The SPEX Speaker

Vol. VII—No. 1 March, 1962

SPEX INDUSTRIES, INC. • P.O. BOX 98, SCOTCH PLAINS, N.J.

A NEW GRAZING INCIDENCE VACUUM SPECTROMETER

D. O. LANDON and A. J. MITTELDORF

WHEN Princeton University's Project Matterhorn physicists first approached us with performance specifications for a vacuum spectrometer, they knew that no conforming instrument existed, even doubted that it was feasible. Yet their stringent demands were quite understandable in view of their own mountain of problems posed by reactions occurring in glowing hydrogen plasmas.

Project Matterhorn is part of the great effort under way to tame the hydrogen bomb, to control fusion in such a manner that the fiery heat erupting will be converted into useful electricity not useless destruction. If hydrogen atoms can be raised to a high enough temperature and held there for a long enough time, they will undergo fusion to helium, a reaction thought to be continuing for millions of years in stars. Thus far, however, man's only way of attaining the conditions has been with a fission bomb detonator.

In the early 50s physicists suggested another way based on the so-called pinch effect theorized in 1934 by W. H. Bennett and developed further by L. Tonks. When a stream of ionized gas is electrified, a magnetic field is generated tending to constrict, or pinch, the stream. First demonstrated in England by A. A. Ware in 1951, it proved to be extremely unstable, breaking up in a few millionths of a second. If the pinch effect could be sustained, this would offer the possibility of ohmic heating of the plasma stream to the temperature desired and, at the same time, keep the heat away from the container which of course it would immediately vaporize. An external, pulsed magnetic field appeared to be a way of stabilizing the pinch effect and it was the basis of Project Matterhorn, part of an all-out effort by the Atomic Energy Commission to produce a machine—soon named the Stellarator—in which to test the ideas.

The first Stellarator was, naturally, small. It was a prototype designed to check the theory of magnetic stabilization with but a fraction of the capacity needed to reach fusion temperatures. So promising did it prove that other, larger ones were built, with progressively stronger magnetic fields, capable of delivering immense jolts of electricity to heat the ionized gas. Based on the design of these instruments labeled A1, A2, B1, etc., a foreign laboratory is now hard at work on a model dubbed "Me2" by the Princeton staff.

The newest Princeton machine is the C Stellarator which, together with its power supplies, control rooms and test instrumentation, fills a good-sized building. Three 20-foot diameter steel flywheels rotating at 900 rpm store the immense energy required to electrify the ion stream every twenty seconds.

To measure the temperature reached in the ion stream, its distribution and duration and to study the reactions spectrographs are largely used. One of the most disturbing questions is the as yet unexplained discrepancy between the input and output energy. Ideally, all of the electricity fed into the ion stream would be converted into heat but, in practice, large amounts are "lost" elsewhere. Although radiation measured in various parts of the spectrum and the production of neutrons account for some of it, calculations indicate that still more is being dissipated. The one region which has not been explored because of its relative inaccessibility is that between 50-500A.

Amenable to measurement or not, the region is important because it is here that the strongest multiply-ionized lines of most elements appear. Because the excitation of the Stellarator is so great, elements such as carbon, oxygen and nitrogen—known impurities in the hydrogen atmosphere of the Stellarator—are not detected at longer wavelengths. All of their energies are released at the short wavelengths. It is theorized that traces of carbon, oxygen and nitrogen act as highly efficient radiators, stealing large segments of the energy that should be heating the hydrogen. Despite the fact that the Stellarator is evacuated to ultra high vacuum levels of 10^{-10} mm Hg and that only the purest of hydrogen is admitted, impurity atoms are known to exist. Spectrographically, it is hoped to track down these impurities, to find out if and how they are reacting and under what conditions. The only region of the spectrum where such studies can be conducted is in the grazing incidence ultraviolet below 500A.

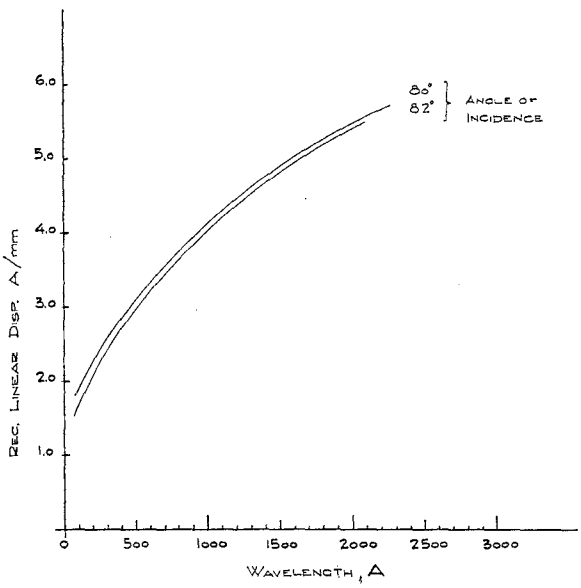
Put simply, though not entirely accurately, this region is a no-man's land separating the x-rays from the ordinary vacuum ultraviolet. The techniques, instrumentation and materials of neither can be used here. It is a region opaque to all known materials, forcing the use of a high vacuum and precluding optical windows of any kind. No known highly reflecting materials exist. There is no known method of producing a continuum here—such as the incandescent lamp in the visible or the hydrogen discharge in the ultraviolet. Only recently has a detector been devised to respond to energy here and not everywhere else as well.

Such then are the conditions for which we were asked to build an instrument capable of being used as a photoelectric scanning spectrometer and a photographic spectrograph, an instrument now affectionately referred to as the GISMO (Grazing Incidence Spectrograph Monochromator).

WITH no adequate reflecting material available, the usual spectrograph, depending on light incident on and reflecting from the surface of a diffraction grating at small angles to the normal, is thus not suitable in this region. Until or unless a material can be found with high reflectivity, spectrographs will have to depend on the phenomenon of reflect-

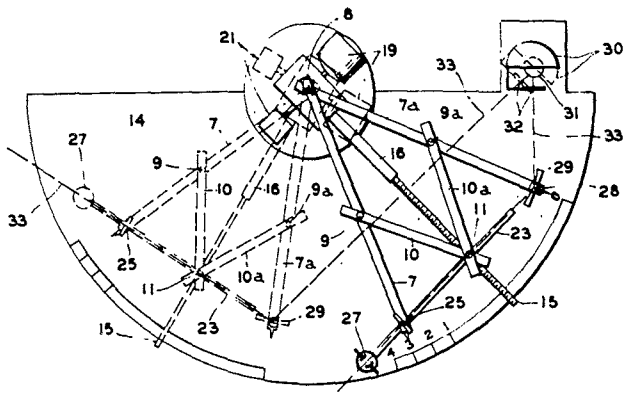
ance at grazing incidence, that is, at very shallow angles, where light is reflected from nearly all materials. The actual angle of incidence must increase the lower wavelength of radiation sought. At least 80° to the normal is necessary to reach a few hundred angstroms and even higher angles—although the exact angles have not been determined—are necessary to reach lower wavelengths.

Although light is reflected at grazing incidence, solving this one problem raises others, the principal one being astigmatism. Light from the concave grating surface is no longer approximately equal to the height of the entrance slit when it is received at the detector. Instead, as the incidence angle increases, the light spreads vertically until at grazing angles a point of light appears as spectral lines several times taller than the grating itself. It stands to reason that the angle of incidence should be kept as low as possible consistent with the lowest wavelength desired. In this case adjustments also had to be provided to permit the angle to be varied from 80° to 86°. This had to be done in such a fashion that, when the instrument was set up as a spectrometer, the readout would be linear in angstroms regardless of the angle of incidence. Put another way, two criteria had to be met simultaneously. The position of the exit slit had to be recorded not in terms of how far along the Rowland circle it had moved but at what wavelength it was reading. And, to complicate matters still further, the angle of incidence was to be made variable at the same time. The dispersion at 80° angle of incidence varies from 1.9 A/mm at 100A to 5.8 A/mm at 2200A; at 82° from 1.6 A/mm at 100A to 5.5 A/mm at 2200A. Yet the readout had to be accurate whatever angle of incidence was chosen.



The mount through which this was accomplished is, as far as known, a new one for which a patent has been applied. It is depicted in the drawing below where it will be seen that basically an entrance slit, an exit slit and a concave grating are all arranged on the usual Rowland circle. The grating is set, however, so that its rulings are almost parallel rather than perpendicular to the light from the entrance slit. The entrance slit is fixed in the main cylinder but all other components are mounted inside the instrument.

The wavelength drive is through a sine-bar which results in a linear motion with respect to wavelength of the exit slit (25) along the Rowland Circle. It is a symmetrical system in which arms (7) and (7A) are of equal length and represent two sides of a triangle. At equal distances from pivot (8)



Grazing Incidence Vacuum Spectrometer Mount

there are two additional arms pivoting at (9) and (9A) whose opposite ends pivot together at (11). Below (11) is a nut which rides along the screw (15) as it is rotated. That is, when the screw is rotated, its mating nut pulls the exit slit (3) towards and away from the grating (29) in scissors fashion. The entire mechanism is pinned to a frame by a stud (28) directly under the grating which, by preventing the grating from moving, constrains the exit slit to a path along the Rowland circle as the screw is rotated. This is accomplished at (8) through a set of bevel gears and a vacuum seal to a motor outside of the chamber.

Light entering the slit at (32) is directed to the concave grating (29), the radius of curvature of which is exactly twice the radius of the Rowland circle or the length of arms (7) or (7A) measured between the pivot point and the exit slit. The diffracted light is directed according to its wavelength and focussed at points along the circle traversed by the exit slit (25). Arm (23), swiveling at (23) and rigidly attached to the exit slit, constrains it so it always is directed toward the grating.

As already stressed, one of the specifications called for a variable angle of incidence. Note that this is readily accomplished by removing the stud at (28) and replacing it elsewhere. Since the sine-bar mechanism is independent of the position of the entire scissors assembly, and the latter swivels around the center of the Rowland circle (8), linear and identical readout is maintained. It is only necessary to change the zero on the counter when switching incidence angle. This is, of course, the direct image and is readily centered photoelectrically at zero on the counter. Further, the exit slit, coupled as it is to the scissors, constantly points to the center of the grating no matter what the angle of incidence.

In actuality, the instrument was found to have a deviation of around 1A when scanned during one of the first runs as shown below. Testifying to the excellence of its construction, readout was still within 3A of the correct wavelength when the exit slit was scanned from high to low wavelengths, backlash amounting to only (2A).

Another requirement was for two such scanning systems to be incorporated in different planes. This would permit "sitting" on two different lines to obtain their intensity ratios. Again the design lent itself nicely to this feature. Through the use of a hollow shaft and a second set of bevel gears at (8), two independent drives were incorporated. The two scissors have arm (9A), that attached to the grating, in common. The scanning parts, however, are independently driven by either one or the other of the two lead screws.

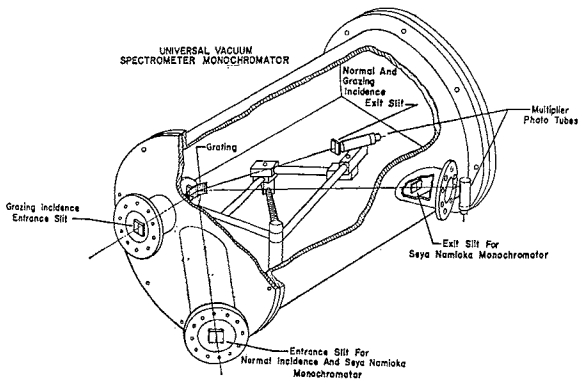
By incorporating two sets of arms, it became possible for the two exit heads to jam into each other in scanning, a prob-

lem overcome by the use of limit switches placed just before the two heads could touch. The exit slits themselves were made unilaterally opening in order to permit the two heads to come as close together as possible so that closely adjoining lines could be read. This meant that when the slit is opened or closed the center of its position would change. Adjusting the slit width from the usual 10 to 100 microns would, at any wavelength setting, change the center of the slit by far less than 1A, the target error.

At grazing incidence it turns out that the image of a straight entrance slit is curved. In order to conform to the image and thus achieve the optimum resolution and signal both exit slits were curved to match the image.

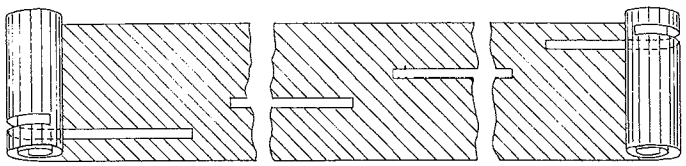
SODIUM salicylate coated multiplier phototubes have, until recently, been the best means of detecting energy in the grazing incidence region. Radiation causes the coating to fluoresce at longer wavelengths which can pass through the quartz or glass envelope. It has been shown a multiplier phototube so treated has a fairly uniform quantum efficiency with wavelength over much of the far ultraviolet. That is, its output is proportional to the energy received and not wavelength dependent.

Though usable, this approach leaves much to be desired in the grazing incidence region because of the severe problem of scattered light of longer wavelengths than those to be measured. A new electron multiplier manufactured by the Bendix Corporation shows much promise in this connection. Its detector is beryllium copper having such a high work function that it responds only to very short wavelengths. In common parlance, it is "solar blind." Its use promises a much better signal-to-noise ratio throughout the vacuum ultraviolet and especially as the direct image is approached where scattered light may all but mask out the radiation under study.



Diagramatic View of the mechanism of the Spectrometer. Actually for Princeton the normal incidence entrance and exit slits were not included. Their instrument had two independently scanning exit slits.

THE optical layout, folding as it does, makes room for a camera covering about 30 inches of spectrum or approximately 10-3000A. For convenience it was required to rack the camera from outside the vacuum to permit a number of exposures to be made in juxtaposition. The usual approach of providing an aperture in front of the focal "plane" was abandoned: it seemed too complicated to provide a removable camera, rackable and with a darkslide that could be inserted and removed from outside the vacuum. The ultimate solution took advantage of the extreme astigmatism in the system. The image at the camera is much greater than the width of the 35-mm film so that more or less even illumination is obtained at any position along the film width. This considered, a "focal plane shutter" arrangement was designed. At either end of the camera a spool was mounted on which a continuous strip of beryllium copper was wound. Apertures were cut out in the strip, in the fashion shown below, permitting several exposures to appear one above the other. The film strip itself is pushed into a slot behind the shutter, and is protected from light for loading and unloading by winding the beryllium copper strip to an opaque section at one end of the spool. The camera is racked through a length of flexible cable by turning a crank on the outside of the vacuum chamber. Position of the exposure is indicated on a dial alongside the crank.



Focal Plane Aperture Arrangement

AS already mentioned, an ordinary high vacuum of 10^{-6} mm Hg would flood the Stellarator with billions of contaminating atoms. The problem was how to house a spectrograph in an ultra-high vacuum. To do this it is necessary to bake out all of the components for many hours at a dull red heat. What happens to a diffraction grating at such temperatures? Physicists at Matterhorn set out to find out before preparing the specifications for our spectrograph. Results were disastrous. Small pieces of original gratings were found unusable after baking. (Mounted on epoxy resins, replicas would melt at bake-out temperatures.)

This, then, represented a very serious obstacle, one that was never completely resolved. An ultra-high vacuum spectrometer being impossible, it was decided to have it open to the Stellarator only for a fraction of a second, long enough to snap a photograph or obtain a photoelectric tracing but sufficiently short, it was hoped, that gases from the spectrograph would not interfere with the operation of the Stellarator.

A bakable impulse operated slit was specified. In the final design, a shutter between the entrance slit and the Stellarator was constructed so it could be opened hydraulically through a bellows arrangement. It serves both as an optical and a vacuum shutter, protecing the spectrograph from light and the Stellarator from gas until opened momentarily.

THE hydraulically actuated slit-shutter may seem puzzling. Would it not be far easier to use a solenoid? This brings up another aspect of the specifications, that calling for a bare minimum of magnetic parts. The Stellarator with its tremendous magnetic fields could distort components of the spectrograph if they were made of magnetic materials. An arm could bend enough to change the wavelength, defocus the instrument, or otherwise temporarily disrupt its operation. Conversely, magnetic components in the spectrograph could adversely affect the configuration of the Stellarator's magnetic field; both considerations plagued us. Not until you try to do without it do you realize how widespread is this phenomenon of magnetism. Several almost inaccessible screws, nuts and washers appeared when probed for with a small magnet. Substitutes had to be found for relays, motors and meters or else they had to be placed at a distance of many feet from the Stellarator. Construction of the spectrograph and housing were principally of stainless steel. The slit components were machined of Inconel-X in order to be bakable.

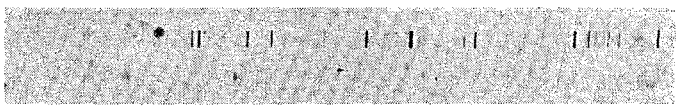
ANOTHER set of specifications for the spectrograph related to its ultimate intended use as a research tool. For this purpose, Matterhorn spectroscopists needed the instrument to peer into several different Stellarators with windows of different heights above the floor. Furthermore, they wanted to be able to look at different portions of the ion stream and from different directions in order to survey it thoroughly. To fulfill these specifications, the spectrograph was constructed with two vacuum ports at right angles around the cylinder. Either one can be used so the entrance slit is either vertical or horizontal. A lifting and tilting feature was added so the instrument, resting on a flexible bellows, between itself and the vacuum console can be moved with jacks to have it point properly.

THE basic vacuum pumps consist of a mechanical fore pump with a capacity of 13 cfm and a water-cooled 6" diameter oil diffusion pump with a capacity of 1500 lps. Measurements of pressure are made through a thermocouple and an ionization gauge, the combination of which permits a continuous range of measurement from 2000 microns down to 10^{-8} mm Hg.

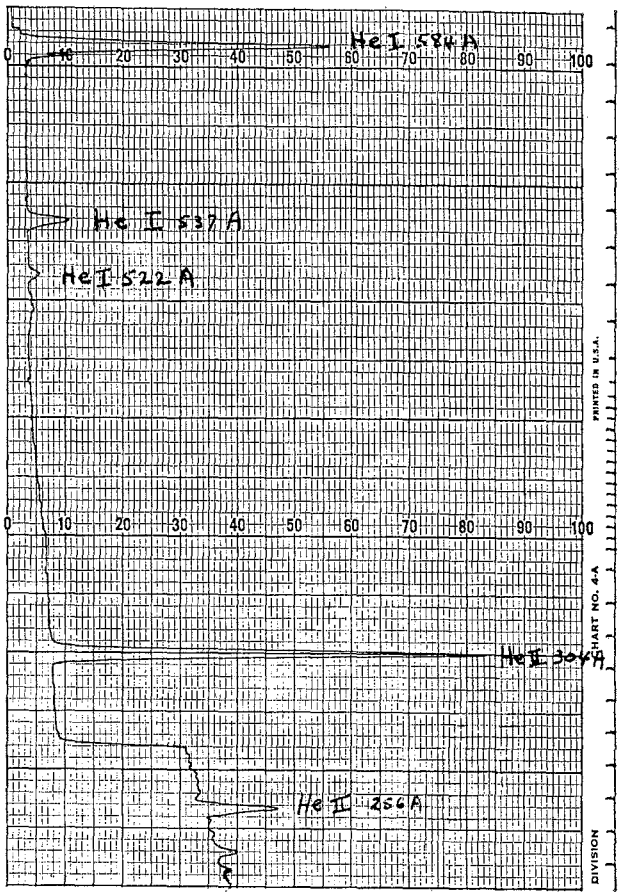
Not only the spectrograph but, conceivably the Stellarator itself could be damaged severely if the vacuum system were to fail permitting high-vacuum oils as well as atmospheric gases to burst into their respective chambers. With this constantly in mind, many protective devices were incorporated. The principal one is a fail-safe, pneumatically-actuated, solenoid-controlled (the need for this magnet outweighed the possible effects it might have on the Stellarator) valve at the throat of the vacuum system. In series with the power line, it would close instantly in the event of power interruption. Another protective device monitors the flow of water out of the oil diffusion pump. Should this slow down, the device turns off the heater to the pump. Still another protector is a sensitive relay on the ionization gauge. When set for a certain pressure, it automatically trips the main valve if that pressure is exceeded.

The vacuum system in the spectrograph can be taken down to the 10^{-5} range in less than one hour after start-up. With the pumping system on between runs, the pressure can be brought down in the main chamber in about 15 minutes. A chevron baffle, which may be cooled down with a refrigerant or liquid nitrogen, takes the vacuum down well into the 10^{-6} range.

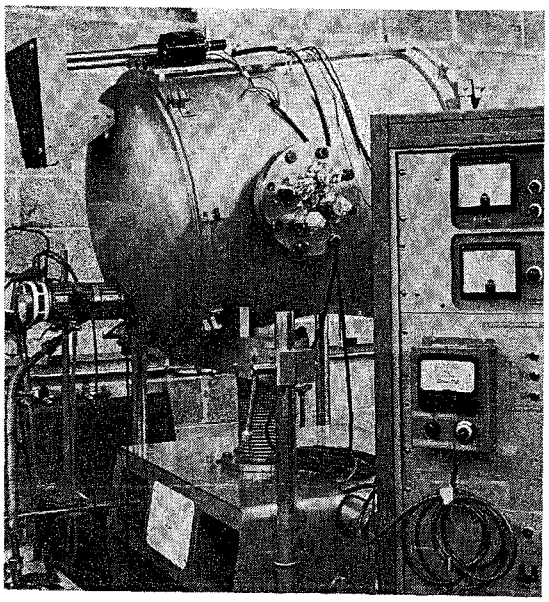
TO a great many scientists at Project Matterhorn, we owe the opportunity of designing and constructing this spectrograph. Special thanks are due Drs. Joseph Hirschberg, Einar Hinnov, Dirck Dimock for their invaluable aid in thrashing out the various ideas. We, in turn, wish them luck needed to solve one of the most baffling problems confronting man today—the increasingly urgent need to find an inexpensive source of non-fossil fuel. That such a source will be forever locked up in the secrets of the deuterium atom, out of reach of mankind, is inconceivable. But right now, as Dr. Hinnov pointed out to a visitor, there are heavy power lines coming in to Matterhorn but not even the poles are up for electricity to leave.



Spectrum taken of Princeton's C Stellarator during pulsing "shake-down". Wavelength interval approximately 250-400A showing many impurity lines of highly ionized carbon and oxygen. Spots on film are typical of the Schumann emulsion used (Eastman SWR).



Spectrometer tracing of hollow cathode discharge in helium. "Pips" at right are automatically triggered by wavelength drive at intervals of 10A.



Installation at James Forrestal Research Center for Stellarator Plasma Analysis (Photo, courtesy Princeton University.)

PLASMA JET SOLUTION ANALYZER

Baer and Hodge (1) have evaluated several of the commonly used solution techniques in emission spectrochemical analysis, pointing out their advantages and disadvantages. A solution analyzer, too new for inclusion in their report, is the Plasma Jet proposed at about the same time by Scribner and Margoshes (2) and Korolev and Vainshtein (3) and later improved by Owen (4). It appears to have a combination of features promising for many analytical problems involving solutions:

1) Its stability is unmatched by other methods. Standard deviations of intensity ratios of well-matched lines range from 0.2 to 1.6% (5).

2) Sensitivity is equal to or better than that of other solution techniques, particularly when helium is used for swirling the plasma column.

3) Although the upper electrode is changed between samples, the lower electrodes need be changed only occasionally as they erode under the high temperature of the jet.

4) It may be run for long periods and can presumably be adapted to on-the-line stream analysis.

5) Once set up, the jet is extremely simple to use. The liquid to be analyzed is atomized from a small container.

6) Used successfully for liquids and slurries, the instrument seems amenable to gas analysis as well.

7) The source is a 10-20 ampere dc arc as used in most spectrochemical laboratories.

8) Operating at around 8000°C, as compared with 6000°C for the usual dc arc, matrix effects may be reduced considerably.

9) Organic liquids, flammable or not, as well as inorganic solutions may be analyzed.

In principle, the Plasma Jet Solution Analyzer is similar to the Stallwood Jet. After the liquid or gas is atomized it is directed upward to a chamber where a tangential stream of gas swirls it as it is forced up the arc column. Argon has been used both for atomizing and swirling the gas although, for the latter purpose, helium has the advantage of lower background and higher sensitivity. Compressed air, nitrogen, and carbon dioxide appear to be other possible gases depending on the application.

Since no graphite enters the highly ionized arc column, there is no production of cyanogen, freeing this region for the use of lines which must be avoided in open arc analysis. On the other hand, the high temperatures do give rise to OH bands. These are many-lined spectra in the region 2811-3000A and 3064-3300A which degrade sharply toward the red, the spacing between lines increasing at the same time. Although it must be stressed that they do not, by any means, obscure the region completely as do cyanogen bands, there is the possibility of line interference in this region.

The Spex Plasma Jet Solution Analyzer is used by mounting it in the lower jaws of most arc stands. At the bottom of the jet extends a hollow rod 1/2" in diameter by 1-1/4" long, sized to fit the jaws. The overall height above the lower jaws to the center of the arc is 2-3/8". The outside diameter of the main body of the assembly is 2".

Water cooling connections are provided; a by-pass from the electrode jaw water is suggested as a convenient source.

For our own 9010 Arc/Spark Stand, we are now building in a second flow meter and toggle valve to control the two different flows of gas. (It is, unfortunately, a factory job to add the flow meter to existing 9010s.) External flow meters may be used directly on the gas cylinder. For the purpose, we supply an assembly having a range suitable for either flow. In practice, an indicated flow of 12-14 lpm for the tangential gas and 4-8 lpm for the atomizing gas have been found suitable. (These indicated flows are calibrated for oxygen; the actual flow depends on the gas or gas mixture chosen.) Depending largely on the bore of the atomizer (small, medium, and large are available), the flow of sample will vary from about 0.2 to over 1 ml per minute.

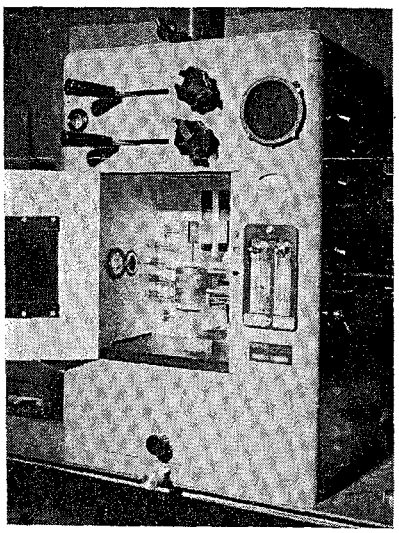
1) Baer, W. K. and Hodge, E. S., *App. Spec. 14,* 141, 1960.

2) Margoshes, M. and Scribner, B. F., *Spec. Acta. 15,* 138, 1959.

3) Korolev, V. V. and Vainshtein, E. E., *Zhur. Anal. Khim., 14,* 658, 1959.

4) Owen, L. E., *App. Spec. 15,* 150, 1961.

5) Scribner, B. F. and Margoshes, M., N.B.S. *Report 7342,* 1961.



Plasma Jet Solution Analyzer in Spex-9010 Arc/Spark Stand. To accommodate this new accessory, a second flow meter and additional plumbing have been incorporated in the stand.

9030 **Plasma Jet Solution Analyzer,** as described above, with medium bore capillary atomizer
Each **$249.00**

9024 **Flow meter assembly,** including regulator and toggle valve; attaches directly to cylinder without further plumbing, range 0-15 lpm; two required for plasma jet operation .....Each **$ 65.00**

PRE-WEIGHED CHEMICALS

PRICE LIST

SPECIFY ACTUAL WEIGHT REQUIRED

Graphite Powder, highest purity specify SP-2 (-100 mesh) SP-2 (-200 mesh) or SP-1 (for briquetting); in 3111 vial (polystyrene 1/2" dia. x 1" long) with 3112 ball (Lucite, 3/8" dia.)

		100	1000	5000
30-100 mg		$16.60	$125.00	$535.00
101-150 mg	±1.5 mg	17.60	136.00	570.00
151-200 mg		18.60	143.00	600.00

Lithium Carbonate, spectrographic grade in 3111 vial with 3112 ball

		100	1000	5000
30-100 mg		$19.80	$142.00	$603.00
101-150 mg	±1.5 mg	22.40	160.00	665.00
151-200 mg		25.00	177.00	730.00

Lithium Carbonate, mixed with SP-2 (-100) powder 1:1 by weight

		100	1000	5000
30-100 mg		$18.80	$137.00	$572.00
101-150 mg	±1.5 mg	20.90	152.00	620.00
151-200 mg		23.00	167.00	665.00

Potassium Bromide, infrared grade, in glass vial (3/4" dia. x 1" long) with stainless steel ball

		100	1000	5000
100-200 mg		$18.80	$147.00	$615.00
201-250 mg		19.30	152.00	640.00
251-300 mg	±2 mg	19.90	157.00	660.00
301-350 mg		20.40	162.00	685.00
351-400 mg		21.00	167.00	705.00

Notes: (1) these vials are sealed in containers together with silica gel to maintain extreme dryness of the KBr.

(2) These vials may be shaken in our No. 5000 Mixer/Mill directly. In the Wig-L-Bug a special adapter (3113K at $6.00) is required.

FLUXING CHEMICALS

Lithium Tetraborate, spectrographic grade, in sealed polyethylene bag. (Sample may be kneaded with lithium borate right in plastic bag, then transferred to graphite fusion crucible No. 7152)

		100	1000	5000
1000 mg		$13.60	$78.00	$420.00
1500 mg	±2 mg	15.60	91.00	465.00
1800 mg		16.80	99.00	490.00

Graphite Powder—SP-1 for pelletizing, in 1/2" dia. x 2" long plastic vial (3116) with 3/8" dia. Lucite ball (3112)

		100	1000	5000
400 mg		$23.80	$188.00	$765.00
900 mg	±2 mg	33.80	277.00	1095.00

In many laboratories, spectroscopy is a production operation and the director is expected to turn out analytical results like any other product, at the lowest cost. Toward this end, we at Spex Industries have tried over the years to introduce time-saving ideas, instruments and standards. The latest is pre-weighed chemicals, powders which are ordinarily weighed out in the laboratory, one portion for each analysis. We think we can prove to you that, by preparing these materials in very large volume using an expensive automatic balance, we can save you money and free your technicians for more important work than repetitive weighings.

Typically, a weighing in a spectrographic lab takes at least one minute including the transfer to the container. At a cost of $10.00 per hour—a figure often quoted to us by laboratory managers who include salary plus overhead—this means that each weighing costs 17 cents. On a large scale, using an automatic balance, we can weigh with equal accuracy, at a fraction of that cost and pass the savings on to you.

Further to reduce costs, we package the chemicals in containers ready for the addition of a sample and either blending or fluxing depending on the application. For the emission laboratory, you can purchase 100 mg units of graphite powder already packaged in plastic vials with a ball included, at a price per 100 of $16.60. You merely add your weighed sample and shake it in a Mixer/Mill or Wig-L-Bug. For the infrared laboratory, you can have high-purity KBr, of the proper particle size and sealed to prevent moisture pickup. It is in a glass container into which you not only mix the sample but can finally store the 13 mm pellet. For fluxing techniques in both X-ray and emission laboratories, you can choose the convenience of having weighed amounts of lithium tetraborate packaged in polyethylene bags. Drop the sample into a bag, knead the two together for a few seconds, then drop the contents (or even the bag as well) into the graphite fluxing crucible.

Please consider the price list which follows as a guide. If you require special mixtures or weights we shall gladly quote. We can work either with our materials or yours.

SPECTRAL LINE INTENSITY TABLES

A tabulation of the relative intensities of 39,000 spectral lines has recently been completed by W. F. Meggers, C. H. Corliss, and B. F. Scribner of the National Bureau of Standards. The compilation provides intensity values on a uniform energy scale for 70 chemical elements over the wavelength range from 2000 to 9000A. Although less extensive than the well-known M.I.T. Wavelength Tables, the new tables will supply spectrochemists with much-needed quantitative intensity values for those elements most commonly encountered in their analyses.

Part I of the tables is available at $3.00 from the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C. Observed and compiled by C. H. Corliss, W. F. Meggers and B. F. Scribner, they are listed as NBS Mono. *32* (1961). Part II should be available shortly.

ELECTRODE LOADER

We think we now have it, a device for quickly and reproducibly loading graphite electrodes through a vibratory tamping action. This loader is somewhat belated perhaps in the wake of No. 1 (which was just too highly priced for most of our pockets) and No. 2 (which impatiently broke loose before finishing school).

After a sample is mixed in one of our small polstyrene vials, the ball is removed and the vial set in a spring clip. The electrode, held inverted, is tightened into a collet and pushed down into the material in the vial. Firmly attached to an eccentric drive, the vial is now vibrated rapidly up and down. In about 15 seconds, the crater is filled with sample matching within a few mg the one loaded before and the one to follow.

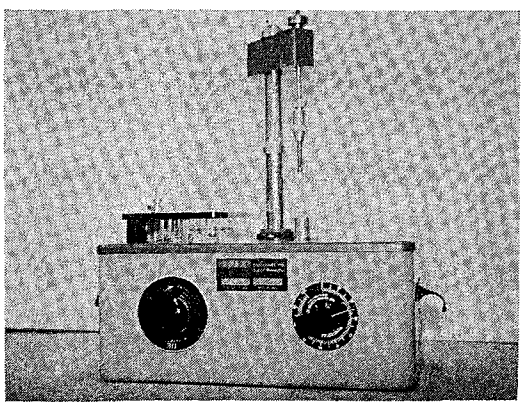
Although the table below illustrates that the loader can operate quite well, it has its limitations. Because the electrodes are loaded in an inverted position, free-flowing powders such as graphite will not pack into a shallow, wide crater. On the other hand, such materials have been successfully tamped into craters with a high depth-to-diameter ratio. Sticky substances such as zinc oxide do not load satisfactorily because they adhere to the outside of the electrode.

TYPICAL PERFORMANCE DATA

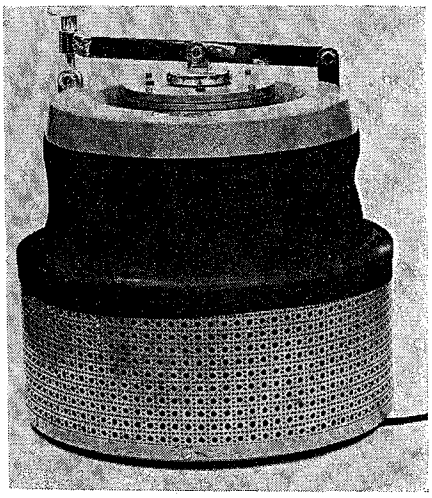
Material	*Electrode*	*Weight (mg) Packed in 15 sec.*			
Graphite SP-2 (-100)	L-3706 3/16"d crater 3/32" dp	21	21	21	21
Graphite SP-2 (-100)	L-3979 1/8"d crater 1/4" dp	34	34	34	—
Lithium Carbonate, Graphite 1:1	L-3912 1/4"d crater 5/32" dp	25	24	24	24

You will be fascinated with its collet for holding the bodies of the electrodes. Like a pin vise, it has an outside tapered screw-on cap which reduces the inside diameter as it is turned. Unlike a screw vise, there are three internal steps conforming to the actual diameters of 1/8", 3/16", and 1/4" electrodes. The operator can interchange electrode sizes as easily as records of different diameters on his home phonograph.

But before hastening to your requisition pad, we suggest that you have us make trial runs with your materials. Only then can you be sure that the Loader will do your job better than by hand. Send your materials together with a few electrodes and we'll return them—loaded to the brim, we hope.



4510 **Electrode Loader,** 115v, 50-60 cy., 60-second timer, adjustable speed motor; one collet has provisions for electrodes 1/8", 3/16" and 1/4" dia. Net weight 9-1/2 lbs., boxed 14 lbs...**$230.00**



THE SHATTERBOX

In this field, when something new is designed you can always count on the first prospect to ask for a bigger one. So it was with our No. 8000 Mixer/Mill about 1000 units ago when we were out scouting for customers. The first man we contacted was delighted by the speed with which blast furnace slag could be pulverized, and he liked the idea of having the material stay put in a closed container, but he simply needed more material than could be ground in a single load.

Well, we now have the answer in the SHATTERBOX, a grinder operating as swiftly, efficiently, and reproducibly as the Mixer/Mill but capable of loads up to 100 ml compared to 10-25 ml. The SHATTERBOX swings its container like a hula hoop at about 900 rpm. Inside the closed hardened steel dish a heavy ring and puck (also of hardened steel) batter the sample, reducing the typical one to usable fineness in 3 minutes or less.

The SHATTERBOX appears to be equally suitable for grinding the same wide ranges of brittle materials now ground in our Mixer/Mills. These include rocks, ores, slags, many metals, ceramics, oxides and finished products. The table below illustrates the results on chunks of blast furnace slag. Note the reproducibility of particle size distribution, so essential to accurate analyses.

Shatterbox Grinding Tests on Blast Furnace Slag

Four 25 gram samples each run for 3 minutes

Mesh	*% through screen*					
+100	2	2	2	1	0*	2**
—100	98	98	98	99	100*	98**
—200	89	89	87	89	98*	80**
—325	68	68	59	71	70*	48**

* 50 grams run for 7 minutes

** 100 grams run for 15 minutes

If it is the next larger size you want, you're a step ahead of us again. SHATTERBOX production is now rolling, however, and if this is the size you've been waiting for, it is ready to take its useful position in your laboratory.

8500 **Shatterbox, grinder and blender,** specify 115v. or 230v., 50-60 cy., 1/3 hp motor, with timer 0-15 minutes; net weight 125 lbs., boxed 200 lbs. .....Each **$925.00**

8501 **Grinding Container,** hardened steel.....Each **$200.00**

8502 **Grinding Container,** hardened steel, provisions for filling with inert gas; gasketed and held closed with four thumb screws .....Each **$245.00**

SPEX INDUSTRIES INC.
P. O. Box 98
Scotch Plains, N. J.

Return Requested

SPECIALISTS IN SPECTROSCOPY

BORON CARBIDE MORTARS and PESTLES

Boron carbide is one of the best materials for hand grinding. Possessing a hardness close to diamond, it is also extremely inert, resisting attack by most acids and alkalies. In addition, boron carbide is unbonded so that the only possible metallic contaminating element is boron itself. Here it differs from, say, tungsten carbide which is usually bonded with cobalt. The material from which the mortars are produced is made by the Norton Company. Compressed under great pressure, its density approaches the theoretical value.

The smallest mortar is mounted in a removable plastic base for ease in handling; 2" d. and larger are encased in stainless steel. Pestles are attached to an aluminum handle.

We can supply mortar and pestle combinations with cavities ranging from 1/2" to 3" in diameter. Also available are double cavity mortars—a crater on both ends of a cylinder. Please write us for price and delivery information on these special sets.

3201 **Mortar and pestle, boron carbide.** Mortar cavity 1/2" d. by 5/32" deep, highly polished. Pestle 1/4" d. ..... Set **$ 46.00**

3202 **Mortar and pestle, boron carbide.** Mortar cavity 1" d. by 1/4" deep, highly polished. Pestle 9/16" d. ..... Set **$ 86.00**

3205 **Mortar and pestle, boron carbide.** Mortar cavity 1-1/2" d. by 3/4" deep, highly polished. Pestle 9/16" d. ..... Set **$268.00**

3203 **Mortar and pestle, boron carbide.** Mortar cavity 2" d. by 1" deep, highly polished. Pestle 9/16" d. ..... Set **$405.00**

3204 **Mortar and pestle, boron carbide.** Mortar cavity 3" d. by 1-1/2" deep, highly polished. Pestle 1" d. ..... Set **$470.00**