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Mr. Jesse C. Johnson, Director
Division of Raw Materials
U. S. Atomic Energy Commission
16th and Constitution Avenue, N. W.
Washington 25, D. C.

Dear Jesse:

Transmitted herewith are three copies of TEI-444, "An application of spectrographic microphotometric scanning," by C. L. Waring, Mona Frank, and A. M. Sherwood, July 1954.

We are asking Mr. Hosted to approve our plan to publish this report in Analytical Chemistry.

Sincerely yours,

Andrew Bradley

for W. H. Bradley
Chief Geologist

Chemistry

This document consists of 29 pages.
Series A.

UNITED STATES DEPARTMENT OF THE INTERIOR

GEOLOGICAL SURVEY

AN APPLICATION OF SPECTROGRAPHIC MICROPHOTOMETRIC SCANNING*

By

C. L. Waring, Mona Frank, and A. M. Sherwood

July 1954

Trace Elements Investigations Report 444

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

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(Including master)	
	79

CONTENTS

TABLES

AN APPLICATION OF SPECTROGRAPHIC MICROPHOTOMETRIC SCANNING

By

C. L. Waring, Mona Frank, and A. M. Sherwood

ABSTRACT

A previously published semiquantitative spectrographic method was modified by applying microphotometric scanning of the spectrograms in such a way that the operator was free to do other work during a large part of the scanning time. The spectrograms of five ash samples from Dakota lignite and one synthetic mixture were scanned for 68 elements; 32 elements were found and those below 10 percent were reported quantitatively.

The tests included a spectrum line study to provide lines least enhanced or depressed by matrix differences, information on accuracy, on human error, and eye fatigue.

In 93 percent of the cases the agreement with the chemical analysis was within a factor-of-10 bracket, 85 percent were within a factor-of-5 bracket, and 79 percent were within a factor-of-2 bracket.

INTRODUCTION

In a semiquantitative spectrographic method for the analysis of minerals, rocks, and ores, described by Waring and Annell (6), the procedure included the analysis of an unknown for 68 elements in one exposure of a 10-mg sample. The final results were obtained by visually comparing spectral lines of the unknown with the same lines on standard plates.

The present study is a modification of the semiquantitative method to include the application of microphotometry to the spectrograms. An increase in the accuracy and precision of the results would make the data obtained more applicable to the geochemical problems being studied. The experiments were conducted with the expectation of applying the procedure to numerous types of samples and reporting the results quantitatively or in narrow concentration ranges.

The spectrograms were scanned and recorded automatically on a commercial microphotometer. The procedure permitted the operator to do other work during a large part of the scanning time, after adjusting the sensitivity of the instrument. Working with an enlarged chart (10 X) was less fatiguing for the operator than using the magnifying glass for plate interpretation. However, because 68 elements are being determined spectrographically at the present time, photographic plates are still being used in most of our analytical work. Determining this large number of elements of rock samples which exhibit non-uniform burning characteristics is still difficult with the presently available commercial direct-reading instruments. A permanent record of the spectrograms has definite reference value in the laboratory. We plan to determine the adaptability of the method with the instruments in the laboratory before attempting the direct reading approach.

The method was applied to the ash of five Dakota lignite auger-hole samples and one synthetic mixture (table 3). As many semiquantitative analyses of the ashes of low rank coals are performed in our laboratory in connection with the geochemical studies of lignites, any improvement in the method could be readily applied to the lignite problem. The major components of the lignites, usually consisting of Fe, Si, Al, Na, and Ca,

may vary considerably from sample to sample. Selection of these samples would fit into our plan of studying numerous lines and selecting those least enhanced or depressed by matrix differences.

A survey of the literature (1,2,4,5) reveals that several similar methods are being applied in other laboratories on different materials with different standardization procedures and visual estimation of results.

EXPERIMENTAL DATA

Preparation of standards

As in the procedure described previously (6), the standard solutions were made to contain 10 mg of the element per milliliter of solution. These solutions were diluted until 0.1 ml of each equals 10, 1, 0.1, 0.01, 0.001, and 0.0001 percent based on a 10-mg sample of the unknowns. A 0.1-ml aliquot of the solution was then added from a micropipette to the sealed electrode cup. This was dried on an asbestos-covered hot plate at a temperature of approximately 100 C. A few milligrams of pure graphite were added to the remaining salts in the electrodes. Duplicate sets of electrodes were prepared, one set was arced (250 v, 12 amp, d-c) for 60 seconds and one for 120 seconds.

The spectrograms were scanned on a recording microphotometer. Data provided by the tracings, including wave length, percent transmittance and width were recorded on cards as shown in table 1. Information on numerous lines was recorded and used in the search for lines least enhanced or depressed by the different elements present. Selected lines are listed in table 2.

Procedure

A synthetic sample of known composition was analyzed with the unknowns. The synthetic sample was prepared by adding standard solutions of the minor elements to the oxides of the major components (table 3). After drying on a hot plate, the material was ignited at 500 C for four hours. The synthetic sample was ground in a boron carbide mortar to pass a 100-mesh stainless-steel screen and thoroughly mixed by passing repeatedly through an 80-mesh stainless-steel screen.

A 10-mg sample of lignite ash obtained according to the method of Fieldner and Selvig (3) was weighed, mixed thoroughly with two parts by weight of pure graphite in the weighing pan, and placed in the electrode cup through a polished stainless-steel funnel. The unknowns were arced (250 v, 12 amp, d-c) for either 60 or 120 seconds, depending on the behavior of the sample in the arc or on past experience with the type of material. Experience has shown that the error is not great if the sample is arced for 60 seconds instead of 120 seconds.

After the plates were processed, the spectrograms were scanned with a recording microphotometer. The proper lines were located on the scannings and the height and, if necessary, the width determined.

The concentrations of the elements were interpolated by direct proportion from the data on transmission versus percent concentration. Information on the previously prepared cards (table 1) obtained from the standard plates was used in the interpolations. The precision and type of data sought did not warrant the use of the more defined logarithmic function-emulsion calibration method, especially in view of the other sources of error.

As previously described (6), on each plate, along with the spectra of the unknowns, were recorded spectra of iron and of aluminum alloy (Aluminum Co. of America standard SA 874). These latter spectra furnished reference points for locating lines and a general index of exposure, of plate sensitivity and development.

The measured transmission, of the chromium line 2780.7 A in the aluminum alloy spectrum, 0.15 percent chromium is used as a rough check on the plate sensitivity and other variables. The normal percent transmittance value of the chromium line is 65 percent. When the percent transmittance of this line varies below 60 percent or above 70 percent, the known variables such as temperature of development tank, age of developer, voltage and current of the source, electrode tips, wall thickness of the lower electrodes, are investigated. If these variables are found to be in order, the amount of light entering the spectrograph is readjusted with the aid of neutral filters to correct for the intensity drift.

DISCUSSION

The semiquantitative method has been applied to numerous types of materials and the results were reported in factor-of-10 brackets. Experience with the method showed that the standardization, sample handling, method of arcing, and development of the plates were satisfactory. The weakest link was the visual plate reading. Microphotometric scanning showed that in 93 percent of the experiments the agreement with the chemical analysis was within a factor-of-10 bracket, 85 percent were within a factor-of-5 bracket, and 79 percent were within a factor-of-2 bracket. The precision gained by the introduction of microphotometry, as indicated

by the tests, warrants reporting the values in brackets of smaller concentration ranges than for the previously reported semiquantitative method, with only a slight increase in total analysis time.

A detailed discussion on the observations of individual lines is given in table 2. Table 4 lists the authors' opinion of the value of the selected lines. The results obtained with the lines marked "good" generally fall within a factor-of-2 bracket of the chemical analysis, and the lines marked "fair" fall within a factor-of-5 bracket. The lines marked "poor" are considered undesirable for accurate testing by this method unless the results are to be reported in factor-of-10 brackets. The line magnesium 2781.4 A, sample F84, table 2, illustrates this point; the chemical value was 0.126 and the spectrographic value was 0.58 percent, both results falling within a factor-of-10 bracket.

The time required for an analysis is greatly influenced by the skill of the analyst and especially the physical nature of the sample to be analyzed. An approximate breakdown of the analysis time is as follows:

Number of samples (952 elements)	14
Quarter, weigh samples and proper reference samples	4 hours
Exposure	1.5 hours
Development	0.75 hour
Plate scanning and estimates	15-20 hours

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Table 1.--Example of scanning information, for aluminum, recorded on card.

Line (A)	10 percent			1.0 percent			0.1 percent			0.01 percent			0.001 percent			0.0001 percent		
	Percent transmission	Line width (mm)		Percent transmission	Line width (mm)		Percent transmission	Line width (mm)		Percent transmission	Line width (mm)		Percent transmission	Line width (mm)		Percent transmission	Line width (mm)	
2568.0	11.5	1	50 1/	21.	Normal		Vis 2/	Normal										
2575.1	8.	2	50	12.5	1	50	79.5	Normal										
2652.5	14.5	1	50	24.	Normal													
2660.4	10.	1	50	16.5	Normal													
3082.2	9.5	4	50	5.5	3	50	11.5	1	50	48.5	Normal		76.	Normal		89.	Normal	
3092.7	10.	5	50	6.	3	50	4.	1.5	50	26.5	1	50	55.	Normal		74.5	Normal	
3059.9	78.	Normal		95.														
3064.3	47.	Normal		87.	Normal													
3066.2	45.5	Normal		88.5	Normal													

1/ Width measured at middle line (50) on chart.

2/ Just visible.

Table 2.--Spectrum lines used and comparison of spectrographic and chemical analyses of ashes.

Element	Line (A)	Sample	Spectrographic		Chemical (percent)	True value (percent)	Remarks
			Microphotometric scanning (percent)	Visual (percent)			
Ag	3280.7	Synthetic	0.00068	0.0001-0.001		0.0005	3280 A satisfactory when background permits its use.
Al	2652.5	Synthetic	10+	10+		13.	High iron may interfere with 3059 A.
	3059.9	C18b	7.1	1.0-10.0	6.75		
	3059.9	F84	10+	1.0-10.0	10.1		
	2575.1	F95	10+	1.0-10.0	11.6		
	3059.9	S29	10+	1.0-10.0	9.3		
	3059.9	SC35	10+	1.0-10.0	7.8		
As	2860.5	Synthetic	0.94	0.1-1.0		0.5	High result is probably due to iron interference.
Au	2352.7						Looked for but not found.
	2428.0						
	2676.0						
	3122.8						
B	2497.8	Synthetic	0.4	0.1-1.0		0.5	2496.7 A may also be used when the boron is over 0.1 percent.
		C18b	0.022	0.01-0.1			
		F84	0.071	0.01-0.1			
		F95	0.066	0.01-0.1			
		S29	0.08	0.01-0.1			
		SC35	0.07	0.01-0.1			

Table 2.--Spectrum lines used and comparison of spectrographic and chemical analyses of ashes--Continued.

Element	Line (A)	Sample	Spectrographic		Chemical (percent)	True value (percent)	Remarks
			Microphotometric scanning (percent)	Visual (percent)			
Ba	2335.3	Synthetic	0.6			0.5	2335 A was satisfactory in this range.
		C18b	0.32		0.27		
		F84	0.69	0.01-0.1			
		F95	0.65	0.01-0.1			
		S29	0.26	0.01-0.1			
		SC35	0.57	0.01-0.1			
Be	2348.6	Synthetic					Be 2348.6 A satisfactory for low Be when iron is not too high.
		C18b	0.0013	0.0001-0.001			
		F84	0.0037	0.001-0.01			
		F95					
		S29	0.00013	0.0001-0.001			
		SC35	0.004	0.001-0.01			
Bi	3067.7						Looked for but not found.
	2897.9						
Ca	3000.9	Synthetic	9.2	1.0-10.0		8.08	High iron interferes with Ca 3000, and 3009 A. 2997 A seems to be the better line.
	3009.2		9.3				
	3000.9	C18b	10.+	1.0-10.0	11.1		
	3009.2		10.+				
	2997.3	F84	4.75	1.0-10.0	2.9		
	3000.9		7.78				
	3009.2		7.66				
	2997.3	F95	6.00	1.0-10.0	7.8		
	2997.3	S29	8.07	1.0-10.0	8.6		
	2997.3	SC35	9.4		14.4		
	3000.9		10.+	1.0-10.0			
	3009.2		10.+				

Table 2.--Spectrum lines used and comparison of spectrographic and chemical analyses of ashes--Continued.

Element	Line (A)	Sample	Spectrographic: Microphotometric scanning (percent)	Spectrographic: Visual (percent)	Chemical (percent)	True value (percent)	Remarks
Cd	3466.2 3261.1 2763.9 2288.0						Looked for but not found.
Ce	4222.6 4186.6 4040.7 4012.4						Looked for but not found.
Co	3453.5	Synthetic	0.05[4]	0.01-0.1		0.05	Background interference apparently is not too serious.
		F95	0.05	0.1-1.0	0.04		
Cr	2835.6	Synthetic	0.043	0.01-0.1		0.05	2835 A is satisfactory.
		C18b	0.07	0.001-0.01			
		F84	0.01	0.001-0.01			
		F95					
		S29	0.01	0.001-0.01			
		SC35	<0.01	0.001-0.01			
Cs	4593.0 4555.5 3347.4 3247.5						Looked for but not found.

Table 2.--Spectrum lines used and comparison of spectrographic and chemical analyses of ashes--Continued.

Element	Line (A)	Sample	Spectrographic		Chemical (percent)	True value (percent)	Remarks
			Microphotometric scanning (percent)	Visual (percent)			
Cu	3247.6	Synthetic	0.0088	0.001-0.01		0.005	3247 and 3274 A are satisfactory when iron is not too high. The line width factor is difficult to apply when copper is high.
	3274.0		0.0089				
	3247.6	C18b	0.1	0.001-0.01			
		F84					
	3274.0	F95	0.0074	0.001-0.01			
	3247.6	S29	0.008	0.001-0.01			
	3274.0	SC35	0.0093	0.001-0.01			
Dy	3454.33						Looked for but not found.
	3407.8						
	3393.58						
Er	4419.6						Looked for but not found.
	3499.1						
	3372.8						
	3264.8						
Eu	2727.8						Looked for but not found.
	2813.1						
F (CaF band)	5291.0						Looked for but not found.
	6036.9						
	6064.4						

Table 2.--Spectrum lines used and comparison or spectrographic and chemical analyses of ashes--Continued.

Element	Line (A)	Sample	Spectrographic: Microphotometric scanning (percent)	Spectrographic: Visual (percent)	Chemical (percent)	True value (percent)	Remarks
Fe	2936.9	Synthetic	6.5	1.0-10.0		8.75	2936 and 2953 A seem best in this range.
	2953.9		7.0				
	2936.9	C18b	10.+	1.0-10.0	8.0		
	2953.9						
	2599.4	F84	10.+	10.+	24.		
	2599.6		10.+	10.+			
	2936.9	F95	10.+	10.+	21.9		
	3008.1	S29	10.+	10.+	24.0		
	2599.4		10.+	10.+	24.0		
	2953.9	SC35	10.+	10.+	15.8		
	2936.9		10.+	10.+			
Ga	2943.7	Synthetic	0.03	0.001-0.01		0.005	As a result of previous work the visual Ga estimates were down-graded one bracket. This work is in agreement. The reason for this enhancement is unknown at present.
		C18b	0.04	0.001-0.01			
		F84	0.07	0.001-0.01			
		F95	0.30	0.01-0.1			
		S29	0.35	0.01-0.1			
		SC35	0.35	0.01-0.1			
Gd	3671.2						Looked for but not found.
	3646.2						
	3358.6						
	3082.0						
Ge	3039.1	Synthetic	0.0075	0.001-0.01			3039 A satisfactory in this range. Ge not detected in other samples.

Table 2.--Spectrum lines used and comparison of spectrographic and chemical analyses of ashes--Continued.

Element	Line (A)	Sample	Spectrographic		Chemical (percent)	True value (percent)	Remarks
			Microphotometric scanning (percent)	Visual (percent)			
Hf	3134.7 3072.8 2820.2						Looked for but not found.
Hg	3125.6 2536.5						Looked for but not found.
Ho	3399.0 3416.5						Looked for but not found.
In	4511.3 3256.09						Looked for but not found.
Ir	2664.8 2849.7 2924.8						Looked for but not found.
K	3446.4	Synthetic	2.35	1.0-10.		2.0	Background corrections apparently are necessary for certain samples.
		C18b	1.	0.1-1.	0.78		
		F84	2.2	1.0-10.	0.68		
		F95	0.48	0.1-1.0	0.68		
		S29	3.2	1.0-10.	1.28		
		SC35	3.2	1.0-10.	0.92		
La	4429.9 3380.9 3337.5						Looked for but not found.

Table 2.--Spectrum lines used and comparison of spectrographic and chemical analyses of ashes--Continued.

Element	Line (A)	Sample	Spectrographic: Microphotometric scanning (percent)	Spectrographic: Visual (percent)	Chemical (percent)	True value (percent)	Remarks
Li	3232.7 2741.3						Looked for but not found.
Lu	2615.4 2619.3 2911.4 3198.1						Looked for but not found.
Mg	2778.3	Synthetic	1.	1.0-10.0		1.5	More work necessary to select the better lines for magnesium determinations. 2779 best in 0.1-1. percent range. 2781 and 2778 are indicated for the 1.0-10. percent range.
	2778.3	C18b		1.0-10.0	4.4		
	2779.9		0.9				
	2778.3	F84	0.764	0.1-1.0	0.126		
	2779.9		0.37				
	2781.4		0.58				
	2778.3	F95	0.923	0.1-1.0	0.13		
	2779.9		1.0				
	2781.4		2.6				
	2779.9	S29	0.78	0.1-1.0	0.138		
	2798.0		0.155				
	2779.9	SC35	0.78	0.1-1.0	0.072		
			0.935				
			0.5				

Table 2.--Spectrum lines used and comparison of spectrographic and chemical analyses of ashes--Continued.

Element	Line (A)	Sample	Spectrographic		Chemical (percent)	True value (percent)	Remarks
			Microphotometric scanning (percent)	Visual (percent)			
Mn	2593.7	Synthetic	0.062			0.5	2801 A satisfactory only when Al is very low. When Al is high 2576 is suggested. 2593 and 2605 are reliable.
	2605.7		0.056	0.01-0.1			
	2605.7	C18b	0.01		0.04		
	2576.1		0.05	0.01-0.1			
	2593.7	F84	0.092		0.2		
	2605.7		0.088	0.01-0.1			
	2593.7	F95	0.68		0.78		
	2605.7		0.7	0.1-1.0			
	2801.1		0.1				
	2593.7	S29	0.066		0.11		
	2605.7		0.062	0.01-0.1			
	2593.7	SC35	0.18		0.2		
	2605.7		0.089				
	2801.1		0.325				
Mo	3170.4	Synthetic	0.004	0.001-0.01		0.005	Use width of 3170 A for high Mo. 3170 A satisfactory for 0.01-0.1 range. Indications are that 3122 A should be used above 0.1 percent.
	3132.6	C18b	0.046	0.01-0.1			
	3170.4	F84		0.1-1.			
	3122.0		0.24	0.1-1.			
	3170.4	F95	0.041	0.01-0.1			
	3122.0	S29	0.1	0.1-1.0	0.15		
	3170.4		0.1	0.1-1.0			
	3122.0	SC35	0.2	0.1-1.0			

Table 2.--Spectrum lines used and comparison of spectrographic and chemical analyses of ashes--Continued.

Element	Line (A)	Sample	Spectrographic		Chemical (percent)	True value (percent)	Remarks
			Microphotometric scanning (percent)	Visual (percent)			
Na	3302.3	Synthetic	3.7	1.0-10.		2.5	Both lines in doublet are satisfactory.
	3302.3	C18b	10.+	10.+	17.		
	3302.3	F84	4.15	1.0-10.	2.6		
	3302.9	F95	0.98	1.0-10.	1.41		
	3302.9	S29	2.85	1.0-10.	3.26		
	3302.9	SC35	3.7	1.0-10.	2.33		
Nb	3358.4						Looked for but not found.
	3094.2						
	2875.5						
Nd	4325.8						Looked for but not found.
	4303.6						
	4247.4						
	3328.3						
Ni	3393.0	Synthetic	0.3			0.5	Slightly high results are probably due to background.
	3423.7		0.1	0.01-0.1			
	3414.8	C18b	0.026	0.01-0.1			
Os	2909.1						Looked for but not found.
	3058.7						
	3301.6						
P	2535.6	Synthetic	0.25	0.01-0.1		0.5	Beware of iron interference.

Table 2.--Spectrum lines used and comparison of spectrographic and chemical analyses of ashes--Continued.

Element	Line (A)	Sample	Spectrographic: Microphotometric scanning (percent)	Spectrographic: Visual (percent)	Chemical (percent)	True value (percent)	Remarks
Pb	2833.1	Synthetic	0.68	0.01-0.1		0.5	Previous work indicated that lead in the low percentages was enhanced by one bracket. Visual results were down-graded one bracket. Confirmation is indicated by these tests. No explanation is available at present.
	2833.1	C18b	0.04	<0.01-0.1			
	2833.1	F84	0.043	<0.01-0.1			
	2833.1	F95	0.059	<0.01-0.1			
	2833.1	S29	0.057	<0.01-0.1			
	2833.1	SC35	0.0575	<0.01-0.1			
Pd	2763.1						Looked for but not found.
	3114.0						
Pr	4241.0						Looked for but not found.
	4225.3						
Pt	4442.6						Looked for but not found.
	2659.4						
Rb	4215.6						Looked for but not found.
	3350.9						
Re	3460.5						Looked for but not found.
	3464.7						
Rh	3280.5						Looked for but not found.
	4374.8						
Ru	2810.0						Looked for but not found.
	4297.7						

Table 2.--Spectrum lines used and comparison of spectrographic and chemical analyses of ashes--Continued.

Element	Line (A)	Sample	Spectrographic: Microphotometric scanning (percent)	Spectrographic: Visual (percent)	Chemical (percent)	True value (percent)	Remarks
Sb	3267.5 2877.9						Looked for but not found.
Sc	3273.6 3353.7 3359.7	Synthetic	0.005 0.001 0.007			0.005	
Si	2506.9	Synthetic	10.+	10.+		11.	If Si 2881 A is 3-4 mm at 50 and 2506 A is normal in width but reads off the scale, use 2435 A because Si is under 10 percent in these cases.
	2435.2	C18b	7.9	1.0-10.	5.35		
	2506.9	F84	10.+	1.0-10.	13.1		
	2506.9	F95	10.+	1.0-10.	15.0		
	2435.2	S29	7.9	1.0-10.	7.1		
	2435.2	SC35	6.88	1.0-10.	4.45		
Sm	4224.4 4256.4						Looked for but not found.
Sn	3175.0 3262.3	Synthetic	0.1 0.0035			0.005	Sn 3262 A satisfactory.
Sr	3646.5	Synthetic	0.008	0.01-0.1		0.05	A survey for more satisfactory strontium lines is in progress.
	3380.7	C18b	1.	0.1-1.0	0.45		
	3464.5		1.				
	3464.5	F84	0.05	0.01-0.1			
	3464.5	F95	0.26	0.1-1.0			
	3464.5	S29	0.07	0.1-1.0			
	3464.5	SC35	0.67	0.1-1.0			

Table 2.--Spectrum lines used and comparison of spectrographic and chemical analyses of ashes--Continued.

Element	Line (A)	Sample	Spectrographic		Chemical (percent)	True value (percent)	Remarks
			Microphotometric scanning (percent)	Visual (percent)			
Ta	3642.1						Looked for but not found.
	3317.9						
Tb	3293.1						Looked for but not found.
	3324.4						
Te	2383.3						Looked for but not found.
	2385.8						
Ti	3236.6	Synthetic	0.085	0.1-1.0		0.5	A survey for more satisfactory titanium lines is in progress.
	3242.0		0.05				
	3242.0	C18b		0.01-0.1	0.06		3242 A is satisfactory in 0.01-1. percent range.
	3242.0	F84		0.1-1.0	0.36		
	3242.0	F95		0.1-1.0	0.36		
	3242.0	S29	0.08	0.01-0.1	0.06		
	3242.0	SC35	0.071	0.01-0.1	0.06		
Th	2837.3	Synthetic	0.07	0.1-1.0		0.5	These lines are satisfactory when uranium is under 1 percent.
	2870.4		0.04	0.1-1.0			
Tl	3529.4						Looked for but not found.
	2767.9						
	2379.6						
Tm	3151.3						Looked for but not found.
	4242.2						

Table 2.--Spectrum lines used and comparison of spectrographic and chemical analyses of ashes--Continued.

Element	Line (A)	Sample	Spectrographic: Microphotometric scanning (percent)	Spectrographic: Visual (percent)	Chemical (percent)	True value (percent)	Remarks
U	4287.9						Under the threshold value.
	4244.4						
	3566.6						
V	3267.7	Synthetic	0.009	0.001-0.01		0.005	3267 and 3276 A are satisfactory for low vanadium.
	3276.1	C18b	0.0036	0.001-0.01			
	3276.1	F84	0.009	0.01-0.1			
	3276.1	F95	0.0087	0.01-0.1			
	3276.1	S29	0.044	0.001-0.01			
	3276.1	SC35	0.03	0.01-0.1			
W	4302.1						Looked for but not found.
	4294.6						
	3049.7						
Y	3327.9	Synthetic	0.0062	0.001-0.01		0.005	3327 A and 3242 A are satisfactory.
	3242.3		0.0075	0.001-0.01			
	3216.7		0.019				
	3327.9	C18b	0.017	0.001-0.01			
	3327.9	F84	0.01	0.01-0.1			
	3327.9	S29		0.001-0.01			
	3327.9	SC35		0.001-0.01			
Yb	3289.4	Synthetic	<0.001	0.001-0.01		0.005	Search for better lines in progress.
	3289.4	C18b		0.001-0.01			
	3289.4	F84	0.002	0.001-0.01			
	3289.4	S29					
	3289.4	SC35					

Table 2.--Spectrum lines used and comparison of spectrographic and chemical analyses of ashes--Continued.

Element	Line (A)	Sample	Spectrographic		Chemical (percent)	True value (percent)	Remarks
			Microphotometric scanning (percent)	Visual (percent)			
Zn	3345.0	Synthetic	0.006	0.001-0.01		0.05	Search for better lines indicated.
	3345.0		0.008	0.001-0.01			
Zr	3391.9	Synthetic	0.03	0.01-0.1		0.05	3392 A satisfactory when background is low. 3273 A satisfactory when copper is low.
	3273.0	F84	0.008	0.1-1.			
	3273.0	SC35	0.036	0.1-1.			
	3273.0	C18b	0.17	0.01-0.1			
	3273.0	S29	0.004	0.001-0.01			

Table 3.--Composition of the synthetic sample.

Element	Percent	Element	Percent	Element	Percent	Element	Percent	Element	Percent
Al	13.	Ti	0.5	Sr	0.05	Y	0.005	Ag	0.0005
Si	11.	As	0.5	Co	0.05	Mo	0.005	Yb	0.0005
Fe	8.75	B	0.5	Mn	0.05	Ge	0.005		
Na	2.5	Ba	0.5	Ni	0.05	Ga	0.005		
Ca	8.08	P	0.5	Pb	0.05	Sn	0.005		
Mg	1.5	Th	0.5	Cr	0.05	V	0.005		
K	2.0			Zr	0.05	Sc	0.005		
U	1.0			Zn	0.05	La	0.005		
				Ce	0.05				
				Cu	0.05				

Table 4.--Summary of lines used, percentage range, and rating of line in spectrographic analysis of ashes.

Element	Line (A)	Range (percent)	Rating
Ag	3280.7	0.0001-0.001	Good
Al	2575.1	10.+	Good
	2652.5	10.+	Good
	3059.9	1.0-10.+	Fair
As	2680.5	0.1-1.0	Fair
B	2497.8	0.01-1.0	Good
Ba	2335.3	0.1-1.0	Good
Be	2348.6	0.0001-0.001	Fair
Ca	2997.3	1.0-10.	Good
	3000.9	1.0-10.	Good
	3009.2	1.0-10.	Fair
Co	3453.5	0.01-0.1	Good
Cr	2835.6	0.01-0.1	Good
Cu	3247.6	0.001-0.1	Fair
	3274.0	0.001-0.01	Fair
Fe	2936.9	1.0-10.	Fair
	2953.9	1.0-10.	Fair
	2599.4	10.+	Fair
	3008.1	10.+	Fair
Ga	2943.7	0.001-0.01	Poor 1/
Ge	3039.1	0.001-0.01	Fair
K	3446.6	0.1-10.	Fair
Mg	2778.3	1.0-10.	Fair
	2779.9	0.1-1.0	Fair
	2781.4	0.1-1.0	Poor
	2798.0	0.1-1.0	Fair

1/ Down-grading the spectrographic results by a factor of 10 will bring chemical and spectrographic in agreement. No explanation is available at present.

Table 4.--Summary of lines used, percentage range, and rating of line in spectrographic analysis of ashes--Continued.

Element	Line (A)	Range (percent)	Rating
Mn	2593.7	0.01-1.	Good
	2576.1	0.01-0.1	Good
	2605.7	0.01-0.1	Fair
	2801.1	0.1-1.0	Fair
Mo	3170.4	0.001-0.01	Good
	3132.6	0.001-0.01	Good
	3122.0	0.01-0.1	Good
	3122.0	0.1-1.0	Good
Na	3302.3	1.0-10.+	Fair
	3302.9	1.0-10.	Fair
Ni	3393.0	0.01-0.1	Poor
	3423.7	0.01-0.1	Fair
	3414.8	0.01-0.1	Fair
P	2535.6	0.1-1.0	Fair
Pb	2833.1	0.01-1.0	Poor 1/
Sc	3273.6	0.001-0.01	Good
	3353.7	0.001-0.01	Poor
	3359.7	0.001-0.01	Good
Si	2506.9	10.+	Good
	2435.2	1.0-10.	Good
Sn	3175.0	0.001-0.01	Poor
	3262.3	0.001-0.01	Fair
Sr	3464.5	0.001-0.01	Poor
	3380.7	0.01-0.1	Fair
Ti	3236.6	0.01-1.	Poor
	3242.0	0.01-0.1	Good
Th	2837.3	0.1-1.	Poor
	2870.4	0.1-1.	Poor
V	3267.7	0.001-0.01	Fair
	3276.1	0.001-0.01	Good

1/ Down-grading the spectrographic results by a factor of 10 will bring chemical and spectrographic in agreement. No explanation is available at present.

Table 4.--Summary of lines used, percentage range, and rating of line in spectrographic analysis of ashes--Continued.

Element	Line (A)	Range (percent)	Rating
Y	3327.9	0.001-0.01	Good
	3242.3	0.001-0.01	Good
	3216.7	0.001-0.01	Fair
Yb	3289.4	0.001-0.01	Fair
Zn	3345.0	0.001-0.01	Poor
Zr	3391.9	0.01-0.1	Good
	3273.0	0.01-0.1	Good