





# MANUAL OF SPECTROSCOPY

*by*  
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# Manual of Spectroscopy

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## PREFACE

Too many chemists, mineral collectors, prospectors, and even assayers struggle with tedious chemical and uncertain flame and blowpipe tests when a spectroscope would give far more prompt results. With electricity universally available, and with present-day instruments and parts so low in cost, spectroscopic equipment should be in every school and laboratory.

This book has been written to assist those who wish to analyze ores, minerals, alloys, and inorganic chemicals, or wish to teach others to do so. In the qualitative analysis of such materials, there is no instrument so rapid and accurate as the spectroscope, although the analyst must remain within its limits of operation. This is also true of the quantitative analysis of these materials. Although speed comes only after some experience, one may very soon acquire the necessary technique for accurate determinations. The author has attempted to point out some of the short cuts to quick spectroscopic success. Direct methods of burning samples are shown; the key lines of each element have been selected, and a new chart-table has been prepared which shows both the spacing of spectral lines and their wave-lengths. An increasing number of schools and universities have courses in spectroscopy, and many industrial plants use spectrographic equipment to speed up the solution of their special problems of analysis.

The bulk and cost of spectrosopes tend to increase in geometric ratio with efficiency, and thus the price of a commercially built high-dispersion instrument is usually beyond the reach of the individual analyst. The parts necessary to make a powerful instrument are few and the essential construction is very simple. The writer believes that instructions for making effective instruments at costs so low as to be within the reach of all, will be appreciated. One section of the book has, therefore, been devoted to such instructions. Largely from his own experience, but also

from the literature (see bibliography) a considerable amount of material has been arranged in what is hoped will be helpful form.

**Acknowledgments.** The writer wishes to express appreciation to those who have assisted in the preparation of the book: especially to his wife, Mary Elizabeth, without whose assistance the work would have been very difficult; to his sons, Windsor C. Cutting, M.D. and Cecil C. Cutting, M.D. for reading the entire manuscript, and for extensive criticisms and suggestions; to Henry Elliott, Ph.D. for reading portions of the manuscript and for constructive ideas; to Leslie Titus, A.B. for spectrum photographs and for reading the entire manuscript in the light of his special chemical and spectroscopic training and experience; to Frederick K. Vreeland, D.Sc. for special data; to his brother, James A. Cutting, M.D. and to the Applied Research Laboratories for photographs and literature; and to the San Jose State College for the use of special equipment.



CHAPTER I  
HISTORY AND THEORY OF SPECTROSCOPY

**I. Historical Review**

Spectroscopy may be considered to have had its origin in the discovery by Newton, about 1670, that sunlight is composed of many colors. His experiment consisted in dispersing a beam of sunlight into its several colors by means of a glass prism. Huygens, a contemporary, developed the theory, contrary to the corpuscular idea of Newton, that light consisted of wave motions. Wave diagrams are still used to illustrate the properties of light.

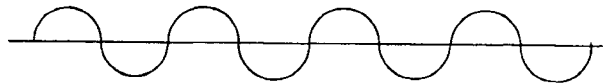


FIGURE 1  
**Light Waves**

It was more than a hundred years before any further advance was made. Early in the 19th century Herschel revived interest in light phenomena by showing that the spectrum continued beyond the visible range. This he demonstrated by placing a thermometer in the infra-red portion of the spectrum. Ritter, in 1801, proved the extension of the spectrum into the ultra-violet by showing that silver chloride was darkened when placed there. Wollaston very soon afterward constructed the first real spectroscope with both prism and slit. With it he discovered the black lines of the solar spectrum. At about the same time, Young discovered the phenomenon of interference, which was later used for determining wavelengths.

Fraunhofer, in 1814, made the first diffraction gratings and used them for the first measurements of light waves. He also mapped the solar spectrum and even extended his observations to the spectra of the stars. Finally he recognized as identical the sodium doublet of the solar spectrum and the doublet of his laboratory flame.

Kirchhoff explained the black lines of the solar spectrum as caused by clouds of metallic vapors surrounding the white-hot orb of the sun. In 1861, he and Bunsen, the inventor of the Bunsen burner, became the originators of spectroscopic analysis by publishing the results of their investigations of the spectra of the elements. Three years later Maxwell contributed his electro-magnetic theory of light, and Balmer, about 1880, discovered a formula for computing the lines of spectral series.

Rowland, in 1881, ruled the first concave gratings and produced with them such superior spectra that accurate mapping of the solar spectrum became possible. His tables of spectral lines represented a great advance in practical spectroscopy. In 1901 Planck propounded his quantum theory of light which revived the corpuscular theory of Newton. Michelson, at about the same time, invented both the echelon and the interferometer, with which he measured the Paris meter bar in terms of the wave-length of the red cadmium line and so established a new and more permanent unit of measurement. In 1912 Bohr harmonized the earlier theories of electron movement with the quantum idea in his "Theory of Spectra and Atomic Constitution."

Since 1915, Meggars of the U. S. Bureau of Standards, has done much to stimulate the use of the spectrograph, especially of the grating type, for both qualitative and quantitative industrial analyses. About thirty of the elements were discovered by means of the spectroscope, and some of them can still be found only with difficulty, if at all, by any other means. Rubidium (red), thallium (green), cesium (blue), and indium (indigo) were all named from the colors of their most brilliant spectral lines.

## **2. The Atom in Spectroscopy**

*The Elements.* Terrestrial forces, operating through eons of time, have formed about 1,000 mineral species, natural compounds, in the earth; man, in the last 200 years, has formed hundreds of thousands of other compounds; all of these are different combinations of 96 elements, each of which has its characteristic atom structure. Atoms, although inconceivably small and quite beyond the range of even the electron microscope, are made up of still smaller bodies, the most important of which are protons, electrons and neutrons.

*Subatomic Particles.* Electrons are particles of almost negligible mass, each carrying a single negative charge of electricity. Protons have about 1,840 times the mass of electrons and each one carries a single positive charge. Neutrons, with only slightly greater mass than protons, carry no charge. Positrons, which have the same mass as electrons and equal but opposite charges, are emitted by bombarded nuclei. The neutrino with electronic mass and no charge, and the negatron with a mass corresponding to that of the neutron and a negative charge, have also been reported.

*Atomic Structure.* It is the electron with which we have chiefly to deal in spectroscopy, since its motions produce light. Each atom consists of a nucleus containing protons, usually neutrons, and one or more electrons revolving around it like tiny planets about a sun. When the elements are arranged in their order of complexity, starting with hydrogen and ending with uranium and the new elements, neptunium, plutonium, americium and curium, their atoms are found to differ, each from its predecessor, by one proton, one electron, and a varying number of neutrons. Normally these electrons revolve about the nucleus in definite orbits, and, according to the theory of Bohr, in concentric shells with a definite number of electrons in each shell, as illustrated in Figure 2.

The periodic chart of the elements, Figure 2, is arranged spirally to show:

1. Atomic numbers in consecutive order

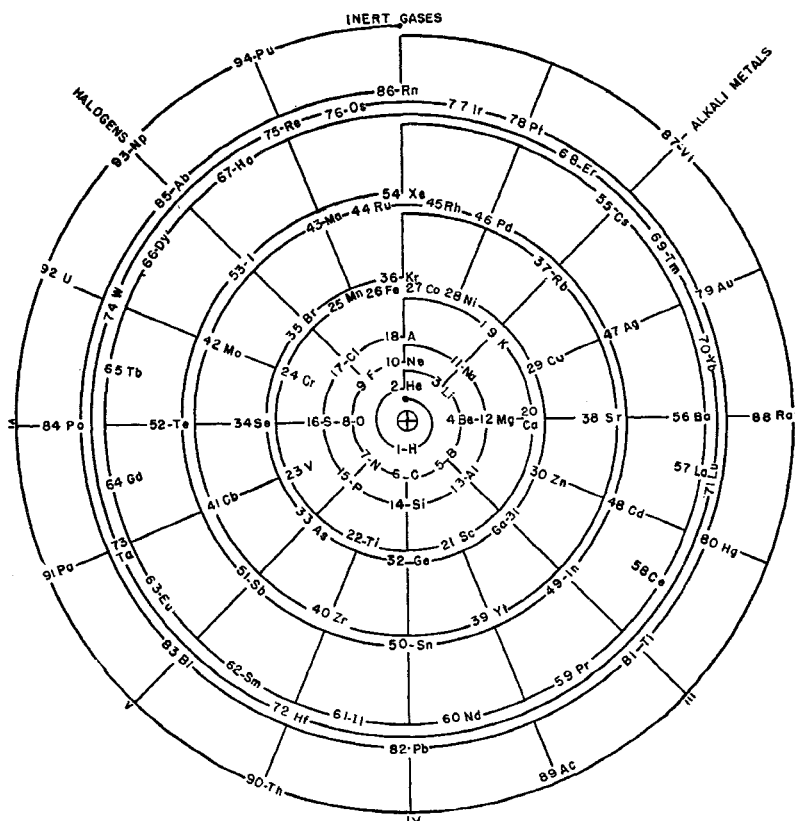


FIGURE 2  
Periodic Chart of the Elements (Spirally Arranged)

2. The theoretical structure of each atom
3. The shell arrangements
4. The number of electrons in the outer shell
5. Chemical valences (Roman numerals)
6. Chemical relationships (Radial positions)

*Light.* In spectroscopy we are always analyzing light. The modern theory of light is complicated, involving emission by the atom of corpuscles or quanta of light in wave form. When an element is heated to incandescence, the motions of the electrons

are accelerated and energy is absorbed. Moreover, the electrons leave their usual orbits and shells and move to higher levels more distant from the nucleus.

When the electrons return or fall again to their former levels, they give off the absorbed energy in the form of light. According to this theory a short fall produces long waves of red or infrared light, and a long fall short waves of violet or ultraviolet light. Upon excitation, the electrons of a given atom can assume only certain positions or orbits; consequently, in falling, they emit light of only certain colors or wave-lengths. Furthermore, since each element has a different number of electrons from the others, each emits a different assortment of colors when heated to incandescence.

Hydrogen, the simplest of the elements, when enclosed in a tube with a high-voltage current passed through it, will glow with a distinctive bluish light. When viewed through a spectroscope this light will be seen to consist, not of a single color, but of several; for the instrument sorts out the different colors of the light source and shows each as a brightly colored line. In the case of hydrogen the lines will be red, blue and violet, and they are said to comprise the hydrogen spectrum, or the visible portion of it, for there are also ultraviolet and infrared lines.

The speed of light has been accurately measured and is the same for all colors, but the number of waves or pulsations per second (frequency) is different for each color and for each shade of that color. The wave-length of the light of any spectral line is also different from that of any other, since it is the quotient of the velocity of light divided by the frequency of its wave.

The wave-lengths of the spectral lines forming the hydrogen spectrum fall into several mathematical series as though each group represented the light given off by electrons dropping from different heights to the same level, as shown in figure 2. In the larger group the short heavy line represents the electron fall causing the very brilliant red spectral line of hydrogen, and it is caused by the short fall of electrons from the third to the second level. The next two lines represent falls causing weaker blue

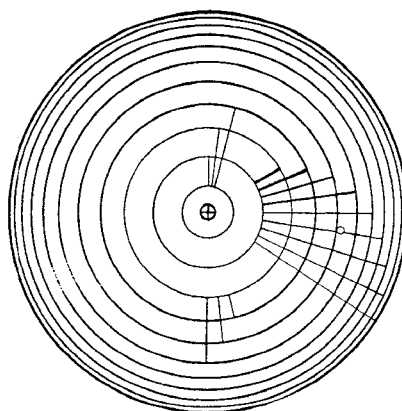


FIGURE 3  
**Hydrogen Atoms**

lines; then come falls causing two violet and finally four ultra-violet ones. Other series to the right and left show electrons falling to the first and third levels. According to the theory, electrons may fall from level to level in short jumps, or even from the top to the bottom level in single long jumps. Mathematical formulae for these series have been developed by which, if a few spectral lines of an element are known, the wave-lengths of the other spectral lines of the series may be predicted.

CHAPTER II  
LIGHT SOURCES

**1. The Electric Arc**

*Light Sources.* For spectroscopic analysis the rock or other sample must first be vaporized by heat and the incandescent vapor then viewed or photographed by means of a spectroscope or spectrograph, since it is only in the vaporized state that each element emits its distinctive colors or imparts them to the flame used for vaporizing it. The original heat source was the Bunsen flame, but its temperature is so low that it can vaporize few of the elements. To-day the electric arc, the spark and the discharge tube have taken its place. For different elements and for different types of samples different sources are required.

The electric arc is a continuous luminous discharge produced at the break in an electric circuit of low voltage. The spark is the discharge occurring at a gap in a high-voltage circuit. A potential of at least 40 or 50 volts is necessary to produce a sustained arc; from 10,000 to 25,000 volts is used in the spark. The discharge or Geissler tube is one filled with the rarefied gas to be analyzed; it is energized by passing a current of high potential through it.

*Sources Required for Different Elements.* Either the electric arc or the spark may be used to produce the known spectra of 65 of the metallic elements. The Geissler tube is employed for the 11 gases: argon, chlorine, fluorine, helium, hydrogen, krypton, neon, nitrogen, oxygen, radon and xenon and liquid bromine. None of the ten non-metallic or semi-metallic elements have very satisfactory visible arc lines: these are arsenic, antimony, boron, carbon, iodine, phosphorus, silicon, selenium, sulfur and tellurium. All of them, however, except iodine and sulfur have ultraviolet arc lines. The spectra of the latter two may be examined in either the discharge tube or in the spark.

Boron has no visible spectrum. A few of the lines and distinctive molecular bands of the *alkali elements* may be brought out in hydrogen or other gas flames, but the only satisfactory way to secure the complete spectrum of an element is to use electrical excitation. The major metals all show strong spectral lines, most of them many such lines.

*The Arc.* By attaining temperatures up to 8000° C, the arc vaporizes all rocks and chemicals and so brings out their full wealth of spectral lines. A potential of nearly 50 volts is necessary to sustain an electric arc, a 110-volt potential is better still. Most operators, in fact, choose 220 volts if available. Direct current is always preferred, but highly satisfactory results may be obtained with 110-volt alternating current. Only carbon electrodes will function with alternating current; with direct current rods of copper, silver, graphite, or other conductive material may be employed. The arc has been used as the chief source for producing spectra since the invention of the Gramm dynamo in 1876.

*Portable Arc.* Prospectors sometimes rig up a generator driven by the automobile engine and carry their spectroscopes with them into the field. Old 12-volt Dodge generators have been successfully rewound to deliver about four times the rated voltage and used in this manner. Regular generators for producing 5 amperes at 100 volts may be purchased or specially wound. The higher the voltage, the less the amperage required to work the arc. Since the number of watts is the product of the number of amperes multiplied by the number of volts, the following table, giving voltages commonly used in arcs, will show that it is customary to use about 1000 watts, except in the case of the high-voltage arc.

#### COMMON ARC CURRENTS

20 amperes at	50 volts or 1000 watts
10 amperes at	110 volts or 1100 watts
5 amperes at	220 volts or 1100 watts
2.5 amperes at	2000 volts or 5000 watts

*Resistor.* A resistor is used in series with the arc to reduce the starting current to about 10 amperes, and to prevent drawing ex-



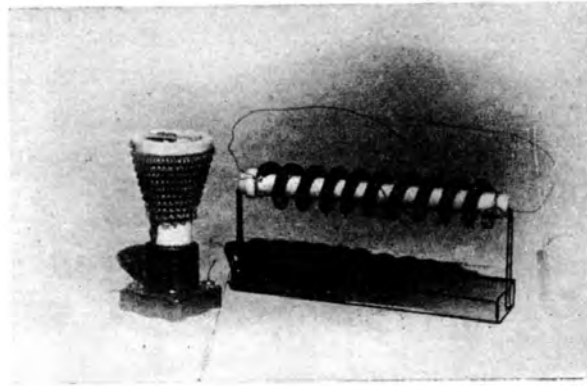


FIGURE 4  
Resistor Types

cessive current and blowing fuses. One 1000-watt heating element of nichrome wire, or two 500-watt elements in parallel, will pass the proper amount of current at 110 volts. Forty feet of ordinary No. 22 *stone* wire will also make a satisfactory resistor, just enough of the wire being used to prevent its becoming red hot and burning out. A shorter length of No. 24 iron wire may be



FIGURE 5  
Coil Winder

employed with the same results. The wire is first coiled about a long iron rod the size of a lead pencil, which may well be mounted and bent at the end to form a handle for turning. Then the coil is slipped off the rod and wound upon a 10-inch length of asbestos-covered gas pipe or porcelain tube.

*Horizontal Arc.* The arc consists merely of two carbon rods or other electrodes held end to end, in any convenient manner, upon insulating supports. Carbons are preferably about  $\frac{1}{4}$  inch in diam-

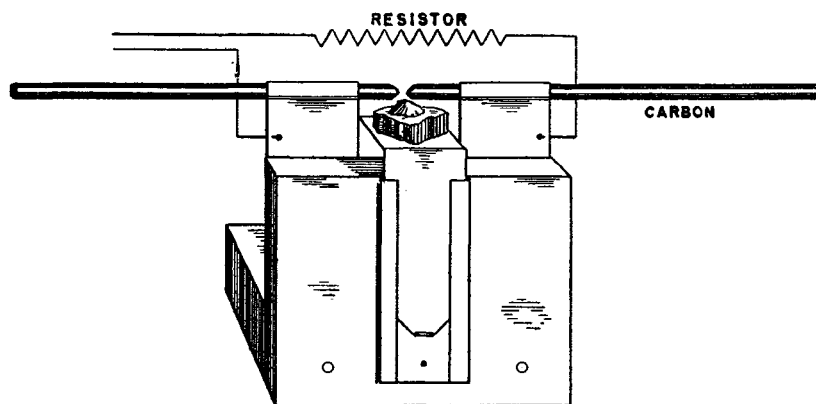


FIGURE 6  
Horizontal Arc

eter, since larger electrodes draw more current than is necessary. The electrodes must be movable so that they may be pulled apart to provide the gap after making initial contact. If the slit in the spectroscope is horizontal, then the carbons should be arranged horizontally; if the slit is vertical, then the carbons are also vertical.

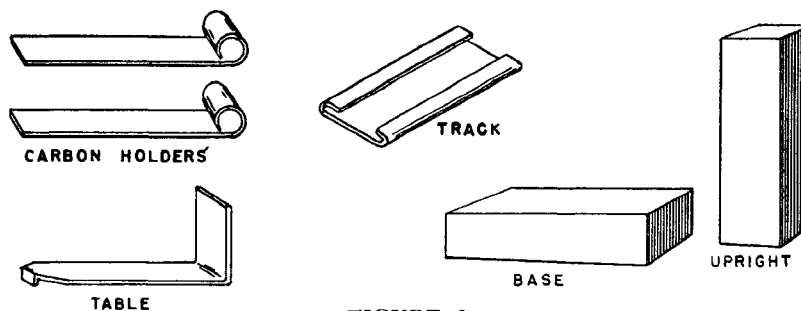


FIGURE 6A  
Arc Parts

The following example will serve to illustrate a simple home-made arc. Nail together at right angles two small boards, each about three inches square, one to serve as a base, the other as an

upright. Next cut out two strips of any sheet metal an inch wide and three inches long for carbon holders. Curl one end of each strip around a spike slightly smaller than the carbons and then pull away the spike. Nail the strips to the upright about an inch apart with the curled ends an inch above the top. These will hold the carbons end to end in a horizontal position.

Prepare another piece of sheet metal, about  $1\frac{1}{4}$  inches wide and 2 inches long, to serve as a track for holding the table strip. Fold over both of the long edges about one-eighth inch. Before flattening the folds completely slide in a double strip of tin and then press flat in a vise. Pull away the strip of tin and nail the track vertically to the middle of the upright on the opposite side from the carbon holders. Finally cut another strip about 1 inch wide and 4 inches long to slide smoothly within the folded track just prepared. Insert the sliding strip and bend it at right angles in the middle so that the horizontal part will form a table between the carbon holders and beneath the carbons upon which to place the sample to be tested. Be sure that the table slides clear of the carbon holders so as not to short out the carbons and so that the ore may be pushed up close beneath the arc, or a little into it. A nib should be bent out at the bottom of the strip to facilitate pushing it up and down after the top becomes hot from the arc.

*Wiring the Arc.* Separate the twisted wires at the end of a heavy drop cord. Connect one terminal directly to one of the carbons or to its holder and connect the other to one terminal of the resistor. Finally connect the opposite terminal of the resistor to the second carbon holder. This provides a circuit for the current to come down one of the twisted wires of the drop cord, pass through the resistor, go to one carbon, jump the gap to the other, and finally go back up the second wire of the drop cord to complete the circuit. The resistor must always be included in the circuit to prevent shorting and blowing fuses.

The remote ends of the carbons may be taped to prevent shocks during manipulation, although if one remembers to touch but one carbon at a time, and the floor is dry, this is unnecessary. There is little danger from a 110-volt circuit, but linoleum should

be used on cement or damp floors to prevent the uncomfortable shocks of a ground circuit. With the use of 220 volts twice the precautions are necessary against shocks and against shorts as well. One should look at the arc flame only through smoked or cobalt-blue glass to avoid injury to the eyes from the ultraviolet radiations.

*High-voltage Arc.* Excellent results are reported by many operators who have used high-voltage alternating-current arcs. In these, the voltage is stepped up by a heavy transformer to about 2,000 volts, and a current of  $2\frac{1}{2}$  amperes is used. There is no condenser in the circuit. In the quantitative analysis of metallurgical products it is claimed to be superior to the direct-current arc.

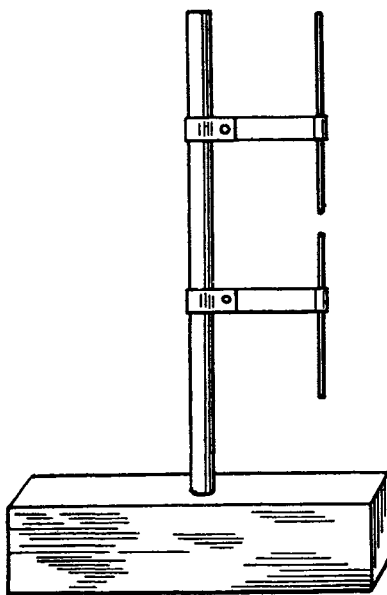


FIGURE 7  
Vertical Arc

*Vertical Arc.* For vertical slits the carbons are clamped, end to end, one above the other on insulating supports. A little cup is

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