

THE SPECTROSCOPE IN DETERMINATIVE MINERALOGY¹

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ABSTRACT

The applications of the spectroscope in determinative mineralogy are discussed. It can be used advantageously as a replacement for chemical and blowpipe tests in the identification of most metallic elements. By means of a direct-current arc the mineral sample is volatilized from a graphite electrode and the metallic constituents are determined objectively by visual observation of their spectral lines. Small amounts of either valuable or undesirable metal constituents in certain minerals may be detected. Elements normally difficult to differentiate chemically often are identified easily. The method is limited to the visible region of the spectrum and does not serve to detect most anionic constituents. A laboratory set-up of the instrument, its calibration and its operation are described.

INTRODUCTION

Minerals are identified on the basis of their chemical and physical properties. Fundamentally, an identification depends upon the determination of a sufficient number of properties to differentiate the mineral from all others through a process of elimination. The constants obtained through the use of the microscope often are sufficient to identify the mineral, but recourse frequently must be made to the determination of its chemical composition and *x*-ray diffraction pattern.

Qualitative chemical tests either by use of the blowpipe or wet methods may be inconclusive unless the unknown is carried through an entire qualitative scheme of analysis. In the latter case the procedure is time-consuming.

As a rapid and satisfactory approach to the problem of the determination of metallic ions in minerals, the Bureau of Mines laboratory, Metallurgical Branch, at College Park, Maryland, for the past ten years has been using the spectroscope and spectrograph as routine analytical tools.

Lee and Wright (6) have discussed the usefulness of the spectrograph in mineral analysis, and numerous other papers have been published on the spectrographic determination of elements present in various minerals. The use of the *spectroscope* as a routine tool in determinative mineralogy apparently has not been emphasized.

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As shown in Fig. 1, over half the elements yield spectra in the visible range when subjected to arc excitation. Thus most of the common and many of the rarer metallic elements occurring in any appreciable concentration in minerals can be rapidly determined.

H																				He					
Li	Be																			B	C	N	O	F	Ne
Na	Mg																			Al	Si	P	S	Cl	A
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr								
Rb	Sr	Y	Zr	Cb	Mo	43	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe								
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	86	Rn								
87	Ra	Ac	Th	Pa	U																				
		Ce	Pr	Nd	61	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu										

FIG. 1

- A—The elements enclosed within the boundary lines yield spectral lines in the visible region.
- B—Fluorine may be detected by adding calcium to the sample (if it does not already contain that element) and observing the CaF_2 band (8).
- C—Boron may be detected by the green color appearing on the test screen due to the BO molecule (10).

THE SPECTROSCOPE AND ACCESSORIES

The set-up of the apparatus used in this laboratory is shown in Fig. 2. The light source (A) consists of a direct-current arc produced between two graphite electrodes. Power at 220 volts is supplied by a motor-generator set. On the wiring diagram for the arc, shown in Fig. 3, (R) is a variable rheostat used to control the power input to the arc and is capable of dissipating approximately 2500 watts. A carbon compression type of rheostat is recommended, since it will give stepless control of the current within the working range of 3 to 15 amperes. The ammeter (A) has a total range of 25 amperes with subdivisions of $\frac{1}{2}$ ampere. The gap (G) between the graphite electrodes is 3 mm.

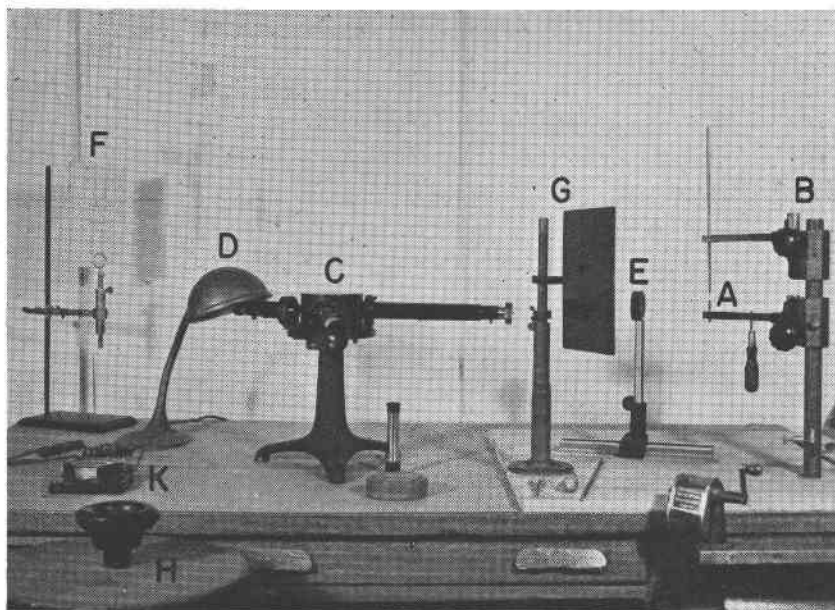


FIG. 2. Arrangement of the apparatus.

- A. Arc
- B. Arc stand
- C. Spectroscope
- D. Light for illuminating the scale
- E. Double convex lens
- F. Electrode positioning screen
- G. Metal shield to protect operator from direct radiation of the burning arc
- H. Rheostat
- K. Ammeter

The arc stand (B in Fig. 2) consists of an upright bakelite post with two steel spring clip arms attached in a horizontal position. These hold the graphite electrodes. Each electrode clamp is connected to a rack and pinion assembly mounted on the bakelite post and actuated by insulated knobs so that the electrodes can be spaced vertically by mechanical

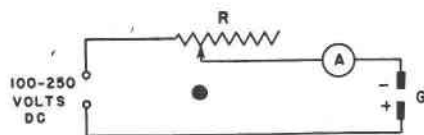


FIG. 3

manipulation while the arc is burning. A permanently mounted clip spreader is used to spread the clip arms when the lower electrode is being changed. This tool is made from a screw driver by cutting off the steel portion approximately 2 inches above the handle. Two flat parallel surfaces are machined on the metal to allow the spreader to fit between the clips of the electrode clamp. Arc stands of various designs are offered for sale by manufacturers of spectrographic equipment.

The graphite electrodes used in this laboratory are 3/16" diameter for the lower and 1/8" or 3/16" diameter for the upper. The lower electrode is cut to a length of 1½", and one end is drilled approximately 7 mm. deep to provide a conical cavity for holding the sample. The upper electrode is pointed by means of the pencil sharpener. "Ordinary-grade" spectroscopic carbons are suitable for most mineral identification work, although traces of several elements present as impurities may be detected in them with the spectroscope.

The spectroscope* (C) shown is a conventional three-arm Bunsen type constructed with a dispersing system consisting of two 60° flint-glass prisms. The telescope arm can be swung through an arc to enable the observer to view any portion of the visible spectrum. By means of a hair line in the eyepiece the exact position of the spectral lines are determined relative to the illuminated scale which is imaged directly above the spectrum. A shielded incandescent light (D) is used to illuminate the scale.

A double convex lens (E) of approximately 5 inches focal length is used to project an image of the light source upon an electrode positioning screen (F) located about 4 feet from the electrodes. The screen is marked to outline the exact position of the electrode image when the center of the arc and the slit are at the same vertical height. The correct vertical height and electrode gap are maintained during arcing by manipulation of the insulated knobs of the arc stand so that the electrode image is kept within the marked boundaries on the screen.

A small hood, not shown in Fig. 2, is mounted above the arc stand to carry away the gases produced by the burning arc.

The cost of the instrument and accessories is not prohibitive. A satisfactory assembly costs no more than a fully equipped petrographic microscope.

Technically trained persons can easily acquire proficiency in the use of the spectroscope in a short time.

* The spectroscope described above is one of several types available on the market. The reader is referred to the general textbooks listed in the bibliography for specific designs and details on the optics of the instruments (1) (2) (3) (11).

OPERATION

The general procedure for an unknown sample is as follows: (1) Place the powdered sample or fragment into the cavity of a previously prepared 3/16" diameter graphite electrode; (2) fit the electrode into the lower electrode clamp; (3) insert a 1/8" or 3/16" diameter graphite rod which has been pointed by means of the pencil sharpener into the upper electrode clamp. The upper electrode clips may be spread apart with a short screw driver or other similar tool; (4) the vertical height of the arc is adjusted to be the same as the spectroscope slit, leaving a gap of 3 to 5mm. between the electrodes; after the master switch is thrown, an arc is struck by making momentary contact between the electrodes with a graphite rod provided with an insulated handle (when striking the arc the free hand of the operator should be kept away from contact with any metallic surface as a safety precaution against possible electrical shock); (6) the current is adjusted to the desired value by means of the rheostat. The current used will depend to a large extent upon the composition of the particular sample. Materials with a high iron content, for example, must be arced at 3 to 5 amperes to prevent loss of the sample due to sputtering. If small amounts of easily volatilized elements are being sought it is best to operate at low current in order that the sample will be consumed less rapidly. Since many mineral samples contain both low-boiling and high-boiling constituents, the arc is usually started at about 3 amperes, operated at 3 to 5 amperes for the first two minutes, then increased to 12 amperes in order to volatilize the more refractory elements. The more volatile elements include the alkalis, Cd, Zn, Bi, Pb, and Tl. Intermediate and more persistent are Au, Ag, Sn, Mn, Ca, Cu, Fe, Ni, V, Cr, Si, Ti and Ba. The most refractory are the rare earths, the platinum group, Al, Be, Mo, Zr, Cb, Ta, W, Th and U. There is, however, in some cases an overlapping between the groups. The individual members of each group are not necessarily arranged according to their volatility. When the more volatile constituents are suspected of being present, the usual procedure is to set the telescope in a position to observe the lines of the particular element in question before striking the arc.

Radiation from an arc source composed of all wave lengths of light in the visible spectral range produces a continuous spectrum. The emission from individual elements volatilizing in the arc produces line spectra which appear as colored images of the slit. With the set-up shown in Fig. 2, the lines are superimposed upon the continuous spectrum background, since no provision is made to prevent the light from the glowing elec-

trodes from entering the slit of the spectroscope. Band spectra resulting from molecular excitation, also may appear superimposed upon the continuous background. The bands consist of a large number of lines lying close together with a crowding toward a limiting line or head. Oftentimes the band heads only are observed, since the remaining portion may be too weak to be detected visually.

Metals in concentrations as low as 0.5 to 1.0 per cent can be readily identified in most cases. With further decreases in concentration, the spectral lines become correspondingly less persistent and may result in a flash or fugitive spectrum not easily detected. However, in a few cases, such as Na and Li, owing to their extremely sensitive lines, concentrations down to at least 0.001 per cent may be detected.

CALIBRATION

The visible portion of the spectrum lies approximately between 3900 and 7800 angstroms. It can be arbitrarily divided into the following groups:

Red.....	7800-6470 angstroms
Orange.....	6470-5850 angstroms
Yellow.....	5850-5750 angstroms
Green.....	5750-4912 angstroms
Blue.....	4912-4240 angstroms
Violet.....	4240-3900 angstroms

A convenient method of calibrating the instrument is to arc metals and metallic salts of high purity and record the scale readings corresponding to their strongest spectral lines. A working curve can then be constructed by plotting wave lengths in angstrom units with respect to scale readings. The parabolic curve obtained demonstrates the nonlinear dispersion of a spectroscope fitted with prisms. With the instrument used in this laboratory, two lines in the red portion of the spectrum differing in wave length by five angstroms can be differentiated only with difficulty, whereas lines appearing in the violet which differ by only two angstrom units are easily distinguished.

The scale of the instrument used in this laboratory is divided and numbered into 17 equal parts, and each part is subdivided into tenths. The D lines of Na at 5896 and 5890 angstroms correspond to 3.0 on the scale and serve as reference points for aligning the instrument. Ca, Fe, Sr, K, Rb, and Cs may be selected in preparing the curve, since they yield spectral lines in the visible range which are easily identified.

TABLE 1

<i>Element</i>	<i>Element</i>	<i>Element</i>
Aluminum	4572.3	5153.3
3961.5	4562.4	5105.6
3944.0	4528.5	Gold
Barium	4186.6	4792.0
5777.7	4165.6	Hafnium
5535.5	4040.8	5552.1
5519.1	4012.4	4800.5
5424.6	Cesium	4620.8
4934.1	6983.4	4336.7
4554.0	6973.3	4232.4
Beryllium	6723.3	4093.2
4572.7	6586.5	Iron
Bismuth	4603.8	5615.7
5552.2	4593.2	5586.7
4722.2	4555.5	5371.5
Cadmium	Chromium	5328.4
5085.8	5791.0	5269.5
4678.5	5409.8	5266.6
Calcium	{ 5348.3	5232.9
5270.3	{ 5345.8	5227.2
{ 5265.6	5328.4	5167.5
5264.2	5298.3	4957.6
{ 5262.2	{ 5208.4	4951.3
5261.7	{ 5206.0	4919.0
{ 5260.4	4289.7	4891.5
4878.2	4274.8	4878.2
4585.8	4254.3	4415.1
4581.5	Cobalt	4404.8
4578.6	4867.9	4383.5
4456.6	4840.3	4337.1
4455.9	4813.5	4315.1
4454.7	4792.9	4282.4
4434.9	Columbium	Lanthanum
4425.4	5344.2	6249.9
3968.5	5276.2	5930.6
3933.7	5095.3	5455.1
Calcium fluoride	5079.0	5302.0
5291.0	4137.1	5183.4
(band head)	4123.9	{ 4921.8
Cerium	4079.7	{ 4921.0
5512.1	4058.9	4899.9
4678.2	Copper	4860.9
4593.9	{ 5220.1	4824.1
	{ 5218.2	

TABLE 1—(continued)

<i>Element</i>	<i>Element</i>	<i>Element</i>
Lead	5805.2	5031.0
5201.0	5476.9	4670.4
Lithium	5146.5	Silicon
6707.9	5142.8	3905.5
6103.6	5099.9	Silver
4971.9	5035.4	5465.4
{ 4603.0	{ 4715.8	5209.0
{ 4602.0	{ 4714.4	Sodium
Magnesium	Platinum	5895.9
5183.6	6710.4	5889.9
5172.9	6326.6	Strontium
5167.3	5478.5	6878.4
4705.0	5475.8	4962.3
Manganese	5368.9	{ 4876.3
4823.5	5301.0	{ 4876.1
4783.4	5227.6	4872.5
4766.4	5059.5	4855.1
4762.4	4552.4	4832.1
4754.1	4520.9	4811.9
Molybdenum	4498.8	4607.4
5570.5	4442.6	4215.5
5533.0	Potassium	4077.7
5506.5	7699.0	Tantalum
4830.5	7664.9	5402.5
4819.3	6939.0	4819.5
4760.2	6911.3	4691.9
4731.5	4047.2	{ 4574.3
4707.3	4044.2	{ 4573.3
4434.9	Praseodymium	4530.8
4411.7	5381.3	{ 4511.5
4381.6	5322.8	{ 4510.9
Neodymium	5220.1	Thallium
5319.8	{ 5110.8	5350.5
5293.2	{ 5110.4	Thorium
5249.5	Rubidium	5049.8
5192.6	6298.6	5017.2
4859.0	6206.5	4919.8
4825.5	4244.4	4863.2
4811.3	4215.6	Tin
4706.5	4201.8	4524.7
4303.6	Scandium	Titanium
4177.3	5526.8	5014.3
Nickel	5239.8	5007.0
5857.8		

TABLE 1—(continued)

<i>Element</i>	<i>Element</i>	<i>Element</i>
4999.5	5492.9	4900.1
4991.1	Vanadium	4883.7
4981.7	5627.7	4854.9
Tungsten	4881.5	4674.8
5514.7	4875.5	4643.7
5224.7	4864.8	Zinc
5053.3	4851.5	4810.5
5015.3	4807.6	4722.2
5006.2	4594.1	4680.1
4843.8	4460.3	Zirconium
4680.5	Yttrium	4815.6
Uranium	5662.9	4772.3
6449.2	5205.7	4739.5
5915.4	5200.4	4710.0
5527.8	5087.4	4687.8

The K doublet at 6939 and 6911 angstroms and the Cs lines at 6983 and 6973 angstroms serve as points to calibrate the red portion of the scale. Ca produces suitable lines in the orange and yellow and Fe gives many lines in the green. A Sr line at 4216 angstroms and a Rb line at 4202 angstroms are near the violet end of the scale.

Several important lines lie beyond the limits of our scale, but within the observed spectral field. They can be detected and identified after a little practice. For example, in the red region the strongest lines of K occur at 7699 and 7665 angstroms, and in the violet a characteristic group of four lines is emitted by Ca at 3968 and 3934 angstroms, and Al at 3962 and 3944 angstroms. Several lines and groups of lines resulting solely from the burning of the electrodes are visible in the spectroscope. At 5165 angstroms is a band head resulting from the C₂ molecule. A band resulting from the CN molecule appears as a group of lines with its head at 4216 angstroms.

DETERMINATIVE TABLES

The wave lengths of the spectral lines that can be used for identification purposes are listed in Table 1. No attempt has been made to list all the lines of each element. Only those most characteristic (in the spectroscope) have been recorded.

Many of the elements commonly found in minerals have some of their

spectral lines arranged in characteristic groups. After a little practice the observer will recognize these characteristic groups and be able to identify rapidly the respective constituents.

These characteristic groups are listed in the following table.

TABLE 2

<i>Element</i>	<i>Characteristic groups</i>
Aluminum	A group of two lines (3961.5 and 3944.0) beyond the scale in the violet. Strong lines of Ca (3968.5 and 3933.7) form a group of four with the Al.
Calcium	A group of three lines in the green (5270.3—[5265.6–5264.2]—[5262.2–5261.7–5260.4]). Also a group of four lines in the blue (4456.6—[4455.9–4454.7]—4434.9–4425.4).
Calcium fluoride	A band head in the green (5291.0) fading toward the red.
Chromium	A group of three in the green ([5348.3–5345.8]—5328.4–5298.3).
Copper	A group of three lines in the green ([5220.1–5218.2])—5153.3–5105.6).
Iron	A group of five lines in the green (5371.5–5328.4—[5269.5–5266.6]—5232.9–5227.2).
Lithium	Two lines in the red (6707.9 and 6103.6).
Magnesium	A group of three lines in the green (5183.6–5172.9–5167.3).
Manganese	A group of five lines in the blue (4823.5–4783.4–4766.4–4762.4–4754.1).
Molybdenum	A group of three lines in the green (5570.5–5533.0–5506.5) also a group of three in the blue (4760.2–4731.5–4707.3).
Potassium	Two doublets—one off the scale in the red (7699.0 and 7664.9) and (6939.0 and 6911.3).
Sodium	The D lines in the yellow (5895.9 and 5889.9).
Titanium	A group of five lines in the green (5014.3–5007.0–4999.5–4991.1–4981.7).
Vanadium	A group of five lines in the blue (4881.5–4875.5–4864.8–4851.5–4807.6).
Zinc	A group of three lines in the blue (4810.5–4722.2–4680.1).

Lines enclosed in brackets are considered as single lines, because in most cases they are not readily resolved.

Many elements when present in major amounts can be quickly identified without a spectroscope through recognition of the colors they impart to the arc. They will be imaged on the test screen in the form of a halo surrounding the center portion of the arc.

The following characteristics will aid in identifying major constituents:

TABLE 3

<i>Element</i>	<i>Color on positioning screen or other characteristics</i>
Al	Greenish blue
Sb	White fumes
As	Garlic odor
Ba	Green
Be	Greenish blue
B	Green
Ca	Orange
CaF ₂	Canary yellow
Cb	Blue with red fringe—pitted lower electrode and white oxide coating
Cr	Green
Cs	Bluish white
Cu	Green
Fe	Blue with yellowish-white fringe—sparks and popping bead
Li	Red
Mg	Green
Mo	Blue—metallic coating on lower electrode
Nd	Light orange-red
K	Bluish-white
Pr	Greenish-gray
Sc	Light orange
Sm	Red
Ag	Green
Na	Yellow
Sr	Red
Ta	Blue with a red fringe—pitting on lower electrode and white oxide coating
Tl	Green (very intense)
Ti	White
U	Bluish white
W	Blue when current is reduced—pitting of lower electrode and yellow oxide coating
Y	Red
Zr	White flashes

N. B. The various greens and reds can be distinguished with practice.

For a detailed study similar to the above the reader is referred to a publication by Wm. Roy Mott (7).

APPLICATION

As a replacement for blowpipe and chemical tests in determinative mineralogy the spectroscopic method offers the following applications:

1. All the common and most of the rare metallic elements may be rapidly identified.

2. Spectroscopic analysis permits the use of a very small sample. In many instances, single mineral grains have been analyzed in order to avoid contamination by associated minerals.

3. The possible interference of one element or a combination of elements with the determination of another depends only upon the complexity of the observed spectra. Elements normally difficult to differentiate chemically are no more difficult than any other spectroscopic determination, e.g., Ce from La and Y.

4. Minor amounts of either valuable or undesirable metals associated with certain minerals or commercial ores may be readily determined.

Applications supplementing petrographic examinations may be summarized as follows:

1. Two or more minerals having similar optical properties may be readily distinguished on the basis of their principal metallic components, e.g., individual members of certain isomorphous mineral groups are more readily distinguished than solely by optical means.

2. The identification of the opaque minerals and minerals having refractive indices lying above the usual range of index oils is considerably simplified and accelerated.

3. Finely divided inclusions in minerals may be more readily identified.

The limitations of the spectroscope may be stated as follows:

1. Most of the anionic constituents of minerals cannot be detected spectroscopically.

2. Spectroscopic examinations are limited to the visible region of the spectrum. If it is desired to determine elements whose principle lines lie in the infra-red or ultra-violet regions, recourse to the spectrograph is necessary.

3. The lines of minor constituents may be obscured if the major constituents have complex spectra, e.g., a small amount of Ni in pyrrhotite.

Specific examples of the application of the spectroscope in mineral identification are numerous. For example, pairs or groups of minerals often confused on the basis of their physical or optical properties can be readily differentiated spectroscopically by the presence or absence of one or two distinguishing elements. The following minerals, arranged in groups of two to four, are typical of those that are difficult to differentiate. These, however, can be identified readily by spectroscopic means.

<i>Mineral</i>	<i>Composition</i>
Fluorite	CaF_2
Opaline silica	SiO_2
Talc	$3\text{MgO} \cdot 4\text{SiO}_2 \cdot \text{H}_2\text{O}$
Pyrophyllite	$\text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2 \cdot \text{H}_2\text{O}$
Chlorite	$9\text{MgO} \cdot 3\text{Al}_2\text{O}_3 \cdot 5\text{SiO}_2 \cdot 8\text{H}_2\text{O}$
Sericite	$(\text{K}, \text{Na})_2\text{O} \cdot 3\text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2 \cdot 2\text{H}_2\text{O}$
Barite	BaSO_4
Celestite	SrSO_4
Chalcopyrite	CuFeS_2
Pyrite	FeS_2
Quartz	SiO_2
Beryl	$3\text{BeO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2$
Nephelite	$\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$
Chromite	$(\text{Fe}, \text{Mg})\text{O} \cdot \text{Cr}_2\text{O}_3$
Franklinite	$(\text{Zn}, \text{Fe}, \text{Mn})\text{O} \cdot (\text{Fe}, \text{Mn})_2\text{O}_3$
Sphene	$\text{CaO} \cdot \text{TiO}_2 \cdot \text{SiO}_2$
Zircon	$\text{ZrO}_2 \cdot \text{SiO}_2$
Pleonaste	$(\text{Mg}, \text{Fe})\text{O} \cdot \text{Al}_2\text{O}_3$
Gahnite	$\text{ZnO} \cdot \text{Al}_2\text{O}_3$
Uvarovite	$3\text{CaO} \cdot \text{Cr}_2\text{O}_3 \cdot 3\text{SiO}_2$
Arizonaite	$\text{Fe}_2\text{O}_3 \cdot 3\text{TiO}_2$
Euxenite	Columbate and titanate of Y, Er, Ce, U, etc.
Fergusonite	$(\text{Y}, \text{Er}, \text{Ce})_2\text{O}_3 \cdot (\text{Cb}, \text{Ta})_2\text{O}_6\text{U}$
Molybdenite	MoS_2
Pyrolusite	MnO_2
Sooty Chalcocite	Cu_2S
Carbonaceous material	C
Witherite	$\text{BaO} \cdot \text{CO}_2$
Aragonite	$\text{CaO} \cdot \text{CO}_2$
Rutile	TiO_2
Cassiterite	SnO_2
Epidote	$4\text{CaO} \cdot 3\text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2 \cdot \text{H}_2\text{O}$
Allanite	$4(\text{Ca}, \text{Fe})\text{O} \cdot 3(\text{Al}, \text{Ce}, \text{Fe}, \text{Di})_2\text{O}_3 \cdot 6\text{SiO}_2 \cdot \text{H}_2\text{O}$
Greenockite	CdS
Carnotite	$\text{K}_2\text{O} \cdot 2\text{UO}_3 \cdot \text{V}_2\text{O}_6 \cdot 8\text{H}_2\text{O}$

Minor amounts of impurities in minerals that may be detected spectroscopically include:

Ti, V in magnetite	Sn in tantalite
Mo in scheelite	Ni in pyrrhotite
Zn in goethite	Cr, Li, V in mica
Cb in rutile	Ag in galena
Li, Na in beryl	Ag in sphalerite
Cr in clay	V in carbonaceous rocks
Zn, Mn in calcite	Fe in quartz and feldspar
Pb, Y in uraninite	Mn in calamine

Frequently, the petrographic laboratory is called upon to furnish rapid spectroscopic examinations of furnace slags, chemical precipitates, metals and alloys and various miscellaneous products other than minerals. While these do not properly fall within the scope of this paper, such application may be of interest to a person engaged in any phase of economic petrography.

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