

The**SPEX**

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Speaker**STANDARDS FOR SPARK SOURCE MASS SPECTROMETRY**E.W. Hobart and J.R. Wieland
Ledoux and Co., Teaneck, N.J. 07666

SPEx Industries, in collaboration with Ledoux and Co., has prepared a series of standards for semiquantitative spark source mass spectrometry. These Spex Spikes comprise minor isotopes of the elements lead, europium, cadmium, molybdenum, nickel and calcium in a matrix of high-purity graphite. By mixing equal parts of the appropriate standard and a finely ground portion of an unknown sample, internal standards, well spaced through the range of masses of elements, are incorporated into the sample mixture. Simple visual examination of the mass spectrum will usually suffice to obtain semiquantitative estimates of most of the elements present in the sample including those in the standard. Since most analytical results are required on a concentration by weight basis, the standards have been prepared to contain 1, 10, 100 and 1000 ppm by weight (ppmw) of the added isotopes.

Spark source mass spectrometry is a powerful technique for trace as well as micro analysis (1,2). Its primary advantages combine high detectivity and applicability to all elements from atomic number 3 (lithium) to 92 (uranium). Nearly every element present in a sample from a few parts per billion to several percent can be estimated in a single analysis. The amount of sample actually consumed in such an analysis is very small, only a few milligrams.

The chief difficulties encountered in spark source mass spectrometry are related to 1) adequate representation of a bulk sample by the very small amount actually analyzed, and 2) standardization.

The usual and simplest approach to improving sample homogeneity is to grind the material to as fine a powder as possible and then, after adding a binder such as graphite or silver, to press solid electrodes for sparking in the instrument (3). A rather elaborate and effective approach, reported by Nicholls, *et al.*, (4), involved repeated grinding of samples and fusions with an appropriate flux to assure homogeneity.

Accuracy of spark source analyses can cover a wide range. Poorest results are based on the assumption that equal atomic concentrations of isotopes in a sample will produce equal blackening on the photoplate (after correction for plate sensitivity factors). Most accurate analyses are based on the rigorous isotope dilution technique in which a known quantity of an isotope of an element is carefully spiked into the sample to serve as an internal standard for other isotopes of that element (5). As for the general method of isotope dilution — that amenable to the determination of all elements including those spiked — Webster (6) has described this in great detail.

Owens and Giardino (7), have reported a detailed study of factors which lead to erroneous results in spark source mass spectrometry. Among these are: emulsion and development uniformity, microsegregation, electrode positioning, fractional volatilization and unequal energy distributions. Proper darkroom techniques, densitometric measurements, and sample preparation are obviously necessary to minimize certain of these errors. The question of standardization, however, remains a difficult problem. As Owens and Giardino point out, electrode positioning can affect the transmission factor of ions through the magnet section of the spectrograph and the extent of this effect will be related to the length of the path through the section. Thus, variations in electrode positioning may play havoc with the apparent sensitivities of heavy ions while having relatively little effects on those of the lighter ones. This being so, attempts at standardization by referring all line intensities to that of one line, (whether due to a minor isotope of a matrix element, an intentionally added internal standard or an impurity of known concentration) become subject to serious error since it is difficult to maintain the spark at one point in the source from sample to sample. In addition, line widths depend on both the path through the magnet and the excellence of focus along the length of the plate. For these reasons, it is desirable to interleave reference isotopes at a number of positions in the mass spectrum.

One of the virtues of spark source mass spectrometry is its applicability to essentially all of the elements. It would make little sense to incorporate five or six internal standards if, in so doing, the capability of determining those elements were to be sacrificed. However, if minor isotopes of elements were added for standardization, the internal standard elements would be determinable by isotope dilution techniques with even more certainty than in the absence of the added minor isotope. It then becomes highly advantageous to have a simple means of spiking unknown samples with known concentrations of several different minor isotopes. Spex Spikes provide a simple and practical way in which to accomplish just this objective.

PREPARATION OF STANDARDS

Many mass spectrometric analyses are performed by mixing well ground samples with graphite, pressing the mixtures into electrodes and sparking between them to generate mass spectra. A simple means of incorporating several minor isotopes into the sample-graphite mixture would be to include known concentrations of the isotopes in the graphite. Spex Spikes, then, were conceived as a series of graded graphite standards suitable for mixing with unknown powder samples and pressing into electrodes for spark source mass spectrometric examination.

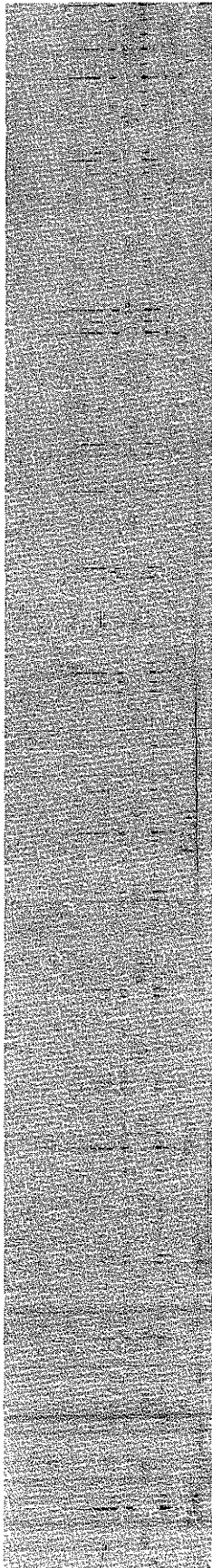


Fig. 1. Portion of a mass spectrogram of several exposures of Spex Spikes MG1, 1000 ppmw standard. The graded densities were obtained by varying the integrated charge. Note the choice of isotopes furnishes reference lines throughout the mass range.

Selection of isotopes to be included required consideration of a number of factors: 1) The mass range should be adequately covered; 2) The isotopes selected should be relatively minor isotopes of the elements; 3) The isotopes selected should not interfere with carbon multiples; 4) The mass number of the isotope should be an odd number; 5) The isotopes selected should be non-radioactive.

Inevitably, certain compromises had to be made in the final selection of isotopes. Those selected were: Pb^{206} , Eu^{153} , Cd^{113} , Mo^{95} , Ni^{62} and Ca^{42} .

The mass range is, indeed, well covered except for the light elements. Of the light elements, Be, F, Na, Al and P are monoisotopic, ruling them out of consideration. Li^6 is interfered by C^{+2} , the magnesium isotopes are all difficult to distinguish from $2C^+$ ions, silicon is such a common matrix element as to be of doubtful utility and boron is of such uncertain isotopic distribution as to limit the usefulness of one of its isotopes. With the exception of Eu^{153} , the isotopes selected are all relatively minor isotopes of the elements. Eu^{153} represents 52.2% of europium but europium itself is a relatively minor rare earth. Pb^{206} and Ni^{62} were selected in preference to Pb^{204} and Ni^{61} because of multiple carbon ions which may be difficult to resolve at masses 61 and 204. The spikes represent a good variety of metallic elements: lead, a heavy metal; europium, a rare earth; cadmium, a metal of intermediate mass; molybdenum, of the second transition series; nickel, a transition metal; and calcium, an alkaline earth.

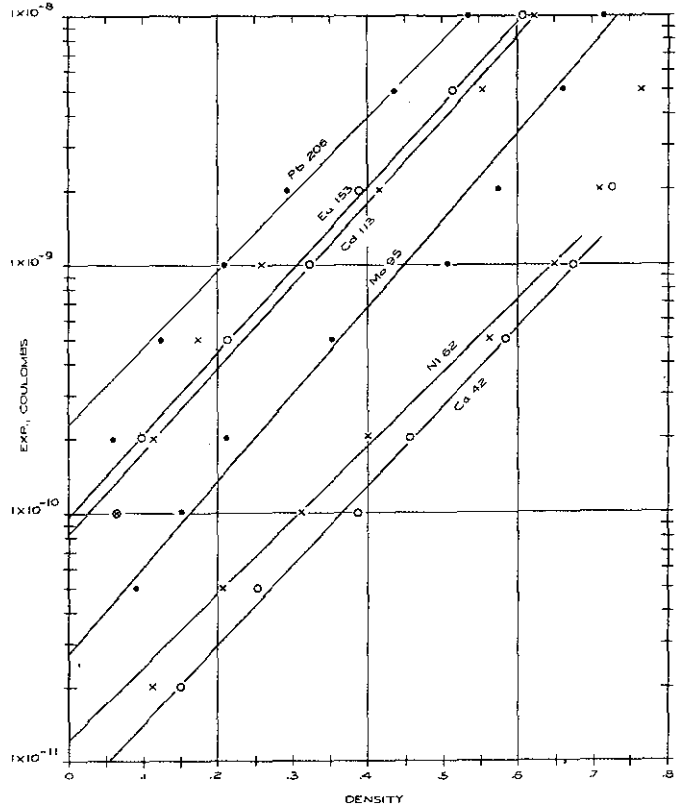


Fig. 2. Plot of Spex Spikes MG1, 1000 ppmw Standard. Density of the photographed lines on Ilford Q2 emulsion is plotted vs. integrated ion beam exposure. At least 6 separate exposures are recorded for each isotope; that they fall so well on a straight line is indicative of the homogeneity of the standard. Note that there is a factor of around 33 between the lowest (Ca) and highest (Pb) atomic number curve. In terms of accuracy, this would imply a maximum error of 3 times in estimating concentration if a low atomic mass were measured against a high atomic element. By spreading the Spikes over the complete atomic weight range, the error of measurement of any single element can be reduced to less than a factor of 2.

Europium, cadmium, molybdenum, nickel, lead and calcium isotopes were obtained from Oak Ridge National Laboratories. The isotopes were thoroughly and controllably dispersed throughout a carrier of 1-micron particle size ultra-pure graphite. Sequential dilutions of a master mix resulted in a set of standards containing 1, 10, 100 and 1000 ppmw of each isotope.

The homogeneity of the 1000 ppm standard was examined by pressing electrodes of the standard and producing a graded series of mass spectra. The resulting spectra were measured densitometrically; Fig. 1 shows the spectra produced and Fig. 2 a plot of the line density versus the log of exposure for all six isotopes. The points fall on reasonably straight and parallel lines. Since each exposure represents a separate portion of the sample, the plots in Fig. 2 demonstrate that a good degree of homogeneity has been achieved.

EVALUATION OF SPEX SPIKES

Equal parts by weight of a Spex Spikes standard and of U_3O_8 standards obtained from the New Brunswick Laboratory of the U.S. Atomic Energy Commission, were mixed in a Wig-L-Bug. The mixtures were pressed into ϕ 7/64" electrodes using the isostatic die of Associated Electrical Industries. The pair of electrodes was mounted in the source of the mass spectrograph. A graded series of exposures ranging from 5×10^{-7} to 1×10^{-12} coulombs, in steps of 5, 2 and 1, were recorded on Ilford Q-2 plates which were subsequently processed in the usual manner. The exposure required to produce a faint line (about 90% T) for each of the standard isotopes was estimated by visual examination of the plate. Similarly, the exposures required to produce lines of similar intensity for isotopes of various elements were estimated. Results are presented in Table 1.

These data would be considered to be quite acceptable for a survey type mass spectral analysis of an unknown sample and were obtained without reference to standard samples other than the Spikes. To verify further the validity of the technique, a sample of tantalum carbide, previously analyzed by emission spectrometry, was mixed with Spex Spikes and analyzed mass spectrographically. The results, in Table 2, are again quite reasonable.

Table 2

Analysis of TaC by emission and Spex Spikes Visual Comparison Technique (values in ppmw)

Element	Emission	MS
W	220	200
Nb	260	200
Cu	48	20
Ni	10	10
Co	5	5
Fe	500	500
Cr	70	50
Mn	17	10
Ca	85	100
Si	30	50

The amounts of the elements were calculated on the basis of the well known and accepted empirical equation accounting for the response of the Q2 emulsion to ions of different masses:

$$\text{ppm} = C \times E_1/E_2 \times (M_2/M_1)^{3/2} \times 100/\%ISO$$

where: ppm = concentration of "unknown" element

C = concentration of standard isotope in Spex Spikes

E_1 = Exposure required to produce a faint line for the standard isotope

E_2 = Exposure required to produce a faint line for the "unknown" isotope

M_2 = Atomic Mass of unknown isotope

M_1 = Atomic Mass of standard isotope

%ISO = Natural abundance of measured isotope

Table 1

Analysis of NBL U_3O_8 Standards Using Spex Spikes Visual Comparison Technique (Values in ppmw)

Element	STD 95-1		STD 95-4		STD 95-6	
	Actual	Found	Actual	Found	Actual	Found
Bi	50	50	5	5	1	0.5
Pb	50	50	5	5	2	2
Sn	50	50	5	10	1	Excessive Background
Cd	5	2	0.5	1	0.2	0.2
Ag	5.1	10	1	0.5	0.5	0.5
Mo	51	100	6	5	2	1
Zn	500	200	50	50	10	5
Cu	51	50	5	5	1.5	2
Ni	110	100	12	10	4	5
Fe	520	500	66	50	22	20
Mn	54	50	6	5	2	1
Cr	95	100	14	10	5	5
V	100	100	11	10	3	5
Ca	25	20	5	contaminated	1	contaminated
B	5	5	0.6	0.5	-	-

The logic of this calculation is straightforward. The higher the concentration of an isotope in a sample, the shorter is the exposure required to see it. Therefore, the concentration is inversely proportional to the exposure. The mass spectrograph is approximately equally responsive to equal atomic concentrations; therefore the photographic density for equal weight concentrations is inversely proportional to the atomic weight of the element. However, Q-2 plates are less responsive to heavy ions, a feature which is approximately compensated by the square root of the ratio of the compared masses (7). The amount of an element present is equal to the amount of an isotope of that element divided by the fraction of the element represented by the isotope. When all these considerations are combined, the above equation results.

In the case of elements, an isotope of which is included in the standard, a different calculation is required. The effect of adding a spike, highly enriched in one isotope of an element, to a sample containing a quantity of that element with a normal isotopic ratio is to alter that ratio. Comparison of the ratios of isotopes before and after spiking can be used to calculate the ratio of the amount of the element in the sample to the amount of the spike. Of course, we must assume that the isotopes are present in the original sample in their natural proportions. The required measurement is of the ratio of the spiked isotope to one of the major naturally occurring isotopes. The expression for calculating the amount of an isotope of an element present (Pb, Eu, Cd, Mo, Ni, Ca) in a sample is:

$$C = (R_2 - R_3)S / (1 - R_2/R_1) \times 100/\% \text{ ISO}$$

- where:
- C = concentration of the element
 - S = concentration of added isotope
 - %ISO = % of the element present as a major isotope in the original sample
 - R₁ = Ratio of major isotope to spiked isotope in the original sample (either measured or taken from tables of natural abundances)
 - R₂ = Ratio of major isotope to spiked isotope in the spiked sample as measured by mass spectrograph.
 - R₃ = Ratio of the major isotope to the spiked isotope in the spiking material.

Inspection of the isotope dilution calculation leads to several noteworthy observations: 1) If the spiked isotope is a very minor natural isotope, R₁ becomes large and the denominator approaches a value of 1; 2) With a highly refined spiking isotope, R₃ approaches zero; 3) If both of these conditions can be fulfilled, the spiked isotope can serve as an internal standard in the usual sense for the major naturally occurring isotope. It is for these reasons that relatively minor isotopes were sought for the Spex Spikes.

As an example of the application of this equation, equal parts of 10 ppm Spex Spike and of NBL U₃O₈ standard 95-4 were mixed. Using conventional photometric techniques, the relative intensity of the Ni⁶² ion line was found to be 4.0 and of the Ni⁵⁸ line, 3.0. Table 4 lists the natural abundances of Ni⁵⁸ and of Ni⁶² which are 67.9% and 3.66% respectively; therefore R₁ = 67.9/3.66 = 18.55. The Ni⁶² used in the Spex Spike contains 1.83% Ni⁵⁸ and 96.16% Ni⁶², therefore R₃ = 0.0190. R₂ was measured to be 0.75.

Table 3
Analysis of U.S. Geol. Surv. BCR-1
Spex Spikes Visual Comparison Technique, ppmw

Element	Average Value (8)	Range (8)	Visual MS
Ag	0.044		<0.5
As	<30		<0.5
Au	0.0007		<0.5
B	—		5
Ba	790	480-1230	500
Be	2.6		1
Bi	<0.5		<0.2
Br	0.18		2 *
Cd	0.067		0.2
Ce	49	40-53	20
Cl	84		50
Co	35.5	29-60	20
Cr	16.3	8-45	10
Cs	1.2		0.5
Cu	22.4	7-33	10
Dy	—		5
Eu	2.2		2
F	485		100
Ga	21.6	16-30	20
Ge	1.2		1
Hf	4.4		5
Hg	0.006		<1
In	0.086		0.2
Ir	0.0007		<0.5
La	28.8	22-36	20
Li	16.9		10
Lu	0.6		0.2
Mn	1350	1040-1600	2000
Mo	3.9		2
Nb	34.1		20
Ni	15	8-30	20
O	44.6%		—
Os	<0.00001		<1
Pb	18	5-35	10
Pd	<0.004		<1
Pr	5		5
Pt	<0.01		<1
Rb	72.8	45-150	50
Re	0.0008		<0.5
Rh	<0.005		<0.5
Sb	0.59		0.5
Sc	36.5		50
Se	0.10		<2
Sm	7.3		5
Sn	1.4		5 *
Sr	345	244-525	500
Ta	0.88		1
Tb	1		1
Th	6.81		5
Tl	0.36		<1
Tm	0.6		0.5
U	1.73		2
V	384	120-700	500
W	0.7		1
Yt	46.2		50
Yb	4.4		2
Zn	132	94-278	100
Zr	185	144-275	200

* Found to be inhomogeneously distributed.

< = Not detected

In Column 2 the range of values reported by many analysts using many techniques is reported.

$$M_1 = \frac{10 (0.75 - 0.0190)}{1 - 0.75/18.55} = \frac{7.31}{0.9596}$$

7.6 ppm Ni⁵⁸

and the Ni content of the sample is

$$7.6 \div 0.679 = 11.2 \text{ ppm Ni in sample.}$$

The certified value of nickel in this sample is 12 ppm.

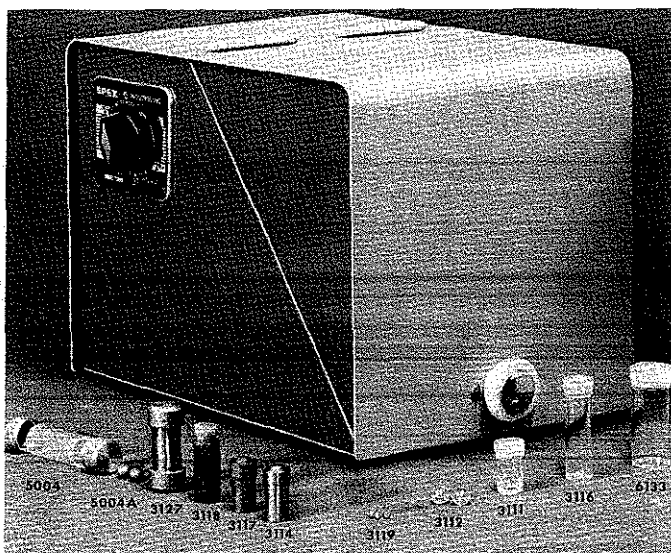
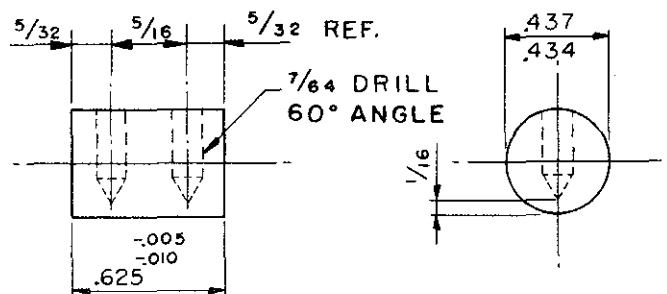
As a final verification of the utility of Spex Spikes, 25 mg of U.S. Geological Survey Standard BCR-1 was mixed with 25 mg of 100 ppm Spex Spike and the usual series of exposures were produced. A complete survey of all trace elements present in the sample was made, referencing the spiked isotopes as internal standards. All estimates were made by visual comparison only. The resulting analysis is presented in Table 3 along with average values reported by Flanagan (8) in his report on this material. Once again, we see that the simple Spex Spikes procedure leads to quite acceptable semiquantitative results for a highly complex material. Densitometric measurements would lead to improved precision and the use of a standard sample would permit relative sensitivity factors to be calculated for quantitative analyses of subsequent samples.

SPEX Spikes, a series of graphite based standards containing 1, 10, 100 and 1000 ppm of six minor isotopes, has proved to be a valuable adjunct to the Mass Spectrometry Laboratory. By the simple expedient of mixing equal parts by weight of the appropriate standard and powdered sample, electrodes can be produced containing internal standards well spaced through the spectrum. Semiquantitative trace analyses can be performed by simple visual examination of the resulting plates. By refining the measurement techniques, even more accurate results can be obtained.

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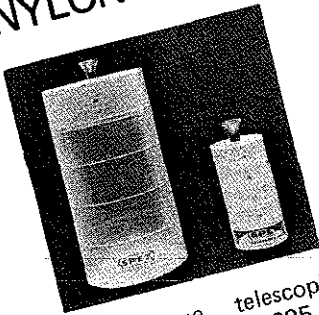
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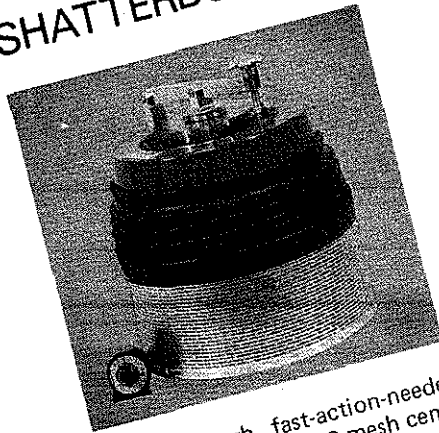


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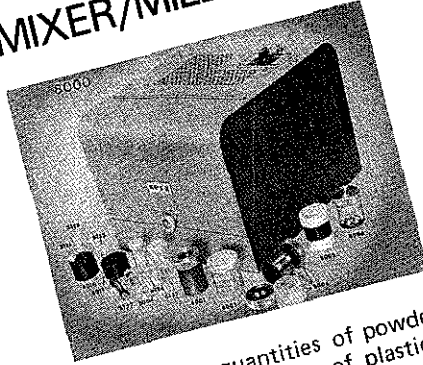
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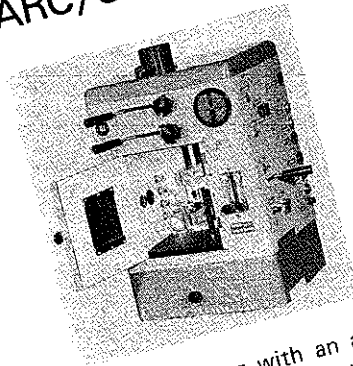
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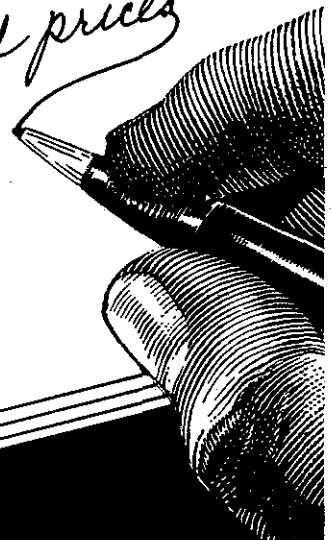
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151-200 mg		27.00	95.00	154.00	648.00

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up to		33.00	115.00	190.00	792.00
400 mg		33.00	115.00	190.00	792.00
900 mg	±2 mg	45.00	155.00	256.00	1135.00

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		100	500	1000	5000
30-100 mg		35.00	108.00	163.00	715.00
101-150 mg	±2.0 mg	41.00	115.00	190.00	878.00
151-200 mg		47.00	122.00	210.00	893.00

LITHIUM CARBONATE-GRAPHITE, -100 mesh powder 1:1 weight

		100	500	1000	5000
30-100 mg		31.00	98.00	154.00	676.00
101-150 mg	±2.0 mg	34.00	103.00	169.00	708.00
151-200 mg		37.00	107.00	180.00	777.00

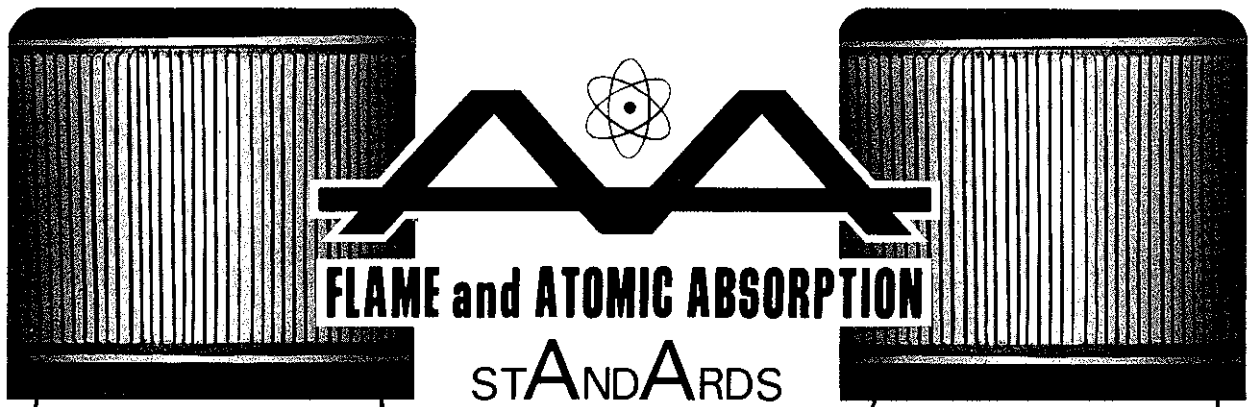
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1800 mg		33.00	125.00	198.00	957.00

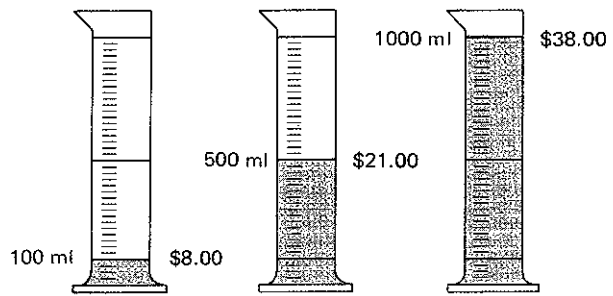
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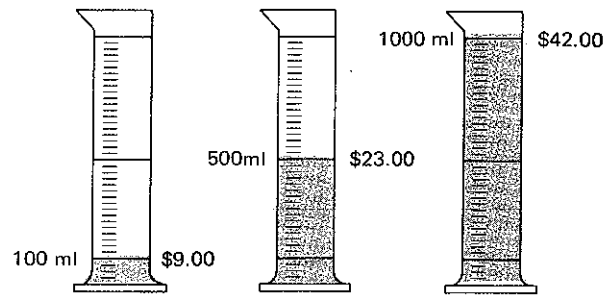
500 µg/ml SOLUTIONS

Be Cd Li Mg Na Zn



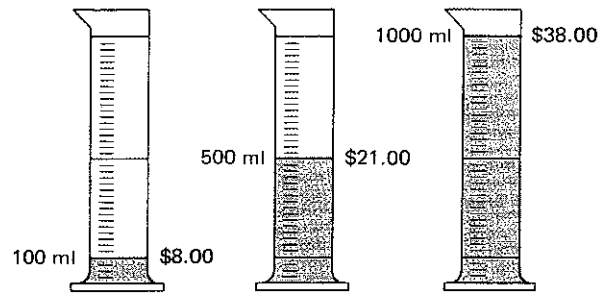
5000 µg/ml SOLUTIONS

Al As B Bi Dy Ge Ho Hg In Mo Se Sb Si Sn Te Ti Tl



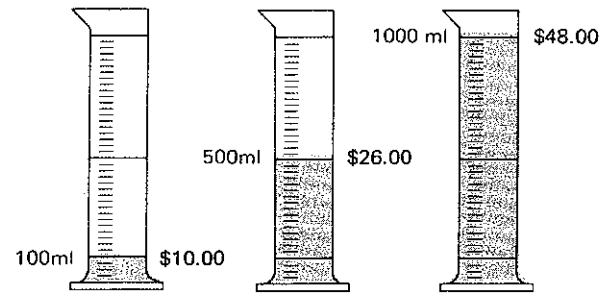
1000 µg/ml SOLUTIONS

Ag Ba Ca Co Cr Cs Cu Fe K Mn Ni Pb Rb Sr V



10000 µg/ml SOLUTIONS

Gd Hf La Nb Pr Sm Ta U W Zr



ADDITIONAL RARE EARTHS AND NOBLE METALS

ELEMENT	Concentration µg ml	Price 100 ml	Price 500 ml	Price liter
Au	1000	\$11.00	\$28.00	\$50.00
Rh	1000	11.00	28.00	50.00
Yb	1000	11.00	28.00	50.00
Er	5000	11.00	28.00	50.00
Eu	5000	11.00	28.00	50.00
Ga	5000	11.00	28.00	50.00
Pd	5000	11.00	28.00	50.00
Pt	5000	15.00	53.00	95.00

ELEMENT	Concentration µg ml	Price 100 ml	Price 500 ml	Price liter
Ru	5000	\$10.00	\$26.00	\$50.00
Sc	5000	11.00	42.00	75.00
Tm	5000	11.00	42.00	75.00
Nd	10000	11.00	42.00	75.00
Re	10000	11.00	42.00	75.00
Tb	10000	11.00	42.00	75.00
Y	10000	11.00	42.00	75.00

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