SPECTROCHEMICAL ANALYSIS OF ROCKS AND MINERALS.*

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ABSTRACT. Spectrochemical methods used in the U. S. Department of the Interior, Geological Survey, laboratory are adapted to the problems of rock and mineral analysis. About 70 chemical elements are determined major constituents qualitatively, minor constituents quantitatively. The finely powdered sample, weighing 20 mg. or less, is burned in a carbon anode, using the direct current arc. Because of the small size of the sample and the inherent sensitivity of the method, sampling errors and trace contamination must be avoided. The spectrograms are complicated by background lines from carbon and from silicon monoxide and cyanogen bands.

Comparison standards used for analyses of siliceous rocks and silicate minerals are generally prepared by incorporating the desired element, preferably in the form of a natural mineral, into a synthetic rock-like mixture of feldspar, quartz, and iron oxide. Intensity of spectral lines of an element is influenced by its state of chemical combination and the nature of the matrix. For accurate results standards must closely approximate the "unknown" sample in chemical and mineralogical composition. Sensitivity of detection varies with the elements. Among the limiting factors may be inherently weak spectral lines, extreme volatility, extreme refractoriness, and interference by lines of other elements present. Tables of sensitivity data are included.

INTRODUCTION.

THIS paper, originally prepared as a talk before the Geological Society of Washington, is written to acquaint geologists with the field of spectrochemical analysis as applied to rock and mineral studies and to stimulate appreciation of the possibilities and limitations of the method. It is not addressed to experienced spectrographers. Information presented in the paper is based on work of the spectrographic laboratory of the Geological Survey, U. S. Department of the Interior.

Today some 70 chemical elements, including many rare earths, can be detected and determined quantitatively in amounts as low as .0001%, and occasionally even lower. Our upper limit for quantitative spectrochemical analysis rests at about 1%, although some laboratories have evolved techniques to determine quantitatively the major constituents in alloys and certain types of ores.

The spectrum emitted by an element is characteristic of that element only, although the intensity and number of spectral

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lines may vary somewhat depending on the energy conditions developed by the particular mode of excitation. Emission spectra are obtained from atoms and molecules that acquire excess energy while in a gaseous or vapor state and then release it as radiant energy. The excess energy may be supplied by an ordinary Bunsen burner flame, such as is used in simple tests for mineral identification, or it may be obtained from an electric arc which creates considerably higher temperatures, or from an electric spark which supplies even higher temperatures and electrical excitation as well. The direct current carbon arc has been widely used for non-conducting materials such as siliceous ores because it is simple to operate and at the same time allows determination of very low concentrations of many elements.

Quantitative spectrochemical analysis is based on the fact that the intensity of light emitted by an element in the arc is a function of the concentration of that element. The degree of blackness, or the density, of the spectral lines produced on the photographic plate is a measure of the concentration of the element emitting light of that particular wavelength. Correction is made for variations in sensitivity of photographic plates and processing by means of calibration standards, whose spectra are exposed on every plate.

Our procedure for spectrochemical analysis of minerals, ores, and rocks is to place 10 to 20 mg. (smaller samples can also be handled) of finely powdered sample in a crater bored into one end of a carbon rod, then burn the sample to completion in the direct current electric arc. The light emitted by the central portion of the arc column is passed through the spectrograph and the resulting spectrum recorded photographically. The spectrograms on the plate are then compared with reference spectra of materials of known composition, usually exposed on the same plate, the comparison being made either by eye or with a photoelectric device known as the densitometer. The spectrographic method, in its use of reference standards, has a basic similarity to colorimetric methods of chemical analysis.

Various other procedures for carrying out spectrochemical analysis are in use which differ from that of the Geological Survey in important details. All statements in this paper refer to the procedure briefly outlined here, and to results obtained by application of this procedure.

FUNDAMENTAL PRECAUTIONS.

Because of the small size of the sample employed, sampling errors are highly significant. Wet chemical analyses are usually made on one gram samples. For spectrographic work only 20 mg. are used. In fact, as little as a few tenths of a milligram of material will often suffice for a spectrochemical analysis. If the sample has not been quartered down carefully, or if it is not homogeneous and finely powdered the values will be unreliable. Depending upon the nature of the material and what he wishes to know about it, the geologist must, of course, sample the material properly in the field whether it is to be analyzed spectrographically or chemically.

Another problem that is particularly worrisome to the spectrographer is contamination. Again the relative size of the sample is a factor, but even more significant is the inherent sensitivity of the method. Sieving can cause serious difficulties because traces of copper from the wire, and tin and lead from the solder, are often picked up in the process. As a result, the determination of very small amounts of these elements in sieved samples cannot be exact. Then too, the ordinary carbon electrodes used are never absolutely pure. Small amounts of magnesium, copper, vanadium, boron, or calcium may be present. Even the purest spectroscopic carbon or graphite electrodes carry traces of magnesium, copper, and lead.

The accuracy aimed for in routine examinations of ores is to obtain one significant figure, which is adequate for most of the problems confronting Survey geologists. In special cases where greater accuracy is required and the expenditure of time for preparing and studying special standards is justified, we can attain two significant figures.

BASIC SPECTRAL FEATURES.

The spectra of some elements commonly encountered in rock analysis are shown in Plate 1, Fig. 1. The spectra on this plate were taken with a Gaertner medium-size quartz spectrograph, which has a small dispersion, allowing the entire spectral range from 2200A to 6800A to be recorded on a single plate.[1] Because of limited space, however, the spectral regions below 2350A and

[1] An Eastman panchromatic "F" sensitized plate was used for this illustration. With an "L" sensitized plate the range can be extended up to 8600A. Unsensitized "O" plates limit the spectrograms to 5000A.

above 4500A do not appear in the reproduction in Plate 1, Fig. 1. The scale is calibrated in millimicrons. As wavelengths are usually reported in Angstrom units, the scale reading is multiplied by 10 to obtain the Angstrom value. The ultraviolet region, at the left, extends up to 4000A; the visible region, from 4000A to 8000A; the infrared lies beyond, to the right.

The first three spectra are of the carbon arc alone, exposed for 20, 40, and 80 seconds, respectively. The carbon spectrum always appears with that of the sample when carbon electrodes are used. (Only rarely are other types of electrodes used.) Up to 3450A the carbon arc spectrum is almost blank except for the single line at 2478A. The sensitive lines of a majority of the elements lie in this clear region. However, with prolonged exposure even this area develops closely spaced background lines of carbon, which show as a group of faint lines to the left of the broad black bands. A comparison of exposures 1, 2, and 3 (20″, 40″, 80″) shows these background lines becoming darker and more numerous as the time of exposure is increased.

In the violet, between 3450A and 4200A, there are three sets of dark, broad molecular cyanogen bands, which are produced by the interaction of the nitrogen in the air with the hot carbon of the electrodes. It is practically impossible to work with this portion of the spectrum, although elements like cobalt, columbium, lead, molybdenum, and tungsten have sensitive lines in this region. Fortunately, these elements have good lines elsewhere. The rare earth elements, on the other hand, have many of their best detection lines in the region of the cyanogen bands. For detailed study of these elements copper electrodes can be used, thus eliminating the bands entirely. The grating spectrograph, with its greater dispersion,[2] is likewise helpful for unravelling the complex rare earth spectra.

Other cyanogen bands occur in the visible region to the right of the three heavy bands, but these are weaker and do not cause serious interference. The most sensitive lines of the alkalies and the alkaline earths are located in this area of the visible and near infrared. For such determinations panchromatic

[2] Our Jarrell-Ash grating spectrograph provides a dispersion of 5A per mm. in the first order spectrum, 2.44 per mm. in the second order, and 1.6A per mm. in the third.

plates with specially sensitized photographic emulsions must be used.

As the earth's crust contains about 60% of silica, it is not surprising that silica is encountered in most rock and mineral analyses. The second set of three spectra (nos. 4, 5, 6 in Plate 1, Fig. 1) shows silica alone, exposed for 20, 40, and 80 seconds. Silica introduces an extensive system of silicon monoxide bands, particularly between 2200A and 2500A, and may cause considerable interference in this region if present in large amounts. In the ultra-violet region, between the silicon monoxide bands on the left and the cyanogen bands on the right[3], sensitive lines of 45 of the 70 determinable elements are located. This is particularly fortunate because there is only moderate background interference from silica in this region.

Aluminum, iron, magnesium, and calcium are also of frequent occurrence in rocks and minerals. The aluminum spectrum (no. 7 in Plate 1, Fig. 1) has very few lines; iron (no. 8) has a complex spectrum with many lines. The wavelengths of the lines in the iron spectrum have been so carefully measured by spectrographers that it has become standard procedure to employ the iron spectrum as a guide or scale in locating and identifying lines of "unknown" elements. The three spectra numbered 9, 10, and 11 (Plate I, Fig. 1), likewise exposed for 20, 40, and 80 seconds, are those of a synthetic mixture of quartz, feldspar, and iron oxide, in proportions approximating the composition of a granite. This base mixture is used in preparing standard series of various elements. It may seem surprising that lines of the rarer elements in such complex spectra can be identified and measured without interference from some or all of the common elements. The "X" spectrum (no. 12 in Plate 1, Fig. 1) is that of a synthetic mixture containing many of the elements looked for in rocks, and is used for reference and orientation.

COMPARISON STANDARDS.

Plate 2, Fig. 2 shows a limited region of some spectra taken with the large Littrow quartz spectrograph. The dispersion

[3] This area of the spectrum is approximately demarcated on Plate 1, Fig. 1 by the arrows at the bottom. The "5" refers to the focal setting of the large Littrow quartz spectrograph when it is used to photograph this region.

of this instrument[4] is three times greater than that of the first, and only one region of the spectrum can be photographed at a time, depending on the focus adjustment. The scale on this instrument is arbitrary and does not indicate wavelength. Plate 2, Fig. 2 includes only a small portion of the ultraviolet region where several good analysis lines of nickel and vanadium are located.

In Plate 2, Fig. 2 are a series of comparison standards for determining nickel and vanadium. For reference, spectra of pure nickel, pure iron, and pure vanadium are included. Of the great number of lines in the vanadium spectrum, only a few can be utilized. The ideal analysis line of an element is sensitive to variations in percentage composition over a wide range, and lies in a region free from interference by lines of other elements. Below these (in Plate 2, Fig. 2) follows a graded series of spectra of silicate mixtures containing 1%, .1, .01, etc., of nickel oxide and vanadium oxide, then the silicate base alone, and last the X mixture.

The most sensitive lines of nickel in the region shown in Plate 2, Fig. 2 are at 30 (3050A) and a doublet at 40.5 (3102A). Nickel can be detected at .0001% as a very faint line. Just beside the nickel doublet is a strong iron triplet which becomes very broad and black in iron-rich materials and makes it difficult to read the neighboring nickel line. The good vanadium lines consist of a set of three at 42.5, 44, and 45.5 (about 3110A) and a triplet at 57 (3180A). The vanadium line nearest the nickel fades out at .001%, the other two disappear at .01%. The triplet is good down to .001% also. The standard for .0001% has been duplicated to show the reproducibility of a spectrum. No lines of nickel or vanadium appear in the base. High purity electrodes were used to obtain these spectrograms. If ordinary electrodes had been used the vanadium lines would never die out, because of the vanadium impurity in the carbon.

Whenever possible, each element is incorporated into the standard mixture in the form of a mineral, for example—boron as tourmaline and tungsten as scheelite. There are very good reasons for this rather tedious procedure. It has been

[4] Dispersion with quartz prism instruments is non-linear; that is, the dispersion decreases toward the higher wavelength regions. The large spectrograph has a dispersion of 2.5A per mm. at 2500A, 7.0A per mm. at 3500A, and 20A per mm. at 5000A.

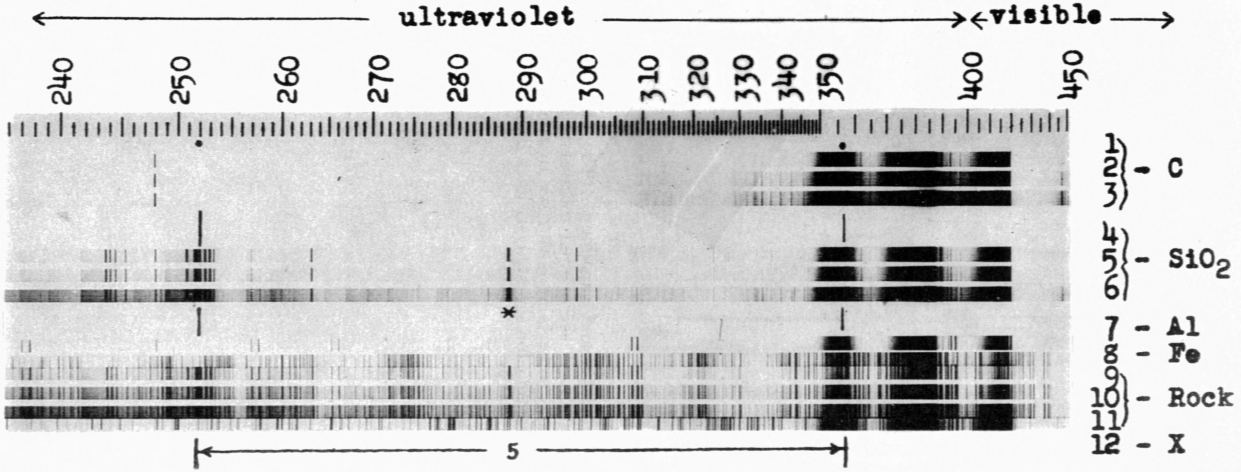

Fig. 1. Some basic spectra. (Spectrographic plate prepared by K. J. Murata, using a Gaertner medium-size spectrograph.) Only wavelengths between 2350A and 4500A are shown. Scale calibrated in millimicrons. Broad black bands at the right are cyanogen. Arrows at the bottom, and the "5", indicate focal setting necessary to photograph this important region with the large Littrow spectrograph. Heavy lines between sets of spectrograms are merely additional reference points, as are the two dots just below the scale at 252 and 355, to mark off the upper and lower limits of this same region. Star locates useful silicon line.

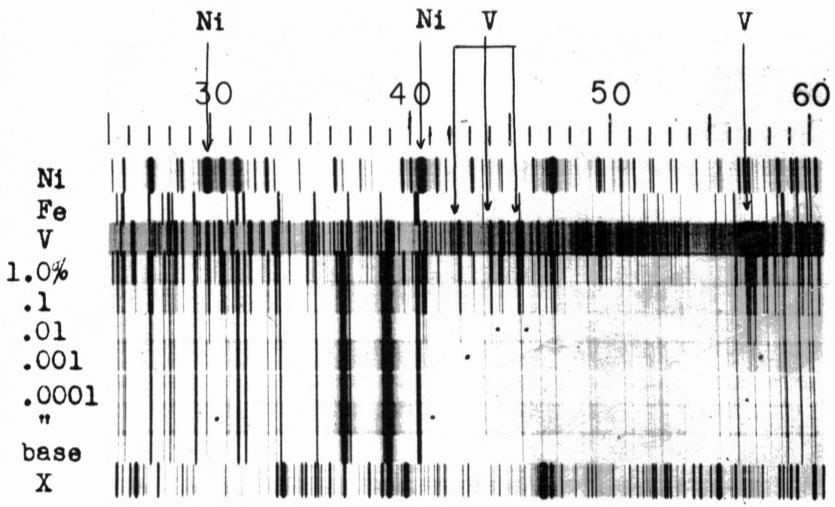

Fig. 2. Comparison standards for nickel and vanadium. (The photographic processing necessary to prepare this figure from the original spectrographic plate has caused a blurring of the background and some faint lines have disappeared entirely.) Dots mark the lowest limit of detection for the analysis lines indicated.

found, time and again, that the state of chemical combination of an element as well as the nature of the matrix have great influence on the intensity of spectral lines.

Just recently we have had occasion to analyze several samples of ludwigite, a ferroan magnesium ferric-iron borate mineral, for tin. By using two series of comparison standards, one a tin-free silicate base and the other a natural tin-free ludwigite base, curves of distinctly different slopes were obtained, as may be seen in Text Fig. 3.

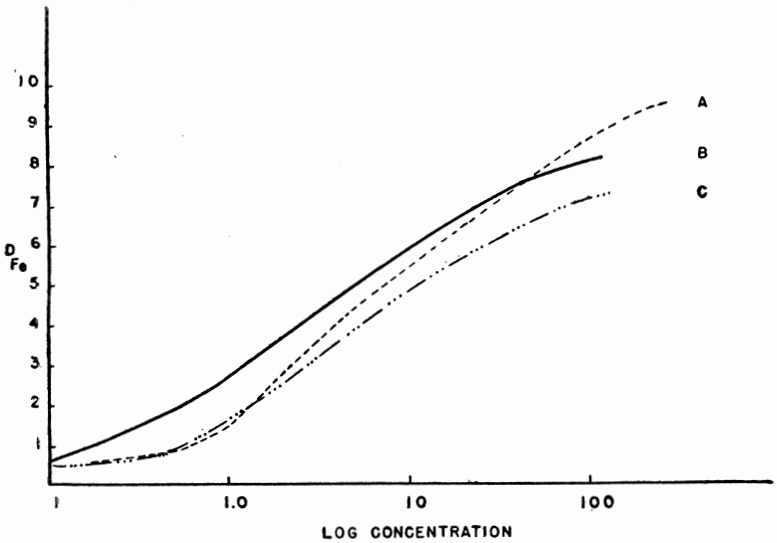

Figure 3. Effect of matrix and state of combination on spectral line intensity of tin. Concentration expressed as micrograms of Sn per sample in electrode (average sample weight, 24 mg.). *A*, tin-free silicate base, with reagent SnO_2; *B*, tin-free ludwigite base, with tin as tin-bearing hulsite; *C*, tin-free ludwigite base, with reagent SnO_2. The ordinate, D_{Fe}, represents line density, using a graded series of Fe lines as comparison standards. (Data by K. J. Murata.)

In the silicate standards, tin had been incorporated as reagent-grade tin oxide in the synthetic mixture of quartz, feldspar, and iron oxide usually employed as a base. In the ludwigite standards tin was incorporated in the form of a chemically analyzed tin-bearing hulsite (magnesian iron borate mineral) in a tin-free ludwigite base. Another, intermediate series of ludwigite standards was also prepared, in which tin as reagent tin oxide was incorporated in a tin-free ludwigite base. In each standard sample the same spectral tin line

(3034.121A) was compared against a graded set of iron spectra, and the density or blackness of the lines was noted. These values were then plotted against the tin concentration.

For any given concentration of tin the density of the spectral lines (and the apparent concentration of the tin) varied with the nature of the standards. Thus, if the percentages of tin in samples of ludwigite had been computed on the basis of the usual silicate standards the values reported would have been accurate at one point only, where the ideal ludwigite (B) in Text Fig. 3 and the silicate (A) curves intersect. Nor would the error be uniform over a wide range in concentration. It will be noted that the two ludwigite curves (B & C) are essentially parallel, showing the effect of similarity in matrix. The spread between the two is due to the difference in the state of chemical combination of the tin.

The need for strictly comparable standards is quite evident, as is the advisability of preliminary petrographic examination of samples to be spectrographed. Not all elements seem to be as sensitive to variations in matrix composition as tin. Many factors are involved, such as relative ionization potentials, vapor pressure, and state of combination of the various elements. (See "References" for Scott's work in this field.)

SENSITIVITY OF DETECTION.

In Text Fig. 4 is shown an extended form of the periodic table. At the extreme left are the alkali metals and alkaline earths; the rare earths at the bottom; the heavy metals near the center; at the right the non-metallic elements, including the halogens and rare gases. Asterisks indicate elements so rare that spectrographic data are not yet available.

The broken line encloses elements that cannot be determined by direct current arc excitation. At present we make no attempt to determine these spectrographically, excepting fluorine which may be determined by means of the CaF^+ molecular band spectrum formed by interaction of fluorine and calcium. Heavy solid blocks enclose elements of high sensitivity. The alkaline earths and the alkalies, except for potassium, rubidium, and cesium, may be detected at .0001% or less. Potassium, rubidium, and cesium lines do not record well because present-day photographic emulsions are not particularly sensitive in the far red portion of the visible spectrum. Those elements

(in Text Fig. 4) not enclosed have average sensitivity, and may be determined down to .001%. Light solid blocks enclose elements of poor sensitivity, of the order of .1% to .01%. Such elements are largely characterized by inherently weak spectral lines. Some are extremely volatile, like arsenic and

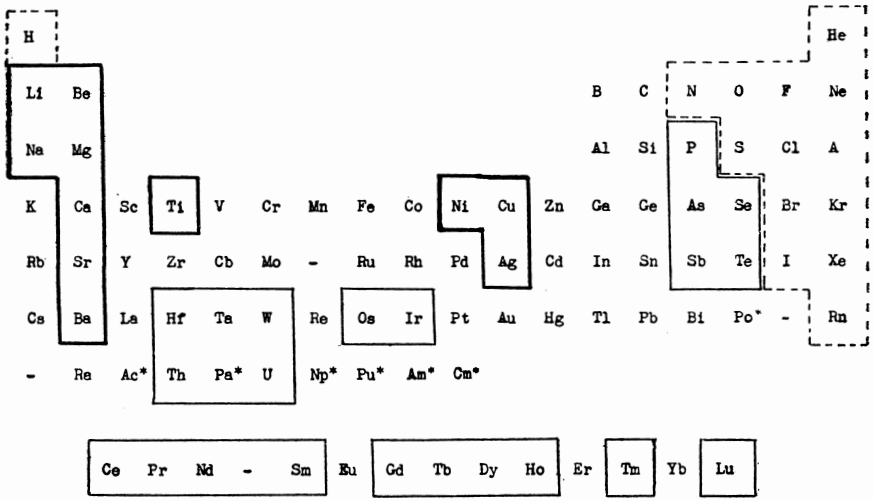

Figure 4. Periodic chart of the elements showing spectrographic sensitivity. (Data by K. J. Murata.)

antimony; others like tantalum, tungsten, and zirconium, are almost impossible to burn to completion.

None of the sensitivities indicated on the chart are absolute limits, but merely indicate present attainments with the large Littrow quartz spectrograph on siliceous materials under the most favorable conditions.

An interesting relation between sensitivity and dispersion is seen in the fact that the large Littrow quartz spectrograph, with its threefold greater dispersion, is in general ten times more sensitive than a medium-size quartz spectrograph. The

greater dispersion seems to result in a dilution of the background in spectrograms, which allows relatively longer exposures to be made with the same photographic emulsion, this in turn allowing much fainter lines to become photographically developed.

Table I lists the minor elements found in rocks and minerals. Beside each element appears the lowest percentage that we can detect with the ordinary procedure used in looking for as many elements as possible in a single spectrogram. Discrepancies between the values on this table and those shown on the periodic chart (Text Fig. 4) will be noted. For example, we cannot determine mercury at all by ordinary methods. It is so volatile that special techniques must be used, when it can be detected at about .001%.

TABLE I.

Spectrographic Sensitivity, Under Ordinary Working Conditions, of Some Minor Elements in Rocks and Minerals.

Ag*	.0001%	Na	.0005%
As	.05	Ni	.0001
Au*	.001	P	.5
B	.001	Pb	.005
Ba	.0001	Pd*	.001
Be	.0001	Pt*	.001
Bi	.001	Rb	.01
Cb	.001	Re	.005
Cd	.01	Rh*	.001
Ce	.1	Sb	.01
Co	.001	Sn	.001
Cr	.001	Sr	.001
Cs	.05	Ta	.1
Cu	.005	Th	.05
Ge	.001	Ti	.0001
Hg*	–	Tl	.005
In	.001	U	.1
Ir*	.01	V	.001
K	.001	W	.01
La	.01	Y	.005
Li	.0005	Yb	.0005
Mn	.001	Zn	.01
Mo	.001	Zr	.01

* Elements capable of greater sensitivity with special techniques. (Data by K. J. Murata.)

In the case of copper, although its lines are very sensitive, we cannot ordinarily go below about .002% because of its presence in even the purest commercial graphite electrodes. Iron interferes with molybdenum determinations in that the sensitive lines of molybdenum coincide with weak iron lines, so .005% molybdenum is the safe lower limit for iron-rich materials. Improvement in the sensitivity of detection of the rare earth elements, such as lanthanum and cerium, may be expected with the use of the new grating spectrograph, which provides greater dispersion.

Platinum may be detected down to about .001% directly in siliceous rocks. Lower concentrations are determined by spectrographing silver assay beads prepared through fire assay methods in which the precious metals of a 29-gram sample, or one assay ton, are gathered into a bead weighing but a few milligrams. By such a combination of techniques concentrations of platinum as low as 10^{-6}% of the original sample may be determined.

The spectrographing of silver assay beads for their content of precious metals is but one specific example of methods involving preliminary chemical concentration. When attempting to determine trace elements in amounts at or below the ordinary limits for spectrographic detection, preliminary chemical treatment should always be considered. By a suitable chemical reaction, traces of an element can often be separated from the bulk of extraneous material in a large sample and concentrated in a small amount of precipitate or solution. In addition to increasing the relative concentration of the element sought, such treatment offers the added advantage of a uniform matrix, since in any one reaction the element always appears with the same reagent and accompanied by the same group of elements. The sensitivity of every trace element can be greatly increased by such preliminary chemical concentration although it may not always be practicable.

A TYPICAL ANALYSIS.

A typical spectrochemical analysis is shown in Table II. It is presented to illustrate the way spectrochemical determinations may be employed to help solve geological problems. In this case, the problem confronting the geologist was the possibility of a genetic relationship between a lateritic soil and an

underlying limestone. Two samples each of the soil and the limestone were spectrographed and analyzed for the constituent minor elements.

TABLE II.

Spectrographic Analysis of Minor Elements in Limestones and Associated Soils.

	1		2	
	Limestone %	Soil %	Limestone %	Soil %
TiO_2	NF*	3.	.0002	2.
ZrO_2	NF	.04	NF	.04
Cr_2O_5	.004	.08	.004	.1
V_2O_5	.003	.05	.006	.02
NiO	NF	.007	.0003	.009
CoO	NF	.005	NF	.005
MnO	.001	.5	.0005	.5
SrO	.006	.0005	.006	.0003
BaO	NF	.0006	NF	.0005
La_2O_3	NF	.03	NF	.01
Y_2O_3	NF	.03	NF	.008
Yb_2O_3	NF	.002	NF	.0005
BeO	NF	.0008	NF	.0006
Ga_2O_3	NF	.001	NF	.001

Looked for but not found: W, Mo, Sn, In, Tl, Ge, Zn, Cd, Pb, As, Sb, Bi, Cu, Ag, Pt, Re, Cb, Ta.

* NF means "not found." The element may be present in amounts below the limit of sensitivity of the method.

For this study special comparison standards were prepared, using a spectroscopically pure calcite base to which was added varying amounts of trace elements. "NF" is indicated wherever the presence of the element could not be detected in the particular sample. The sensitivity of the elements differs widely, as shown diagrammatically in Text Fig. 4. Up to .01% lanthanum could escape detection, whereas nickel would show up at .0001%. Consequently, although no rare earths are reported in the limestone, it does not prove that very small amounts may not be present. Listed separately below are elements that could not be found in any of the samples.

If it be assumed that the soil is derived from the limestone, a very definite trend becomes apparent, and one which would be expected—the concentration in the soil of those elements that are rather inert and difficultly soluble, namely titanium, zirconium, and the rare earths. As strontium forms relatively

soluble compounds, it shows an opposite trend. Manganese is usually present in limestone as readily dissolved manganous carbonate, so it would seem as if oxidizing conditions had been present to allow for its concentration, say as insoluble manganese dioxide, in the soil. This assumption is supported by the presence of much red iron oxide in the soil. Iron, being a major constituent, was not determined.

This example shows how spectrographic analysis for minor elements has furnished the geologist with information about some fifteen elements, over and above the major elements disclosed by chemical analysis, and how the consideration of the properties of these minor elements may throw additional light on the chemical aspects of a geological problem.

REFERENCE MATERIAL.

A few references, selected for their geochemical interest or their general treatment of fundamentals, have been appended. The Eastman Kodak publication describes the fundamental properties of photographic films and plates suitable for spectrographic analysis. Sawyer's "Experimental Spectroscopy" is an excellent basic reference on techniques and instruments, largely non-mathematical in treatment. Standen's paper deals particularly with zinc ores and alloys. The data included in his tables, however, as well as the general discussion, should be of value to all geologists interested in the spectrochemical method. Strock's paper on the cathode-layer method is an outgrowth of the geochemical research program initiated by Professor V. M. Goldschmidt at the University of Göttingen. Strock discusses in detail the problems of spectrochemical analysis of rocks and minerals utilizing the cathode-layer effect in the carbon arc method.

In conclusion, it may be emphasized that spectrochemical analysis is a valuable research tool when applied with due consideration for the limitations inherent in the method and those imposed by the wide and variable composition of rocks and minerals. Great possibilities undoubtedly lie ahead both in perfecting the method and in its application to geology.

ACKNOWLEDGMENT.

The writer is indebted to Mr. K. J. Murata, in charge of the spectrographic laboratory of the U. S. Geological Survey, for

advice and criticism and to Dr. Waldemar T. Schaller, Chief Chemist, for critical review of the manuscript.

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