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Speaker

COMMERCIAL SPECTROGRAPHIC STANDARDS APPLIED TO THE ANALYSIS OF HIGH-PURITY CHEMICALS

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When a high-purity material is required for research study, the cost of analytical characterization may be of little moment. In contrast, commercial offering of a high-purity chemical demands that the analytical strategies adopted do not price the product out of its intended markets. The authors and their collaborators speak of "practical analysis of high-purity chemicals" to delineate the pressing economic factors underlying such cost-sensitive analyses. During the past few years at J. T. Baker, an attack has been mounted on the economic broad-based lot characterization of both inorganic and organic chemicals in high-purity forms (1-7). By the term "high-purity chemicals" we refer to compounds with a *total* detectable impurity content of less than 0.05%, that is, less than 500 parts per million. Products introduced by this company so far include clinical laboratory standards (4), ULTREX[®] chemicals for advanced applications in analysis and materials science (1-2), and so-called MOS-Low Sodium grade acids and solvents intended for the fabrication of metal oxide semiconductor (MOS) devices. Well-characterized high-purity chemicals at modest cost are being welcomed across the spectrum of the laboratory and advanced technologies.

It is noteworthy that the higher the purity of a chemical produced by a batch process, the more imperative is the assessment of each discrete lot and, in our view, the more important the disclosure to the customer of the actual analytical values and nature of the methods by which they were obtained. For acids (6) and other corrosives and for atmosphere-sensitive products, analysis only after final packaging is appropriate; thereby, any contamination of the product or interaction with the container is included in the analytical values.

The assessment of a high-purity chemical should not be based on a single analytical technique or on the determination of a limited group of impurities. In this sense, so-called spectrographic grade chemicals are looked upon as of *special* purity, not high-purity because an emission spectrograph

detects cations primarily. For an inorganic chemical to be certified as "high purity" the determination of both metal and non-metal impurities is considered essential. Also, the chemical must be subjected to further general tests, such as particulate matter, loss on drying, or residue on ignition.

The selection of practical approaches to the determination of impurities in high-purity chemicals involves a number of factors, including the total sample that can be allowed for analysis and the costs in terms of manpower and instruments. In a continuing program procedures are preferred that can be applied to a variety of compounds with little or no modification. Survey techniques that allow the simultaneous detection or determination of a number of impurities are especially valued. For trace metals and a few non-metals, arc emission spectrography represents, in our view, the comprehensive survey method of first choice. With a single sample, over 25 elements can thereby often be detected or determined. This survey method obviously must be backed up by other techniques where special or general problems are encountered. Other important but more restricted techniques in our studies include atomic absorption photometry, cold-vapor atomic absorption photometry, ultraviolet-visible solution photometry, and polarography. Such techniques are employed, for example, for elements that may not be recovered in some preconcentration step, that have strong lines in a spectral region not usually recorded (alkali metals), that can be determined with superior detection limits by another technique (alkali metals, cadmium, mercury, zinc), or that have the favorable strong line coincident with that of a matrix element (e.g., cadmium when indium is selected as a collector ion or when indium oxide is the matrix).

In this article some of the approaches that we have taken to apply arc spectrography to high-purity chemicals are delineated and special reference is made to SPEX semi-quantitative standards and spectrographic-grade products in this work.

*Paper VIII in the Series, "The Practical Analysis of High-Purity Chemicals"

products has been routine for many years. Reports on applications to high-purity chemicals began to appear with the 1950s and the monograph of Alimarin (8) provides many working procedures. As a general purpose method we prefer DC-arc excitation under total burn and controlled-atmosphere (argon-oxygen) conditions with SA No. 1 plates. Where available, common element standards are employed that have a matrix identical or similar to the compound being characterized. For example, the SPEX 4-part "Al" standard, 3-part "Ca" standard, and 3-part "Zn" standard serve as references for the direct arcing of high-purity oxides and salts of aluminum, calcium, and zinc, respectively. To augment the 3-part standards, a fourth level (0.0001%) is secured by dilution with spectrographic grade calcium carbonate or zinc oxide, respectively.

Direct arcing does not provide adequate detection limits for many elements where a high-purity chemical is being assessed, and preconcentration becomes necessary. Solutions can be evaporated, organic materials can be ashed, and certain metals can be concentrated by precipitation or extraction.

Inorganic acids are major products in industry and the laboratory and high-purity forms are finding increasing applications. For such acids (ULTREX and Low-Sodium MOS grades), we have reported (6) a procedure for the evaporation of up to 100-gram samples of the concentrated acid under contamination-free conditions, followed by spectrographic evaluation of the residue. In this evaporation, spectrographic-grade graphite powder is added as a collector, a few drops of ULTREX sulfuric acid to convert to less volatile sulfates (omitted with hydrofluoric acid), indium as an internal standard (as SPEX indium oxide dissolved in ULTREX hydrochloric acid), and mannitol to retain boron (for hydrochloric and hydrofluoric acids). The spectrum is examined for 33 elements against the recently introduced SPEX-7step "G-7" common element standard in graphite containing indium as an internal standard. Where an impurity is present only at the ppm level, visual comparison suffices for quality control purposes. Densitometry is to be recommended for impurities present at higher levels and in application studies, such as etching or leaching, where greater precision is required. This 7-part standard is stepped at one-third decade concentration levels, resulting in improved precision over the decade-stepped 3-part or 4-part standards. Especially in the visual comparison of line intensities, interpolation is much more reliable with the closer steps of the 7-part standard. Selection of a single element, indium, as the internal standard, and a single line of that element for the calculation of line-pair ratios affords simplicity to the procedure. At the trace level involved, the associated compromise is not considered serious. This preconcentration by evaporation markedly improves the detection limits (10,000-fold for a 100-gram sample!), but at the expense of time. The time required depends on the volatility of the acid and the number of elements present significantly above their detection limit, thereby requiring densitometry. The total elapsed time for the concurrent analysis of *three* acid samples is 4 to 10 hours, but the actual working time is 3 to 5

hours. This approach has been extended in our laboratory to the study of trace elements in water, plant effluents, and high-purity organic solvents. We have confirmed the completeness of recovery achieved with the procedure for a number of high-purity acids. (Table 1).

For organic materials, ashing is an effective means of concentrating trace metals. Fiftyfold concentration can be readily achieved in this way. Some of the conditions suitable for ashing have been summarized by Koch and Koch-Dedic (9) and by Gorsuch (10). In most cases we have preferred a procedure involving pre-ashing of a sample, 0.5-gram moistened with 0.5 ml of high purity sulfuric acid, in a pre-ashing chamber, addition of 10 mg of high-purity lithium carbonate, and final ashing in a muffle furnace at 450C overnight, and arcing of the total residue. The spectrographic plates obtained are compared with SPEX 3-part "Li" standard (with a fourth, lower part secured by dilution with lithium carbonate). We have published this procedure for the spectrographic evaluation of trace metals (and boron and silicon) in the ULTREX key chelating agent, EDTA (1).

Collection via precipitation is a most powerful tool in the application of emission spectrography to high-purity inorganic salts. Usually one or more chelating precipitants are coupled with a polyvalent metal ion as the collector. The 8-quinolinolthionalide-tannic acid system introduced by R. L. Mitchell and coworkers (11), and confirmed by other workers (12,13), is

Table I.
Recovery from Hydrochloric Acid (6)

Element	Sample*		Spiked Sample†, µg		% Recovered	
	Found, µg	Calculated	Found	Calculated		
Antimony	<0.5	<0.5	<0.5	3. 3-3.8	3.0	79-91
Aluminum	0.7	1	0.7	4.1	4.7	115
Boron	0.3	0.2	0.1	3.5	3.7	106
Calcium	1.0	2.0	2.0	5.0	5.6	112
Chromium	0.1	0.5	0.2	3.6	3.3	92
Cobalt	<0.1	<0.1	<0.1	3. 3-3.4	3.3	97-100
Copper	0.2	0.3	0.2	3.5	3.3	94
Iron	0.3	1.0	0.7	4.0	4.7	118
Lead	<0.1	<0.1	<0.1	3. 3-3.4	3.4	100-103
Magnesium	0.3	0.5	0.5	3.7	4.0	108
Manganese	0.07	0.1	0.1	3.39	3.3	97
Mercury	<1.0	<1.0	<1.0	3. 3-4.3	3.3	77-100
Nickel	<0.1	<0.1	<0.1	3. 3-3.4	3.3	97-100
Potassium	<1.0	<1.0	<1.0	3. 3-4.3	3.3	77-100
Silicon	2.0	3.0	2.0	5.6	5.6	100
Sodium	10.0	10.0	9.0	13.0	13.0	100
Tin	<0.1	<0.1	<0.1	3. 3-3.4	3.1	91-94
Zinc	<0.1	<0.1	<0.1	3. 3-3.4	3.3	97-100

*Hydrochloric Acid, Lot UA 156; values for three 100-gram samples from lot.

†10 mg. of the 0.33% level (G-1A) of the standard, that is, 3.3µg of each element, added to 100 gram of the acid and the single spiked sample analyzed.

probably the most common. Both aluminum and indium can serve as the collector ion. We prefer the latter since indium is seldom encountered in practical samples and aluminum is often of interest. This system, as modified by us, involves suitable dissolution of a 5-gram sample, addition of an indium salt solution as the collector and of 8-quinolinol solution. The pH is adjusted to 1.8, then ammonium acetate, tannic acid, and thionalide solutions are added, and the pH is brought to 5.9 with aqueous ammonia. The mixture is allowed to stand overnight and is then filtered. The precipitate is washed, pre-ashed, ashed, and the residue arced. The plates are read against the SPEX 3-part "In" standard (with a fourth, lower part secured by dilution with indium oxide). By this procedure the detection limits are improved about 500 times. When this technique is applied to high-purity chemicals, it is imperative that the precipitants be adequately free of metal impurities as confirmed by ashing and emission spectrography. 8-quinolinol is the principal component of the precipitant system, therefore a major contributor to the blank. Early in our studies on the practical characterization of high-purity compounds, we found it appropriate to purify 8-quinolinol specially for both precipitation and extraction. In view of the success achieved, an ULTREX grade of this compound was introduced for which the sum of about 30 divalent and trivalent metals is typically less than 0.5 ppm.

Extraction is also of interest as a preconcentration technique with emission spectrography; a variety of extractants, alone or in combination or sequence, have received attention (8). A single example will suffice: Kuz'min and co-workers have reported (14) a scheme for the determination of various trace elements in phosphorus pentoxide, involving hydration to phosphoric acid, alkalization with ammonia, extraction with 8-quinolinol into a mixed solvent, evaporation of the extract with graphite powder added, mineralization with nitric acid, and finally spectrographic assessment. Kuz'min conducted extensive recovery studies and established that even at high phosphate concentrations 11 elements are extracted with recoveries of at least 80% and zinc with a 60% recovery. With a 5-gram sample, the detection limits are improved about 500 times. We have confirmed the completeness of the recovery for five of the elements, and have applied the scheme to phosphorus pentoxide of extreme purity, which has found application in studies of the Apollo lunar rocks (2) and which is receiving increasing attention as a superior dopant for semiconductors. We have also relied on this approach in the characterization of Low-Sodium MOS-grade phosphoric acid, and also of ULTREX phosphorus trichloride and oxychloride, recommended as semiconductor dopants, following oxidation or hydrolysis of the sample, or both. The 7-part "G-7" standard (graphite base) is appropriate in these analyses. Low blanks are assured by specifying ULTREX 8-quinolinol, ULTREX acids, high-purity ammonia and ultrapure water.

As a concentration technique for trace metals, removal of the matrix, as distinguished from separation of the trace impurities, is often not feasible. Where the former approach is suitable, it is important to undertake studies to assure that

losses of the elements to be determined do not occur. The approach is of special merit where the matrix can be volatilized because attention is required only for those few elements that might be evolved under the given conditions. Boron compounds are amenable to matrix removal (15) and we have applied it to high-purity boron oxide and boron tribromide with which advanced electronic devices are doped. For boron oxide, boron is first volatilized from the sample solution as methyl borate; graphite powder and also indium (internal standard) are then added; the mixture is finally taken to dryness, and arced. The plates are read against the 7-part "G-7" standard.

It is sometimes feasible to remove a matrix by crystallization or precipitation (8). Caldararu (16) applied this approach in the spectrographic assessment of aluminum metal and we have extended the method to anhydrous aluminum chloride. Metal-free hydrogen chloride gas is passed through the solution of the salt in water. Aluminum chloride is thereby crystallized and the supernatant liquid is decanted. The salt is redissolved in water and the process is repeated twice. The combined supernatant liquids are evaporated with the addition of ULTREX nitric acid. The nitrate residue is converted to oxides by ignition and arced. The plate is assessed against the 4-part SPEX "Al" standard. Caldararu reported (16) data for 10 elements of interest and we have made recovery studies for certain key elements. With a 5-gram sample, the detection limits secured are improved about 500 times.

One of the important measures to be taken in the adoption of a preconcentration step with spectrography is to assure that the recoveries of the elements of interest are sufficiently complete at the concentration levels to be encountered with the actual samples. In recovery studies the addition of elements as stock solutions of one or several elements can be tedious. With a dry mix, it is imperative that a high degree of homogeneity be achieved. Either way, only high-purity forms must be employed and the addition mixture or combined solutions must be analyzed accurately to correct for contributions of impurity contents of the additives. Although commercial common-element mixtures and derived spectrographic standards have undoubtedly previously been used in recovery studies to facilitate the addition of a large number of elements at the trace level, we failed in a cursory literature study to turn up relevant papers. Obviously this technique, where feasible, obviates the major expenditure of time and effort that is otherwise required in making additions. It is necessary to assure a high degree of mixing and equilibration of the added common-element mix with the sample via digestions, ashing, fusion, evaporation, etc. This approach (6) has proved effective in a recovery study for the evaporation and spectrographic analysis of inorganic acids (see above). Few earlier workers have reported detailed recovery studies; most have relied on the accumulated knowledge concerning the loss of trace elements upon heating of acidic solutions. Facing the problem squarely, we studied over 15 elements. In so doing we measured the errors that could result with spikes obtained by adding measured volumes of one or more solutions. What was

done was to add to a known weight of the sample of the acid, a weighed amount of one part of the 7-part "G-7" common element standard. In this way the elements of interest were added in a precisely known amount. Since this standard is referenced in the reading of the plates of both spiked and unspiked samples, the results reflect closely actual losses of the elements. The situation was further improved by arcing on a single plate both the residue from evaporation of the spiked sample and the identical amount of the same part of the "G-7" standard with which the sample had been spiked, thereby reducing minor variations in photometric development and arcing at different times. Table I records the results of such a spiking experienced for hydrochloric acid. The procedure with a number of high-purity acids was verified in this way to give recoveries exceeding 75% for some 18 elements (6).

The applications delineated in this article for SPEX common element standards in the J. T. Baker programs for high-purity chemicals speaks well for the reliability of these standards. However, any spectrographic laboratory seeking quantitative data should independently attempt to confirm the accuracy of the commercial standards employed. We have approached this problem informally in various ways, including comparison of one batch of a standard with the next, comparison of one standard with another in a different matrix, comparison of standards from different firms, and also comparison of the commercial standards with laboratory-prepared standards for a few key elements.

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HIGH PURITY MATERIALS

Material	Cat. No.	Purity	Form	Price
ALUMINUM (Al)	1112-6	6-9s	Sheet, 1x100mm	5.20/2g 17.70/10g
	1112	5-9s+	Rod, 4x75mm (approx. 7 rods)	8.10/10g 27.50/50g
	1112P	5-9s	Powder, 90-150 μ	4.80/10g 16.30/50g
ALUMINUM OXIDE	1212-6	6-9s	Powder, Al ₂ O ₃	5.00/2g 17.00/10g
	1212-4	4-9s	Powder, Al ₂ O ₃ ·xH ₂ O 0.01-1 micron	7.00/50g 23.80/250g
ANTIMONY (Sb)	1113-6	6-9s	Powder	6.00/g 20.40/5g
	1113	5-9s+	Pieces, Irregular	6.50/10g 22.25/50g

Material	Cat. No.	Purity	Form	Price
ANTIMONY OXIDE	1213-6	5-9s+	Powder, Sb ₂ O ₃ 45 microns	8.00/10g 27.20/50g
	1213	5-9s	Powder, Sb ₂ O ₃	4.40/2g 15.10/10g
ARSENIC (As)	1114-6	6-9s	Lumps, Beta-form, air stable	8.00/2g 27.20/10g
	1114	5-9s+	Pieces, Irregular	7.90/5g 26.75/25g
ARSENIC OXIDE	1214	5-9s+	Powder, As ₂ O ₃	5.00/5g 17.50/25g
BARIUM CARBONATE (Ba)	1215	5-9s	Powder, BaCO ₃	8.00/5g 27.25/25g
BARIUM CHLORIDE	1315	5-9s		5.55/5g 18.90/25g

Material	Cat. No.	Purity	Form	Price
BERYLLIUM (Be)	1116-4	4-9s	Flake	8.90/g 30.25/5g
	1116	3-9s	Chip	4.50/10g 15.30/50g
BERYLLIUM OXIDE	1216	5-9s	Powder, BeOxH ₂ O	7.80/20g 26.60/100g
BISMUTH (Bi)	1117	6-9s	Shot, Irregular	9.00/50g 30.60/250g
	1117P	6-9s	Powder, 45 microns	7.20/20g 24.50/100g
BISMUTH OXIDE	1217	5-9s	Powder, Bi ₂ O ₃	5.50/10g 18.70/50g
BORON (B)	1118-5	5-9s+	Granule, 150 microns	146.20/g
	1118	3-9s	Powder	8.75/5g 29.80/25g
BORIC ACID	1218	4-9s	Powder, H ₃ BO ₃	6.00/50g 20.50/250g
BORON OXIDE	1218-0	5-9s	Powder, B ₂ O ₃	9.85/g 33.50/5g
CADMIUM (Cd)	1119-6	6-9s	Powder, 45 microns	10.00/10g 34.00/50g
	1119	5-9s+	Splatters	6.50/50g 22.10/250g
CADMIUM OXIDE	1219	5-9s+	Powder, CdO	11.60/5g 43.20/25g
CALCIUM CARBONATE (Ca)	1220	5-9s	Powder, CaCO ₃ xH ₂ O	6.40/5g 21.75/25g
CALCIUM FLUORIDE	1320	3-9s+		9.70/10g 33.00/50g
CERIUM (Ce)	1165I	3-9s+	Ingot	5.80/10g 19.75/50g
	1165	3-9s	Powder	9.60/g 32.65/5g
CERIUM OXIDE	1266-5	5-9s	Powder, CeO ₂	9.40/g 31.95/5g
	1265	3-9s+	Powder, CeO ₂	4.40/10g 15.00/50g
CESIUM CHORIDE (Cs)	1221	3-9s	Powder	5.50/10g 18.70/50g
CESIUM CARBONATE	1331	3-9s	Powder	5.50/10g 18.70/50g
CESIUM CHROMATE	1441	3-9s	Powder	5.50/10g 18.70/50g
CHROMIUM (Cr)	1122	5-9s	Shot, Irregular	7.50/20g 32.50/100g
	1122F	5-9s	Flake	5.50/10g 19.70/50g
	1122P	4-9s	Powder, 150 microns	9.00/50g 30.60/250g
CHROMIUM OXIDE	1222	5-9s	Powder	9.30/5g 31.60/25g
COBALT (Co)	1123	5-9s	Powder	7.60/5g 25.80/25g
	1123R		Rod, 3mmx15cm (27gm)	57.00/rod 190.00/5 rods
COBALT OXIDE	1223	5-9s	Powder	5.90/5g 20.00/25g
COLUMBIUM			(SEE NIOBIUM)	

Material	Cat. No.	Purity	Form	Price
COPPER (Cu)	1125-6	6-9s	Bars 3/4" (approx. 70gm)	14.45/bar
	1125	5-9s	Rods, 5x150mm (approx. 27g/rod)	9.42/rod 32.00/5 rods
	1125P	5-9s	Powder	8.50/10g 28.90/50g
COPPER OXIDE	1225	5-9s+	Powder	11.25/5g 38.25/25g
COPPER HYDROXY FLUORIDE	1325	4-9s	Powder, Cu(OH)F, boron-free for determination by carrier dist.	7.20/10g 24.40/50g
DYSPROSIUM (Dy)	1166	3-9s	Ingot	4.90/2g 16.70/10g
DYSPROSIUM OXIDE	1266	3-9s+	Powder, Dy ₂ O ₃	6.80/5g 22.75/25g
ERBIUM (Er)	1167	3-9s	Ingot	6.10/g 20.75/5g
	1167P	3-9s	Powder	5.54/2g 18.90/10g
ERBIUM OXIDE	1267	3-9s+	Powder, Er ₂ O ₃	6.20/5g 20.70/25g
EUROPIUM (Eu)	1168	3-9s	Ingot	20.70/g 70.40/5g
EUROPIUM OXIDE	1268-5	5-9s	Powder, Eu ₂ O ₃	106.00/g
	1268	3-9s	Powder, Eu ₂ O ₃	7.75/g 26.50/5g
GADOLINIUM (Gd)	1169	4-9s	Powder, 250-450 microns	11.50/g 39.10/5g
GADOLINIUM OXIDE	1269-5	5-9s	Powder, Gd ₂ O ₃	7.70/g 26.20/5g
	1269	3-9s+	Powder, Gd ₂ O ₃	7.30/10g 25.00/50g
GALLIUM (Ga)	1126	6-9s	Splatter	7.10/g 24.10/5g
GALLIUM OXIDE	1226	6-9s	Powder, Ga ₂ O ₃ , 1-2 microns	6.13/g 20.80/5g
GERMANIUM (Ge)	1127	5-9s+	Pieces, Irregular	4.75/5g 16.20/25g
	1127P	5-9s	Powder	8.70/10g 29.60/50g
GERMANIUM OXIDE	1227	5-9s	Powder, GeO ₂	7.20/20g 24.50/100g
GOLD (Au)	1128P	5-9s	Powder	7.70/g 29.20/5g
	1128	5-9s+	Splatter	6.20/g 21.10/5g
HAFNIUM (Hf)	1129W	3-9s+	Wire, Excluding Zr, 2-3% (0.125mm dia.)	6.80/2g 23.12/10g
	1129	3-9s	Sponge, Excluding Zr, 1-3%	8.15/5g 27.75/25g
HAFNIUM OXIDE	1229	3-9s	Powder, HfO ₂ (Low Zr)	8.00/g 27.20/5g
HOLMIUM (Ho)	1170	3-9s	Ingot	7.50/g 25.50/5g
	1170P	3-9s	Powder	8.05/g 27.40/5g
HOLMIUM OXIDE	1270	3-9s+	Powder, Ho ₂ O ₃	9.40/5g 32.00/25g

Material	Cat. No.	Purity	Form	Price
INDIUM (In)	1130	6-9s	Splatter	7.20/5g 24.50/25g
	1130P	5-9s	Powder	6.30/5g 21.50/25g
	1230	5-9s+	Powder, In ₂ O ₃	6.20/10g 21.00/50g
IODINE (I)	1181	6-9s	Crystals	5.40/20g 18.40/100g
IRIDIUM (Ir)	1131-5	5-9s	Sponge	44.50/g 151.30/5g
	1131	3-9s+	Powder	20.25/g 84.40/5g
IRON (Fe)	1132-5	5-9s	Sponge	5.50/10g 18.70/50g
	1132P	5-9s	Powder, 150 micron	6.50/10g 22.00/50g
	1132	5-9s	Rod, 5x150mm (approx. 24g/rod)	15.00/rod 62.50/5 rods
IRON OXIDE	1232	5-9s	Powder	7.50/10g 25.50/50g
LANTHANUM (La)	1171	3-9s	Powder, Z150 microns	9.40/g 32.00/5g
LANTHANUM OXIDE	1271	5-9s+	Powder, La ₂ O ₃	4.20/20g 14.25/100g
LEAD (Pb)	1133	6-9s	Shot, Irregular	8.00/50g 27.20/250g
	1133P	5-9s	Powder	8.00/20g 27.20/100g
LEAD OXIDE	1233	5-9s+	Powder, PbO	5.60/10g 19.00/50g
LEAD SULFIDE	1333	4-9s+	Powder	7.75/10g 26.40/50g
LITHIUM CARBONATE (Li)	1234	5-9s	Powder, Li ₂ CO ₃ ,	7.30/10g
			Spectrographic Buffer	24.80/50g 44.00/100g 85.00/200g 150.00/lb.
LITHIUM FLUORIDE	1134	5-9s+	Powder, LiF	7.75/5g 26.40/25g
LUTETIUM (Lu)	1172	3-9s	Ingot	140.00/g
	1172P	3-9s	Powder	74.00/g
LUTETIUM OXIDE	1272	3-9s+	Powder, Lu ₂ O ₃	25.25/g 86.00/5g
MAGNESIUM (Mg)	1135	4-9s+	Rods, 3x75mm (approx. 1.1g/rod)	5.40/20g 18.40/100g
MAGNESIUM OXIDE	1235	5-9s+	Powder, MgO	5.70/5g 19.30/25g
MANGANESE (Mn)	1136-4	4-9s+	Flake	8.00/50g 27.20/250g
	1136	3-9s+	Powder	4.00/100g 13.60/500g
MANGANESE OXIDE	1236	5-9s+	Powder	10.00/5g 34.00/25g
MANGANESE SULFIDE	1336	4-9s+	Powder	7.90/2g 26.80/10g
MERCURY (Hg)	1137-9	9-9s	Liquid	8.20/20g 27.40/100g
	1137	7-9s	Liquid	6.50/50g 22.00/250g

Material	Cat. No.	Purity	Form	Price
MERCURY OXIDE	1237	5-9s+	Powder, HgO	5.90/2g 20.10/10g
MOLYBDENUM (Mo)	1138	4-9s+	Powder	8.00/100g 27.25/500g
		5-9s	Powder	9.25/5g 31.50/25g
NEODYMIUM (Ne)	1173	3-9s	Powder, 250-450 microns	19.90/g 57.70/5g
NEODYMIUM OXIDE	1273-5	5-9s	Powder, Nd ₂ O ₃	10.60/g 36.00/5g
	1273	3-9s	Powder, Nd ₂ O ₃	4.90/10g 16.70/50g
		5-9s	Powder	7.30/10g 24.80/50g
NICKEL (Ni)	1139-5	5-9s	Sponge	7.30/10g 24.80/50g
	1139	4-9s	Powder	4.00/100g 13.60/500g
		5-9s	Powder	6.00/10g 20.40/50g
		5-9s	Powder, NiO	7.40/20g 25.20/100g
NICKEL OXIDE	1239	5-9s	Powder, NiO	6.40/20g 21.75/100g
NIOBIUM (Nb)	1124	3-9s+	Powder	6.40/20g 21.75/100g
NIOBIUM OXIDE	1224	4-9s+	Powder, Nb ₂ O ₅	8.50/50g 29.00/250g
PALLADIUM (Pd)	1141	5-9s	Powder	15.60/g 53.20/5g
	1141-3	3-9s	Powder	10.00/2g 34.00/10g
PALLADIUM OXIDE	1241		Catalyst Grade Powder	10.60/g 36.00/5g
AMMONIUM PHOSPHATE (P)	1242	5-9s	Powder, NH ₄ H ₂ PO ₄	9.00/100g 30.60/500g
PLATINUM (Pt)	1143	5-9s	Powder	29.00/g 98.50/5g
POTASSIUM CARBONATE (K)	1244	5-9s	Granules, K ₂ CO ₃ x H ₂ O	9.00/20g 30.50/100g
POTASSIUM CHLORIDE	1344	5-9s+	Powder, KCl	6.30/5g 21.40/25g
PRASEODYMIUM (Pr)	1174	3-9s	Wire, 1mm dia.	128.70/g
	1174P	3-9s	Powder	5.48/2g 18.65/10g
PRASEODYMIUM OXIDE	1274-5	5-9s	Powder, Pr ₆ O ₁₁	10.60/g 36.00/5g
	1274	3-9s+	Powder, Pr ₆ O ₁₁	7.80/20g 26.50/100g
RHENIUM (Re)	1145	4-9s+	Powder	4.79/g 16.30/5g
AMMONIUM PERRHENATE	1245	4-9s+	Granule	6.50/g 22.10/5g
RHODIUM (Rh)	1146-5	5-9s	Powder	80.00/g 240.00/5g
	1146	4-9s	Powder	21.12/g 72.00/5g 47.50/g
RHODIUM OXIDE	1246	4-9s	Powder	47.50/g 5.50/10g
RUBIDIUM CHLORIDE (Rb)	1247	3-9s	Powder	18.70/50g
RUTHENIUM (Ru)	1148	3-9s+	Powder	7.58/g 25.80/5g

Material	Cat. No	Purity	Form	Price		
SAMARIUM (Sm)	1175	3-9s	Powder,	16.35/g		
			75-150 microns	55.60/5g		
SAMARIUM OXIDE	1275	3-9s+	Powder, Sm ₂ O ₃	4.50/10g 15.30/50g		
SCANDIUM (Sc)	1176	3-9s+	Ingot	197.80/g		
			1176P	3-9s	Powder	99.70/g
SCANDIUM OXIDE	1276-5	5-9s	Powder, Sc ₂ O ₃	35.90/g 122.00/5g		
			1276	3-9s	Powder, Sc ₂ O ₃	12.41/g 42.10/5g
					1149-6	6-9s
SELENIUM (Se)	1149	5-9s+	Pellets	9.50/50g 32.30/250g		
			SELENIUM OXIDE	1249	5-9s+	Powder, SeO ₂
SILICON (Si)	1150	6-9s+	Pieces, Irregular	5.20/10g 17.70/50g		
			1150P	6-9s	Powder	10.10/5g 33.15/25g
					SILICON OXIDE	1250
SILVER (Ag)	1151	6-9s	Shot	5.65/5g 19.20/25g		
			1151P	5-9s	Powder	7.30/2g 24.80/10g
					SILVER CHLORIDE	1251
SODIUM CARBONATE (Na)	1252	5-9s	Powder, Na ₂ CO ₃	7.50/2g 25.50/10g		
SODIUM CHLORIDE	1352	5-9s	Powder, NaCl	6.50/10g 22.10/50g		
STRONTIUM CARBONATE (Sr)	1253	5-9s	Powder, SrCO ₃	9.50/5g 32.40/25g		
STRONTIUM CHLORIDE	1353	5-9s	Powder, SrCl ₂	6.86/2g 23.30/10g		
STRONTIUM FLUORIDE	1153	5-9s	Powder, SrF ₂	7.10/2g 24.10/10g		
TANTALUM (Ta)	1154	4-9s+	Powder	5.80/20g 19.70/100g		
TANTALUM OXIDE	1254	4-9s	Powder, Ta ₂ O ₅	8.80/10g 30.00/50g		
TELLURIUM (Te)	1155-6	6-9s	Bars	9.10/10g 30.95/50g		
			1155	5-9s+	Pieces, Irregular	4.60/20g 15.60/100g
					TELLURIUM OXIDE	1255
TERBIUM (Tb)	1177	3-9s	Ingot	14.60/g 39.70/5g		
			1177P	3-9s	Powder	15.25/g 52.00/5g
					TERBIUM OXIDE	1277-5
1277	3-9s	Powder, Tb ₄ O ₇	7.25/g 24.70/5g			
		THALLIUM (Tl)	1156	5-9s+	Rods, 10x33mm (approx. 25g/rod)	12.50/25g 43.75/125g

Material	Cat. No.	Purity	Form	Price		
THALLIUM OXIDE	1256	5-9s+	Powder, Tl ₂ O ₃	5.50/10g 18.70/50g		
			THORIUM (Th)	1157	3-9s	Powder
THORIUM OXIDE	1257	4-9s	Powder, ThO ₂	8.40/10g 28.50/50g		
THULIUM (Tm)	1178	3-9s	Powder, 250-350 microns	58.70/g		
THULIUM OXIDE	1278	3-9s+	Powder, Tm ₂ O ₃	16.20/g 55.00/5g		
TIN (Sn)	1158	6-9s	Pellets	6.50/10g 22.00/50g		
			1158P	6-9s	Powder	8.00/5g 27.20/25g
TIN OXIDE	1258	6-9s+	Powder	10.00/5g 34.00/25g		
TITANIUM (Ti)	1159	3-9s+	Sponge	5.20/20g 17.70/100g		
			1159P	3-9s	Powder	5.00/10g 17.00/50g
TITANIUM OXIDE	1259	4-9s	Powder, TiO ₂	4.40/20g 15.00/100g		
TUNGSTEN (W)	1160	4-9s	Powder	3.00/100g 10.20/500g		
			1160-5	5-9s	Powder, Low-Molybdenum	7.20/10g 24.50/50g
TUNGSTEN OXIDE	1260	4-9s+	Powder	4.00/100g 13.60/500g		
URANIUM (U)	1161	4-9s+	Chunks, Irregular	7.20/10g 24.50/50g		
URANIUM OXIDE	1261	3-9s+	Powder	8.60/5g 29.25/25g		
VANADIUM (V)	1162	3-9s	Pellets	7.80/20g 26.50/100g		
VANADIUM OXIDE	1262-5	3-9s+	Powder	7.40/10g 25.20/50g		
			1262-5	4-9s+	Powder	3.25/g 11.05/5g
YTTRIUM (Y)	1179	3-9s	Ingot	4.50/2g 15.30/10g		
YTTRIUM OXIDE	1279-6	6-9s	Powder, Y ₂ O ₃	5.00/g 17.00/5g		
			1279	5-9s	Powder, Y ₂ O ₃ , 1-2 microns	4.50/2g 15.30/10g
YTTERBIUM (Yb)	1180	3-9s	Powder, 350 microns	10.60/g 36.00/5g		
YTTERBIUM OXIDE	1280	3-9s	Powder, Yb ₂ O ₃	4.40/2g 15.00/10g		
ZINC (Zn)	1163	5-9s+	Splatter	4.40/20g 15.00/100g		
			1163P	6-9s	Powder	6.00/20g 20.40/100g
					ZINC OXIDE	1263
ZIRCONIUM (Zr)	1164-4	4-9s	Rod	52.50/rod 9.00/20g		
			1164	3-9s+	Sponge	30.50/100g 11.00/20g
ZIRCONIUM OXIDE	1264	3-9s+	Powder, ZrO ₂ , low Hf	37.50/100g		

NOTE: We have rolled back electrode prices in response to the Presidential Ruling. When the freeze is lifted, should the manufacturers' increases be reinstated, the price list in our June, 1971 SPEX SPEAKER will become current.

SPECTROSCOPIC PREFORMED ELECTRODES

HIGH PURITY GRAPHITE						
Dia"	Description	Spex	AGKSP	Ultra	ASTM	Price per 100
1/4	necked crater, 5/32" dp.	4000	L-3912	100-L	S-12	22.00
3/16	necked crater, 3/16", dp.	4001	L-3903	101-L	S-13	22.00
1/4	crater, 3/16" dp.	4002	L-3900	103	S-8	20.00
1/4	angular platform, center post	4003	L-3948	104-L	P-2	28.00
3/16	necked crater, 3/32" dp.	4004	L-3906	105-S		19.00
3/16	necked crater, 3/16" dp.	4005	L-3909	105-D	S-14	19.00
1/4	flat necked upper	4007	L-3960	101-U	C-8	20.00
1/4	undercut, center post upper	4008	L-3963	104-U	C-7	22.00
3/16	double-ended, 1/16" r., 2" l.	4009	L-3955	5712		24.00
1/4	pointed upper, 120°	4010	L-3966	108	C-2	20.00
1/2	solution disc, 1/8" thick	4011	L-4075	106	D-1	14.00
1/2	platrode, extruded	4012	L-4078	1907	D-3	18.00
1/4	porous cup, .025" floor, 1-1/2" l.	4014	L-3927	203		24.00
1/4	porous cup, chamfered floor, 7/8" l.	4015	L-3933	204	PC-1	23.00
1/4	crater, 1/16" dp.	4016	L-3982	4196	S-5	21.00
1/8	pedestal, 1-1/2" l.	4017	L-3919	1964	S-1	16.00
1/4	anode cap, 9/32" dp., thin wall	4018	L-3918	1998	S-3	16.00
1/8	pointed upper	4019	L-4036	1992	C-1	16.00
1/8	crater, 1/4" dp.	4020	L-3979	5440		16.00
1/4	necked crater, 1/16" dp.	4021	L-4012	1988	S-4	22.00
1/4	boiler cap	4022	L-3915	300		26.00
3/16	boiler cap	4023	L-3916	301		26.00
1/4	flat rod	4024	L-3921	107	C-3	18.00
1/4	mandrel for rotating electrode, 2" l.	4026	L-3970	2022		22.00
1/2	solution disc., .200" thick	4027	L-4072	861	D-2	16.00
1/2	platrode, molded	4028	L-4081	1909		18.00
3/16	necked crater, 3/16" dp.	4029	L-4000	5915		19.00
3/16	necked crater, 3/32" dp.	4030	L-4006	7250		19.00
1/4	mandrel for Combination Analyzer	4032	SP-1003	2025		22.00
1/8	necked crater, 1/8" dp.	4033	L-3905	781		19.00
1/8	crater, .059" dp.	4034	L-3975	2509		16.00
1/8	crater, 3/16" dp.	4035	L-3977	5000		16.00
1/4	porous cup, .025" floor	4037	L-4928	5680		24.00
1/4	anode cap, 9/32" dp.	4038	L-4024	1990	S-2	16.00
3/16	rounded upper, 1/16" r.	4039	L-3951	105-U		17.00
3/16	rounded upper, 1/16" r., 2" l.	4040	L-3954	5710		18.00
1/4	rounded upper, 1/16", r.	4041	L-3957	100-U	C-5	20.00
1/4	necked crater, 3/16" dp.	4042	L-4018	1989		22.00
1/8	pedestal, 1" l.	4043	L-4042	1993		12.00
1/4	anode cap, 1/32" dp.	4044	L-4030	1991		16.00
1/4	center post crater, 1/4" dp.	4046	L-4054	1995		21.00
1/4	curved platform, center post	4049	L-3945	1335		24.00
1/4	anode cap, 1/16" dp.	4070	L-4031	5630		16.00
1/8	flat rod	4071	L-3922	1509	C-6	14.00
3/16	flat rod	4072	L-3923	2615	C-9	14.50
1/8	rounded upper, 1/32" r.	4073	L-4037	5001		17.00
1/4	vacuum cup, 3/8" post, 2" l.	4074		6010		35.00
3/4	O.D. Teflon Cup for 4074 or 4075	4074A		6010A		12.00/10
1/4	vacuum cup, 5/8" post, 2" l.	4075		6011		35.00
1/4	tapered mandrel, 1-1/8" l.	4078	L-3969	2021	D-4	18.00
1/4	.054" micro-cup, .062" dp.	4079	L-4257	115		22.00
1/4	.096" micro-cup, .075" dp.	4080	L-4259	117		22.00



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