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## INFRARED SPECTROSCOPY...NOW and SOON

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### Introduction

The interaction of electromagnetic radiation with matter is observed by all of us and studied by specialists in many branches of the sciences. Infrared waves make objects warm; light waves cause them to be visible; x-rays penetrate less dense forms of matter, casting shadows of more dense hidden objects.

To the absorption spectroscopist, infrared exhibits a tremendous range of applications. It is a near-magic probe into the motions of atomic groups in molecules. It's the doctor's stethoscope, the designer's inspiration, and the builder's model in molecular diagnosis and synthesis. In the short span of two decades of rapid extension in application, it has served to unravel the structure of new drugs so that life-saving ones could be manufactured. It has pointed the way to production of better fuels, rubber, fibers, plastics and coatings; it has contributed to fundamental knowledge of energies involved in molecular bonds. In short, wherever an improved understanding of molecular structure helps mankind's progress, infrared spectroscopy is a tremendously valuable aid.

### Technical Background

Electromagnetic radiation is absorbed by molecules whenever the energy in the radiation corresponds exactly to the energy of some motion of groups within the molecule. If this motion is a stretching vibration between different atoms or groups of atoms, or a twisting, rocking or bending along an axis about which there is a dipole moment, the frequency of change of electric moment involved corresponds to energy in the infrared region of the electromagnetic spectrum.

The masses and bond strengths of the particular groups in motion determine the exact amount of energy involved, and therefore determine the specific frequency of infrared radiation absorbed. This correspondence of energy to frequency is expressed by Planck's law,  $E = h\nu$ , which states simply that the energy in radiation is equal to a constant ( $h$ ) times the frequency ( $\nu$ ) of the radiation.

The way in which the masses and bond strengths of the atomic groups within the molecule are related to frequency is most readily seen by analogy to molecular models wherein the

Clara D. Smith has two families: one at home consisting of her husband, Ray, and three children; the other at Battelle consisting of hundreds of thousands of organic molecules. Equally successful with both families, she finds time to coach Linda Sue, 6, at the piano and swim and sail with two younger boys, Stevie and Derrin. As an infrared spectroscopist, Mrs. Smith was first employed by Esso Research Laboratories in Linden, N. J. Since 1949, she has headed the well-staffed and beautifully-equipped laboratory at Battelle Memorial Institute. Last year, with E. R. Mueller, she won the Carbide and Carbon Award administered by ACS. Other honors have come regularly starting with a Phi Beta Kappa key and cum laude graduation from Ohio State in 1945. Always anxious to lend her energetic and respected hand to organizational work, she is chairman of the Committee on Infrared Spectral Data of the Coblenz Society and is active on Committee E-13 of ASTM, the Advisory Board of the Documentation of Molecular Spectra, the American Chemical Society and the Optical Society of America.



bonds are considered as springs of certain strengths joining the atoms. This system may be considered, to a first approximation, to follow Hooke's law and can be expressed by equations which hold for simple harmonic vibrations, i.e.,

$$\nu = \frac{1}{2\pi c} (K/\mu)^{1/2}$$

where  $\nu$  is the frequency in  $\text{cm}^{-1}$ ,  $c$  is the velocity of light,  $\mu$  is the reduced mass of vibrating atoms

$$\left( \frac{1}{\mu} = \frac{1}{m_1} + \frac{1}{m_2} \right)$$

and  $k$  is the force constant which exists between the atoms.

Of course, this equation relates only to one pair of masses joined by a particular strength bond. A molecule of several atoms will thus have many ways in which pairs of atoms may vibrate, bend, rock or twist relative to each other and relative to other pairs or larger groups of atoms. Actually, for a non-linear molecule, where  $n$  is the number of atoms, there are  $3n-6$  modes of vibration. This does not include combination and difference frequencies and harmonics which are also observed.

Accordingly, a molecule exhibits absorption at many different frequencies. For a given molecule, if the amount of absorption is plotted against frequency (or wavelength), the pattern yields a characteristic spectrogram, or "fingerprint" of the molecule. So powerful is this identification tool that, for organic molecules, only stereoisomers defy distinction by infrared spectroscopy.

### Instrumentation

Basically, the equipment required for infrared spectroscopy is a source unit, optics for passing the beam through the sample and for dispersing it into separate wavelengths, and a detector for measuring the changes in energy transmitted.

Simple in principle, infrared components are, nevertheless, expensive. The cartoon depicting one man in Purchasing exclaiming to another, "Good heavens, those guys out in the lab are paying \$450 a pound for rock salt!" has its root in the necessity of using unusual synthetic crystals as prism materials. The news release a few years ago about scientists with "gold-lined" equipment stemmed from the occasional practice of using gold-surfaced mirrors to maintain high reflectivity in corrosive atmospheres.

Far more exciting than these eye-catchers is the steady struggle by instrument designers to translate the very feeble energy decrease by sample absorption into a reliable electrical signal. There has been a large array of obstacles in the path of development of a stable, directly-recording percent transmission infrared spectrometer. These include such physical factors as the continuously decreasing source energy toward longer wavelengths from the near-infrared energy peak; the difficulty in developing a sufficiently sensitive and stable thermocouple or bolometer; the susceptibility of prism materials to clouding by moisture and to dimensional changes from ambient temperatures; and absorption bands of carbon dioxide and water vapor in the air which obscure parts of the spectrum.

The changes in intensity of the source energy with wavelength are compensated for by automatic programming of slits with mechanical and electrical cams. Spectrometer cases are temperature-controlled to minimize wavelength errors. Spectrometers are evacuated or flushed with dry nitrogen to diminish atmospheric absorption bands. Double-beam spectrometers have been developed which continuously compare the sample beam signal with a reference-beam signal. Compensations of source fluctuation, atmospheric absorption and electronic variations are thus achieved.

### Spectral Interpretation

As pointed out earlier, the observed infrared absorption bands result from certain motions of the atoms in molecules. It was recognized as long ago as the turn of the century that there are also characteristic frequencies for particular structural groups. To quote from the writings of W. W. Coblentz at the time, "There is something . . . call it a particle, a group of atoms, ion or nucleus . . . in common, with many of the compounds studied, which causes absorption bands that are characteristic of the great groups of organic compounds." It has since been repeatedly shown that there are atomic groupings whose motions are free enough of interference from the rest of the molecule to be recognizably consistent. Charts showing the results of empirical correlation of functional groups with frequency appeared during the forties, and refinements are still being added. A 1954 publication, "The Infrared Spectra of Complex Molecules" by L. J. Bellamy remains the most comprehensive summary of these correlations to date.

We have seen that two main factors establish the absorption frequency for a given atomic grouping: the masses of the atoms and the strength of the bonds joining them. The stretching frequencies of the lightest atom, hydrogen, are the highest. Thus in the  $3700 \text{ cm}^{-1}$  to  $2100 \text{ cm}^{-1}$  range, the O-H, N-H,  $\equiv\text{C-H}$ , S-H, B-H, P-H, Si-H bands are readily recognized. A novice can unambiguously distinguish O-H and N-H from aromatic and aliphatic C-H and distinguish the last two from each other. Moreover, interpretations of the exact position and shape of the bands permit the practiced spectroscopist to tell a great deal about the environment of the group.

Next down the frequency scale, from about  $2400 \text{ cm}^{-1}$  to  $2100 \text{ cm}^{-1}$ , comes distinctive absorption from the vibrations involving strong bonds between heavier atoms: for example,  $\text{C}\equiv\text{C}$  and  $\text{C}\equiv\text{N}$  groups.

There is a region from  $2000 \text{ cm}^{-1}$  to  $1850 \text{ cm}^{-1}$  which is fairly free of fundamental absorption bands and clearly separates the hydrogen-stretching vibrations and the triple-bond-stretching vibrations from the complex region which follows.

Between  $1850 \text{ cm}^{-1}$  and  $1200 \text{ cm}^{-1}$ , there is an overlapping of different double-bond stretching groups starting at the high frequency end with  $\text{C}=\text{O}$  which overlaps  $\text{C}=\text{C}$  and  $\text{C}=\text{N}$  which in turn are superimposed on hydrogen-bending vibrations. This region becomes quite intricate because of the many double-bond groups possible and because some of these shift markedly with such variables as electronegativity and mass of substituent atoms. Of course, the very same shifting of bands which makes it impossible to assign a specific frequency to a certain  $\text{C}=\text{O}$  group or  $\text{C}=\text{N}$  group—which would be convenient for new workers in the field—is the spectrally enriching effect that enables the practiced spectroscopist to tell a great deal about the environment of a group. Such data permit him to narrow the possibilities and pinpoint the compound.

Single-bond stretching modes occur at still lower frequencies and are generally very sensitive to substituent groups. Here again the spectrum is complicated (and enriched) by hydrogen-bending frequencies which spread throughout the 6-15 micron region. This single-bond region, with bands arising from skeletal modes is the portion of the spectrum commonly considered the "fingerprint" of the molecule. It extends roughly from  $1250 \text{ cm}^{-1}$  to  $660 \text{ cm}^{-1}$ . Actually, the  $660 \text{ cm}^{-1}$  (15-micron) cut-off is not realistic as far as vibrational bands are concerned since some halogens, sulfur, and many heavier atoms have characteristic frequencies at still longer wavelengths. In practice, however, the sodium chloride prism which gives good performance from 2-15 microns ( $5000\text{-}660 \text{ cm}^{-1}$ ) becomes opaque at longer wavelengths. Therefore, the mass of empirical correlations have been established without full consideration of the potentialities of the spectral range beyond 15 microns.

The type of structural group information just discussed is most useful for samples such as complete unknowns, natural products, and complex mixtures. The need for specific identification of a compound may call for a different kind of information. For the simplest compounds precise identification may be arrived at from structural group analysis. For example, a spectrum with bands characteristic of hydroxy groups and aromatic rings and with no evidence for aliphatic C-H would suggest immediately that an unknown might be phenol. The spectrum of phenol could then be taken from a reference file for point-by-point comparison for confirmation. On the other hand, a spectrum may be devoid of all major functional groups except aliphatic C-H bands. In the past, the spectroscopist would have to recall it from memory or search through all possible reference spectra of isomeric alkane combinations to identify such a compound. Today, spectrum sorting aids are an enormous time-saver.

Currently used are several forms of sorting systems. These range from notebook systems which private laboratories have reference-indexed by major bands, to needlesort systems on edge-punched cards and to electrical sorters of coded IBM spectral data cards. Reference spectra are commercially available on coded cards for needle-sorting by absorption band position.<sup>1</sup> Coded IBM cards for rapid sorting of all of the commercially available spectra are available from the American Society for Testing Materials. These and other spectral data systems have been recently summarized by the Coblenz Society.<sup>2</sup>

Frequently, a desired compound can be completely identified by the sorting technique. If the unknown spectrum is not on file, or if it is closely similar to other spectra (usually of a related structure), the spectra isolated by the sorting procedure may still provide considerable information about the type of compound being hunted.

The coded systems usually provide considerable information in addition to wavelength position of absorption bands. Codes for elements present and physical constants are useful. A reliable empirical formula from elemental analysis is an invaluable aid to specific compound identification.

### Quantitative Analysis

Quantitative analysis by infrared rests on the fact that the amount of light absorbed by a given group (or molecule) is proportional to the number of such groups (or molecules) in the beam. This proportionality can be put into absolute terms of concentration if the path length is known and if the absorption coefficient of the pure compound under the same experimental conditions is known.

The special analytical techniques necessary to cover the range of materials the infrared spectroscopist encounters—solids, liquids, gases, trace analysis, and multi-component systems—would require a book in itself. Nor is there any single good reference to the wealth of analytical applications of infrared. Recently, a move toward bringing useful analytical schemes together was started by the Coblenz Society in cooperation with *Analytical Chemistry*. Infrared analysts are encouraged to submit a special short-form summary of methods to the Coblenz Society. These are to be reviewed and published in groups in *Analytical Chemistry*.

### Future

A look to the future is always hazardous, but the future of infrared seems so solidly bright that a few comments on trends are probably in order.

At the present state of development, the infrared spectroscopist must view his field with mixed reactions. There is pride

and wonder at the rapid strides and increase in industrial application, at the improvement in instruments and the healthy beginning of spectrum publishing systems. Frustration enters when the signal-to-noise ratio isn't high enough, or resolution good enough, or scanning time fast enough for a special application. The spectroscopist is dismayed when, after obtaining a fine spectrum on an important sample, he can't find a spectral match in the published literature.

The spectroscopist wants to work on ways of increasing his effectiveness in distinguishing among products belonging to large classes of materials such as cellulose, proteins, bacteria, and synthetic high polymers. The ways in which he can hope to do this are varied. There are obvious extensions of the present conventional spectral region. Recent instrumentation advances, centered around an infrared-sensitive photoconductor, PbS, permit rapid, high resolution spectra in the very near infrared. This region starts at 0.7 microns and usually is considered to extend to 3.0-3.2 microns. The most important information to be found here arises from overtones and combinations of hydrogen frequencies. Band intensities and symmetry in solvents of varying polarity make very detailed observation of structural variations possible. A reference library of spectra is being prepared<sup>3</sup> and researchers are actively pursuing the potentialities of this near infrared region.

Beyond the 15-micron cut-off of sodium chloride, prisms made of potassium bromide, cesium bromide and cesium iodide are being used. Investigators of the cesium bromide region (11-35 microns) are optimistic about its potentialities. One of these investigators<sup>4</sup>, who has just completed a comprehensive review of the field, prophesies thus: "I think we're on the threshold of a great future for the far infrared region for both analytical applications and structure elucidation; it will be used as a complement to the 2-15 micron range. It is exceptionally sensitive to phenomena arising from crystal lattice structure. Another 2-3 years will apparently be required before its potentialities can even be properly assessed, let alone fulfilled."

In addition to these promising extensions of the spectral range covered by chemical spectroscopists, there is much yet to be gained from a more detailed examination of the 2-15 micron region. As photo-conductive materials are extended in range of sensitivity (lead telluride is useful to 6 microns and new intermetallic alloys are continuously being investigated) instruments of higher resolution and greater stability will be realized.

Improved instrumentation can be translated into increased application in several ways. The wealth of information available in the double-bond stretching region is yet to be completely explored and the extension of use of CaF<sub>2</sub> optics and gratings can be expected to be pursued for many years before this store of information is fully tapped.

A fairly recent approach<sup>5</sup> to increasing the sensitivity of graphical presentation of minute spectral differences has been to plot the data as first and second derivatives of the observed absorptions. The value of this procedure in enhancing inflections on the side of peaks and otherwise discriminating between overlapping bands is so obvious that the fact that it has hardly been exploited must attest to the busy state of the field—so much to be done! But it can be expected that the combination of improved instrumentation, and derivative methods may increase application of infrared to members of classes of materials such as woody structures, viruses, and bacteria.

In reference to the busy day of the applied spectroscopist, there has been an interesting recent development which holds a promise of releasing the experienced spectroscopist from the more routine applications of infrared. Easy-to-operate, low-cost

spectrometers have become available. They provide spectra of suitable quality to answer many of the chemist's questions about purity and structure of his daily samples. It can be hopefully expected that these spectrometers will increase the realization of infrared's usefulness by putting it at the chemist's fingertips. At the same time it will release the infrared spectroscopist for the needed exploration and development of his field. There can be no doubt about the better things to come!

## WIG-L-BUG CONTAMINATION

Dr. Harold L. Lovell of the Pennsylvania State University points out that, when used with the KBr pellet technique, the polystyrene vials give rise to a "blank" spectrum. Of course, such a blank when expected in routine applications can often be ignored. But in examining completely unknown spectra, contamination must be considered. For other than routine applications, we therefore recommend our stainless steel or the new agate vial for grinding samples with KBr.

While on this topic, it might be well to mention the fact that, occasionally, water bands appear when a sample is ground in the Wig-L-Bug. This appears to be related to the particular batch of KBr employed and also to the grinding time. Since in but seconds the Wig-L-Bug grinds samples to a much smaller particle size than normally obtained with a hand mortar, determine and use the shortest possible grinding time for the particular weight and type of sample. If anyone has any additional information on this general subject, we shall appreciate receiving it.

## References

<sup>1</sup>National Research Council—National Bureau of Standards System and Documentation of Molecular Spectroscopy System.

<sup>2</sup>*Applied Spectroscopy*, Vol. 11, No. 2, p. 108.

<sup>3</sup>Beckman Instruments, Inc., Fullerton, California.

<sup>4</sup>Freeman Bentley, WADC, Dayton, Ohio.

<sup>5</sup>Collier and Singleton, *Infrared Analysis by the Derivative Method*, *J. of App. Chem.*, 6, 1956, pp. 495-510.

## SPEX SEMI-QUANTITATIVE STANDARDS

Introduced as the first products of our spanking new company in 1955, the SPEX SEMI-QUANTITATIVE STANDARDS have now been in existence for about 2 1/2 years. The other day we checked our records on the vital sales statistics thinking that you would be interested in knowing where and to what extent they are in use.

Almost every state in the United States wound up with a colored tack to indicate that the standards have been shipped there. Mining companies in Montana, Utah and Nevada; universities in New Hampshire, South Dakota, Alabama and Colorado; oil, chemical and metal producers in Louisiana and Oklahoma; railroads in Virginia and West Virginia; testing laboratories in New Jersey, Illinois and Texas; electronic manufacturers in Massachusetts, Pennsylvania and Indiana; aircraft companies in Connecticut, Kansas and Georgia; crime laboratories in New York, Washington, D. C., Florida; health laboratories in Ohio and California; atomic energy and other government laboratories in New Mexico, Tennessee, South Carolina and Washington. This is a representative—but certainly not complete—list of the spectrographic laboratories using our semi-quantitative standards.

More surprising to us is the distribution among other countries of the standards. These include: Belgium, Canada, England, France, Germany, India, Israel, Japan, Mexico, the Netherlands, Northern Rhodesia, Sweden, Switzerland, Union of South Africa and Yugoslavia.

As for numbers, the table below shows how many of each kind of standard have been bought to date:

SPEX MIX (Base containing 43 elements at 1.34% .....	288
G STANDARDS (graphite base) .....	328
L STANDARDS (lithium carbonate base) .....	49
Z STANDARDS (zinc oxide base) .....	116

Spex Mix, of course, is popular because of its versatility. Do-it-yourself spectrographers have been enabled to set up their own standards for materials of immediate concern to their employers. In addition, we have reports of people using such materials as silica, aluminum oxide and sulfate, barium carbonate and germanium oxide as matrices in combination with Spex Mix.

Considering our own sets of already prepared semi-quantitative standards, the G or graphite-base have been the most popular. There are several reasons for this: Graphite is so pure that it is possible to extend the range of added elements down to 1 ppm; it is also a good buffer and reducing agent. The L standards are largely the result of the pioneering work of Weaver and Brittain. (*Anal. Chem.* 21, 1038-41, 1949) and are being used principally for the analysis of ashes of oil and other organic materials. Having their origin in the research of G. W. Standen (*Ind. Eng. Chem. Anal. Ed.*, 16, 675-80, 1944), the Z standards are used for both inorganic and organic materials. The built-in internal standard of zinc permits high semi-quantitative accuracy.

May we quote a prominent spectrographer who wrote an unsolicited letter to us a few weeks ago? He said, "Honestly, I think that your Spex Standards are the best item to be added to the field in some time." Credit actually belongs to the above-mentioned, and to a group under the direction of the late Dr. L. C. Gaddum who used standards similar to ours at the Florida Agricultural Experiment Station in the early 1940s.

# DOCUMENTATION OF MOLECULAR SPECTROSCOPY (DMS) New Punched Card System for Infrared Now Available

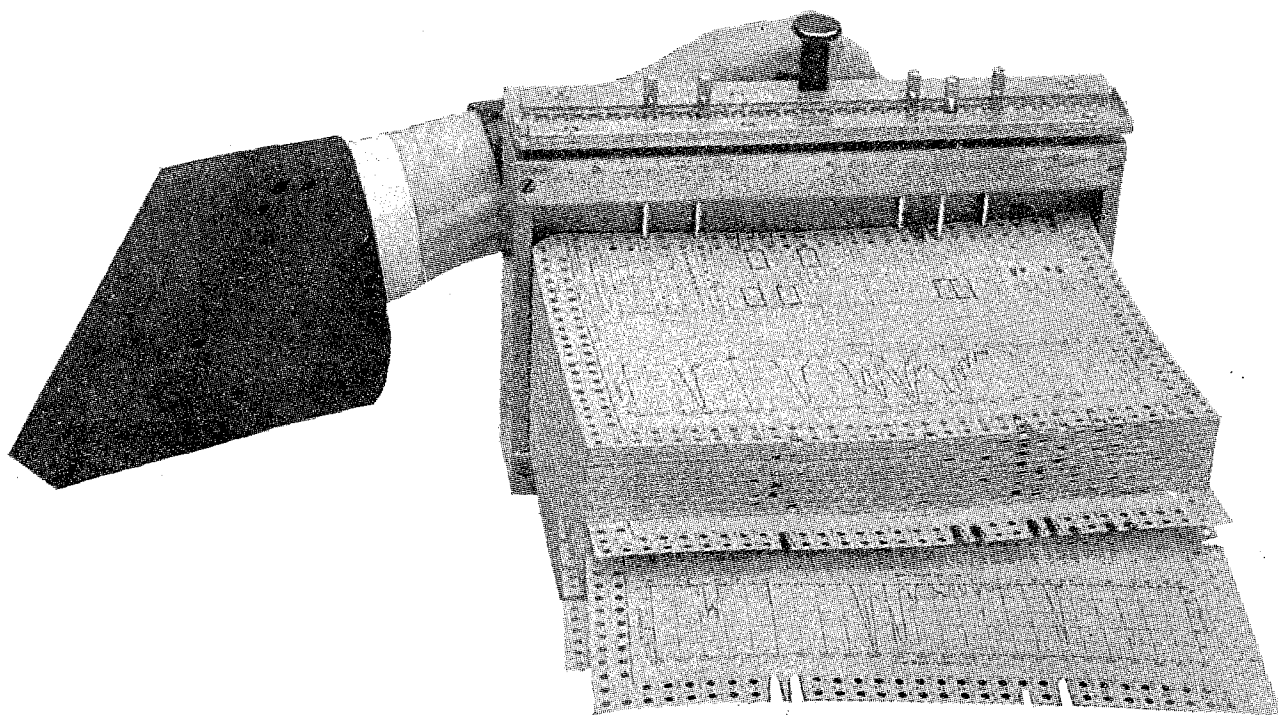
By DAVID N. KENDALL, Ph.D.,  
Infrared Consultant to Spex Industries, Inc.

Every practicing spectroscopist is aware of the necessity of having at hand the largest possible reliable collection of spectra and appropriate literature references. It is impractical for the individual laboratory to attempt to make such a collection. The best current answer to this problem is a punched card system, incorporating spectra submitted by laboratories around the world, monitored by distinguished scientists, and published in standard format on a dependable schedule.

Now available to absorption spectroscopists is the DMS key-sort card system comprising infrared and literature cards. The data are prepared by well-qualified experts working under the direction of a British and a German group, the former headed by Dr. H. W. Thompson, the latter by Dr. H. Kaiser. It is

Three types of punched cards are included in the DMS system:

1. *Literature cards, yellow*, giving abstracts of important articles on spectroscopic problems or containing useful spectra.
2. *Spectral cards* of two types:
  - (1) *Rose-colored cards* for the infrared spectra of organic compounds of definite constitution.
  - (2) *Rose-colored cards* for the infrared spectra of polymers and natural substances whose constitution has not yet been established, technical products and mixtures.



gathered from industrial and academic laboratories in Europe, the U.S.A., and Canada, and published in England by Butterworths Scientific Publications and in Germany by Verlag Chemie GMBH. Raman spectral cards are to be dove-tailed into the system at a later date.

Each spectral or literature card of the DMS system records the relevant data using a completely new classification of organic substances in a convenient coded form. The new classification encompasses all organic compounds, technical products, and mixtures—even natural substances and polymers whose constitution has not yet been established. A cross-reference between the literature and spectral cards is provided for those literature articles which also include spectra. Indexed information is given in clear text.

The DMS system is a service sold on an annual subscription basis. The subscriber receives 2,000 cards a year in batches of 500 at intervals of about three months. Current subscribers in this first year of operation have received 1,000 cards by February of 1957 and 2,000 cards by August, 1957.

“Blank” cards are also available to subscribers to record and code spectra which they themselves have measured. On these, only the basic framework and headings are printed, together with the skeleton structure and substituent keys.

The front side of a spectral card gives the name, main physical properties, and molecular and structural formulae of the compound together with the author or source of the spectrum, and the conditions under which the spectrum was measured. The reverse side of the card shows the spectrum, a table of frequencies of the chief bands and their approximate intensities. The infrared spectra are drawn on a standard format with abscissa linear in wave numbers, covering the range 200 to 4000  $\text{cm}^{-1}$  with a scale break at 2000  $\text{cm}^{-1}$ . The ordinate is percent transmittance. The card edges contain two rows of holes, with 203 holes in all. These can be cut so as to make deep, shallow, or “middle slots.” Details of the unique and practical coding system will not be elaborated here. Interested readers may consult the article by Professor H. W. Thompson, *J. Chem Soc.*, 1955, pp. 4501-4509.

Each subscriber to the DMS system is provided with a complete *Instruction and Coding Manual*. For users who prefer to employ their own code or make some modification to the present one, the cards can be ordered with the holes which should be slotted *marked* on all cards, but *not cut*. Most users, however, will probably want the slots cut out. A slotting service is available whereby subscriber's cards are slotted according to the DMS code.

The literature cards relate to spectra recorded on spectral cards, or to important developments of technique or theory. An abstract of the article, names of authors, year of publication, nature of the work, spectral range studied, state of aggregation of the sample, and details of the equipment, method, or theoretical aspects are all included. In addition to cross-reference to the spectra cards, it is possible to select from the literature deck a card or cards dealing with a specific topic of interest.

The principal advantages of the DMS system to spectroscopists are:

1. It provides an ever-expanding collection of carefully selected spectra gathered from industrial and academic

laboratories in the U.S.A., Europe, and Canada, including many previously unpublished and unobtainable.

2. It is a keysort card system. Individually named, numbered, and indexed, each card contains a wealth of useful data. The hand sorting apparatus required is inexpensive.
3. The DMS System offers a complete classification of organic substances in a convenient coded form, enabling all organic compounds to be included. Inorganic compounds are also to be included in the future.
4. Literature references are at your fingertips.
5. It is very practical for the small laboratory possessing no sorting machine as well as for the large laboratory where workers in special fields will usually retain numbers of cards in defined groups, thus simplifying the sorting operations.
6. ASTM is now indexing the DMS System on IBM cards. About 300 are now completed and about 1800 will be available within the year.

**Note:** A duty of 11½% will be paid by the Subscriber directly to the U. S. Post Office upon arrival of the cards. They are classified under Par. 1410 by U. S. Customs.

## PRICE LIST

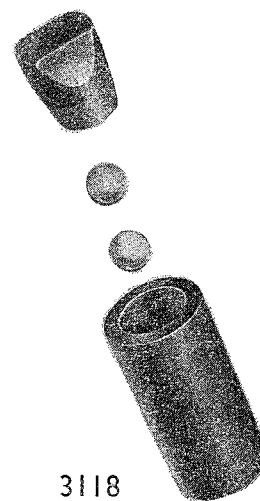
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