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Speaker

FLUXING TECHNIQUES IN EMISSION AND X-RAY SPECTROSCOPY

T. J. CULLEN, U. S. Metals Refining Co., Carteret, N. J.

A. J. MITTELDORF, Spex Industries Inc.

Almost as knotty a problem as the population explosion is the technical literature explosion. In soaring numbers, papers appear in many languages, in well-known as well as obscure journals, in house organs, in trade magazines, in government publications, in texts. From this confusing assortment the scientist must ferret out the good from the bad ideas, those with current or potential use in his laboratory from inappropriate ones. But locating information is hard enough; recognizing the good ideas in print is even harder.

And so, when a paper by Tingle and Matocha (1) appeared routinely in "Analytical Chemistry" a few years back, its impact was not immediately felt. The authors thoroughly outlined a universal method of spectrographic analysis in which quantitative figures could be attained on almost any sample with no prior knowledge of its composition and with but a single set of standards. True, they did not claim to devise the technique in "stroke-of-genius" style. But, by eclectically blending the best features of already known methods, modifying and simplifying them at each step, they brought to a high state of perfection what has come to be known as the lithium borate technique. In this, samples are first fluxed with $\text{Li}_2\text{B}_4\text{O}_7$ to form a glass which is then crushed, mixed with graphite powder and pelletized. Run against similarly prepared standards on a direct reading spectrograph, samples are analyzed for a large number of elements with precision and accuracy fully equal to the best routine wet chemical methods, at a fraction of the time and cost.

Emission Techniques

The origins of the technique are uncertain. Having been so long used for dissolving wet-chemical samples, fluxing certainly must have been transplanted to the spectrographers' laboratory very early. Yet, as a method for eliminating variables, the first references appear to be those by Applied Research Laboratories who, under contract to the U. S. Signal Corps Laboratories in the late '40s, used a flux consisting of one part of lithium carbonate to one part of boric acid for the analysis of mica and titanate refractories. In 1952, Hasler and Barley (2, 2A) of ARL applied the method to the analysis of steel-making slags, the composition of which controls the speed of conversion of pig iron to steel in the open hearth furnace. In 1954, Gillette, Boyd and Shurkas (3), of ARL too, reported that the method could be extended to nickel ores and slags for the determination of nickel (0.07-2%), iron, silicon, and chromium. They also stated that the methods had been applied

to the analysis of zinc and copper slags and concentrates as well as iron ores. Here, then, was an indication of the universality of the method—its potential application to the analysis of almost any material with little, if any matrix effect. A further hint as to the feasibility of the method for production control was the authors' time study showing that 20 slag samples could be analyzed in one day using a Quantometer. This represented 13 minutes of preparation time and 6 minutes for the actual analysis in duplicate.

In 1953, Price (4), of the British Cast Iron Research Association, proposed a fluxing method in which borax alone was employed. From our 1961 vantage point, one of its disadvantages is immediately obvious: the need for a very high (100:1) flux ratio. To offset the loss of sensitivity resulting from such a high dilution, Price used dc arc excitation, rather than the more precise spark, for the determination of the major constituents in slags and ores. When, in contrast, Landergren and Muld (5), of the Geological Survey of Sweden, put their chips on lithium tetraborate, they were able to use a flux ratio of but 7:1 for the analysis of similar substances. Spark excitation then served for the major elements and coefficients of variation around 3% of the amounts present were obtained.

Lounamaa (6), also in Sweden, in 1955, extended the lithium borate fusion method to the analysis of copper and lead making slags. Following the lead of the workers at ARL, he used spark excitation for the determination of copper, lead, zinc, aluminum, calcium, iron, magnesium, and silicon. Having tried a number of fluxing agents before settling on lithium borate, Lounamaa pointed out its superiority in forming homogeneous melts with most non-metals and, at the same time, requiring small flux ratios. He also stated that, provided the fluxing temperature is maintained below 900° , losses of zinc and lead are not serious. Because they form a separate phase, sulfides cannot be fluxed directly. They have to be either converted to sulfates or fluxed with another material such as pyrosulfate.

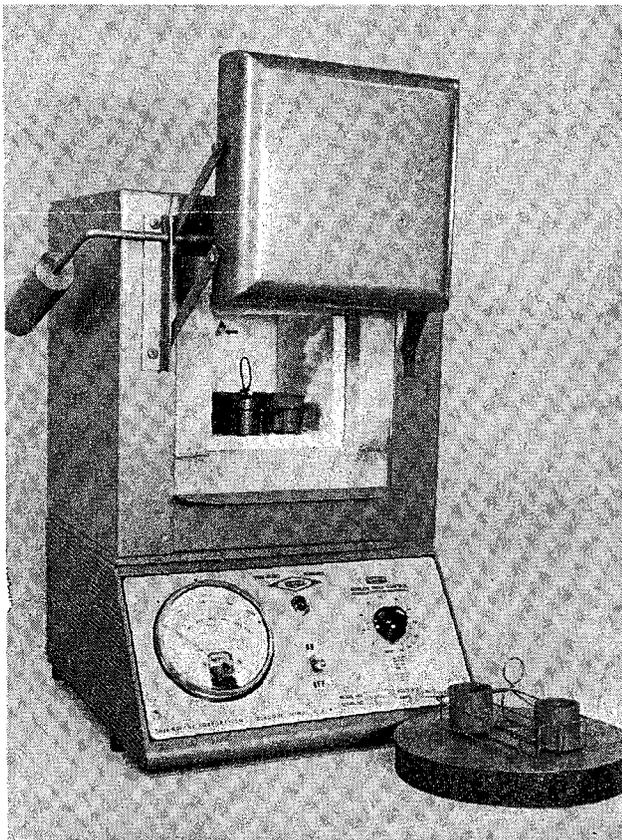
Feldman (7), in 1960, suggested ammonium bisulfate as a fluxing agent. His application differs, however, from the others reported here in that his purpose was to solubilize and so homogenize samples rather than convert them to a common matrix. After fusion of the material to be analyzed, the flux is vaporized at under 300° , a temperature so low that most elements normally considered volatile are retained. The residue

can then be dissolved and run against standard solutions similarly prepared.

While a flux can probably be found to form a glass with almost any material, lithium borate works with many materials in the form of oxides, silicates, carbonates, etc. It is thus of special interest in the handling of slags, ores, concentrates, rocks, and minerals. Metals can often be treated after being dissolved in a suitable acid. Carbon in samples presents no problem. It can either be burned off beforehand or, during fluxing, will rise to the surface where it will burn off.

Often NBS samples serve as convenient standards as well as check materials in the technique. Such samples as the various glasses, refractories, clays, and limestone are found useful for determining the common elements. However, since the method is designed to be quantitative, most spectrographers prefer to brew their own standards, designing them to resemble closely their anticipated samples.

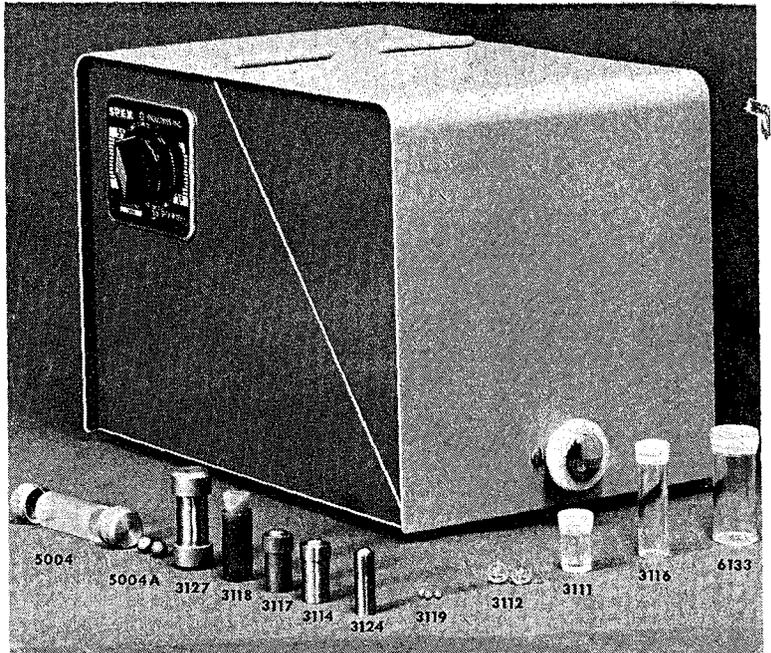
In the latest version of the Alcoa optical emission method (8)*, Tingle and Matocha choose a flux ratio of 8:1, taking 0.200 g of sample to 1.60 g of lithium borate. The two are mixed for a few seconds in an agate mortar then transferred to a graphite crucible. Several of these are loaded into a wire tray which is placed in an electric furnace at 1050°C for 8 minutes. The tray is removed and the beads are allowed to solidify in the crucibles.



7150 Furnace with 7151 Crucible Racks and 7152 Graphite Crucibles.

After being shattered by a few hammer blows, each bead, together with a tungsten carbide ball, is placed in a tungsten

* As herein described the procedure, adapted to many commercially available items, differs somewhat from Alcoa's. As such, neither Alcoa nor Tingle and Matocha are responsible for or endorse it.

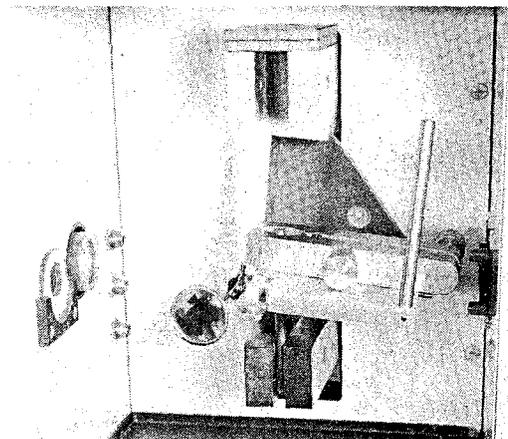


5000 Mixer/Mill showing the various grinding and mixing vials available.

carbide grinding vial and ground in a Mixer/Mill until, from experience, it is known that 80-85% will pass a 200 mesh screen. This eliminates an extra procedural step specified in the original paper (1) in which a -300 mesh cut was screened out for analysis. The newer procedure has the additional advantage of working with coarser material which is easier to remove from the grinding vial.

To 0.300 g of the unscreened material is added 0.900 g of pelleting grade graphite (National SP-1) and the two are mixed in a plastic container together with a plastic ball again in the Mixer/Mill. The entire sample is finally briquetted at 80,000 psi in a 1/2" (13 mm) dia. die. Once prepared the pellet should be sparked immediately or kept desiccated because it is hygroscopic.

When sparked in an arc stand, the pellet is conveniently held in a spring clip which, in turn, fits the lower electrode jaws. Alternatively, a Spex No. 3300 Clamp may be used to spark the pellet in a Petrey stand. Either way, the pellet may readily be resurfaced after a burn by rubbing it lightly with Whatman No. 2 filter paper.



Pellets 1/2" dia. are conveniently held on a Petrey Stand using the No. 3300 Clamp.

A high-voltage uni-directional spark is applied from a National L-3966 graphite counter electrode. Typical spark parameters are 0.007 microfarads capacitance, 20 microhenries inductance, 4 breaks per half cycle. A 5 second prespark is followed by a 20 second exposure on the Quantometer used at the Alcoa Research Laboratories.

A unique adaptation of this method is that using adhesive tape as described by Danielsson, Nilsson and Sundkvist (9) this past summer. Here instead of briquetting, the powdered bead is fed to a moving tape and sparked further along as it passes between two electrodes. After extensive studies, they decided on quite a complex buffer for their analysis of slags. It consists of lithium borate, strontium borate, cobalt oxide, and beryllium carbonate. Strontium is added to counteract the increased intensity resulting from samples containing alkalis. Cobalt and beryllium serve as internal standards. With a claimed precision of better than 1% of the amounts determined, this tape method appears to be still another milestone in the progress of emission spectroscopy.

Although Alcoa developed the lithium borate technique for the analysis of their raw materials: bauxite, schist, bayer mud, sinter feed, and sinter mud for silicon, iron, titanium, sodium, calcium as well as aluminum, others have modified it for their own materials. Charles Hodgkins, of the Esso Research Center in Linden, N. J., is one. He has successfully adapted the method to the analysis of petroleum ashes. Using a Quantometer, too, he now reports highly accurate figures to supplant the semi-quantitative ones furnished in the past. Recently, Mr. Hodgkins put two rock samples "through the mill" as though they were oil ashes. Identical standardization was used on these samples, which, unknown to him, were the well-known G-1 and W-1 pulverized rocks of the U. S. Geological Survey. His results, shown in the following table, were excellent but, needless to say, they could be improved still further if his standards more closely resembled the samples.

	W-1		G-1	
	Accepted	Esso, spec.	Accepted	Esso, spec.
Fe	7.76	7.26	1.37	1.41
Si	24.6	28.8	33.9	41.4
				(extrapolated)
Al	7.94	8.24	7.54	7.63
Na	1.53	1.87	2.46	2.65
Mg	3.98	4.88	0.24	0.2
Ca	7.85	6.58	0.97	0.79

X-Ray Techniques

Meanwhile, parallel investigations have been conducted on fusion techniques for X-ray analysis. Claisse (10), in 1957, returned to borax and, like Price, used a 100:1 flux ratio. In the range above 1% concentration, he reported excellent results for the determination of iron, manganese, and zinc in all minerals with the exception of sulfide bearing ones. Incidentally, he chose to use the face of a cast disc rather than grind and press a pellet. Others have found that, unless temperature conditions are exactly proper, such discs have a tendency to crack on casting.

Just this past summer Andermann, then of Applied Research Laboratories, having had extensive experience with lithium borate for emission spectroscopy, submitted a suggested method to ASTM based on this flux for the X-ray analysis of finished cements. Using what he terms a Minimum Flux Ratio of but 1:1, he claimed sensitivities down to 0.01% for oxides of manganese and strontium; to 0.05% for titanium and phosphorus; to 0.02% for sulfur; to 0.1% for magnesium. The method has

been detailed in a recent issue of *Analytical Chemistry* (11). Especially impressive is the improvement in precision obtained with a fluxed sample vs. one ground and briquetted but unfluxed. Andermann further showed that, with no correction factors applied, acceptable accuracy could be attained, but this could be improved appreciably with a simple correction factor so that the mean errors for, say, silica determinations, would be reduced to but 2.2% of the amount present.

What ground rules can be applied for the use of various fluxes in X-ray analysis? Borax, a low absorbing material, is particularly suited to the determination of low atomic numbered elements producing soft radiation. A 100-fold dilution, as used by Claisse, is sufficient to remove or substantially reduce matrix effects for these elements. On the other hand, higher atomic elements will exhibit matrix effects in a straight fusion, as pointed out by Claisse. He suggested the addition of 2g of barium oxide to 10g of borax (to 100 mg of sample) to offset the effect.

Fusion with lithium borate is similar to that with borax from the X-ray standpoint. The flux ratio can be varied to effect a compromise between matrix effect and sensitivity. Both are used primarily with non-metallic samples and both materials are somewhat hygroscopic requiring occasional resurfacing and storage in a desiccator.

Many variations have been proposed. In Andermann's method for cements (13) recently submitted to ASTM, 1.00 g of sample and an equal quantity of lithium borate are mixed in an agate mortar before being transferred to a graphite crucible. Fusion is carried out at 940°C for 5 minutes after which the molten contents are cast in a copper ring on top of a massive copper block. After grinding the resulting disc, the mixture is loaded into a die on top of a wad of cellulose paper pulp and pressed at 20,000 psi for 30 seconds. The cellulose and a spray of acrylic lacquer (Krylon) around the edges of the pellet make for a less brittle briquet. Adler and Rose, at the U. S. Geological Survey in Washington, suggest blending into the mixture about 7% boric acid as an alternate means of strengthening pellets. Using a pressure of 40,000 psi, they routinely obtain solid, easily handled discs and feel that, so prepared, they are superior to those containing cellulose. Incidentally, for the analysis of rocks and ores, Adler and Rose prefer a flux ratio of 5 parts lithium borate to one of sample. Before being pressed, the material is almost entirely -325 mesh, obtained with the Spex No. 5004 tungsten carbide grinding vial.

As contrasted with both borax and lithium borate, potassium pyrosulfate, recommended by Cullen (12) in 1959, is highly absorbing to X-rays and so is capable of removing severe absorption and enhancement effects of elements determined as well as interfering elements.

Nothing could be more varied than the samples coming in for analysis by Cullen at United States Metals Refining Co. which buys copper-bearing scrap, paying for it on the basis of a carefully monitored copper assay. Heaps of insulated wire, brass and bronze turnings, automobile radiators, plumbing and the like are tossed into a furnace from which, smelted, they emerge as metal ingots and slag. The slag, which may contain 0.5-70% copper, 0-70% iron, 0-30% silica, 0-20% zinc lends itself beautifully to fusion by potassium pyrosulfate followed by X-ray analysis. A flux ratio of 50:1 was found necessary to overcome inter-element effects while, at the same time, permitting the determination of low copper with the required accuracy. A ratio of 25:1, by contrast, was found insufficient to ward off absorption and enhancement effects of the highly variable samples.

In practice 100 mg of slag is fused in platinum at around 400°C together with 5.00 g of potassium pyrosulfate. When silica is appreciable sodium fluoride, added to the fusion, volatilizes the silica. After cooling, the bead is readily ground using polystyrene vials (Spex No. 6133) and three 3/8" dia. acrylic balls (Spex No. 3112) for a period of ten minutes. Because of the high volume of production samples, Cullen grinds four samples simultaneously in his No. 8000 Mixer/Mill using a No. 8011 adapter. Briquets 1-1/4" dia. are prepared at 25 tons total pressure (about 40,000 psi).

The technique permits the determination of copper to an accuracy of $\pm 0.5\%$ in the range from 0.5-100% copper. Lead and zinc are also routinely determined and eventually may be salvaged. Iron, tin, and nickel down to around 0.1% and arsenic, which could volatilize in higher melting slags, are measured routinely, too. For each of the elements, a straight-line calibration curve is obtained with no inter-element effects. The instrument is set up once only, with a tungsten-target tube, scintillation counter, and lithium fluoride crystal. The relatively poor sensitivity for arsenic is the result of the use of the K-beta line, the K-alpha being interfered with by lead L-alpha. No background corrections are needed except in the case of lines appearing in the area of the white radiation, i.e., tin K-alpha. Not only are slags analyzed by this technique but semi-refined copper, flue dusts, blister copper, cupola black and other plant intermediates upon the composition of which depends plant production. It is interesting to note that the accuracy of the zinc determination exceeds that using routine wet chemistry.

One of the advantages of pyrosulfate—its ability to fuse metals and sulfides directly—has already been mentioned. Another is its relatively low fluxing temperature (300-700°C) compared with 900° for lithium borate. Its main drawback is in the determination of low atomic numbered elements which, absorbed by the potassium, suffer a loss in sensitivity. Standards of pyrosulfate must be kept desiccated although some X-ray spectrographers have experimented with some success, with surface treatment such as plastic sprays and even pelletizing between sheets of 1/4-mil Mylar film.

Among the various fluxes the choice will depend on both the material being analyzed and the elements to be determined. It is entirely conceivable that separate fusions might be required when traces as well as major constituents of both high and low atomic number are to be determined.

As illustrated by Cullen's pyrosulfate technique, the preparation of samples for X-ray spectroscopy is basically similar to that for emission with the exception that the sample is finally briquetted in a large die, 1-1/4". Normally, as in emission, the die need not be evacuated for X-ray spectroscopy. In the new vacuum spectrometers, however, a pellet so produced may "explode". Here, of course, an evacuable die is advantageous to prevent entrapped air bursting the pellets.*

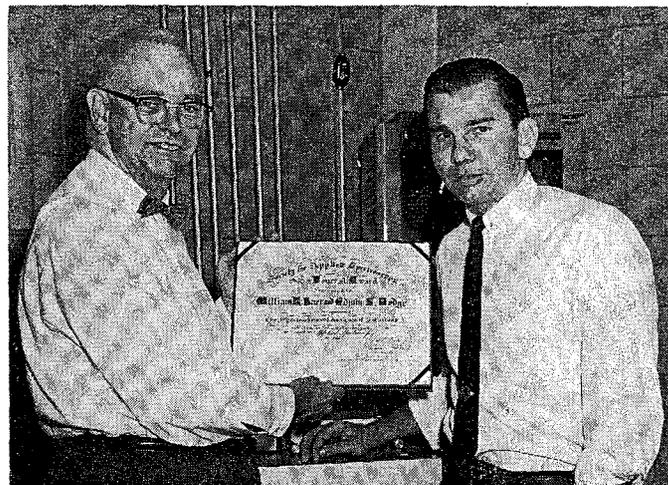
* We are indebted to Mr. Michael Yanak of International Nickel Research Laboratories for the conclusions reached in an experiment aimed at trying to find out if there was any other advantage in pressing X-ray pellets under vacuum. Working with NBS Burned Magnesite, Argillaceous Clay and Burnt Refractory, he found that, although evacuation did produce denser pellets (12% denser on the average), X-ray intensities of light elements (magnesium, aluminum, silicon, titanium) showed no corresponding increase.

Variations are the spice of a spectrographer's life and we apologize for having omitted many useful and interesting ones that must have occurred to others. Whatever the variations, fluxing methods are bound to mushroom in a great many laboratories where the problem of getting more accurate results faster grows as fast as the ever-increasing backlog of samples.

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11. Andermann, George, *Anal. Chem.*, 33, 1689, 1961.
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HODGE AND BAER RECEIVE AWARD



The first recipients of the Spex Industries sponsored annual SAS award of \$100.00 for the paper describing the best piece of research or development in the field of spectroscopy as reported in *Applied Spectroscopy* during 1960, are W. K. Baer and E. S. Hodge of Mellon Institute, for their paper entitled "The Spectrochemical Analysis of Solutions: A Comparison of Five Techniques." Recognizing the bewildering choice of methods open to spectrographers when they need to set up a specific method, the authors carefully evaluated the most common ones pointing out their good points and drawbacks. Unprejudiced and based on solid data, their advice will be heeded for a long time to come by spectrographers who, having had the ground work laid, are certain to be deeply appreciative. Presentation was made during the Eastern Analytical Symposium in New York City on November 15, by N. F. Gordon, President of SAS.

EQUIPMENT USED FOR FLUXING TECHNIQUES

Price List

(Pictured on page 3)

7150 **Furnace**, Thermolyne. Temp. range to 1100°C (2000°F); intermittent, 1000°C (1850°F) continuous; stepless control allows settings to be made quickly and accurately; pilot light indicator; working space 4" x 4" x 4-1/2" deep. Specify either 115 v, 50/60 cy. or 230 v, 50/60 cy.Each \$151.00

7151 **Rack and Handling Tongs** for 6 No. 7152 graphite crucibles, heli-arc welded high-temperature wire; tongs for placement in and removal of rack from furnaceSet \$ 19.00

7152 **Crucible, graphite**, 1-1/4" dia. x 1" long, 9 ml capacity.
100 \$ 20.00
300 \$ 45.00

3300 **Petrey Stand Clamp**, for holding pellets and other small or irregular shape samples for sparkingEach \$ 33.00

3301 **Pellet Holder**, for mounting pellets in electrode jaws; rhodium plated brass.
13 mmEach \$ 6.00
1/2 inchEach \$ 6.00

6005 **Lithium tetraborate**, anhydrous100g \$ 4.00
1 lb. \$ 11.50

4062 **Graphite powder**, SP-1, for briquetting use, highest purity1 oz. \$ 6.00
4 oz. \$ 20.00
8 oz. \$ 35.00
1 lb. \$ 60.00

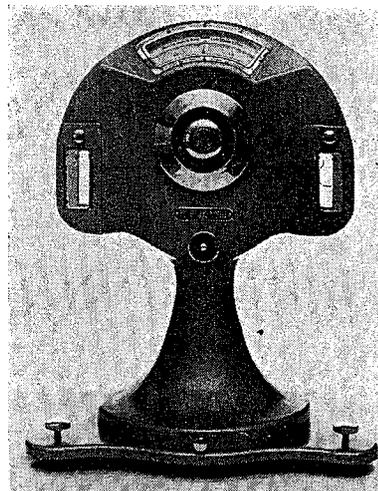
5000 **Mixer/Mill**, for grinding and mixing samples, 115 v, 50/60 cy., or 230 v, 50/60 cy. (specify), accommodates vials up to 3/4" dia. x 2-3/8" long, dual timer with settings up to 6 min. or 60 min.Each \$143.00

5004 **Tungsten carbide grinding vial**, consisting of two tungsten carbide lined end caps, six replaceable center sections of Lucite, two 1/4" dia. tungsten carbide ballsEach \$ 30.00

3116 **Vial**, polystyrene with polyethylene cap 5 ml capacity.
100 \$ 5.00
1000 \$ 40.00
5000 \$150.00

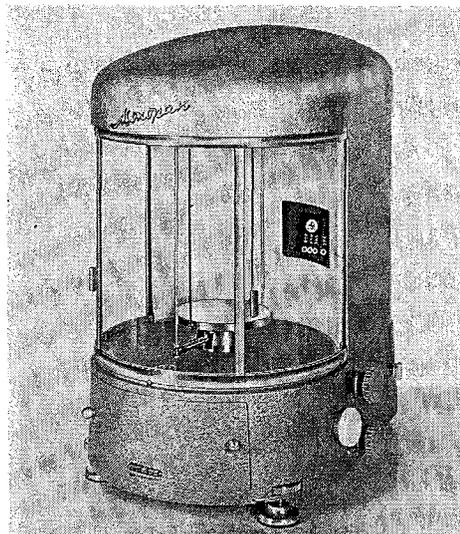
3112 **Ball**, clear acrylic
100 \$ 1.80
1000 \$ 12.00
5000 \$ 50.00

Standards We regularly formulate special standards for individual requirements. For the Lithium Borate technique, we cordially invite your inquiries. To give you a typical example of the cost of such standards, we will prepare 100 g each of four standards containing 12 common elements mixed in an unfused $\text{Li}_2\text{B}_4\text{O}_7$ base in a ratio to be specified for \$96.00. For formulating more costly elements the price may be somewhat higher.



7001 **Sauter Ultramatic Balance**, for fast, repetitive weighings of materials up to 2.5 g with an accuracy of 0.5 mgEach \$288.00

7004 **Sauter Monopan Balance**, Constant Load Substitution, for Analytical Weighings up to 230g. This balance has an accuracy for differential weighings within optical range of 0.03mg and a limit of error over entire range of 0.10mg. Easy operation is insured with suspended side sliding doors; extra-large, clear projection scale right in field of view of load pan is recessed to prevent distracting reflections; 3-position arrestment control protects knife edges from damage during "pre-weighings." Complete with cleaning accessories and protective plastic cover; specify 110 or 240 vacEach \$760.00



SHIPMENT AND STORAGE OF PLATES AND FILM

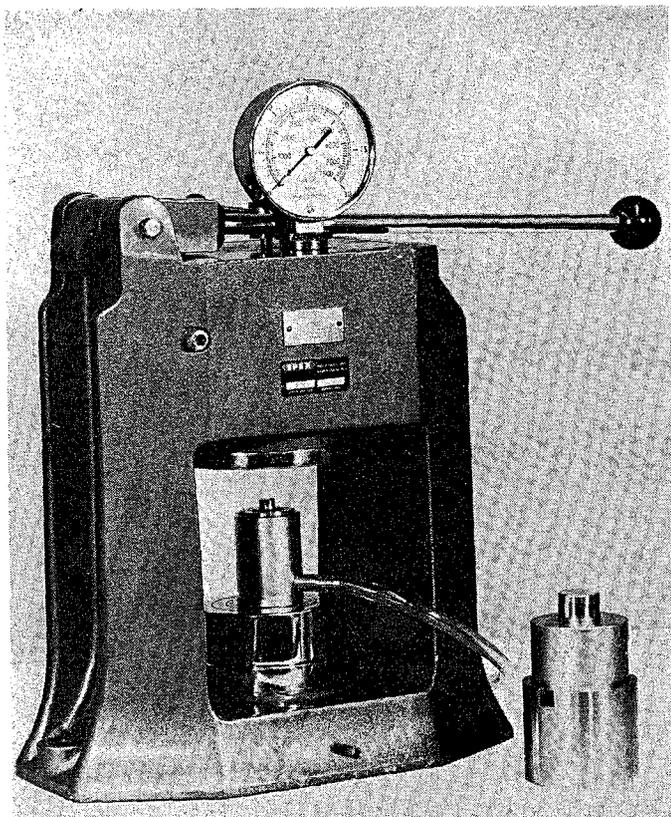
It is well known that the shelf life of photographic emulsions is strongly temperature dependent. Mr. Cyrus Feldman of Oak Ridge National Laboratory showed (*Spec. Acta*, 7, 349, 1956) that over a year there was a significant deterioration of SA #1 films kept at room temperature while portions of the same batch kept at freezer temperatures showed very little change. With this in mind, most spectrographers store their plates and film at low temperatures. To reduce condensation on the cardboard box, it is first sealed in a polyethylene bag.

While plates can thus be preserved once received, what can the spectrographer do to protect his plates during shipment? The problem for most emulsions exists only in hot weather when they may travel for days inside a truck or be stored on a loading platform in the baking sun. Where possible, the best solution is to order enough plates and films in cool weather to protect in your freezer during the warmer months. Incidentally, this procedure has an additional advantage: we offer a substantial discount on plates ordered in case lots and film in lots of 6 rolls or more.

DELIVERED PRICES EFFECTIVE NOVEMBER 29, 1961

	Case East*	Lots West*	3-Doz. East*	Pkg. West*	Single Doz. Pkg.
4"x10" Plates					
SA #1	\$142.50	\$152.50	\$19.20	\$20.70	\$7.75
SA #2	158.00	168.00	21.20	22.70	8.50
33	158.00	168.00	21.20	22.70
103-O)					
103-F)	355.00	365.00	17.00
1-N)					
2"x10" Plates					
SA #1	180.00	190.00	12.85	14.35	5.20
SA #2	198.00	208.00	14.20	15.70	5.70
33	198.00	208.00	14.20	15.70
103-O)					
103-F)	430.00	440.00	11.00
1-N)					

*East or West of the Mississippi River.



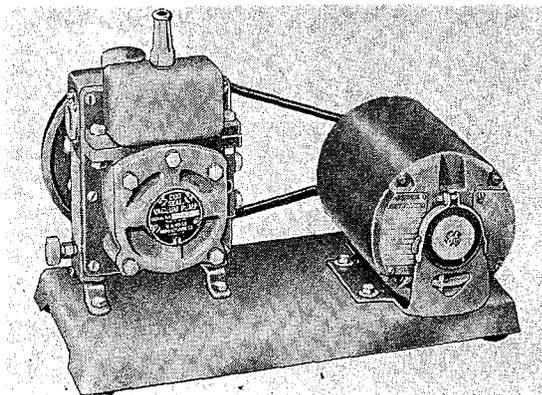
RH30-1 Press, 30-ton hydraulic, vertical opening 5-3/8", platen stroke 1"; 15" wide x 16" high x 9" deepEach \$575.00

KB-01 Evacuable Die, may be used either with or without vacuum; optically flat, polished and parallel hardened tool steel faces; produces pellets 13 mm dia. x up to 1/4" thickEach \$152.00

3622 Die, optically flat, polished and parallel hardened tool steel faces; produces pellets 1-1/4" dia. x up to 5/16" thickEach \$215.00

3623 Evacuable Die, may be used either with or without vacuum; optically flat, polished and parallel hardened tool steel faces; produces pellets 1.235" dia. x up to 5/16" thick. Recommended for use with vacuum x-ray spectrometers such as PhilipsEach \$243.00

3624 Vacuum Pump, Welch Duo-Seal No. 1400B with belt guard (not shown), 1/3-hp. motor, 1725 rpm, open motor, base, pulley, belt tightening provision, supply of Duo-Seal oilEach \$148.00



LOW TUNGSTEN STEEL STANDARDS

The British Bureau of Analysed Samples, Ltd. has made available a new set of steels especially standardized for tungsten. Covering the range of 0.7-3.4%, they thus supplement the Mild Steel Residual Series which contain from 0.02-0.20% tungsten (plus small amounts of C, Si, S, P, Mn, As, Ni, Cr, Mo, Cu, V, Ni, Cr, Mo, Cu, Co, Sn, B, Ti, Al, Zr, Ta, Nb, and Pb) and the Low Alloy Steel Series which are standardized for Si Mn, Ni, Cr, Mo, V, and Cu but contain no W.

BSS 21-24 Low Tungsten Steel Standards, set of 4 rods, 3/4" dia. x 3" long; Tungsten 0.70%, 1.29%, 2.16%, 3.41%. Each contains about 0.02% C, 0.13% Si, 0.036% S, 0.014% P, 0.07% Mn, 0.02% Mo and 0.01% VSet of 4 rods \$ 67.00

BCS 281-284 Low Tungsten Steel Standards, granules, analysis as aboveSet of 4, 100 granules \$ 45.00

tricks of the trade

ANALYSIS OF DUCTILE IRONS FOR Mg AND Ni

Some time ago the Bureau of Analysed Samples released a set of ductile iron bars (Spex No. BSS 41-46) to permit direct sparking for magnesium and nickel, thus avoiding the chemical separation procedure normally used. Techniques for the analysis were developed by Mr. Argyle of the British Cast Iron Research Association, using both a Quantometer and a spectrograph with point-to-plane technique, a graphite counter electrode and a 3 mm gap. Other conditions are as follows:

Source—ARL High Precision, Capacitance 0.007 microfarads, no added inductance, 200 discharges per second, RF current 13 amp.

Line Pairs

Quantometric Analysis		Spectrographic Analysis	
Mg	2802.7	Mg	2802.7
Fe	2714.4	Fe	2799
Ni	2270.2	Ni	3101
Fe	2714.4	Fe	3100.7

Precision and Accuracy

Coefficients of Variation of Ni and Mg Determinations

B.S.S. No.	Mg, %	Coef. Var., %	Ni, %	Coef. Var., %
41	0.012	2.8	0.32	2.4
42	0.024	9.5	0.39	1.8
43	0.039	5.9	0.52	1.7
44	0.053	6.3	0.64	1.6
45	0.078	4.2	0.96	1.6
46	0.128	4.4	1.42	1.2

Accuracy of Ni and Mg Determinations Obtained by Use of B.S.S. Nos. 41-46

Mg %	Accuracy	Ni %	Accuracy
0.006	± 0.003	0.30	± 0.015
0.020	± 0.003	0.40	± 0.017
0.040	± 0.004	0.50	± 0.019
0.060	± 0.006	0.60	± 0.019
0.090	± 0.007	1.00	± 0.024
0.150	± 0.010	1.50	± 0.024

Method of Preparation of Samples

It is appreciated that the preparation of routine samples must be carefully carried out otherwise serious errors can arise from various causes.

Routine samples should, strictly speaking, be prepared in such a way that their microstructures match those of the standards. This can be accomplished by casting the metal in greensand or drysand molds without any chilling. At B.C.I.R.A. samples measuring 1.5" diameter by 3" long are cast in greensand molds and they find that this method of casting gives mainly pearlitic or ferritic matrices. They have no reason, however, to suspect that major microstructural differences have

USES OF THE NO. 8000 MIXER/MILL IN CEMENT LABORATORIES

(Furnished by Mr. W. G. Hime, Portland Cement Association Res. Labs., Skokie, Ill.)

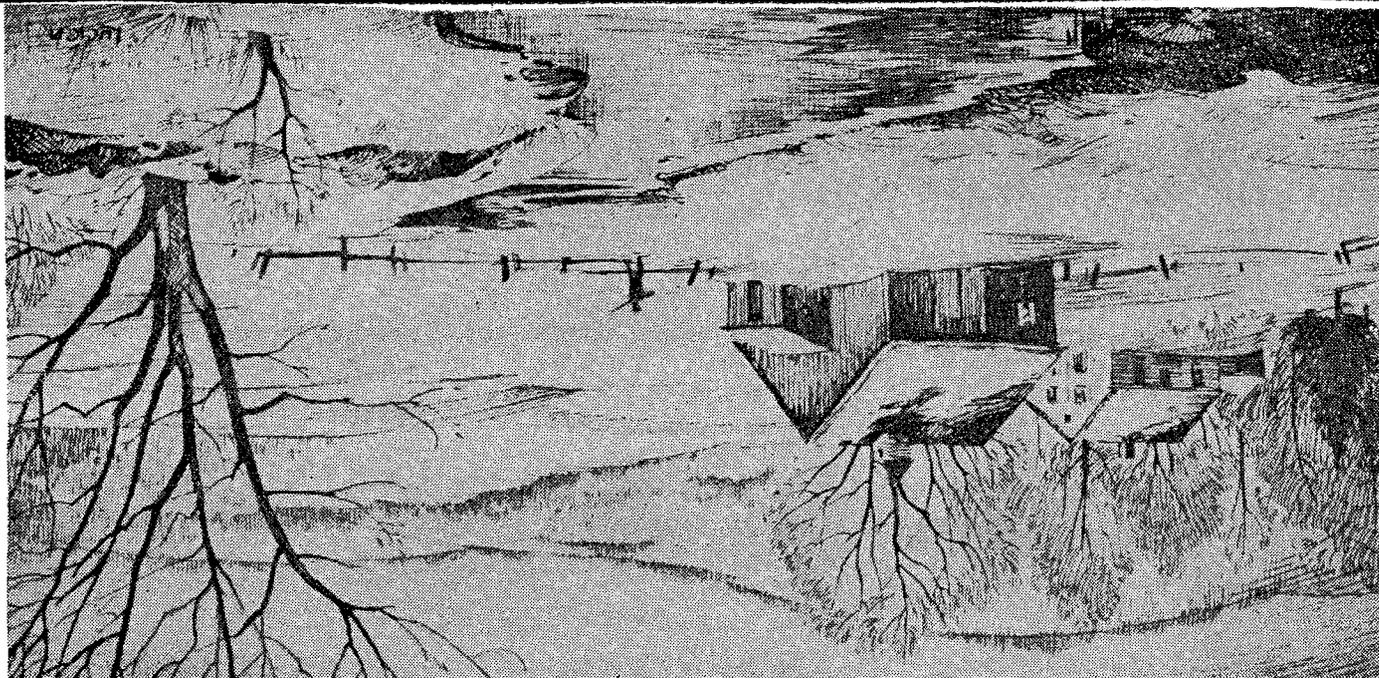
1. Mixing—to check cement formulations especially for setting properties. Plastic jars (No. 8002) and balls (No. 3112) are used.
2. Grinding
 - A. Prior to chemical determination of free lime. Three grams of the raw cement are ground in the No. 8001 tool steel vial for 2 minutes. An alcohol-glycerine extraction is made of the material, most of which passes -325 mesh. The advantage of this grinding method is that a closed container is used for a short period of time reducing the pick-up of CO₂ from the atmosphere.
 - B. Prior to X-ray spectrochemical determinations of Fe, Mg, Al, Ca and Si. Five to 15 grams of raw cement are ground in the No. 8004 tungsten carbon grinding vial using two WC ball-pestles. Ultimate particle size of 200-325 mesh is satisfactory for a tungsten-target x-ray tube but smaller particle size may be necessary with a chromium-target tube.
 - C. Prior to x-ray diffraction for the qualitative and quantitative determination of compounds. Grinding is usually done dry although grinding in a slurry of 1,1,1-trichloroethylene, producing finer particles, is being studied.
 - D. Thixotropic nature. Slurries in water are made to determine the thickening properties of different formulations.
 - E. Analysis of raw materials. Dolomite and calcite rocks are ground dry prior to chemical and X-ray analysis.

ANALYSIS OF FOILS

Mr. A. Liebman, RCA Tube Division, Harrison, N. J., suggests a method for analyzing thin metallic foils. The foil, ranging in thickness from 1 to 5 mils, is attached using double adhesive faced cellophane tape to platform and sparked as it rotates. The platform acts as a heat sink and, by rotating, prevents sparking through. It is of course necessary to have the foil contact the rotating platform electrically. This is conveniently done by leaving the center portion of the foil free of the adhesive tape. Although we built a special large platform for his work, it should be possible to use the No. 3402 platform together with our Combination Analyzer set up as a Platrode. Here, the platform is offset vertically to permit sparking to the periphery rather than the center.

any influence on the determination of nickel and magnesium and in order to clarify the position they are proposing to make comparisons between unchilled and chilled magnesium-treated irons and they will inform us of any conclusions which they may reach.

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