

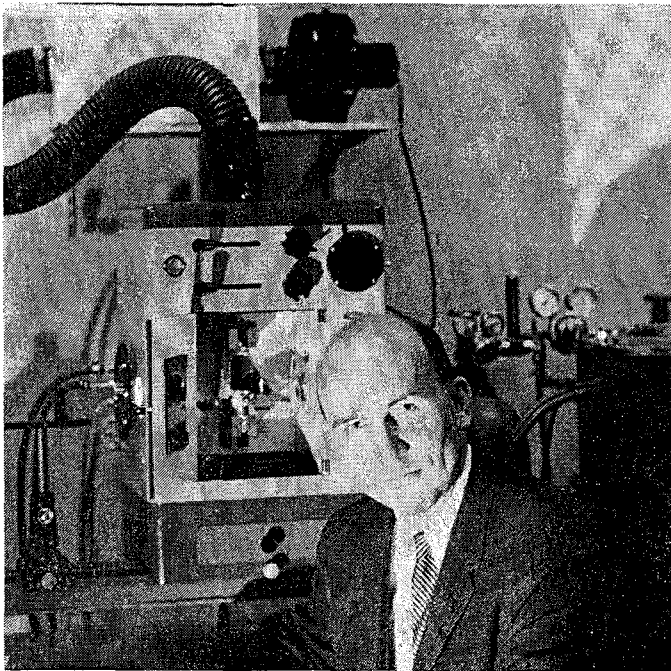
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## A UNIVERSAL METHOD FOR SPECTROCHEMICAL ANALYSIS OF FERROUS ALLOYS

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A method has been developed for direct spectrochemical analysis of any ferrous alloy for all of its constituents, with the prior knowledge only of the approximate percentage of iron in the sample. It makes use of a "black-burn" point-to-plane spark in an inert gas. Instead of plotting intensity ratios, the ordinary practice in preparing analytical curves, a corrected ratio based on the iron content is used. The method has been applied successfully on some thirty-two different standards, ranging from 1.4% to 97% in iron content. The principles employed are applicable equally well to other matrices, such as copper, aluminum, zinc or nickel.



In the opinion of many who first heard it delivered in College Park, Md., in June, 1962, this paper by Arno Arrak represents a significant advance in the science of spectrochemistry. Already corroborated in several laboratories, its importance lies in the evidence that the matrix effect, that omnipresent intruder, may have at last found its master. If so, the need for both standards and analytical procedures could be slashed considerably. Although the proof of his method is based on extensive studies with ferrous alloys, it appears to be equally applicable to others as well.

Among his numerous articles, Mr. Arrak has published several other noteworthy ones of lasting impact on the spectrographic community. Perhaps the most striking one is a technique for quantitatively focusing a spectrograph (*Spec. Acta.*, 12, 1003, 1959). As far as we are aware, it is the only method by which the researcher can tell exactly how far the slit must be moved to attain critical focus. With little more effort, he can chart the full focal curve of his instrument and so appraise whether the entire spectrum is in focus at the same setting.

At present a member of the Quality Control Department of Grumman Aircraft in Bethpage, L. I., Arrak received his bachelor's degree from Dartmouth and in 1959 his Master's in chemical engineering from Pratt Institute. His introduction to spectrochemistry was through additional graduate studies at Columbia University.

for which standards are available, there is always some doubt about the validity of the analysis, even when all elements present can be determined. To overcome these limitations a means of eliminating the matrix effects is desirable, and this has been a goal of spectrochemical research for more than a decade.

Recently, a number of papers have appeared on the use of controlled atmospheres to reduce matrix effects (1-5). Although no universal method evolved from this work, considerable improvement over results obtained in air was reported by most authors. Our investigation of the inert atmosphere spark was prompted by these encouraging reports in the literature.

Both nitrogen and argon atmospheres were studied. The sparking conditions chosen produce a so-called "black burn"

**A** METHOD of spark analysis universally valid for all ferrous alloys would have great value in simplifying the spectrochemical analysis of steels. More effective use could be made of a limited number of standards. More diversified and unusual compositions could be analyzed with accuracy and speed. A major obstacle retarding the development of such a method has been the interference by so-called "matrix effects." It is generally believed that these effects are mainly due to variable surface oxidation rates of different alloys, to differences of metallurgical structure, and to the dependence of the internal standard line intensity upon the iron content of highly alloyed steels. Because of this, separate sets of standards and of analysis procedures have been required for low alloy, stainless, tool, and high temperature steels. As a result, when a new alloy comes along that does not fit into any of the categories

(2), a good indicator of adequate shielding from air (Fig. 2). This commonly appears in an inert atmosphere, although it may appear, under special conditions, in air (6). Introduction of the gas into the spark gap is with a device shown in Fig. 1, designed by F. E. Hurley of Grumman. It is similar to an earlier design of J. Marjoribanks and W. Walsleben of Grumman Quality Control Laboratories (unpublished), which had been in routine use for some ten years; its principal application was in the analysis of aluminum alloys where good shielding was found to be essential for obtaining a satisfactory black

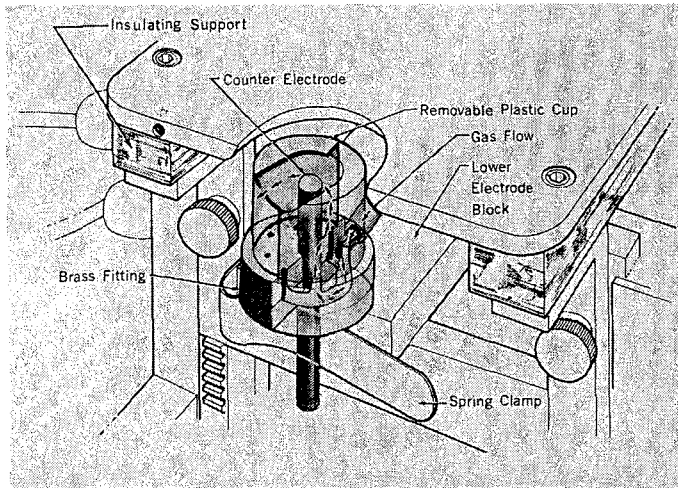
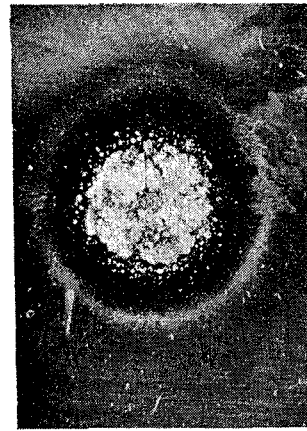


Fig. 1: Grumman method of introducing gas into the spark gap of the Petrey stand. The arrangement shown was designed by F. E. Hurley of Grumman Spectroscopy Laboratory. Gas enters the brass fitting from the rear and is made to impinge on the sample surface through small holes drilled into its top. The sample surface should be flush with the top of the plastic cup to prevent mixing with air.

burn. When used for sparking steels or nickel-base alloys with nitrogen or argon it produces uniform black burns on all samples sparked. The appearance of the black burn is independent of alloy type but it does depend upon the atmosphere, as may be seen from Fig. 2. Only when the samples are sparked in air is there an appreciable dependence of burn type upon the alloy sparked. Thus, low alloy steels and nickel-base alloys give two different types of burns in air as may be seen from Fig. 3. Apparently, this reflects the varying course of surface oxidation reactions during sparking of these samples, as well as the influence of the respective oxides upon the spark pattern formed. Further confirmation is the observation that burns on 18 Cr—8 Ni stainless steels resemble those on nickel base alloys, whereas burns on less highly alloyed stainless steels and on some tool steels show an intermediate, sometimes variable, character. The disappearance of these differences when nitrogen or argon is substituted for air suggested that an inert atmosphere should effectively eliminate the influence of surface oxidizability on analytical results. When the densitometric data on steels sparked in nitrogen and in argon were studied, it turned out, however, that systematic differences between analytical curves for low alloy, stainless, and tool steels persisted, even when the ratio method of plotting curves (7) was used. Since surface oxidizability as a factor was now presumably eliminated, it was suspected that excitation conditions within the discharge itself might vary and thus give rise to the observed inconsistencies in the data. To check this possibility the intensity ratio of Fe III 3013.125A: Fe I 3016.19A was measured in all spectra obtained up to that point. This ratio had been recommended as an excitation index for tool steels sparked in nitrogen by Majkowski and Schreiber (3). In our

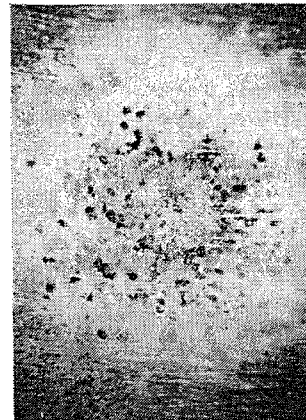


Black Burns  
in Argon

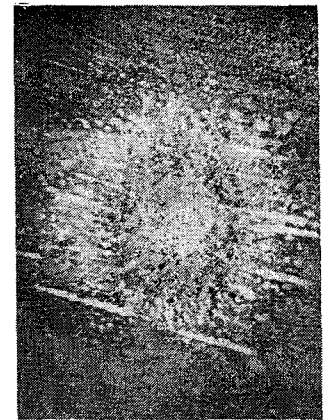


Black Burns  
in Nitrogen

Fig. 2. Black burns in nitrogen and in argon. The black deposit is graphite from the counter electrode. The structure and distribution of this graphite deposit depend upon the atmosphere as can be seen. It will form when sparking is carried out in an inert atmosphere under most conditions, although it may fail to form when the number of breaks per half cycle is high. The appearance and uniformity of the graphite deposit reflect the effectiveness of shielding by the atmosphere; if, in a series of burns there are one or two where the graphite failed to deposit or deposited very lightly, shielding was lost during that shot and the exposure should be disregarded.



Nickel Base and  
Stainless Steel in Air



Low Alloy Steel  
in Air

Fig. 3: Spark burns in air.

instrumentation, however, chromium and vanadium interferences prevented us from using it, and as alternates we chose the ratios Fe II 2813.61A: Fe I 3009.570A and Fe II\* 2495.860A: Fe I 3009.570A as excitation indices for nitrogen and argon atmospheres, respectively (8). Fluctuations of these ratios are not only small, but free of any systematic trends, an indication that the variation of excitation conditions cannot explain the observed changes in analytical curves with alloy type.

Metallurgical variations or the failure of the ratio method to compensate adequately for changes of iron line intensities with iron concentration, alone or in combination, might ac-

\*Although both an Fe I and an Fe II line are listed by Moore at that wavelength, the observed line behaves like a typical Fe II line.

count for these differences. Possibly some other minor influences, such as sample heating effects, surface irregularities, etc., might also contribute.

Of these effects, the ratio method can most readily be subjected to an experimental test. Relative intensities of lines selected as possible internal standard lines were plotted against the concentration of iron in the standards, without regard to the alloy in which the iron occurs. Among the standards was a wide variety of steels as well as some nickel alloys, included to provide the most severe limiting cases for study. Experimental points from the nickel base alloys were plotted with a square symbol. A point was plotted whenever a reading could be obtained on a line. Since the exposure time and excitation conditions for all spectra were identical the resulting smooth curve is a "constant time" analytical curve for iron which relates iron concentration to the intensity of the internal standard line used (Fig. 4). As may be seen, producing a smooth curve is only feasible with an inert sparking atmosphere; it fails when sparking is carried out in air, probably because of the disturbing influence of variable surface oxidation rates. It is clear that the "constant time" analytical curves for iron reflect the true dependence of the iron line intensities upon the iron concentration, for straight line relationships covering almost two decades on the concentration scale can scarcely be expected to arise by accident. For the ratio method to be valid, however, such "constant time" plots for the internal standard line should have a unit slope in logarithmic coordinates. None of the lines studied satisfy this condition, indicating that the ratio method will fail when these lines are used for internal standard lines.

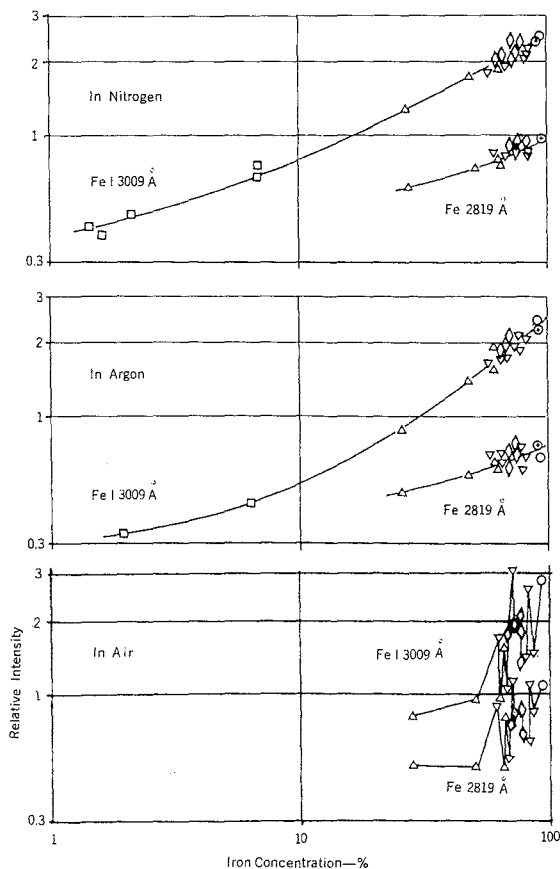


Fig. 4: Relative intensities of Fe 3009A and Fe 2819A as functions of iron concentration in nitrogen, argon and air atmospheres. Note that a smooth curve can be drawn only in the inert gas examples. Other iron lines were found to behave similarly.

Standard	Composition range and type
⊙ BAS SS1 — SS8	Low alloy steels: Fe 94-97, balance Cr, Ni, Mo, V, Mn, Si, Cu, Al, etc.
○ NBS 805A, 816A, 1161 — 1164, 1168	
◇ NBS 836 — 841	Tool steels: Fe 69-79, Cr 2-8, Mo .8-8, V .6-3, W 2-18, Co 3-12, Mn, Si, etc. balance.
▽ NBS 845 — 850	Stainless steels: Fe 62-83, Cr 3-24, Ni .3-25, Mn, Si, Mo, Ti, Nb, W, etc.
△ NBS 1184 — 1187	Stainless and high temp. alloys: Fe 27-66, Cr 16-21, Ni 9-24, Mo 1.5-6, W 1.4-2.4, also Co, Nb, Mn, Si, etc.
□ NBS 1188, 1189, 1191, 1192	Nickel base alloys: Fe 1.4-6.6, Cr 15-20, Ni 55-72, Mo 4-7, Ti 2-3, Al .7-1.5, Co 11-13, Mn, Si, etc.

### The Empirical Variable Internal Standard Principle

THE "constant time" analytical curves for iron can be reproduced from plate to plate when uniform sparking conditions are maintained. Since they are assumed to represent the true dependence of iron line intensities upon the concentration of iron, they form a basis for correcting the intensity ratios of analytical to internal standard line intensities for changes with the concentration of iron.

In order to effect this correction, the "constant time" analytical curve for the internal standard line used is first transferred to a working curve strip, which is then attached parallel to the intensity scale of the calculating board as shown in Fig. 5. To calculate corrected intensity ratios the percent transmittance of the internal standard line is not brought to coincide with 1.0 on the intensity scale of the calculating board (as is usually done), but is lined up instead with the iron concentration of the standard (or sample) on the iron correction working curve strip on the board.

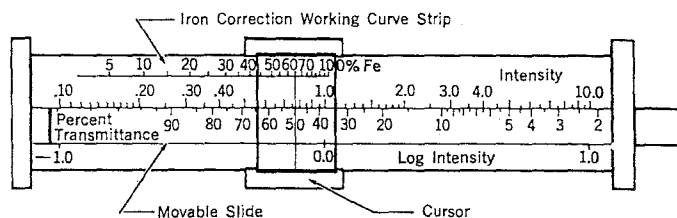


Fig. 5: Hypothetical calculating board with an iron correction working curve strip to be used in applying the variable internal standard technique. The hairline of the cursor is first brought over the iron concentration of the standard on the iron correction working curve strip on the board. The percent transmittance of the internal standard line for which the strip was constructed is then brought under the hairline and corrected intensity ratios are read off from the intensity scale in the usual manner. The drawing shows a setting for a standard containing 65% iron whose internal standard line reading is 50% T.

Analytical curves plotted in terms of such corrected intensity ratios are then universally valid, provided that metallurgical structure and other factors do not lead to additional matrix effects. Experience has shown that in order to use such curves successfully, the iron content of the material analyzed should

be known within two or three percent. While errors of this magnitude in the iron content do not affect the analytical results, an error of four or five percent (depending upon the alloy) may have some effect upon the derived concentrations of alloying elements.

In practice, all low alloy steels are analyzed without even correcting for their iron content, while high alloy steels are analyzed by first assuming an iron content typical of the alloy and then comparing this value to the iron value obtained by difference after concentrations of other elements have been calculated. If the assumed and calculated iron values do not agree within two or three percent, the process is repeated with a new assumed iron value until the concentrations of alloying elements converge to stable values. Usually one or two iterations are sufficient for this purpose.

THE spark parameters used are listed in Table 1 and information on the lines studied is given in Tables 2 and 3. Typical analytical curves, plotted in terms of corrected intensity ratios to Fe I 3009.570A, are shown in Figs. 6 to 9. When this line was too weak to read (as in nickel base alloys) the filtered part of Fe II 2592.779A or Fe II 2730.738A was read and "theoretical" corrected intensity ratios to Fe I 3009.570A were derived on the assumption that the relative positions of all "constant time" analytical curves remain fixed with respect to one another. The behavior of the analytical curve for each line is briefly described in Table 3. All points for which a reading could be obtained were plotted on the curves, regardless of how widely the concentrations in different sets of standards varied.

In general, the data obtained are highly self-consistent and the appearance of the analytical curves does not betray the very heterogeneous nature of the standards employed in constructing them. Lines which yield poor analytical curves are afflicted either by self-reversal, line interference, high background from iron or the surrounding atmosphere, or a combination of these. Usually it is possible to find another line of the same element which is free of these difficulties but, in the case of silicon, line interferences cannot be avoided and must be allowed for (see Appendix).

TABLE 1: SUMMARY OF EXPERIMENTAL CONDITIONS

<b>Spark Parameters:</b>	Inductance—residual Secondary resistance—residual Capacitance—.005 microfarads RF current—9 amperes Breaks per half cycle—4 Spark breakdown voltage approx. 15kv Secondary gap—magnesium electrodes, in series with analytical gap, air quenched but not irradiated
<b>Spectrograph:</b>	Hilger large Littrow glass and quartz, model E 478
<b>Wavelength Region:</b>	2470 A to 3500 A
<b>Slit Width:</b>	12 microns (line width 30 microns)
<b>Filter:</b>	Two step rhodium on quartz—100%T and 15%T at the slit
<b>Analytical Gap:</b>	3 millimeters
<b>Counterelectrodes:</b>	1/4" diameter graphite 120 degree cone (for nitrogen) and 60 degree cone (for argon), hemispherically tipped
<b>Emulsion:</b>	Spectrum Analysis No. 1 plates, devel- oped in D-19 with continuous agita- tion for four minutes at 20°C
<b>Photometry:</b>	Jarrell-Ash model 2000 microphotome- ter with barrier-layer cell detector and galvanometer readout
<b>Sample Surface Preparation:</b>	80 grit aluminum oxide belt on a high speed sanding machine
<b>Gas Flow Rates:</b>	Nitrogen—8 liters per minute; argon— 10 liters per minute. Both gases in- troduced into the analytical gap by the arrangement shown in Fig. 1. Flow rates measured with a float- ing-ball type gas flow meter calibrated for O <sub>2</sub> flow rate.
<b>Exposure:</b>	15 second prespark period, 25 second exposure time.

TABLE 2: SOME IRON LINES STUDIED\*

Wavelength, A	Excitation potential (ev)		Remarks
	Low level	High level	
2592.779 II	?	?	Both can be used as internal standard lines when readable. They are a little too strong for low alloy steels but are advantageous for highly alloyed steels whose iron content is low.
2730.738 II	1.07	5.59	
2813.613 II	3.21	7.59	Intensity ratio to Fe I 3009.570A serves as excitation index with spectra exposed in nitrogen.
2495.860 II**	?	?	Intensity ratio to Fe I 3009.570A serves as excitation index with spectra exposed in argon.
2819.294 I	2.75	7.12	Blend of these two lines may be used as an internal standard line if iron content of the material is not too low. An appreciable background under the line is responsible for the small slope of the "constant time" analytical curve. The observed line has an intensity best suited for steels with at least 70% or more iron content. (Fig. 4)
2819.333 II	3.14	7.52	
3009.570 I	0.91	5.01	Can be used for an internal standard line whenever readable. Useful with a wider range of compositions than any of the other lines studied. (Fig. 4)

\*Wavelengths and excitation potentials were taken from references [12], [13], and [14]. Excitation potentials given are referred to the ground state of the atom or ion, and do not include the ionization potential in the case of Fe II lines.  
\*\*Although the line is listed as a blend with an Fe I line by Moore, the observed line behaves like a typical Fe II line.

TABLE 3: ANALYTICAL LINES USED\*

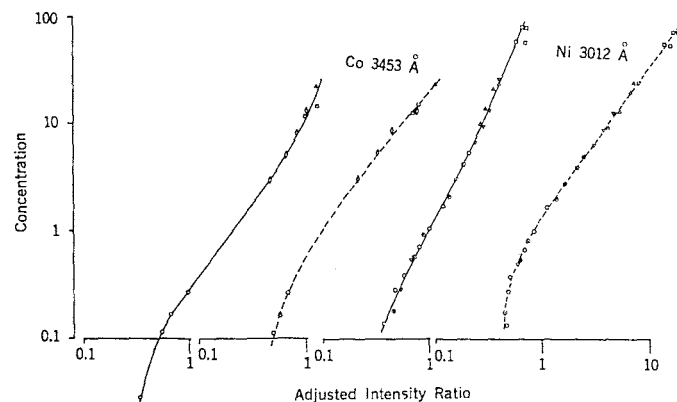
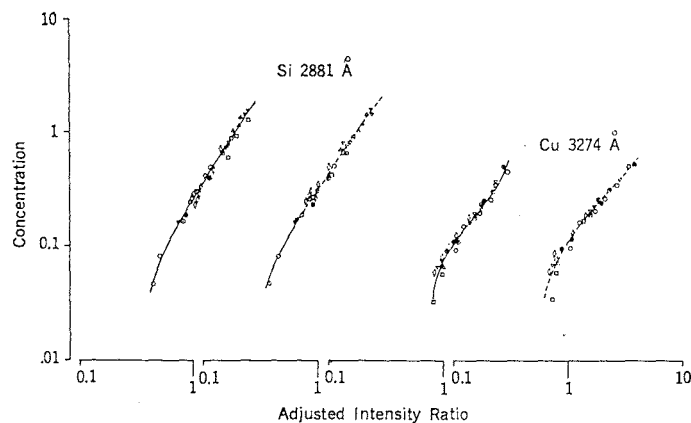
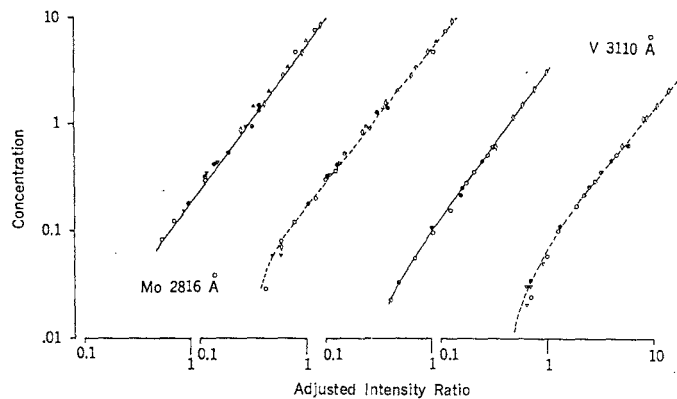
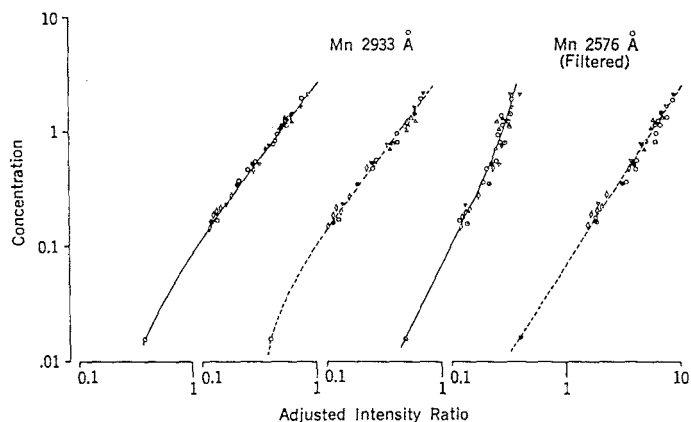
Dispersion A/mm	Wavelength, Å	Excitation potential (ev)		Remarks
		Low level	High level	
4.4	Mn II 2933.063	1.17	5.38	Interference from Cr 2932.705A not detected. At lowest concentrations curvature due to the presence of spark background observed.
2.8	Mn II 2576.104	0.00	4.79	Self-reversed at higher concentrations in N <sub>2</sub> but not in argon. More sensitive than 2933.063A. Analytical curve not affected by background and still straight at the lowest concentration plotted (.016% Mn).
4.2	Si I 2881.578	0.78	5.06	Cr 2881.931A, Mo 2881.370A, and W 2881.606A interferences identified. By plotting the adjusted summation: $S \equiv [\%Si] + .0165 [\%Cr] + .005 [\%W] + .0015 [\%Mo]$ , the contributions of the interfering elements to Si intensity are allowed for. The same relation allows correction of measured Si concentrations for interferences. (See Appendix).
2.6	Si 2516.123	0.03	4.93	Ti III 2516.01A and Mo 2516.109A interferences identified. Slope in N <sub>2</sub> is extremely poor. In argon the curve is usable when Ti and Mo are not present.
3.8	Cr II 2818.359	4.14	8.52	Possible interference by Mo 2818.300A not detected. Above 15% Cr, points from different sets of standards tend to segregate slightly.
3.8	Cr II 2822.012	4.13	8.50	Same intensity and same multiplet as 2818.359A. Mo 2822.029A interference detected in low alloy and tool steels.
4.8	Ni I 3012.004	0.42	4.52	Fe 3011.94A underlies and causes curvature at low Ni. Self-reversed at higher concentrations in N <sub>2</sub> . Analytical curve covers amazingly wide concentration range.
3.8	Mo II 2816.154	1.66	6.04	Al II 2816.179A interferes when Al content is above one percent. Analytical curve is straight from 0.1% to 10% Mo.
5.8	Mo I 3170.347	0.00	3.87	Fe II 3170.346A underlies and causes curvature at low and intermediate values of Mo, and a poor slope. At high concentrations in N <sub>2</sub> the slope continues to be poor owing to self-reversal, while in argon the curve gradually bends over and acquires a useful slope.
5.4	V II 3110.706	0.35	4.31	Analytical curve is straight from 0.1% to 3.1% vanadium content.
6.4	Cu I 3273.962	0.00	3.77	The fact that the transition is to the ground state makes self-reversal in nitrogen probable. The data on the limited concentration range available in the standards are consistent with this interpretation.
5.9	Nb II 3194.977	0.32	4.19	W 3195.076A interferes but only when several percent of tungsten is present. Free of vanadium interference in stainless steels.
4.2	Nb 2883.178	?	?	Free of any vanadium interference but weaker than either 3094A or 3194.977A.
5.2	Al I 3082.155	0.00	4.00	Mo 3082.220A may interfere but could not be checked. Few data available.
6.2	Ti II 3241.986	0.00	3.81	Background in argon higher than in nitrogen. All available data fit a single analytical curve. Possible W and Nb interferences could not be checked.
7.3	Co I 3453.505	0.43	4.00	All data fit a single analytical curve. Self-reversed in N <sub>2</sub> at higher concentrations. Possible Cr and V interferences could not be checked.
2.5	W II 2488.771	0.92	5.87	No significant interference at high W concentrations. Heavy background from neighboring iron line causes curvature at low concentrations, and necessitates correction for iron, if iron content of samples is variable. Possible Nb interference could not be checked.
6.4	Zr II 3273.047	0.16	3.93	Readable above 0.1% Zr. Not enough data to check interferences.

\*Wavelengths and excitation potentials in this table were taken from references [12], [13], and [14]. Excitation potentials refer to the ground state of the atom or ion and do not include the ionization potential in the case of singly ionized atoms.

Systematic deviations that still persist when allowance is made for all known line interferences are few in number. Their magnitude is not large on the log intensity scale, and their practical importance is small, except at high concentrations of a few alloying elements. Conceivably, some of these residual deviations may yet be explained in terms of unsuspected line interferences. Others, associated with nickel-base materials, may be simply due to the questionable assumption that the relative positions of the "constant time" analytical curves for iron do not shift. Since the intensities that had to be measured span the latitude of the photographic emulsion used, microphotometry had to be pushed to its limits and further work with direct readers is needed to settle these remaining questions.

**I**N a high voltage spark the sample vapor is ejected from the electrodes by explosion-like sputtering processes and then travels through the gap at a high speed, independent of the current flowing in the spark channel (9, 10). Collisions with the molecules or atoms of the surrounding gas tend to slow down and stop the expansion of this vapor cloud. The ability of a gas to retard the expansion (its stopping power) depends upon the molecular weights of the gas and metal atoms and upon their collision cross sections. Steinhaus, Crosswhite, and Dieke observed the motion of iron vapor in nitrogen as well as in argon by methods of time-resolved spectroscopy (11) and noted that apparently the vapor moves six to eight times faster in nitrogen than in argon (the average speed in argon, over a distance of 1.5 mm, was 100 meters/second). So rapid is the

expansion in nitrogen that the spark channel is surrounded with an envelope of cool metal vapor before the end of each spark discharge, and lines ending near the ground state begin to show self-absorption towards the end of the discharge cycle. In argon, the expansion of metal vapor is so slow that self-absorption cannot develop, discharge terminating before an appreciable vapor cloud can form around the spark channel. Our data tend to confirm those of Steinhaus et al. (11). Lines from transitions to or near the ground state become self-reversed in nitrogen but not in argon. When the analytical curves taken in nitrogen and in argon are compared it is evident that the following analytical lines are affected by self-absorption at high concentrations in nitrogen: Co 3453A, Mn 2576A, Cu 3274A, Mo 3170A, Ni 3012A, Ni 3414A, and possibly others. In each case, no evidence of self-absorption could be deduced from the visual appearance of the lines. In contrast to nitrogen, no analytical curves obtained in argon showed evidence of self-reversal at any concentration level. An argon atmosphere is thus peculiarly well suited for analytical procedures requiring determination of major elements. For minor or trace elements, either nitrogen or argon would serve equally well, although argon has a somewhat higher general spark background (except in the cyanogen band region). When nitrogen is used care should be taken to avoid lines that are subject to self-reversal at high concentrations of alloying elements. All lines from transitions to the ground state or to low levels just above ground state are suspect, and their behavior should be carefully checked before they are used for a particular analytical purpose.



**Figs. 6 to 9:** Analytical curves obtained by the empirical variable internal standard technique, for some of the analytical lines in Table 3. Data from nitrogen and from argon are shown side by side for each line, with the curve in nitrogen (solid) on the left and the curve for argon (dashed) on the right.

It has been shown that universal analytical curves, valid for all ferrous alloys, can be constructed by using very diverse steel and nickel base standards if they are sparked in nitrogen in argon under conditions producing a black burn, provided that allowance is made for the variation of the internal standard line intensity with iron concentration.

#### Appendix: The "Concentration Subtraction" Method of Correcting for Line Interferences

The "concentration subtraction" method of correcting analytical curves for line interferences is based on the assumption that the apparent increase in the concentration of an element due to a coincident line on the analysis line is proportional to the concentration of the interfering element in the sample. The proportionality relation is determined by plotting the distance of points obtained from standards containing the interfering element against the interfering element concentration. If elements A, B, and C interfere with the analysis line of element E, and we are using that line to determine the concentration of E in a sample, it is clear that what we actually read off the analytical curve is the value of the adjusted summation

$$(1) \quad S_1 = [\%E] + k_1[\%A] + k_2[\%B] + k_3[\%C]$$

where  $k_1$ ,  $k_2$  and  $k_3$  are constants. When the constants have been determined, the contributions from the interfering lines of A, B, and C are easily determined if their concentrations in the sample are known. Subtracting these terms from the measured value of  $S_1$  gives then the concentration of E. When the analytical curve is plotted this procedure is reversed, the interference terms are added to the concentration of E and the resultant adjusted summation is plotted on the analytical curve.

This procedure has been followed in constructing the analytical curve for Si 2881A which, in our spectrograph, is subject to interference from Cr, W and Mo. The adjusted summation of silicon and interfering element concentrations that was actually plotted is

$$(2) \quad S_2 = [\%Si] + .0165[\%Cr] + .005[\%W] + .0015[\%Mo]$$

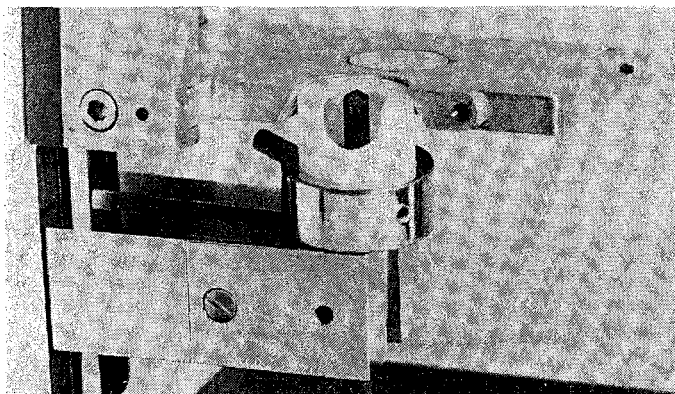
The constants were determined graphically from data obtained in nitrogen, but they are evidently good in argon as well.

The advantage of using interference correction functions of the form of equation (1) lies in the fact that all standards that are available can be used for constructing the analytical curve, even if they are afflicted by severe line interference. In addition, there is evidence that with similar types of equipment, and excitation conditions, the same function will describe data taken in different laboratories. This evidence is based on the observation that when an adjusted summation of the type of equation (1) was used to describe the interference of Cr and V with Fe III 3013.125A line (8), another laboratory that checked our work found it to be a valid description of their results as well.

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#### 3310 Atmosphere Chamber for Petrey Stand

Slightly modified so it is adaptable to most commercial Petrey Stands, the 3310 Atmosphere Chamber is modeled after that described by Arrak in this issue of the SPEAKER and designed by Frank Hurley of Grumman Aircraft's Quality Control Laboratory. The chamber is constructed of Delrin. A rim on its bottom mates with a groove in the chromium-plated brass part to enable the unit to be operated upside down or on its side. Included with the chamber is a 2- and 3-mm spacer for pushing the lower electrode into position. This may or may not be necessary depending on the particular spacing attachment built into the Petrey Stand itself.

**3310 Atmosphere Chamber for Petrey Stand.** Complete with 2- and 3-mm spacer for positioning lower electrode; length of Tygon tubing ..... \$ 65.00

**9024A Regulator, Flow-meter, Toggle valve assembly.** For controlling the flow of argon through the Atmosphere Chamber. Fits directly on standard gas cylinder. .... \$ 65.00

**1200 Low Alloy Steels Standards,** set of eight blocks 1-1/2" x 1-1/2" x 1". Si range 0.10-0.96; Mn 0.16-1.54; Ni 0.19-4.98; Cr 0.19-2.97; Mo 0.17-1.51; V 0.12-0.65; Cu 0.13-0.55% ..... \$124.00

## CORRECTION

Mr. E. O. Brown of the Marquette Cement Company Research Laboratories in Chicago has called our attention to an implication which we did not intend when reporting his technique described in the last issue of the Spex Speaker. The high speed grinder in use in his laboratory, for preparing cement samples for x-ray spectrochemical analysis, is not a Spex Shatterbox but a similar foreign instrument.

Ed. note: The Shatterbox is employed for this purpose by a number of other cement companies including Huron Portland Cement Company, Lone Star Cement Company, Louisville Cement Company, Oklahoma Cement Company and Permanente Cement Company.



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