An Evaluation of Whole-Order ½-order, and ⅓-order Reporting in Semiquantitative Spectrochemical Analysis

GEOLOGICAL SURVEY BULLETIN 1084-H

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By PAUL R. BARNETT

CONTRIBUTIONS TO GEOCHEMISTRY

GEOLOGICAL SURVEY BULLETIN 1084-H

UNITED STATES GOVERNMENT PRINTING OFFICE, WASHINGTON : 1961

UNITED STATES DEPARTMENT OF THE INTERIOR

STEWART L. UDALL, *Secretary*

GEOLOGICAL SURVEY

Thomas B. Nolan, *Director*

For sale by the Superintendent of Documents, U.S. Government Printing Office
Washington 25, D.C. - Price 15 cents (paper cover)

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AN EVALUATION OF WHOLE-ORDER, ½-ORDER, AND ⅓-ORDER REPORTING IN SEMIQUANTITATIVE SPECTROCHEMICAL ANALYSIS

By Paul R. Barnett

ABSTRACT

The results of 507 spectrochemical determinations in 63 samples are evaluated by the percentage of successful assignments to the correct range and by calculation of the logarithmic standard deviation. The intervals in terms of the standard deviation are approximately the same whether computed by the percentage of hits or by the usual method for replicate determinations (after converting the data to logarithms). The size of the reporting interval is from 4 to 6 times the standard deviation for the whole order, 2.7 times the standard deviation for the ½-order, and still approximately twice the standard deviation for the ⅓-order of magnitude breakdown.

INTRODUCTION

Spectrochemical analysis is a null method in which the light intensity emitted at a specific wavelength by an excited atom in an unknown is compared with the intensity emitted at the same wavelength by an atom of the same element in a sample or series of samples of known concentration. In the quantitative spectrochemical analysis a microphotometer is used to interpolate between the "standards." Because such interpolation is time consuming many spectrochemists have devised methods of making visual comparisons of the unknown with the standards. Most of these methods consist of recording on a photographic negative the spectra of a series of prepared standards of decreasing concentration in the element or elements to be determined in the unknown. The spectrum of the unknown is recorded on a separate negative and the appropriate line in this spectrum compared visually with the corresponding line in the standard spectrograms.

As in any work of this type the analyst must decide upon the size and nature of the intervals between standards. Because concentrations may range from less than a part per million to more than 100,000 parts per million, a linear spacing is seldom used. Instead an exponential, or logarithmic, interval is usually employed. Waring and Annell (1953) use standards spaced at intervals of powers of 10 (0.0001, 0.001, 0.01, . . . percent) and report each result as a concentration range, 0.001 to 0.01 percent or 0.01 to 0.1 percent, for example. Mitchell (1948, p. 68) uses a spacing of one-half this size by varying the trace elements in the prepared standards in steps of $\sqrt{10}$ (3.16) times, "* * * which gives even concentration intervals and a convenient gradation." Harvey (1950, p. 152) uses standards with concentrations of 0.0001, 0.0003, 0.001, 0.003, . . . percent, (a factor of approximately $\sqrt{10}$), and reports the results as a range covering a factor of 10, that is, 0.03–0.3 percent or 0.1–1 percent. Allen (1952, p. 921) reports in the ranges 0.5–5.0 percent, 0.05–0.5 percent, 0.005–0.05 percent, 0.005–0.005 percent, and less than 0.0005 percent, but divides each range into three parts by suffixing plus and minus signs. This gives roughly a factor of two between the limits of reporting ranges. In work done in the Geological Survey laboratory, standards are used in which the concentrations of the trace elements increase by a factor of $\sqrt[3]{10}$, or approximately 2.15. The results are reported as the approximate midpoints of the ranges defined by the above standards, that is, 0.015, 0.03, 0.07, 0.15, . . . percent.

An analysis performed by the use of standards differing in concentrations by a factor of 10 has come to be known as 1-step or whole order of magnitude semiquantitative analysis, one with the use of standards differing by a factor of 3.16 ($\sim \sqrt{10}$) as 2-step or 1/2-order of magnitude, and one with standards with a factor of 2.15 ($\sim \sqrt[3]{10}$) as 3-step or 1/3-order.

EVALUATION OF RESULTS

The matching may be considered to have resulted in a correct analysis (a "hit") whenever the reported value and the accepted value lie between the same consecutive standards (that is, in the same bracket or range) and as a "miss" if they fall in different brackets. However, a high proportion of hits is not necessarily desirable. It is obvious that as the interval between standards is increased the percentage of hits will increase, but if the interval is made too great much valuable information is lost. At the extreme a bracket extending from 100 percent to 0 percent would result in all hits and no misses. As the interval is made smaller the percentage of hits decreases. If it is made sufficiently small any hit would be essentially fortuitous and the number of hits would approach zero. An analysis with no hits could conceivably be very accurate, with values having a very small deviation from the accepted values.

As pointed out by Youden, Connor, and Severo (1959) the closeness of the true value of the unknown to a concentration of a step of the standard is an important consideration in making measurements by matching. Matching will be most successful when the unknown coincides with one of the standards and least successful when it lies half way between two successive standards. In reporting by brackets rather than by matching, a true value at the midpoint of the bracket is most likely to be placed in the proper bracket and a true value coinciding with a step of the standard is least likely to be correctly placed. In spite of this reversal of conditions, the conclusion is valid that for an interval equal to 2σ (two times the standard deviation of the method) the analysis of unknowns will result in 60.9 percent hits. This is the average value of the area under the normal curve between plus 1σ and minus 1σ as the unknown varies from the central value of the interval to the limiting value. Table 1 gives the results of calculation of percentage hits for various intervals expressed in terms of σ. The paired values up to 10σ are used in constructing the curve in figure 28. This curve is the same as curve *A* as drawn by Youden, Connor, and Severo (1959).

Waring and Annell (1953) had 10 misses in 304 determinations or 97 percent hits. From table 1 this would indicate that their bracket (of factor 10) is approximately 30 times the σ of their method. A. T. Myers, R. G. Havens, and P. J. Dunton (1961) using an interval of 1/3-order of magnitude had 69.3 percent hits in 682 tries. From figure 28 it is found that this interval is about 2.6σ.

TABLE 1.—*Interval versus percentage hit*

Interval (in multiples of σ)	Percent hits	Interval (in multiples of σ)	Percent hits
0. 0	0	5. 5	85. 45
0. 25	9. 9	6. 0	86. 7
0. 50	19. 5	6. 5	87. 7
1. 0	36. 2	7. 0	88. 6
1. 5	50. 7	7. 5	89. 3
2. 0	60. 0	8. 0	90. 0
2. 5	68. 2	8. 5	90. 6
3. 0	73. 4	9. 0	91. 1
3. 5	77. 2	9. 5	91. 6
4. 0	80. 0	10	92. 0
4. 5	82. 2	20	96. 0
5. 0	84. 0	30	97. 3

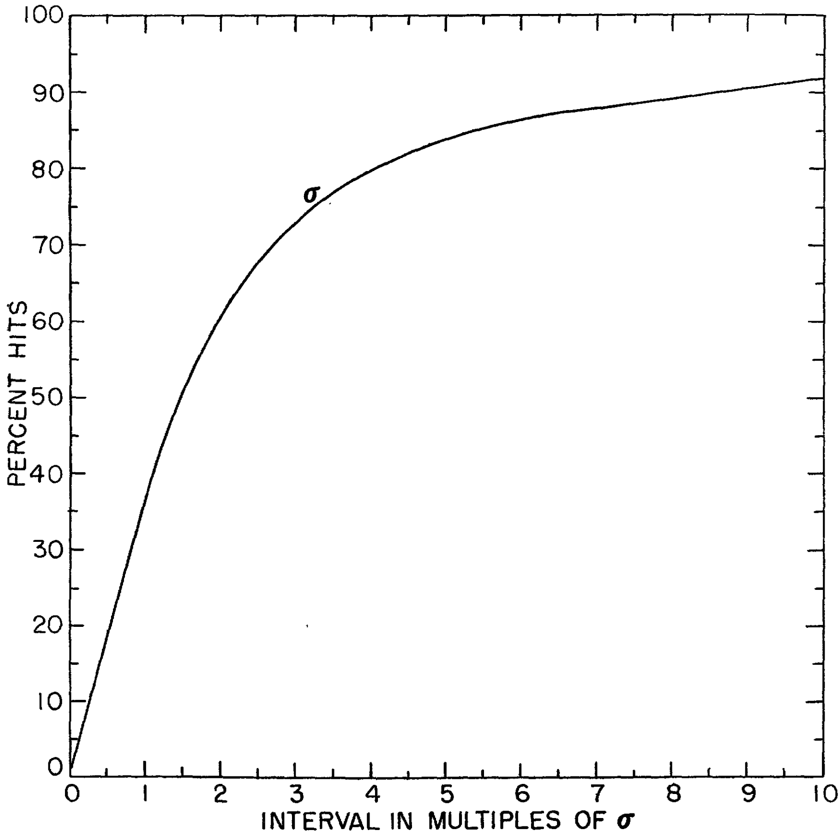

FIGURE 28.—Percentage hits versus interval.

EVALUATION OF SPECTROCHEMICAL ANAYLSES OF 30 SAMPLES OF VEIN MATERIAL AND METAMORPHIC ROCK

Myers, Havens, and Dunton made 264 determinations on elements in a suite of 30 samples of vein material and metamorphic rock. They used a standard interval of 1/3-order of magnitude and achieved 67 percent hits. From figure 28 it is seen that 67 percent success in bracketing gives an interval of about 2.4σ. When the spectrochemical results are grouped into brackets with a factor of 10, the percentage of hits increases to 86 and the interval is 5.7σ. The 30 samples of this suite were analyzed by Nancy M. Conklin, using standards at intervals of 1/2-order of magnitude. This analysis resulted in 71 percent hits or an interval of 2.8σ.

A complete tabulation of the results of the analyses of these 30 samples is given in table 4.

EVALUATION OF SPECTROCHEMICAL ANALYSES OF 26 NATIONAL BUREAU OF STANDARDS STANDARD SAMPLES AND 7 OTHER MISCELLANEOUS SAMPLES

The samples listed and described in table 5 were analyzed by the author, who used three sets of standards of different intervals. The first standard had an interval of a whole order, the second ½-order and the third ⅓-order of magnitude. The results of these analyses are given in table 6. This set of 33 samples was analyzed in an attempt to duplicate or approximate the results obtained with the suite of 30 silicate rock samples differing more widely in composition. The statistical evaluation of the results is given in table 2, along with that of the suite of 30 samples and of the combined sets. As expected, a decrease in the size of the reporting bracket resulted in a decrease in the percentage of hits. For the whole-order reporting of the results on the 63 samples of the combined sets, the reporting interval is 5.7 times as large as the standard deviation of the method. For the ½-order the interval is 2.7 times the standard deviation and for the ⅓-order the reporting interval is still more than twice the standard deviation.

In their development of the argument for the method of evaluating results obtained by matching techniques, Youden, Connor, and Severo (1959) point out that the distribution of the *fractional values* is usually approximately rectangular. Figure 29 is a histogram of the fractional values of the 507 accepted true concentrations of trace elements in the 63 samples of the combined sets. The distribution does not appear to be rectangular, but is strongly suggestive of a rough logarithmic distribution. Curve *A* in figure 30 is the cumulative distribution curve (on logarithmic probability paper). Curve *B* is the cumulative logarithmic distribution and Curve *C* is the cumulative rectangular distribution. Comparison of the three curves shows that the fractional values have a distribution considerably closer to logarithmic than to rectangular. This does not invalidate the foregoing method of evaluation of the spectrographic data. It simply means that the *logarithmic* fractions of the true values of the unknowns have a rectangular distribution and that the σ's are logarithmic.

TABLE 2.—*Intervals in multiples of σ for various reporting brackets*

	Number of determinations	Percent hits			Interval (σ)		
		Whole-order	½-order	⅓-order	Whole-order	½-order	⅓-order
Suite of 30	265	86	71	66	5.7	2.8	2.4
Set of 33	242	85	70	57	5.3	2.7	1.8
Combined sets	507	86	70	62	5.7	2.7	2.1

579108—61——2

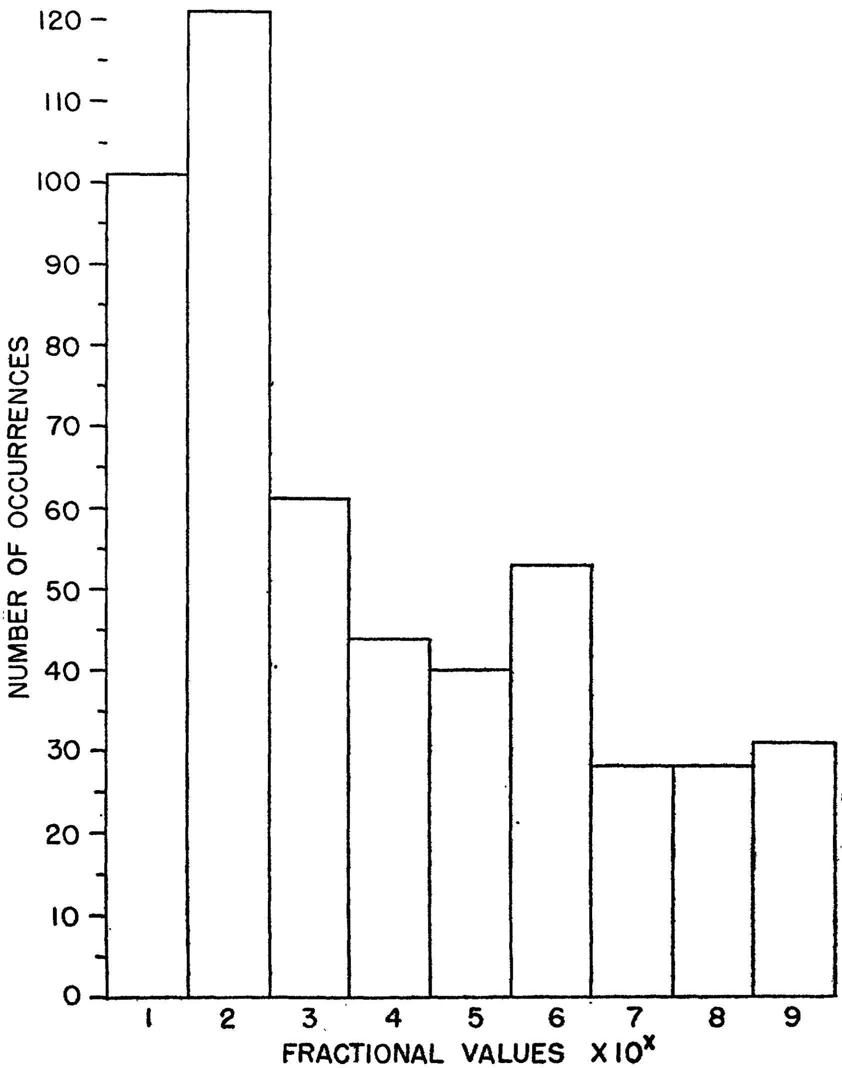

FIGURE 29.—Frequency of fractional values.

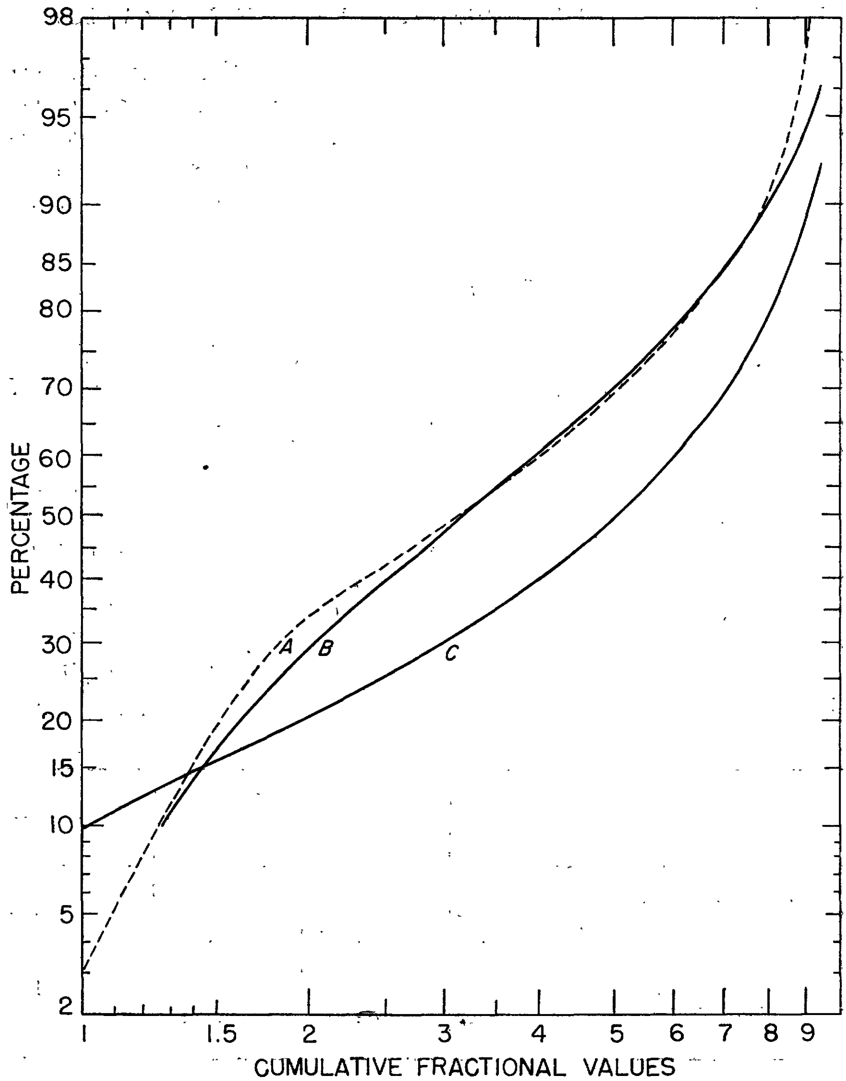

FIGURE 30.—Cumulative distribution of fractional values on largarithmic probability paper. Curve *A* is constructed from the 507 accepted true concentrations of elements in the 63 samples of this study. Curve *B* represents cululative logarithmic distribution and Curve *C* rectangular distribution.

EVALUATION BY LOGARITHMIC TRANSFORMATION

The conclusion that the fractional values of the unknowns are logarithmically distributed suggests converting the accepted values to logarithms and the spectrochemical value or range to the logarithmic midpoint of the range. These duplicate measurements lend themselves easily to calculation of standard deviation (σ) by use of the formula

$$\sigma=\sqrt{\frac{\text{sum of } d^2}{2n}}$$

where d is the difference between duplicates and n is the number of sets of duplicate measurements. Table 3 illustrates this method of calculating the logarithmic standard deviation (L.S.D.), the interval in terms of the L.S.D., and the geometric deviation (G.D.). G.D., a term suggested by E. M. Shoemaker, is the antilogarithm of the L.S.D. and is the factor by which a result may be multiplied or divided to find the limits of plus one and minus one logarithmic standard deviation.

The L.S.D., the interval in terms of the L.S.D., and the G.D. for the individual elements are given in tables 4 and 6.

The 507 spectrographic determinations by each of three standards with different intervals were evaluated by this logarithmic transformation technique and the results are as follows:

	Reporting interval		
	Whole-order	½-order	⅓-order
Logarithmic interval	1.000	0.500	0.333
Logarithmic standard deviation	.254	.187	.159
Interval in multiples of logarithmic standard deviation	4.0	2.7	2.1
Geometric deviation	1.80	1.54	1.44

The size of the interval in terms of the standard deviation as determined by the percentage of hits is compared with the size of the interval in terms of the logarithmic standard deviation as follows:

	Reporting interval		
	Whole-order	½-order	⅓-order
Interval in terms of σ [1]	5.7	2.7	2.1
Interval in terms of L.S.D	4.0	2.7	2.1

[1] As pointed out above this is actually logarithmic.

TABLE 3. *Calculation of logarithmic standard deviation of cobalt analyses*

[Chemical analyses by Claude Huffman, Jr.]

Sample number	Chemical	Spectrochemical			Logarithm				Differences between (6) and (7) (8) (9)			Square of differences, respectively		
		Whole-order	½-order	⅓-order	Chemical	Whole-order	½-order	⅓-order						
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)	(15)
	(Percent)	*(Percent)*	*(Percent)*	*(Percent)*										
21	0.018	0.03	0.01	0.015	$\bar{2}.26$	$\bar{2}.50$	$\bar{2}.00$	$\bar{2}.17$	0.24	0.26	0.09	0.0576	0.0676	0.0081
27	.017	.03	.01	.015	$\bar{2}.23$	$\bar{2}.50$	$\bar{2}.00$	$\bar{2}.17$	.27	.23	.06	.0729	.0529	.0036
28	.008	.03	.01	.015	$\bar{3}.90$	$\bar{2}.50$	$\bar{2}.00$	$\bar{2}.17$	.60	.10	.27	.3600	.0100	.0729
30	.012	.03	.01	.015	$\bar{2}.08$	$\bar{2}.50$	$\bar{2}.00$	$\bar{2}.17$	.42	.08	.09	.1764	.0064	.0081
32	.021	.03	.03	.015	$\bar{2}.32$	$\bar{2}.50$	$\bar{2}.50$	$\bar{2}.17$	.18	.18	.15	.0324	.0324	.0225
33	.007	.03	.01	.015	$\bar{3}.85$	$\bar{2}.50$	$\bar{2}.00$	$\bar{2}.17$	.65	.15	.32	.4225	.0225	.1024
35	.04	.03	.03	.03	$\bar{2}.60$	$\bar{2}.50$	$\bar{2}.50$	$\bar{2}.50$	.10	.10	.10	.0100	.0100	.0100
37	.018	.03	.03	.015	$\bar{2}.26$	$\bar{2}.50$	$\bar{2}.50$	$\bar{2}.17$	.24	.24	.09	.0576	.0576	.0081
38	.016	.03	.03	.015	$\bar{2}.20$	$\bar{2}.50$	$\bar{2}.50$	$\bar{2}.17$	.30	.30	.03	.0900	.0900	.0009
39	.006	.003	.01	.007	$\bar{3}.78$	$\bar{3}.50$	$\bar{2}.00$	$\bar{3}.83$	.28	.22	.05	.0784	.0484	.0025
40	.016	.03	.03	.015	$\bar{2}.20$	$\bar{2}.50$	$\bar{2}.50$	$\bar{2}.17$	.30	.30	.03	.0900	.0900	.0009
41	.05	.03	.03	.03	$\bar{2}.70$	$\bar{2}.50$	$\bar{2}.50$	$\bar{2}.50$	.20	.20	.20	.0400	.0400	.0400
42	.012	.03	.01	.015	$\bar{2}.08$	$\bar{2}.50$	$\bar{2}.00$	$\bar{2}.17$	.42	.08	.09	.1764	.0064	.0081
44	.013	.03	.01	.015	$\bar{2}.11$	$\bar{2}.50$	$\bar{2}.00$	$\bar{2}.17$	.39	.11	.06	.1521	.0121	.0036
46	.018	.03	.01	.015	$\bar{2}.26$	$\bar{2}.50$	$\bar{2}.00$	$\bar{2}.17$	.24	.26	.09	.0576	.0676	.0081
Sum												1.8739	0.6139	0.2998
Sum/2n=sum/30												.0625	.0205	.0100
L.S.D												.250	.143	.100
Interval in multiples of L.S.D												4.0	3.5	3.3
G.D												1.78	1.39	1.26

The intervals in terms of the standard deviation are approximately the same whether computed by the percentage hits or by the usual method for duplicate determinations (after converting the data to logarithms). This interval decreases as the reporting interval decreases. The size of the reporting interval is from 4 to 6 times the standard deviation for the whole order, 2.7 times the standard deviation for the ½-order, and still approximately twice the standard deviation for the ⅓-order of magnitude breakdown. The largest bracket obviously is least discriminative.

It appears that for greater efficiency the size of the interval should be even less than the ⅓-order. This could be done without increasing appreciably the length of time required for a determination. The practicable extent to which the size of the interval can be reduced would seem to depend, at least in part, upon the number of spectrograms that can be recorded on one standard plate. Youden, Connor, and Severo (1959) also show that "* * * making the width of the interval less than sigma does not cut the average absolute value of the 'miss' appreciably." These two considerations probably are the most important ones in helping the spectrographer determine the size of the reporting interval he should use.

COMPARATIVE DATA

Comparisons were made of spectrochemical results when reported as whole-order, ½-order, and ⅓-order with results by other quantitative methods. Samples of 30 vein materials and metamorphic rocks were used for the comparison shown in table 4. Table 5 lists a variety of other samples (mostly National Bureau of Standards standard samples) used for the comparison shown in table 6.

TABLE 4.—*Results of semiquantitative spectrochemical analysis of 30 samples of vein material and metamorphic rocks*

Silver and cobalt

Sample number	Silver (in percent)				Cobalt (in percent)			
	Fire assay [1]	Spectrochemical			Chemical [2]	Spectrochemical		
		Whole-order	½-order	⅓-order		Whole-order	½-order	⅓-order
17	0.00014	0.0003	0.0003	0.00015	--	--	--	--
18	.00054	.003	.003	.0015	--	--	--	--
19	.00014	.003	.001	.0015	--	--	--	--
20	--	--	--	--	--	--	--	--
21	.0047	.003	.003	.007	0.018	0.03	0.01	0.015
22	--	--	--	--	--	--	--	--
23	.0014	.003	.003	.0015	--	--	--	--
24	.00069	.003	.003	.0015	--	--	--	--
25	--	--	--	--	--	--	--	--
26	.00014	.0003	.0003	.0003	--	--	--	--
27	.0062	.003	.01	.007	.017	.03	.01	.015
28	.0042	.003	.01	.007	.008	.03	.01	.015
29	.00014	.0003	.0003	.0007	--	--	--	--
30	.0015	.003	.003	.003	.012	.03	.01	.015
31	--	--	--	--	--	--	--	--
32	.0062	.003	.01	.007	.021	.03	.03	.015
33	.0048	.003	.003	.007	.007	.03	.01	.015
34	--	--	--	--	--	--	--	--
35	.0180	.03	.01	.015	.04	.03	.03	.03
36	--	--	--	--	--	--	--	--
37	.0057	.03	.01	.015	.018	.03	.03	.015
38	.0096	.03	.03	.015	.016	.03	.03	.015
39	.0012	.003	.003	.003	.006	.003	.01	.007
40	.0121	.03	.01	.015	.016	.03	.03	.015
41	.0243	.03	.03	.015	.05	.03	.03	.03
42	.0036	.003	.01	.007	.012	.03	.01	.015
43	.00069	.003	.003	.003	--	--	--	--
44	.0031	.003	.001	.003	.013	.03	.01	.015
45	.0016	.003	.001	.003	--	--	--	--
46	.0067	.003	.01	.007	.018	.03	.01	.015
L.S.D.		.341	.295	.257	--	.250	.143	.100
Interval in multiples of L.S.D.		2.9	1.7	1.2	--	4.0	3.5	3.3
G.D.		2.19	1.97	1.81	--	1.78	1.39	1.26

See footnotes at end of table.

TABLE 4.—*Results of semiquantitative spectrochemical analysis of 30 samples of vein material and metamorphic rocks*—Continued

Copper and manganese

Sample number	Copper (in percent)				Manganese (in percent)			
	Chemical [3]	Spectrochemical			Chemical [4]	Spectrochemical		
		Whole-order	½-order	⅓-order		Whole-order	½-order	⅓-order
17	0.03	0.03	0.03	0.03	0.10	0.3	0.1	0.15
18	.15	.3	.1	.15	.18	.3	.3	.3
19	.14	.3	.1	.15	.12	.3	.3	.3
20	.0095	.03	.01	.015	1.48	3	3	3
21	.15	.3	.1	.15	.41	.3	.3	.7
22	.02	.03	.01	.015	1.10	3	1	1.5
23	.09	.03	.1	.07	.40	.3	.3	.7
24	.08	.03	.1	.07	.46	.3	.3	.7
25	.03	.03	03	.03	3.69	3	3	3
26	.03	.03	.03	.03	.58	.3	.3	.7
27	.87	3	1	1.5	.062	.3	.1	.15
28	.14	.3	.1	.15	.046	.03	.1	.07
29	.11	.3	.1	.15	.023	.03	.1	.07
30	.19	.3	.3	.3	.054	.3	.1	.15
31	.03	.03	.03	.03	.085	.3	.1	.15
32	.23	.3	.3	.3	.062	.3	.1	.15
33	.09	.3	.1	.15	.35	.3	.3	.7
34	.09	.03	.1	.07	1.5	3	3	3
35	1.07	3	1	1.5	.14	.3	.1	.15
36	.22	.3	.3	.3	.070	.3	.1	.15
37	.95	3	1	1.5	.085	.3	.1	.15
38	.62	.3	.1	.7	.031	.03	.1	.07
39	.46	.3	1	.7	.015	.03	.03	.03
40	1.16	3	1	1.5	.015	.03	.03	.03
41	.89	3	1	1.5	.37	.3	.3	.3
42	.32	.3	.3	.3	.12	.3	.3	.3
43	.10	.3	.1	.15	.07	.3	.3	.15
44	.16	.3	.1	.15	.36	.3	.3	.7
45	.43	.3	.3	.7	.24	.3	.3	.3
46	.85	3	1	1.5	.062	.3	.1	.15
L.S.D.		.259	.0955	.0979	----------	.294	.202	.204
Interval in multiples of L.S.D.		3.9	5.2	3.4	----------	3.4	2.5	1.6
G.D.		1.82	1.25	1.25	----------	1.97	1.59	1.60

See footnotes at end of table.

TABLE 4.—*Results of semiquantitative spectrochemical analysis of 30 samples of vein material and metamorphic rocks*—Continued

Molybdenum and nickel

Sample number	Molybdenum (in percent)				Nickel (in percent)			
	Chemical [5]	Spectrochemical			Chemical [5]	Spectrochemical		
		Whole-order	½-order	⅓-order		Whole-order	½-order	⅓-order
17	0.04	0.03	0.03	0.07	0.0065	0.003	0.003	0.007
18	.22	.3	.3	.3	.01	.03	.01	.015
19	.038	.03	.03	.03	.0075	.003	.003	.007
20	.018	.03	.01	.015	.0065	.003	.003	.007
21	.60	.3	.3	.7	.0060	.03	.01	.03
22	.060	.03	.03	.07	.0020	.003	.01	.007
23	.18	.3	.1	.15	.011	.003	.003	.007
24	.072	.03	.1	.07	.0075	.003	.01	.007
25	.062	.03	.03	.07	.0085	.003	.003	.003
26	.060	.03	.03	.07	.0080	.003	.003	.007
27	.56	.3	1	.7	.022	.03	.01	.03
28	.43	.3	.3	.3	.011	.03	.01	.015
29	.04	.03	.03	.03	.0065	.003	.01	.007
30	.14	.3	.3	.15	.016	.03	.01	.015
31	.006	.003	.01	.007	.009	.003	.01	.007
32	.31	.3	.3	.7	.039	.03	.03	.03
33	.42	.3	.3	.7	.022	.03	.01	.03
34	.058	.03	.03	.07	.009	.003	.01	.007
35	.22	.3	.3	.3	.035	.03	.03	.03
36	.054	.3	.1	.15	.0085	.03	.01	.015
37	.21	.3	.3	.3	.018	.03	.03	.015
38	.15	.3	.3	.3	.018	.03	.03	.03
39	.07	.3	.1	.15	.014	.03	.01	.015
40	.22	.3	.3	.3	.016	.03	.01	.015
41	.84	.3	1	.7	.053	.03	.1	.07
42	.17	.3	.3	.15	.017	.03	.01	.015
43	.092	.3	.1	.15	.009	.003	.01	.007
44	.14	.3	.3	.3	.016	.03	.01	.015
45	.17	.3	.3	.3	.019	.03	.03	.03
46	.95	.3	1	.7	.041	.03	.03	.07
L.S.D.		.229	.154	.133		.256	.194	.154
Interval in multiples of L.S.D.		4.4	3.2	2.5		3.9	2.6	2.2
G.D.		1.69	1.43	1.36		1.80	1.56	1.43

See footnotes at end of table.

Table 4.—*Results of semiquantitative spectrochemical analysis of 30 samples of vein material and metamorphic rocks*—Continued

Lead and uranium

Sample number	Lead (in percent)				Uranium (in percent)			
	Chemical [6]	Spectrochemical			Chemical [7]	Spectrochemical		
		Whole-order	½-order	⅓-order		Whole-order	½-order	⅓-order
17	0.10	0.03	0.1	0.07	0.63	0.3	1	0.7
18	.46	.3	1	.7	.58	.3	1	.7
19	.06	.03	.03	.07	.26	.3	.3	.3
20	.009	.003	.01	.007	----------	----------	----------	----------
21	.81	.3	.3	.3	5.57	3	3	7
22	.21	.3	.3	.15	.33	.3	.3	.3
23	.23	.3	.3	.3	.58	.3	.3	.3
24	.08	.03	.1	.07	.66	.3	.3	.7
25	.05	.03	.03	.015	.067	.03	.1	.07
26	.07	.03	.03	.03	.084	.03	.03	.07
27	1.05	3	1	1.5	.79	.3	1	.7
28	.46	.3	1	.7	1.95	3	1	1.5
29	.11	.03	.1	.07	.033	.03	.03	.07
30	.63	.3	1	.7	.72	.3	1	.7
31	.03	.03	.03	.03	----------	----------	----------	----------
32	.73	.3	1	.7	1.59	3	1	1.5
33	.69	.3	.3	.3	4.59	3	3	7
34	.13	.03	.1	.07	.50	.3	.3	.3
35	.62	.3	1	.7	3.61	3	3	3
36	.06	.03	.1	.07	.28	.3	.3	.3
37	.29	.3	.3	.3	.58	.3	1	.7
38	.37	.3	.3	.7	1.15	3	1	1.5
39	.08	.3	.1	.15	.11	.3	.1	.15
40	.70	.3	1	.7	2.79	3	3	3
41	.60	.3	1	.7	3.70	3	3	3
42	.16	.3	.3	.3	1.11	.3	1	.7
43	.10	.3	.1	.15	.29	.3	.3	.3
44	.28	.3	.3	.3	2.49	3	3	3
45	.73	.3	1	.7	1.37	3	1	1.5
46	1.45	3	3	1.5	6.36	3	10	7
L.S.D.		.247	.147	.145	----------	.198	.128	.084
Interval in multiples of L.S.D.		4.0	3.4	2.3	----------	5.1	3.9	4.0
G.D.		1.77	1.40	1.40	----------	1.58	1.34	1.21

See footnotes at end of table.

TABLE 4.—*Results of semiquantitative spectrochemical analysis of 30 samples of vein material and metamorphic rocks*—Continued

Vanadium and zinc

Sample number	Vanadium (in percent)				Zinc (in percent)			
	Chemical [8]	Spectrochemical			By chemistry [9]	Spectrochemical		
		Whole-order	½-order	⅓-order		Whole-order	½-order	⅓-order
17	0.02	0.03	0.01	0.015	----------	----------	----------	----------
18	.03	.03	.03	.07	----------	----------	----------	----------
19	.03	.03	.03	.03	----------	----------	----------	----------
20	.01	.03	.01	.03	----------	----------	----------	----------
21	.03	.03	.03	.07	0.086	0.03	0.03	0.07
22	.05	.03	.03	.03	----------	----------	----------	----------
23	.08	.03	.03	.03	----------	----------	----------	----------
24	.03	.03	.01	.03	----------	----------	----------	----------
25	.02	.03	.01	.015	----------	----------	----------	----------
26	.04	.03	.03	.03	----------	----------	----------	----------
27	.03	.03	.03	.03	.19	.03	.1	.07
28	.02	.03	.03	.03	.056	.03	.03	.03
29	.01	.03	.01	.015	.062	.03	.1	.03
30	.02	.03	.01	.015	.16	.3	.1	.15
31	.02	.03	.01	.015	.11	.03	.1	.07
32	.03	.03	.03	.03	.088	.03	.1	.07
33	.05	.03	.03	.03	----------	----------	----------	----------
34	.03	.03	.01	.03	----------	----------	----------	----------
35	.05	.03	.03	.07	.11	.03	.1	.07
36	.03	.03	.03	.03	.044	.03	.03	.03
37	.03	.03	.03	.03	.092	.03	.1	.07
38	.02	.03	.03	.03	.082	.03	.1	.07
39	.02	.03	.03	.03	.076	.03	.1	.07
40	.03	.03	.03	.03	.089	.03	.1	.07
41	.08	.03	.03	.07	.10	.03	.03	.03
42	.04	.03	.03	.03	----------	----------	----------	----------
43	.03	.03	.01	.03	.041	.03	.03	.03
44	.05	.03	.03	.03	.16	.03	.1	.07
45	.03	.03	.03	.03	.16	.3	.1	.15
46	.06	.03	.03	.07	.084	.03	.03	.07
L.S.D.		.151	.171	.132	----------	.311	.165	.161
Interval in terms of L.S.D.		6.6	2.9	2.5	----------	3.2	3.0	2.1
G.D.		1.42	1.48	1.36	----------	2.05	1.46	1.45

[1] D. L. Skinner, analyst.
[2] Claude Huffman, Jr., analyst.
[3] W. D. Goss, anlayst.
[4] D. L. Skinner and E. C. Mallory, analysts.
[5] Claude Huffman, Jr., and R. F. DuFour, analysts.
[6] C. E. Thompson and H. M. Nakagawa, analysts.
[7] H. H. Lipp, J. S. Wahlberg, and J. P. Schuch, analysts.
[8] C. E. Thompson, analyst.
J. S. Wahlberg, analyst.

Table 5.—*List of 26 National Bureau of Standards standard samples and 7 miscellaneous samples analyzed by the author*

Sample	*Material*
NBS 1a	Argillaceous limestone.
26	Iron ore, Crescent.
27b	Iron ore, Sibley.
28a	Iron ore, Norrie.
29a	Iron ore, magnetite.
56b	Phosphate rock, Tennessee brown.
76	Burned refractory (40 percent Al_2O_3).
77	Burned refractory (60 percent Al_2O_3).
78	Burned refractory (70 percent Al_2O_3).
79	Fluorspar.
80	Glass.
81	Glass sand.
88	Dolomite.
89	Lead-barium glass.
91	Opal glass.
93	Borosilicate glass.
97	Flint clay.
98	Plastic clay.
99	Soda-feldspar.
102	Silica brick.
103	Chrome refractory.
104	Burned magnesite.
112	Silica carbide.
120	Phosphate rock, Florida land pebble.
128	Glass (soda-lime).
165	Glass sand.
733 B	Enstatite.
734 B	Diopside.
755 B	Olivine.
Soil #3	Soil.
Soil #7	Soil.
G-1	Westerly granite.
W-1	Centerville diabase.

TABLE 6.—*Results of semiquantitative spectrochemical analysis of 26 National Bureau of Standards standard samples and 7 miscellaneous samples*

Aluminum and calcium

Sample	Aluminum (in percent)				Calcium (in percent)			
	Accepted value	Spectrochemical			Accepted value	Spectrochemical		
		Whole-order	½-order	⅓-order		Whole-order	½-order	⅓-order
NBS 1a	2.21	3	3	3	----	----	----	----
26	.54	.3	.1	.7	1.83	3	3	3
27b	.31	.3	.3	.3	----	----	----	----
28a	----	----	----	----	----	----	----	----
29a	.24	.3	.3	.3	.069	.03	.03	.03
56b	----	----	----	----	----	----	----	----
76	----	----	----	----	.19	.3	.1	.15
77	----	----	----	----	.186	.3	.1	.15
78	----	----	----	----	.27	.3	.3	.3
79	.01	.03	.03	.03	----	----	----	----
80	.17	.3	.1	.15	3.32	3	3	3
81	.14	.03	.1	.07	.021	.03	.03	.03
88	.035	.03	.03	.03	----	----	----	----
89	.095	.03	.1	.07	.15	.3	.1	.15
91	3.18	3	3	3	7.49	3	10	7
93	1.03	3	1	1.5	----	----	----	----
97	----	----	----	----	.07	.03	.03	.03
98	----	----	----	----	.15	.3	.1	.15
99	----	----	----	----	.257	.3	.1	.3
102	1.04	3	1	1.5	1.64	3	3	3
103	----	----	----	----	.56	.3	1	.3
104	.44	.3	.1	.15	2.39	.3	1	.7
112	.23	.3	.1	.15	.03	.03	.1	.07
120	.46	.3	1	.7	----	----	----	----
128	1.00	3	1	1.5	3.40	3	3	1.5
165	.087	.03	.1	.03	----	----	----	----
733 B	----	----	----	----	----	----	----	----
734 B	----	----	----	----	----	----	----	----
755 B	----	----	----	----	----	----	----	----
Soil #3	----	----	----	----	----	----	----	----
Soil #7	----	----	----	----	----	----	----	----
G1	----	----	----	----	1.00	.3	1	.7
W1	----	----	----	----	7.86	3	10	7
L.S.D.		.246	.180	.152	----	.236	.185	.170
Interval in terms of L.S.D.		4.1	2.8	2.2	----	4.2	2.7	2.0
G.D.		1.76	1.51	1.42	----	1.72	1.53	1.48

TABLE 6.—*Results of semiquantitative spectrochemical analysis of 26 National Bureau of Standards standard samples and 7 miscellaneous samples*—Continued

Chromium and iron

Sample	Chromium (in percent)				Iron (in percent)			
	Accepted value	Spectrochemical			Accepted value	Spectrochemical		
		Whole-order	½-order	⅓-order		Whole-order	½-order	⅓-order
NBS 1a					1.14	0.3	1	0.7
26								
27b	0.001	0.003	0.003	0.0015				
28a								
29a	.0014	.003	.01	.007				
56b								
76					1.66	3	3	1.5
77					.63	.3	1	.7
78					.55	.3	1	.7
79					.10	.3	.1	.15
80					.045	.03	.03	.03
81					.05	.03	.03	.03
88					.059	.03	.03	.03
89					.034	.03	.03	.015
91					.057	.03	.03	.03
93					.053	.03	.03	.03
97	.054	.03	.03	.03	.68	.3	.3	.7
98	.014	.03	.01	.015	1.43	3	1	1.5
99					.047	.03	.03	.03
102					.46	.3	.3	.3
103								
104	.018	.03	.03	.03	4.94	3	10	7
112					.45	.3	.3	.3
120	.007	.03	.01	.015	.62	.3	.3	.7
128					.027	.03	.03	.015
165					.013	.003	.01	.007
733 B	.35	.3	.3	.3				
734 B	.47	.3	.3	.3				
755 B	.014	.03	.01	.015				
Soil #3								
Soil #7								
G1	.002	.003	.003	.003	1.33	3	1	1.5
W1	.013	.03	.01	.015	7.69	3	3	7
L.S.D		.250	.237	.188		.220	.152	.128
Interval in terms of L.S.D		4.0	2.1	1.8		4.5	3.3	2.6
G.D		1.78	1.73	1.54		1.66	1.42	1.34

TABLE 6.—*Results of semiquantitative spectrochemical analysis of 26 National Bureau of Standards standard samples and 7 miscellaneous samples*—Continued

Potassium and magnesium

Sample	Potassium (in percent)				Magnesium (in percent)			
	Accepted value	Spectrochemical			Accepted value	Spectrochemical		
		Whole-order	½-order	⅓-order		Whole-order	½-order	⅓-order
NBS 1a	0.59	3	1	1.5	1.32	3	1	1.5
26					1.97	3	3	1.5
27b								
28a								
29a					.057	.03	.1	.07
56b								
76	1.14	3	1	1.5	.35	.3	.3	.7
77	1.75	3	1	1.5	.30	.3	.3	.3
78	2.35	3	1	3	.31	.3	.3	.7
79					.078	.03	.01	.015
80					1.95	.3	1	.7
81					.0096	.003	.003	.007
88					12.96	3	10	7
89	6.97	3	3	7	.018	.03	.01	.015
91	2.70	3	3	3	.005	.03	.01	.015
93					.016	.03	.01	.015
97					.16	.3	.1	.15
98	2.63	3	3	3	.43	.3	.3	.3
99	.34	.3	.3	.7	.032	.03	.03	.03
102					.13	.3	.1	.15
103					9.81	3	10	7
104								
112								
120					.08	.3	.1	.15
128	.82	3	1	1.5	2.01	3	1	1.5
165					.0018	.003	.003	.003
733 B								
734 B								
755 B								
Soil #3								
Soil #7								
G1	4.56	3	3	7	.235	.3	.3	.3
W1	.564	3	1	1.5	3.98	3	3	3
L.S.D.		.292	.147	.161		.272	.192	.181
Interval in terms of L.S.D.		3.4	3.4	2.1		3.6	2.6	1.8
G.D.		1.96	1.40	1.45		1.87	1.56	1.52

TABLE 6.—*Results of semiquantitative spectrochemical analysis of 26 National Bureau of Standards standard samples and 7 miscellaneous samples*—Continued

Manganese and sodium

Sample	Manganese (in percent)				Sodium (in percent)			
	Accepted value	Spectrochemical			Accepted value	Spectrochemical		
		Whole-order	½-order	⅓-order		Whole-order	½-order	⅓-order
NBS 1a	0.029	0.03	0.03	0.03	0.29	0.3	0.3	0.7
26	---------	---------	---------	---------	---------	---------	---------	---------
27b	---------	---------	---------	---------	---------	---------	---------	---------
28a	.435	.3	.3	.3	---------	---------	---------	---------
29a	.023	.03	.03	.03	---------	---------	---------	---------
56b	---------	---------	---------	---------	---------	---------	---------	---------
76	.015	.03	.01	.015	.28	.3	.1	.3
77	.0054	.003	.001	.0015	.64	.03	.03	.07
78	.004	.003	.001	.0015	.39	.3	.1	.15
79	.0023	.003	.003	.003	---------	---------	---------	---------
80	.0023	.003	.003	.003	---------	---------	---------	---------
81	---------	---------	---------	---------	---------	---------	---------	---------
88	.0046	.003	.003	.003	.06	.03	.03	.07
89	.068	.03	.1	.07	4.23	3	3	3
91	.006	.003	.003	.007	6.29	3	10	7
93	---------	---------	---------	---------	3.09	3	3	3
97	.0015	.003	.003	.003	.089	.3	.1	.15
98	.004	.003	003	.007	.21	.3	.3	.7
99	---------	---------	---------	---------	7.96	3	10	7
102	---------	---------	---------	---------	.04	.03	.03	.03
103	.16	.03	.1	.07	---------	---------	---------	---------
104	.33	.3	.3	.3	---------	---------	---------	---------
112	---------	---------	---------	---------	---------	---------	---------	---------
120	.026	.03	.03	.03	.10	.3	.1	.15
128	---------	---------	---------	---------	---------	---------	---------	---------
165	---------	---------	---------	---------	---------	---------	---------	---------
733 B	.14	.3	.1	.15	---------	---------	---------	---------
734 B	.085	.03	.1	.07	---------	---------	---------	---------
755 B	.12	.03	.1	.07	---------	---------	---------	---------
Soil #3	---------	---------	---------	---------	---------	---------	---------	---------
Soil #7	---------	---------	---------	---------	---------	---------	---------	---------
G1	.019	.03	.01	.015	2.45	3	3	3
W1	.15	.3	.1	.15	1.56	3	1	1.5
L.S.D		.210	.185	.154	---------	.304	.289	.235
Interval in multiples of L.S.D		4.8	2.7	2.2	---------	3.3	1.7	1.4
G.D		1.62	1.53	1.43	---------	2.01	1.94	1.72

TABLE 6.—*Results of semiquantitative spectrochemical analysis of 26 National Bureau of Standards standard samples and 7 miscellaneous samples*—Continued

Silicon and titanium

Sample	Silicon (in percent)				Titanium (in percent)			
	Accepted value	Spectrochemical			Accepted value	Spectrochemical		
		Whole-order	½-order	⅓-order		Whole-order	½-order	⅓-order
NBS 1a	6.59	3	3	3	0.096	0.03	0.1	0.07
26	2.35	3	1	1.5	.042	.03	.03	.03
27b	.61	.3	.3	.7	.014	.03	.01	.015
28a	---	---	---	---	---	---	---	---
29a	1.34	.3	1	.7	.09	.03	.1	.07
56b	4.72	3	3	3	---	---	---	---
76	---	---	---	---	1.32	.3	1	.7
77	---	---	---	---	1.76	3	1	1.5
78	---	---	---	---	2.02	3	1	1.5
79	.88	.3	1	.7	.0018	.003	.003	.003
80	---	---	---	---	.012	.03	.01	.015
81	---	---	---	---	.057	.03	.1	.07
88	.145	.03	.03	.03	---	---	---	---
89	---	---	---	---	.006	.03	.01	.015
91	---	---	---	---	.011	.003	.01	.007
93	---	---	---	---	.016	.03	.01	.015
97	---	---	---	---	1.43	.3	.3	.7
98	---	---	---	---	.86	.3	.3	.7
99	---	---	---	---	.010	.003	.01	.007
102	---	---	---	---	.096	.3	.1	.15
103	3.85	3	3	3	.56	.3	1	.7
104	1.19	.3	.3	.7	.018	.003	.003	.003
112	---	---	---	---	.025	.03	.03	.03
120	3.5	3	3	3	.04	.03	.03	.03
128	---	---	---	---	.010	.003	.003	.007
165	---	---	---	---	.018	.03	.01	.015
733 B	---	---	---	---	---	---	---	---
734 B	---	---	---	---	---	---	---	---
755 B	---	---	---	---	---	---	---	---
Soil #3	---	---	---	---	---	---	---	---
Soil #7	---	---	---	---	---	---	---	---
G1	---	---	---	---	.156	.3	.1	.15
W1	---	---	---	---	.659	.3	.3	.3
L.S.D.		.283	.240	.198	---	.302	.208	.165
Interval in multiples of L.S.D.		3.5	2.1	1.7	---	3.3	2.4	2.0
G.D.		1.92	1.74	1.58	---	2.00	1.61	1.46

TABLE 6.—*Results of semiquantitative spectrochemical analysis of 26 National Bureau of Standards standard samples and 7 miscellaneous samples*—Continued

Vanadium and zirconium

Sample	Vanadium (in percent)				Zirconium (in percent)			
	Accepted value	Spectrochemical			Accepted value	Spectrochemical		
		Whole-order	½-order	⅓-order		Whole-order	½-order	⅓-order
NBS 1a	----------	----------	----------	----------	----------	----------	----------	----------
26	----------	----------	----------	----------	----------	----------	----------	----------
27b	0.004	0.003	0.01	0.007	----------	----------	----------	----------
28a	----------	----------	----------	----------	----------	----------	----------	----------
29a	.0014	.003	.003	.003	----------	----------	----------	----------
56b	----------	----------	----------	----------	----------	----------	----------	----------
76	.012	.03	.03	.03	0.05	0.03	0.03	0.07
77	.018	.03	.03	.03	.067	.03	.03	.07
78	.026	.03	.1	.07	.089	.03	.03	.03
79	----------	----------	----------	----------	----------	----------	----------	----------
80	----------	----------	----------	----------	.0022	.003	.003	.003
81	----------	----------	----------	----------	.023	.03	.01	.03
88	----------	----------	----------	----------	----------	----------	----------	----------
89	----------	----------	----------	----------	.0037	.003	.003	.003
91	----------	----------	----------	----------	.007	.003	.003	.007
93	----------	----------	----------	----------	.0096	.03	.01	.015
97	.022	.03	.03	.015	.185	.03	.03	.07
98	.014	.03	.03	.015	.030	.03	.01	.015
99	----------	----------	----------	----------	----------	----------	----------	----------
102	----------	----------	----------	----------	.016	.03	.01	.03
103	----------	----------	----------	----------	.05	.003	.01	.007
104	----------	----------	----------	----------	----------	----------	----------	----------
112	----------	----------	----------	----------	.027	.03	.03	.03
120	.0084	.003	.01	.007	----------	----------	----------	----------
128	----------	----------	----------	----------	----------	----------	----------	----------
165	----------	----------	----------	----------	.003	.003	.003	.003
733 B	.005	.003	.01	.007	----------	----------	----------	----------
734 B	.0085	.03	.01	.015	----------	----------	----------	----------
755 B	----------	----------	----------	----------	----------	----------	----------	----------
Soil #3	----------	----------	----------	----------	----------	----------	----------	----------
Soil #7	----------	----------	----------	----------	----------	----------	----------	----------
G1	.0018	.003	.003	.003	.02	.03	.03	.03
W1	.024	.03	.03	.03	.009	.003	.01	.007
L.S.D.		.217	.221	.178	----------	.313	.252	.217
Internal in terms of L.S.D.		4.6	2.3	1.9	----------	3.2	2.0	1.5
G.D.		1.65	1.66	1.51	----------	2.06	1.79	1.65

TABLE 6.—*Results of semiquantitative spectrochemical analysis of 26 National Bureau of Standards standard samples and 7 miscellaneous samples*—Continued

Miscellaneous minor elements (in percent)

Sample	Element	Accepted value	Spectrochemical		
			Whole-order	½-order	⅓-order
NBS 80	As	0.07	0.3	0.03	0.15
89	As	.26	.3	.3	.7
91	As	.135	.3	.3	.3
93	As	.155	.3	.3	.7
128	B	.47	.3	.3	.3
79	Ba	.06	.03	.03	.03
89	Ba	1.25	3	1	1.5
97	Ba	.013	.003	.01	.007
98	Ba	.05	.03	.03	.015
99	Ba	.009	.003	.01	.007
G1	Ba	.14	.03	.1	.07
W1	Ba	.032	.03	.01	.015
733 B	Co	.0087	.003	.003	.007
734 B	Co	.0036	.003	.003	.003
735 B	Co	.017	.03	.01	.015
W1	Co	.0036	.003	.003	.003
NBS 97	Cu	.0024	.003	.003	.003
98	Cu	.007	.003	.003	.007
120	Cu	.0008	.003	.001	.0015
Soil #3	Cu	.035	.03	.03	.03
Soil #7	Cu	.013	.03	.01	.015
G1	Cu	.0011	.003	.001	.0015
W1	Cu	.011	.03	.01	.015
NBS 120	Mo	.0013	.003	.003	.0015
120	Ni	.0016	.003	.003	.003
733 B	Ni	.05	.03	.03	.03
734 B	Ni	.025	.03	.01	.015
755 B	Ni	.25	.3	.3	.15
W1	Ni	.009	.003	.01	.007
NBS 79	Pb	.23	.3	.3	.3
91	Pb	.090	.3	.1	.15
Soil #3	Pb	.13	.03	.03	.07
Soil #7	Pb	.016	.03	.03	.015
NBS 1a	Sr	.10	.3	.3	.15
G1	Sr	.019	.03	.03	.03
W1	Sr	.020	.03	.03	.03
NBS 79	Zn	.35	.3	.3	.3
91	Zn	.064	.03	.03	.07
Soil #3	Zn	.036	.03	.03	.03
Soil #7	Zn	.039	.03	.03	.03
L.S.D			.251	.189	.166
Interval in multiples of L.S.D			4.0	2.6	2.3
G.D			1.78	1.54	1.47

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