

## OPTIMIZING ACCURACY IN EMISSION SPECTROCHEMICAL ANALYSIS\*

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UNLIKE most of the other unsolved problems in spectrochemical analysis, that of optimizing accuracy has been and will remain a long-term one, subject to steady, if undramatic, improvement over the years. As sources of error are isolated, their magnitude estimated, new offsetting techniques, equipment and correction factors are introduced. A few are found to work; fewer become widely accepted. Spectroscopy has thus evolved to a position of equal rank with wet chemistry. Today, it is known that limitations hamper the accuracy of both approaches and often favor one over the other. But no longer is the wet chemical figure sanctified, the spectrochemical one discarded as untrustworthy. In modern spectrochemistry, accuracy in the order of 1% of the amount determined is routinely achieved in photoelectric spark analysis of metals. The error is of the same magnitude as the inhomogeneities of the minute sample taken. What deviations persist appear to be real, in the sample rather than in the method.

Take as an example a typical low-alloy steel, type 4130, whose constituents are mainly above 0.1% and so amenable to spark analysis. Accuracy of analysis is excellent and control of the end-product is equally good. When a rod of 4130 from any American steel mill is machined there is no question but that the shape of the tool, the depth of the cut, the speed of the lathe spindle—even tool wear—can all be standardized and, of course, automated. And when the final product is heat-treated under controlled conditions, it will develop a hardness of Rockwell C-60 within tight bounds.

The same cannot be said about materials which depend on the presence or absence of trace elements for their properties. Transistors manufactured from ultra-pure silicon or germanium with the addition of a trace of another element must be selected before shipment. Partly because of compositional variations, one transistor will break down at 50 volts while a batch-mate will rate 100 volts and command a premium price.

So it is here, in the realm of trace-element analysis and control, that accuracy needs coaxing. Dropping sharply from 1% variations attainable at the 0.1% concentration levels, the accuracy at the ppm level often plummets to a poor but reluctantly acceptable 100% of the amount found. No spectrochemist is embarrassed when he reports 1 ppm iron in a sample on one occasion and 2 ppm the next time. Yet a jump from 1 to 2 ppm, like one from 1% to 2% or 10% to 20%, can easily account for

a significant difference in the physical properties of many materials.

To encourage consistency, ASTM is currently considering a series of all-encompassing definitions which do not distinguish between an element at a trace, minor or major level of concentration. A quantitative determination is to be one where the inferred accuracy is identical regardless of the percentage of the element.

TABLE I

## Several Definitions Under Study by ASTM E-2, Sub. 3

**Qualitative Analysis:** Survey of a sample for all elements in which concentrations may or may not be estimated.

**Semi-quantitative Determination:** Measurement of an element within a confidence level of  $\frac{2}{3}$  to  $1\frac{1}{2}$  times the actual amount present; for those elements not detected, the limit of detection is furnished.

**Quantitative Determination:** Measurement of an element within a confidence level of  $\pm 15\%$  of the actual amount present.

**Trace Analysis:** Quantitative or semi-quantitative measurement of concentration between 1 and 100 ppm.

**Micro Analysis:** Quantitative or semi-quantitative measurement of concentrations in samples smaller than 1 mg.

**Ultra-Trace Analysis:** Quantitative or semi-quantitative measurement of concentrations below 1 ppm.

Table I represents, on the one hand, lofty goals in trace analysis but, on the other, merely a statement of existing conditions for elements at moderate concentration levels. Thus are we inspired to our present undertaking: an attempt to ferret out and isolate the sources of error that are bothersome mostly at trace concentration levels.

Before stumbling along the error-strewn path, however, let's take the straightaway development of spectroscopy as a chemical method. Like other physical analytical tools, a spectrograph measures not concentration as the weight of one element compared with another but an optical property related to concentration. Summed up briefly though somewhat unprecisely, the integrated light intensity emitted by an element at a particular wavelength is assumed to bear a fixed relationship with concentration of that element. To achieve this artificial relationship, it is obvious that the sample and reference standards must be excited for the same length of time, at the same distance from

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the slit of the spectrograph, and under identical electrical, geometrical and optical conditions. Try as he does to control all variables, however, the analyst finds that his results may not always meet his accuracy requirements. Are there unsuspected breeding grounds for errors? Are some accepted shortcuts and assumptions not really acceptable? It seems worthwhile to sort out a few of these surreptitious areas and to assign a figure of demerit to them relative to how seriously they can interfere with results.

### SENSITIVITY

As a starter, forcing a spectral line out into the open improves its measurability and reduces attendant errors. The greater the line:background, the more reliable the determination. Many means for accomplishing this are known; at least brief mention is warranted of four important ones.

The spectrograph itself is properly adjusted at a position of maximum line:background. As shown by several authors, a grating instrument should be set to an order where the bandpass at the minimum practical slit width (10 microns) equals the natural width of the line. With a dc arc, Doppler broadening sets a limiting width of 0.1-0.01A for the middle to heavyweight elements. At a dispersion of 1A/mm, 0.01A is the bandpass equivalent of a 10-micron slit. A typical 3-meter spectrograph with a 600 groove/mm grating needs to be set to the 5th or 6th order to reach 0.01A bandpass.

A photographic plate restricts sensitivity by its inherent graininess and fog. Intuition to the contrary, it is not always wise to rely on the fastest and consequently most grainy and foggy emulsion to attain optimum sensitivity. Frequently SA #1, with its fine grain, is the best emulsion. The test, once more, is the relative intensity of the line over background.

Much has been learned about carriers to boost accuracy through sensitivity. Fluorides are now widely accepted for determining silicon and boron. Gallium oxide and silver chloride are routinely employed as carriers in the atomic energy field for flushing impurities out of a refractory matrix. Acting as a mechanical rather than chemical carrier, boiler cap electrodes boost the sensitivity of highly volatile elements such as mercury and arsenic.

Characteristically, all carriers are selective; they enhance the production of certain lines at the expense of others. In sharp contrast, the Stallwood Jet has proved universally applicable in trimming errors for all elements by combining three functions: 1) the sample is bathed in an atmosphere free of nitrogen, notorious not only for prolifically fathering cyanogen bands but also for producing overall fogging in the ultraviolet. In place of nitrogen, a gas mixture can be selected with an excitation potential matched to that of the elements being determined. 2) The Stallwood Jet constrains the arc to a non-wavering column so all excited atoms leave their trail markers when emerging from the electrode crater. 3) Combined with a deep-cratered electrode, it helps samples burn in layers, discouraging volatile elements from coming forth in one explosive burst. Typically dc arc transmittance variations have been routinely reduced from 15% to 4% in hundreds of labs where Stallwood Jets work full time.

### FOCUS

Arrak (1) discovered that focusing a spectrograph need not depend on "eyeball" estimation of how far in or out the entrance slit is to be moved. His "separation of beams" technique is objective, graphically pinpointing the slit position of optimum focus. A mask placed over the center of the grating produces, in effect, two gratings side by side. Properly focused, the double grating generates normal spectral lines. Defocused, the lines appear doubled, the spacing between them a linear function of the degree. By plotting slit position vs. line separation,

the exact slit position where the two lines merge is readily interpolated. A measuring microscope or eyepiece is the only tool needed.

The slit position of best focus for one spectral region may differ from that for another. Provisions for tilting the camera are thus offered on modern spectrographs. With plane grating spectrographs, focus may depend on the rotation of the grating, optical laws notwithstanding. Apparently, some grating blanks are not perfectly flat but show a radius of curvature. To be sure, this may be many meters long but it, nevertheless, alters the focal setting. Incidentally, over-tightening a grating in its mount will distort it to create resolution as well as focus problems.

To study the effect of focus on spectrochemical precision, a 3/4-meter Czerny-Turner spectrometer was set up with a mercury penlight source and deliberately defocused. The intensity of the source is quite steady, permitting individual photoelectric measurements to be made reliably as contrasted with analytical spectrochemistry equipment where fluctuations in the source and photography demand statistical treatment of many data. As indicated in Table 2, the intensity of two background corrected lines does reach a maximum at the position of best focus. That the lines do not bear a one-to-one relationship defocused is shown in the last column.

Whether or not visual focusing is sufficient in spectrochemical work is not easy to say. The work reported in Table 2 was repeated photographically with a 3.4-meter Ebert spectrograph. Here, of course, the focal depth is much greater and the slit position was varied in increments of 2 mm. The data were inconclusive because of the difficulty in making background corrections. It appears that for uncorrected lines of about the same intensity, focus is not critical. When, however, a weak line is background corrected, focus becomes quite critical in its bearing on accuracy.

TABLE II  
Effect of Focus on Intensity

Slit position, mm	Hg 3125A I*	Hg 3110A I <sub>c</sub> *	I/I <sub>c</sub>
-0.5	117	7	0.17
-0.3	147	11	0.075
-0.2	200	12	0.060
0	285	16	0.056
+0.2	285	16	0.053
+0.3	285	13	0.046
+0.5	222	10	0.045

\* Background corrected

### COMA AND RESOLUTION

Coma is an optical aberration which disrupts the shape and resolution of lines at increasing distances from the optical axis or the coma-free position on a plate. (The two positions are not necessarily identical, as will be shown.) The actual resolution of lines in the center region of a plate is therefore better than at the ends, a fact often overlooked by spectroscopists. If low concentrations of boron and chromium are to be determined in a particular sample, the wavelength region would be set to cover 2400 to 4300A to bracket the *raies ultimes* of both elements. For another sample in which barium is sought instead of boron, the grating is usually cranked about to pick up Ba 4554A. In so doing would the chromium analytical curve remain fixed? The answer is decidedly negative. All four curves shown in Fig. 1 can be directly obtained photoelectrically and may simply be redrawn from normal recording microphotometer transmittance tracings.

In Fig. 1A the intensity vs. wavelength is plotted for an ideal line. Perfectly symmetrical, the curve sweeps upward from a region of no background without inflections. The half-intensity

FIG. 1

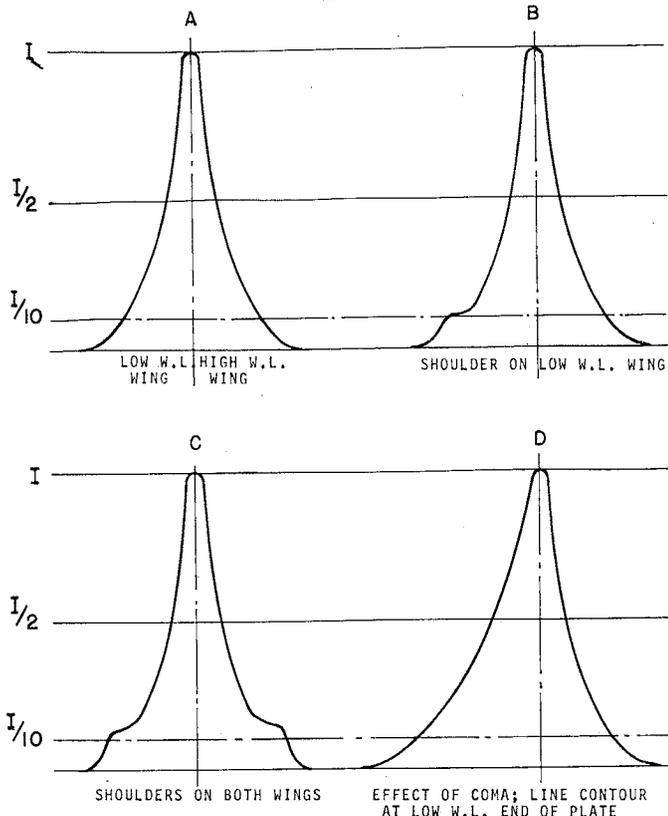


Fig. 1—Line tracings illustrating A) an ideal shape; B) resolution loss at low intensity on one side of line only; C) the same on both sides; D) coma.

width is about equal to the theoretical resolving power,  $m\lambda$ , where  $m$  is the total number of lines on the grating multiplied by the spectral order. With a 150 mm wide, 1200 groove/mm grating a spectrograph will produce lines, in the center of a plate, having a half-width close to the theoretical 0.016A.

Fig. 1B shows a typical imperfection due to the grating, mirrors or optical adjustment. Here a shoulder emerges on one side of the line. This may or may not be "grass" which can be resolved into many components by narrowing the slit. Note that the line shape is asymmetrical and that at an arbitrary  $1/10$  relative intensity, a quantitative figure can be derived to assay resolution loss. In Fig. 1C, the same aberration may be noticed, this time unevenly distributed on either wing. Fig. 1D is typical of coma. The side of the line toward the center of the plate drops steeply as in Fig. 1A; on the other side, the line fades slowly. Instead of coming to a uniform point at either end, lines photographed in a region of coma have skewed ends, the flatter side of the point extending toward the outside of the plate. The line in Fig. 1D is at the low wavelength end of the plate; its mirror image would appear at the high wavelength end.

As already inferred, the width of both wings of a line is indicative of resolution and furnishes a yardstick for the degree of coma. For simplicity, however, we chose to measure the full  $1/10$  width for the plot in Fig. 2. As expected, the width increases rapidly toward the end of the plate. It will be noted, too, that the coma-free point does not coincide with the physical or optical center. The coma-free point is related to the rotation of the grating and therefore varies with the wavelength range to which the instrument is set.

It is evident from Fig. 2 that the maximum density of a line—the figure read on a microphotometer for conversion to intensity and ultimate insertion as a point on an analytical curve—

will be greater in the coma-free section of a plate than at the ends. Fig. 3 demonstrates this for three separate lines read in different regions of the plate. For this work a 2024 aluminum alloy was sparked at different grating angles. Note that Al 3066A, Si 2881A and Si 2516A all have a minimum transmittance value approaching the coma-free position.

FIG. 2

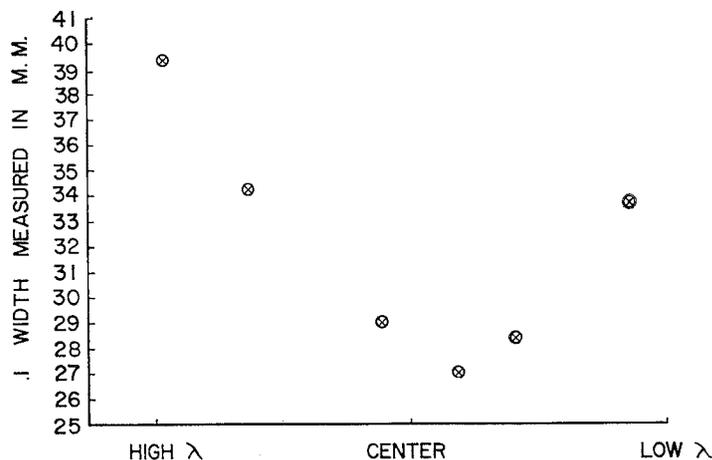


Fig. 2—The width of a spectral line at 0.1 of its maximum intensity is a good indication of the coma. Note that the coma-free point does not coincide with the center of the plate and that the degree of coma is worse at high than at the low wavelength end of the plate.

FIG. 3

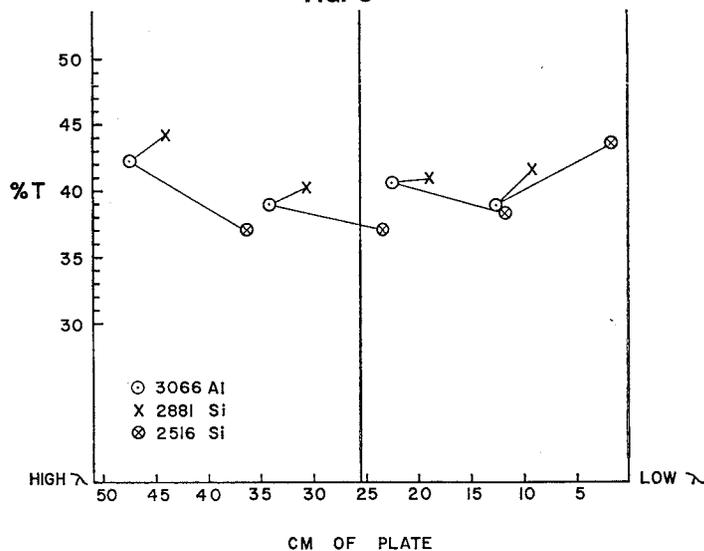


Fig 3—Transmittance readings for 3 lines in different regions of the plate.

Spectrochemical analysis relies on intensity ratio and it remains to be shown that this varies with the plate position. In Fig. 4, the same silicon lines were ratioed to the aluminum internal standard. The separation between one pair is 185A, the other 550A. Therefore, the coma differences would be expected to be more pronounced in the 2516/3066A. Note that the former line pair varies from around 0.9 to 1.0. The latter drops from a high of 1.2 at the high wavelength end to 0.8 at the low end. At the high end the intensity peak of the denominator (Al 3066A) drops because of coma while at the low end Si 2516A decreases. The overall change of 50% would result in an error of that amount in calculated concentration.

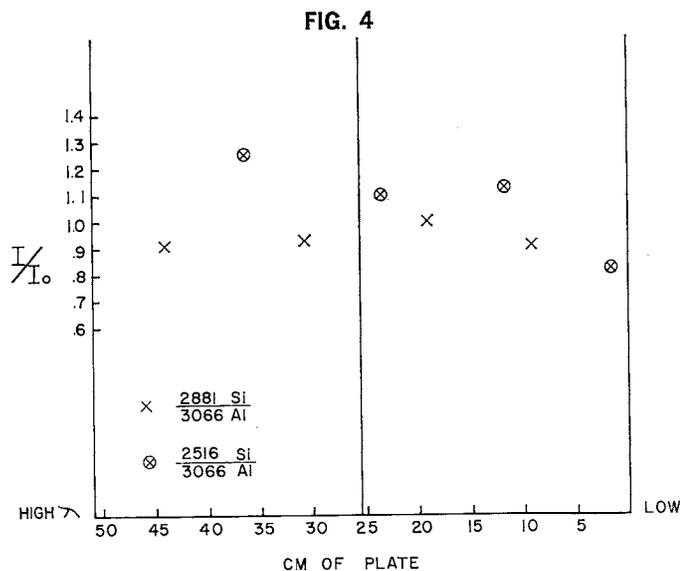


Fig. 4—Silicon lines ratioed to an aluminum internal standard at different grating angles.

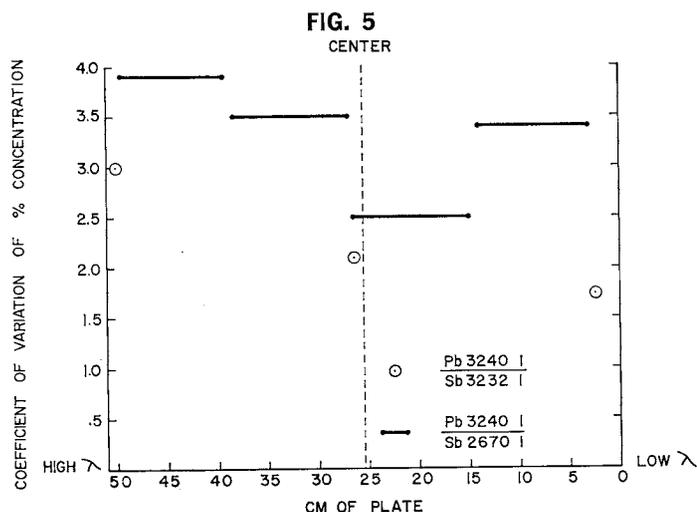


Fig. 5—The intensity ratio of 2 lines widely separated in wavelength not only differs markedly when positioned at various portions of the plate; its error follows the degree of coma. The error for close lines, while following the same trend, is not nearly so severe. The reason for this is not known.

Evidence shown thus far seems clear-cut, easily explained. The data in Fig. 5, however, have not been interpreted so successfully. It shows that the reproducibility of line ratios seems to be a function of coma. In a series of ten runs, at various regions on the plate, the error of measurement of the antimony content of a lead standard is worse when the lines are moved to either end of the plate. Secondly, the variation of the antimony line, separated by only 8Å from the internal standard, is consistently better than that separated by 600Å or 10 cm on the plate. But why?

While those two questions must remain unanswered, hopefully temporarily, the answer to a more basic one is known. Resolution loss, whether caused by coma or anything else, reduces the sensitivity of lines and reproducibility of lines in the "shadow" of strong diffuse lines. Two supposedly identical instruments, one with better resolution than the other, will therefore differ in the accuracy of trace-element determinations. For

weak lines, signal-to-noise will be directly related to the resolution and the resolution can easily vary by a factor of five depending on the excellence of the optics of a spectrograph. In any one spectrograph, the last few inches of spectra should be avoided when highest reproducibility and sensitivity are sought.

### BACKGROUND CONTROL

Background itself is a little understood subject, its implications often not taken into account. One of the rules of thumb to which spectrochemists refer is to ignore background if it is above an arbitrary 70% transmittance. While true that it would hardly pay to correct for such a low background against a line of 10%T., a line at 65% T. in a 70% haze definitely needs to be corrected. Further, when setting up an analytical curve for a series of standards, if the transmittances at the lowest concentrations require background corrections, corrections must be made at all other concentrations.

Luckily, this is a paper on unsolved problems for background correction falls squarely in this category. As Slavin pointed out (2), background arises from spectral and non-spectral sources, from line emissions as well as continua. In the usual method of background correction, however, it is tacitly assumed that background arises only from a line emitted in close proximity to the line under measurement. This assumed, it is logical that the same characteristic curve applies to both and background intensity can then simply be subtracted. But Slavin reminded us that the best grating is never 100% efficient; a small fraction of the light it receives is reflected as from a mirror. Light from this internal source now bounces around to fog the plate unevenly. When light originates from an incandescent source, the scattering is pronounced because such a wide and intense band strikes the grating. A spark source, on the other hand, with its high energy excites molecular bands of air, water vapor, metal oxides, etc. Ignoring these differences, the practicing spectrochemist tends to lump all background into a single correction term. Certainly, if he knew that the background adjoining a line at 3500Å arose from scattered radiation of 4500Å, he would prepare a separate characteristic curve and thus obtain a better correction term.

All considered, background corrections represent probably one of the severest limitations imposed on trace-element accuracy. Before sensible recommendations can be made, a thorough investigation must be conducted to learn the origins of background with sparks and arcs under various conditions. Only then will it be possible to circumvent a serious hurdle in spectrochemical analysis.

### PHYSICAL NATURE OF SAMPLE

Among the hapless scapegoats persistently blamed for precision troubles is the physical structure of the sample over which the analyst rarely can exercise any control. In metals, it is the history of the sample that is cited for contempt. Heat treatment, casting temperature, addition of grain refiners and scavengers to the melt, method of shaping all are known to bear heavily on the intensity ratio of the spectral lines representing their composition. Although metallurgical history can now be reckoned with by "black burns" in an inert atmosphere, too little attention has been paid to its counterpart in powder-dc arc analysis. Such things as crystalline structure, degree of hydration, particle size, shape and distribution hinder accuracy but few controlled experiments have been conducted to get an idea of the order of magnitude of the trouble they can cause.

Aluminum oxide is a good example of crystalline form variations. It exists in at least three well-known and commercially valuable forms. The heat conductivity of two, alpha and gamma, is markedly different and their arcing characteristics differ accordingly. Yet the one is often "doctored" with impurities to

become a series of reference standards for the analysis of the other.

That this is decidedly not to be recommended for quantitative work is seen in Table III. Spex Mix was diluted with both forms of alumina in the same proportions so that 49 elements were all present at the same concentration level. The materials were burned ten times with a Stallwood Jet-stabilized dc arc. Lines of six elements were measured, enough to predict that all elements would probably behave similarly. The data indicate that the intensity ratio for all six elements in the low-temperature alpha alumina is significantly lower than that for gamma-alumina, the form obtained by firing in a higher temperature atmosphere. Although not tabulated, the line transmittances give the reasons for the dramatic change. The intensity of the added lines remains relatively constant. The intensity of the aluminum internal standard, on the other hand, is much lower in the more refractory form. The heat of the dc arc simply excites the aluminum in the low-temperature alpha material to a greater degree than the aluminum in the gamma form.

From a practical viewpoint, Table III looms out as a problem faced by every spectrochemist called upon to run "what-is-its" and instructed to write a concentration figure plainly in the appropriate box. Unless the sample and standards are normalized—by solution in a common acid, by fluxing uniformly, by melting, or by some other means—quantitative analysis is simply not possible. Faced with a decision, the spectrochemist should report results as semi-quantitative whenever he cannot convert samples and standards to a common form.

**TABLE III**  
**EFFECT OF CRYSTALLINE FORM ON INTENSITY RATIO**  
**Intensity Ratio for Two Forms of Alumina**

Line Pair	Alpha Alumina	Gamma Alumina
In 3256A	0.95	1.77
Al 3060A		
Cd 3261A	0.53	0.70
Al 3060A		
Sb 3268A	0.64	1.31
Al 3060A		
Ge 3269A	0.31	0.55
Al 3060A		
Sn 3262A	0.75	1.44
Al 3060A		
V 3183A	0.40	0.79
Al 3060A		

**ALTHOUGH** we have been able to consider only a few sources of error, surely, others will occur to readers. Won't you take the time to jot them down for the benefit of your colleagues? The isolation and presentation of a problem can go a long way toward its solution. We'll gladly find space in these columns for any findings you offer.

#### References

- 1) Arrak, Arno, *Spectro Acta*, 12, 1003, 1959.
- 2) Slavin, Morris, *Appl. Spectros.*, 16, 173, 1962.

## FLUXING TECHNIQUE FOR X-RAY ANALYSIS

Although subject to variations and itself in a state of flux, a procedure practiced widely for quantitative analysis of minerals in the United States and Canada is that of fluxing and pelletizing samples. A typical procedure follows:

Fuse a mixture of 9 parts by weight of  $\text{Li}_2\text{B}_4\text{O}_7$  with 2 parts of CeO for 30-60 minutes at  $1100^\circ\text{C}$ . Grind this material without metallic contamination to pass 100 mesh. Add 1100 mg of this powder to 200 mg of the ground sample and fuse for 30 minutes in Spex #7152 graphite crucibles. To correct for ignition losses, add enough of the original ground  $\text{Li}_2\text{B}_4\text{O}_7$ -CeO flux to the fusion to return it to 1300 mg. Add 120 mg of boric acid to strengthen the final briquet and grind the material so that about 60% passes 325 mesh. Using the #8011 adapter, four such samples can be ground simultaneously in Spex #5004 tungsten carbide vials in a #8000 Mixer/Mill; grinding time is around 10 minutes. Alternatively, seven samples can be handled in #8503 hardened steel containers in the #8500 Shatterbox where the grinding time is reduced to about two minutes. The ground material is placed in a #3622 die and pressed in the #C-30 press at thirty tons to yield the final pellet.

Though too new to be unequivocally recommended, the #3619 Spec-Cap should eliminate one step in the procedure. Pressed into the Spec-Cap, the pellet itself need not be strong and so the addition of boric acid may prove unnecessary. The material is simply loaded into the Spec-Cap which, in turn, is placed at the bottom of the die and pressed normally.

Preweighed  $\text{Li}_2\text{B}_4\text{O}_7$ -CeO mixture, 9:2 by weight, fused at  $1100^\circ$ ; ground and packaged in #3116 vial,  $\frac{1}{2}$ " dia. x 2",  $1100 \pm 5$  mg ea.

		100	\$ 45.00
		1000	330.00
6005	Lithium Tetraborate, anhydrous	100 g	4.00
		1 lb	11.50
C-30	Hydraulic Press, 30-ton capacity		590.00
3622	Die, hardened steel, produces pellets 1.235" o.d. x up to 5/16" thick	Each	215.00
8000	Mixer/Mill	115 vac	Each 390.00
		230 vac	Each 400.00
5004	Tungsten Carbide Vial	Each	36.00
8500	Shatterbox, 115 or 230 vac		960.00
8503	Grinding Vial, hardened steel, 15 ml cap set of 7		275.00
8503R	Rack for holding 7 #8503 vials	Each	85.00

## TYPING ALUMINUM ALLOYS WITH THE SPEX LAZYPROBE

Employing the so-called spark transfer method\* the Lazyprobe is an electronic sampling apparatus designed for the analysis of large, immovable or inaccessible objects. Sampling is accomplished by first placing a copper or high-purity graphite electrode in the holder with the safety switch open. The entire assembly is then positioned over the spot from which the sample is to be obtained, then pushed gently toward the specimen until the electrode touches its surface. After alignment, the probe is retracted, the safety switch depressed, and the assembly again slowly pushed toward the sample's surface. The spark discharges automatically when the EMF impressed on the capacitor is sufficient to overcome the resistance of the air gap between the electrode and the sample surface. This process is then repeated about ten times, or as many times as necessary to obtain a sufficient sample for the particular analysis. It is advantageous to coat the entire periphery of the electrode to obtain the most representative sampling.

Upon completion of sampling, the electrode is placed in the covered container section of the Lazyprobe to avoid contamination. This precaution is extremely important as only a micro sample is obtained; a minute amount of contaminant can obscure or invalidate the results. The final spectrographic analysis is by a typical micro method. Enough sample is easily obtained to bring detection limits down to about 0.1% for most elements.

The only requirement for the use of the probe is that the sample be an electrical conductor. Usually only washing and or mild sanding of the surface removes the shallow scar. A few advantages of the probe are:

**PORTABILITY:** Samples need not be brought to the laboratory as the probe will operate any place with 115 vac.

**SIMPLICITY OF OPERATION:** There are no critical adjustments nor intricate techniques required to obtain a sample.

**VERSATILITY:** With the tripod legs removed, analysis of material from holes or crevices can be performed. The size or contour of the material has no effect on the sampling procedure.

**MICRO-ANALYSIS:** Weldments, inclusions, platings can be analyzed. The diameter of the sampled area is about 1 mm.

**NON-DESTRUCTIVE:** Samples can be obtained from finished parts with little if any defacement.

As a practical application, the Lazyprobe transfer method was tested in our lab to type aluminum alloys. The presumption was made that, with virgin stock, the manufacturer's quality control of constituent concentrations will hold these values at the median of the specified range so the vast majority of alloys will be well within specifications. This is true for most aluminum alloys whose make-up is from relatively inexpensive constituents.

For the work 24 types of aluminum alloys, 12 casting and 12 wrought, were chosen. Table I contains the analytical lines that referenced the respective elements, with the lowest detection limits for an average sample obtained with the probe (15 sparks) and a National L-3903 cratered electrode arced in a Stallwood Jet.

TABLE I

Analytical Lines

Element	Wavelength, A	Detection Limit, %
Aluminum	3082.2	
Silicon	2881.6	0.1
Manganese	2953.7	0.1
Nickel	3414.8	0.05
Copper	3247.5	0.01
Magnesium	2802.7	0.01
Zinc	3345.0	1
Chromium	4254.4	0.01

From the experimental data a system of classification was developed that relies on a preliminary separation of the alloys into three major groups depending on their silicon content. It was found that the following relations would suffice.

TABLE II

Preliminary Classification

Silicon, %	Si 2881.6A, %T
Less than 0.2	Greater than 50 in first filter step
0.2 to 2	Less than 50 in second filter step
Greater than 2	Less than 50 in third filter step

Densitometry revealed that, with the aluminum line at 3082.2A in the range 25-50% T in the third filter step the approximations in Table II were valid. The elements shown in Table III were then visually estimated utilizing the stated criteria. After obtaining approximate concentrations of the various elements, the flow diagrams shown in Tables IV, V, VI were followed to identify the particular alloy.

Data in Tables IV and VI reveal that certain alloys are indistinguishable by the proposed scheme. The best recourse for their separation is a quantitative determination of a constituent which differs significantly in each. Inspection of a table of alloy compositions shows that the magnesium content will resolve most conflicts.

TABLE III

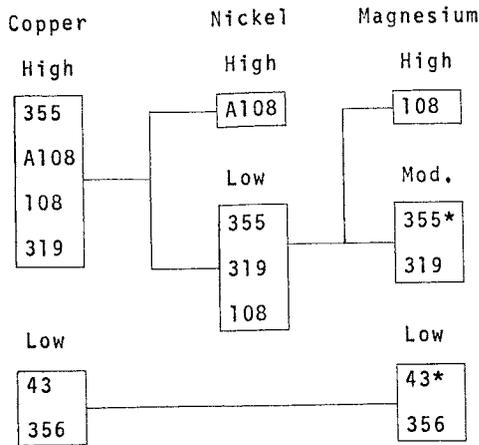
Element Concentration Determinations Using a 3-Step Neutral Filter

Element Line	Detection at given Transmittance step	% Concentration
Manganese 2953.7A	N.D. at 25%	Low < 0.1
	Visible at 25%	Mod. 0.1-0.5
	Visible at 4%	High > 0.8
Copper 3247.5A	N.D. at 4%	Low < 0.01
	< Al (3082.2A)	Mod. 0.1-1.0
	> Al (3082.2A)	High > 1.0
Magnesium 2802.7A	N.D. to faint trace at 4%	Low < 0.2
	< Al (3082.2A)	Mod. 0.2-2.0
	> Al (3082.2A)	High > 2.0
Nickel 3414.8A	N.D. at 100%	Low < 0.05
	Visible at 100%	High > 0.05
Zinc 3345.0A	N.D. at 100%	Low < 2.0
	Visible at 100%	High > 2.0
Chromium 4254.4A	N.D. at 4%	Low < 0.1
	Visible at 4%	High > 0.2

\* A comprehensive report on the Lazyprobe itself has been prepared by Arno Arrak, Grumman Aircraft Engineering Co., Bethpage, N. Y. Copies are available from him.

**TABLE IV**

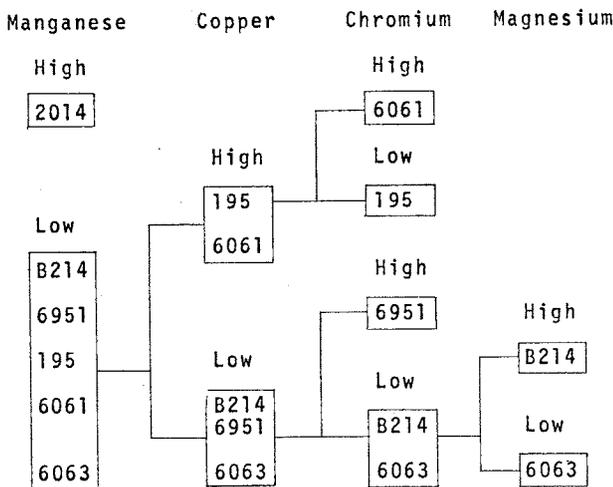
Flow Diagram for Alloys with High Silicon



\*These alloys are separable only by a quantitative magnesium determination

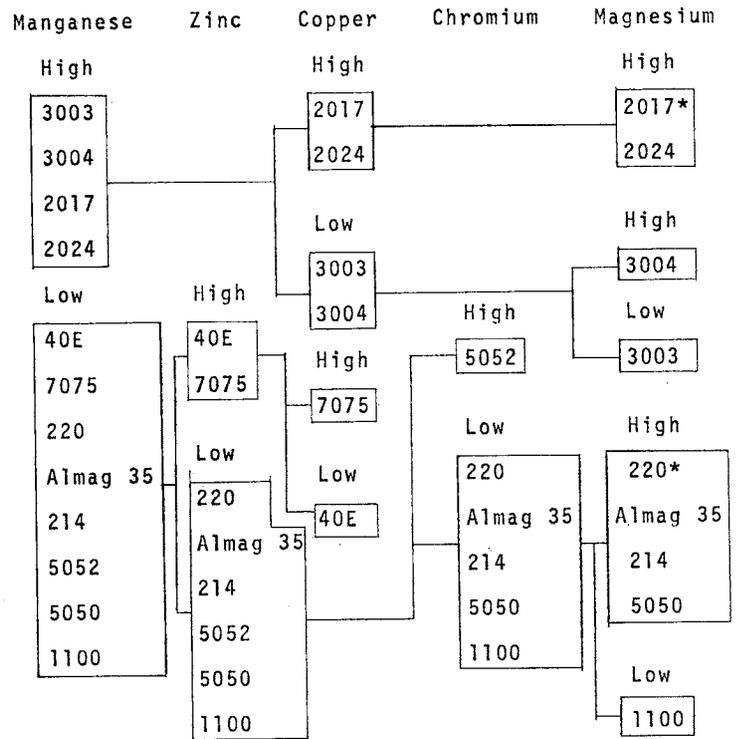
**TABLE V**

Flow Diagram for Alloys with Moderate Silicon



**TABLE VI**

Flow Diagram for Alloys with Low Silicon



\* These alloys are separable only by a quantitative magnesium determination

- 9050 Lazyprobe, spark transfer sampler, 115 vac ..... Each \$155.00
- 4001 Graphite electrodes ..... 100 \$ 22.00
- 9050-1 Copper electrodes ..... 100 \$ 22.00

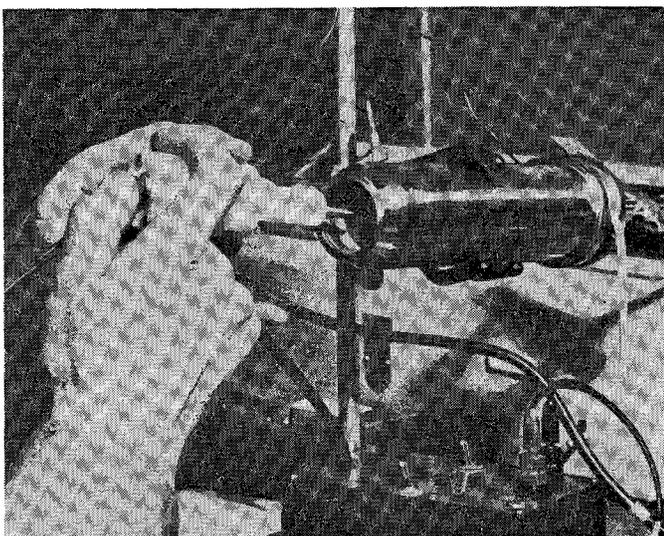


**AT YOUR SERVICE**

Lou Casper, who has been travelling among the middle eastern states in our behalf for the past several years, is presently poised for a cross continental hop to open and manage our new western regional sales office at:

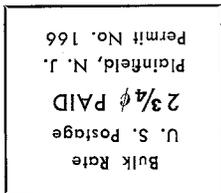
**23725 Sylvan St.  
Woodland Hills  
California 91364**

Picking up the eastern trail markers will be Elias Todd. Both fellows are anxious to be of any help they can.



Return Requested

Add'l. 1/8 cent pd.



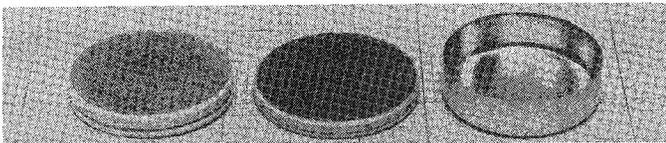
Metuchen, N. J. 08841  
3880 Park Avenue  
INDUSTRIES, INC.



### WINE BOTTLE SPECTROSCOPY

Charles Matocha of the Alcoa Research Laboratories in New Kensington, Pa., spotted a link between wine and spectroscopy on a visit to the Aluminum Company of America's Richmond, Va. plant. There wine bottle caps are made, automatic kick presses stamping them out by the jillion. Half way along the production line, Matocha noticed that such a cap just might be useful for holding powders in which to press pellets.

The product, a Spec-Cap, makes possible the pressing of powders that normally refuse to stay together. Pressed at around 40,000 psi (24 tons), the final planchet gives excellent spectroscopic results. It is strong, needs no binder, can be marked readily with an identifying number, is protected from moisture in handling, can be made conductive for emission analysis.



**3619 Spec-Cap**, aluminum container for briquetting powders; 1.085" o.d. x 0.325" thick; final pellet is 1.235" o.d. x 3/16" thick when pressed in #3622 or #3623 die.

300	\$10.00
1800	\$36.00

**Correction:** P. 19 of our May 1965 catalog erroneously lists the price of the #3619 Spec-Caps as \$10.00/100. The above-listed price of \$10.00/300 is the proper current price.

### CHEMIST NEEDED

Young, Brilliant, Ambitious, Eager, Willing and Able—of course!

Inspiring to staff (presently one technician), ingenious at improvisations;

Minimal in demands, maximal in production—of course!

Having the writing flair of a Hemingway and speaking ability of Churchill, in addition to performing the following:

Supervise laboratory operations, including preparation of semi-quantitative powder standards, pure materials, pre-weighed chemicals and master plates.

Conduct laboratory development and "shake-down" programs for new instruments and procedures. For a starter, we have a backlog of work on the Plasma Jet, DTA, TGA and 1967 things still under wraps.

After-hour stimulation may be derived from SAS, ASTM and the likes of EAS and the Pittsburgh Conference—of course!

Kidding aside, we need someone well grounded in the basics of chemistry (conversion of high-purity chemicals is tops on our priority list) and physics with an affinity for hard work and aspirations to stretch and grow with us.