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To all the friends who stopped by our booth at the Pittsburgh Conference, we want to say, "Thanks"—a warm, sincere and heartfelt thanks. We were gratified to learn that your results with our semi-quantitative standards were even better than we had promised. We felt our chests puffing a trifle upon hearing your kind words on our accessories. We decided to make the SPEAKER a quarterly after hearing that you appreciated it. And of course, we were most delighted to learn that you liked doing business with us.

We have enjoyed all this. But most rewarding is the knowledge that we have helped people save that one irreplaceable commodity—time. As we have stated before, we hope to continue to make life simpler and easier for the practicing spectrographer—emission, absorption and x-ray.

## SPECTROCHEMICAL ANALYSIS FOR TRACE ELEMENTS

### II. When Sample Size is Limited

By A. J. MITTELDORF

#### Introduction

In the introduction to Part I of this article, we spoke of the *double* problem of trace-element analysis. At one extreme, the sample could be unlimited in size—drinking water for example. At the opposite extreme, the sample could be exceedingly small—a particle of dust. Part I dealt with the former. In it, we discussed techniques for obtaining the optimum sensitivity with such samples. We showed how the important consideration was the line-to-background ratio. Anything we could do to increase that ratio would improve sensitivity. Such techniques as using replicate exposures, concentration by ashing or chemical separation, the use of high-resolution, low-fog photographic emulsions, all resulted in increasing the line-to-background ratio and, consequently, improved sensitivity. Further, we showed that a fine entrance slit at the spectrograph should be used and the spectrograph itself should be set to its highest effective dispersion. Practically all of the recommendations had the effect of improving the signal-to-noise ratio but at the expense of speed.

By way of contrast, *speed* is the keyword when a minute sample is to be analyzed. For the analysis of a sample, the size of which is limited, boils down to getting adequate registration on a photographic plate. The sample is usually so small that nothing can be done to it chemically. Indeed, sometimes the sample is so small that even maneuvering it into an electrode is a feat in itself.

Typical examples of limited sample analysis in metallurgy are the examination of a microscopic inclusion in a casting and the identification of a surface deposit. In criminology, analyses of limited samples are everyday affairs. Some months ago I was shown a section of the skull of a murdered woman, on the surface of which was a shiny, greyish spot. Apparently, she had been

struck with a metallic object. A pewter candlestick, found nearby, was just about proved to be the murder weapon when the spectrum of scrapings from the skull matched that of the candlestick. Biological research is another place where limited samples occur. Many biological projects end up with a speck of a newly isolated enzyme or vitamin. Then comes the arduous task of analyzing it. Through spectrochemical analysis, vitamin B-12 was found to be a cobalt compound and carbonic anhydrase was confirmed as a zinc metallo-organic. Atomic energy research is still another field where tiny samples abound. In recent years, a dozen or so new elements have been discovered. The birth announcement of almost every one of these elements was its unique emission spectrum.

The limited sample differs from the unlimited one in still another respect. In the former, we are concerned with *major* elements in a *tiny* sample; in the latter, with *trace* elements in a *large* sample. The matrix effect thus becomes important when dealing with a limited sample. Suppose a sample, the total weight of which is but a few micrograms, consists of 99% sodium and 1% iron. The iron would undoubtedly escape detection because of the low ionization potential of the sodium. If the spectrographer were asked to compare the iron content of this sample with one equally small which consisted of 99% aluminum and the same 1% iron, he might be thrown completely off the track by the appearance of the two spectrograms. For, in the latter, iron lines undoubtedly would appear.

The conclusion to the above is that quantitative, even semi-quantitative reports on limited samples are usually not possible. With fingers crossed, we report "major" or "principal" or "minor" elements. But on unlimited samples, semi-quantitative reports are indeed possible. We can ash organic samples or

evaporate down several water samples, mix the resulting powders with graphite in a fixed ratio and then analyze the samples by referring to standards such as the Spex Z or G sets. Here we can report values such as .003% or .00005% and obtain excellent comparisons among the samples.

## Analytical Techniques

So much for the differences in the two problems. We have already stated that to analyze limited samples, we must obtain the maximum speed from our apparatus. Let us examine every step in the spectrochemical procedure to see how we can squeeze the highest speed out of each.

### THE EXCITATION SOURCE

The dc arc with the sample burned to completion has been found to be the best overall source. The sample is ordinarily made electrically positive for the anode is the hotter of two graphite electrodes. Certain elements, however, have more intense lines near the cathode and it is often desirable to take advantage of this fact. The spectrograph is allowed to "see" only the light emitted from a thin layer directly above the cathode.<sup>1</sup> To utilize the "cathode layer" phenomenon, it is advisable to use a stigmatic spectrograph.

A source approaching, and in certain instances surpassing, the sensitivity of the dc arc is the copper spark. With this technique,<sup>2</sup> a drop of the solution to be analyzed is placed atop the faced-off ends of two 1/4" copper rods. After drying, the rods are sparked for 5 to 10 seconds. Because the source requires solutions, it is not so universally applicable as the dc arc. Where applicable, excellent results have been obtained. For example as little as 20 millimicrograms of Ag, Al, Bi, Cu, Fe, Mg, Mn, Pb, Sn and Tl can be detected.

Other sources occasionally used are enclosed ones like the hollow cathode tube. The principal advantage here is that the sample may be excited over and over again. In contrast, an atom once excited in an open source disappears.

### THE SPECTROGRAPH

To obtain the best speed on any spectrograph, all possible light from the source should be focussed on the slit. In addition to using condensing lenses, sometimes large reflectors are placed behind the source in order to collect as much of the light as possible and direct it to the spectrograph.

As was pointed out in Part I, it is a good idea to mask out the incandescent electrodes so that their light does not result in unwanted background. In the past, too little attention has been paid to this. But many spectrographers have recently augmented the train on their optical bench with an adjustable mask and an additional condensing lens in order to mask out the electrodes.<sup>1,3</sup> At least one manufacturer (Bausch and Lomb) incorporates this optical improvement in their arc-spark stand.

On the subject of external optics, the new fused silica offered by Corning should be mentioned. Synthetically prepared, this material has about the same transmittance as natural quartz down to about 2300A where the latter starts to fall off considerably. It transmits about 80% of the incident light down to about 1800A. For the determination of traces of cadmium, lead, zinc, carbon, etc., this new glass may offer decided advantages.

With speed again in mind, a small spectrograph is preferred over a larger one. The f-number of spectrographs generally varies directly with their size so that the larger the spectrograph, the slower it is. But, the larger grating instruments should be used where their speed is greatest. In other words, the blaze of a grating will determine its speed so that a particular grating will

be many times as fast in one order than in another. If in the 3rd order, a particular spectrograph is five times as fast at 2881A than at any other order, the spectrograph should be set for the 3rd order for the determination of silicon in a limited sample. The slit should be set as wide as possible, consistent with the resolution required for the analysis.

One scheme to increase the speed of a spectrograph was recently tried successfully. A university needed the greatest possible speed in a spectrograph over a wavelength interval of about 100A. At the same time, a dispersion of 2.5 A/mm was specified. As has been mentioned, such high speed and high dispersion do not ordinarily go together. To increase the speed, a cylindrical lens (axis horizontal) was used right at the camera, condensing the light vertically as it emerged from the grating of the instrument. In other words, light on a 10-mm high slit wound up as spectral lines but 1-mm high, giving a theoretical 10-fold increase in illumination at the camera. While the scheme had the disadvantage of having a limited wave length coverage, it was shown to give this 10-fold increase in apparent speed for the wavelength interval of interest. Moreover, any 100A interval could be chosen by setting the instrument so that the desired range was behind the lens.

### THE PHOTOGRAPHIC EMULSION

With speed again the goal, a high-sensitivity photographic emulsion is required. An ideal emulsion would be extremely fast over several thousand angstroms and still retain good resolution and other characteristics. Unfortunately, compromises have to be accepted. Eastman-Kodak manufactures a number of emulsions having high speed over comparatively narrow wavelength bands. Examples are given below:

<i>Emulsion</i>	<i>Approx. W.L. Range, A</i>
SWR	60-2000
103-0	2300-5000
1-L	2300-8200
1-N	6500-8200

Eastman will also sensitize certain emulsions for the region 2100-2500A through the use of fluorescent dyes. The speed of 103-0 in this region may be so increased. This emulsion, so treated, is called 103-0, U.V.

Iford, Ltd. (England) provides emulsions which supplement Eastman's, especially in the region below 2300A. The Q-3 plates are the fastest in this region. Let us repeat that the region between 1850 and 2500A promises to have much more usefulness now that optics are available which transmit here.

One scheme that is sometimes used to increase the sensitivity of an emulsion is to "prefog" it. The characteristic curve (density vs. exposure) consists of a "toe" region at low density values followed by a straight line as the exposure level increases. Deliberately fogging a plate takes the emulsion out of its region of low sensitivity. This scheme, however, is seldom used because of the inconvenience in fogging a plate reproducibly.

Practically speaking, many experienced spectrographers use the 103-0 emulsion as a general-purpose tool when running tiny samples. It has a wide wavelength coverage and its speed, determined in a recent series of tests by A.S.T.M., ranges from 5 to 40 times that of Spectrum Analysis #1, depending on the wavelength.

In the visible region, mention should be made of the new Eastman emulsion, Tri-X. While its characteristics have not as yet been compared with spectroscopic emulsions, it seems to be much faster than the 1-L and 1-N types.

# NEW STEEL STANDARDS

## MILD RESIDUAL SERIES

A set of seven British Chemical Standard steel samples has just been released by the Bureau of Analysed Samples, Ltd. The set contains 19 elements determined by wet chemical as well as spectrochemical means and covers the ranges below:

** Al	0.008-0.066%	* Ni	0.01-0.23%
† B	0.001-0.01	** Pb	0.002-0.020
* C	0.15-0.23	† Si	0.19-0.37
* Co	0.01-0.25	* Sn	0.005-0.11
* Cr	0.02-0.18	† Ta	0.001-0.020
* Cu	0.01-0.21	** Ti	0.002-0.108
** Mg	Tr. -0.007	* V	0.005-0.10
† Mn	0.47-0.51	* W	0.01-0.28
* Mo	0.01-0.19	** Zr	Tr. -0.045
† Nb	0.01-0.15		

\* Standardized values.

\*\* Temporary certificate issued for these elements.

† Preliminary spectrographic results given for these elements.

Because of the urgent need for these standards, they have been made available with a provisional certificate pending the completion of the round-robin tests by committees of the British Iron and Steel Research Association and other analysts. As American distributors, we shall pass on to our customers the final certificates when issued.

The standards may be purchased in two forms. For spectrographers, rods 3/4" x 3" are available. (Incidentally, the Spex Petrey Stand Clamp #3300 is ideal for holding these rods.) For wet chemical standardization, turnings are available. They are listed as follows:

1210	Steel Standards, Mild Residual Series, set of seven rods 3/4" dia. x 3" long .....	\$120.00
1211	Steel Standards, Mild Residual Series, set of seven turnings, each 100 grams .....	\$130.00

## LOW ALLOY SERIES

The above standards are actually the second set issued by the B.A.S. primarily for the spectrographer. The first set of eight Low Alloy Steels contains 10 elements covering a wide range of composition as follows:

Al	0.027-0.57%	Mo	0.007-1.41%
C	0.1% nominal in each	Ni	0.048-5.15
Cr	0.044-3.07	Si	0.013-0.81
Cu	0.090-0.49	V	0.034-0.80
Mn	0.016-1.42	Sn	0.004-0.012

These Low Alloy Steel standards have been shown to yield excellent accuracy for a wide range of steels and are especially useful in those laboratories where occasional steel samples are run without prior knowledge of alloy type. An individual certificate accompanies each rod in which all analysis data are tabulated. In addition, a composite table of average values is printed inside the sturdy box in which the standards are packaged.

1200	Steel Standards, Low Alloy Series; set of eight rods 1/2" dia. x 6" long .....	\$92.00
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### A NOTE ON THE BUREAU OF ANALYSED SAMPLES, LTD.

Located in Yorkshire, England, the Bureau of Analysed Samples was formed in 1935 to ensure a continuous supply of British Chemical Standards—analytically standardized samples of irons, steels, non-ferrous alloys, ores, slags, etc.—which, since 1916, had been supplied by Messrs. Ridsdale & Co. Standards are prepared under rigorous laboratory conditions under the auspices of an Honorary Advisory Committee and over 100 cooperating laboratories representing independent associations, government organizations (our own National Bureau of Standards is included) and manufacturers, sellers and buyers. Approximately 100 standards are currently available.

## Some Examples

A few months ago, our laboratory was asked to determine the calcium content of a strain of amoebae. Biological studies were being conducted on the calcium uptake of these one-celled animals. For this purpose, radioactive labeled calcium compounds were to be used. But before data from the labeled calcium could be extracted, the biologists had to have figures on the original (or blank) level of calcium in the amoebae.

Our spectrographic problem was not only to see the calcium lines but also to provide quantitative results. The biologists cooperated by painstakingly pipetting 100, 250, 500 and 1000 of the amoebae into separate graphite electrodes. For the purpose, we preburned  $\frac{1}{4}$ " dia. electrodes with shallow cups to porosify them. The amoebae were transferred with a droplet of water. The water seeped through the porous electrode leaving the amoebae in the cup.

Incidentally, in the event the question comes up, the average calcium content of an amoeba turned out to be 0.0015 micrograms. Our results were obtained by preparing a working curve from standards of distilled water containing added amounts of a calcium salt. Blanks were carried through but, surprisingly, calcium was not detected in them.

But let us fill in the details of the analysis which was performed for Dr. I. M. Heller of Sloan-Kettering Institute. Our equipment consisted of a 1-1/2 meter Wadsworth mount Jarrell-Ash spectrograph, a dc arc and a Model 2000 Microphotometer. To obtain the maximum speed from the instrument, we used the widest of the three fixed slits (40-micron) and a condensing lens placed in front of the electrodes. The emulsion chosen was 103-0, the speed of which is some 30 times that of SA #1 at 4227A where the principal calcium line appears.

Because of the difficulties involved in preparing samples, we could not run moving plate studies to determine the optimum exposure conditions. But we reasoned that a short exposure at 8 amperes would be required to volatilize the calcium. Accordingly, we settled on 10 seconds.

A second example of an unusual problem involving a limited sample had to do with a surface coating on a prototype photomultiplier. We were handed two identical—or what appeared to be identical—tubes. We were asked to examine the coating on the inside surface of one of the tubes. On close scrutiny, and with a little imagination, it became evident that one tube did have a slight brownish tinge while the other was clear. This tinge was an evaporated coat of a photosensitive palladium-antimony alloy. A bead of the alloy had been placed in a boat in the tube. After the tube was evacuated, the boat was heated and the alloy evaporated. The question then was: does the evaporated coat have the same composition as the original bead, or did the antimony evaporate preferentially?

We attacked the problem by breaking open the tube and dissolving the deposit in a small volume of acid. This was then transferred to an electrode. A weighed chunk of the bead was dissolved similarly and placed in another electrode. The exciting thing about the spectrographic analysis was the results which we were able to calculate. First of all, we determined that the antimony had evaporated preferentially as had been suspected. The original bead was 4:1 palladium; the deposit 10:1 antimony. From the weights and line readings, we were then able to determine the weight of the deposit and also its thickness.

## Conclusions

This subject of trace-element analysis should rightly be considered just begun rather than concluded. Metallurgists, biologists,

chemists and physicists are rapidly learning that many of the properties that they had assigned to materials are grossly inaccurate because the materials themselves were not pure. Perhaps the most spectacular example of this is the transistor properties of silicon. One of the more common elements, silicon had been studied and its electrical properties supposedly known for decades. But when *pure* silicon was prepared, within the past two years, it was found to have decidedly different properties, so wonderfully different that silicon transistors will surely displace many vacuum tubes in the years ahead.

The role of the spectrograph will become increasingly important as pure materials find their place in technology. Its problems will become increasingly taxing as demands are made to maintain the quality of these pure materials.

A point-by-point Check-List for improving sensitivity is recapped below:

## Improving Spectrographic Sensitivity When Sample Size is Limited

### I. Spectrograph

- A. A small spectrograph of high speed should be used.
- B. When a grating spectrograph is employed, it should be used in the order where its speed is fastest.
- C. The slit of the spectrograph should be wide.
- D. The external optics should be so arranged as to direct and focus as much light as possible on the slit.
- E. A lens and diaphragm with which the images of the hot electrodes may be removed is helpful.
- F. For detecting elements whose spectral lines fall below 2500A the new Corning glass should be used in place of natural quartz for lenses, cover glasses and prisms.

### II. Source

- A. For most work, the dc arc with the sample electrically positive is used.
- B. For special purposes, the cathode layer technique will prove helpful.
- C. The copper spark method may be used to advantage on solutions.

### III. Photography

- A. The emulsion with the highest speed in the wavelength region of interest should be employed.
- B. Coating the emulsion with a fluorescent dye will improve its speed between 2100 and 2500A.
- C. Prefogging an emulsion will improve its apparent speed but, at best, is a complicated procedure.

## References

- 1 Strock, L.W., "Spectrum Analysis with the Carbon Arc Cathode Layer," Hilger, London (1936).
- 2 Fred, Mark; Nachtrieb, N.H.; Tomkins, F.S., *J.O.S.A.* 37: 279 (1947).
- 3 Twyman, F., and Simeon, F., *Trans. Opt. Soc.*, 31: 169 (1929-30).
- 4 Green, Milton, *J.O.S.A.*, 41: 862 (1951).

# tricks of the trade

## FACING-OFF METAL DISCS

Although A.S.T.M. methods usually call for a lathe for facing off metal samples, many laboratories prefer an inexpensive belt sander which saves a great deal of time. The procedure is to label and use a separate coarse and fine belt for each different type of alloy. Belts are conveniently stored by hanging above the sander. Sample discs are first sanded on the coarse and then on the fine belt. With a little practice, a very smooth surface is readily obtained. Care should be taken to spark the samples with the sanding scratches perpendicular to the direction of the optical bench. The best reproducibility is so obtained. We shall be glad to furnish additional information.

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## ROTATING SECTOR

We've all seen the rotating sector wheel "stand still" as the spark frequency matched its speed. Even the ac ripple of a dc arc will cause this stroboscopic effect. To avoid it, many spectrographers continually throw the motor switch on and off or adjust the speed-control rheostat. This may be done automatically by using a small "blinker", the gadget used on store displays and Christmas tree lights. Buy three or four—they cost about 25¢ apiece—and pick out one whose on-off cycle is right for your motor. They require a warm-up period of about a minute before they stabilize.

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## PETREY STAND MIRROR

When sparking to a small sample, it is not easy to direct the discharge to the exact spot desired. A small mirror placed on the optical bench is helpful here. It is also useful for getting as many burnings as possible out of a metal disc before it is refinished.

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## DC ARC REPRODUCIBILITY

It's an old story that dc arc results are not as precise as those using other methods. Part of the reason is the tendency of the arc to wander around the wall of the electrode cup. At a recent talk before the Society for Applied Spectroscopy, Dr. R. L. Mitchell told of his work with a narrow (about 1/8" diameter) electrode. The excellent reproducibility he reported on thousands of biological samples per year prompted several spectrographers to try using narrow electrodes. They were amazed at the improvement in reproducibility so obtained. In addition, they stated that cyanogen band interference was reduced because less graphite is "seen" by the spectrograph.

We are now supplying a typical electrode for such uses. Manufactured by United Carbon Products, it is 1/8" diameter by 1-1/2" long with a crater 1/4" deep by .096" diameter. It is listed

under our catalog number 4020 and sells for \$19.00 per 100. The counter electrode is simply a 1/8" rod broken off.

## PHOTOGRAPHIC EMULSIONS

Over the years Eastman-Kodak has done a magnificent job in providing spectrographers with emulsions to fit their applications. As a result, a whole host of emulsions is available. While the experienced spectrographer knows which are the common, most used types, the inexperienced man finds it hard to decide which emulsion to use for a specific job.

From Eastman's standpoint, they would like to see us use fewer emulsions so that, by concentrating their production efforts on these, they can provide a more consistent product. While we cannot advise spectrographers on exactly which emulsion to use—so much depends on individual preferences—we can tell, from our own sales records which ones are purchased in largest quantities. Naturally, these are the ones we stock for immediate delivery in most instances.

- SA #1 An emulsion of high but somewhat non-uniform contrast with wavelength; 2300-4300A; most popular emulsion for quantitative use; speed moderate.
- SA #2 Moderate, uniform contrast; 2300-5000A; most widely used for qualitative analysis; speed moderate.
- 33 Moderate, uniform contrast; 2300-5000A speed faster than SA #2.
- 103-0 Moderate, uniform contrast; 2300-5000A; extremely fast; useful for trace element analysis of small samples; somewhat grainy.
- 103-F Moderate, uniform contrast; 2300-6800A; extremely fast; useful for trace element analysis of small samples; extended wavelength range; somewhat grainy.
- I-L Moderate contrast; 2300-8200A; very fast. Used for visible region analysis.
- I-N Moderate contrast; 6500-8200; very fast. For visible and near infra-red lines such as the principal lines of K, Cs, Rb.

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## NEW DIMENSIONS OF REGULAR GRADE GRAPHITE RODS

Spectrographers will be happy to learn that the National Carbon Co. regular grade graphite rods in both the 1/4" and 3/16" diameters, are now ground down to exactly the same diameter as the high purity grade rods. In the past, special collets had to be used to machine the regular grade rods and, even then, the rods frequently jammed in the collet or the wall thickness was not constant from one electrode to the next.

National Carbon Co. has just published a new catalog on preformed electrodes. About 40 shapes are now available as stock items.

Write us for a copy of the catalog.

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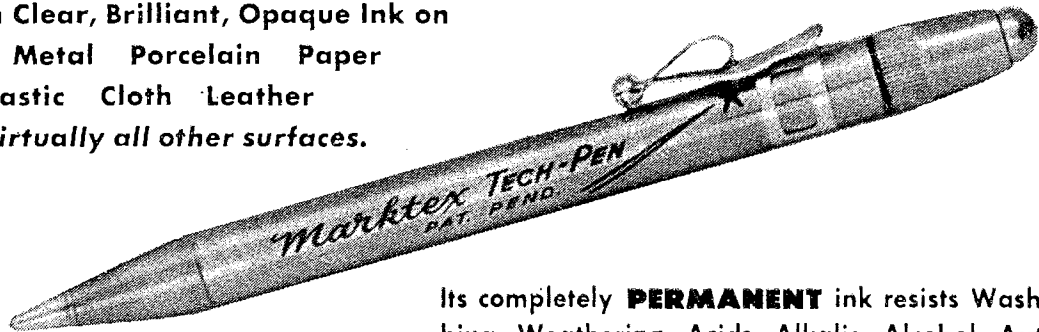
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| 3911 | Refills for Tech-Pen (designate color)  |        |
|      | Each .....  | \$ .7  |

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