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A NEW SPECTRAL BAND

Blustery fall days have a way of driving the wanderlust from our fancies just as spring had planted it there. At this time we think of "getting together" and look forward to the social and technical meetings which are so much part of our life. This year the prospect of a national society of spectrographers is particularly stimulating. Spectroscopy seems to be grown up at last, soon to take its place alongside the American Chemical Society, the American Institute of Physics and other established scientific groups.

Why do we birds want to flock together? In 82 years the ACS has grown from a handful of planners to a point where it just busted the seams of its Washington headquarters. Why? Because 17,000 members benefit from its many services.

Likewise, spectrographers stand to benefit as soon as their local groups team up. APPLIED SPECTROSCOPY, the journal now published by the New York society, will surely gain stature as the publication of a national association. It will attract greater prestige and recognition from the public, advertisers, the press and fellow scientists. We shall all find a broader market for the exchange of ideas and speakers and thereby help strengthen infant local groups. Our publication needs the backing of a national society to improve its own position and that of spectrographers themselves.

With the advance of knowledge in the spectroscopic sciences being our constant goal, each step that promotes a closer bond among members is an accomplishment to be valued. Numerous annual meetings, held throughout the United States and Canada are difficult to organize and have become increasingly difficult for all of us to attend. Proposed are two annual meetings: The Pittsburgh Conference in spring and another in fall elsewhere by rotation. This will certainly allow greater attendance at fewer but better meetings and should be a feature strongly favored by management.

At these crossroads, where the profession of spectroscopy can either push forward or merely plod along, we feel that every effort should be expended to see the former become a reality. We salute our many friends who have worked so hard to build the national society. Towards that end we pledge our cooperation and encourage your support of the proposed constitution.

MEETING NOTICES

Seventh Annual Spectroscopy Seminar. Sponsored by the Department of Mechanical Engineering, University of Florida, Gainesville, Fla. Jan. 8-10, 1959. Further information may be obtained from Prof. W. T. Tiffin at the University of Florida.

Tenth Annual Analytical Conference. Jointly sponsored by the Spectroscopy Society of Pittsburgh and the American Chemical Society, March 2-6, 1959, Penn-Sheraton Hotel. Contact Dr. Fritz Will III, Alcoa Research Laboratories, New Kensington, Pa., for further information.

Seventh Meeting on Mass Spectroscopy. ASTM Committee E-14, May 17, 1959, Hotel Statler, Los Angeles, Calif. Contact A. G. Sharkey, U. S. Bureau of Mines, 4800 Forbes St., Pittsburgh, Pa.

Tenth Annual Symposium on Spectroscopy of the American Association of Spectrographers, Conrad Hilton Hotel, Chicago, Ill. June 1-4, 1959. Contact H. M. Wilson, Continental Can Company, Inc., 7622 S. Racine Avenue, Chicago 20, Ill., for submission of papers.

LOOMIS 20-TON HYDRAULIC PRESS

The pressed pellet technique is used extensively in three branches of applied spectroscopy—optical emission, x-ray and infrared. For all three, the sample in powder form is compacted to a uniformly dense button having smooth, parallel faces. To prepare such buttons, not only is a sturdy, convenient-to-operate press required but the instrument must be designed so that the platen moves exactly parallel to the head. This feature insures reproducible pellets and also proper alignment of the hardened dies which might otherwise be damaged.

EMISSION—

A typical example of the use of the press in emission spectroscopy is the ASTM method for the analysis of alkaline earth titanates (E-2 SM 10-3). Here a 1/2" diameter pellet is formed at a pressure of 80,000 psi (15,700 pounds) after the sample is mixed with lithium carbonate. Another example is the recently published method by the Alcoa Research Laboratories on a universal quantitative method of analysis (*Anal. Chem.* 30: 494, 1958 by W. H. Tingle and C. K. Matochka). Here the sample —325 mesh powder is pelletized in a 1/2" die at 44,000 psi (8,600 pounds) after it is mixed with graphite powder to make it conductive.

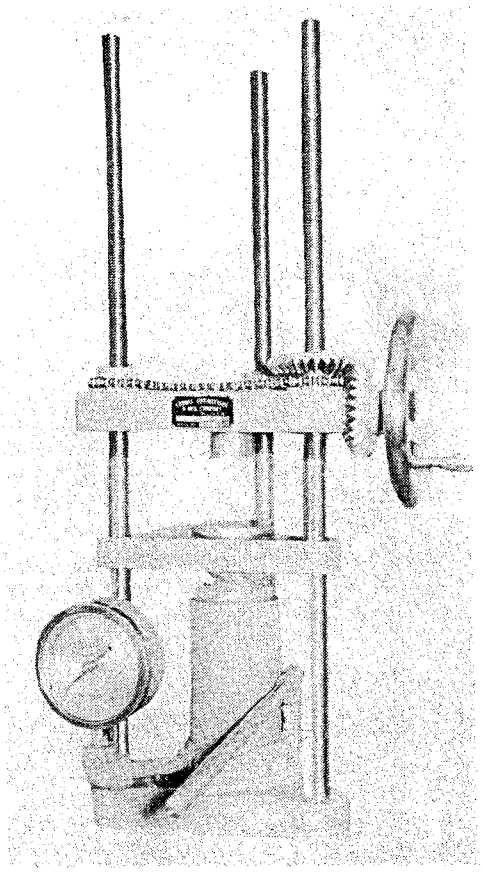
X-RAY—

For x-ray fluorescence, the standard disc is 1-1/4" in diameter. The 20-ton capacity of the Loomis Press permits an actual pressure of 32,800 pounds on this size disc. Adler and Axelrod (Norelco *Reporter* III, 65, 1956) describe a method using such pellets pressed at 20,000-30,000 psi for the x-ray analysis of rocks and minerals. Pellets are especially valuable for the determination of light elements, eg., silicon, calcium, magnesium, sulfur and phosphorus by x-ray techniques. Since the radiation of these elements is completely absorbed by layers above the particles, only those on the surface are effectively sampled by the x-ray beam. Thus by compacting the powder at 30,000 psi, the surface area is increased and sensitivity of the light elements is improved by as much as 50%. For both light and heavy elements, pressed pellets improve the reproducibility of the determinations by permitting the x-ray detector to sample a more uniform surface.

INFRARED—

In infrared spectroscopy, the major use of the Loomis Press is for the preparation of KBr pellets. The sample is first ground in a Wig-L-Bug with potassium bromide. It is then placed in an evacuable die (these are available from Beckman Instrument, Perkin-Elmer, Baird-Atomic) and pressed at around 40,000 psi. For micro work, a 1/4" diameter die is often used. This is especially fragile and the precision features of the Loomis Press assure its long-life as it forms pellets optically clear, with accurately parallel faces.

To obtain extreme rigidity, the Loomis 20-ton Hydraulic Press has three stanchions instead of the usual two. The extra stanchion minimizes cocking of the moving platen as pressure is applied. In order to make sure that the head remains parallel to the hydraulically operated platen, the former is adjusted by means of a handwheel which, through a chain drive, moves the three supporting nuts as a single unit. The die plunger is bolted to the center of the head to facilitate pelletizing and to assure equalization of stress on the instrument and die.



Loomis Press with KBr Die Mounting Plates

SPECIFICATIONS

Maximum vertical opening between platen and head	22-3/8″
Horizontal clearance between any two stanchions	9-9/16″
Vertical stroke of moving platen	6-1/2″
Overall height	44-3/16″
Overall width	23-7/8″
Depth	16″
Weight	280 pounds
Weight, crated for shipment	350 pounds
Maximum pressure	40,000 pounds
Gauge	Maximum indicating, calibrated in 500 pound units

PRICE LIST

3620 **Loomis 20-ton Hydraulic Press,** with three stanchions and handwheel to adjust the head and maintain it parallel to platen at all times; capacity 40,000 pounds indicated on maximum indicating gauge; platens ground smooth and parallel within 0.002″; centering bolt to attach die plunger in true center and so equalize stress **$635.00**

3621 **Die for preparing 1/2″ diameter pellets,** hardened tool steel; with push-out device for ejecting discs **$138.00**

3622 **Die for preparing 1-1/4″ diameter pellets,** hardened tool steel; push-out device for ejecting discs **$210.00**

Note: If the Press is to be used with evacuable dies supplied by infrared spectrometer manufacturers, a special centering plate may be required. Please furnish name and serial number of your instrument so we can quote properly.

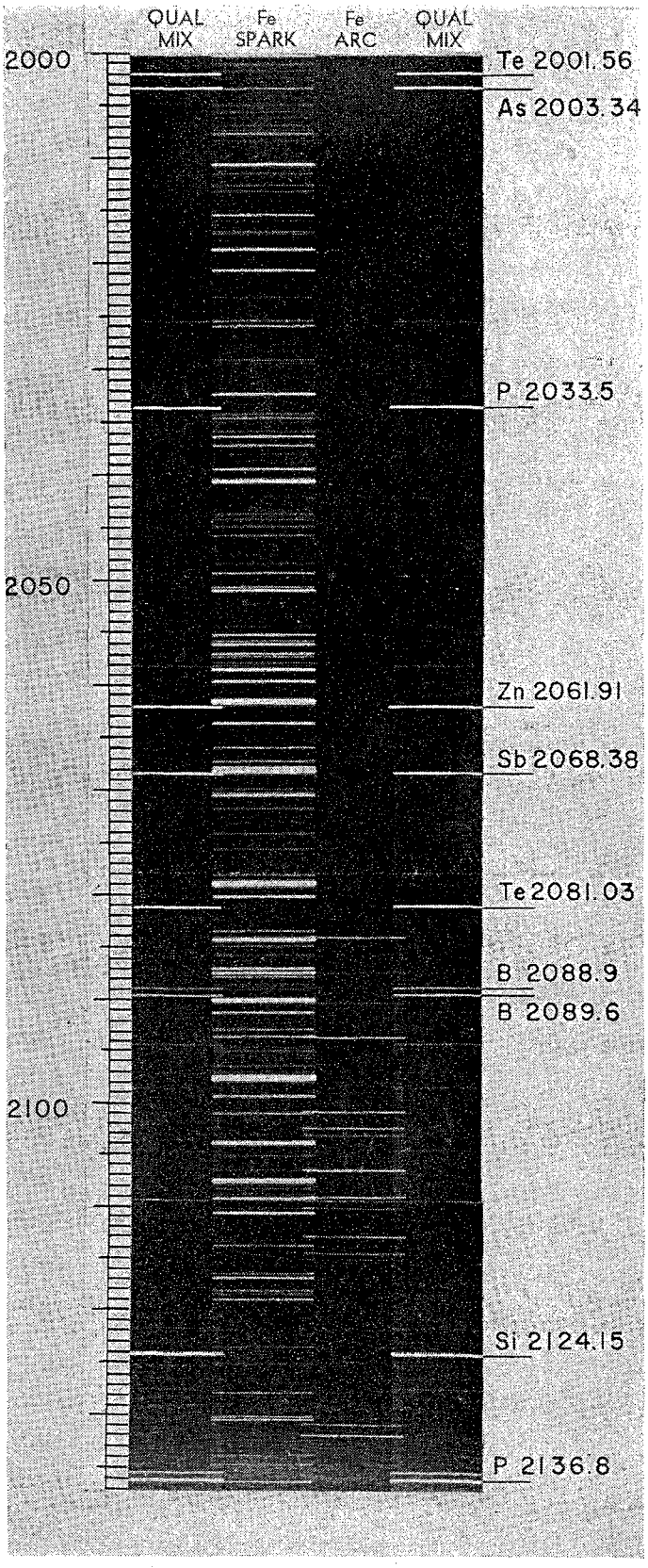


Chart from Qual Mix Atlas taken on Q-2 plate in region below 2300A. Note crisp lines and high contrast.

QUAL MIX ATLAS

Qual Mix is a powder containing 43 of the common elements so blended that a few lines of all of the elements appear when the material is burned in a dc arc as directed. Qual Mix was developed especially for the modern grating spectrograph which, because of its superior resolution above 3400A, has helped open this region to effective analysis. Although it was introduced only last year, it is now an accepted standard for qualitative analysis in hundreds of laboratories.

While extremely useful alone, the powder is even more valuable accompanied by the new Qual Mix Atlas. This is a series of 20 spectrogram enlargements taken on a large grating spectrograph, covering the region 2000-4650A. The charts consist of four spectrograms, as may be seen in the accompanying photo, taken with a Hartmann diaphragm to facilitate identification of the lines. The top and bottom are of Qual Mix; the inner two of an iron arc and spark. Alongside one of the Qual Mix exposures is a wavelength ruling divided into single Angstrom units. The element lines are identified according to wavelength to the nearest hundredth of an Angstrom.

In addition to these spectrum charts, the Atlas contains:

1) general directions for burning Qual Mix, including suggested filters to cut out overlapping orders and recommended photographic emulsions.

2) a table of persistent lines arranged according to wavelength.

3) a table of persistent lines arranged according to element.

4) a table giving the approximate % concentration of the elements in Qual Mix.

5) two chapters on qualitative spectrochemical analysis especially for trace elements; techniques to optimize conditions are stressed for different types of equipment.

The entire book is attractively and sturdily bound in a rigid, leather grained, washable vinyl cover. A multiple plastic ring binder is used, enabling the 8-1/2" x 11" book to be opened flat for ease in referring to it. The spectra themselves are printed on heavy card stock assuring many years of usefulness in the busy laboratory.

1025 **Qual Mix Atlas,** 20 charts containing spectra of Qual Mix with lines of 43 elements identified, as described in above text.
Each **$24.75**

1020 **Qual Mix,** preparation of 43 elements for qualitative analysis.
Per two grams **$20.00**

NEW!

NOBLE METALS ELEMENT KIT

The Noble Metal Standards will add ten elements to our standards for spectrochemical analysis, thus bringing the total number of elements covered to 69. The first group of standards covered the most common 43 elements; the next, the rare earths, scandium and yttrium. The present ones include hafnium, rhenium, rhodium, platinum, palladium, iridium, ruthenium, gold, gallium and indium.

Currently available is the Noble Metals Element Kit, the first in a group of qualitative and semi-quantitative standards. The Kit contains adequate quantities of each of the elements to last many years in the average spectrochemical laboratory. Some of these are in the form of powders, others solutions.

It will be noted that osmium is omitted from the elements in the Noble Metal Standards. Fumes of this element are extremely poisonous and may cause blindness. With proper precautions, it can, however, be burned in a spectrographic arc and, upon request, we shall help those interested obtain small quantities of osmium or its salts.

1040 **Noble Metals Element Kit,** contains small quantities of high-purity salts of the following elements: gallium, gold, hafnium, indium, iridium, palladium, platinum, rhenium, rhodium, ruthenium; boxed in a plastic container.
Per Kit **$50.00**

RARE EARTH QUAL MIX

Analagous with our Qual Mix, Rare Earth Qual Mix contains 16 elements so proportioned that a few lines of each of the elements appear when burned in a dc arc. Consistent with the Rare Earth L Standards and Element Kit, the new standard contains the identical 16 elements. These include all of the rare earths (with the exception of promethium of which there are but a few milligrams in existence) plus scandium and yttrium. Rare Earth Qual Mix will shortly be augmented by an Atlas (similar to the No. 1025 Atlas announced elsewhere in this issue), which will extend its usefulness. In the meanwhile, however, it will greatly help the spectrographer spot the analysis lines of the rare earth elements which, because of their complex spectra, are often quite troublesome.

1033 **Rare Earth Qual Mix,** preparation for the qualitative determination of 14 rare earth elements plus scandium and yttrium.
Per two grams **$20.00**

1030 **Rare Earth Element Kit,** contains quantities ranging from 100 mg to 2 grams of the same elements as in 1033.
Per Kit **$50.00**

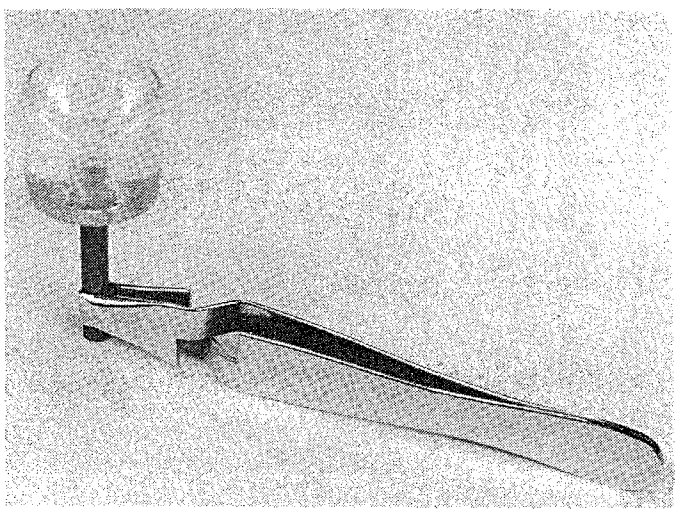
1031 **Rare Earth Spex Mix,** contains the same elements as in 1033, each at exactly 5.28%; for semi-quantitative analysis of "pure" rare earths.
Per two grams **$36.00**

1032 **Rare Earth L Standards,** for semi-quantitative analysis of mixtures and ordinary unknowns containing rare earths.
Per set of 4 standards **$49.00**

ELECTRODE TWEEZERS

Here, at last, is a pair of tweezers specifically designed for picking up and holding graphite electrodes from 1/8" to 5/16" in diameter. Constructed of Type 430 stainless steel, it may be washed in dilute acids for the most exacting trace-element work. Self closing, the tweezers will stand up while grasping an electrode so that the latter may be readily filled with or without the use of one of our plastic funnels. You will want several pairs of these handy tweezers, at least one for removing spent electrodes and separate ones for handling unused preforms.

3503 **Tweezers,** stainless steel, for handling spectroscopic graphite electrodes.
Per Pair ..... **$ 2.50**



CALCULATING BOARD

In photographic spectrochemical analysis, transmittance readings of photographed spectral lines are converted into intensity. Since it is ordinarily necessary to compensate for variations in the burn from sample to sample, intensity ratios of certain line pairs are usually taken. Often additional compensations are made by correcting for background behind the spectral lines of interest.

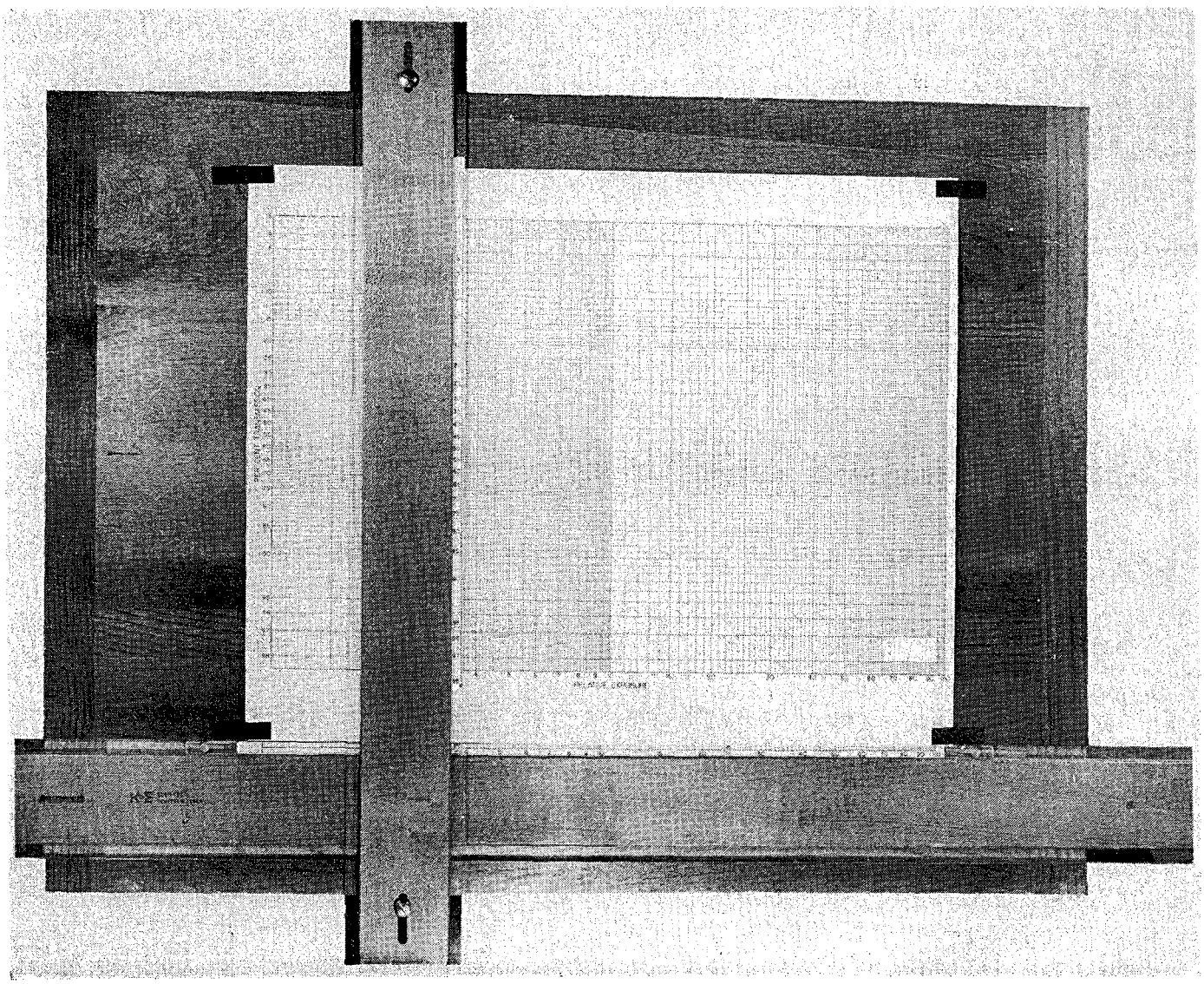
These calculations can be performed rapidly and accurately on the Spex Calculating Board. Constructed from standard drafting room components, the 31″ x 23″ Board consists of a hardwood base on which a vertical slider is moved through a parallel attachment, the mechanism for which is underneath the base and out of the way. The horizontal slider is a draftsman's straight edge moved in a groove cut into the Board. Both sliders have transparent, plastic edges to aid in interpolating and overcoming parallax.

Although the Calculating Board was designed specifically for use with our Seidel Kit, it is perfectly suitable for use with the H&D curve plotted on ordinary log-log coordinates. For the Seidel system, the Seidel-scale is mounted on the vertical slider. For the H&D, a 2-cycle logarithmic scale is mounted on the vertical slider. For both systems, a 2-cycle logarithmic scale is mounted on the horizontal slider. The scales are sandwiched between the transparent edge of the slider and a clear Lucite guard, to keep the scale clean.

The Board stands at an angle to facilitate reading. It weighs under 12 pounds so that it may be readily stored to conserve space when not in use. Our low price reflects the choice of common drafting room parts.

3610 Calculating Board, for converting % transmittance to intensity ratio, completely assembled and with full instructions.
Each **$93.00**

1102 Seidel Emulsion Calibration Kit
Each ..... **$ 8.00**



tricks of the trade

ANALYZING THIN METAL FOILS

A novel use of our Combination Analyzer is to analyze thin metal foils. Ordinarily, if a thin sheet is sparked, it gets so hot that its spectrum is hardly recognizable. If, on the other hand, it is placed on the table adapter of the Combination Analyzer, it may be sparked while turning horizontally with far less local heating. Resulting spectra are compatible with those from thicker standards.

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ANALYSIS OF REFRACTORY MATERIALS

James E. Scott of Nuclear Materials and Equipment Corporation in Apollo, Pa., has developed a noteworthy method for the semi-quantitative determination of metals in refractories which combines the multiple addition technique using Spex Mix with the carrier distillation technique developed back in 1946 by Scribner and Mullin. Mr. Scott "Wig-L-Bugs" 80% of the refractory material with 20% of a carrier such as AgCl, NaCl, BaF_2 and then places 100 mg of the mixture in an anode cap. After heating the material for 10 minutes under an infrared lamp, it is arced for 60 seconds in a dc arc. Using ordinary densitometer and calculation techniques, Mr. Scott obtains an accuracy of 1/2 to twice the amount of element present. He claims that the method offsets the usual requirement that standards have to be prepared for every refractory oxide. A further advantage is that the continuum from such elements as U, Zr and Th is avoided with the result that the detectability and accuracy of elements in the few ppm range are sharply enhanced.

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WINTERTIME MIXING IN THE MIXER/MILL AND WIG-L-BUG

Winter brings on electrostatic problems. Charges build up in the plastic vials in both the Mixer/Mill and Wig-L-Bug, sometimes preventing adequate mixing. This is easily overcome by coating the vial with a layer of graphite powder to make its surface conducting. Simply place a small quantity of graphite together with a plastic ball in the vial and let the instrument run for a few seconds.

THE NEAR FAR ULTRAVIOLET

Since our recent announcement that we are stocking Ilford Q-1, Q-2 and Q-3 plates 4" x 10", several specific applications of the emulsion have come to our attention. T. M. Hess of the Dow Chemical Company, Midland, Michigan, has developed a procedure using the Q-2 emulsion for the determination of Zinc at 2139A in calcium phosphate. The method is 30 times as sensitive as an earlier one which employed conventional plates and the zinc 3345A line. As little as 0.5 ppm of zinc may now be determined and the method is being extended both to other matrices and other metals. Incidentally, a close, strong copper line precludes the use of 2139A for zinc in the presence of copper.

Several labs use Q plates for the determination of selenium. At the Geological Survey in Washington, C. L. Waring, H. W. Worthing and K. V. Hazel report in *Anal. Chem.* that 0.01% selenium may be determined in minerals. Another laboratory reports that selenium may be determined in free-machining stainless steel in the range 0.1-0.3% using the 2063A line as recorded on Q-2 plates. Both of the above methods use a dc arc directly on the material to be analyzed.

J. E. Scott, Nuclear Materials and Equipment Company, Apollo, Pa., reports that the sensitivity for cadmium 2288A in uranium oxide is improved at least 5-fold to 0.1 ppm by using the Q-3 plate.

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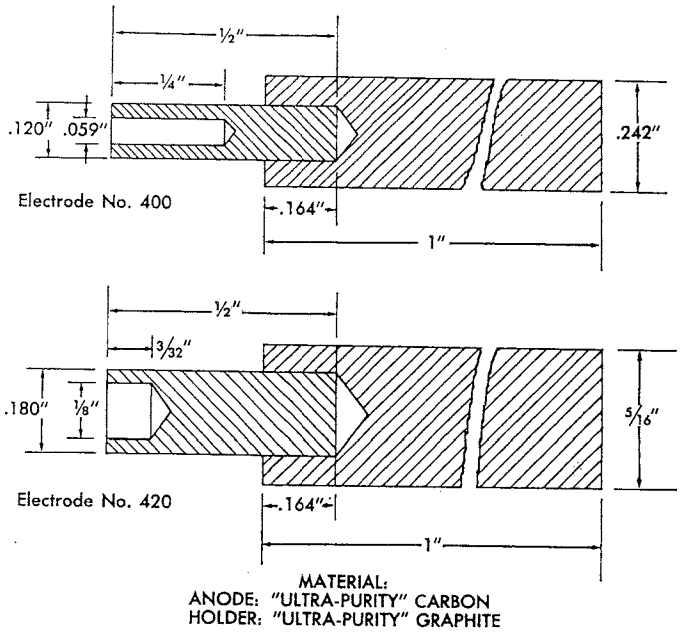
UNRAVELING SPECTRAL ORDERS

Especially when higher orders of a grating spectrograph are employed, there may be confusion from lines of overlapping orders. Frequently, these may be filtered out with a sharp cut-off photographic emulsion or a filter at the slit. Sometimes, however, an appropriate filter is not available. In these instances, one trick is to cover a tiny portion of the slit so that it will not interfere with subsequent densitometry. A comparison between the unfiltered line and its filtered portion will tell the spectrographer at a glance its approximate wavelength. A typical example is the use of a large spectrograph in the region 2500-5000A. Around 5000A, second order lines from 2300-2500A will appear. Unfortunately, a filter with a sharp cut-off below 2500A is not available. In its place a piece of ordinary glass (e.g., microscope slide) may be used at the very bottom of the line. Cutting off all radiation below 3100-3500A but practically transparent above, the filter will attenuate a second order line but not a first.



UNITED INTRODUCES NEW CARBON-GRAPHITE ELECTRODES

Announcement has been made by United Carbon Products Company of the manufacture of two new double section carbon-graphite preformed spectroscopic electrodes for dc arc analysis. Both of these new electrodes, designated No. 400 and 420, afford the burning advantages of an amorphous carbon cup while holding down overall costs due to the good machinability of the lower graphite section.

A recent article by J. W. Mellichamp and J. J. Finnegan, Signal Corps Engineering Laboratories at Fort Monmouth, N. J., (*App. Spec.* 11, 158, 1957) shows the advantages of such electrodes in reducing the burn-off time by almost a factor of two and improving the sensitivity of nearly all elements determined in silicon metal. The comparison is with typical *graphite* electrodes. The advantages are due to the lower conductivity and therefore higher temperatures of carbon electrodes.

It is felt that these electrodes will find wide use in a variety of analyses. Available from our stock, they are priced at $11.25 per box of 25 for the No. 400 and $12.50 per box of 25 for the No. 420.

CLARA SMITH DMS CONSULTANT

Clara D. Smith is now serving as our technical consultant on the DMS System of cataloging organic compounds through their infrared spectra. Highly respected not only for her technical competence but also for her drive in the cooperative efforts of the Coblentz Society, ASTM and other organizations, Mrs. Smith has recently opened her own laboratory at 900 E. Mulberry, Evansville, Indiana. Services include development of analytical methods, research studies, running and interpreting spectra. A unique service is to help a new laboratory get under way by spending time there.

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