

The**SPEX INDUSTRIES, INC. · 3880 PARK AVENUE · METUCHEN, N. J. · ☎ 201-549-7144****Speaker****THE STABILIZED PLASMA JET FLUID ANALYZER**

By A. J. MITTELDORF and D. O. LANDON

COMMERCIALY available for only a year, the plasma jet is already considered one of the most significant advances made in recent spectrochemical technology. It appears applicable to the analysis of all gases and liquids: organic, inorganic, flammable, corrosive or just plain water. With little or no preparation, the fluid is aspirated into a chamber then arced with ordinary dc as a vortex of gas drives the resulting mixture through an orifice. The plasma jet offers the most sensitivity of any liquid method known. It is the most precise of any spectrochemical source known.

Like all solution techniques, the plasma jet has the built-in advantage of ease of sample and standard preparation. Synthetic reference materials are conveniently made at any concentration range with whatever elements desired, with little fear of contamination. Compare this with the tribulations of trying to wedge known amounts of elements into molten metal where all nature appears to be at odds with you. Volatile elements disappear up the chimney. Reactive ones stubbornly oxidize then float defiantly on the surface. Refractory metals demand temperatures so high they must first be alloyed to reduce their melting point.

This is not to say that the preparation of solution standards is always a snap. Tin is often incompatible in acid mixtures and may precipitate unexpectedly. With organometallics, such as those used to prepare synthetic petroleum products, the task of solubilizing a string of elements often entails a good deal of frustration. A few days after you have heaved a sigh of relief at finally getting everything into solution, a suspicious deposit appears in the bottom of the container.

Yet, all considered, the preparation of solution standards is far easier and less prone to matrix effects than either powders or metals. Until the advent of the plasma jet, however, the spectrochemical analysis of liquids was always sensitivity-limited. The spark and interrupted arc were the only sources which could be directed to the sample. A dc arc would be squelched almost immediately by the steam generated during the burn. With flammable materials, the power of even the spark source had to be throttled to prevent ignition. Otherwise the source would revert to a flame rather than the desired electrical spark.

The plasma jet adds sensitivity to the convenience of liquids and at the same time contributes a bonus in stability. Conservative estimates by a good many spectrographers place its sensitivity as ten times as great as any other solution technique, fractional ppm being the detection threshold for most elements reported. As for stability, nobody has a figure to report because the jet is more stable than any current method of checking it.

There are still more advantages. The plasma jet operates at electron temperatures of 8,000-10,000° K, at dc currents employed routinely in the spectrographic laboratory. Well above the normal dc arc, these temperatures seem to be responsible for reducing matrix effects significantly; the actual extent is, at this time, not known, however.

With the jet, trace elements in alcohol and acetone have been detected. These highly flammable materials behave as though they were non-flammable and there is little danger that the sample, kept cool in its container, will ignite. OH bands have been seen in water and alcohol but not in carbon tetrachloride, inviting the spectrographer to work up a method for, say, determining water in organic materials. Plastics such as polypropylene may be analyzed after being dissolved in hot decalin and aspirated from a heated stage. One can determine trace elements either deliberately added to bolster properties like resistance to ultraviolet or present as unwanted impurities.

The jet is already being groomed for on-stream analysis. Because the radiation is so constant, it may be possible to record instantaneous intensity values rather than the usual integrated intensity ratios. Owen hopes to determine U_{238}/U_{235} in UF_6 gas in this fashion (1).

BEFORE delving into the rather nebulous beginnings of the plasma jet as a spectrochemical tool, it might be well to describe its earlier history. In the 1920s, H. Gerdien (2) in Germany developed the first plasma jet shown in Fig. 1.

After a dc arc is struck in a closed chamber between a graphite rod and a ring, a tangential stream of water or noble gas blows the arc through the orifice of the cathode. In so doing, the arc is heated for two reasons. The cooling effect of the water makes the outer shell of the arc less conductive, thermally pinching the current into the center. A magneto-hydrodynamic pinch then constricts the arc still further jolting the plasma temperature still higher. This latter effect is perhaps better known by its counterpart in wires. A hundred years ago Faraday discovered that current flowing in the same direction in two parallel wires would cause the wires to be attracted magnetically. Similarly, the positive ions in the plasma are attracted, incidentally keeping the stream away from the ring which it would surely vaporize otherwise.

Interest appeared to have lapsed in the plasma jet until the 1950s when its high temperature potentialities began to be appreciated. Here was a device which might alloy reluctant metals in the vapor phase; release metals from their ores; cut metals more efficiently than an arc; machine refractory metals; spray refractories on to base metals. Some of these are, indeed, commercial realities today.

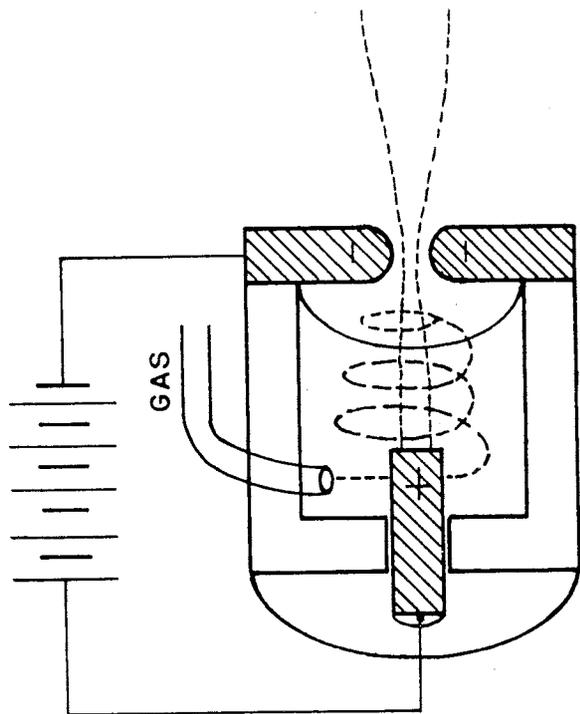


Fig. 1—Initial design of plasma jet. Note pinching of discharge at cathode protecting it from excessive erosion.

In 1959, Margoshes and Scribner (3) in the United States and Korolev and Vainshtein (4) in Russia independently hit upon the idea of analyzing liquid samples by atomizing them into the jet stream. Fig. 2 below depicts the almost identical schemes devised by each pair of researchers:

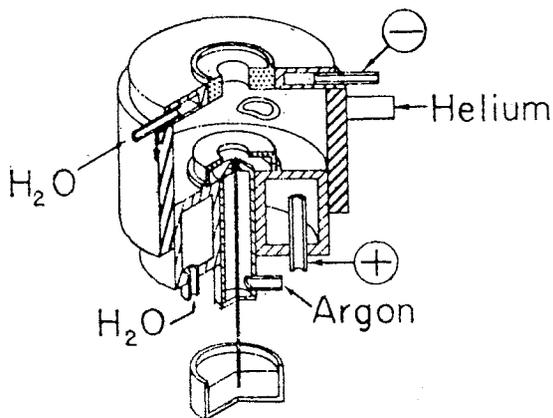


Fig. 2—Original design of Margoshes and Scribner for introducing liquids for analysis by the plasma jet.

Both the Americans and Russians showed that spectra could be produced from the aspirated liquid. As initially envisioned, however, the plasma jet proved to be unstable and unevenly intense. After squeezing through the cathode ring, the jet swelled, lost intensity and tended to swirl erratically. Owen's (5) contribution corrected this by adding an external cathode as depicted in Fig. 3.

Here the arc, first striking the cathode ring, is transferred to the upper tungsten rod maintained at the same potential. The external jet in this arrangement is quite stable and uniformly intense along its length which can be stretched out to several cm.

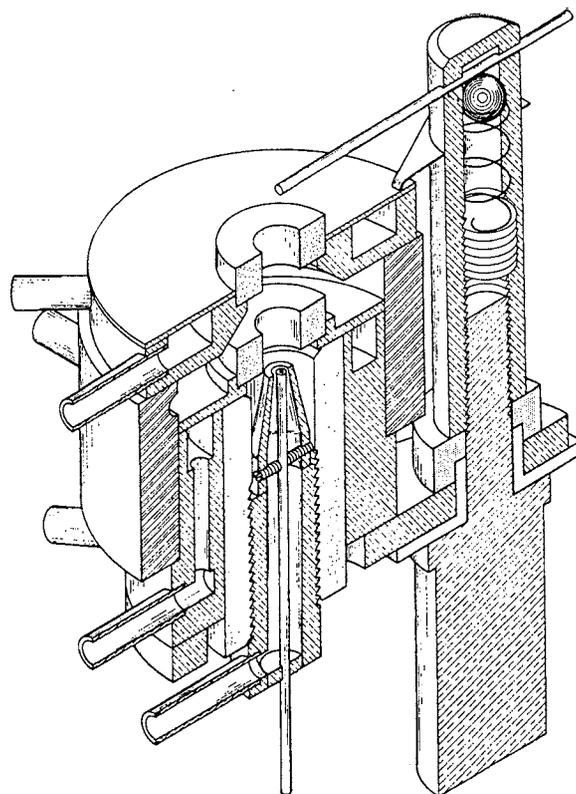


Fig. 3—Owen's design in which the top of the chamber is connected electrically to an external cathode.

Exchanging ideas with Owen, Scribner and Margoshes (6) proposed two changes. The upper cathode was placed axially in line with the center of the graphite ring. Secondly, this ring was allowed to assume a potential dictated by the ion stream inside. It was left to "float" electrically and has since become known as the control ring.

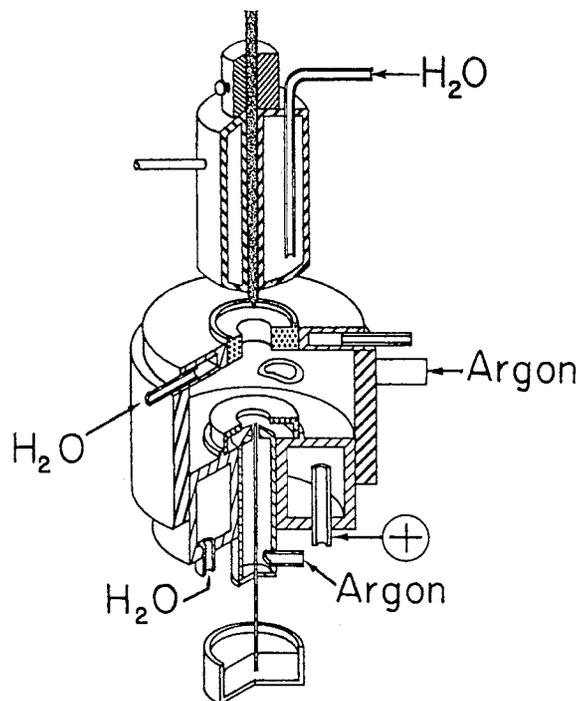


Fig. 4—Scribner and Margoshes prefer to call this a "gas stabilized arc" device, its external cathode differentiating it from the plasma jet.

The Spex version, designed to fit in the jaws of most arc stands is essentially the same as that of Scribner and Margoshes but modified for ease in disassembly and convenience in use. Also, the hole in the anode ring is stepped as later suggested by Sirois and Owen. Small at the bottom, large at the top, the hole in the anode ring acts as a stop when the cathode is inserted. The most convenient way to strike an arc is with a spark discharge of the type used in most commercial dc arc sources. If, however, the upper cathode rod is pushed down too far, the arc might strike the atomizer tip, damaging it. The small hole also protects the atomizer from radiant heat during the actual arcing period.

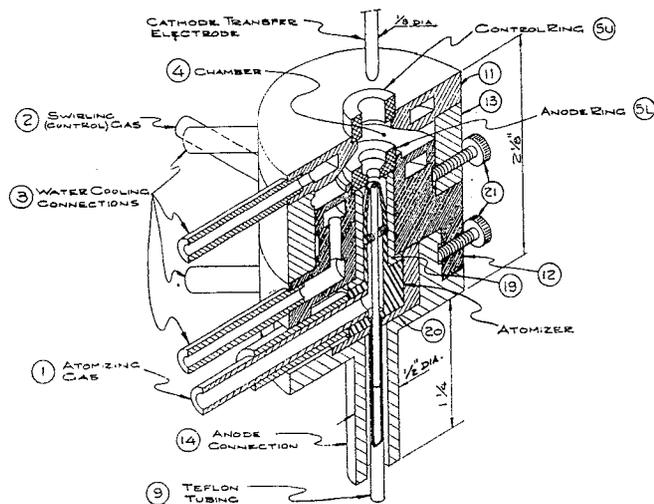


Fig. 5—The Spex No. 9030 Plasma Jet Solution Analyzer fits most arc stands. Like that in Fig. 4, the cathode is external and the upper ring "floats."

Margoshes and others feel it wise to distinguish clearly between the plasma jet and what they refer to as the gas stabilized arc. The former is of the original type where the plasma, developed inside a confined space, is then forced outward by gas pressure alone. The latter utilizes an external cathode as a return for the electrical path. By this definition, the Spex instrument falls in the second category as a gas stabilized arc. It seems to the authors, however, that both can be considered as versions of a plasma jet. Plasma signifies an ionized gas and the jet, whether blown mechanically or given an assist by an external electrical path, is still a jet. Properly or not, like our democracy which in reality is a republic, the Spex Plasma Jet Solution Analyzer No. 9030 seems destined to remain so named.

While our stabilizing approach has been to use an external electrode, a Japanese researcher, Yamamoto (7), apparently achieved stability through an external magnetic field. As shown in Fig. 6, the configuration of his plasma jet differs in that the solution is atomized in at an angle and the control gas, instead of being introduced tangentially, is directed through an annular orifice around the consumable electrode which, incidentally, is made the cathode rather than the customary anode. It appears, however, that, in the Yamamoto device, apart from stability, the plasma light intensity tapers. In the American stabilized jets, the distribution of intensity along the length of the plasma is reasonably constant.

As already indicated, the operation of the plasma jet involves two gases. While the last word has not, by any means, been heard on which gas to use when, it appears that the noble gases with their high ionization potential are preferable. Margoshes and Scribner, in their earlier work, used Ar for

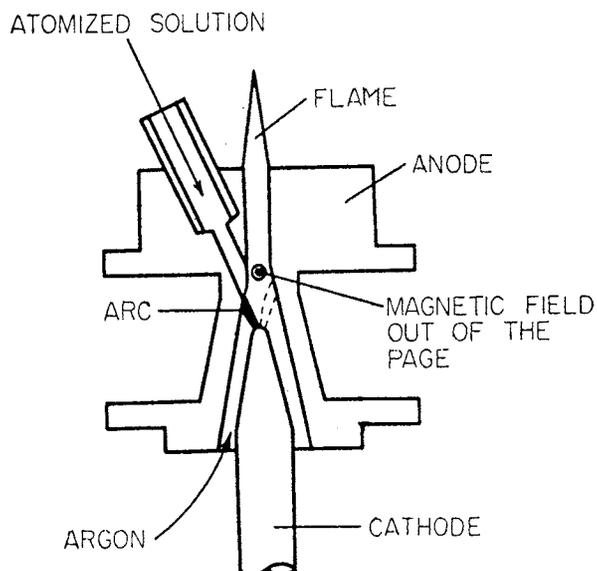


Fig. 6—Yamamoto's method for stabilizing an arc through an external magnetic field. The dashed lines show the arc when no magnetic field is present.

both the control and atomizing gas. Most of our own work has been with He for control, Ar for atomizing. Now Owen, recommending that He may be used for both, has designed a compact control package which accurately regulates the flow of He through micrometer-head needle valves rather than the ball-float flow meter. With his device, pictured in Fig. 7, a small stream of gas is allowed to flow constantly to protect the capillary tip from accidental arcing. Also, the unit has a by-pass valve which assists in transferring the arc to the upper cathode.

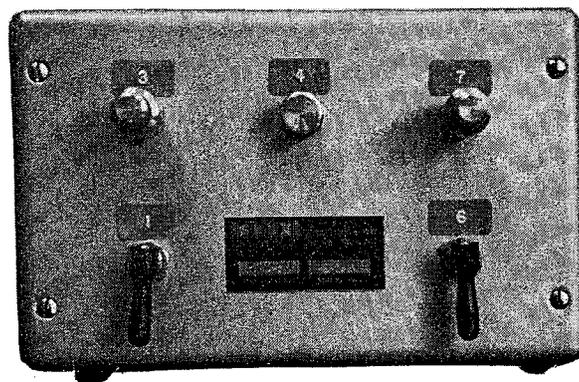


Fig. 7—Spex-Owen Manifold No. 9032.

The high temperature of the jet changes the ordinary arc *raies ultimes* as will be noted later in this article. Like other relatively unexplored areas, however, a good deal more careful work is required to establish the exact sequence of persistent lines and correlate these with various combinations of gases.

What to use for an upper electrode is another unanswered question. The jet column is usually kept between 0.5 and 1 cm long and, over most of the length, the intensity is reasonably constant. With a tungsten cathode, only within a mm or so from the cathode do W lines appear. Thoriated tungsten seems to improve the jet stability. Graphite has the advantage of being inexpensive but ablates and burns away more rapidly than W.* Further, it burns away most rapidly, not at the

*With organic materials, a graphite cathode grows longer as more graphite, probably pyrolytic, deposits during the burn.

end as one would expect, but about 1 cm from the end. If burned too long, the graphite end may drop off altogether and, if it is one of those days, may land inside the jet assembly. All in all, it appears that tungsten or an alloy may prove, in the long run, to be the best choice of cathode.

High Temperature Alloys

At General Electric's Flight Propulsion Laboratory in Evendale, Ohio, Mr. E. H. Sirois worked extensively with the Spex Plasma Jet for determining rare earth additives in high-temperature chromium-base alloys. Even in the low ppm range, such additives are known to affect the oxidation resistance, strength and ductility of the base alloy. How to keep tabs on minute traces accurately and precisely plagued the spectrographic laboratory until the jet was assigned the task. All other solution techniques failed on two counts: lack of sensitivity and/or precision. Methods involving solid samples did not permit ready standardization. It is not easy to add a few ppm of an element to a complex alloy and to be sure either that all of it has dissolved or that the base material was free of it to begin with.

Relying upon solutions which could be nicely synthesized and standardized and the plasma arc which is known to be more sensitive than older solution techniques, the jet appeared promising. A good deal of development work followed but without going through the pitfalls, Sirois eventually found that he could determine 10 ppm Yt with a precision of ± 0.5 ppm. Taken up ultimately in a 10% acid mixture (largely perchloric), the entire alloy was aspirated with Ar and swirled with He. The cathode was of graphite; Yt 3710A was exposed on SA1 plates for 30 seconds on a Wadsworth spectrograph.

Sirois states that, in his opinion, the plasma jet can be made so stable that it is unnecessary to employ correction factors such as internal standardization. For example, in determining Yt, a plot of the intensity of its 3710A line *vs.* concentration served as the analytical curve. To be sure, a good deal of preliminary work was required before the excellent precision and sensitivity evolved. In order to establish conditions which would give a constant atomizing rate, Sirois set up a liquid flow meter as shown in Fig. 8. He takes the unit, its hoses intact, out of the arc stand and dips the Teflon tube into the glass flow meter. The atomizing rate is then adjusted to obtain a fixed position of the ball. This procedure is especially valuable in switching from one atomizer to another in which the bore diameter may not be identical.

Further, finding the timer on his source unit erratic, he finally resorted to the use of a stopwatch to measure the exposures. Taking such precautions and doing much preliminary work, Sirois was rewarded with a method easily 10 times as sensitive and far more precise than any solution method known.

Blood

With the skyrocketing number of medicines and drugs on the market has inevitably come a need for greater government control to prevent deleterious reactions. For new drugs the Federal Drug Administration is placing the burden of proof of both effectiveness and harmlessness on the manufacturer. The use of heavy metals and others is scrutinized for possible absorption into the blood stream of the patient.

Along these lines, Mr. George Heinze of Johnson and Johnson Research Laboratories in New Brunswick, N. J., was assigned the problem of determining what if any Al or Mg would be absorbed in the patient's blood upon his taking oral doses of antacid formulations containing these metals. Blood from

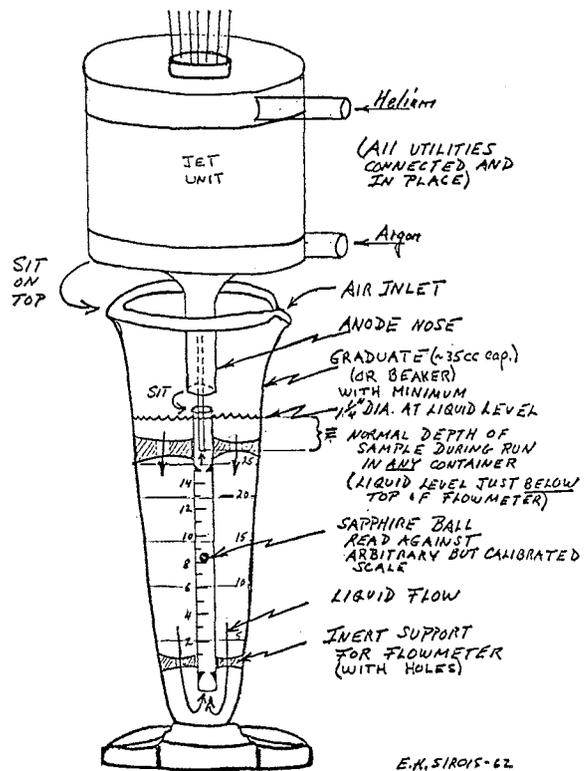


Fig. 8—Sirois apparatus for calibrating aspiration rate.

volunteers was analyzed colorimetrically in before-and-after tests but the least amount detectable above blank concentrations was around 10 ppm, far above the accepted normal values for whole blood. Over a period of several months, many other methods were tried, but, in all of them the blank level from reagents and glassware disrupted the determination. Spectrographic techniques in which the blood was ashed beforehand fell under the same limitation.

Noting a spectrographic technique which did not depend on ashed samples, Heinze was anxious to try the plasma jet and one day visited our laboratory laden with several pints of blood. To the blood had been added an anti-coagulant and a hemolizing agent to break up the red cells. Without further preparation, the samples were run in a 15 ampere dc jet using an Ar-He system. Initial tests differentiated a blood sample to which 3 ppm Al was added from its blank, (0.2-0.5 ppm). Al 3962A line was seen on an SA3 plate after an exposure of 60 seconds during which about 1 ml of blood was aspirated.

Sea Water

The bountiful ocean has fascinated chemists as well as explorers through the ages. Here is a wealth of water and minerals challenging the resourceful to be separated and claimed. Over the years, pushing his techniques to ever more sensitive levels, the analytical chemist has discovered in the sea most of the elements in the periodic chart. One of the pioneers in the use of the Stallwood Jet, Dr. O. I. Joensuu of the Marine Laboratory of the University of Miami, decided to see what the Plasma Jet could do with sea water. His current interest is in strontium and, with no separation or concentration, he is able to detect better than 1 ppm at the 4078A line. A Dual Grating spectrograph registers the line from a 20-ampere discharge for about a minute. Joensuu reports that the sensitivity for Sr as well as for Mg and the rare earths is at least ten times as good in the plasma jet as it is in the porous cup or rotating disc techniques.

Plating Baths

With its unique combination of high corrosion resistance, electrical and thermal conductivity, excellent bonding properties and stability at high temperatures, Au plating is turning out to have wide industrial applications. Unlike in jewelry, where only appearance is a criterion, here the physical characteristics of the plated surface are uppermost. It must be uniform in thickness over whatever complicated surface is to be plated. It must be adherent, free of imperfections, smooth and remain so under the conditions to which the part is to be subjected.

To provide for such rigorous specifications, it has been found that not only must the Au content in the bath be maintained constant but that some trace elements must be kept below ppm levels. For example, a few ppm of Cr seems to affect the throwing power of the bath—the ability of the Au to plate uniformly in crevices, around corners, at sharp edges, etc. Ag, again at very low concentration, produces a flaky, non-adherent coating.

Until recently, Richard Hunsberger, of Western Electric in Laureldale, Pa., had used a rotating electrode for the analysis of Au plating solutions but, having done the necessary groundwork, is about to switch to the plasma jet. He finds that Cr can be detected at levels at least ten times as low in the jet. Equally of interest, Au itself seems capable of being assayed. Where hours were previously needed to plate out the Au and weigh it on a Pt cathode, the plasma jet can do the job in minutes, preliminary results indicating an accuracy well within the requirements. Mr. Hunsberger adds germanium dioxide as an internal standard to the gold cyanide bath for the determination.

Copper Nickel Alloys

Joseph Irwin of Anaconda-American Brass Research Laboratories in Waterbury, Connecticut, is currently experimenting with the plasma jet for the determination of Fe, Ni and Mn in 70:30 cupro-nickels. A nitric acid solution of the alloy is employed. Although still in the developmental stage, the method shows considerable promise, excellent analytical curves having been obtained for these elements. One annoyance Mr. Irwin experienced is with an old dc arc source. Despite its 220v. open circuit voltage, the source occasionally does not sustain the plasma arc during an entire burning period. It appears necessary to add an inductive ballast of around 5 millihenries to the dc output but before the spark ignition circuit, a practice established in most modern arc units.

Petroleum Products

Our own work has been principally with gas oils, the intermediate refinery materials from which gasolines and other petroleum products emerge. In a refinery, two elements stand out as being particularly troublesome: S and V. The former with its habit of reverting to sulfuric acid and corroding reaction lines, is readily determined and so kept under control. On the other hand, vanadium's specialty is to poison the catalytic cracking catalysts that convert gas oils to modern gasolines. Traces down to the range of fractional ppm eventually force the shut-down of a cracking tower in which the catalyst must be replaced. Accordingly, refinery operators have placed ever more stringent limitations on the V content of crude oils, straining every known method of analysis.

Before the plasma jet was found to be applicable, the best method of determining V involved ashing the sample and run-

ning the ash spectrochemically. Not only is this a lengthy procedure, but V is lost to varying extents in the ashing depending on the particular organo-metallic constituents present. No other methods including atomic absorption are capable of dealing with elements that form refractory oxides such as Be, Al, Si, V, and Nb.*

With the cooperation of spectrographers in several oil companies who supplied standards, we set up the Spex Plasma Jet Solution Analyzer for the analysis of gas oils. Table I is a summary of conditions eventually decided upon:

TABLE I

Spectrograph	Home-built, 3.4-meter Ebert with 15,000 1/in grating, 2nd order; 30 micron slit SA #1
Plate	18 ampere dc, lower electrode positive
Source	Control—He, 10 l/min as indicated on gage calibrated for air
Gases	Atomizing—Ar, 3 l/min
Gap	6mm
Exposure time	10 seconds preburn, 60 seconds exposure
Atomizer	Large-bore, 0.018" dia. (Spex 9030-4140)
Flow rate	Under the conditions given, approximately 1 g/min aspirated
Cathode	Graphite, 3/16" rod

Table II lists the detectability limits for each of the five elements sought.

TABLE II

Sensitivity for Elements in Gas Oils

Element	Analytical Line A	Minimum conc. detectable ppm
V	II 3110.7	0.05
Cu	I 3247.5	<0.05
Fe	II 2599.4	<0.2
Ni	I 3002.5**	<0.3
Pb	I 2833.1	<0.3

It is evident from Table II that we, too, have found the plasma jet approach at least ten times as sensitive as any other solution method. In fact it appears to be even more sensitive than most powder-dc arc methods. We were, naturally, interested in attaining the utmost in sensitivity and investigated several parameters in this connection. The one obvious parameter was dc arc current. The data in Table III were obtained using a 1P28 photomultiplier instead of a photographic plate at the camera. In this and subsequent experiments the PM tube was run at 1000 volts from a Hamner Model N-4035 High-Voltage Supply and read out on a Keithley Model 414 Microammeter.

*It may be interesting to point out that electron spin resonance appears to be a method aside from the one soon to be described but it involves exceedingly costly equipment quite apart from the emission spectrograph standing by in almost every major refinery.

**Dr. S. A. Bartkiewicz, Esso Research Laboratories, Baton Rouge, La., reports that in preliminary experiments on white oils he was able to detect 0.22 ppm of nickel at the V-1 line, 2287.1A using a photoelectric spectrometer. It would appear that, with a photographic instrument and a Q-2 plate, the detection limit for this line would be better than our figure of 0.3 ppm.

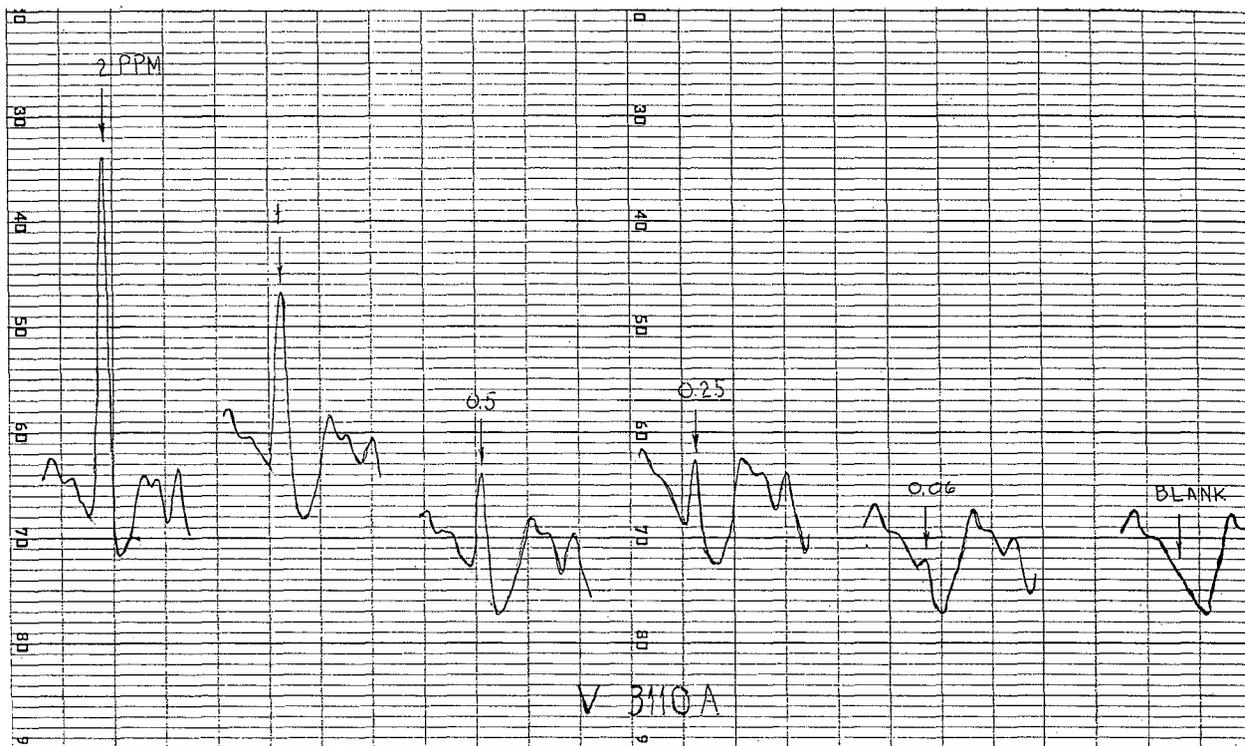


Fig. 9—Tracings of V3110 at levels from 0.06 - 1 ppm.

Fig. 9 depicts unretouched tracings taken on a recording densitometer of the V 3110A line at different concentration levels. Note particularly the difference between the 0.06 ppm line and the blank. Although this line is not visible, it is repeatedly found with the microphotometer. Note also the repeatability of the background structure from sample to sample.

The data in Table III were obtained by running a sample of mineral oil and Varsol free of V, then re-running it after adding 100 ppm of V.

TABLE III

Signal-to-Noise vs. Current

dc Current amps	Relative Intensity of background	Signal-to-noise, V 2908.8A: background
10	1.0	1.41
15	1.3	1.80
20	1.7	1.85
25	2.0	1.87

It will be noted from Table III that, while the intensity of the background rises roughly with the increase in current, the line: background levels off at around 20 amperes. Although data are not presented, all five elements behaved in much the same fashion,* dictating our choice of that current for more subsequent work.

Another important parameter is the flow rate of the tangential gas. On theoretical grounds, the intensity of radiation should, and indeed does, increase with the gas flow. The greater the rate, the greater the pinch and the hotter the central core of the plasma. From the spectrochemical standpoint, however, the significant factor is not intensity but the line-to-background

*This is not to say that all elements in all solutions behave similarly. A water sample containing P showed quite the reverse effect, its signal-to-noise ratio diminishing rapidly with increasing current above 12 amperes.

ratio. As shown in Fig. 10, there is a continuous improvement for iron and for vanadium. Two of the others are unaffected and copper shows an increase to a constant level. It appears that the relationship is explainable on the basis of excitation level. Only the iron and vanadium are ionized lines. The optimum intensity of these appears to be above the flow rate tested. The others are all unionized arc lines. Obviously, a comprehensive study of this parameter is called for.

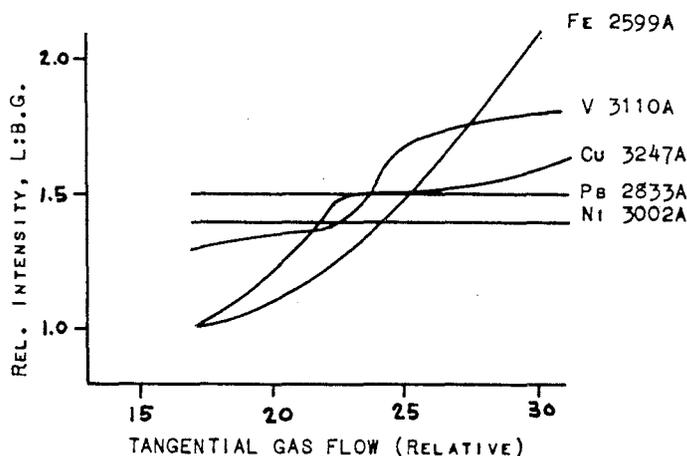
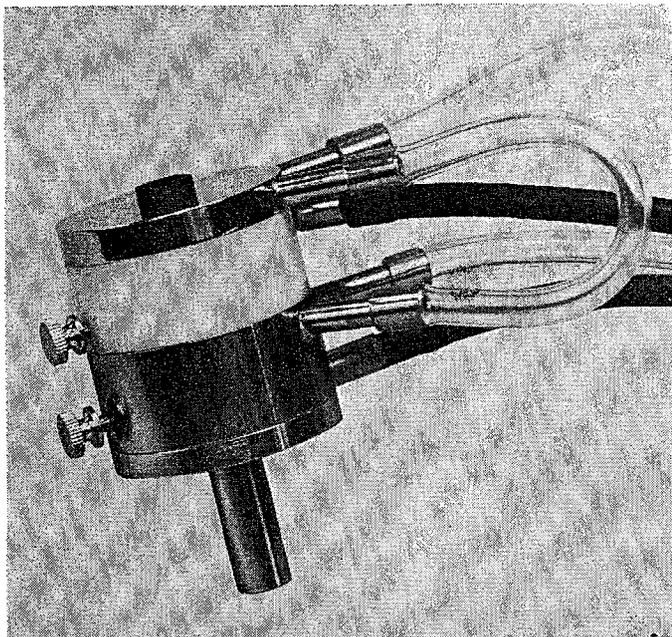


Fig. 10—Line: Background ratio as a function of the tangential gas flow.

WE are most grateful to all the spectrographers upon whom we called for help in assembling this plasma jet story. Our special thanks go to E. H. Sirois and L. E. Owen for their constructive critical reviews of our draft. To Owen's own comment that "... none of us would be likely to be using a plasma jet in spectrochemistry if Marvin Margoshes had not started the ball rolling," we would add only our confirming tribute.



PLASMA JET ANALYZER PRICE LIST

- 9030 **Plasma Jet Solution Analyzer**, fits in most arc/spark stands. Includes water-cooled jet assembly with atomizer (medium bore unless otherwise specified), and graphite upper control rings, graphite lower anode rings, Teflon sample holder\$249.00
- 9031 **Plasma Jet Unit in Stand**. Complete excitation unit including 9030 atomizer assembly; vertically adjustable, water-cooled upper electrode holder; interlocking door switch; neutral filter on viewing window\$750.00
- 9031A **Adapter** to fit user's optical bar. Specify 1) manufacturer of bar and spectrograph; 2) vertical height from top of bar to center of optical path; 3) direction (left or right) of light as operator views Plasma Jet Unit\$ 45.00
- 9032 **Spex-Owen Manifold**, micrometer-controlled gas system for using single gas in both atomizer and control sections of Plasma Jet; purging device for reducing inter-sample contamination...\$250.00
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- 9030-7 **Heated solution container**, aluminum, 115v., 50-60 cy., useful for analyzing substances which dissolve at elevated temperatures, or to reduce viscosity of some liquids, or melt materials such as waxes\$ 55.00
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10 \$ 40.00

Note: If you are ordering the Plasma Jet along with our 9010 Arc/Spark Stand 9028 Water cooling connections, 9019 and 9029 Flow meters should be ordered at the same time so they can be built into the stand.

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ERRATA

Within a few days after a SPEAKER is mailed out, we routinely receive a card from a sharp-eyed grammarian whose self-imposed duty it is to guard the purity of the English language. (We leave it to you to find at least one such error in the last issue.) Then, almost equally inevitably, comes the letter to turn our faces red in embarrassment. This is from a real friend who, having spotted an error which may seriously affect the text, wants to bring it to the attention of others who may slip on it. No matter how many amateur proof-readers read and re-read the galley and final proof, some error seems to worm its way in obtrusively. In Vol. VII, No. 4, two were belatedly discovered in Table 4 by several spectrographers to

whom we are indeed grateful. Would you kindly cut out the following corrected table and tape it over the one in that issue? Thank you.

TABLE 4

Analysis of Pure Materials

Base	% of each element in base	Dilution to add 0.500% of each element	Dilution to add 0.100% of each element
Spex Mix	1.28	1 base+ 1.56x	1 base+ 11.8x
RESM	5.28	1 base+ 9.56x	1 base+ 51.8x
NMSM	9.32	1 base+ 17.6x	1 base+ 92.2x

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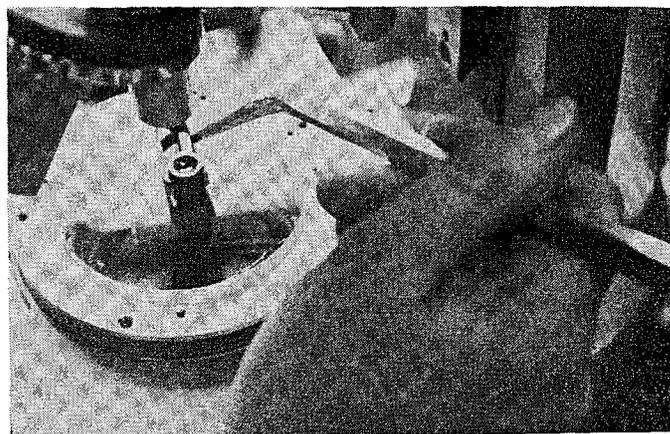
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A tiny boron carbide mortar and pestle set is now available for use in grinding samples under the microscope. Only 3/8" in diameter and 1/2" high with a cavity 1/4" in diameter, it is readily held on the microscope stage with the self-gripping tweezers supplied. The pestle has an offset handle facilitating manipulation while the sample is viewed. In this application, unbonded, highly-polished boron carbide with its unique combination of properties—a hardness surpassed only by diamond, inert to practically any material, tough and strain-free—is unsurpassed as a grinding medium.

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3206 Mortar and pestle, boron carbide. Mortar cavity 1/4" d., highly polished. Pestle, 1/8"d. cemented to 60° offset aluminum handle....Set \$ 50.00

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