as it is usually termed.

Naphthenic acids: These acids are derived from waste lye obtained in the refining of asphalt base gas oil, by treatment with sulfuric acid. Inspections of a batch of naphthenic acids suitable for this purpose follow:

Gravity	14.3° Bé.
Flash	240° F.
Viscosity at 100° F.	349
Naphthenic acids (using 280 as combining weight)	76.2 per cent
Neutralization value, mg. KOH per gm.	153.7

Water: Fresh water is suitable for all washes and for making the solutions required. Salt water is objectionable due to the quantities of chlorides and sulfates it contains, which would cause the precipitation of insoluble lead salts.

Caustic soda: This material is employed as a 40° Bé. lye solution.

Lead Acetate: A technical grade of lead acetate of the quality known as "white, broken." This material is used as a solution containing 50 per

cent by weight of lead acetate. A temperature of 148° F. is convenient to cause rapid solution.

EQUIPMENT REQUIRED:

Kettle for preparation of the lead soaps: The process is most readily carried on in a regular steam-jacketed grease mixer. The kettle should be equipped so that water may be circulated through the kettle jacket for cooling purposes.

Skimming Pump: Some means of siphoning off supernatant liquor and wash waters from the heavy lead soaps which fall to the bottom of the kettle is required. A pump and skimming line or "swing line" is found to be most convenient.

Lead Acetate Storage: Solutions of this material cannot be stored in iron vessels as the iron goes into solution, precipitating spongy metallic lead. Lead-lined vats or wooden tanks equipped with lead-lined steam coils are used to advantage.

FORMULA:

Gross Formula for Soap

	Per Cent by Weight
Oleic acid	11.19
Naphthenic acids	11.19
Water	44.76
Caustic soda, 40° Bé.	8.25
Lead acetate, 50 percent solution	24.61

Net Formula for Soap

Oleic acid	29.79
Naphthenic acids	29.79
Caustic soda (dry)	7.69
Lead acetate (technical)	32.73

PROCEDURE:

In order to prepare the lead oleo-naphthenate a mixture of 50 per cent commercial oleic acid and 50 per cent of naphthenic acids is neutralized with caustic soda until a faint alkaline reaction to phenolphthalein can be observed. A small amount of the free acid mixture may then be added to bring the batch to a slightly acid condition. The sodium salts of the naphthenic and oleic acids dissolved in water may then be converted to lead soaps by treating with a 50 per cent solution of lead acetate. The addition of lead acetate is continued as long as the precipitation of insoluble lead soap occurs. The lead soap is then washed and dried. The actual average yield from the net formula is 75.3 per cent.

The oleic acid and naphthenic acid are charged in the kettle and double the amount of fresh water is added, with thorough agitation.

Steam is then turned on in the kettle jacket until the temperature reaches 125° F. The 40° Bé. caustic soda solution is then run into the acid-water mixture. While this process is being carried out the temperature rises to about 180° F. Near the end of the neutralization process, care must be used to avoid passing the neutral point. The caustic soda is added until a slight pink color is obtained when a small portion of the reacting materials are brought together with a drop of phenolphthalein solution on a glass or porcelain plate. One-half per cent by weight of the total amount of mixed acids may then be added to the kettle to be sure that the mass is slightly acid.

With the temperature at about 180° F. the lead acetate solution is allowed to flow into the soda soap solution and agitation is continued for about two minutes.

On allowing the mixture to stand in the kettle the lead soaps sink to the bottom and a fairly clear supernatant liquor should form. At this point the addition of the lead acetate solution must be carefully watched and stopped as soon as no more lead

precipitate is formed. If a sample of the supernatant liquid is mixed with a few drops of sodium chromate solution and a heavy yellow precipitate is obtained, the indications are that all of the organic acids have been carried down in the form of lead soaps.

The batch is allowed to settle for about one hour.

The supernatant liquor is then siphoned or pumped off. Two hundred per cent by volume of fresh water is added to the lead soaps and the mixture agitated and heated to 180° F. The batch is allowed to stand and the wash water skimmed off.

The soaps are washed as before but no heat is applied.

The lead soaps may be dehydrated by applying the steam to the kettle and allowing the temperature to rise to about 215° F., when considerable foaming may be noticed. At about 230° F. the foaming decreases and the soaps take on a clear appearance. The temperature may then be allowed to rise to 300° F. in order to completely dehydrate the material.

The lead soap should be drawn as quickly as possible after being dehydrated, as prolonged heating oxidizes and darkens it, impairing the color of the finished product.

A three-quarter to one per cent solution of the lead soap in suitable motor oil may then be made by warming the mixture to about 130° F.[4]

LEAD-PETROLEUM COMPOUNDS USED IN LUBRICATING COMPOSITIONS

Diggs [5] has assigned to the Standard Oil Company of Indiana his patent for a steam cylinder oil containing lead soap. By the use of about 1 per cent of oil soluble lead soaps he claimed it possible to control the drop size of the oil in the hydrostatic lubricator. Arveson [6] has assigned to the Standard Oil Company of Indiana his patent for a lubricant consisting of lubricating oil, and the lead soaps of mahogany acids. The finished lubricants ordinarily contained about three and one-half per cent of lead. McCabe and Mead [7] have developed a lubricating oil heavier than kerosene and containing an alkyl compound of a metal such as lead or tin. Pope [8] has proposed a method of refining lubricating oils to render them resistant to sludging in service, in which a small quantity of an organic lead compound is added to a hydrocarbon heavier than butane, and the mixture with lubricating oil subjected to prolonged heating. Doell [9] has developed a fluid lubricant consisting of lubricating oil carrying in solution lead salts of unsaturated fatty acids (such as oleic acid) together with the lead salt of sulfonic acid. Arveson [10] has proposed a similar lubricant consisting of heavy metal compounds of petroleum sulfonic acids dispersed in viscous lubricating oils.

Lead Oleate and Lead Tetra Ethyl Motor Oils

Helmore's [11] invention relates to means for preventing the deterioration of lubricating oils, when used under conditions of high temperature, by increasing the resistance of the oil to molecular oxidation by the addition (to the oil) of small amounts of chemical substances having the property of inhibiting oxidation.

[4] U. S. Patent 1,781,167 (Oct. 12, 1926).
[5] U. S. Patent 1,869,800 (Aug. 2, 1932).
[6] U. S. Patent 1,871,942 (Aug. 16, 1932).
[7] U. S. Patent 1,857,761 (May 10, 1932).
[8] U. S. Patent 1,882,887 (Oct. 18, 1932).
[9] U. S. Patent 1,867,695 (July 19, 1932).
[10] U. S. Patent 1,871,941 (Aug. 16, 1932).
[11] British Patent 398,222 (Sept. 11, 1933); also: British Patent 295,230.

It is well known that one of the chief defects experienced with lubricants under such conditions, for example, in internal combustion engines, is the formation in the oil, often after a very short period of running, of an oxidizing sludge which precipitates in the oil reservoir and may choke the filter and oil channels, and otherwise impair lubrication. Helmore believed that the oxidation of the oil in this way was caused by exposure of the oil surface at high temperature to oxygen, and that oxidation proceeds most rapidly on cylinder walls and piston surfaces which are at high temperatures, and from which thin films of oil are continually sheared. Further oxidation at lower temperatures may occur in the oil fog present in the crankcase where the finely sub-divided oil presents a great surface to oxygen. Further surface oxidation occurs in other metallic surfaces, such as crankcase walls, journal housings, connecting rods, and the under side of the piston. The formation of gum in the piston ring grooves, by causing the piston rings to become embedded and jammed in the grooves, produces a vicious circle whereby the formation of gum is accelerated owing to the sweeping of flame past the piston ring, which by raising the the temperature between the piston and the walls, accelerates oxidation still further and increases wear on the piston and cylinder.

Helmore found by a method of direct oxidation of certain lubricating oils, at various surfaces over a range of temperatures, that the rate of oxidation of oils at any particular temperature is decreased to a marked extent by the addition of certain elements and their compounds of group IV periodic classification; other than lead tetra ethyl, e. g., tin, cerium and their compounds, in oil soluble or finely divided states. For example, in his experiments, tin oleate added to a Venezuelan asphaltic base red oil in the proportion of approximately 1 per cent by weight was found to decrease the rate of oxidation of the oil in air threefold. Other compounds of this group such as tin naphthyl, tin ricinoleate, cerium oleate and cerium resinate, were also found to give beneficial results with certain commercial oils. He claimed in a previous patent [11a] the use of tetra ethyl lead in lubricating oils, and also other members of group IV, Periodic Classification, and these substances may be used either alone or in conjunction with tetra ethyl lead.

The substances claimed in this invention are not added for the purpose of altering the consistency of the oil in any way or to affect its other properties, except in so far so to increase its resistance to oxidation.

Helmore found sludge preventing properties are to be obtained with lubricating oil having dispersed therein a small proportion of tin; preferably an organic compound of tin, such as tin oleate, tin naphthyl, tin ricinoleate, tin phenyl, tin methyl iodide.

An important property required of lubricating oil for use in internal-combustion engines is that it should have a high spontaneous ignition temperature, in order that it may not be burnt in the engine cylinder when subjected to high temperatures and pressures. Lead tetra ethyl has been found particularly suitable for this latter purpose. Thus, in order that the oil may have both the properties of sludge preventing and a high spontaneous

[11a] British Patent 295,230.

ignition temperature, according to Helmore, it would have dispersed therein a small proportion of a mixture of an organic compound of tin and an organic compound of lead. Furthermore, lead tetra ethyl raises the temperature at which aldehydes and other oxidation products occur.

If the material to be added is used in metallic form, or in a form which is not soluble in the oil, it should be finely comminuted to approximately colloidal size so that it may remain in suspension in the oil. Preferably, however, materials are selected which are soluble in oil, such as the specific materials mentioned above.

In selecting the quantity of material to be used, the main consideration is that it shall not unduly affect the viscosity of the oil.

It has been found that the property of each substance added to the oil may be up to about 1 per cent by weight of the oil.

Investigations have included an examination of Venezuelan asphaltic base oil, Russian, Pennsylvania bright stock, castor oil, high molecular weight hydrocarbons, oleic acid and olive oil. Proprietory oils have also been successfully treated including blended vegetable and mineral oils. The oils have been submitted to temperatures between 125 and 250° C. in a sealed bulb with air for a period up to thirty days, and when treated with tin compounds such as tin phenyl, tin oleate, tin naphthyl, tin ricinoleate, tin methyl iodide, the amount of oxidation has been found to be reduced up to 40 per cent measured by the amount of oxygen absorbed in the bulb. Accompanying this reduction in oxidation rate there was less sludging and less viscosity increase. In addition to the above mentioned compounds, tests were made with oil treated with tin dust, and this was found to act as an inhibitor of oxidation, usually to a lesser extent than the oil soluble derivatives specified above.

In one example, tin phenyl was added to an aviation lubricating oil designated by the British Air Ministry as P4. The tin was added in a proportion of 1 per cent by weight of the oil, and the mixture was warmed gently to a temperature of about 50° C. Two samples were then tested, one with the treated oil, and the other of the untreated oil in a similar bulb and in a similar manner to that described above. Both samples were heated to a temperature of about 200° C. for a period of two hours. It was found that the untreated oil was oxidized to a considerably greater extent than the treated oil. For example, 12 milligrams of oxygen were absorbed by the untreated oil per gram of the oil, while 7 milligrams of oxygen only were absorbed by the treated oil. The viscosity increase of the oil was 23 per cent as compared with 10 per cent and the treated oil was considerably less discolored.

In the second test, there were mixed with 1 liter of oil, ten grams of lead tetra ethyl, 5 grams of lead oleate and 10 grams of tin oleate. The oil used was sold under the Trade Name "Castrol XXL." After the preliminary mixing, two samples of the oil, one treated and the other untreated, were tested in bulbs in a similar manner to that described above. The samples were heated to a temperature of 250° C. for a period of two hours. A 20 per cent reduction in the rate of oxidation of the treated oil, and a

considerably higher spontaneous ignition temperature was obtained with the treated oil. The viscosity increase was also less in the treated oil than in the untreated oil.

In order to improve the properties of lubricating oil, it has been proposed to dissolve in the oil, asphaltum or resin, and to eliminate the gumming properties of these additional materials by incorporating ashes of lead, or zinc or tin. There has also been proposed as a substitute for petroleum, oil or soap water for use with boring and shaping machines, an aqueous solution formed by boiling in water, soda, lime, zinc, tin or similar metals, chlorine solution, and oil or fat.

HYPOID GEAR LUBRICANTS

In view of the sliding action between pinion and ring gear teeth of hypoid gears, and the high unit pressures developed at the point of contact, either at high speed on the road or due to shocks incident to gear shifting, there is a tendency towards scuffing and scoring providing the lubricant does not possess definite, active extreme pressure properties. The necessity for adequate extreme pressure lubrication during the break-in period has been definitely established, although in some cases after hypoid gears are once properly broken-in, the more active types of extreme pressure lubricants may be dispensed with and more normal types of high film strength lubricants utilized with considerable success.

Prior to 1929, the Packard Motor Car Company started using hypoid gears in the rear axles of their cars and were convinced that lead soap lubricants containing sulfur were necessary for proper lubrication. Even before this, Mack trucks were made with hypoid gears in the rear axles of some units. Here again, lubricants containing a combination of lead soap and sulfur were definitely required. The Gleason Gear Works established a testing laboratory and made break-down tests in hypoid gear rear axles. Loads and speeds were imposed which were sufficiently high to quickly cause scuffing and scoring of the new hypoid gear sets. With lubricants containing lead soap and sulfur freedom from failure was possible. In spite of the fact that the roller bearing manufacturers considered the lead soap base oils to be abrasive in nature on account of their free lead oxide content, or the presence of sulfur, sulfur compounds, or lead sulfide, many such lubricants were approved and used with success in Mack trucks equipped with hypoid gears.

In 1937, considerable interest in extreme pressure lubricants for hypoid rear axles has been aroused in view of the adoption of hypoid gears by the majority of the large automotive manufacturers. The advantages of a lower center of gravity due to placing the pinion at a lower position in the hypoid design, the elimination of tunnels in the floor boards, the quieter action of hypoid gears and the tendency to reduce the size of power transmitting parts with relation to increased engine power are items which favored the general adoption of hypoid gears. It is evident, however, that unit pressures and gear tooth surface speeds are such that, even with improved metallurgical

Name of Hypoid Gear Lubricant	ZN	ST Leaded Lubricant	WR Summer	WHT No. 0	WR Winter	HYPOY	PB No. 20	PB No. 15	PN 612
Color	Gr. Black	Black	Black	Black	Black	Dark green	Black	Black	Black
Odor		Fish	Fish, Pine tar	Tarry	Still Bottoms	Tarry	Faint	Faint	Pine tar
Gravity A.P.I.	20.4	.....	8.4	.....	22.3	21.3	10.0	12.2	15.9
Pour point	21.4	15	25	.....	10	10	35	20	20
Visc. S.U. at 210° F.	101	.....	205	.....	277	119	249	154	131
Ash, per cent		.....	5.30	12.8	0.04	0.33	.....	.....	.889
Oil, per cent	82.70	.....	.....	67	.....	.....	92	93.0	96.2
Lead (per cent PbO)	0.05	5.60	2.98	9.4	None	0.10	1.83	1.44	.857
Tests on Oil:									
Gravity A.P.I.	22.6	16.9	.....	18.3	.....	.....	13.9	13.0	16.2
Viscosity 210° F.	94	122	.....	.....	.....	.....	270	187	150
Viscosity 100° F.	1165?	.....	.....	961	.....	.....	.....	.....	.....
Pour point		.....	.....	.....	.....	.....	.....	.....	20
Color			Black	Black			Black	Black	
Probable origin	Naphthene	Naphthene	Naphthene	Naphthene	Naphthene	Naphthene	Naphthene	Naphthene	Naphthene
Sulfur:									
Total, per cent	1.63	1.86	2.02	2.56	0.13	0.12	2.57	2.45	.....
In oil, per cent	0.95	1.96	.....	.....	.....	.....	2.70	2.47	.....
Soap, per cent (as lead oleate)	0.20			.....	.....	.....	5.3	5.0	.....
Timken Test	34,800	26,100	26,100	37,700	20,300	.....	27,140	25,710	25,710

composition of the hypoid gears, ordinary low film strength lubricants will not suffice and special lead soap and sulfur gear lubricants are essential.

In the following sections the discussion will be limited in so far as possible to lead soap and sulfur lubricants; although occasional mention of other extreme pressure lubricants, such as chlorine compounds and organic phosphorus compounds which will be treated more exhaustively in a following chapter, may be made.

By 1929, a large number of lead soap sulfur lubricants were on the market and in a general way were considered satisfactory for use in conjunction with hypoid gears. These lubricants were analyzed and their Timken film strength determined at a surface speed of 400 feet per minute. The results of these tests are shown in the table on page 662.

The following table gives a typical specification for hypoid gear lubricants and lists eight commercial lubricants which were offered by various manufacturers as complying with requirements:

Test	MC Specifications	WH No. 0	FI.	AM-G	53	58	60	61	DUP
Sp. Gr.	0.99–1.01	1.059	1.013	0.986	.949	1.01	1.01	...	1.02
Flash, ° F.	320 min.	345	385	400	510	375	445	445	330
Fire, ° F.	360 min.	405	435	445	...	420	495	490	360
S.U.V. at 210° F.	95–105	126	125	169	139	137	127	127	146
Channel, ° F.	5 max.	0	0	5	5	5	10	0	10
Percent lead	5–6	8.2	6	3.6	.97	5.5	5.5	5.5	...
Percent free oleic acid	.5 max.	5 0	.75	5.3	...	...	...	...	8.25
Color	Jet black	O.K.	O.K.	O.K.	Green	O.K.	O.K.	O.K.	O.K.
Odor	Inoffensive	O.K.	O.K.	O.K.	O.K.	O.K.	O.K.	O.K.	O.K.
Pour point ° F.	.....	35	15	25	20	20	25	20	50

Clayden [12] pointed out that some automobile manufacturers had been troubled by complaints of noise and actual failure of differential gears. He considered that, in the case of these normal spiral bevel gears, these two difficulties were interrelated and both were due to excessively heavy tooth pressures which accelerated wear, produced noise and in some cases caused seizure of parts. He considered there were many soaps which were as good as, or in some cases even better, with regard to lubricating value, than castor oil, which up to this time was considered one of the best compounding materials for rear axle lubricants. Clayden revealed the use of combinations of red lead (Pb_3O_4) and tallow or red lead and fish oil. He proposed two theories to explain why lead soaps aided in gear lubrication where the high unit pressures forced practically all of the oil from between the gear tooth surfaces in contact. One was that the soap, probably due to it being a polar body, improved the ability of the oil to enter the interstices of the metal; the other was that the presence of even minute quantities of another metal might have an effect similar to that of introducing another metal between the two steel parts. Clayden did not comment on the use of combinations of lead soap and sulfur in gear lubricants.

Edwards [13] has pointed out that there was a tendency towards undesirable ingredients in certain lead soap lubricants which increased the rate of wear of either gears or bearings. He considered the abrasive material resulting from scuffing of gear teeth, when a lubricant was used that could

[12] *Automotive Ind.*, 481 (Oct. 5, 1929).
[13] *S.A.E. Journal*, **28**, 50 (1931).

not maintain an adequate film, was more detrimental than even a poorly manufactured compound containing uncombined lead oxide or free sulfur which might be effective in preventing scuffing.

Edwards discussed the manufacture of lead base lubricants consisting, first, of chemically combining suitable fatty oils with lead oxide or litharge; after which the lead soap thus produced was added in any desired percentage to refined lubricating oil. He considered that a satisfactory lead base lubricant should be characterized by the following qualities:

Ability to withstand high unit pressures without excessive wear, scoring, or scuffing.
A low channel test, permitting easy gear shifting at low temperatures.
A low coefficient of friction at high unit pressures. This prevents excessive lubricant temperatures in the rear axle housing.
Should promote smooth and quiet operation of rear axle gears.

Edwards listed the specifications of three manufacturers of automobiles for lead-soap lubricants, as follows:

	A	B	C
Specific gravity	1.000	Not given	Not given
Flash, ° F.	Min. 320	Not given	Not given
Fire, ° F.	Min. 360	Not given	Not given
S.U.V. at 211° F.	(95 to 105)	(90)	(90 to 100)
Pour point, A.S.T.M., ° F.	Not given	0	Max. 15
Channel test, ° F.	Max. 5	Not given	Not given
Lead soap, per cent	Not given	Min. 12	Not given
Lead reduced to free lead, per cent	5 to 6	Not given	3 to 4
Free lead, per cent	Not given	Not given	None
Free oleic acid, per cent	Max. 0.50	Not given	Not given
Free fat by weight, per cent	Not given	Not given	Max. 2
Total fat by weight, per cent	Not given	Not given	9 to 12
Sulfur in mineral oil, per cent	Not given	Not given	Max. 1
Sulfur added, per cent	Not given	(Min. 2.5)	Not given
Corrosion	Not given	Bad	None
Color	Jet black	Not given	Not given
Odor	Inoffensive	Not given	Pine tar

In a paper on methods for testing rear axles Wooler,[14] in 1929, mentioned that graphite and lead soaps which were frequently regarded as satisfactory ingredients of gear lubricants, produced a lapping effect which considerably increased bearing wear. He considered it reasonable to assume that such materials also caused lapping of gear teeth which would eventually cause the teeth to lose their correct profile and become noisy.

Later, Wooler[15] stated that lead soap oils were apparently a necessary evil, and were undoubtedly the salvation of hypoid-gear rear axles. He considered that with lead base oils much greater loads could be carried even though such lubricants were more abrasive than straight mineral oils. He reported results with the Timken film strength testing machine for various lubricants, showing that, at an arbitrary pressure of 20,000 pounds, a straight mineral oil scored very badly, was improved by the addition of 2 per cent of free sulfur, and that with 2 per cent of lead oleate no scratches were evident at this pressure. With a mixture containing 2 per cent each

[14] *S.A.E. Journal*, **25**, 75 (1929).
[15] *S.A.E. Journal*, **28**, 53 (1931).

of sulfur and lead oleate, fine scratches were produced on the test block. These were attributed to the sulfur and may have been caused by the formation of lead sulfide which acted as a lapping agent. The coefficient of friction for the 150 S.U.V. at 210° F. Pennsylvania stock, containing 2 per cent of sulfur, was only 0.077 at 20,000 pounds pressure, as compared with 0.11 for the same oil containing no sulfur but 10 per cent of lead oleate. It would appear from this that, while scoring could be effectively prevented by the use of lead oleate or sulfur, the oleate would give higher frictional results. Wooler gave the following list of characteristics for a lubricant suitable for heavily loaded gears and hypoid gears:

Freedom from separation on standing and in service.
Minimum tendency for abrasiveness.
Ability to carry certain loads on a suitable test machine at specified surface speeds without scuffing.
Freedom from channeling at low temperatures.
Should not be excessively costly.
Should not produce etching, discoloration, or corrosion of bearing surfaces.

Becker [16] explained the function of free sulfur in a mineral oil operating under extreme pressure conditions, as follows: "When a film of such a lubricant is ruptured, the sulfur forms metallic sulfides on the exposed portions of the bearing surfaces."

These sulfides, which may become removed, are very mild lapping agents acting in much the same manner as graphite, mica, or rouge. The combination of chemical action and abrasion may finally result in nearly 100 per cent bearing area, and consequently greatly decrease localized loads and temperatures. Becker considered that the success of hypoid gears would depend on the possibilities for effecting a satisfactory compromise between film strength or load carrying capacity and wear increase.

Havre [17] has pointed out that mineral oils have a particular attraction for sulfides, graphite, and pulverized coal. It has, therefore, very frequently in the past been proposed to sulfurize the surfaces of journals and bearings. In some cases the practice has been to apply powdered sulfur to bearings that are liable to overheat. The formation of hydrogen sulfide and metallic sulfides will increase the adherence of the lubrication oil. Graphite is known to have considerable adhesiveness for both metal and mineral oils. Havre considered that thick lubricating greases, made at high temperatures from animal fat and sodium hydroxide, are capable of forming very resistent lubricating films but do not impart extreme pressure characteristics.

In 1931 Mougey [18] and Almen reported tests, run by the General Motors Research Laboratories, which indicated that in the lubrication of automobile gears the pressures were of such magnitude that ordinary mineral oil films would not cause complete separation of the gear teeth. Although normal gears operate on straight mineral oils, this was considered possible only because of the hardness of the surfaces and the low rate at which heat was

[16] *Ibid.*, 63.
[17] *Génie civil*, **90**, 45-48 (1927).
[18] "Extreme Pressure Lubricants." Presented before American Petroleum Institute (Nov. 12, 1931).

generated on account of the small relative motion between the gear teeth. For hypoid gears, the sliding action is much more pronounced than for spiral bevel gears. Therefore, ordinary oils are not sufficient and special extreme pressure lubricants must be used.

In well lubricated bearings the loads seldom exceed about 2,000 pounds per square inch and the surface speeds are usually sufficiently high to maintain an oil film for separating the two surfaces. In automobile gears the pressures between the gear teeth may reach values as high as 400,000 pounds per square inch with surface velocities ranging from zero to as much as 400 feet per minute. Mougey and Almen have investigated lead soap lubricants containing sulfur. They found that the presence of large amounts of sulfur in successful lead lubricants was beneficial from the standpoint of load carrying capacity and that those with smaller amounts of sulfur did not carry extreme loads. They made experimental mixtures by adding sulfur to mineral oils containing little natural sulfur and found that this addition caused the oil to carry heavy loads whether lead soap was present or not. They added lead soap to mineral oils low in sulfur but were not able by this means to greatly improve load carrying capacity. In some cases, however, where the oils were high in natural sulfur high load carrying capacities resulted.

When lead soap was added to sulfur free mineral oil, a high film strength on the General Motors testing machine was not obtained, but combinations of lead soap and castor oil produced a high film strength. Lead soap, when added to a mineral oil containing sufficient sulfur, produces an active extreme pressure lubricant, which apparently will pass through the transition from fluid film lubrication to true extreme pressure lubrication, with the production of less frictional heat than straight sulfurized mineral oils without lead soaps being present. Mougey and Almen considered that the superior load carrying capacities of such lubricants were due to the formation of a film of separating material formed directly on the surface of the gear teeth. They found a large number of materials which had high load carrying capacity such as carbon tetrachloride, dichlor ethyl ether, etc. When these were used, no doubt the separating film was iron chloride instead of iron sulfide.

Wolf and Mougey,[19] in 1932, again stressed the importance of developing suitable extreme pressure lubricants. At that time, they found available on the market lubricants prepared by blending saponifiable oil containing chemically combined added sulfur, with petroleum oil; or straight sulfur-mineral oil lubricants in which the sulfur was combined directly with the petroleum oil. They also investigated lubricants containing lead soap and sulfur in which high film strength was dependent on added sulfur or a high content of natural sulfur in the mineral oil. They pointed out that, in most cases, the sulfur naturally occuring in refined mineral oils was too firmly bound to be readily available for extreme pressure lubrication activity. Also, sulfur chemically combined with saponifiable oil may also act in this manner.

[19] "Extreme Pressure Lubricant, Correlation of Service Data with Laboratory Testing Methods," Presented before the American Petroleum Institute (Nov. 17, 1932).

Bridgeman [20] has presented an excellent discussion of extreme pressure lubricants, including a description of the testing machine developed at the Bureau of Standards. Load carrying capacity, coefficient of friction (which controls the amount of heat generated in the gear teeth), stability, tendency to cause wear, and corrosiveness were considered important factors relative to the serviceability of hypoid gear lubricants.

McKee, Bitner, and McKee [21] have presented a more detailed account of the design and operation of the extreme pressure lubricants tester developed at the U. S. Bureau of Standards. This includes the results of a large number of tests on various extreme pressure lubricants, together with their conclusion that it was possible to obtain information with this tester which was in reasonable agreement with ratings based upon service performance in automotive gears. It is not our intention to present in this volume a description of the Bureau of Standards tester, now known as the S.A.E. Lubricant Tester, but it may be mentioned that with this apparatus lubricants are tested between two rotating roller bearing cups, in much the same manner as the action of gear teeth. It was found that the wear on these test rolls had little effect on the area of contact. For this reason, the applied loads were reasonably accurate measures of the pressures on the rubbing surfaces. Computations of the pressures for various loads, using the Hertz [22] formula for the elastic deformation and typical values for the physical properties of the steel (modulus of elasticity = 30,000,000; Poisson's Ratio = 0.3), indicated that the average pressure on the rubbing surfaces was as high as 180,000 pounds per square inch for a load of 2,400 pounds, and 115,000 pounds per square inch for a load of 1,000 pounds. The effect of the distribution of pressure due to the film of extreme pressure lubricant is not taken into consideration in these calculations. These figures are of interest, however, in showing that the values of the pressures at the time of seizure are much higher than those obtained with other testing devices, and are more in line with the pressures at which hypoid gear failures occur.

Special Hypoid Gear Lubricants

In the test developed by the Gleason Works, a hypoid rear axle assembly was made use of. The axle was driven through a standard propeller shaft and universal joints by a short jack shaft, which was in turn driven by a 75 H.P. motor through a 16 inch driving belt. Load was applied to the axle by means of a 45 KW. generator, and the electrical load of this generator was absorbed by a band of grid resistances. The power input to the driving motor was measured by a voltmeter and an ammeter. The generator load was recorded by means of a wattmeter.

[20] "The Present Status of the Extreme Pressure Lubrication Problem." Presented before the Society of Automotive Engineers (Jan. 22, 1934).

[21] "An Apparatus for the Determination of Load Carrying Capacity of Extreme Pressure Lubricants." Presented before the Society of Automotive Engineers (1933).

[22] *J. of Math. (Crelle)*, 92 (1881); *ges. Werke, Leipsig*, 1, 155 (1895).

LUBRICANT "A."

This lubricant had the following composition:

	Per Cent by Weight
Lard oil	4.625
Sulfur	.375
Oleic acid	2.640
Litharge (PbO)	1.040
100/25 S.U.V. at 210° F. Calif. bright stock	91.320

In this compound oleic acid and litharge were caused to react together and for the formation of normal lead oleate; the sulfur, as flowers of sulfur, was heated together with the lard oil alone at a temperature of about 400° F. in order to produce a permanent combination. The sulfurized lard oil, lead oleate and Bright Stock were then mixed together to produce a product having the following tests:

Gravity	17.5
Saybolt viscosity at 210° F.	139
Pour point	20° F.
Corrosion test (copper)	O.K.

This lubricant, "A," was tested by the Gleason Works on February 25th, 1930, using a Packard hypoid rear axle having a ratio of 13-61, offset 2 inches. The drive shaft was operated at 1200 R.P.M., which is the equivalent of a road speed of 25 miles per hour. The generator operated at 790 R.P.M. with an average output of about 58.4 KW.

After running the set for 5 minutes with the lubricant at a temperature of 60° F., the load was applied. After 15 minutes of operation the temperature of the lubricant increased to 145° F., loud grinding noises were heard and on subsequent inspection the gears were found to be scored. It was noted that there was a considerable layer of foam on the oil.

LUBRICANT "B."

This was a medium bodied soda base fiber grease prepared from high quality cylinder stock and contained about 5 per cent of soda soap. This grease was tested according to the procedure as mentioned above, and the gears were found to have scored after a test period of less than 15 minutes. Final temperature of the oil was 140° F.

LUBRICANT "C."

This lubricant consists of 95 per cent of "650" Pennsylvania Steam Refined Stock and 5 per cent of acidless tallow oil. Its Saybolt Viscosity at 210° F. is approximately 180. Under the foregoing test conditions, this lubricant was found to fail after a test period of 25 minutes; the final oil temperature being 170° F. There was no layer of foam on the oil.

LUBRICANT "D."

The properties of this sample are listed below:

Specific gravity	1.015
Flash, ° F.	4.20
Fire, ° F.	475
S.U.V. at 210° F.	137
Percent lead	4.87
Channel test ° F.	5
Pour point, ° F.	20
Percent free oleic acid	5.75
Percent sulfur (total)	1.51
Color	Jet black

This experimental lubricant had the following composition:

	Per Cent by Weight
Litharge	5.50
Oleic acid	6.90
Black oil	86.9
Sulfur	.50
Stockholm tar	.2

In preparing this lubricant the oleic acid and PbO were heated together, the black oil added and the sulfur incorporated with the mixture at a temperature of about 250° F.

A test was made using the Pierce Arrow Hypoid rear axle, gear ratio 12 to 53, offset 2½ inches and a drive shaft speed of 1200 R.P.M., corresponding to a road speed of 25 miles per hour in reverse. The generator speed was 825 R.P.M., which gave an output of 60 KW. Starting with an oil temperature of 62° F., the run was continued for a period of 12 hours, after which the gears were inspected and were found to be in a satisfactory condition. The final temperature of the oil was only 98° F.

WH No. 0 HYPOID GEAR LUBRICANT.

This lubricant, having a Saybolt Viscosity of 99 at 210° F. and a pour test of 20° F., was approved as a successful hypoid lubricant. This product has the following tests:

Gravity	8.4
S.U.V. at 210° F.	105
Pour point, ° F.	35
Percent lead	4.87
Percent sulfur (total)	1.26

The Pierce Arrow hypoid gears having a ratio of 12 to 53 were used, the drive shaft rotation being in the forward direction at a speed of 1200 R.P.M. The generator R.P.M. was 835 and its output 60 KW. After running for a period of 1 hour and 55 minutes, the oil temperature had increased from 74° F. to 281° F. On inspection, the oil was found to be foamy and the condition of the gears satisfactory regardless of the high temperature produced.

In order to check the foregoing results a check test was made. Pierce Arrow gears were used, but the direction of rotation of the drive shaft was

reversed. The drive shaft speed was 1200 R.P.M., generator speed was 835 R.P.M., and the generator output 60 KW, as in the previous test. After a test period of 1 hour and 40 minutes the oil temperature, which was 85° F. at the start, had increased to 331° F., and on inspection the oil was found to be very foamy and the gears scored.

The conclusion is that the accelerated laboratory service tests are not very reliable for proving the effectiveness of hypoid gear lubricants.

Sodium Soap-Lead Soap S.A.E. 250 E.P. Gear Lubricants

FORMULAE.

The following formulae indicate the possibilities for incorporating lead oleate with other greases having soap bases for preventing leakage from gear cases:

Lubricant	1	2	3	4	5	6	7	8
Tallow	5.0	3.42	3.25	4.5	3.63	..	..	3.00
NaOH	.75	.51	.49	.67	.54	..	..	.46
180 S.U.V. at 210° S.R. stock	64.0	70.07	67.5	66.5	72.0	50	11	60.00
Sulfurized fatty oil	11.28	12.0	11.5	...	...	25	25	20.00
Lead oleate	.9425	1.0	4.95	4.93	5.0	..	..	1.54
650 S. R. stock	18.0	13.0	12.3	23.4	18.83	..	59	15.00
Sodium base gear grease	...	...	...	...	...	25%	..	...
Aluminum stearate	...	...	...	...	...	..	5%	...
Visc. of oil at 100° F.	7030	...	...	7050	...	..	..	...
MacMichael viscosity	245/76° F. 24/120° F.	360/76° F. 285/80° F.		135/65° F.	130/82° F.	30/85° F.		
Channel, ° F.				24 O.K. at 30				

Timken Test: 400 Feet per Minute

O.K.	17,140	....	20,000	....	20,000	22,260	22,860
Failed	20,000	....	25,710	....	22,860	25,710	25,710

Nature of Reaction Between Lead Oleate and Sulfur Dispersed in Mineral Oil

If basic lead oleate in mineral oil is heated to temperatures above 250° F. any or all of the following reactions may occur. Experiments carried out by the author, in November, 1929, indicated that in general the reactions will take place in about the order named:

I. $$2R_2Pb\cdot PbO + S = 2R_2Pb\cdot PbS + O_2$$
(R indicates oleic acid radicle)

II. $$\text{Mineral oil} + S = H_2S + \text{Mineral oil less } H_2$$

III. $$H_2S + R_2Pb\cdot PbS = \underset{\text{Oleic acid}}{2RH} + \underset{\text{Lead sulfide}}{2PbS}$$

Better stability is obtained if the temperatures to produce only the complex sulfide, indicated in Equation I, are maintained and the reaction so

regulated as to produce this material in a colloidal condition. With high temperatures the insoluble lead sulfide is formed and it precipitates as the lubricant stands.

In another method of manufacture the fatty radicle may be treated with sulfur, at elevated temperatures, in a separate vessel and then combined with solutions of lead oleate in mineral oil at substantially lower temperatures.

On February 18, 1932, four years after the above experiments were carried out, Gallsworthy [23] assigned to the Texas Company his patent dealing with a method of treating lead soap with sulfur to precipitate finely divided lead sulfide therein. He found that when a lead soap was treated with sulfur at a sufficiently high temperature the soap was decomposed by the sulfur with a precipitation of fine particles of lead sulfide. He preferred to employ only enough sulfur to precipitate a portion of the lead, thus permitting the remainder of the soap to remain in the lubricant. This investigator made use of lead oleate or lead fish oil soap and heated them together with mineral oil to a temperature of 300 to 350° F. Then, he added the sulfur and continued heating for about 3 hours. The quantity of sulfur employed ranged from 0.25 per cent to 5.0 per cent of the weight of the soap and would normally constitute about 1 per cent of the finished lubricant. The quantity of lead sulfide formed would represent about 7.5 per cent by weight of the original lead soap. In other cases, Gallsworthy preferred to prepare a soap base of this character and then blend it with a 70 S.U.V. at 100° F. mineral oil at compounding temperatures of 100 to 300° F. His finished lubricants for high pressure gear applications were intended to contain approximately 1 per cent of added sulfur. In one of his claims, a lubricant consisted of lead soap and 0.25 to 5 per cent sulfur, as lead sulfide, dispersed therein. The lead sulfide is mentioned as having a particle size of 8 to 25 microns.

Many early patents have been granted covering compositions of lead soaps and various other lubricants. In one,[24] a compound of lead, such as lead oxide, was heated with animal fat and mineral oil was then added. In 1873, Trossin [25] proposed the use of alloys of lead, tin, bismuth, and cadmium for the lubrication of engine pistons subjected to the action of superheated steam. Stratford [26] has produced lubricating compositions in accordance with the following ranges:

Lead oleate or other heavy metal soaps	1 to 10 per cent
"Solubilizing" agent of a phenolic nature	1 to 3 per cent
Viscous mineral oil as required.	

Abrams [27] assigned to Arthur Ballard his patent for a lubricant, for use under extreme pressure conditions, formed from free sulfur particles suspended in mineral oil by means of freely mobile jel particles of soap. It was

[23] U. S. Patent 1,957,259 (May 1, 1934).
[24] U. S. Patent 932,855.
[25] British Patent 3,401 (Oct. 21, 1873).
[26] U. S. Patent 2,031,986.
[27] U. S. Patent 1,913,300 (June 6, 1933).

claimed that this lubricant was satisfactory for heavily loaded gears, particularly worm gears and hypoid gears.

Lead-Fish Oil Soap Base Hypoid Gear Lubricant

Formula	Per Cent by Weight
Lead-fish oil soap	22.0
Flowers of sulfur	2.0
125 S.U.V. at 210° Naphthene Bright Stock	75.5
Stockholm tar	0.5

The properties of this compound are:

Gravity (1.005 sp. gr.)	9.3° Bé.
S.U.V. at 210° F.	148
Pour point, ° F.	20
Channel point, ° F.	10
Percent lead (Pb)	5.72
Percent sulfur	1.82
Color	Green-black

The cost of this product is estimated as follows:

Per Cent by Weight		Cents	Cents
22.0	Lead soap	22 lbs. at 16.50 per lb.	363.000
2.0	Flowers of sulfur	2.0 lbs. at 5.0 per lb.	10.000
75.5	B. stock	9.69 gals. at 10.0 per gal.	96.900
.5	Stockholm tar	.0563 gal. at 42.542 gal.	2.394
	Cost of materials for 100 lbs.		472.294
	Cost of materials for 1 lb.		4.722
	Cost of compounding 1 lb.		0.660
	Total cost of 1 pound ex-kettle		5.382

Chemical Analyses of Hypoid Gear Lubricants (1929-1930)

The following are the results of analysis of various hypoid gear lubricants:

	WH-No. O	FL Lead Base Lubricant
Physical Tests:		
Appearance	Heavy black oil	Heavy black oil
S.U.V. at 210° F.	126	125
Odor	Tarry	Soapy
Sulfur	2.58	....
Composition (per cent by weight):		
Oil	67.0	79.2
Asphaltic matter (insoluble in petroleum ether)	9.0 (Sp. Gr. 1.072)	1.5
Combined fat	6.0	9.1
Water	Trace	Trace
Lead (PbO)	9.4	6.5
Insoluble carbonaceous matter	8.0	
Undetermined	0.6	3.7
Total	100.0	100.0

	WH-No. O	FL Lead Base Lubricant
Ash (as sulfates)	12.8	9.0
Lead ($PbSO_4$)	94.3	96.8
Iron ($Fe_2(SO_4)_3$	2.0	....
Acid insoluble	1.6	1.9
Undetermined	2.1	1.3
Total	100.0	100.0
Fatty Acid from Soap:		
Iodine value (Hanus)	80.7	90.9
Neutralization value (Mgms. KOH)	178	201
Melting point	32° C.	Liquid
Sulfur (S)	2.45	
Mineral Oil:		
Gravity, ° A.P.I.	18.3	16.6
S.U.V. at 100° F.	961	1669

	DUPX Hypoid Compound
Physical Tests:	
Appearance	Black viscous oil
S.U.V. at 210° F.	146
Pour point, ° F.	50
Sulfur (S)	2.58
Odor	Pine tar
Composition (per cent by weight):	
Combined fat and free fatty acids	11.0
Oil (soluble in petroleum ether)	74.6
Moisture	.4
Asphaltenes (insoluble in petroleum ether) (soluble in CS_2)	5.6
Lead (PbO)	5.5
Organic matter (insoluble in CS_2)	2.0
Undetermined	0.9
	100.0
Mixed Fatty Acids:	
Iodine No. (Hanus)	134.1
Acid value (Mgms. KOH)	168
Appearance	Liquid, black
Sulfur (S)	0.75
Oil:	
S.U.V. at 100° F.	981
Gravity,° A.P.I.	18.1
Ash (Sulfates)	7.4
Lead ($PbSO_4$)	100.7%

	AM-Gear Lubricant
Physical Tests:	
Appearance	Black, viscous oil
Pour point	20° F.
Odor	Tarry
S.U.V. at 210° F.	167
Total sulfur (S)	2.52*

* This material when acidified with a mineral acid liberates some hydrogen sulfide.

	AM-Gear Lubricant
Composition (per cent by weight):	
Combined fat	9.25
Moisture	None
Mineral oil	81.80
Asphaltic matter (soluble in CS_2) (insoluble in petroleum ether)	4.40 (5.21% sulfur)
Lead (PbO)	4.10
Undetermined	.45
Total	100.00
Ash (Sulfates)	5.5
Silica (SiO_2)	2.0
Iron ($Fe_2(SO_4)_3$)	.9
Lead ($PbSO_4$)	95.4
Undetermined	1.7
Total	100.0
Fatty Acids from Combined Fat	
Acid value (mgms. KOH)	197
Iodine value (Hanus)	96.0
Color	Black
Percent sulfur (S)	0.38
Consistency at 60° F.	Liquid
Mineral Oil:	
Gravity, ° A.P.I.	17.8
S.U.V. at 100° F.	1141
Percent sulfur (S)	2.51

	WH Compound No. 52
Physical Tests:	
Gravity at $\frac{60° \text{ F.}}{60° \text{ F.}}$	1.09
Color	Black
Flash (o.c.)	465
S.U.V. at 210° F.	712
Pour point, ° F.	60
Odor	Pine tar
Composition (per cent by weight):	
Combined fat	11.0
Oil (by difference)	70.2
Lead (PbO)	12.7
Asphaltic matter (insoluble in petroleum ether)	4.0
Filler	2.0
Water	0.1
Total	100.0
Mineral Oil:	
Gravity, ° A.P.I.	18.1
S.U.V. at 210° F. (approx.)	100
Fatty Acids from Soap:	
Acid value (mgms. KOH)	196
Iodine value	84

	WH Compound No. 52
Ash:	13.0
Lead (PbO)	96.0
Undetermined	4.0
Total	100.0

Lead Naphthenate Base Hypoid Gear Lubricant

In view of the greater solubility and stability of blends of lead naphthenate with mineral oils as compared with certain lead oleate compounds, some manufacturers have preferred to use the naphthenate in compounding their hypoid gear lubricants. A typical formula would be:

	Per Cent by Weight
Lead naphthenate	18.0
Sulfur	1.5
1000 S.U.V. at 100° F. black oil	80.0
Stockholm tar	.5

The sulfur is first dissolved in the black oil by heating to about 250° F., after which the oil is cooled and blended with the balance of the ingredients at temperatures below 200° F. to prevent excessive reaction between the sulfur and the lead soap which would produce lead sulfide.

Notes on Hypoid Gear Lubricant

Many Hypoid Gear Lubricants foam badly in use. This causes leakage around the grease retainer washers and in some instances the lubricant has leaked onto the brake linings with objectionable results. The usual remedy is to keep the level of lubricant in the rear axle housing one inch or more below the filling hole. It seems likely that in some hypoid lubricants free fatty acids may be liberated by the action of the sulfur on the lead soap, forming lead sulfide which separates, in addition to free oleic acid.

Some hypoid greases do not foam in use when uncontaminated, but when mixed with other lubricants foam objectionably, possibly due to contamination with fatty acids. It is likely that the fatty acids greatly affect the surface tension and tend to increase the tendency to form aero-sols.

Results of tests made by several automobile manufacturers have shown that lead base hypoid gear lubricants are very effective in quieting noisy spiral bevel gear rear axles.

In one case a lubricant was made consisting of a blend of 12 per cent by weight of sulfurized lard oil (8 per cent by weight sulfur), 47 per cent 650 Pennsylvania steam refined stock and 40 per cent 100 S.U.V. at 100° F. pale oil with 1 per cent of lead fish oil soap. This product had high film strength, but after standing about one month both the lead soap and sulfurized fatty oil had precipitated to the bottom of the containers. There was some evidence that the lead soap was acting as a catalyst and greatly accelerating oxidation of the fatty oil, thus causing it to assume a condition which rendered it insoluble in this particular mineral oil blend.

In 1928 C. G. Williams [28] reported his experience with a lead oleate lubricant on a 4-inch spindle on a grinder. This bearing finally exploded and after replacement it was noted that there were further tendencies toward explosions and formation of sparks. It was reported that the total pressure on these bearings reached values of 96,400 to 385,360 pounds, and that in the case of a 9-inch shaft the surface velocity was 1250 feet per second. As a possible explanation of the explosion it was suggested that one of several explosive lead compounds might have been formed; for instance, lead hyponitrite, $PbNO_2$; lead nitrohydroxylamine, PbN_2H_3; lead imide, PbNH; lead azide, PbN_4; and lead trinitride. Since the foregoing loads and speeds will never be encountered in automotive lubrication practice it is not to be expected that any of the present lead base lubricants utilized in rear axles should ever cause explosions.

LEAD BASE LUBRICANTS FOR WORM GEARS

In view of their tendency to oxidize, lead soaps should not be generally used as ingredients of worm drive lubricants. It is known that localized temperatures in worm gears may readily attain values in excess of 500° F. Under such conditions hydrocarbon lubricants are prone to oxidize, and if it is true that lead salts catalyze oxidation reactions then the reason for difficulties which may be experienced should be clear. However, in some tests carried out with a Leyland worm drive rear axle operating under torque values up to 2000 pounds-feet, a lead soap oil combination was found to be superior to mineral oil and nearly the equivalent of pure castor oil. The oils tested had the following properties:

	Mineral Oil	Castor Oil	Lead Soap Compounded Oil
S.U.V. at 120° F.	1006	582	843
S.U.V. at 200° F.	158	35	123
Specific gravity	0.906	0.958	0.997
Ash	0.02	Nil	4.83 (Lead 83 per cent)
Percent sulfur	.....	.....	1.7

The results of efficiency tests made in the worm gear are tabulated below:

Worm Shaft R.P.M.	Worm Shaft Torque lbs./ft.	Worm Shaft H.P.	Efficiency of worm drive — Mineral Oil	Castor Base Oil	Lead Compounded Oil
131	600	15	.878	.897	.878
135	800	20.5	.880	.803	.890
138	1000	28.5	.883	.800	.886
143	1200	38.7	.890	.813	.896
147	1400	39.1	.890	.820	.900
153	1600	46.6	.880	.928	.907
159	1800	54.6	...	.931	.913
168	2000	63.2	...	.931	.916

These tests indicated that mineral oils would not carry the loads safely permitted by compounded oils. It appears evident that the temperatures

[28] *Mech. Eng.*, 129 (Feb., 1932).

developed when using castor oil would be considerably lower than for mineral oil, and possibly lower than for the oil compounded with lead soap.

Evidence in this country has indicated that the addition of acidless tallow oil, or lard oil, to steam refined cylinder stocks improves their lubricating properties for use on steel and bronze combinations. As little as 5 per cent of the saponifiable oil produces an improvement, but 10 per cent causes even more superior results. Sulfurized fatty oils are no doubt of some value in worm gear applications. Chlorine compounds have been found advantageous with some bronzes, but serious trouble has been experienced with other bronzes containing lead when chlorine compounds were added to the lubricant being used.

LEAD BASE COMPOUNDS CONTAINING SOLID LUBRICANTS

Many combinations of either normal or basic lead salts of fatty acids may be made with lubricating oils, sodium, calcium, or aluminum soaps, together with solid lubricants such as graphite, bentonite, asbestos, and mica. In 1886, Dewrance [29] proposed a mixture consisting of two parts of graphite or soapstone with one part of lead soap as a lubricant for steam cylinders. Cothias [30] developed a lubricant which he claimed was capable of transmitting electricity, consisting of graphite, lead oxide (or lead ore containing iron sulfide), metallic lead, a wax, and stearin. Paddock [31] prepared a lubricant having extreme pressure properties by mixing together graphite, castor oil, mineral oil, white lead, sulfur, and lead acetate. According to the inventor this product was capable of greatly reducing friction and cooling bearings which were running hot on account of overloading or improper design. Hutton's [32] grease was prepared with tallow, linseed oil, sulfur, graphite, white lead, and petrolatum. In 1934, Brunstrum [33] developed a grease suitable for use in lubricating plug valves. Its composition may be indicated by the following ranges:

	Per Cent by Weight
Graphite	35–65
Anhydrous calcium soap	2–15
Lead mahogany soaps	15–45

Hendrick [34] in 1877, was well aware of the lubricating value of lead soaps. His lubricating grease consisted chiefly of lubricating oil with a minor portion of the lead soap of whale oil. He made use of a fire heated retort, placing in it about equal parts of lead carbonate and whale oil. He then applied heat while stirring and noted that as the heat increased to about 250° F. considerable foaming took place and that at 350 to 400° F. the reaction was substantially complete and the product was clear and amber in color. At this stage he incorporated the mineral oil. Hendrick also

[29] British Patent 2,178 (Feb. 15, 1886).
[30] British Patent 4,561 (Mar. 9, 1900).
[31] British Patent 9,973 (May 30, 1900).
[32] U. S. Patent 1,568,606 (Jan. 5, 1926).
[33] U. S. Patent 1,982,200 (Nov. 27, 1934).
[34] U. S. Patent 197,129 (Nov. 13, 1877).

carried out the reaction between red lead and animal or vegetable oils at 380 to 400° F. for the preparation of lubricating bases. He found, in these cases, that the reaction would become exothermic, requiring careful temperature control to avoid sudden increases in temperature amounting to 100° F. or more.

Lead Base Railroad Lubricants

Railroads have long recognized the lubricating value of lead oleate and lead soaps in their locomotive journal box greases, their car journal box oils, and even in valve oils.

FORMULA FOR UP SUMMER CAR OIL

	Per Cent by Volume
65 S.U.V. at 210° F. lubricating distillate	97
Normal lead oleate	3

SPECIFICATIONS FOR UP SUMMER CAR OIL

Flash, ° F.	350 min.
Fire, ° F.	400 min.
S.U.V. at 210° F.	60– 65
S.U.V. at 130° F.	310–320
Pour point, ° F.	35 max.
Copper strip corrosion test	O.K.
Percent insoluble in P.E.	0.2 max.
Percent volatile material	1.5 max.
Percent PbO	.75 min.

FORMULA FOR UP CAR OIL WINTER

This product is similar to the Summer Grade oil with the exceptions as noted below:

	Per Cent by Volume
350 S.U.V. at 100° F. lubricating distillate	97.5
Normal lead oleate	2.5

SPECIFICATIONS FOR UP CAR OIL WINTER

Flash, ° F.	275 min.
Fire, ° F.	325 min.
S.U.V. at 210° F.	45– 50
S.U.V. at 130° F.	135–145
Pour point, ° F.	10 max.

"Galena" Car Oils (Lead Soap Journal Grease)

In 1909, Gill [85] described the use of journal oils which were combinations of mineral oils, animal oils, and suitable oxides and carbonates of lead. He noted that, in some instances, the uncombined lead inorganics would separate from the oil and proposed to increase the consistency of such lubricants to a point where objectionable separation would not take place. A typical formula reported by Gill was:

[85] U. S. Patent 932,855 (Aug. 31, 1909).

	Pounds
Fish oil	750
Lead oxide (or carbonate)	600
Tallow	14,000–28,000

The fish oil and lead oxide were stirred together and heated to 200 to 300° F. At this temperature the desired amount of tallow was added, while stirring, and the material allowed to cool. According to Gill,[35a] various grades of mineral lubricating oil were introduced into cakes of the above product in a non-homogeneous manner. The lead soap grease cake was provided with a large number of holes, or cells, which opened onto the surface of the grease cake. These held the oil which was fed thereby to the bearing surface. Penniman [36] has proposed journal box oils consisting of mineral lubricating oils of the lowest practical viscosity mixed with about 10 per cent of lard, or other animal oil of great adhesive ability, or with palm or castor oil, or with metallic soaps such as lead oleate. Sulfurized oils or oxidized hydrocarbons of high film strength were also considered applicable.

[35a] U. S. Patents 756,176 and 787,915.
[36] British Patent 306,532 (Feb. 23, 1928).

Chapter X

Miscellaneous Metallic Soap Base Greases

ZINC BASE GREASES

Zinc soaps are very seldom incorporated with lubricating oils to produce greases without the addition of other soaps, as they have little ability to thicken the oil. Their chief purpose is to modify the properties of soda soaps to give a smoother grease with better lubricating properties.

Zinc soaps are used in pharmacy; in the preparation of certain varnishes; and for the waterproofing of leather, canvas, textiles, etc.[1] Zinc oleate may be prepared by dissolving 64 parts of sodium oleate in 480 parts of water. Thirty-two parts of zinc sulphate are dissolved in 64 parts of boiling water. These two solutions are then stirred together, when the zinc soap will be precipitated. The precipitate should be washed with hot water until free from sodium sulfate.

Zinc stearate is made by a similar process by mixing a boiling solution of 100 parts of zinc acetate with a similar solution of 279 parts of sodium stearate. A white insoluble product is formed, which may be dried and ground to give a fine, white powder.

Mixed Soda-Zinc Base Transmission Lubricant

Formula

	Pounds	Per Cent by Weight
Animal fatty acids	900	8.504
Zinc oxide	75	.708
Caustic soda, solid	107	1.011
Water	21	.200
Asphalt base black oil, 410 vis. at 100° F.	6642	62.761
Heavy asphalt base steam refined stock	2834	26.778
Oil of citronella	4	.038

PROCEDURE:

The animal fatty acids are melted in a steam jacketed grease mixer.

The zinc oxide, separately worked to a smooth paste with a small amount of the lubricating oil mixture, is added to the kettle.

Forty-three gallons of 30° Bé. caustic soda solution is then run in, while agitating.

The temperature is brought slowly to about 300° F., the batch expanding until the kettle is practically full. After heating at this temperature for several hours the mass "breaks," indicating that practically all of the moisture has been evaporated.

The remainder of the oil is run into the batch slowly, maintaining a temperature of 300° F. If low pressure steam is used in the kettle jacket, several days may be required to complete the batch.

After all of the oil has been added a small sample may be taken for test. If the product is satisfactory, the batch is cooled while stirring to a temperature of 140° F., when it may be drawn into containers.

[1] French Patent 322,788; French Patent 368,755; German Patent 194,726.

Some manufacturers incorporate a small amount of zinc soap with heavy calcium base greases. Such greases are known to give satisfactory service for the lubrication of crankpins on donkey engines used in logging and construction work. The zinc soap may be produced either by the action of zinc oxide on fatty acids or by treating a solution of soluble alkali soap with zinc acetate.

Jarman's Zinc Soap Base Lubricant

In 1927, Jarman [1a] discovered that the addition of small amounts of the zinc soap of degras would appreciably lower the pour point of waxy motor oils. His soap was prepared: (1) directly, by mixing zinc oxide and degras together and boiling until saponification was nearly complete or (2) indirectly, by the usual double decomposition procedure. In the latter case, he saponified the degras with sodium hydroxide and dissolved the soap in water. Zinc sulfate solution was then added, the zinc soaps were precipitated and then washed to remove sodium sulfate. A stock solution of 10 per cent of the zinc-degras soap was then made with mineral oil. Ten per cent of this base when added to a waxy Mid-Continent oil would reduce its pour point from 35° F. to 0° F., according to the inventor, the final blend containing 1 per cent of the zinc degras soap. He claimed, however, lubricants containing as much as 10 per cent of the zinc degras soap. This idea has possibilities for the development of automotive gear lubricants of low channeling characteristics.

Miscellaneous Zinc Base Lubricants

In 1887, Wass [2] prepared a complex lubricant by dissolving rosin and stearin in mineral lubricating oil and heating together with zinc oxide and lead oxide. Further saponification with sodium hydroxide was suggested if a more consistent lubricant was required.

Mond [3] found that certain dyes when added to lubricating oils would separate on standing. To stabilize such mixtures he proposed the addition of water insoluble soaps such as zinc, calcium, magnesium, or aluminum salts of oleic, stearic or benzoic acids.

For reducing the pour point of waxy motor oils, and increasing their lubricating value, zinc or magnesium salts of acids obtained from the oxidation of paraffin wax has been proposed by Sullivan.[4]

Zinc oleate has also been suggested as a means of promoting the miscibility of mixtures of mineral oil and castor oil. MacLaren has assigned to the Standard Oil Company of Indiana [5] his patent for this idea in which mineral oil and a solubilizing agent such as the oleate or stearate of zinc, aluminum or magnesium are blended together. The blend is heated at 150

[1a] U. S. Patent 1,767,076 (June 24, 1930).
[2] British Patent 3,833 (March 14, 1887).
[3] British Patent 357,179 (June 17, 1930).
[4] U. S. Patent 1,789,026 (Jan. 13, 1931).
[5] U. S. Patent 2,034,405 (March 17, 1936).

to 175° F., cooled to about 38° F., and then mixed with the vegetable castor oil.

BARIUM SOAP BASE GREASES

The barium salts of the higher fatty acids are hydrolized by water. They have not been used extensively as lubricating greases bases in view of their relatively high cost and lack of specific points of superior service qualities. The stearate and palmitate are substantially insoluble in alcohol. The oleate, stearate or palmitate may be readily prepared by usual double decomposition methods, although for grease making it has been found more expedient and less costly to make use of barium hydrate, combining it directly with fatty acids or fats.

Deguide's Barium Soap Base Grease

Deguide[6] appears to be the first to recognize the full importance of lubricating greases prepared with barium soap bases. He has, in 1932, proposed a soap for use in manufacturing a consistent lubricating grease made by saponifying a fat with crystallized barium hydroxide, $Ba(OH)_2 \cdot 8H_2O$. Rape seed oil, tallow, or a mixture of these, is heated under pressure in an autoclave with the calculated theoretical quantity of barium hydroxide to effect substantially complete saponification. The resulting soap may then be dissolved or emulsified with mineral oil to form the desired lubricating grease.

Ott, Clarke, Marter,[7] Barium Soap Base Grease

These inventors have assigned their patent to the Union Oil Company. It is somewhat similar to that above but involves a different manufacturing technic.

A saponifiable fat is treated with an excess of barium hydroxide, sufficient to produce a preponderant proportion of basic barium soap. This reaction is carried out at an elevated temperature and for a time sufficiently long enough to effect complete saponification. Mineral oil, having good solvent properties with respect to basic barium soap, is then added. A fatty acid, oleic or stearic, is then added to neutralize any excess barium hydroxide remaining in the grease. Then the lubricant is cooled to a temperature suitable for the incorporation of water, after previously heating for further formation of basic barium soap. The hydration is apparently quite important, as in the case with calcium base greases, and the batch must be handled with equal care. In this lubricant the proportions of materials used are controlled so that the finished grease contains a major portion of lubricating oil and a minor proportion of the basic barium soap. No comparable data is available to indicate the relative yields for greases made with similar quantities of the barium soap and normal calcium soaps.

[6] British Patent 398,402 (Feb. 14, 1932).
[7] U. S. Patent 2,033,148 (March 10, 1936).

MAGNESIUM SOAP BASE GREASES

Magnesium Stearate Base Lubricants

For more than ten years magnesium stearate has been offered to the grease making industry at a price of about 25 cents per pound. On account of this relatively high cost its popularity with most grease manufacturers has not been great. The methods of manufacture, when using magnesium stearate, do not differ greatly from those in use when aluminum stearate is the base used. In most instances, however, particularly when making soft greases, chill pans may be dispensed with. It appears that magnesium stearate does not impart as high melting points as other soap bases; for instance, calcium and aluminum. A normal magnesium stearate base grease containing about 7 per cent by weight of magnesium stearate and made with 100 S.U.V. at 100° F. pale oil will have a melting point ranging from 140 to 150° F.

The percentage of magnesium stearate normally required to make a grease having a worked A.S.T.M. Penetration of 200 to 300 at 77° F. is 5 to 6, which is approximately .40 pound per gallon of oil. The oil may be mixed with the stearate in a grease kettle without heating. When the soap is thoroughly dispersed the mixture is heated to 320 to 350° F. and maintained within this range for (from) one half to one hour. The grease may be run directly into drums which may require (up to) four days to cool or preferably, for the more consistent grades, chill pans may be used with a cooling period of (about) 24 hours.

The magnesium stearate should completely dissolve at about 200° F. but a temperature of 350° F. appears necessary to secure greases of satisfactory transparency. Oils ranging in viscosity from 75 S.U.V. at 100° F. to 1000 S.U.V. at 100° F. may be employed for making magnesium stearate greases.

Magnesium stearate, in common with many other metallic oleates and stearates, has been found useful in lowering the pour point of waxy motor oils. Beim [8] has proposed the heating of a blend of two different grades of lubricating oil maintained under a vacuum of 10 pounds, at a temperature of 425° F., to remove volatile fractions. While still hot, from 0.3 to 1.0 per cent of magnesium stearate is added and the mixture agitated and cooled. It is then filtered and drawn off for use.

An unusual use of a lubricant has been patented by Ducamp.[9] He discovered that the addition of mercury cyanide to lubricating oil used in an internal combustion engine reduced detonation. He found, further, that fatty acids, preferably oxidized, or alkali or alkaline earth soaps, such as calcium or magnesium oleate, had a similar action.

Fell [10] has developed textile fiber lubricants consisting of oils emulsified with ammonia and fats treated with magnesium chloride.

[8] U. S. Patent 1,784,809 (Dec. 16, 1930).
[9] British Patent 338,780 (Feb. 16, 1929).
[10] British Patent 13,580 (July 1, 1905).

Carpmael [11] has discovered that the chemical combination of magnesium and methyl alcohol known as magnesium alcoholate has the property of gelatinizing fatty and mineral oils, thus forming greases. In examples, 120 pounds of methyl alcohol, containing 0.5 to 1.0 per cent of water, is mixed with a solution of 1 pound of magnesium in 30 pounds of methyl alcohol; and 1 to 2 pounds of magnesium alcoholate solution is added to 10 pounds of castor oil, and 0.1 pound of water, dissolved in a little alcohol, is stirred in.

LUBRICANTS CONTAINING MISCELLANEOUS METALLIC SOAPS

Chromium oleate has been proposed by Griffith [12] as an ingredient of motor oils for the prevention of sludge formation in the crankcase. Less than 1 per cent by weight is recommended and it is possible that such a soap has some effect on the oiliness characteristics of the lubricant and piston ring sticking. Chromium oleate is a useful ingredient of other lubricating greases where it has been found to be effective in reducing rusting and corrosion.

Iron soaps, such as iron oleate and iron stearate, have been suggested by Koethen [13] as a means of increasing the consistency of lubricants and improving their lubricating value.

To prevent motor oils from forming sludges, Helmore [14] has discovered that tin oleate, or the tin compounds of naphthenic acid, or ricinoleic acid, or tin phenyl or tin methyl iodide, when added to crankcase oils are effective agents. Lead compounds proposed by Helmore have already been discussed.

Yacco,[15] in a search for agents which would increase the thermal conductivity of internal combustion engine cylinders, developed the following lubricant:

	Pounds
Ammonium oleate	1
Ammonium stearate	1
Copper oleate	½
Mixed vegetable oils	7.75
Mineral oil	90

The mercury salts of naphthenic acids have been found to be useful antiknock compounds. Ducamp [16] has developed the method of dissolving mercuric oxide in naphthenic acid, after which the mecuric naphthenic is dissolved in lubricating oil and used for the lubrication of internal combustion engines.

Boner [17] has studied various soaps of stearic acid and lard oil. He

[11] British Patent 259,431 (Dec. 29, 1925).
[12] U. S. Patent 2,032,279.
[13] U. S. Patent 1,758,446 (May 13, 1930).
[14] British Patent 398,222 (March 9, 1932).
[15] British Patent 395,867 (June 24, 1932).
[16] British Patent 368,025 (Feb. 4, 1931).
[17] *Ind. Eng. Chem.*, **29**, 59 (1937).

concludes that four metals (aluminum, cobalt, magnesium, and nickel) are capable of producing clear or transparent lubricating greases. He found that the soaps of cadmium, calcium, lead, mercury and strontium normally resulted in opaque greases. Since he prepared greases with 10 per cent of these soaps, and the first group of four soaps were of metals having relatively low atomic weights, it was concluded that the low metal content will tend to improve transparency. Eight soaps were found to be unsatisfactory with regard to grease forming characteristics; these were barium (the basic soaps of this metal have been found satisfactory by other investigators), cerium, chromium, copper, iron, manganese, silver, tin and zinc. It appears that, in general, divalent soaps of various metals are superior from the standpoint of grease formation to trivalent metals. It will be recalled that there is some doubt that the aluminum stearate utilized in making commercial lubricants is aluminum tri-stearate but is probably a mixture of the basic soaps, mono- and di-stearates.

Boner, in studying the above soaps and in incorporating them with oils, utilized temperatures of 230 to 260° F., dehydrated soaps, and greases containing 0.5 per cent water, and others containing 0.5 per cent of stearic acid. It is possible that further research on utilizing basic soaps would result in more promising products.

Chapter XI

Inorganic-Hydrocarbon Lubricants

In this chapter those lubricants consisting of various solid lubricants dispersed in hydrocarbon lubricants will be discussed. Powdered or colloidal metals, soapstone, mica, asbestos, clay, inorganic salts, powdered alloys, graphite, and talc, are examples of the inorganics which will be considered. While, in general, these inorganics act as lapping agents and tend to increase the rate of wear of anti-friction bearings, severe operating conditions have forced their use in many cases. Thus, in tractor roller lubrication we find certain bearings equipped with spiral type rollers. Asbestos filled lubricants in actual service have been found to greatly prolong the life of such bearings; even though laboratory wear tests indicate more wear, due to the presence of asbestos, than for a normal petroleum oil with or without a dispersed soap. This apparent anomaly may be explained by the fact that the asbestos acts as an extreme pressure lubricant, thus preventing scoring when enormously high loads are imposed on a roller, whereas a normal lubricant would permit scoring to take place. Other solid inorganic lubricants may be considered to act in a similar manner, and for this reason are worthy of careful consideration. It will be noted that many of the lubricants of the past 75 years fall in this classification, and that for more modern lubricants the tendency has been to eliminate the solid lubricants, apparently on account of their more or less abrasive characteristics. However, with the advent of the hypoid gear, overloaded bearings due to increased production, and certain types of modern design, lubricating engineers are again carrying out researches with the object in view of utilizing the desirable characteristics of the solid lubricants and eliminating their objectionable features.

It is likely that the trend toward better utilization of solid lubricants will be in the direction of producing them in colloidal sizes, thus decreasing the dangers due to abrasion. It is possible that there will be discovered a range between the conditions where the chemically active extreme pressure lubricants are beneficial, and normal fluid film lubrication, where colloidally dispersed solid lubricants will prove most advantageous. The development of colloidal graphite and the general recognition of its superior quality as compared with fine flake graphite, or most ground graphite, may be considered as an example of what may follow for other solid lubricants when satisfactory means of dispersing them in a colloidal condition have been developed.

While it is fully recognized that graphite is a form of carbon, and according to proper chemical nomenclature should be classed as an organic material, it has for the sake of convenience been placed in this chapter along with other solid lubricants of definitely inorganic character.

The greases considered in this chapter are further characterized by depending on the dispersion of solid materials in a finely divided state to produce yield value, and in nearly all instances contain no soap. The greases discussed in the following sections may be more properly termed lubricating pastes, than unctuous lubricating greases.

ALUMINUM LUBRICANTS

It is believed that Spence,[1] in 1919, was first to seriously consider the use of powdered aluminum as an ingredient of lubricating greases for machinery bearings. Spence preferred finely divided aluminum such as is made by grinding and pounding in the presence of stearic acid. In examples of his lubricants aluminum powder was mixed with water, animal, vegetable, or mineral oils, greases, emulsions, or solutions of soap. He suggested the use of a deflocculating agent or combinations of aluminum with mica, graphite or other solid lubricants. A typical formula developed by Spence is:

	Pounds
Powdered aluminum	5
Petrolatum (or 47.5 pounds petrolatum and 47.5 pounds lubricating oil)	95

In another formula Spence recommended a compound consisting of 5 per cent by weight of powdered aluminum with 95 per cent of solidified oil containing either calcium or sodium soaps. It appears likely that even though the powdered aluminum in these formulae may be of colloidal size, the lubricants when applied to Babbit bearings may exert an objectionable lapping and wearing action.

ANTIMONY LUBRICANTS

Archer, Hardy, and Archer,[2] in 1886, developed the following lubricating composition:

	Pounds
Tallow	67
Petroleum oil	45
Black antimony (Sb_2S_3)	1
Gum camphor	1

ASBESTOS LUBRICANTS

It is evident that asbestos has been used as an ingredient of lubricating compositions for approximately 75 years. A fluid lubricant was manufactured by Colby,[3] in 1865, and consisted of a mixture of asbestos, graphite, ether, and other solvents.

In 1875, Otto[4] patented his anti-friction compound consisting of

[1] British Patent 158,922 (Aug. 16, 1919).
[2] British Patent 14,835 (Nov. 16, 1886).
[3] U. S. Patent 49,983 (1865).
[4] U. S. Patent 169,031 (Sept. 29, 1875).

asbestos, graphite, glycerin, bolus, paper pulp, talc, and condensed linseed oil. Another early lubricant [5] was composed of asbestos, glue, rosin, and vegetable fiber. A railway journal bearing lubricant having cooling characteristics was invented by Hyatt [6] and consisted of a mixture of powdered asbestos with palm oil or tallow. Hyatt [7] also prepared a dry asbestos paste mixed with petroleum lubricating oil. After studying the galvanic action taking place between the piston rod and stuffing boxes of steam engines, Gedge [8] developed his "anti-galvanic" compound consisting of fish or whale oil, iodine dissolved in alcohol, asbestos, and mica. Swain's [9] lubricant consisted of asbestos fiber, candle wax, and lubricating oil. Strickler [10] manufactured at Dodge City, Kansas, in 1892, a lubricating composition consisting of pulverized asbestos, talc, mica, and lubricating oil. Gregory and Williams [11] developed a fibrous lubricant base containing asbestos, powdered wood, and coconut fiber, impregnated with mineral lubricating oils.

In 1909, Hoy [12] patented a railway journal box lubricant quite similar to that developed by Hyatt in 1873. It consisted of a mixture of asbestos, graphite, and lubricating oil. Jackson [13] patented the following complicated mixture in 1914:

	Per cent
Natural asbestos	5 –50
Granular soapstone	2½–10
Lubricating oil or grease	90 –38½
Ground borax	2½– 1½

Asbestos lubricating compositions similar to those marketed today were patented by Warrell,[14] in 1915. Warrell's process consisted of carrying out prolonged agitation of hot dry asbestos or soapstone with heavy lubricating oil, until a permanent magma was formed.

Tractor Roller Greases

A Mississippi Valley grease manufacturer has for many years manufactured a series of asbestos base lubricants intended for use in tractor rollers. These products are dispersions of asbestos floats in a steam refined cylinder stock of 180 to 200 S.U.V. at 210° F. Their formulae were:

Grade	0 Per Cent	1 Per Cent	2 Per Cent	3 Per Cent	4 Per Cent
Asbestos	4–6	9–11	17–20	23–26	43–46
Maximum free alkali	.15	.15	.15	.15	.30
Maximum free acid	.2	.2	.2	.2	.2
Ash	3.5–5.5	8–10	14–17	20–23	37–40

[5] U. S. Patent 368,636.
[6] British Patent 3,684 (Nov. 12, 1873).
[7] British Patent 478 (Feb. 5, 1874).
[8] British Patent 1,809 (May 26, 1875).
[9] U. S. Patent 424,233 (1890).
[10] U. S. Patent 476,574 (June 7, 1892).
[11] British Patent 28,077 (Dec. 22, 1904).
[12] British Patent 28,026 (Dec. 1, 1909).
[13] British Patent 30,080 (Dec. 31, 1914).
[14] U. S. Patent 1,133,204 (March 24, 1915).

Asbestos Base Tractor Roller Lubricant No. 1

Formula

650 Penn. S.R. stock 1020 gallons at $.2780	$280.50
Asbestos floats (JM) 2300 pounds at $.0425	97.75
Cost of materials	$378.25
Cost of materials per pound	3.78
Compounding cost per pound	.13
Cost of grease per pound of ex kettle	$ 3.91

The Asbestos Floats used in greases of this type may vary greatly in composition; samples from two different greases were analyzed with these results:

Sample	I Per Cent	II Per Cent
Acid insoluble material	39.1	55.0
Iron	6.7	21.4

Wear Tests

That tractor roller greases, prepared with pulverized asbestos, may cause increased wear in service, where they are not necessary, is indicated by the following Timken wear tests. Three asbestos greases were found to increase wear more than tenfold at an arbitrary pressure of 35,700 pounds per square inch; this unit pressure, of course, becoming much less as wear proceeded.

	Loss in Weight of Block First Hour Mg.	Loss in Weight of Ring First Hour Mg.	Total Loss in Weight First Hour Mg.	Loss in Weight of Block Second Hour Mg.	Loss in Weight of Ring Second Hour Mg.	Total Loss for Second Hour Mg.	Total Loss in Weight in two Hour Period Mg.
Commercial No. 3	6.1	8.4	14.5	5.1	4.7	9.8	24.3
JM No. 1 Grease*	5.4	7.7	13.1	2.9	3.7	6.6	19.7
SP Roller Grease	8.2	10.2	18.4	4.1	9.2	13.3	31.7
No. 2 Sodium Soap Grease	0.2	0.7	0.9	...	...	...	...

* Johns-Manville Asbestos Floats Grade No. 323, 23 per cent.
650 Pennsylvania Steam Refined Stock 77 per cent.

Conditions of Test:

Velocity of Timken machine	140 feet per minute
Pressure, pounds per square inch	35,700

The temperature of the grease was 140° F. at the start of the run. The temperature of the oil on the ring increased from 140° F. to 250° F. during the first fifteen minutes for the three sampels of asbestos greases. Grease No. 2, containing Latex, heated the ring only to 190° F. after 40 minutes of operation.

No. 2 Sodium Soap Base Grease (Latex)

Formula

	Gallons	Lbs. Net	Per Cent by Weight
Tallow	60	455.3	5.44
45° Bé. caustic soda	13.25	66.9	.80
1000 red oil	100	776.8	9.28
150 at 210° California bright stock	64	500.5	5.98
S.R. stock	696	5289.9	63.20
Floyd's E.P. base	151	1255.5	15.00
Latex	3.2	25.1	.30
		8370.0	100.00

Tractor Roller Grease JM-1

Formula

	Gallons	Lbs. Net	Per Cent by Weight
650 Pennsylvania S.R. stock	1020	7700	77.0
Johns-Manville asbestos floats		2300	23.0
		10,000	100.0

Soda Soap Tractor Roller Grease

Formula

	Per Cent by Weight
Dark fiber grease No. 3	42.35
Dark fiber grease No. 4	42.35
Floyd's base	15.0
Rubber latex emulsion	0.30

Tests

Penetration at 77° F. (unworked)	395
Penetration at 77° F. (worked)	435
Timken test at 400 R.P.M. Failed	30,000 pounds
Timken test at 400 R.P.M. O.K.	27,140 pounds

Tractor Roller Grease DA No. 0

Analysis

Gravity ° A.P.I.	24.5
Color	Black
S.U.V. at 210° F.	218
Flash, ° F.	580
Fire, ° F.	575
Ash, per cent	0.462

Note: The ash appears to be mainly finely ground asbestos.

Tractor Roller Grease DA No. 3

Analysis

Physical Tests:

Appearance	Smooth, stringy
Color	Very dark green
Odor	None
Penetration, A.S.T.M. (unworked) at 77° F.	350
Penetration, A.S.T.M. (worked) at 77° F.	370
Dropping point ° C.	200

Composition (per cent by weight):

Oil	78.8
Filler	20.6
Moisture	0.6
Free alkali	0.0
Total	100.0

Oil Extracted:

Color	Green black
Gravity ° A.P.I.	24.5
S.U.V. at 210° F.	218
Flash ° F.	580
Fire ° F.	665

Ash:

Total percent on original	20.6

The ash appears to be all filler, which resembles finely ground asbestos.

SN Sodium Base Tractor Grease (1932)

Analysis

Physical Characteristics:

Appearance	Black tacky fiber grease
Odor	Soapy
Dropping point, Ubbelohde, ° C.	123

Composition (per cent by weight):

Soap	4.82
Free alkali (as sodium hydroxide)	0.08
Petroleum ether soluble, principally petroleum oil	83.03
Petroleum ether insoluble but benzol soluble (principally hard asphalt)	4.60
Benzol insoluble (carbonaceous matter)	6.60
Undetermined	0.87
Total	100.00

Ash (per cent by weight):

Sodium as sodium carbonate (Na_2CO_3)	69.63
Sodium sulfate (Na_2SO_4)	26.97
Iron as Fe_2O_3	4.00
Gain in analysis	0.60
Total	100.00

ZN Tractor Roller Grease

Analysis

Physical Tests:

Appearance	Light greenish brown
Odor	Fatty
Penetration at 77° F. (cone unworked)	371
Penetration at 77° F. (cone worked)	347
Dropping point ° C.	85

Composition (per cent by weight):		
Soap		11.60
Oil		86.81
Moisture		0.75
Free alkali		0.07
Free fat		0.29
Insoluble (fillers)		0.01
Gain or loss		0.47
	Total	100.00
Mineral Oil Extracted:		
Color		Opaque
Gravity ° A.P.I.		20.7
S.U.V. at 100° F.		335
Ash (total percent on original):		1.24
Acid insoluble		Trace
Iron and aluminum as R_2O_3		1.41
Calcium oxide as CaO		91.00
Undetermined		7.59
	Total	100.00
Fatty Acids from Soap:		
Iodine value (Hanus)		52
Acid value (Mg. KOH)		207
Melting point ° C.		40
Color		Light brown

CARBON BASE LUBRICANTS

Lubrication engineers have noted that finely divided carbon, soot, pulverized coal, and coke are readily wetted by lubricating oils. There is some evidence to support the contention that the addition of such materials to lubricants will improve their film strength and tend to reduce bearing temperatures. Their abrasive properties, of course, must be taken into consideration. In 1873, Norris [15] made a lubricating compound by pulverizing coal and mixing it with lubricating oil, petroleum residuum, talc, ammonium chloride, sodium hydroxide, and water. In 1893, another [16] United States patent was taken out covering compositions consisting of coal soot and lubricating oil. Pulverized bituminous coal dispersed in lubricating oil was also suggested as a suitable lubricant for machinery bearings by Ferguson.[17] Colloidal carbon, colloidal graphite, and naphthalene dispersed in xylene make an effective valve or top cylinder lubricant according to Wagner.[18]

CLAY BASE LUBRICANTS

Clays are ordinarily considered as the decomposition and disintegration products of various rocks. In general, they may be described as being the natural hydrated compounds of alumina and silica, and will normally become plastic when mixed with sufficient water. In most cases the object of

[15] U. S. Patent 145,812 (Dec. 23, 1873).
[16] U. S. Patent 496,742 (1893).
[17] U. S. Patent 1,531,913 (March 31, 1925).
[18] British Patent 405,145 (April 18, 1932).

lubrication engineering and bearing design is to prevent entry of earth and clay to bearings as they are known to greatly increase wear. However, for rough bearings, very finely divided clay has often been suggested as a base for various lubricating compositions. For finely finished bearings. or anti-friction bearings, lubricants containing clay are unsuitable. Steel mill lubricants on analysis will often reveal the presence of clays.

In 1869, a mixture of the following materials was marketed by Hull and Vail [19] as a lubricant and was no doubt suitable for much of the rough machinery used at that time:

White clay	1 pound
Graphite	2 ounces
Petroleum residuum	10 pounds
Paraffine wax	2 "

A mixture of elutriated kaolin, animal fat, lubricating oil, and an alkali was proposed by Smith [20] for lubricating purposes. Thomas [21] also proposed dry powdered China clay (kaolin) as a filler for lubricating greases. A mixture of putty powder with graphite and mineral oil has been suggested by Sandwith [22] as a lubricant for preventing friction between automobile inner tubes and casings. In 1919, Wheelock [23] assigned to the Swan Process Oil Company his patent for an anti-squeak compound consisting of an oil absorbing insoluble earthy base together with a low viscosity mineral oil to form an unctuous pasty mass. Sherrick,[24] in investigations of preparing clays to make them suitable as bases for lubricants, found that best results could be obtained by treating bentonite with strong acid to obtain gelatinous colloids. The finished filler offered to grease manufacturers consisted of this clay in combination with an emulsion of water and lubricating oil.

STEEL MILL GREASES

The manufacture of iron and steel rolled shapes is carried out in rolling mills, where the metal is passed through a train of rolls, composed of two or more "roll stands." The rolls are usually made of chilled iron or steel and are cylindrical; in the stands they are set one above the other, with their axes parallel, and are held in housings so that there is a measured space between them. The rolls are driven by motors or steam engines through a system of reduction gears, in such a way that they rotate in opposite directions. The gears are connected to the rolls by means of short shafts connected by coupling boxes. The rolls are provided with journals, or necks, as they are usually termed, which are nearly as large as the rolls themselves.

Iron and steel are rolled at temperatures which are high enough to soften the metal, and the rolling operation results in compression and elongation

[19] U. S. Patent 74,366 (1869).
[20] U. S. Patent 208,697 (Sept. 6, 1878).
[21] British Patent 1,187 (March 25, 1879).
[22] British Patent 13,037 (June 10, 1903).
[23] U. S. Patent 1,362,161 (April 7, 1919).
[24] U. S. Patent 1,758,265.

of the piece being worked on. Above the roll necks are placed bearings, or brasses, which constitute a quarter bearing. The brasses rest against the ends of the roll screws which are used to bring the rolls closer together when desired. When iron and steel shapes, or sheets, are being rolled the bearing pressures are tremendous, and good lubrication is imperative.

Reversing mills have only two rolls, one set above the other. After each pass of the metal through the mill the power is reversed so that the material is rolled back in the opposite direction.

Continuous or non-reversing mills have three rolls arranged in a vertical position. The material passes between the lower and middle rolls in one direction and returns between the upper and middle rolls. Bar mills, rod and wire mills, hoop and billet mills, are usually of the continuous type, having several stands of two rolls arranged in tandem, which in some cases are driven at progressively increasing speed. Rolling mills are designated by the name of the material which they manufacture.

The rolls used in the production of bars, rails, rods, structural iron and other shapes are equipped with water pipes, so arranged that a stream of water may be kept flowing over the roll necks to prevent overheating. The lubricant used for these necks is known as cold neck grease, and is generally a mechanical mixture of tallow or suet alone or compounded with various petroleum derivatives. In some cases the cold neck grease is packed directly against the necks, being contained in small burlap bags of about two pounds capacity. The grease feeds gradually by melting through the burlap to the neck surfaces.

The rolls used for making sheet steel, tin plate, sheet iron for automobile bodies, and sheets suitable for the purpose of being galvanized, are known as hot rolls. Usually, no water is employed to cool the hot roll necks, due to the nature of the work being done; consequently they run at very high temperatures. At times, however, small quantities of water are used on the ends of the necks to control the temperature.

Hot Neck Grease

The conditions to be met in the lubrication of the large hot roll bearings or necks are enormous pressures, suddenly applied, and very high temperatures, due to the heating of the rolls by contact with the heated metal which is being fabricated. On account of the large size of the necks the speeds of the bearing surfaces are comparatively high. Very little water can be used to cool the necks of these rolls, as it would cause irregular cooling of the sheets and rolls with consequent warping and uneven rolling.

To compensate for the increased expansion at the center of the roll, they are turned so as to have a slight concavity when cold. At working temperatures the rolls are very nearly parallel. Clearances of two typical rolls 40 inches long and 28 inches in diameter, used for rolling tin, are given below:[24a]

[24a] Iron Age, (Oct. 11, 1923).

Distance from end of roll Inches	Clearances between rolls (Operating temperature) Inches	Clearances between rolls (Cold) Inches
5	0.002	0.0078
10	.007	.0156
15	.009	.020
20	.009	.025

Sheets of tin and light gauge steel are usually rolled in packs of two or more sheets. When finishing a pack, if one neck of the roll be greased without greasing the other, the pack will always draw a long horn next to the neck that is dry. The reason for this is that the friction of the ungreased neck is greater and consequently heats and expands the roll on that side. The statement has been made in a report by the Carnegie Institute of Technology that from 60 to 80 per cent of the power required in rolling sheets is used in overcoming roll neck friction.

It is evident from a consideration of these points that the choice of lubricants used on hot necks is made with great care. After a user has found a satisfactory lubricant it is not to be expected that he would change readily to a product with which he is not familiar, as an inferior lubricant might cause the rolls to operate at a higher temperature, resulting in irregular roll clearances, and, consequently, the production of sheets of uneven thickness. Greatest trouble is experienced where the roll necks are of small diameter and subjected to sudden heating and expansion. Uneven expansion is the cause of many rolls being broken. Methods have been worked out to heat the rolls to operating temperatures by electricity.

In some tests to determine the operating temperatures of the cast iron rolls used for rolling tin, thermocouples were inserted in the roll and the following temperatures were obtained:

Location	Temperature range ° F.
Center of roll on surface	703–736
Center of roll	641–674
End of rolling surface	510–542
Bearing surfaces	330–447

In rolling steel plates it is often noticed that the rolls are blue or indigo in color. This corresponds to a temperature of about 559° F. The necks as indicated above are cooler than the center of the roll, but the grease is often seen burning on them.

Properties of a Satisfactory Hot Neck Grease

Melting Point. The melting point of a well known satisfactory hot neck grease before being used is 210° F. Another grease which is also used with satisfaction has a melting point of 410° F. These greases may be applied either by first melting the material and swabbing it on the roll necks or the product may be cut into bars approximately 2 inches by 2 inches by 10 inches, which may be placed on the roll neck, the heat causing it to melt and run onto the bearing surfaces. The latter method is the most popular. After being used the grease drips from the journal and cools in

pits below the rolls, where it may be collected at the end of a week's run, remelted in a fire-heated vat, poured onto a cooling floor of concrete, and allowed to cool in a layer 2 inches in depth. After the grease has chilled sufficiently it is cut into bars and stacked for future use. On repeated use hot neck greases show a progressive increase in melting point. For this reason new unused grease is usually incorporated with old grease before being used for the first time.

Since the temperature of the hot necks may vary from 350° to 550° F. the melting point of the grease will to some extent determine the number of applications for a given period of time. It is also evident that if the melting point of the grease is 450° F. satisfactory lubrication will not be secured if the rolls are operating at a temperature of 350° F. However, the rolls operate at the lower temperature for only a small part of the time, when beginning the week's work, and during this period fresh, soft grease is used. The melting point of the grease, for the most satisfactory service, should be approximately 400° F.

Flash Point. The flash point of a satisfactory hot neck grease should be not below 450° F.; preferably 500° F. or higher.

Consistency. The consistency of the grease should not be so high as to prevent lubrication at ordinary temperatures. The asphalt penetrations of new and used neck greases are given later. The A.S.T.M. penetration of a satisfactory used grease was 2, and the unused grease 42. Greases having a penetration of 70 before being used have been found to be satisfactory.

Adhesiveness. Satisfactory hot neck greases should have very high adhesive properties, particularly at high temperatures. It is believed that the function of the mineral filler and soap or pitch used is to prevent the melted grease from running off the bearing surface too rapidly. The grease should form a heavy lubricating film, not easily ruptured by high bearing pressures suddenly applied.

Water. It is necessary that the grease should be insoluble in water, as it sometimes comes in contact with moisture or cooling water. The grease should not contain water, as its presence tends to reduce the adhesive properties of the product, as well as cause foaming due to the formation of steam at the high operating temperatures of the necks.

Stability and Re-use. The grease should not be greatly altered in its physical characteristics under repeated use, as it is collected and re-used many times. Furthermore, the filler should not readily settle out when the grease is melted.

Materials. A search of the literature has shown that various mixtures of the following materials are used as hot neck lubricants:

Heavy mineral lubricating oil (500 flash or higher)	
Petroleum residuum	
Asphalt	
Pitch	Stearin (Candle tar)
	Wool (or Cholesterol)
Tar	Coal
	Pine

Rosin
Wool grease
Fillers.....Clay
Talc
Lime
Mica
Graphite
Soap
Calcium
Sodium
Magnesium
Aluminum

Properties of Metallic Soaps Subjected to Heat

In order to show the properties of metallic soaps, with particular reference to use at high temperatures on roll necks, Table 1, showing the properties of various metallic stearates, is included:

TABLE 1.—Action of Metallic Soaps on Heating

Metallic soap of	Appearance after heating 4 hours at 600° F.	Loss by vaporization (per cent)	Approximate melting point ° F.
Sodium	Hard, black and charred	26.7	500
Calcium	Light brown, hard, honeycomb structure	17.7	430
Magnesium	Very hard, brown; little changed	8.0	430
Potassium	Hard, dense, black	25.3	430
Aluminum	Hard, black, cinder	31.0	280
Lead	Black, cinder	50.0	230

TABLE 2.—Analyses of Commercial Rolling Mill Greases

	Sheet mill grease	Tin mill grease	Hot neck grease	Hot roll neck grease	Cold roll neck grease	Bar and plate mill grease
Penetration	158	57	41	7	Heavy liquid	69
Melting point, ° F.	116	123	136	292	56	204
Ductility	23	136	87	0	....	0
Flash point, ° F.	650	580	540	645	495	430
Solubility in CS_2, per cent	74.4	99.2	99.8	79.4	97.6	58.8
Solubility in CCl_4	74.4	99.1	99.7	79.0	96.0	58.8
Solubility in 86° gasoline	59.5	76.2	71.3	41.2	82.3	51.1
Fixed carbon	7.3	13.2	14.4	20.2	8.4	.2
Loss at 325° F., per cent	.3	0	0	.1	.5	Boiled out
Penetration after loss	91	48	35	7	Liquid	....
Loss at 600° F. (1½ hours)	Boiled out	1.9	1.8	.4	Boiled out	Boiled out
Penetration of residue	108	30	28	7	Liquid	....
Melting point of residue, ° F.	107	139	147	298	70	....
Mineral matter	....	.5	....	....	31	14.3
Ash	26.15	.97	.27	14.66	2.15	12.2
CaO	3.64	Trace	....	1.53	.09	4.40
$Al_2O_3 + Fe_2O_3$	2.54	Trace	....	3.42	.06	4.86
Insoluble matter	16.14	Trace	....	8.68	.88	3.06
Saponifiable matter	6.73	Trace	Trace	....	12.61	3.06
Specific gravity	....	1.044	1.033	....	....	....
Combined fat	....	....	....	16.33	....	38.36
Free fatty acids	....	....	....	1.43	....	1.29
Water	....	....	....	....	....	5.00
Mineral oil	....	....	....	....	....	40.09

Typical Hot Neck Grease

Formula	Per cent by Weight
Paraffine base residue, 650 flash	70
Hydrated lime	15
American talc	15

PROCEDURE:

The paraffine base residue or still grease is blown with air at a temperature of 450° to 550° F. to give a material having the following characteristics:

Penetration	143
Loss at 600° F. (1½ hours)	1.26 per cent
Penetration of residue	169
Melting point	150° F.

The talc and lime are mixed together and added to the hydrocarbon material, which is drawn into containers while in a molten condition.

Analyses of Commercial Rolling Mill Greases

The chemical analyses of several rolling mill greases are presented in Table 2.

The analysis of a hot neck grease used in one large steel mill is:

Penetration	122
Melting point	159° F.
Ductility	3.75
Solubility in CS_2	64.35 per cent
Solubility in CCl_4	64.41 " "
Solubility in 86° gasoline	49.49 " "
Loss at 325° F. (5 hours)	.07 " "
Penetration after loss	117
Mineral matter	31.38 per cent
Burns at	620° F.

After extracting the tarry hydrocarbons, they and the remaining mineral matter were analyzed with the following results:

	Per cent
CaO	50.45
MgO	7.44
SiO_2	16.28
R_2O_3	11.15
Loss on ignition	18.60

Tarry Hydrocarbons

Penetration	293
Melting point	99° F.
Ductility	64.25
Flash	510° F.
Solubility in CS_2	99.7
Solubility in CCl_4	99.75
Solubility in 86° gasoline	83.6
Fixed carbon	5.40
Loss at 325° F.	.12
Penetration after loss	181
Loss at 600° F. (1½ hours)	158
Penetration of residue	250
Melting point of residue	92° F.
Sulfur	.69 per cent

Analyses of European Hot Neck Greases

These greases gave the following results:

	Phillips Block Grease	Miller Block Grease
Appearance	Dull black, hard block grease, slightly greasy to the touch	Black, fairly hard, greasy feel
Melting point (Ubbelohde)	83° C.	80° C.
Ash	31.5 per cent by weight	3.0 per cent by weight
Nature of ash	Essentially limestone (silica, alumina and ferric oxide, lime and magnesia) together with a small quantity of sodium carbonate	Mainly ferric oxide and sodium carbonate
Alkali content of ash (as: NaOH)	2.6 per cent by weight (calculated on the ash)	35 per cent by weight (calculated on the ash)
Water content	3.0 per cent by weight	1.0 per cent by weight
Acid value	Alkaline	20.4 mgms. KOH/1 gm.
Total free and combined fatty acids	31.8 per cent by weight	58.8 per cent by weight
Nature of fatty acids	Probably derived from wool grease	Probably derived from wool grease
Iodine value of fatty acids	23	42
Melting point (capillary tube)	42–44° C.	.....
Unsaponifiable matter	33.7 per cent by weight (by difference)	35.2 per cent by weight
Nature of unsaponifiable matter	Black viscous material	Black viscous material
Cholesterol test on unsaponifiable matter	Positive	Positive
Test for vanadium on ash of unsaponifiable matter	Absent	Absent
Graphite content		2.0 per cent by weight

English Hot Neck Grease

The following analyses indicate the nature of hot neck greases in general use in England:

Special Hot Neck Grease

Ash	21.1 per cent by weight
Nature of ash	Mainly calcium carbonate, together with a small amount of sodium carbonate
Alkali content of ash (as: NaOH)	8.7 per cent by weight (calculated on the ash)
Total free and combined fatty acids	5.1 per cent by weight
Nature of fatty acids	Dark resinous material, probably derived from wool pitch

Approximate Compositions	Duplex Per Cent by Weight	Glycoline Per Cent by Weight	Phillips' Per Cent by Weight	Fletcher's Per Cent by Weight
Alkali (as: NaOH)	0.6	0.2	0.8	1.0
Limestone	3.0	18.	30.	...
Wool grease	50.	57.	53.	88.
Wool pitch	45.6	25.	13.	8.
Water	0.8	Trace	3.	1.
Graphite	...	...	...	2.

It will be observed from the above that these black block greases are all prepared from fatty materials of the animal fatty acid pitch type, most probably derived from wool grease. It will be appreciated that it is difficult to deduce the exact formulae from the analytical figures, to which these greases were made, because of the wide variations in the properties of the raw materials on the market. For example, the figures for the total free and combined fatty acids will include the saponifiable matter from both wool grease and any wool pitch used; and also, the unsaponifiable matter will include the unsaponifiable matter from both the wool grease and the wool pitch. The following compositions have been calculated on the assumption the wool grease containing 60 per cent by weight of saponifiable matter and wool pitch containing no appreciable amount of saponifiable matter has been used. It must, however, be remembered that similar results would be obtained if fatty acid pitches containing appreciable quantities of saponifiable matter had been used.

The special hot neck grease indicated above consists essentially of wool grease pitch containing about 10 per cent by weight of lime together with a small amount of sodium hydroxide. There is no definite indication of the presence of wool grease as such. No petroleum products were found. The unsaponifiable matter was characteristic of that obtained in general from fatty acid pitches.

Caplan's Formulae for Hot Roll Mill Greases [25]

Caplan gives the following formula for Starting Up Grease:

	Per cent by Weight
Petroleum pitch	40
Melting point 163° C.	
Flash point 515° F.	
Degras	10
Heavy cylinder oil	30
Flake graphite	20

This grease is made by melting the pitch in a fire kettle and gradually adding the degras, cylinder oil and graphite.

This "Starting Up Grease" is swabbed on the necks of the rolls until the necks have warmed up. When the necks are warm, fresh "Hot Neck Grease" is applied:

	Per cent by Weight
Petroleum pitch	47
Melting point 163° C.	
Flash point 535° F.	
Degras	7
Soapstone	27
Heavy cylinder oil	19
Sp. Gr. 18.50° Bé.	
Flash 504° F.	
Fire 570° F.	
Vis. 204 Saybolt at 210° F.	

[25] *Petroleum Engr.*, 121 (Sept., 1930).

This Hot Neck Grease is made exactly like the "Starting Up Grease." The soapstone is added at the end of the process.

PIPE JOINT COMPOUND

This product is a viscous paste of green-brown color and may be applied by a brush to oil well drill joints or to pipe threads, where the joints are to be broken within a reasonable length of time.

Powdered rottenstone	650 pounds
Boiled linseed oil	27 gallons
160 S.U.V. at 210° F. naphthene steam refined stock	17 gallons

COPPER LUBRICANTS

The use of powdered metals such as copper, zinc, and lead was proposed by Enlivisle,[26] in 1899. A typical formula included the following ingredients:

	Parts by Weight
Stearin or tallow	50
Soapstone or powdered mica	20
Graphite	2
Neatsfoot or other oil	10
Powdered lead, zinc, copper	10
Oil of tar	10

In 1921, Minton [27] patented a lubricant for yarns, fibrous materials, and the working parts of weaving machinery. It consisted of powdered mica and powdered tin, zinc, copper, nickel, silver, platinum, or other metal, but excluding powdered aluminum. A mixture containing equal proportions of mica and a single metal was preferred; but two parts of mica and one of a mixture of metals, in equal proportions, may be used. The mixture may be mixed with an oleaginous base, such as tallow or soap, to form a paste. Pearlman [28] has patented a lubricant for machinery consisting of:

Copper or bronze powder	1 liter
Petrolatum	2 liters
Lubricating oil	1 liter

GRAPHITE CONTAINING LUBRICANTS

Graphite, also known as black lead or plumbago, is probably the most important of all solid lubricants. It is not appreciably affected by temperature and is resistant to the action of acids and alkalies. Both natural and artificial graphites are employed in lubricants. Amorphous graphite was originally produced by Dr. Acheson in the electrical furnace. This graphite is almost chemically pure and is of a soft unctuous, noncoalescing nature. Ordinarily, this quality of graphite will contain 99 per cent or more

[26] British Patent 16,292 (Aug. 27, 1899).
[27] British Patent 179,344 (Feb. 17, 1921).
[28] British Patent 206,706 (Nov. 15, 1922).

of pure carbon. In the No. 1340 Acheson graphite more than 98 per cent of the graphite particles are less than 1/338 inch in diameter. Acheson produces deflocculated graphite by kneading it with water in the presence of tannic acid. Under such conditions the graphite particles become dispersed, breaking down into particles 1000 times smaller in diameter. Acheson estimated that during deflocculation an original particle of graphite would break down into 700,000 colloid particles. Dispersions of this sort of graphite in lubricating oil are sold under the names of Oildag, Olesol, or Kollag. According to Holde colloidal graphite particles range in size from 1 to 6 microns, but the majority are sub-microns less than 1 micron in size (1 micron = .001 mm.).

Graphite may be applied in three ways: (1), dry application; (2), mixed with semi-solids such as greases; (3), mixed with liquids such as mineral or fatty oils. The dry application has been adopted on certain reciprocating parts of lace making machines, in place of oil in order to avoid staining the fabric. Other applications are for lubrication of glass molds and chocolate machine bearings.

Bachman and Brieger [28a] have presented a simple and logical explanation of the role of graphite in lubrication. They claim that when graphite is added to lubricating oils a layer is formed on the bearing surfaces and that this layer adsorbs all oils nearly alike and all more strongly than metals adsorb lubricating oils. For this reason, graphite should improve oils and greases as lubricants and should improve the poorer products to a greater extent.

Film Strength Graphite Motor Oil

Timken film strength tests have been made on two lubricants, as shown below, and indicate that for the percentages given no appreciable extreme pressure properties were evident.

	Pyro-Oil	Kollag
Percent carbon residue	0.24	8.00
Percent graphite by filtration	0.17	7.70
Timken film strength, pounds per square inch		
O.K. at	2,900	2,900
Failed	4,275	4,275
(Speed 400 feet per minute, temperature 140 to 150° F.)		

It was noted that the graphite apparently caused a fine abrasive or lapping action on the test specimens.

Railway bearings, high speed fan bearings, dictating machines, pitman bearings on jaw crushers, worm gears, steam cylinders and valves, internal

[28a] *Kolloid Z.*, **39**, 334-46 (1926).
See also: "Memorandum on Solid Lubricants,—Report of the Lubricants and Lubrication Enquiry Committee." Bulletin 4, Dept. of Scientific and Industrial Research, England.
Thomsen, T. C., "Practice of Lubrication," 2nd ed., 30-1, McGraw-Hill Book Co., New York (1926).
Archbutt and Deeley, "Lubrication and Lubricants," 5th ed., 180-7, C. Griffin & Co., London (1927).
Hyde, J. H., "Graphite, Its Effect on Lubricants, 1920,—Report of the Lubricants and Lubrication Enquiry Committee." Dept. of Scientific and Industrial Research, England. Abstr. in *J. Soc. Chem. Ind.*, **40**, 37.
Ind. Eng. Chem., **18**, 497-9.
J. Chem. Education, **3**, 909-14.

combustion engines, and ropes, chains and gears, metal cutting and wire drawing, are some of the applications where colloidal graphite has been found useful. While graphite in general is not applicable to anti-friction bearings, there is some evidence to show that in three or four point contact ball bearings and roller bearings, which develop end thrust, extreme pressure conditions may exist and the utilization of colloidal graphite may be found advantageous.

E. G. Acheson [29, 30, 31] has been granted a large number of patents relating to the production of deflocculated graphite. These covered the utilization of tannic acid as a peptizing agent. An excess amount of tannin [32] was found desirable. To make graphite miscible with water an oil treatment with gallotanic acid was proposed by Dr. Acheson in 1906.[33] Tannic acid was also recommended for the deflocculation of graphite in the presence of non-aqueous liquids miscible with water.[34] In 1909, Acheson [35] proposed the following procedure for progressive addition of deflocculating agents: action of friction, the addition of a soluble substance, the addition of a colloid accelerator, and the addition of dextrin. In 1910, Acheson [36] patented dispersions of deflocculated graphite in mineral oil.

Townsend [37] has assigned to the Acheson Oildag Company a process for treating aqueous suspensions of deflocculated graphite, which consists of precipitating the body of graphite in a reversible condition by means of a volatile liquid reagent miscible with water. Acheson [38] pointed out that small additions of colloidal graphite to pale mineral oils caused little color depreciation. Also, that a pigmented oil compound with deflocculated graphite [39] could be made by utilizing a sufficient quantity of the graphite, or by employing a water and oil emulsion as the dispersion medium. Marks [40] has deflocculated graphite by heating it with water and working in lubricating oil at temperatures below 210° F.

It has also been suggested that graphite may be ground to a colloidal condition by mixing it with fatty acids and passing the mixture through a colloid mill.[41] Darling [42] had developed a method for purifying natural graphites by selective suspension in mineral oil. A graphite mill capable of exerting pressures amounting to about 1.5 kilograms per millimeter is considered desirable by one manufacturer [43] in order to produce colloidal graphite.

[29] British Patent 7,775 (Dec. 10, 1906).
[30] British Patent 7,776 (Dec. 31, 1906).
[31] British Patent 27,312 (Oct. 28, 1907).
[32] U. S. Patent 1,082,386 (May, 1910).
[33] U. S. Patent 844,989 (Dec. 10, 1906).
[34] U. S. Patent 986,793 (1909).
[35] U. S. Patent 964,478 (1909). See also British Patent 6,463 (March 15, 1910).
[36] U. S. Patent 966,636 (Aug. 9, 1910).
[37] U. S. Patent 1,116,957 (Jan. 18, 1910).
[38] U. S. Patent 1,448,246 (Aug. 28, 1919).
[39] U. S. Patent 1,590,067.
[40] British Patent 4,155 (Feb. 19, 1912).
[41] British Patent 208,267.
[42] British Patent 6,863 (March 21, 1904).
[43] U. S. Patent 2,045,501.

Miscellaneous Lubricating Compounds Containing Graphite

Dispersions of colloidal graphite in lubricating oils have been discussed and in this section suspensions of supercolloidal graphite will be considered. Apparently the advantages of graphite lubricating oils have been such as to attract the attention of many investigators. One of the early graphite oil compositions [44] consisted of graphite, lard oil, and benzol. In 1873, Smith [45] patented a mixture of graphite with tallow and turpentine. King's [46] lubricating compound was prepared with graphite, mineral oil, and animal oil. Lyon [47] preferred graphite and pulverized shale suspended in a mixture of castor and lard oils. In a patent granted to French [48] the following formula for a graphite lubricating composition was given:

Plumbago	6 ounces
Rosin	6 "
Tar	1 ounce
Glaubers salts	1 "
Tallow	3 ounces
Petroleum oil	1 gallon

Various mixtures of graphite with castor oil and other materials appear to have been popular in the past. Baldwin's [49] shock absorber lubricant consisted of one part of graphite and two parts of castor oil, lard oil, or neatsfoot oil. Thompson [50] blended tallow and castor oil together with graphite. Downs [51] prepared a lubricant consisting of graphite particles first coated with castor oil and subsequently placing the coated particles in an oil with which castor oil is not miscible.

Bierbaum [52] has developed a method of making lubricants consisting of mixing graphite with asphalt and then diluting this mixture with lubricating oil. In a later patent [53] he proposed mixing a wet mass of pulverized graphite with petrolene, derived from asphalt, and then dispersing the graphite mass in lubricating oil.

Powdered graphite mixed with boiled linseed oil and mineral oil composed the lubricant patented by Riedel,[54] in 1885. Maloney [55] patented a complex mixture of the following ingredients:

Paraffin oil	50 gallons
Graphite	15 pounds
Ether	1½ gallons
Acetone	30 "
Turpentine	5 "
Kerosene	15 "
Creosote oil	50 "

[44] U. S. Patent 544,223.
[45] U. S. Patent 144,875 (Nov. 25, 1873).
[46] U. S. Patent 148,568 (March 17, 1874).
[47] U. S. Patent 181,080 (May 26, 1876).
[48] U. S. Patent 195,890 (May 29, 1877).
[49] U. S. Patent 1,737,555 (Dec. 3, 1929).
[50] U. S. Patent 335,546 (1886).
[51] U. S. Patent 697,996 (Sept. 30, 1901).
[52] U. S. Patent 1,186,167 (March 30, 1914).
[53] U. S. Patent 1,207,843 (April 7, 1915).
[54] U. S. Patent 311,529 (1885).
[55] U. S. Patent 1,384,703 (April 26, 1920). See also U. S. Patent 1,309,492.

Straten's [65] lubricating oil consisted of:

Turpentine	50	gallons
Lubricating oil	50	"
Creosote oil	50	"
Ether	5	pounds
Powdered graphite	28	"

A lubricant suitable for loom shuttles, and wooden loom parts was developed by Holdridge [57] and was composed of graphite, sperm oil, and turpentine. Fifty parts of purified soot with fifty parts of mineral oil, rosin, or tallow was patented by Fresenium.[58] Tennant [59] claims a colloidal graphite oil consisting of powdered graphite blended with a blown fatty oil, such as blown rape seed oil, ground in a mill.

Chapman and Knowles [60] have patented mixtures of powdered graphite with mineral oil and glycerin. A graphite lubricant intended for use on open bearings on bakery machinery has been developed by Silhavy.[61] It consists of powdered graphite and a mineral oil having a gravity of about 46° Bé. with an initial boiling point above 325° F. and a final boiling point of less than 500° F. A graphite motor oil has been patented by Kidder [62] consisting of 0.05 to 0.005 per cent of colloidal graphite in a light motor oil. Bohner [63] has developed a graphite lubricant for bakery apparatus and other high temperature food cooking equipment made by suspending powdered graphite in white oil or liquid petrolatum. Pettit [64] has proposed suspensions of graphite in red engine oil, stabilized by an emulsion of kerosene, lime water and paraffine wax. It is claimed that close temperature control is essential and various mixtures of the above are prepared at temperatures from 110 to 160° F. A special lubricating distillate having a specific gravity of 0.843 and obtained by cracking pentadecane, and then distilling for a fraction boiling between 220 and 350° C. has been found by Copthorne [65] to be a suitable medium for dispersing deflocculated graphite.

Bierbaum [66] has made use of a method for freeing natural flocculated graphite from earth by adding water, mixing well, and then adding a hydrocarbon oil. The product thus obtained is intended for use as a lubricant.

Graphite Gold Dredger Grease

For the lubrication of underwater tumbler bearings on gold dredgers certain types of graphite greases have been proved advantageous. The analysis of one of the most successful greases for this purpose is given below

[56] U. S. Patent 1,369,492 (Nov. 12, 1919).
[57] British Patent 7,908 (July 23, 1904).
[58] British Patent 19,338 (Aug. 26, 1913).
[59] British Patent 416,626 (Jan. 9, 1934).
[60] British Patent 6,289 (Mar. 15, 1906).
[61] U. S. Patent 1,880,987.
[62] U. S. Patent 1,879,874 (Sept. 27, 1932).
[63] U. S. Patent 1,877,491 (Sept. 13, 1932).
[64] U. S. Patent 1,222,128 (Sept. 18, 1916).
[65] U. S. Patent 1,580,233 (April 13, 1926).
[66] U. S. Patent 1,340,316 (Oct. 6, 1914).

and consists chiefly of a mixture of 12 per cent by weight of fine flake graphite in a mixture of fat and waxes.

A.S.T.M. penetration at 77° F. (unworked)	165
A.S.T.M. penetration at 77° F. (worked)	175
Ubbelohde dropping point ° C.	55
Moisture	Trace
Percent graphite	12.1
S.U.V. at 210° F.	85
Analysis of Wax and Fat Mixture:	
Saponification value	23
Acid, mg. KOH/gm.	0.67
Percent wax	56.6
Melting point of wax ° C.	71
Analysis of Fatty Acids:	
Acid value	200
Iodine value	32
Melting point ° C.	48

Graphite Dredger Grease No. 4

Formula

	Per Cent
Cup grease No. 4	60
Acheson's graphite No. 39	25
Asbestos floats	15
	100

Tests. Experimental Sample:

Worked penetration	180
Ubbelohde dropping point ° C.	113 (235° F.)
Water, per cent	1.2
Ash, per cent	14.4
Acid insoluble filler, per cent	31.4
Ash content of acid insoluble filler, per cent	5.8 (based on total grease)
Graphite, per cent by difference	25.6
Asbestos estimated from ash (1.5 per cent deducted as CaO)	12.9
Total filler, per cent as determined by analysis	38.5

Suggested Specifications. Graphite Dredger Grease No. 4:

Worked penetration	180–210
Ubbelohde dropping point ° F.	200 (93° C.)
Graphite, per cent by analysis, min.	23
Ash, per cent	14.5–18.5
Average soap, per cent	12
Free alkali, per cent	....
Water, per cent max.	2.0
Standard sample	Must conform

Graphite Glass Mold Oils

Various mixtures of graphite with naphthene pale oils and degras have found application in the glass industry.

Glass Bottle Mold Swabbing Oil

	Per cent by Weight
200 S.U.V. at 100° F. naphthene pale oil	99.2
Acheson No. 38 graphite	0.8
A.P.I. gravity	23.4

Glass Mold Swabbing Oil

	Per cent by Weight
Lampblack	0.35
Dixon's microfyne graphite	0.35
200/100° F. naphthene pale oil	95.3
Degras	4
A.P.I. gravity	21.7
Flash ° F.	350
Fire ° F.	395

Graphite Spring Lubricants

Various combinations of ground and fine flake graphite, in combination with suitable amounts of calcium base grease, intended for the lubrication of automobile springs, were discussed in detail in Chapter V. In this section a few graphite spring lubricants and brake lubricants will be mentioned. Thompson [67] developed a graphite spring stabilizing compound consisting of about 50 per cent of fine graphite mixed with varnish and liquid glue to form a preparation having great adhesive properties and resisting the action tending to squeeze the lubricant from between the spring leaves. Burton [68] has formulated a "rust solvent" suitable for application to springs. It consists of a light lubricating oil, graphite, and a chlorinated hydrocarbon such as carbon tetrachloride, chloroform, or tetrachlorethane. It was suggested by Burton that from 2 to 25 per cent of this material added to heavy gear greases would greatly reduce their channel temperatures and permit easy shifting of gears at low temperatures. Since the organic chlorides and graphite both have extreme pressure properties this would also constitute an extreme pressure gear lubricant. Reid [69] has proposed a spring lubricant consisting of the following substances:

Light hydrocarbon oil	3 quarts
Engine oil	1 quart
Carbon disulfide	2 ounces
Graphite	1 ounce
Camphor	1 "

A penetration oil composed of deflocculated graphite in light coal tar oil has been proposed by Custer.[70] Burton [71] has made a penetrating oil with lubricating oil of 100 S.U.V. at 100° F. and a volatile, non-inflammable, chlorinated hydrocarbon thinner, together with suspended graphite. A

[67] U. S. Patent 1,410,967 (Sept. 7, 1920).
[68] British Patent 353,295 (Aug. 26, 1930).
[69] British Patent 414,847 (Apr. 23, 1930).
[70] U. S. Patent 1,574,642 (Feb. 23, 1926).
[71] U. S. Patent 1,857,235 (May 10, 1932).

mixture of low viscosity mineral oil, "pine fatty acids" and deflocculated graphite has been patented by King.[72]

Ordinarily, brake linings should never be lubricated with oily materials; however, several patents have been granted for such preparations to be applied to brake surfaces. Wieser [73] has suggested the following preparation for application to brake linings:

	Parts by Weight
Turpentine	96
Castor oil	16
Graphite	8
Oil of citronella	1

Addyman [74] has proposed a brake lubricant and rust remover consisting of one half ounce of ground graphite dispersed in 400 cc. of the following mixture:

	Liters
Lubricating oil	500
Light paraffine oil	20
Petroleum naphtha	20
Carbon disulfide	20

de Dion and Bouton [75] have suggested the use of consistent lubricants, prepared by mixing graphite with unctuous lubricants, as a clutch lubricant.

In an earlier patent by Burton,[76] a spring lubricant having the following formula was described:

Crude petroleum	3 quarts
Kerosene, 2 drams ethyl butyrate	18 ounces
Graphite, 5 minims oil cassia	6 "
Ethyl ether	7 "
Amyl acetate	3 drams
Ethyl acetate	3 "

Steam Cylinder Lubricants

The manufacturers of colloidal graphite claim that it is a suitable material for incorporation with steam cylinder oils. It is well known that the valves of Corliss steam engines are particularly difficult to lubricate and it is claimed that some of the advantages of colloidal graphite for this application are: reductions in the amount of oil fed, which amount to as much as 50 per cent; the consequent reduction in carbon formation and elimination of oil from the exhaust steam; improved action of the valves; improved packing lubrication; and a tendency to reduce dry spots on cylinder walls where highly superheated steam is used.

An early graphite steam cylinder lubricant was patented by Colgan,[77] in 1883, and consisted of cylinder oil, beeswax, paraffine, and graphite. Miller [78] preferred the use of a simple dispersion of graphite in the steam cylinder oil. Jenkin's [79] cylinder oil, patented in 1912, was quite similar to

[72] U. S. Patent 2,025,517.
[73] U. S. Patent 1,573,468 (Feb. 16, 1926).
[74] British Patent 359,602 (Aug. 1, 1930).
[75] British Patent 29,373 (Aug. 31, 1904).
[76] U. S. Patent 1,462,799 (July 24, 1923).

that of Colgan, and consisted of cylinder oil, paraffine, and graphite. A steam cylinder lubricant composed of lampblack and graphite together with lard oil, horsefat, and benzol, was patented by Olivera.[80] Chapman's [81] steam cylinder lubricant consisted of graphite, suspended in glycerin.

Graphite Railroad Lubricants

As mentioned in other sections of this volume, graphite is a frequent addition to such products as driving journal compounds, air brake cylinder lubricants, pantograph greases (33 per cent graphite), railroad gear greases, mine car greases, and railway automatic signal bearing greases. Colloidal graphite may be used to advantage in valve oils and special lubricants for locomotive motion parts; also, as an ingredient of present locomotive pressure gun greases. It is claimed that the use of colloidal graphite in valve oil permits smaller quantities of oil being fed to the cylinders, which in turn reduces the quantity of carbon deposited in valve ports and in piston ring grooves.

Oetling [82] has evolved an air brake cylinder lubricant consisting of graphite, glycerin, and distilled water. The object of the glycerin is, no doubt, to prevent freezing and sticking of the brake piston at low temperatures. An air cylinder grease was developed by Gifford.[83] Its ingredients are graphite, a neutral fat, and a non-drying oil. Brown and Rider [84] have proposed a lubricant for pneumatic systems consisting of graphite dispersed in a strong salt water solution to prevent freezing at low temperatures.

A graphite railroad grease was developed by Mott,[85] in 1886, and had the following composition:

	Pounds
Paraffine wax	60
Tallow	100
Lubricating oil	210
Graphite	1½

Miscellaneous Waxy Lubricants Containing Graphite

The patent literature is replete with references to various mixtures of wax and graphite. Paraffine wax and beeswax appear to be most popular. Colgan [86] manufactured a lubricating paste or stick consisting of petroleum oil, paraffine wax, and graphite. A more complex graphite lubricant consisted of lubricating oil, ammonium chloride, potash, tartar, paraffine wax,

[77] U. S. Patent 280,134 (June 26, 1883).
[78] British Patent 644 (June 10, 1901).
[79] British Patent 14,147 (July 12, 1912).
[80] British Patent 17,288 (July 26, 1912).
[81] U. S. Patent 905,649 (Aug. 20, 1906).
[82] British Patent 23,893 (Nov. 25, 1901).
[83] British Patent 11,527 (June 13, 1894).
[84] British Patent 5,039 (Dec. 29, 1876).
[85] British Patent 8,933 (July 8, 1886).
[86] U. S. Patent 241,932 (Mar. 18, 1881).

bay wax, and unctuous fatty oil, and graphite.[87] A more simple lubricant was compounded with paraffine wax, tallow, lubricating oil, and graphite.[88] Donati [89] preferred the use of ceresin wax, sodium carbonate, a yellow dye, steatite, and graphite.

As a suitable lubricant for electric motor and generator commutators Jones [90] has developed a lubricant comprising paraffine wax, and graphite, together with a small amount of spermaceti, added after boiling, and before cooling the mixture. Holm [91] prepared a solid lubricant from paraffine wax, graphite, magnesium oxide, and oil of citronella.

Cook [92] patented a composition of beeswax, glycerin, and graphite. The lubricant patented by LaRoche [93] also contained beeswax, glycerin, and graphite, and in addition kerosene, crude petroleum, or creosote oil, salts of tartar, and water.

Colgan [94] also patented a grease made consistent by a mixture of beeswax, and paraffine wax, and added lubricating oil and graphite. Williams [95] has developed a beeswax and graphite containing lubricant made in accordance with the following formula:

	Parts
Paraffine	30
Beeswax	20
Rosin	1
Castor oil	2
Graphite	40

Graphite Wire Drawing Compounds

In many instances lubrication engineers have recommended graphite as an ingredient of wire drawing compounds. The graphite is frequently mixed with powdered soap and powdered talc. The sodium soap of olive oil, mixed with graphite, is known to have been used with success in certain drawing operations. Aquadag, a dispersion of deflocculated graphite in water, has been used in drawing the metal filaments used in electric lamps. The dies in this case require a certain amount of lubrication to produce a satisfactory thread and this lubricant is apparently one which has been found satisfactory.[96] For metal tube drawing operations graphite greases, such as normal calcium base cup greases containing from 3 to 25 per cent of fine graphite, have been recommended. Braun [97] has assigned to the Westinghouse Lamp Company his patent for a wire drawing lubricant. It consists of a homogeneous mixture of powdered graphite mechanically held in suspension by a gelatinous medium, which is obtained by boiling

[87] U. S. Patent 324,018 (1885).
[88] U. S. Patent 366,383.
[89] U. S. Patent 650,037 (Nov. 10, 1898).
[90] U. S. Patent 680,155 (Jan. 7, 1901).
[91] U. S. Patent 650,108.
[92] U. S. Patent 635,303 (Jan. 20, 1898).
[93] U. S. Patent 177,525 (Apr. 7, 1876).
[94] British Patent 3,149 (June 26, 1883).
[95] U. S. Patent 1,735,368 (Nov. 12, 1929).
[96] British Patent 17,722 (1911).
[97] U. S. Patent 1,409,658 (Jan. 3, 1919).

agar-agar, or equivalent gum, with a liquid such as water to produce a jelly-like consistency. Iredell [98] has developed a wire drawing lubricant consisting of powdered graphite, Turkey Red oil, and agar-agar dissolved in water. Cachemaille [99] dissolves 0.5 kg. of agar-agar in 30 kg. of water and adds 20 kg. of graphite in the preparation of a drawing lubricant for fine tungsten wire. Montgomery [100] violently mixed water with an emulsifiable oil and fatty acid; then, added a pulverized insoluble solid, in the preparation of a drawing lubricant.

Dry Graphite Lubricating Compositions

Many graphite compositions, varying in nature from powders to hard cakes or firm bearing materials, have been developed. In some instances presses capable of applying pressures up to 30 tons, similar to those used for the production of oilless bearings from powdered copper, may be utilized for the manufacture of graphite bearing compositions. A graphitic "self lubricating bearing" consisting of petroleum coke, graphite, and a coal tar solvent, has been developed by Adams [101] and assigned to the U. S. Graphite Company. These materials are pressed together at pressures of 5 to 20 tons per square inch into bearings of any desired shape and are then baked at 500 to 1500° C. Kirkegaard [102] has employed shellac as a binder for graphite in the preparation of bearings. This process, owned by the National Graphite Lubricant Company, includes the drying of the shellac and graphite mixture as one of its phases. In the process assigned to the General Electric Company by Guay,[103] resins are utilized as binders for the graphite. A composition consisting of gum copal, graphite, and shellac has been patented by Smalley.[104] In another invention,[105] a formalin compound is utilized to bind powdered graphite together. Villepigue [106] has patented a process for high-speed turbines, in which the heat is so great that the use of lubricating oils is inadmissible. The bearings are covered with incrustations of graphite conglomerated by means of a tragacanth product which can vary according to requirements. A mixture of powdered graphite and spongy platinum is preferably employed by the inventor.

Hunt [107] has formulated a graphite lubricant for chains consisting of 20 pounds of graphite, one half pint of weak acetic acid, two pounds of lampblack, 6½ pounds of water, together with small quantities of gum tragacanth, turpentine, sperm oil, and neatsfoot oil. A solution of a gum, triethanolamine, and graphite are the components of Thomsen's [108] lubri-

[98] U. S. Patent 1,724,134.
[99] British Patent 137,329 (Jan. 2, 1920).
[100] U. S. Patent 1,617,455 (Feb. 15, 1927).
[101] U. S. Patent 1,589,919 (June 22, 1926).
[102] U. S. Patent 1,185,682.
[103] U. S. Patent 1,304,477 (Dec. 2, 1918).
[104] British Patent 8,823 (June 10, 1884).
[105] British Patent 23,622 (Oct. 25, 1907).
[106] British Patent 27,019 (Dec. 27, 1905).
[107] British Patent 22,037 (Oct. 13, 1903).
[108] U. S. Patent 1,895,014 (Jan. 24, 1933).

cant. A composition suitable for the lubricating of ordnance projectiles was prepared from graphite mixed with petrolatum, or molasses and water. It was forced through a die to form a rod suitable for insertion in grooves cut in the driving band of the projectile, by its inventors Hadfield and Jack.[109] A lubricant composed of powdered graphite, acetone, motion picture film scrap, and amyl acetate has been developed by McKee.[110] Lubricants patented by Chapman and Knowles [111] have included glycerin alone, or in combination with graphite, or with such substances as sodium hydroxide and borax, which prevent the formation of incrustations in steam generators and are recommended for use on the internal parts of condensing steam engines. A similar composition, but intended as a lubricant for use in hydraulic turbines or pumps, and having a specific gravity equal to or greater than that of water, has been proposed by Kaplan.[112] It was obtained by using glycerin, graphite, or talc, either alone or mixed with usual lubricants.

As lubricants for hot air cylinders, Adams [113] recommended a mixture of graphite, and soapstone, not essentially different than the mixture of black lead and talc suggested by Newton [114] six years earlier in 1861. Goold's [115] bearing composition consisted of graphite and coal tar. A mixture of powdered graphite and sodium chloride was suggested by Kurtz,[116] in 1880, as a lubricant for certain machinery bearings. A lubricant which no doubt had extreme pressure properties was composed of graphite, calcium phosphate, sodium and potassium silicates, and sodium carbonate. It was developed by Justice [117] in 1881. Aylsworth [118] has assigned to the Condensite Co. of America his process of treating flake graphite (200 mesh) with an oxidizing agent to obtain an "expanded graphite" of increased volume and decreased density. A fire extinguishing lubricant made up of the following materials has been patented by Wiley:[119]

	Per cent by Weight
Powdered graphite	75
Talc	20
Borax	5

Miscellaneous Graphite Greases

Various mixtures of graphite dispersed in fats or soap base greases have been suggested by many investigators. The following references will indicate the scope of this utilization of graphite. Sawrey [120] employed

[109] British Patent 18,514 (1903).
[110] U. S. Patent 1,603,086 (Oct. 12, 1926).
[111] British Patent 9,777 (Apr. 26, 1906).
[112] British Patent 157,367 (May 18, 1914).
[113] British Patent 2,907 (Oct. 17, 1867).
[114] British Patent 17 (Jan. 3, 1861).
[115] British Patent 145,117 (Nov. 19, 1919).
[116] U. S. Patent 237,437 (Apr. 7, 1880).
[117] British Patent 4,556 (Oct. 18, 1881).
[118] U. S. Patent 1,191,383 (Dec. 14, 1914).
[119] U. S. Patent 1,139,073 (Oct. 18, 1919).
[120] British Patent 2,386 (1880).

tallow or other fat as the base of his lubricant containing graphite, talc, salt, and lubricating oil. Roberts [121] mixed graphite with tallow and petrolatum. Bull [122] proposed similar mixtures of black lead and grease.

In a steel mill grease patented by Hicks and Elliott,[123] in 1888, extreme pressure properties were imparted to an animal fat grease containing graphite, peat, clay, and India rubber or pitch. Degras was the principal fat ingredient of Thorpe's [124] lubricant:

	Parts by Weight
Degras	20
Kerosene	16
Water	75
Turpentine	8.7
Aqua ammonia	4.4
Graphite	30

Sodium or calcium soap base greases in combination with graphite and other materials form the basis of a patent obtained by Hoar:[125]

Grease	20 pounds
Graphite	2 "
Pulverized charcoal	4 "
Ammonia	½ pint

Einstein [126] has developed a lubricant containing 10 to 15 per cent of powdered graphite dispersed in mineral oil. It is obtained by first mixing the graphite with sodium oleate, and then treating with an aluminum sulfate solution to deposit aluminum oleate on the graphite particles and then mixing with the mineral oil.

Graphite and Sulfur Compositions

In certain lubricants intended for application to automobile rear axle gears graphite and sulfur, together with various metallic soaps, have been utilized for the past 25 years. More recently combinations of lead soap, graphite, and sulfur were offered as hypoid gear lubricants. In 1874, Monckton [127] proposed a lubricant for dynamo bearings consisting of fish oil, and oily dispersion of sulfur and powdered graphite. Upper's [128] lubricating compound contained sulfur, graphite, tallow, rosin, and sodium chloride.

IRON CONTAINING LUBRICANTS

Most any lubricant after use on an iron journal will contain traces or more of iron, either as fine iron particles or organic iron compounds. In common with other solid lubricants finely divided iron may be considered

[121] British Patent 20,476 (Sept. 16, 1896).
[122] British Patent 835 (Apr. 16, 1855).
[123] British Patent 9,477 (July 5, 1888).
[124] U. S. Patent 2,003,564 (July 4, 1935).
[125] U. S. Patent 1,618,708 (Feb. 22, 1927).
[126] British Patent 366,128 (Oct. 30, 1930).
[127] British Patent 265 (Jan. 21, 1874).
[128] U. S. Patent 179,744 (Mar. 13, 1876).

objectionable from the standpoint of abrasiveness. Iron soaps, however, may have a proper place in lubrication. Since 1861, investigators have from time to time proposed the use of iron or iron compounds as lubricant ingredients. Finely ground iron ore was frequently used in early lubricants; for instance, Perrott [129] employed it in conjunction with tallow and lubricating oil. A dispersion of powdered iron, graphite, alumina, and silica in lubricating oil was patented by Harmon [130] as a lubricant, although in the light of present knowledge it would probably be considered a grinding or lapping compound. As a suitable lubricant for bicycle and motorcycle chains a mixture of iron ore, petroleum, and a fat such as tallow was proposed by Gartzke.[131] Trubshaw [132] also experimented with lubricating compositions made with powdered Fe_2O_3, iron ore, and fats. Becker [133] has developed a lubricant carrying material consisting of steel, or other metal wool treated with sulfur and mineral oil at 300 to 500° F. to cause the metal wool to retain large quantities of the oil. The composition was intended to be utilized as a lubricating pad.

KIESELGUHR LUBRICANTS

The persistence of certain investigators in offering mild abrasive compositions as lubricants is noticeable in the references applicable to kieselguhr, Celite, and silica. In 1883, Balene [134] produced calcined kieselguhr, sifted it through silk and mixed it with palm oil or other oils to produce a "lubricant." Tanner [135] prepared a compound for application to bearings consisting of the following:

Tallow and oil	3 pounds
Tripoli (silica)	2 "
Lampblack	1 ounce

Jordan [136] has proposed the use of kieselguhr as a means of filtering reclaimed motor oil; but it is not anticipated that any appreciable amount of this solid would remain in the treated oil.

LEAD CONTAINING LUBRICANTS

Powdered metallic lead or various lead oxides (such as PbO, PbO_2, Pb_3O_4, Pb_2O_3, or Pb_2O) have been suggested as suitable additions to otherwise normal calcium, sodium, aluminum, or lead soap greases. Such "lead lined" lubricants may contain from 2 to 65 per cent of the various lead oxides or powdered lead. In these lubricants, the lead or lead oxide is mechanically dispersed and prevented from separating by the character

[129] British Patent 158 (Jan. 21, 1861).
[130] U. S. Patent 482,249 (1892).
[131] British Patent 10,830 (May 12, 1898).
[132] British Patent 10,182 (1895).
[133] U. S. Patent 1,562,138.
[134] British Patent 1,432 (Mar. 19, 1883).
[135] U. S. Patent 136,945 (1873).
[136] U. S. Patent 1,393,223.

of the dispersing medium. It is claimed that in use such lead lubricants may actually deposit lead on the wearing surfaces. In some cases, lubricants of this class have been utilized with success at temperatures of 400 to 590° F. In the absence of oxygen it is claimed that they may be used at temperatures up to 1000° F. Some of the specific lubricants falling in this classification are: lead base pressure gun grease; tool joint and casing compound; wire rope lubricant; outside gear grease; lead base steel mill grease; pipe joint lubricant; lead base gasket compound; pump and valve packing lubricants; plug valve lubricants; pump and gland packings; asbestos-lead packing compounds; and lead base brake and belt compositions.

Lead Base Slow Speed Gear and Steel Mill Grease

FORMULA:

The following is a typical formula for this kind of grease:

	Per Cent by Weight
Lime tallow soap	15
Water	1
Lead sub-oxide	40
500 S.U.V. at 100° F. naphthene red oil	44

Medium Grade Lead Base Wire Rope Lubricant

FORMULA:

This product adheres well to each strand of a wire cable and tends to reduce wear and corrosion. It is sufficiently soft to be applied by means of a brush or swab.

	Per Cent by Weight
Lime tallow soap	6.5
Water	.5
Lead sub-oxide	25
500 S.U.V. at 100° F. naphthene red oil	68

Lead Base Cup Grease

FORMULA:

This product may be used for general purposes but when applied to babbit bearings will act as a lapping agent. It is useful when applied to piston rod glands and packings. It will have a penetration value of about 275 A.S.T.M. at 77° F.

	Per Cent by Weight
Lime tallow soap	14.5
Water	1
Lead sub-oxide	22.5
100 S.U.V. at 100° F. naphthene pale oil	62

All of the foregoing lubricants have definite extreme pressure properties when tested on the Timken apparatus.

Analysis of Four Lead Base Lubricants

	Slow Speed Gear Lub.	Heavy Grade Wire Rope Lub.	High Speed Gear Lub.	Lead Base Cup Grease
Physical Tests:				
Appearance	Soft, not very fluid	Semi-fluid, chunky	Quite fluid (smooth)	Standardized Graphite Cup Grease No. 3
Color	Gray black	Gray black	Gray black	Gray black
Odor	Similar to lard oil	None	None	None
Composition (per cent by weight):				
Soap	12.7	3.4	5.3	13.2
Kind of soap	Lead	Lead	Lead	Calcium
Oil	40.0	47.0	71.2	55.2
Moisture	0.1	None	0.4	1.0
Filler	38.0	36.5	19.6	22.5
Nature of filler	Resembling lead sub-oxide	Resembling lead sub-oxide	Resembling lead sub-oxide	Resembling lead sub-oxide
Saponifiable matter	4.2	None	None	13.3
Undetermined, loss or gain	5.0	13.1	3.5	5.2
	100.0	100.0	100.0	100.0
Oil Extracted:				
Gravity, ° A.P.I.	20.2	19.3	19.1	23.8
S.U.V. at 100° F.	470	401	483	95
Ash, total per cent on original	39.8	35.3	24.7	26.1
Nature of ash	Lead oxide (PbO)	Lead oxide (PbO)	Lead oxide (PbO)	Approx. 95 per cent Lead oxide (PbO); 5 per cent Calcium (CaO)
Timken Film Strength, Pounds per Square Inch:				
	O.K. at 17,160 Failed at 20,000			

Newell[137] has developed a composition for packing glands and the like consisting of a fibrous material treated with a mixture of graphite, lubricating oil, lead carbonate, and minium (Pb_3O_4). Verriaud[138] has assigned to the G.M.C. Metallic Grease Company his patent for lubricants consisting of mixtures of cup grease, lead dust, babbitt dust, and powdered white lead (basic lead carbonate). A typical formula developed by Verriaud is:

Calcium base cup grease	50 pounds
Lead dust	1 pound
Babbitt dust	.25 "
White lead	1 ounce

[137] British Patent 6,564 (Mar. 28, 1893).
[138] U. S. Patent 1,454,002 (June 29, 1922).

White [139] has developed a packing material consisting of a crumpled sheet of metal such as lead, coated with oil and a powdered lubricant.

MERCURY BASE LUBRICANTS

The role of mercury as a lubricant is not completely understood. It is possible, however, that since mercury tends to form amalgams, it has sufficient affinity for bearing metals to assist the formation of lubricating films. Possibly, organic mercury compounds may be formed from the constituents of the hydrocarbon lubricant which are more adherent to the bearing surfaces than the hydrocarbon or fatty oils alone. Newton [140] proposed an amalgam type lubricant composed of mercury, sodium or potassium, or other alkaline earth metals. Wise [141] prepared a lubricant by mixing together boiled linseed oil, beeswax, collodion, and graphite, and then dispersed mercury in this mixture. With respect to collodion, graphite, and mercury Haddan's[142] composition was identical but the linseed oil and beeswax were replaced by camphor and castor oil. Haddan [143] prepared, in 1877, a grease in cake form containing mercury, graphite, beeswax, boiled linseed oil, and collodion. Birkbeck [144] and Webster prepared a base for improving the oiliness of lubricating oils with which it was mixed in the proportions of four ounces per gallon of oil. It consisted of:

	Pounds
Lard	16
Tallow	1
Sulfur	40
Mercury	16

It is likely that appreciable quantities of mercury sulfide are formed when utilizing this lubricant and that it may have appreciable extreme pressure characteristics. Brown's [145] mercury lubricant was made with one pound of mercury and two pounds of petrolatum. Ducamp [146] dissolved mercury cyanide in glycerol and dispersed this material in motor oil to improve its lubricating qualities.

MICA LUBRICANTS

Mica is a lustrous, laminated, hard, silicate mineral and is represented by that class of minerals known as Muscovite, ($3Al_2O_3$, K_2O, 6 SiO_2, $2H_2O$). The formulae of calcium resinate base axle grease and sodium soap base fiber greases, containing this powdered mineral, have already been given. Since many micas contain fluorine compounds it may be possible that this element causes some form of chemical activity during lubrication

[139] U. S. Patent 1,002,349 (Sept. 5, 1911).
[140] British Patent 1,719 (June 28, 1865).
[141] British Patent 1,601 (Apr. 24, 1877).
[142] British Patent 2,099 (May 30, 1877).
[143] British Patent 2,098 (May 30, 1877).
[144] British Patent 242,520 (Mar. 31, 1925).
[145] British Patent 346,379 (Jan. 9, 1930).
[146] U. S. Patent 1,868,053 (July 19, 1932).

processes in addition to the abrasive and extreme pressure effects of the small lamina of mica. A method of pulverizing mica forming very fine flakes, has been patented by Boult.[147]

A simple dispersion of ground mica in lubricating oils was prepared by Zerbe,[148] of Lebanon, Pennsylvania, in 1874. In 1876, a compound claimed to be suitable for steam cylinder lubrication was formulated by Scott and Zerbe [149] and was prepared with powdered mica, lubricating oil, fat or grease. A lubricant manufactured by first mixing mica, graphite, or talc, with castor oil and then blending the mixture with mineral oil was developed by Downs.[150]

A dry powdered lubricant consisting of pulverized mica, ammonium chloride, charcoal, or carbon, was patented by Norris.[151] Lubricants for both high and low temperature applications were prepared by McCallum and Holmes [152] using the following formulae:

	Low Temperatures Part	High Temperatures Parts
Tallow	1	2
Chalk	1	1
Mica	$\frac{1}{8}$	1

Tallow filled with powdered mica, graphite, potassium ferrocyanide, dextrin, litharge, sodium carbonate, together with paraffine wax and glycerin was the complex mixture patented by Cooke,[153] in 1891. Minton [154] has developed a special lubricant for fibers used in weaving, braiding, or rope-making, or for wires in wire-weaving, consisting of 10 to 90 parts of mica mixed with 90 to 10 parts of powdered aluminum. The mixture may be incorporated with soap or tallow to form a paste, or with water, oil or other liquid. Scott and Kenyon [155] manufactured a lubricant for railway journal bearings. It consisted of tallow, paraffine wax, stearin, palm oil, or grease, together with mica, talc, clay or similar materials to be used in the place of pulverized shale, which they also suggested as a filler. A compound for dusting automobile inner tubes was patented by Schnell [156] and consisted of a dry powdery mixture of graphite, mica, and lampblack.

FREE SULFUR LUBRICANTS

Many of the early lubricants contained free sulfur, the particle size of which was greater than colloidal dimensions. For instance, in 1895, Belknap [157] proposed a lubricant consisting of one gallon of mineral oil,

[147] British Patent 19,892 (Oct. 22, 1895).
[148] U. S. Patent 150,739 (Feb. 7, 1874).
[149] British Patent 3,773 (1876).
[150] British Patent 9,313 (Apr. 22, 1902).
[151] U. S. Patent 267,451 (Nov. 14, 1882).
[152] British Patent 26,328 (Nov. 29, 1902).
[153] U. S. Patent 454,588 (1891). See also U. S. Patent 455,012.
[154] British Patent 140,174 (Feb. 19, 1919).
[155] British Patent 494 (Feb. 1, 1882).
[156] U. S. Patent 1,519,268 (Dec. 16, 1924).
[157] U. S. Patent 317,490 (May 12, 1885).

a pound of graphite, one quarter pound of paraffine wax, one pound of tallow, and one pound of finely powdered flowers of sulfur. The mixture was compounded at temperatures of 150 to 160° F. to produce a paste suitable for application to hot boxes, propeller shafts and the like. Prior to this, in 1872, Trossin [158] utilized melted sulfur alone for the lubrication of hot air engine pistons. For a similar use Baird [159] claimed improvement over Trossin by incorporating various other lubricants with sulfur. The Tolhurst [160] grease consisted of a mixture of tallow, wax, petrolatum, powdered sulfur, and castor oil. A lubricant patented by Fink [161] consisted of equal quantities of an animal, vegetable, or mineral oil, with the following base:

Lime water	12 gallons
Chalk	10 pounds
Ammonium chloride	4 ounces
Ammonium carbonate	4 "
Sodium borate	2 "
Sulfur	10 pounds

More recently Abrams [162] has proposed lubricants, for use under extreme pressure conditions, formed by suspending free sulfur particles in oil or water by means of a mass of freely mobile gel particles. In the oil medium these particles are composed of soap. In water he uses starch, dextrin, gum, etc. This lubricant is said to be satisfactory for worm and hypoid gears.

Another lubricant contained powdered sulfur greater than colloidal size. It consisted of a lubricating gel composed of water, starch, gum, glue, or casein, together with the powdered sulfur. It was also patented by Abrams.[163] This investigator [164] also developed soap base lubricants in which the soap gel prevented settling of the suspended sulfur particles. In another lubricating grease Abrams [165] suspended free sulfur particles of greater than colloidal dimensions in a mixture of carnauba wax and oil.

LUBRICANTS CONTAINING VARIOUS INORGANIC SALTS

To those not familiar with the early patent art concerning lubricating compositions it will no doubt be surprising to find that a wide variety of inorganic salts have been proposed as ingredients of these compounds. These will be discussed briefly in the following paragraphs.

Ammonium nitrate, (NH_4NO_3), one part, sodium carbonate one part, and 18 parts of grease or lubricating oil were the constituents used in a lubricating composition proposed by Wood.[166]

[158] British Patent 2,884 (Nov. 2, 1870).
[159] British Patent 1,516 (May 18, 1872).
[160] British Patent 23,344 (Oct. 11, 1897).
[161] British Patent 1,525 (Feb. 2, 1886).
[162] U. S. Patent 1,913,300 (June 6, 1933).
[163] U. S. Patent 1,913,299 (June 6, 1933).
[164] U. S. Patent 2,007,137.
[165] U. S. Patent 2,016,532 (Oct. 8, 1935).
[166] U. S. Patent 1,398,759 (Aug. 4, 1920).

Ammonium chloride, (NH_4Cl), has been utilized by Grof [167] in the preparation of a "fire proof lubricating oil" which was made by boiling one gallon of mineral oil for one hour with the following materials:

Graphite	6 ounces
Sodium tungstate	6 "
Ammonium phosphate	5 "
Ammonium chloride	16 "
Sal soda	16 "
Ammonium sulfate	½ ounce
Lubricating oil	1 gallon

Alumina, (Al_2O_3), was incorporated with other aluminum compounds, tallow, oils, and greases in the preparation of a grease formulated by Accles and Scott.[168]

Barium sulfate, ($BaSO_4$), has been used by Fletcher [169] for raising the specific gravity of 500 S.U.V. at 100° F. mineral oil above that of muddy water in the preparation of an oil well drilling compound. Other oil compounds with heavy pulverized mineral substances were investigated by Fletcher and assigned by him to the Hughes Tool Company.

A lubricating base has been suggested by Heath [170] consisting of 80 per cent of barium sulfate and 20 per cent of a colloidal clay.

Calcium antimoniate, ($Ca_2Sb_2O_7$), mixed with a mineral oil of low specific gravity and then combined with a heavy mineral oil was the subject of a patent for a lubricant granted to Smith.[171]

Calcium carbonate, ($CaCO_3$), while normally considered objectionable in lubricating greases was found by Langer [172] to be a suitable ingredient. He used it in the preparation of a non-inflammable lubricating emulsion which consisted of mineral oil and 30 to 60 per cent of a saturated solution of either calcium hydroxide or the carbonate. Morgan's [173] lubricant consisted of equal parts of calcium carbonate and paraffine wax.

Calcium antimony tartrate mixed with mineral oil has also been utilized by Smith [174] as a "fire proof lubricant."

Iron chromite, ($FeCr_2O_4$), has been suggested by Lake.[175] His lubricant, for chains and bearings of bicycles, was prepared by mixing a powdered metallic chromite, such as chromite of iron, copper, or manganese, with a suspending or binding medium, such as glycerin, oil, or paraffine. Ketchum [176] has also proposed the use of iron chromite dispersed in lubricating oil.

Potassium iodide, (KI), together with rape seed oil, lard, and butter

[167] U. S. Patent 1,181,976 (Mar. 8, 1916).
[168] British Patent 5,344 (Dec. 7, 1881).
[169] U. S. Patent 1,587,944 (June 8, 1926).
[170] U. S. Patent 1,881,542 (Oct. 11, 1932).
[171] U. S. Patent 748,317 (May 28, 1903).
[172] British Patent 156,517 (1919). See also British Patent 156,537.
[173] British Patent 2,436 (Feb. 12, 1889).
[174] U. S. Patent 736,162 (Aug. 11, 1903).
[175] British Patent 17,553 (Oct. 14, 1891).
[176] U. S. Patent 467,938 (1892).

has been patented by Fontaine [177] as a lubricant. An alkali is utilized to saponify the fat for consistent products.

Magnesium oxide, (MgO), mixed with refined suet constitutes a lubricant proposed by Mege.[178]

Fink [179] has also made use of magnesium oxide in lubricants. His process was to emulsify a saponifiable oil with a mixture of:

Calcium oxide	14 pounds
Pulverized French chalk	20 ounces
Potassium carbonate	22 "
Calcined magnesia	16 "
Sodium carbonate	10–20 "

He also patented [180] a similar composition in 1872, but eliminated the magnesia, the product consisting of lime, pulverized chalk, water, and potassium carbonate, (K_2CO_3).

Potassium nitrate, (KNO_3), together with sodium carbonate and graphite has been utilized as a lubricant.[181]

Silicon monoxide, (SiO), dispersed in mineral oil represents a class of lubricants patented by Potter [182] and assigned to the Westinghouse Company.

Silver oxide, (Ag_2O), or finely divided metallic silver, together with a fatty lubricant has been patented by Forsyth.[183]

Sodium carbonate, (Na_2CO_3), has been employed as an ingredient of many soap base lubricants, some of which have been referred to in the previous chapters. Park [184] has proposed a cutting lubricant consisting of a mixture of sodium carbonate, hydrated lime, and water.

Sodium chloride, (NaCl), has also been frequently referred to in lubricants of the prior art. A typical example is the lubricant of Thorswik.[185]

	Per Cent by Weight
Sodium chloride	25
Borax	2
Powdered coal	10
Water	63

Zinc sulfate, ($ZnSO_4$), together with sodium chloride, castor oil, and water, were mixed together by Thomas [186] and then treated with a solution of sodium carbonate, borax, and potassium prussiate. Mixtures of zinc sulfate with graphite, borax, sodium bicarbonate, and lubricating oil have also been patented.[187]

[177] British Patent 1,252 (June 1, 1855).
[178] British Patent 2,157 (July 17, 1869).
[179] U. S. Patent 945,711 (Oct. 8, 1909).
[180] U. S. Patent 127,328 (May 28, 1872).
[181] U. S. Patent 343,943 (1886).
[182] U. S. Patent 830,739 (Sept. 11, 1906).
[183] British Patent 2,564 (June 26, 1878).
[184] U. S. Patent 204,076 (Mar. 2, 1878).
[185] U. S. Patent 1,249,249 (Mar. 2, 1917).
[186] U. S. Patent 184,927 (Oct. 14, 1876).
[187] U. S. Patent 406,883 (1889).

SULFUR BASE LUBRICANTS

The probable action of sulfur when used in a lubricant to form metallic sulfides on the surfaces of bearings has already been discussed in connection with the subject of hypoid gear lubricants and extreme pressure products. A few uses of sulfur in lubricants, particularly those of early origin, will now be mentioned. In 1865, Taylor [188] patented a mixture of sulfur, lubricating oil, and fine emery. Williams [189] mixed sulfur, tallow, lime, graphite, and various clays together in the preparation of a lubricating compound. Bartlett's [190] grease had the following formula:

Sulfur	3 pounds
Lard oil, or other oil	1 gallon
Sodium chloride	5 pounds

Sulfur, powdered graphite, alum, and sodium chloride have also been patented.[191] A similar product [192] consisted of flowers of sulfur, graphite, sodium chloride, and gypsum. Mixtures of sulfur, tallow, graphite, paraffine wax, and a heavy lubricating oil were covered by a patent,[193] in 1885. An early lubricant intended for the application to wagon axles consisted of powdered sulfur, peat, and lubricating oil.[194] Mixtures of sulfur, lubricating oil, and potassium prussiate were patented [195] in 1890.

TALC LUBRICANTS

Talc, also known as steatite, soapstone, or French chalk, ordinarily consists of hydrogen magnesium silicate ($H_2Mg_3Si_4O_{12}$). It has long been used as a filler for lubricating greases having stearates or oleates of calcium, sodium, aluminum, lead, or barium as a base. Various talc lubricants not incorporated with soap base compounds are listed below. Hamill,[196] in 1863, was one of the first to utilize mixtures of pulverized soapstone and petroleum. Mixtures of talc and chalk have long been used as "anti-attrition compounds" to prevent the squeaking of shoe soles.[197] Sawyer's [198] lubricant consisted of talc, graphite, tallow, fullers earth, and lard oil. Another composition [199] consisted of slaked lime, magnesia, talc, calcium chloride, and mineral oil. Kaplan [200] has suggested the use of a composition having a specific gravity greater than that of water consisting of talc, graphite, glycerin, and mineral oil as a lubricant for hydraulic turbines or water pumps.

[188] U. S. Patent 50,049 (1865).
[189] U. S. Patent 164,117 (May 11, 1875).
[190] U. S. Patent 167,730 (July 22, 1875).
[191] U. S. Patent 287,643 (1883).
[192] U. S. Patent 294,840 (Mar. 11, 1884).
[193] U. S. Patent 317,490 (1885).
[194] U. S. Patent 357,227 (1887).
[195] U. S. Patent 423, 522 (1890).
[196] U. S. Patent 38,822 (1863).
[197] U. S. Patent 207,874.
[198] U. S. Patent 268,546 (Dec. 5, 1882).
[199] U. S. Patent 487,203 (1892).
[200] British Patent 157,367 (May 8, 1914).

Various compositions of waxes with talc have been patented. Paraffine wax, mineral oil, alcohol, and talc was patented in 1885,[201] Spermaceti wax and talc was patented in 1881.[202] Clarke [203] proposed mixtures of paraffine wax, lubricating oil, and talc. Starke [204] has suggested a mixture of paraffine wax, talc, and graphite or asbestos as a lubricant for electrical equipment.

A lubricating grease was made by Maguire,[205] in 1878, by mixing together fish oil, tallow, graphite, sodium nitrate, and lime, and then adding talc. Bullock [206] has developed a lubricant for cutting tools consisting of talc, sodium carbonate, and water.

ZINC GEAR LUBRICANTS

Prendergast, Sonsthagen, and Pearson [207] have developed a series of lubricants for wheels subject to varying pressure, such as gear wheels, friction gears, and roller bearings. They were prepared with various proportions of a metal, metal oxide, or metal compound, electropositive with respect to steel or other metal of the gears in order to make the lubricant anodic with respect to the gears. The proportions used were based on the following relationship:

$$A = c - \frac{K(a-b)}{6 + \sqrt[4]{c^2}} + 100$$

in which:

A is the percent of electropositive component,
a is the potential in volts of the electropositive component,
b is the potential in volts of the metal in the gears,
c is the percentage of water in the complete lubricant,
K is a constant of 1312, 1180, or 770,

depending on whether the working conditions are ideal, normal, or bad respectively. Electropositive substances specified were zinc, zinc oxide, zinc carbonate, cadmium, and cadmium oxide. These agents were dispersed in mineral lubricating oils, fatty oils or lubricating greases by the foregoing investigators. One or more of the components of the lubricant may be in the form of a colloidal material. For normal working conditions, with iron or steel gears, 28.5 per cent of zinc oxide and 1 per cent of water was preferred as a dispersion in mineral oil. Normally, a coating of zinc on the steel gear surfaces is obtained.

A zinc containing lubricant has been recommended for application to the Keenok pinion, developed by British engineers in 1929, for use in transmitting power in automobiles. Various gear greases have been marketed in this country for more than fifteen years which contained either zinc soaps or zinc oxide. European grease makers have for many years

[201] U. S. Patent 316,703 (1885).
[202] U. S. Patent 245,695 (1881).
[203] British Patent 670 (Jan. 15, 1886).
[204] British Patent 2,347 (1904).
[205] U. S. Patent 211,755 (Nov. 22, 1878).
[206] U. S. Patent 1,449,379 (March 27, 1923).
[207] British Patent 327,097 (Sept. 22, 1928).

added small percentages of zinc oxide to their sodium and calcium base greases in order to improve their lubricating characteristics and render them slightly opaque.

Carter [208] has developed an extreme pressure lubricant consisting of equal parts of mineral oil and lard oil, together with zinc oxide, talc, and a binder consisting of a calcium compound.

A fifty per cent mixture of zinc oxide in lubricating oil has also been suggested by Pendergast.[209] In 1921 Hartmann, Vaudt, and Adler [210] proposed a packing material for gasometers consisting of a mixture of the following:

	Per Cent by Weight
Zinc oxide	40
Lead oxide	40
Olive oil	10
Gourd-core oil	10

Watts [211] has patented the addition of 2 to 10 per cent by weight of zinc oxide to lubricating oils or greases. A lubricant consisting of 10 parts by weight of finely divided zinc white with one part of mineral oil was patented by Judd,[212] in 1920.

Finely divided metallic zinc is frequently used for lubricating packings. Entwistle [213] has developed a water-proof mixture for lubricating hydraulic rams. It consists of suitable proportions of stearin, talc, graphite, neatsfoot oil, powdered zinc, and oil of tar.

Rattigan [214] has proposed a fibrous packing impregnated with zinc metal dust as a lubricant. Newbern [215] has developed a lubricant composed of lubricating oil, flake graphite, powdered zinc, white lead, and ammonia. Zollinger [216] heats castor oil in a vacuum with finely divided zinc to produce a composition for lubricating leather.

[208] U. S. Patent 1,810,905 (June 23, 1931).
[209] U. S. Patent 1,913,953 (June 13, 1933).
[210] British Patent 182,959 (Apr. 21, 1921).
[211] U. S. Patent 2,043,638 (June 9, 1936).
[212] U. S. Patent 1,449,608 (May 11, 1920).
[213] British Patent 16,292 (Aug. 27, 1894).
[214] U. S. Patent 1,838,189 (Dec. 29, 1931).
[215] U. S. Patent 1,839,159 (Dec. 29, 1931).
[216] British Patent 112,624 (1916).

Chapter XII

Organic Lubricants and Extreme Pressure Compounds

OILINESS

The theory of fluid or viscous lubrication, which is based on the laws of hydro-dynamics, has been fairly well developed [1] but the application of the laws pertaining to the physico-chemical forces have not been fully investigated. In discussions of oiliness it is well to keep in mind two distinct types of lubrication, namely, viscous and boundary lubrication. When the rubbing surfaces of a bearing are completely separated by a relatively thick film of lubricating oil molecules, this lubrication may be termed viscous, but if the film is extremely thin and disrupted the conditions of boundary lubrication apply. According to Clark, Lincoln, and Sterrett,[2] lubrication engineers recognize the following conditions applicable to boundary lubrication:

When bearings are at rest, or in a static condition, at the moment of starting boundary lubrication conditions will exist. Viscous lubrication is essentially a kinetic phenomenon.

When machine parts have slowed down and are just about to become static, boundary conditions may also apply.

In reciprocating and rocking motions, for instance, crankshaft bearings, where heavy shock loads are imposed, boundary conditions may exist.

Rapid changes in speed or load may be sufficient to cause momentary metallic contact.

When the viscosity of the lubricant is too low, due either to the use of an oil which is excessively thin or on account of reduced viscosity due to high temperatures, metallic contact and boundary conditions may occur.

An inadequate supply of oil or lubricant may cause boundary lubrication.

Friction will, of course, occur in both boundary and viscous lubrication. Ewing,[3] Kingsbury,[4] and others,[5] have made investigations of oiliness and

[1] *Boswall, "The Theory of Film Lubrication,"* Longmans, Green and Company (1928).

[2] *Proc. Am. Petroleum Inst.*, **16**, (Nov. 11, 1935).

[3] *Proc. Royal Inst. Gt. Brit.*, **13**, 387 (1892).

[4] *Trans. Am. Soc. Mech. Eng.*, **24**, 143 (1903). See also:
Sub-committee on Lubrication, *Mech. Eng.*, **41**, 537 (1919).

[5] Rayleigh, *Phil. Mag.*, (6), **35**, 157 (1918). See also:
Tomlinson, *Phil. Mag.* (7), **7**, 904 (1929); and *ibid.*,
Murison, **17**, 201 (1934).
Dunstan and Thole, *J. Inst. Petroleum Tech.*, **55**, 191 (1918).
Deeley, *Proc. Phys. Soc.* (*London*), (2), 32, 1-s (1920).
Langmuir, *J. Am. Chem. Soc.*, **38**, 2221 (1916); **39**, 1848 (1917); *ibid.*,
Blodgett, **56**, 495 (1934); **57**, 1007 (1935).
Lubricant and Lubrication Inquiry Committee, Department of Scientific and Industrial Research (England) (1920).
Hardy, *Kolloid Z.*, **46**, 268 (1918).
Barnard and Forrest, *Ind. Eng. Chem.*, **16**, 327 (1920).
Wilson and Barnard, *ibid.*, **14**, 682 (1922).
Wilharm, *ibid.*, **18**, 463 (1926).
Becker, *ibid.*, **18**, 471 (1926).
Stanton, *Engineer*, **135**, 678 (1923).
Bragg, *Nature*, **115**, 266 (1925).
Parsons and Taylor, *Ind. Eng. Chem.*, **18**, 493 (1926).
Woog, *Compt. rend.*, **180**, (1924); **181**, 772 (1925).

the factors which produce differences in coefficient of friction under identical operating conditions, viscosities being the same. The friction reducing properties of various "oiliness dopes" are applicable in the case of greases as with lubricating oils. The relationships between oiliness, film strength as in the case of extreme pressure lubricants, wear, and seizure have not been fully established or adequately defined. A gear lubricant, for instance, may have a high degree of oiliness and under normal operating conditions maintain low lubricant temperatures, but under extreme loading might fail and cause scuffing where a high film strength E. P. lubricant would prevent failure but temperatures might become quite high. The best extreme pressure lubricants should also have satisfactory oiliness characteristics. Some lubrication engineers believe that by adequately increasing the oiliness of lubricants there will be a tendency to minimize loss of bearing metal by wear and reduce the possibilities for seizure and bearing failure. It is probable that the region where extreme pressure lubricants are required overlaps the region of boundary lubrication where oily lubricants are desirable; in any case, the two conditions are closely related.

The effects of oiliness are ordinarily attributed to the ability of the lubricant to be sufficiently active to neutralize unbalanced forces acting between metal bearing surfaces. It is believed that this is accomplished by polar molecules, which may contain an atom, or group of atoms, possessing residual valence. The polar groups are attracted by the forces exerted by the bearing surfaces and probably assume regular oriented positions. Clark and his associates consider that oiliness and the ability of the polar molecules to orient themselves are closely related. The ability of the polar films to adhere to bearing surfaces is not related to the viscosity of the lubricant; therefore, a very dense grease may not have the oiliness characteristics of a relatively soft cup grease properly compounded with a suitable oiliness material. It is felt that grease research chemists should give careful consideration to improving the oiliness of lubricating greases, and that this is a field which has been investigated to only a slight extent. The popular conception is that long-chain polar molecular molecules are more firmly attached to bearing surfaces than normal oil molecules, and that the polar end adheres to the surface with the free ends extended into the lubricant layer. It is likely that these chains assume parallel positions and are at a right angle to the bearing surface. Hardy has shown that the field of force extending from the boundary surface approximates 20,000 Ångstrom units. Kingsbury considered that the laws of viscous lubrication applied to films as thin as 6,250 Ångstrom units. According to Trillat the forces existing between polar molecules and metal surfaces may extend through layers of four or five hundred molecules to a thickness of about 9,000 Ångstrom units.

[5] Dallwitz-Wegner, *Kolloid Z.*, **38**, 193 (1926).
Trillat, *Rev. Sci.*, **64**, 552 (1926).
Blom, *Petroleum Z.*, **25**, (46), Suppl. 3 (Nov. 13, 1929).
Tausz et al, *Z. angew. Chem.*, **43**, 570 (1930).
Hersey, *J. Wash. Acad. Sc.*, **23**, 297 (1933).
Department of Scientific and Industrial Research (England), Technical Papers 1 (1930) and 2 (1934).
Bridgeman, *Physics*, **5**, 125 (1934).

Clark, and his associates, have reported the following coefficients of friction (static or break-away torque), using an Almen machine:

	Coefficient of Friction
S.A.E. 30 mineral oil	0.215
S.A.E. 30 plus 0.75 per cent methyldichlorostearate	0.194
S.A.E. 30 plus 0.5 per cent oleic acid	0.200

Under loads of 3000 pounds per square inch of projected bearing area they obtained the following data, from which it was concluded there was no direct relationship between film strength and oiliness, as measured by starting torque on the Almen machine:

Compound Added	Timken Film Strength (Pounds)	Decrease in Torque (Per cent)
Trichlororesorcinol	125.0	74.1
Methyldichlorostearate	28.0	60.9
Chlorostearic acid	64.0	20.0
Ethyldichlorophthalate	38.0	24.0
Trichloroanisole	13.8	30.4
Oleic acid	10.3	33.3
Hexyl chloride	8.8	4.5 increase
o-Chloropenetole	14.8	8.0 increase

Using a Deeley testing device, equipped with three pegs and a disk, and making determinations at (from) 10 to 100 pounds per square inch, the following static coefficients of friction were obtained. They indicate that, for a series of oils from the same source, the lower the viscosity the higher the friction.

	Deeley Value
750 S.U.V. at 100° F. Mid-Continent lubricating distillate	0.187
F.F.F. Pennsylvania bright stock	0.181
Heavy Californian lubricating distillate (600 S.U.V. at 100° F.)	0.155
Light Californian lubricating distillate (300 S.U.V. at 100° F.)	0.168
Heavy Mexican lubricating distillate	0.150
Light Mexican lubricating distillate	0.155
Lighter Mexican lubricating distillate	0.155
Very light Mexican lubricating distillate	0.170
600 S.U.V. at 100° F. Red oil	0.155
600 S.U.V. at 100° F. Red oil plus 0.5 per cent sperm oil	0.149
600 S.U.V. at 100° F. Red oil plus 1 per cent sperm oil	0.145
600 S.U.V. at 100° F. Red oil plus 2 per cent sperm oil	0.142
600 S.U.V. at 100° F. Red oil plus 3 per cent sperm oil	0.138
600 S.U.V. at 100° F. Red oil plus 5 per cent sperm oil	0.133
600 S.U.V. at 100° F. Red oil plus 7.5 per cent sperm oil	0.121
600 S.U.V. at 100° F. Red oil plus 10 per cent sperm oil	0.115
600 S.U.V. at 100° F. Red oil plus 15 per cent sperm oil	0.106
600 S.U.V. at 100° F. Red oil plus 20 per cent sperm oil	0.102
Sperm (straight)	0.098
600 S.U.V. at 100° F. Red oil plus 5 per cent lard oil (containing 4.5 per cent free fatty acid)	0.096
600 S.U.V. at 100° F. Red oil plus 1 per cent oleic acid	0.100
600 S.U.V. at 100° F. Red oil plus 1 per cent aluminum oleate	0.089
600 S.U.V. at 100° F. Red oil plus 10 per cent sperm plus 3.5 per cent castor oil	0.102
Straight Castor oil	0.121
Castor plus 5 per cent lard oil plus 5 per cent light Mexican distillate	0.104

It is of note, that in the case of the sperm oil additions above, the first 0.5 per cent has a greater relative effect than increased amounts, and that the 20 per cent blend is practically the same as for 100 per cent sperm oil. Aluminum oleate is effective in reducing friction and small percentages of fatty acids are as effective as large amounts of fat.

The effects of fatty acids in reducing the coefficient of friction of lubricants was studied by Southcombe and Wells,[6] in 1918, who had in mind the addition of these materials to plastic greases as well as to fluid lubricating oils. These investigators compounded hydrocarbon lubricating oils with a minute amount, *e. g.*, 1 to 2 per cent, of a substance other than soap which lowers the interfacial tension between oil and water. Where it is desired to prevent the product from forming an emulsion with water, acids of lower molecular weight, such as butyric or other volatile fatty acids, cinnamic acid, or water-soluble naphthenic acids, are added to the mineral lubricating oil. A product, which is emulsifiable with water and suitable for marine-engine lubrication, was obtained by the use of higher fatty acids, such as those obtained from rape oil, wool grease, or whale oil. Olein and other commercial fatty acids are preferably employed, the products so obtained being very similar to those obtained by compounding mineral oils. The hydrocarbon oil may be derived from petroleum, coal, coal-tar, shale, colophony, etc. Petroleum jelly is also used. The fatty acids suggested by Southcombe and Wells were termed "germs" and their process was called the "germ process." In 1923, they [7] proposed to refine lubricating oils in such a manner as to leave up to 2 per cent of petroleum organic acids in the finished oil in order to reduce the coefficient of friction. It appears likely that lubricating greases made with petroleum residues, containing appreciable amounts of organic acids, should have superior oily properties; providing their conversion to soaps does not render them inactive. Such evidence as is available, however, indicates that such soaps reduce rather than increase the frictional values.

In 1933, Southcombe and Wells [8] obtained additional protection for their germ lubricants. They considered that hydrocarbon lubricating oils were improved by the addition of 0.1 to 0.25 per cent by weight of a saturated fatty acid having at least 16 carbon atoms. Stearic, arachidic, and cerotic acids are specified. For example, 0.1 per cent of cerotic acid or 0.2 per cent of stearic acid is added to a mineral lubricating oil. Not only did Southcombe and Wells recognize the importance of free fatty acids but Wisner,[9] in 1924, proposed mixing American lubricating oils with from 0.5 to 5 per cent of saturated fatty acids. As suitable acids he recommended stearic, arachidic, cerotic, and myristic. A typical formula called for 1 part of stearic acid and 99 parts of mineral oil.

McGill [10] has assigned his patent dealing with oils of improved "oiliness" to the Standard Oil Company of Indiana. He proposed the addition of

[6] British Patent 130,377 (Feb. 5, 1918).
[7] U. S. Patent 1,467,695 (Mar. 11, 1923).
[8] British Patent 412,101 (Nov. 7, 1933).
[9] British Patent 244,086 (Dec. 5, 1924).
[10] U. S. Patent 1.837.279 (Dec. 22, 1931).

50 grams of hydroxystearic acid to each liter of oil. Moran [11] has proposed the addition of Nicolet and de Milt's acid to mineral oils to increase their oiliness and film strength. Burwell [12] has suggested a penetrating oil of low viscosity to which he added aliphatic acids derived from petroleum and containing 4 to 12 carbon atoms (molecular weight, 160 to 165).

A railway car journal oil having an S.U.V. at 0° F. of 200 seconds and a minimum S.U.V. at 100° F. of 70 seconds with a pour point not above — 60° F. has been proposed by Ott.[13] In this product he blends a high viscosity index, SO_2 insoluble lubricating oil with a fatty acid to improve oiliness. The stereometrical isomeride obtained from the reaction between nitrous acid and oleic acid has been proposed as an oiliness compound by Lincoln.[14] He recommended the addition of 35 to 100 grams of this fatty material to each gallon of lubricating oil. Pseudopimaric acid, ($C_{20}H_{30}O_2$), having a melting point of 169 to 181° C. has been suggested by Brennan [15] as an oiliness material.

Various organic esters are known to improve the frictional characteristics of lubricants. Frolich [16] has developed the use of from 0.25 to 2.5 per cent of the following: the isopropyl esters of oxidized paraffine wax, methyl caprylate, amyl butyrate, glycol stearate, secondary butyl naphthenate, isopropyl benzoate, phenyl acetate, glycerol stearate, isopropyl oleate, and ethyl stearate. Revere [17] assigned to the Texas Company his patent covering the use of esters of hydroxystearic acid. Ellis [18] has experimented with improving lubricating oils by adding to them the reaction products of organic acids having an alkyl group such as alkylolamine. To increase viscosity and improve lubrication Schuman [19] has suggested the polymerization products of vinyl ether with an unsaturated alcohol, having molecular weights of at least 1000.

Johnson [20] has suggested the use of partly depolymerized hydrocarbon products as agents for improving lubricating oils. He [21] has also proposed hydroxyalkyl mixed ethers. Edlund [22] has developed the use of mixed ethers in which one of the radicles is a tertiary alkyl group and the other is the residue of a primary or secondary alcohol. Lubricants containing large quantities (2 to 10 per cent) of organic compounds to increase oiliness, but not viscosity (mineral oils having 35 to 65 S.U.V. at 100° F.), have been patented by Penniman.[23] Southcombe and Wells have suggested oil soluble organic bases [24] and sulfonic [25] acids as means of reducing the

[11] U. S. Patent 1,850,561 (Mar. 22, 1932).
[12] U. S. Patent 1,895,374 (Jan. 24, 1933).
[13] U. S. Patent 1,929,189 (Oct. 3, 1933).
[14] U. S. Patent 1,945,614 (Feb. 6, 1934).
[15] U. S. Patent 2,042,035.
[16] British Patent 402,262 (Dec. 29, 1932).
[17] U. S. Patent 2,031,227 (Feb. 18, 1936).
[18] U. S. Patent 2,018,758 (Oct. 29, 1935).
[19] U. S. Patent 2,020,703.
[20] British Patent 411,894 (Dec. 12, 1932).
[21] British Patent 317,770 (Mar. 19, 1928).
[22] British Patent 393,753 (June 8, 1932).
[23] U. S. Patent 1,967,255 (July 24, 1934).
[24] British Patent 168,076.
[25] British Patent 165,897.

frictional properties of oils. Ralston [26] has found that the use of 0.1 to 1.0 per cent of a retone derivative may be used with advantageous results. His compounds had the following structure, in which R is a closed ring and R′ is an alkyl radical of not less than eleven carbon atoms:

$$R-\underset{\underset{O}{\|}}{C}-R'$$

For cutting lubricants, mold oils, and other applications where a high degree of oiliness is required the Alox Chemical Corporation [27] have developed products consisting of heavy saturated aliphatic mineral hydrocarbons oxidized in the liquid phase under pressures up to 320 pounds per square inch at 100 to 150° C. in the presence of a catalyst. The reaction is carried out until compounds insoluble in petroleum and in the reaction mixture begin to form, separating the saponifiable components, treating them with steam and drying. Hydrocarbons suitable for oxidation are waxes from shale or brown coal or petroleum, and the heavy distillates of petroleum. The saponifiable components may be separated from the reaction mass by: (1), saponifying with caustic alkali, separating the soap solution, diluting to clear it, and then acidifying; or (2), extraction by ethyl or higher alcohol, or acetone or other ketone. Suitable proportions for addition to engine or bearing oils are 0.25, 0.50, 0.75, 2.5 to 10.0 per cent; up to 50 per cent may be added to cutting oils; 10 to 2 per cent with 90 to 98 per cent of petroleum oil of viscosity of at least 40 Saybolt at 100° F. is suitable for moulds. Mackenzie and Haskell [28] have proposed the oxidation of paraffine wax, or the neutral saponifiable material derived from the oxidation of paraffine wax, as agents for increasing oiliness. The quantities of this material to be used have been suggested by Mackenzie [29] as 2 to 5 per cent of the weight of the total lubricating composition. Burwell [30] has also made oily bases by the oxidation of high molecular weight hydrocarbons in the liquid phase utilizing a suitable catalyst to promote reaction. The insoluble material formed is separated, purified, and the saponifiable and unsaponifiable material isolated. Henriksen and Lincoln [31] have suggested the condensation products obtained from two halogenated fatty acids as oily bases. An alkali condensation agent is employed to promote the reaction. Sommerville [32] has studied the action of small amounts of the condensation products obtained from ketones with nitrogenous bases. He [33] also produced condensation products of aldehydes with nitrogenous bases.

Later, Sommerville [34] proposed the addition of fatty oils together with small amounts of the condensation product of an aldehyde with an amine.

[26] U. S. Patent 2,033,543.
[27] British Patent 335,152 (Dec. 18, 1928).
[28] U. S. Patent 1,705,298 (Mar. 12, 1929).
[29] U. S. Patent 1,904,065.
[30] U. S. Patent 1,863,004 (June 14, 1932). See also U. S. Patent 2,043,923.
[31] U. S. Patent 1,939,994 (Dec. 19, 1933).
[32] U. S. Patent 1,617,826 (Feb. 15, 1927).
[33] U. S. Patent 1,594,983 (Aug. 3, 1926).
[34] U. S. Patent 1,767,264 (June 24, 1930).

Cassidy [35] has developed the process of heating a mixture of naphthenic acid and acridine orange base, then heating and mixing with lubricating oil. Polymerized styrene to the extent of about 0.5 per cent has been suggested by Wulff [36] as a means of improving lubricating oils.

ORGANIC EXTREME PRESSURE BASES

E. P. Bases may be classified under three headings: (1), organic sulfur compounds; (2), organic chlorine compounds; and (3), organic phosphorus compounds. Examples of the application of various extreme pressure ingredients (sulfur, graphite, etc.) have already been mentioned in connection wtih gear lubrication, wire drawing, and metal cutting and turning operations. It is claimed by some technologists that the utilization of sulfur, for instance revealed in early cutting patents, is not tenable with respect to hypoid gear lubrication. Metal working is an old process while extreme pressure lubrication as applied to hypoid gears is a recent development necessitated by the invention of these newly designed devices. As already pointed out, extreme pressure lubricants are those containing a relatively small amount of a material which will greatly increase their load carrying capacity. As previously mentioned, they may be considered as a separate class from oiliness compounds. The following discussions will apply to various uses of E.P. bases regardless of whether the application is metal cutting, metal drawing, or gear lubrication. If special lubricants are advantageous with regard to one, the possibility is they are applicable with respect to the others, and will be given consideration in the following sections. It will be remembered, of course, that extreme pressure lubricants containing solid materials have already been presented. In the following pages, those bases capable of being blended with lubricating greases or oils to form products free from solid matter will be given consideration.

SULFUR BASE CUTTING COMPOUNDS

Cutting lubricants, or cooling liquids as they are sometimes called, are compounded oils or aqueous emulsions which are used wherever the cutting of metal is carried on. They possess lubricating and cooling properties in different degrees, and the various classes may be listed as follows:

Cutting Oils. These are such oils as lard oil, cottonseed oil, degras or rape oil, or mixtures of such oils, free from water and soap, with mineral lubricating oils. These compounded oils do not usually form emulsions with water.

Cutting Emulsions. Emulsions used in cutting operations are made by mixing some form of soluble oil which usually consists of a mineral oil, soda or potassium soap, free fatty acid, and glycerin or alcohol, in conjunction with water.

Paste Cutting Compound. These are solid emulsions of mineral oil, soda or potassium soap, and water.

Soluble Oils. These products are oily liquids which form emulsions when mixed with water.

[35] U. S. Patent 1,860,850 (May 31, 1932).
[36] U. S. Patent 1,998,350 (Apr. 16, 1933).

Cooling

During the operation of cutting metal, heat is developed, which not only causes a rise of temperature of the edge of the cutting tool, but also the material which is being machined. On cooling, the metal being machined will contract, and often the dimensions will differ from the measurements taken during the cutting operation. This difficulty is particularly noticeable in the machining of such metals as aluminum, which has a high coefficient of expansion.

If the tool heats to much, the edge will wear rapidly. It is known that the heat generated at the point of the tool is largely conducted into the body of the tool; and when the tool is of large section the heat is more easily radiated and dissipated than in the case of a tool with small cross section. Efficient cooling of the tool edges reduces wear and enables a greater output to be obtained.

Finish

In many cases in metal cutting it is highly desirable that the cutting oil should be such that a smooth finish is imparted to the completed work. Where a perfect finish is desired, experience has shown that the cutting oil used must possess that quality known as oiliness. This is the reason for the extended use of various animal and vegetable oils, or compounds of such oils with mineral oils. In some cases such oils as rape seed and cottonseed are preferable to either mineral or animal oils in producing a smooth finish. The use of a small amount of sulfur, either in free solution or in loose combination, with highly unsaturated vegetable or animal oils, results in production of very satisfactory, smooth finishes. It is believed that the function of the sulfur in such compounded oils is to modify the surface tension of the oil to the metal, resulting in a product which adheres more satisfactorily to the cutting edge of the tool.

Dies, taps, reamers, broaches, and other form tools are known to have a longer life, when used on tough metals, if the cutting oil employed is a mixture of mineral oil and sulfurized vegetable or animal oil. For finishing, rifling, etc., a mixture of castor oil and light-bodied mineral oil in the ratio of 1 to 3 has been known to give good results. The addition of an equal volume of turpentine to this mixture causes perfect solution to take place, and, for some materials, is said to give a better finish, particularly on guns and other hard material.

Lubrication

Where the metal being operated on is of brittle material, lubrication is of minor importance, as the material is removed in the form of a fine powder or chips. In the case of cast iron, the use of a solution of soluble oil in water is of some advantage, due to the fact that the chips, dust, etc., are washed away from the working parts and thus prevented from being dispersed in the air. For tough metals such as wrought iron, the material being removed is in the form of spiral shavings, and it is necessary that

efficient lubrication be maintained, as the chips and shavings grind heavily over the cutting edge of the tool. For heavier cuts the metallic friction is greater, making it more important that the lubrication of the nose of the tool be efficiently maintained, as otherwise the shavings will produce considerable friction, which may result in destruction of the tool and a rough finish.

Washing Action

In many cases found in machine tool work the material being cut is subjected to a continuous stream of cutting lubricant, which washes away the chips as fast as they are formed. If the cutting emulsion used is too weak, the chips will not be effectively carried away. In the boring of gun tubes, etc., a solution of 25 pounds of soda soap and 50 pounds of sodium carbonate in 200 gallons of water, is said to give satisfactory results.

Rust Prevention

Satisfactory cutting oils should not cause rusting; in fact, the oil should inhibit rusting. Cutting oils containing large quantities of lard, or other animal or vegetable oils, often become rancid, with the formation of small quantities of fatty acids which attack the metal and cause corrosion. Some soluble oils when used as emulsions are particularly objectionable from a corrosion standpoint.

Selection of Cutting Lubricants

In selecting a satisfactory cutting lubricant for a particular piece of work, it is necessary to take into consideration the cutting speed and depth of cut. Low speeds and shallow cuts require little cooling or lubrication. Low speeds and heavy cuts, particularly when tough metal is being operated on, require a lubricant of great oiliness. At high speeds and shallow cuts the cutting lubricant should have good cooling properties, and, consequently, emulsions of soluble oils and sulfur-base cutting oils are frequently employed. Turpentine is sometimes used for aluminum. High speeds and heavy cuts demand a lubricant of good cooling properties as well as high lubricating quality. Heavily compounded sulfur base oils are of value in this work.

Where the material is brittle, as in the case of cast iron, emulsions of soluble oil in water are frequently employed. For tough materials the highly compounded oils are of value. For automatic screw cutting machines a light sulfur base oil is generally employed, although emulsions containing from $2\frac{1}{2}$ to 20 per cent of soluble oil are sometimes used. The richer mixtures are used for severe service and the more dilute solutions for light service, where there is little danger of rusting, such as for brass and aluminum.

Cutting oils may be applied by hand in a drop feed system by some means of overhead gravity feed. The larger machine shops almost invariably utilize some system of automatic circulation of the lubricating oil to and from

the machine tools. In most modern plants the turnings are collected and centrifuged for the recovery of the cutting oils adhering to them.

Certain cutting lubricants have been known to cause "oil rashes" on the skin of the workmen, which may be due either to plugging of the small glands at the roots of the hairs or to injury of the skin produced by metallic particles suspended in the cutting oil. To prevent such skin diseases and infections, suitable washing accommodations for those workmen who must come in contact with the oil should be provided. Care should also be taken in the handling and selection of cutting oils to secure fresh, clean oils or lubricants which have been satisfactorily filtered on recovery. Frequent cleaning of the machines with removal of rancid lubricant from all parts is essential. The use of such antiseptics as carbolic acid is sometimes of value in preventing skin infections. A solution containing not more than one or two per cent of this material should be used. It is possible that where the oil attains a temperature of over 300° F. for short periods considerable sterilizing action takes place.

Sulfur Base Cutting Oils

In most cutting oils of this type sulfur is heated with some kind of animal or vegetable oil in order to effect a loose combination of the sulfur with the unsaturated oil. The nature of the reaction between such an oil as lard oil and sulfur is by no means clear. At 270° to 300° F. oleic acid readily takes up about 10 per cent of sulfur without evolution of hydrogen sulfide; at higher temperatures, such as 400° to 575° F., hydrogen sulfide is evolved. It would thus appear that at the lower temperature addition products are formed and at the higher temperatures substitution products are produced.

In some sulfur base cutting oils sulfur is dissolved directly in the mineral oil. On heating mineral oil with sulfur, no evolution of hydrogen sulfide takes place below about 350° F. At 300° F., light asphalt base lubricating oils hold in solution as much as 15 per cent by weight of sulfur; on cooling to room temperature and filtering, about 3 per cent of sulfur remains in the oil. On cooling to 32° F., only one per cent of sulfur remains in the oil.

Sulfur Base Cutting Oil

A satisfactory sulfur base cutting oil may be made as follows:

Formula

Base

	Per Cent by Weight
Lard oil, No. 1	85
Flowers of sulfur	15

Finished Oil

Sulfur	4.43
Lard oil	15.94
Mineral oil, 100 to 200 vis. pale oil	80.00

PROCEDURE:

Charge in a fire-heated grease mixture 355 pounds of lard oil, and heat to a temperature of 380° to 420° F.

At this temperature sift in gradually 63 pounds of ground sulfur. The lower the temperature and the shorter the process, the lighter will be the color of the finished base.

After heating for about fifteen minutes a drop of the oil should be tested by placing on a cold, bright surface. Heating should be continued until there is no precipitate of sulfur.

The base should be left in the kettle to cool and after cooling 1,254 pounds of lard oil, having an acid value of 18 to 22 per cent, calculated as oleic, should be run in.

About 880 gallons of 100 vis. at 100° F. pale oil should be mixed with the ingredients in the kettle.

This product will be found to be satisfactory for general use. In some cases for particularly heavy service an oil of 200 to 300 viscosity at 100° F. may be found to be of advantage.

Sulfur Base Cooling Oil

A satisfactory sulfur base cooling oil may be made as follows:

Formula	Per Cent by Weight
Finely ground sulfur	0.90
Asphalt base red oil, 250 vis. at 100° F.	99.10

PROCEDURE:

The mineral oil is first charged in a steam jacketed kettle and heated to 245° F.

The sulfur is then sifted in. Agitation may be accomplished, if so desired, by means of a small pump so arranged as to take suction at the bottom of the kettle and discharge back into the kettle just below the surface of the oil. Circulation should be carried on while heating the oil. For ten minutes the batch of oil is kept at a temperature of 250° F., when the steam in the jacket is turned off.

The batch is slowly cooled over a period of about three hours to a temperature of 110° F., when it may be drawn into barrels or other containers for shipment.

Heavy Duty Sulfur Base Cutting Oil

A heavy duty sulfur base cutting oil may be made as follows:

Formula	Per cent by Weight
Sulfur	10.0
Herring oil	26.6
Paraffine oil, 100 vis	81.7

PROCEDURE:

The herring oil is first placed in a small fire-heated kettle and mixed with the sulfur. The temperature is then raised to about 300° F., when a soft, rubbery material is formed.

The sulfurized herring oil may then be dissolved in the mineral oil by heating to about 150° F.

Sulfur Base Cutting Oil Patents

The following patent abstracts pertain to sulfur base cutting oil:

Pressell [36a] has assigned to E. F. Houghton & Co. a patent for cutting compound base and process of preparing same, for use on metal cutting

[36a] U. S. Patent 1,367,428 (Mar. 25, 1919).

tools, comprising a stabilized base of animal fat, sulfur and paraffine oil, capable of forming a permanent dilute solution of hydrocarbon mineral oil. The formula for the "refrigerant base," by which term it is generally known, is given below:

	Per cent by Weight
Wool fat or distilled wool grease	30.00
Sulfur	13.00
Lard oil	26.00
28° Paraffine oil	30.00

These ingredients are heated together at a temperature of about 350° F.

Claflin [36b] has patented a heat-absorbing lubricant composed of a colloidal solution of sulfur in mineral oil. It is said that this lubricant is especially adapted for screw-cutting machines. It is made by mixing together 5 pounds of sulfur with a small quantity of cylinder stock and about 20 gallons of light oil, in the order given below:

Fine sulfur	5 pounds
Steam refined stock	1 quart
Steam refined stock	10¼ gallons

Heat to 383° to 428° F. in a mixing kettle and run in

Paraffine oil, 34° Bé	20 gallons

From two to three per cent of sulfur is said to remain in colloidal solution.

Bezzenburger [36c] has secured a patent for a sulfur base cutting oil in which an unsaturated animal or vegetable oil is treated with sulfur at a high temperature. He claims that the sulfur is taken up in the unsaturated bonds of the saponifiable oil. A typical formula follows:

	Per cent by Weight
Sulfurized corn oil base (containing 10% of sulfur)	10 to 25
Mineral oil	60 to 75
Lard oil	15

Compounded Cutting Oils

Cutting Oil

The following cutting oil is compounded in any ordinary compounding vat:

Formula	Per cent by Weight
28° Bé. Paraffine oil (140-170 vis. at 100° F.)	98.0
Lard oil, No. 1	1.75
Cooling base	.25
Sulfur, Degras, Lard oil	equal parts

Heavy Duty Cutting Oil

The following is a satisfactory oil of this character:

[36b] U. S. Patent 1,401,760 (Mar. 22, 1920).
[36c] U. S. Patent 1,516,879 (Nov. 25, 1924).

Formula	Per cent by Weight
Texas red oil, 300 vis.	98.0
Lard oil, No. 1	1.75
Cooling base (as in No. 1)	.25

Dark Cutting Oil

The following is a satisfactory oil of this character:

Formula	Per cent by Weight
Black oil, 500 vis.	98.0
Lard oil, No. 1	1.75
Cooling base (as in No. 1)	.25

Thread Cutting Oil

The following is a satisfactory oil of this character:

Formula	Per cent by Weight
Cooling base	5.0
Paraffine oil, 170 vis.	40.0
Inedible oil	40.0
Lard oil	15.0

The cooling base consists of the following ingredients heated together:

	Per cent by Weight
Soy bean oil	85.0
Sulfur	15.0

Acid Base Cutting Oil

The following is a satisfactory oil of this character:

Formula	Per cent by Weight
Lard oil, No. 2	30.0
Paraffine oil, 28° Bé.	70.0

Add 2 ounces of hydrochloric acid for each 50-gallon barrel. Mix the ingredients well at 260° F. and cool to barreling temperature.

Thread-Cutting Oil

The following is a satisfactory oil of this character:

Formula	Per cent by Weight
Whale oil	20.0
Acidless tallow oil	5.0
Pale oil, 100 vis.	75.0

A very small amount of nitro-benzene may be used for scenting this oil.

Cutting Compounds or Pastes

Satisfactory cutting compounds should be made from cleaning pure materials and should not turn rancid, decompose, or develop offensive odors

on storage. When used as an emulsion it should not gum or rust the machines or work. In mixing cutting compound, 100 pounds of the material should be added to every 30 gallons of water, at a temperature of about 120° to 150° F. It should be stirred vigorously until thoroughly dissolved and care should be taken that no small particles remain undissolved. The solution thus formed should be of a creamy or milk-like appearance, to which water may be added as follows:

For automatic and hand-screw machines, turret lathes, and threading, 20 gallons.
For milling machines and engine lathes, 100 gallons.
For automatic machines and turrret lathes doing shape work on which no threading is done, 100 gallons.
For threading bolts and tapping nuts, couplings, unions, etc., 70 gallons.
For screw cutting and turning on engine lathes, 170 gallons.
For drilling purposes, 350 gallons.

Cutting Compound

The following is a satisfactory cutting compound:

Formula

	Pounds	Per cent by Weight
Water	250	48.00
Caustic soda	6	1.22
Silicate of soda, 40° Bé.	27	5.18
Commercial oleic acid	86	16.50
Ohio pale oil, 100 vis.	151	29.10

PROCEDURE:

In a single walled grease mixer mix the commercial oleic acid, the 100 Viscosity Pale Oil and nine-tenths of the water.

Without heating, run in the sodium silicate solution.

Dissolve the caustic soda in the balance of the water and add to the contents of the kettle.

The kettle used should have a draw-off of at least 8 inches in diameter. After stirring for 15 or 30 minutes the compound should develop a consistency equal to that of a No. 2 or 2½ cup grease.

Cutting Compound Paste

The following is a satisfactory paste:

Formula

Oleic acid	5 gallons
Lard oil, No. 2	10 "
Paraffine oil, 28° Bé.	10 "
Raw degras	35 pounds
Sal soda	35 "
Water	210 "
Oil of mirbane	2 ounces

PROCEDURE:

Charge in a steam jacketed mixer all of the oils and the degras and bring to 80° F.

Mix the sal soda (crystallized sodium carbonate) and water together and bring to 110° F., when all of the soda should be dissolved.

Add the soda solution to the ingredients in the mixer and heat to 130° F.

Heating should be continued until a temperature of 210° F. is reached, which completes the batch.

While the cutting compounds are made with a soap base and do not, strictly speaking, fall in this chapter they have been included for the sake of convenience in making reference to them.

Sulfur Base Cutting Oil Patents

While free sulfur and sulfur compounds have long been used as ingredients of lubricants, it was not until (about) 1888 when Sommer[37] completed the development of his "sulphochlorinated" compounds suitable for use in cutting lubricants, that they were given much consideration by lubricating engineers. In his original process, the hydrochloric acid formed, when fats were treated with sulfur chloride, was neutralized with any suitable alkali. But in 1888, Sommer proposed the addition of certain terpenes and olefines which reacted with the excess sulfur chloride or hydrochloric acid as it was liberated. Propylene, butylene, beta-amylene, and beta-hexylene were some of the materials preferred. Another [38] early cutting and cooling lubricant was prepared by mixing 13 parts of glycerin with 2 parts of sulfur. It was not until 1918 that further advances in the art of making sulfur base cutting oils were published. Pressell [39] had found that a cooling base for cutting operations could be made by bringing together and stabilizing a mixture of animal fat, sulfur, and paraffine oil. Claflin's,[40] and Bezzenburger's,[41] sulfur base cutting oils have been discussed.

Oldacre [42] recognized that the amount of sulfur which could either be dissolved or combined with mineral oil was limited. He developed a method of treating mineral oil with sulfur chloride to introduce the sulfur molecule. He found that heat was necessary to promote the reaction and that a temperature of 170° F. was satisfactory for red and pale oils of medium viscosity. For heavy steam refined stocks he preferred lower temperatures. In order to eliminate the hydrochloric acid and other gas formed he suggested blowing air through the mixture while the reaction was taking place. The air also served to agitate the mixture. Ordinarily, in order to combine about 3.5 per cent of sulfur with mineral oil, Oldacre would start with a mixture of 7 per cent sulfur chloride and 93 per cent mineral oil. Treatment of the mineral oil in this manner also effected a reduction in pour point. No objectionable odor was produced, due to hydrogen sulfide remaining in the oil, as in other processes where sulfur is heated with the oil at high temperatures. Nelson,[43] in 1933, assigned to the Texas Company his patent for preparing sulfur base cutting oil. His process was to mix mineral oil having a minimum viscosity of 70 S.U.V. at 100° F. with sulfur and heating under pressure at 75 to 150 pounds per square inch. It was possible to combine as much as 10 per cent of sulfur with mineral oil in this manner. Nelson mixed the sulfur and oil and heated

[37] U. S. Patent 419,726 (Jan. 21, 1890). See also British Patent 12,795 (Sept. 4, 1888).
[38] U. S. Patent 632,363.
[39] U. S. Patent 1,367,428 (Mar. 25, 1919). See also British Patent 129,132 (July 15, 1918).
[40] U. S. Patent 1,401,760 (Mar. 22, 1920).
[41] U. S. Patent 1,516,879 (Nov. 25, 1924).
[42] U. S. Patent 1,604,068 (Oct. 19, 1926).
[43] U. S. Patent 1,929,955 (Oct. 10, 1933).

at temperatures between 350 and 450° F. for 2 to 10 hours under the pressure mentioned above. He usually obtained a viscosity increase of 25 to 100 seconds S.U.V. at 100° F. The hydrogen sulfide formed was extracted by means of a vacuum or the oil was blown with sulfur dioxide. Later, Nelson [44] obtained a similar patent extending his temperature ranges and providing for cooling.

Sulfur chloride was utilized in another process [45] for making cutting oil. A base was first made by treating 90 parts of vegetable or animal oil with sulfur chloride in the presence of about 6 per cent of mineral oil or grease. About 25 per cent of this base was then blended with 75 per cent of a low viscosity mineral oil in the preparation of the finished cutting oil. Gallsworthy [46] developed what he termed "a stable cutting oil" by treating a solution of free sulfur in mineral oil with sulfur chloride at specified elevated temperatures. Cushman [47] and Doell developed a formula for a cutting oil consisting of free sulfur dissolved in aromatic mineral oil capable of being completely miscible with sulfur dioxide. Adams [48] assigned to the Standard Oil Company of Indiana his patent covering a cutting oil consisting of mineral oil containing about 0.75 per cent natural sulfur, with added free sulfur in solution to bring the total sulfur content to at least 2 per cent by weight.

Various processes for chemically combining sulfur with fatty oils, which may subsequently be dissolved in mineral oil, have already been discussed; however, the following modern modifications of some of these processes will be of interest. Gregory's [49] process was to heat fish oil and sulfur together at 175° C., and then gradually increase the temperature to 220° C. over a period of one to two hours. The plastic mass so formed was mixed with additional sulfur and a small amount of lead oxide to accelerate the reaction. The mass was then heated at 155° C. under pressure of 50 to 40 pounds per square inch for one hour.

The use of pressure to assist combination of sulfur with organic material has been adopted by other investigators. For instance, Kaufman [50] heats from 5 to 25 per cent of sulfur with pine oil in an autoclave to effect combination. He [51] also found it possible to combine as much as 25 to 30 per cent of sulfur with pine oil by this process. Kobbe [52] prepared a sulfur base cutting oil by heating terpenes together with sulfur and then adding this base to mineral oil. Becker [53] assigned to the Standard Development Company his patent for a sulfur base lubricant consisting of 75 to 95 parts of a fatty compound with 25 to 5 parts of sulfur. These ingredients were combined by heating at a temperature of 430° F. The finished product was

[44] U. S. Patent 2,045,306.
[45] U. S. Patent 1,971,243.
[46] U. S. Patent 1,964,983 (July 3, 1934).
[47] U. S. Patent 1,806,933 (May 26, 1931).
[48] U. S. Patent 1,824,523 (Sept. 21, 1931).
[49] British Patent 123,114 (Nov. 5, 1917).
[50] U. S. Patent 2,043,961.
[51] U. S. Patent 2,043,962.
[52] U. S. Patent 1,844,400 (1932).
[53] U. S. Patent 1,590,800 (1926).

liquid at 300° F., having a S.U.V. at 300° F. of not less than 49. Davis [54] developed, for the Otis Elevator Company, a sulfur base cutting oil made by agitating 10 gallons of lard oil with 20 pounds of sulfur at 450 to 475° F. and then adding 4 to 7 gallons of 100 S.U.V. at 100° F. pale oil. After cooling, sufficient oil is added to make a fifty gallon batch for each ten gallons of lard oil. Lard oil, acetic acid, and small amounts of sulfur, soda, and borax, constitute the composition of a cutting oil patented by Marble.[55] Patch [56] has patented a long series of cutting compounds containing "tall oil," which is a by-product of European paper mills. It is obtained during the process of converting wood fiber into cellulose by the sulfate process. It is sometimes called Swedish rosin oil. It is claimed to have chemical properties ranging between those of fatty acids and naphthenic acids. Typical formulae proposed by Patch and Leaper are listed below:

Formula 1

	Per cent
Tall oil	20
Recovered wool grease (Degras)	5
Mineral oil	75

Formula 2

Oleic acid	6
Tall oil	8
35° Caustic soda solution	4
Ethyleneglycolmonoethyl (or monobutyl) ether	4

Formula 3

Distilled Tall oil	12
28° Paraffine oil	81
35° Bé. Caustic soda solution	3.6
Denatured alcohol	3.4

Formula 4

Sulphonaphthenic sludge, derived from the treatment of white mineral oils	20
Distilled Tall Oil	20
28° Paraffine oil	44
35° Bé. caustic potash solution	8
Carbolic crystals	5
Diethyleneglycol	3

Formula 5

Castor oil	10
Tall oil	6
Recovered wool grease (Degras)	1.7
Mineral oil	45.4
Water	27.4
35° Caustic soda solution	9.5

Formula 6

Tall oil	20
Mineral oil	45
Water	27
35° Caustic soda solution	8

[54] U. S. Patent 1,689,011 (1928).
[55] U. S. Patent 1,777,803 (1930).
[56] U. S. Patent 1,919,125 (1933).

Formula 7	Per cent
Crude tall oil	30
Mineral oil	30
Sulphonaphthenic sludge, from white mineral oil	20
Sulfur	15
Pine oil	5

This sulfur base cutting oil may be manufactured by heating a mixture of the compounds to a temperature at which the sulfur will be completely dissolved and combined with the other ingredients, for instance 350° F. In certain cases, it has been found advantageous to dissolve the sulfur in the saponifiable material, or in a portion thereof, by bringing it to a temperature in the neighborhood of 350° F., and thereafter slowly adding the sulfur during the heating. When the mixture of saponifiable material and sulfur is at the temperature given, and the sulfur has thoroughly dissolved in the saponifiable material, the mineral oil, or if desired a portion of the saponifiable material and the mineral, which has previously been heated to a temperature in the neighborhood of 300° F., is added to the mixture of saponifiable matter and sulfur, the mass being agitated during the mixing process. Variations in the percentage of sulfur used in the above formula as well as in the percentages of the other constituents may be made. If desired, the sulphonapthenic sludge may be replaced by other mineral oil and the pine tar may be omitted without departing from the invention.

E. P. BASES CONTAINING SULFUR

Churchill [57] has investigated sulfur containing lubricants for hypoid gears and has found that the seizure which may take place when the lubricant is of a normal type may be prevented by non-corrosive sulfur containing lubricants. As an example of his invention 88.5 parts by weight of corn oil is mixed with 11.5 parts of flowers of sulfur and the mixture heated while agitating until an exothermic reaction takes place. In general, this will occur at about 320° F. The source of heat may be removed at this point, and the temperature may continue to rise to about 400 to 420° F. Churchill believed that under these conditions addition products were formed. At 320° F. traces of hydrogen sulfide were evolved and at 400° F. the quantity was appreciable. Since small amounts of water were produced, it appears that some substitution of sulfur in the fatty molecule has taken place. The batch is stirred and cooled for about six hours or more until it is found that a sample of the base will not tarnish a copper strip immersed in it for a period of two minutes. Churchill claimed that by careful attention to details it was possible to prepare a base which would not corrode copper when immersed for three hours at 210° F. Cottonseeed oil, menhaden fish oil, linseed oil, and other vegetable or fish oils may be substituted for the corn oil. Churchill produced sulfurized oleic acid and found that this material would etch copper but would not tarnish or discolor it. (sic.). As

[57] U. S. Patent 1,974,299 (Sept. 18, 1934).

typical formulae for hypoid gear lubrication Churchill proposed the following:

Formula 1	Per Cent
Sulfurized base	20
150 to 160 vis. at 210° F. black mineral oil	79
Pine oil	1
Total	100

Formula 2	Per Cent
Sulfurized base	10
70 to 100 vis. at 210° F. black mineral oil	88.5
Pine oil	1.5
Total	100.0

Werder [58] has referred to the disadvantages of carbon tetrachloride and sulfurized fish oils as bases for hypoid gear extreme pressure lubricants. He proposed the treatment of vegetable and animal fatty oils with sulfurous chloride, (S_2Cl_2), in the preparation of a satisfactory base. In making his product, 90 per cent by volume of cottonseed oil was mixed with 10 per cent of sulfurous chloride at temperatures of 50 to 60° F. for approximately 1 hour. The mixture was then heated for about six hours at 150° F. During this time hydrochloric acid fumes are produced and agitation assisted in their elimination. For motor oils and greases about 6 per cent of this sulfurized base was recommended. A concentrated base may be made containing about 25 per cent of the base and 75 per cent of motor oil or grease. Farrington and Humphreys [59] have assigned to the Standard Oil Co. of California their patent for E.P. lubricants. Thiocarbonates were found to satisfy the requirements of extreme pressure lubrication and in admixture with hydrocarbon or fatty oils afforded lubrication of hypoid gears. The use of sulfonated anthracene has been patented [60] as an E.P. base. In the preparation of a sulfurized lubricant Becker [61] has segregated pressure distillate residue and treated it with sulfur while heating.

It is interesting to note that as early as 1893 the soaps of sulfuretted fatty oils were manufactured by Wetter.[62] He pointed out that fatty acids, resins, and esters of these belonging to the unsaturated series were capable of being heated with sulfur at temperatures ranging from 120 to 160° C. to produce chemical combination. He utilized alcoholic solutions of alkali for saponifying the sulfuretted fats. Combintaions of stearic acid and 0.5 to 5.0 per cent of sulfur have been investigated by Wisner [63] as E.P. bases. Palmer and Powers [64] have sulfurized terpene in the presence of an agent which will combine with the sulfur to form a basic reacting material. Beale [65]

[58] U. S. Patent 1,971,243 (Aug. 21, 1934).
[59] U. S. Patent 2,020,021 (Nov. 5, 1935).
[60] British Patent 193,722.
[61] U. S. Patent 1,812,766 (June 30, 1931).
[62] British Patent 10,707 (July 1, 1893).
[63] British Patent 247,520 (Feb. 10, 1935).
[64] U. S. Patent 1,926,687 (Sept. 12, 1933).
[65] U. S. Patent 1,990,365 (Feb. 5, 1935).

has assigned to the Standard Oil Company of Indiana his process for making top cylinder lubricants from alkylolamine salts of fatty acids; for instance, sulfurized oleic acid and the foregoing compound are blended in the proportions of 100 to 1000 cc. of the base with 5 gallons of gasoline. Gallsworthy [66] has developed a sulfur containing compound which serves as a cutting oil base, or for other purposes, obtained by the reaction of mineral oil, oxidized paraffine wax, and sulfur at temperatures in excess of 330° F.

Henriksen and Lincoln [67] treated orthotolyl stearamide with about 15 per cent by weight of sulfur chloride for ten minutes at 300° F. One per cent of the purified material was added to an S.A.E. 40 motor oil having a 20° F. cold test. It was found that a reduction of 5° F. was thereby obtained. The blend had a Timken test of 30.5 pounds (17,500 pounds per square inch).

COMMERCIAL METHOD FOR THE PREPARATION OF SULFURIZED LARD OIL

This method requires the use of a small still equipped for injection of superheated steam. Lard oil, cottonseed oil, or other oil, is charged and steamed to 350 to 400° F. and maintained at that temperature for about three hours. Then, 12 per cent by weight of flowers of sulfur are added to the still (it may be pumped in as a slurry with oil), and agitated with steam for about one half hour at 350° F. Finally, the temperature is increased by the means of fire and steam to 400 to 410° F. Steaming is continued at this temperature for 1½ hours. The batch is steamed at 320 to 350° F. for three hours to remove traces of hydrogen sulfide. This product, when made with lard oil, will have the following characteristics:

Sulfur, percent by weight	8.43
Cu strip corrosion test, 3 hours at 212° F.	Slight stain only
S.U.V. at 210 ° F.	336
Timken test, on 20 per cent by volume of sulfurized lard oil and 80 per cent S.R. stock; 400 feet per minute:	
O.K.	17,140
Failed	20,000

E.P. BASES CONTAINING CHLORINE OR OTHER HALOGENS

A wide variety of organic compounds containing chemically combined chlorine have been patented as agents for improving the film strength of lubricating oils and greases. Even carbon tetrachloride, which of course is too volatile for use in most lubricants, has been referred to as an extreme pressure base. Sommer,[68] in 1891, prepared chlorinated fats as bases for lubricants. Later a lubricating composition was patented [69] containing a minor portion of a chlorine substitution product of a waxy carbocyclic compound in which the halogen was attached to a ring. In 1912, Aylworth [70]

[66] U. S. Patent 1,987,397.
[67] U. S. Patent 1,959,054 (May 15, 1934).
[68] U. S. Patent 461,513 (1891).
[69] U. S. Patent 1,029,254.
[70] U. S. Patent 1,028,926 (1912).

developed non-inflammable lubricants containing the chlorine substitution products of naphthalene. In 1929, Carpmael [71] developed various chlorinated paraffines for lubricating and greasing textile fibers. It is quite possible that these materials would also have E.P. properties when blended with lubricating greases and applied to metal parts. Some examples of Carpmael's chlorine compounds follow:

1. **Trichlorinated hard paraffine.** Obtained by direct chlorination of hard paraffine of average molecular weight 367, is heated in an autoclave with aqueous sodium hydroxide. The product, which may be added to emulsions used in the textile and leather industries, contains hydroxy groups and one chlorine atom per molecule and is unsaturated.
2. **Tetrachlorinated hard paraffine.** Obtained by chlorination of hard paraffine of average molecular weight 367, is heated in an autoclave with aqueous sodium carbonate. The product is saturated and contains three chlorine atoms per molecule.
3. **Heptachlorinated hard paraffine.** Obtained by chlorination of a hard paraffine of average molecular weight of 367, is heated in an autoclave with potassium carbonate.
4. **The same chlorinated hard paraffine used in Example 3.** This is heated under reflux with aqueous sodium hydroxide.
5. **The same trichlorinated hard paraffine used in Example 1.** This is heated in an autoclave with milk of lime.
6. **A tetrachlorinated soft paraffine.** Obtained by chlorinating soft paraffine of average molecular weight of 300, is heated in an autoclave with aqueous barium hydrate.

Vidal [72] has prepared lubricants from a modified acid, obtained by treating oleic acid with alkaline hypochlorite or hypobromite, and decomposing the product with acid. This modified acid is treated alone, or in admixture with organic substances, with a further quantity of hypochlorite or hypobromite to obtain liquids or thick pastes which are completely soluble in water. The pastes may be dried without loss of solubility. Organic substances which may be used are hydrocarbons such as petrol, kerosene, petroleum, solar oil, pale oil, petrolatum oils, paraffine, benzol, toluene, xylene, cumene, cymene, coal oil, chlorinated hydrocarbons such as carbon tetrachloride, chloroform, di-, tri-, tetra-, or penta-, chlorethane or chlorethylene, chlorbenzene, ortho or para dichlorbenzene. Prutton [73] has assigned his patent covering halogenated extreme pressure bases, to the Lubri-Zol Corporation. He has developed the use of cholrinated carbon ring compounds, added to oils and greases in quantities up to 20 per cent, and claims that such components act on bearing surfaces to form a film which is not ruptured under high pressures.

Vallee [74] has assigned to the Sharpless Solvents Corporation his patent for an E.P. base having a high penetrating value consisting of amylene dichloride. Ralston [75] has studied the Friedel-Crafts' synthesis reaction products of coal tar fractions and a fatty acid chloride having at least six carbon atoms.

Henriksen and Lincoln [76] have made an elaborate study of halogenated

[71] British Patent 343,948 (Sept. 11, 1929).
[72] British Patent 289,001 (Apr. 19, 1927).
[73] U. S. Patent 1,986,651 (Jan. 1, 1935). See also U. S. Patent 1,986,645 (Jan. 1, 1935).
[74] U. S. Patent 1,926,961 (Sept. 12, 1933)
[75] U. S. Patent 2,033,546 (Mar. 10, 1936).
[76] U. S. Patent 1,936,670 (Nov. 28, 1933).

organic compounds as means for increasing oiliness and film strength. Many of these compounds are worthy of further consideration as agents for improving the lubricating qualities of greases. Halogenated diphenylene oxide was one of the materials found by these investigators to have a high Timken film strength. Other compounds investigated were the halogenated aromatics (a chlorinated ester condensation product with an aromatic, and chlorinated glycerin condensation product with an aromatic hydrocarbon),[77] combinations of halogenated organic acids and diphenylene oxide,[78] condensation products from halogenated waxes, and halogenated organic acids,[79] halogenated esters of organic acids with monohydric alcohols, and halogenated esters of organic acids with dihydric alcohols,[80] condensation products formed by the action of alkali condensing agents on two halogenated waxes,[81] halogenated amides of aliphatic acids plus sulfo chlorinated amides of aliphatic acids,[82] and low viscosity oils plus halogenated fatty acids.

By treating commercial orthotolyl stearamide with excess chlorine to increase its weight by 10 to 20 per cent, Henriksen and Lincoln found that temperatures of approximately 250° F. were required. The chlorinated orthotolyl stearamide when blended with 99 per cent of S.A.E. motor oil gave a product having a Timken film strength of 28 pounds (16,250 pounds per square inch). These investigators also studied primary, secondary and tertiary amines converted to amides with suitable organic acids. They investigated organic amides obtained by replacing 1 hydrogen atom in ammonia with an organic acid radical, or by replacing the OH of the carboxyl group by NH_2. Ralston, Christensen, Hoffman, Selby, and Conquest [83] have also made studies of the nitriles, amides, and ketones, as agents for imparting extreme pressure properties. Henriksen and Lincoln found that all of the above amides when chlorinated, or sulfurized, were capable of increasing film strength. Stearic acid may be chlorinated by the direct action of chlorine and further chlorinated as follows: 150 grams of dichlorstearic acid, and 70 grams of diphenylene oxide are dissolved in 300 cc. of carbon disulfide. Thirty-five grams of anhydrous aluminum chloride is then added slowly and the mixture refluxed for three hours at 120° F. The mixture is then treated with dilute nitric acid and washed with ether. The final condensation product will contain about 6 per cent of chlorine, and when 1 per cent of it is added to a mineral oil the Timken film strength may be increased from 18 to 54.9 pounds (10,500 to 31,250 pounds per square inch).

Lard oil (100 grams) may be chlorinated for about 12 hours and then mixed with 10 grams of naphthalene and 10 grams of anhydrous aluminum chloride. Excess chlorine is driven off by heating. The Friedal-Crafts'

[77] U. S. Patent 1,939,993 (Dec. 19, 1933).
[78] U. S. Patent 1,939,979 (Dec. 19, 1933).
[79] U. S. Patent 1,939,995 (Dec. 19, 1933).
[80] U. S. Patent 1,944,941 (Jan. 30, 1934).
[81] U. S. Patent 1,945,615 (Feb. 6, 1934).
[82] U. S. Patent 2,041,076 (May 19, 1934).
[83] *Natl. Petroleum News*, 59, (Dec. 9, 1936).

reaction products obtained when blended with 99 parts of mineral oil will produce a Timken film strength of 25,000 pounds per square inch.

Lincoln [83a] describes the chlorination of stearic acid. To the chlorinated acid, sodium ethoxide is added, and after the reaction is complete the sodium is removed from the carboxyl group with an inorganic acid, and the mixture freed from excess alcohol, inorganic acid, and inorganic salt by water washing. This condensation product when added to the extent of 1 per cent to motor oil or grease will reduce the static coefficient of friction by about 50 per cent and increase the Timken film strength from about 10,500 to 26,000 pounds per square inch. In the above reaction it is probable that the alkali condensing agent reacts with the acid hydrogen of the fatty acid forming a soap. The inorganic acid used hydrolizes the soap and frees the fatty acid. The final condensation product consists of two or more molecules of halogenated fatty acid together with free fatty acid and free halogenated acids. The sodium ethoxide condensing agent removes the halogen from (say) two halogenated acids freeing the alkali halide and opening two bonds on two molecules. These combine and form a molecule containing two or more carboxyl groups.

As an example of a halogenated wax-halogenated organic acid condensation product used for increasing film strength Lincoln and Henriksen [83b] have described the following procedure for the preparation of such material from paraffine wax, carnauba wax, beeswax, animal and vegetable oils:

A 124 to 126° F. melting point hydrocarbon wax was chlorinated until the chlorinated product had a melting point of 60° F. This chlorination was done by direct addition and substitution. In another container a batch of commercial stearic acid was chlorinated by direct substitution until the chlorinated product had a cold test of 13° F. Equal quantities of these two chlorinated products were mixed and treated with sodium ethoxide in an alcohol solution. Sufficient sodium ethoxide was added to cause the removal of part of the halogen from the chlorinated wax and from the chlorinated stearic acid in addition to that required to form the sodium soap of the fatty acids present. When the chlorine atoms are removed from the two molecules they can combine with each other or be saturated with the free OC_2H_5. It is thus possible to substitute a number of fatty acid molecules on a saturated hydrocarbon molecule. Other type molecules are also present.

After condensation is complete, the alcohol is removed by distillation and the sodium atom forming the soap with the carboxyl group is replaced by treating with dilute hydrochloric acid. The product is then water washed to remove traces of hydrochloric acid. A solvent, such as a light naphtha or other solvents, may be used to facilitate washing. After washing, the solvent is removed by distillation. The product may be used after the removal of the solvent or may be further purified by low pressure distillation. When 1.5 per cent of this condensation product was added to 150 S.U.V. at 210° F. steam refined cylinder stock, the Timken film strength

[83a] U. S. Patent 1,939,994.
[83b] U. S. Patent 1,939,995.

at 390 feet per minute was increased from 10,500 pounds to 23,000 pounds per square inch.

E.P. BASES CONTAINING PHOSPHORUS

Bunge [84] and Macura prepared phosphorus compounds of low temperature coal tar fractions. Other halogens and compounds were suggested including phosgene, boron trichloride, phosphorus oxychloride, phosphorus sulphochloride, other chlorides of phosphorus, silicon tetrachloride, chromyl chloride, sulphuryl chloride, and chlorides of tin, antimony, arsenic, and bismuth. Graves [85] has studied the esters of phosphoric acid. He has pointed out that certain phosphates, such as oleyl lauryl phosphate, when added to lubricating oils assist in maintaining an effective lubricating film at excessively high unit pressures. A patent describing the phosphates proposed by Graves has been assigned to the E. I. du Pont de Nemours & Company, Inc. du Pont bases for lubricating oils and greases are described below:

No. 22.

This material is a liquid easily soluble in oil. It melts at 12° to 14° C. and boils at about 305° C. When added to lubricating oil it does not appreciably change the physical characteristics of the oil. When compounded with 1 per cent of this base a medium oil will withstand 20 to 30 pounds load (35,000 pounds per square inch) on the Almen machine and 40 to 45 pounds on the Timken tester.

The torque developed in the Almen test is low (about 2.8 pounds feet at 35,000 pounds per square inch), being similar in this respect to the better chlorine extreme pressure lubricant bases. It is non-corrosive, and does not form corrosive decomposition products. The wear test on the Timken machine showed 0.016 of a square inch, with an actual bearing load of 18,700 pounds at the end of this test.

No. 152.

This extreme pressure lubricant base is a pale yellow solid soluble to about 1 per cent in most oils. It melts at about 135° C. It has no effect on the physical properties of the oil with which it is compounded except to increase the color slightly. It is not quickly soluble so that mixing is preferably done by heating the oil.

The Almen test on a 0.25 per cent solution of this material in a medium oil indicates a film strength of 25 to 30 pounds (40,000 pounds per square inch). The torque developed is lower than that found with chlorine base lubricants.

The Timken test indicates a film strength of over 50 pounds at 0.25 per cent concentration. The wear rate (0.026 with a final pressure of 11,500 pounds per square inch) is much lower than that for chlorine base oils (0.035 with a final pressure of only 8,600 pounds per square inch), while the film strength is considerably higher.

No. 153.

This base is a liquid which congeals at about −25° C. The boiling point is about 290° C. at 10 mm. pressure. It readily dissolves in oil to upward of 10 per cent. The Almen test on 1 per cent solutions shows a film strength of 20 pounds (30,000 pounds per square inch) and the torque is satisfactorily low.

On the Timken machine this assistant, in 1 per cent concentration, has a film strength of 40 to 45 pounds. The wear test showed 0.011 of a square inch, with a final pressure of 27,200 pounds per square inch. This is the lowest wear value we have encountered with any extreme pressure lubricant base.

The tests with this assistant indicate it is very suitable for use in motor oils for increasing the adhesion of the oil. It does not affect the properties of the oil body, is non-corrosive (apparently also inhibiting corrosion somewhat), odorless and is effective at relatively low concentrations, *i. e.*, 1 per cent or less.

[84] British Patent 285,000.

[85] U. S. Patent 2,005,619 (June 18, 1935).

No. 162.

This assistant is a liquid miscible with oil at 25° C. It dissolves readily and has no apparent effect on the body of the medium. At concentrations of 0.5 to 1 per cent it has a film strength on the Almen machine of over 40 pounds and develops exceptionally low torque, about 2.7 pounds feet at bearing pressures of 65,000 pounds per square inch.

This torque is less than that developed by the best chlorine type bases and only half that found with sulfur treated oils. It is non-corrosive and, in fact, somewhat inhibits corrosion.

When tested on the Timken machine the film strength of 1 per cent solutions is about 30 pounds, the wear is low, 0.016 of a square inch with a final pressure of 18,700 pounds per square inch. This assistant is more suitable for use in gear lubricants than in motor oils because of its high film strength and low friction at comparatively low speeds.

The properties of the du Pont extreme pressure lubricant bases are summarized as follows:

EPL Base	Melting Point, ° C.	Boiling Point, ° C	Specific Gravity	Solubility at 25° C. in S.A.E. 30 Oil— Paraffinic	Naphthenic
22	12 to 14	305	1.175	7.5	12.5
152	134 to 136	...	....	1.0	1.3
153	−25	290	1.170	14.0	17.5
162	−3 to +8	...	.980	miscible	miscible

Naamlooze Vennootschap de Bataafsche Petroleum Maatschappij has patented high pressure lubricants containing phosphoric or arsenic compounds which preferably have an organic radical with a long chain, or chains, and in the form of an ester, other than those in which the phosphorus or arsenic atom in the compound is joined by an aryl radical by means of sulfur or an oxygen atom.[86] Boughton[87] has pointed out that phosphoric acid lubricants including moistened phosphorus pentoxide, (P_2O_5), 85 per cent orthophosphoric acid, (H_3PO_4), and various mixtures comprising chiefly substances containing the orthophosphate radical, (PO_4), have long been used as lubricants for glass surfaces. Unfortunately, they are subject to being highly hydroscopic with the result that they absorb water from the air, become thin and tend to lose their lubricating value. He, therefore, suggested the use of metaphosphoric acid, or its alkali metal salt sodium metaphosphate, ($NaPO_3$), in water soluble form, such as that prepared by heating mono sodium dihydrogen phosphate to the fusion point and redness where the following reaction occurs:

$$NaH_2PO_4 = NaPO_3 + H_2O$$

Mixtures with borates were also suggested. In one example 100 grams of water-soluble sodium metaphosphate are dissolved in 500 cc. of water and 20 grams of sodium borate are added and dissolved. The solution is then boiled to a volume of about 150 cc., and 10 cc. of 85 per cent orthophosphoric acid is added. The mixture is then boiled until the boiling temperature is about 108° C., when it is allowed to cool.

The Standard Oil Development Company has patented the use of tricresyl phosphate as an extreme pressure agent for addition to other lubri-

[86] British Patent 424,380 (Feb. 20, 1935).

[87] U. S. Patent 1,917,089 (July 4, 1933).

Laboratory Tests on E. P. Gear Oils containing Sulfur, Phosphorus and Chlorine

Sample	Trans. S.A.E. 250	90% Trans. 250 2% Acidless Tallow Oil 8% Floyd B.	10% Lubrizol Semi-EP Base 90% 650 Penn.	10% Lubrizol Full EP Base 90% 650 Penn.	2% Tricresyl Phosphate 98% 650 Penn.	1% DuPont GD-162 99% 650 Penn.	1% DuPont GD-153 99% 650 Penn.	100% 650 Penn.	Anglamol No. 40—5.7 650 Penn.—91.3 100 Red—3.0	Chlorinated Wax 8% 650 Penn 92%
Composition (per cent by weight):										
Flash, o.c. ° F.	410	....	535	505	585	500	570	....	480	540
Fire, ° F.	....	....	655	645	680	650	665	....	....	....
Gravity, ° A.P.I.	18.4	17.6	22.6	22.2	23.7	24.4	24.2	24.6	22.0	18.4
S.F.V. at 100° F.	776	656	289	285	348	378	353	382	320	321
S.U.V. at 210° F.	256	257	172	170	186	206	200	204	174	176
Centrifuge Test:										
Per cent separation, 16 hours	None	None	0.05	0.05	None	None	None	None	.02	.05
Pour point, ° F.	....	....	50	20	25	25	30	40	45	50
Cu. Strip Corrosion Tests:										
3 hours at 210° F.	....	....	O.K.	Slight Corrosion	O.K.	O.K.	O.K.	O.K.	O.K.	O.K.
24 hours at 300° F.	....	....	Corrosion	Corrosion	O.K.	O.K.	Stained	O.K.	....	....
Steel Strip Corrosion Tests:										
24 hours at 300° F.	....	....	Corrosion	Corrosion	O.K.	O.K.	O.K.	O.K.	....	....
Timken Film Strength:										
O.K. at pounds per square inch	....	....	17,140	20,000	17,140	17,140	17,140	7,140	22,860	22,860
Failed at pounds per square inch	....	....	20,000	22,860	20,000	20,000	20,000	8,575	25,700	25,700
Tests after Oxidation, Shell Sludging Test, at 250° F., 8 Days										
Centrifuge Test:										
Per cent separation, 16 hours	0.05	0.05	0.1	0.1	0.1	0.1	0.1	0.05	None	.03
S.U.V. at 210° F.	465	611	222	223	212	243	223	237	196	200
Increase in viscosity during sludging test	209	354	50	53	26	37	23	33	22	24
Costs of E.P. base, cents per pound (freight charges not included)	....	....	15	22	16	100	42	....	10.75	20
Approximate cost of E.P. base in 1 gallon gear oil (freight charges not included)	....	....	11.5c	16.1c	2.4c	7.6c	3.2c	....	4.7c	13.0c
Nature of E.P. base	....	S	Cl.	Cl.	Ph.	Ph.	Ph.	....	Cl.	Cl.

cants.[88] van Dijck has assigned to the Shell Development Company a patent also claiming extreme pressure lubricant bases containing phosphorus and as alternatives, arsenic, selenium, or tellurium, or their derivatives.[89] Tricresyl phosphate is mentioned as a typical example of a phosphate E.P. base. Mono-carboxylic esters are also referred to. The du Pont Company has also developed esters of phosphorus acid for use in high pressure lubricants. They have carried out extensive experiments of blends of straight mineral oil, sulfur containing oil, and various phosphates, phosphites, and thiophosphates, as well as nitro-, and amine-, substituted compounds.

Worner [90] has prepared halogenated phosphorus compounds for use in lubricants.

Diphenyl and diphenyl oxide, as well as their derivatives, have been proposed as bases for improving film strength. According to Dow,[91] they are particularly useful as ingredients for lubricants for steam cylinders, internal combustion engines, valve stems, piston rod packings and the like. He recommended them for use at temperatures above 600° F. Diphenylene oxide boils at 287 to 288° C. and is fluid at temperatures above 80° C. About 1 pint of diphenylene oxide per gallon of cylinder oil was recommended by Dow. In a later patent [92] diphenyl oxide, having a boiling point of about 500° F. and considered suitable for lubricating purposes at temperatures up to 850° F., was claimed. The same proportions as were recommended in the previous patent were retained. The use of diphenyl, naphthalene, pyrene, picene, and phenanthrene were also referred to. Grebe and Sloesser [93] have developed a method for preparing diphenyl oxide for use as a lubricant. It consists of heating the commercial material at about 400° C., and below its decomposition temperature, distilling off a product at 200 to 350° C. at an absolute pressure of 5 to 10 mm. of mercury.

ORGANIC COMPOUNDS FOR IMPROVING THE TEMPERATURE-VISCOSITY CHARACTERISTICS OF LUBRICATING GREASES

It is well known that, aside from the effect of the soap base on the consistency and viscosity of soap base lubricating greases at low temperatures, the Viscosity Index or temperature-viscosity susceptibility of the lubricating oil constituent is a most important factor. It is for this reason that considerable research should be carried out with a view to increasing the Viscosity Index of oils used in greases. The use of high V.I. oils will not only improve such factors as gear shifting ability at low temperatures, but will tend to maintain higher working viscosities during summer when the lubricant temperatures may become quite high. Polymerized isobutylene

[88] British Patent 424,380 (Feb. 20, 1935).

[89] Canadian Patent 357,650 (May 5, 1936). See also Dutch Patent 36,239 (Sept. 16, 1935) and French Patent 770,289 (June 25, 1934).

[90] British Patent 1,719 (Jan. 23, 1906).

[91] U. S. Patent 1,867,968 (July 19, 1932).

[92] U. S. Patent 1,918,593 (July 18, 1933).

[93] U. S. Patent 1,905,850 (Apr. 25, 1933).

is one of the most promising materials for improving the temperature-viscosity characteristics of greases. Other materials are condensed paraffines, rubber of diolefines, more or less condensed. Johnson [94] adds polymerized isobutylene to a mineral oil and treats the mixture with a condensing agent of the Friedel-Crafts' type. The oil is mixed with a small amount of the polymerized isobutylene and stirred and heated at 40 to 80° C. with aluminum chloride. The condensing agent is separated from the resulting oil by settling or washing with water. The oil is then treated with a bleaching earth and finally is filtered. The hydrocarbon oil may be treated with the condensing agent alone, and then mixed with a solution of polymerized isobutylene, in the same or a different hydrocarbon oil, and the mixture heated to complete condensation. For example, an oil obtained by splitting off hydrogen chloride from chlorinated paraffine wax or by cracking hard or soft paraffine wax is heated at 60° C. with iron, tin, or zinc chloride. A solution of polymerized isobutylene in lubricating oil, or cracked gas oil, is added and the mixture heated at 80° C., after which the sludge is allowed to settle and the lubricating oil is decanted. A diluent such as ligroin may be used. The product may be mixed with ordinary lubricating oils to improve the temperature-viscosity curve. Johnson [95] has polymerized unsaturated fatty oils such as soya bean oil, olive oil, palm oil, or bone oil, by means of Friedel-Crafts' agents at 0 to 200° C. to obtain materials for improving the Viscosity-Index of lubricants. Condensing agents specified may be aluminum chloride, zinc chloride, tin tetrachloride and boron fluoride. Conversion may be effected by heating the fatty substance: (1), to 300 to 500° C., preferably in the presence of a splitting catalyst, such as copper or copper chromite, and may be in presence of hydrogen and under pressures up to 50 atmospheres; or (2), to 200 to 300° C., in the presence of hydrogen at 50 to 300 atmospheres and reducing catalysts, *e. g.*, copper, copper chromite, or mixtures of copper oxide and aluminum oxide, and dehydrating the alcohols formed. The condensation may be with aromatic hydrocarbons, gaseous or liquid olefines and mineral or tar oils. In examples: (1), 100 kg. of soya bean oil is led over a copper aluminum catalyst at 425° C. and the oxygen-free product is stirred at 120° C. with 6 kg. of aluminum chloride for 2 hours; (2), 100 kg. of a mixture of olefines, obtained by the destructive hydrogenation of olive oil at 230° C. and 200 atmospheres in presence of copper chromite, is polymerized by heating to 120° C. for 1 hour with 8 kg. of aluminum chloride. 100 kg. of a product of the destructive hydrogenation of brown coal tar is added, and the mixture stirred for 1 hour at 90° C.

Johnson [96] has also condensed such materials as paraffine wax, montan wax, etc., with lead, sodium and ammonium oleates and stearates, together with rubber, balata, styrene and indene, using a high frequency electrical field to form products having molecular weights of 500 to 10,000. He [97]

[94] British Patent 411,198 (Dec. 23, 1932).
[95] British Patent 394,073 (Feb. 26, 1932).
[96] British Patent 407,379 (June 10, 1932).
[97] British Patent 415,526 (Apr. 18, 1933).

has also condensed stearyl and cetyl alcohol utilizing thionyl chloride as a condensing agent. To increase the Viscosity-Index and to lower pour points highly polymerized vinyl esters of oleic, stearic or palmitic acids have been suggested.[98] Sloane [99] has made condensation products, having an average molecular weight greater than 1200, for use as pour point depressors from paraffine wax and naphthalene. Condensation of the waxy hydrocarbons may be effected through their active derivatives such as olefines, or halides, in the presence of aluminum chloride, or zinc chloride, or by blowing with oxygen-containing gas, or by electrical methods. In a specific instance, chlorinated paraffine wax is condensed in a kerosene medium at about 70 to 150° F. The product consists of: (a), lighter fractions and unreacted wax boiling (say) up to 700° F.; (b), an inactive waxy material having a mean molecular weight below 1000 to 1200; and (c), the undistilled viscous or semi-solid highly active substance. The lighter fractions, (a), are removed by distillation, preferably under vacuum, and are suitable as lubricants. The active fraction, (c), is isolated from the remaining crude depressant, preferably by extraction with solvents of the class of "wax precipitants," in which the active fraction is insoluble. This extraction may be effected either by using sufficient of the solvent to dissolve both the wax and the lighter ends, or by using the solvent in such amount as to dissolve only the lighter oils, the wax being subsequently extracted from the residue by a larger quantity of the solvent.

In order to improve the Viscosity-Index of motor oils McCoull [100] has developed a method for blending a small amount of a low boiling point mineral oil with normal oil and adding about 2 per cent of oxidized paraffine wax, apparently to increase oiliness, as well as improve the temperature-viscosity relationship. Vinyl compounds polymerized to such an extent that their average molecular weight is at least 1000 have been proposed by Wulff [101] as a means of increasing the viscosity of mineral oils.

OXIDATION INHIBITORS

The effect of incorporating small quantities, usually less than 1 per cent, of anti-oxidants to soap base lubricating greases has been discussed briefly in the Appendix. It is believed that further research will prove the desirability of adding suitable anti-oxidants to lubricating greases made with calcium, sodium, or aluminum soap bases and intended particularly for anti-friction bearings. There is no doubt but that such compounds as will be discussed in the following sections, particularly various organic amines, diphenyl derivatives, certain phenolic compounds and derivatives, offer encouraging possibilities for decreasing oxidation and retarding rancidification of lubricating greases.

In a spring lubricant patented by Cabot [102] reference is made to colloid-

[98] British Patent 413,637.
[99] British Patent 402,253 (Dec. 8, 1932).
[100] U. S. Patent 1,767,147 (Jan. 24, 1930).
[101] U. S. Patent 2,020,714 (Nov. 12, 1936).
[102] British Patent 369,197 (Oct. 9, 1930).

ally dispersed asphaltic residues, a polar grease, such as degras, and aniline; the latter being termed an anti-oxidant. Murrill and Evans [103] have made extensive researches of products suitable for preventing the oxidation of fatty materials. In 1931 they proposed the use of the condensation product of an aldehyde and alkylene diaryl diamine. It was claimed that this material would inhibit the break down of fats and greases. They have also proposed the condensation product of an aldehyde with an alkylene-diaryldiamine.[104] Dialkylene diamines and piperazine [105] have been suggested by Murrill as fat stabilizers. He [106] has also patented compounds with fatty acid radicals plus small amounts of a material having the type formula $N(ROH)_2\ H_{3-x}$ in which R is an alkylene group and OH is attached to a carbon atom other than the one attached to the nitrogen atom. Tattersall [107] has had successful results with peracetic acid and its derivatives as fatty oil anti-oxidants. Morton [108] has proposed the derivatives of hydrogenated glyoxaline as fat anti-oxidants.

In 1927, Somerville [109] developed the use of para amido phenol as an anti-oxidant for transformer oils. Rogers [110] has successfully prevented excessive oxidation and the formation of emulsifying constituents in white oils by adding small amounts of alpha naphthol. He [111] also had positive results with 0.04 per cent of pyrogallol. As a fat anti-oxidant Murrill [112] has successfully developed the use of phenyl phenolate. He [113] has also proposed the addition of combinations of phenyl phenolate, phenyl phenol and organic bases as suitable fat stabilizers. Story's [114] anti-oxidants were beta naphthol and dibenzyl disulfide. Buc [115] has proposed an anti-oxidant consisting of phenolic compounds containing tertiary aliphatic hydrocarbons as side chains.

Various organic nitrogen compounds, particularly amine derivatives, have been found successful as fat anti-oxidants and color stabilizers. Adams [116] has assigned to the Standard Oil Company of Indiana his patent covering color stabilizers consisting of aliphatic amines having an alkyl group containing not less than 4 carbon atoms.

Evans [117] has reported that the addition of a small amount of an alkylene diaryl diamine to a lubricating oil would tend to stabilize it and prevent oxidation. He also pointed out that such compounds were applicable to vegetable and animal oil compositions containing fatty acids or glycerides,

[103] U. S. Patent 1,789,927 (Jan. 20, 1931).
[104] U. S. Patent 1,789,926 (Jan. 20, 1931).
[105] U. S. Patent 1,822,108 (Sept. 8, 1931).
[106] U. S. Patent 1,822,934 (Sept. 15, 1931).
[107] U. S. Patent 1,828,708 (Oct. 20, 1931).
[108] U. S. Patent 1,805,953 (May 19, 1931).
[109] U. S. Patent 1,634,054 (June 28, 1927).
[110] U. S. Patent 1,793,134 (Feb. 17, 1931).
[111] U. S. Patent 1,793,135 (Feb. 17, 1931).
[112] U. S. Patent 1,852,787 (Apr. 5, 1932).
[113] U. S. Patent 1,852,788 (Apr. 5, 1932).
[114] U. S. Patent 2,000,105.
[115] U. S. Patent 2,031,930.
[116] U. S. Patent 1,888.023.
[117] U. S. Patent 1,752,945 (Apr. 1, 1930).

including cutting oils and the like. He prepared the alkylene diaryl diamines by condensation of alkylene dihalides, such as ethylene dichloride, with aromatic amines such as aniline. From 0.2 to 0.5 per cent of the diamine was recommended to prevent excessive oxidation. Later,[118] he pointed out that alkylene diaryl diamines were also suitable as stabilizers and found that 0.5 per cent of ethylene-di-para-tolyl-diamine was particularly effective in preventing oxidation of oils and greases used for insulating electric cables. The action of these stabilizers is obscure but apparently they retard oxidation at elevated temperatures in the presence of oxygen and electrical potentials. Ihrig [119] has proposed the use of small amounts of the nitrogenous bases from petroleum as suitable anti-corrosive agents in lubricants. Rogers [120] has successfully employed small amounts of amino compounds having a plurality of aromatic rings attached to the nitrogen atom. Hoffman and Dunkel [121] added 0.1 per cent by weight of toluonitrile to lubricating oils to prevent objectionable oxidation.

Voorhees' [122] color stabilizer was an aliphatic amine having the following constitution:

$$H_2N\ \begin{matrix} H & H \\ C & C \\ H & H \end{matrix}\ NH_2$$

Shoemaker's [123] anti-oxidant had the following structure:

$$X\cdot\left(N\begin{matrix} R \\ R_1 \end{matrix}\right)_2$$

in which X is a divalent alkaline metal such as calcium, R is an alkyl group and R_1 is an aryl or aralkyl group.

Koegebehm [124] has investigated the use of hydroxy diphenyl in quantities less than 1 per cent as a means of preventing oxidation of mineral oils and improving their film strength characteristics.

Of particular interest to grease chemists are the oxidation inhibitors for fats and soaps developed by Calcott [125] and assigned to du Pont de Nemours and Company. From 0.001 to 0.1 per cent of hydroxy diphenyl methane was found by this investigator to be particularly advantageous in preventing rancidification. From 0.001 to 0.1 per cent of para hydroxy diphenyl ether [126] was also discovered as being a suitable anti-oxidant. Calcott [127] proposed di-para-para'- hydroxy diphenyl. His associates, Douglass and Walker,[128] developed the use of 0.001 to 0.2 per cent of para: para'-diethoxy diphenylamine to prevent the rancidification of soaps and soap stocks.

[118] U. S. Patent 1,752,946 (Apr. 1, 1930).
[119] U. S. Patent 1,768,910 (July 1, 1930).
[120] U. S. Patent 1,774,845 (Sept. 2, 1930).
[121] U. S. Patent 1,856,186 (May 3, 1932).
[122] U. S. Patent 1,973,676.
[123] U. S. Patent 2,012,918 (Aug. 27, 1935).
[124] U. S. Patent 1,784,359 (Dec. 9, 1930).
[125] U. S. Patent 1,913,367 (June 13, 1933).
[126] U. S. Patent 1,913,368 (June 13, 1933).
[127] U. S. Patent 1,993,771 (Mar. 12, 1935).
[128] U. S. Patent 1,987,321 (Jan. 8, 1935).

Traces of mercaptans, having at least seven carbon atoms, have been proposed by Sloane [129] as suitable color stabilizers for white oils. According to Hoffman and Dunkel [130] lubricating, transformer, and other oils are protected against the action of air, especially when under raised temperature conditions, by the addition of quantities not exceeding 2 per cent of an acid amide, such as urea or oxamide, or a nitrile, either alone or in mixture with each other, or with the substances. In an example, 0.1 per cent of urea is added to a compressor lubricating oil.

As an anti-corrosive agent for coating metallic surfaces Fowler and Edser [131] have proposed a material comprising neutral soap from wool fat (adeps lanae) and a non-volative thinning agent, such as a lubricating oil or a mineral grease such as petrolatum.

[129] U. S. Patent 1,966,050 (July 10, 1935).
[130] British Patent 284,616 (Nov. 24, 1925).
[131] British Patent 258,795 (Mar. 24, 1925).

Chapter XIII

Miscellaneous Organic Lubricants

In this chapter those lubricating greases and compositions will be presented which depend on fats and blown oils, natural and synthetic waxes, resins, rubber, asphaltic residues, coal tar, ammonium compounds, and carbohydrates, for their consistency or some special property. Such lubricants will, of course, contain no predominating quantity of metallic soap, or solid lubricants, except as incidental components. In view of their miscellaneous character, lubricating compositions containing glycerol or its derivatives, and textile lubricants, which are frequently soluble oils with soap emulgators, will be discussed in this chapter.

SOAP FREE LUBRICANTS CONTAINING FAT

Many investigators have made extensive researches with a view to developing stable, miscible mixtures of castor oil with mineral oils. Since such products are normally fluids, and are more properly termed oil blends than greases, they will not be considered here.

In order to obtain consistent lubricating greases Hutchinson,[1] in 1873, mixed together mineral oils and animal and vegetable oils, and exposed the mixture in shallow pans to promote oxidation and thickening. Kingzett [2] made thick products by blowing turpentine and fatty material with air at 50 to 60° F. Mixtures of tallow and kerosene were patented in 1886.[3] Warren [4] blended greases or fat with vegetable oils or mineral oil and incorporated ozokerite or ozokerite pitch. Mixtures of animal fats, vegetable oils, camphor, and naphtha or mineral oil were proposed by Hennequin and Cayeux.[5] Even fish scales, combined with mineral oil, have been suggested as a lubricant by Saunders.[6] Sterratt [7] mixed together tallow, tar and sodium chloride solution to produce a lubricant.

Various mixtures of thick palm oil have been suggested as greases. Smith's [8] lubricant was:

	Parts by Weight
Palm oil	4½
Cottonseed oil	10
Crude petroleum	25

[1] British Patent 3,287 (Oct. 10, 1873).
[2] British Patent 274 (Jan. 24, 1876).
[3] U. S. Patent 351,352 (1886).
[4] British Patent 4,021 (Oct. 6, 1879).
[5] British Patent 11,679 (June 3, 1905).
[6] U. S. Patent 646,548 (1899).
[7] British Patent 16,411 (1898).
[8] U. S. Patent 214,594 (Oct. 28, 1878).

The residuum obtained from the distillation of palm oil, mixed with mineral oil has been proposed as a grease by Bowen.[9] A lubricant for automobile gears, consisting of a relatively large proportion of an unsaponified hydrogenated fatty oil, such hydrogenated castor oil, together with suitable amounts of mineral oil has been developed by Nill.[10] Various lubricants containing wool fat or degras to increase viscosity and improve lubricating qualities have been evolved from time to time. Boileau [11] recommended mixtures of neutral wool fat and mineral oil for the lubrication of transmissions of the planetary type, using as much as 12 per cent of the wool grease in his blend. Tongue,[12] also, used recovered wool grease and mineral oil mixtures.

The advantages of blown oils in increasing the adhesiveness and consistency of lubricants has not been fully appreciated. Blown castor, blown rape seed oil, and other oxidized and polymerized oils, may frequently be used with considerable success, particularly when service at low temperatures must be considered, or greases which must adhere well in the presence of moisture must be developed. Bijur [13] has suggested a chassis lubricant consisting of 3000 S.U.V. at 100° F. naphthenic pale oil and blown rape seed oil. Brewer [14] has developed greases which are semi-solid between the temperatures of −18 and 340° F. consisting of 9 parts of lubricating distillate with 1 part of stearin, the mixture having been distilled for 12 hours, extracted with benzol and the residue mixed with further amounts of distillate.

Wire Cable Compound

A satisfactory lubricant for wire cables may be made by following the formula given below:

	Per Cent by Weight
100 to 120 penetration blown asphalt	23.00
Degras	23.00
Raw linseed oil	5.30
Fish oil	2.70
300 S.U.V. at 100° F. naphthenic pale oil	23.00
Pine tar	23.00

LUBRICANTS CONTAINING WAX

Paraffine wax, on account of cheapness, is the most common wax used for mixing with other materials to produce consistent greases. Simple mixtures of lubricating oil and paraffine wax have long been used.[15] Mixtures of oil well rod wax with lard oil and castor oil were proposed by Hopkins.[16] Similar compositions consisting of mineral oil, castor oil,

[9] British Patent (1912).
[10] U. S. Patent 1,617,077.
[11] U. S. Patent 1,423,512 (Jan. 5, 1922).
[12] British Patent 2,326 (Aug. 20, 1862).
[13] U. S. Patent 1,944,164 (Jan. 23, 1934).
[14] British Patent 288,796 (Mar. 17, 1927).
[15] U. S. Patent 226,153.
[16] U. S. Patent 428,610.

olive oil and paraffine wax, were patented by Burns.[17] Clark[18] patented the following mixture:

	Pounds
Paraffine wax	1
Mineral oil	1
Alcohol	.001
Powdered talc	.005

A lubricant for use in conjunction with ordnance consisted of paraffine wax alone.[19] Tennant[20] made a lubricant consisting of paraffine wax and colza oil. Stearin and paraffine wax have been employed for making a shell, or casing, to be filled with a softer lubricant.[21] Humphries and Baulk[22] manufactured a loom grease in the form of sticks consisting of sperm oil solidified with paraffine wax. A lubricant for boring and cutting operations has been developed by Groves[23] and contained a dispersion of paraffine wax in mineral oil, turpentine, linseed oil, carbon disulfide and aniline. Constantinesso[24] has developed a hydraulic fluid consisting of 6 to 9 parts of paraffine wax and one part of a low pour point mineral oil. To improve lubricating value and produce a product suitable for cutting metals, Gallsworthy[25] has dispersed from 15 to 30 per cent of oxidized paraffine wax in mineral oil.

A lubricant, suitable for application to automobile door latches, which is adhesive yet of such a nature that it does not readily soil clothing has been patented by Rosen.[26] It is claimed that this lubricant remains solid at ordinary temperatures and is sufficiently sitcky and viscous to remain in place for long periods of time. This product has the following formula:

	Parts by Weight
Paraffine wax	50
Petrolatum	30
Lubricating oil	20

The paraffine wax is melted and the petrolatum added while stirring, the mineral oil being run in last. The liquid mixture is poured into molds of cylindrical shape producing sticks of grease for the purpose as indicated above.

Petrolatum, or vaseline, has been utilized in various greases of the past, and more recently has been favored as an ingredient of anti-corrosive compounds and slushing compounds for preventing rust of newly fabricated metal parts. On account of its low melting point, it has been superseded by soap base lubricants. In 1877, Thompson[27] suggested a formula for a

[17] British Patent 499 (Jan. 8, 1910).
[18] British Patent 5,901 (April 3, 1884).
[19] British Patent 8,598 (1884).
[20] British Patent 1,582 (1898).
[21] British Patent 16,628 (Dec. 18, 1884).
[22] British Patent 7,335 (Mar. 26, 1902).
[23] British Patent 358,202 (Aug. 14, 1930).
[24] British Patent 129,362 (Nov. 1, 1917).
[25] U. S. Patent 1,874,956 (Aug. 30, 1932).
[26] U. S. Patent 1,920,161 (July 25, 1933).
[27] British Patent 1,838 (May 11, 1877).

gun grease, consisting of 9 parts of petrolatum and one part of paraffine wax. Smith's [28] lubricating grease was prepared with petrolatum, sperm oil and benzol. Ames [29] has developed an automobile spring lubricant containing petrolatum, mineral oil and turpentine.

In addition to waxes derived from petroleum many other mineral, vegetable, and animal waxes have been used as ingredients of various lubricating compositions. In one case,[30] myrtle wax was utilized. For coating metallic cartridges mixtures of ozokerite, Japan and other waxes, such as paraffine wax, dissolved in gasoline, benzol or carbon disulfide, have been patented by the Auto Ordnance Company.[31] Hannay [32] has experimented with hydrocarbon textile lubricants composed of ozokerite, olive oil, paraffine wax and beeswax. To make montan wax more suitable for use in lubricating compounds, Pungs [33] has carried out a bleaching process consisting of oxidation with chromic acid. Emulsions of montan wax with water to form consistent lubricating greases have been developed by Heitmann.[34] Johnson [35] has developed wax-like lubricants by utilizing a decarboxylating catalyst on a higher aliphatic carboxy acid at temperatures of 100 to 450° C. He has also suggested the use of calcium montanate as a base for lubricating greases. Johnson [36] has subjected ceresin to silent glow electrical discharge to improve its characteristics as lubricating grease base. Carnauba wax has been utilized as a base for a grease suitable for lubricated plug valves. Webster [37] has assigned to the Merco Nordstrom Valve Co. his patent covering the following formula:

	Per Cent
Castor oil	77 to 78
Carnauba wax	23 to 22

Warren prepared lubricants by mixing ozokerite or ozokerite pitch with grease, or animal, vegetable or mineral oil. He [38] has proposed to stabilize such mixtures, to prevent their deterioration, by incorporating a small quantity of carnauba wax and mixing by means of compressed air. Carroll [39] has utilized mixtures of carnauba wax, petrolatum and coconut oil as lubricants for wood bearings.

Many lubrication engineers have expressed their preference for lubricants containing wool fat or degras. Fowler and Edser [40] have developed methods for purifying wool fat to improve its color and reduce its acid content, by treatment with sodium peroxide or sodium perborate solutions. Snow [41] blends wool grease with castor oil to prepare lubricating composi-

[28] U. S. Patent 627,278 (Feb. 6, 1899).
[29] British Patent 409,058 (Nov. 25, 1932).
[30] U. S. Patent 203,839.
[31] British Patent 141,594 (Aug. 25, 1919).
[32] British Patent 5,875 (1882).
[33] U. S. Patent 1,787,951 (Jan. 6, 1931).
[34] British Patent 232,259.
[35] British Patent 410,087 (Oct. 8, 1932).
[36] British Patent 410,833 (Aug. 17, 1932).
[37] U. S. Patent 1,559,592.
[38] British Patent 6,172 (1886).
[39] British Patent 11,946 (July 2, 1900).
[40] British Patent 253,995.
[41] British Patent 326,359 (Mar. 19, 1929).

tions. A lubricant composed of wool grease, paraffine wax and mineral oil has been suggested by Schmidt.[42] Herzog [43] has developed lubricants containing oily fractions from wool fat or the sulfonated derivatives of such a fraction.

Pungs [44] has suggested the preparation of solid lubricating greases by mixing tar oils, mineral, animal, or vegetable oils with bleached montan wax or a derivative of this wax; the free acids in the bleached wax being neutralized by means of an alkali. In an example, 80 parts of mineral oil, 5 parts of rape oil and 8.5 parts of a montan wax, bleached by oxidation by means of chromic acid and having the acid value 140, are mixed together and warmed to 100° C. until a perfectly clear solution is formed. The solution is allowed to cool down to about 75° C. and a solution of 1 to 1.2 parts of sodium hydroxide, in 5 to 6 parts of water, is added, in a thin stream while vigorously stirring, a perfect emulsion being formed immediately. The stirring is then interrupted and the mass allowed to cool, furnishing a solid grease with a flow point of about 90° C. Instead of the mixture of mineral and rape oil a fish oil, such as train oil, can be employed. In another example, 10 parts of a product obtained by esterification with ethylene glycol of a montan wax, bleached by oxidation and then showing an acid number of 20, are dissolved in 100 parts of mineral fuel oil, and mixed while stirring at about 90° C. with 0.5 part of sodium hydroxide dissolved in 12 parts of water. After a short time a solid grease of the consistency of soft soap is formed showing a flow point of 143° C.

Strather [45] developed a lubricant for tobacco presses, consisting of spermaceti and petrolatum. Honan [46] has assigned to the Westinghouse Electric Company his patent covering a compound containing 50 per cent of spermaceti wax and a volatile vehicle, together with petrolatum. Stryker [47] has assigned to the Texas Company the patent for a lubricant for planetary type transmissions, consisting of mineral oil and from 0.25 to 2 per cent of carnauba wax, bayberry wax, beeswax, japan wax, and spermaceti wax. Hutchinson's [48] lubricant, for a similar purpose, was prepared with motor oil, castor oil, castor machine oil, paraffine oil, neatsfoot oil, and alcohol. Winsor [49] made lubricants with beeswax, tallow, lubricating oil, and borax. Lepper [50] proposed the use of beeswax, rosin, lubricating oil, lard oil and tallow. For tapping aluminum and for cutting operations Kern [51] developed a lubricant consisting of beeswax, gasoline, and turpentine. It had a pasty consistency. A valve packing grease was recommended by Weisgerber [52] which had the following formula:

[42] U. S. Patent 1,780,315.
[43] U. S. Patent 1,543,384 (June 23, 1925).
[44] U. S. Patent 1,776,193 (Sept. 16, 1930).
[45] U. S. Patent 280,879 (1883).
[46] U. S. Patent 1,606,788 (Nov. 16, 1926).
[47] U. S. Patent 1,732,780 (Oct. 22, 1929).
[48] U. S. Patent 1,500,193 (July 8, 1924).
[49] U. S. Patent 173,840 (Jan. 29, 1876).
[50] U. S. Patent 278,248 (May 23, 1883).
[51] U. S. Patent 1,041,484 (Jan. 31, 1912).
[52] U. S. Patent 1,384,714 (Jan. 10, 1919).

Tallow	25 pounds
Soap	1 pound
Beeswax	1 "
Mineral oil	1 gallon

LUBRICANTS CONTAINING RESINS

The use of sodium abietate and calcium abietate in lubricating greases has been discussed in preceding chapters. In this section various mechanical mixtures of rosin with other lubricating materials will be presented. In 1863, Bauer [53] obtained an oil by distillation of pitch at 500° F. which he mixed with water and glue to produce an adhesive lubricant. Gladdin [54] mixed petroleum residues with rosin and lubricating oil. Vanderslice [55] proposed a method of forming a lubricant by boiling a suitable lubricating oil with wood, the rosin being extracted and dissolved in the oil. Patch [56] has developed a cutting lubricant containing "tall oil," also known as Swedish rosin oil and obtained as a by-product of the paper industry. The following formula [57] was patented in 1907:

	Parts by Weight
Stearin	1
Paraffine wax	2
Rosin	3
Mineral oil	4
Petrolatum	3

A steel mill neck grease has been developed by Donovan [58] having the formula:

Pitch	10 pounds
Black cylinder oil	3 gallons
Linseed oil	1 quart
Rosin	28 pounds

Claypoole [59] has assigned to the Texas Company his patent for a rubber ized cable oil having the following composition:

	Per Cent
Mineral oil	85 to 90
Rosin	10 to 15
Rubber	0.5 (max.)

LUBRICANTS CONTAINING RUBBER HYDROCARBONS

There is no doubt but that the addition of rubber hydrocarbons to lubricating compounds greatly increase their cohesiveness and adhesiveness. These characteristics were recognized by engineers even before petroleum lubricants were commercially available. When mineral lubricating oils became generally available it was not surprising to find that engineers made mixtures

[53] U. S. Patent 38,459 (1863).
[54] U. S. Patent 132,759 (Nov. 5, 1872).
[55] U. S. Patent 787,545 (Apr. 26, 1904).
[56] U. S. Patent 1,919,125 (July 18, 1933).
[57] British Patent 20,059 (Sept. 9, 1907).
[58] British Patent 238,630.
[59] U. S. Patent 1,807,060.

with gum rubber in order to increase the adhesiveness of the lubricant and render it more suitable and lasting for the rough machinery of nearly a hundred years ago. For instance, Hyde [60] heated para rubber and mineral oil at 80 to 150° F. in the preparation of an adhesive lubricant. Newton [61] developed a formula calling for caoutchouc and mineral oil heated together at 200° F. and combined with animal or vegetable oils, water, graphite or sulfur. Brinks [62] developed hydrocarbon solvents for dissolving caoutchouc and then dispersed the rubber in mineral oil. A dispersion of 1 per cent of caoutchouc (sheet rubber) in mineral oil was recommended as a machinery lubricant by Ewing and Coleman.[63] The following formula was proposed by Chard [64] in 1878:

	Ounces
25° Mineral oil	12
Caoutchouc	2
Sulfur	2
Graphite	4
Sodium carbonate	2
Beeswax	4

These components were blended at a temperature of about 140° F. and then stirred while cooling. Chard considered the rubber produced a more durable lubricant and served to hold the other ingredients in suspension.

A lubricant for steam engines was patented in 1880 and contained gambia, terra japonica, catechu and water.[65] Small proportions of India rubber were mixed with mineral and animal oils at ordinary temperatures by Brinks.[66] Rubber latex in liquid form has been suggested by Rigby [67] as a means of increasing the viscosity of mineral oils. The author has already discussed his patents covering the use of latex in soap base greases. A mixture of petroleum lubricating oil with India rubber, tallow, suet, and bergamot perfume has been proposed by Wilkins.[68] McLeod's [69] lubricating compound consisted of a mixture of 8 parts of rubber and 1 part of tallow. One ounce of rubber cement, five ounces of gasoline, and twenty-four ounces of lubricating oil were mixed together to form a base for increasing the viscosity of lubricating oils. About one ounce of this base per gallon of lubricating oil was recommended by Anderson.[70] J. Y. Johnson [71] has made comprehensive investigations of rubber hydrocarbons suitable for use in lubricants. He has proposed the destructive hydrogenation of rubber, polyisobutylene, etc., and the addition of these to various waxy lubricants in order to decrease their brittleness. He [72] has also made solid lubricating

[60] British Patent 2,668 (Nov. 27, 1855).
[61] British Patent 2,176 (July 31, 1862).
[62] U. S. Patent 444,626 (1891).
[63] British Patent 1,752 (June 18, 1870).
[64] U. S. Patent 199,789 (Dec. 22, 1877).
[65] U. S. Patent 261,180 (July 18, 1882).
[66] British Patent 17,163 (Oct. 30, 1889).
[67] British Patent 343,548 (Nov. 16, 1929).
[68] British Patent 2,012 (June 2, 1875).
[69] U. S. Patent 1,387,437 (Mar. 8, 1919).
[70] U. S. Patent 1,619,964 (Mar. 8, 1927).
[71] British Patent 411,893 (Dec. 12, 1932).
[72] British Patent 401,295 (May 6, 1932).

greases by mixing fats, grease, and the like, with polymerized styrene indene, terpene, cyclohexene, tetrahydronaphthalene, or hydrogenated polymers of styrene, rubber, diolefines, butadiene, or isobutylene.

To stabilize lubricating greases with regard to oxidation and to increase the viscosity of oils Johnson [73] has incorporated one or more of the following polymerized hydrocarbons having molecular weights over 800: polymerized indene, polymerized styrene, hydrogenated rubber or cyclo-rubber. hydrogenated polymerized diolefines such as butadiene or isoprene. A suitable addition is 0.5 to 30 per cent by weight. The polymerized hydrocarbon may be formed, *in situ,* by heating styrene, indene, or other polymerizable hydrocarbon in the lubricating oil. The hydrogenation may also be carried out, *in situ,* maybe simultaneously, with the polymerization. In examples: (1), 1, 2, and 4.5 per cent of styrene polymerized as an emulsion are added to different portions of a lubricating oil obtained by alkylating naphthalene by lower olefines; (2), 2 to 5 per cent of rubber hydrogenated in presence of nickel, while dissolved in cyclohexane, is added to spindle oil; (3), 3 kgs. of styrene and 100 kgs. of spindle oil are heated together for 20 hours at 150° C.; (4), 2 kgs. of solid polymerized styrene, obtained by heating styrene to 100° C. for several hours or by polymerizing at ordinary temperature an emulsion of styrene in presence of accelerators such as hydrogen peroxide or benzoyl, is added to lubricating oil.

Kormann and Milliff [74] have dissolved nitrated naphthalene and caoutchouc in toluol, applying sufficient heat to carry out an endothermic reaction and then dissolving this material in lubricating oils, and cooling to obtain a consistent grease. Higgins [75] has made a rubber and oil blend as follows:

Caoutchouc dissolved in gasoline	1 pound
Mineral oil	25 gallons
Coconut oil	8 ounces
Castor oil	50 gallons
Cottonseed oil	50 "

A cooling composition has been proposed by Strauch [76] consisting of a water in mineral oil emulsion, the emulsifying agent being rubber or asphalt. Allen [77] has made a lubricating grease for machinery bearings consisting of petrolatum mixed with, or dissolved in, an air-evaporating solvent such as petroleum naphtha, gasoline, benzol, or other solvent. To this lubricant he also added beeswax, or paraffine wax, dissolved in benzol or xylol. Werder [78] has developed an automobile spring and body lubricant consisting of heavy lubricating oil, a light petroleum solvent, and carbon dioxide, together with rubber cement, maintained under pressure in a suitable container. Greases of this kind are sometimes known as "balloon grease."

[73] British Patent 379,717 (April 29, 1931).
[74] U. S. Patent 1,918,403 (July 18, 1933).
[75] U. S. Patent 1,780,157 (Nov. 4, 1930).
[76] U. S. Patent 1,909,080 (May 16, 1933).
[77] British Patent 248,463 (Dec. 4, 1924).
[78] U. S. Patent 1,995,371 (Mar. 26, 1935).

LUBRICANTS CONTAINING BITUMENS AND MINERAL OILS

It is not our purpose to present a complete discussion of lubricating oil blends, but to merely point out a number of instances in which petroleum, or natural bitumens, mineral oil residue, or polymers, have been utilized either as fluid compositions or consistent greases. The addition of low viscosity, volatile solvents to heavy lubricants to improve fluidity at low temperatures is not at all new. In 1885, it was suggested that benzol could be added to mineral oil in order to reduce its pourpoint.[79] Wilson [80] has assigned to the Standard Oil Company of Indiana his patent for a low pourpoint oil having a S.U.V. at 100° F. of 180 and an initial boiling point of 350° F., with 11 per cent by volume distilled off at 460° F., combined with 6 to 10 per cent of kerosene. Whiting [81] has suggested a means of reducing the pourpoint of oils by blending in hydrocarbon compounds containing a substituted halogen atom such as benzyl chloride. Lockhart [82] has proposed a lubricant for engines consisting of a mixture of light and heavy lubricating oils. Recent research has indicated that greatly improved temperature-viscosity characteristics are possible when blending light and viscous materials together when compared with distillates containing none of the low boiling point material or residue. This is suggested as a possible means of improving the low temperature properties of lubricating greases. Lockhart [83] has proposed a steam cylinder lubricant consisting of petroleum residue together with approximately 4 per cent of a light naphtha, and normal lubricating oils lighter than spindle oil. Eastlake [84] has prepared lubricating oils which consist of a "lubricating element" substantially free from solvent with the spent residue of used lubricating oil. Watkins [85] found that asphaltic or naphthenic base oils were superior to others for lubricating furnace parts operating at a temperature of 2000° F. in a non-oxidizing atmosphere. Apparently this was on account of the nature of the carbon residue formed. Klees [86] has patented unsaturated, non-benzenoid hydrocarbons blended with naphthenic and paraffinic oils of similar boiling point. Secondary alcohol blended with lubricating oil has been claimed to improve spreading properties according to Lyons,[87] this blend having been proposed as an automobile spring oil. As a hydrocarbon commutator lubricant, having "spark preventing properties," Fisher [88] has developed blends of naphthene mineral oil with paraffine base oil and kerosene. They are scented with oil of wintergreen. Hobson [89] has developed steel mill hot neck greases containing volatile solvents which evaporate after application

[79] U. S. Patent 318,389 (1885).
[80] U. S. Patent 1,547,141.
[81] U. S. Patent 915,506 (Oct. 1, 1904).
[82] U. S. Patent 1,369,678.
[83] U. S. Patent 1,330,882 (Aug. 28, 1918).
[84] U. S. Patent 1,352,502 (Dec. 7, 1917).
[85] U. S. Patent 1,587,568 (June 8, 1926).
[86] U. S. Patent 1,727,109 (Sept. 3 1929).
[87] U. S. Patent 1,610,458 (Dec. 14, 1926).
[88] U. S. Patent 1,469,039 (Sept. 25, 1923).
[89] U. S. Patent 2,031,368.

to the hot journal surface leaving a very viscous film of lubricant. Terry and Halloran [90] have proposed the distillation of asphalt under a sufficiently high vacuum to prevent decomposition, then about one-half of the residue is destructively distilled to form a brittle non-sticky pitch. Lubricating oil is then extracted from the pitch with a volatile petroleum solvent. Various means of producing heavy asphaltic material have been proposed utilizing air-blowing processes for residues maintained at high temperatures in a still, or consisting of steam distillation for the production of the so-called steam refined asphalts.

In 1906,[91] a process was developed for preparing oxidized petroleum lubricants which consisted of distilling, simultaneously, dehydrogenating and polymerizing hydrocarbons by heating them to a suitable temperature and then blowing cold air through the mass, the heating being discontinued, and the distillation completed by means of the heat generated by the exothermic reaction. The air may be enriched with oxygen, etc., according to the nature of the substance under treatment. The temperature of the reaction may also be controlled by cooling the liquid. The process is applicable to naphthas, naphtha residues, coal tars, wood tars, schist tars, schist oils, etc., and may be used for the production of condensed hydrocarbons suitable for use as lubricants, and of pitch suitable for the manufacture of agglomerates and briquettes. Cyclic hydrocarbons, such as benzene, naphthenes, naphthalene, etc., are unaffected by this reaction and may be purified and distilled by admixture with hydrocarbons capable of modification by this process. Naturally-occurring mixtures of cyclic and open chain compounds may be separated in this way; for example, hexahydrides occurring in petroleum may be distilled off, the residue being a pitch.

Various catalysts have been proposed for assisting the air oxidation or polymerization of petroleum residues to render them suitable as ingredients of lubricants. For instance, Harger [92] has developed a method for making greases in which lubricating oils, together with a catalyst consisting of a manganese, lead or mercury compound, are maintained at a temperature below the cracking point (50 to 300° C.) and blowing with air or oxygen until 0.2 to 1.0 per cent of organic acid has been produced. The product thus obtained may be blended with other hydrocarbon lubricating oils, vegetable, or animal oils, or fats, alkali, soap, or graphite.

In other cases Harger considers catalysts unnecessary and the air or oxygen may be at or above or below atmospheric pressure.

Buraczwski [93] has prepared lubricants by polymerizing mixtures of benzene, ligroin, petroleum lubricating oils, turpentine, etc., by treating with formaldehyde and sulfuric acid. Plauson's [94] solidified oils were made from a crude bitumen, ozokerite, beeswax, montan wax, carnauba wax, or Japan wax treated with an aldehyde, such as a formaldehyde or furfural, in presence of an alkali, with or without addition of ketone, such as acetone

[90] U. S. Patent 1,721,719.
[91] British Patent 28,689 (April 19, 1906).
[92] British Patent 167,789 (Feb. 18, 1920).
[93] British Patent 6,391 (Mar. 15, 1912).
[94] British Patent 186,950 (April 13, 1921).

or methyl ethyl ketone, or a divalent or trivalent alcohol such as glycerol or glycol. The product, after removal of the alkali by washing, may be added to mineral, tar, animal or vegetable oil, yielding viscous or paste-like products which may be used as lubricants. Instead of aldehydes, their polymerization products or substances, such as hexamethylenetetramine, capable of liberating aldehyde may be used. Burch [95] has prepared a lubricant by mixing an asphaltic oil and a paraffine, the latter having the characteristics of dodecane, then heating to 400 to 450° F. and blowing air through the mixture. In addition to air blowing, mechanical agitation may be carried out and thick plastics, or finely divided solids, added. Blowing of mixtures of mineral oil and nitrobenzene at high temperatures in the presence of alkali has been investigated by Boleg.[96] According to Duyck [97] nitrobenzene may be added to lubricants having a blue outer tone to improve their color.

Crane and Moir [98] have prepared greases which are the residues of fire and steam distillation of petroleum. Cabot [99] has suggested mixtures of lubricating oil, having at least 14 carbon atoms, with a polymer and a light solvent to reduce the viscosity as required. From 0.2 to 8 per cent of petrolene (asphaltenes or other material isolated from petroleum residues) has been suggested by Matsunawa [100] as a means of increasing the viscosity of mineral oils and as a base for greases. Dengler [101] has made a study of the optimum temperature conditions for distilling and oxidizing petroleum in order to obtain a material of improved lubricating value. The process consists of reducing a paraffine base crude petroleum by distillation, without substantial decomposition, until the residuum has a flash test within the range of 375° to 475° F., then intimately contacting the residual oil with a current of air at temperatures between 400° to 515° F. In an example of Dengler's process, 400 barrels of residue are charged in a still and oxidized for 50 to 60 hours at 450° F., the air rate being 30,000 cubic feet per hour. The finished product has an A.S.T.M. Penetration of 65 to 75 at 77° F., and a melting point of 280 to 300° F. A lubricant intended particularly for application to the curved rail sections of street railways has been developed by Gray.[102] For this purpose he selected cylinder stock residuum, having a viscosity of 200 S.U.V. at 210° F., and blended it with 40 per cent of a naphthene base residual having a viscosity of about 550 S.F. at 122° F.

To increase the viscosity index (improve the temperature-viscosity characteristics) and to reduce the pour points of waxy oils, Davis [103] has developed a synthetic material formed by the condensation of a waxy hydrocarbon material with an aromatic hydrocarbon. Up to 10 per cent of this material may be used. It is prepared by first chlorinating paraffinic

[95] U. S. Patent 1,238,101 (1916).
[96] U. S. Patent 850,898 (Mar. 8, 1904).
[97] British Patent 2,046 (1870).
[98] British Patent 2,765 (July 10, 1878).
[99] U. S. Patent 1,935,588 (Nov. 14, 1933).
[100] U. S. Patent 1,893,094 (Jan. 3, 1933).
[101] U. S. Patent 1,781,444 (Nov. 11, 1930).
[102] U. S. Patent 1,716,310 (June 4, 1929).
[103] U. S. Patent 1,815,022 (July 14, 1931).

materials such as paraffine wax, petrolatum or other aliphatic hydrocarbons, either pure or in a mixed state, by means of passing chlorine through it while at a temperature between about 140 and 300° F. for a prolonged period, after which it is found that the paraffine contains 10 to 12 per cent of chlorine. Aromatic hydrocarbon material such as naphthalene or anthracene is then mixed with an equal weight of aluminum chloride and the chlorinated oil is slowly added while maintaining a low condensation temperature of about 140 to 160° F. The proportion of the chlorinated product to the aromatic material may vary considerably, about .10 to .5 parts of the latter to 1.0 of the former. The reaction is carried out at room temperature for about 24 hours and it is then desirable to increase the temperature slowly to about 200° F., to complete the reaction and vent the hydrochloric acid vapors which are formed.[104]

GEAR AND PINION GREASES

The base of these products is usually the residuum obtained from the stills in which asphalt and mixed base crude oils are being distilled. The consistency of the products may vary depending upon the extent to which the distillation has been carried. The usual products appearing on the market are so stiff that they must be melted before being applied. Satisfactory greases should be highly adhesive and should stick to exposed gears under adverse conditions of dust and temperature. The thinner-bodied pinion greases are frequently employed as wire cable coatings. The use of a small quantity of pine tar is frequently encountered in these products, but it is said that the use of large quantities of pine tar impairs the adhesive properties of the product.

Gear shield or pinion grease finds extended use in steel mills or other places where the gears are exposed to great heat. This requires that the grease after being applied while hot to the gear teeth should chill and form a cushion which does not drip off at relatively high temperatures. Such lubricants are sometimes known as gear glazes.

Pinion Grease (Hard)

Formula	Pounds	Per cent by weight
Asphalt base fuel oil, 16° Bé.	1200	60.00
Lump rosin, grade F	790	39.50
Pine tar	10	.50

These materials are melted together in a steam jacketed vat to a temperature of about 200° F. After cooling to about 150° F. the mixed product is drawn into containers.

This product is an adhesive grease for the lubrication of enclosed or open gears, as on winches, logging locomotives, and similar equipment, also rotary gear-driven kilns, hoists and crushers. It has been found satisfactory for the lubrication of the exposed master gears and pinions on tractors of the crawler type.

[104] U. S. Patent 1,632,027 (June 14, 1927).

Pinion Grease (Soft)

Formula	Pounds	Per cent by weight
Asphalt base fuel oil, 16° Bé.	1400	70.35
Lump rosin, grade F	580	29.14
Pine tar	10	.51

This product flows slowly at normal operating temperatures and has been used for the satisfactory lubrication of enclosed or open gears, as on winches, logging locomotives, and similar equipment.

Gear Grease, Recommendation of Bureau of Mines Committee on Standardization of Petroleum Specifications (December 29, 1920)

This specification covers the grade of petroleum oil used by the United States Government and its agencies for the lubrication and protection of chains, wire ropes and gears of cranes, dredges, steam shovels and all other heavy equipment, for the lubrication and protection of the gears and ropes of balloon hoists, and for swabbing the wires and cables of aircraft.

The oil shall be a petroleum product only, free from vegetable or animal oils or products derived from them. It shall be entirely free from fillers, such as talc, resin, and all materials of every nature not related to the original product.

PROPERTIES AND TESTS:

Viscosity.—The viscosity at 210° F. shall be within the following limits: 900 to 1,100 seconds.

Protection.—When applied to a plate of polished steel the lubricant shall protect the steel for a period of 30 days when immersed in a 10 per cent salt solution.

All tests shall be made according to the methods for testing lubricants adopted by the Committee on Standardization of Petroleum Specifications.

Gear Shield Lubricant No. 1

Formula	Per cent by weight
Pine tar	5.0
Light black oil	40.0
Petroleum pitch, melting point 240° F.	55.0

Gear Shield Lubricant No. 2

Formula	Per cent by weight
Pine tar	5.0
Black oil	25.0
Petroleum pitch, melting point 240° F.	70.0

Gear Grease

Formula	Per cent by weight
Black oil, 200 vis. at 100° F.	50.0
Petroleum pitch, melting point 240° F.	50.0

Special Gear Grease

Formula	Per cent by weight
Raw degras	65.0
Black oil, 200 vis. at 100° F.	10.0
Powdered graphite	25.0

Curve Grease

Formula	Per cent by weight
Asphalt base fuel oil, 16° Bé.	83.76
Lump rosin, grade F	16.24

Analyses of Commercial Gear and Pinion Greases

The analyses of a few commercial gear and pinion greases are given below.

No. 1	Per cent by weight
Water	Trace
Heavy asphaltic base oil	96.1
Saponifiable matter	1.51
Ash	.25
Filler	None
Rosin	None
Viscosity at 210° F.	955
Flash	590° F.
Burn	580° F.

No. 2	Per cent by weight
Water	Trace
Saponifiable matter	1.71
Rosin	None
Filler	None
Ash	.21
Asphalt	86.4
Viscosity at 210° F.	886
Flash	270° F.
Burn	405° F.

No. 3	Per cent by weight
Water	None
Ash	Trace
Filler	None
Saponifiable matter	1.88
Rosin	None
Flash	460° F.
Burn	530° F.
Viscosity at 210° F.	908

No. 4	Per cent by weight
Water	.13
Fe_2O_3	.16
Combined fat	.83
Heavy black oil	98.88
Viscosity at 210° F.	186
Cold test	28° F.
Melting point, approximately	162° F.

No. 5	Per cent by weight
Water	2.6
Black oil	61.1
Asphalt	21.2
Rosin (or rosin oil)	6.9
Ash (lime)	8.2
Melting point	208° F.

Rock Bit Drill Oil

Formula	
100 to 120 Steam refined asphalt	186 gallons
600 S.U.V. at 100° F. naphthene lubricating distillate	114 gallons

This blend should have a Saybolt Viscosity of 700 to 730 at 210° F.

Standardized Gear Shield Greases (Outside Gear Greases)

	Formula		
	Per cent by weight 10 to 20 Penetration Air Blown Asphalt	Per cent by weight 160 S.U.V. at 210° F. Naphthene S.R. Stock	S.U.V. at 210° F. of Finished Grease
Grade A (light)	24.03	75.97	300–400
Grade B (medium)	40.13	59.87	900–1000
Grade C (heavy)	46.00	54.00	1550–1750
Grade D (extra heavy)	50.71	49.29	3500–4500
Special	50.00*	50.00	850–1150

*(S.R. unblown 40–50 penetration asphalt)

Grade A is suitable as a Railway Center Plate Grease.

Grade C may also be sold as Electric Car Gear Grease, or Railroad Gear Grease.

Heitmann [105] has investigated the possibilities for making consistent greases by injecting saturated or superheated steam, cold or heated air, or other gas, into a body of lubricating oil and adding the desired proportion of water, with continued injection of steam and maintaining the injection until a stable emulsion of 50 pounds of polymerized mineral oil or saponifiable oil with 50 pounds of water is produced, using steam at 1 to 2 atmospheres. Solid products may be obtained by first treating a mixture of mineral oil with agents affecting the viscosity; for example, higher alcohols, montan wax, ceresin, or "Landine." The injection is maintained while the aqueous liquid is added from above in a finely-divided state. Subsequently, he proposed the manufacture of similar greases by treating mineral oils with an electrical discharge and then emulsifying with water.[106]

In 1927, Heitmann [107] reported that the best emulsifying agents for mineral oils used in making this type of grease were polymerized oils such as Voltol, or wool fat alcohols, montan wax, etc. His procedure for preparing the Voltol oil was: Hydrocarbon oil is introduced into a vessel in which are a plurality of rotatable electrodes separated by a dielectric. The vessel is filled with hydrogen at a sub-atmospheric pressure. The electrodes are subjected to a potential ranging between 4300 and 4600 volts at 500 cycles per second, and a current intensity of 19 to 23 amperes. This current varies with the pressure in the apparatus. The oil in the chamber is caused to trickle over the electrode surfaces by the rotation thereof. The oil is kept at a temperature of between 60 and 80° C. The electrical discharges arising from the individual electrode plates convert the rotating electrodes which are covered with oil into a flame roll of rose violet color. The oil falling onto and trickling over the electrodes is subjected to the electrical field previously mentioned. The finished emulsified solid greases may have formulae as follows:

50 parts by weight electrically treated oil; 50 parts water.
42 parts by weight light mineral oil; 6 parts wax; 2 parts electrically treated oil; 50 parts water.
42 parts by weight tar oils; 6 parts paraffine wax; 2 parts electrically treated oil; 50 parts water.

Heitmann [108] has also patented the process in which gas or air is passed under pressure into a body of lubricating oil in order to break it up and form finely divided particles; water is then added to produce an emulsion of the desired composition and the injection of gas continued until a stabilized emulsion is obtained. For bearings operating at high temperatures Heitmann [109] has proposed a lubricant of the foregoing type utilizing a highly viscous bitumen as the dispersion agent.

[105] British Patent 250,562 (April 11, 1925).
[106] U. S. Patent 1,593,762 (July 27, 1926).
[107] U. S. Patent 1,632,027 (June 14, 1927).
[108] U. S. Patent 1,724,653.
[109] U. S. Patent 1,984,214 (Dec. 11, 1934).

LUBRICANTS CONTAINING COAL TAR DERIVATIVES

Under this heading various lubricants containing coal tar, coal tar derivatives, phenol, cresylic acid, naphthalene, volatilized coal tar products, anthracene, and coal tar dyes and odorants will be given brief consideration. In 1897, Thompson [110] prepared an axle grease consisting of three parts of coal tar and one part of fish oil. Tar, naphtha, or similar materials were simultaneously oxidized and distilled by first heating such a mixture to a suitable temperature and then blowing it with dry, hot air, free from carbonic acid and combined or not with oxygen. The distillation was completed by means of the heat generated by the exothermic reaction, the temperature of the mixture reaching approximately 350° F. Wallbaum [111] has manufactured emulsions of solid hydrocarbons such as asphalt, coal tar pitch, paraffine and the like while melting with a saponifiable substance, heating to temperatures higher than 100° C. and pouring the hot mixture into a solution of water, naphthenic acids, and ammonia, while agitating. Coal tar oils may be extracted from coal by digestion with an oil at 300 to 350° C. The solvent material may be distilled with high pressure steam to yield a viscous product suitable for use in lubricants.[112] The oils obtained by this distillation [113] may be mixed with superheated steam and passed through a tube containing such catalysts as, carbon, silicic acid, iron, aluminum, nickel, copper, zinc, tin, and particularly magnesium compounds.[114] Melamid [115] has prepared lubricants from coal tar products by treating them with anhydrous aromatic sulfonic acids in the presence, or not, of methyl, ethyl, or other alcohols. Ethers and esters are produced by the reaction of the phenolic, acidic, and alcoholic groups due to the condensing action of the sulfonic acids.

Lubricants have been prepared by condensing and polymerizing organic bases with phenols, coal tar oils, or distillates from coal tar pitch, by Klever.[116] Rial [117] has assigned to the Union Oil Company his patent for preparing castor oil-mineral oil blends which calls for treatment with carbocyclic (cresylic, anthracenic and naphthenic oils) compounds. Jeyes [118] grease had the following formula:

Tallow	200 pounds
Naphthalene	75 "
Mineral oil	70 gallons

A rust preventing compound has been developed by Benton.[119] He utilizes naphthalene dispersed in white oil. Southcombe and Wells [120] have hydro-

[110] British Patent 28,713 (Dec. 4, 1897).
[111] British Patent 16,182 (Dec. 19, 1904).
[112] U. S. Patent 1,014,103 (Dec. 3, 1910).
[113] British Patent 131,588 (Aug. 9, 1918).
[114] British Patent 156,140 (Dec. 12, 1919).
[115] British Patent 146,898 (Mar. 11, 1919).
[116] U. S. Patent 1,350,814.
[117] U. S. Patent 1,587,859 (June 8, 1926).
[118] British Patent 1,857 (Feb. 2, 1889).
[119] U. S. Patent 1,943,808.
[120] British Patent 320,899 (July 19, 1928).

genated carbonaceous materials such as coal, wood, and vegetable matter, oils, etc., by adding organic acids, preferably those containing more than five carbon atoms, for example oleic acid. Carpmael [121] has studied the action of silent electric discharge on alkylated naphthalenes. For example, a mixture of ethyl and propyl naphthalenes was permitted to flow slowly, at atmospheric or reduced pressures, through a quartz ozonizer for 20 hours with silent discharge produced by an alternating current of 3,500 cycles per second, at 9000 volts. Methyl naphthalene may also be treated in this manner to produce lubricants. Products obtained by destructively hydrogenating lignite may be treated in the liquid phase, by the electric current at 5000 volts and 2000 cycles, to produce thick lubricants according to a process developed by Johnson.[122]

Fats, oils, tars, and resinous oils may be solidified, according to Blass,[123] by incorporating anthracene with them. Coal tar derivatives are frequently used with success to color lubricating oils and greases. Lubberge,[124] for instance, has suggested coloring textile lubricants with methyl violet-oleate in order to detect their presence on fabrics. Vellner [125] has suggested coloring lubricating greases for identification purposes, using 2 ounces of scent and the necessary dye for each 100 pounds of lubricant. Coal tar dyes specified are yellow, green, red, blue, and orange aniline dyes in any combination with scents, such as oil of cloves, wintergreen, peppermint, terpineol, or lavender, and ionone or violets. Primuline [126] bases have been recommended for producing fluorescence in lubricating oils and greases. For identification purposes Ferguson [127] has proposed the addition of such compounds as quinine, vesculine, phenolphthalein, cresol-phthalein, brom thymol blue, etc.

LUBRICANTS CONTAINING NITROGEN COMPOUNDS

In view of the fact that the organic acid salts of ammonia are not stable, and when dispersed in lubricating oils do not produce high yields of grease, they are not particularly suitable as grease bases. Various amines and amides have, however, been used with success in producing low melting point, smooth textured greases. Ammonia is often utilized to control the pH value of emulsions and form ammonia soaps therein. The alkaline effects of free ammonia may be advantageous in certain types of lubricants. For instance, Wild [128] has developed an oily lubricant for neutralizing the acid residues left in gun barrels after firing smokeless powder and to prevent rusting. It consists of mineral oil, alcohol, and strong ammonia. Hutch-

[121] British Patent 325,832 (Nov. 28, 1928).
[122] British Patent 322,935 (Jan. 21, 1929). See also British Patent 323,580.
[123] U. S. Patent 877,289 (Feb. 27, 1906).
[124] British Patent 5,786 (April 29, 1911).
[125] British Patent 359,590 (July 29, 1930).
[126] U. S. Patent 1,962,564.
[127] British Patent 361,310 (June 18, 1930).
[128] U. S. Patent 768,835 (Sept. 22, 1933).

inson [129] has proposed a lubricant consisting of wool fat, 1 part; mineral oil, 6 parts; and ammonia water 20 parts.

Ammonium oleate has been suggested by Duckham [130] as an ingredient of an emulsified lubricant for internal combustion engine cylinders. It consists of heavy mineral oil, an animal fat such as lanoline, emulsified by means of ammonia, or an ammonium salt such as ammonium oleate. Ammonium oleate has also been suggested by Dahlberg [131] as an emulgator for cutting oils. Mond [132] has studied lubricating greases in which ammonium stearate is the base. In an example, 45 parts by weight of lubricating oil containing 4.5 parts of glycerol di-stearin is blended with 50 parts of water containing one fourth part of ammonium stearate.

Parr [133] has utilized the following materials in making a lubricant: Irish moss, carbolic acid, coconut oil, olive oil, and ammonia. Sorenson [134] found that dispersions of ammonia soaps in lubricating oil were particularly useful in preventing chattering of clutch bands in planetary type transmissions. Auer [135] has studied dispersions in mineral oil made by heating fat, fatty oil, or resins in a vacuum with amines. In an example, mineral oil is heated with linseed oil, which has been solidified with benzidine or lithium sulfite, to form a viscous oil or with aqueous solutions of dianisidine hydrochloride and the ammonium soap of linseed oil fatty acids are mixed together and form a water insoluble condensation product which is washed, dried, and dissolved in mineral oil. The products are viscous oils, or their gels, and may be used as lubricating oils or greases. Mineral oil and water emulsions may be produced according to Klever [136] by incorporating 25 per cent of oleic acid and adding ammonia. Kosters [137] has manufactured lubricants by boiling an amide of a higher fatty acid, such as the amide of stearic acid, or an acidyl derivative of an aromatic base (the anilide of stearic acid) with water and adding to this mixture oil or fat, which is to be emulsified, together with an alkali salt of a fatty acid. A thick emulsion suitable for lubrication of textile fibers may be prepared by following the formula given below:

	Grams
Stearamid	400
Sodium stearate	240
Water	4000
Heavy cylinder oil	2000
Water	4000

This product is a viscous, yellow gel containing about 20 per cent of oil. For the 400 grams of stearamid, 300 grams of stearanilid may be substituted. Mills [138] has proposed emulsions consisting of plant slime, or a com-

[129] U. S. Patent 907,055.
[130] British Patent 8,387 (April 15, 1908).
[131] U. S. Patent 1,881,173 (Oct. 4, 1932).
[132] British Patent 329,266 (Nov. 5, 1928).
[133] British Patent 3,889 (Sept. 25, 1880).
[134] U. S. Patent 1,414,313 (Mar. 25, 1922). See also U. S. Patent 1,414,227.
[135] British Patent 337,751 (April 24, 1929).
[136] British Patent 17,118 (Aug. 1, 1896).
[137] British Patent 4,688 (1906). See also U. S. Patent 867,141 (1907).
[138] British Patent 14,119 (June 16, 1909).

pound containing an aldehyde. Ammonium compounds, or an amide swelled in water and treated with formaldehyde, may be added and the finished product used as a cylinder lubricant, cutting oil, or textile lubricant. Johnson [139] has suggested the use of sulfonated amides of aliphatic acids as ingredients of textile lubricants. He [140] has also proposed sulfonated Tall oil (resinous oil) or sulfonated amides or anilids for the same purpose.

Nill [141] has developed a process for making anilids, for example, stearanilid by distilling aniline oil at a temperature of approximately 185° C. and passing the vapors through stearic acid which has been heated to a temperature of about 235° C. He [142] has also studied the grease making characteristics of anilids and has patented a composition which is substantially free from water and comprises a mineral oil having dissolved therein a neutral anilid of a fatty acid to increase the consistency and raise the melting point of the composition.

Sullivan [143] has assigned to the Standard Oil Company of Indiana his patent for a lubricant for a grease which yields no ash on analysis. It consists of the fatty acid soaps of a non-cyclic organic base, having two amino nitrogen atoms bonded to a carbon atom, capable of forming carbonates stable at 210° F. dispersed in lubricating oil. Nill [144] has also prepared grease substantially free from water which is made with mineral oil mixed with a toluide of a fatty acid to stiffen the mixture and increase the melting point. Sullivan and Gill [145] have proposed the use of an amide, derived from fatty acids and derivatives of aniline, having alkyl groups substituted in the ring as agents for decreasing the pour-point of waxy lubricating oils. Nill,[146] in order to secure still higher melting points than was possible with amides and anilids, has proposed the addition of metallic soaps together with amides of fatty acids to mineral oils.

LUBRICANTS CONTAINING GLYCERIN

Glycerin, a trihydric alcohol, may be found in almost any soap base grease made with whole fats rather than fatty acids, as it is one of the products of fat saponification. Glycerin is also known as glycerol, $(C_3H_5(HO)_3)$, and it or its derivatives are sometimes added to non-soap lubricants to reduce the freezing point, increase fluidity at low temperatures, or for other special purposes. Johnson [147] has made a study of non-freezing lubricants made by mixing or emulsifying oils or fats with mineral or vegetable oils and then adding a glycol or a hydroxy-containing ether of a glycol. Soap dispersion agents may be used such as the salts of high molecular weight sulfonates or an alkylolamine. In an example, Johnson has reported the use of a mixture

[139] British Patent 341,053 (Aug. 7, 1929).
[140] British Patent 340,272 (July 19, 1929).
[141] U. S. Patent 1,659,150.
[142] U. S. Patent 1,659,149 (Feb. 14, 1928).
[143] U. S. Patent 1,830,970 (Nov. 10, 1931).
[144] U. S. Patent 1,853,571 (April 12, 1932).
[145] U. S. Patent 1,870,074 (Aug. 2, 1932).
[146] U. S. Patent 1,937,462 (Nov. 28, 1933).
[147] British Patent 340,294 (July 27, 1929).

of spindle oil, tallow, rosin, or the acid oxidation products of paraffine wax, mixed with ethylene glycol, borax, benzyl alcohol, and an aqueous solution of potash to form a homogeneous mass which is suitable for lubrication of locomotive axles, electrical contacts and railway switches, or used in hydraulic presses or brakes, or as a rust preventative coating. The ethylene glycol may be replaced by a corresponding mono ethyl, or cresyl ether, or by polyglycol, and glycerin may be used in conjunction with these compounds. Noack [148] has carried out extensive experiments to develop explosion-proof lubricants for use in compressors, valves, etc., containing a mixture of higher polymers of aliphatic alcohols having three or more hydroxyl groups free from di-, and tri-polymers. The higher polymers are made by heating the glycerol, or similar alcohols, to 250 to 275° C. in the presence of condensing agents such as heavy metal salts; for example, mercuric chloride or sulfate, or cuprous or ferric chloride; removing the water formed by treating the reaction mixture with a stream of inert gas, and further heating to form a highly viscous product. Small amounts of di-, and tri-glycerins are removed by distillation in vacuum. Glycerin may be replaced by chlorinated glycerin or by a mixture of glycerin and higher alcohols containing more than three hydroxy groups. Inert gases specified by Noack are carbon dioxide, nitrogen, or rare gases. To form a lubricant the highly polymerized glycerins are preferably mixed with water and a hygroscopic substance such as magnesium chloride or a lactate. If desired, the lubricant may be brought to the consistency of about No. 3 Cup Grease, by incorporating tragacanth, talcum, etc.[149]

Many fluid lubricants containing glycerin to prevent freezing [150] have been patented; for instance, propylene glycol,[151] mixtures of carbon disulfide with glycerin,[152] and, in particular, Massa's [153] refrigerator oil consisting of a suitable mineral oil blended with glycols. Jackson [154] has proposed a lubricating grease consisting of glycerin and gelatin. For dressing and increasing the pliability of fabrics and textile fibers Claessen [155] has proposed the use of di-, and poly-glycerides.

A fluid for transferring energy in hydraulic presses, hydraulic brakes or shock absorbers has been developed by Johnson.[156] It consists of animal, vegetable, or mineral oils and ethers having melting points below 0° C. and boiling points above 50° C. Ethers specified are propyl, butyl, heptyl, and alkyl and aryl ethers of glycols, or polyglycols, or higher polyhydric alcohols. Agents retarding oxidation may be added. In examples, he gives mixtures of 1 pound of castor oil with 2 pounds of either mono butyl, or mono ethyl, ether of ethylene glycol, or polyglycol; and 1 pound of castor oil with 1 pound of di-butyl ether. The castor oil may have been rendered miscible

[148] British Patent 398,474 (Feb. 6, 1932).
[149] U. S. Patent 2,015,867.
[150] British Patent 1,544 (April 11, 1876).
[151] U. S. Patent 1,498,021.
[152] British Patent 2,863 (July 14, 1879).
[153] U. S. Patent 1,696,642 (Dec. 25, 1928).
[154] U. S. Patent 280,625 (1883).
[155] British Patent 9,579 (May 2, 1908).
[156] British Patent 348,642 (Feb. 10, 1930).

with other vegetable oils, or with mineral oils, and be used mixed therewith. For example, 30 liters of castor oil, made miscible with mineral oil, 30 liters of ethylene glycol mono cresyl, and 10 liters of mineral oil may be used. The oils may be rendered neutral by treating with ethylene oxide or its homologues, or by treatment with alkali hydroxides, carbonates, phosphates, etc., and may be dissolved in alcohol or other solvent. Commercial ethers are preferably purified by alkalies and distillation. Vegetable or animal oils are preferably preserved against oxidation by addition of urea, aldol-a-naphthylamine, or the like agent.

For similar purposes as above Johnson [157] has carried out extensive investigations of various mixtures of glycols with sulfonation products of organic compounds containing at least six carbon atoms. The mixture may also contain water, ethers of glycols, monohydric alcohols, and alcohols containing more than two OH groups.

These mixtures do not freeze, are less viscous than oils at low temperatures, do not cause rust, and do not attack rubber joints or packings. Glycols include the hydroxyalkyl ethers, or polyglycols, and the ethers both of glycols and of polyglycols. Examples of formulae are:

	Parts by Weight
Ethylene glycol	63
Water	30
Polyglycol ether (plus 10 to 20 per cent of neutralized sulfuric ester of castor oil)	7
Ethylene glycol	52–53
Polyethylene glycol ether	6
Water	25
Neutral sulfuric ester of ricinoleic acid	16–17

	Parts by Volume
Ethylene glycol	28
Butanol	12
50 Per cent aqueous solution of a neutral sulfuric ester of ricinoleic acid	20

Teeple [158] has assigned to the Crane Packing Company his patent covering a lubricant for gasoline pump bearings consisting of 25 to 80 per cent of blown castor oil and 10 to 65 per cent of glycerin. Nonamaker and Welsbach [159] have proposed a lubricant which has the chemical action of a reducing agent, and consists of glycerin, a glycol and sodium thiosulfate.

LUBRICANTS CONTAINING CARBOHYDRATES

While the carbohydrates (monosaccharides and polysaccharides) are subject to charring at elevated temperatures they have, in some cases, been utilized for the lubrication of bearings and gears where high temperatures are not encountered. These products are normally insoluble in mineral oil and in one sense may be considered as solid lubricants. Certain derivatives

[157] British Patent 333,200 (Apr. 26, 1929). See also U. S. Patent 1,984,421.
[158] U. S. Patent 1,854,237 (April 19, 1932).
[159] U. S. Patent 1,822,449 (Sept. 8, 1931).

of cellulose have, however, been colloidally dispersed in lubricating oils to form satisfactory gel type greases. Starch, $(C_6H_{10}O_5)_x$, may be found in some lubricants; for instance, Hagedorn [160] has developed lubricants consisting of vegetable, animal, or mineral oils together with derivatives of a polysaccharide. Curtis,[161] in 1868, proposed mixtures of petroleum oil and rye flour. Another early lubricant [162] intended as a substitute for tallow was made with castor oil, animal fat, and wheat flour. Coati [163] claims a non-inflammable lubricant made by emulsifying starch, water and oil. Pitlik [164] has made a boiler lubricant consisting of mineral oil, dextrin and water.

Various extracts of carbohydrate material have been suggested as agents for improving lubricants or producing emulsions. As examples, Hodge [165] has made a lubricating oil compound containing one-fourth ounce of slippery elm bark per gallon of oil. Goss [166] has utilized the aqueous extract of slippery elm bark for preparing water and oil emulsions.

Baldwin [167] has formulated a cutting oil as follows:

	Parts by Weight
Wormwood salts	80
Ammonium hydrochlorate	10
Phenol	1
Water	50

Morgan and Brown [168] proposed a soap bark lubricant containing mineral lubricating oils and "quillia saponaria" in suitable proportions to form a grease. An adhesive lubricant was developed by Pearson and Taunton [169] containing the liquid extract of the locust bean. Whole crushed cotton seeds mixed with fiber waste and mineral oil has been suggested as a packing and lubricating composition.[170]

Cellulose, in a more or less refined condition, or compounds of cellulose, have been frequently suggested as bases for lubricating compositions. In 1890, Nevill [171] developed a lubricant consisting of tar, pitch, and paper pulp. In 1928, cellulose naphthenate [172] was suggested as a suitable agent for addition to transformer oils, about 9 per cent of the naphthenate being recommended. The object of this addition was to increase viscosity, and other esters, or mixed esters, or ether-esters, of polymerized carbohydrates (starch or cellulose) were also suggested. Seven per cent of triethyl cellulose dissolved in vegetable castor oil was proposed as a lubricant.

[160] U. S. Patent 1,882,816 (Oct. 18, 1932).
[161] U. S. Patent 78,189 (1868).
[162] U. S. Patent 209,203.
[163] British Patent 20,387 (Sept. 7, 1912).
[164] British Patent 10,022 (May 19, 1893).
[165] U. S. Patent 1,230,579 (Nov. 29, 1916).
[166] U. S. Patent 1,708,058 (April 9, 1929).
[167] British Patent 2,327 (Sept. 4, 1871).
[168] British Patent 2,922 (Aug. 19, 1875).
[169] British Patent 20,368 (Oct. 29, 1893).
[170] U. S. Patent 601,180.
[171] British Patent 13,398 (Aug. 26, 1890).
[172] British Patent 337,639 (Nov. 29, 1928).

Hickman [173] has assigned to Kodak Ltd. a patent for a similar lubricant which is particularly suitable for automobile transmissions and rear axles, consisting of substantially a cellulose ether or ester, vegetable or fish oils, and a heavy mineral oil. Cellulose derivatives specified are triethyl cellulose, and the acetate, stearate, acetate-stearate, propionate, butyrate, laurate, naphthenate, and nitrate of cellulose. The mineral oil preferably forms 25 to 50 per cent of the finished lubricant. In an example, 6 pounds of triethyl cellulose is soaked in 64 pounds of castor oil for 15 to 25 hours, dissolved by heating to 210° C., cooled, and masticated, and then masticated with 30 pounds of crank-case oil; the grease does not bleed. Similarly sperm oil, or a mixture of sperm and castor oils, may be used. Malm and Fordyce [174] have also assigned to Kodak Ltd. their patent for greases containing cellulose mixed esters, such as cellulose propionate-stearate and cellulose butyrate-stearate. These esters are soluble in mineral and vegetable oils, and, in particular, cellulose propionate-stearate is soluble in hot mineral oil to give on cooling a product resembling heavy greases. Staud and Fuess [175] have developed for the Eastman Kodak Co. lubricants containing acetone-soluble cellulose acetate, diethyl phthalate and ethyl lactate.

Craik [176] has also investigated the preparation of thick lubricating greases made by dissolving cellulose esters of saturated fatty acids, having 12 to 18 carbon atoms in the molecule, in mineral lubricating oils. Such esters as cellulose stearate, cellulose palmitate, or cellulose dilaurate are suggested. Craik prefered those esters containing two ester groups per molecule of cellulose (taken as $C_6H_{10}O_5$). Lubricants containing nitrocellulose and di-butyl-phthalate have been developed by Custer.[177] It will be recalled that Tall oil, a sulfite cellulose material derived from paper mill waste liquor has been suggested as an ingredient of emulsified cutting oils.[178]

Raw carbohydrates have also been utilized in lubricating greases, but are not favored as quality ingredients. In 1876, Benedict [179] suggested mixing ground cork with various gear lubricants. Marshall [180] proposed mixtures of animal, vegetable, or mineral oil, combined with the gelatinous and fibrous material from the cactus plant. Even ground corn stalks have been suggested.[181] Rush or reed pith has been incorporated with mineral oil to form a lubricant.[182] Wood sawdust, peat, or bark mixed with mineral oil, was suggested by Haas [183] in 1902. Kimmel and McMahon [184] developed a lubricant consisting of 10 parts of heavy lubricating oil with 1 part of "coarse cut wood fibre".

[173] British Patent 398,936 (April 18, 1932). See also U. S. Patent 1,963,901.
[174] British Patent 392,775 (July 27, 1932).
[175] U. S. Patent 1,866,732.
[176] British Patent 416,513 (Mar. 17, 1933).
[177] U. S. Patent 1,976,716 (Oct. 16, 1934).
[178] U. S. Patent 1,495,074.
[179] U. S. Patent 176,582 (Feb. 16, 1876).
[180] U. S. Patent 339,200 (1886).
[181] British Patent 15,758 (1898).
[182] British Patent 18,810 (Aug. 22, 1906).
[183] British Patent 18,783 (Aug. 26, 1902).
[184] U. S. Patent 1,581,804 (April 20, 1926).

USE OF GUMS IN LUBRICATING GREASES

The term gums is generic and applies to a variety of vegetable materials having a gummy or sticky character, most of which may be considered as carbohydrates. True gums are water-soluble and in order to disperse them in mineral oil lubricants special solvents are sometimes necessary. There is some evidence that gums tend to stabilize calcium base greases, and further research in this direction should prove fruitful. Young [185] has developed a lubricant for automobile springs having superior adhesive properties. It is composed of motor oil, crude oil and gum camphor. Gums which offer possibilities for use in lubricating greases are: Arabic, Copal, Dammar, Gamboge, Kauri, Mastic, Myrrh, Olibanum, Sandarach, Thus, Tragacanth, Yacca, and Senegal. Ester gums, which are usually harder than the natural gums, are made by combining acid resins and alcohols, such as abietic acid and glycerin. Their use in lubricating greases has not been fully explored. Such synthetic gums as Bakelite, Formite, Margalite, and the like, offer possibilities but have not been thoroughly studied.

NON-SOAP GREASES CONTAINING ANIMAL MATTER, FREE ANIMAL FAT, OR FATTY DERIVATIVES

The utilization of animal and vegetable fibers in soap base lubricants has already been discussed and in this section brief comments on non-soap base greases will be given. As early as 1855, Wilkenson [186] proposed greases containing wool or cotton fiber. Hicks [187] proposed greases made from tallow, wool, and cotton fiber. Schultz [188] also investigated various combinations of grease and fibrous materials. In 1861, Dumesnil [189] suggested mucilage as an agent for increasing the adhesiveness of lubricants.

Engel [190] has invented a tallow substitute made by melting a consistent fat and then immersing a spongy substance in the fat and cooling. In some cases, as for instance in the lubrication of steel cables, a lubricant which will oxidize and form a tough, leathery film, may be desirable. Hodson [191] has developed lubricants of this kind. Polymerized linseed or perilla oils, linseed oil, tung oil, and the like are suitable.[192]

Mixtures of linseed oil, fish oil, lard oil, and turpentine have been patented by Hart.[193] For lubricating fabric transmission bands, Boileau [194] has recommended mineral oil compounded with 2 to 12 per cent of a fat such as degras, sperm oil or lard oil. For lubricating leather, Stiepel [195] has proposed a mixture of palm oil, sperm oil, lard, and olive oils. Mixtures of

[185] U. S. Patent 1,555,899 (Oct. 6, 1925).
[186] British Patent 2,311 (Oct. 16, 1855).
[187] British Patent 1,893 (Feb. 7, 1887).
[188] British Patent 27,564 (Dec. 16, 1903).
[189] British Patent 1,845 (July 23, 1861).
[190] U. S. Patent 1,012,830 (Aug. 3, 1911).
[191] U. S. Patent 2,028,155.
[192] British Patent 250,947.
[193] British Patent 2,030 (Sept. 8, 1955).
[194] U. S. Patent 1,418,695 (June 6, 1922).
[195] U. S. Patent 1,934,100.

tallow, rape seed oil and rosin for use as an axle lubricant were suggested by Reddan.[196] Draper [197] has proposed lubricants consisting of lard oil, mineral oil, and about 10 per cent of chloroform to prevent freezing at low temperatures. Mackle [198] has heat treated mixtures of 85 per cent of cotton seed oil, and 15 per cent of castor oil to produce lubricants. Thomas and Anquetil [199] have evaporated mixtures of oleic and stearic acids to obtain thickened lubricants. A lubricating grease consisting of tallow, cocoanut oil and water has been patented by Lowther.[200] Hall [201] has proposed mixtures of beef fat and colza oil. Clark's [202] axle grease was made with suet, lard and water. Dougall [203] first treated oils, fats, greases and waxes with an alkali and then sulfurous acid to render them suitable as lubricants.

TEXTILE LUBRICANTS

These products, ordinarily intended for lubricating textile fibers and rendering yarns more pliable in order to facilitate weaving and spinning operations, cannot properly be termed lubricating greases. However, for the sake of completeness, and on account of the fact that some of the ideas involved in their composition may serve as the starting points for the development of novel lubricating grease compositions, the following list of patents covering textile emulsions is presented:

Inventor	Textile Lubricant
Sulfonated Products	
Johnson [204]	Water soluble sulfonic compounds derived from mineral oil with non-mineral oily vehicle.
Howes [205]	Sulfonated castor oil.
Fischer & Reddish [206]	Purification of mahogany sulfonates.
Hoel [207]	Non-frothing stable emulsion of mineral oil and saponified sulfonated sperm oil.
Reddish [208]	White oil and mahogany sulfonates.
Melamid [209]	Phenol alcohols with aromatic sulfochlorides.
Vidal [210]	Sulfonated fatty oils.
Bohme Akt.-Ges.[211]	Sulfonated alcohols.
Jackson [212]	Mahogany sulfonates.
Fet. u. Glz. Fabrik [213]	Sulfonated oleic acid.
Cavaillon [214]	Sulfonated olive oil.
Bohme Akt.-Ges.[215]	Cetyl glucoside and sulfonated castor oil.

[196] U. S. Patent 276,711 (May 1, 1883).
[197] U. S. Patent 228,181 (1879).
[198] U. S. Patent 1,191,328 (Oct. 17, 1914).
[199] British Patent 986 (April 4, 1870).
[200] British Patent 1,175 (April 27, 1865).
[201] British Patent 388, (Feb. 11, 1865)
[202] British Patent 2,541 (Oct. 14, 1864).
[203] British Patent 2,390 (Sept. 19, 1865).
[204] U. S. Patent 1,619,074 (Mar. 1, 1927).
[205] U. S. Patent 1,677,389 (July 17, 1928).
[206] U. S. Patent 1,731,716 (Oct. 15, 1929).
[207] U. S. Patent 1,875,001 (Aug. 30, 1932).
[208] U. S. Patent 1,909,721 (May 16, 1933)
[209] British Patent 143,185 (May 12, 1919).
[210] British Patent 285,473 (Feb. 18, 1927).
[211] British Patent 341,978 (July 19, 1929).
[212] British Patent 349,962 (Feb. 3, 1930).
[213] British Patent 400,587 (April 14, 1931).
[214] British Patent 370,810 (Sept. 7, 1931).
[215] British Patent 393,769 (July 7, 1932).

Inventor	Textile Lubricant
Esters	
Johnson [216]	Di- or triethyleneglycol esters of aliphatic monohydric alcohols of at least 8 carbon atoms.
Bohme [217]	Alkyl esters of sulfonic acids.
Johnson [218]	Derivatives of polyhydric alcohols.
Hydrogenated Compounds	
Zemmerli [219]	Hydrogenated phenols, hydrogenated naphthalenes or hydrogenated terpenes.
British Celanese, Ltd.[220]	Hydrogenated coconut or palm kernel oils.
Nitrogen Compounds	
British Celanese, Ltd.[221]	Amides of carboxylic acids.
Johnson [222]	Amines and ammonium bases having one aliphatic radical.
British Celanese, Ltd.[223]	Amino bases of at least 7 carbon atoms, amino group not directly attached to aryl residue or to carbon atom linked to a second nitrogen atom.
Johnson [224]	Insoluble amines of carboxy or sulfonic amides. containing at least one hydrogen atom connected to a nitrogen atom, but no hydroxy groups, together with polyethylene glycols.
Chamberlain & Speakman [225]	Aiiliphatic alcohol, with ether or amine of more than 8 carbon atoms derived from wool fat.
Halogen Compounds	
Carpmael [226]	Chlorinated paraffine wax.
Carpmael [227]	Chlorinated paraffine wax.
Carpmael [228]	Chlorinated paraffine wax.
Terpenes	
Deutsche Hyd.[229]	Esters of cyclohexanol with aliphatic acids; for example, borneol, fenchol, or terpineol esters of cocoanut, palm kernel, bornyl montanate or stearate.
Basle [230]	Terpineol treated at below 10° C. with chlorsulfonic or fuming sulfuric acid.
Fats, Waxes and Oils	
Carroll [231]	Paraffine wax, beeswax, myrtle wax, stearic acid, spermaceti, and fossil wax.
Hopkinson [232]	Wool fat.
Wallbaum [233]	Animal, vegetable and mineral oils with tar oil, beeswax, and wool fat.
Boyeux [234]	Linseed oil, beeswax, etc.

[216] British Patent 404,931 (June 9, 1932).
[217] British Patent 350,595 (Mar. 20, 1929).
[218] British Patent 375,842 (Mar. 22, 1931).
[219] British Patent 277,649 (Sept. 16, 1926).
[220] British Patent 398,243 (Mar. 11, 1932).
[221] British Patent 355,726 (Mar. 21, 1930).
[222] British Patent 372,325 (Nov. 27, 1930).
[223] British Patent 378,473 (Feb. 11, 1931).
[224] British Patent 380,851 (April 10, 1931).
[225] British Patent 400,681 (May 4, 1932).
[226] British Patent 367,545 (Feb. 6, 1931).
[227] British Patent 372,784 (Feb. 6, 1931).
[228] British Patent 372,785 (Feb. 6, 1931).
[229] British Patent 390,534 (Oct. 7, 1931).
[230] British Patent 398,086 (Jan. 20, 1933)
[231] British Patent 10,314 (May 31, 1892).
[232] British Patent 29,612 (1897).
[233] British Patent 4,116 (Feb. 19, 1906).
[234] British Patent 233 (Jan. 3, 1907).

Inventor	Textile Lubricant
Miscellaneous	
U. S. Pat. 2,026,735 [235]	Linoleic acid.
Singer [236]	Mono-, di-, and tri-acetin with glycerin.
Johnson [237]	Esters of water-insoluble organic acids.
U. S. Pat. 1,938,804 [238]	Cyclohexanol, soap, mineral oil and water.
Langer [239]	Emulsion of mineral oil and water.
Pelly [240]	Polyhydric alcohols and mono-glycerides of oleic acids as emulgators.
I. G. Farben.[241]	Condensation products of higher fatty acids obtained from oxidation of paraffin oil. Ethionic acid heated with stearic acid.
Barton [242]	Nitrobenzene.
U. S. Pat. 2,002,885 [243]	Lecithin.
Sandqvist [244]	Petrolatum plus 5 per cent of phytosterin.
Bollmann[245]	Soya bean lecithin.
Bollman & Rewald [246]	Lecithin.
Groves [247]	Tetrahydronaphthalene, olive oil, mineral oil and wax.
Adams [248]	Emulsion for use in hard water.
Bullock [249]	Method of forming emulsions.
U. S. Pat. 1,791,057 [250]	1 to 2 per cent Hydroquinone to produce non-inflammable wool oil.

[235] U. S. Patent 2,026,735.
[236] British Patent 1,605 (Jan. 22, 1904).
[237] British Patent 380,431 (April 10, 1931).
[238] U. S. Patent 1,938,804.
[239] British Patent 164,303.
[240] British Patent 188,364.
[241] British Patent 366,916 (Oct. 31, 1930).
[242] British Patent 1,487 (June 5, 1871).
[243] U. S. Patent 2,002,885.
[244] British Patent 329,305 (Feb. 14, 1929).
[245] British Patent 348,873 (Mar. 20, 1930).
[246] British Patent 369,990 (Dec. 18, 1930).
[247] British Patent 361,860 (Aug. 18, 1930).
[248] U. S. Patent 2,039,377.
[249] U. S. Patent 1,578,187 (Mar. 23, 1926).
[250] U. S. Patent 1,791,057.

Chapter XIV

Grease Plant Management and Research

CAPITAL RATIO FOR LUBRICATING GREASE INDUSTRY

According to a report [1] issued by the National Association of Lubricating Grease Manufacturers the capital invested in the grease industry and the total sales value of the products produced were:

Capital Invested	$4,148,184.82
Total Sales (yearly)	7,329,378.85

It is evident from these figures that $0.566 has been invested in the grease industry for each $1.00 of sales. This value compares favorably with that of a similar industry, the manufacture of soap, for which the capital ratio for 1904 to 1919 was $0.61, and the value for all chemical engineering industries for this period which was $0.85. It appears likely that the comparatively low capital ratio of the grease industry may be due to the fact that it takes refined basic materials, fats and lubricating oils, and converts them to products finished and ready for sale to the final user. The capital ratio is a convenient means by which each grease plant manager may check his position with the average for this country. In planning for any new improvements of the grease plant it is well to give careful consideration to the capital ratio as it is a useful index of the financial stability of the organization. If the cost of making extensions is estimated to increase the existing capital ratio then considerable doubt may be cast on the wisdom of making such a capital expenditure. Some of the factors which tend to increase the capital ratio to abnormally high values are:

Excessive inventories of fats, lubricating oils, and chemicals.
Inefficient processes or grease making apparatus, poor means of handling products within the plant, or poor production management.
Inefficient utilization of storage, container conditioning, and grease kettle space.
Excessive investment in laboratory and technical equipment.

COSTS

Operating and manufacturing costs have already been briefly discussed; for instance, see Chapter V, showing the derivation of a total manufacturing cost of 0.276c per pound (not including cost of materials) for a grease plant having a monthly production of about 900,000 pounds. Of the total direct expenses at this plant about 92 per cent was expended for labor (does not include overhead). Considering overhead expenses, but not

[1] See Report of Meeting of the N.A.O.L.G.M. Inc., Chicago (Oct. 16, 1934).

cost of ingredients, the manufacturing cost of 0.276c per pound may be allocated as follows:

	Per Cent
Total labor	33.0
Materials (not used in greases)	2.8
Overhead	64.2

If the average cost of grease making materials is taken at 1.25c per pound (this will vary greatly and will be much more for independent plants which do not manufacture their own lubricating oils) then the value of the grease in containers will be about 1.53c per pound. The average selling price of normal greases is about 5.8c per pound. The selling price may now be broken down as follows:

	Cents per Pound	Per Cent
Manufacturing labor	.092	1.590
Manufacturing material (not grease ingredients)	.008	.137
Manufacturing overhead	.180	3.120
Materials used in grease	1.250	21.550
Transportation of grease to customer (estimated)	.900	15.500
Sales costs	2.200	37.920
Estimated profit	1.170	20.183
Total selling price	5.800	100.000

If it is true that about 60 per cent of the sales costs may be considered as salaries, then labor represents roughly 27.5 per cent of the total selling price of a pound of grease taken at 5.8c per pound. It is of note, however, that these values represent averages rather than typical cases. For example, some grease manufacturers obtain much higher prices for their lubricants than others. In one case a manufacturer will sell a normal grease for 14 or 16c per pound whereas another factory operating on a quantity production basis, will sell at 3.5c. De Long [2] has found from a study of the costs of twenty-three chemical industries that the wage cost is about 24 per cent of the gross income for the products.

It will be noted from the above table that about 21.5 per cent of the selling price of grease is represented by the cost of raw ingredients used in manufacturing the grease. Data from other sources indicates that this value may run as high as 39 per cent. In any case these values are not excessive as compared with averages reported for all chemical manufacturing concerns which are approximately 50 per cent. However, since the labor ratio for the grease industry is not excessive (particularly for manufacturing is quite low) grease manufacturing management should be most vitally concerned with the purchase of raw materials. A careful development of purchase specifications, and the distribution of these to many suppliers will often result in considerable savings. Furthermore, a careful study of market trends for fats and lubricating oils will often indicate when a grease plant may stock up before prices become abnormally high. This, of course, implies that a modern grease plant has adequate storage for raw materials consumed over periods of two months or more. In some cases

[2] *Chem. Met. Eng.*, **32**, 919 (1925).

it is possible for the sales departments to prepare estimates of the volume of business which is anticipated for periods of six months or a year in advance. With these available, a grease plant manager is in a position to purchase raw materials to much better advantage, both from the standpoint of market trends and from purchase in quantity lots.

The average wages paid in chemical industries range from about $1000 to $1900 per year, with an average of about $1450. It is estimated that there are about 600 men actively engaged in the manufacture of grease in this country, the annual pay roll being (roughly) a million dollars. This, of course, is for labor only and does not include management and technical supervision. It is considered that from the standpoint of value of products each man should represent the manufacture of about $12,500 worth of grease. Thus, for the yearly manufacture of 10,000,000 pounds of grease, the plant should operate with 46 men. These would comprise:

One	Grease Plant Superintendent
One	Assistant Grease Plant Superintendent
One	Accountant
One	Clerk
One	Shipping Clerk
Two	Assistant Shipping Clerks
Ten	Car and Truck Loaders
One	Stock Clerk
Four	Barrel Washers
Two	Painters
One	Chief Technologist
Two	Grease Chemists
One	Grease Research Chemist
Three	Grease Makers
One	Superintendent of Filling Floor
Two	Fillers
One	Weigher
Two	Can Filling Machine Operators
One	Gauger and Sampler
One	Messenger boy
Two	Coopers
One	Mechanic
Two	Steam Plant Engineers
Two	Laborers or Janitors

Too often, the grease plant is not adequately manned, and many of the above functions may be merged, particularly where the grease plant is a part of a large oil refinery and service departments will not only serve the grease plant but other refinery departments such as cracking department, crude stills, lubricating oil plant, etc. It is obvious, of course, that where any of the above positions can be dispensed with, without decreasing efficiency or production, labor costs may be greatly reduced. As a matter of fact, in several grease plants less than half of the above indicated employees are used; and, in order to lower the manufacturing cost to about 0.25c per pound, a grease plant producing ten million pounds of grease per year must either pay very low salaries or reduce the pay roll to about twenty men.

ORGANIZATION

A complete grease manufacturing and sales organization must include the following functions:

Accounting, finance and credit.
Grease manufacturing.
Sales.
Transportation.
Sales service.
Engineering.
Research and development.

In this volume we are chiefly concerned with grease manufacturing which has been presented in the foregoing chapters, as well as research and development. There remains, however, the discussion of a few important items with respect to management and particularly its relation to research and development. Any successful grease plant must be controlled by a responsible management having due appreciation of all data pertinent to the manufacture of grease. The management should be particularly alert to new developments as the art and science of lubrication is changing rapidly and an unprogressive policy may mean that the successful grease plant of today will be the obsolete plant of tomorrow. Grease plant employees must be selected with great care and proper attention should be paid to obtaining men who will develop through foremanship to even more responsible positions.

TECHNICAL STAFF

Grease making is essentially a technical operation and therefore it would seem logical that the number of technical men in the organization should be as great as its size will permit. Four chemists, for a plant producing ten million pounds annually, to care for research, development and control is certainly not excessive, even though other industries may be satisfied with a smaller number. Companies capitalized at less than one-quarter of a million dollars must, of course, keep the number of technical men at a minimum. For special problems they may call in consultants who can solve their problems in many cases at less expense than they can maintain extensive research laboratories of their own.

For a grease plant the technical work will fall under the following headings:

Manufacturing control. Penetration tests, melting points, water determinations, calculation of kettle charges, standardization of formulae and manufacturing processes. (See Appendix).

Testing and Analysis. Development of purchase specifications, testing and approving incoming materials, analysis of competitive products and special analytical work.

Research and Development. Development of new uses for existing greases, development of new greases for newly discovered uses and the development of new applications of grease. Process development.

Sales service and sales development. Service station, garage, and industrial plant visitation to determine the exact conditions under which greases are used and the requirements they must meet. The extent to which a grease plant man will be required for this work will depend on the character of the sales engineers attached to his company. In view of the policies of some companies all, or nearly all, of the technical

data is kept in the hands of the manufacturing department; in others well qualified lubrication engineers, versed in grease manufacture and attached to the sales departments, render it unnecessary for plant employees to make field investigations, as these engineers are capable of reporting all necessary information.

In the organization of technical men the Chief Technologist should be in charge and the senior man. The research and development work should at first be started on a small scale and it may be increased as it is shown that investments along this line are profitable. In selecting men for the technical organization care should be taken to not secure two or more men who are more or less specialized along the same lines. One man may be a good analyst, another better at research, and another more adaptable to plant and process development and engineering. These factors should be carefully weighed in employing a new man and all factors well represented to secure a well balanced technical group. Grease making is closely allied with organic chemistry. Therefore, the research man should be well versed along this line and have a good background of colloidal chemistry. A physical chemist or rheologist, a chemical or lubricating engineer, and a mechanical engineer are all needed to round out a satisfactory organization. At the start recent college graduates, whose salary requirements may not be great, may fill most of these positions. But, as the success of the group is established their efforts should be substantially rewarded. The chief technologist should be a man of considerable experience and able to inspire outstanding work in his associates. The technical group should be charged with the full responsibility for the quality of the products shipped. If this is well understood, the group should work harmoniously together and serve as watch dog for the grease production. If products are manufactured which do not comply with the specifications developed by this technical group, and there is need to ship them notwithstanding, then the management should approve the deficient batches or abide by the technical group's negation.

The management should not expect a too rapid development of new ideas and new products. It is often a difficult matter to adapt a process which has been successful in glassware in the laboratory to full scale production in the grease kettles. Very frequently, the chief cost of development work is in just this phase, and it may be necessary to make several commercial scale batches before the process can be standardized and specification ranges established. An appreciation of these facts by the management is sometimes lacking, and sales demand may force the development of several new products within less than a month's time, whereas from one to six months may be needed to fully consider all phases of even one of the problems. Such rapid development work is frequently carried out and, unfortunately, is seldom profitable as the necessary technical work must be carried out at subsequent periods. When this work is done piecemeal it is more costly than when done systematically.

Soap contents are very important from the standpoint of control and cost. It is probably best to have the weigher report to the control chemist the exact weight of each batch of grease; this, compared with the formula

used by the grease maker, the consistency tests, and other data, should indicate whether a satisfactory yield is being obtained without the necessity of making a chemical analysis. Such an analysis in some cases, would cost more than the value of the loss or gain in grease which it is sought to control. Each grease plant must work out its own list of specifications and requirements, approved by the management, and meeting the needs of the individual plant. The policies vary so greatly in this respect from plant to plant, that it may be stated no universal rule applies. A thorough study of suggested manufacturing specifications presented in this book should, however, do much to bring plants into agreement as to what is considered essential and what is extraneous.

Where careful control is insisted on, three one-pound samples should be taken from different containers of each batch shortly after it has been filled. One of these samples is filed for a period of not less than six months and serves as a reference sample in case any complaint is made. Each drum of grease, and each carton, should be stenciled with a batch identifying number and date and the three samples should be similarly marked. The remaining one-pound samples are sent to the laboratory for final approval by the control chemist; one for water determination, ash, free fatty acids and other chemical tests, while the other is used for making necessary consistency tests. Thus time is saved by having a separate sample which may be brought to 77° F. in a thermostatically controlled water bath, releasing the second sample for the immediate attention of the analytical chemist.

RESEARCH PROGRAM

In some firms the research program, particularly new greases for development, are dictated by the sales department. This practice has both good and bad features. The sales staff is, of course, in close touch with the needs of their field, but may not have a full appreciation of those problems, of a more fundamental nature, which may be of great importance with respect to revolutionary changes in lubricating grease manufacture. It is considered best practice to split the problems, allocating about 70 per cent of the time and money available to meeting direct sales needs, and utilizing the balance to studying what might be termed "kite flying" investigations. These are, more or less, pure research in contrast to the applied research demanded by the sales departments.

It is desirable that manufacturing and sales executives should realize the possibilities offered by lubricating grease research. If they fully appreciate these possibilities they will ordinarily provide adequate financial assistance and moral support. In some of these cases the chemist must adopt the technique of the scientific detective to establish the cause of contamination. In one example, a shipment of grease badly contaminated with dirt was returned to the manufacturers and damages claimed on account of the contamination. Examination by the chemist revealed the presence of a fish scale, a small piece of oakum and wood splinters. Since none of these materials were considered likely to have been introduced into the grease

at the time of manufacture, a further investigation was made relative to the means of transportation, and it was subsequently learned that in loading a drum of the grease aboard ship it had been dropped from the sling, the head forced open and the grease spilled on the dock. The lubricant had then been shoveled back into the drum, the head closed, and the shipment continued without notifying the shipper. In the case of high pressure chassis lubricant, clogging of the small screens on the grease pumps frequently occurs, due to the presence of various kinds of contaminants. In some cases, these materials were found to be wood slivers from the paddle used by the customer for transferring the grease from the drum to the grease gun container.

Lubricating greases which have been carried in stock for many years frequently oxidize, dry out, or separate oil; all of which may result in complaints. The remedies for these difficulties are obvious of course; the incorporation of suitable anti-oxidants and color stabilizers, the use of improved emulgators having a greater affinity for water, and insistence on more rapid turn-over of stocks.

Bearing failures are seldom due to the quality of the lubricant. More often than not they may be traced to mechanical defects, such as improper design, utilizing bearings which are not large enough for the purpose in hand, improperly fitted bearings, excessive pressures developed by belts which are too tight, and repair jobs in which old and worn parts have been used as replacements.

The decision as to whether or not comprehensive and fundamental researches should be undertaken to develop new types of lubricating greases, or to utilize the technical staff for merely analyzing and duplicating competitors' products, may depend on the character and personal qualifications of the research staff. Unfortunately, executives in many cases demand too much from their technical organizations. If results cannot be immediately converted to monetary profits they may consider research as being of little use and wasteful of the company's resources. It seems likely that the grease industry is too prone to carry out applied researches for the solution of some immediate problem without giving proper attention to fundamental researches. By-products and new ideas may be developed from the latter course which may prove much more profitable and valuable than could be predicted at the initiation of the problem.

In the larger companies a research and development committee, made up of representatives of the sales, manufacturing, and technical departments should give consideration to the more important research problems. This committee should decide what problems are of sufficient importance to undertake, and after they have been prosecuted for some time should indicate whether or not they should be continued.

COMPLAINTS

The technical staff will frequently be called on to investigate complaints. Where the batches of grease have been given proper identification symbols,

and these designations can be traced, a reference to the retained sample and the tests made may be all that will be required. In many cases, the complaints will be due to contamination with such material as moisture from rain or snow, dirt, grit or other contaminants. A careful examination of complaint samples, and the skilful use of the microscope will often reveal the nature of the contaminating material and give a clue as to where contamination occurs.

Excessive moisture in cup and gear greases will, in some cases, cause objectionable frothing, foaming, and possible loss of lubricant. This may be due to mixing different types of greases, or to contamination with rain water while the grease is in storage, or in use in gear housings. Contamination of ball and roller bearing greases with grit or dirt is, of course, a serious matter and every effort to maintain the lubricating greases in a cleanly condition should be made. Greases which are broken down, or separated, present an objectionable appearance, and may be wasteful since they will pass through the bearings or leak through housings. Greases which are too consistent will be channeled in ball bearings and may produce high frictional losses or will not promote proper lubrication due to lack of mobility.

Where grease feed lines are of small dimensions the flow characteristics of the grease at operating temperatures are quite important. In addition, if the grease is forced through small orifices separation of soap can take place with certain greases, resulting in clogging of the line and complaints by the customer.

DEVELOPMENT PROBLEMS OF THE FUTURE

The following suggestions are indicative of the course which may be taken by grease research of the future. It is not intended to discuss these items in detail but to merely list problems which undoubtedly will be given considerable study during the next decade:

Investigations of various ingredients for producing a higher degree of oiliness. A study of such materials in the presence of various soaps is greatly needed.

Various agents for increasing film strength and imparting extreme pressure properties of soap base lubricating grease require further study. The effects of materials for reducing friction under high pressure conditions should be considered simultaneously.

The improvement of lubricating greases from the standpoint of increased melting points and decreased yield values or internal friction.

Improvement of lubricating greases with respect to service characteristics at subzero temperatures and simultaneous improvement of non-leaking ability.

The development of soap base greases of greater mechanical stability. Some greases appear to be broken down by the disruptive forces existing in various bearings.

Development of greases having soap bases which are stable and do not separate from the oil phase when subjected to temperatures sufficient to evaporate nearly all of the water content.

The development of new types of non-soap base greases, such as those made with various organic polymers, and cellulose derivatives.

The discovery of greases made with new bases which will impart high melting points (above 275° F.) and which are not readily emulsified by or soluble in water. Soda soap greases may be satisfactory from the standpoint of melting point, but are

not sufficiently water resistant; whereas calcium soap base greases are resistant to moisture but ordinarily have melting points below 210° F. The greases made with 10 per cent of the stearates of calcium, cobalt, lead, magnesium, nickel, and strontium, all have melting points of less than 215° F.; whereas 10 per cent of mercury stearate will produce a grease having a melting point of only 225° F.

It is, therefore, obvious that the search for superior grease bases, whether they are soaps or other compounds, must be continued.

Appendix

Analytical Methods and Tests

In this section details will be presented for conducting the most ordinary chemical laboratory analyses and physical laboratory tests applicable to lubricating greases. It is intended that the directions given herewith should be sufficiently clear to enable any well trained chemist to carry out the various methods indicated.

Special service tests for automobile lubricating oils and gasoline have been more or less standardized, but, as will be pointed out in this discussion, the service testing of lubricating greases has been largely a matter of special technic developed as occasion necessitated by the grease manufacturer, the automobile manufacturer, institution, or consumer. Because casual observation of service results in equipment on the road often leads to erroneous results and comparisons, laboratory service tests are frequently resorted to in order to secure more precise data from which definite conclusions may be drawn. In some cases, a series of greases differing only slightly in their usual properties must be studied to determine their relative service value. Road tests for thousands of miles might be needed to reveal practical differences, but accelerated laboratory service tests can be devised which enable definite comparisons to be made in a few hours.

Obviously, service tests may be used for proving that under a given set of conditions one grease is superior to another. The time-honored tests for melting point, penetration, percent water, percent soap and percent ash, while of greatest value as control tests to secure uniform quality, leave too much to the investigator's imagination in predicting service performance. Carefully performed laboratory service tests are believed to be invaluable as indicators, pointing the direction in which definite improvements in greases may be made. The following tabulation records a large number of tests which may be applied to lubricating greases. Some are of a routine nature and others are only rarely conducted. The chemical laboratory tests, as shown, are classified under two headings: A, control tests and analyses of a routine nature; and B, analytical quality tests. Physical laboratory tests may be similarly classified.

Chemical Laboratory Tests

A. *Control Tests*

1. Percent water, A.S.T.M. D95-30
2. Corrosion (copper strip)
3. Saponification number
4. Percent free alkali (free acid) A.S.T.M. D128-27 (See Grease Analysis)

B. *Quality Tests*

1. Grease analysis, A.S.T.M. 128-27
2. Percent fillers
3. Percent sulfur
4. Percent mineral oil
   Mineral oil tests:
   Gravity

S.U. viscosity
Color
Flash point
Fire point
5. Percent soap
6. Kind of soap
7. Total mixed fatty acids:
Neutralization number
Iodine number
Solidification point
Micro-examination
Acetyl number
S.U. viscosity
Special tests for unsaturation
Valenta test
8. Oxidation tests
9. Corrosion tests (special)
(Long periods, presence of water, etc.)
10. Bacteriological tests

Physical Laboratory Tests

A. *Control Tests*

1. Penetration test at 77° F. A.S.T..M. D217-33T
2. Saybolt viscosity
3. MacMichael viscosity
4. Melting point:
Gillette
Ubbelohde
5. Density
6. Color
7. Texture
8. Adhesion tests
9. Channel tests

B. *Quality Tests*

1. Coefficient of friction
2. Film strength
3. Mechanical stability tests
4. Susceptibility to boiling water
5. Aeration tests
6. Resistance to capillary action
7. Microscopic examination
8. Ultra-microscopic examination
9. Separation by centrifuge
10. Separation by ultra filtration
11. Diffusion tests
12. Hygroscopic tests
13. Pressure viscosity at constant shearing rates
14. Pressure viscosity at constant pressures
15. Stability to heat

CHEMICAL LABORATORY TESTS

Control Tests

Water Content

The methods published by the American Society for Testing Materials, Philadelphia, Penna., and known as the "A.S.T.M. Standards on Petroleum Products and Lubricants," have been well standardized and, in most cases, are to be preferred for the examination of lubricating greases. Methods of sampling and testing lubricants published in the U. S. Federal Stock Catalog, Section IV (Part 5),[1] are similar and may also be consulted. These are quoted as follows:

F-5q. METHOD 300.13—WATER IN PETROLEUM PRODUCTS AND OTHER BITUMINOUS MATERIALS. [A.S.T.M., D95-30]

F-5q (1). This method of test determines water existing in a sample of bituminous material by distilling the sample with a volatile solvent. This method is suitable for a variety of materials but is especially applicable to petroleum, fuel oil, road oil, coal tar, water-gas tar, coke-oven tar, and other petroleum products or bituminous materials.

APPARATUS

F-5q (2). 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.

[1] U. S. Federal Standard Stock Catalog, VV-L-791 (October 3, 1933).

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.

F-5q (3) a. The metal still 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.

F-5q (3) b. The glass flask shall be of the short-neck, round-bottom type, made of well-annealed glass, having an approximate capacity of 500 ml.

F-5q (4). 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.

F-5q (5). 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° from the vertical axis of the condenser.

F-5q (6). The trap shall be made of well-annealed glass constructed in accordance with figure 20 (*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.

F-5q (7) 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, determined in accordance with method 100.14 for distillation of gasoline, naphtha, kerosine, and similar petroleum products (paragraph F-5E).

Five per cent shall distill at a temperature not below 194° F. (90° C.) nor above 212° F. (100° C.).
Ninety per cent shall distill below 410° F. (210° C.).

F-5q (7) 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, determined in accordance with the method 100.14 for distillation of gasoline, naphtha, kerosine, and similar petroleum products.

Ninety-eight per cent shall distill between 248° F. (120° C.) and 482° F. (250° C.).

SAMPLE

F-5q (8). 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

F-5q (9). When the sample to be tested contains less than 10 percent 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 percent 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. 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 2 to 5 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.

F-5q (10). 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, A.S.T. M. method."

ACCURACY

F-5q (11). 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.

Corrosion Tests [1]

F-5ll. METHOD 530.31—CORROSION TEST AT 212° F. (Copper strip)

F-5ll (1). Place a clean strip of mechanically polished pure sheet copper about ½ in. wide and 3 in. long in a clean test tube. Add enough of the sample to be tested to cover the strip completely. Close the tube with a vented stopper and maintain for 3 hours at 212° F. Rinse the copper strip with sulfur-free acetone and compare it with a similar strip of freshly polished copper.

F-5ll (2). Discoloration of pitting indicates corrosion.

F-5mm. METHOD 530.4—CORROSION TEST FOR SOLID OR SEMISOLID PRODUCTS

F-5mm (1). This method of test shall be used for the detection of corrosive properties of greases or other solid or semisolid products.

F-5mm (2). Clean, well-polished sheets of copper shall be used for this test. In general, sheets about 1 in. (25 mm) square will be found most convenient.

F-5mm (3). Place a small portion of the grease to be tested on each of the metal plates, taking care not to cover the whole surface of the metal plates. Cover each plate with an inverted watch glass and let stand at room temperature for 24 hours. There must be no evidence of corrosion on the plate, either when examined with the sample in place or after removing it by washing with benzol. Either the development of a green color in the material on the copper or a brown stain remaining on the copper after washing with benzene which passes the corrosion test, method 530.23 (sec. F-5kk), is evidence of corrosion.

F-5nn. METHOD 530.6—CORROSION TEST AT ROOM TEMPERATURES

F-5nn (1). This method of test shall be used for the detection of corrosive compounds in soluble cutting oils.

F-5nn (2). Clean strips of mechanically polished sheet steel and copper about ½ in. wide and 3 in. long shall be placed separately in suitable clean tubes or sample bottles. The cutting oil under test shall be added so that the strips are but partly

immersed and the tubes or bottles shall be closed with corks. The containers shall then be manipulated so as to wet completely the metal strips with the cutting oil. The tubes or bottles shall be set up in an approximately vertical position and allowed to remain in the laboratory at room temperature (70° to 90° F.) for 48 hours.

F-5nn (3). At the end of the 48-hour period the metal strips shall be removed from the containers and washed with benzol which passes corrosion test 530.23 (sec. F-5kk). The presence of corrosive compounds is indicated by corrosion or discoloration of the steel or copper strips when compared with freshly polished ones.

F-5nn (3) a. A soluble cutting oil shall be reported as passing the test when the exposed strips show at most an extremely slight discoloration.

F-5nn (3) b. A soluble cutting oil shall be reported as not passing the test when the exposed strips of either metal show more than extremely slight discoloration.

Saponification [1]

F-oo. METHOD 540.11—SAPONIFICATION NUMBER. [A.S.T.M., D94-28]

F-5oo (1). This method is one of the means used for the identification of unmixed animal and vegetable oils, and for measuring the quantity of fatty material in compounded products. It may also be used in the study of noncompounded petroleum products.

APPARATUS

F-5oo (2). The saponification shall be carried out in a wide-mouthed flat-bottom extraction flask, or Erlenmeyer flask of chemically resistant glass, of 250 to 300-ml capacity, fitted to a reliable condenser properly connected with a good cork. The boiling shall preferably be carried on by means of an electric hot plate.

SOLUTIONS REQUIRED

F-5oo (3) a. *Alcoholic solution for saponification.*—Dissolve 58 g of potassium hydrate "purified by alcohol" in 500 ml of 95 per cent purified ethyl alcohol. Allow the solution to settle in a dark place. Draw off the clear solution or filter through an asbestos filter and make up to one liter with 95 per cent alcohol. The solution so prepared shall stand at least 20 hours before it is standardized.

F-5oo (3) b. *Alcohol.*—Purify 95 per cent ethyl alcohol with silver oxide in the following manner: Dissolve 1.5 g of silver nitrate c.p. in about 3 ml of water and add to 1 liter of alcohol in a glass-stoppered bottle, and mix thoroughly. Dissolve 3 g of potassium hydrate (by alcohol) in 10 to 15 ml of warm alcohol. After cooling, add slowly to the alcoholic silver nitrate solution, stirring slightly. Allow the precipitated silver oxide to settle, siphon off the clear solution and distill on a steam bath.

F-5oo (3) c. *Standard hydrochloric acid solution.—One half normal solution.*

F-5oo (3) d. *Phenolphthalein solution.*—One gram phenolphthalein in 100 ml alcohol and water.

PROCEDURE

F-5oo (4). *Blank determinations.*—Determination shall be made in duplicate in the alcoholic potash solution in the following manner: Measure accurately into the flask 25 ml of alcoholic potash solution from a calibrated pipette. The tip and outside of the pipette shall be wiped off with a clean filter paper before the solution is delivered. Then rinse out with 25 ml of neutral alcohol. If a standard burette is used, allow 60 seconds total time for drawing and draining. Connect the flask to a suitable condenser and boil for three hours. Before disconnecting the flask, wash out the condenser with a few milliliters of neutral alcohol. If a Soxhlet is used as the condenser the tip shall be washed off into the flask. Titrate while hot with 0.5 *N* HCl using three drops of phenolphthalein indicator. The total number of milliliters of 0.5 *N* HCl required for the blank represents the strength of the alcoholic potash solution.

F-5oo (5) a. *Test sample.*—For straight fats or oils use a sample of from 2 to 3 g of the material, for oils containing over 30 per cent of fatty oils use about 5 g and for oils containing less than 30 per cent of fatty oils use about 10 g.

F-5oo (5) b. *Procedure.*—Weigh the oil accurately, by difference, from a small beaker into the saponification flask. Add 25 ml of alcoholic potash solution and 25 ml

of neutral alcohol in the same manner as for the blank, connect to the condenser and boil for three hours. Titrate while hot. Calculate the saponification number from the difference between the number of milliliters of 0.5 *N* HCl required for the determination and the average of the two blanks, using the formula:

$$\text{Saponification number} = \frac{\text{difference, milliliters} \times 28.05}{\text{weight of oil, grams}}$$

Petroleum ether may be used with compounded cylinder oils in the quantity of 50 ml providing a Soxhlet extraction flask is used periodically to collect this petroleum ether. The Soxhlet flask shall be so adjusted, through the addition of glass rodding or beads when necessary, so that it will just overflow with the full quantity of petroleum ether.

F-500 (6) a. The percentage of fatty oil (or fat) in a compounded petroleum product can be calculated from the saponification number of such a product only when the saponification number of the fatty oil is known. If the saponification numbers of both fatty oil and compounded oil are known, the following formula shall be used:

$$\text{Percentage of fatty oil} = \frac{100 \times \text{saponification number of compounded oil}}{\text{saponification number of fatty oil}}$$

F-500 (6) b. For this determination the following values of saponification number may be used:

Fatty oil:	Saponification
Lard oil	192–198
Tallow	193–198
Neatsfoot	193–204
Fish	140–193
Sperm	120–140
Castor	176–187
Rapeseed	170–179
Soya bean	189–197
Peanut	186–197
Cottonseed	191–197
Blown rapeseed	195–216
Blown cottonseed	210–225
Degras	110–210

Note.—The above calculations and data presume a knowledge of the nature of the fat present. In an unknown sample, and particularly when a low saponification number leaves doubt as to whether fat is actually present, the fatty acid may be extracted (as the soap) and recovered for gravimetric determination. For this purpose, the saponified and neutralized solution, obtained as above, shall be handled as in a grease analysis, as described in the second paragraph of section 19 of method 541.2 of analysis of grease (paragraph F-5qq). The fatty acid is thus obtained and may then be used for subsequent identification tests.

ACCURACY

F-500 (7). It may be expected that the same operator will find variations of ± 0.3 saponification number on compounded oils containing less than 30 per cent compounding; ± 0.5 saponification number on compounded oils containing over 30 per cent compounding; and ± 1.0 saponification number for unmixed fatty oils. Corresponding permissible variations for different operators will be ± 0.5, ± 1.0, and ± 2.0 saponification numbers, respectively. For dark oils, the errors may be double this magnitude.

F-5pp. METHOD 540.2—FATTY OIL

F-5pp (1). The percentage of fatty oil shall be calculated from the saponification number (method 540.11 (sec. F-500)) by means of the following formula:

$$\text{Percentage of fatty oil} = \frac{100 \times \text{saponification number of the sample}}{\text{saponification number of fatty oil}}$$

F-5pp (2). The saponification number of the fatty oil used in compounding shall be assumed to be 195, unless the manufacturer of the sample under examination submits an affidavit stating the kind of fatty oil used and its saponification number.

Quality Tests

A.S.T.M. Grease Analysis [1]

F-5qq. METHOD 541.2—ANALYSIS OF GREASE. [A.S.T.M. D128-27]

F-5qq (1). These methods of analysis permit determinations sufficiently accurate for referee purposes of all the constituents of greases likely to be covered by specifications. These constituents are fillers and ash, soap bases, soap, fat, water, excess alkali or acid, petroleum products and unsaponifiable matter. No quantitative determination of glycerin is described, but a procedure is outlined for its qualitative detection.

SAMPLE

F-5qq (2). The size of sample for the soap determination (methods (*1*) and (*2*) for determination of fillers, soap, fat, petroleum oil, and unsaponifiable matter) shall be from about 8 to 30 g depending on the consistency of the grease, which is chiefly determined by the percentage of soap present. Ten to twenty grams are usually a convenient amount for No. 3 cup grease, while thin transmission and other greases require a larger sample. The original sample should be stirred or mixed until uniform.

F-5qq (3). Samples need not be weighed more closely than 0.1 g.

SOLVENTS

F-5qq (4). The petroleum ether shall have an end point not higher than 200° F., and 125 ml of it shall not consume more than 0.2 ml of 0.5 N potassium hydroxide when a blank test is made as follows:

(Section IV, part 5)

One hundred and twenty-five ml of petroleum ether shall be boiled with 10 ml of 0.5 N alcoholic potassium hydroxide and 50 ml of neutral 50 per cent alcohol for 1½ hours on a hot plate, using a glass tube of about 7 mm internal diameter and 75 cm long as a reflux condenser. After cooling, the solutions shall be titrated with 0.5 N hydrochloric acid and phenolphthalein. Not less than 9.8 ml of 0.5 N acid shall be required for neutralization. The amount of alkali consumed in this test shall be deducted as a blank correction in the fat determination on solution (E).[20]

F-5qq (5). The alcohol shall be prepared from commercial 95 per cent grain or denatured grain alcohol by distilling from sodium hydroxide, diluting to 50 per cent (or 70 per cent) by volume with distilled water and neutralizing exactly with sodium or potassium hydroxide and phenolphthalein.

F-5qq (6). The phenolphthalein solution shall be prepared by dissolving 1.0 g of phenolphthalein in 50 ml of strong redistilled alcohol, adding 50 ml of water, and neutralizing with sodium or potassium hydroxide.

F-5qq (7). Concentrated c.p. hydrochloric acid shall be used when "concentrated hydrochloric acid" is specified. Hydrochloric acid (10 per cent) shall contain 10 per cent by weight of absolute HCl, with a permissible variation of ± 0.5 per cent.

PROCEDURE

(A) Determination of Ash

Note.—The determination of the total ash should not in general be regarded as of any great importance. It is, however, sometimes required. This determination is often unsatisfactory on account of interaction between sodium carbonate derived from the soap, and inorganic fillers. There is always likelihood of reaction with the porcelain crucible itself on account of the long-continued heating necessary to burn out all carbon. Moreover, if much sodium or potassium carbonate is present, the ash is fusible and often encloses carbon,

[20] Solutions designated by capital letters in parentheses refer to the solutions given in chart.

making complete removal of the latter very difficult. Results will always be low in the presence of easily reducible oxides of volatile metals. There is also uncertainty as to when calcium carbonate has been completely ignited to calcium oxide. Ash determinations made on the same sample in different laboratories are likely to vary widely. For these reasons it is usually preferable to make separate determinations of the percentage of fillers and of soap, from which data a calculation is easily made giving all the information to be gained from a direct determination of total ash.

F-5qq (8). The percentage of ash shall not be included in the total of the analysis.

(1) Rapid routine method of ash determination

F-5qq (9). From 2 to 5 g of grease shall be placed in a weighed porcelain crucible, and the sample weighed to the nearest 0.1 g. If lead or zinc coap is known to be absent, a platinum crucible is more convenient. The combustible matter shall be burned off slowly and the residue finally ignited until the ash is free of carbonaceous matter. The crucible and contents shall then be cooled in a dessicator and weighed, and the result reported as percentage of ash.

(2) Alternative method of ash determination

F-5qq (10). The sample shall be burned as in method (*1*) until the ash is nearly free from carbon. The crucible and contents shall then be cooled, the soluble portions dissolved in a little water, and a slight excess of dilute sulfuric acid added, running the acid in carefully from a pipette inserted under a small watch glass covering the crucible. The crucible and contents shall then be warmed on the steam bath until effervescence has ceased. The watch glass shall then be rinsed with water into the crucible. The solution when tested with methyl orange for free acidity shall show free acid present. The contents of the crucible shall be evaporated to dryness, and the whole ignited at a low red heat, adding a few small pieces of dry ammonium carbonate to drive off the excess of sulfur trioxide. After cooling and weighing, the result shall be reported as "percentage of ash as sulfates."

The alternative method gives more concordant results than the first method, but requires more time and manipulation.

(B) QUALITATIVE EXAMINATION OF ASH

F-5qq (11). An easily fusible ash, dissolving completely in water to give a strongly alkaline solution, indicates a grease containing sodium, potassium, or both. A white infusible ash, practically insoluble in water, but imparting to it an alkaline reaction, may indicate calcium, with or without magnesium or aluminum. Zinc is shown by the yellow color of the ash while hot, and lead may be indicated by the presence of metallic globules, or by the yellow color of the ash when cold.

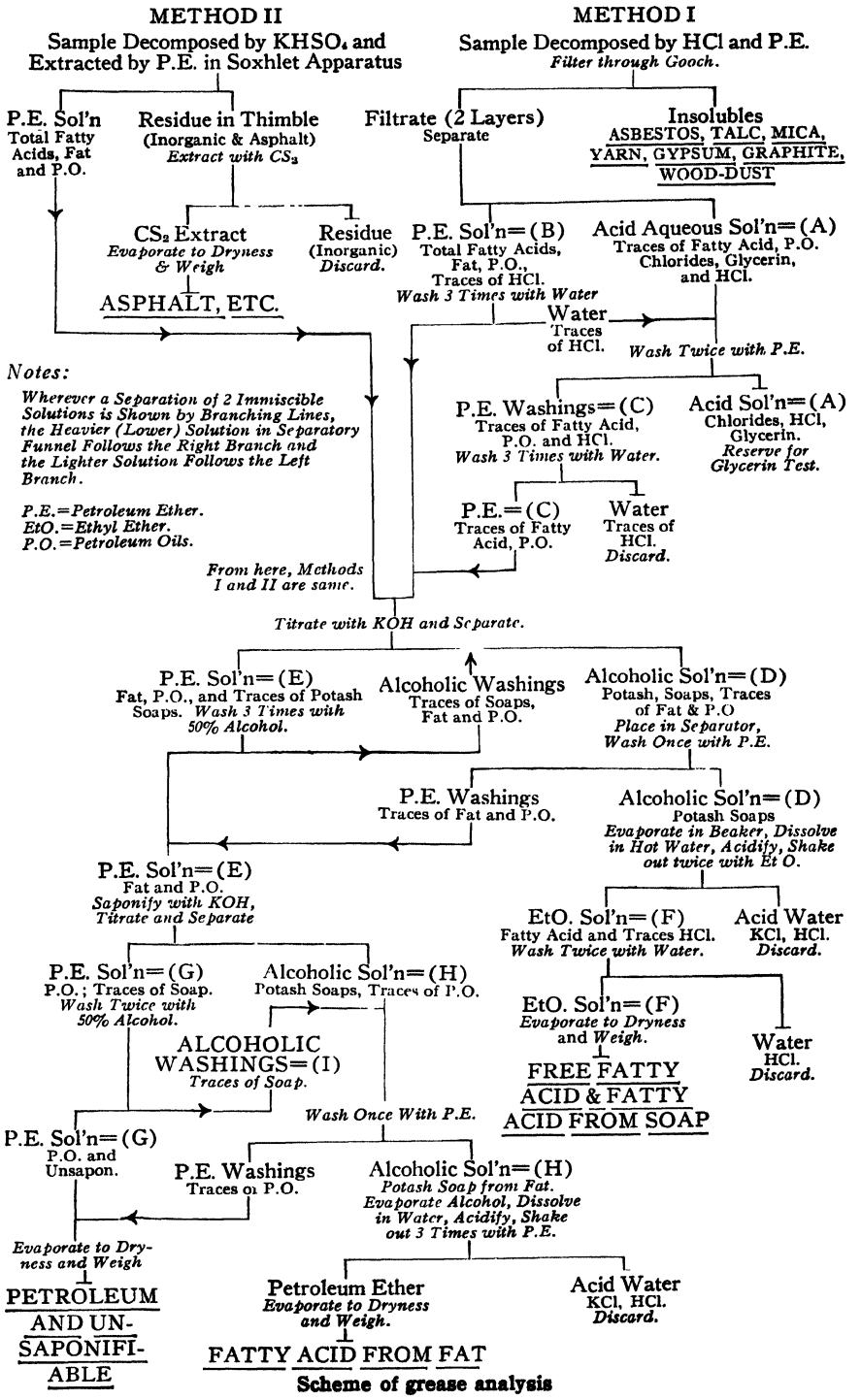
F-5qq (12). The ash shall be dissolved in dilute nitric or hydrochloric acid, and the presence of the several bases confirmed by suitable chemical tests, any standard scheme of qualitative analysis being followed.

(C) QUANTITATIVE EXAMINATION OF ASH

F-5qq (13). For the quantitative examination of ash any standard analytical procedure shall be used, the choice of methods being based on the information gained from the qualitative tests. If only one base is present, a quantitative determination is in general unnecessary.

(D) DETERMINATION OF FILLERS, SOAP, FAT, PETROLEUM OIL, AND UNSAPONIFIABLE MATTER

F-5qq (14). *Choice of method.*—Cup, fiber, and sponge greases, with or without graphite, and all comparatively light-colored greases, including axle greases, shall be examined by method (*1*). Dark greases containing residuum, asphaltic oils and asphalt, tars, etc., shall be examined by method (*2*), as these ingredients usually cause stubborn emulsions if a shaking-out process is applied at the start.



Scheme of grease analysis

(1) Determination of filler, soap, fat, etc.

F-5qq (15). If no fillers are present, paragraphs F-5qq (15) and (16) shall be passed over and the grease sample introduced directly from a weighing bottle into a separator, the weight of the sample being obtained by difference. (See par. F-5qq (2).) The sample shall then be shaken with 75 ml of petroleum ether and 50 ml of 10 per cent hydrochloric acid until the grease is completely decomposed, after which paragraph F-5qq (17) shall be followed. If fillers are present, the sample (see par. F-5qq (2)) shall be weighed in a small beaker, 50 ml of 10 per cent hydrochloric acid added and the beaker warmed on the steam bath, stirring until all soap lumps have disappeared and the upper layer is clear. If undissolved mineral matter or other filler is present, both layers shall be filtered through a Gooch crucible provided with a suitable mat, the beaker and crucible washed with water and petroleum ether, and the crucible finally washed with strong alcohol, the alcohol washings being collected separately and discarded. The crucible and contents shall then be dried at 120° C. and weighed, and the result reported as "percentage of insoluble matter" (graphite, mica, talc, asbestos, gypsum, wood pulp, etc.).

F-5qq (16). If gypsum is present as a filler, the results obtained in the determination described in paragraph F-5qq (15) will be too low because of the solubility of calcium sulphate in hydrochloric acid. A sample of about 5 g. of grease shall be weighed in a small beaker, and decomposed with 50 ml of petroleum ether and 25 ml of concentrated hydrochloric acid, warming on the steam bath until all gypsum is dissolved. The contents of the beaker shall be cooled and poured into a separatory funnel, the beaker rinsed with water and a little petroleum ether, and the solutions in the funnel allowed to separate clearly. The lower layer shall then be drawn into a beaker, and the petroleum ether in the separator washed with two portions of 20 ml each of 10 per cent hydrochloric acid, and the washes added to the strong acid solution in the beaker. The hydrochloric acid solution in the beaker shall then be evaporated on the steam bath almost to dryness, diluted to 150 ml with water, heated to boiling and treated with 10 ml of 10 per cent barium chloride solution. The barium sulfate shall then be filtered off and ignited in the usual way, and from the weight of the barium sulfate the percentage of calcium sulfate shall be calculated, reporting it as $CaSO_4 . 2H_2O$.[21]

F-5qq (17). *Determination of soap.*—The solution resulting from the decomposition of the filler-free grease or the combined filtrate and washes from the determination of fillers (par. F-5qq (15)), except the alcohol, which is discarded, shall be placed in a separator, allowed to clear, and the aqueous layer (A), which contains all the bases as well as glycerin, shall be drawn into another separator. The petroleum ether layer (B) shall be washed three times with 25 ml of water to remove hydrochloric acid, adding the washings to (A). Solution (B) shall then be run into a 250-ml separator. The aqueous solution (A) shall then be washed twice with 20 ml of petroleum ether (C), whereupon (A) is set aside for examination for glycerin. (See par. F-5qq (18).) Solution (C) shall then be washed once with 15 ml of water, which is rejected, and (C) shall be added to (B). If (B) and (C) are comparatively light colored, an approximate determination of free fatty acids and fatty acids from soap may now be made by titrating the solution in the separator with 0.5 N alcoholic potassium hydroxide and phenolphthalein, using 200 as the average neutralization value of the fatty acid (that is, 1.0 g of fatty acid requires 200 mg of absolute KOH for neutralization). If solutions (B) and (C) are dark, a few drops of phenolphthalein and sufficient 0.5 N alcoholic potassium hydroxide shall be added to make the alcoholic layer distinctly alkaline after vigorous shaking. If the solutions have been titrated, a slight excess of alkali shall be added before separating. The conservative addition of strong alcohol at this point may aid in securing rapid and sharp separation. The two solutions shall be allowed to separate sharply and the lower alcoholic layer (D) drawn off into another separator. The upper petroleum ether layer (E) shall be washed three times, with 30, 25, and 20 ml of neutral 50 per cent alcohol, respectively, adding these washes to (D). Solution (D) shall be washed with 25 ml of petroleum ether, after which (D) is drawn off into a beaker and the petroleum ether added to (E). Solution (D) shall then be evaporated to a small volume to remove alcohol, the residue of potassium soap washed into a separator with hot water, acidified with hydrochloric acid and shaken out twice with 50 and 25 ml of ethyl ether (F), respectively. Solution (F)

[21] Chalk and other forms of alkaline earth carbonates are not determined by this method, but are considered under the determination of free alkalinity, par. F-5qq (24).

shall be run into another separator and washed twice with 20-ml portions of water, which is discarded. Solution (F) shall then be transferred to a weighed beaker and evaporated to dryness on the steam bath, blowing with air to remove all traces of petroleum ether. The residue consisting of free fatty acid and fatty acid from soap shall be heated for a short time on a steam bath, adding and evaporating 5 ml of absolute alcohol to remove the last traces of water, and weighed. The exact neutralization value shall then be determined on as large a sample of these fatty acids as possible. From the total fatty acid found here, the free acid if any (see par. F-5qq (23) and (25)) shall be deducted and the remainder calculated to percentage of soap, the ash analysis being used as a guide in distributing the bases, if more than one are present.

The fatty acids may be identified to some extent by special tests, such as odor, crystal form, melting point, iodine number, neutralization value, color reactions, etc.

If the grease is appreciably oxidized, the fatty acids obtained by the ethyl ether extraction are likely to be dark in color and hard to identify. For further study and identification, the neutralized acids may be extracted again (qualitatively) with petroleum ether and dilute HCl, thus eliminating the small amount of more darkly colored matter.

F-5qq (18). *Detection of glycerin.*—In case it is desired to determine whether a grease has been made from whole fats or from fatty acids, solution (A) shall be neutralized with dry sodium carbonate and sufficient excess added to precipitate calcium or other metals. The whole mass shall then be evaporated to dryness, the residue extracted several times with strong alcohol, the combined alcoholic extracts filtered, and the alcohol exaporated. The residue will then contain most of the glycerin, with a little sodium chloride. The presence or absence of glycerin in the residue shall be confirmed by suitable qualitative tests.

Since fats on saponification yield between 10 and 11 per cent of glycerin, and this analytical scheme does not include a quantitative determination of glycerin, the total percentages in greases made from fats should always be less than 100.[22]

F-5qq (19). *Determination of fat.*—The petroleum ether solution (E) (see par. F-5qq (17)), containing free fat, petroleum oils, and unsaponifiable matter, shall be concentrated to a volume of about 125 ml in a 300-ml Erlenmeyer flask, 10 ml of 0.5 N alcoholic potassium hydroxide and 50 ml of strong neutral alcohol added, and the whole boiled on a hot plate with an air condenser for 1½ hours. The uncombined alkali shall be titrated with 0.5 N hydrochloric acid, and from the alkali consumed, corrected for the blank determination, as specified in section 4, the percentage of free fat shall be calculated, using 195 as the average saponification value (that is, 1.0 g of fat requires 195 mg of absolute KOH for saponification).

The titrated solution (G) shall be placed in a separator, the alcoholic lower layer (H) drawn off into another separator, and the remaining traces of soap removed as described for (B) and (C), paragraph F-5qq, making only two washes with 30 and 20 ml of 50 per cent alcohol (I). Solutions (H) and (I) shall be combined, washed once with a little petroleum ether, which is added to solution (G), and solutions (H) and (I) then evaporated to a small volume and the fatty acid isolated as described for (D), paragraph F-5qq. The percentage of fat may be checked by weighing and titrating the free fatty acid.[23]

F-5qq (20). *Determination of petroleum oil.*—The petroleum ether solution (G), now containing all the hydrocarbon oils and unsaponifiable matter, shall be evaporated to dryness in a weighed beaker as described for (F) paragraph F-5qq, the residue weighed, and the result reported as "petroleum oils plus unsaponifiable matter."

An approximate determination of the viscosity of the petroleum oil may be made by using a 2 or 5-ml pipette which has been standardized against oils of known viscosities. If a complete characterization of the petroleum products is required, a new sample of from 150 to 200 g. of grease shall be decomposed as described above, except that all quantitative operations shall be omitted, as well as the isolation of the free fatty acids, and the use of standard alkali and acid is not necessary.

If the grease contains rosin oil, beeswax, degras, spermaceti, spermatin, montan wax, candle pitch, and other materials containing a large amount of unsaponifiable substances, the petroleum oils isolated from solution (G) will contain the unsaponi-

[22] A close approximation to the amount of glycerin present may be calculated and included in the analysis, by taking 11 per cent of the weight of the fatty acids from soaps, providing, of course, that the grease was made from neutral fats.

[23] Multiplying the weight of fatty acid by 1.045 gives a very close approximation to the weight of fat from which it was derived. This factor varies very little with the molecular weight of the fat.

fiable matter, and the constants found will differ from those of the petroleum products used in making the grease. In most cases, no further separation is possible except in the hands of skillful and experienced operators who can devise special methods to suit the individual conditions.

(2). Determination of Filler, Soap, Fat, Etc.

F-5qq (21). *Method for dark greascs.*—The sample shall be weighed in a 3-in. porcelain dish, 10 g. of granulated acid potassium sulphate added and 10 g. of clean dry ignited sand. The dish and contents shall be heated on the steam bath with frequent stirring until all water is driven off, 2 hours usually being sufficient. After cooling and breaking up lumps with a small pestle, the mixture shall be transferred quantitatively to an extraction thimble, using a little petroleum ether to wash the last traces into the thimble, which should be already placed in a Soxhlet apparatus. The thimble shall be extracted thoroughly with petroleum ether, the extract concentrated somewhat if necessary, and the free fatty acid and fatty acid from soap titrated with 0.5 N alcoholic potassium hydroxide, as in solutions (B) and (C) in method (*1*) paragraph F-5qq. From this point on, the procedure is identical with that given in method (*1*) paragraph F-5qq.

F-5qq (22). *Determination of asphalt and tarry matter.*—The thimble shall be extracted a second time with carbon disulphide, the extract exaporated to dryness, heated for 1 hour to 120° C. and weighed, the results being reported as asphaltic and tarry matter. The residue in the thimble shall be discarded.

(E) Determination of Free Alkali and Free Acid

F-5qq (23). From 10 to 30 grams of grease shall be weighed in a small beaker, dissolved as completely as possible in 75 ml of petroleum ether by stirring with a spatula, the mixture washed into a 250-ml Erlenmeyer flask with a small amount of petroleum ether, and the beaker rinsed with 50 ml of 50 per cent alcohol, the alcohol being poured into the flask, after which a few drops of phenolphthalein solution shall be added and the whole shaken vigorously. If the alcoholic layer, after settling for a few seconds, is pink, 10 ml of 0.5 N hydrochloric acid shall be added, the solutions boiled on a hot plate for 10 minutes to expel carbon dioxide, and the excess acid titrated back with 0.5 N alcoholic potassium hydroxide. The free alkalinity shall be calculated in terms of hydroxide of the predominating base.

F-5qq (24). If chalk or any other form of alkaline earth carbonate, or lead carbonate, is present as a filler, it will be detected by effervescence on adding the hydrochloric acid. As the amount of such carbonates is likely to be considerable, the volume of 0.5 N hydrochloric acid added shall be increased sufficiently to dissolve all carbonate and leave a slight excess of acid. The solution shall be boiled for 2 minutes and the excess acid titrated back with 0.5 N alcoholic potassium hydroxide, and from the acid consumed, its equivalent in calcium carbonate, etc., shall be calculated, disregarding any other forms of alkalinity which may have been present.

F-5qq (25). If the original alcoholic layer is not pink, the solution shall be titrated carefully in the cold with 0.5 N alcoholic potassium hydroxide, shaking well after each addition. The acidity shall be calculated as oleic acid.

F-5qq (26). If soaps of iron, zinc, aluminum, or other weak bases are present, a determination of free acid is not possible, since these metallic soaps react with potassium hydroxide. Up to the present no means has been devised whereby this determination can be made directly.

(F) Determination of Water

F-5qq (27). The quantity of water shall be determined in accordance with method for water in petroleum products and other bituminous materials (paragraph F-5q).

Estimation of Dirt [2]

The determination of dirt is of interest, particularly if the impurities are present in the form of black specks or pieces of black scale. Some of the

[2] McConville, H. A., "Practical Tests for Industrial Greases," 3rd Annual Convention, Lubrication and Grease Manufacturers Association (Oct. 14, 1935).

particles found are very hard and certainly undesirable to have present in a ball bearing grease. There is also the effect on the customer to consider, as the presence of black specks in a yellow grease is quite conspicuous and makes him question the quality of the grease.

A measured volume of grease equal to about 10 c.c. is transferred to a clean milk white titration plate. A sheet of cellophane is placed over the grease and plate, held in place by two or three rubber bands and the grease is worked out with a spatula to a uniform layer covering approximately 3 square inches. This is done readily by working through the cellophane.

A qualitative estimate of dirt or foreign material may be obtained by observing the sample with a hand magnifying glass. A quantitative measure may also be obtained by placing a mask with suitable opening, (use 1" square openings) over the sample and scanning the surface under a binocular microscope at $\times$ 12 magnification. The number of particles visible is recorded as the cleanness rating. Use the average of four or five 1" square surfaces and the count will check very well in any one grease sample. A count below 20 is good, from 20 to 40 fair, and over 40 is rated poor and objectionable for lubricating grease for ball bearing use.

Determination of dirt quantitatively by dissolving a weighed sample of grease in toluol and filtering through filter paper, using a steam funnel, is frequently resorted to. Brush the dirt thus collected onto a watch glass and weigh. More than a trace of dirt may be cause for rejection.

Examination of the Mixed Fatty Acids

The various methods for examining and identifying the mixed fatty acids obtained in the foregoing grease analysis have been briefly described and discussed in Chapter II.

Oxidation Tests

Both the soap stock, and mineral lubricating oils used in greases are subject to more or less oxidation. The extent of oxidation will depend on the presence of impurities, which may act as oxidation catalysts, the nature of the soap itself, which may act as a catalyst, and the degree of unsaturation of the grease making materials. Two general methods of carrying out tests for determining the resistance of grease to oxidation are in use. There are large number of tests in which the lubricant is heated in an oven, under carefully controlled temperature conditions, for specified periods of time and the change in penetration, viscosity, texture, or other characteristics, measured. Other methods have been used for measuring the rate of oxygen absorption, while maintaining a sample of the grease under specified air or oxygen pressure, at controlled temperatures, in a suitable bomb.

Fluid Grease Sludging Test

One liter of the lubricant is placed in an iron vessel immersed in an oil bath heated by electrical resistance coils and maintained at 250° F.

(plus or minus 2° F.) for eight days (192 hours). The sample of lubricant is stirred by means of a small propeller, driven by an electric motor, throughout the oxidation period.

After oxidation the lubricant is thoroughly mixed and cooled to room temperature and a 100 ml. sample centrifuged without dilution with benzol, but otherwise in accordance with the requirements of the A.S.T.M. method D96-30. A comparison of the amount of sediment as determined in the original sample with that found in the oxidized grease is an indication of the instability and formation of insoluble oxidized products in the lubricant. The presence of various soaps, of course, greatly influence the results.

The results of making the following tests on the original sample and the oxidized lubricant are important indications of the extent of oxidation:

Organic Acidity
Saybolt Viscosity at 210° F.
Corrosion Tests
Timken, S.A.E. or other film strength tests
MacMichael Viscosity
Pressure Viscosity Tests

Bomb Method

The bomb used in this test is quite similar to that utilized for studying gum formation in gasolines. The samples, so distributed on small glass dishes as to present a total free area of ten square inches, are placed in the bomb on a small stainless steel rack.

To flush air from the bomb, it is five times alternately filled with dry oxygen and opened to the air. Typical conditions for a bomb test are:

Initial oxygen pressure, 110 pounds per square inch.
Bomb is immersed in constant temperature bath at 175° F.
A small steel strip may be included with the grease.

After ten to four hundred hours, pressure drops of from two to eighty-five pounds will be noticed. There is usually a period of slow oxidation, termed the induction period, after which the absorption of oxygen is quite rapid. When all of the material in the grease which rapidly absorbs oxygen has been oxidized the rate of oxygen again assumes a low value. Results of tests of this kind indicate that the fats and soap making materials and compounds produced during saponification are more readily combined with the oxygen than the relatively inert refined mineral oils.

McConville Oven Test

Examination of oxidized greases shows that oxidation in all cases is accompanied by an increase in acidity and sometimes by a drop in the flow point of the grease. With this as a basis an accelerated oxidation test has been developed as follows:

A drying oven about 18 inches square was fitted up for the introduction of moisture, oxygen and light and the results of various combinations of heat, light and oxygen and moisture were tried. The oxygen was bubbled through sulfuric acid at a rate of about 120 bubbles a minute to absorb the

moisture. When moisture was to be purposely introduced into the oven the oxygen was bubbled through water in the interior of the oven. A thin layer of grease was exposed on watch glasses about 5 inches in diameter for periods of one week. Heat alone at 180° F. gave very little change. Heat and oxygen produced a small increase in acidity but no change in flow point. Heat, light, and oxygen gave a marked increase in acidity and lowering of flow point. The introduction of moisture somewhat lessened the rate of oxidation.

A comparison of results obtained on one grease is given:

	Flow Point ° C.	Acidity
Untreated grease (original)	148	.035 per cent NaOH
Heat alone (1 week at 180° F.)	142	.035 per cent NaOH
Oxygen and heat (1 week at 180° F.)	147	.42 per cent (Oleic)
Oxygen and light (60 watts) heat (1 week at 155° F.)	132	2.54 per cent (Oleic)
Oxygen and light (100 watts) heat (1 week at 155° F.)	115	2.85 per cent (Oleic)
Oxygen and light (200 watts) heat (1 week at 160° F.)	115	3.41 per cent (Oleic)
Oxygen through water in oven, light (200 watts) heat (1 week at 165° F.)	130	3.10 per cent (Oleic)
Grease in motor in storage 4 years	83	5.19 per cent (Oleic)

It will be noted that one week's time was not sufficient to duplicate the oxidation in storage over a period of 4 years. Oxidation is not always accompanied by darkening of color as some greases increase in acidity without any color change.

A modification of the foregoing test is as follows:

The grease is packed in a No. 203 Conrad type single row all steel ball bearing after thoroughly cleaning the bearing in carbon tetrachloride so that it turned absolutely free. The bearing is packed level with the sides with grease, the ease of turning noted, then the increased resistance to turning after oxidation, as well as could be determined by rotating the bearings by hand. This gives similar results to those obtained by the bearing companies, who pack their bearings with grease, store them for 18 months and then note the increased resistance to turning. But it does not give an accurate means of measuring the change that takes place.

McConville's apparatus consists of two No. 203 bearings, as mentioned above, mounted about six inches apart on the two ends of a steel shaft. These bearings are held on by a washer and lock nut and should slide on the shaft quite easily, depending on the nut and washer to keep the center of the bearing from turning. The shaft and bearings are mounted in a special fixture made of steel about $\frac{1}{2}$ inch thick. It has a bottom piece of steel about 6 inches long, 2 inches wide and $\frac{1}{2}$ inch thick, to which have been welded two arms about 2 inches wide extending upward on a curve resembling a parabola to a height of about 6 inches. The bearings on the shaft fit into holes cut in the side arms. In the center of the shaft a pullley $1\frac{1}{2}$ inches in diameter is mounted and to this pulley is attached a piece of flexible stranded copper wire $\frac{1}{8}$ inch in diameter and about 6 inches long fastened to an aluminum pan on which weights can be placed. The side arms were designed as

described above originally so that the apparatus could be fastened to a bench leaving the weighing pan some distance to travel to the floor, if necessary. This apparatus has now been adapted to conducting tests on the behavior of greases at sub-zero temperatures, by mounting in a suitable box. Using the apparatus for its original purpose of measuring degree of oxidation the box, of course, is not necessary. The resistance to turning by loading the pan with weights until the shaft just begins to turn is measured. The procedure used is to clean the desired number of No. 203 bearings thoroughly, pack 2 bearings with each kind of grease to be tested, pressing the grease in well around the balls with the small, flat blade of a penknife, rotating the bearing 4 or 5 times by hand, pressing in the grease again and then filling the bearing level with the sides with grease. Each pair of bearings is then placed in the testing apparatus and the number of grams to cause initial turning of the shaft is noted. Each bearing is then placed on a watch glass 2 inches in diameter and the bearings then placed as close together as possible on a tray ready for the oxidation oven.

The oven used for oxidation, is about 18 inches square and has a De Khotinsky temperature control mounted on the side. In the top of the oven is a hole 1½ inches in diameter through which is inserted a part of the socket for the electric light bulb. The action of light speeds up the reaction to a considerable extent. This is borne out by observation that some greases packed in glass containers oxidize badly when only exposed to the sunlight, and the same is true of oils, this tendency being overcome when dark glass containers are used. At the top of the oven above the light bulb there is a large flat standard light reflector, such as is in common use, to throw the light down on the bearings. An ordinary 100 watt white frosted bulb is used for the light. The use of a sodium vapor lamp has been considered for giving quicker results, but as the apparatus for using it is complicated, costly, and is not readily obtainable by everyone, its use was not warranted even if it might save time. The bearings on the tray are placed under the light about 6 inches away from the bulb, a thermometer is inserted in the oven and with the light on, the temperature in the oven is raised to 175° F. This temperature was selected because it is quite often reached by motors in service and most sodium and sodium-calcium ball bearing greases are recommended for and should stand a continuous temperature of 175° F. Oxygen is bubbled through a bottle containing water at the rate of about 2 bubbles a second from a ¼ inch diameter tube and introduced into the oven. This adds a small amount of moisture, the presence of which causes some greases to become sticky; a condition also found in service. The bearings are thus subjected to the combined action of heat, light, oxygen, and moisture for a period of two weeks. They are then removed from the oven, cooled to room temperature, and again placed in the testing apparatus where the number of grams weight required to turn the shaft is noted. One or two greases have been found with practically no change taking place after this treatment, but in the majority of cases there is a decided increase in resistance to turning; in some extremes the ratio of weights being 10 to 1 after oxidation. These tests should be conducted both

on an original sample of the grease as received and on a sample after working thoroughly in a motor at a temperature not high enough to change the structure of the grease. A check of these two conditions is desirable because quite often in the manufacture of motors, when they are packed with grease in the production line, they may have only a short test run, and then under extreme conditions might be placed in storage for some time before being used. The grease is thus in practically the same as the original unworked condition. The other case is where the grease has become worked in the motor and changes with use. This change may be caused by oxidation or may be due to some other cause. As a result of these two checks an estimate of the stability of a grease in storage and in use can be predicted.

At this point it might be well to mention the storage stability test as developed by *Norma-Hoffman Bearings, Inc.* This method determines the suitability of greases by measuring the oxygen absorption of the grease. A sample of grease weighing 13 to 15 grams is spread in a thin layer on several trays of either glass or some metal such as used in ball bearings. Those trays are placed in a steel bomb fitted with an accurate pressure gauge.

PHYSICAL LABORATORY TESTS

Control Tests

Penetration Tests for Consistency

The following is procedure as specified by The American Society For Testing Materials:

F-5e. METHOD 31.11—PENETRATION OF GREASES AND PETROLATUM. [A.S.T.M. D217-33T]

F-5e (1). This method shall be used to measure the original consistency or the worked consistency of no. 0 cup grease and all harder greases, and of petrolatum (unworked).

F-5e (2). 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.

APPARATUS

F-5e (3). The consistency shall be determined by measuring the penetration on what is customarily known as an asphalt penetrometer, such as that described in the Proceedings of the American Society for Testing Materials (vol. VII, p. 626, 1907), and illustrated in Plate XXV. It is desirable, however, to modify this penetrometer by placing a flat metal plate or transite board on top of the present grid base to give a flat support for the container. If many tests are to be made, it is preferable to replace the ordinary grid base with a special flat base 6 in. in diameter.

This plate should have a cork insert in the center to prevent dulling the tip of the cone if it should drop.

F-5e (4). The needle used in the asphalt penetration tests shall be replaced with the cone shown in Plate XXV. The cone 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

[5] The cone may be combined with the plunger if desired, providing the total weight of cone and plunger and the outside dimensions of the cone are not thereby changed.

dimensions without tolerances are intended to be suggestive only. The outside surface of the cone and tip shall be given a very smooth finish.

F-5e (5). The total moving weight in the test shall be 150 g. If the asphalt plunger weighs 47.5g (50g—2.5g needle) the cone, or the cone plus an attached weight, must weigh 102.5g. It is in general desirable 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

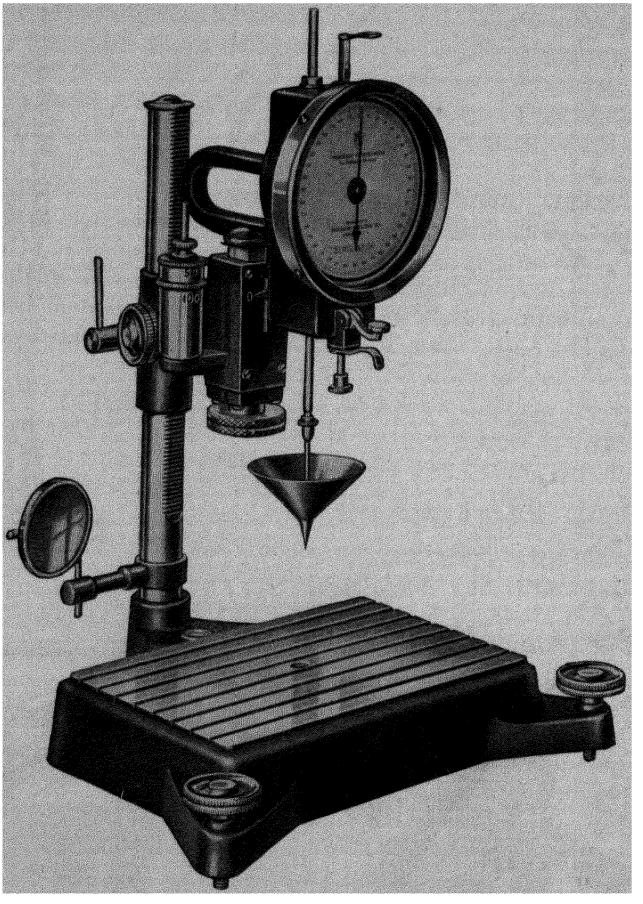


PLATE XXV.—Lubricating Grease Penetrometer
(Precision Type)

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° F. (25° C.).

F-5e (6). The grease worker shown in Plate XXVII shall be used to work the grease to constant consistency in tests where the worked consistency is to be measured.

F-5e (7). 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.

SAMPLES

F-5e (8). 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

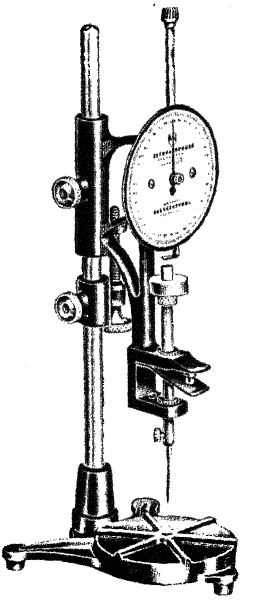
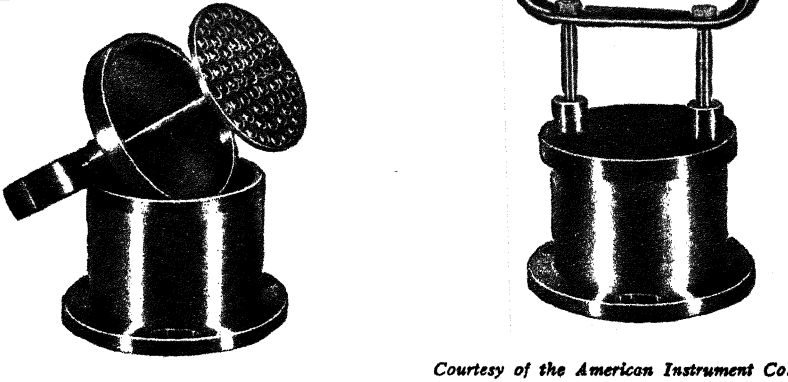


PLATE XXVI.—Grease Penetrometer (Miniature type suitable for routine control work; shown with needle, cone to be used for grease testing)

Courtesy of the Precision Scientific Co.



Courtesy of the American Instrument Co.

PLATE XXVII.—A.S.T.M. Grease Worker

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 petrolatum shall be tested for original (unworked) consistency after melting and cooling to the temperature of test.

PROCEDURE

(A) Original (Unworked) Consistency

F-5e (9). 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.

F-5e (10). 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.

F-5e (11). 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 5 seconds. The penetration is read from the scale, the units being tenths of a millimeter.

F-5e (12). 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 the 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.

F-5e (13). 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 per cent. If the mean deviation does exceed 3 per cent, the average of 10 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

F-5e (14). 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.[6] 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.

Operation of the "Precision" Junior Penetrometer

The following notes applicable to the use of the "Precision" Junior Penetrometer will be found of interest in addition to the procedure as specified by the A.S.T.M.[8]

Level the instrument carefully before using. After this is done, avoid shifting the location of the Penetrometer, otherwise it will be necessary to re-level it.

Insert penetrating instrument into chuck and tighten securely.

Set to zero. Pull up the depth gauge as high as it will go. If the dial needle does not stop exactly at zero, loosen the needle lock-nut and reset the needle to zero. Be sure to tighten the needle lock-nut very securely.

[6] In general quite similar results may be obtained by working the grease thoroughly for 5 minutes with a spatula on a flat plate, provided care is taken to prevent working in much air. For referee tests, however, the standard worker must be used.

[8] M.B.K.—TS 3518. Published by the Precision Scientific Company, Chicago (Jan., 1935).

Add necessary weights to test rod. In adding weights to make up the required test load, note that the weight of the test rod itself is 47.5 grams. When the specified load for grease penetrations with a grease cone, for example, is 150 grams, no weights need be added, since the combined weight of the cone (102.5 grams) and test rod (47.5 grams) makes up the required load as specified by A.S.T.M.

Place prepared sample in position on base.

Adjust height of mechanism head to bring the point of the penetrating instrument exactly into contact with the surface of the sample. This may easily be done by contacting the point with its image as reflected from the surface of the sample. To intensify the image, use a mirror so as to throw a beam of light across the center of the sample. First make a coarse adjustment, then a micrometer adjustment as follows:

Coarse Adjustment. Grasp the mechanism bracket, and loosen both the upper lock screw and lower lock screw. Now move the entire mechanism head up or down to the approximate position for the test; then tighten the lower lock screw.

Micrometer Adjustment. With the upper lock screw loosened, make the final contact adjustment by means of the micrometer adjusting screw. When this is done, be sure to tighten the upper lock screw.

Release test rod, allowing penetrating instrument to descend into the sample. To do this, release the clutch spring and hold in its released position during the specified time required for the test, using a stop watch for noting the time.

Observe the dial reading. To read the depth of penetration, push down the depth gauge—gently—as far as it will go. The dial reading now indicates the depth of penetration directly in tenths of millimeters. For example, if the pointer comes to a rest at the fourth mark past the 270 point, the depth of penetration is 274 tenth-millimeters, or 27.4 millimeters.

With the "Precision" Junior Penetrometer, penetration measurements can be made to a total depth of 39 mm. on a single reading. On depths greater than 38 mm., the dial needle makes more than a complete revolution.

Return dial needle to zero, as directed above. If the original zero setting has been accurate, the dial pointer will return exactly to zero, and subsequent readings will check against standard gauge blocks.

H. V. Schroeter has found from work with the Kissling and A.S.T.M. methods for determining the consistency of greases that the A.S.T.M. method was not satisfactory. Penetration tests on numerous samples and under varying conditions made with a new needle designed to overcome the objections to the A.S.T.M. apparatus checked with results obtained from the Kissling method. Softness was determined by the penetration depth expressed in mm. Drop-point and flow-point determinations were not sufficient to fully characterize consistency. Penetration tests at 20° and 50° C. are also necessary. The ring and ball method used with asphalt was found to be inapplicable to greases. It was concluded from work done on the measurement of greases under pressure in capillary tubes that it should be possible to obtain from these data an absolute measure of the physical values which determine the consistency properties of greases. It was also established that the Hagen-Poiseuille law does not hold for greases. A relationship seemed to be apparent between consistency measurements obtained by penetration methods and two special constants calculated from flow investigations. The relationship developed by Schroeter must be clarified, however, by further experimental data.

B.E.C. Penetration Test

The Bearing Engineers Committee has developed a penetration test applicable to determining the penetration of small samples of grease taken from a bearing. The B.E.C. tester and procedure is in general principle similar to the A.S.T.M. apparatus and method, but tests may be made on

one cubic inch of greases rather than approximately one pound as in the case of the A.S.T.M. procedure. It appears possible that a conversion factor for converting the results obtained with the B.E.C. tester to terms of the A.S.T.M. is quite likely, and the committee is working along this line.

[4] The procedure for using this B.E.C. Penetrometer to determine the difference in penetration between an unworked sample of grease and one which has been subjected to test in the B.E.C. grease tester is as follows:

Fill specimen holder with sample of unworked grease, using utmost care to avoid air pockets.
Level off with care so that grease is flush with faces of specimen holder.
Adjust cone so that when indicator points to zero, tip of cone will just touch the top face of the specimen holder.
Swing head into position, making sure that the head is tight against stop, lock in position.
Release pressure, and allow to penetrate for five seconds.
Read depth of penetration in hundredths of millimeters.
Repeat this procedure with a grease worked in B.E.C. grease tester.
Calculate percentage increase or decrease in penetrations.

Note: Do not allow grease to go over top of cone as this will give false readings.

KARNS-MAAG CONSISTENCY

The relationship between the results of Karns-Maag consistencies and A.S.T.M. unworked penetrations is shown in the following table for one series of cup greases. Since the details of making the Karns-Maag test are so much different than the use of the A.S.T.M. cone, it is not surprising that a correlation, as listed below, will not apply to all manner of lubricating greases.

Karns-Maag Consistency at 75° F.	A.S.T.M. Penetration at 77° F.	
	Unworked	Worked
10	150	184
12	166	202
14	190	220
16	204	232
18	214	244
20	224	255
25	246	...
30	262	...
35	280	...
40	300	...
45	315	...

Mechanical Stability Tests

The manufacturers of anti-friction bearings have long felt the need for a device for determining the service characteristics and in particular the stability of lubricants. Some greases were found to dry out and harden in service resulting in serious damage to anti-friction bearings. In other instances, the oil and soap would separate, the soap deposit caking on the bearing races. Experiments have shown that some anti-friction bearing greases would increase in volume in the bearing and be forced therefrom. In some cases electric motor bearings after assembly and storage would not

[4] Ahlberg, C. R., 4th Annual Convention, National Association of Lubrication and Grease Manufacturers (Oct. 12, 1936).

operate freely when delivered to a customer. It was found that this difficulty was due to oxidation processes which promoted the deposition of resinous material on the bearing surfaces. The Bearing Engineers Committee pooled their data and developed a service testing apparatus known as the B.E.C. Grease Testing Machine. In principle this machine works a specific quantity of lubricating grease in a medium size ball bearing operating at normal speed and at different test temperatures. The machine is also capable of yielding data relative to the following important items:

Starting and running torque.
Oil separation (soap deposits).
Change in grease structure and texture.
Change in consistency.
Channeling of the lubricant in the bearing.
Leakage of lubricant past the bearing seal.
Aeration and resulting increase in volume while in service.

B.E.C. Mechanical Stability Testing Machine

Gillette [5] has described the B.E.C. machine and the procedure for making tests, as follows:

"The machine consists of a base, a vertical column, a constant speed 3450 R.P.M. motor and an extension to the motor spindle, upon which is mounted a plate shielded, Conrad Type, size 204 S.A.E. bearing. This bearing is mounted in a grease cup which is free to turn with the bearing. A flexible cord connecting this cup with the torque scale spring, provides a means for determining the torque. The grease cup is surrounded by an oil bath which is a counterweighted, adjustable container in which the oil is maintained at the various test temperatures. The oil temperature is recorded by the dial type thermometer.

"The test procedure consists of mounting the bearing upon the shaft with the shield side down and packing the test quantity of grease into the bearing and around the shaft. The grease cup is then assembled on the bearing and the grease in the cup leveled off.

"The oil bath is raised to the stop on the column, with the damping vane on the grease cup in front.

"The flexible cord from the torque spring is attached to the cup by means of the slot provided.

"For the start of the initial test, the oil temperature is adjusted to 80° to 85° F. The motor is started and the starting torque is observed. The running torque is recorded at intervals throughout the test. The test is continued for twenty minutes, at which time the torque approaches a constant.

"With the bearing still running, the temperature of the oil bath is raised to 150° F. at the rate of 10° to 12° F. per minute and another 20-minute test is conducted.

"With soda soap and mixed base greases the temperature of the oil bath is again raised, this time to 220° F., where another 20-minute test is made.

"During all of these tests, separation of oil and soap, change in structure, change in consistency and aeration are noted, in addition to the torque readings.

"If desired, the machine may be stopped after each 20-minute run to determine the extent of grease leakage past the bearing shield, at each test temperature.

"The interpretation of results can best be explained by first stating the physical characteristics desired in an ideal ball bearing grease. Such a grease should have a low torque, should not change in structure nor consistency, should not increase in volume due to aeration, and should not leak past the bearing closure. The grease should maintain these characteristics in operation, from room temperature to 220° F. Obviously, the more nearly a grease approaches these conditions, the more suitable it becomes for general ball bearing lubrication. On the other hand, the fact that a grease does not meet all of these requirements does not eliminate it as ball bearing lubricant. It simply limits its usefulness to certain definite applications. For instance,

[5] Gillette, C. R., 4th Annual Convention, National Association of Lubrication and Grease Manufacturers (Oct. 12, 1936).

high torque is always objectionable but is sometimes tolerated where power consumption is not a factor. A grease which changes from a smooth to a fibrous structure in operation is limited to a temperature below which this change takes place because except for low speeds, fibrous greases will not remain in a ball bearing but are immediately thrown out. A decrease in consistency might be objectionable from the standpoint of leakage past the bearing closures or of increased fluid friction which would be serious in high speed bearings. Aeration of the grease with its resulting increase in volume would be objectionable insofar as it affected grease leakage.

"Thus, these results obtained with the B.E.C. Test Machine, indicating the mechanical stability, when considered in conjunction with the physical and chemical stability of the original grease, enable us to make a fair estimation of the results to be expected under service conditions."

Melting Point Determinations

A knowledge of the melting point characteristics of a cup grease is of value to the user of lubricants in selecting greases for any type of lubrication, but particularly when choosing greases for the lubrication of bearings operating at from 100° to 210° F. It is obvious that the method of applying the grease to the bearing is also of importance. Such methods as compression cups, constant pressure compression cups, pressure lubricating systems, gravity or pin cups, swabbing, and packing are all employed for bringing the grease lubricant in contact with the surfaces to be lubricated. The melting point of the grease, its consistency at operating temperature, and the method of applying are the most important factors in determining the rate of consumption of a grease in a given bearing. It should be understood that cup greases do not have a definite melting point, but gradually soften as the temperature is raised. This may be clearly demonstrated by determining the consistency of a grease by the A.S.T.M. method at various temperatures up to 200° F. The author in examining cup greases by this method has found that they vary considerably in softening under heat. Most cup greases soften gradually as the temperature is raised until they become so soft or semifluid that the consistency cannot be determined. In this melted condition the grease is a viscous liquid rather than a plastic solid. Other cup greases have been found to harden on heating due to the slight evaporation of water causing the grease gel to become more firm. This property depends on such factors as the ratio of lime to fat in the grease, the amount of water, and the viscosity and origin of the mineral oil in the grease.

There are many methods of determining the melting point of greases, a search of the literature on the subject showing that many very ingenious methods have been devised. In general it may be said that there is no accepted method for determining the melting point of a grease, although most manufacturers have adopted a method for their own products. A method described by Gillette [5a] is interesting; and while in itself the test means but little, however, when made in conjunction with a complete analysis of a grease it is of value in checking up a manufacturer's product as to uniformity for successive lots of grease of the same grade. It also indicates to some extent the temperature at which the grease will function. The method as outlined by Gillette makes use of an open capillary tube,

[5a] Ind. Eng. Chem., 1, 351 (1909).

4 mm. inside diameter and about 8 cm. long, graduated at 1 cm. and 5 cm. from one end. In making this test the tube is inserted in the grease, and a plug of grease is drawn up 1 cm. into the tube, using suction if necessary to draw up this plug. The tube is then attached, with a rubber band, to a thermometer so as to bring the grease plug beside the bulb. The thermometer and tube are then immersed in a beaker of water, so as to bring the bottom of the tube 5 cm. below the surface of the water. The water is then heated at the rate of 3° per minute, and when the melting point is reached, the plug, which is under pressure of 5 cm. head of water, flies up the tube. Of course the test is dependent on a number of factors and is of value chiefly as an indicator. The nature and amount of soap, oil and water in the grease as well as the process used in manufacturing the grease, affect the melting point.

Archbutt and Deeley [5b] give several methods of determining the melting point of greases. These may be listed as follows:

Pohl's Method: By this method the point of incipient fusion at which a substance begins to melt is determined by coating the bulb of the thermometer with the melted grease, allowing it to solidify, then placing the thermometer inside of a test tube. The apparatus is supported over a Bunsen flame which plays on an asbestos disk. The temperature at which the melted grease collects at the bottom of the thermometer bulb is taken as the melting point. This method is not adaptable to cup greases since they cannot be melted for coating the bulb without altering their true melting point.

Stock's Method: The grease is placed in a small test tube which just slips over the bulb of the thermometer which is supported in a larger test tube and mounted over a Bunsen flame. The temperature at which the grease flows down the sides to the bottom of the small test tube is the melting point.

The following method is used by many of the manufacturers of grease in this country and is particularly useful for hard high melting point greases: A small cube of grease about $\frac{1}{16}$ of an inch square is placed on the surface of mercury in a 25 c.c. porcelain crucible. The crucible is heated on an electric hot plate, and the temperature at which the cube begins to melt down is noted on a thermometer the bulb of which is in the mercury.

Another method which is often used, and which after the operator has become thoroughly experienced gives very good results, is to place a very small bit of the grease on the side of the bulb of an accurate thermometer. The thermometer is supported in a 1 inch test tube by means of a stopper. The tube is then placed in a liter beaker of water, glycerin or water-white mineral oil, depending on the melting point to be taken. The temperature is then raised at the rate of 2° to 3° F. per minute by means of a Bunsen flame. If the bit of grease has been drawn out to a fine point it is possible to note a fairly definite melting point when this point melts down or draws in.

One manufacturer determines the melting point by coating the thermom-

[5b] "Lubrication and Lubricants," 224.

eter with the grease to be tested and then suspending it freely in an empty test tube which is immersed in a light oil. This oil bath is then gradually heated at rate of 5° F. per minute and the temperature observed. When a drop of grease forms on the end of the thermometer and falls to the bottom of the tube, the temperature is noted as the melting point of the grease.

NOTES ON COMPARISON OF FOUR DROPPING POINT METHODS

Ubbelohde. Consists in observing the temperature during heating, where the first drop separates from the grease which is supported around the bulb of the thermometer. The sample is placed in a glass cup provided with an orifice 3 mm. in diameter. When within 20° C. of the melting point the heating rate is 1° C. per minute.

Cube Method. This is the temperature during heating at which small portions of grease melt. These particles of grease are supported on cover glasses which are floated on the surface of mercury.

Gillette. Consists in observing the temperature at which a plug of grease held in a capillary tube, flies up the tube when heating same in a water bath.

Southern Pacific Company. This is the temperature at which grease, when heated, slides out of a capillary tube or the first drop breaks away from the tube.

Results. Shows dropping or melting temperatures obtained by various methods on two wheel bearing greases:

	Wheel Bearing Grease A			Wheel Bearing Grease B		
Method	1st Test	2nd Test	Average	1st Test	2nd Test	Average
Ubbelohde	177° C.	190° C.	184° C.	199° C.	195° C.	197° C.
Cube	180	180	180	199	204	202
Gillette	68	81	75	100+	100+	100+
S.P. Co.	202	200	201	174	182	178

It is of note that the results obtained by the Ubbelohde and Cube method check fairly closely for the high melting point wheel bearing greases shown in the above tabulation. It is considered, however, that the Ubbelohde method is to be preferred, inasmuch as it gives more information as to adhesiveness, and possible separation of oil from the grease.

NOTES ON COMPARISON OF FOUR OTHER MELTING POINT METHODS

The following data, obtained from more than fifteen different laboratories, clearly indicates the need for further standardization and study of dropping point methods applicable to lubricating greases. Method I, makes use of a modified form of Thiele tube; Method II, is the cube method in which a bit of grease is supported in a mercury bath; Method III, is the one in which the grease is held to the thermometer bulb by means of a small spiral of copper wire; and Method IV, is a modified form of Ubbelohde test in which the cup is made of brass to minimize breakage which frequently occurs with the normal glass cup. All of the results are reported in terms of ° F.

SAMPLES OF LUBRICATING GREASES

Method I—Modified Thiele Tube

Laboratory No.	A	B	C	D	E	F	G
1	269	307	465	288	206	209	104
2	282	235	Above 370	276	207	212	120
3	274	232	449	273	203	207	103
4	359	291	470	310	215	217	137
5	351	287	400+	372	211	208	122
6	298	239	400+	272	206	210	129
7	354	309	457	284	194	212	113
8	319	317	434	269	195	197	109
9	306	...	...	270	199	206	118
10	353	303	400+	273	204	211	98
11	260	290	448	284	203	205	115
12	306	314	400+	279	206	211	136
13	352	349	459	288	209	217	134
14	291	284	400	270	208	208	125
15	318	312	447	278	195	194	92
Average	313	284	448	286	204	208	117

Method II—Mercury Bath

1	339	330	482	275	206	210	100
2	328	287	Above 430	251	202	210	Too Soft
3	270	258	460	260	189	220	84
4	345	320	469	289	213	212	141
5	369	370	400+	303	220	223	Too Soft
6	238	258	...	320	224	250	120
7	369	324	477	270	221	223	Too Soft
8	347	346	464	275	213	210	129
9	352	311	483	295	206	239	136
10	Not used as believed hazardous to health						
11	340	340	463	272	202	208	110
12	309	304	400+	270	202	209	118
13	360	363	463	268	211	217	Too Soft
14	355	340	465	305	208	218	102
Average	332	320	469	281	209	218	116

Method III—Spiral Copper Wire

1	310	295	451	265	196	198	104
2	334	324	Above 370	272	204	205	116
3	340	240	426	282	204	209	103
4	350	337	465	286	213	212	139
5	311	292	410	270	198	195	100
6	339	334	400+	270	205	203	130
7	340	333	462	271	199	203	120
8	333	323	444	274	201	203	117
9	340	323	434	284	205	203	118
10	327	309	425	257	200	199	98
11	340	340	451	280	200	203	119
12	325	311	430	271	201	201	109
13	324	316	431	288	206	209	123
14	335	340	453	283	202	205	120
15	340	326	449	285	204	201	105
16	310	300	442	280	202	200	110
17	314	307	422	223	199	198	109
18	345	340	465	305	208	218	102
Average	331	316	441	275	203	204	113

SAMPLES OF LUBRICATING GREASES—(*Continued*)

Method IV—Modified Ubbelohde

Laboratory No.	A	B	C	D	E	F	G
1	340	335	408	247	211	209	121
2	331	317	Above 410	216	208	206	111
3	340	325	459	239	212	208	126
4	328	316	Above 392	230	205	207	110
5	334	299	Above 392	235	200	203	118
Average	333	314	...	230	206	206	116

THE SCHRODER SOFTENING POINT METHOD [6]

The increasing use of loaded greases as lubricants for machinery involving high temperatures and high pressures, has made it imperative to know definitely at what temperature a grease will soften or "flow" and begin to function as a lubricant.

In some recent work with one of these greases, it was impossible to obtain check results with any of the softening point or melting point apparatus on the market today. This particular grease, in order to function properly, required close control of its properties. Therefore the following apparatus was developed by our technical service department, to determine its "flow point."

By "flow point," we mean that temperature at which the grease changes from a solid to a more or less plastic mass and begins to function as a lubricant.

THE NEW METHOD AND APPARATUS

To make a determination, the grease is rolled into a thin sheet about ¼ inch thick. The end of a thin walled glass tube, ¼ inch inside diameter, is then pressed into the grease, forcing a pellet of the grease into the end of the tube. Most greases can be loaded into this "softening tube" in this manner, but with high softening point greases, the glass tube must be heated slightly before pressing into the grease. Two inches of mercury are then placed on top of the grease and the loaded tube is then mounted in the inner chamber of the apparatus along with the thermometer, both supported by means of the two-hole rubber stopper.

The jacket of the apparatus is then filled with sulphuric acid, glycerin or other bath liquid, and mounted over a gas burner or electric hot plate by means of a laboratory apparatus support. The bath thermometer is then placed in the side tubulature, projecting through a rubber stopper into the bath.

The jacket of the apparatus is novel in that the side arm facilitates thorough circulation of the heating liquid. The jacket has a small vent hole at the top.

As the entire bath is protected from the air, there is an absence of fumes even when high temperatures are employed. Glycerin can therefore be used for much longer periods of time in this apparatus than is the case when used in the ordinary melting point tubes.

The rate of applying heat can be regulated by means of the auxiliary thermometer in the jacket. One degree C. per minute has been found satisfactory, although 2 or even 3° C. per minute can be used in many cases. Where greases have been loaded with materials of high specific heat and low thermal conductivity, the rate of heat application should be slow, or a marked thermal differential will exist between the sample and the jacket and so give erroneous results.

When the flow point is reached, the grease suddenly softens, and under the weight of the mercury, the grease and mercury drop from the loaded sample tube. The tem-

[6] Reprinted from: "The Laboratory," Fisher Scientific Co.

perature read on the thermometer beside the sample (in the air chamber) is taken as the "flow point" of the grease.

To prevent the molten grease from adhering to the inner tube, a small metal cup is mounted below the softening tube. After each determination, this cup can be replaced by a clean one and the apparatus is ready for a new test.

The Schroder Softening-Point Apparatus is made of Pyrex glass and costs seven dollars and fifty cents, (1937) without thermometers.

Other Uses of the Apparatus

While this apparatus has been designed especially for use with loaded greases, it has worked very satisfactorily as a softening point and melting point apparatus. Pitches, vaselines and similar materials have given results capable of being checked very closely. For the determination of melting points of organic compounds, the glass sample tube has been replaced by the customary narrow bore melting point tube, with very good results.

B.E.C. Melting Point Determination (Modified Ubbelohde Method)

While the A.S.T.M. penetration or consistency tests may not meet all of the B.E.C. requirements it is at least standardized so as to indicate the consistency of the greases tested, but as to the melting point determination there seems to be considerable confusion, for many of the grease laboratories use their own methods for reporting melting or dropping point. Since this confusion can be eliminated by the standardization and the use of the *Modified Ubbelohde Melting Point Apparatus* which the B.E.C. has adopted, details of this method are presented. This method is used with considerable success in many of the European countries. If this procedure is adopted officially by the N.A.L.G.M. for checking this property of grease it would make it also an international method.

Scope. This method of test covers the determination of the melting point of greases, petrolatums, and similar petroleum products.

I. Apparatus

The apparatus shall consist of the following:

A standard A.S.T.M. Pensky-Martens high flash thermometer (200° F. to 700° F.).

A 0 to 150° C. thermometer calibrated in 1° dimensions. (A thermometer of the appropriate range calibrated in ° F. can, if desired, be substituted for this thermometer.)

A glass tip is employed for holding the grease to be tested. A brass tip is used in combination with the 200 to 700° F. thermometer, and it may also be used with the 0 to 150° C. thermometer.

Test tubes, beakers, etc.

II. Procedure

Press some of the material to be tested into the brass tip, taking care to avoid the inclusion of air bubbles. For greases having an anticipated melting point of 300° F. or over, use the 200 to 700° F. thermometer, and for greases having an anticipated melting point of less than 300° F. use the low-range thermometer. Push the brass tip onto the lower end of the thermometer, and secure the tip in place with the set screw. The excess material should be smoothed off the end of the tip. The apparatus is then suspended by means of a cork in a 4 by 18 cm. test tube so that the end of the thermometer bulb extends to within 1 in. + 0.1 in. of the bottom of the test tube. The test tube should be suspended vertically in a transparent oil bath. The oil should be heated at a rate such that the thermometer will show a rise of 8 to 10° F. per minute until a point is reached approximately 20° F. below the anticipated melting point of the material. At this point, the heating rate should be decreased to 2 to 4° F. per minute.

The melting point is the temperature at which the first drop of material falls to the bottom of the test tube.

Adhesion Tests

Knopf [7] has described his Adherometer and test procedure as follows:

"The adherometer is a device designed to simulate the sliding of a transmission gear on its shaft, except that in the instrument the shaft moves and the bore remains stationary, the relative motion being at about the same velocity as that of the sliding gear when hard shifting is experienced. The device is described as follows:

"A steel cylinder with a lapped bore of 0.628 inch is provided with two thermometer wells, which also act as air ducts in connection with vent passages to allow air to escape from the lower end of the cylinder as the bullet is being pushed down by the spike plunger, which is impelled by the same mechanism as in the spike test. The adherometer cylinder is thermally insulated by an air space between it and a brass jacket (2 inches in diameter), and by cork and fiber disks at the bottom. Six pins hold the cylinder concentrically in its jacket and are rounded so as to cut heat conduction to a minimum.

"The lubricating pin having a milled groove to hold the test lubricant is used to apply the lubricant to the bore of the cylinder. The bullet is 0.005 inch smaller in diameter than the cylinder bore, leaving a 0.0025 inch clearance on each side. The sizing plug is 0.002 inch smaller than the bullet and like the bullet has spherical ends. The body of the lubricating pin is 0.002 inch smaller than the plug. A slight excess of lubricant is deposited by the pin on the cylinder wall, but after the pin is withdrawn and the plug passed through the bore, only 0.001 inch of excess film is left on its walls. A magnet is employed for light lubricants to keep the bullet from slipping down of its own weight. The top end of the bullet for all tests is 0.375 inch down from the top of the cylinder, at a point fixed by the 0.625 inch thickness of the cork and the 1-inch magnet extension. The spike for the adherometer has a 0.437-inch head to provide sufficient clearance for it to pass safely down the cylinder bore without touching. Its shank has notches cut at points 2.125 and 3.125 inches from the head end, which are the correct distances from the top of the bullet when at A and B. This is a gauge for setting the contactors so that the buzzer will sound when the bullet is 0.5 inch above and 0.5 inch below the mid-travel position, which actually is 0.25 inch below the middle of the cylinder. As in the spike test, the two readings will be taken at A and B, regardless of how much the holder and scale platform depress. Special holders are provided for cooling and holding the bullet and pin.

Adherometer Test Procedure

"Corks are inserted in each end of the cylinder bore, extending inside about 0.25 inch. To reduce errors to a minimum the bullet starts from the same end, T, of the cylinder for all tests. The cylinder is placed in its jacket with end T down and the large cork left off to make cooling more rapid. A thermometer (reading to 0.2° F.) is inserted in the well. The cylinder, bullet, and lubricating pin are placed in a cooling cabinet which is held at 34° to 36° F., care being taken to see that no piece is allowed to cool as low as 32° F., for fear of moisture or frost deposit.

"When the cylinder temperature has dropped below 40° F. (35° F. for light products) the thermometer is removed and the cylinder wrapped in a cloth to keep it cool. The corks are removed and the bore is lubricated thoroughly from both ends, care being taken to make the last pass with the sizing plug in the direction the bullet travels during the test. The previously coated bullet is now inserted in the T end, followed by a cork which holds the bullet 0.25 inch from the end of the cylinder and away from the vent. The cylinder is placed in its jar, bullet end down, and the other end is sealed by a small cork. The outfit is then placed in the cabinet and the thermometer inserted. Now the cylinder is cooled, with the cabinet held at −5° F., and when the temperature of the adherometer reaches −2° or −3° F. the thermometer and top cork are removed; the cylinder is inverted, bringing the bullet (end T) at the top. Then the jar and cylinder are placed in the cold wooden holder, adding the wooden ring; the small cork is replaced by the large cork, which is forced down against the top end of the cylinder; the magnet is inserted and pushed down until the bullet reaches the proper point for starting; and the thermometer is inserted. The temperature of the cylinder is brought to exactly 0° F. by several applications of the hand to brass jacket. The

[7] *An. Ed. Ind. Eng. Chem.*, **6**, 130 (Mar. 15, 1934).

temperature is held at exactly 0° F. for 5 minutes or more, after which the thermometer and magnet are removed. The cylinder and holder (held by its base) are quickly carried to the spike-test machine. The dial is adjusted to zero and the bullet pushed down by the small-headed spike. Compensating weights are added as in the other test to equal the amount of the scale reading at about 0.25 inch above station A. The large cork is not removed during the test.

"The average of the A and B reading is taken as the adhesiveness or relative shifting ability of the lubricant if used in transmissions in cold weather. Winter lubricants are usually run at 0° F., whereas summer products are run at 20° F. The adherometer test enables one to classify lubricants with regard to shifting ability, and when correlated with actual transmission-shift tests furnishes valuable data.

"The adherometer is convenient to operate and is accurate within 1 per cent. Furthermore, the results of a second and third passage of the bullet through the cylinder check with the first test, if the temperature is the same in all tests. Only one determination is usually necessary. The instrument besides measuring the shifting ability of gear lubricants, including those intended for steering gears, also gives information relative to the viscosity of motor oil at very low temperatures, classifying them as to their ability to start easily."

Channel Tests

Knopf [7a] has developed a spike test which he describes as follows:

"The essential parts of this device are: a cylinder of seamless tubing (1.300 inch bore) for holding the test lubricant, thermally insulated by a dead-air space between it and a seamless brass jacket (2 inch diameter); the assembly is held and insulated by a wooden holder, which rests on an accurate dial weighing scale. A spike-shaped plunger with 0.5-inch diameter head and 0.25-inch shank, is pushed, head on, into the cold lubricant by means of an impelling weight, the velocity of descent of which is controlled by the passage of light oil through a needle valve, which regulates the flow of oil from below the piston to the upper end of the containing cylinder. The velocity of the descent is held constant for all tests, being arbitrarily chosen as 26.5 (26 to 27) seconds for a 4-inch travel of the spike. The riser bottle furnishes make-up oil to the cylinder when the piston-rod is withdrawn. The impelling force (about 35 pounds) is due to the combined mass of the piston, rod, and weight, which force is weakened during the test by an amount equal to the resistance between the spike and the lubricant, or the upward force on the spike as indicated by a scale.

"Compensating weights of various denominations are placed on the weight to keep the net effective force constant, thus maintaining the standard velocity of descent. The lever, and weights, counterbalance the combined mass of the test jar and holder, thus making it possible to read directly on the dial the viscidity of spike-test resistance in ounces.

"A reamer insures an exact outage of 1 inch in the test jar, or a constant quantity (4.375 inches) of lubricant for all tests. The scale is read precisely at points 0.5 inch above and 0.5 inch below the mid-travel position, which is defined as 2.25 inches below the original level of the lubricant. These two reading stations, A and B, are announced by the sounding of a buzzer. The buzzer circuit is completed by a brass plate on the weight coming in contact with a U-shaped contactor, carried on a rod mounted in roller bearings the bottom end of the rod being attached at the end of a lever. The vertical distance between the nut and the mid-travel position of the test jar remains fixed; hence, the buzzer sounds at A and B, regardless of the amount of depression of the scale platform, which for the full scale reading amounts to about 1 inch or more.

"The device, therefore, consists of a constant velocity of descent of a spike-shaped plunger; a well-insulated test jar, of accurate bore, for holding the test sample; and a means of counterbalancing the jar and holder so that the weighing device reads viscidity direct, at definite stations (A and B), 0.5 inch above and below the mid-travel position of the spike head. The viscidity is defined as the average resistance offered by the lubricant to the passage of the spike through it.

PROCEDURE OF SPIKE TEST.

"A sample of test lubricant, at approximately room temperature, is placed in a spike-test jar to a depth of about 0.75 inch from the top and sealed with a cork stopper.

[7a] An. Ed. Ind. Eng. Chem., 6, 130 (Mar. 15, 1934).

Another jar is filled with the same sample or a similar lubricant, also at room temperature, into which a Redwood thermometer is inserted with its bulb near the middle of the sample.

"The test jar and its dummy are now placed in a cooling cabinet with a breeze of air striking both jars similarly so as to insure the same rate of cooling. Other jars, containing similar lubricants of approximately the same consistency, may be cooled at the same time, the dummy indicating the temperature of all test jars. When the tests are to be made at 0° F., rapid cooling and satisfactory results are secured by holding the cabinet temperature at about —5° F. When the dummy reads 3° to 5° F. the cabinet temperature is brought to —1° F. and held here until the dummy reads exactly 0° F. The dummy is then held at 0° F. for 5 minutes, after which one of the test jars is placed in the holder, and the wooden ring added, both the latter being also at 0° F. The spike is cooled to 0° F. and held until needed in an insulated billet which was also cooled along with the jars and holder. The holder is placed on the scale platform and the pointer brought to zero by means of proper counterbalance weights, sliding poise, and dial adjustment. The cork is removed from the jar. The cold spike is now placed, as quickly as possible, in the chuck and the weight released by pulling the trigger. Compensating weights are held in readiness and added to the descending weight, equal to (or slightly greater than) the scale reading, until the spike reaches a point 0.25 inch above station A. A few ounces more or less than the A and B readings will not materially affect the rate of descent, inasmuch as each ounce represents only about 0.2 per cent of the total weight of 35 pounds.

"The viscidity, or spike-resistance, of the lubricant is the average of the A and B readings or equivalent to the resistance at mid-travel position. As soon as the weight comes to rest, the spike is disconnected, the gate valve is opened, and the weight raised to its top position, where it is held by a catch.

"Lubricants of considerable fluidity flow behind the head during the 60-second period, the amount being in proportion to the fluidity or nonchannel properties of the lubricant. On withdrawing the spike, the quantity adhering to it is noted, and the number of inches the lubricant strings out also observed. A combination of these two factors constitutes a basis for grading the lubricant as to its nonchannel and pick-up characteristics. Grade A means excellent; B is good; C is fair (tendency to channel and poor feeding); D is poor (with channel and practically no pick-up); while E is very poor. Petrolatum, for example, has an E grade with perfect channel and no pick-up at 0° F.

"The resistance in ounces is something definite and can be checked within 2 to 5 per cent accuracy; the grading method, being based on judgment and correlation with gear-set test, requires considerable experience and is difficult to describe accurately.

"As we are measuring consistency, and therefore cohesiveness chiefly, we eliminate as much as possible all resistances due to adhesiveness by means of the large head and small shank of the plunger. With light winter lubricants there is sometimes a slight error due to the fact that the lubricant flows behind the head on the down stroke.

"The viscidometer is invaluable as a control instrument in the manufacture of gear lubricants, as well as a means of comparing various products with regard to their low-temperature consistencies. When correlated with the rear-axle test, the results of the spike test enable one to predict accurately just how a certain lubricant will function at low temperatures. The spike test, however, does not give accurate information relative to its shifting resistance when employed in a transmission, being only fairly indicative of shifting ability.

"The shifting resistance present in automobile transmissions in cold weather is due almost entirely to the adhesiveness of the lubricant in the clearance space between the sliding gear and the shaft on which it slides, and is also due, to a great extent, to the lubricant adhering to the tooth surfaces of the mating gears. In some designs, the sliding gear is partly immersed in the lubricant bath, but the resistance from this source is relatively small as compared with those mentioned above.

"Shifting resistance tests, in which a transmission is employed, are very inconvenient to run, and yield results which may not check within 20 or 25 per cent. The sleeve test was developed and used in this laboratory for some time to determine the relative adhesiveness of various lubricants. Although this device is more convenient and gives more consistent results than the shifting tests, it still falls short of being a practical control instrument. The sleeve test, or concentric cylinder viscometer, measures absolute viscosities of lubricants, with a range of 1500 to 100,000,000 Say-

bolt seconds. It has been found invaluable for the calibration of other viscosity instruments as well as for obtaining readings on heavy lubricants at very low temperatures when other instruments are difficult to manipulate."

Quality Tests

Coefficient of Friction

Static Coefficient of Friction

The static coefficient of friction is best determined by means of an inclined plane testing device such as the one developed by Wilharm and which may be constructed at a cost of approximately $500.

Coefficient of Static Friction

The inclined plane apparatus for determining the static coefficient has been developed to a fairly accurate instrument by Wilson and Barnard [8] and a modification of it was used by Rhodes and Allen for the study of sodium base greases. The results of these tests are given in Chapter VII.

Kinetic Coefficient of Friction

The Herschel Oiliness Machine.[9] The following discussion of oiliness* and the use of the Herschel coefficient of friction apparatus is of interest. This device has been used with success for determination of the kinetic coefficient of friction of lubricating greases, particularly at high unit pressures:

In mathematical discussions of lubrication,[1] it is always assumed that viscosity is the only property of the lubricating oil which affects the friction, and that the bearing metal has no effect. It is perhaps on this account that friction machines have ordinarily been used to study the laws of lubrication of journal bearings, or to test the quality of lubricants rather than of bearing metals.

Experience, however, has shown that when bearing pressures are very high, speeds are very low, or there is an inadequate supply of lubricant, both the "oiliness" of the lubricant and the friction reducing properties of the bearing metal must be considered.

"Without attempting to decide what is the exact cause of oiliness,[2] it may here be considered as the joint property of metals and lubricant which causes a difference in the coefficient of friction when two lubricating oils, having the same viscosity at the temperature of the oil film, or two bearing metals of equal smoothness, are used under identical conditions. As suggested by this definition, the simplest method of measuring oiliness is by a friction machine, although various other methods have been proposed to avoid the polishing of metal surfaces to the required degree of smoothness.

THE JOURNAL FRICTION MACHINE

"It is desirable in any friction machine to provide for readily changing the rubbing surfaces in order to maintain them at a constant smoothness. With machines used for testing oiliness, rubbing surfaces must also be changed in order to determine the effect of different metals.

[8] *Ind. Eng. Chem.*, **14**, 683 (1922).

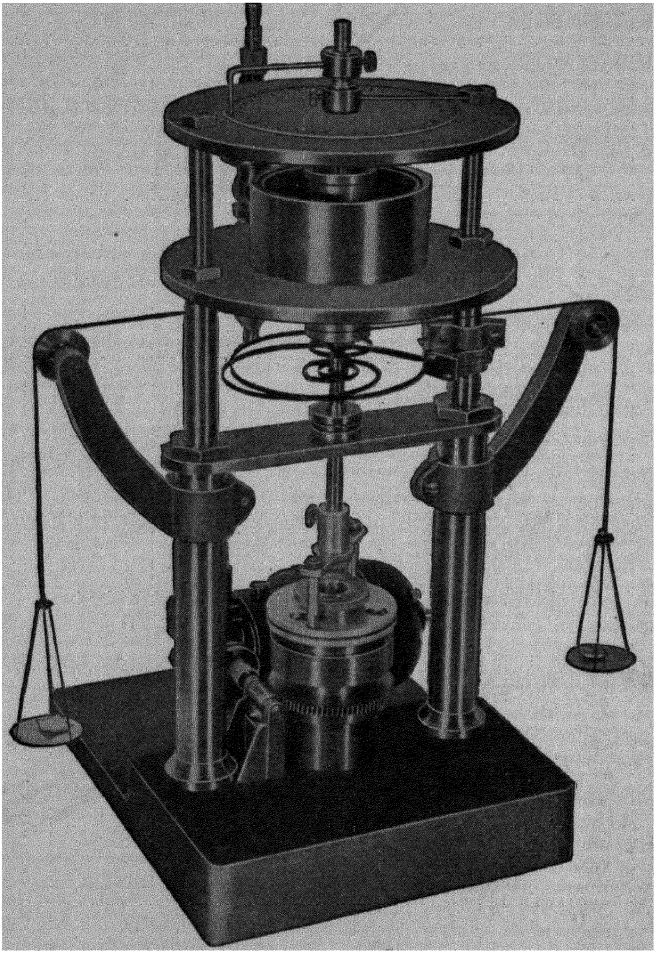
[9] The American Instrument Company, Bull. 112.

* Prepared from data furnished by Winslow H. Herschel.

[1] For example, M. D. Hersey, On the Laws of Lubrication of Journal Bearings, Trans. A. S. M. E., 37, pp. 167-202; 1915. A. G. M. Mitchell, Viscosity and Lubrication, Chapter III, in "Mechanical Properties of Fluids" (a collective work); London, Blackie; 1923.

[2] For a discussion of possible explanations of oiliness, see paper by Hans Karplus, Petroleum Zeit., 25, pp. 375-386; 1929.

"The journal friction machine presents considerable difficulty in testing oiliness, since the rubbing surfaces cannot be repolished without changing the clearance and hence the friction. If sufficient care is taken, however, in design and operation, differences in oiliness may be determined by a journal bearing operated at low speeds and under high pressures."[3]



Courtesy of the Amsler Instrument Co.

PLATE XXVIII.—The Herschel Oiliness Machine.

THE DICKINSON MACHINE

"The difficulty that polishing the surfaces changes the clearance has been avoided in a novel machine, suggested by Dr. H. C. Dickinson, of the Bureau of Standards,[4] in which three one-half-inch balls of steel or other material, symmetrically placed in a frame, are pressed against circumferential grooves on a rotating cylinder of bearing

[3] A. Kingsbury, A New Oil Testing Machine and Some of Its Results, Trans. A.S.M.E., 24, pp. 145-167; 1903. S. A. McKee, The Effect of the Addition of Kerosene on the Oiliness of Lubricating Oils, Jour. S. A. E., 19, pp. 356-360; 1926.

[4] See C. F. Marvin, Jr., A Machine for Comparing the Lubricating Properties of Oils at High Pressures, Jour. S. A. E., 18, pp. 287-289; 1925.

metal. The motion of the frame is restricted, and its tendency to rotate is a measure of the friction.

THE DEELEY MACHINE

"In the machine of R. M. Deeley,[5] a heavily loaded tripod rests upon a disk which is rotated slowly by hand, by means of a crank and suitable gearing. The tripod is supported by three $\frac{5}{32}$-inch pegs. When the disk is rotated, the pegs are carried with it by the friction, thus rotating the tripod. The movement of the tripod actuates a vertical spindle to which a spiral spring and pointer are attached. When slipping takes place between the pegs and the disk, due to the stress in the spring, the reading of the pointer, moving over a graduated scale, gives a value from which the coefficient of friction of rest may be calculated.

"Using the Deeley machine with pegs flat at the bottom, Dover[6] found that the coefficient of friction decreased as the pressure increased. With the high pressures obtained when a tripod or slider rests on spherical surfaces, it was found by Hardy and Doubleday[7] that the coefficient of friction is independent, not only of the pressure, but is also independent of the temperature over a considerable range of temperatures.

COEFFICIENT OF REST OR MOTION

"The question might arise in designing a friction machine for testing oiliness whether it is desirable to measure the coefficient of friction of rest or motion. Wilson and Barnard,[8] who used both a simplified Deeley machine for measuring the coefficient of friction of motion, and a slider on an inclined plane for measuring the coefficient of friction of rest, decided in favor of the latter. On the other hand, Woog[9] concluded that it was preferable to measure the coefficient of friction of motion, because results of static tests were so apt to be discordant due to defects in the rubbing surfaces, and to accidental vibrations from any cause. In determining static coefficients there was also the difficulty of deciding the exact moment at which the slider started to move.

"Woog's tests were made with steel on steel and do not show any comparison between different metals. Table 1 shows coefficients of friction as obtained by Deeley.

TABLE 1.—COEFFICIENTS OF FRICTION OF REST ACCORDING TO DEELEY

Oil	Coefficient of Friction—Mild Steel on Cast-Iron	Coefficient of Friction—Mild Steel on Lead Bronze
H. B. clock oil	0.271	0.275
Bayonne oil	.213	.234
Typewriter oil	.211	.294
Victory red	.196	.246
F. F. F. cylinder	.193	.236
Manchester spindle	.183	.262
Castor	.183	.159
Valvoline cylinder	.143	...
Sperm	.127	.180
Trotter (neatsfoot)	.123	.152
Olive	.119	.196
Rape seed	.119	.136

Deeley, in commenting on Table 1, says:

"Rape and olive oil give the best results and mineral oils the worst, castor coming between the mineral oil and the rest. Rape and olive oil are of equal friction-reducing value between mild steel and cast-iron, but rape is the better oil between mild steel and the lead-bronze." As the lead-bronze contained only 4 per cent each of lead and of zinc, 5.7 per cent of tin and 85.9 per cent of copper, it seems quite possible that the lower friction on cast-iron, with all of the fatty oils except castor, may have

[5] R. M. Deeley, Proc. Physical Soc. of London, 32, part 2, pp. 1s to 11s; 1920.

[6] M. V. Dover, Ind. Eng. Chem., 18, pp. 499-501; 1926. M. V. Dover and J. H. Cromwell, Ind. Eng. Chem., 19, pp. 94-96; 1927.

[7] W. B. Hardy and I. Doubleday, Proc. R. S., 101, pp. 487-492; 1922.

[8] R. E. Wilson and D. P. Barnard, 4th, The Mechanism of Lubrication, II, Ind. Eng. Chem., 14, p. 682; 1922.

[9] Paul Woog, Contribution, a l'étude du Graissage, Paris, Librarie Delagrave, p. 216; 1926.

been due to the fact that the fatty acids contained in the oils caused less corrosion on the cast-iron than on the copper contained in the bronze.

THE HERSCHEL OILINESS MACHINE

"The Herschel oiliness machine [10] resembles the Deeley machine in some respects, but the disk is motor driven, at very low speeds, so that the coefficients of friction of rest and of motion may be obtained. The slip takes place between the rotating disk and a slider supported on three one-half-inch steel balls, such as used in ball bearings. *As the steel balls are a commercial article and readily replaced, and the disk is easily polished, a uniform smoothness of rubbing surfaces may be obtained without difficulty.* The speed of slipping ordinarily used is 0.24 foot per minute, but it was found that this could be considerably increased or decreased without changing the friction.

"The disk, which may be made of any bearing metal which it is desired to investigate, is 2 inches in diameter by $\frac{1}{8}$ inch thick, with a $\frac{1}{8}$-inch hole in the center. Disks of these same dimensions are used in the Amsler Wear Test Machine,[11] so that, if desired, wear tests may be made on the edge of a disk in the Amsler machine, and an oiliness test made afterwards on the face of the same disk.

"The slider is similar to that used by Koethen [12] in tests of static friction. Since a very high pressure is assured by the "point contact" between the disk and the steel balls, the slider is not weighed as in the Deeley machine. On the contrary, in order to make it as light as possible so as to avoid scratching the disk, it is made mainly of aluminum, the three steel balls being clamped between two aluminum disks so that the balls do not rotate. The slider weighs 129 grams and when loaded to weigh 179 grams there was no change in the coefficient of friction.

"When the machine is started the slider and the pointer at the top of a vertical shaft rotate with the same angular velocity as the motor driven disk until slipping begins, the rotation of the shaft winding up one flat spiral spring and unwinding another. The pointer moves over a graduated circle, and the springs are so adjusted that the readings of the pointer give the coefficient of friction direct. When the tension in the springs is sufficient to cause slipping, the reading of the coefficient of friction of rest is taken.

"It has been found that this first reading is unreliable, presumably because it varies with chance irregularities in the smoothness of the rubbing surfaces, or with variations in the oil film. After continued running the reading generally falls, as would be expected from the effect of "running in," which smooths the surface of the disk in the track of the balls. In other cases, however, the friction increases with the time of running. This may be due to corrosion which has frequently been shown by a green discoloration of the oil on bronze, or it may be due to a poor lubricant or unsuitable combination of metals, as steel on steel.

"It has been found, in accordance with Kingsbury and Hardy, that with a straight mineral oil the oiliness improves with the viscosity. It has also been found that stearic acid reduces friction more than does oleic acid, when there is no corrosion.

"With a mineral oil of medium viscosity, the coefficient of friction of motion was found to decrease from 0.18 on metal containing no lead, to 0.10 on 95 per cent lead. A metal containing zinc was also found to give lower friction than would have been anticipated from its lead content.

"With any metal the friction could be reduced by the use of a fatty oil (when not corrosive), the greatest reduction in friction being obtained with metals high in lead. The lowest value thus far obtained is 0.02, with lard oil on 95 per cent lead. This is also Hardy's lowest value, obtained with recinolic acid on bismuth."

Film Strength

THE TIMKEN EXTREME PRESSURE LUBRICANT TESTER

The Timken Roller Bearing Company, of Canton, Ohio, has developed an apparatus for testing the load carrying capacity of lubricants that has

[10] Winslow H. Herschel, Proc. A. S. T. M., 28, part II, pp. 687-9; 1928.
[11] Bureau of Standards, Research Paper No. 13, p. 357; 1928.
[12] F. L. Koethen, Ind. Eng. Chem., 18, pp. 497-9; 1926.

some very interesting features. It was originally developed for use in the company's own laboratory as a means for obtaining accurate information on the effect of different lubricants on Hypoid and spiral bevel gears in automotive axles, and to test greases in connection with many intricate industrial bearing problems.

The apparatus consists essentially of a cast iron base which supports the testing mandrel, two levers and a container holding about a gallon of the lubricant to be tested. In detail, the tank is mounted on top of the base, and just under it is an electric heater, used to raise the oil to any desired temperature up to 210° F.

The oil flows from the tank, over the test piece, to a sump in the base by gravity, the rate of flow being adjusted by means of a valve in the line. From the sump, the oil is pumped back to the tank by a small pump located in the base, and belt driven from the testing mandrel. The latter extends through the base, and may be either direct or belt driven by a variable speed fractional horsepower motor. The mandrel is mounted in two Timken bearings, so arranged as to hold it rigidly in alignment and is tapered at the test end to receive the tapered cup of a Timken bearing. This cup, which forms one of the test pieces is held in place firmly by a nut on the end of the mandrel, which is threaded to receive it.

The lever system, which is the most important part of the device, consists of two levers, one above the other. The upper is called the load lever, and the lower the friction lever, for reasons that will appear. The upper, which carries the test block is pivoted on a knife edge, mounted in the lower lever. The latter is also pivoted on a knife edge and is provided with a stop at the unloaded end. By virtue of this arrangement the test block is always parallel to the revolving cup and the unit loading over the length of both pieces is always constant. The test block is a small piece of metal about $\frac{1}{2}$ inch square by $\frac{3}{4}$ inch long hardened to Rockwell C-60 and ground. It is inserted in a notch in the loading lever and held in place by a wedge. In case bearing metals are to be tested both the test block or the cup or both can be made of the appropriate material. The friction lever is provided with a vernier scale, and a sliding weight for obtaining accurate measurements and both levers are equipped with hangers for carrying adjustable weights. A welded steel housing is provided to cover the end of the mandrel and levers.

The operation of the device is substantially as follows: Assuming both test pieces in place, the driving motor or line shaft is brought up to the desired speed, and the lubricant heated to the required temperature, is allowed to flow over the test block. The loading lever is then loaded by means of weights until the desired unit pressure is obtained between the test pieces, a chart being furnished with the apparatus which gives the necessary weights on the loading lever per 1,000 pounds of unit pressure. If only the load carrying capacity of the oil is to be determined, the test block is removed after 10 minutes' running, and examined for signs of scuffing. The degree of scuffing determines the relative load carrying capacity of the lubricant. In case the coefficient of friction of the lubricant is desired, weights are added to the friction lever until it moves away from the stop. Loading the upper lever causes the friction on the test block to move it forward horizontally, unbalancing the friction lever until it rests on the stop. This condition is compensated by adding the weights on the friction lever, until the system is again in balance and the lever is off the stop. The coefficient of friction of the lubricant is then calculated from the amount of weight it takes to balance the levers. It would be a simple matter if extreme accuracy in this regard was needed to provide a make or break electric circuit between the lever and the stop that would indicate, by means of a lamp, or some other device, exactly when the corerct weight is reached. The same holds true, of course, in case different bearing materials are being tested. Not the lest interesting feature of the machine is the permanence of the records it provides. The condition of the test block can be examined at leisure, as a means of analyzing the effectiveness of the lubricant under test.

Typical Timken Film Strength Tests. The relation between Timken film strength and viscosity of a given series of non-compounded mineral oils has been reported by Maag as follows:

Bé. at 60° F.	Viscosity at 210° F.	Load on Lever Arms — Pounds — O.K.	Score
27.4	40 sec.	1	2
21.0	48 "	5	6
22.0	65 "	7	8
26.9	73 "	9	10
26.6	99 "	11	13
25.8	124 "	13	15
25.7	153 "	15	17
22.6	180 "	18	20

Resistance to Capillary Action

Filter Paper Test for Bleeding of Cup Greases

In this test a small sample of the grease to be tested is carefully packed in a $\frac{5}{8}$-inch brass ring, and the grease and ring placed at the center of a close-textured, analytical type filter paper. The paper is supported by a flat tin surface directly below the ring, and air is permitted to circulate both below and above the balance of the paper surface. After four hours the area of filter paper showing oil stain is measured. Insofar as the author is aware this test has not yet been correlated with stability in storage nor is its definite relation to stability in service known. It is reasonable to assume, however, that the test affords a fair measure of the resistance of a grease to the capillarity presented by the filter paper. Whether or not the effects of dust located on the edges of bearings exert a comparable effect has not been investigated, but it is quite likely that correlation is possible.

The brass ring used in this test complies with the requirements given in A.S.T.M. D36-26 for the brass ring used in the Ring and Ball Method for determining the softening point of bituminous materials. This grease stability test may also be applied to lubricants which have been dehydrated by heating for ten minutes at 210° F., and then poured into the ring resting on a cold metal surface. The ring and sample are then placed on the filter paper after cooling to room temperature. In the typical results given below the "F" series greases are European products while the various grades of cup grease were commercial products made on the standardized formulae discussed in Chapter V.

Name of Grease	Age of Grease (Months)	Percent Soap	Percent Free Alkali	Separation as Observed in Container	Filter Paper Test — Original Grease	Filter Paper Test — Grease after Dehydration
Cup No. 00	1	9.8	.07	none	15.2	15.2–18.1
Cup No. 0	8	13.6	.24	none	13.5–14.1	28.6–30.2
Cup No. 1	11	11.5	.09	slight	13.2–12.6	14.9–10.1
Cup No. 2	12	14.3	.15	none	10.7	12.0–12.6
Cup No. 3	12	17.7	.18	none	8.6	12.0–17.3
Cup No. 4	6	25.0	.34	none	5.3– 6.2	28.3–30.6
Cup No. 5	11	35.6	.20	none	2.8	4.9– 6.2
F. 1					20.8	65
F. 2					19.5	68
F. 2a					15.8	58
F. 3					18.7	about 80
F.A. 2					18.0	36.8
F.B. 2					18.7	38
R.B.		18.9			6 1	4.5

Separation of the Oil Phase

Ordinarily the lighter greases, containing less soap, are more subject to separation of the oil phase than for the more consistent greases.

Storage Test

A 5-pound can of grease received from the manufacturer may show no separation of free oil on opening. If, however, a hollow about the size of a tea cup is made in the center of the can near the top, and the can stored in a warm room for six weeks, oil to the extent of less than one per cent, or to as much as 12 per cent in some cases, may be poured off and weighed or measured.

Screen Tests

A weighed quantity of greases is placed in a cone-shaped section of wire gauze, which in turn is placed in a funnel. The apparatus is then mounted in a constant temperature box and the amount of oil which drips into a beaker is weighed after given time intervals.

Herschel Bleeding Test for Cup Greases

An interesting and useful method of determining the bleeding characteristics of cup greases has been worked out by Herschel and others.[10] For complete details of this method the original paper ("A Procedure for Measuring Bleeding of Cup Greases.") should be consulted.

Separation of the Oil Phase by Ultra Filtration

Experiments were made with an Alemite, screw-type grease gun, connected to a pipe fitting (cap) in which a $\frac{3}{8}$-inch hole had been bored. Over this hole was placed a disk of 130 mesh steel wire gauze which supported a small disk of filter paper. The grease gun was filled with pressure gun lubricant, and a pressure of 500 pounds per square inch was applied as indicated on a pressure gauge connected to the grease chamber. After 20 minutes oil to the extent of about 1 per cent of the grease in the system had filtered through the paper. The flow of soap free oil then ceased due to clogging of the filter with the heavy grease base. It was concluded from this experiment that the filter area was too small and that with a greater area with respect to the volume of grease, greater amounts of free oil could be collected.

With this object in mind, small Alundum thimbles (Alundum R.A. 98, coarse porosity) were tried as a filtering medium, using the same Alemite gun as a source of pressure. Considering that the system held 25 grams of grease, it was found that after two hours, 17.8 per cent of soap free oil was filtered from lime base grease using a pressure of only 10 pounds per square inch. A heavy residue, resembling No. 4 Cup Grease, was left on the interior of the thimble and greatly retarded the oil flow after about one hour.

Standardized pressure gun grease in a similar test yielded 9.3 per cent of oil.

A grease prepared from 9 per cent of Aluminum Stearate in 300 Pale Oil, gave 25 per cent of separated oil in the two hour period.

[10] *Proc. Am. Soc. Testing Materials*, **33**, Pt. I., 343-347 (1933).

The Timken Separation Test

Maag [11] has described the following test for determining the heat stability of ball and roller bearing greases which is used by British Timken Limited:

"25 grams of grease are melted in the standard Pensky-Martens Flash Point Cup, and heated at the rate of 10° F. per minute to 250° F. The grease is maintained at this temperature for half an hour.

"During the heating and maintaining the grease is to be stirred at the rate of 2 revolutions per second for alternate minutes.

"25 grams of grease are heated to 250° F. and held at that temperature for half an hour, then poured into a silica tube, which when cool is suspended over a conical measure, graduated in c.c.'s., in which the mineral oil that separates out in 48 hours is collected.

"Greases showing separation to be rejected.

"After the grease has been held at a constant temperature of 250° F. for half an hour it is poured into a silica tube, approximately 3 inches long by 1 inch inside diameter, one end of which has been previously sealed with a cork.

"After half an hour atmospheric cooling, the cork is removed, and the grease, supported by two pieces of flattened wire, approximately $\frac{1}{8}$ inch wide, wound vertically round the tube, is suspended over a conical measure graduated in c.c.'s.

"The oil collected after 48 hours × 4 = per cent separation on 100 grams of grease.

Hydroscopic Tests

Moisture Absorption Test

The moisture absorption test is made by taking Petri culture dishes about $2\frac{1}{2}$ inches in diameter and cutting them down to a height of $\frac{3}{16}$ inch. This gives a thin layer of grease exposing as much surface as possible. The dishes are filled with grease and the top leveled off. They are weighed and placed in a dessicator under 95 per cent relative humidity. This is obtained by using a glycerin solution of 1.04 specific gravity. At the end of a definite period of time, say one week or one month, the dishes are reweighed and from the gain in weight the percent of moisture absorbed is calculated. A high rate of absorption may lead to other troubles. Under accelerated oxidation tests the introduction of moisture causes the greases to become sticky and gummy and this condition is sometimes encountered in bearings in storage. When grease oxidizes in a bearing in storage a certain amount of soluble volatile organic acids may be formed. It is considered that the combination of moisture with these acids gives a conducting solution, which with dissimilar metals present in bearings may produce electrolytic action and be the cause of pitting or corrosion. It is also possible that only the water soluble organic acids are dangerous and, therefore, that a determination of total acidity in a grease, which includes not only acetic and butyric acids but also the higher organic acids such as oleic and stearic, does not give a true picture. Tests have indicated that a sample of badly oxidized grease taken from a motor in storage four years had a total acidity of 5.19 per cent (calculated as oleic acid). Of this amount 3.42 per cent (calculated as oleic) were water soluble acids.

[11] Paper presented at A.I. & S.E.E. Pittsburgh Convention (Sept. 24, 1935). See also: *Iron & Steel Engr.*, (Dec. 1935).

Pressure Viscosity

General Discussion of the Rheology of Greases

To those concerned with the manufacture and testing of lubricating greases a thorough understanding of the distinction between fluid and plastic flow is essential. The plastic nature of grease is probably its most important characteristic which distinguishes it from fluid lubricating oil. In the case of fluid oils continuous deformation will occur regardless of how small the shearing force is which may be applied to it. If we place an oil in a viscosity tube to which pressure may be applied it is possible to determine the various rates of flow for different applied pressures. If these quantities of oil which flow in a unit of time are plotted on plain coordinate paper against the applied pressures, a straight line will be obtained which passes through the origin. As discussed in Chapter V, in the section on gear greases, lubricating greases containing soaps do not follow this rule but require a certain initial force or yield value in order to start flow, and hence do not plot as a straight line through the origin, but show a curved section through the origin, terminating in a straight line the slope of which represents viscosity. The point at which this line, when extrapolated, passes through the pressure axis indicates the pressure at which flow will start or the "yield value." The equation for viscous flow (length and diameter of orifice and viscosity constant) may be reduced to the simple form:

I. $$Q = Cp$$

in which:

Q is the volume of grease to pass through the orifice in unit time;
C is a constant;
p is the pressure drop through the orifice.

In the case of plastic flow of greases the straight portion of the curve is indicated by the following equation:

II. $$Q = M\,(P - Y)$$

in which:

M is a constant;
Y is the yield value;
P is the total pressure applied to the grease.

In Equation I, for the viscous flow of oils, when Q is equal to zero, then the pressure drop is zero, and the plotted line passes through the origin. For the plastic flow of greases, Equation II, when Q is equal to zero, Y then becomes equal to P, or in other words, the extended straight portion of the curve starts on the pressure axis at a point equal to the yield value.

Fundamentals of Viscosity and Fluidity

The law of Poiseuille for viscous flow of fluids in a circular tube is based on the following assumptions:

Flow occurs without radial components.

Shearing force per unit area along any cylindrical surface concentric with the axis of the tube is

$$\mu \frac{du}{dr}$$

The motion is steady $\left(\frac{du}{dt} = O\right)$

Pressure uniform over sections perpendicular to axis.

Definition: The viscosity of a substance is measured by the tangential force on a unit area of either of two horizontal planes at a unit distance apart required to move one plane with unit velocity with reference to the other plane, the space between being filled with the viscous liquid

$$\left(T = \mu \frac{du}{dr}\right)$$

The coefficient of fluidity is the reciprocal of the coefficient of viscosity

$$\left(\varphi = \frac{1}{\mu}\right)$$

Symbols:

r = radius
x = distance in direction of motion
u = velocity in x-direction
p = pressure
μ = absolute viscosity
φ = coefficient of fluidity
ρ = density
T = shearing force per unit area
Q = volume per second
t = time integral
V = total volume discharged in time t.
$\frac{pv}{g}$ = velocity head
g = acceleration of gravity.

The equation of equilibrium of a ring of length dx and thickness dr is

$$2\pi\mu\left[(r + dr)\left(\frac{du}{dn} + \frac{d^2u}{dr^2}dr\right) - r\frac{du}{dr}\right]dx + 2\pi r\left[p - (p - dp)\right]dx$$

$$\frac{d^2u}{dr^2} + \frac{1}{r}\frac{du}{dr} + \frac{1}{\mu}\frac{dp}{dx} = O \tag{1}$$

$$u = C_1 r^2 + C_2 \tag{1a}$$

Substituting in the differential equation, $C_1 = \frac{1}{4\mu}\frac{dp}{dx}$ and from the boundary condition that $u = O$ at $r = R_1$, $C_2 = +\frac{R^2}{4\mu}\frac{dp}{dx}$

$$u = \frac{1}{4\mu}(R^2 - r^2)\frac{dp}{dx} \tag{2}$$

$\left(\frac{dp}{dx}\right.$ is positive for a decrease in pressure in the direction of motion.)

Perhaps a better way to obtain this equation is to consider the forces acting on a central core of radius r.

$$(3)\qquad \begin{cases} 2\pi r \cdot T \cdot dx + [p - (p - \varphi)]\,\pi r^2 = O \\ T = -\dfrac{r}{2}\dfrac{dp}{dx} = \mu \dfrac{du}{dr} \end{cases}$$

At the center, $r = O$, $T = O$ and hence $\dfrac{du}{dr} = O$. Integrating,

$$u = -\frac{1}{4\mu}\frac{dp}{dx} r^2 + C.$$

$$r = R,\ u = O,\ C = \frac{1}{4\mu}\frac{dp}{dx} R^2$$

$$(2)\qquad u = \frac{1}{4\mu}\frac{dp}{dx}(R^2 - r^2).$$

The volume discharged per unit time is

$$Q = \int_o^R u \cdot 2\pi r - dr = \frac{2\pi}{4\mu}\cdot\frac{dp}{dx}\int_o^R (R^2 - r^2) r \cdot dr$$

$$(4)\qquad = \frac{\pi}{8\mu}\cdot\frac{dp}{dx} R^4;\ U_m = \text{mean velocity} = \frac{Q}{\pi R^2} = \frac{R^2}{8\mu}\frac{dp}{dx}$$

which is the law of Poiseuille. The volume in time t is:

$$(5)\qquad V = \frac{\pi}{8\mu}\frac{dp}{dx} R^4 t.$$

The kinetic energy being carried past a cross-section in unit time is:

$$(6)\qquad E = \int_o^R \rho \cdot \frac{u^2}{2} \cdot u \cdot 2\pi r \cdot dr$$

$$E = \cdot\frac{\pi\rho}{64\mu^3}\left(\frac{dp}{dx}\right)^3 \int_o^R (R^2 - r^2)^3 r \cdot dr$$

$$(6a)\qquad = \frac{\pi\rho R^8}{512\mu^3}\left(\frac{dp}{dx}\right)^3 = \pi\rho R^2 U_m{}^3$$

The average kinetic energy per unit weight or the velocity head is:

$$(7)\qquad \frac{p_v}{\rho g} = \frac{E}{Q\rho g} \cdot = = 2 \cdot \frac{U_m{}^2}{2g}$$

2. Capillary Tube Viscometer (Primary Method).

The law of Poiseuille assumes that the coefficient of viscosity μ is a physical constant of the liquid and that its value does not depend upon the velocity or other properties of the flow nor upon the size, length or characteristics of the tube. The validity of this assumption has been checked experimentally and it has been found that for $\dfrac{2u\rho R}{\mu} < 2000$ (approximately), depends only upon pressure and temperature for true fluids.

In the derivation of Poiseuille's equation, only the conditions at a single section normal to the motion were considered. The obvious difficulties of making connections for pressure measurements along the capillary tube itself has led to the use of a capillary tube connecting two chambers and this method requires corrections for "end effects."

p_u = loss of head at entrance
p_t = total energy
p_s = static pressure
ρu_m^2 = velocity head $\left(= 2\rho \frac{U_m^2}{2}\right)$
Δp = measured pressure drop
L = length of capillary tube.

a. Liquids. The loss of head per unit weight is $\frac{\Delta p}{\Delta g}$ and, if this loss is very great and the tube well insulated, the temperature of the liquid, and hence the viscosity, may be altered during passage through the tube. In the case of water, a loss of head of 778 feet is necessary to increase the temperature 1° F. It is apparent that the temperature correction is usually negligible. Assuming that the temperature remains constant and that the entire velocity head is lost as heat at discharge

$$\Delta p = p_u + L \cdot \frac{dp}{dx} + \rho u_m^2; \quad \rho = \text{const.}; \quad u_m = \text{const.} \tag{8}$$

$$\frac{dp}{dx} = -\frac{8\mu Q}{\pi R^4} \tag{4}$$

Since the loss at entrance probably depends upon the velocity head, we may substitute

$$p_u + \rho u_m^2 = m\rho u_m^2 = m\rho \cdot \frac{Q^2}{\pi^2 R^4} \tag{9}$$

$$\mu = \frac{1}{8L}\left(\frac{\pi \cdot \Delta p \cdot R^4}{Q} - \frac{m\rho Q}{\pi}\right) \tag{10}$$

The coefficient m has a value of about 1.13 on the basis of ρu_m^2 or 2.26 for $\frac{1}{2}\rho u_m^2$.

b. Slippage. It has been thought by some experimenters that slippage might occur at the wall of the tube in which case $u \neq 0$ at $r = R$. The bulk of the evidence is in favor of zero slippage at least for liquids but there is some reason to believe that gases made up of large molecules may show a slip. The equation derived by Helmholtz for the viscous flow of a liquid with slippage is

$$Q = \frac{\pi \cdot \Delta p}{8\mu L}(R^4 + 4\lambda R^3)$$

where λ is a coefficient depending upon the nature of the fluid as well as upon that of the boundary surface.

c. Viscometry. Bingham [12] gives viscometer rather than viskosimeter as the name for an instrument for measuring viscosity.

There are two general classes of viscometers, namely, primary instruments which are used to measure the viscosity directly and secondary instruments which are calibrated over the range of use by means of liquids of known viscosity. The principal requirement for a primary instrument is that the torque, speed or volume, as the case may be, can be expressed analytically as a function of viscosity. Some primary methods are:

Oscillations of a disc, sphere or cylinder.
Falling sphere or rising bubble.
Efflux through horizontal and vertical tubes of small diameter.
Rate at which one substance penetrates another under influence of capillary action, diffusion or solution tension.
Decay of oscillations of a liquid in a U-tube.
Decay of waves upon a free surface.
Decay of vibrations in a viscous substance.
Rate of crystallization.

The majority of the secondary instruments are of the secondary type in which the time of efflux of a given volume of liquid is measured.

3. Viscosity and Fluidity of Mixtures and Emulsions.

a. Emulsions—Viscosities Additive.

Consider a series of vertical laminae of viscous material arranged alternately and completely filling the space between two planes and subjected to a horizontal shearing stress. Suppose that all of the laminae of viscosity μ_1 have the same thickness s_1 and those of viscosity μ_2 the thickness s_2 and that the corresponding shearing stresses per unit area are T_1 and T_2. Then the total shearing force is

$$TS = S_1T_1 + S_2T_2 + \text{— —}; \quad T_1 = \mu_1\frac{u}{R}; \quad T_2 = \mu_2\frac{u}{R}. \tag{11}$$

The velocity of the upper surface is u and the apparent viscosity μ' is

$$\mu' = \frac{TR}{u} = \frac{R}{u}\left(\frac{S_1T_1 + S_2T_2 + \text{—}}{S}\right) \tag{12}$$

But $\frac{s_1}{s}$ is the percentage by volume of the substance (1) and $\frac{s_2}{s}$ is the percentage of (2) so that

$$\mu' = \frac{R}{u}\left(a\cdot\mu_1\frac{u}{R} + b\mu_2\frac{u}{R} + = a\mu_1 + b\mu_2\right) \tag{13}$$

where a and b are the percentages by volume. The viscosities are additive. This case is of particular interest in connection with emulsions and other poorly mixed substances.

b. Fluid Mixtures—*Fluidities Additive.*

Laminae assumed to be arranged parallel to the direction of shear, each liquid in layers of constant thickness r_1 and r_1. The shear is constant

$$T = \frac{\mu_1u_1}{r_1} = \frac{\mu_2u_2}{r_2} = \text{— — — — —} \tag{14}$$

If the viscosity is to be computed from the volume of flow, then we must consider the line BS' with a velocity u' which the surface BS would have if the laminae were replaced by a homogeneous fluid having the same volume of flow. The relation between the effective velocity and the volume of flow is

$$Q = \frac{u'R}{2}$$

If the apparent viscosity corresponding to Q is μ' then

$$T = \frac{\mu' u'}{R} = \frac{2\mu' Q}{R^2} \tag{15}$$

Had the less viscous substance been in contact with the lower surface, the velocity u'' corresponding to BS'' should have been used.

$$U = U_1 + U_2 + \text{— — — — — —} = U'' \text{ (approx.)} \tag{16}$$

$$\frac{TR}{\mu'} = TR\varphi' = U_1 + U_2 + \text{— — — — —}$$

$$= \frac{Tr_1}{\mu_1} + \frac{Tr_2}{\mu_2} + = \text{— — —} = Tr_1\varphi_1 + Tr_2\varphi_2 + \tag{17}$$

$$R\varphi' = r_1\varphi_1 + r_2\varphi_2 + \text{—} \tag{18}$$

$$\varphi' = a\varphi_1 + b\varphi_2 \tag{19}$$

since $\frac{r_1}{R}$ and $\frac{r_1}{R}$ are the percentages of the substances present.

It should be noted that this equation may be written

$$\frac{1}{\mu'} = a \cdot \frac{1}{\mu_1} + b\frac{1}{\mu_2} \tag{20}$$

The above analysis is strictly true only in the case of an infinite number of layers. The exact equation is

$$\varphi = a\varphi_1 + b\varphi_2 + \frac{ab}{r}(\varphi_1 - \varphi_2) \tag{21}$$

Equations (19) and (20) are strictly applicable to the disc type of viscometer in which the actual velocity u is measured.

In homogeneous mixtures, the two equations lead to the same result and experiments with disc and efflux viscometers indicate that this is usually the case in fluid mixtures.

Bingham's conclusions regarding mixtures are as follows:

1. The viscosity of a thorough mixture of chemically indifferent fluids must always be less than would be expected on the assumption that viscosities are additive.

2. The viscosity of an emulsion must be greater than that of a perfect mixture of the same concentration.

If the solid sheets are broken up into fragments, the deformation of the liquid would tend to change the form of the cube into that of a parallelopiped

but the solid cannot be deformed so that the shearing force can only rotate the cube around its center. The failure of the solid to change its shape with the flow of the liquid will necessitate transverse motions in the liquid by way of readjustment, hence the viscosity of a suspension will always be greater than it would be if the fluidities were strictly additive. If the amount of transverse motion in a suspension is proportional to the number of suspended particles of a given size and for each particle the amount of transverse motion bears a constant ratio to the amount of shear, it will follow that the fluidity curves of suspensions must be linear.

4. Fluidity and Temperature, Volume and Pressure.

a. Temperature. For liquids, the fluidity-temperature curves should be straight lines if the fluidities of a mixture are additive ($T = A\varphi$). Bingham found that the fluidity-temperature curve can be represented by an equation of the form

$$T = A\varphi + C - \frac{B}{\varphi + D}$$

This equation represented the experimental results on eight substances with a maximum percentage difference of 0.07 per cent. The fluidity of liquids generally increases with temperature.

The viscosity of a gas generally increases with temperature. Sutherland has developed a formula which agrees very well with data on gases but is somewhat less suitable for vapors

$$\mu = \frac{1}{\varphi} \alpha \frac{T^{\frac{3}{2}}}{1 + \frac{C}{T}}$$

If the constant C is small as compared with the absolute temperature, $\mu = KT^{\frac{3}{2}}$ which agrees with the results indicated by the kinetic theory.

b. Volume. For liquids, a decrease in pressure or an increase in temperature is usually associated with an increase in fluidity. In other words, an increase in volume tends to increase fluidity.

Batchinski found that the relation between fluidity and molecular volume is $\varphi = C(V - \pi)$ where V is the molecular volume and π and C are constants. The constant π can be defined as the limiting value which the molecular volume of any liquid can have as its fluidity approaches zero. The difference $(V - \pi)$ may be called the molecular free volume.

5. Diffusional and Collisional Viscosity.

Bingham objects to the assumption that the viscosity of a liquid results from an attraction between the molecules because any retardation by attraction of molecules behind will be compensated for by acceleration by molecules ahead. He offers the following explanation:

a. Collisional Viscosity. In a very viscous liquid, the molecular free path is so greatly reduced that diffusion between adjacent layers is comparatively slight whereas the volume of the molecules themselves is a considerable portion of the total volume of the liquid. Given a layer of molecules C whose translational velocity is higher than that of another layer D,

collisions must occur between the two layers due to the flow. On collision, the translational velocity is partly communicated to the slower moving molecules of layer D so that the molecules of the layer D have a mean resultant velocity in the direction of flow, the remainder of the translational motion being converted into disordered motion or heat. When the system has reached a steady state, any layer D imparts to the layer E below it the same amount of translational momentum that it has received from the layer C above it, except for the amount of energy which is being continually changed into heat and it is this disappearance of translational momentum which gives rise to the new type of viscous resistance known as collisional viscosity.

It is a confirmation of the above conception that the fluidity is decreased in just the proportion that the concentration is increased either by lowering the temperature or raising the pressure.

In fluids at ordinary temperatures and pressures, the viscous resistance is evidently the sum of the diffusional and collisional resistances.

6. Colloidal Solutions. A small percentage of a colloid may completely change the flow characteristics of a liquid and the viscometer is an important means of recognizing the colloidal state. One per cent of agar agar will give water the properties of a stiff solid.

There are two types of colloidal structures.

1. Soap Gels. Structure requires time to form and the fluidity at a given moment depends upon the previous history of the solution.

2. Suspensions such as graphite in oil in which the forces bringing about the setting of the gel are absent.

Taking only the simplest case of a suspension of relatively large spherical particles, the dissipation of mechanical energy or head is somewhat as follows:

Spheres in the same stratum of liquid have the same velocity and the same direction of rotation and do not touch, but spheres in adjacent strata have different velocities and collisions will take place with a frequency depending upon the radii of the spheres, their number per unit volume, and upon any attractions or repulsions which may exist between them.

The rotation of the spheres will have the same sense, and hence the surfaces coming in contact will be moving in opposite directions and the viscous resistance in the liquid between them will equalize their velocities and dissipate their rotational energy as heat. The contact of the two particles brings the laws of ordinary friction into play and the spheres will not rotate relative to each other unless the torque exceeds a certain value. The pair of spheres rotate for a time as a unit and if during this time additional particles are acquired, the clots may increase in size until they fill the tube and at this point viscous flow of the material as a whole stops and plastic flow begins.

Definitions

The following glossary and list of definitions relative to consistency and plasticity will be found helpful to those interested in the manufacture of

lubricating greases. They were suggested by a special committee of the Society of Rheology.

CONSISTENCY

Consistency is that property of a material by which it resists permanent change of shape and defined by the complete flow-force relation. If in non-turbulent flow, the ratio of flow to force is constant, the material is said to be fluid; otherwise it is plastic

FLUID

A substance which is continuously deformed under any finite shearing stress, so long as the stress is maintained. Specifically a gas or a liquid.

FLUIDITY

The measure of the rate with which a fluid is continuously deformed by a shearing stress. The property of flowing freely. Quantitative.—The fluidity is measured by the relative velocity, in centimeters per second, imparted to either of two parallel planes, one centimeter apart, by a shearing stress of one dyne per square centimeter, the space between the planes being filled with the fluid. The unit of measurement is the rhe.

VISCOSITY

The measure of the resistance to continuous deformation in a fluid. The property of fluids by which they resist continuous deformation, especially characteristic of thick liquids like molasses or pitch. The reciprocal of fluidity. Quantitative.—The viscosity is measured by the shearing stress in dynes per square centimeter required to move either of two parallel planes, one centimeter apart relative to each other, with a velocity of one centimeter per second, the space between the planes being filled with the fluid. It is measured in poises, or preferably in centipoises, the centipoise being the approximate viscosity of water at 20° C. (1.005 cp.).

VISCID

Having a high viscosity. Opposite of fluid.

SOLID

A solid, in contradistinction to a fluid, is a substance which will continuously resist deformation provided that the shearing stress is below a certain value, which is called the yield value. At shearing stresses below the yield value, the deformation consists of the temporary elastic deformation and the permanent pseudo-plastic deformation.

PLASTICITY

Plasticity is that property which enables a material to be permanently deformed without rupture under the application of pressure, provided that the ratio of flow to force is variable.

(NOTE.—A solid is plastic when it can be readily molded into a desired form, to cause flow. The shearing stress must exceed the yield value. Liquids do not exhibit plasticity because they do not retain their shape and therefore cannot be molded advantageously. Plasticity may not be defined quantitatively because it is a complex property made up of yield value and mobility or their equivalent.)

YIELD VALUE

The minimum shearing stress in dynes per square centimeter required to produce continuous deformation in a solid. One of the quantitative factors in plasticity.

MOBILITY

The measure of the rate with which a solid is continuously deformed after the yield value has been exceeded. The second quantitative factor in plasticity. Quantitative.—The mobility is measured by the velocity, in centimeters per second, given to either of two planes, one centimeter apart, by a shearing stress of one dyne per square centimeter in excess of the yield value, the space between the planes being filled with the plastic material. Mobility is a more general term than "fluidity," the latter being the mobility when the yield value is zero.

STIFFNESS

The measure of the resistance to continuous deformation in a solid in excess of the yield value. The reciprocal of mobility. Characteristics of glutinous substances such as grease, glue, rubber, etc. Quantitative.—The stiffness is measured by the shearing stress, in dynes per square centimeter in excess of the yield value, required to shear either of two parallel planes, one centimeter apart, with a velocity of one centimeter per second, the space between being filled with the plastic material. Stiffness is a more general term than viscosity, the latter being the stiffness when the yield value is zero.

ELASTICITY

When substances have been compressed they tend to recover their volume as soon as the compression is removed. All fluids have perfect elasticity of volume. Elasticity of shape refers to spontaneous return of a substance after deformation to its former shape. The term is used in two distinct senses. According to one, a substance is highly elastic which is easily deformed and quickly recovers; that is, it depends upon the amount of the deformation or recovery. According to the second usage, that substance is the most elastic which most completely recovers its shape after deformation. Elasticity should be measured according to the first definition, and the lack of perfect recovery used to calculate the plasticity. Quantitative.—Elasticity of volume is measured by the compression in cubic centimeters of one cubic centimeter of material by a pressure of one dyne per square centimeter. The elasticity of shape is measured by the displacement in centimeters given to either of two parallel planes, one centimeter apart, by a shearing stress of one dyne per square centimeter, the space between being filled with the elastic material. The reciprocal of rigidity.

RIGIDITY

The property of bodies in which they resist an instantaneous change of shape. The reciprocal of elasticity. In physics a rigid body is assumed to be not only rigid but stiff as well. Quantitative.—The rigidity is measured by the shearing stress required to displace elastically either of two parallel planes, one centimeter apart, to the extent of one centimeter, the space between the planes being filled with the elastic material.

COHESION

The force, molecular in origin and perhaps the resultant of the attractions and repulsions between the electrical charges, which causes the particles of a substance to attract each other and tend to stick together. Tensile strength and surface tension are related, but not an exact measure.

ADHESION

The force which causes the particles of unlike substances to attract each other and tend to stick together. Methods have been proposed for measuring the adhesion between solids and liquids, that is, their wetability.

STRENGTH

The maximum stress required to overcome the cohesion of a material. Quantitative.—A complex property made up of tensile strength and shearing strength. The force required to break a bar of unit cross-section under tension; that is, the tensile strength, depends not only upon the cohesion but also upon the consistency and, therefore, at least to some extent, upon the rate of application of the load. Strength involves the idea of resistance to rupture, while hardness involves resistance to deformation. Distinction may be made between tensile strength and compressive strength.

LENGTH

Length is a quality possessed by a solid having a low yield value and low mobility, ductile. A long substance has the characteristics of a very viscous liquid. It is capable of being drawn out into ropes, threads, or fibers. Compare shortness, toughness, and tackiness.

SHORTNESS

Shortness is a quality possessed by a solid which has a high yield value and high mobility, non-ductile; for example, butter. A short solid "necks down" rapidly and shows little tendency to be drawn out into threads. Emulsions are all short. The opposite of length. Cohesion is an essential factor, and the mobility is low.

TOUGHNESS

The ability to withstand large and sustained shearing stresses without yielding. Toughness involves low mobility (or fluidity) and adequate yield value. Automatically, a certain cohesion is involved. See also tackiness, length, and shortness.

TACKINESS

Stickiness. A quality possessed by a solid having a low yield value and high mobility, by means of which contact readily results in adhesion; for example, glue, varnish, printer's ink, and gold size under working conditions are tacky or sticky substances. When a material dries out, sets up or gels, or hardens, due to chemical or other change, it loses tack or stickiness. Compare length, shortness, and toughness.

HARDNESS

A property applied to solids and very viscous liquids to indicate solidity and firmness in substance or outline. A hard substance does not readily receive an indentation or impression. It is unyielding to a bruising, cutting, or penetrating instrument. In viscous liquids the hardness is most appropriately measured by the viscosity, but the hardness of solids must be measured by the two properties of yield value and mobility and there must be different kinds of hardness. Being a complex property, it is unfortunate that most of the methods in use attempt to express this property by a single numerical value. But it is, at any rate, recognized that the methods in use do not measure the same kinds of hardness.

In grading minerals the hardness is based upon the ability of one mineral to scratch another. This scratch hardness involves the factor of cohesion. The "scleroscope hardness" test depends upon the amount of rebound of a steel sphere dropped upon the surface to be tested and involves the factor of elasticity. Since very viscous liquids like pitch with complete absence of yield value and low tensile strength may yet possess a certain form of hardness, it seems reasonable to assume that a low mobility is essential and distinguishes hardness from toughness, since a tough substance may be deformed whereas a merely hard substance may, or may not. Rigidity is sometimes regarded as a form of elastic hardness.

SOFTNESS

The opposite of hardness. A soft substance is easily deformed permanently without fracture. Contrast with brittleness. It is not identical with weakness; for example, a soft pencil lead may possess considerable strength.

PASTY

Having the consistency or appearance of paste. The latter use is not scientific as, for example, a pasty complexion. Applied to a rather short material.

GLUTINOUS

Having the consistency of glue, applied to a long, a stringy substance.

LIVERY

Having the consistency or the appearance of liver. The latter use is unscientific. Applied to certain materials, such as paints, which sometimes develop an excessive yield value on standing.

GREASY

Like grease or oil. Smooth, seemingly unctuous to the touch. Slippery. Talc and graphite have a greasy feel. The property seems to be caused by the layer of viscous fluid, or soft solid particles, which forms the lubricating film between the two surfaces sliding over each other.

LEAN

Non-plastic, lacking the ingredient required to give the desired yield value; for example, pastry is lean when it lacks sufficient lard, that is, shortening.

FAT

Very plastic or capable of becoming so on the addition of a dispersing agent. Applied to clay for example.

SLIPPERY

Applied to a surface over which objects readily slip or slide. A smooth and well-lubricated surface is slippery. Ice is more slippery than glass, presumably due to the film of water acting as a lubricant.

LUBRICANT

A substance used to reduce friction or making surfaces slippery. The lubricant tends to eliminate the effects of asperities of the surfaces, to prevent adhesion between the surfaces and to introduce a material, either solid or fluid, of sufficient consistency to maintain a film which can be easily sheared. The opposite of abrasive.

BODY

The appearance given by consistency, density, or opacity, of possessing much of the substantial or valuable ingredient; for example, wine has body due to color; silk due to its weight; lubricating oil due to viscosity; and paint due to the use of a blown, that is, a bodied oil or of a soap such as aluminum stearate, which produces a false body. The bodied oil gives a low mobility and false body results in a high yield value.

SOLUBILITY

The conventional definition of solubility is the percentage of dissolved substance contained in a saturated solution at a given temperature. But the term cannot be given the conventional definition when it is applied to greases for the following reason: Greases are quite different from ordinary solutions in that they do not obey the laws of true solutions. Colloidal solubility may be defined as the percentage concentration of colloid (soap) required to give a yield value when brought into colloidal solution at a given temperature. Generally speaking, the colloidal solubility increases with the temperature.

PLASTICIZER

A substance which lowers the consistency of a mixture. Some substances are of too high a consistency to act as colloidal solvents, even though they are able to disperse the colloids. Thus camphor converts nitrocotton into a plastic mass rather than a colloidal solution. It is, therefore, known as a plasticizer.

MELTING POINT

The melting point is the transition point between solid and liquid phases. The melting point or the softening temperature of a colloid does not have the conventional meaning given above, since it does not refer to a change of state but to a more or less rapid change in consistency. When a pure liquid like ethanol is cooled without crystallization, it becomes more and more viscid and finally vitreous, but it is quite arbitrary to speak of a definite solidifying temperature. With colloids, however, there appears to be a temperature at which the yield value appears and this may be regarded as the analogue of the melting point. The melting temperature and the solidifying temperature should be identical. In a colloidal solution the temperature of incipient melting and complete melting may, of course, be quite different.

WORKABILITY

Certain substances change in consistency during the period of application or of manufacture; for example, plaster suffers an increase in consistency as it is being applied to the walls, due to water absorption. A material shows the property of workability which is readily manageable during the operation in question.

THICKEN

To increase the consistency of a material. Opposed to thin.

INSPISSATE

To increase to a desired consistency, thicken. Opposed to temper.

TEMPER

To reduce or bring to a desired consistency, plasticity, softness, or tractability, as by adding solvent, dispersing agent, deflocculating agent, by stirring, mixing, kneading, pugging, grinding, etc. The hardness of steel is tempered by heating.

CUT

The physical action by means of which a material is divided or severed. The action of cutting is partly plastic flow, but also a breaking apart by the wedging or prying action of the knife or tool. In general, the less the factor of plastic flow has in the process, the less will be the energy used up in the operation of cutting. This

efficiency is often gained by lowering the angle of the wedge, by lubrication of the cutting edge, or by reducing the consistency of the material.

BRITTLENESS

The liability to break without appreciable deformation. Opposed to toughness. A material only exhibits brittleness as the shearing stress exceeds the cohesion of the material. A brittle material may be either solid or liquid but it has low cohesion and low mobility. Spun glass is brittle to the extent of being friable, that is, easily crumbled; but a spider's web is fragile and not brittle.

DUCTILITY

The capacity for deformation or extension without rupture, by drawing or other process involving tension. A property of low yield value, fairly low mobility with adequate cohesion.

Pressure Viscosity at Constant Shearing Rate

It will not be possible in this section to present details for constructing and operating constant shear type pressure viscosimeters. The one developed by Arveson [13] is quite elaborate and the technic chiefly adapted to research work. Such an instrument may be constructed at a cost of $700 to $1500. Arveson has pointed out that the apparent viscosity of lubricating greases are not constant values but vary with the shear rate. It is evident that, in bearings operating at fairly high surface speeds, there is sufficient breakdown in the structure of the grease as to liberate sufficient oil, or the products of the disrupting forces are of such low viscosity, that the bearing is lubricated in fairly close agreement with the theories of fluid film lubrication, and, as previously stated, the viscosity of this portion of the grease is only a fraction of that determined at low shearing rates, as in the constant pressure viscosity test made at fairly low pressures.

Arveson gives the following shear rates for a two inch shaft mounted concentrically in its bearing:

Clearance (Inches)	Rate of Shear (Reciprocal Seconds)
.01	18,800
.001	188,000
.0001	1,880,000

In the Arveson viscometer, the shear rate is fixed and for a given capillary tube the pressures developed at the entrance to the tube are measured by means of a mercury column.

A small piston is driven mechanically downward at a constant rate into a cylinder filled with the grease being tested. The grease is forced downward onto the mercury bed which is held in an annular space around the entrance of the orifice tube. When equilibrium is established, there is no flow of mercury through its tube to the manometer and the flow of the grease is through the capillary. The rate of flow is predetermined by selecting the gears which are used for driving the piston at a constant rate of speed.

For this viscometer, the apparent viscosity is given by the following Poiseuille equation:

[13] *Ind. Eng. Chem.*, **24**, 71 (Jan., 1932)

$$n_a = \frac{\pi PR^4}{8LV/t} = \frac{\frac{PR}{2L}}{\frac{4V/t}{\pi R^3}} = \frac{F}{S}$$

in which

F is stress in dynes per cm.^{2}
S is shearing rate in reciprocal seconds;
n_a is the apparent viscosity in poises;
P is pressure in dynes per cm.^{2}
R is the radius of the capillary in cm.;
L is the length of the capillary in cm.;
V/t is the rate of efflux in cm.^{3} per second.

In brief, the results of Arvesons work on lime base cup greases demonstrated that as the rate of shear increases, the apparent viscosity approaches a value somewhat greater, but of the same order, as that of the oil content of the grease. That this is true, has also been pointed to by results of starting torque and temperature rises in tests on ball bearings filled with greases, which, at equilibrium conditions, were of the order of the oils used in manufacturing the greases tested.

Shear Rate

The term shearing rate as applied to plastic material such as greases may be confusing, so that some explanation may be needed:

$$\text{Rate of shear} = \frac{4V/t}{\pi R^3}$$

in this equation V/t is the efflux rate in cm.^{3} per second and R is the radius of the capillary tube in cm. The unit of shear measurement is reciprocal seconds. For example, if grease is passing through a $\frac{1}{8}$-inch (0.32 cm.) tube at a rate of 10 c.c. per second, then the shear rate is found by substituting in the above equation as follows:

$$\text{Rate of shear} = \frac{4 \cdot 10}{\pi 0.16^3} = 3110 \text{ reciprocal seconds.}$$

Results of Viscosity Determinations at Constant Shearing Rates

Tests on a series of cup greases indicated that (roughly) the following apparent viscosities were obtainable at various shearing rates as shown in the table:

	Apparent Viscosity in Poises				
Percent Soap	5	10	15	20	25
Shear 0.1 sec.$^{-1}$	1000	10000	60000	90000	....
Shear 1.0 sec.$^{-1}$	300	3000	9000	12000	50000
Shear 100 sec.$^{-1}$	10	80	300	600	800
Shear 100,000 sec.$^{-1}$	2	5	6	8	10

Such data is of interest in selecting the grease of the most desirable soap content to meet a given shear condition, or the converse. In one instance, it was desired to know the increase in pressure necessary to main-

tain a given shear rate in a grease distributing system, with an increase of 3.5 per cent in the soap content. Calculations indicated the following:

	Original	Modified Case
Percent soap content of grease	10.1	13.6
Shear, sec.$^{-1}$	100	100
Apparent viscosities at S = 100	52	145
Pounds per square inch	200 (known)	560 (found)

GERMAN CONSISTOMETER

German investigators realizing that pour points and dropping points of greases throw little light on the nature of their consistency, have developed a constant shear rate consistometer. Since the relation between dropping point and consistency is not very well defined it is necessary, and important, that methods of determining grease viscosities at various temperatures should be developed and used. With the German tester an idea is obtained of the approximate ratio between cohesion and adhesion of the various greases. The phenomenon is often observed, when lubricating with grease, that one grease during lubrication or passage through a bearing becomes quite thin and soft and runs rapidly out of the bearing, while another grease of the same kind and chemical composition, with the same drop point, softens much less and holds longer in the bearing. This effect is evidently connected with the change of the greases as a result of pressing and mixing.

This consistency meter has been designed and constructed for testing under these conditions.

It consists of a press cylinder 28 mm. in diameter in which a piston moves, the length of the stroke of which is 60 mm., so that for filling, about 38 grams of grease is required. The piston, equipped with a leather cup, is moved backwards and forwards by means of a spindle traveling in axial direction. The hollow spindle (allowing for the insertion of a thermometer) is driven by an electric motor turning a worm and worm wheel at a speed of 15 revolutions per minute and effecting the stroke of the piston in about 100 seconds. The press cylinder is equipped with a manometer indicating up to 50 atmospheres. The press cylinder is jacketed so as to be able to test the sample at any desired temperature by circulating steam or hot or cold liquids through the jacket.

The press cylinder has an interchangeable nozzle in which a tube of 150 mm. length with a bore of 4 mm. in diameter is inserted. In this tube interchangeable needles may be placed so as to be able to utilize the apparatus for substances of different consistency or even for liquids. It is evident that in comparative tests the same needle must be used. The nozzle, which can be unscrewed, is equipped with a manometer indicating the pressure which results mainly from the adhesion of the moving grease from the place of the manometer to the free end of the tube. The principal manometer on the press cylinder indicates the total pressure required for deformation, and movement of the grease in the discharge tube.

A wattmeter is connected in the circuit of the electric motor for driving the consistometer; this wattmeter, on the forward stroke of the piston, indicates the power required for pressing the grease forward and for overcoming the mechanical and electrical resistances of the entire apparatus. Since the mechanical and electrical resistances are practically constant (the no-load power may be determined before and after the test) this constitutes also an electrical measurement of the consistency.

The consistency meter is used as follows:

With the nozzle removed the piston is moved up by the electric drive. A gun with a large opening filled with the grease to be tested, by means of a trowel, is screwed on the end of the press cylinder and the piston of the press cylinder is then moved

back. This is done by hand by means of a gear on the shaft of the electric motor. Simultaneously, the piston of the gun is pressed home according as the piston of the press cylinder is moved back, so that the grease fills the press cylinder entirely and no air pockets are formed. The grease gun is then unscrewed and the nozzle with manometer is screwed on. By means of a push button the motor is then switched in and the grease pressed forward by the electric motor at a uniform speed. The maximum pressure on both manometers is recorded and at the same time the maximum watt consumption.

Two kinds of tests at normal temperatures may be made, one test with fresh samples, in which the grease is not used again, and another test with the used sample, in which the grease forced out once, is forced out again a predetermined number of times, or until a steady state is attained.

With fresh samples, the degree of uniformity of working of the consistency meter is quite apparent. The structure and consistency of certain samples of grease are not uniform. Especially a stringy sodium base grease is in itself not uniform and, as is known, a separation or rearrangement of the soap and oil components of the majority of greases takes place after these have been left to stand for a longer or shorter time, which sometimes leads to distinct separations of oil. Consequently, it is advisable to mix the grease by hand before starting the test, to be sure that soap and oil are to a certain extent uniformly distributed in the grease.

In regard to the testing of the same sample, the following points are noteworthy: the considerable change of the consistency as a result of repeated forcing out; the change in the ratio of the pressures in the press cylinder and discharge tube; the appreciable change of the consistency and the adhesion as measured at the discharge manometer.

Results Obtained with the German Grease Consistometer

Typical results obtained with the German Constant Shear Consistometer, are shown in the following table, for nine commercial cup greases. The pressures for the original grease and those for the same grease after passing through the device ten times are recorded.

Grease No.	Measurement	Manometer 1	Manometer 2	Difference Between Manometer 1 and Manometer 2	Ratio Manometer 1 / Manometer 2	Difference 1-10 Measurements Manometer 1	Difference 1-10 Measurements Manometer 2	Penetration Unworked	Penetration Worked	Penetration Difference Worked and Unworked
1	1st	10.6	6.2	4.4	0.58	5.8	3.2	211	...	..
	10th	4.8	3.0	1.8	....					
2	1st	10 3	5.8	4.5	0.56	6.0	2.9	212	235	23
	10th	4.3	2.9	1.4	....					
3	1st	8.7	4.9	3.8	0.56	5.9	2.9	212	238	26
	10th	2.8	2 0	0.8	....					
4	1st	12.7	8.2	4.5	0.64	8.7	5.5	212	256	24
	10th	4.0	2.7	1.3	....					
5	1st	9.0	5.3	3.7	0.59	6.3	2.8	224	252	20
	10th	3.3	2.5	0.8	....					
6	1st	13.7	8.9	4.9	0.64	5.3	4.9	198	226	28
	10th	8.5	5.0	3.5	....					
7	1st	9.5	5.4	4.1	0.57	7.2	3.5	206	261	55
	10th	2.3	1.9	0.4	....					
8	1st	14 2	9 6	4.6	0.68	4.1	3.6	200	231	31
	10th	10.1	6.0	4.1	....					
9	1st	11.8	7.2	4.6	0.64	5.0	3.1	200	238	38
	10th	6.8	4.1	2.7	....					

Manometer 1: Pressure on grease at inlet of orifice in Kg./cm.^{2}
Manometer 2: Pressure on grease at orifice outlet in Kg./cm.^{2}

Other results on a No. 4 Fiber Grease are shown in the table:

Watts (Motor Input)	Pressures (Kg./cm.²) Inlet	Outlet
	Sample tested repeatedly	
112	15.3	8.8
111	14.6	8.2
111	13.1	7.8
110	12.0	7.4
109	12.0	7.0
108	11.8	7.0
106	11.2	6.8
105	10.0	6.4
104	9.6	5.9
104	9.2	5.2
	Six check tests on fresh samples	
111	15.5	9.1
111	15.3	8.7
111	15.0	8.5
112	15.2	8.5
111	15.0	8.6
110	15.2	8.5

Note: All tests made at room temperature. The diameter of the needle placed in the 4 mm. orifice is 3 mm.

Pressure Viscosity at Constant Pressure

This method of testing lubricating greases is not at all new. Prior to 1912, Kunkler had standardized a consistency test for greases [14] in which the sample was placed in a vertical viscometer tube, 30 mm. in diameter and 150 mm. long, fitted with a small jet, or orifice, at the bottom. The tube was provided with a sliding piston for forcing the grease through the orifice and could be so loaded as to weigh 50, 100 or 200 grams. The time required for the piston to drop a specified distance (forcing out of the orifice a definite amount of grease) with a given loading was taken as a measure of the consistency.

In 1921, a similar device was in use by the Buick Motor Company; the tube was $1\frac{1}{8}$ inches in diameter, $3\frac{1}{2}$ inches long, and the orifice was $\frac{1}{8}$ inch in diameter. For summer cup grease, the "consistency" was specified at 2.25 to 2.75 pounds, and for winter cup grease, the range was 1.75 to 2.25 pounds. The test was conducted as follows:

Sample to be at a temperature of 65 to 75° F. Fill the chamber with the grease to be tested and screw on the cap. Place the apparatus on an iron tripod so that the plunger is in a vertical position and push the plunger down about one inch to insure representative pressure conditions. Then, wipe off the grease at the bottom of the outlet orifice and apply weights to the top disk until the grease just starts to flow through the orifice. This "flow" should just be noticeable after the proper weight has been applied for one minute.

The applied weight plus the weight of the plunger (or moving portion of the apparatus) expressed in pounds is taken as the body of the grease.

Care should be taken to have the plunger of the apparatus strictly in a vertical position in order to avoid any binding or side pressures.

[14] Archbutt and Deeley, "Lubrication and Lubricants," 3rd ed., page 193.

The ordinary range of room temperatures seems to have little or no effect on the results obtained by this method.

The body of a grease is changed by its standing in a container which absorbs oil (e. g., pasteboard boxes, etc.). Care should be taken to avoid any errors due to this source.

Porter and Gruse [13a] have reported the use of the plastometer for determining the consistency of cup greases. For more detailed information than is presented the reader is referred to the original papers. By means of the plastometer the time rate of flow of the grease through an orifice of definite size may be determined at different pressures. Consistency may be defined as the "degree of solidity" of a grease. The consistency will determine the rate of flow or resistance to motion under any given condition.

With all true fluids, like oils, consistency in the above sense is defined completely by viscosity. Where we are dealing with stream line flow, if the pressure inducing flow is increased or reduced, the flow rate will be proportionately increased or reduced. No matter how small the pressure, some flow will take place, at a rate proportional to the pressure. Because of this proportionality between pressure and flow, a viscosity determination can be made under any suitable pressure—for instance, under the gravity head of the liquid, as in commercial viscosimeters such as the Saybolt instrument. This single determination will permit predicting what the flow rate will be in the same apparatus under any pressure.

As previously mentioned, this simple relation between pressure and flow rate does not hold for greases. At small pressures, as under gravity head in an ordinary viscosimeter, no flow might take place with a certain grease. The conclusion might be that the grease had infinite viscosity, that is, it would give an enormous resistance to motion. If, however, the grease is subjected to a gradually increasing pressure a point will be reached at which it begins to flow, and from there on the flow rate will rapidly increase. To obtain, therefore, the relation between the pressure and flow rate for a grease, tests will have to be made at more than one pressure. A determination at a single pressure in the case of greases would not permit predicting the flow rates at other pressures.

Thus it is seen that an oil is a viscous liquid, that is, a body in which the smallest stress (shearing force) will cause a constantly increasing change of form. The ratio between stress and flow is measured by viscosity. A grease, on the other hand, is a plastic solid, that is, a body in which no continuous alteration of form is produced by stresses up to a certain minimum value. This minimum value has been termed by Bingham "internal friction," and by others "yield value." If the stress exceeds the minimum value referred to, flow will take place at a rate which is proportional to the excess of shearing force above the minimum required to start flow. Thus, if we consider the excess of force only, we have the same relationship between pressure and flow as obtains with an oil. This relation between pressure and flow is defined in the case of an oil as viscosity, or its reciprocal fluidity. With plastic solids such as greases, the similar relation between excess pressure and flow is generally defined as mobility.

[13a] *Ind. Eng. Chem.*, **17**, 853 (Sept., 1925).

PRESSURE VISCOSIMETERS (CONSTANT PRESSURE TYPE)

The Saybolt Furol instrument is of little value in determining viscosities at low temperature, and pour test data are of practically no value at all, in deciding whether or not a lubricant will function properly at low temperatures.

The most satisfactory, as well as accurate, method of determining the viscosity of lubricants at any temperature from —30° F. to 210° F. is by means of the Pressure Viscosimeter.

Instruments of varying design have been employed by a number of experimenters who also do not follow the same methods of operation. In basic principle however, all instruments are very similar in that pressure is used to force the lubricant through an orifice.

The Sinclair Pressure Viscosimeter. The tube is mounted in an insulated cabinet the temperature of which can be held at temperatures ranging from −25° F. to 225° F.

The tube is a nickeled steel cylinder (2 inches diam. × 8 inches long) the top cap of which is connected to a ¾-inch pipe communicating with a constant pressure air tank.

Accurate and convenient control of air pressure in the tank is accomplished by means of the two needle valves, in the supply line and relief pipe respectively. The stop and waste valve enables one to apply pressure to the lubricant in the tube, or it will cut off and instantaneously release the pressure within the tube.

The hinged cover is operated by lever and rod making it unnecessary to open the cabinet door during the test.

Method of Operation. About 100 cc. of the lubricant to be tested is brought to the temperature of the tests. Fill the tube to about half full (full when testing light lubricants at high temperatures) having the nickel tube screwed in place and holding the cover tight against the Saybolt orifice. Screw the tube in place, attach rod to cover and lever and connect the electric instrument to the nickel plug (or insert pour test thermometer). Bring the lubricant to the desired temperature and after a constant value has existed for 15 minutes, the determination may be made.

Open the stop and waste cock, gradually build up the air pressure in the tank and tube until the desired pressure is obtained, adjusting the pressure by means of the two needle valves. When the pressure remains constant for a minute and the thermometer still reads the desired test temperature, then the orifice cover is quickly opened by latching the lever, simultaneously snapping a stop watch. When the lubricant in the cup reaches about ¾ full, the pressure within the tube is suddenly relieved by throwing the stop and waste cock handle and simultaneously stopping the watch; after which the lever is unlatched, closing the orifice cover.

Remove the cup of expelled lubricant and replace with a clean cup, care being taken to avoid having the cabinet door open longer than necessary.

Check pressure and temperature and when both are again constant make a check run. Weigh the lubricant carefully and record to the closest gram.

The formula generally employed is the one developed by Parsons and Taylor, two pioneers in the work. It is:

$$\text{Pressure Viscosity, in Saybolt Seconds} = PV = \frac{31{,}500 \times \text{minutes} \times \text{pounds/sq. in.}}{\text{grams}}$$

THE BULKLEY GREASE CONSISTOMETER

Bulkley and Bitner [15] have developed a two compartment pressure viscosimeter which is of considerable value in indicating the breakdown of

[15] *J. Rheol.*, (3) **1**, 269 (April, 1930).

the grease structure due to working. The pressure is applied to the instrument by means of a leveling bulb and hose filled with mercury. There are two similarly constructed reservoirs for containing the grease under test, joined by a glass capillary tube through which the flow is measured. The two vessels are fitted with screw covers having suitable outlets for pressure connections. The capacity of each reservoir is about 25 cm.[3] The instrument is mounted in a constant temperature bath when in use. The mercury leveling tube is provided with a horizontal glass arm, and is used for measuring the volume of grease which flows, this being equal to the amount of mercury displaced during the test run. For the calibration methods used and details of manipulation the original paper by Bulkley should be consulted.

The following results were reported for a normal cup grease (No. 3) having a worked A.S.T.M. penetration at 77° F. of 241:

Temperature	Pressure grms./cm.[2]	Rate of Flow cm.[3] per Second
95° C.	100	0.06
	200	0.16
	300	0.26
90° C.	160	0.00
	200	0.04
	300	0.11
	400	0.19
	500	0.27
75° C.	400	0.005
	450	0.005
	500	0.010

English Constant Pressure Type Grease Consistometer

English investigators have developed a grease worker and constant pressure type viscometer for studying the breakdown of lubricating greases.

The grease worker itself consists of a metal cylinder 1 inch in diameter and 4 inches high mounted vertically on a brass plate and fitted with a screw cap carrying a plunger. The plunger takes the form of a steel rod bearing at its lower end a circular brass plate pierced with a number of $\frac{1}{16}$-inch holes and fitting into the cylinder with a clearance of about $\frac{1}{64}$ inch. The upper end of the plunger is connected to the driving system of a Geryk pump which is actuated by an electric motor.

The Consistometer takes the form of a glass tube 10 mm. in diameter and about 20 cm. in length, to one end of which is joined a piece of capillary tubing of 1.8 mm. bore forming a jet. The capillary is joined by heating and expanding one end and fusing on to the 10 mm. tube and has a length of 2.6 cm. not including the expanded portion. A line is etched around the tube 11.8 cm. from the open end of the capillary.

The jet fits tightly into a short test tube, or small flask, by means of a rubber bung which also carries an exit tube. A length of rubber tubing connects this exit tube with a three way tap which communicates to the air and to a manometer for measuring rates of flow of air. The manometer is filled with colored toluene and by means of suitable capillary tubes can register rates of flow over a very large range. The consistometer tube and the chamber into which the jet connects are immersed in a water bath for the control of temperatures.

The upper end of the consistometer tube is connected by means of rubber pressure tubing to a montejus (egg) which connects with a mercury manometer and is pumped up to the necessary pressure by means of a cycle pump.

Several tubes have been prepared and calibrated using the original tube as a standard. This calibration has been carried out using a high viscosity mineral oil.

In carrying out a test the consistometer tube is one-half to three-fourths filled through the wide end with the grease under examination by spatula, grease gun or suction, taking care to avoid air bubbles as much as possible. After filling, the grease is drawn down slowly to the lower end of the tube by gentle suction, the jet is inserted into the rubber bung and thus connected to the test tube or flask. The exit tube is connected up to the three way tap which is open to air and the apparatus then placed in the water bath which is maintained at 60° F. When sufficient time has elapsed for temperature equilibrium to be attained (usually one-half hour is allowed) the upper end of the tube is connected to the "egg" and air gradually pumped in until a pressure of 40 cm. mercury is indicated. As there is no tap between the tube and the "egg" the pressure on the grease rises gradually. If the 40 cm. pressure were turned on to the grease at once there would be a great liability for blow holes to result and erroneous results would follow.

With the rise in pressure the grease commences to flow through the jet. As soon as the desired pressure is reached, the manometer is connected to the consistometer and by fitting a suitable capillary a measurable difference in level is obtained. As the amount of grease in the tube lessens, the pressure being kept constant throughout the flow, the rate of flow tends to increase and for this reason the readings on the manometer are taken when the level of the material reaches the etched line. This ensures the same height of grease being in the tube for all experiments when readings are taken. Once the measurements have been recorded the consistometer is disconnected from the manometer and opened to air and the tube allowed to empty itself under the air pressure.

Having obtained the initial consistency of the sample, the worker is filled with the material, the cap screwed on and the motor started. By suitable gearing the wheel of the driving unit is run at 85 to 90 revolutions per minute, the plunger making one down and one up stroke per revolution.

After 1000 revolutions, working is stopped, a portion of the grease removed from the body of the apparatus and filled into the consistometer from which all traces of the unworked grease have been removed. After allowing time for the material to come to 60° F. the rate of flow is determined and as much as possible of the grease is removed from the consistometer and returned to the worker, the motor being then restarted. Further samples are withdrawn after 5000, 10,000, 15,000 and 20,000 revolutions and examined in a similar manner and curves can be drawn plotting rates of flow against number of revolutions.

NOTE (1). During the working of greases, some of the material collects between the top surface of the plunger and the lower surface of the screw cap. This material does not get properly worked and must not be taken for consistency tests. All samples for this test should be taken from the body of the worker.

NOTE (2). When the graduated capillaries are affixed to the manometer care must be taken that the rubber connections are absolutely tight otherwise low results will be obtained. Also the capillaries should be examined occasionally to make sure no blocking has occurred.

	Rate of Flow cc./min. at 60° F. Pressure 40 cm. Mercury			
No. of Revolutions	No. 4 Fiber Grease (Sodium Base)	No. 4 Sponge Grease (Sodium Base)	No. 4 Sponge Grease	Cup Grease No. 3
0	0.006	0.12	0.18	0.005
1000	10.6	2.5	0.035	2.9
5000	13.4	4.0	0.12	4.0
10000	14.5	5.0	0.15	4.5
15000	15.1	7.4	0.055	4.5
20000	16.0	21.8	0.055	4.7
Appearance after working	rather soft	soft and oily	Grease has stiffened up	rather soft
Appearance after 20 hours working in Hardy Spicer Universal Joint	Grease became yellowish emulsion		Good. Little change	

From the foregoing and following tests on sodium base greases, at various temperatures, it is evident that there is little relation temperature-viscosity curves and degree of working-viscosity curves.

Temperature ° F.	Rate of Flow cc./min. Pressure 40 cm. Mercury: No. 4 Fiber Grease Worked 100 Times	No. 4 Fiber Grease Unworked	No. 4 Sponge Grease Unworked	No. 4 Sponge Grease Unworked
60	6.4	0.005	0.01	0.24
80	16.5	0.08	0.08	0.77
100	39.4	0.35	0.72	3.5
120	....	3.28	....	17.6
140	....	5.7	9.5	30.0
160	....	14.7	25.9	....
180	....	>50.0	>50.0	....

Temperature ° F.	Rate of Flow cc./min. Pressure 40 cm. Mercury: No. 4 Sponge Grease Worked 100 Revolutions	No. 4 Sponge Grease Unworked	No. 4 Sponge Grease (German) Unworked
60	0.005	0.69	0.97
80	0.10	1.3	4.0
100	0.31	6.2	13.9
120	0.99	29.4	35.3
140	3.19	....	....
160	6.67	....	....
180	7.5	....	....
200	17.6	....	....

Stability to Heat

For over fifty years many heat tests have been proposed to indicate the stability of lubricating greases. These range from simply heating a sample of the grease in a small vessel, and noting the character of the grease on cooling, to more refined procedures in which the time and temperature conditions may be very accurately controlled and the extent of deterioration of the grease accurately measured.

English Heating Test

The test is carried out in such a manner that the grease is spread onto a tin lid until it is nearly full. Then this lid, filled with grease, is heated for one hour in a drying oven at 105 to 110° C. The grease should not show any oil separation after standing 24 hours.

This test is long, but not nearly as severe as the Timken test, and can be fulfilled by greases which are still slightly alkaline, especially autoclave greases.

Timken Grease Test

The 1933 Timken specifications distributed in England read as follows:

"25 grs. of grease are heated to 250° F. (121° C.) and held at that temperature for one-half hour. The grease is then poured into a silica tube and allowed to cool. When cool, the silica tube is suspended over a conical measure graduated in cubic centimeters in which the mineral oil which separates out in 48 hours is collected.

"The result of the test is expressed as a percentage separation on 100 grs. of grease.

"**Manipulation.** The grease is melted in the flash point cup and maintained at 250° F. for one-half hour. It is then poured into a silica tube 3 inches long and 1 inch inside diameter, one end of which has been previously sealed by means of a cork. When the grease solidifies the cork is removed and the grease supported by two pieces of flattened wire wound vertically around the tube. It is then placed in the mouth of a conical measure graduated in ccs. The separated oil after 48 hours × 4 = percent separation on 100 grs. of grease.

"Greases which show above 4 per cent separation should be rejected."

ENGLISH HEAT TEST T-1

20 to 25 grs. of grease was heated in a suitable container, the best being a Cleveland flash cup, stirring with a thermometer to the flash point of the mineral oil employed in the manufacture of this grease (This heating is eventually carried to 200° C.). Then, the fluid grease is poured into a larger watch glass and allowed to congeal. The specification is that, after 24 hours standing, no oil should separate. One can, however, in one to two hours determine whether the specifications are to be fulfilled or not. It is usually fulfilled in cases where the neutralization number of the grease is not over $2\frac{1}{2}$, corresponding to $1\frac{1}{4}$ per cent oleic acid. In such cases, no oil separation takes place after 24 hours if the surface of the grease forms a completely flat meniscus. If a considerably arched meniscus appears, which is comparable to that of mercury in a glass vessel, the grease will ordinarily separate appreciable oil.

Other heat tests are described in Chapter V, under the sections dealing with ball bearing greases.

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