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IN REPLY REFER TO:

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DEPARTMENT OF THE INTERIOR
GEOLOGICAL SURVEY
WASHINGTON 25, D. C.

October 31, 1952

AEC - 423/3

Dr. Phillip L. Merritt, Assistant Director
Division of Raw Materials
U. S. Atomic Energy Commission
P. O. Box 30, Ansonia Station
New York 23, New York

Dear Phil:

Transmitted herewith are six copies of Trace Elements Investigations Report 274, "Spectrographic identification of mineral grains," by J. N. Stich, October 1952.

We plan to publish this report as a Geological Survey circular. We are asking Mr. Hosted whether the Commission approves this plan.

Sincerely yours,

Arthur P. Butler, Jr.
for W. H. Bradley
Chief Geologist

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Geology - Mineralogy

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UNITED STATES DEPARTMENT OF THE INTERIOR

GEOLOGICAL SURVEY

SPECTROGRAPHIC IDENTIFICATION OF MINERAL GRAINS *

By

J. N. Stich

October 1952

Trace Elements Investigations Report 274

This preliminary report is distributed without editorial and technical review for conformity with official standards and nomenclature. It is not for public inspection or quotation.

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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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By

J. N. Stich

ABSTRACT

A qualitative spectrographic method of analysis of single mineral grains, impurity inclusions, X-ray powder spindles, and other small samples weighing 1 mg or less is described. Samples weighing as little as 0.01 mg have been analyzed. When 1 mg of sample is available, the accuracy of the method approaches that of a semiquantitative one. During a 10-month period, 20,000 determinations of 69 metallic and metalloid elements in over 300 small samples have been made with the method. In conjunction with petrographic, physical, and X-ray methods, these determinations of chemical composition have been useful in establishing the mineral identity of small single grains. A direct-current arc is used. The solution procedure of standardization involving only one element per plate greatly facilitates the selection of reliable analytical lines. A small carbon electrode cut from 1/8-in-diameter graphite stock is used; this is convenient for the loading and arcing of X-ray powder spindles. The medium quartz prism spectrograph is preferable to a grating instrument for qualitative microanalysis as its complete spectrum coverage for a single exposure includes the most sensitive lines of the alkali elements.

INTRODUCTION

In connection with the investigation of radioactive raw materials conducted by the Geological Survey on behalf of the Atomic Energy Commission, a very large number and variety of samples are received for spectrographic analysis.

Among these samples are many which are available only in 1-mg amounts or less and consist chiefly of mineral grains and impurity inclusions hand-picked under a petrographic microscope from various rocks and ores being studied. Because these small samples are composed of grains selected individually and occasionally include but one grain each, they are also referred to as "single-grain" samples.

Although many of these grains can be identified on the basis of such properties as crystal structure, refractive index, hardness, specific gravity, and X-ray diffraction pattern, frequently their chemical composition must also be determined in order to establish their mineral identity (Lee and Wright, 1939).

Owing to the small size of the samples, which generally limits the duration in the arc of their characteristic spectra to a few seconds or less, the application of a visual spectroscope (Peterson et al., 1947; Jaffe, 1949) is restricted to the determination of one or two major elements at a time. Also, unless larger samples are used, many elements, such as arsenic, boron, and phosphorus, whose principal emission lines occur in the ultraviolet range not visible to the eye, cannot be detected with the spectroscope.

The use of the more sensitive spectrograph, however, permits the rapid qualitative analysis of single-grain samples for a complete range of metallic and metalloid elements in concentrations down to 0.1 percent and lower. Another advantage of the spectrograph is that, in the form of a spectrogram recorded on a photographic plate, it produces an objective and permanent record of the analysis of a sample.

The author is indebted to his associates of the Geological Survey for their help and cooperation, particularly to A. W. Helz for his invaluable technical aid and to C. L. Waring whose suggestions contributed materially to the scope of this paper. Special thanks are due also to C. S. Annell for the use of his standard solutions, to Alice Dowse Weeks and Mary E. Thompson for providing many of the samples, to Evelyn Cisney and Joseph Berman for the X-ray diffraction pattern checks, and to A. M. Sherwood for most of the chemical microanalyses.

DISCUSSION

In addition to inclusions and small single grains, the types of samples analyzed have included magnetic separates, fragments of mineralized wood, placer sand, sediments, chemical precipitates, distilled water residues, furnace slags, and corrosion products. Samples weighing as little as 0.01 mg have been analyzed. During a period of ten months, 20,000 determinations of 69 metallic and metalloid elements (table 1) in over 300 small samples have been made with the method.

The single-grain samples are received in the form either of the grains themselves or more often of ethyl cellulose X-ray powder spindles containing the grains.

The practice of arcing X-ray spindles rather than duplicate samples of the grains is advantageous because it reduces the amount of separation work required and also permits the data from the spectrograms of the arced spindles to be checked directly with that from the X-ray diffraction patterns previously obtained.

The spectrographic results are reported qualitatively as "major", "minor", and "trace", corresponding approximately to brackets of 10 percent and over, 1 - 10 percent, and 0.1 - 1 percent concentrations. With single-grain samples and X-ray spindles these results are used specifically (1) to confirm the chemical compositions of minerals tentatively identified by their X-ray diffraction patterns and other properties, (2) to determine impurities in the minerals, and (3) to differentiate between minerals producing similar X-ray diffraction patterns, such as metatorbernite and metazeunerite. If no patterns were obtained, the spectrographic reports aid further study and purification work.

Some of the minerals which have been analyzed and whose identifications have been confirmed by checks of the spectrographic results with the X-ray diffraction patterns are:

Uranium-bearing and rare-earth minerals

Allanite
Autunite
Bastnaesite
Beta-uranotil
Brannerite
Carnotite
Cuprosklodowskite
Cyrtolite
Euxenite
Florencite
Huttonite
Kasolite
Parisite
Phosphuranylite
Rauvite
Saleeite
Samarskite
Sklodowskite
Soddyite
Thorite
Torbernite
Tyuyamunite
Uraninite
Uranocircite
Uranophane
Zeunerite

Miscellaneous minerals

Apatite
Apophyllite
Azurite
Barite
Biotite
Calcite
Cerussite
Chrysocolla
Conichalcite
Franklinite
Gibbsite
Goethite
Halite
Hematite
Hewettite
Hydromica
Ilmenite
Kaolinite
Magnetite
Melanovanadite
Montroseite
Orpiment
Pyrargyrite
Pyrochlore-microlite
Quartz
Realgar
Rutile
Smaltite-cloanthite
Soda-niter
Sphalerite
Talc
Topaz
Vosenite
Zircon

METHODS

The spectrographic method of microanalysis initially applied to this wide variety of minerals utilized a 220 d-c volt arc at 10 amp and an Eagle-mounted 3-meter grating spectrograph. The d-c arc was chosen because, in general, it gives the highest sensitivity (Nachtrieb, 1950). As the method involved a determination of major and minor elements in very small samples, the use of the same excitation conditions favorable to the determination of trace elements in larger samples was indicated.

The standards used consisted of four mixtures containing 10 different elements each, prepared in 1, 0.1, and 0.01 percent series of concentration, with silica as the base material. Because the use of 1-mg amounts of each of the standard mixtures in the preparation of the standard plates was based on taking 1 mg of the unknown sample, 10 percent standard spectrograms were obtained by using 10-mg amounts of the 1 percent mixtures. The arrangement of the elements within each mixture followed that described by Van Tongeren (1938).

This procedure had two serious limitations: (1) as the exposures were made in the generally useful range of 2450-3850 A, the single-exposure spectrum coverage of only 1400 A did not include the most sensitive lines of the alkali elements; and (2) the complexity of the spectrograms of the 1 percent standard mixtures, which made it difficult to select and identify the reliable analytical lines for each element.

The disadvantage of limited wavelength coverage for a single exposure applied also to the Wadsworth-mounted 21-ft grating spectrograph. With its grating set to cover the ultraviolet range down to 2250 A, the upper wavelength limit effective is at 4750 A, which falls short of the 5890-7700 A region where the persistent K, Li, and Na lines are located. The detection limits of K and Na with the grating spectrographs set in the ultraviolet range are only about 10 percent on the basis of 1 mg of sample.

When a medium quartz prism spectrograph accomodating 14-in plates and permitting a single-exposure coverage of the entire range from 2080 to 10,000 A became available, the method was transferred to this instrument. The d-c arc was retained. At the same time the solution standardization procedure described by Waring and Annell (1951) was adopted. (See Procedure.)

Because of the complete spectrum coverage of the prism spectrograph for a single exposure, which makes available the K lines at 7665 and 7699 A and the Na lines at 5890 and 5896 A, it is possible to detect K and Na as low as 0.1 percent in 1-mg samples. This is a distinct advantage, especially in seeking to differentiate between various uranium minerals, such as carnotite and tyuyamunite. (See table 4.) In addition the Ca lines at 8498, 8542, and 8662 A are available. As many of the samples submitted for analysis contain high concentrations of uranium, these lines are useful because their intensities are not depressed by uranium nearly as much as are those of the Ca lines at 3159 and 3179 A.

The use of standard solutions, involving only one element per plate and producing relatively simple spectrograms, greatly facilitated the selection of reliable analytical lines. (See table 2.) To a considerable extent, the accuracy of the method depends upon the utilization of the proper lines. (See Results and Tables.)

Checks of many of the X-ray diffraction patterns and some chemical checks, obtained in those few cases where enough sample was available for a chemical microanalysis, have been made (tables 3 and 4). The agreement of the chemical and spectrographic results are adequate for purposes of mineral identification. It has been our experience in applying the method that the agreement is likely to be good for samples weighing about 1 mg; for such samples the results may become semiquantitative rather than qualitative.

Although the standardization of the method is on a semiquantitative basis (Waring and Annell, 1951), the results have been reported only in qualitative terms, defined in the Discussion, for the following reasons: (1) many of the samples weigh less than 1 mg, necessitating upgrading of the element concentration estimations with respect to the standard spectrograms that are based on 1 mg of sample; (2) in the case of the X-ray powder spindles the exact weights of the samples are not known; (3) the omission of a flux (see Procedure) increases the possibility of the sample matrix influencing a determination; and (4) the number of chemical checks is insufficient for a statistical study of the accuracy of the method.

The dispersion of the medium quartz prism spectrograph used is 4.6 A/mm at 2500 A, 8.4 at 3000 A, 17.2 at 3500 A, 21.5 at 4000 A, 42 at 5000 A, 75 at 6000 A, and 111.5 at 7000 A. The low dispersion from 5000 A up is not troublesome because this region is generally free from strong lines other than those of the alkali elements which are readily resolved.

Another medium quartz prism spectrograph was available having a lower dispersion of 8.7 A/mm at 2500 A, 14.8 at 3000 A, 22.5 at 3500 A, 33 at 4000 A, 61 at 5000 A, 100 at 6000 A, and 167 at 7000 A. This spectrograph permits a complete spectrum coverage for a single exposure on a 10-in. plate. Tests were made to determine whether this instrument was also applicable to the microqualitative method in spite of its lower dispersion than the first spectrograph. Under similar excitation, optical, and exposure conditions (Procedure), a plate was exposed to 1-mg samples of four minerals producing complex spectra (carnotite, calcium uranylvanadate, brannerite, and allanite). Then, utilizing the solution method of preparing the standard electrodes, standard plates were made for the elements Ca, K, Na, Fe, Ti, Si, Th, U, V, Ce, La, and Y, constituting the major and minor components of these minerals.

The series of spectrograms obtained for each element represented concentrations of 10 percent, 1 percent, and 0.1 percent based on 1 mg of sample. By means of these standard plates, the positions of the analytical lines were readily determined. In spite of the low dispersion of the instrument, no difficulty was experienced in identifying these lines in the complex spectrograms of the "unknown" samples. The analytical results obtained checked with those previously reported for the same samples tested with the prism spectrograph with higher dispersion. It is thus indicated that, in conjunction with the solution standardization procedure, the lower dispersion medium prism spectrograph also can be used effectively for the qualitative analysis of single grains, X-ray spindles, and other small samples weighing 1 mg or less.

Prior to adopting the solution method of standardizing the medium quartz prism spectrographs, the use of pure minerals as standards had been considered. For the following reasons, however, it was adjudged impracticable: (1) the very large number of mineral standards required; (2) the difficulty of obtaining pure minerals; (3) the problem of diluting these minerals to obtain sets of spectrograms corresponding to various series of decreasing element concentrations; (4) the presence of impurities in the unknown samples analyzed and the fact that the samples are often mixtures of two or more distinct minerals; (5) the need for an independent and all-inclusive procedure of analysis in cases where the sample is a newly discovered mineral in the process of identification or where it is a chemical precipitate or other material;

and (6) the complexity of the resulting spectrograms thus again making it difficult to select reliable analytical lines.

PROCEDURE

As described by Waring and Annell (1951), the solution standardization procedure involves the preparation of a standard plate for each element, with each plate showing a series of spectrograms representing element percentages decreasing in multiples of ten from 10 percent to 0.0001 percent, based on an unknown sample weighing 1 mg.

The standard solutions are prepared from "C.P." and "Specpure" grade chemicals usually to give a concentration of 10 mg of the element per milliliter of solution (see Appendix for the compositions); 0.01 ml of each 10 mg/ml solution is equivalent to 10 percent of the element in a 1-mg sample. These solutions are consecutively diluted so that 0.01 ml of each is then equivalent to 1.0, 0.1, 0.01, 0.001, and 0.0001 percent of element based on a 1-mg sample.

In the preparation of a series of standard electrodes representing 10 - 0.0001 percent element concentrations in a 1-mg sample, the bottoms of the electrode craters are first sealed with paraffin added in the form of a few drops of a 1 percent benzene solution of the wax (Waring and Annell, 1951). Then 0.01-ml aliquots of the 10 - 0.0001 mg/ml solutions are introduced into the electrodes, using a micropipette equipped with an extension whose tip fits into the craters. The electrodes are dried in a brass holder placed on an asbestos-covered hot plate adjusted to a temperature of approximately 100 C.

Provision of reference spectra for the standard plates follows the arrangement used by Waring and Annell (1951). Prior to arcing of the electrodes in the order of decreasing element concentration, a spectrum of iron is recorded on the plate. Following arcing of the last standard electrode, a spectrum of 1 mg of aluminum alloy (Alcoa aluminum standard no. 874) weighed out in the form of filings is also registered. The spectrograms are 2.3 mm high and positioned so that the bottom of one spectrogram nearly coincides with the top of the succeeding one. The arrangement is similar for the unknowns except that the number of spectrograms included between the reference spectra is limited to two or three. In running a suite of unknowns, a half dozen such series of spectrograms and reference spectra can be recorded on a plate.

The concentrations of elements in the unknown samples are estimated by visual comparison of certain lines of these elements in the unknown spectrograms with those on the standard plates. When the sample weight is less than 1 mg, the estimations are upgraded by the necessary factor. If, for example, the sample weighs 0.5 mg, the preliminary estimations are multiplied by two. The plates are read on a viewing desk under a microscope or on a viewing box with a magnifying eyepiece.

Besides furnishing reference points additional to those provided by the iron spectrum, the aluminum alloy spectrum serves as a useful check on exposure, emulsion speed, and plate processing. The Cr line at 2780.703 A, in particular, is a sensitive indicator of the optimum conditions. When these are being maintained, the intensity of the line falls within a transmission range of 80 to 95 percent, measured with a Jarrell-Ash microphotometer. The other components of the alloy--Cu, Fe, Mg, Mn, Si, Ti, and Zn--present in known trace amounts also produce lines whose intensities may conveniently be used for supplementary reference when estimating concentrations of these elements in the unknowns.

The power for the d-c arc is supplied by a full-wave mercury vapor rectifier (220 volts direct current and 15 amp maximum). An inductance for giving smooth operation of the arc and a rheostat for controlling the current are used in series with the arc. The rheostat has three fixed values, 74, 38, and 19 ohms, producing three possible arc currents of approximately 2, 4, and 9 amp. The value of the inductance is not critical; in this case an old 7.5-kw autotransformer is used for the purpose.

The standard electrodes, the aluminum alloy samples, and the unknowns are arced at 9 amp. The iron arc is operated at 4 amp. All arcings are with a gap of 4 mm for a period of 60 seconds at 8 percent transmission controlled by a rotating-sector disk. The slit width is 10 microns, with the arc focused on the collimating lens. The emulsion used is Eastman I-L, developed in D-19 for 4 minutes at 18 C ± 1/2 C.

The samples are weighed on a 5-mg torsion microbalance and generally range from 0.1 to 1 mg. A microfunnel drawn from 7-mm glass tubing is used to transfer the samples from the detachable balance pan to the electrode craters. Spindles are transferred directly by sliding them off the pan.

The weight of sample in a spindle is determined approximately by subtracting from the total weight of the spindle and its contents the estimated weight of the spindle alone. Spindle weights can be estimated to within about ± 0.1 mg by comparison with an available series of previously weighed blank spindles. A supplementary criterion for estimating the weight of sample in a spindle is the fact that usually the weight ratio of spindle to sample is about 1:1. As spindles and their contents usually weigh from 0.5 to 1 mg, the actual sample weight ranges from 0.25 to 0.5 mg.

No powdered graphite or other flux is added to the sample because the nature of the samples precludes effective mixing.

The determination of fluorine in the samples of low calcium content requires a separate exposure (Waring and Annell, 1952). Two-tenths of a milligram of calcium as calcium chloride in solution is added to the electrodes which are dried prior to introduction of the unknown samples. The addition of calcium causes the formation in the arc of the calcium fluoride molecule which emits the bands used for the fluorine estimation.

The carbon microelectrode used has a deep, thin-walled, cylindrical crater that is convenient for the loading and arcing of X-ray powder spindles, in addition to other materials. The half-inch depth of the crater corresponds to the length of the average spindle, thereby eliminating any necessity for cutting it up. This depth also tends to prevent mechanical loss during arcing of the sample when it consists of refractory grains. After fusing, the sample adheres to the graphite and burns away to completion along with the carbon, thus fulfilling an essential requirement of qualitative analysis (Nachtrieb, 1950). The dimensions of the electrode, cut and drilled with a Bausch and Lomb electrode shaper, are as follows:

1/8-in. spectroscopic carbon rod	1 3/4 in. long
Crater, outside diameter	0.090 in.
Crater, inside diameter	0.052 in. (no. 55 drill)
Crater, depth	1/2 in.
Outside shoulder below crater rim	5/8 in.

The upper electrode, the cathode, is cut from a 1/4-in. spectroscopic carbon rod. Its arc end is hemispherical with a 0.06-in. radius. Tests showed that this upper electrode produces less background in the spectrograms than would be caused by electrodes of smaller dimensions.

The iron arc arrangement consists of a 1/4-in. iron rod as the lower positive electrode and an upper carbon electrode the same as that used for the samples.

RESULTS AND TABLES

Table 1 lists the sensitivities on the basis of 1-mg samples for the 69 elements determined with the method. The values are those indicated by the solution standard plates on the basis of 1 mg of sample. Where less than 1 mg of sample is available, the sensitivities are reduced by a factor equal to that applied in upgrading the estimations of element concentrations. (See Procedure.) Interference by the lines of other elements also decreases the sensitivities listed. For example, when uranium is a major or minor component of a sample, the thorium line at 2837.299 A cannot be used because of interference by uranium lines at 2837.328 and 2837.187 A. The less sensitive thorium lines at 2703.962 and 2565.597 A then must be utilized, thus reducing the visual detection limit of thorium from 0.1 percent to 1 percent. The maximum sensitivities of Al, B, Ca, Cu, Li, Mg, Si, Ti, and V are limited by the presence of these elements as residual impurities in the regular grade carbon electrodes. The method is not designed for extreme sensitivity but rather for a means by which major and minor concentrations of 68 elements in small samples may be checked in one exposure.

Table 2 lists the wavelengths (Harrison, 1939) and individual sensitivities of the analytical lines used. Wherever practicable two or more lines are listed for each of the major, minor, and trace concentration ranges reported. In spectrographic quantitative analysis the utilization of various lines for different ranges of element concentrations is necessary (Harrison et al., 1948); in a qualitative method the use of such lines in addition to the persistent lines is advisable, especially where the concentration of an element lies on or close to the boundary of demarcation between percentage brackets. The choice of the lines was guided by the various tables in the literature (Waring and Annell, 1952;

Waring and Mela, 1952; Harrison, 1939; Brode, 1943; Harvey, 1947), by considerations of freedom from interference by the lines of closely associated elements, and by checks of the solution standard plates with spectrograms of 1-mg samples of National Bureau of Standards standard samples and other standards available. Due to interference by the cyanogen bands, a number of persistent lines cannot be used, for instance those of K at 4047 and 4044 A and of In at 4511 A.

Table 3 lists the results of chemical microanalyses of some samples originally analyzed by the spectrographic microqualitative method. These samples were available in amounts of only a few milligrams, and the qualitative spectrographic results shown were intended to serve as a guide for the subsequent quantitative chemical analyses. Comparison of the 31 determinations of 13 elements in the eight samples indicates seven disagreements of the spectrographic results with the chemical in the magnitude of one percentage bracket. These disagreements were not considered too serious because it was still possible to identify the minerals from the data at hand.

Table 4 lists a number of the mineral identifications of small single-grain samples that have been accomplished by agreement of the spectrographic microqualitative analyses with the data from the X-ray diffraction patterns. The spectrographic determinations of more than 40 different elements have thus been checked on a qualitative basis.

The appendix tabulates the compositions of the standard solutions (Waring and Annell, 1952).

FUTURE PROGRAM

Plans for further improvement of the microqualitative method include an investigation of the possible application of a controlled arc atmosphere designed to eliminate the cyanogen bands obscuring a large number of persistent lines. The sensitivities for elements such as Cs, Rb, Th, U, and various rare earths might thus be increased. The preparation of standard spectrograms on the basis of 0.5 mg of sample rather than 1 mg, involving the dilution by a factor of two of the present standard solutions, is also being considered. Such spectrograms, by representing a sample weight closer to that usually used, would aid in making the method more nearly semiquantitative.

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Table 1.--Sensitivities on the basis of 1-mg samples for the elements determined by the microqualitative method

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

* A second exposure is required for the fluorine determination.

Table 2.--Wavelengths and sensitivities of the analytical lines used in the microqualitative method

Element	Wavelength (in A)	Sensitivity in percent	Element	Wavelength (in A)	Sensitivity in percent
Ag	3280.683	0.001	Bi	3067.716	0.01
	3382.891	0.01		2938.298	0.1
	5465.487	10.0		2897.975	0.1
				3024.635	1.0
Al	3092.713	0.001		2989.029	1.0
	3082.155	0.01		2780.521	10.0
	2660.393	1.0		2627.906	10.0
	2652.489	1.0			
	2575.100	1.0	Ca	4226.728	0.1
	2567.987	1.0		3179.332	0.1
				3158.869	0.1
As	2349.84	0.1		8542.089	1.0
	2860.452	1.0		8662.140	10.0
	2780.197	1.0		8498.018	10.0
	2898.71	10.0			
	2492.91	10.0	Cd	3261.057	0.1
				2980.628	1.0
Au	2675.95	0.01		2288.018	1.0
	2427.95	0.1		2265.017	1.0
	3122.781	1.0		4799.918	10.0
	2748.26	1.0		2981.34	10.0
	2700.89	10.0		2836.907	10.0
	2641.49	10.0			
			Ce	4222.599	1.0
B	2497.733	0.01		3272.253	1.0
	2496.778	0.01		3056.777	10.0
				3055.243	10.0
Ba	4934.086	0.1		2651.006	10.0
	6141.716	1.0			
	5853.679	1.0	Co	3453.505	1.0
	3071.591	10.0		3412.633	1.0
	2634.783	10.0		3412.339	1.0
				3044.005	1.0
Be	3131.072	0.001		2521.363	1.0
	3130.416	0.001		2663.529	10.0
	2348.610	0.001		2649.940	10.0
	3321.343	0.1		2519.822	10.0
	2350.685	10.0			

Table 2.--Continued

Element	Wavelength (in A)	Sensitivity in percent
Cr	4289.721	0.1
	4274.803	0.1
	4254.346	0.1
	2843.252	0.1
	2835.633	0.1
	2860.934	1.0
	2855.676	1.0
	2769.915	10.0
	2731.908	10.0
Cs	8521.10	10.0
Cu	3273.962	0.001
	3247.540	0.001
	2618.366	1.0
	2492.146	1.0
	2766.371	10.0
	2441.637	10.0
Dy	3407.80	1.0
	3393.583	1.0
	3385.027	1.0
	3280.10	1.0
	3251.260	1.0
Er	3264.781	0.1
	3230.585	0.1
	2964.518	1.0
Eu	2813.95	0.1
	2906.676	1.0
	2727.780	1.0
	2685.65	10.0
	2678.28	10.0
F (CaF band heads)	6064.4	1.0
	6036.9	1.0
	5291.0	10.0
Fe	3020.640	0.1
	2599.396	0.1
	2739.546	1.0
	2598.369	1.0
	3100.304	10.0
	3099.971	10.0
	3099.897	10.0
Ga	2943.637	0.1
	2944.175	1.0
	2874.244	1.0
	2719.653	10.0
	2500.173	10.0
Gd	3100.508	0.1
	3034.059	1.0
	3032.850	1.0
	3027.612	1.0
	2840.236	10.0
	2809.720	10.0
Ge	3039.064	0.1
	2651.575	0.1
	2651.178	0.1
	3269.494	1.0
	2691.344	1.0
	2740.431	10.0
	2589.188	10.0
	2533.229	10.0
Hf	3134.718	0.1
	3072.877	0.1
	3012.902	0.1
	2861.696	1.0
	2861.012	1.0
	2683.353	10.0
	2531.193	10.0
Hg	2536.519	1.0
	3131.833	10.0
	3131.546	10.0
	3125.663	10.0

Table 2.--Continued

Element	Wavelength (in A)	Sensitivity in percent	Element	Wavelength (in A)	Sensitivity in percent
Ho	3456.00	0.1	Mn	2576.104	0.01
	3453.13	1.0		2605.688	0.1
	3416.46	1.0		2593.729	0.1
	3398.98	1.0		2939.304	1.0
				2933.063	1.0
In	3256.090	0.1		3044.567	10.0
	3039.356	0.1		2584.308	10.0
	3258.564	1.0			
	2753.878	10.0	Mo	3193.973	0.1
	2710.265	10.0		3170.347	0.1
				2816.154	0.1
Ir	3220.780	1.0		3208.834	1.0
	2639.712	1.0		3158.165	1.0
	2543.971	1.0		2784.992	10.0
	2534.457	10.0		2763.620	10.0
	2533.131	10.0			
			Na	5895.923	0.1
K	7698.979	0.1		5889.953	0.1
	7664.907	0.1		3302.988	10.0
				3302.323	10.0
La	3337.488	0.1			
	3344.560	1.0	Nb	3225.479	0.1
	3303.11	1.0		3094.183	0.1
	3265.67	1.0		2931.469	1.0
	2808.39	10.0		2876.947	1.0
	2610.335	10.0		2875.392	1.0
				2746.104	10.0
Li	6707.844	0.01		2745.729	10.0
	6103.642	1.0			
	3232.61	1.0	Nd	4303.573	1.0
	2741.31	10.0		4247.367	1.0
				3328.270	10.0
Lu	3077.60	0.1		3275.218	10.0
	2615.42	0.1			
	2619.26	1.0	Ni	3050.819	0.1
	2613.40	1.0		3002.491	0.1
	2685.08	10.0		2992.595	1.0
	2392.19	10.0		2943.914	1.0
				2907.459	10.0
Mg	2852.129	0.001		2798.653	10.0
	2802.695	0.001			
	2795.53	0.001	Os	3058.66	1.0
	2782.974	1.0		2909.061	1.0
	2779.834	1.0		3301.559	10.0
	2776.690	1.0		2838.626	10.0

Table 2.--Continued.

Element	Wavelength (in A)	Sensitivity in percent
P	2553.28	1.0
	2535.65	1.0
	2554.93	10.0
	2534.01	10.0
Pb	2833.069	0.1
	2802.003	0.1
	2614.178	0.1
	2873.316	1.0
	2823.189	1.0
	2663.166	1.0
	2393.794	10.0
Pd	3421.24	0.1
	3242.703	0.1
	3251.640	1.0
	2763.092	1.0
	3002.652	10.0
	2922.492	10.0
Pr	4241.019	1.0
	4225.327	1.0
	5322.778	10.0
	5259.743	10.0
Pt	3064.712	0.01
	2997.967	0.1
	2659.454	0.1
	2650.857	1.0
	2646.886	1.0
	2803.239	10.0
	2639.350	10.0
Rb	7947.60	1.0
	7800.227	1.0
Re	3464.722	1.0
	3460.47	1.0
	2715.470	10.0
	2674.337	10.0
Rh	3434.893	0.1
	3396.85	1.0
	3283.573	1.0
	3280.55	1.0
	3271.612	10.0
	3263.144	10.0
Ru	2874.984	1.0
	2735.718	1.0
	2678.758	1.0
	2886.536	10.0
	2734.349	10.0
Sb	2598.062	0.1
	2877.915	1.0
	2769.939	1.0
	2718.893	10.0
	2670.643	10.0
Sc	2560.227	0.1
	2552.359	0.1
	2555.799	1.0
	2545.204	1.0
	2822.131	10.0
Si	2881.578	0.01
	2516.123	0.01
	2987.648	1.0
	2435.159	1.0
	2532.378	10.0
	2438.782	10.0
Sm	3273.477	1.0
	3272.803	1.0
	3254.378	1.0
	3254.290	1.0
	3253.931	1.0
Sn	3034.121	0.1
	2839.989	0.1
	3009.147	1.0
	2863.327	1.0
	2850.618	1.0
	2661.248	10.0
	2571.592	10.0

Table 2.--Continued.

Element	Wavelength (in A)	Sensitivity in percent
Sr	4607.331	1.0
	3464.457	1.0
	4305.447	10.0
	3380.711	10.0
Ta	2685.11	0.1
	2635.929	0.1
	2714.674	1.0
	2675.901	1.0
	2902.046	10.0
	2891.843	10.0
Tb	4278.51	1.0
	3324.40	1.0
	3219.95	1.0
	3218.93	1.0
Te	2385.76	1.0
	2383.25	1.0
Th	2837.299	0.1
	2832.319	0.1
	2870.413	1.0
	2842.815	1.0
	2703.962	1.0
	2565.597	1.0
	4391.114	10.0
Ti	3241.986	0.01
	3239.038	0.01
	3236.573	0.01
	3234.516	0.01
	3088.025	0.01
	3078.645	0.1
	3072.971	0.1
	3072.107	0.1
	2956.131	1.0
	2941.995	1.0
	2619.939	10.0
	2605.151	10.0
Tl	2767.87	1.0
	5350.46	10.0
	2918.32	10.0
Tm	3133.89	0.1
	3131.26	0.1
	4242.15	1.0
	3462.20	1.0
	3362.61	1.0
U	4244.372	1.0
	4241.669	1.0
	2882.741	1.0
V	3185.396	0.1
	3183.982	0.1
	3183.406	0.1
	3202.381	1.0
	3198.012	1.0
	3214.750	10.0
	3212.434	10.0
W	2946.981	1.0
	2944.395	1.0
	2947.384	10.0
	2589.167	10.0
Y	3242.280	0.01
	3216.682	0.01
	3200.270	0.1
	3195.615	0.1
	2984.256	1.0
	2422.185	1.0
	3086.858	10.0
	3055.22	10.0
Yb	3289.37	0.01
	2970.564	0.1
	3031.11	1.0
	2653.75	1.0
	3107.897	10.0

Table 2.--Continued.

Element	Wavelength (in A)	Sensitivity in percent	Element	Wavelength (in A)	Sensitivity in percent
Zn	3345.020	1.0	Zr	3438.230	0.1
	3302.588	1.0		3391.975	0.1
	3282.333	1.0		2758.813	1.0
	2800.869	1.0		2752.206	1.0
	4810.534	10.0		2889.430	10.0
	2770.984	10.0		2888.036	10.0
	2770.865	10.0			

Table 3.--Comparison of chemical quantitative and spectrographic qualitative microanalyses of samples of uranium minerals (TWC 2050, TWS 712, TWX 319, Lot 3218)

Lab. no. Mineral	75083 Kasolite		75084 Uranophane		75085 Cuprosklodowskite		75086 Soddyite		75087 Beta-uranotil	
Location	Belgian Congo		Yancey, N. C.		Jachymov, Bohemia		Belgian Congo		Jachymov, Bohemia	
Element	Chem %1/	Spec 2/	Chem %1/	Spec 2/	Chem %1/	Spec 2/	Chem %1/	Spec 2/	Chem %1/	Spec 2/
Ca	0.71	Trace	7.3	Minor					5.2	Minor
Cu					5.3	Minor				
Pb	29.0	Major								
Si	4.9	Major	6.2	Major	4.8	Major	4.1	Major	6.1	Major
U	43.5	Major	50.8	Major	53.2	Major	68.0	Major	49.3	Major

1/ Chemical results = oxides reduced to elements

2/ Major, approximately 10 percent or more; Minor, approximately 1 - 10 percent; and Trace, approximately 0.1 - 1 percent

Table 3.--Continued

	Lab. no. 75088		Lab. no. 66779 Lot no. 595 (TWC 1959, TWS 569)		Sample no. JJK/1/52 Lot no. 0-22 (TWC 2052, TWS 786)	
Mineral	Sklodowskite		Brannerite		Calcium uranylvanadate	
Location	Belgian Congo		Dean's Mine, Mono County, Calif.		Small Spot mine, Mesa County, Colorado	
Element	Chem. % 1/	Spec. 2/	Chem. % 1/	Spec. 2/	Chem. % 1/	Spec. 2/
Al					0.2	Trace
Ca	2.5	Minor	2.0	Trace	4.5	Minor
Fe			1.8	Minor		
Mg	1.3	Minor				
Pb	4.9	Minor				
Si	6.5	Major	0.23	Trace		
Th			4.4	Minor		
Ti			19.7	Major		
U	50.3	Major	33.8	Major	49.3	Major
V					12.2	Major
Er			--	Minor		
Y			--	Trace		
Rare earths			6.5			

1/ Chemical results = oxides reduced to elements

2/ Major, approximately 10 percent or more; Minor, approximately 1 - 10 percent; and Trace, approximately 0.1 - 1 percent

Table 4.--Mineral identifications accomplished by agreement of spectrographic microqualitative analyses with data from X-ray diffraction patterns

Minerals for standard X-ray patterns

(TWS-738, Lot 0-70)

Sample no.	T. E. X-ray film no.	Mineral identification	Spectrographic report 1/		
			Major	Minor	Trace
EC-52	2286	Azurite	Cu	--	Si Al
EC-53	2287	Chrysocolla	Cu Si	Al	Mg
EC-54	2293	Apophyllite	Ca Si	K F	Al Na
EC-56	2294	Cerussite	Pb	--	Ca
EC-59	2313	Franklinite	Zn	Fe Mn	Al
EC-64	2314	Pyrargyrite	Ag Sb	Si	Zn Cu
EC-65	2315	Realgar	As	--	Ca
EC-76	2283	Talc	Mg Si	--	Fe Al

1/ Major, approximately 10 percent or more; Minor, approximately 1 - 10 percent; and Trace, approximately 0.1 - 1 percent.

Table 4.--Continued.

Uranium-bearing minerals

Sample no.	Lot no.	T. E. X-ray film no.	Mineral identification	TWS Report	Spectrograhic [1]		
					Major	Minor	Trace
FK-26F	1193	1986	Quartz and carnotite	549	Si	U V Mg Fe	Na K Al
YC-1-51	1635	1975	Tyuyamunite and clay (?)	542	U V Si	Ca Al Fe	Mg Cu
J-59	1128	2389	Calcium uranylvanadate	770	U V	Ca Si Na	Mg Al
ADW-170	0-20	1919	Rauvite	533	U V	Ca	Al Ti Ba Si
T-38a	1128	2476	Rauvite and quartz	789	U Si V	Ca	Al Na Fe
DN-31A	1708	2067	Uranophane and phosphuranylite	597	U Si	Ca Al Na K P As	Mg
J-102C	1128	2424	Autunite	774	U	P Ca Si Na	Cu Mg Fe Ba Al
CS-3-4-1(1)	1629	2244	Autunite-uranocircite	677	U	P Si Ba Ca	Na Fe Mg Al
J-103	1128	2409	Autunite, saleeite, and torbernite	774	U P Ca	Mg Si Na	Cu Al Fe

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[1] Major, approximately 10 percent or more; Minor, approximately 1 - 10 percent; and Trace, approximately 0.1 - 1 percent.

Table 4.--Continued

Uranium-bearing minerals

Sample no.	Lot no.	T. E. X-ray film no.	Mineral identification	TWS Report	Spectrographic 1/ Major	Minor	Trace
J-81	1128	2346	Zeunerite	770	U As	Cu Ba Si	Na
J-102B	1128	2423	Torbernite	774	U	P Cu Si Ca Na	Fe Mg As K Ba Al
4493-C(3)	551	2251	Uraninite and smaltite-cloanthite	690	U	Si As Co Ni Fe Ba	Cu Na Y Al Mg
2904-G(1)	551	2405	Huttonite	761	Th Si U	Ca Fe Na	Pb K B Mg Al Mn
4419-H(3)	551	2166	Brannerite, thorite, and samarskite	727	Th Ti	U Si Nb Y	Fe Bi Na Ni Ca

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1/ Major, approximately 10 percent or more; Minor, approximately 1 - 10 percent; and Trace, approximately 0.1 - 1 percent.

Table 4.--Continued

Rare-earth minerals

Sample no.	Lot no.	X-ray film no.	Mineral identification	TWS Report	Spectrographic 1/		
					Major	Minor	Trace
JNS-2	0-1	T. E. 2498	Allanite	778	Si Fe	Al Ce La Ca Na Mg Th Nd Pr	Mn K
P-59-Ba	0-51	T. E. 1692	Bastnaesite	522	Ce	La Y Ca Al Fe Si P	Mg Mn Pb Th
51P-72a	1613	T. E. 2266	Florencite	687	P Al Ce	Si Th La Fe Ca Zr	Ti Gd Pb Y Ba K
51P-72b	1613	T. E. 2272	Florencite and rutile	687	P Ti	Si Ce Al Zr La	Th Pb Fe K Ba Y Ca
4428	3036	T. E. 2192	Parisite	658	Ce La	Ca Nd	Fe
U.S.N.M. no. 80183	0-70	B-255 and B-256	Cyrtolite	746	Zr Si Hf	Y Fe Ca	U Mn Th Na Al Yb Gd B

1/ Major, approximately 10 percent or more; Minor, approximately 1 - 10 percent; and Trace, approximately 0.1 - 1 percent.

Table 4.--Continued

Miscellaneous minerals

Sample no.	Lot no.	T. E. X-ray film no.	Mineral identification	TWS Report	Spectrographic 1/		
					Major	Minor	Trace
J-102A	1128	2426	Quartz and hydromica	774	Si	K Fe Al Mg Na	Ca Ti
W-88	1128	2477	Kaolinite	789	Si Al	K Mg Na	Ca Fe Ti
AT-20	1126	2367	Gibbsite	757	Si Al	Na	Mg Ca K Cu Fe Ni
4099	3035	2043	Topaz and impurities	576 and 579	Al Si	F Pb	Ca Fe
JNS-1	0-1	2479	Biotite	778	Si Fe	Al Mg K Ca Na	Li Ti
J-56	1128	2377	Apatite and hydromica	770	Ca Si	Fe Mg K P Al Na	Mn V Ti Ba
ADW-173	0-20	1922	Hewettite	533	V	Ca	Ti Ba Si
4477-A	551	2148	Melanovanadite, quartz and barite (?)	723	V	Ca Si Ba	Fe Mg Na Ti Al

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1/ Major, approximately 10 percent or more; Minor, approximately 1 - 10 percent; and Trace, approximately 0.1 - 1 percent.

Table 4.--Continued

Miscellaneous minerals

Sample no.	Lot no.	T. E. X-ray film no.	Mineral identification	TWS Report	Spectrographic 1/ Major	Minor	Trace
51-AWe-107	3035	2023	Vosenite	571	Fe Mg B	Mn Si	Ca Na Ti Ni
51-P-67	1613	2246	Hematite, rutile, and ilmenite	687	Fe Ti	Si Mn	Na Mg Al Ca
51-P-67m	1613	2241	Magnetite	687	Fe	Si Na	Ti Mg Al Mn Ca
J-102D	1128	2393	Goethite	770	Fe	Si Na	Cu Ca Al K Mg
ADW-186	0-20	1913	Conichalcite and quartz	561	Cu Ca As Si	V	Al Mg Mn Ba
4385	3036	2193	Orpiment	655	As	--	Al Mn Si Ti V
K-35-3	1637	2486	Sphalerite	787	Zn Si	Mg Na	K
WE-186A	3035	2196	Pyrochlore-microlite	654	Ca Ta	Nb Na	Fe Mn Si Ti

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1/ Major, approximately 10 percent or more; Minor, approximately 1 - 10 percent; and Trace, approximately 0.1 - 1 percent.

APPENDIX

(Waring and Annell, 1952)

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.	
Cd	$CdCl_2 \cdot 2\frac{1}{2}H_2O$, C. P.	
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.

Element standardized	Compound used	Solution
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.
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.

Element standardized	Compound used	Solution
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	
Nb	Nb, metal, J and M	48 percent HF. Diluted to volume with HNO_3, conc.
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.
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.

Element standardized	Compound used	Solution
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.
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.

Element standardized	Compound used	Solution
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.	