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A SEMIQUANTITATIVE SPECTROGRAPHIC METHOD FOR THE ANALYSIS OF MINERALS, ROCKS, AND ORES (II)

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CHEMISTRY

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A SEMIQUANTITATIVE SPECTROGRAPHIC METHOD FOR THE ANALYSIS OF MINERALS, ROCKS, AND ORES (II)*

by

C. L. Waring and C. A. Annell

ABSTRACT

The scope of the semiquantitative spectrographic method for the analysis of minerals, rocks, and ores previously described as determining 55 elements, has now been increased to 68 elements which can be estimated in one exposure of a 10-mg sample. Fluorine, the 69th element, requires a separate exposure for some materials. The method has been used to complete about 185,000 determinations in the past two years. Listed in this report are 336 chemical check analyses that indicate approximately 8 percent disagreements in the magnitude of one 10 percent bracket. No chemical and spectrographic results differ by a factor of more than 10.

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*This report concerns work done on behalf of the Division of Raw Materials of the U. S. Atomic Energy Commission.

INTRODUCTION

In the program of investigating radioactive minerals, rocks, and ores conducted by the Geological Survey on behalf of the Atomic Energy Commission, it is often desirable to know the trace-elements content and the major constituents of a very large number of samples in a limited time.

Trace Elements Investigations Report 143, "A semiquantitative spectrographic method for the analysis of minerals, rocks, and ores," by C. L. Waring and C. A. Annell, presents a procedure for the rapid analysis of solid samples of varied constituents. These investigations are being continued in order to afford reliable basic data leading to better analytical methods applicable to these diversified materials.

The purpose of this paper is to describe advances in the semiquantitative method which have resulted in increasing the number of elements determined from 55 to 69 (see tables 1 and 2) and to compare chemical and spectrographic analytical data on a large number of samples.

The method has been applied to the following materials:

Minerals

Allanite	Galena	Samarskite
Apatite	Garnet	Scheelite
Auerlite	Hewettite	Schroeckingerite
Bastnaesite	Huebnerite	Sphalerite
Betafite	Hummerite	Sphene
Bostonite	Idocrase	Thorite
Brannerite	Magnetite	Torbernite
Carnotite	Martite	Uraninite
Chalcopyrite	Melilite	Uranothorite
Corvusite	Microlite	Uranophane
Cyrtolite	Monazite	Vanoxite
Davidite	Montroseite	Volborthite
Euxenite	Perovskite	Zippeite
Feldspars	Pitchblende	Zircon
Fergusonite	Pyrite	

Rocks

Clays
Coal ash
Granite
Leach products
Lignite ash
Limestone
Pegmatites
Phosphate rocks
Sandstones
Shale
Sulfur ores

Miscellaneous

Arsenic trioxide
Bone fragments
Cadmium furnace residue
Carnotite ore mill pulps
Cottrell dust
Various precipitates
Nickel alloys
Phosphoric acid residues
Sea-water residues
Slags
Tap- and mine-water residues

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DISCUSSION

No significant changes, except the addition of elements, have been introduced into the method since the original description.

The estimation of fluorine requires a separate exposure on samples of low calcium content. Two milligrams of calcium as calcium chloride solution are added to the electrodes and dried prior to loading the sample. The addition of calcium aids in the formation of the calcium fluoride molecule which produces the molecular band effect recorded by the photographic emulsion.

In analyzing the minerals euxenite and samarskite a 2-minute arcing time instead of the customary 1-minute period gives higher accuracy for such elements as Cb, Ta, Er, and U. These and other elements or their

compounds having high boiling points constitue over 50 percent of the weight of the two minerals and therefore require a longer arcing time to vaporize into the arc stream.

The time required for an analysis varies with the type of material under test and the skill of the analyst. A trained person can complete the analysis of 14 samples (300 elements) of phosphate rock in 16 hours. However, this rate of speed is not recommended as a daily practice as eye fatigue may result. Samples producing more complicated spectra require additional time. Analysis time on 14 samples is broken down approximately as follows:

Quarter and weigh samples and proper references	4 hr
Exposure	1½ hr
Development	45 min
Plate interpretation	10 hr

The ease or difficulty with which a spectrum can be analyzed will depend upon the number, kind, and quantity of elements present in the sample. Samples containing relatively large amounts of the transition elements often give very complex spectra with heavy background. For some materials this results in serious interference with good analysis lines of the less abundant elements and necessitates referring to less sensitive lines. This procedure may involve similar scrutiny for a large number of elements and thus prolong the analysis.

Additional testing will be necessary to explain the following observed effects:

1.0 percent Ni in calcium matrices seems to be enhanced.

10.0 percent Ni in calcium phosphate matrices seems to be depressed.

1.0 percent Ni in feldspar matrices seems to be depressed, and slightly enhanced at less than 1.0 percent.

In schroeckingerite Ca and U seem to be depressed.

RESULTS AND TABLES

The method has been used to complete about 185,000 determinations during the past two years. Comparisons of chemical and spectrographic results for 336 determinations are shown in tables 3 - 13. The disagreements are approximately 8 percent in the magnitude of one bracket. Approximately 4 percent of the disagreements are regarded as borderline cases because of doubt as to which of two adjacent brackets an element belonged.

Noted in the chemical analyses were several disagreements especially for the following elements: Pb, Mn, Mg, V, Al, Fe, Na, Zr, and Ca. Standard plates for these elements have been remade and better agreement with the chemical analyses has been observed.

Table 1.--Standard sensitivities for the elements determined by the semiquantitative method.1/

Note: It is possible to detect some elements below the values listed, as the standard reference plates were prepared on the basis of 10 percent increments.

	Percent		Percent
Ag	0.0001	Mo	0.001
Al	0.0001	Mn	0.001
As	0.1	Na	0.001 2/ (0.1)
Au	0.01	Nd	0.01
B	0.001	Ni	0.01
Ba	0.0001	Os	0.1
Be	0.0001	P	0.1
Bi	0.001	Pb	0.01
Ca	0.001	Pd	0.01
Cb	0.01	Pr	0.01
Cd	0.01	Pt	0.01
Ce	0.1	Rb	0.01 2/ (10.0)
Co	0.01	Re	0.1
Cr	0.001	Rh	0.01
Cs	0.1 2/ (1.0)	Ru	0.01
Cu	0.0001	Sb	0.01
Dy	0.01	Sc	0.001
Eu	0.01	Si	0.0001
Er	0.01	Sm	0.1
F	0.1 3/	Sn	0.01
Fe	0.001	Sr	0.01
Ga	0.01	Ta	0.1
Gd	0.01	Tb	0.01
Ge	0.001	Te	0.1
Hf	0.1	Th	0.1
Hg	0.1	Ti	0.001
Ho	0.01	Tl	0.1
In	0.001	Tm	0.01
Ir	0.1	U	0.1
K	0.01 2/ (1.0)	V	0.01
La	0.01	W	0.1
Li	0.0001 2/ (0.1)	Y	0.001
Lu	0.01	Yb	0.0001
Mg	0.0001	Zn	0.01
		Zr	0.001

1/ Better sensitivity for many of these elements may be obtained by special methods.

2/ A second exposure is required for the high sensitivity listed.

3/ A third exposure is required for the fluorine estimation.

Table 2.--Arc lines used in the semiquantitative method

Element	Wave lengths (in A)	Element	Wave lengths (in A)	Element	Wave lengths (in A)
Ag	3382.9	Cd	3466.2	Eu	2727.8
	3280.7		3261.1		2813.1
			2763.9		2814.0
Al	3092.7		2288.0		2816.2
	3082.2				2906.7
	3059.9	Ce	4222.6		4435.6
	2660.4		4186.6		
	2652.5		4040.7	F (CaF band)	5291.0
	2575.1		4012.4		6036.9
	2568.0				6064.4
		Co	3465.8		
As	2780.2		3453.5	Fe	3100.31
	2349.84		3449.2		3099.97
	2288.12		3405.1		3099.9
			3283.5		3020.65
Au	2352.7		3243.8		2599.4
	2428.0				2598.38
	2676.0	Cr	4289.7		
	3122.8		4274.8	Ga	2944.2
			4254.3		2874.2
B	2497.8		2780.7		
	2496.7		2769.9	Gd	3671.2
			2731.9		3646.2
Ba	5535.55				3358.6
	4554.04	Cs	8521.1		3082.0
	3071.6		4593.0		
			4555.5	Ge	3039.1
Be	3321.3		3347.4		2691.4
	2348.6		3247.5		2651.2
Bi	3067.7	Cu	3274.0	Hf	3134.7
	2897.9		3247.6		3072.8
			2824.4		
Ca	4456.6		2492.2	Hg	4358.3
	4226.7		2293.9		3650.2
	3179.3				3125.6
	3158.9	Dy	3645.42		2536.5
			3454.33		
Cb	3358.4		3407.8	Ho	3399.0
	3094.2		3393.58		3416.5
	2875.5				3453.1
		Er	4419.6		3456.0
			3499.1		4254.4
			3372.8		

Table 2.--Continued

Element	Wave lengths (in A)	Element	Wave lengths (in A)	Element	Wave lengths (in A)
In	4511.3	Mo	4251.9	Pt	4442.6
	3256.09		3194.0		3064.7
	2710.3		3170.4		3042.6
			2816.1		2659.4
Ir	2664.8				
	2849.7	Na	5895.9	Rb	4215.6
	2924.8		5890.0		4201.8
	3220.8		3302.9		3350.9
			3302.3		7800.2
K	7698.9				7947.6
	7664.9	Nd	4325.8		
	4047.2		4303.6	Re	3460.5
	4044.1		3328.3		3464.7
	3447.7				
	3446.4	Ni	3492.9	Rh	3280.5
			3433.6		3283.6
La	4429.9		3414.8		3396.9
	4333.8		3002.5		3434.9
	3380.9		2320.1		4374.8
	3337.5				
		Os	2909.1	Ru	2810.0
Li	6707.9		3058.7		2810.6
	3232.7		3301.6		3428.3
	2741.3				3436.7
		P	2554.9		4297.7
Lu	2613.4		2553.3		
	2615.4		2535.7	Sb	3267.5
	2619.3		2534.0		2877.9
	3911.4				2598.1
	3198.1	Pb	2873.3		2528.5
			2833.1		2311.5
Mg	4351.9		2663.2		
	2852.1		2614.2	Sc	3911.8
	2795.5				3907.5
	2779.9	Pd	2763.1		3369.0
	2776.7		3114.0		3019.3
			3242.7		2552.4
Mn	2798.3		3421.2		
	2605.7		3634.7	Si	2987.7
	2593.7				2881.6
	2576.1	Pr	4241.0		2528.5
			4225.3		2524.1
			4206.7		2516.1
					2435.2

Table 2.--Continued

Element	Wave lengths (in A)	Element	Wave lengths (in A)	Element	Wave lengths (in A)
Sm	4424.4	Ti	3372.8	V	4379.2
	4256.4		3242.0		3185.4
			3239.0		3184.0
Sn	3262.3		3234.5		3183.4
	2863.3		3224.2		
	2839.9			W	4302.1
		Tl	3775.7		4294.6
Sr	4607.3		3529.4		3049.7
	3464.5		3519.2		
	3351.3		2767.9	Y	3242.3
	2569.5		2379.6		3195.6
Ta	4574.3	Tm	3131.3	Yb	2653.7
	3642.1		3133.9		2891.4
	3311.2		3362.6		3107.9
			3462.2		3289.4
Tb	3293.1		4242.2		3988.0
	3324.4				
	4278.5	U	4287.9	Zn	4680.1
	4318.9		4241.7		3345.0
			3566.6		3302.6
Te	2383.3		2837.328		3282.3
	2385.8		2837.187		
				Zr	3556.6
Th	4619.5				3438.2
	4019.1				3391.9
	2837.3				

Table 3.--Comparison of chemical and spectrographic analyses for phosphorus of various phosphate rocks (chemical results as oxides converted to element).

Sample no.1/	Percent P chemical	Percent P spectrographic
128	2.3	1.0 - 10.
129	13.8	10.+
130	15.6	10.+
131	3.8	1.0 - 10.
132	13.7	10.+
133	14.9	10.+
134	1.9	1.0 - 10.
135	9.9	10.+
27B-1	0.87	0.1 - 1.0
27B-2A	2.7	1.0 - 10.
27B-2B	2.3	1.0 - 10.
27B-2C	6.3	1.0 - 10.
27B-3	8.	1.0 - 10.
27B-4	8.3	1.0 - 10.
27B-5A	6.2	1.0 - 10.
27B-5B	6.0	1.0 - 10.
PC-1	5.2	1.0 - 10.
PC-2	6.1	1.0 - 10.
PC-3	3.7	1.0 - 10.
PC-4A	0.24	0.1 - 1.0
PC-4B	6.1	1.0 - 10.
PC-5A	5.4	1.0 - 10.
PC-5B	4.8	1.0 - 10.
PC-6	1.9	1.0 - 10.

Several materials comprise "Florida phosphates." The typical ore is a clastic phosphorite with admixed quartz and clay. It has a high content of Ca and P. Above the ore in the deposit is a "leached zone," consisting mainly of quartz but with some aluminous phosphate and only minor amounts of calcium.

1/ Lot 712, TWS 345, TWC 1376
Lot 850, TWS 346, TWC 1378
Lot 851, TWS 347, TWC 1379

Table 3.--Continued.

	Sample no. 2/	Percent P chemical	Percent P spectrographic
Fossil manatee bones from the leached zone of the Bone Valley formation, Hillsborough County, Florida, which consist mainly of apatite.	74-M3	16.0	10.+
	M4	16.0	10.+
	M5	16.2	10.+
	M6	15.8	10.+
	M7	16.4	10.+
	M8	16.0	10.+
	M9	16.1	10.+
	M10	16.7	10.+
	M11	16.0	10.+
	M12	16.0	10.+
	N-1	16.0	10.+
	N-2	16.5	10.+
	N-13	10.4	1.0 - 10.
Samples from caves on Mona Island, West Indies, varying from red and white limestone to almost pure earthy phosphorite (hydroxylapatite).	M-1-A 3/	0.23	0.1 - 1.0
	M-1-B	0.07	Not detected 4/
	M-1-C	0.07	Not detected
	M-2	0.17	Not detected
	M-3	18.22	10.+
	M-4	14.26	10.+
	M-5	17.51	10.+
	M-6	11.57	10.+
	M-7-A	16.32	10.+
	M-7-B	6.95	1.0 - 10.0
	M-8	16.72	10.+

2/ Lot 669, TWS 173, Lot 0, TWS 238

3/ Lot 535, TWS 298

4/ Detection limit of P is approximately 0.1 percent.

Table 4.--Comparison of chemical and spectrographic percentage analyses of phosphate rock from Florida with chemical results as oxides converted to elements. (For description of Florida phosphates, see first part of table 3.)

Element	No. 1 1/		No. 2		No. 3		No. 4		No. 5	
	chem.	spec.	chem.	spec.	chem.	spec.	chem.	spec.	chem.	spec.
P	11.3	10.+	12.6	10.+	12.7	10.+	15.8	10.+	15.5	10.+
Ca	19.6	10.+	18.3	10.+	17.7	10.+	19.4	10.+	19.8	10.+
Fe	1.26	0.1-1.0	1.19	0.1-1.0	0.46	0.1-1.0	0.87	0.1-1.0	1.25	0.1-1.0
Al	0.32	0.1-1.0	0.44	0.1-1.0	0.66	0.1-1.0	0.33	0.1-1.0	0.07	0.1-1.0
Si	4.1	1.0-10.	3.9	1.0-10.	7.6	1.0-10.	4.0	1.0-10.	2.9	1.0-10.
Na	0.75	0.1-1.0	0.82	0.1-1.0	0.39	0.1-1.0	0.22	0.1-1.0	0.16	0.1-1.0
K	0.25	0.1-1.0	0.28	0.1-1.0	0.025	0.01-0.1	0.033	0.01-0.1	0.016	0.01-0.1
Mg	0.63	1.0-10.0	0.69	1.0-10.0	0.09	0.01-0.1	0.16	0.1-1.0	0.14	0.1-1.0
Mn	0.023	0.01-0.1	0.023	0.01-0.1	0.023	0.01-0.1	0.031	0.01-0.1	0.108	0.01-0.1
V	0.006	0.01-0.1	0.006	0.01-0.1	0.0056	0.01-0.1	0.011	0.01-0.1	0.014	0.01-0.1
Ti	0.036	0.01-0.1	0.012	0.01-0.1	0.006	0.01-0.1	0.018	0.01-0.1	0.036	0.01-0.1
Cr	0.00	0.001-.01	0.00	0.001-.01	0.00	0.001-.01	0.00	0.001-.01	0.00	0.001-.01

1/ Lot 524, TWS 61, TWC 908

Table 4.--Continued.

Element	BP-1 1/		BP-2		BP-3		BP-5		BP-6		BP-7		BP-8	
	chem.	spec.	chem.	spec.	chem.	spec.	chem.	spec.	chem.	spec.	chem.	spec.	chem.	spec.
Si	25.4	10.+	18.6	10.+	23.8	10.+	28.8	10.+	36.8	10.+	38.2	10.+	38.8	10.+
P	4.8	1.0-10.0	5.7	1.0-10.0	5.1	1.0-10.0	3.1	1.0-10.0	1.1	1.0-10.0	1.4	1.0-10.0	1.5	1.0-10.0
Al	9.5	10.+	12.8	10.+	10.7	10.+	8.8	10.+	5.3	10.+	4.2	10.+	3.6	10.+
Zr	0.08	0.1-1.0	0.08	0.1-1.0	0.1	0.1-1.0	0.1	0.1-1.0	0.09	0.1-1.0	0.1	0.1-1.0	0.1	0.1-1.0
Fe	1.8	1.0-10.0	2.0	1.0-10.0	1.7	1.0-10.0	1.5	1.0-10.0	1.2	1.0-10.0	1.7	1.0-10.0	1.5	1.0-10.0
Ca	0.68	0.1-1.0	0.64	0.1-1.0	0.05	0.01-0.1	0.08	0.01-0.1	0.29	0.1-1.0	0.16	0.01-0.1	0.05	0.01-0.1
Mn	0.00	0.001-0.01	0.00	0.001-0.01	0.00	0.001-0.01	0.01	0.001-0.01	0.00	0.001-0.01	0.01	0.001-0.01	0.01	0.001-0.01
Mg	0.18	0.1-1.0	0.23	0.1-1.0	0.29	0.1-1.0	0.17	0.1-1.0	0.15	0.1-1.0	0.12	0.1-1.0	0.13	0.1-1.0
Na	0.21	0.1-1.0	0.30	0.01-0.1	0.25	0.01-0.1	0.045	0.001-0.01	0.13	0.01-0.1	0.19	0.01-0.1	0.12	0.01-0.1
Ti	0.44	0.1-1.0	0.69	0.1-1.0	0.50	0.1-1.0	0.40	0.1-1.0	0.24	0.1-1.0	0.21	0.1-1.0	0.20	0.1-1.0
Cr	0.03	0.01-0.1	0.11	0.01-0.1	0.13	0.01-0.1	0.06	0.01-0.1	0.06	0.01-0.1	0.03	0.01-0.1	0.04	0.01-0.1
V	0.01	0.001-0.01	0.01	0.001-0.01	0.00	0.001-0.01	0.01	0.001-0.01	0.00	0.001-0.01	0.00	0.001-0.01	0.00	0.001-0.01

1/ Lot 2 - 23, TWS 86, TWC 780

Table 5.--Comparison of chemical and spectrographic percentage analyses of northwest phosphates from Montana and Idaho (chemical results as oxides converted to elements). 1/

Northwest phosphates are from the Phosphoria formation mainly in Montana and Idaho. They are dark-colored pelletal phosphorites, consisting of carbonate fluorapatite, with minor admixed calcite and/or dolomite, clays, mica, and organic matter. When samples representing phosphatic facies are analyzed, the relative amounts of minor constituents will be greater.

Element	RAH-47-10		RAH-47-46		LES-47-36		VEM-47-253	
	chem.	spec	chem.	spec.	chem.	spec.	chem.	spec.
Si	7.06	1.0-10.0	8.28	1.0-10.0	2.21	1.0-10.0	4.10	1.0-10.0
Cr	0.11	0.1-1.0	0.13	0.1-1.0	0.07	0.1-1.0	0.11	0.1-1.0
V	0.09	0.1-1.0	0.04	0.01-0.1	0.01	0.01-0.1	0.02	0.01-0.1
Ti	0.09	0.01-0.1	0.11	0.01-0.1	0.02	0.01-0.1	0.04	0.01-0.1
P	11.5	1.0-10.0	12.2	10.+	15.3	10.+	10.4	1.0-10.0
Mn	0.04	0.001-0.01	0.03	0.001-0.01	0.01	0.001-0.01	0.06	0.01-0.1
Ca	26.6	10.+	29.0	10.+	35.0	10.+	32.3	10.+
Mg	0.20	0.1-1.0	0.22	0.1-1.0	0.15	0.1-1.0	0.55	0.1-1.0
Fe	0.70	1.0-10.0	0.55	0.1-1.0	0.44	1.0-10.0	0.49	0.1-1.0
Al	1.46	1.0-10.0	1.44	1.0-10.0	0.42	1.0-10.0	0.75	1.0-10.0

1/ Lot 1202, TWS 124, TWC 964; Lot 1204, TWS 125, TWC 965; Lot 1205, TWS 126, TWC 966; Lot 1206, TWS 127, TWC 967.

Table 5.--Continued.

Element	RAH-82-47 chem. spec.	RAH-87-47 chem. spec.	RAH-100-47 chem. spec.	LES-117-47 chem. spec.	LES-179-47 chem. spec.	VEM-111-47 chem. spec.
Al			2.2 1.0-10.0		0.39 0.1-1.0	0.58 1.0-10.0
Ca					15. 1.0-10.0	
Mg	8.9 10. +	1.1 1.0-10.0				
Si				4.25 1.0-10.0		

Element	VEM-485-47 chem. spec.	VEM-488-47 chem. spec.	RMC-127-47 chem. spec.	RAH-47-179 chem. spec.	VEM-519-47 chem. spec.	VEM-523-47 chem. spec.
Al	0.88 0.1-1.0 and 1.0-10.0	5.7 1.0-10.0				
Ni				0.37 1.0-10.0		
Si					8.3 1.0-10.0	9.4 10. +
V			0.43 0.1-1.0			

Lot 1134, TWS 223, TWC 1134; Lot 1209, TWS 130; Lot 1211, TWS 131, TWC 1286

Table 6.--Comparison of chemical and spectrographic analyses of a vanadium mineral (chemical results as oxides converted to elements).

Red-brown vanadium mineral associated with hummerite, Hummer mine, Jo Dandy group, Paradox Valley, Montrose County, Colo. This is probably a new mineral but it is mixed with fine-grained clay from which it cannot be separated mechanically.

Element	ADW no. 79 1/	
	Chemical percent	Spectrographic percent
Si	2.5	1.0-10.0
Fe	5.5	1.0-10.0
Mg	2.5	1.0-10.0
K	2.0	0.1-1.0
Al	0.5	0.1-1.0
Na	1.1	0.1-1.0
Sr	0.6	0.1-1.0
Ca	0.64	0.01-0.1

1/ Lot 0-20, TWS 270, TWC 1421

Table 7.--Comparison of chemical and spectrographic percentage analyses of miscellaneous samples (chemical results as oxides converted to elements).

e-nt	Montroseite 1/ chem.	spec.	Hummerite 2/ chem.	spec.	Ore 3/ chem.	spec.	Idoqrase 4/ chem.	spec.	Newettite 5/ chem.	spec.	Euxenite 6/ chem.	spec.	Pitchblende 7/ chem.	spec.
	1.59	1.0-10.0	0.10	0.1-1.0	0.054	0.1-1.0			0.07	0.1-1.0				
					0.25	0.1-1.0								
											9.1	1.0-10.0		
			0.03	0.01-0.1	3.88	1.0-10.0	24.4	10.+	4.6	1.0-10.0				
	6.8	1.0-10.0			0.007	0.01-0.1	3.3	1.0-10.0						
			5.5	1.0-10.0					0.96	0.1-1.0				
			3.6	1.0-10.0										
							0.047	0.01-0.1						
													8.4	1.0-10.0
	2.82	1.0-10.0	0.31	0.1-1.0	0.014	0.01-0.1	17.3	10.+	0.23	0.1-1.0				
					0.072	0.1-1.0								
											.82	0.1-1.0		
											30.	10.+		
					47.5	10.+								
	51.7	10.+	37.4	10.+	10.9	1.0-10.0			45.9	10.+				

/ Bitter Creek mine, Montrose County, Colorado
/ Jo Dandy mine, Montrose County, Colorado
/ Hand-picked sample from ore-bearing sandstone, May Day mine, Mesa County, Colorado
/ Olmstedville, Essex County, New York
/ Jo Dandy mine, Montrose County, Colorado
/ Euxenite sample from Kragero, Norway, U.S.N.M. R7144
/ Great Bear pitchblende, Canada

Table 8.--Comparison of chemical and spectrographic analyses of silicate rocks for zirconium (chemical results as oxides converted to elements).1/

The samples were radioactive Tertiary intrusives associated pitchblende deposits of the Central City district, Colorado.

Sample no.	Percent Zr chemical	Percent Zr spectrographic
P - 9	< 0.01	0.001 - 0.01
P - 14	0.14	0.01 - 0.1
P - 21	0.06	0.1 - 1.0
P - 23A	< 0.01	0.001 - 0.01
P - 23B	< 0.01	0.001 - 0.01
P - 27	0.05	0.01 - 0.1
P - 29	0.06	0.01 - 0.1
P - 94	0.05	0.001 - 0.01
P - 118	0.10	0.01 - 0.1

1/ Lot 0-50, TWS 336, TWC 1336

Table 9.--Comparison of chemical and spectrographic percentage analyses of ash from Dakota lignite samples (chemical results as oxides converted to elements).1/ Auger hole samples from Southwest North Dakota.

Ele-ment	C - 18b		F - 84		F - 95		S - 29		SC - 35		SC - 36	
	chem.	spec.	chem.	spec.	chem.	spec.	chem.	spec.	chem.	spec.	chem.	spec.
Si	5.35	1.0-10.0	13.1	10.+	15.	10.+	7.1	1.0-10.0	4.45	1.0-10.0	10.2	1.0-10.0
e	8.0	1.0-10.0	23.6	10.+	21.9	10.+	24.2	10.+	15.8	10.+	8.7	1.0-10.0
a	11.1	1.0-10.0	2.9	1.0-10.0	7.8	1.0-10.0	8.6	1.0-10.0	14.4	1.0-10.0	10.2	1.0-10.0
	4.4	1.0-10.0	0.126	0.1-1.0	0.126	0.1-1.0	0.138	0.1-1.0	0.072	0.1-1.0	0.108	0.1-1.0
	0.04	0.01-0.1	0.2	0.1-1.0	0.78	0.1-1.0	0.11	0.01-0.1	0.20	0.1-1.0	0.37	0.1-1.0
	6.75	1.0-10.0	10.1	10.+	11.6	10.+	9.3	1.0-10.0	7.8	1.0-10.0	12.6	10.+
i	0.06	0.01-0.1	0.36	0.1-1.0	0.36	0.1-1.0	0.06	0.01-0.1	0.06	0.01-0.1	0.06	0.01-0.1
a	17.8	1.0-10.0	2.6	1.0-10.0	1.41	1.0-10.0	3.26	1.0-10.0	2.33	1.0-10.0	2.56	1.0-10.0
	0.78	0.1-1.0	0.68	0.1-1.0	0.68	0.1-1.0	1.28	1.0-10.0	0.92	1.0-10.0	0.92	0.1-1.0
					0.118	0.1-1.0						
					0.20	0.1-1.0						
o					0.04	0.1-1.0						
r	0.45	0.1-1.0										
	0.27	0.1-1.0										
							0.15	0.1-1.0				

1/ Lot 0-99, TWS 473, TWC 1587

Table 10.--Comparison of chemical and spectrographic percentage analyses of the lead content of carnotite-bearing sandstones from various localities (chemical results as oxides reduced to elements) 1/

Sample no.	Locality	Chemical Pb	Spectrographic Pb
LRS-6-48	Mine D, Montrose County, Colo.	0.10	0.1-1.0
7	Butterfly mine, Montrose County, Colo.	0.16	0.1-1.0
21	Radium No. 5 mine, San Miguel County, Colo.	0.11	0.1-1.0
22	Raven mine, San Miguel County, Colo.	0.003	0.1-1.0
26	Calamity No. 13 mine, Mesa County, Colo.	0.013	0.01-0.1
32	Vanadous No. 1 mine, San Miguel County, Colo.	0.004	0.01-0.1
33B	Bear Creek mine, San Miguel County, Colo.	0.010	0.01-0.1
34	Primus claim, San Miguel County, Colo.	0.22	0.1-1.0
35A	Dunning-Greysill mine, San Juan County, Colo.	0.004	0.01-0.1
39A	Stone No. 1 claim, Montrose County, Colo.	0.10	0.1-1.0
43	Bitter Creek mine, Montrose County, Colo.	0.12	0.1-1.0
60	Club mine, Montrose County, Colo.	0.045	0.01-0.1
67	Wild Steer mine, Montrose County, Colo.	0.11	0.1-1.0
69	Eastside mine, Navajo Indian Reservation, Ariz.	0.011	0.01-0.1

1/ Lot 0-21, TWS 175

Table 11.--Comparison of chemical and spectrographic percentage analyses of red and gray clays from the Colorado Plateau (chemical results as oxides converted to elements).1/

Red and gray clays consisting chiefly of hydro-mica, quartz, and clacite from a zone underlying vanadium-bearing ore at Bitter Creek mine, Montrose County, Colorado.

Element	Red clay		Gray clay	
	chem.	spec.	chem.	spec.
Ca	1.7	1.0-10.0	1.8	1.0-10.0
Si	28.0	10 +	30.0	10 +
Fe	3.84	1.0-10.0	1.7	1.0-10.0
Al	5.6	1.0-10.0	4.7	1.0-10.0
Mg	2.46	1.0-10.0	2.42	1.0-10.0
Cu	1.37	1.0-10.0	1.45	1.0-10.0
Na	0.03	0.1-1.0	0.07	0.1-1.0
K	5.0	1.0-10.0	4.6	1.0-10.0
Ti	0.35	0.1-1.0	0.36	0.1-1.0
V	0.04	0.01-0.1	0.04	0.01-0.1

1/ Lot 0-20, TWS 118

Table 12.-- Comparison of chemical and spectrographic percentage analyses of Hyatt pegmatite samples, Larimer County, Colo. (chemical results as oxides reduced to elements).1/

Hyatt pegmatite samples from NE1/4NW1/4 sec. 28, T. 6 N., R. 71 W., Larimer County, Colorado. Samples from the plagioclase-perthite-quartz wall zone of the pegmatite. The purpose of analyzing the samples was to determine the presence in the wall zone of minor elements that crystallized in relative abundance in the next inner zone.

Element	B6090-A chemical	B6090-B chemical	B6090-A and B spectrographic
Si	33.8	--	10. +
Al	8.25	8.24	1.0-10.0
Na	4.35	4.28	1.0-10.0
K	3.22	3.3	0.1-1.0
Fe	0.50	0.51	0.1-1.0
Ca	0.25	0.24	0.01-0.1
Mg	0.07	0.08	0.01-0.1
V	0.02	0.02	0.01-0.1
Ti	0.006	0.006	0.001-0.01
Mn	0.031	0.031	0.01-0.1
Ba	0.045	0.045	0.01-0.1

1/ Lot 1521, TWS 316, TWC 1303

Table 13.--Comparison of chemical and spectrographic percentage analyses of modern and fossil manatee bones (chemical results as oxides converted to elements).1/

Samples M4, M8, and M12 were fossil manatee ribs from the Bone Valley formation of Florida. Samples M13 A and B were modern manatee rib. The analyses were used to compare the modern and fossil bones.

Sample no.	Mg		Sr	
	chem.	spec.	chem.	spec.
M - 4	0.2	0.1-1.0	0.17	0.01-0.1
M - 8	0.82	0.1-1.0	0.14	0.01-0.1
M - 12	0.3	0.1-1.0	0.14	0.01-0.1
M - 13A	0.6	0.1-1.0	0.13	0.01-0.1
M - 13B	0.6	0.1-1.0	0.14	0.01-0.1

1/ TWS 173, 238, 321, TWC 1256, TWC 1257

APPENDIX

Composition of standard solutions

The following standard solutions were made from compounds and elements available in the laboratory. Many of the compounds and elements used were Johnson, Matthey and Co. "Specpure" grade (J and M). The compounds were dissolved in distilled water unless otherwise noted.

Element standardized	Compound used	Solution
Ag	$AgNO_3$, reagent	
Al	$AlCl_3 \cdot 6H_2O$, C.P.	Compound dried in oven at 140 C. and dissolved in cold acidified H_2O.
As	As_2O_3, Nat. Bur. St. No. 83a	1:1 HNO_3, heated. Diluted to volume with H_2O.
Au	Au, metal, J and M	Aqua regia. Boiled down several times with HCl (conc.) to drive off HNO_3. Diluted to volume with H_2O.
B	H_3BO_3, C.P.	
Ba	$BaCl_2 \cdot 2H_2O$, C.P.	
Be	Be, metal, J and M	Dilute HCl.
Bi	Bi, metal, J and M	1:1 HNO_3, diluted to volume with H_2O.
Ca	$CaCl_2 \cdot 2H_2O$, anal. reag.	
Cb	Cb, metal, J and M	48 percent HF. Diluted to volume with HNO_3, conc.
Cd	$CdCl_2 \cdot 2\frac{1}{2}H_2O$, C. P.	

Element standardized	Compound used	Solution
Ce	CeO_2, J and M	H_2SO_4, conc., heated to form amber, $Ce(SO_4)_2$. 6 percent H_2SO_3 added to form colorless $Ce_2(SO_4)_3$. Diluted to volume with H_2O.
Co	$CoCl_2 \cdot 6H_2O$, C.P.	
Cr	Cr, metal J and M	1:1 H_2SO_4
Cs	CsCl, C.P.	
Cu	CuO, reagent	Dilute HCl.
Dy	Dy_2O_3, J and M	1:1 HCl. Diluted to volume with H_2O.
Er	Er_2O_3, J and M	1:1 HCl. Diluted to volume with H_2O.
Eu	Eu_2O_3, J and M	1:1 HCl, heated. Diluted to volume with H_2O.
F	$CaCl_2$, C.P. and NaF, C.P.	H_2O (2 solutions).
Fe	Fe, metal, J and M	Dilute H_2SO_4.
Ga	Ga, metal, C.P.	Aqua regia. Diluted to volume with H_2O.
Ge	GeO_2, C.P.	HF, 48 percent. H_2SO_4, conc., added and heated to drive off HF. Diluted to volume with H_2O.
Gd	Gd_2O_3, J and M	Dilute HCl.
Hf	HfO_2, J and M	Dilute H_2SO_4, heated and H_2O_2, 3 percent, added until dissolved. Diluted to volume with H_2O.
Hg	$HgCl_2$, reagent	
Ho	Ho_2O_3, J and M	1:1 HCl, heated. Diluted to volume with H_2O.

Element standardized	Compound used	Solution
In	In, metal, J and M	HNO_3, conc. Diluted to volume with H_2O.
Ir	Ir metal powder, C.P.	Fused with 3 parts KOH and 1 part KNO_3. Fusion dissolved in aqua regia. SiO_2 filtered off. Filtrate boiled down to small volume. Crystals of K_2IrCl_6 separate upon cooling and dissolve in H_2O.
K	$HKC_8H_4O_4$, Nat. Bur. Stand.	
La	La_2O_3, J and M	Dilute HCl.
Li	Li_2CO_3, reagent	Dilute HCl.
Lu	Lu_2O_3, J and M	1:1 HCl, heated. Diluted to volume with H_2O.
Mg	Mg, metal, J and M	Dilute HCl.
Mn	$MnCl_2 \cdot 4H_2O$, C.P.	
Mo	Mo, metal, J and M	Aqua regia, heated. Diluted to volume with H_2O.
Na	NaCl, reagent	
Nd	Nd_2O_3, J and M	1:1 HCl. Diluted to volume with H_2O.
Ni	Ni, metal, J and M	1:1 HNO_3, heated. Diluted to volume with H_2O.
Os	Os metal powder, C.P.	Os metal powder heated with aqua regia in flask fitted with reflux condenser.
P	$NaH_2PO_4 \cdot H_2O$ C.P.	
Pb	$Pb(NO_3)_2$, C.P.	
Pd	Pd, wire, J and M	Aqua regia. Diluted to volume with H_2O.

Element standardized	Compound used	Solution
Pr	Pr_6O_{11}, J and M	1:1 HCl. Diluted to volume with H_2O.
Pt	Pt, sheet	Aqua regia. Boiled down several times with HCl, conc., to drive off HNO_3. Diluted to volume with H_2O.
Rb	RbCl, J and M	
Re	Re, metal, J and M	HNO_3, conc. Diluted to volume with H_2O.
Rh	$RhCl_3$, dry, C.P.	Dilute HCl.
Ru	$(NH_4)_2RuCl_5$, J and M	Hot H_2O.
Sb	SbI_3, C.P.	Acetone + HCl, dil.
Sc	$Sc_2(SO_4)_3 \cdot 5H_2O$, J and M	
Si	SiO_2, pure	Na_2CO_3 fusion. Diluted to volume with H_2O.
Sm	Sm_2O_3, J and M	Dilute HCl.
Sn	$SnCl_2 \cdot 2H_2O$, reagent	
Sr	$SrCO_3$, reagent	Dilute HCl.
Ta	Ta, metal, J and M	48 percent HF + HNO_3, conc. Diluted to volume with H_2O.
Tb	Tb_4O_7, J and M	1:1 HCl, heated. Diluted to volume with H_2O.
Te	$H_2TeO_4 \cdot 2H_2O$, C.P.	1:6 HNO_3, heated.
Th	$Th(NO_3)_4 \cdot 4H_2O$, C.P.	
Ti	TiO_2, C.P.	48 percent HF + H_2O_2. H_2SO_4, conc., added and heated to drive off HF. Diluted to volume with H_2O.

Element standardized	Compound used	Solution
Tm	Tm_2O_3, J and M	1:1 HCl, heated. Diluted to volume with H_2O.
Tl	$TlNO_3$, C.P.	
U	$(UO_2)(C_2H_3O_2)_2 \cdot 2H_2O$ C.P.	
V	NH_4VO_3, C.P.	1:1 HCl. Diluted to volume with H_2O.
W	W, metal	48 percent HF + HNO_3, conc., heat. Diluted to volume with H_2O.
Y	Y_2O_3, J and M	1:1 HCl and heat. Diluted to volume with H_2O.
Yb	Yb_2O_3, J and M	1:1 HCl, heated. Diluted to volume with H_2O.
Zn	ZnO, reagent	Dilute HCl.
Zr	$ZrOCl_2 \cdot 8H_2O$, C.P.	