QUANTITATIVE ANALYSIS WITH SEMIQUANTITATIVE STANDARDS

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THE primacy of the emission spectrograph in providing reliable analytical results has been jolted in recent years by atomic absorption and X-ray fluorescence; the former for determining trace amounts with speed and accuracy, the latter for determining elements in concentrations of 1% and above. But most instances of significantly better results by these methods are those for which some or all of the following apply:

1—sample composition is well established and standards are available or easily prepared
2—element concentrations are between 0.1 and 10%
3—samples are presented in convenient form (solutions, for instance) making sample preparation unnecessary or quite simple
4—fewer than 10 elements are determined for a given analysis

But the emission spectrograph is still unsurpassed for:
1—determining the composition of miscellaneous unknowns such as deposits, sludges, ashed organic materials, rocks and ores which are difficult or impossible to analyze by other means
2—analyses of samples having elements in concentration ranges from 0.001 to 30% inclusive
3—handling a variety of shapes, sizes and physical forms of samples
4—analysis of samples containing more than 10 elements where reliable results are most quickly and easily obtained.

Our aim here is to acquaint newcomers to the field with standards and techniques which make the task of analyzing problematical samples less formidable. Perhaps en route we may also reacquaint the more experienced hand at such analyses with another trick or two.

ALL instrumental methods suffer because the very physical and chemical properties which establish the uniqueness of every element known to man, and make them valuable to his technology, also conspire to prevent him from quantitatively analyzing for one in the presence of another without standard materials closely matching the samples. This stumbler, known as the matrix effect, causes the same concentration of any element in two different materials to produce spectra that appear so grossly different that estimates of concentration, based solely on spectral intensity, may prove embarrassingly incorrect by a factor of 100 or more. Early spectroscopists showed their aversion for, and their inability to cope with, the matrix effect by reporting results in strangely ambiguous terms; strong, moderate, weak were deemed adequate substitutes for percent concentrations.

This unlikely system of reporting persisted for many years after it was discovered that by properly diluting samples with suitable pure substances, such as graphite, the matrix effect could be reduced appreciably. And there was adequate reason. First, the most widely used spectrographs were prismatic. Reflected light from the faces of the collimating lens and prism gave rise to "Littrow fog" which swamped weak spectral lines. This, plus the fact that the sample had to be diluted about 100 times, was sufficient to reduce analytical lines below their sensitivity limit. Grating spectrographs, despite—or because of—their inherently better signal-to-noise ratios, were for the most part tools of the atomic and molecular spectroscopist. Even so, unblazed gratings, with relatively poor dispersion were responsible for a good deal of "fog" in their own right and offered little improvement in the situation. The dc arc, usually powered by low, unregulated, and not well filtered current, contributed its share to non-reproducible results. Electrodes, often home-made and not to very stringent tolerances of purity and dimensions, added to the confusion.

WHAT then is the sum of the changes which brought spectrochemical semiquantitative analysis to the present state of "perfection"?

The introduction of long focal length plane grating spectrographs with finely ruled, high efficiency, blazed gratings improved the S:N attainable by about 2 orders of magnitude and added immeasurably to spectroscopists' ability to look at a highly diluted sample and extrapolate back to the original concentrations with a satisfactory degree of accuracy. The constant-current dc arc has largely supplanted earlier types and has improved reproducibility. The inert atmosphere of a Stallwood Jet has simultaneously improved S:N ratios by suppressing the background and cyanogen bands from the spectra and, at the same time, increased reproducibility of exposure by uniformly volatilizing elements from the sample. In many cases the Stallwood Jet has actually increased the sensitivity of elements by other means, not clearly understood, but somehow associated with energy transfer characteristics of the atmosphere surrounding the arc. Finally, graphite products, now manufactured under carefully controlled conditions to assure purity and mechanical tolerances of the highest order, need no longer be of any concern to the spectroscopist as a source of possible error in semiquantitative analysis.

ONE of the pioneers in the development of semiquantitative methods of analysis was C. E. Harvey (1), then at Applied Research Laboratories, who determined the sensitivities of many of the common elements in various matrices by analyzing synthetic mixtures of the salts of these elements through hundreds of carefully controlled experiments. Plotting the intensity ratio of a line to its background vs. % concentration for each element in the presence of other elements, Harvey calculated concentration factors which could be applied directly to densitometric measurements and yield results directly in concentration without the usual requirements for constant standardization. Unfortunately, experimental results were valid only for a single element matrix and failed to provide meaningful results for more complex materials. Further, wide variations in S:N of individual spectrographs were not accounted for.

It also soon became apparent that much greater sensitivity, for many elements, resulted from reduction of background (rather than its enhancement to pose as an internal standard which Harvey advocated). To our knowledge these shortcomings were never overcome and the method is seldom applied today.

Through the years since then other workers recognized that predicting the effects of the matrix on other elements in the sample, as Harvey did, was less precise than reducing these effects. For example, ASTM still recommends a method developed by Smith and Matarella (2) at least 15 years ago. In their method the sample is diluted 24:1 with a Li_2CO_3-graphite matrix, where Li also acts as an internal standard, and compared with synthetic standards containing up to 22 elements. Determinations of concentrations from 0.1 to 100% are possible with accuracies of from 5-17%.

F. M. Smith (3) developed a method with Gallium as the internal standard in a graphite-germanium oxide base. Instead of a single dilution with the buffer/diluent, he made multiple dilutions of the sample so that they might more nearly approximate his standards containing 20 elements. Smith reported precision of 15% and accuracy to within 20% of the amount present.

The most elegant procedure, to date, developed by Tingle and Matocha (4) (5), of Alcoa, provides for the greatest reduction of the matrix effect by fluxing the sample with lithium tetraborate. Thus all prior sample history is removed and a wide range of materials is accommodated by the method. The lithium acts as an internal standard and, by sparking the sample instead of arcing it, improved precision is realized. Six major constituents from 0.1 to 70% (as the oxides) and about fifteen minor elements are routinely determined by this method at Alcoa laboratories throughout the country with precision and accuracy of 1-2%.

Each of the methods described briefly have a common failing (with the exception of Harvey's which was described above). They are all designed to meet the specific needs of a particular laboratory. Despite their good to excellent accuracy and precision, none has been advocated for work with completely unknown samples.

THE Spex Standards and methods are designed with just that kind of complete unknown in mind. Judging their popularity from the rate of reorder, we conclude they are busily at work turning out acceptable to excellent results in agricultural, ceramics, electronics, metals, mining, petroleum, and pharmaceutical industries.

When introduced in 1954, Spex Standards contained 43 elements in either a graphite or zinc oxide matrix at 0.1, 0.01, 0.001, and 0.0001% concentration levels, and were derived from Spex Mix which contained the same common elements each at a 1.34% concentration. (Incidently, a forerunner of Spex Mix was "Medication," a mixture of about 20 elements prepared as a standard for a program at the University of Florida Experimental Agricultural Station directed by L. C. Gaddum and assisted by Richard Corrigan and Louis Rogers in the early 1940's.) They were meant for visual estimations with samples diluted 10, 100, or 1000 times with graphite. Estimates, multiplied by the proper dilution factor, provided results accurate within ½ to 2 times the actual amount present.

TABLE 1

Element	USGS G-1		USGS W-1		BAS Manganese Ore		NBS Lead Barium Glass		NBS Borosilicate Glass	
	A	B	A	B	A	B	A	B	A	B
Si	33.9	31	24.6	28	3.87	2.7	30.5	23	37.7	33
Ca	.97	.87	7.8	-----	.557	-----	.15	.16	----	----
Mg	.24	.25	3.98	3.8	.422	.4	.018	.017	.0157	.012
Fe	1.37	1.4	7.76	8.5	1.49	1.8	.034	----	.0532	.05
Mn	.021	<.05	.14	.18	51.3	-----	.068	.064	----	----
Al	7.54	6.7	7.94	8.7	.641	.73	.095	.076	1.03	1.0
Ti	.144	.16	1.647	.85	.012	.03	.06	.10	.016	.01
Cr	-----	-----	-----	-----	.893	.84	----	----	----	----

In Column A appear the % concentrations as standardized. In Column B appear the % concentrations as determined. The first two standards are ground rocks prepared by the U. S. Geological Survey; the others are by the British Bureau of Analysed Samples, Ltd. and the National Bureau of Standards.

TABLE 2

ANALYSIS OF NBS BOROSILICATE GLASS (a) AND B.A.S. MANGANESE ORE (b) WITH SPEX G STANDARDS

Method	Si, %		Mg, %		Fe, %		Al, %		Ti, %		Mn, %	
	a	b	a	b	a	b	a	b	a	b	a	b
Unweighed	M	M	.02	M	.2	M	M	M	.2	—	—	M
Weighed	30	7	.03	.2	.1	.8	1	1	.05	—	—	15
Dens. Meas.	33	2.7	.012	.4	.05	1.8	1.0	.73	.01	—	—	Not determined
Standardized Values	37.7	3.87	.016	.422	.053	1.49	1.03	.64	.016	—	—	51.3

M = Major

Unweighed refers to method requiring 40 minutes elapsed time for both samples.
Weighed refers to method requiring 80 minutes elapsed time for both samples.

Dens. Meas. refers to method requiring 150 minutes elapsed time for both samples.
Standardized Values are those furnished by the National Bureau of Standards and the British Bureau of Analysed Samples, respectively.

We have since expanded the series to encompass 49 elements. In addition, while graphite was very suitable as a diluent for preparing samples and standards to analyze inorganic materials, it did not always serve particularly well in analyzing organic materials. The pioneering work of Weaver and Brittain (6), in analyzing petroleum products with Li_2CO_3 as buffer/diluent and internal standard, led us into the development of L Standards for analyzing organic materials. They also contain the 49 elements of the Spex Mix in a lithium carbonate base. G. W. Stander (7) had pointed out the utility of still another matrix, ZnO, for organic materials. Our initial Z Standards resulted from that work. As increasing numbers of laboratories were becoming interested in the Rare Earths and Noble Metals, we kept pace with two new sets of standards containing 16 Rare Earths, in Li_2CO_3 and 10 Noble Metals, in graphite.

THE fastest method of evaluating data available in the form of an exposed spectrographic plate is still visual examination. Such estimation of sample composition, with or without Spex Standards, is plagued by a number of problems not the least of which is the limitation imposed by the experience of the analyst. Another difficulty with the customary 4-part Standards, each diluted 10:1 with the matrix, is the ease with which a concentration level can be misjudged when the sample line intensity falls between steps of the standards. Changing contrast factors of spectroscopic emulsions with wavelength can further compound the problem of interpolation.

All of these factors can be overcome if estimations are taken out of the hands of the analyst and trusted to machines. Thus, in 1962 Spex analyzed five standard materials by methods conforming very closely to our visually oriented procedures, except that: densitometric measurements were made of analytical lines to reduce errors of judgment in interpolation between standards, and a lithium line served as an internal standard. The results of that analysis published in THE SPEX SPEAKER, Vol. VII, No. 2, are repeated in Table 1. We have never treated the data statistically but analyses, with minor exceptions, fall well within the ½-2 times the amount present limits set by acceptable semiquantitative procedures at that time.

Despite these very encouraging densitometric results we noted that spectroscopists seemed to prefer their visual procedures; perhaps because, at the time, they were not being pressed for better results. Economic considerations always play a role in the administration of any laboratory. Thus, if one analysis can be obtained with an accuracy of ½-2 times the amount present in 40 minutes, would it be worthwhile to undertake a method promising 20% of the amount present, if that would take 150 minutes? Many laboratories thought not.

To emphasize time considerations, in 1965 we reanalyzed the NBS borosilicate glass and the BAS manganese ore by four methods involving a sequence of more precise techniques and, of course, increased time expenditure. Table 2 is reprinted from THE SPEX SPEAKER, Vol X, No. 1.

In the unweighed method a scoop of material was mixed with a scoop of graphite, as diluent and buffer, arced and compared visually with our G Standards. The second technique was essentially the same except for the weighing and multiple dilution which provided a graded series of samples. The densitometric analysis with which these visual results are compared were taken from Table 1 data. At no time did the analyst estimating visually have access to the densitometric measurements or results.

NOT until the summer of 1968 were we impressed with the importance of accuracy even at the expenditure of greater analytical time. A vendor-customer dispute developed over the purity of electronic alloys. Although both analyses were based on the Spex G Standards, the accuracy was not confining enough to resolve the question of whether or not the product met specifications. Naturally, we were pleased to cooperate.

Our investigations began with a review of the analyses presented in Tables 1 and 2. It occurred to us that the 4-part standards failed to take into account two of the basic properties of spectroscopic emulsions. The first is the emulsion latitude that rarely permits analyses over a concentration range greater than 100 to 1. Our standards with concentrations varying by a maximum of 1000:1 outstripped the emulsion by one order of magnitude. The second was the non-linear characteristics of emulsion calibration curves obtained for %T values below 5 and above 85%. This was evidenced by the fact that our 1 ppm standard invariably showed transmission values for many analytical lines greater than 85%; the 1000 ppm standard invariably less than 5%. As a consequence, more often than not, analytical curves had to be drawn on the basis of only two values. Somehow we had to increase the number of plottable concentration values in order to assure a suitable analytical curve.

Someone recalled that the amount of silver in the developed image of a line on a photographic plate was directly proportional to the energy transferred to it during the exposure. Would integrating the line help? Yes, it did. When we took the average of the areas under a typical analytical line in four exposures of each of our G Standards and plotted them against concentration we were rewarded with remarkably smooth curves, Fig. 1, encompassing at least three points. A recording microphotometer with a Disc integrator provides the best integration. Lacking the latter, however, one may estimate area fairly well by determining that of the trapezoid sketched lightly about a "typical line" as presented by the microphotometer. In Fig. 2, the hatched area under the trace above the line C must be about equal to the two hatched areas between the tracing and lines A and B, for best results. Wings and background can be ignored as long as uniform treatment is given each line in that regard.

Helpful as this procedure seemed to be in improving the precision of the measurements and expanding the analytical curve, it lacked one essential ingredient to make it convenient in practice: day-to-day repeatability. It required a new set of analytical curves for each plate prepared. To generate these wasted too much time; we searched on, faced with the dilemma we started with—how to gather more points onto an analytical curve.

The best way to accomplish that, it seemed to us, was to prepare standards containing intermediate concentration levels between existing 10:1 dilution steps. In order to plot equally spaced points on the common log-log analytical curves a $\sqrt{10}$ factor seemed appropriate but difficult to implement. We rounded off to a factor of 3. Thus, a new series of standards was born, although not tested. 1968 came to a close with the scoreboard reading:

COMPARISON OF 1954 AND 1969 STANDARDS

	G Standards	G-7 Standards
DILUTION STEPS	0.1, 0.01, 0.001, 0.0001	0.1, 0.033, 0.01, 0.0033 0.001, 0.00033, 0.0001
ANALYTICAL METHODS	visual estimation transmittance meas. integrated intensities	visual estimation transmittance meas. integrated intensities intensity ratio meas.
ACCURACY	±30-50%	±15-30%

Plotting %T and integrated intensity **vs.** %C once again generated some interesting curves for Mg and Fe (Fig. 3 and 4). Note that: 1) it was possible to plot as many as five concentration steps on an analytical curve, 2) precision improved markedly with integrated intensities and 3) an unexpected bonus accrued—it became simpler for an analyst to determine concentrations in unknowns by visual means because interpolation between steps, matching sample to standard, was more precise.

As a test of this, we assisted a completely inexperienced "eyeball" analyst, by locating the proper analytical lines in sample and standards, and asking him to estimate concentrations in a Burnt Refractory sample, NBS #78. Results:

TABLE 3

NBS #78 BURNT REFRACTORY ANALYZED USING SPEX G-STANDARDS 4 PART AND 7 PART

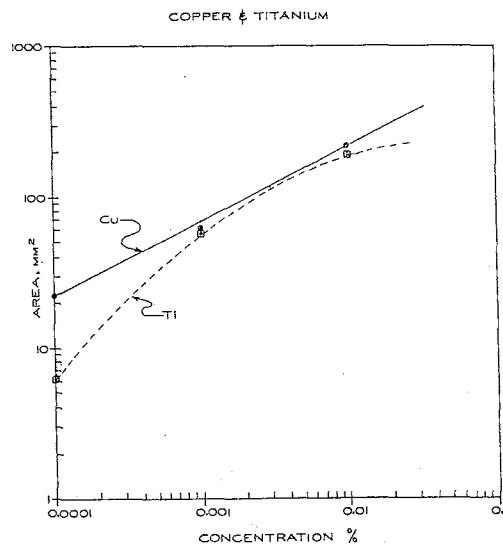| Element	Nominal %	Estimated % with 4-part stds.	Estimated % with 7-part stds.
Fe	0.55	3	1.0
Mg	0.307	1	0.5
Si	9.67	15	10.0
Al	37.0	30	30.0

Fig. 1 Integrated intensities vs. % conc. for titanium and copper in 4-part Spex G Standards demonstrating the ability of this method of recording results to encompass at least three concentration values of the standards and thus provide a more reliable analytical curve than by plotting % T vs. % conc., a method previously advocated.

Having come this far we opted for a possible further improvement in precision by adding an internal standard to the now 7-part G Standards. Indium oxide was tapped because it was present in our standards at the 0.1% level and so was easy to incorporate at this stage of our experiment; it also seemed to offer a few more possible lines than Li, for instance. As materials for internal standardization should be, In was seldom sought, was available in high purity and was easily handled. Fig. 3 and 4 illustrate the demonstrable improvement in precision over other methods of plotting analytical curves. The ultimate test, of course, required the analysis of "unknowns" with the new standards. Results are tabulated in Tables 4 and 5.
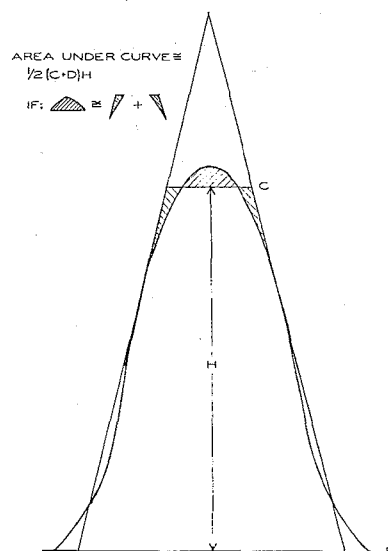

Fig. 2 Means employed for obtaining integrated intensity values in the absence of a Disc integrator. Ignoring any asymmetry of the line, draw a triangle tangent to the line and estimate area under the curve by calculating the area of the trapezoid.
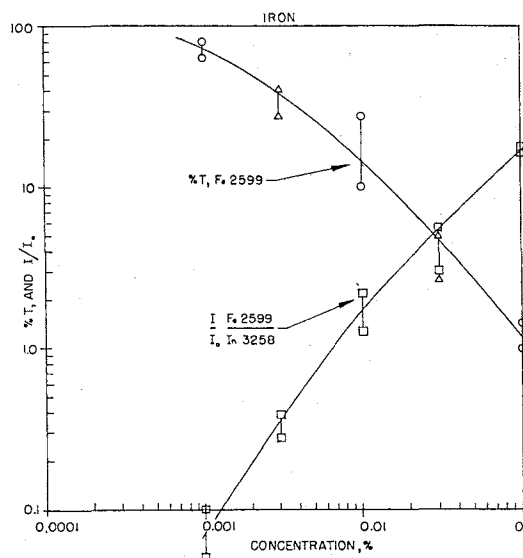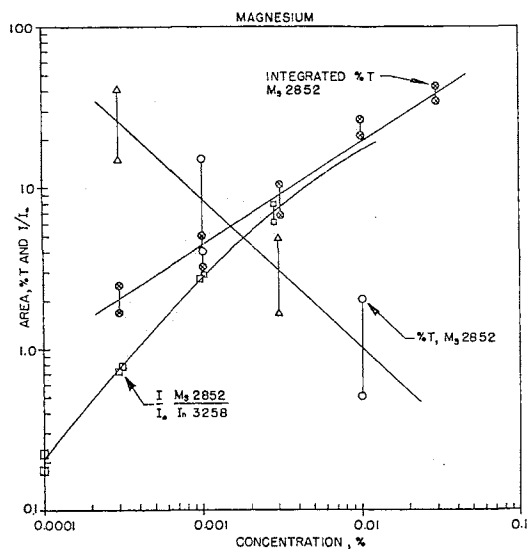
Fig. 3, Fig. 4 Magnesium and iron analytical curves drawn from data collected on both 4-part and 7-part G Standards. They illustrate the improved precision attainable when progressing from % T vs. % conc. through integrated intensity vs. % conc and finally to I/I₀ vs. % conc. The final method is possible with the new 7-step standards because they contain an internal standard, indium.

TABLE 4

ANALYSIS OF NBS BOROSILICATE STANDARD, #93, USING SPEX G-7 STANDARDS WITH INTERNAL STANDARD

Element	Nominal Conc., %	Analyzed Conc., %	Average Error, %	Coefficient of Variation
Fe	0.053	0.044	17.0	14
Mg	0.016	0.014	12.5	18
Si	37.7	36.1	4.3	2.4
Al	1.03	0.99	3.9	37

TABLE 5

ANALYSIS OF NBS BURNT REFRACTORY, #78, USING SPEX G-7 STANDARDS WITH INTERNAL STANDARD

Fe	0.55	0.469	16.3	0
Mg	0.307	0.430	40.0	42
Si	9.67	11.55	19.5	3
Al	37.0	37.0	0	0.6

When compared to the analysis presented in Tables 1 and 2, of the same material (NBS #93), decided improvement in accuracy is evident for at least two elements, Mg and Si; for other elements the analyses indicate a standoff. The overall picture looks very good. With only a single exception accuracies hover about 20% of the amount present and precision has the same order of magnitude. We believe that an unexplained diluted sample bias, not the standards, is the cause of discrepancies in precision and our work toward improvement continues. Further reports will follow to keep you abreast of progress.

TABLE 6

CONDITIONS OF ANALYSIS

Spectrograph	3.4-m Ebert, 600 gr/mm grating, Order II
Slit width	20 microns
Spectral Range	2400-3400 A
Filter at slit	2 step — 100% T, and 25% T
Source	dc arc, 10 amp
Time	arc to completion, ~90 sec
Atmosphere	30:70 Ar/O₂ in Spex Stallwood Jet
Photographic Emulsion	SA #1
Electrodes	3/16″ undercut lower, rounded upper

Table 6 summarizes the conditions judged optimum for most elements in most materials. Samples are screened to -200 mesh. Dilution is with a graphite-InO₂ mixture, prepared from -100 mesh, SP-2X graphite. Relatively coarse particle size inhibits materials from blowing out of the electrode cup. Of course, too coarse a mixture may exhibit segregation.

W E HAVE introduced a new series of standards for semi-quantitative spectrochemical analysis of diversified materials which provide for the determination of many elements to ±20% of the amount present by: increasing the number of standards in a series, decreasing the concentration steps from 10:1 to 3:1; and, by adding an internal reference, Indium oxide. The advantages accrued in providing such standards apply not only to the more sophisticated methods which require emulsion calibration and plotting of normal analytical curves as I/I₀ **vs.** % C, but also, to general cases where visual estimates, or % T **vs.** % C curves are drawn and to another, not commonly used but recommended procedure, in which integrated intensities **vs.** % C are plotted.
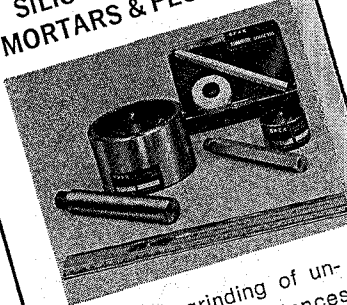
A complete analytical scheme, including sample preparation, arcing techniques and general recommendations may be found in our current catalog of Spectrochemical Standards and High Purity Materials. If you have not received a copy please request one.

References:

1. C. E. Harvey, "A Method of Semiquantitative Spectrographic Analysis," Applied Research Laboratories, 1947.

2. R. W. Smith and S. P. Matarella, chapter in "Methods for Emission Spectrochemical Analysis," ASTM, Philadelphia, 1968, 128.

3. F. M. Smith, AEC Research and Development Report, HW-59864, 1959.

4. W. H. Tingle and C. K. Matocha, Anal. Chem., 30, 494, 1958.

5. SPEX SPEAKER, VI, No. 4, 1961.

6. J. R. Weaver and R. R. Brattain, Anal. Chem., 21, 1038, 1949.

7. G. W. Standen, Ind. Eng. Chem. Anal. Ed., 16, 675, 1944.

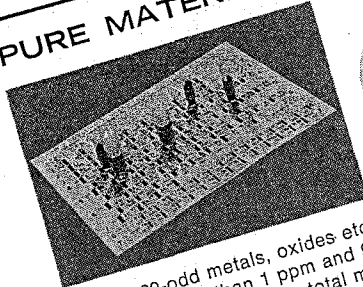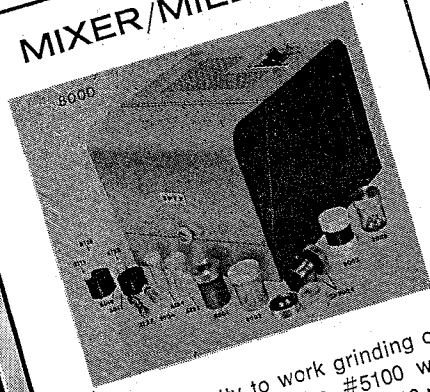# May we give you a hand?

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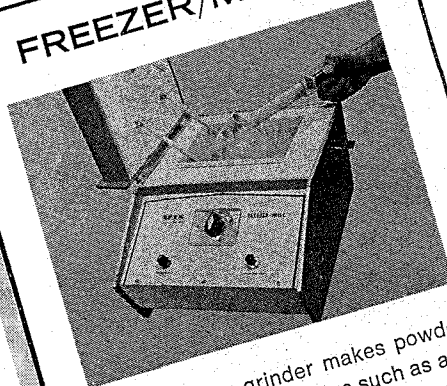
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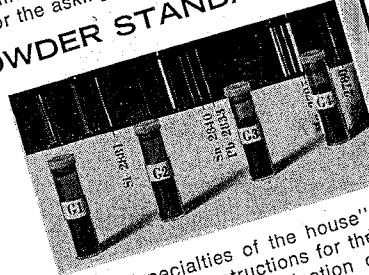
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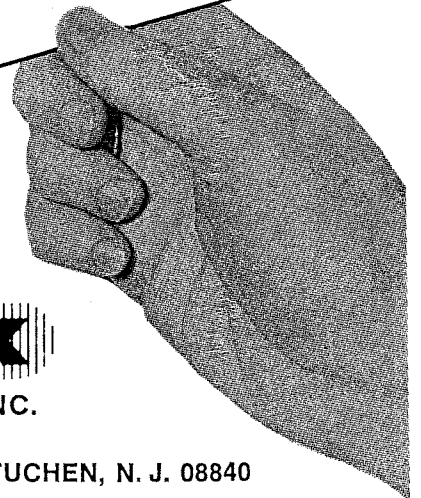
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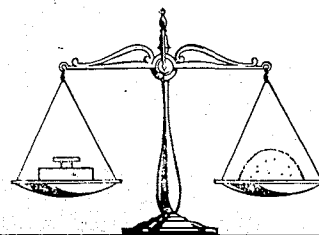
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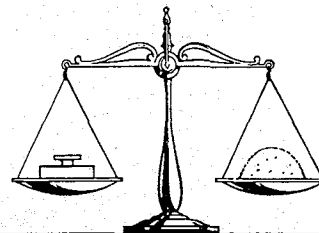
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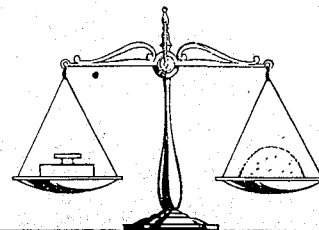
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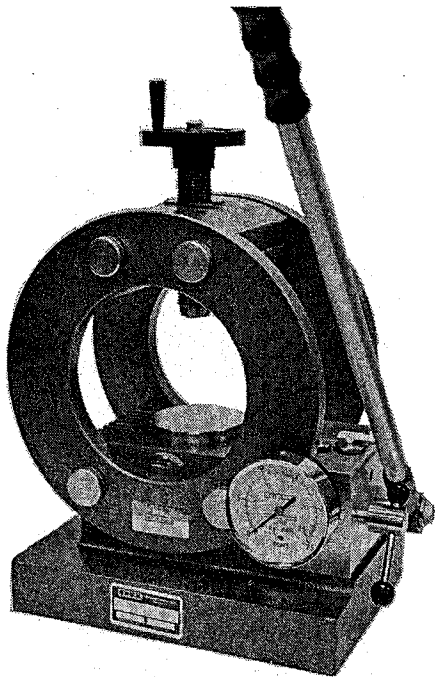
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THE 1967 WILLIAM F. MEGGERS AWARD

On March 4, 1969, at the Pittsburgh Conference in Cleveland, the 1967 William F. Meggers Award was presented by Dr. C. L. Grant, President of the Society for Applied Spectroscopy, and Mr. A. J. Mitteldorf, President of Spex Industries, Inc., to Drs. Michael L. Parsons and James D. Winefordner. This award, originally called the Journal Award and sponsored since its inception seven years ago by Spex Industries, Inc., recognizes the outstanding paper published in Applied Spectroscopy during each calendar year. The paper honored for 1967 was entitled "Optimization of the Critical Instrument Parameters for Achieving Maximum Sensitivity and Precision in Flame-Spectrometric Methods of Analysis."

Dr. Parsons is now Assistant Professor of Chemistry at Arizona State University and Dr. Winefordner is Professor of Chemistry at the University of Florida.

When asked what had prompted them to undertake this research, Drs. Parsons and Winefordner indicated a belief that many flame methods were devised with little or no attention to the optimization of important parameters. In this paper, they attempted to provide the description of an approach to the selection of parameters to be optimized and a statistical means of optimization. Selection of this paper for the Meggers Award surely indicates that they accomplished their goal.

Left to right: A. J. Mitteldorf, C. L. Grant, M. L. Parsons, J. D. Winefordner.