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VACUUM SPECTROSCOPY*

II. APPLICATIONS

Challenge of this Region

In Part I of this paper, we discussed the history of vacuum spectroscopy and described some of the current instrumentation used in this region of the spectrum between about 50 and 2000Å. The present paper deals mainly with applications, showing why there is the intense interest in the region today.

Why to the Air Force is revealed by P. H. Wyckoff, Chief, Atmospheric Physics Laboratory of the Air Force, Cambridge Research Center. In the Foreword to a paper (1), Wyckoff states, "At altitudes of 50 miles or higher, we enter into an entirely new realm of the atmosphere which many prominent scientists like to call the chemosphere. It is a region of intense ultraviolet radiation from the sun, and because of this huge influx of energy, it is a region of chemical change and radical reactions. Constituents of our atmosphere, which we are accustomed to consider as stable near the earth's surface, become readily dissociated and recombine to form compounds with vastly different properties. Some of these gases are deadly poisons, some of these have violent effects upon the combustion processes of reciprocating and jet engines, and others are corrosive to metal and plastic components of an aircraft. . . . In order to study these phenomena at short range [our] approach has been to synthesize these conditions in the laboratory through the use of known mixtures of pure gases that are irradiated by [far] ultraviolet light."

The same radiation that is filtered out by the upper atmosphere is responsible for the reactions occurring there. Photodissociation and photoionization take place in the ionosphere, a band circling the earth 25 to 250 miles high. Here hardly any molecular nitrogen or oxygen exists. Normal atmosphere gases are torn apart by the intense Lyman alpha line radiation at 1216Å, by x-rays and cosmic rays to produce compounds and ions rare at the surface of the earth.

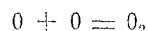
Spectroscopists have learned how to create these high altitude compounds in their low altitude laboratories by subjecting atmospheric gases to various radiation sources. Characteristic absorption spectra in the far ultraviolet are then obtained to help unlock the intriguing mysteries of space.

* Presented as an invited paper to the Canadian Association for Applied Spectroscopy, Montreal Section, at McGill University, on Nov. 19, 1957.

To conventional spectroscopists, such studies may appear deceptively simple. First let us dispel that illusion. Working in the far ultraviolet is much more difficult than working in other regions. Unlike elsewhere, there is but one source of "white" light covering the far ultraviolet. This is the Lyman source which erodes its electrodes rapidly and becomes unstable. The familiar hydrogen continuum becomes many-lined below 1650Å and the rare gases exhibit continua over only narrow spectral ranges. Temperamental in other regions, photographic emulsions are even more so below 2000Å and are subject to scratching, distortion and variable sensitivity. Recently, a photoelectric detector was discovered which offers much promise. It is a sodium salicylate covered multiplier phototube. A phosphor, sodium salicylate was found to have the property of constant quantum efficiency over a wide range of the far ultraviolet. That is to say, its electrical output is identical whether it is irradiated with the same amount of energy at 2000Å or 1000Å.

While on the subject of the difficulties facing the vacuum spectrographer, it would be well to remind those of us who may be used to running dozens of samples a day, that our patience might be strained in the vacuum region. Changing samples, loading and unloading film—speedily done in other regions—are all time-consuming operations in far ultra-violet work.

Despite the obstacles, a good deal of practical information is currently being obtained. Reaction kinetics may seem to be a highly theoretical subject, remote from any practical implications. Just the opposite is true, however. Consider the problem of reentry of missiles into our atmosphere. Here the nose of the missile is subjected to heat of such intensity that most solids would instantly vaporize. Part of the heat is caused through the triggering by the missile of the reaction



which releases a good deal of energy. Preventing or inhibiting this reaction is thus of immediate military concern.

A unique method of studying the reaction has been devised at the General Electric Missiles & Ordnance Department in Philadelphia by a group under the direction of Dr. L. A. Myerson. The first step is to dissociate molecular oxygen into atomic oxygen, the reverse of the above reaction.

This may be accomplished in an RF field or through flash photolysis, using a flash of light around 1750Å. This wavelength is equivalent to the 8 eV required for dissociation.

Having dissociated, the oxygen atoms recombine spontaneously. To measure the recombination rate, Dr. Myerson is trying three approaches: 1) studying the intensity changes with time of the molecular oxygen continuum around 1750Å; 2) measuring the intensity change of the atomic oxygen line at 1302Å; 3) through flash spectroscopy where photographs of the spectrum are taken at different delay intervals after a flash dissociates the molecule. The last technique will be discussed at greater length later.

Free Radicals and Mechanism of Organic Reactions

The "military" is thus interested in vacuum spectroscopy in connection with upper atmosphere research programs. Others have more down-to-earth interests. One of the greatest single areas where vacuum spectroscopy is being put to use is in free-radical research. So important is this field considered that Dr. Herbert Broida was recently placed in charge of an exhaustive three-year research study at the National Bureau of Standards in which scientists throughout the world have been invited to participate. A conference on free radicals was held in May in Canada under the auspices of the National Research Council. Another took place last Fall at the NBS in Washington.

Although free radicals were discovered in 1900 by Moses Gomberg, knowledge concerning their eccentricities has lagged. Free radicals may be defined as molecules in which one of the atoms has one of its valences unoccupied. They thus exhibit a valence one less than normal, e.g., carbon appears trivalent rather than quadrivalent. The missing electron means that a free radical has an *odd* number of electrons, giving rise to, among other phenomena, characteristic absorption bands. The more stable and complex free radicals have been studied through their absorption in the near ultraviolet. The simplest, least stable radicals absorb only in the vacuum ultraviolet where, because of technical difficulties, very limited studies have been conducted.

Most free radicals are extremely reactive, another fact which has discouraged their study. Explosions and flames are examples of where such reactions take place. Methods of observing the antics of free radicals have, nevertheless, been devised over the years. An ingenious one was worked out by Paneth and Hofedetz in 1929 (2). In an evacuated tube, they decomposed tetramethyl-lead by heat to form the methyl free radical. At some distance along the tube was placed a lead mirror. Any free methyl reaching the lead mirror would tarnish it. Thus, at close distances to the source of tetramethyl lead, methyl free radicals would react with the lead mirror. Increasing the distance, a point would be reached where no free radicals would have survived the journey and the lead mirror would remain shiny. Mean free path calculations gave these pioneers data on the half-lives of the methyl free radical. A method had at last been found for making such measurements.

All free radicals are not, however, so easy to track down, a fact that hampered research in this field. Later, other techniques were devised, a notable one being the use of trapping the free radicals as they are produced on a surface cooled by liquid air. Here their life expectancy is improved considerably.

Today the state of the art of free radical study is such that most work is still in basic research. Since absorption is theoretically known to occur in the far ultraviolet, the best new approach at detection makes use of vacuum ultraviolet spectroscopy. Dr. G. B. Herzberg and his colleagues at the

National Research Council in Ottawa have worked out a brilliant scheme for producing and then running down the free radicals using flash photolysis and absorption in the far ultraviolet. First they form the free radicals with a flash lamp of extremely high intensity. Once produced, the free radicals stand ready for detection for about a millisecond before they vanish. In this method, a Lyman source is switched on a fraction of a millisecond after the initial flash occurs. Placed in a cell between this source and the spectrographic slit, the material absorbs radiation from the Lyman source, producing characteristic spectra of the free radicals formed by the flash source. This cycle, causing the spectra to be recorded only when the Lyman source is on, is repeated as often as required to produce good spectrograms of the material under observation.

Going one step further, Dr. L. S. Nelson of the Bell Telephone Laboratories, has designed a flash lamp using a sapphire envelope (3, 4) which permits irradiation down to about 1450Å. In this region, a great deal more energy is available for producing free radicals. (See Fig. 1).

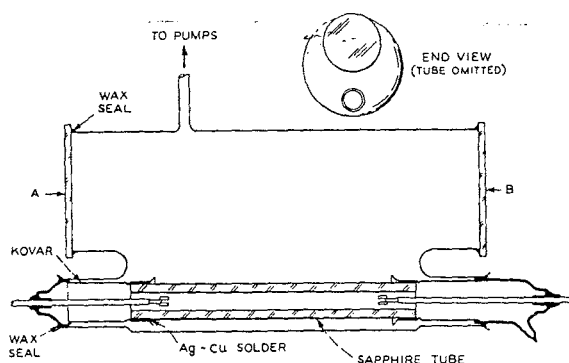


Fig. 1. Absorption cell used for flash photolysis experiments with a sapphire flash lamp (4).

We have stated that most of the current work on free radicals is basic. Unlike many basic research adventures, however, the rewards here can almost be predicted. For instance, a better understanding of free radical reactions is virtually certain to spark the development of many superior reactions. Today, so many plant processes depend on free radical reactions that the potentialities here are vast.

Other possible fruits of free-radical study are exotic fuels which will utilize the reaction heats of free radicals. No longer confined to science fiction pages are suggestions that aircraft fuels of the future may be obtained from the very atmosphere through which the aircraft travels. We hear of "photonic" energy in which the energy is derived from light. Such energy is, of course, being used already by telephone companies for powering rural transmission lines. Another, even more exotic fuel, is ordinarily locked in unreacted atoms in our ionosphere. Thus the reaction mentioned earlier in which two free radicals of oxygen combine to form molecular oxygen offers energy for the taking. Will it be possible some day to utilize as a fuel the very same energy which destroys most rockets and meteors as they enter our atmosphere from outer space?

Other Research Problems

One major research area has already been mentioned: the study of free radicals. Other types of problems are enumerated below:

1. Determination of absorption coefficient.
2. Photoelectric studies of metals.
3. Photoionization of gases.

4. Determination of ionization and excitation potentials and dissociation energies.
5. Excitation spectrum of phosphors.

A good deal of the work in these areas is highly theoretical. Results are advancing our knowledge of the nature of atoms and molecules. But, as so often happens, practical results often emerge unexpectedly. The discovery that sodium salicylate has a constant quantum efficiency is a good example of this. It stemmed from a study of the excitation spectrum of many phosphors.

The work on excitation potentials and dissociation energies has many ultimate goals. A better understanding of the mechanism of flames and explosions, for instance, may pave the way to more efficient fuels for jet and rocket aircraft.

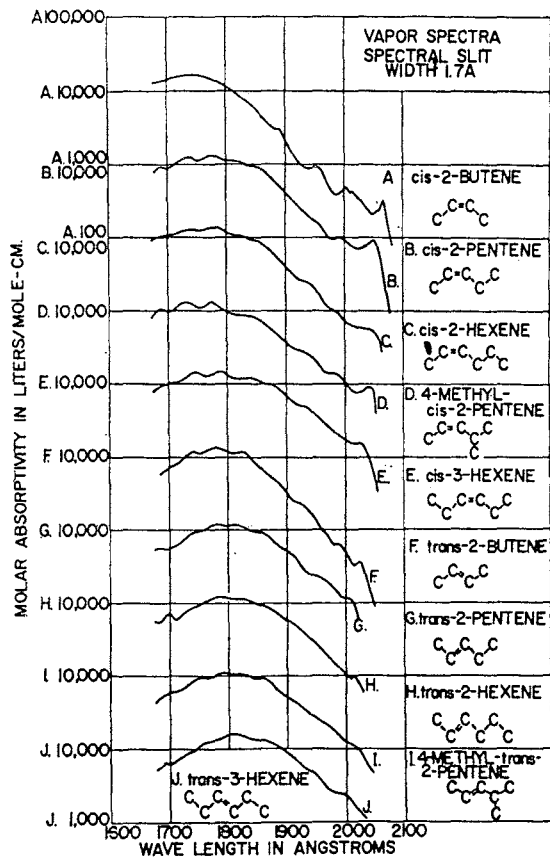


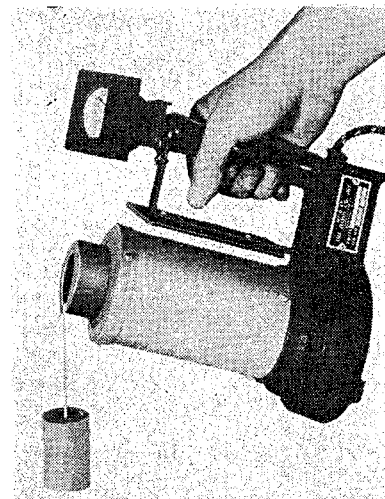
Fig. 2. Far ultraviolet spectra of vapors of C_4 to C_6 1, 2-dialkylethylenes (5).

An Analytical Tool

A paper published in 1955 (5) is about the most complete account of the absorption applications of far ultraviolet radiation as a tool in analytical chemistry. It is by Jones & Taylor of the Wood River (Illinois) Research Labs of the Shell Oil Company. Among other things, these chemists found that, in this region, the spectra of unsaturated hydrocarbons consist of broad, almost structureless bands. The spectra of members of a particular series were found to be so similar that this region may be used to determine the total concentration of this hydrocarbon series. On this basis, the authors indicate the possibilities of running a total olefin determination. Vacuum ultraviolet analysis thus offers a tool to complement more specific measurements on saturated hydrocarbons in the ultraviolet, visible and infrared. On the other hand, Jones and Taylor found that, with such substances as conjugated diolefins,

(continued on page 4)

JELRUS
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FURNACE



ULTRA-PURITY CRUCIBLE NOW AVAILABLE WITH "HANDY-MELT" FURNACE

A furnace beautifully suited to the needs of spectrochemists is the Jelrus "Handy-Melt" pictured above. Standards and samples may be prepared with it in but minutes after it is plugged into the nearest electrical outlet. The melting crucible becomes the pouring ladle and a built-in thermocouple permits the operator to pour all samples and standards at identical temperatures. This, of course, is essential in minimizing segregation and matrix errors.

Until now, the furnace had one shortcoming. Its crucible was not made of spectrographic grade graphite. This resulted in contamination from trace elements present in the crucible. Now, at a nominal cost, we are making available a graphite crucible manufactured by United Carbon Products and purified after machining to their rigorous ultra-purity standards.

The furnace has a maximum temperature of 2000°F (1093°C), permitting melting of most non-ferrous metals. A safety control panel is available as an accessory. With it, the muffle life may be prolonged and the thermocouple and meter are protected. Up to 1½ pounds of brass, 8 ounces of aluminum or 25 Troy ounces of silver may be melted. The actual volumetric capacity of the muffle is about 90 ml.

7100	Handy-Melt Furnace, 1250 watts, 115 volts ac or dc; complete with ordinary graphite crucible but without Control Panel	Each	\$97.50
7101	Handy-Melt Furnace, same as above for 230 volts ac or dc	Each	\$100.00
7102	Control Panel for 7100; protects furnace by maintaining temperature below 1500°F.	Each	\$22.50
7103	Control Panel for 7101; same as above for 230 volts	Each	\$22.50
7104	Crucible, graphite, ordinary grade (one furnished with 7100 or 7101)	Each	\$2.65
7105	Crucible, graphite, ultra-purity, spectrographic grade	Each	\$15.00
7106	Muffle, spare, 115 volts	Each	\$17.00
7107	Muffle, spare, 230 volts	Each	\$18.00

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individual members of the series did give rise to characteristic spectra which could be used for specific identification. Fig. 2 and Fig. 3 show typical results.

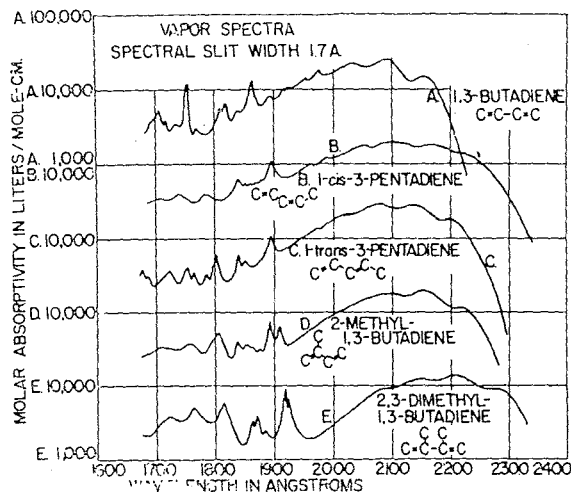


Fig. 3. Far ultraviolet spectra of C_4 to C_6 conjugated diolefins (5).

In emission, very little has, as yet, been done in the vacuum region. Spectra of many elements have been studied and reported in the region below 2000A, but the region has been put to little practical use. This, despite the fact that several elements have their strongest lines here. I think the answer to this little riddle lies in the very nature of progress. Twenty years ago, the spectrographer was content with buying a basic spectrograph and setting up his own auxiliary equipment. Now, he is used to buying a complete instrument in which the sample goes in one end and the answer comes out the other. Vacuum spectroscopy has a long way to go to reach this landmark.

But first let us question once more the significance of the vacuum ultraviolet, especially from the standpoint of why certain data must be obtained here and cannot be obtained in other spectral regions. The excitation potential of an atom is defined as the energy required to elevate an electron from one orbital level to another. This is usually expressed in electron volts. Quantum mechanics has shown that 1 to 100 ev corresponds to the region from 12,500 to 125A—the near infrared to the far ultraviolet.

The excitation potential of many atoms lies above 6.3 ev or below 2000A. Thus the most sensitive lines of many of the non-metals, the metalloids, the halogens and the gaseous elements, usually require the vacuum ultraviolet region.

An example of the usefulness of this region dates back to 1936 when G. R. Harrison, successfully hurdling the high-vacuum obstacles, showed that selenium and sulfur could be determined in both nickel and steel.

But it remained for the application of photoelectric detection to put these determinations on a routine basis. Applied Research Laboratories recently introduced the Quantovac, a variation of the more familiar Quantometer, designed for photoelectric measurements mainly in the region 1600-2000A. The Quantovac may be flushed with dry nitrogen instead of being evacuated so that the high-vacuum problems are avoided. Dry nitrogen is transparent above 1600A. Table 1 gives the lines that are normally used for steel analysis.

Table 1.

QUANTOVAC (Applied Research Laboratories)

Standard arrangement of multiplier phototubes:

1657A	Carbon	2714A	Iron
1783	Phosphorus	2933	Manganese
1820	Sulfur	3082	Aluminum
1890	Arsenic	3110	Vanadium
2316	Nickel	3133	Molybdenum
2516	Silicon	3175	Tin
2677	Chromium	3242	Titanium
		3274A	Copper

The Mond Nickel Co. Development and Research Laboratory in Birmingham, England has made another compromise with the vacuum problem. Here a permanently evacuated spectrograph is used with the arc-spark stand alone flushed with nitrogen. With this arrangement, only a very short nitrogen path is introduced. K. M. Bills of the Mond Nickel Company reports that, using this instrument, 40 samples per day of cast iron may be analyzed for sulfur and phosphorus. Photoelectric integration results in a limit of detection of 0.002% for sulfur and 0.005% for phosphorus. At levels of around 0.02% concentration, a precision of better than 10% of the amounts present is obtained.

Hilger recently announced its Fluorite Vacuum Polychromator, also called the CPS Evaluator. It is an instrument functionally similar to the Quantovac, but based on the design of Bills. With it, Hilger claims satisfactory accuracy for determinations of carbon down to 0.003%, phosphorus to 0.005% and sulfur to 0.003% in routine analyses of steels. Typical precision values are given in Table 2. The time required for determining all three elements in a particular steel sample is given as under two minutes, a factor of at least ten better than conventional wet chemical methods.

Table 2.

Fluorite Polychromator
also called
CPS Evaluator
(Hilger)

Element	Spectral Line, A	Conc. Range, %	Std. Dev., %
Carbon	1657	0.01-1.7	2.5
Phosphorus	1783	0.005-0.1	10
Sulfur	1820	0.005-0.06	6

Because of the increasing emphasis on the role of gases in metals, vacuum spectroscopy is certain to blossom. As instruments and techniques of analysis become available, we will see these elements gradually added to chemical specifications. A further prod to such progress is the development of vacuum melting and casting techniques. Free of entrapped alloyed gases, metals so produced have many improved properties in ductility, strength, porosity, corrosion resistance, etc. As is so often the case, pressure from the end-product will result in pressure on developing techniques for controlling the quality of that end-product. Vacuum metallurgy, in other words, is greatly encouraging the development of vacuum spectroscopy.

Instrumental Progress

Thus, a tremendous amount of work awaits the researchers who are going to probe the vacuum ultraviolet in the years ahead. To repeat what was inferred earlier, many more researchers would be eager to tackle these problems were the instrumentation more advanced. May I recap the engineering difficulties perhaps to whet the appetite of some of the enterprising spectrographers who may want to contribute their welcome ideas.

1. *The Source*—In absorption, the ideal source is a continuum of uniform intensity with wavelength in the region of interest. For the vacuum ultraviolet, no such constant source has yet been discovered. (The Lyman source leaves much to be desired.) Is it possible by employing the correct electrodes, the correct excitation and the correct atmosphere to invent such a source? If not, is it practical to devise a combination of sources so that each will cover a particular region with a short overlap into the next? It goes without saying that the source or sources must be long-lived and foolproof, steady, without hazard and inexpensive.

2. *Background*—One of the severe optical problems in the vacuum ultraviolet is scattered light. Those of us who are used to seeing crisp black lines on an otherwise clean plate are disappointed when we see our first grey-on-grey far ultraviolet spectrum. The background results from scattered light reflected from blackened surfaces and the grating itself. Actually, the trouble probably lies in the fact that aluminum (or sometimes platinum), which is used to coat the grating, reflects not much better than the baffles which are mounted in a vacuum spectrograph to prevent spurious reflections. Although aluminum is the best overall reflector known, its reflectance drops from about 80% at 2000Å to 25% at 1200Å. Needed is a coating which may be applied to gratings and mirrors which would make them highly reflecting in the far ultraviolet.

3. *Conveniences*—Lumped in this category are all the vacuum controls, switches, gauges, safety devices, valves and bleeds which are presently necessary to understand and cope with in order to obtain vacuum spectra. Any contribution which reduces the complexity of the controls or eases the mind of the operator will greatly help the art of vacuum spectroscopy. The operator must now be constantly on the alert lest a failure occur in the safety device protecting another safety device which, in turn, monitors the water flow to the diffusion pump.

4. *Electronics*—The improvements in related instruments are gradually overflowing into vacuum spectrographic instrumentation. Witness the Quantovac and the CPS Evaluator which use similar circuitry to that in other direct reading spectrographs made by ARL and Hilger.

In conclusion, let me describe a new instrument designed to overcome many of the problems mentioned earlier. It is the Model 240, 2-meter vacuum spectrograph manufactured by Paul M. McPherson of Acton, Massachusetts. It is pictured in Fig. 5. Designed to be as flexible as possible, this spectrograph may be used as a monochromator, a scanning monochromator, a photoelectric spectrometer or a spectrograph with a coverage from about 1000Å to 6800Å. To provide additional flexibility, gratings may be interchanged simply by lifting one out and replacing it. This is important where optimum speed or dispersion at a specific wavelength is required.

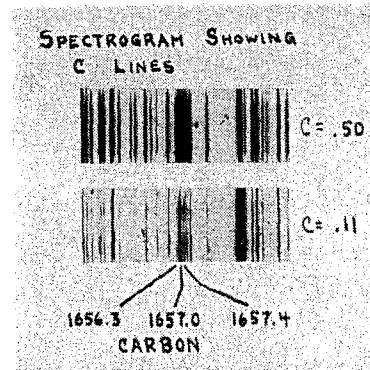


Fig. 4. Typical Spectrogram showing Carbon lines of Steel in the Vacuum Ultraviolet.

(Courtesy ARL)

In Fig. 5, note the long tube on the right which houses the entrance slit. Alongside the entrance slit is a compartment which accommodates either a camera or an exit slit. With the camera, the instrument becomes a spectrograph. On a 35-mm film strip 11 inches long, ten exposures may be taken. The camera is racked through a vacuum-tight linkage to the handwheel just below the entrance slit. Above the handwheel is a counter which indicates the film racking position in mm. To convert the instrument into a monochromator, the camera is replaced by an exit slit assembly. Two or more slits and multiplier phototubes may be used in order to read intensity ratios continuously.

As a scanning monochromator, a motor drives a screw at a preselected speed through a gear box. The screw, in turn, makes the necessary scanning motions and focusing adjustments smoothly. Wavelengths are easily interpolated from a second Veeder counter. Alternatively, hand scanning or rapid wavelength adjustment may be made manually by turning the inner handwheel shown in the photograph.

At the center of the Model 240 is the vacuum console. Switches and meters shown control and indicate the pressure obtained by the fore and diffusion pumps. Better than 2×10^{-5} mm of mercury may be obtained. Safety devices on the instrument include a thermal relay which turns the diffusion pump heater off should the water cooling fail. A special baffle at the throat of the diffusion pump may be refrigerated to condense out any possible fumes.

One neat convenience on the instrument permits rapid change of film or sample. Note the black knob on the upper right of the instrument. It actuates a flapper valve which seals off the film chamber. Air may thus be bled only into the latter in order to change film or adjust exit slits. Similarly, the entrance slit may be isolated from the main part of the instrument, permitting rapid change of samples for absorption measurements. After the film is changed or another sample is inserted, an auxiliary roughing pump is used to bring the pressure down in these chambers to a level where the flapper valves may once more be opened. A thermocouple gauge tells the operator when the valves may be opened.

Still another convenience is a mounting plate at the entrance slit designed to take the source and samples through an O-ring seal. It is possible to isolate gas samples from the main body of the instrument by using a window in front of

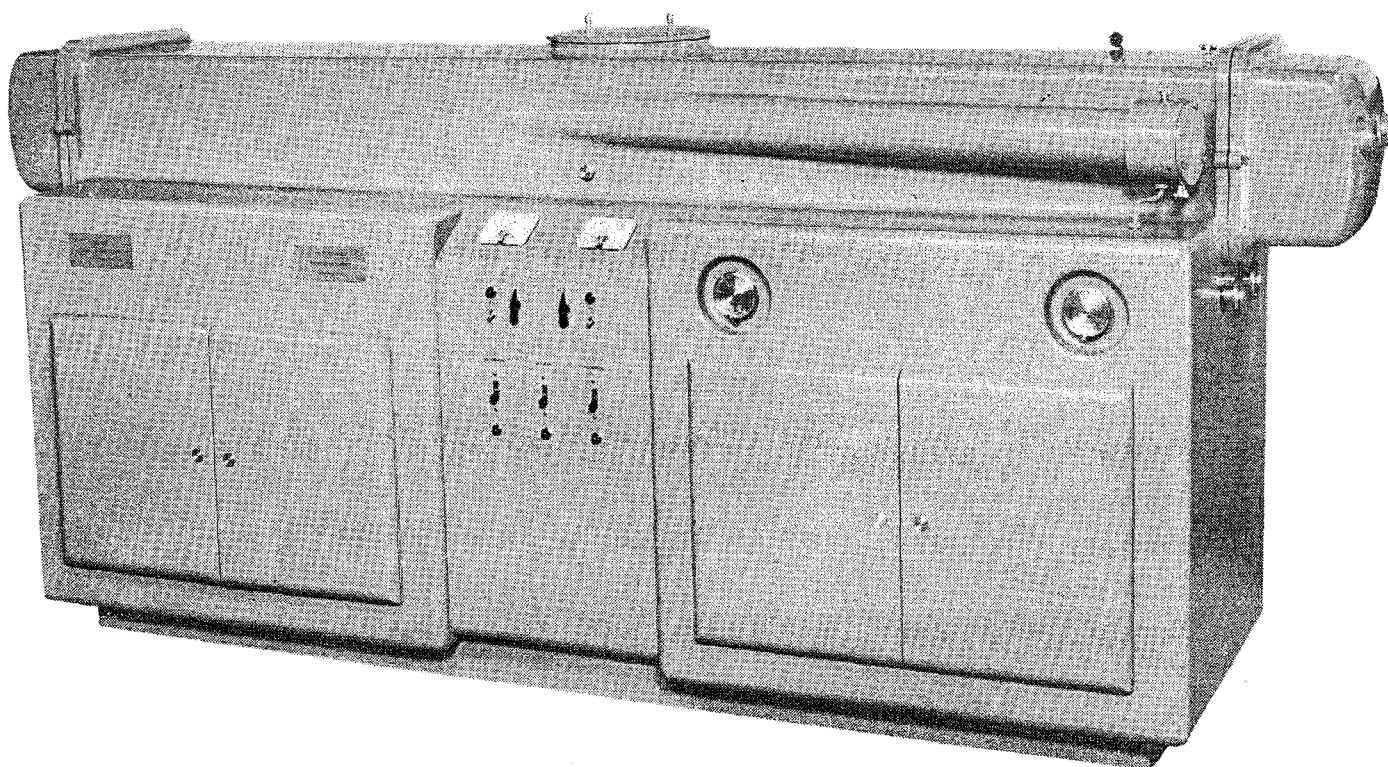


Fig. 5. Model 240 Vacuum Spectrograph

the entrance slit. Also possible is the use of differential pumping whereby no window is used. In this technique, the gas is fed at a low pressure into a cell and then removed rapidly by vacuum pumps. Any one of four entrance slits of different widths may be placed in the optical path while the instrument is operating under full vacuum.

The wavelength range of the McPherson 240 is about 1000-6800Å. The lower limit is set by the reflectivity of gratings when used at normal incidence. The upper limit, well up in the visible, permits the use of the instrument here. Equally important, it allows the operator to make many adjustments visually—another time saver when he is working in the invisible ultraviolet.

To summarize, the McPherson 240 makes it a lot more convenient than with previous instruments to perform research and development in the far ultraviolet. The next step is up to the researchers themselves.

—A. J. MITTELDORF.

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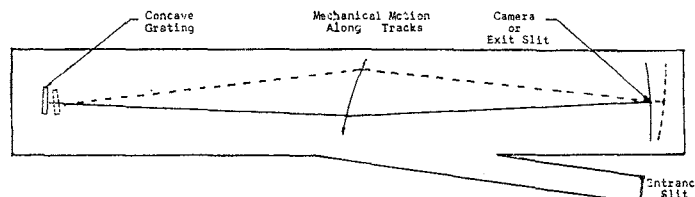


Fig. 6. The optical lay-out of the McPherson Model 240. The solid lines show the position of the components when the wavelength is set at one extreme; the dotted lines, at the other extreme. At any one setting, the Rowland circle may be drawn from the center (on the track) through the entrance slit, the grating and the camera or exit slit.

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In the short time since we were appointed exclusive United States representative for the Documentation of Molecular Spectroscopy (DMS), a considerable number of companies and institutions have entered subscriptions to this excellent punched card system of cataloging organic compounds through their infrared spectra. Subscribers approve the systematic collection of spectral and literature data from industrial and academic laboratories in Europe, the U.S.A., and Canada and the significant fact that all data are carefully monitored for correctness by a well-qualified group working under the direction of Dr. H. W. Thompson in England and Dr. H. Kaiser in Germany.

Each subscriber receives 2000 cards a year, in batches of 500 quarterly. With each batch of cards, a DMS Newsletter is also issued. This newsletter corrects mistakes that have been found, gives useful hints on sorting and on the use of the system such as presenting a general method of presorting the cards into convenient groups. It also discusses the coding of difficult compounds such as complex sugars, heterocyclic compounds, sulfoximines, etc. Whenever mistakes are uncovered for which correction cannot be made by simple slotting changes in a card, a replacement card is issued.

All of us concerned with the DMS System are always anxious to improve the service. In this regard, we solicit suggestions

from subscribers and non-subscribers alike. As an example of a recent suggestion, all future spectrum cards will include the structural diagram of the compound on *both* sides of the card.

A.S.T.M. is issuing "Wyandotte" cards corresponding to the spectral cards of the DMS System. Those laboratories with I.B.M. machines will therefore be able to sort their DMS cards mechanically by the use of auxiliary Wyandotte cards. A numerical list of DMS cards is being published to provide rapid cross-reference between the Wyandotte and DMS cards.

"Methods for Emission Spectrochemical Analysis" Second Edition published

Committee E-2 of the American Society for Testing Materials has recently published the second edition of the book which has become the bible of practicing spectrochemists. Among the additions in the new edition are methods for the analysis of stainless steels, refractory materials, slags, petroleum products and other miscellaneous nonmetallic materials. A section contributed by Committee D-2 is devoted to the analysis of Diesel locomotive lubricating oils. Another important addition is entitled "Suggested Practice for Describing and Specifying the Excitation Source." Many methods have been upgraded from Suggested to Tentative or Recommended. The book is available from ASTM, 1916 Race St., Philadelphia 3, Pa. It is priced at \$5.25 to members and \$7.00 to non-members.

QUAL-MIX

A NEW PREPARATION

FOR

SIMPLIFYING QUALITATIVE SPECTROCHEMICAL ANALYSIS

At the SAS Annual Meeting last November, we announced a new standard for qualitative analysis. Although we said that the atlas or master charts for the material would not be available until around March, 1958, we were pressed with requests for the QUAL MIX itself. Accordingly, we are making it known that QUAL MIX is now available for immediate shipment.

QUAL MIX contains 43 elements so blended that about three lines of each element will appear on the spectrographic plate in the region 2100 to 4700A. The elements are the same as are present in our semi-quantitative standards, ELEMENT KIT and SPEX MIX. A consistent scheme of qualitative, semi-quantitative and quantitative analysis is thus achieved. Among the criteria we aimed for in the mixture are the following:

1. Lines to be as sharp as possible without reversal.
2. Ordinary dc arc and almost any electrode to be employed.
3. No sputtering of the material during burning.

4. A definite end-point to indicate when the burning is complete.
5. Matrix is to be non-toxic with non-interfering lines.
6. A blend balanced for modern grating spectrographs especially. For example, the region 3500-4700A to be fully utilized.
7. Background to be kept to a minimum; masking of lines by cyanogen and general incandescence to be minimized.

Our tests were both on a large grating instrument and a Littrow. How well we met the criteria will have to be judged by the user himself. The matrix of QUAL MIX is a mixture of potassium carbonate and potassium phosphate. This combination, the result of innumerable trials of various other promising compounds, seems best to satisfy the above criteria.

The atlas which is in preparation will consist of a series of prints showing typical burnings of QUAL MIX alongside dc arc and spark burnings of iron. Lines of each of the elements will be identified together with their wavelengths. A wavelength scale will be given with marks every 10A.

1020	QUAL MIX, preparation to facilitate qualitative spectrochemical analysis for 43 elements per 2 grams	\$20.00
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Because of the demand, the Bureau of Analysed Samples originally released the set of seven Mild Steel Residual Series of standards before final certificates were issued for several of the elements. At this date, although 10 of the elements are still given with but approximate figures, a number of new tentative values have been assigned. We are publishing these

as a guide both to the many users of the standards and to all other spectrographers and wet chemists interested in them. Just as soon as we have the final values, they, too, will be made available.

- 1210 **Steel Standards, Mild Residual Series**, set of seven rods, 3/4" dia. x 3" long \$120.00
- 1211 **Steel Standards, Mild Residual Series**, set of seven, turnings, 100 grams each \$130.00

TEMPORARY CERTIFICATE OF ANALYSIS

Sample No.	Ni	Cr	Mo	Cu	W	Co	Sn	V	B
S.S.11 (B.C.S. 271)	0.01-	0.04 ₅	0.19-	0.01-	0.01 ₅	0.01 ₅	0.11-	<0.005	0.013-
S.S.12 (B.C.S. 272)	0.03 ₅	0.02 ₅	0.17-	0.01 ₅	<0.01-	0.25-	0.02-	<0.01-	0.003 ₅
S.S.13 (B.C.S. 273)	0.03-	0.07 ₅	0.04 ₅	0.16 ₅	0.28-	0.02-	0.06 ₅	0.04 ₅	0.002-
S.S.14 (B.C.S. 274)	0.13-	0.18 ₅	0.07-	0.04-	0.04-	0.19-	0.02-	0.01 ₅	0.008-
S.S.15 (B.C.S. 275)	0.08-	0.16 ₅	0.09 ₅	0.21-	0.05-	0.06-	0.04-	0.05-	0.001 ₅
S.S.16 (B.C.S. 276)	0.18-	0.10-	>0.01-	0.12 ₅	0.20 ₅	0.02 ₅	0.01 ₅	0.05 ₅	0.004 ₅
S.S.17 (B.C.S. 277)	0.23 ₅	0.03-	0.01 ₅	0.10-	0.12 ₅	0.12 ₅	>0.005	0.10 ₅	>0.001-

Approximate content of elements NOT STANDARDIZED

Sample No.	C	Si	P	Mn	Ti	Al	Zr	Ta	Nb	Pb
S.S. 11 (B.C.S. 271)	0.22	0.30	0.015	0.47	0.00 ₅	0.01 ₀	0.04 ₅	0.008	0.110	0.002 ₅
S.S. 12 (B.C.S. 272)	0.18	0.35	0.019	0.48	0.01 ₅	0.06 ₅	0.03 ₀	0.001 ₅	0.005 ₅	0.003 ₀
S.S. 13 (B.C.S. 273)	0.14	0.19	0.018	0.48	0.07 ₀	0.06 ₀	trace	0.020	0.0003	0.002 ₀
S.S. 14 (B.C.S. 274)	0.18	0.32	0.020	0.51	0.09 ₅	0.03 ₅	0.00 ₅	0.006 ₅	0.050	0.007 ₅
S.S. 15 (B.C.S. 275)	0.16	0.33	0.019	0.49	0.03 ₅	0.02 ₀	0.01 ₅	0.003	0.035	0.005 ₀
S.S. 16 (B.C.S. 276)	0.23	0.19	0.016	0.48	0.00 ₅	0.02 ₅	0.00 ₅	0.006 ₅	0.055	0.006 ₅
S.S. 17 (B.C.S. 277)	0.16	0.37	0.021	0.50	0.03 ₅	0.01 ₅	0.04 ₀	0.0006	0.011	0.006 ₅