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Infrared Absorption

OF INORGANIC SUBSTANCES

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To the Memory of
My Father
LEON LEWIS EMANUEL
(1901–1940)

Preface

Since 1952, an increasing number of detailed studies of the infrared absorption spectra of inorganic materials have appeared in the literature. In many of these studies characteristic frequencies which are useful in the analysis and identification of inorganic substances have been observed. In addition, some of the studies have afforded valuable information on the nature of the structures and of the intermolecular forces.

The present work represents a compilation and review of empirical data and interpretations of the infrared absorption spectra of inorganic substances. No effort has been made to cover the theory of spectra nor, in any great detail, the experimental methods, both of which have been treated extensively in the literature. Rather, it is intended that this volume should serve as a source book of spectral information for persons engaged in this phase of spectroscopy.

Part I summarizes current experimental techniques with reference to the controlling factors and cites the varied applications of infrared spectroscopic studies of inorganic materials. In Part II, as many correlations and assignments as are available from the experimental data of various observers are presented. The inorganic substances discussed have been grouped into four main cate-

gories based on the number of elements involved: elemental substances, binary compounds, ternary compounds, and compounds containing more than three elements. A glossary and an extensive annotated bibliography with index follow.

The author wishes to thank those who have assisted in the preparation of this work, and particularly Dr. G. A. Crosby, Dr. R. S. Claassen, Dr. F. P. Hudson, and Mr. H. A. Stein, who read portions of the manuscript critically. Thanks are also due to The American Chemical Society, The American Institute of Physics, The Faraday Society, The National Research Council of Canada, D. Van Nostrand Co., Inc., Pergamon Press Ltd. and *Physical Review* for permission to use certain copyrighted material. Finally, the author expresses appreciation to her husband for assistance and encouragement throughout the entire course of this work.

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Albuquerque, New Mexico

March, 1961

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PART I

Introduction

INFRARED ABSORPTION SPECTRA associated with various structural groups of organic materials have been so thoroughly studied that identification of many organic compounds and mixtures presents very little problem. This method of analysis has the advantages of being rapid and of requiring only small amounts of sample. The ease of solubility of organic materials in non-aqueous solvents has, to some extent, been responsible for the large volume of work which has been completed with these substances.

On the other hand, studies involving inorganic materials have not met with corresponding success. Until recently inorganic samples, suitable for analysis, were difficult, if not impossible, to prepare. Consequently, such data as were available on the vibrational spectra of inorganics were obtained primarily by Raman or reflectance methods. In the past six to eight years, however, techniques have been developed which yield discrete spectra resulting in a considerable increase in accumulated data. A number of valuable correlations are now available and fundamental studies on inorganic structures and intermolecular forces have been initiated. This section presents a survey of the usual techniques employed in infrared spectroscopy of inorganic substances and describes some of the many applications of the spectral data.

TECHNIQUES

Manuals, books, and review articles¹⁻⁷ containing extensive descriptions and discussions of instrumentation, and instruction books for the various commercial instruments, are available to the infrared spectroscopist. This section has, therefore, been confined strictly to methods of sample preparation and related factors.

Infrared absorption spectra may be obtained on gases, liquids, or solids. The practicality of infrared spectrophotometry as an analytical tool for gases is well established. Gases are particularly easy to investigate in the infrared region because concentrations are controllable by simply varying the pressure. In addition, spectra of gaseous materials are less affected by intermolecular forces than are those of solid and liquid samples.

The freedom of rotation of gaseous molecules generally introduces fine structure into the absorption bands as a result of rotational energy changes along with the usual vibrational energy changes. This frequently makes the spectra more complicated, but aids in the assignment of characteristic frequencies and consequently in identification of the molecule.

Special sampling techniques and cell construction for use in vapor-phase spectroscopy are described in the literature. For example, Taylor, Benedict, and Strong⁸ described a porcelain-lined

1. Harrison, G. R., Lord, R. C., and Loofbourow, J. R., "Practical Spectroscopy," Prentice-Hall, New York, 1948.
2. Jones, R. N., and Sandorfy, C., "Infrared and Raman Spectrometry: Applications," in A. Weissberger's "Technique of Organic Chemistry," Vol. IX, Interscience, New York, 1956.
3. Lord, R. C., McDonald, R. S., and Miller, F. A., *J. Opt. Soc. Amer.*, **42**, 149 (1952).
4. Robinson, D. Z., *Anal. Chem.*, **23**, 273 (1951); **24**, 619 (1952).
5. West, W., "Spectroscopy and Spectrophotometry," in A. Weissberger's "Physical Methods of Organic Chemistry, Technique of Organic Chemistry," Vol. I, Part II, 2nd Ed., Interscience, New York, 1949.
6. Willard, H. H., Merritt, L. L., and Dean, J. A., "Instrumental Methods of Analysis," 3rd Ed., D. Van Nostrand, New Jersey, 1958.
7. Williams, V. Z., *Rev. Sci. Instr.*, **19**, 135 (1948).
8. Taylor, J. H., Benedict, W. S., and Strong, J., *J. Chem. Phys.*, **20**, 1884 (1952).

absorption cell of the Pfund type capable of heating 3 meters of gas to temperatures above 500° C. Similarly, Randall, Greene, and Margrave⁹ described a simple cell for measuring the spectra of magnesium halides at 1000° C and nickel chloride at 850° C.

The liquid state of inorganic substances is seldom used in obtaining spectra, since few inorganics normally occur in the liquid state, and it is not usually convenient to transform a solid or gas to a liquid simply for the purpose of analysis. However, some progress has been made in the study of inorganic materials in solution. The usual solvent for inorganic determinations is water. Aside from the fact that water attacks the more common infrared cell materials, it also has a strong absorption at wavelengths greater than 1.5 microns. Cells of silver, barium, and thallium halides have made possible the measurement of infrared spectra of aqueous solutions. Non-aqueous solutions of inorganic substances are rarely used for infrared measurements, since solubilities of inorganic compounds in the common solvents are quite low. The current trend of molten salt spectroscopy accompanied by the newer designs of the necessary high-temperature cells will in all probability increase the number of spectral observations in the liquid phase. One notable advance in this direction is the platinum screen technique of Greenberg and Hallgren¹⁰ for the infrared study of fused salts. The salt under consideration is placed in a fine-mesh platinum screen which has been electrically heated to the appropriate temperature. The screen is then placed vertically into the spectrometer so that excess salt runs off, leaving a uniform film supported in the interstices. The technique has been used at temperatures up to 800° C in vacuum and in the atmosphere. Some of its advantages are: (1) the infrared radiation penetrates only the sample, not the cell material; (2) spectra can be obtained over varying temperatures; (3) the corrosive action of the salt at the high temperature does not appreciably affect the screen; and (4) elimination of moisture from the screen is relatively easy when

9. Randall, S. P., Greene, F. T., and Margrave, J. L., *J. Phys. Chem.*, **63**, 758 (1959).

10. Greenberg, J., and Hallgren, L. J., *J. Chem. Phys.*, **33**, 900 (1960).

compared to elimination of moisture from other matrix materials used in normal infrared studies.

The most extensively studied physical state for the examination of inorganic compounds via infrared spectroscopy is the solid state. Solids may be handled in a number of ways: powder film, mull, or alkali-halide pellet techniques are most frequently used. In the powder film technique a thin film of the finely pulverized sample is deposited upon a salt plate by evaporation of the liquid from a solution or suspension of the solid. The particle size is quite important, the optimum size being that which prevents excessive scattering and yet transmits adequately for measurement of the absorption. Generally, methods which are required to give the ideal particle size are rather complicated.

Hackskaylo¹¹ introduced a refinement of the film technique which produced well-resolved spectra, free of extraneous absorption bands which arise from the solvent or the dispersing medium. Solutions of soluble inorganic salts (e.g., potassium thiocyanate and ammonium heptamolybdate) were brushed onto salt plates which were heated to a temperature intermediate between the decomposition point of the compound and the vaporization temperature of the solvent. As the solvent evaporated, a thin film of suitable particle size remained on the plate. The technique was extended to insoluble compounds. Actually, in some instances, soluble compounds behaved more favorably if treated as insoluble ones. A slurry or thin paste is prepared by mixing the compound with a liquid of low vaporization temperature. The mixture is spread onto salt plates and the liquid allowed to vaporize. As the evaporation proceeds, crystals explode into smaller particles and leave a fluffy coating of solid on the plate. Excess solid is removed by smoothing over with another salt plate.

Thin crystalline samples of the alkali hydroxides have been prepared by melting a small amount of the compound between cleaved halves of an alkali halide plate and allowing recrystallization to occur after pressing the halves together.¹² The hydroxide

11. Hackskaylo, M., *Anal. Chem.*, **26** (9), 1410 (1954).

12. Wickersheim, K. A., *J. Chem. Phys.*, **29**, 1197 (1958).

forms a thin filler in a crystalline sandwich. The samples ranged in thickness from 10 to 30 microns. Samples of LiOH, NaOH, and KOH were coarse-grained with single crystal grains up to two millimeters in diameter. This structure was not exhibited by cesium or rubidium hydroxide. For all hydroxides except lithium hydroxide, moisture was excluded by sealing the edges of the plates. The formation of alkali carbonate was minimized by working in a carbon dioxide-free atmosphere. Interaction between the salt plate and the hydroxide was considered negligible.

The mull technique has found rather wide usage in infrared spectroscopy. The procedure involves grinding the sample with a viscous liquid, which has few absorption peaks in the region under study. The solid particles are suspended in the liquid medium, the refractive index of which is higher than that of air, in order that light scattering may be reduced to a minimum. The common mulling agents are mineral oil, perfluorokerosene, and hexachlorobutadiene. The problem of excluding moisture and carbon dioxide during the grinding process has been dealt with in a number of ways, among which is conducting the entire process in a cold, dry box or room. Erley¹³ proposed the use of 2,2-dimethoxypropane as a drying agent for samples which are slightly acid or are stable in a slightly acid environment. For such a mull, the grinding process will generally remove the products of the interaction between the drying agent and water, since they are volatile. One inhibiting feature is the fact that 2,2-dimethoxypropane is a combustion hazard when stored at room temperature and must be handled with extreme care.

More recently, the alkali halide pellet or disk technique has been applied to the infrared examination of inorganic compounds. The solid being investigated is ground to micron size with the alkali halide, usually potassium bromide, and the mixture pressed into a clear pellet or disk at room temperature *in vacuo* under pressures of about 20,000 pounds per square inch for 5 or more minutes. This technique was originally accepted with enthusiasm, since potassium bromide has no absorption peaks in the 2–25 micron

13. Erley, D. S., *Anal. Chem.*, **29** (10), 1564 (1957).

range. More discrete spectra were obtained, the pellets could be stored in a dry box for future reference, and the possibility of quantitative analysis was suggested. This early enthusiasm was soon curtailed as various investigators encountered anomalous behavior in the infrared spectra of inorganic samples prepared by the pellet technique.

Kalbus¹⁴ made an extensive study of the factors influencing the appearance of the spectra. Difficulties encountered when the alkali halide pellet technique was used were attributed to anion exchange between the inorganic compound and the alkali halide and to pressure or orientation effects on the sample as a result of compression. Some of the factors affecting the exchange were enumerated—ionic size and type, magnitude and time of application of pressure, particle size, grinding time, surface moisture, and sample solubility. Table 1 summarizes the data obtained.

The relative merits of the mull and pellet procedures were discussed in considerable detail by Baker.¹⁵ Although this work was concerned primarily with spectra of organic compounds, some of the findings may be extended to the field of inorganic spectroscopy. Baker suggested that discrepancies between spectra of organic compounds prepared by the two techniques are due either to an induced physical isomerization of the compound or to the fact that it becomes amorphous in the alkali halide pellet. Several factors influence these changes: the crystal energy of the organic phase; the energy of grinding of both sample and matrix; the matrix lattice energy and particle size; the ability of the sample to recrystallize in the pellet; and the relative stability of its polymorphic forms. Each of these factors is discussed at length and experimental data substantiating the arguments are presented. Despite the serious limitations of the pellet procedure, it possesses certain advantages over other methods of sample preparation. When used with caution and together with other techniques, valuable qualitative information can be obtained. In particular, steps

14. Kalbus, G. E., *Dissertation Abstracts*, 17, 2413 (1957). See also Melloche, V. W., and Kalbus, G. E., *J. Inorg. Nucl. Chem.*, 6, 104 (1958).
15. Baker, A. W., *J. Phys. Chem.*, 61, 450 (1957). See also literature citations therein.

TABLE 1. BEHAVIOR OF INORGANIC SALTS CONTAINED IN POTASSIUM BROMIDE PELLETS ^a(After Meloche and Kalbus ¹⁴)

Anion	Cation	Behavior
CO ₃ ⁻²	Normal: Ag, Sr, Cd, Co(III), Mn(II), Li, Fe(II), Mg, K, Na, Ca, Ba, Pb	No exchange
	Basic: Cu, Mg, Ni, Cr(III), Be, Zn	No exchange
NO ₃ ⁻	Ag, Ni(II), Cd, Mg, Co(II), Li, Bi(III), Zn, Fe(III), Hg(II), Pb	Exchange evident
	Na, Al, Ca, Cr(III), Cu(II), Sr, Ba, Th(IV), Hg(I)	No exchange
PO ₄ ⁻³	Anhydrous: K, Ca, Sr, Ag, Cd, Ba, Bi(III), Pb	No exchange
	Hydrated: Li, Na, Mg, Mn(II), Fe(III), Ni, Co(II), Cu, Zn	No exchange
SO ₄ ⁻²	Anhydrous: Hg(I), Hg(II), Pb, Cu(II), Ag	Exchange evident
	Hydrated: Cu, Zn	Exchange evident
	Anhydrous: Na, Sr, Ba	No exchange
	Hydrated: Mn(II), Co(II), Li, Mg, Ni, Ca	No exchange
CrO ₄ ⁻² , NO ₃ ⁻ , NH ₄ ⁺ MoO ₄ ⁻² , Cl ⁻ , Br ⁻ , I ⁻ CO ₃ ⁻²		No exchange ^b
SCN ⁻	NH ₄ ⁺	Bands of KHCO ₃ , not of K ₂ CO ₃ Exchange facilitated by CHCl ₃ ; eliminated in dry state

^a Samples of 0.8-1.5 mg. of the inorganic salt weighed to the nearest 0.05 mg. were added to 300 ± 30 mg. of potassium bromide.

^b Exchange from halides, if it did occur, could not be observed, since the absorption bands would appear too close to the original bands to be distinguishable.

which eliminate moisture and minimize distortions due to grinding and pressure yield clear-cut spectra which are not otherwise obtainable.

For some inorganic salts, particularly hydrates and hygroscopic materials, the elimination of moisture is extremely difficult, if not impossible. Tai and Underwood¹⁶ combined the technique of freeze drying with the KBr-disk method to obtain good spectra of a number of inorganic sulfates. An aqueous solution of the sample under study was placed in a tube which was rotated in a dry ice-acetone bath until a thin layer of frozen matter formed. The sample tube was then connected to a vacuum system and pumped-on for at least 12 hours. The water was removed rapidly enough to keep the solutions frozen without external cooling. The pumping time can be greatly reduced by warming the tubes after the first two hours, provided the sample is sufficiently heat-stable. After all the water was removed, the freeze-dried product was pressed into a potassium bromide pellet. Generally, prior to formation of the pellet, both the alkali halide and sample should be dried as completely as possible.

The particle size of the matrix must be small enough to obtain pellet transparency and to prevent too violent a grinding action on the particles of the sample. The particle size of the sample should be of the order of one micron. This ideal condition is difficult to attain by hand grinding. For most samples, Harshaw KBr (250–300 mesh) pulverized with the sample in a mechanical vibrator (such as the Wig-L-Bug amalgamator) yields an appropriate particle size. Although it is not absolutely necessary to dry the mixture after the grinding process, a minimum of contact with moisture should be permitted. Distortions of spectra due to amount of pressure and loading time are generally, though not always, minimized by using pressures not in excess of 20,000 pounds per square inch for a loading time of 5 to 15 minutes.

It may be worth while to mention at this point that crystal changes giving rise to distorted spectra have also been encountered in mulls, although less frequently than in pellets. Spectra

16. Tai, H., and Underwood, A. C., *Anal. Chem.*, **29** (10), 1430 (1957).

obtained from vigorously hand-mulled samples are different, in a number of cases, from those obtained from vigorously machine-mulled samples.¹⁵ Although anomalous behavior in alkali halide pellet spectra has been observed and attributed to cation exchange, mixed crystal formation, or surface absorption, some evidence also exists for interchange when oil mulls are pressed between salt plates, particularly when sufficient moisture is present to foster the reaction.¹⁷ Thus, any comparison of solid-state spectra, whether obtained by mull or pellet technique, should make allowance for possible crystal changes and interaction.

The growth of suitable single crystals of appropriate thicknesses has made possible the examination of many elemental substances and ionic and intermetallic compounds. Unfortunately, the techniques for growing such crystals are by no means suited to general use. Furthermore, the spectra of crystals are often complicated by the presence of peaks due to reflection and scattering. These complications are of general occurrence and, unless they are accurately controlled, lead to difficulties in interpreting the infrared spectra of the substance.

Samples which cannot be dealt with by any of the above means may possibly be handled by the recently developed diamond cell microtechnique.¹⁸ A small crystal weighing as little as 4×10^{-6} gram can be crushed into a thin film between diamond surfaces and the assembly mounted into a commercial infrared beam-condensing unit. Minimum pressures to produce the film range from 1 to 100 atmospheres. The inertness of the diamond surface permits the handling of corrosive materials. By using sapphire instead of diamond, the useful range of the cell can be extended to frequencies higher than 1900 cm^{-1} .

17. Padgett, W. M., Talbert, J. M., and Hammer, W. F., *J. Chem. Phys.*, **26**, 959 (1957).
18. Method of Lippincott, Welsh, and Weir, as reported in *Chem. Eng. News*, **38**, No. 39, 120 (1960).

APPLICATIONS

Review of the literature reveals a variety of applications of the infrared spectra of inorganic materials. These include (1) qualitative^{14, 19-21} and quantitative analysis;⁴ (2) study of molecular structure; (3) measurement of band intensities;²² (4) study of solid-state reactions to determine whether a product is a new compound or a physical mixture of reactants;²³ (5) calculation of thermodynamic quantities;²⁴ (6) investigation of impurity levels in semiconductors;²⁵⁻²⁷ and (7) determination of isotope ratios in tracer experiments.²⁸ Other applications can be found in the review articles cited in the bibliography. Only the qualitative and quantitative analyses and molecular structure studies will be discussed here as examples of the application.

As early as 1952, Miller and Wilkins¹⁹ demonstrated the usefulness of spectral data for the qualitative analysis of inorganic mixtures by assigning characteristic frequencies for 33 polyatomic ions present in over a hundred different pure inorganic compounds. The quality of the spectra ranged from sharp, intense bands to ill-defined ones. The effect of varying the positive ion associated with a given anion was observed. Individual sulfates demonstrated enough variation among themselves to make distinction possible. On the other hand, nitrates showed no appreciable shifting as the cation was varied. However, it was suggested that a combination of infrared absorption, emission, and x-ray analysis

19. Miller, F. A., and Wilkins, C. H., *Anal. Chem.*, **24**, 1253 (1952).
20. Brame, E. G., Cohen, S., Margrave, J. L., and Meloche, V. W., *J. Inorg. Nucl. Chem.*, **4**, 90 (1957); Brame, E. G., Margrave, J. L., and Meloche, V. W., *J. Inorg. Nucl. Chem.*, **5**, 48 (1957).
21. Brame, E. G., Johnson, F. A., Larsen, E. M., and Meloche, V. W., *J. Inorg. Nucl. Chem.*, **6**, 99 (1958).
22. Mecke, R. J., *J. Chem. Phys.*, **20**, 1935 (1952).
23. Duval, C., and Lecomte, J., *Compt. rend.*, **234**, 2445 (1952).
24. Giguere, P. A., and Liu, I. D., *Can. J. Chem.*, **30**, 948 (1952).
25. Burstein, E., *et al.*, *J. Phys. Chem.*, **57**, 849 (1953).
26. Kaiser, W., *et al.*, *Phys. Rev.*, **91**, 1380 (1953); *Naturwiss.*, **40**, 497 (1953).
27. Tanenbaum, M., and Briggs, H. B., *Phys. Rev.*, **91**, 1561 (1953).
28. Kluyver, J. C., and Milatz, J. M., *Physica*, **19**, 401 (1953).

be used in the qualitative analysis of mixtures containing more than two components, since this combination provides more valid information than one method alone. Kalbus¹⁴ examined 122 inorganic compounds and found that identification was generally possible for carbonates, nitrates, and sulfates, but was not as specific for others. Brame *et al.*²⁰⁻²¹ undertook the task of securing spectra of a variety of inorganic materials—peroxides, peroxide hydrates, superoxides, oxides, nitrides, carbides, borides, and octacyano complexes of tungsten and molybdenum—for identification purposes. The results of the spectral studies showed characteristic absorption bands for some of the structures, but failed to do so for others. The important correlations made are discussed here in Part II.

Quantitative analysis of inorganic substances has not developed as rapidly as qualitative. The absorption spectrum of a mixture is generally an additive function of each component, so that theoretically it should be possible to determine the component concentrations. Experimentally, difficulties are encountered due largely to the presence of scattered energy from the sources employed in the spectrophotometers used. A number of adjustments and modifications of instruments and methods have been proposed to correct for the stray energy. Other sources of errors are reflection losses, solvent absorption, non-homogeneity of sample, misalignment of cells, and non-linearity in the detecting and recording systems of the instrument.

An evaluation of infrared spectrophotometers for quantitative analysis has been made by Robinson.⁴ It was pointed out that errors occur in the measurement of transmission, and in the reading of the zero and 100% lines. The effect of such errors can be calculated. If a plot is made of the per cent transmission versus the per cent error in concentration, assuming a probable error of 1% in the zero line, 100% line, and the transmission, a minimum occurs at about 40% transmission. Any transmission between 25% and 60% gives about the same error.

Two other factors are involved in the adaptation of infrared techniques for quantitative analysis, namely, the law of additivity of optical density and the Beer-Lambert law. The law of addi-

tivity of optical density simply states that the optical density of a mixture is the sum of the optical densities of its components:

$$\left(\log \frac{I_0}{I}\right)_{\text{mixture}} = \sum_{i=1}^N \left(\log \frac{I_0}{I}\right)_i$$

where $\left(\log \frac{I_0}{I}\right)_i$ is the optical density and there are N components.

For the law of additivity to hold, Beer's law must hold for all components but one. The fact that the transmission of light through a medium containing a number of molecules is a function of the number of molecules in the light beam is the basis of every concentration measurement. If there is no appreciable molecular interaction and if the light is monochromatic, Beer's law is an approximation of this function. Deviations from Beer's law generally occur when the spectrometer slits are wider than the width of the absorption band measured, since under this condition it is possible to have changes in absorption coefficients over a slit width.

A useful technique in infrared quantitative analysis is the method of differential analysis. By this method small differences in similar substances can be detected by comparing them simultaneously in a double-beam spectrophotometer. Some method should be employed to eliminate the effect of differences in cell thickness; for example, measuring the solution in the sample cell against the solvent in the blank cell, followed by measuring the solution in the blank cell against the solvent in the sample cell.

The availability of microsampling techniques makes possible the analysis of microquantities of solids, liquids, and gases. Essentially, to effect analysis of microquantities the thickness of the sample in the beam or the sample area must be reduced. When the sample thickness is reduced, a loss in recorder deflection occurs, unless scale expansion devices are used with the spectrometer. Reduction in sample area by means of a small cell lowers the amount of energy available to the detector. One approach to the problem has been the use of beam-condenser optics, which reduce the size of the beam as it passes through the sample, thus making possible the analysis of smaller samples.

Spectra obtained as a function of temperature have proved use-

ful in studies of molecular structure. In addition, study of the polarized spectra of crystalline samples give valuable information in this regard. For molecules of appropriate symmetry, vibrations which are in a direction parallel to an axis of orientation can be differentiated from those which are perpendicular. Interpretation of polarized spectra provides information about the crystal structure also. These spectra have, indeed, been useful in locating bonds which are difficult to find by x-ray diffraction methods. Here again, technical difficulties arise in obtaining and properly orienting the crystalline film or sample. The experimental data indicate that the radiation absorbed in a band is polarized along a given axis. This is not always easily or correctly related to the direction of the bond or to the direction of the bond vibration. Consequently, information provided by infrared polarized radiations of a sample is most valuable when used to complement information secured by other means of analysis.

PART II

Spectra-Structure Correlations

THE CORRELATION OF STRUCTURE OF MOLECULES with their spectra is one of the ultimate aims of infrared analysis. In this section is presented a discussion of the vibrational spectra of a variety of inorganic materials for which absorption data are available. The materials are divided into categories based on the number of kinds of atoms present. Where more than one kind of atom is present, further subdivision is based on the principal non-metallic constituent. Miscellaneous structures, such as intermetallic compounds, are discussed under the subheading "Other Binary (or Ternary) Compounds." Some consideration is also given to metal-organic complexes. In view of the immense volume of empirical data that exists in the literature, there are unavoidable omissions of source material, particularly with respect to publications prior to 1950. However, an attempt has been made to cite as many review articles and references to the earlier work as possible. Although this review represents extensive coverage of the important achievements in the field, the reader should regard this section only as an aid in locating the types of spectral studies reported in the literature for various inorganic materials, realizing that any further usage or interpretation should be made in conjunction with the original accounts.

ELEMENTAL SUBSTANCES

Vibrations of linear non-polar molecules, e.g., O_2 , N_2 , H_2 , cause no change of electric moment—a condition necessary for absorption in the infrared. Therefore, such molecules should possess no rotation-vibration spectra. However, these symmetrical diatomic molecules have been found to have absorption bands when measured under certain conditions.²⁹ The intensity of the absorption is proportional to the square of the gas pressure. Absorbing gases, when pressurized with infrared-inactive gases, exhibit an induced absorption which has an intensity proportional to the pressure of the inactive gas. Apparently then, this pressure-induced absorption is due to the interaction of the radiation field with an induced dipole moment resulting from the distortion of molecules upon collision.

Considerable interest has centered around the absorption of hydrogen. In the region $660\text{--}1250\text{ cm}^{-1}$ compressed hydrogen shows a remarkable absorption, which has been ascribed to the pure rotation spectrum by Ketelaar *et al.*³⁰ The fundamental vibrational absorption of hydrogen, induced by intermolecular forces, has been studied over a wide range of pressures and temperatures. As the temperature is lowered, resolution of the band is improved. Allin, Hare, and MacDonald³¹ investigated the infrared absorption of hydrogen in the liquid and solid states. The molecule is distorted asymmetrically by intermolecular forces as the temperature drops, giving rise to induced infrared absorption. In the crystalline state, this distortion is produced not only by random orientation of the freely rotating molecule but also by the lattice vibrations of the crystals. Variations in the fundamental absorption of solid hydrogen at $13.6 \pm 0.2^\circ\text{K}$ occurs with changes

29. Welsh, H. L., Crawford, M. F., MacDonald, J. C. F., Chishom, D. A., *Phys. Rev.*, **83**, 1264 (1951).

30. Ketelaar, J. A. A., Colpa, J. P., and Hooge, F. N., *J. Chem. Phys.*, **23**, 413 (1955).

31. Allin, E. J., Hare, W. F. J., and MacDonald, R. E., *Phys. Rev.*, **98**, 554 (1955).

in the ortho-para ratio.³² More structure is observed as the per cent composition of para-hydrogen is decreased.

While there are published works on other non-metallic molecules—ozone,^{33, 34} sulfur,³⁵ oxygen and nitrogen^{36, 37}—most of the work on elemental substances has been devoted to metallic and semiconducting structures. The optical constants of many polyvalent metals show a marked dependence on frequency in the infrared region at wavelengths greater than 10 microns. Theoretical discussions of the optical properties of metals appear in the book literature.^{38, 39}

A number of inconsistencies in the measured optical constants exist in the literature. These arise, in general, from the fact that the methods used in determining the constants are all too often dependent on the previous chemical and mechanical treatment of the surface. Haas⁴⁰ has tabulated the optical constants of a number of metals and alloys at various wavelengths extending into the infrared.

Infrared absorption measurements have also been used as a means of investigating the anomalous skin effect^{41, 42} in metals. According to classical theory, the absorptivity of a metal should

32. Hare, W. F. J., Allin, E. J., and Welsh, H. L., *Phys. Rev.*, **99**, 1887 (1955).

33. Nexsen, W. E., *Dissertation Abstr.*, **16**, 771 (1956).

34. Paetzold, H. K., *Z. Naturforsch.*, **11a**, 128 (1956).

35. Barrow, G. M., *J. Chem. Phys.*, **21**, 219 (1953).

36. Yaroslavskii, N. G., and Karyalin, A. V., *Doklady Akad. Nauk S.S.S.R.*, **85**, 1103 (1952).

37. Coulon, R., Oskengorn, B., Robin, St., and Vodar, B., *J. phys. rad.*, **14**, 63 (1953).

38. Seitz, F., "The Modern Theory of Solids," Chapter XVII, McGraw-Hill Book Co., Inc., New York (1940).

39. Givens, M. P., "Optical Properties Of Metals," p. 313ff. in "Solid State Physics," Vol. 6, F. Seitz and D. Turnbull, Editors, Academic Press, Inc., New York (1958).

40. Haas, G., "Optical Constants of Metals," Section 6–102 in "American Institute of Physics Handbook," D. E. Gray, Editor, McGraw-Hill Book Co., Inc., New York (1957).

41. Pippard, A. B., *Proc. Roy. Soc. (London)*, **191A**, 385, 399 (1947).

42. Reuter, G. E. H., and Sondheimer, E. H., *Proc. Roy. Soc. (London)*, **195A**, 336 (1948).

decrease without limit as the mean free path of the conduction electrons increases. Observed absorptivities of metals, however, are of the same order of magnitude at liquid helium temperature as at room temperature. The classical theory is valid as long as the mean free path, l , is small compared to the depth of penetration, δ , of the electromagnetic field. When $l > \delta$, anomalous behavior results. Biondi⁴³ has measured the optical absorption of copper and silver at 4.2°K in the region 33,333–3030 cm^{-1} ($0.3\text{--}3\mu$), where $l > \delta$. Consistent with the anomalous skin effect, infrared absorptivity was independent of wavelength at values greater than 1.5μ .

Notable contributions to the understanding of phenomena associated with semiconductors have been made through infrared absorption spectroscopy. By studying the temperature dependence of the absorption spectrum of such materials in terms of existing theories, it has been possible to obtain valuable information for the assignment of the appropriate band structures. Furthermore, examination of the infrared absorption spectra of semiconductors before and after heat treatments and irradiation provides clues to the nature of the mechanisms responsible for absorption. Extensive coverage of the theory and experimental findings appear in a recent book by Moss.⁴⁴

BINARY COMPOUNDS

Borides, Carbides, and Nitrides

Information on the physical and chemical properties of borides, carbides, and nitrides is very limited; this is particularly true of spectral information. The infrared spectra of Mo_2B , ZrB_2 , TiB_2 , B_4C , SiC , Li_3N , Cu_3N , BN , AlN , CrN , Mg_3N_2 and Zr_3N_2 have been investigated in the region $624\text{--}5000\text{ cm}^{-1}$.²⁰ Of the boride structures studied, only the tetragonal molybdenum boride showed band structure. A weak, broad band with its center at 1389 cm^{-1}

43. Biondi, M. A., *Phys. Rev.*, **102**, 964 (1956).

44. Moss, T. S., "Optical Properties of Semiconductors," Academic Press, Inc., New York (1959).

was found. Only two carbides—rhombohedral boron carbide and hexagonal silicon carbide—were examined. Although this is hardly sufficient data to establish that the observed band is characteristic, it is interesting to point out that a shift toward longer wavelengths occurs as the mass is increased. The 1053 cm^{-1} medium intensity band of B_4C is shifted, as would be expected from the mass effect, to an 833.3 cm^{-1} band of medium intensity for SiC . The shift is small and may be attributed partly to differences in crystal structure as well as to the mass effect.

Two strong intensity bands were noted for all nitride samples except copper nitride and chromium nitride. Copper nitride exhibits strong absorption in the neighborhood of 625 cm^{-1} , the limit of the rock-salt scanning region used; chromium nitride showed no absorption for the region. For the series Li_3N , BN , AlN a progressive shift toward longer wavelength of the strong intensity bands with mass increase was observed. Similarly, an absorption band shift was noted for Mg_3N_2 and Zn_3N_2 . The predicted change in position of the bands as a result of mass effect was not followed when the entire group of nitrides was considered. Presumably, the differences in crystal structure between the two sets of nitride samples make the mass effect prediction invalid. For different crystal structures, relative positions of the atoms are changed. This results in changes of the force constants involved and consequent shifts in the absorption frequencies. Notwithstanding the crystal structure effect, the strong intensity bands observed apparently represent a metal-nitrogen vibration, particularly since the shift of the main band was of reasonably large magnitude. Table 2 shows the crystal structures and band centers located for the samples studied.

Halides

Inorganic halides have been used widely as prism and cell window material for the infrared region. For a number of these compounds, the infrared absorption spectrum has been determined under a variety of conditions. The approximate range of transmission of the halides most commonly used for prism and window construction is given in Table 3. The useful range of

TABLE 2. CRYSTAL STRUCTURE AND ABSORPTION BAND CENTERS OF BORIDES, CARBIDES, AND NITRIDES

(After Brame *et al.*²⁰)

Compound	Crystal Structure ^a	Absorption Bands ^b	
Mo ₂ B	tetragonal	7.2(1389)w	
B ₄ C	rhombohedral	9.5(1053)m	12.9(775.2)w
SiC	hexagonal	12.0(833.3)m	
Li ₃ N	hexagonal	6.8(1471)sh	7.0(1429)s
		11.6(862.1)m	15.2(657.9)m
Bn	hexagonal	7.28(1374)s	12.3(813)m
AlN	hexagonal	8.45(1183)w	9.46(1057)w
		14.0(714.3)m	
Mg ₃ N ₂	cubic	4.8(2083)m	7.1(1408)m
		15.2(657.9)m	
Zn ₃ N ₂	cubic	9.1(1099)m	11.15(897.1)w
Cu ₃ N	cubic	16.0(625)s	

^a Wyckoff, R. W. G., "Crystal Structure," Vol. I., Interscience Publishers, New York, 1951.

^b The location of the band centers is given in microns and cm^{-1} ; the wave number (cm^{-1}) location is in parentheses. The band intensity is represented by the lettering: sh = shoulder; w = weak; m = medium; s = strong.

TABLE 3. APPROXIMATE RANGE OF INFRARED TRANSMISSION OF SOME COMMONLY USED PRISM AND WINDOW MATERIALS ^a

Material	Range of Transmission	
	Wavelength, μ	Wavenumber, cm^{-1}
LiF	0.11–6	90,910–1667
CaF ₂	0.12–8.5	90,090–1176
NaCl	0.2–15	50,000–667
KBr	0.21–28	49,750–357
KRS-5 (TlBr + TlI)	0.6–40	16,670–250
CsBr	0.3–40	33,333–250
CsI	–55	–182

^a Taken in part from: Willard, Merritt, Dean, "Instrumental Methods of Analysis," p. 156, D. Van Nostrand Company, Inc., Princeton, N. J., 1958.

transmission is determined by other factors and will, in general, be somewhat narrower than the ranges given.

Fluorides. The hexafluorides of S, Se, Te, Mo, W, U, Np, Pu, Re, and Ir have been shown to have the O_h symmetrical structures of regular octahedra. Accordingly, there are six fundamental vibrations: three Raman-active modes of species a_{1g} , e_g , and f_{2g} ; two infrared-active modes of species f_{1u} ; and an inactive f_{2u} mode. Observed and calculated values of these fundamentals have been reported in the literature⁴⁵⁻⁵¹ and are tabulated in Table 4. Spectra of certain fluorides of sulfur, namely, sulfur monofluoride,⁵² sulfur tetrafluoride,⁵³ and sulfur hexafluoride,^{46, 54, 55} have been measured in the infrared region. Sulfur monofluoride exhibits the following vibrations: 1332 cm^{-1} , combination band of 807 and 526 ; 807 cm^{-1} , asymmetric F—S stretching; 745 cm^{-1} , symmetric F—S stretching; and 526 cm^{-1} , S—S stretching. Both infrared and Raman work on the tetrafluoride seems to indicate that the SF_4 molecule belongs to point group C_{2v} . The spectrum of the hexafluoride has a number of unexplainable peaks, perhaps due to the presence of SF_4 and S_2F_{10} . However, the observation of the predicted infrared and Raman bands, together with electron diffraction data, is compatible with the octahedral model for SF_6 .

45. Burke, T. G., Smith, D. F., and Nielsen, A. H., *J. Chem. Phys.*, **20**, 447 (1952).
46. Gaunt, J., *Trans. Faraday Soc.*, **49**, 1122 (1953).
47. Gaunt, J., *Trans. Faraday Soc.*, **51**, 893 (1955).
48. Hawkins, N. J., Mattraw, H. C., and Sabol, W. W., *J. Chem. Phys.*, **23**, 2191 (1955).
49. Malm, J. G., Weinstock, B., and Claasen, H. H., *J. Chem. Phys.*, **23**, 2192 (1955).
50. Gaunt, J., *Trans. Faraday Soc.*, **50**, 209 (1954).
51. Mattraw, H. C., Hawkins, N. J., Carpenter, D. R., and Sabol, W. W., *J. Chem. Phys.*, **23**, 985 (1955).
52. Matutano, J. R. B., and Otero, C., *Anales real Soc. espan. fis. y quim. (Madrid)*, **51B**, 223 (1955).
53. Dodd, R. E., Woodward, L. A., and Roberts, H. L., *Trans. Faraday Soc.*, **52**, 1052 (1956).
54. de Lattre, A., *J. Chem. Phys.*, **20**, 520 (1952).
55. Edelson, D., and McAfee, K. B., *J. Chem. Phys.*, **19**, 1311 (1951).

TABLE 4. FUNDAMENTAL VIBRATIONAL FREQUENCIES (cm^{-1}) OF SOME HEXAFLUORIDES ^a

	ν_1	ν_2	ν_3	ν_4	ν_5	ν_6	References ^b
MoF ₆	741(736)	645(641)	741(742)	260(269)	322(319)	234(240)	45,(46,47)
WF ₆	772(769)	672(670)	712(712)	258(256)	316(322)	215(216)	45,(46,47)
ReF ₆	753	600	716	393	246	170	50
IrF ₆	696	643	718	276	260	205	51
UF ₆	656(668)	511(532)	640(626)	200(189)	200(202)	130(144)	45,(46,47)
NpF ₆	648(665)	528(536)	624(623)	200(186)	206(202)	164(136)	49,(48)
PuF ₆	628(625)	523(522)	615(617)	203(205)	211(211)	171(176)	49,(48)
SF ₆	775	644	932	613	524	344	46,47
SeF ₆	708	662	780	437	405	260	46,47
TeF ₆	701	674	752	327	313	197	46,47

^a ν_1 , ν_2 , ν_5 —Raman-active, ν_3 , ν_4 —infrared active, ν_6 —inactive.

^b The references in parentheses refer to the values listed in parentheses.

Considering their similarity to methane, CH_4 , it seems natural to assume that the tetrafluorides of carbon, silicon, and germanium (CF_4 , SiF_4 , and GeF_4 , respectively) would form a regular tetrahedron with each of the four fluoride atoms at the vertices and the remaining atom in the center. For the regular tetrahedron (point group, T_d) there are four fundamental vibrational frequencies, two of which are infrared-active. Indeed, for these three tetrafluorides, two intense infrared bands have been located.⁵⁶ The number of total bands observed for GeF_4 is small when compared with those observed for CF_4 and SiF_4 . For all three compounds, the ν_4 fundamental is the only one in which PQR band structure is discernible with prism instruments. The fundamental vibrational frequencies are recorded in Table 5.

TABLE 5. FUNDAMENTAL VIBRATIONAL FREQUENCIES OF CF_4 , SiF_4 , AND GeF_4

	CF_4^a	SiF_4^b	GeF_4^c
ν_1	904 cm^{-1}	800 cm^{-1}	738 cm^{-1}
ν_2	435	268	205
ν_3	1283	1031	800
ν_4	632	391	260

^a Woltz, P. J. H., and Nielsen, A. H., *J. Chem. Phys.*, **20**, 307 (1952).

^b Jones, Kirby-Smith, Woltz, P. J. H., and Nielsen, A. H., *J. Chem. Phys.*, **19**, 242 (1951).

^c Caunt, A. D., Short, L. N., and Woodward, L. A., *Trans. Faraday Soc.*, **48**, 873 (1952).

The trifluorides of nitrogen and phosphorus were investigated by Wilson and Polo⁵⁷ for the region 250–5000 cm^{-1} . Chemically, NF_3 and PF_3 are quite dissimilar in their tendency to form addition compounds. For example, PF_3 forms a ferrohemoglobin complex, HbPF_3 , and a nickel complex, $\text{Ni}(\text{PF}_3)_4$, while NF_3 does not. This would seem to indicate that the two molecules have significantly different electronic structures. These investigators measured the absolute intensities of the infrared vibration bands

56. Woltz, P. J. H., and Nielsen, A. H., *J. Chem. Phys.*, **20**, 307 (1952).

57. Wilson, M. K., and Polo, S. R., *J. Chem. Phys.*, **20**, 1716 (1952).

of NF_3 and PF_3 in an attempt to gain information on the structure. If one assumes for these molecules the C_{3v} symmetry of ammonia, NH_3 , then six normal vibrational modes may be described: $\nu_1(a_1)$, a totally symmetric stretching vibration; $\nu_2(a_1)$, a totally symmetric bending vibration; $\nu_3(e)$, a doubly degenerate stretching frequency; and $\nu_4(e)$, a doubly degenerate bending frequency. All four fundamentals are allowed in the infrared and Raman spectrum. The spectrum of neither molecule was rich in overtone and combination bands, and assignment was reasonably straightforward. Pace and Pierce⁵⁸ observed the infrared and Raman spectra of NF_3 and obtained data which substantiate the findings of Wilson and Polo (Table 6).

TABLE 6. FUNDAMENTAL VIBRATIONAL FREQUENCIES (cm^{-1}) OF NF_3 AND PF_3

	PF_3^a	NF_3^a		
			Infrared	Raman
$\nu_1(a_1)$	892	1032	1031	1050
$\nu_2(a_1)$	487	647	642	667
$\nu_3(e)$	860	905	907	905
$\nu_4(e)$	344	493	497	515

^a Wilson and Polo.⁵⁷

^b Pace and Pierce.⁵⁸

Most molecules of the XY_3 type have been found to have the planar or pyramidal structures, the Y atoms being equivalent. For NF_3 and PF_3 the pyramidal model is indicated; however, for BF_3 spectral data favor the plane symmetrical structure.⁵⁹ For the trifluorides of chlorine and bromine, ClF_3 and BrF_3 , respectively, there is strong indication that neither model applies.⁶⁰ Rather, a planar T-shaped model seems more plausible. For such a structure, six fundamental vibrations are allowed: three totally

58. Pace, E. L., and Pierce, L., *J. Chem. Phys.*, **23**, 1248 (1955).

59. See discussion in Herzberg, F., "Infrared and Raman Spectra of Polyatomic Molecules," p. 298, D. Van Nostrand Co., Inc., New York, 1945.

60. Claassen, H. H., Weinstock, B., and Malm, J. G., *J. Chem. Phys.*, **28**, 285 (1958).

symmetric vibrations of species a_1 , two fundamentals of species b_1 , and one of species b_2 . All six were observed in the spectra of ClF_3 (vapor): $\nu_1(752)$, $\nu_2(528)$, $\nu_3(326)$, $\nu_4(703)$, $\nu_5(434)$, and $\nu_6(364)$; while only two were observed in the spectra of BrF_3 (vapor): $\nu_1(674)$ and $\nu_4(613)$. The calculated values for BrF_3 are $\nu_1(662)$, $\nu_2(528)$, $\nu_3(300)$, $\nu_4(601)$, $\nu_5(384)$, $\nu_6(289)$.

The spectrum of gaseous BrF_3 was recorded in the KBr region and a tetragonal pyramidal structure suggested on the basis of the data obtained along with supporting data from the Raman spectrum of liquid BrF_3 .⁶¹ No definite structural assignment could be made because insufficient data were obtained on the low-frequency infrared-active fundamentals. Measurements are necessarily made at low pressures, because BrF_3 strongly attacks the cell windows. Nine Raman lines (684, 629, 569, 539, 480, 410, 365, 310, 241) were designated as fundamentals, strongly suggesting the C_{4v} square pyramid structure in preference to a trigonal bipyramid model. The infrared spectra of liquid BrF_3 and liquid BrF_5 were reported by Haendler and co-workers.⁶² Only a limited region of the infrared was studied because of the extreme activity of the samples. Three bands were located for both compounds: BrF_3 —1337, 1250, 1154 cm^{-1} and BrF_5 —1355, 1298, 1187 cm^{-1} . Another pentafluoride, PF_5 , was studied by Pemsler and Planet.⁶³ The data obtained suggest a trigonal bipyramid structure for the molecule.

Spectral studies on other fluorides⁶⁴⁻⁶⁷ warrant citation but will not be discussed in detail here. Fluorides of hydrogen and its isotopes will be treated at the end of this section with the corresponding compounds of the other halogens.

61. Burke, T. G., and Jones, E. A., *J. Chem. Phys.*, **19**, 1611 (1951).

62. Haendler, H. M., BuKata, S. W., Millard, B., Goodman, E. I., and Littman, J., *J. Chem. Phys.*, **22**, 1939 (1954).

63. Pemsler, J. P., and Planet, W. G., *J. Chem. Phys.*, **24**, 920 (1956).

64. Nielsen, A. H., and Jones, E. A., *J. Chem. Phys.*, **19**, 1117 (1951) ClF .

65. Edelson, D., *J. Am. Chem. Soc.*, **74**, 262 (1952) S_2F_{10} .

66. Gaunt, J., and Ainscough, J. B., *Spectrochim. Acta*, **10**, 57 (1957) SbF_5 ; *ibid.*, 52 NbF_5 .

67. Jones, D. A., Jones, R. V., and Stevenson, R. W., *Proc. Phys. Soc. (London)*, **65B**, 906 (1952) LiF , CuF_2 , CaF_2 , CdF_2 , SrF_2 , PbF_2 , BaF_2 .

Chlorides, Bromides, and Iodides. The vibrational spectra of a number of inorganic halides were reviewed in the classic work of Herzberg. More recently, several articles containing theoretical discussions of the optical properties and lattice vibrations of alkali halides and other non-metallic crystals have appeared.⁶⁸ Infrared absorption information on the alkali halides was obtained indirectly through the use of their reflection spectra and the findings were analyzed by Lax and Burstein.⁶⁹ An explanation for the auxiliary bands appearing in the infrared absorption spectra of these compounds was proposed, on the assumption that an appreciable deformation of charge distribution exists about the atoms during their lattice vibration.

In the region of the fundamental vibration frequency, spectra for NaCl, NaBr, NaI, KCl, KBr, RbCl, and CsCl have been taken of the molten salts in an atmosphere of argon using optics of cesium bromide or iodide.⁷⁰ The calculated frequencies in cm^{-1} are: NaCl (366), NaBr (302), NaI (258), KCl (281), KBr (213), RbCl (228), CsCl (209). It was possible from the data obtained, along with other data on lithium halides, to extrapolate values for five other compounds: KI (173), RbBr (166), RbI (128), CsBr (139), CsI (101).

The polarized infrared spectrum of hydrated cupric chloride, $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$, was investigated in an attempt to determine whether water is predominantly ionically or covalently bonded to the metal ions in such complex structures.⁷¹ If the bonding is ionic, it would be expected that the water dipole would point toward the metal; if the bonding is covalent, approximately tetrahedral MOH angles would be anticipated. The polarized infrared spectral data favor the latter.

68. Dexter, D. L., "Theory of the Optical Properties of Imperfections in Non-metals," p. 353ff. in "Solid State Physics," Vol. 6, F. Seitz and D. Turnbull, Editors, Academic Press, Inc., New York, 1958. See also references cited therein.

69. Lax, M., and Burstein, E., *Phys. Rev.*, **97**, 39 (1955).

70. Rice, S. A., and Klemperer, W., *J. Chem. Phys.*, **27**, 573 (1957).

71. Rundle, R. E., Nakamoto, K., and Richardson, J. W., *J. Chem. Phys.*, **23**, 2450 (1955).

Molecules of divalent metals and halogens have been investigated to some extent. Klemperer and Lindeman⁷² reported the frequency of the antisymmetric motion in gaseous mercuric chloride, bromide, and iodide. These are linear symmetrical triatomic molecules for which knowledge of the three fundamental frequencies is sufficient to calculate the force constants. The vibrational frequencies (in cm^{-1}) observed are: HgCl_2 , ν_1 (360), ν_2 (70), ν_3 (413); HgBr_2 , ν_1 (225), ν_2 (41), ν_3 (293); HgI_2 , ν_1 (156), ν_2 (33).

Gaseous magnesium bromide and magnesium chloride have been examined spectrally at an elevated temperature of 1000°C .⁷³ Based on electron diffraction measurements these molecules are also linear and should exhibit two infrared-active fundamentals at frequency ν_3 for the antisymmetric stretching vibration and ν_2 for the bending vibration. Only one absorption peak was observed for these halides and it has been ascribed to the antisymmetric stretching vibration. Similar results were obtained with nickelous chloride at 850°C .

Halides of Hydrogen and Its Isotopes. Rather extensive studies have centered around the hydrogen halides and their deuterium and tritium analogs.⁷⁴⁻⁸⁶ Many of the studies are concerned pri-

72. Klemperer, W., and Lindeman, L., *J. Chem. Phys.*, **25**, 397 (1956).
73. Randall, S. P., Greene, F. T., and Margrave, J. L., *J. Phys. Chem.*, **63**, 758 (1959).
74. Shelton, R. D., and Nielsen, A. H., *J. Chem. Phys.*, **19**, 1312 (1951).
75. Coulon, R., *J. phys. radium*, **13**, 371 (1952).
76. Mills, I. M., Thompson, H. W., and Williams, R. C., *Proc. Roy. Soc. (London)*, **A218**, 29 (1953).
77. Benesch, W., and Elder, T., *Phys. Rev.*, **91**, 308 (1953).
78. Keller, F. L., and Nielsen, A. H., *J. Chem. Phys.*, **22**, 294 (1954).
79. Hornig, D. F., and Osberg, W. E., *J. Chem. Phys.*, **23**, 662 (1955).
80. Mayburg, R. H., Gordon, S., and Katz, J. J., *J. Chem. Phys.*, **23**, 1277 (1955).
81. Palik, E. D., *J. Chem. Phys.*, **23**, 217 (1955).
82. Adams, R. M., and Katz, J. J., *J. Opt. Soc. Am.*, **46**, 895 (1956).
83. Jones, L. H., and Robinson, E. S., *J. Chem. Phys.*, **24**, 1246 (1956).
84. Van Horne, B. H., and Hause, C. D., *J. Chem. Phys.*, **25**, 56 (1956).
85. Kuipers, G. A., Smith, D. F., and Nielsen, A. H., *J. Chem. Phys.*, **25**, 275 (1956).
86. Hiebert, G. L., and Hornig, D. F., *J. Chem. Phys.*, **28**, 1316 (1958).

marily with the evaluation and re-evaluation of data for the calculation of molecular constants. Others have been conducted for the purpose of explaining some rather interesting physical and chemical problems. The phenomenon of pressure broadening of absorption lines constitutes a major problem in infrared atmospheric transmission and in quantitative gas analysis in the infrared region. Some qualitative features of molecular interactions in pressure-broadening phenomena have been determined using HCl and CH₄ gases with He, Ne, Ar, Kr, Xe, SF₆, O₂, H₂, N₂, CO, CO₂, N₂O, SO₂, and HCl as broadeners.⁷⁷ The data seem to indicate (1) an interaction between the induced dipole of the broadener molecule and some undefined property of the absorbing gas regardless of the foreign gas used, and (2) an interaction between the quadrupole moment of the broadener and the dipole of the absorber when unsymmetrical foreign molecules were used to broaden the absorption lines of a polar molecule. The increased absorption in the infrared associated with the pressure-broadening effect has been used to calculate relative optical collision diameters.

The infrared absorption spectrum of hydrogen fluoride has been studied to gain information concerning the degree of polymerization under various conditions. The intensity of the 1025 cm⁻¹ band increases with pressure to the extent that polymeric formation is undoubtedly the prevailing situation.⁷⁵ Examination of HF from 3000–4250 cm⁻¹ at temperature from 32–88° C, and pressures from atmospheric to vacuum give results that indicate only a few polymeric species, even under the optimum polymer formation conditions of low temperature and high pressure.⁷⁴

Fundamental vibrations of hydrogen halide crystals at -250° C were determined, to delineate the structures of the low-temperature phases present.⁷⁹ Both HCl and HBr were shown to form zigzag hydrogen-bonded chains, the angles between adjacent molecules being 107° and 97° for the HCl and HBr, respectively. Apparently HI has a different structure, but this was not conclusively determined. Mixed crystals of HCl and HBr at 63° K give rise to a spectrum which consists of a band characteristic of chains of similar molecules plus a line resulting from the end molecule in

any chain.⁸⁶ This structure is repeated in the HCl and HBr regions. The fundamental frequency of HBr with the H bonded to HCl is higher than when it is bonded to another HBr molecule; and that of HCl is lower when the H is bonded to HBr than to another HCl.

The ionic crystalline monohydrates of the hydrogen halides have been investigated in the infrared region at -195°C . The spectra of OH_3F , OH_3Cl , OH_3Br , OH_3I , and OD_3Cl exhibit fundamental frequencies consistent with a symmetrical pyramidal structure⁸⁷ (Table 7).

TABLE 7. FUNDAMENTAL FREQUENCIES (cm^{-1}) OF IONIC CRYSTALS
HYDROGEN HALIDE MONOHYDRATES

(After Ferriso and Hornig⁸⁷)

	OH_3F	OH_3Cl	OH_3Br	OH_3I	OD_3Cl
ν_1 OR ν_3	3150	3235	3250	3350	2445
ν_2	1150	1150	1150	1160	785
ν_3 OR ν_1	2468	2590	2610	2635	2000
ν_4	1705	1700	1705	1705	1255

Oxides

The classic work of Herzberg summarizes the earlier observations of the vibrational spectra of oxides and peroxides. Included among these are oxides of carbon, hydrogen, nitrogen, fluorine, and chlorine and hydrogen peroxide. No attempt will be made here to review these observations, except as necessary for discussion. Attention will be directed toward the more recent studies which have been conducted to gain further insight into the vibrational frequencies and structure of these and related molecules.

Compounds of Hydrogen and Its Isotopes. In addition to the spectra of water and deuterium oxide, infrared spectra of tritium oxide, THO, and TDO have been obtained.⁸⁸ Vapor-phase

87. Ferriso and Hornig, D. F., *J. Chem. Phys.*, **23**, 1464 (1955).

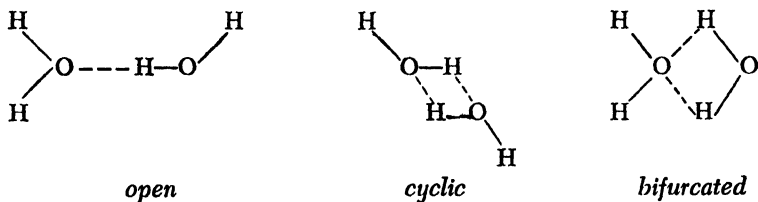
88. Staats, P. A., Morgan, H. W., and Goldstein, J. H., *J. Chem. Phys.*, **24**, 916 (1956).

studies in the 700–7000 cm^{-1} region yield the following assignments: T_2O : ν_2 –1017.2 cm^{-1} , ν_3 –2438 $\pm$ 5 cm^{-1} ; THO : ν_2 –1358.8 cm^{-1} , ν_3 –3895 $\pm$ 10 cm^{-1} ; TDO : ν_3 –2829 $\pm$ 5 cm^{-1} . The values given are observed frequency plus the anharmonicity correction, and are in agreement with calculated values of ω except for ν_2 of THO , the calculated value of which was found to be 16 cm^{-1} higher than the value given here. This discrepancy was not explained and is greater than the estimated error in locating the center of the band.

The matrix isolation technique has been used to study the hydrogen bonding of water. The technique warrants some discussion. It generally consists of the dispersion of a chemically reactive material in a large amount of inert solid substance, the matrix, at a temperature sufficiently low to retard or prevent molecular diffusion of the sample. A glass or crystalline solid of suitable inertness, rigidity, transparency, and volatility can serve as the matrix. Theoretically, any molecule which is difficult to prepare because of its high reactivity or is unstable under the more conventional means may be handled in this way. The method has been tested using the free radical NO_2 , and the hydrogen bonding molecules— HBr , HCN , HN_3 , NH_3 , and H_2O —with nitrogen, argon, and xenon as matrices at 20° K.⁸⁹ The vibrational frequencies obtained when these molecules are isolated in the matrix in molar ratios of inactive to active material ranging from 100:1 to 500:1 are essentially the same as the frequencies in the gas phase. Using nitrogen at 20° K as a matrix, the stretching and bending modes of the hydrogen-bonded polymers of water were studied by Thiel, Becker, and Pimental.⁹⁰ Absorption frequencies for the monomers are 3725, 3627, 1600 cm^{-1} ; for the dimers, 3691, 3546, 1620 cm^{-1} ; and for the higher polymers, 3510, 3355, 3318, 3222, and 1633 cm^{-1} . The three possible structures of the dimeric molecule are as shown:

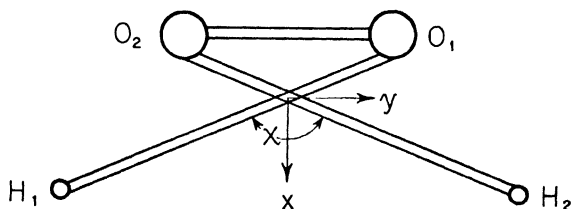
89. Becker, E. D., and Pimental, G. C., *J. Chem. Phys.*, **25**, 224 (1956).

90. Thiel, M. V., Becker, E. C., and Pimental, G. C., *J. Chem. Phys.*, **27**, 486 (1957).



The observed frequencies agree with the cyclic structure for the dimer rather than the open or bifurcated configuration. A slight decrease in the absorption coefficients of the bending modes was noted in the higher polymers, while an increase by a factor of about twelve was observed in the stretching modes. This intensity can hardly be explained on the basis of the enhanced ionic character of the O—H group only, and thus lends support to the currently accepted assumption of linearity of the H—bond.

Hirota⁹¹ has studied the rotational structure of the infrared absorption spectrum of hydrogen peroxide vapor. The H_2O_2 molecule is known to have C_2 symmetry. Its vibrational motions are (1) a symmetric O—H stretch, a symmetric H—O—O deformation, and an O—O stretch of class A except for internal rotation and (2) an antisymmetric O—H stretch and an antisymmetric H—O—O deformation of class B. The internal rotation of H_2O_2 was treated on the assumption that its potential energy can be approximated by a Fourier expansion. Considering the H_2O_2 molecule to have the structure:



with the z axis chosen through the centers of mass of two OH bonds, and with x the angle between the projections of these bonds on the plane perpendicular to the z axis, then using the two-

91. Hirota, E., *J. Chem. Phys.*, 28, 839 (1958).

term Fourier expansion: $V = (V_1/2) (1 + \cos \chi) \div (V_2/2) (1 + \cos 2\chi)$, and data obtained from infrared and microwave spectra, Hirota calculated values for V_1 and V_2 . These values were respectively 244 cm^{-1} (700 cal/mole) and 319 cm^{-1} (910 cal/mole). The doublet structure of the infrared absorption spectrum was attributed to the existence of a double minimum in the hindering potential, V . The height of the potential barrier for the *cis*-configuration was definitely greater than that for the *trans*-configuration.

Spectra of air in the region $400\text{--}5000 \text{ cm}^{-1}$ have been obtained with varying conditions of pressure, temperature, and concentration of carbon dioxide and water.⁹² New lines originating from levels of high energy were observed in the pure rotation spectrum and the ν_2 vibrational region of water and in the 2000 cm^{-1} and $500\text{--}909 \text{ cm}^{-1}$ region of the absorption spectrum of carbon dioxide. The observations were made with a high resolution spectrometer and represented the first attempt to study superheated steam under such resolution.

Oxides of Carbon. A number of studies have been made on carbon monoxide and carbon dioxide. The purposes of these have been largely (1) to determine molecular constants,⁹³⁻⁹⁵ (2) to investigate absorption after thermal excitation,⁹⁶ (3) to study spectra of the chemisorbed oxide,⁹⁷⁻⁹⁸ and (4) to measure quantitatively infrared intensities of the pressurized and unpressurized gases.⁹⁹ The Raman spectra and the infrared spectra of carbon

92. Taylor, J. H., Benedict, W. S., and Strong, J., *J. Chem. Phys.*, **20**, 1884 (1952).
93. Rossman, K., Rao, K. N., and Nielsen, H. H., *J. Chem. Phys.*, **24**, 103 (1956).
94. Nielsen, A. H., and Lagemann, R. T., *J. Chem. Phys.*, **22**, 36 (1954).
95. Rossman, K., France, W. L., Rao, K. N., and Nielsen, H. H., *J. Chem. Phys.*, **24**, 1007 (1956).
96. Tourin, R. H., *J. Chem. Phys.*, **20**, 1651 (1952).
97. Yang, A. C., and Garland, C. W., *J. Phys. Chem.*, **61**, 1504 (1957).
98. Eischens, R. P., Pliskin, W. A., and Francis, S. A., *J. Chem. Phys.*, **22**, 1986 (1954).
99. Renner, S. S., and Weber, D., *J. Chem. Phys.*, **19**, 807, 817, 1351 (1951).

suboxide have been reported independently by Rix¹⁰⁰ and Long.¹⁰¹

Oxides of Nitrogen. Perhaps the most widely studied oxides are those of nitrogen. These oxides are difficult to prepare in pure form free from water and other nitrogen oxides. However, their interesting chemical and physical behavior has led to extensive investigation of their molecular structures. Bands suitable for analytical purposes have been found in the 650–2500 cm^{-1} range for many of these compounds. The spectrum of N_2O_5 has been recorded with a fast-scanning spectrometer.¹⁰² A strong band was observed at 1240 cm^{-1} and another band at 1315 cm^{-1} . Initially the band at 1315 cm^{-1} was weak while the 1240 cm^{-1} band was intense. As the scanning was continued the 1315 cm^{-1} band became more intense, while the 1240 cm^{-1} band diminished in intensity. This is strong indication that the 1315 cm^{-1} band is that of HNO_3 formed by interaction of the N_2O_5 molecule with water.

Nitric oxide is a particularly interesting compound, being a stable diatomic molecule possessing an unpaired electron. The lowest electronic configuration for the molecule gives a $^2\Pi$ state as ground state. The components of the ground state are only slightly separated, giving rise to a unique infrared spectrum. Fletcher and Begun¹⁰³ observed the fundamental vibration of N^{15}O (1760–1920 cm^{-1}) under high resolution and calculated molecular constants which were in good agreement with those obtained by earlier studies. They also observed the spectra of the isotopic N_2O molecules: $\text{N}^{14}\text{N}^{15}\text{O}^{16}$, $\text{N}^{15}\text{N}^{14}\text{O}^{16}$, and $\text{N}^{15}\text{N}^{15}\text{O}^{16}$ in the 500–4000 cm^{-1} region.¹⁰⁴ Frequencies of three fundamental bands and fifteen overtone and combination bands were tabulated. Approximate zero-order frequencies were calculated for

100. Rix, H. D., *J. Chem. Phys.*, **22**, 429 (1954).

101. Long, D. A., Murfin, F. S., and Williams, R. C., *Proc. Roy. Soc. (London)*, **A223**, 251 (1954).

102. Nightingale, R. E., Downie, A. R., Rotenberg, D. C., Crawford, B. C., and Ogg, R. A., *J. Phys. Chem.*, **58**, 1047 (1954). (See also literature citations therein.)

103. Fletcher, W. H., and Begun, G. M., *J. Chem. Phys.*, **27**, 579 (1957).

104. Begun, G. M., and Fletcher, W. H., *J. Chem. Phys.*, **28**, 414 (1958).

the isotopic molecules, using known anharmonic constants and isotopic relations. Table 8 shows the agreement between observed and calculated values.

TABLE 8. ZERO-ORDER FREQUENCIES (cm^{-1}) OF ISOTOPIC NITROUS OXIDE MOLECULES

(After Begun and Fletcher ¹⁰⁴)

N_2^{14}O		$\text{N}^{14}\text{N}^{15}\text{O}$		$\text{N}^{15}\text{N}^{14}\text{O}$		N_2^{15}O	
Obs.	Calc.	Obs.	Calc.	Obs.	Calc.	Obs.	Calc.
1300.3	1299.7	1295.6	1298.5	1285.1	1281.4	1280.3	1280.6
596.5	596.5	582.8	582.8	593.2	592.9	578.9	579.2
2276.9	2277.0	2228.6	2226.9	2254.5	2256.7	2206.3	2205.9

Oxides of Halogens and Sulfur. The absorption spectra of ClO, BrO, and IO were observed by Durie and Ramsay ¹⁰⁵ during photolysis of mixtures of chlorine, bromine, or iodine with oxygen. Nielsen and Woltz ¹⁰⁶ have interpreted the spectrum of ClO₂ in the 250–5000 cm^{-1} region as follows: polymer band–290 cm^{-1} ; ν_1 –943.2 cm^{-1} ; ν_2 –455 cm^{-1} ; ν_3 –1110.5 cm^{-1} . The ν_2 bending mode at 455 cm^{-1} has the doublet character required if the molecular model is to be an obtuse triangle, as suggested by electron diffraction measurements. The band at 290 cm^{-1} was difficult to assign, but the suggestion was made that it may be ascribed to a polymeric form of ClO₂.

The dioxide of sulfur at 95° K has crystal structure C_{2v}^{17} with two molecules per unit cell (orthorhombic). In such a structure the SO₂ each occupy sites of C₂ symmetry. The fundamental vibrations, ν_1 (a_1) and ν_2 (a_1), yield one infrared active component, while the b_1 fundamental, ν_3 , is split into two components by the crystal-line field, both of these components being infrared-active. Wiener and Nixon ¹⁰⁷ measured the infrared transmission of solid SO₂ at

105. Durie, R. A., and Ramsay, D. A., *Can. J. Phys.*, **36**, 35 (1958).

106. Nielsen, A. H., and Woltz, P. J. H., *J. Chem. Phys.*, **20**, 1878 (1952).

107. Wiener, R. N., and Nixon, E. R., *J. Chem. Phys.*, **25**, 175 (1956).

93° K. Spectral studies performed by Giguere and Falk¹⁰⁸ are in agreement (Table 9).

TABLE 9. INFRARED ABSORPTION FREQUENCIES OF SULFUR DIOXIDE (cm⁻¹)

Assignment	Gaseous * SO ₂	Solid SO ₂ (Wiener and Nixon)	Solid SO ₂ (Giguere and Falk)
ν_2 , bending	518	521	528-35 (doublet)
$\nu_1 - \nu'$		1142	
ν_1 , symmetric stretch	1151	1147	1144
$\nu_3 - \nu''$		1316	
ν_3 , asymmetric stretch	1361	1308 1330 } doublet	1310-22 (doublet)
$\nu_1 + \nu_3$		2460 2436 } doublet	2455

* Polo, S. R., and Wilson, M. K., *J. Chem. Phys.*, 22, 900 (1954).

An interesting study of the absorption of SO₂ and CO₂ in aqueous solution was made by Jones and McLaren.¹⁰⁹ Solutions were prepared by bubbling the gas through water at room temperature. The infrared and Raman spectra show that in the aqueous phase, SO₂ is present mostly as SO₂ rather than as H₂SO₃. This is consistent with data obtained by conductance and vapor pressure measurements and from ultraviolet absorption data. The results obtained are as shown below with comparable data from Herzberg on the gas phase. (After Jones and McLaren.¹⁰⁹)

		Aqueous	Gaseous
SO ₂	ν_3	1332 cm ⁻¹	1361 cm ⁻¹
	ν_1	1157	1151
CO ₂	ν_3	2342	2349

108. Giguere, P. A, and Falk, M., *Can. J. Chem.*, 34, 1833 (1956).

109. Jones, L. H., and McLaren, E., *J. Chem. Phys.*, 28, 995 (1958).

The frequency shift for CO_2 is not appreciable; however, for SO_2 the shift from gaseous to aqueous phase is 29 cm^{-1} . Carbon dioxide, a linear, symmetric molecule, having no dipole moment, will not interact as strongly with water as will SO_2 , which is a non-linear, symmetric molecule with a dipole moment. This is actually manifested in the observed difference in magnitude of the ν_3 shift in the two molecules.

Oxygen Compounds of Metals. The infrared spectra of Li_2O_2 , $\text{Li}_2\text{O}_2 \cdot 8\text{H}_2\text{O}$, Na_2O_2 , $\text{Na}_2\text{O}_2 \cdot 8\text{H}_2\text{O}$, $\text{Na}_2\text{O}_2 \cdot 8\text{D}_2\text{O}$, NaO_2 , KO_2 , $\text{CaO}_2 \cdot 8\text{H}_2\text{O}$, SrO_2 , and BaO_2 were obtained by Brame and co-workers¹¹⁰ in order to search for a possible existence of the O_3^- ion as well as to investigate the peroxide and peroxide hydrate structures. No characteristic sharp bands were observed for peroxide, O_2^{2-} , or superoxide, O_2^{-1} . This is consistent with the theory that both ions are homopolar and consequently have no infrared-active vibrations. There were no absorption bands attributable to the O_3^{2-} ion. Similarly, Giguere and Harvey¹¹¹ were unable to show any evidence of discrete O_3^{2-} ions in the infrared spectrum of K_2O_3 .

Brame and his co-workers¹¹² were able to locate at least one strong absorption band in each of four oxide samples investigated. This band, observed in B_2O_3 , Al_2O_3 , Cr_2O_3 , and Ga_2O_3 , was probably due to a metal-oxygen vibration, the band shifting to longer wavelengths with increase of mass in the series.

The infrared spectrum of osmium tetroxide, OsO_4 , has been obtained under conditions of its own vapor pressure and temperatures up to about 130°C .¹¹³ Four bands— $\nu_1(a_1)$ 971 cm^{-1} *; $\nu_2(e)$ 328 cm^{-1} ; $\nu_3(f_2)$ 959.7 cm^{-1} ; and $\nu_4(f_2)$ 328 cm^{-1} —were positively attributed to OsO_4 . It was assumed that the molecular

110. Brame, E. C., Cohen, S., Margrave, J. L., and Meloche, V. W., *J. Inorg. and Nucl. Chem.*, **4**, 90 (1957).

111. Giguere, P. A., and Harvey, K. B., *J. Am. Chem. Soc.*, **76**, 5891 (1954).

112. Brame, E. G., Margrave, J. C., and Meloche, V. W., *J. Inorg. Nucl. Chem.*, **5**, 48 (1957).

113. Hawkins, N. J., and Sabol, W. W., *J. Chem. Phys.*, **25**, 775 (1956).

* Data of Langseth and Quiller, *Z. Physik. Chem.*, **B27**, 79 (1934).

model is an XY_4 model of T_d point group symmetry. If this is the proper model, one would expect to find in the $660\text{--}580\text{ cm}^{-1}$ region the overtone band, $2\nu_4$, the combination band, $\nu_2 + \nu_4$, and the difference bands involving the stretching frequency less the deformation frequency. The spectrum obtained was quite simple, having the two stretching vibrations, ν_1 and ν_3 , of almost equal values and the two deformations, ν_2 and ν_4 , of equal value and containing none of the overtone or combination bands.

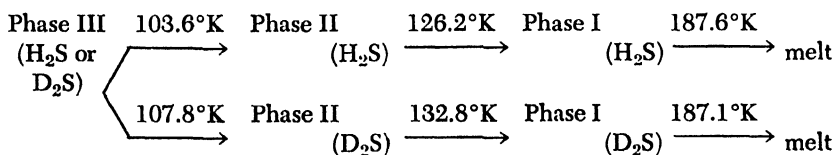
Other Binary Compounds

The infrared spectra of gaseous hydrogen sulfide, H_2S , and the deuterated species, HDS and D_2S , have been studied under high resolution in a number of regions. The reader is referred to the original works and the references cited therein.¹¹⁴⁻¹²¹ The H_2S , HDS , and D_2S molecules have spectra which are similar to those obtained for the corresponding oxygen analogs. Actually, the Group VI elements O, S, Se, and Te form with hydrogen a series of asymmetric top molecules with interesting spectral similarities. Hydrogen selenide, when examined in the far infrared, exhibits bands suggesting that the molecule is an almost accidentally symmetric rotor with apex angle $2\alpha \approx 90^\circ$ and $a \approx b \approx 2c$, where a , b , and c are reciprocals of the moments of inertia.¹²²

114. Allen, H. C., and Plyler, E. K., *J. Research Natl. Bur. Standards*, **52**, 205 (1954) H_2S (6290 cm^{-1}); *J. Chem. Phys.*, **22**, 1104 (1954) H_2S (5100 cm^{-1}); *J. Chem. Phys.*, **25**, 1132 (1956) H_2S (1250 cm^{-1}).
115. Innes, K. K., Cross, P. C., and Bair, E. J., *J. Chem. Phys.*, **21**, 545 (1953) H_2S (8700 cm^{-1}) and (11000 cm^{-1}).
116. McCubbin, T. K., *J. Chem. Phys.*, **20**, 668 (1952) H_2S ($100\text{--}143\text{ cm}^{-1}$).
117. Allen, H. C., Blaine, C. R., Plyler, E. K., and Cross, P. C., *J. Chem. Phys.*, **24**, 35 (1956) H_2S ($2200\text{--}2800\text{ cm}^{-1}$).
118. Ordway, G. L., Cross, P. C., and Bair, E. J., *J. Chem. Phys.*, **23**, 541 (1955) H_2S ($7480\text{--}7880\text{ cm}^{-1}$).
119. Savage, C. M., and Edwards, T. H., *J. Chem. Phys.*, **27**, 179 (1957) H_2S (3846 cm^{-1}).
120. Crosby, G., Bair, E. J., and Cross, P. C., *J. Chem. Phys.*, **23**, 1660 (1955) HDS (6140 cm^{-1}).
121. Allen, H. C., Plyler, E. K., and Blaine, L. R., *J. Research Nat'l. Bur. Standards*, **59**, 211 (1957) D_2S (2000 cm^{-1} and 2778 cm^{-1}).
122. Palik, E. D., *J. Chem. Phys.*, **23**, 980 (1955).

The most nearly symmetric member of the entire series is hydrogen telluride, H_2Te . The particular chemical instability of the gas presents experimental difficulties in obtaining its high resolution spectrum. However, Rossmann and Straley¹²³ have succeeded in stabilizing it for a long enough period to obtain spectra and data sufficient to calculate molecular constants. They identified two overlapping fundamentals, ν_1 and ν_3 , extending from $1800\text{--}2200\text{ cm}^{-1}$ and a fundamental ν_2 centering at about 870 cm^{-1} . The angle H-Te-H was reported as $89^\circ 30'$ with H-Te distance equal to $1.7 \times 10^{-8}\text{ cm}$, $I_A = I_B = 4.6 \times 10^{-40}\text{ g cm}^2$ and $I_c = 9.2 \times 10^{-40}\text{ g cm}^2$.

The infrared spectra of H_2S in phases III, II, and I have been studied at 66°K , 112°K , and 140°K , respectively, and that of D_2S in phase III at 66°K .¹²⁴ Extremely sharp lines were observed in the spectra of phase III, suggesting an ordered structure which presumably consisted of eight molecules in a primitive unit cell of D_{4h} symmetry. Broader bands, indicating disordered arrangement of the hydrogen atoms, were noted in phases II and I. The three phases are related as shown below:



From the spectra of the crystalline form it was concluded that the fundamental frequencies of gaseous D_2S must be very nearly 855 cm^{-1} for ν_2 and 1910 cm^{-1} for ν_3 . Examination with a polarizing microscope showed phase III to be anisotropic, while phases II and I are perhaps isotropic.

XH₃ Molecules. Molecules of the type XH_3 , where X is N, As, P, or Sb and H is hydrogen or deuterium, have been examined in

123. Rossmann, K., and Straley, J. W., *J. Chem. Phys.*, **24**, 1276 (1956).

124. Reding, F. P., and Hornig, D. F., *J. Chem. Phys.*, **27**, 1024 (1957).

the infrared region to some extent.¹²⁵⁻¹²⁸ The ammonia molecule, NH_3 , is a symmetrical pyramidal structure of point group C_{3v} and, as such, has two totally symmetric a fundamentals and two doubly degenerate e fundamentals. All are active in the infrared and Raman. Infrared absorption spectra of ammonia containing equimolar proportions of deuterium and hydrogen seem to indicate a disproportionation. The infrared region seems particularly suitable to determine the molecular species arising from partial deuteration of ammonia. The $700\text{--}1000\text{ cm}^{-1}$ region contains only one fundamental vibrational mode for NH_3 and ND_3 . The intensities are strong and the only other observations in this region are the relatively much weaker rotational components. Arsine, AsH_3 , has band structure in the region $857\text{--}1130\text{ cm}^{-1}$. Phosphine, PH_3 , has two fundamentals, ν_1 , and ν_3 , which lie near 2381 cm^{-1} . The four fundamentals of stibine, SbH_3 , and the two high-frequency fundamentals of deuterio-stibine have been observed and sufficient analysis made to determine the size and shape of the molecule and to correct the band centers for anharmonicity and thus indicate the normal frequencies of the molecule with a reasonable degree of accuracy (Table 10).

TABLE 10. FUNDAMENTAL BANDS OF XY_3 MOLECULES (cm^{-1})

	NH_3^a	ND_3^a	PH_3^b	SbH_3^c	SbD_3^c		SbH_3^c	SbD_3^c
ν_1	3335.9 3337.5	2419	2322.9	1890.9	1358.8	ω_1	1988.9	1409.4
ν_2	931.58 968.08	748.6 749.0	992.0	781.5	561.1	ω_2	795.9	568.5
ν_3	3414	2555	2327.7	1894.2	1362.0	ω_3	1974.5	1403.5
ν_4	1627.5	1191.0	1122.4	830.9	592.5	ω_4	844.7	599.5

^a Herzberg; ^b (127); ^c (128).

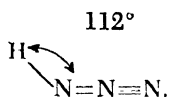
125. Stedman, D. F., *J. Chem. Phys.*, **20**, 718 (1952) ($\text{NH}_3\text{--ND}$).

126. Nielsen, H. H., *Discussions Faraday Soc.*, No. 9, 85 (1950) AsH_3 .

127. McConaghie, V. M., and Nielsen, H. H., *J. Chem. Phys.*, **21**, 1836 (1953) PH_3 .

128. Haynie, W. H., and Nielsen, H. H., *J. Chem. Phys.*, **21**, 1839 (1953) $\text{SbH}_3 + \text{SbD}_3$.

HN₃ Molecules. The infrared spectra of gaseous and solid hydrazoic acid, HN₃, and deuterohydrazoic acid, DN₃, have been investigated by Dows and Pimentel.¹²⁹ In the solid state of both species, hydrogen bonding was manifested through frequency shifts of the N—H and N—D stretching vibrations, 216 and 151 cm⁻¹, respectively. In carbon tetrachloride solutions of HN₃ up to 0.3M, hydrogen bonding was not evident. The structure of HN₃ is known from data obtained from infrared and microwave studies.¹³⁰ An angle of 112° exists between the N—H bond and the three colinear nitrogen atoms,



The molecule has C_s symmetry with five in-plane vibrations (type a') and one out-of-plane vibration (type a''), all of which are active in both infrared and Raman. The molecule approaches the symmetric top model and, if treated as such, one might expect the out-of-plane vibration to be a perpendicular band and the in-plane vibrations to be hybrids of superimposed parallel and perpendicular bands. The parallel bands should be similar to those of a linear molecule, i.e., the Q branch should be weak. Calculations of the separations of the P and R branch maxima indicate 26 cm⁻¹ for both HN₃ and DN₃, while the Q branch separation of the perpendicular bands should be 36 cm⁻¹ apart for HN₃ and 22 cm⁻¹ apart for DN₃. Table 11 shows the frequencies observed for gaseous and solid states of HN₃ and DN₃. (On warming solid hydrazoic acid, an irreversible phase transition occurs above about 120°K.)

Upon substitution with deuterium, the ν_3 fundamental acquires more of a bending motion. The combination band $\nu_3 + \nu_4$ is not appreciably affected and is observed both in HN₃ and DN₃; however, the first overtone of ν_4 is observed only in HN₃ and that of ν_3 observed only in DN₃. Hydrogen bonding is suggested from the following observations in going from the gaseous to the solid

129. Dows, D. A., and Pimentel, G. C., *J. Chem. Phys.*, **23**, 1258 (1955).

130. Amble, E., and Dailey, B. P., *J. Chem. Phys.*, **18**, 1422 (1950).

TABLE 11. FREQUENCIES OF GASEOUS AND SOLID HN_3 AND DN_3
(After Dows and Pimentel¹²⁹)

Assignment	$\text{HN}_3(\text{g})$	$\text{HN}_3(\text{SII})$	$\text{HN}_3(\text{SI})$	$\text{DN}_3(\text{g})$	$\text{DN}_3(\text{SII})$	$\text{DN}_3(\text{SI})$
ν_1	3336 m	3120 vs	3090	2480 m	2329 s	2308
$\nu_2 + \nu_4$	3250 w	3305 w				
$2\nu_3$				2358 m	2442 m	2412
$\nu_2 + \nu_7$		2435 w			2380 w	
$\nu_3 + \nu_4$	2402 m	2402 m		2170 w	2202 w	
$2\nu_4$	2304 s	2298 w	2349			
ν_2	2140 vs	2169 vs	2162	2141 vs	2165 vs	2155
ν_3	1274 m	1280 m	1299	1183 m	1229 m	1230
ν_4	1150 vs	1182 s	1180	955 s	958 s	977
$\nu_2 - \nu_4$	950 vw					
ν_6	672 w			638 w		
ν_5	522 w			498 w		
ν_7		266 (?)			215 (?)	

states: (1) bands associated with N—H and N—D stretching modes shift to lower frequencies and are more intense relative to other bands, and (2) all fundamentals ν_1 , ν_2 , ν_3 , and ν_4 shift to higher frequencies.

Gray and Waddington¹³¹ have observed the fundamental vibrational frequencies of the azide ion, N_3^- , with various cations: Li, Na, K, Rb, Cs, Ca, Sr, and Ba. The symmetric stretching vibration, ν_1 , and the first overtone of the ν_2 deformation mode were observed in the Raman, while the fundamental ν_2 and the asymmetric stretching frequency, ν_3 , were observed in the infrared. The ν_1 symmetrical stretching frequency for the compounds containing monovalent cations decreased from 1368.7 cm^{-1} for lithium to 1328.6 cm^{-1} for cesium. For the divalent cations the range was 1380.5 cm^{-1} for calcium to 1354.4 cm^{-1} for barium. The values ν_2 and ν_3 for the entire series of compounds were $642 \pm 8 \text{ cm}^{-1}$ and $2076 \pm \text{cm}^{-1}$, respectively. Ammonium azide, which has been studied in some detail by Dows and co-workers,¹³²

131. Gray, P., and Waddington, T. C., *Trans. Faraday Soc.*, **53**, 901 (1957).

132. Dows, D. A., Whittle, E., and Pimentel, G. C., *J. Chem. Phys.*, **23**, 1475 (1955).

has the values: ν_1 -1345 cm^{-1} , ν_2 -661 (650) cm^{-1} , and ν_3 -2041 cm^{-1} . An additional assignment of a combination band in NH_4N_3 at 1830 cm^{-1} has thrown some light on the question of the free rotation of the ammonium ion in the solid state. The behavior of the ammonium ion in a lattice has received considerable attention. Studies made with solid NH_4Cl and NH_4Br suggest that the ion does not undergo free rotation in the lattice.¹³³ On the other hand, studies with NH_4I indicate a temperature-dependent, one-dimensional free rotation between the two ions.¹³⁴

On the basis of proton magnetic resonance studies, the ammonium ion is hindered with respect to rotation in solids.¹³⁵ Dows *et al.*¹³² investigated the ammonium azide molecule in an attempt to gain an understanding of the contrasting behavior already reported for the ammonium halides. The crystal structure of ammonium azide, NH_4N_3 , at room temperature is D_{2h}^7 with four molecules per unit cell. Four azide ions surround each ammonium ion at approximately tetrahedral angles. Two slightly different distances (2.94 Å and 2.99 Å) exist between the nitrogen atom of each ammonium ion and the nearest nitrogen atoms of the four neighboring azide ions. The ammonium ion has site symmetry C_2 . According to the site group approximation, nine nondegenerate infrared and Raman-active vibrations are expected. The linear, symmetric azide ions are equally distributed between two sets of C_{2h} symmetry sites. Both sets give equivalent results; however, the frequencies for the two kinds of azides are not necessarily identical. In the approximation, four nondegenerate vibrations are allowed: two arising from the degenerate bending mode of the hypothetical free azide ion, one active only in the Raman, and one active only in the infrared. A torsional motion of the ammonium ion, ν_6 (420 $\pm$ 20 cm^{-1}), shows that it does not rotate freely in crystalline NH_4N_3 . A combination band at 1830 cm^{-1} , involving ν_6 and ν_4 , the NH bending mode, was also assigned. The relative magnitudes of the frequencies of these torsional modes of NH_4^+ are consistent, at least qualitatively, with the hydrogen bond ener-

133. Wagner, E. L., and Hornig, D. F., *J. Chem. Phys.*, **18**, 305 (1950).

134. Plumb, R. C., and Hornig, D. F., *J. Chem. Phys.*, **21**, 366 (1953).

135. Gutowsky, H. S., Pake, and Bersohn, *J. Chem. Phys.*, **22**, 643 (1954).

gies inferred from frequencies of the N—H stretching modes in ammonium azide, chloride, and bromide.

The technique of infrared spectroscopy has been used further to study the species formed in the glow-discharge decomposition of hydrazoic acid, HN_3 , when the products are allowed to condense on a surface cooled to 90°K .¹³⁶ Spectra were taken at several temperatures up to 230°K , and absorption bands attributable to HN_3 , NH_4N_3 , NH_3 and two intermediates were observed. The intermediate species were assumed to be N_2H_2 and $(\text{NH})_x$, where x may be as high as 4.

Other Binary Nitrogen Compounds. The hydrazine molecule, N_2H_4 , has been examined in the infrared from $400\text{--}6667\text{ cm}^{-1}$. The spectra of the liquid and of the solid at -70°C were observed. The symmetry of the molecule is C_2 and one expects twelve non-degenerate fundamental modes: four N—H stretching vibrations, six N—H deformation vibrations, an N—N stretching mode, and a torsional oscillatory mode. The identification and assignment of vibrational bands were not complete because of extensive overlapping in the observed spectra.

The molecule N_4S_4 has been studied in sufficient detail to make complete assignment of the frequencies of its normal vibrational modes.¹³⁷ The data were analyzed in terms of several proposed structures and were found to be consistent with a cage structure of D_{2d} symmetry. Force constants were calculated for the N—S bond stretch, and the N—N bond stretch, the N—N—S angle bend, the N—N bond to N—N bond, the N—S bond to N—S bond, and the N—N—S angle to N—N—S angle.

XH₄ Molecules. Molecules and ions of this type where X is C, Si, Ge, N, P, or Al and H is hydrogen and/or its isotopes have been examined in the infrared region by several investigators.^{138–140} Some of the observed frequencies are tabulated in Table 12.

136. Dows, D. A., Pimentel, G. C., and Whittle, E., *J. Chem. Phys.*, **23**, 1606 (1955).

137. Lippincott, E. R., and Tobin, M. C., *J. Chem. Phys.*, **21**, 1559 (1953).

138. Meal, J. H., and Wilson, M. K., *J. Chem. Phys.*, **24**, 385 (1956).

139. Pistorius, C. W. F. T., *J. Chem. Phys.*, **27**, 965 (1957).

140. Morgan, H. W., Staats, P. A., and Goldstein, J. H., *J. Chem. Phys.*, **27**, 1212 (1957).

TABLE 12. SOME OBSERVED FREQUENCIES (cm^{-1}) OF XH_4 TYPE MOLECULES AND IONS

	Symmetry	$\nu_1(a_1)$	$\nu_2(e)$	$\nu_3(f_2)$	$\nu_4(f_2)$
CH_4^a	T_d	2914.2	1520	3020	1305
CD_4^a	T_d	2084.7	1075	2259.3	995.86
SiH_4^a	T_d	2180	970	2183	910
SiH_4^b	T_d	2187	974.6	2190.6	914.2
SiD_4^b	T_d	1545	689	1597	681
GeH_4^a	T_d	1990	833	2110	933
NH_4^{+a}	T_d	3040	1680	3145	1400
NH_4^{+c}	T_d		1649	3146	1404
ND_4^{+a}	T_d	2214	1215	2346	1065
ND_4^{+c}	T_d		1186	2350	1066
NT_4^{+c}	T_d		976	2022	913
NHT_3^{+c}				3084 (?)	1345 (?)
NDT_3^{+c}				2319 (?)	
PH_4^{+a}	T_d	2304	1040	2370	930
AlH_4^{-a}	T_d	1790	799	1741	769
SiHD_3^b	C_{3v}	1573	683	2182(a_1)	851(e)
				1598(e)	683(a_1)
SiH_2D_2	C_{2v}	1587	944(a_1)	2189(a_1)	862(b_1)
			844(a_2)	2183(b_2)	743(b_2)
				1601(b_1)	682.5(a_1)

^a (139) ^b (138) ^c (140)

Note: For the structures of symmetry type other than T_d , the species representation, wherever it differs from that of T_d , is indicated parenthetically to the right of the observed frequency.

Digermane and Disilane. These two compounds, Ge_2H_6 and Si_2H_6 , are analogous to ethane, C_2H_6 . A knowledge of their molecular structures and intermolecular forces would contribute to the understanding of the chemistry of germanium and silicon compounds, just as knowledge of ethane structure has to the chemistry of carbon compounds. The spectrum of gaseous Si_2H_6 was observed in the $350\text{--}4000\text{ cm}^{-1}$ region¹⁴¹ and that of gaseous and solid Ge_2H_6 observed in the $400\text{--}4000\text{ cm}^{-1}$ region.¹⁴² Like ethane, one expects the structure of these molecules to be D_{3h} or D_{3d} (or

141. Gutowsky, H. S., and Stejskal, E. O., *J. Chem. Phys.*, **22**, 939 (1954).142. Dows, D. A., and Hexter, R. M., *J. Chem. Phys.*, **24**, 1029 (1956).

free rotation about the Si—Si or Ge—Ge axis). Four of the five infrared-active fundamentals of disilane were observed and assigned: $\nu_5 - 2154.4 \text{ cm}^{-1}$, $\nu_6 - 842.7 \text{ cm}^{-1}$ (both parallel bands) and $\nu_7 - 2181.1 \text{ cm}^{-1}$, $\nu_8 - 945.7 \text{ cm}^{-1}$ (both perpendicular bands). The infrared fundamentals of gaseous digermane are: $a_{2u} - 2078, 755$, $e_u - 2114, 898, 407 \text{ cm}^{-1}$; $a_{1u} - 144 \text{ cm}^{-1}$; $a_{1g} - 2070, 765, 229 \text{ cm}^{-1}$; and $e_g - 2150, 875, 417 \text{ cm}^{-1}$. The solid spectrum at 90°K showed only the fundamentals active in the gas phase.

Binary Intermetallic Compounds. The binary intermetallic compounds of interest are those which have been studied primarily with reference to their electrical properties. Absorption processes are generally more easily interpreted and understood than are the electrical properties of these materials. Consequently, increasing emphasis is being placed on the study of infrared absorption of intermetallics. A number of review articles¹⁴³ and books^{38, 39, 44} cover the work already published in this field. Blunt, Frederikse, and Hosler¹⁴⁴ have investigated n and p -type magnesium stannide, Mg_2Sn , between 2 and 15μ . Both n and p -type show a relatively strong absorption with a maximum at approximately 1333 cm^{-1} at room temperature (300°K). The room-temperature absorption is much larger than absorption at low temperature due to the high concentration of intrinsic carriers ($\sim 4 \times 10^{17} \text{ cm}^{-3}$). For p -type samples, no absorption band was found in any of the low temperature spectral curves; however, a faint absorption was still observable in the n -type samples at 85°K and 5°K . The value of the energy gap, obtained by extrapolating the absorption edge to $\alpha = 0$, is $\sim 0.22 \text{ ev}$ at 300°K , 0.30 ev at 85°K , and 0.33 ev at 5°K . Electrical measurements give a value of 0.33 ev at 0°K . The value deduced from photoconductivity threshold 2381 cm^{-1} is about 0.03 ev lower.

143. Hrostowski, H. J., "Infrared Absorption of Semiconductors," Chapter 10 in *Semiconductors*, N. B. Hannay, Editor, Reinhold Publishing Corporation, New York, 1959.

144. Blunt, R. F., Frederikse, H. P. R., and Hosler, W. R., *Phys. Rev.*, **100**, 663 (1955).

The Group III-Group IV¹⁴⁵⁻¹⁴⁹ and Group III-Group VI¹⁵⁰ compounds have been studied by a number of investigators, with the result that, at least for some of the structures, important correlations between absorption and band structure exist. The IIIB-VB compounds (InSb, GaSb, and AlSb) have the zincblende structure, equivalent to the diamond structure when the two components are the same. Indium antimonide has a small forbidden energy gap and high electron mobility. Between 3333 and 1429 cm^{-1} the absorption edge changes from sample to sample^{147, 151, 152} in both *n* and *p*-types. The absorption edge shift with temperature is greater for *n*-type samples than for *p*-type.

Samples of *p*- and *n*-type gallium antimonide¹⁴⁹ and aluminum antimonide¹⁴⁸ do not show the anomalous absorption edge change from sample to sample observed with indium antimonide. For GaSb, the absorption edge occurs at approximately 5556-6667 cm^{-1} . Measurements taken at 10°K, 80°K, and 300°K, show clearly the shift of absorption edge, while the long wavelength tail changes very little with the temperature variation. The forbidden energy gap is 0.775 eV at 0°K. For AlSb, a sharp absorption edge is observed at 12,220 cm^{-1} , corresponding to a forbidden energy gap of 1.52 eV at room temperature.

Typical of the studies made in an attempt to determine energy gaps, carrier mobilities, and other properties of importance in semiconductor application is the work of Black *et al.*¹⁵³ on M_2^{VB} -

145. Avery, D. G., Goodwin, D. W., Lawson, W. D., and Moss, T. S., *Proc. Phys. Soc. (London)*, **67B** (1954) InSb.
146. Cunnell, F. A., Edmond, J. T., and Richards, J. L., *Proc. Phys. Soc. (London)*, **67B**, 848 (1954) AlSb, GaSb, InSb, InAs, InP, GaAs.
147. Breckenridge, R. G., Blunt, R. F., Hosler, W. R., Frederikse, H. P. R., Becker, J. H., and Oshinsky, W., *Phys. Rev.*, **96**, 571 (1954) InSb.
148. Blunt, R. F., Frederikse, H. P. R., Becker, J. H., and Hosler, W. R., *Phys. Rev.*, **96**, 578 (1954) AlSb.
149. Blunt, R. F., Hosler, W. R., and Frederikse, H. P. R., *Phys. Rev.*, **96**, 576 (1954) GaSb.
150. Harbeke, G., and Lautz, G., *Optik*, **14**, 547 (1957) Ga_2Te_3 , In_2Te_3 .
151. Tanenbaum, M., and Briggs, H. B., *Phys. Rev.*, **91**, 1561 (1953).
152. Burstein, E., *Phys. Rev.*, **93**, 632 (1954).
153. Black, J., Conwell, E. M., Seigle, L., and Spencer, C. W., *J. Phys. Chem. Solids*, **2**, 240 (1957).

TABLE 13. OPTICAL PROPERTIES OF $M_2^{VB}-N_3^{VIB}$ COMPOUNDS

(After Black, Conwell, Seigle, and Spencer 153)

Compound *	Lattice	Space Group	Short Wave— Length Edge, μ	Remarks	Optical Energy Gap (ev at 300°K)
As_2Te^*	Monoclinic	$C_{2h}^3(A-m)$	1.1	Polycrystalline	1.0
Sb_2Se_3	Orthorhombic	$D_{2h}^{16}(Pbnm)$	< 2	0.1 mm thick sample; $T \cong 30\%$ from 2-15 μ	1.7
Sb_2Se_3	Orthorhombic	$D_{2h}^{16}(Pbnm)$	~ 1.0	21% T, illumination to cleavage plane, 6% T, $\perp$	1.2
Sb_2Te_3	Rhombohedral	$D_{3d}^5(R\bar{3}m)$		Poor T, 0.04 mm thick sample transmitted a little from 3.6-5 μ	0.3
$Bi_2S_3^*$	Orthorhombic	$D_{2h}^{16}(Pbnm)$		0.6 mm thick sample. Results indicate better samples need be used.	1.3
Bi_2Se_3	Rhombohedral	$D_{3d}^5(R\bar{3}m)$		Transmits < 5% from 3.5-5 μ ; 0.03 mm thick sample, illumination $\perp$ to plane.	(0.4 at liq. N_2 °T.) 0.35
Bi_2Te_3	Rhombohedral	$D_{3d}^5(R\bar{3}m)$	Falls slowly below 8.2	$\sim 0.1\%$ T, illumination to cleavage plane at 300°K, 1.4-4% T at liq. N_2 °T. (0.06 mm thick)	0.16 (78°K)
$As_2Se_3^*$			0.77	Amorphous	1.6

* All samples were single crystals except those whose formulas appear with an asterisk.

N_3^{vib} compounds. Data obtained by optical and electrical measurements are tabulated in Table 13.

TERNARY COMPOUNDS

Boron and Carbon Compounds

Boric Acid. Polycrystalline mulls and single crystals of boric acid, $\text{B}(\text{OH})_3$, and deuteroboric acid, $\text{B}(\text{OD})_3$, have been examined in the $500\text{--}3600\text{ cm}^{-1}$ region by Bethel and Sheppard and the results compared with earlier work.¹⁵⁴ The data support a slightly distorted C_{3h} crystal site symmetry for the molecule and C_{6h} for the layer structure of boric acid. The results of this work have been confirmed by the independent works of Hornig and Plumb¹⁵⁵ and Servoss and Clark.¹⁵⁶

In the work of Hornig and Plumb, spectra were obtained at 25° and -195°C of $\text{B}(\text{OH})_3$ as randomly oriented crystallites and at 25°C as a single crystal. The spectra were interpreted as skeletal vibrations of $\text{B}(\text{OH})_3$ molecules plus vibrations of the hydrogens associated with the hexagonal $\text{O—H}\dots\text{O}$ rings. The symmetry about the boron atoms is almost trigonal. The masses and forces in the boric acid molecule are similar to those in the boron trifluoride molecule, BF_3 , and consequently one might expect similar skeletal frequencies and the same isotope effect (Table 14).

Borates. Samples of Mg, Ca, Ni, Mn, Sr, and Ba orthoborates and of Na, Pb and Ba metaborates absorb in the $2000\text{--}700\text{ cm}^{-1}$ region.¹⁵⁷ The spectra indicate that the BO_2 ion does not exist; rather, the ion is present as the polymerized unit, $(\text{BO}_2)_n$, where n depends on the age of the sample. For freshly prepared samples, one may postulate $n = 2$ or 3 , but the exact structure cannot be accurately determined. For aged samples, the extent of polymerization is certainly greater. The aqueous borate ion has been observed in Raman spectra and from the number, shape, and position

154. Bethel, D. E., and Sheppard, N., *Trans. Faraday Soc.*, **51**, 9 (1955).

155. Hornig, D. F., and Plumb, R. C., *J. Chem. Phys.*, **26**, 637 (1957).

156. Servoss, R. R., and Clark, H. M., *J. Chem. Phys.*, **26**, 1175 (1957).

157. Duval, C., and Lecomte, J., *Bull. soc. chim. France*, **101**, 1952; *J. Opt. Soc. Amer.*, **44**, 261 (1954).

TABLE 14. COMPARISON OF SKELETAL FREQUENCIES (cm^{-1}) OF B(OH)_3 AND BF_3 (After Hornig and Plumb ¹⁵⁵)

Vibration	B(OH)_3	BF_3 (Herzberg)
ν_1	885	888
ν_2	666(B^{10}) 632(B^{11})	720(B^{10}) 691(B^{11})
ν_3	1490	1497(B^{10}) 1446(B^{11})
ν_4	552	482(B^{10}) 480(B^{11})

of the lines the ion has been described as B(OH)^- with tetrahedral symmetry.¹⁵⁸

Borates of lanthanum, scandium, and indium having $\text{B}^{10}/\text{B}^{11}$ ratios varying from the natural abundance 18/82, up to about 96/4, yield four fundamental frequencies which fall within the ranges: $\omega_1 = 939 \text{ cm}^{-1}$, $\omega_2 = 740\text{--}790 \text{ cm}^{-1}$, $\omega_3 = 1265\text{--}1330 \text{ cm}^{-1}$, and $\omega_4 = 606\text{--}675$.¹⁵⁹ Lanthanum borate, unlike the other two salts, has less than threefold site symmetry and was the only one of the three borates for which the totally symmetric frequency ω_1 was observable.

Metal Carbonates. The infrared spectra of various carbonates, basic carbonates, bicarbonates and related materials have been studied by Hunt and co-workers¹⁶⁰ and by Miller and Wilkins.¹⁹ A strong band in the $1450\text{--}1410 \text{ cm}^{-1}$ region and a less intense one in the $880\text{--}850 \text{ cm}^{-1}$ region are characteristic of normal carbonates. For the $880\text{--}850 \text{ cm}^{-1}$ band, an almost linear relationship exists between the observed frequency and the logarithm of the mass of the cation. Lithium carbonate was the only compound which did not follow this relationship. The studies with bicarbonates, basic carbonates, and related materials suggest that suffi-

158. Edwards, J. O., Morrison, G. C., Ross, V. F., and Schultz, J. W., *J. Am. Chem. Soc.*, **77**, 266 (1955).

159. Steele, W. C., *J. Chem. Phys.*, **25**, 1184 (1956).

160. Hunt, J. M., Wisherd, M. P., and Bonham, L. C., *Anal. Chem.*, **22**, 1478 (1950).

cient spectral changes occur to make possible use of infrared spectra for aids in identification purposes. However, considerably more study is needed to make significant correlations. Louisfert and Pobequin¹⁶¹ have successfully differentiated between crystal forms of calcium carbonate by means of infrared absorption and found the method to be more sensitive than the usual x-ray diffraction methods.

The infrared absorption spectra of carbonates which crystallize in the aragonite-type lattice show considerable fine structure in the 880–800 cm^{-1} region when the carbon isotope ratio is altered. In the aragonite-type lattice the nearest neighbor carbonate ions occupy positions vertically above one another, the plane of the ion being taken as horizontal. The distance between nearest neighbors is decidedly less than in the calcite-type lattice, where the nearest neighbors are in the same plane. Decius¹⁶² has observed fine structure in Nujol mull spectra of two compounds, barium carbonate and potassium nitrate, which crystallize in the aragonite-type lattice. This structure is present when the N^{15} or C^{13} content is increased to the order of 50%, but is not observed with sodium nitrate, which has a calcite-type structure. A force-constant coupling the out-of-plane bending modes of nearest neighbors in the aragonite-type lattice was postulated. Calculated and observed spectra are in good agreement.

Metals Carbonyls. Interesting structural problems arise in the infrared study of the metal carbonyls, $\text{M}_x(\text{CO})_y$. In particular, the question occurs as to whether these compounds have $\text{M}-\text{C}\equiv\text{O}$ or $\text{M}=\text{C}=\text{O}$ structures or, in some cases, bridged carbonyl structures. It is commonly accepted that the CO molecule, which absorbs in the 2100 cm^{-1} region, has partial triple bond character. Crawford and Cross¹⁶³ quote absorption of $\text{Ni}(\text{CO})_4$ at 2039 cm^{-1} . Jones¹⁶⁴ has also observed nickel carbonyl absorptions at 2057 cm^{-1} and 2128 cm^{-1} . Other metal carbonyls have reportedly shown similar band pairs in the triple-bond region. This offers strong,

161. Louisfert, J., and Pobequin, T., *Compt. rend.*, **235**, 287 (1952).

162. Decius, J. C., *J. Chem. Phys.*, **23**, 1290 (1955).

163. Crawford, B., and Cross, P. C., *J. Chem. Phys.*, **6**, 525 (1938).

164. Jones, L. H., *J. Chem. Phys.*, **28**, 1215 (1958).

although not unequivocal, evidence that the $M-C\equiv O$ structure exists in metal carbonyls. The appearance of additional absorption bands near 1830 cm^{-1} in certain metal carbonyls¹⁶⁵⁻¹⁶⁷ seems to indicate the existence of bridged ring systems in which some of the carbonyl groups are ketonic while others exist as $-C\equiv O$ units. The bridge carbonyl structure has been further suggested from x-ray diffraction data. Dahl and Rundle¹⁶⁸ have investigated the infrared spectra of iron tetracarbonyl with a view to elucidating the structure of the molecule. Solution spectra obtained were essentially the same as reported by others. An alkali halide pellet spectrum showed two distinct bands in the 1800 cm^{-1} region and a large increase in the intensity ratio of the 1800 cm^{-1} to 2000 cm^{-1} bands compared to the ratio in solution. An intense band at 1875 cm^{-1} was found in the polarized infrared spectrum of a single crystal. The intensity of the 1800 cm^{-1} band throws some doubt on the possibility of its being a combination or overtone—a suggestion that has been made by some investigators. On the basis of this work there is some evidence for the association of some carbonyls with more than one iron atom in the crystalline state. On the other hand, the question of differences in solid and solution spectra cannot be ignored in the interpretation, and consequently the structure of iron tetracarbonyl cannot be considered as delineated.

Infrared spectral studies throw some light on the structure of carbonyl derivatives of cobalt. An investigation of cobalt hydrocarbonyl, $HCo(CO)_4$, dicobalt octacarbonyl, $Co_2(CO)_8$, cobalt carbonyl anion, $[Co(CO)_4]^-$, and tetracobalt dodecacarbonyl, $Co_4(CO)_{12}$, leads to the following inferences: (1) the hydrogen of $HCo(CO)_4$ is very weakly bound to the rest of the molecule; (2) $Co_2(CO)_8$ is structurally a transconfiguration of two trigonal bipyramids joined at the edge so that the apical and equatorial carbonyls are shared as bridge carbonyls; (3) $Co_4(CO)_{12}$ has a somewhat simpler structure but also possesses bridge carbonyls; and

165. Sheline, R. K., *J. Am. Chem. Soc.*, **73**, 1615 (1951).

166. Cable, J. W., and Sheline, R. K., *Chem. Rev.*, **56**, 1 (1956).

167. Friedel, R. A., Wender, I., Shuffler, S. L., and Sternberg, H. W., *J. Am. Chem. Soc.*, **77**, 3951 (1955).

168. Dahl, L. F., and Rundle, R. E., *J. Chem. Phys.*, **27**, 323 (1957).

(4) the cobalt carbonyl anion shows only one band in the carbonyl region and may be represented by a tetrahedral model.¹⁶⁷ For the tetracobalt dodecacarbonyl molecule, infrared data show bands attributable to both terminal and bridge carbonyl groups, the ratios of the areas under the bands corresponding to a 2:1 terminal to bridge relationship.

The structure of the $M(CO)_6$ molecules appears to be more definite. Only one intense $C\equiv O$ stretching frequency is observed in the vapor phase of $Cr(CO)_6$. This indicates strongly an equivalence of all the CO bonds and implies a regular octahedral structure for the $Cr(CO)_6$ molecule and other hexacarbonyls.¹⁶⁹ Assuming for these molecules an O_h symmetry, it can be shown that thirteen fundamental vibrations are allowed, four of which are infrared-active. Three of these have been observed directly by means of infrared spectroscopy; the fourth, occurring at about 100 cm^{-1} , is beyond the cutoff of the usual commercially available instruments.¹⁷⁰ Calculated and observed spectral frequencies are in good agreement.¹⁷¹

Halogen Compounds

Compounds of halogen, hydrogen, and some other element. The ternary compounds of the form MH_3X (or MD_3X), where M is Si or Ge, have been found to have the expected C_3 symmetry. Thus, there are six fundamentals: three totally symmetric a_1 vibrations and three doubly degenerate e vibrations. All fundamentals, overtones, and combinations are allowed in both the Raman and infrared. The $M-H$ vibration is almost independent of the rest of the molecule and occurs at approximately $2100-2200\text{ cm}^{-1}$. Substitution of deuterium for hydrogen in the molecule gives the expected lower frequency vibration. The $M-X$ vibration, involving masses of more equal magnitude, is dependent on the rest of the molecule, as would be expected. The observed fundamen-

169. Shufler, S. C., Sternberg, H. W., and Friedel, R. A., *J. Am. Chem. Soc.*, **78**, 2687 (1956).

170. Hawkins, N. J., Matraw, H. C., Sabol, W. W., and Carpenter, D. R., *J. Chem. Phys.*, **23**, 2422 (1955).

171. Murata, H., and Kawai, K., *J. Chem. Phys.*, **27**, 605 (1957).

tal frequencies of SiH_3F , SiH_3Cl , SiH_3Br , GeH_3Cl , and GeD_3Cl are shown in Table 15.

TABLE 15. OBSERVED FUNDAMENTAL FREQUENCIES (cm^{-1}) OF MH_3X MOLECULES

		SiH_3F^a	SiH_3Cl^a	SiH_3Br^a	SiH_3Br^b	GeH_3Cl^c	GeD_3Cl^c
a_1	M—H stretch	2206	2201	2200	2200	2121.2	1522
	MH_3 bend	990	949	930	927	847.7	614
	M—X stretch	872	551	430	428	422.6	421
e	M—H stretch	2196	2195	2196	$\sim 2100^d$	2129.4	1530
	M— H_3 bend	943.4	954.4	950.4	946	874.6	630
	M— H_3 rock	728.1	664.0	632.6	629	604.1	434

^a Newman, C., O'Loane, J. K., Polo, S. R., and Wilson, M. K., *J. Chem. Phys.*, **25**, 855 (1956).

^b Mayo, D. W., Opitz, H. E., and Peake, J. S., *J. Chem. Phys.*, **23**, 1344 (1955).

^c Lord, R. C., and Steese, C. M., *J. Chem. Phys.*, **22**, 542 (1954).

^d Estimated from data in reference (b) by authors in Reference (a).

The absorption spectra of several compounds of nitrogen, hydrogen and halogen have been observed in the $400\text{--}3333\text{ cm}^{-1}$ region. As an example, the crystalline and liquid states of $\text{N}_2\text{H}_5\text{Cl}$, $\text{N}_2\text{D}_5\text{Cl}$, $\text{N}_2\text{H}_5\text{Br}$ and $\text{N}_2\text{D}_5\text{Br}$ were studied by Decius and Pearson.¹⁷² From x-ray diffraction data, $\text{N}_2\text{H}_5\text{Cl}$ has been found to be an orthorhombic crystal of space group C_{2v}^{19} with 16 molecules per unit cell. All fundamental frequencies are allowed in both the infrared and Raman. If one ignores intermolecular coupling, the following frequencies are expected: five hydrogen stretching modes in the $3200\text{--}2400\text{ cm}^{-1}$ region, four H—N—H deformations in the $1400\text{--}1600\text{ cm}^{-1}$ region; four NH_3^+ or NH_2 bending modes in the $1000\text{--}1200\text{ cm}^{-1}$ region, an N—N stretching frequency below 1000 cm^{-1} , and a torsional mode at a considerably lower frequency.

According to these authors, rather broad, complex absorption was observed in the hydrogen stretching region for the N—H or N—D stretch. The arrangement of N_2H_5^+ ions in the unit cell is not known, but it was suggested that hydrogen bonds between —NH_3^+ groups of one ion and the nitrogen of the —NH_2 group

172. Decius, J. C., and Pearson, D. P., *J. Am. Chem. Soc.*, **75**, 2436 (1953).

of another ion perhaps are important in the structure determination. The possibility of hydrogen bonding to the halide ions was eliminated, since bromide ion would not be expected to take part in such bonding; yet the spectra of $\text{N}_2\text{H}_5\text{Cl}$ and $\text{N}_2\text{H}_5\text{Br}$ were quite similar. The bending and deformation region revealed a number of peaks, the total not exceeding that predicted by site symmetry selection rules. However, it was not possible to make detailed assignments. Data and assignments for the spectra of $\text{N}_2\text{H}_5\text{Cl}$ and $\text{N}_2\text{D}_2\text{Cl}$ are shown in Table 16.

TABLE 16. VIBRATIONAL MODES AND FREQUENCIES (cm^{-1}) OF $\text{N}_2\text{H}_5\text{Cl}$ AND $\text{N}_2\text{D}_2\text{Cl}$

(After Decius and Pearson¹⁸⁶)

Vibrational Mode	$\text{N}_2\text{H}_5\text{Cl}$ Liquid, 95°	$\text{N}_2\text{H}_5\text{Cl}$ Solid, 25°	$\text{N}_2\text{D}_2\text{Cl}$ Solid, 25°
N—N stretch	950	975	940
NH_3^+ bend	1080	1101	790
	1100	1124	805
Bend	1220	1246	1000
Bend	1405	1417	1100
Deformation	1495	1500	1133
Deformation		1584	
Deformation	1600	1638	1190
NH_3^+ stretch		2716	
NH_3^+ stretch		2950	2170
NH_3^+ stretch		3034	
NH_2 stretch		3150	2390
NH_2 stretch		3261	2440

Absorption and reflection spectra of NH_4Cl , NH_4F , NH_4Br , and NH_4I at 20° and —150° were recorded in the 1400–3400 cm^{-1} region by Bovey.¹⁷³ The data support the following symmetries for the low-temperature form of the ammonium ion: T_d in NH_4Cl , V_d in NH_4Br and NH_4I , and C_3 or C_{3v} in NH_4F . Martinez and Wagner¹⁷⁴ have reported a similar vibrational study of phosphonium and deuterio-phosphonium iodides at —78°C, room temperature,

173. Bovey, L. R. H., *J. Opt. Soc. Am.*, **41**, 836 (1951).

174. Martinez, J. V., and Wagner, E. L., *J. Chem. Phys.*, **27**, 1110 (1957).

and 190°C in the range 400–4000 cm^{-1} . The low-temperature spectra could be interpreted on the basis of a V_a site symmetry for the PH_4^+ ion and the room temperature spectra were compatible with the x-ray structure, D_{4h}^7 .

Infrared absorption spectra of bifluorides (NaHF_2 , KHF_2 , and NH_4HF_2) are identical when obtained in KCl discs, but different when obtained as Nujol mulls.¹⁷⁵ This behavior has been attributed to the formation of mixed crystals in the alkali halide technique. Discs of KCl, KBr, KI, and NaBr give somewhat different spectra, but for a given matrix, absorption is essentially the same for the three bifluoride molecules. Two infrared-active vibrations were reported for the bifluoride ion: the antisymmetric stretching vibration near 1500 cm^{-1} and the deformation vibration near 1200 cm^{-1} . This is in accord with the vibrational frequencies found by Cote and Thompson¹⁷⁶ and Newman and Badger¹⁷⁷ for the HF_2^- ion in potassium bifluoride.

Compounds of halogen, oxygen and some other element. The ternary compounds of the form ZO_xX_y , where x is a halogen atom, x and y are small integers (1–3), and Z is another element (possibly a different halogen), have been examined to some extent in the infrared region. Twelve absorption bands have been observed and identified for the nitrosyl fluoride molecule, NOF , in the 260–5500 cm^{-1} region¹⁷⁸ (Table 17). The assumed model for NOF , like that of NOCl and NOBr , is a non-linear unsymmetrical structure with nitrogen at the apex of a comparatively flat triangle.

The thionyl halides, SOF_2 and SOCl_2 , are known to have C_s symmetry from electron diffraction, microwave, and vibrational studies. Assignments for the fundamentals of SOF_2 , using spectral data obtained between 3000 and 300 cm^{-1} are: ν_1 , (a'), 1333 cm^{-1} ; $\nu_2(a')$, 808 cm^{-1} ; $\nu_3(a')$, 530 cm^{-1} ; $\nu_4(a')$, 410 cm^{-1} ; $\nu_5(a'')$,

175. Ketelaar, J. A. A., Haas, C., and van der Elsken, J., *J. Chem. Phys.*, **24**, 624 (1956).

176. Cote, G. L., and Thompson, H. W., *Proc. Roy. Soc. (London)*, **A210**, 206 (1951).

177. Newman, R., and Badger, R. M., *J. Chem. Phys.*, **19**, 1207 (1951).

178. Woltz, P. J. H., Jones, E. A., and Nielsen, A. H., *J. Chem. Phys.*, **20**, 378 (1952).

TABLE 17. OBSERVED FREQUENCIES (cm^{-1}) OF NOF, NOCl, AND NOBr

Assignment	NOF ^a	NOCl ^b	NOBr ^b
ν_2 (bending)	521	332	265
ν_3 (N—X)	765.86 ^c	592	542
ν_1 (N—O)	1844.03 ^c	1799	1801
$2\nu_2$	1037	664	
$\nu_2 + \nu_3$	1290	923	807
$2\nu_3$		1184	
$\nu_1 - \nu_2$		1207	
$2\nu_2 + \nu_3$		1248	
$\nu_1 + \nu_2$	2365	2131	2066
$\nu_1 + \nu_3$	2612	2395	2340
$\nu_1 + \nu_2 + \nu_3$	3087		
$2\nu_1$	3652	3568	3567
$2\nu_1 + \nu_3$	4174	4160	4103
$2\nu_1 + \nu_2$	4441	3898	3831
$3\nu_1$	5417	5303	5303

^a (178)^b Burns, W. G., and Bernstein, H. J., *J. Chem. Phys.*, **18**, 1669 (1950).^c Magnuson, D. W., *J. Chem. Phys.*, **20**, 380 (1952).

748 cm^{-1} ; and $\nu_6(\text{a}'')$, 390 cm^{-1} .¹⁷⁹ The four totally symmetric a' vibrations were ascribed to an S—O stretching mode, an S—F stretching mode, a mode in which all angles increase simultaneously, and a mode in which the F—S—F angle increases while the O—S—F angles decrease. The two antisymmetric a'' vibrations were attributed to an S—F stretch and a deformation of the SOF_2 pyramid.

Infrared spectral data have been used further to elucidate the structure of the nitryl halides, NO_2X . The analysis is consistent with the formula X—NO_2 rather than X—O—N—O , the assumed model being a planar structure of C_{2v} symmetry. The fundamental frequencies of NO_2Cl and NO_2F are compared with those of the similar nitric acid molecule, NO_2OH , in Table 18. The oxytrichlorides of vanadium and phosphorus have C_{3v} symmetry. As such, six fundamentals active in both Raman and infrared are allowed. The spectrum of vanadium oxytrichloride has been

179. O'Loane, J. K., and Wilson, M. K., *J. Chem. Phys.*, **23**, 1313 (1955).

TABLE 18. FUNDAMENTAL FREQUENCIES (cm^{-1}) OF NO_2Cl , NO_2F AND NO_2OH^a [After Dodd, Rolfe, and Woodward, *Trans. Faraday Soc.*, **52**, 145 (1956)]

	$\nu_1(a_1)$	$\nu_2(a_1)$	$\nu_3(a_1)$	$\nu_4(b_1)$	$\nu_5(b_1)$	$\nu_6(b_2)$
$\text{NO}_2\text{OH}(\text{IR})$	1320	886		1710	583	765
(R)	1300	925	680	1675	610	(767) ^c
$\text{NO}_2\text{F}(\text{IR})$	1312	822	460	1793	570	742
$\text{NO}_2\text{Cl}(\text{IR})^b$	1293	794	411	1685	367	651

^a The OH may be considered as a single unit in this analysis.^b These are values observed by Ryason, R., and Wilson, M. K., *J. Chem. Phys.*, **22**, 2000 (1954). However, in this reference the $\nu_3(a_1)$ and $\nu_6(b_2)$ frequencies are interchanged.^c From the first harmonic.

observed by Miller and Cousins,¹⁸⁰ and the fundamental frequencies compared with the corresponding data of Delwaulle and François¹⁸¹ for phosphorus oxytrichloride (Table 19). Other halogen-containing ternary molecules have been studied and specific data regarding these may be obtained by referring to the articles of references 182 through 190.

TABLE 19. FUNDAMENTAL FREQUENCIES (cm^{-1}) OF VOCl_3 AND POCl_3 (After Miller and Cousins¹⁸⁰)

Activity		Description	VOCl_3	POCl_3
a_1	R(p)IR	V=O stretch	1035	1290
		V—Cl stretch	408	486
		V—Cl ₃ def.	165	267p?
e	R(dp)IR	V—Cl stretch	504	581
		V—Cl ₃ def.	249	337
		Cl ₃ —V—O rock	129	193

180. Miller, F. A., and Cousins, L. R., *J. Chem. Phys.*, **26**, 329 (1957).181. Delwaulle, M. L., and François, F., *Compt. rend.*, **220**, 817 (1945).182. de Lattre, A., *J. Chem. Phys.*, **19**, 1610 (1951), Na_3AlF_6 , KPF_6 , Na and NH_4 fluoerrates.183. de Lattre, A., *J. Chem. Phys.*, **20**, 1180 (1952). Complex fluorides.184. Lagemann, R. T., Jones, E. A., and Woltz, P. J. H., *J. Chem. Phys.*, **20**, 1768 (1952), CF_3OF .

Other Ternary Compounds

A few correlations for identification and analytical purposes have been made for ternary compounds containing key elements other than boron, carbon, or halogen. These may generally be located in some of the works already cited. (See, for example, Herzberg or references 14 and 19–21.) Some of the more recent studies on nitrogen-containing compounds, hydroxides, ferrites, manganites, and titanates will be reviewed here.

Nitrogen compounds. Spectra of nitric acid and its solutions have been observed by Marcus and Fresco.¹⁹¹ A nitronium ion frequency was observed at 2360 cm^{-1} . The band intensity is enhanced by addition of nitronium fluoborate, sulfuric acid, phosphorus pentoxide, or small amounts of acetic anhydride, and is reduced by sodium nitrate, potassium dihydrogen phosphate, water, and larger amounts of acetic anhydride. Absorption behavior of the nitronium ion in the various media has been used to provide information about the existing equilibria. When the concentration of nitronium ion was large, a combination band was observed near 3745 cm^{-1} . Bands arising from undissociated nitric acid are not observed when a small amount of nitric acid is added to 100% sulfuric acid. Because of the observed behavior in different media, hydrogen-bonded structures were assumed to exist in the nitric acid solutions. The existence of such structure is supported by effects observed in Raman work.

The vibrational frequencies of the nitrite ion (C_{2v} symmetry)

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185. Perkins, W. D., and Wilson, M. K., *J. Chem. Phys.*, **20**, 1791 (1952), SO_2F_2 .
 186. Nielsen, A. H., Burke, T. G., Woltz, P. J. H., and Jones, E. A., *J. Chem. Phys.*, **20**, 596 (1952), F_2CO , FClCO , and Cl_2CO .
 187. Martz, D. E., and Lagemann, R. T., *J. Chem. Phys.*, **22**, 1193 (1954), SOCl_2 and SO_2Cl_2 .
 188. Pemsler, J. P., and Smith, D. F., *J. Chem. Phys.*, **22**, 1834 (1954), $\text{HF} \cdot \text{ClF}_3$ and $\text{DF} \cdot \text{ClF}_3$.
 189. Katzin, L. I., *J. Am. Chem. Soc.*, **76**, 3089 (1954), Cs_2CoCl_4 .
 190. Madden, R. P., and Benedict, W. S., *J. Chem. Phys.*, **25**, 594 (1956), ClO_3F .
 191. Marcus, R. A., and Fresco, J. M., *J. Chem. Phys.*, **27**, 564 (1957).

have been assigned, using spectral data obtained from microcrystalline samples of NaNO_2 , KNO_2 , and AgNO_2 and aqueous solutions of NaNO_2 . The assignments are: $\nu_1(a_1)$, symmetric stretching – $1345 \pm 20 \text{ cm}^{-1}$; $\nu_2(a_1)$, bending – 816 ± 2 , $809 \pm 2 \text{ cm}^{-1}$; $\nu_3(b_1)$, antisymmetric stretching – $1236 \pm 15 \text{ cm}^{-1}$; and $\nu_1 + \nu_3(B_1)$ combination – $2550 \pm 30 \text{ cm}^{-1}$.¹⁹²

Ternary compounds containing the carbon-nitrogen linkage have also been investigated in the infrared region. Rotational spectral data and results of electron diffraction and x-ray studies have furnished bond length values for carbon-nitrogen and carbon-oxygen systems. It is known that frequencies due to carbonyl bonds are sensitive to chemical effects. Layton, Kross and Fassel¹⁹³ have correlated the bond lengths of a number of such systems with the stretching frequency involved. The stretching frequency of molecules containing the C—N linkage shows a linear dependence on the length of the C—N bond. A plot of C—O bond length versus stretching frequency reveals a slight curvature particularly for frequencies beyond 1700 cm^{-1} and bond lengths below 1.20 Å. Data for typical inorganic systems are shown in Table 20.

TABLE 20. BOND LENGTHS AND STRETCHING FREQUENCIES OF SOME INORGANIC C—O AND C—N SYSTEMS
(After Layton, Kross, and Fassel¹⁹³)

C—O			C—N		
	Bond length, Å	Frequency (cm^{-1})		Bond length, Å	Frequency (cm^{-1})
Ni(CO) ₄	1.15 ± 0.03	2045	CNBr	1.158	2187
			CNCl	1.163	2213
COS	1.16 ± 0.05	2050.5			
CO	1.128	2169.8	CNI	1.159	2158
C ₃ O ₂	1.19	{ 2190 (s) 2258 (as)	SCN [−]	1.15	2066
CO ₂	1.16		(CN ₂)	1.16 ± 0.02	2235
		2349.3 (as)	HCN	1.1574	2110

192. Weston, R. E., and Brodasky, T. F., *J. Chem. Phys.*, **27**, 683 (1957).

193. Layton, Jr., Kross, R. D., and Fassel, V. A., *J. Chem. Phys.*, **25**, 135 (1956).

The spectrum of tritium cyanide, TCN, has been obtained in the 400–4000 cm^{-1} region and the fundamental frequencies, ν_1 , ν_2 , and ν_3 , compared with those of HCN and DCN¹⁹⁴ (Table 21).

TABLE 21. COMPARISON OF OBSERVED AND CALCULATED FREQUENCIES (cm^{-1}) FOR HCN, DCN, AND TCN

(After Staats, Morgan, and Goldstein¹⁹⁴)

	ω obs.	Anharmonicity Correction	ω	ω Calculated
HCN ^a ν_1	2095.5 (Raman)	29.2	2124.7	2125.6
ν_2	711.7	15.0	726.7	—
ν_3	3311.7	134.5	3446.2	3448.8
DCN ^a ν_1	1928	25.4	1953	1955.8
ν_2	568.9	10.7	579.6	579.5
ν_3	2629.3	70.9	2700.2	2700.3
TCN ν_1	1724	27.5 ^b	—	1751.9
ν_2	513	8.4 ^b	—	521.4
ν_3	2460	47.2 ^b	—	2507.2

^a Data of A. E. Douglas and D. Sharma, *J. Chem. Phys.*, **21**, 448 (1953).

^b Calculated from ω calc. — ω obs.

The anharmonicity corrections for ν_3 are approximately proportional to the reduced masses computed for the H—CN stretch, treating the molecule as diatomic. The corrections for ν_1 are essentially constant and those for ν_2 vary with ν_2 .

Hydroxides. Infrared spectroscopic studies provide information concerning the —OH fundamentals and the combination bands in the adjoining spectral regions. A band at approximately 3333 cm^{-1} was observed in the absorption spectra of $\text{Ca}(\text{OH})_2$, $\text{Ni}(\text{OH})_2$, $\text{Cd}(\text{OH})_2$, $\text{Y}(\text{OH})_3$, $\text{La}(\text{OH})_2$, $\text{Nd}(\text{OH})_3$, $\beta\text{-Be}(\text{OH})_2$, $\epsilon\text{-Zn}(\text{OH})_2$, $\gamma\text{-Al}(\text{OH})_3$, α - and $\gamma\text{-FeOOH}$, $\text{B}(\text{OH})_3$, and $\alpha\text{-AlOOH}$.¹⁹⁵ Similarly, bands for vanadyl hydroxide, $\text{VO}(\text{OH})_2$, have been observed at 3276 cm^{-1} and 3529–66 cm^{-1} and for $\text{Cu}(\text{OH})_2$ at 3312

194. Staats, P. A., Morgan, H. W., and Goldstein, J. H., *J. Chem. Phys.*, **25**, 582 (1956).

195. Glemser, O., and Hartert, E., *Z. anorg. u. allgem. Chem.*, **283**, 111 (1956).

cm^{-1} and 3574 cm^{-1} .¹⁹⁶ The spectra of the hydroxides of alkali metals have been observed by Wickersheim¹¹ and Phillips and Busing.¹⁹⁷ Hydroxide stretching frequencies for LiOH , $\text{LiOH}\cdot\text{H}_2\text{O}$, and NaOH were reported by the latter authors as 3678 cm^{-1} , 3574 cm^{-1} , and 3637 cm^{-1} , respectively, from infrared measurements; and 3664 cm^{-1} , 3563 cm^{-1} , and 3663 cm^{-1} , respectively, from Raman measurements.

Rather complex absorption patterns have been observed in the —OH stretching region for Ca(OH)_2 and Mg(OH)_2 , both molecules having the same structure. From neutron diffraction studies, the crystal structure of Ca(OH)_2 is known to have, per unit cell, two OH^- ions with bonds parallel to the Z-axis and related by a center of symmetry. Two fundamental OH stretching modes are expected to appear in the vibrational spectrum, namely an anti-symmetric mode which is infrared-active and a symmetric mode which is Raman-active. It is also anticipated that intensities of the absorption bands should be dependent upon the angle between the incident radiation and the OH bond. Using single crystals of Ca(OH)_2 , Busing and Morgan¹⁹⁸ made the following observations: (1) decreased intensity of the low-frequency absorption and increased intensity of the high-frequency absorption at 4164 cm^{-1} occur as predicted when the sample is cooled to liquid nitrogen temperatures; and (2) an unexpected decrease in intensity of the 3720 cm^{-1} band occurs. An explanation of the details of the spectra could not be proposed from the model assumed.

Ferrites, Manganites, and Titanates

In order to understand the properties of ferromagnetic semiconductors, it is advantageous to have as many relations between structure and electromagnetic response as possible. The electric and magnetic properties of these materials are dependent on their atomic and ionic configurations in the crystal lattice. Information about the position and valence of ions can be obtained from

196. Cabannes-Ott, C., *Compt. rend.*, **242**, 2825 (1956).

197. Phillips, B. A., and Busing, W. R., *J. Phys. Chem.*, **61**, 502 (1959).

198. Busing, W. R., and Morgan, H. W., *J. Chem. Phys.*, **28**, 998 (1958).

the vibrational spectra as well as from electronic and magnetic dipole spectra. Recently, vibrational studies of some ferrites, manganites, and titanates have appeared in the literature. In a typical study, the infrared spectra of seven ferrites of the type MFe_2O_4 , where M represents a divalent metal ion (specifically Co^{++} , Fe^{++} , Mg^{++} , Ni^{++} , Zn^{++} , Mn^{++} , and $Ni_{0.3}^{++}Zn_{0.7}^{++}$) were analyzed.¹⁹⁹ As in the spectra of most metallic oxides, two absorption bands arising from interatomic vibrations were observed in the region below 1000 cm^{-1} . Force constants were calculated for the bond stretching between metal and oxide ions. The ferrites used in this study were considered as continuously bonded crystals, i.e., the atoms are bonded to all nearest neighbors by equivalent forces, and the frequency distribution of vibrations can be given by a Debye or Born-von Karman treatment of the classical mechanical problem. X-ray diffraction studies of the ferrites indicate a structure equivalent to that of spinel. The ferrites of Co, Fe, and Ni are believed to be predominantly inverse; $ZnFe_2O_4$ is a normal spinel; and some doubt exists as to the configuration of the other ferrites. Theoretical vibrational analysis indicates that only two high-frequency modes, ν_1 and ν_2 , and two low-frequency oscillatory modes, ν_3 and ν_4 , are infrared-active. Indeed, two high-frequency modes were observed at approximately 570 cm^{-1} and 380 cm^{-1} ; the lower-frequency modes were inaccessible in the spectral range covered. Measurements of the integrated vibrational band intensities were compatible with predominantly ionic bonding for the ferrite structures.

The absorption spectra of copper and nickel manganites also show bands in the region $\sim 570\text{ cm}^{-1}$ and $\sim 460\text{ cm}^{-1}$. The structures are normal and inverse spinel, respectively.²⁰⁰ Tables 22A and 22B show the observed spectral frequencies of the ferrites and manganites.

The spectrum of barium titanate in the $5000\text{--}300\text{ cm}^{-1}$ region was obtained using the technique of deposition of the finely pulverized material onto salt plates from an isopropyl alcohol suspen-

199. Waldron, R. D., *Phys. Rev.*, **99**, 1727 (1955).

200. Dasgupta, S., and Sinha, A. P. B., *Trans. Faraday Soc.*, **53**, 909 (1957).

TABLE 22A. INFRARED SPECTRAL FREQUENCIES (cm^{-1}) OF FERRITES
(After Waldron ¹⁹⁹)

	ν_1	ν_2	Other ν 's
CoFe_2O_4	575	374	shoulder <i>ca.</i> 320
FeFe_2O_4	570 $\left\{ \begin{array}{l} 579^{\text{a}} \\ 610^{\text{a}} \end{array} \right.$	370–80	
MgFe_2O_4	565	406 $\left\{ \begin{array}{l} 380 \\ 430-40 \end{array} \right.$	shoulder <i>ca.</i> 735
MnFe_2O_4	550	392	shoulder <i>ca.</i> 645, 320
NiFe_2O_4	587	396	
ZnFe_2O_4	555 $\left\{ \begin{array}{l} 575 \\ 540 \end{array} \right.$	393	shoulder <i>ca.</i> 325
$\text{Ni}_{0.3}\text{Zn}_{0.7}\text{Fe}_2\text{O}_4$	563	409	

^a Liquid N_2 temperature. The usual narrowing of bands was not observed at liquid N_2 temperature.

sion.²⁰¹ Two broad bands were observed, one centered near 550 cm^{-1} and the other starting near 450 cm^{-1} and reaching its maximum below 300 cm^{-1} . Strong absorption near 600 cm^{-1} is common to all titanates. The fact that this band is also found in rutile, TiO_2 , suggests a connection with a vibrational mode of the TiO_4 tetrahedra. It has been proposed by Megaw²⁰² that a change from the cubic form, which exists above the Curie point of 120°C , to the tetragonal is associated with a change in the character of the bonds around the Ti and O atoms. It was expected, therefore, that (1) a change would occur in the spectrum if heated to 150°C and (2) the spectra of the ferroelectric tetragonal BaTiO_3 and the hexagonal form which is not ferroelectric would be different. No changes were observed by Mara, Sutherland, and Tyrell²⁰¹ when tetragonal BaTiO_3 was heated to 150°C , nor was there any apparent difference in the spectra of the two forms (hexagonal and tetragonal) over the $5000\text{--}700 \text{ cm}^{-1}$ region. A somewhat more detailed study of the infrared absorption of BaTiO_3 and

201. Mara, R. T., Sutherland, G. B. B. M., and Tyrell, H. V., *Phys. Rev.*, **96**, 801 (1954).

202. Megaw, H. D., *Acta Cryst.*, **5**, 739 (1952).

TABLE 22B. INFRARED SPECTRAL FREQUENCIES (cm^{-1}) OF MANGANITES
(After Dasgupta and Sinha ²⁰⁰)

NiO + Mn ₂ O ₃ unheated	942.6 < 963.4 > s 921.7 < > doublet	833.3 m; sh	571.4 m; b	467.1 { 473 458 } vs; b
MiMn ₂ O ₄	947.9 ± 26 w; b		578.1 s; sh	465.3 { 473.0 457.9 } vs; b
CuO + Mn ₂ O ₃ unheated	957.8 < 988.1 > s 927.6 < > doublet	845.3 w	554.4 < 578 530.8 > doublet	466.2 { 479.4 457.9 } vs; b
CuMn ₂ O ₄	941.6 ± 17.4 w; b	858.4 vw; shoulder	< 578.0 — 552.5 — s; b > 530.8 < triplet	465.3 s; sh
sh = sharp b = broad	s = strong w = weak	vs = very strong vw = very weak		

related materials was subsequently reported by Last.²⁰³ Thin single crystals and alkali halide disc spectra were observed. Bands attributed to TiO_3 vibrations appeared at 495 cm^{-1} and approximately 340 cm^{-1} in the single crystal spectra of BaTiO_3 . A third expected vibrational mode, Ba moving against TiO_3 , was not observed in the region covered, but on the basis of specific heat data it was estimated that this frequency is approximately 225 cm^{-1} . The band at 495 cm^{-1} was studied over a range of temperatures (-190° to $+175^\circ\text{C}$). Band splitting related to the change of crystal symmetry appeared as the crystal changed from the cubic to tetragonal, orthorhombic, and rhombohedral phases.

The general features of spectra of the perovskite titanates (SrTiO_3 , PbTiO_3 , and CaTiO_3) and the perovskite niobates (KNbO_3 and NaNbO_3) were similar to those of BaTiO_3 . Such differences as were detected were attributed to differences in unit cell size and symmetry. Three ilmenite titanates (CdTiO_3 , ZnTiO_3 , and MgTiO_3) showed a high frequency band which was broader and less distinct than the corresponding band in the perovskite modifications. Also, the low-frequency band exhibited double structure. The observed band centers are shown in Table 23. Band centers in BaTiO_3 shifted to lower frequencies when a matrix of higher refractive index (n , measured at 540 cm^{-1}) was used in the pressed disc technique. For the halides: KBr ($n = 1.48$); AgCl ($n = 1.92$); TlCl ($n = 2.06$); and TlBr ($n = 2.25$), the absorption maxima were 545 and 400 cm^{-1} ; 540 and 380 cm^{-1} ; 532 and 365 cm^{-1} ; and 535 and 360 cm^{-1} , respectively.

COMPOUNDS CONTAINING MORE THAN THREE ELEMENTS

Inorganic substances containing more than three elements may be broadly grouped into two classes, namely: (1) *non-complex compounds* such as certain minerals, salts of heteropoly acids, and simple structures, and (2) *complex compounds*, such as coordination compounds and metal organic structures.

203. Last, J. T., *Phys. Rev.*, **105**, 1740 (1957).

TABLE 23. INFRARED ABSORPTION BAND CENTERS (cm^{-1}) OF BaTiO_3 AND SOME RELATED MATERIALS
(After Last 203)

Single Crystal Spectra	ν_1	Pressed Disc Spectra	ν_1	ν_2
BaTiO_3 (cubic)	495	BaTiO_3 (tetragonal)	545	400
BaTiO_3 (tetragonal)	517; 495	BaTiO_3 (hexagonal)	555	365
BaTiO_3 (orthorhombic)	520; 495(sh)	PbTiO_3 (tetragonal)	590	405
BaTiO_3 (rhombohedral)	632; 490	SrTiO_3 (cubic)	610	395
PbTiO_3 (tetragonal)	610(sh); 535	CaTiO_3 (orthorhombic)	540; 700(sh)	360(b)
		KNbO_3 (orthorhombic)	660; 550(sh)	375
		NaNbO_3 (orthorhombic)	675; 510(sh)	375
		CdTiO_3 (ilmenite)	575(b)	425; 335
		ZnTiO_3 (ilmenite)	590(b)	400; 315(sh)
		MgTiO_3 (ilmenite)	600(b)	475; 350

Notation: ν_1 = high frequency stretching
 ν_2 = lower frequency bending
(sh) = shoulder
(b) = broad

Non-Complex Compounds. The spectra of a number of minerals were recorded by Hunt, Wisherd, and Bonham.¹⁶⁰ For the purpose of identification, spectra of the ammonium salts of several heteropoly acids were obtained in the 1200–625 cm^{-1} region by Sharpless and Munday.²⁰⁴ An equation for calculating integrated molar extinction coefficients for the compounds suspended in KBr discs was derived. The KBr disc technique has also been used for obtaining spectra of solid sulfamic acid and the sulfamate ion from 500–4000 cm^{-1} at various temperatures.²⁰⁵ Assignments of vibrational modes were made for the zwitterion model of the acid $\text{H}_3\text{N}^+\text{SO}_3^-$ with site symmetry in the crystal of C_1 . Spectra of the sulfamate ion H_2NSO_3^- , observed in $\text{H}_2\text{NSO}_3\text{K}$ and the corresponding deuterated compounds, was interpreted on basis of the C_s model. According to x-ray data,²⁰⁶ sulfamic acid belongs to the space group $\text{V}_h^{15}\text{-Pbca}$ with eight molecules per unit cell. According to Halford's tables,²⁰⁷ this corresponds to C_1 site symmetry. The eighteen fundamental frequencies, their overtones, and combinations are allowed in both infrared and Raman. If, in the crystal, the molecule exists as the zwitterion, $\text{H}_3\text{N}^+\text{SO}_3^-$, the actual site symmetry should not be very different from C_{3v} . On the other hand if the molecular form, $\text{NH}_2\text{SO}_2\text{OH}$, exists in the crystal, the site symmetry should be very nearly C_s . The zwitterion model would have a simpler spectrum than the molecular model, if one neglects factors such as hydrogen bonding, intermolecular coupling, etc. The zwitterion form should have only eleven fundamentals instead of eighteen. In addition to this, there should appear a broad, intense band in the low frequency N—H stretching region and no hydrogen modes below about 1000 cm^{-1} . The molecular form would be expected to exhibit a sharper, higher frequency N—H stretch, NH_2 deformation bands below 1000 cm^{-1} , an OH stretching mode at a frequency higher than the N—H stretching frequency, and an altered NSO_3 spectrum due to the single-bonded S—OH.

204. Sharpless, N. E., and Munday, J. S., *Anal. Chem.*, **29**, 1619 (1957).

205. Vuagnat, A. M., and Wagner, E. L., *J. Chem. Phys.*, **26**, 77 (1957).

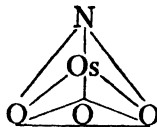
206. Kanda, F. A., and King, A. S., *J. Am. Chem. Soc.*, **73**, 2315 (1951).

207. Halford, R. S., *J. Chem. Phys.*, **14**, 8 (1946).

The observed spectrum suggested the zwitterion configuration. This observation supports the work of Dupuis,²⁰⁸ who cited evidence for the existence of the zwitterion form of the acid in the solid state.

Two osmium-nitrogen compounds of considerable chemical stability have been studied in the infrared region.²⁰⁹ The metal-nitrogen stretching frequency in potassium osmiate, $\text{K}[\text{OsO}_3\text{N}]$, and potassium nitrilopentachlorosmate, $\text{K}_2[\text{OsCl}_5\text{N}]$ was identified by noting the isotopic shift with the N^{15} analogs. For the four compounds, the observed frequencies are: $\text{K}[\text{OsO}_3\text{N}]$ – 1023 cm^{-1} (strong, sharp), 890 cm^{-1} (strong), 858 cm^{-1} (strong); $\text{K}[\text{OsO}_3\text{N}^{15}]$ – 1023 cm^{-1} (strong, sharp), 992 cm^{-1} (strong, sharp), 890 cm^{-1} (strong); $\text{K}_2[\text{OsCl}_5\text{N}]$ – 1073 cm^{-1} (strong, sharp); $\text{K}_2[\text{OsCl}_5\text{N}^{15}]$ – 1073 cm^{-1} (strong, sharp), 1041 cm^{-1} (strong, sharp). The band at 1023 cm^{-1} for the osmiate ion and the band at 1073 cm^{-1} for the nitrilopentachlorosmate ion were attributed to the metal-nitrogen vibration. The isotope N^{15} shifts the 1023 cm^{-1} band to 992 cm^{-1} (991 cm^{-1} , calculated value) and similarly the 1073 cm^{-1} band was shifted to 1041 cm^{-1} (1039 cm^{-1} , calculated value). The high frequency of the Os—N vibration implies considerable multiple bond character in accord with the great chemical stability of these compounds. The spectrum confirmed

the structure of the osmiate ion,



as a distorted

tetrahedron in agreement with x-ray data and chemical evidence.

The vibrational spectra of the solid and gaseous borine carbon-yls, $\text{B}^{11}\text{H}_3\text{CO}$, $\text{B}^{10}\text{H}_3\text{CO}$, $\text{B}^{11}\text{D}_3\text{CO}$, and $\text{B}^{10}\text{D}_3\text{CO}$ have been reported by Bethke and Wilson.²¹⁰ These molecules have point group symmetry C_{3v} and as such there are four fundamentals of species a_1 and four of species e . Table 24 summarizes the observed data.

208. Dupuis, T., *Compt. rend.*, **243**, 1621 (1956); **242**, 2924 (1956).

209. Lewis, J., and Wilkinson, G., *J. Inorg. and Nuclear Chem.*, **6**, 12 (1958).

210. Bethke, G. W., and Wilson, M. K., *J. Chem. Phys.*, **26**, 1119 (1957).

TABLE 24. OBSERVED FREQUENCIES (cm^{-1}) OF BORINE CARBONYL
(After Bethke and Wilson ²¹⁰)

Species and Activity	Description	Assignment	B ¹⁰ H ₃ CO	B ¹¹ H ₃ CO	B ¹¹ H ₃ CO ^a	B ¹⁰ D ₃ CO	B ¹¹ D ₃ CO
a_1	B—H stretch	ν_1	2385	2379	(2360)*	1694.8	1679.0
IR,	C—O stretch	ν_2	2166.0	2164.7	2164	2169.0	2168.5
Raman, polarized	BH ₃ bend	ν_3	1083.1	1073.4	1105	883	862
	B—C stretch	ν_4	707.0	691.4	692	629.5	624.8
e	B—H stretch	ν_5	2456	2441	2440	1852	1837
IR, $\perp$	BH ₃ bend	ν_6	1100	1100	1392	801.5	801.3
Raman, depol.	Unsym. bend	ν_7	818.8	809.3	809	718.0	708.5
	Sym. bend	ν_8	313.7	313.2	(315)*	266.0	266.0

^a Cowan, R. D., *J. Chem. Phys.* 18, 1101 (1950).

* Estimated.

Molecules of the type $\text{NaXO}_2(\text{Ac})_3$, where X is uranium, neptunium, plutonium and americium were examined in the infrared region by Jones.²¹¹ Utilizing the symmetric and antisymmetric stretching frequencies of the O—X—O groups, approximate X—O force constants were calculated and found to decrease in the order $k_{\text{U-O}} > k_{\text{Np-O}} > k_{\text{Pu-O}} > k_{\text{Am-O}}$, respective values being 0.705, 0.698, 0.675, and 0.612 megadyne cm^{-1} . The X—O bond distance decreases in the same order. It was suggested that the decrease in bond distance accompanied by the decrease in force constant occurs because the bond, though shortened by contraction of the electron shells of the metal is weakened by interaction with the extra valence shell electrons. The data appear in Table 25.

TABLE 25. VIBRATIONAL FREQUENCIES (cm^{-1}) OF $\text{NaXO}_2(\text{Ac})_3$ COMPOUNDS

(After Jones²²⁵)

	NaAc ^a	NaUO ₂ (Ac) ₃	NaNpO ₂ (Ac) ₃	NaPuO ₂ (Ac) ₃	NaAmO ₂ (Ac) ₃
ν_1 (C—H stretch)	2933	2933	2935	2927	
ν_2 (CH ₃ def.)	1425	1413	1410	1415	
ν_3 (C—O stretch)	1408	1472	1472	1470	1467
ν_4 (C—C stretch)	924	948	948	948	948
ν_5 (CO ₂ def.)	645	678	677	677	675
ν_6 torsion about C—C bond					
ν_7 (C—H stretch)	2999	2978	2980	2980	
ν_8 (C—O stretch)	1578	1537	1536	1540	1541
ν_9 (CH ₂ def.)	1440	(1450)?			
ν_{10} (CH ₃ rock)	1012	1004	1004	1004	
ν_{11} (CO ₂ rock)	465				
ν_{12} (C—H stretch)	2999	2978	2980	2980	
ν_{13} (CH ₃ def.)	1488	(1480)?			
ν_{14} (CH ₃ rock)	1045	1055	1054	1054	1052
ν_{15} (CO ₂ rock)	615	612	610	609	
ν_1 (O—X—O) ^b		856	844	818	749
ν_3 (O—X—O)		931	934	930	914
$(\nu_1 + \nu_3)$ (XO ₂ group) ^b		1781	1770	1739	

^a Jones, C. H., and McLaren, E. H., *J. Chem. Phys.*, **22**, 1796 (1954).

^b Observed only on very thick samples.

211. Jones, L. H., *J. Chem. Phys.*, **23**, 2105 (1954).

Complex Compounds

It would be difficult in a work of this nature to include all the investigations which have appeared in the literature on the spectral studies of complex structures. However, some of the typical studies involving the inorganic coordination compounds and the metal-organic complexes will be presented. It is hoped that the reader will supplement the discussion here with ample use of the references recorded in the bibliography of Part III. Typical complexes involving only inorganic moieties are the hexamine metal complexes, $M(\text{NH}_3)_6^{+y}\text{X}_y$, where M is a metal of valency y and X is a univalent anion. Kobayashi and Fujita²¹² observed absorption maxima of the hexammine metal complexes, $[\text{Ni}(\text{NH}_3)_6]\text{Cl}_2$, $[\text{Cr}(\text{NH}_3)_6]\text{Cl}_3$, and $[\text{Co}(\text{NH}_3)_6]\text{Cl}_3$. A strong band observed in the neighborhood of 3000 cm^{-1} was attributed to the N—H stretching mode. The formation of the NH_3 -metal bond is associated with a considerable shift of the NH stretching frequency within the NH_3 . In coordination compounds, it is expected that the more covalent character of the nitrogen-metal bond will result in a lower frequency for the N—H stretching vibration. The N—H frequencies of the complexes lie between that of pure ammonia and $\text{NH}_3\text{—H}^+$, the strongest covalent bond. The shifts of N—H frequencies to longer wavelengths decrease in the order of Co, Cr, Ni, while the complex stability increases in this order. Force constants calculated for the Ni—N, Cr—N, Co—N bonds were 3.62×10^5 , 4.38×10^5 , and 5.36×10^5 dyne cm^{-1} respectively. The vibrational modes of the NH_3 ligand have been observed and studied in the works of Mizushima *et al.*,^{213, 214} Hill and Rosenberg,²¹⁵ and Caglioti *et al.*²¹⁶

212. Kobayashi, M., and Fujita, J., *J. Chem. Phys.*, **23**, 1354 (1955).

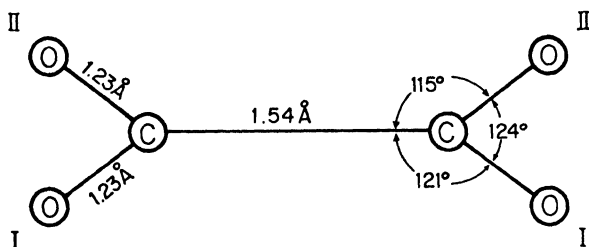
213. Mizushima, S., Nakagawa, K., and Sweeny, D. M., *J. Chem. Phys.*, **25**, 1006 (1956).

214. Mizushima, S., Nakagawa, I., and Quagliano, J. V., *J. Chem. Phys.*, **23**, 1367 (1955).

215. Hill, D. G., and Rosenberg, F., *J. Chem. Phys.*, **24**, 1219 (1956); *ibid.*, **22**, 148 (1954).

216. Caglioti, V., Silvestroni, P., Sartori, G., and Scrocco, M., *Ricerca sci.*, **26**, 1743 (1956).

The oxalate ion²³⁰ is considered as planar and the structural parameters are as shown:



The inequality in the angles $\text{C}-\text{C}-\text{O}_\text{I}$ and $\text{C}-\text{C}-\text{O}_\text{II}$ is perhaps due to experimental error in the analysis and the ion has been assigned V_h symmetry. The formation of the $\text{M}-\text{O}$ bond lengthens the $\text{C}-\text{O}_\text{I}$ bond and strengthens the $\text{C}-\text{O}_\text{II}$ bond to an extent dependent on the strength of the metal-oxygen bond. This results in shifts of $(\text{C}-\text{O}_\text{I})$ and $(\text{C}-\text{O}_\text{II})$ to lower and higher frequencies, respectively. The relation between the strength of the $\text{M}-\text{O}$ bond and the shifts of the bending modes is somewhat complex, but the $\text{M}-\text{O}$ bond becomes stronger in the order of the metals, Ni^{+2} , Co^{+2} , Cu^{+2} , Fe^{+3} , Cr^{+3} , Co^{+3} , Al^{+3} . For the divalent ions, this is in the order of the stability constants of the oxalates. For the trivalent ions, stability constant data for the oxalate chelates was not available; however, the order is consistent with the stability constant order of other complexes in which the metals are coordinated by oxygen. Metal complexes involving more complicated ligands than the oxalate ion are constantly being investigated by spectral methods in order to gain more information bearing on the problem of metal-ligand bonding.

A critical review of some of the significant results in the study of transitional metal complexes via infrared spectroscopy has been made by Cotton.²³¹ This summary treats many inorganic coordination compounds as well as the following organic complexes:

230. Jeffrey, G. A., and Parry, G. S., *J. Am. Chem. Soc.*, **76**, 5283 (1954).

231. Cotton, F. A., "The Infrared Spectra of Transitional Metal Complexes," in Lewis and Wilkins, "Modern Coordination Chemistry," Interscience Publishers Inc., New York, 1956.

metal sandwich compounds, cyclo-pentadienyl metal carbonyls, nitrosyls, alkyls, etc., β -diketones, oxalato and related complexes, alkene and alkyne complexes, complexes of amino acids, urea, ethylenediamine-tetraacetic acid, dimethylglyoxime, and 8-hydroxyquinoline, and complexes of large organic ligands, such as derivatives of tetraphenylporphin.

GLOSSARY

Brief explanatory notes and definitions of many of the terms used in the preceding parts are given below. For more detailed discussions of the concepts, techniques, and notation the following list of general references is recommended.

- Bellamy, L. J., "The Infrared Spectra of Complex Molecules," John Wiley & Sons, Inc., New York, 1954, or Methuen and Co., Ltd., London, 1954.
- Hershenson, H. M., "Infrared Absorption Spectra-Index for 1945-1957," Academic Press, New York, 1959.
- Herzberg, G., "Infrared and Raman Spectra of Polyatomic Molecules," D. Van Nostrand Co., Inc., New York, 1945.
- Lewis, J., and Wilkins, R. G., "Modern Coordination Chemistry," Interscience Publishers Inc., New York, 1960. (In Chapter 5, contributing author F. A. Cotton discusses the infrared spectra of transitional metal complexes.)
- Lothian, G. F., "Absorption Spectrophotometry," The Macmillan Company, New York, 1958.
- "Report on Notation for the Spectra of Polyatomic Molecules," *J. Chem. Phys.*, 23, 1997-2011 (1955).
- West, W., "Chemical Applications of Spectroscopy" in A. Weissberger's "Technique of Organic Chemistry," Vol. IX, Interscience Publishers, Inc., New York, 1956. (An excellent section discussing the location of spectra in the literature is given by authors R. N. Jones and C. Sandorfy on pp. 327-331 of this volume.)
- Wilson, E. B., Decius, J. C., and Cross, P. C., "Molecular Vibrations," McGraw-Hill Book Co., New York, 1955.

Absorption coefficient (absorbancy index, extinction coefficient, absorptivity). A quantity (index), a , defined by the expression:
$$a = \frac{1}{dc} \log_{10} \frac{I_0}{I}$$
 where d is the length of the absorbing path, c is the concentration of material in any specified units, and $\log I_0/I$ is the absorbance (optical density). When d is expressed in cm. and c in moles per liter, the index is called the molar absorbancy index or molar extinction coefficient, ϵ .

Absorption edge. The limit or discontinuity appearing in the intensity of an absorption spectrum.

Absorptivity. The fraction of the incident radiation absorbed by a substance. The ratio of radiant energy absorbed by a substance to that absorbed by a black body under the same conditions. See also under **absorption coefficient**.

Amorphous. Having no definite form; non-crystalline.

Anharmonic oscillator. An oscillator in which the restoring force does not vary linearly with the displacement of the system from its equilibrium position. The **anharmonicity** may be mechanical or electrical. If electrical, the variation of dipole moment (infrared) or polarizability (Raman) with internuclear distance is non-linear. **Anharmonicity corrections** must be used for exact calculations of band centers and intensities.

Anisotropic. Having different properties for different orientations.

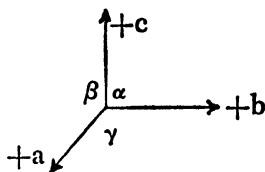
Black body. A body whose absorptivity is unity, i.e., it absorbs all of the radiant energy incident on it for all wavelengths.

Bending modes. Vibrational modes which involve changes in the interbond angles of a molecule. Such modes are associated with lower force constants and thus appear at lower frequencies than do **stretching modes**. Bond-bending vibrations are sometimes called **deformations**, which are described by many authors using such terms as scissors, wag, rock, twist, etc.

Chelate compounds. Compounds which are formed when a metal ion enters into ring formation with a substance containing two or more electron donor groups.

Crystallites. Small, generally imperfectly formed crystals. Component grains of a polycrystalline material.

Crystal systems. Crystals may be classified according to their symmetry into seven major divisions. For the various systems, certain relationships exist among the crystallographic axes—*a*, *b*, *c*—and the axial angles—



(1) *Triclinic* (no symmetry greater than 1-fold),

$$a \neq b \neq c$$

$$\alpha \neq \beta \neq \gamma \neq 90^\circ$$

- (2) *Monoclinic* (one two-fold axis along *b* and no higher symmetry),

$$a \neq b \neq c$$

$$\alpha = \gamma = 90^\circ, \beta \neq 90^\circ$$

- (3) *Orthorhombic, Rhombic* (three two-fold axes along *a*, *b*, and *c*),

$$a \neq b \neq c$$

$$\alpha = \beta = \gamma = 90^\circ$$

- (4) *Tetragonal* (one four-fold axis along *c*),

$$a = b \neq c$$

$$\alpha = \beta = \gamma = 90^\circ$$

- (5) *Cubic, Regular, Isometric* (four three-fold axes),

$$a = b = c$$

$$\alpha = \beta = \gamma = 90^\circ$$

- (6) *Hexagonal* (one six-fold axis), Three horizontal axes a_1, a_2, a_3 equal and at an angle of 120° to each other, a 4th axis unequal and normal to the plane of a_1, a_2, a_3 .

- (7) *Trigonal Rhombohedral* (one three-fold axis),

$$a = b = c$$

$$\alpha = \beta = \gamma \neq 90^\circ$$

These seven systems give rise to 32 different symmetry classes or **point groups** and 230 **space groups** with which a crystal may be associated. The classification of a crystal into these groups is dependent upon the presence of certain elements of symmetry.

Curie point (temperature). The critical temperature above which ferromagnetic materials lose their spontaneous magnetization. The term is also applicable to ferroelectric materials which often show several Curie points.

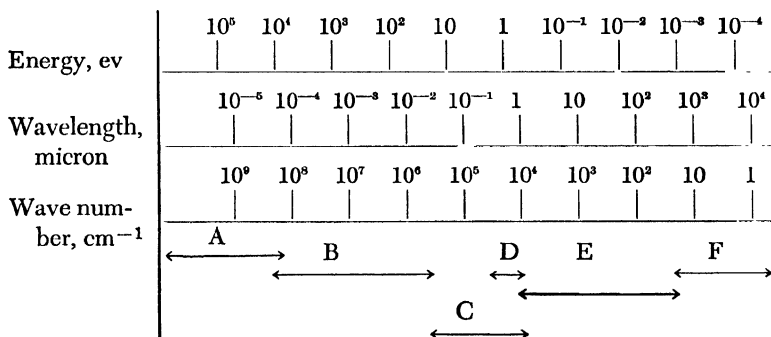
^{-d}**x**. A symbol denoting deuterium substitution; the subscript *x* is a number indicative of the extent of deuteration. E.g., ammonia — d_2 and ammonia — d_3 refer respectively to the replacement of two and three hydrogens with deuterium atoms.

Deutero-. Prefix denoting the replacement of hydrogen in a compound by its isotope of mass 2, deuterium. Sometimes deuterio-, particularly so used in *Chemical Abstracts*.

Dipole. A unit in which the effective centers of opposite electrical or magnetic charges are separated by a small distance. The **dipole**

moment of such a unit is mathematically the product of one of the charges and the separation distance.

Electromagnetic Spectrum, Subdivisions of Interest in Molecular Structure Studies.



The interactions of the electromagnetic radiation in the regions A, B, C, D, E, and F with matter are as follows:

Region	Effect
A—Gamma rays	Ionization and nuclear transformation
B—X-rays	Electronic transitions within inner orbits
C—Ultraviolet	Electronic transitions involving outer orbits
D—Visible	Electronic transitions involving outer orbits
E—Infrared	Vibrational transitions (also rotational in the far infrared)
F—Microwaves	Rotational transitions

Energy gap. The energy range between the bottom of the conduction band and the top of the valence band of a semiconductor. Sometimes referred to as the **forbidden energy gap** or **band**, implying the energy range in which there are no electronic levels.

Ferroelectric. Pertaining to materials which exhibit spontaneous polarization (dipole moment).

Ferromagnetic. Pertaining to materials which have permeabilities appreciably greater than 1, exhibiting a permanent magnetic moment.

Ground state. The lowest energy state of a system.

Hygroscopic. Term applied to substances which absorb water from the atmosphere.

Intermolecular coupling. Interaction between molecules.

Isomers. Substances having the same number and kind of atoms but different structural arrangement of the atoms in the molecule.

Different physical and/or chemical properties are exhibited by the isomeric forms.

Isotropic. Having the same properties in all directions.

Lattice (space lattice). A particular arrangement of structural units (atoms, groups of atoms, or molecules), such that the environments of all points are identical. There are 14 different types of lattices determined by lengths and orientations of the primitive translations which carry the unit cell into its neighbor. For the cubic system, for example, there are three possible lattice types—simple, face-centered, and body-centered.

Matrix. A medium in which a sample is dispersed for the purpose of examining its spectrum. In infrared spectroscopy, matrix materials may be alkali halide powders, as in the pellet technique, or gases, in which the sample is frozen, as in the matrix isolation technique.

Mean free path. The mean (average) distance which is traversed by a particle of a system between successive collisions with other particles.

Mobility. Motion of charged particles subject to force fields. The term is generally applicable to all processes of electrical conduction, whether the charge is carried by holes, electrons, ions, or the like.

Non-polar molecule. A molecule which does not have a dipole moment. Electrons between the bonded atoms are evenly distributed. The bonding is atomic or homopolar. Such bonding is also often referred to as covalent.

Normal modes of vibration. Simple oscillatory motions in which all atoms undergo their maximum displacement at the same time and pass through their equilibrium positions at the same time, i.e., the frequency and phase of the motion for each coordinate of the system are equal. There are $3n-5$ or $3n-6$ (n = number of atoms) genuine normal modes of vibration for molecules, depending on whether the system is linear or not. The five or six non-genuine vibrations are simply translations or rotations of the molecule as a whole.

The lowest frequency normal mode is called the fundamental mode. Modes whose frequencies are integral multiples of the fundamental are called overtones or harmonics. The number ascribed to the harmonic is always identically its frequency divided by that of the fundamental, the $(n + 1)^{\text{th}}$ harmonic being the n^{th} overtone.

Two or more normal modes may have the same frequency, in which case they are called **degenerate vibrations**. Under such conditions, it is possible to construct an infinite number of normal modes by superimposing the degenerate modes with different phases. It is, then, no longer required that the atoms move in straight lines although they continue to move with the same frequency. The **degeneracy** is defined as the number of linearly independent modes of a given frequency.

Ortho-para ratio of hydrogen. In the hydrogen molecule, it is possible to have two different nuclear spin combinations. The molecular form in which the nuclei have antiparallel spins is para-hydrogen; the form in which the spins are parallel is ortho-hydrogen. At room temperature the temperature-dependent ortho-para ratio is 3:1.

Photoconductivity threshold. The frequency of radiation which is just sufficient to cause increased electric conductivity in a substance when illuminated.

Point groups. Molecules may be classified into symmetry groups by the number and nature of symmetry elements they possess. In molecular spectroscopy, the 32 possible symmetry groups (point groups) are described most often in the Schoenflies notation; in crystallographic work the Hermann-Mauguin system is used. The Schoenflies notation and associated symmetry elements are:

Symbol	Symmetry Element
C_n (Cyclic)	An n -fold rotation axis
D_n (Dihedral)	An n -fold rotation axis and n two-fold axes $\perp$ to it
S_n	An n -fold rotation-reflection axis
T (Tetrahedral)	Four 3-fold axes directed toward the vertices of a tetrahedron
O (Octahedral)	Three mutually perpendicular 4-fold axes

Subscripts v, h, d, i, s, denote vertical reflection plane containing the symmetry axis, horizontal reflection plane $\perp$ to the axis, dihedral reflection plane bisecting the angle between two 2-fold axes, inversion, and reflection plane respectively. Thus: Molecular symmetry C_{2v} indicates that the molecule possesses a 2-fold rotation axis and two vertical planes of symmetry through the axis.

In crystal studies, numerical superscripts are used to denote the space groups isomorphous with the point group. E.g., D_{4h}^6 and D_{4h}^{17} are both space groups isomorphous with the point group D_{4h} .

Each point group can be reduced into a number of non-equivalent irreducible representations. Vibrational modes are found to belong to one of these representations. The notation used is as follows:

Non-degenerate fundamentals	a, b
Doubly-degenerate fundamentals	e
Triply-degenerate fundamentals	f

For combination bands capital letters are used. For non-degenerate modes the letter a or b is used according as the mode is symmetrical or antisymmetrical with respect to rotation about the principal axis. Subscripts 1, 2 or g, u, and superscripts ' and '' are used with both non-degenerate and degenerate modes and are related to the symmetry or asymmetry of the mode with respect to a certain operation of the point group.

Polar molecule. A molecule which possesses a dipole moment. The moment is generally produced by the uneven distribution of electrons between the bonded atoms. The bonding is largely ionic or heteropolar. Such bonding is also often referred to as **electrovalent**.

Polarizability. When an external electric field produces or alters the dipole moment of a molecule or atom, the process is called polarization. The polarizability of an isotropic molecule is defined by the equation, $\hat{\mu} = \alpha \hat{E}$, where $\hat{\mu}$ is the induced moment and E is the field strength.

Polymerization. Reaction or process by which compounds of higher molecular weight but same percentage composition are formed from simpler compounds. E.g., $n\text{HF} \longrightarrow (\text{HF})_n$.

PQR branches. When a molecule is excited by infrared radiation, vibrational and rotational changes occur concomitantly. Consequently, each vibrational band, when well-resolved, may appear as three branches. As the vibrational quantum number, ν , increases, a P, Q, or R branch appears according as the rotational energy is increased, unaffected, or decreased, respectively. The frequencies contained in the three branches diminish in the order $P > Q > R$, the rotational quantum number, J changes by $-1, 0$, and $+1$ in the same order.

Quadrupole moment. Atoms or molecules which have no dipole moment may have electric moments of higher order. The higher order moments may be thought of as arrangements of dipoles with dif-

ferent orientations. For example, two dipoles combined form a quadrupole, four an octupole. The strength of attraction between atoms or molecules having a quadrupole moment decreases very rapidly as the separation distance increases.

Raman effect. An effect associated with the incoherent scattering of monochromatic light by a substance on which the light is incident. Additional lines appear in the spectrum of the incident light near each of its prominent lines. A necessary condition for a molecule to exhibit the Raman effect is that the molecule experiences a change in polarizability.

Selection rules. A set of rules, derived from quantum mechanics, which govern the possibility of transitions between states of a system. **Forbidden** transitions violate the selection rules while **allowed** transitions are in accord with them. The differences in energy of two vibrational levels between which transitions take place determine the frequencies of infrared absorption bands. Allowed transitions in the infrared depend on the non-vanishing of the integral $\int \psi_a^* M \psi_b d\tau$ where ψ_a and ψ_b are wave functions of the upper and lower states respectively and M is the dipole moment operator. When the integral has a non-zero value, the **intensity** of the transition is dependent upon the magnitude of that value. A forbidden transition assumes the vanishing of this integral.

Semiconductors. Solids whose electrical resistivity at room temperature lies intermediate between that of conductors and insulators. The electrical charge carrier concentration varies with thermal agitation over some range of temperature. Pure semiconducting substances which are void of impurities are termed **intrinsic**, while those whose electrical properties are dependent on impurities are termed **extrinsic**. Extrinsic semiconductors having an excess of negative charge carriers (electrons) are called *n*-type semiconductors; those having an excess of positive charge carriers (holes) are called *p*-type semiconductors.

Skin effect. An effect in which the depth of penetration of electric currents in a conductor decreases with increasing current frequency.

Spectrum. A graphic representation, visual display, or photograph of the intensity distribution of radiant energy with respect to wavelength, frequency, or some related quantity. Various types of spectra may be observed depending on the type of matter-light interaction involved:

Absorption spectrum. Energy is removed from the light beam and reappears as heat or some other form,

Emission spectrum. Energy is released from the substance after it has received proper excitation with heat, bombardment, etc.,

Diffraction spectrum. The incident beam is distributed (spread) by the substance into other beams,

Polarized spectrum. An absorption spectrum obtained with plane-polarized light, i.e., the electric vectors of all components of the incident beam lie in the same plane. Similarly, in emission spectroscopy the substance may be excited with polarized light, followed by determination of the polarization of the emitted light,

Raman spectrum. See **Raman effect**; a spectrum so obtained.

Reflectance spectrum. The direction of the incident beam is changed as it is impinged upon the surface of the substance.

Stability constant. For the equilibrium reaction, $M + nL \rightleftharpoons ML_n$, between a metal ion M and a ligand L , the stability constant β_n for the complex, ML_n , is defined by $\beta_n = [ML_n] / [M] [L]^n$, where the brackets denote concentrations.

Stretching modes. Vibrational modes which involve extensions of bonds between atoms. The mode may be **symmetric** or **antisymmetric** according as the atoms move in-phase or out-of-phase, respectively.

Symmetry elements and operations. Associated with every element of symmetry there is an operation which will transform the molecule (or any geometrical figure) into a configuration that is indistinguishable from the original. Examples of symmetry elements are (1) the identity, (2) center of symmetry, (3) rotation axes, (4) reflection planes and (5) rotation-reflection axes.

Zwitterion. A dipolar ion, i.e., an ion which carries both positive and negative charge. While electrically neutral, the unit has a dipole moment, e.g., the zwitterion form of sulfamic acid, $H_3N^+SO_2O^-$.

PART III

Bibliography of Unclassified References

THIS BIBLIOGRAPHY was constructed largely by searching the section of *Chemical Abstracts* entitled "Electronic Phenomenon and Spectra," for the period January 1952 through December 1958. It is divided chronologically into sections which correspond to the year of publication of the original works. Numbers appearing at the end of an entry refer to the volume and column index of *Chemical Abstracts*. For example, 50-75 indicates that the work was abstracted in column 75 of Volume 50 of *Chemical Abstracts*. Papers are included on the theory, techniques, spectral data, and applications of infrared absorption for a large variety of inorganic materials. Emission, Raman, and reflectance spectra are included only where they appear as part of or in support of an absorption study.

An effort has been made to provide annotations to the bibliography, particularly in the cases of entries whose titles give little information concerning the nature of the work performed. A subject index which was prepared from the material in *Chemical Abstracts*, or the original article is presented at the end of the reference list. Review articles and papers, which for various rea-

sons were unavailable for proper indexing, are included in the index under the heading *General*. A supplementary reference list whose entries appear in the subject index is presented at the end. These references were reported since the original construction of the bibliographic section and therefore are not included in the main body of the bibliography. This supplementary list represents coverage of material appearing in *Chemical Abstracts* or elsewhere through April, 1960. References to published works on metal-organic complexes are not indexed.

1950

1. Bardeen, J., "Theory of Infrared Absorption in Silicon and Germanium," *Phys. Rev.*, **79**, 216. 46-6923.
2. Herzberg, G., and Reid, C., "Infrared Spectrum and Structure of the HNCO Molecule," *Discussions Faraday Soc.*, No. 9, 92. 46-3403.
The spectrum of HNCO was examined from 450-8000 cm^{-1} using LiF, NaCl, and KBr prisms and out to 13,220 cm^{-1} using a 21 ft. grating spectrograph.
3. Hornig, D. F., "Infrared Spectra of Crystals at Low Temperatures," *Discussions Faraday Soc.*, No. 9, 115. 46-3408.
Data and discussion for CO_2 and HCN.
4. Hunt, J. M., Wisherd, M. P., and Bonham, L. C., "Infrared Absorption Spectra of Minerals and Other Inorganic Compounds," *Anal. Chem.*, **22**, 1478.
Infrared spectra of minerals and other inorganic compounds (64 samples) are obtained for the region 625-5000 cm^{-1} . Spectral positions of the principal absorption bands are tabulated.
5. Lohman, J. B., and Hornig, D. F., "The Infrared Spectrum of Crystalline Hydrogen Sulfide," *Phys. Rev.*, **79**, 235. 46-6924.
6. Nielsen, H. H., "The Low-Frequency Fundamental Bands in the Spectrum of Arsine," *Discussions Faraday Soc.*, No. 9, 85. 46-3405.
Discussion of the arsine bands in the region 859-1130 cm^{-1} .
7. Taylor, W. J., Beckett, C. W., Tung, J. Y., Holden, R. B., and Johnston, H. L., "Raman and Infrared Spectra of Pentaborane," *Phys. Rev.*, **79**, 234. 46-6924.
8. Woltz, P. J. H., Jones, E. A., and Nielsen, A. H., "Some New Measurements on the Infrared and Raman Spectra of Silicon Tetrafluoride," *Phys. Rev.*, **79**, 416. 46-6924.

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9. Bovey, L. F. H., "The Infrared Absorption and Reflection Spectra of the Ammonium Halides," *J. Opt. Soc. Am.*, **41**, 836. 46-343.

The results of absorption and reflection spectra of NH_4Cl , NH_4F , NH_4Br , and NH_4I at 20° and -150° in the range $1400\text{--}3400\text{ cm}^{-1}$ are interpreted in terms of crystal structure.

10. Burke, T. G., and Jones, E. A., "Spectra of Bromine Pentafluoride," *J. Chem. Phys.*, **19**, 1611. 46-4366.

Spectrum of gaseous BrF_5 was observed from $400\text{--}700\text{ cm}^{-1}$ using KBr optics. With supporting data from the Raman spectrum of liquid BrF_5 , a tetragonal pyramid structure is suggested.

11. Burke, T. G., and Smith, D. F., "The Infrared and Raman Spectra of Molybdenum Hexafluoride," *Phys. Rev.*, **83**, 485. 47-6754.

An abstract of a paper presented at the American Physical Society meeting.

12. Callomon, H. J., McKean, D. C., and Thompson, H. W., "Intensities of Vibration Bands. III. Nitrous Oxide," *Proc. Roy. Soc. (London)*, **A208**, 332. 46-5435.

The absolute band intensity has been determined at 2222 cm^{-1} and has been correlated with the polar properties of the N—N and N—O linkages.

13. Caunt, A. D., Short, L. N., and Woodward, L. A., "Raman and Infrared Spectra of Germanium Tetrafluoride," *Nature (London)*, **168**, 557. 46-10892.

The most intense infrared absorption band of GeF_4 (gaseous) is located at 800 cm^{-1} .

14. Coté, G. L., and Thompson, H. W., "Infrared Spectra and the Solid State. III. Potassium Bifluoride," *Proc. Roy. Soc. (London)*, **A210**, 206. 46-10891.

Spectra are reported for the $1250\text{--}5000\text{ cm}^{-1}$ range between 293° and 90°K .

15. Coté, G. L., and Thompson, H. W., "Infrared Spectra and the Solid State. IV. Fluoborates," *Proc. Roy. Soc. (London)*, **A210**, 217. 46-10891.

Spectra of ammonium, potassium, and sodium fluoborates are reported for the range $400\text{--}5000\text{ cm}^{-1}$.

16. Dowd, J. J., "Optical Properties of Selenium," *Proc. Phys. Soc. (London)*, **64B**, 783. 46-10896.

The absorption coefficient of amorphous selenium has been measured in the near infrared region.

17. Duval, Cl., Lecomte, J., and Morandat, J., "The Infrared Absorption Spectra of Metallic Salts Including the XO_2 Group (Chlorites and Nitrites)," *Bull. soc. chim. France*, **745**. 46-5436.

18. Edelson, D., and McAfee, K. B., "The Infrared Spectrum of Sulphur Hexafluoride," *J. Chem. Phys.*, **19**, 1311. 46-3856.
19. Gebbie, A., and Saker, E. W., "Properties of Amorphous Selenium in the Infrared," *Proc. Phys. Soc. (London)*, **64B**, 360. 46-9423.

The infrared transmission of selenium is reported from the visible to 2500 cm^{-1} region.

20. Jones, L. H., Badger, R. M., and Moore, G. E., "The Infrared Spectrum and the Structure of Gaseous Nitrous Acid," *J. Chem. Phys.*, **19**, 1599. 46-5969.

The infrared spectrum of nitrous acid indicates the existence of two tautomeric forms believed to be cis and trans. An analysis of the spectral observations is given.

21. deLattre, A., "Infrared Spectrum of Some Complex Hexafluorides," *J. Chem. Phys.*, **19**, 1610. 46-4366.

Absorption data is presented for Na_3AlF_6 , KPF_6 and sodium and ammonium fluoerrates.

22. Loferski, J. J., and Miller, P. H., Jr., "Infrared Properties of Tellurium," *Phys. Rev.*, **83**, 876. 47-6754.

An abstract of a paper presented at the American Physical Society Meeting.

23. Louisfert, J., "Infrared Absorption Spectra of Metallic Carbonates in Powder Form," *Compt. rend.*, **233**, 381. 46-1352.

Comparison is made between absorption bands and estimated intensities of natural crystals and synthetic preparations of finely divided metal carbonates.

24. Magnuson, D. W., "Infrared and Microwave Studies of Nitrosyl Fluoride," *Phys. Rev.*, **83**, 485. 47-6754.

An abstract of a paper presented at the American Physical Society Meeting.

25. Mathis, R., "The Infrared Spectrum of Hydrogen Sulfide at 1.6μ ," *J. phys. radium*, **12**, 764. 46-3405.

26. Matsumura, O., "Infrared Absorption Bands of Water of Crystallization," *Mem. Faculty Sci. Kyusyu Univ.*, **1B**, 1. 46-4365.

Absorption spectra were observed for the following hydrates in the $2857\text{--}6667\text{ cm}^{-1}$ region.

$\text{CdSO}_4 \cdot 8/3\text{ H}_2\text{O}$, $\text{NiSO}_4 \cdot \text{H}_2\text{O}$, $\text{NiSO}_4 (\text{NH}_3)_2 \text{SO}_4 \cdot 6\text{H}_2\text{O}$, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, $\text{CuSO}_4 (\text{NH}_3)_4 \cdot \text{H}_2\text{O}$, $\text{CuSO}_4 \cdot 2\text{H}_2\text{O}$, $\text{KAl} (\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ and $\text{KNa C}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$.

27. Milatz, J. M. W., Kluyver, J. C., and Hardebol, J., "Determination of Isotope Ratios, e.g., in Tracer Work, by an Infrared Absorption Method," *J. Chem. Phys.*, **19**, 887-8. 46-341.

The $\text{C}^{13}/\text{C}^{12}$ isotope ratio is determined to within 0.002 units.

28. Murray, R. B., "The Near Infrared Spectrum of Nitrous Oxide with Long Absorbing Paths," *Phys. Rev.*, **83**, 486. 47-6754.

An abstract of a paper presented at the American Physical Society Meeting.

29. Newman, R., and Badger, R. M., "The Polarized Infrared Spectrum of Potassium Bifluoride at -185°C ," *J. Chem. Phys.*, 19, 1207. 46-3406.

Polarized infrared spectra of KHF_2 crystals are interpreted at 25°C and -185°C in the $700\text{--}5800\text{ cm}^{-1}$ region.

30. Nielsen, A. H., and Jones, E. A., "Analysis of Infrared Spectrum of Chlorine Monofluoride," *J. Chem. Phys.*, 19, 1117. 47-10348.
31. Nielsen, A. H., and Lagemann, R. T., "High Dispersion Measurements on the ω_3 of $\text{C}^{14}\text{O}_2^{16}$ at 4.5μ ," *Phys. Rev.*, 83, 245. 47-6754.

An abstract of a paper presented at the American Physical Society Meeting.

32. d'Or, L., and Tarte, P., "Spectroscopic Studies of Nitrous Acid. The Infrared Spectrum of Gaseous Nitrous Acid," *Bull. soc. roy. sci. Liege*, 20, 478. 46-7875.

Spectral data for ordinary and deuterated nitrous acid were obtained from gaseous mixtures of $\text{NO--NO}_2\text{--H}_2\text{O}$ and $\text{NO--NO}_2\text{--D}_2\text{O}$. Structural features are discussed.

33. Paul, W., Jones, D. A., and Jones, R. V., "Infrared Transmission of Galena," *Proc. Phys. Soc. (London)*, 64B, 528. 46-9985.

In the $833\text{--}5000\text{ cm}^{-1}$ range, reflection, absorption, and transmission data are reported.

34. Penner, S. S., and Weber, D., "Quantitative Infrared Intensity Measurements. I. Carbon Monoxide Pressurized with Infrared-inactive Gases," *J. Chem. Phys.*, 19, 807. 46-341.

Measurements of infrared intensity of CO pressurized with H_2 , He, A, O_2 and N_2 have been made for systems having total pressures up to 700 lbs./sq.in. Values of integrated absorption for the fundamental of CO is reported as $237\text{ cm}^{-2}\text{ atm}^{-1}$, and for the first overtone as $1.64\text{ cm}^{-2}\text{ atm}^{-1}$.

35. Penner, S. S., and Weber, S., "Quantitative Infrared Measurements. II. Studies on the First Overtone of Unpressurized Carbon Monoxide," *J. Chem. Phys.*, 19, 817. 46-341.

Cells of varying lengths and at different gas pressures were used to measure quantitatively the absorption of unpressurized CO for the first overtone. A value of $1.69\text{ cm}^{-2}\text{ atm}^{-1}$ was reported.

36. Penner, S. S., and Weber, D., "Quantitative Line-Width Measurements in the Infrared. I. Carbon Monoxide Pressurized with Infrared-Inactive Gases," *J. Chem. Phys.*, 19, 1351. 46-2401.

Quantitative infrared-intensity measurements on CO pressurized with gases are reported. For pressures up to 100 lbs./sq.in. correlations are made between pressure, absorption and optical density.

37. Penner, S. S., and Weber, D., "Quantitative Linewidth Measurements in

the Infrared. II. Unpressurized Carbon Monoxide," *J. Chem. Phys.*, **19**, 1361. 46-2401.

38. Richardson, W. S., "The Infrared Spectra of DC^{12}N and DC^{13}N ," *J. Chem. Phys.*, **19**, 1213.

A tabulation is presented for the fundamental infrared frequencies of DC^{12}N , DC^{13}N , HC^{12}N and HC^{13}N .

39. Robinson, D. Z., "The Experimental Determination of the Intensities of Infrared Absorption Bands. IV. Measurements of the Stretching Vibrations of OCS and CS_2 ," *J. Chem. Phys.*, **19**, 881. 46-342.
40. Rossini, F. D., "American Petroleum Institute Spectral Data and Standard Samples," *Applied Spectroscopy*, **6**, No. 1, 3. 46-2899.

A report of the available catalogs of spectral data and other materials on infrared, ultraviolet, Raman, and mass spectral data.

41. Shelton, R. D., and Nielsen, A. H., "Infrared Study of the Polymer Bands of Hydrogen Fluoride Vapor," *J. Chem. Phys.*, **19**, 1312. 46-3856.

LiF , CaF , and NaCl optics were used in examining the HF polymer bands from $425\text{--}3000\text{ cm}^{-1}$. Temperatures ranged from $32\text{--}88^\circ\text{C}$ and pressures from atmospheric to vacuum.

42. Simon, I., "Optical Constants of Germanium, Silicon, and Pyrite in the Infrared," *J. Opt. Soc. Amer.*, **41**, 730.

Reflection method is applied to investigate optical properties of semiconductors. Data are reported for the region $600\text{--}2400\text{ cm}^{-1}$.

43. Smith, R. A., "Electric and Optical Properties of Certain Sulfides, Selenides, Tellurides," *Semiconducting Materials*, **198**. 49-15455.

The electrical and optical properties of sulfides, selenides, and tellurides of Pb and Bi are given along with various hypotheses to explain the experimental results.

44. Taylor, J. H., "Two New Absorption Bands of N_2O ," *J. Chem. Phys.*, **19**, 1314. 46-35.

45. Thompson, H. W., and Williams, R. L., "Vibration-Rotation Bands of Nitrous Oxide," *Proc. Roy. Soc. (London)*, **A208**, 326. 46-5435.

A re-investigation of the absorption spectrum of nitrous oxide gas near 2222 cm^{-1} has been reported. The results are discussed analytically.

46. Vincent-Geisse, J., "Variation in the Fine Structure of an Infrared Absorption Spectrum Observed With a Slit of Variable Width," *Compt. rend.*, **233**, 1179. 46-10889.

47. Welsh, H. L., Crawford, M. F., MacDonald, J. C. F., and Chisolm, D. A., "Induced Infrared Absorptions of Hydrogen, Nitrogen, and Oxygen in the First Overtone Regions," *Phys. Rev.*, **83**, 1264. 46-340.

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48. Allen, H. C., Jr., and Cross, P. C., "The Asymmetric Rotor. X. The Hydrogen Sulfide Spectrum in the 5150 cm^{-1} region," *J. Chem. Phys.*, **20**, 1709. 47-4739.
49. Bass, A. M., and Benedict, W. S., "A New Infrared Band System of FeO ," *Astrophys. J.*, **116**, 652. 47-3694.
50. Benedict, W. S., Claassen, H. H., and Shaw, J. H., "Absorption Spectrum of Water Vapor Between 4.5 and 13 Microns," *J. Research Natl. Bur. Standards*, **49**, 91 (Research Paper No. 2347). 47-966.

The absorption spectrum has been measured with a 3600 line/inch replica echelette grating as dispersing element. The absorbing path lengths and concentrations of water vapor were varied.

51. Boyd, D. R. J., and Thompson, H. W., "The Fundamental Vibration Band of Hydrogen Iodide," *Spectrochim. Acta*, **5**, 308. 47-8515.

Greater resolving power has been used in re-examining the HI fundamental vibration band (near 2222 cm^{-1}). New values have been obtained for the molecular rotational constants.

52. Briggs, H. B., "Infrared Absorption in High-Purity Germanium," *J. Opt. Soc. Amer.*, **42**, 686. 48-10425.

Spectral curves for single crystals of Ge and for thin films of Ge on glass are presented for the range $400\text{--}5000\text{ cm}^{-1}$.

53. Briggs, H. B., and Fletcher, R. C., "New Infrared Absorption Bands in p-Type Germanium," *Phys. Rev.*, **87**, 1130. 47-11969.

Bands were found at 2941 and 2128 cm^{-1} in p-type germanium. The absorption is proportional to the concentration of free holes.

54. Burke, T. G., Smith, D. F., and Nielsen, A. H., "The Molecular Structure of MoF_6 , WF_6 , and UF_6 from Infrared and Raman Spectra," *J. Chem. Phys.*, **20**, 447. 46-7432.

The region $250\text{--}5000\text{ cm}^{-1}$ was examined for MoF_6 and WF_6 spectra; $588\text{--}5000\text{ cm}^{-1}$ for UF_6 spectrum. An interpretation of the observed spectra is given.

55. Caunt, A. D., Short, L. N., and Woodward, C. A., "Raman and Infrared Spectra of Germanium Tetrafluoride," *Trans. Faraday Soc.*, **48**, 873. 47-4743.

56. Chatterjee, S. D., Ghosh, N. N., and Naha, A. M., "Near-infrared Absorption by Solutions of Inorganic Substances," *J. Chem. Phys.*, **20**, 344. 46-8963.

Description of an infra-red colorimeter for the range $9091\text{--}12,500\text{ cm}^{-1}$ is given.

Absorption data are given for aqueous and ammoniacal solutions of CuSO_4 , NiSO_4 , also for aqueous solutions of CuCl_2 , $\text{Cu}(\text{NO}_3)_2$, metal sulfates, and metal NH_4 sulfates of Cu, Ni, and Fe(II), and several other inorganic molecules.

57. Chisolm, D. A., MacDonald, J. C. F., Crawford, M. M., Welsh, H. C., "Induced Infrared Absorption in Hydrogen and Hydrogen-foreign Gas Mixtures at Pressures up to 1500 Atmospheres," *Phys. Rev.* **88**, 957. 47-3120.
58. Clark, M. A., and Cashman, R. J., "Transmission and Spectral Response of Lead Sulfide and Lead Telluride," *Phys. Rev.*, **85**, 1043. 46-5973.
Transmissions of PbS, 0.48 mm crystal, at 2000-5000 cm^{-1} and of PbTe, 0.11 mm crystal at 1667-5000 cm^{-1} have been observed at 23°-72°, and -194°. Comparison is made at each temperature between the transmittance and the spectral response of photocells made of the same compound.
59. Cohn, H., "Infrared Spectrum of Crystalline Potassium Perchlorate. Reassignment of the Lower Fundamental Frequencies of the Perchlorate Ion," *J. Chem. Soc.*, 4282. 47-4739.
A Nujol paste was used to measure infrared transmission of potassium perchlorate. The lower fundamental frequencies of the perchlorate ion were reassigned.
60. Coulon, R., "The Infrared Spectrum of Gaseous Hydrofluoric Acid Between 1400 cm^{-1} and 700 cm^{-1} ," *J. phys. radium*, **13**, 371. 47-403.
Three absorption bands are reported. The intensity of the band occurring at 1025 cm^{-1} increases with pressure to the extent that polymeric formation in HF is assumed without doubt.
61. Dayal, B., "The Vibration Spectrum of Rutile," *Proc. Indian Acad. Sci.*, **32A**, 304. 47-7896.
62. Duval, C., and Lecomte, J., "Infrared Absorption Spectra of Ions of the Type BO_2^- and BO_3^- ," *Bull. soc. chim. France*, 101. 47-42.
Spectral data for the 625-2000 cm^{-1} region have been obtained for samples of Mg, Ca, Ni, Mn, Sr, and Ba orthoborates and for Na, Pb, and Ba metaborates.
63. Duval, C., and Lecomte, J., "Some Solid-State Reactions Studied by Infrared Absorption Spectra," *Compt. rend.*, **234**, 2445. 46-10890.
Infrared spectroscopy techniques have been applied to powdered solids to determine whether a chemical reaction has occurred after mixing.
64. Eberhardt, W. H., and Burke, T. G., "An Absorption Band of Nitrosyl Chloride at 30 μ ," *J. Chem. Phys.*, **20**, 529. 46-8964.
The spectrum of NOCl has been examined from 250-10,000 cm^{-1} .
65. Edelson, D., "The Infrared Spectrum of Disulfur Decafluoride," *J. Am. Chem. Soc.*, **74**, 262. 47-1492.
66. Fabricand, B. P., Carlson, R. O., Lee, C. A., and Rabi, I. I., "The Rotational Spectrum of Potassium Chloride," *Phys. Rev.*, **86**, 607. 47-6757.
An abstract of a paper presented at the American Physical Society meeting.
67. Florinskaya, V. A., and Pechenkina, R. S., "Reflection and Transmission

Spectra of Different Modifications of Silica in the Infrared Region," *Doklady Akad. Nauk S.S.S.R.*, **85**, 1265. 47-1491.

Spectra of quartz crystals, cut parallel and perpendicular to the optical axis, fused quartz, quenched crystalline quartz, pure artificial cristobalite, and pure artificial tridymite were recorded up to 714 cm^{-1} .

68. Giguère, P. A., and Liu, I. D., "Infrared Spectrum, Molecular Structure, and Thermodynamic Functions of Hydroxylamine," *Can. J. Chem.*, **30**, 948. 47-7320.

The absorption spectra were obtained in the region $400\text{--}6667\text{ cm}^{-1}$ for the gaseous and solid states. An analysis was made and a structure suggested.

69. Giguère, P. A., and Liu, I. D., "The Infrared Spectrum of Hydrazine," *J. Chem. Phys.*, **20**, 136. 46-6488.

Spectral data is given for the absorption of hydrazine from $400\text{--}6667\text{ cm}^{-1}$. The spectrum of the liquid and of the solid at -70°C were measured.

70. Granier-Mayence, J., "Absorption Spectra at Low Temperature. Study of Gaseous Nitrous Oxide and of Gaseous and Liquid Nitric Oxide," *Ann. Phys.*, **7**, 453. 47-3115.

71. Gross, E. F., and Karryev, N. A., "Absorption of Light in a Crystal of Cuprous Oxide in the Infrared and the Visible," *Doklady Akad. Nauk S.S.S.R.*, **84**, 261. 46-8963.

Absorption spectrum of Cu_2O is reported in the region $714\text{--}1429\text{ cm}^{-1}$.

72. Gruen, D. M., "Interpretation of the Visible and Near-Infrared Spectra of NpO_2^+ and PuO_2^{++} Ions," *J. Chem. Phys.*, **20**, 1818. 47-1487.

73. Gutowsky, H. S., and Liehr, A. D., "Infrared Spectra of Phosphorus Trifluoride, Phosphorus Oxyfluoride, and Phosphorus Pentafluoride," *J. Chem. Phys.*, **20**, 1652. 46-10896.

Spectra were determined at room temperature using NaCl and KBr optics.

74. Hansler, R. C., Bell, E. E., and Oetjen, "Infrared Rotational Spectrum of Hydrogen Sulfide," *Phys. Rev.*, **88**, 174. 48-8024.

An abstract of a paper presented at the American Physical Society meeting.

75. Harris, L., and Beasley, J. K., "The Infrared Properties of Gold-Smoke Deposits," *J. Opt. Soc. Am.*, **42**, 134. 46-4365.

Porous structures of colloidal-gold particles are examined under infrared radiation. An interpretation of the optical properties is given.

76. Howard, J. N., and Chapman, R. M., "The Pressure Dependence of the Absorption by Entire Bands of Water Vapor in the Near Infrared," *J. Opt. Soc. Am.*, **42**, 423. 46-7430.

Absorption has been determined as a function of total pressure, water

vapor density, and optical path length in the region from 1429–10,000 cm^{-1} .

77. Howard, J. N., and Chapman, R. M., "Near-Infrared Absorption by Entire Bands of Carbon Dioxide," *J. Opt. Soc. Am.*, **42**, 856. 49–7977.
78. Hyde, C. E., and Hornig, D. F., "The Measurement of Bond Moments and Derivatives in HCN and DCN from Infrared Intensities," *J. Chem. Phys.*, **20**, 647. 46–10890.

The absolute infrared absorption intensities of the fundamentals for HCN and DCN have been measured.

79. Jolly, W. L., "Absorption Spectroscopy in Liquid Ammonia," *U. S. Atomic Energy Comm., Nat'l. Sci. Foundation, Wash., D.C. UCRL-2008*, 3. 47–11993.

Spectral curves are given for:

- 1) Solid NH_4I , KI, and NaI in liquid NH_3 ,
 - 2) H_2O , aqueous NH_3 , and liquid NH_3 in the region 7143–16667 cm^{-1} ,
 - 3) CuSO_4 in H_2O and aqueous NH_3 and $\text{Cu}(\text{NO}_3)_2$ in liquid NH_3 from 833–25,000 cm^{-1} ,
 - 4) KMnO_4 in H_2O and liquid NH_3 , and
 - 5) Solutions of Na in liquid NH_3 from 4000–10,000 cm^{-1} .
80. Jones, D. A., Jones, R. V., and Stevenson, R. W. H., "Infrared Transmission Limits of Single Crystals of Some Fluorides," *Proc. Phys. Soc. (London)*, **65B**, 906. 47–3120.

Measurements from 769–2000 cm^{-1} were made for LiF, synthetic CuF_2 , natural CaF_2 , CdF_2 , SrF_2 , PbF_2 , and BaF_2 .

81. Keller, W. D., Spotts, J. H., and Biggs, D. L., "Infrared Spectra of Some Rock-Forming Minerals," *Am. J. Sci.*, **250**, 453. 46–7875.

Infrared absorption data for 54 samples of carbonate, sulfate, phosphate, and silicate minerals are given.

82. Lagemann, R. T., Jones, E. A., and Woltz, P. J. H., "The Infrared and Raman Spectra of CF_3OF ," *J. Chem. Phys.*, **20**, 1768. 47–4740.

The region 400–5000 cm^{-1} was investigated for the CF_3OF spectrum. Assignments were made for nine of the twelve fundamental vibration frequencies.

83. deLattre, A., "Infrared Spectrum of Some Complex Fluorides," *J. Chem. Phys.*, **20**, 1180. 46–8964.

Bands for K, Cu, Zn, and Ni fluosilicates are reported at 488 ± 6 and 735 ± 6 cm^{-1} . A new band for KBF_4 is reported at 523 cm^{-1} . Bands for anhydrous and hydrated aluminum fluorides and KAHF_4 are discussed.

84. deLattre, A., "Infrared Spectrum of Sulphur Hexafluoride in the Lithium Fluoride Region," *J. Chem. Phys.*, **20**, 520. 46–8964.

85. Lord, R. C., "Far-Infrared Transmission of Silicon and Germanium," *Phys. Rev.*, **85**, 140. 46–4365.

The transmission bands observed with 2mm thick samples of Si and Ge between 250–833 cm^{-1} are discussed.

86. Lord, R. C., McDonald, R. S., and Miller, F. A., "Notes on the Practice of Infrared Spectroscopy," *J. Opt. Soc. Am.*, **42**, 149. 46–4913.

Techniques for spectroscopy in the infrared are described in detail.

87. Louisfert, J., and Pobeguín, T., "Differentiation of Calcium Carbonates by Means of Infrared Absorption Spectra," *Compt. rend.*, **235**, 287. 47–2040.

Various forms of CaCO_3 were subjected to infrared spectral analysis. A quantitative method is described as being more sensitive than the usual X-ray diffraction methods.

88. Magnuson, Dale W., "The Infrared Spectrum of Nitrosyl Fluoride. II. Rotational Analysis of ν_1 and ν_3 ," *J. Chem. Phys.*, **20**, 380. 47–6754.
89. McCubbin, T. K., "The Spectra of HCl , NH_3 , H_2O , and H_2S from 100–700 Microns," *J. Chem. Phys.*, **20**, 668. 46–10890.
90. Mecke, R., "Infrared Spectroscopy and the Character of Chemical Binding," *J. Chem. Phys.*, **20**, 1935. 47–4739.

A method is developed for obtaining bond moments in the ground state by extrapolation of values calculated for the transition moments of the bond. Other mathematical expressions are given.

91. Miller, F. A., and Wilkins, C. H., "Infrared Spectra and Characteristic Frequencies of Inorganic Ions," *Anal. Chem.*, **24**, 1253. 46–10892.

The infrared spectral curves of 159 inorganic salts are given. They include metaborates, tetraborates, boron nitride, carbonates, bicarbonates, nitrites, nitrates, phosphates, orthoarsenates, sulfites, sulfates, bisulfates, thiosulfates, chromates, metavanadates, cyanides, cyanates, thiocyanates, metasilicates, silicofluorides, silica-gel, oxides, selenites, selenates, chlorates, perchlorates, bromates, iodates, periodates, dichromates, molybdates, tungstates, permanganates, ferrocyanides, chlorides, and cobaltinitrites.

92. Moore, G. E., and Badger, R. M., "The Infrared Spectra and Structure of the Chloramines and Nitrogen Trichloride," *J. Am. Chem. Soc.*, **74**, 6076. 47–3697.

Spectra of gaseous NH_2Cl , NH_2DCl , NHCl_2 , and NCl_3 were obtained in the region 400–7140 cm^{-1} .

93. Moss, T. S., "Optical Properties of Tellurium in the Infrared," *Proc. Phys. Soc. (London)*, **65B**, 62. 46–10896.

Absorption constant of tellurium layers is given for the near infrared region.

94. Newman, R., and Badger, R. M., "Infrared Spectra of Cyanuric Acid and Deuterocyanuric Acid," *J. Am. Chem. Soc.*, **74**, 3545. 46–10891.

Spectra of sublimed films of $(\text{HNCO})_3$ and $(\text{DNCO})_3$ along with the polarized spectra of (101) single crystals of the acid are reported.

95. Newman, R., "Polarized Infrared Spectrum of Sodium Nitrite," *J. Chem. Phys.*, **20**, 444. 46-7430.
96. Nielsen, A. H., Burke, T. G., Woltz, P. J. H., and Jones, E. A., "The Infrared and Raman Spectra of F_2CO , $FClCO$, and Cl_2CO ," *J. Chem. Phys.*, **20**, 596. 46-10891.
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Interpretation is made of the ClO_2 spectrum in the $250-5000\text{ cm}^{-1}$ region.

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Reflection spectra for KH_2PO_4 , KH_2AsO_4 , $NH_4H_2PO_4$, and $NH_4H_2AsO_4$ are given for the $400-10,000\text{ cm}^{-1}$ region at room temperature. Spectra for $NH_4H_2PO_4$ and $NH_4H_2AsO_4$ are also given at liquid $-N_2$ temperatures for the $667-5000\text{ cm}^{-1}$ region.

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Spectra for NF_3 and PF_3 are reported in the range 250 cm^{-1} – 5000 cm^{-1} .

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The spectral curves are obtained from $667\text{--}3000\text{ cm}^{-1}$. Calculations are made and a compilation of stretching and deformation force constants for CH_4 , CH_3F , CH_3Cl , SiH_4 , SiH_3F , and SiH_3Cl is presented.

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 $\text{Co}(\text{NH}_3)_5\text{H}_2\text{OCl}_3$, $\text{Co}(\text{NH}_3)_5\text{H}_2\text{OBr}_3$, $\text{Co}(\text{NH}_3)_5\text{H}_2\text{OI}_3$,
 $\text{Co}(\text{NH}_3)_5\text{N}_3\text{Cl}_2$, $\text{Co}(\text{NH}_3)_5\text{N}_3\text{Br}_2$, $\text{Co}(\text{NH}_3)_5\text{N}_3\text{I}_2$,
 $\text{Co}(\text{NH}_3)_5\text{FCl}_2$, $\text{Co}(\text{NH}_3)_5\text{FBr}_2$, $\text{Co}(\text{NH}_3)_5\text{FI}_2$, $\text{Co}(\text{NH}_3)_5\text{ClCl}_2$,
 $\text{Co}(\text{NH}_3)_5\text{BrCl}_2$, and $\text{Co}(\text{NH}_3)_5\text{BrBr}_2$.

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463. Bues, W., and Gehrke, H. W., "Vibrational Spectra of Metals, Glasses, and Crystals of Sodium Di-, Tri-, and Tetra Phosphates," *Z. anorg. u. allgem. chem.*, **288**, 291. 51-7147.

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464. Bues, W., and Gehrke, H. W., "Vibrational Spectra of Molten, Glassy, and Crystalline Highly Polymeric Phosphates," *Z. anorg. u. allgem. chem.*, **288**, 307. 51-7859.

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474. Dalby, F. W., and Nielsen, H. H., "Infrared Spectrum of Water Vapor. I. The 6.26 μ Region," *J. Chem. Phys.*, **25**, 934. 51-4144.
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$\nu_2(a_1)$ 822 cm^{-1}	$\nu_4(b_1)$ 1793 cm^{-1}	$\nu_6(b_2)$ 742 cm^{-1}

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481. Dupuis, T., "The Infrared Absorption Spectra of Sulfamic Acid, Heavy Sulfamic Acid, and Their Salts," *Compt. rend.*, **243**, 1621. 51-4821.

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484. Eischens, R. P., Francis, S. A., and Pliskin, W. A., "The Effect of Surface Coverage on the Spectra of Chemisorbed Carbon Monoxide," *J. Phys. Chem.*, **60**, 194. 50-6918.

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Two Diatomic Molecules in Electrostatic Interaction," *Compt. rend.*, **242**, 1871. 50-9875.

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Spectra for kaolinite, dickite, halloysite, montmorillonite, nontronite, attapulgite, illite, vermiculite, muscovite, biotite, talc, pyrophyllite, and chrysolite have been recorded for the 833-1250 cm^{-1} .

492. Gaunt, J., "The Analysis of Heavy Water by Infrared Spectrometry," *Spectrochim. Acta*, **8** (2), 57. 51-135.
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- a doublet at 1310-22, asymmetrical stretching;
- a doublet at 528-35, bending.

An overtone at 2287 and a combination band at 2455 are also reported.

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The absorption spectra of $\text{Ca}(\text{OH})_2$, $\text{Mg}(\text{OH})_2$, $\text{Ni}(\text{OH})_2$, $\text{Cd}(\text{OH})_2$, $\text{Y}(\text{OH})_3$, $\text{La}(\text{OH})_3$, $\text{Nd}(\text{OH})_3$, $\beta\text{-Be}(\text{OH})_2$, $\epsilon\text{-Zn}(\text{OH})_2$, $\nu\text{-Al}(\text{OH})_3$, α - and $\gamma\text{-FeOOH}$, $\text{B}(\text{OH})_3$, and $\alpha\text{-AlOOH}$ (deposited from iso-propyl alcohol suspensions) were obtained and analyzed. A band at $\sim 3\mu$ was observed in all cases.

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502. Haase, H., "Infrarot Bibliographie," The Hague: M. Nijhoff. 80 pp.
503. Haeusler, C., "Study of the Ammonia Band at 4μ ," *Compt. rend.*, **242**, 1153. 50-9875.
504. Harrick, N. J., "Use of Infrared Absorption to Determine Carrier Distribution in Germanium and Surface Recombination Velocity," *Phys. Rev.*, **101**, 491. 50-4626.
505. Harrick, N. J., "Use of Infrared Absorption in Germanium to Determine Carrier Distributions for Injection and Extraction," *Phys. Rev.*, **103**, 1173. 50-4626.
506. Hartert, E., and Glemser, O., "Infrared Spectroscopic Determination of Metal-Oxygen Distances in Hydroxides, Basic Salts, and Salt Hydrates," *Z. Elektrochem.*, **60**, 746. 51-857.
507. Hartmann, H., and Schlafer, H. C., "Intercombination Spectra of Transition Metal Complex Ions," *Rec. trav. chim.*, **75**, 648. 52-9746.
508. Hass, M., and Sutherland, G. B. B. M., "The Infrared Spectrum and Crystal Structure of Gypsum," *Proc. Roy. Soc. (London)*, **A236**, 427. 51-81.

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510. Hidalgo, A., and Serratos, J. M., "Application of Infrared Absorption Spectra to the Study of Clay Minerals," *Ion*, **16**, 645. 51-2396.

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515. Houziaux, C., "Infrared Absorption Spectra of Some Natural Glasses Between 2 and 24 Microns," *Geochim. et Cosmochim. Acta*, **9**, 298. 51-6337.

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517. Jones, L. H., "Determination of Accurate Water Vapor Calibration Points for Prism Spectrometers in the Region 1330-2100 cm^{-1} ," *J. Chem. Phys.*, **24**, 1250. 50-12651.
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METAL-ORGANIC COMPLEXES

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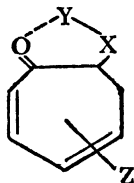
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- S-61. Ebsworth, E. A. V., Hall, J. R., MacKillop, M. J., McKean, D. C., Sheppard, N., and Woodward, L. A., "Vibrational Spectra and Structure of Trisilylamine and Trisilylamine- d_9 ," *Spectrochim. Acta*, **13**, 202. 53–6759.

Gaseous $N(SiH_3)_3$ and $N(SiD_3)_3$ were studied from 4000–400 cm^{-1} . The data suggest a planar structure for NSi_3 .

- S-62. Fabbri, G., and Cappellina, F., "Infrared Spectrum of Potassium Octacyanotungstate in the Crystalline State and in Aqueous Solution," *Atti accad. nazl. Lincei, Rend., Classe sci. fis., mat. e nat.*, **25**, 299. 53–17668.

- S-63. Falk, M., and Giguère, P. A., "Infrared Spectra and Structure of Selenious Acid," *Can. J. Chem.*, **36**, 1680. 53–6757.

Methanolic solutions of H_2SeO_3 and D_2SeO_3 and films of H_2SeO_3 on AgCl plates were examined from 3000–400 cm^{-1} . The data are consistent with the structure $(\text{HO})_2\text{SeO}$.

- S-64. Farmer, V. C., "The Infrared Spectra of Talc, Saponite, and Hectorite," *Mineral. Mag.*, **31**, 829. 53–1918.

The spectra of these minerals in the region 400–4000 cm^{-1} are similar.

- S-65. Filimonov, V. N., "Electronic Absorption Bands of ZnO and TiO_2 in the Infrared Region of the Spectrum," *Optika i Spektroskopiya*, **5**, 709. 53–9818.
- S-66. Frasco, D. L., "Interpretation of the Vibrational Spectra of Solid Hydroxyl-Ammonium Salts," *Dissertation Abstr.*, **19**, 1219.
- S-67. Gailar, N. M., " ν_2 Band of HDO and the Design and Construction of an Infrared Spectrometer," *Dissertation Abstr.*, **19**, 839.
- S-68. Garing, J. S., "High-Resolution Study of the 6-, 10-, and 16- μ Vibration-Rotation Bands of Ammonia," *Dissertation Abstr.*, **19**, 839.
- S-69. Gatehouse, B. M., "Survey of the Infrared Spectra of NO_2 in Metal Complexes," *J. Inorg. and Nuclear Chem.*, **8**, 79. 53–5868.
- S-70. Gatehouse, B. M., and Comyns, A. E., "Infrared Spectra of Uranyl Nitrate Hydrates and Rubidium Uranyl Nitrate," *J. Chem. Soc.*, 3965. 53–1918.

Spectra of hydrated uranyl nitrate and hydrated rubidium uranyl nitrate were obtained from 700–3700 cm^{-1} .

- S-71. Gatehouse, B. M., Livingstone, S. E., and Nyholm, R. S., "Infrared Spectra of Some Nitrate and Other Oxyanion Coordination Complexes," *J. Inorg. and Nuclear Chem.*, **8**, 75. 53–5868.
- S-72. Gilfert, J. C., and Williams, D., "Pressure Modulation of Infrared Absorption. I. Entire Vibration-Rotation Bands," *J. Opt. Soc. Am.*, **48**, 765.

Pressure-modulation absorption bands of CO , N_2O , methane, and propane are discussed.

- S-73. Gillis, R. G., "The Intensity of Infrared Absorption Bands—A Bibliography," *Australian, Commonwealth, Dept. Supply Defence Standards Labs. Tech. Memo*, **2**, 48 pp. 53–14673.
- S-74. Griffith, W. P., Lewis, J., and Wilkinson, G., "Infrared Spectra of Transition Metal-Nitric Oxide Complexes. II. Complexes Involving NO^- Ion," *J. Inorg. and Nuclear Chem.*, **7**, 38. 53–50.
- S-75. Groskaufmanis, A., Shakalina, A., and Liepina, L., "Vibrational Spectrum of the O—H Group, and Bound Water in the Molecule," *Latvijas Valsts Univ. Kim. Fak. Zinātniskie Raksti*, **22**, No. 6, 107. 53–12831.

Spectra between 1.25 and 1.65 μ indicate that O—H exists in aqueous solutions of many aluminum salts. Salts investigated were $\text{Al}(\text{OH})_2\text{Cl}$, $\text{Al}(\text{OH})\text{Cl}_2$, AlCl_3 and other neutral aluminum salts.

- S-76. Harbeke, G., and Lautz, G., "The Infrared Absorption of Gallium Telluride and Indium Telluride," *Z. Naturforsch.*, **13A**, 775. 53-5878.

Absorption spectra were obtained at temperatures from 20°-650°K.

- S-77. Harihan, T. A., "The Force Constants of Some Polyatomic Molecules and Ions," *J. Indian Inst. Sci.*, **40**, 89. 53-18627.

Calculations are made for OPBr_3 , SPBr_3 , CaBr_4^{-2} , InBr_4^{-2} , TlBr_4^{-2} , TlBr_4^{-2} , and CdBr_4^{-2} .

- S-78. Heilmann, G., "Optical Constants n and k of Lithium Fluoride in the Region of the Infrared Reststrahlen Band," *Z. Naturforsch.*, **13a**, 238. 53-2785.

- S-79. Hexter, R. M., "Infrared Absorption Spectra of Crystalline Brucite $[\text{Mg}(\text{OH})_2]$ and Portlandite $[\text{Ca}(\text{OH})_2]$," *J. Opt. Soc. Am.*, **48**, 770. 53-4902.

- S-80. Hooge, R. N., and Ketelaar, J. A. A., "Assignment of the Vibrational Frequencies of S_2X_2 Molecules," *Rec. trav. chim.*, **77**, 902. 53-3881.

Molecules of the S_2X_2 type where X is H, methyl, or halogen were studied. A band at ca. 250 cm^{-1} was assigned to the SS stretching mode.

- S-81. Horák, M., "Technique of Measurement of Infrared Spectra of Solids," *Chemi (Prague)*, **10**, 710. 53-21125.

Review.

- S-82. Hornig, D. F., White, H. F., and Reding, F. P., "The Infrared Spectra of Crystalline H_2O , D_2O , and HDO ," *Spectrochim. Acta*, **12**, 338. 53-4901.

Samples prepared by freezing the liquid or condensing the vapor were studied at temperatures to -190°C in the region $3700\text{--}200\text{ cm}^{-1}$.

- S-83. Huld, L., and Staffin, T., "Infrared Absorption of Photogenerated Free Carriers into Germanium," *Phys. Rev. Letters*, **1**, 236. 53-59.

- S-84. Huld, L., and Staffin, T., "Valence Band Structure of Silicon," *Phys. Rev. Letters*, **1**, 313. 53-874.

- S-85. Jones, L. H., "Vibrational Spectrum and Structure of Metal-Cyanide Complexes in the Solid State. IV. $\text{K}_3\text{Cu}(\text{CN})_4$," *J. Chem. Phys.*, **29**, 463. 53-1920.

- S-86. Jones, W. D., "Vibrational Relaxation Time Studies on Carbon Monoxide, by the Infrared Spectrophone Method," *Dissertation Abstr.*, **19**, 456.

- S-87. Josien, M. L., Grange, P., and Lascombe, J., "Study of Hydrofluoric Acid Dissolved in Carbon Tetrachloride and Some Aromatic Solvents by Infrared Spectroscopy," *Compt. rend.*, **246**, 3339. 53-2785.

Bands corresponding to polymerized $(\text{HF})_n$ molecules were located at 3550 and 3300 cm^{-1} .

- S-88. Kirillov, E. A., Nesterovskaya, E. A., and Gol'denberg, A. B., "The Infrared Region of the Absorption Spectrum of Interior Centers in

Fine-Grained Silver Bromide and Silver Chloride Emulsions," *Pratsi Odes'k. Derzhav. Univ. im. I. I. Mechnikova, Ser. Fiz.*, **148**, No. 6, 15. 53-21186.

S-89. Klier, M., "Temperature Dependence of the Optical Constants of Lithium and Sodium Fluorides in the Infrared," *Z. Physik*, **150**, 49. 53-21204.

S-90. Kostyshin, M. T., "Optical Constants of Germanium in the Region 2-25 Microns," *Optika i Spektroskopiya*, **5**, 312. 53-1927.

S-91. Kriegsmann, H., and Forster, W., "Spectroscopic Investigations of Silicon Compounds. V. The Vibration Spectrum of Trisilylamine," *Z. anorg. u. allgem. Chem.*, **298**, 212. 53-7762.

S-92. Krogh-Moe, J., "The Infrared Spectra of Some Vitreous and Crystalline Borates," *Arkiv Kemi*, **12**, 475. 53-2785.

Alkali halide disks containing crystalline and vitreous samples of alkali borates were examined between 5000 and 667 cm^{-1} .

S-93. Lafon, J., "Raman and Infrared Spectra of the Double Guanidine-Aluminum Sulfate," *Compt. rend.*, **247**, 2120. 53-21160.

S-94. Lazarev, A. N., "Absorption Spectrum of KH_2PO_4 in the Region of Hydroxyl Valence Vibration," *Zhur. Tekh. Fiz.*, **27**, 426. 53-4902.

Absorption data for the region 4000-1818 cm^{-1} is reported.

S-95. Lehmann, W. J., Ditter, J. F., and Shapiro, I., "Infrared Spectra of Partially Deuterated Diboranes," *J. Chem. Phys.*, **29**, 1248. 53-6758.

S-96. Lely, J. A., and Kröger, F. A., "Optical Properties of Pure and Doped Silicon Carbide," *Semiconductors and Phosphors, Proc. Intern. Colloq., Garmisch-Partenkirchen*, 514. 53-16709.

S-97. Lewis, J., Irving, R. J., and Wilkinson, G., "Infrared Spectra of Transition Metal-Nitric Oxide Complexes. I. Complexes Involving Donation From the NO^+ Ion," *J. Inorg. and Nuclear Chem.*, **7**, 32. 53-50.

S-98. Linton, H. R., and Nixon, E. R., "Vibrational Spectrum of Disilyl Sulfide," *J. Chem. Phys.*, **29**, 921. 53-3880.

A value of 100 degrees has been estimated for the Si-S-Si angle on basis of infrared and Raman spectral data obtained for $\text{SiH}_3\text{SSiH}_3$ and its deuterated analog.

S-99. Linton, H. R., and Nixon, E. R., "Infrared Spectrum and Force Constants of Silyl- d_3 -Iodide," *Spectrochim. Acta*, **12**, 41. 53-4903.

Gaseous SiD_3I was studied from 4000-300 cm^{-1} .

S-100. Lippincott, E. R., Valkenburg, A. V., Weir, C. E., and Bunting, E. N., "Infrared Studies on Polymorphs of Silica and Germania," *J. Research Natl. Bur. Standards*, **61**, No. 1, 61 (Research Paper 2885). 53-5876.

The region 4000-300 cm^{-1} was investigated for coesite, cristobalite, quartz, vitreous silica, hexagonal and tetragonal GeO_2 and vitreous germania.

S-101. Lygin, V. I., "Variation of the Infrared Absorption Spectra of the Hydroxyl Groups of Porous Glass and Silica Gel During Thermal De-

hydration," *Vestnik Moskov. Univ., Ser. Mat., Mekh., Astron., Fiz., Khim.*, 13, No. 1, 223. 53-2799.

- S-102. Majumdar, A. K., Duval, C., and Lecomte, J., "A New Case of Isomerism Revealed by Infrared Spectrometry," *Compt. rend.*, 247, 302. 53-3879.

Two isomeric forms of $\text{Co}(\text{NH}_3)_3(\text{NO}_2)_3$ were revealed by means of infrared spectrometry.

- S-103. Mathieu, J. P., "Vibration Spectra and Structure of Coordination Complexes," *J. Inorg. and Nuclear Chem.*, 8, 33. 53-5864.

A review.

- S-104. Matossi, F., "Infrared Absorption of n-Conducting Indium Arsenide," *Z. Naturforsch.*, 13a, 767.

- S-105. McDonald, R. S., "Surface Functionality of Amorphous Silica by Infrared Spectroscopy," *J. Phys. Chem.*, 62, 1168. 53-2804.

- S-106. McKean, D. C., "Force Constants, Interbond Angle, and Solid State Spectrum of Disiloxane," *Spectrochim. Acta*, 13, 38. 53-6762.

- S-107. Meyer, H. J. G., "Infrared Absorption by Conduction Electrons in Germanium," *Phys. Rev.*, 112, 298. 53-3895.

- S-108. Miloslavskii, V. K., and Kovalenko, N. A., "Absorption of Zinc Oxide in the Infrared Region of the Spectrum," *Optika i Spektroskopiya*, 5, 614. 53-10958.

A maximum at 1818 cm^{-1} is observed in samples of ZnO when absorption is measured from $10,000\text{--}625\text{ cm}^{-1}$.

- S-109. Mitra, S. S., "Infrared and Raman Spectra of Brucite, $\text{Mg}(\text{OH})_2$," *Dissertation Abstr.*, 19, 841. 53-2784.

- S-110. Mitsuishi, A., Yoshinaga, H., and Fujita, S., "Far-Infrared Absorption of Sulfides, Selenides, and Tellurides of Zinc and Cadmium," *J. Phys. Soc. Japan*, 13, 1235. 53-2785.

- S-111. Mitsuishi, A., Yoshinaga, H., and Fujita, S., "Far-Infrared Absorption of Ferrites," *J. Phys. Soc. Japan*, 13, 1236. 53-2785.

The region $500\text{--}10\text{ cm}^{-1}$ was investigated for ZnFe_2O_4 , NiFe_2O_4 , CoFe_2O_4 , and MgFe_2O_4 .

- S-112. Mizushima, S., Nakagawa, I., Schmelz, M. J., Curran, C., and Quagliano, J. V., "Infrared Absorption Spectra of Inorganic Coordination Complexes. XVII. Infrared Spectra of Platinum (II) and Palladium (II) Ammine Complexes," *Spectrochim. Acta*, 13, 31. 53-6757.

The assignment of bands in the $5000\text{--}667\text{ cm}^{-1}$ region for 9 inorganic complexes is reported.

- S-113. Nakagawa, I., Mizushima, S., Saraceno, A. J., Lane, T. J., and Quagliano, J. V., "Infrared Absorption Spectra of Inorganic Coordination Complexes. XV. Normal Vibrations of Sulfamic Acid, $\text{H}_3\text{N}^+ - \text{SO}_3^-$," *Spectrochim. Acta*, 12, 239. 53-4902.

- S-114. Nakamoto, K., Fujita, J., and Murata, H., "Infrared Spectra of Metallic

- Complexes. V. Nitro and Nitrito Complexes," *J. Am. Chem. Soc.*, **80**, 4817. 53-12832.
- S-115. Narasimhan, P. T., "Infrared Intensities and the Nature of the Chemical Bond. I. Variation of Hybridization With Bond Length in SO_2 , NH_3 , and PH_3 ," *Proc. Natl. Instl. Sci. India, Pt. A*, **24**, 55. 53-857.
- S-116. Newman, R., "Optical Properties of n-Type Indium Phosphide," *Phys. Rev.*, **111**, 1518. 53-2811.
- S-117. Ockman, N., "The Infrared and Raman Spectra of Ice," *Advance in Phys.*, **7**, 199. 53-8793.
- Review.
- S-118. Orssmann, K., "High-Resolution Study of Some Vibration-Rotation Bands of $\text{C}^{12}\text{O}_2^{16}$ and H_2Te ," *Dissertation Abstr.*, **19**, 341.
- S-119. Pistorius, C. W. F. T., "Potential Field and Force Constants of Octahedral Molecules," *J. Chem. Phys.*, **29**, 1328. 53-7762.
- Calculations are made for SF_6 , SeF_6 , TeF_6 , ReF_6 , IrF_6 , UF_6 , NpF_6 , PuF_6 , TiCl_6^{-2} , SeCl_6^{-2} , SnCl_6^{-2} , SbCl_6^{-2} , SnBr_6^{-2} , MoF_6 and WF_6 .
- S-120. Pitavskii, L. P., "Anomalous Skin Effect in the Infrared Region," *Zhur. Ekspt. i Teoret. Fiz.*, **34**, 942. 53-1912.
- S-121. Plyler, E. K., and Acquista, N., "Transmission and Reflectance of Cesium Iodide in the Far-Infrared Region," *J. Opt. Soc. Am.*, **48**, 668. 53-3878.
- S-122. Powell, J. M., and Kurnick, S. W., "Indium Antimonide Infrared Filter," *J. Appl. Phys.*, **29**, 1129. 53-874.
- Transmission data for InSb (n-type) with 10^{17} donors/c.c. are reported at 0° and 25° .
- S-123. Prigent, J., "Infrared Absorption Spectra of Anhydrous Uranyl Halides," *Compt. rend.*, **247**, 1737. 53-10961.
- S-124. Pryce, M. H. L., and Runciman, W. A., "The Absorption Spectrum of Vanadium Corundum," *Discussions Faraday Soc.*, No. **26**, 34. 53-15765.
- S-125. Sartori, G., Furlani, C., and Damiani, A., "Vibrational Frequencies of Water in Complexes," *J. Inorg. and Nuclear Chem.*, **8**, 119. 53-5868.
- S-126. Saum, G. A., "Fundamental Optical Absorption in the II A—VI B Compounds," *Dissertation Abstr.*, **19**, 342.
- S-127. Saumagne, P., and Josien, M. L., "Valence Vibrations of H_2O , D_2O , and HDO Molecules in Organic Solvents," *Bull. soc. chim. France*, **813**. 53-18626.
- S-128. Schatz, P. N., "Potential Function of NF_3 ," *J. Chem. Phys.*, **29**, 481. 53-858.
- S-129. Schatz, P. N., and Levin, I. W., "Absolute Infrared Intensities of the Fundamental Vibrations of NF_3 ," *J. Chem. Phys.*, **29**, 475. 53-858.
- S-130. Serratos, J. M., and Bradley, W. F., "Determination of the Orientation of OH Bond Axes in Layer Silicates by Infrared Absorption," *J. Phys. Chem.*, **62**, 1164. 53-2804.

- S-131. Sheldon, J. C., and Tyree, S. Y., "Addition Compounds of Metal Halides with POX_3 Compounds," *J. Am. Chem. Soc.*, **80**, 4775. 53-3880.
- S-132. Siebert, H., "Infrared Spectra of Cobalt (III) Complexes With Ammonia and Anions of Oxyacids as Ligands," *Z. anorg. u. allgem. Chem.*, **298**, 51. 53-7762.

Co(III) complexes of NH_3 and anions of oxyacids (CO_3^{2-} , HCO_2^- , $\text{C}_2\text{O}_4^{2-}$, NO_2^- , NO_3^- , SO_4^{2-} , ONO^- , SO_3^{2-}) were studied in the region 4000-600 cm^{-1} .

- S-133. Smith, D. F., "Molecular Properties of Hydrogen Fluoride," *Proc. U. N. Intern. Conf. Peaceful Uses At. Energy*, 2nd, Geneva, **28**, 130. 54-68.

Pressure-broadening of HF lines by other gases is described. Absorption shifts related to complexes and polymer structures are discussed.

- S-134. Spinar, L. H., and Margrave, J. L., "Absorption Spectra of Gaseous Alkali Metal Hydroxides at High Temperatures," *Spectrochim. Acta*, **12**, 244. 53-4902.

Temperatures up to 1000° have been employed in the absorption studies of LiOH, NaOH, KOH, and RbOH.

- S-135. Stammreich, H., Bassi, D., Sala, O., and Siebert, H., "The Vibrational Spectrum of the Dichromate Ion," *Spectrochim. Acta*, **13**, 192. 53-6757.

- S-136. Steger, E., "Spectroscopic Investigation of Condensed Phosphates and Phosphoric Acids. V. Structural Differences of the Trimetaphosphate Ion in Crystal and in Solution," *Z. anorg. u. allgem. Chem.*, **29**, 305. 53-1918.

- S-137. Stekhanov, A. I., "Structure of the OH Vibration Band in Crystal Spectra Containing a Hydrogen Bond," *Izvest. Akad. Nauk S.S.S.R., Ser. Fiz.*, **22**, 1109. 53-869.

Infrared and Raman spectra reveal a large, complex-structured band for crystals containing the OH group. At low temperature the band was found to consist of narrow bands and sharp lines. Among the 20 crystals studied were gypsum, Seignette salt, and NaH_2PO_4 .

- S-138. Štokr, J., and Schneider, B., "Vibrational Spectra of Some Halogenated Silanes. II. Vibrational Spectra of Trichlorohalogen Silanes," *Chem. listy*, **52**, 985. 53-49.

Raman and infrared data are reported for the region 450-2000 cm^{-1} .

- S-139. Tarte, P., "Researches on the Deformation Frequencies of OH. I. Infrared Spectra of Basic Copper Salts," *Spectrochim. Acta*, **13**, 107. 53-6757.

- S-140. Vincent-Geisse, J., Queyrel, M., and Lecomte, J., "Infrared Optical Constant Determination of Very Absorbing Solids," *Compt. rend.*, **247**, 1330. 53-21190.

Results are reported for quartz and calcite.

- S-141. Waddington, T. C., "Infrared Spectra, Structure, and Hydrogen Bonding in Ammonium Salts," *J. Chem. Soc.*, 4340. 53-3880.

Spectra-structure correlations are made for the following ammonium salts: NH_4N_3 , NH_4HF_2 , NH_4NCO , NH_4NCS , NH_4NO_3 , NH_4ClO_4 , NH_4BF_4 , NH_4BPh_4 , NH_4PF_6 , $\text{NH}_4\text{SO}_3\text{F}$, and $\text{NH}_4\text{SO}_3\text{CH}_3$.

- S-142. Wickersheim, K. A., "Preparation of Thin Crystalline Samples of the Alkali Hydroxides for Infrared Absorption Studies," *J. Chem. Phys.*, 29, 1197. 53-5867.

- S-143. Yamamoto, G., and Sasamori, T., "Calculation of the Absorption of the 15μ Carbon Dioxide Band," *Sci. Repts. Tohoku Univ. Fifth Ser.*, 10, 37. 53-13776.

- S-144. Yaroslavskii, N. G., and Stanevich, A. E., "Rotational Spectrum of Water in the Long-Wave Infrared Region $50\text{--}1500\mu$ ($200\text{--}7\text{ cm}^{-1}$)," *Optika i Spektroskopiya*, 5, 384. 53-10958.

- S-145. Yatsenko, A. F., "Infrared Absorption Spectra of Perovskite-Type Ferroelectric Crystals," *Izvest. Akad. Nauk S.S.S.R., Ser. Fiz.*, 22, 1456. 53-5878.

Absorption data and force constants are reported for PbTiO_3 , BaTiO_3 , CaTiO_3 , SrTiO_3 , PbZrO_3 , BaZrO_3 , SrZrO_3 , BaSnO_3 , SrSnO_3 , and WO_3 .

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- S-146. Adams, R. V., and Douglas, R. W., "Infrared Studies on Various Samples of Fused Silica With Special Reference to the Bands Due to Water," *J. Soc. Glass Technol.*, 43, 1471. 53-15766.

- S-147. Akhmanova, M. V., "Infrared Absorption Spectra of Minerals," *Uspekhi Khim.*, 28, 312. 53-11979.

Review.

- S-148. Allpress, J. G., and Hambly, A. N., "Infrared Spectra of Uranyl Compounds. I. Uranyl Nitrates," *Australian J. Chem.*, 12, 569. 54-5245.

Anhydrous and hydrated uranyl nitrates were studied between 3800 and 700 cm^{-1} .

- S-149. Arneth, R., "Absorption of ZnO Single Crystals in the Infrared," *Z. Physik*, 155, 595. 53-18635.

Pure and doped ZnO crystals were studied between $10,000$ and 700 cm^{-1} .

- S-150. Bán, M. I., "Vibrational Symmetries of Tetracyanonickelate (II) Ion," *Acta Chim. Acad. Sci. Hung.*, 19, 35. 53-19557.

- S-151. Barraclough, C. G., Lewis, J., and Nyholm, R. S., "Stretching Frequencies of Metal-O Double Bonds," *J. Chem. Soc.*, 3552. 54-6306.

The region $900\text{--}1100\text{ cm}^{-1}$ has been associated with a stretching

frequency of the metal-oxygen bond. Compounds studied contained Cr—O, Mn—O, V—O, Re—O, Mo—O, or Ti—O bonds.

- S-152. Benesi, H. A., "Infrared Spectrum of Magnesium Hydroxide," *J. Chem. Phys.*, **30**, 852. 53-13776.

- S-153. Benesi, H. A., and Jones, A. C., "An Infrared Study of the Water-Silica Gel System," *J. Phys. Chem.*, **63**, 179. 53-10958.

Infrared spectrum of physically adsorbed water is compared with that of "unbound" water.

- S-154. Bernard, M. P., "Infrared Absorption Spectrum at Low Temperature of Crystalline D-Substituted Seignette Salt ($7500\text{--}5300\text{ cm}^{-1}$)," *Compt. rend.*, **248**, 1153. 53-15766.

- S-155. Bertin, E. P., Mizushima, S., Lane, T. J., and Quagliano, J. V., "Infrared Absorption Studies of Inorganic Coordination Complexes. XXI. Nitrosopentammine Cobalt Halides and Nitrate," *J. Am. Chem. Soc.*, **81**, 3821. 54-4149.

- S-156. Bertin, E. P., Penland, R. B., Mizushima, S., Curran, C., and Quagliano, J. V., "Infrared Absorption Studies of Inorganic Coordination Complexes. XX. Cobalt (III) Ammine Complexes Containing Some Coordinated Anions," *J. Am. Chem. Soc.*, **81**, 3818. 54-4149.

- S-157. Biermann, W. J., and Gilmour, J. B., "Hydronium Ion Absorption in the region 1.7μ ," *Can. J. Chem.*, **37**, 1249. 54-69.

- S-158. Block, H. A., "Vibrational Study of the Hexammine-Cobalt (III) Ion," *Trans. Faraday Soc.*, **55**, 867. 54-6305.

Potassium chloride pellets containing $\text{Co}(\text{NH}_3)_6\text{Cl}_3$ were examined from $5000\text{--}416\text{ cm}^{-1}$.

- S-159. Bor, G., and Markó, L., "Infrared Spectrum of Dicobalto Octacarbonyl," *Spectrochim. Acta*, **747**. 54-4149.

Principal absorption bands for $\text{Co}_2(\text{CO})_8$ in the LiF region are reported.

- S-160. Boyle, W. S., "Far Infrared Magneto-optic Effects from Impurities in Germanium," *Phys. and Chem. Solids*, **8**, 321. 53-12849.

Magneto-optic effects were observed in Ge-doped with arsenic and phosphorus.

- S-161. Braunholtz, J. T., Hall, G. E., Mann, F. G., and Sheppard, N., "Infrared Spectra and Hydrogen Bonding in Compounds Containing $\text{X}(\text{O})$. OH Groups," *J. Chem. Soc.*, **868**. 53-11994.

Bands associated with OH vibrations are studied for compounds of the type $\text{X}(\text{O})$. OH, where X is P, As, S, Se, and C.

- S-162. Buchanan, R. A., "Near Infrared Spectra of Crystalline Alkali Hydroxides," *J. Chem. Phys.*, **31**, 870. 54-6304.

- S-163. Burnside, P. B., "Pure Rotational Absorption Spectrum of Deuterium Sulfide," *Dissertation Abstr.*, **19**, 3329. 53-18626.

- S-164. Caldwell, R. S., and Fan, H. Y., "Optical Properties of Tellurium and Selenium," *Phys. Rev.*, **114**, 664. 53-18638.

Studies were made on Te and trigonal and amorphous Se to wavelengths of 152μ using polarized radiation.

- S-165. Catalano, E., and Milligan, D. E., "Infrared Spectra of H_2O , D_2O , and HDO in Solid Argon, Krypton, and Xenon," *J. Chem. Phys.*, **30**, 45. 53-9812.
- S-166. Church, C. H., "High-Resolution Electric Field-Induced Infrared Absorption Spectra of H_2 and D_2 ," *Dissertation Abstr.*, **20**, 1398. 54-4147.
- S-167. Claassen, H. H., "Force Constants of Metal Hexafluorides," *J. Chem. Phys.*, **30**, 968. 53-17668.
- S-168. Cleek, G. W., and Scuderi, T. G., "Effect of Fluorides on Infrared Transmittance of Certain Silicate Glasses," *J. Am. Ceram. Soc.*, **42**, 599. 54-5245.
- S-169. Cleek, G. W., Villa, J. J., and Hahner, C. H., "Refractive Indexes And Transmittances of Several Optical Glasses in the Infrared," *J. Opt. Soc. Am.*, **49**, 1090. 54-1069.

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- S-170. Colpa-Boonstra, J. P., "Rotational Spectra of Pure Molecular Hydrogen Induced by Pressure," *Colloq. intern. centre natl. recherche sci. (Paris)*, No. 77. 53-21155.
- S-171. Cotton, F. A., Down, J. L., and Wilkinson, G., "Infrared Spectra of Manganese Carbonyl Hydride and Deuteride," *J. Chem. Soc.*, 833. 53-10960.

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- S-172. Coulon, R., and Hai, V., "Some New Results on the Infrared Spectra of Mixtures of Compressed Gases," *Coll. internl. centre natl. recherche Sci. (Paris)*, No. 77, 99. 54-2937.
- S-173. Courtoy, C. P., "Infrared Spectra of Carbon Dioxide at High Dispersion and Constant Molecular Concentration," *Ann. soc. sci. Bruxelles, Ser. I*, **73**, 5. 53-15762.

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- S-176. Dows, D. A., "Torsional Vibrations in Carbon Dioxide and Nitrous Oxide Crystals," *Spectrochim. Acta*, **13**, 308.

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- S-178. Dunlap, J. L., "Infrared Intensities, Bond Moments, and Bond Moment Derivatives for PF_3 and POF_3 ," *Dissertation Abstr.*, **20**, 1398. 54–4147.

- S-179. Duval, C., and Lecomte, J., "Infrared Absorption Spectra of Mixed Crystals: CuSO_4 and ZnSO_4 and Their Interpretation," *Compt. rend.*, **248**, 1977. 53–14686.

- S-180. Duyckaerts, G., "The Infrared Analysis of Solid Substances," *Analyst*, **84**, 201. 53–16691.

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- S-185. Fateley, W. G., Bent, H. A., and Crawford, Jr., B., "Infrared Spectra of the Frozen Oxides of Nitrogen," *J. Chem. Phys.*, **31**, 204. 54–1069.

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- S-187. Folman, M., and Yates, D. J. C., "Infrared Studies of Physically Adsorbed Polar Molecules and of the Surface of a Silica Adsorbent Containing Hydroxyl Groups," *J. Phys. Chem.*, **63**, 183. 53–10964.

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- S-190. Fujii, S., "Infrared Spectra of Coal," *Nenryô Kyôkaishi*, 38, 442. 54-5233.
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- S-193. Gandy, H. W., "Optical Transmission of Heat-Treated Strontium Titanate," *Phys. Rev.*, 113, 795. 53-13789.
- S-194. Garland, C. W., "Effect of Poisoning on the Infrared Spectrum of Carbon Monoxide Adsorbed on Nickel," *J. Phys. Chem.*, 63, 1423. 54-5249.
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- S-198. Goubeau, J., Heubach, E., Paulin, D., and Widmaier, I., "The Vibration Spectra of Some Silicon Cyanates," *Z. anorg. u. allgem. Chem.*, 300, 194. 53-21155.
- S-199. Goubeau, J., and Hummel, D., "Vibrational Spectra of Various Boron-Oxygen Compounds," *Z. physik. Chem. (Frankfurt)*, 20, 15. 54-1070.
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- S-209. Jahn, A., "Inorganic Compounds of Nitric Oxide. X. The N—O Stretching Frequencies of Nitric Oxide Complexes of Iron Group Metals," *Z. anorg. u. allgem. Chem.*, **301**, 301. 54-4150.
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- S-213. Kessler, F. R., "Infrared Absorption of Free Carriers Produced in Semiconductors by the Photo Effect," *Phys. and Chem. Solids*, **8**, 275.
- S-214. Ketelaar, J. A. A., "Infrared Spectra of Compressed Gases," *Spectrochim. Acta*, **14**, 237. 53-14673.

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