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The X-ray spectrometer and what it reveals

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Thesis

THE X-RAY SPECTROMETER AND WHAT IT REVEALS

Submitted by

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THE X-RAY SPECTROMETER AND WHAT IT REVEALS.

I. Introduction and Historical Survey.
II. Construction.
III. Operation.
IV. Application.
V. Results Shown.
VI. Future Possibilities.
VII. Conclusion.

The X-Ray Spectrometer and What It Reveals

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The X-ray Spectrometer and What It Reveals.

When a vacuum tube is electrically excited the negative electrode or cathode sends out a stream of negatively charged particles called electrons. The electrons make up what is known as cathode rays. When they are made to impinge upon a target called the anticathode X-rays are produced.

X-rays have continued to baffle those who have attempted to diffract or refract them, and it is only within the last few years that investigators have succeeded in reflecting them. It has become known that the failure of X-rays to suffer reflection like ordinary light is due to the fact that the wave lengths are very short and the irregularities in ordinary polished surfaces scatter the rays so that they are not reflected as a whole.

In 1912 Dr. Laue of Munich suggested using carefully broken natural crystals for "reflecting" X-rays. They were known to have surfaces smoother and flatter than anything artificial and he said that the grouping of the atoms should be able to produce interference effects with X-rays in a way analagous to that in which diffraction gratings deal with light waves. Friedrich and Knipping soon after made the first tests and justified Laue's theory. Later W. L. Bragg got beautiful X-ray reflections by using mica crystals. This simple epoch-making experiment served as the starting point of a long series of experiments by various scientists and they have practically proved that X-rays are similar to light but have a shorter wave-length.

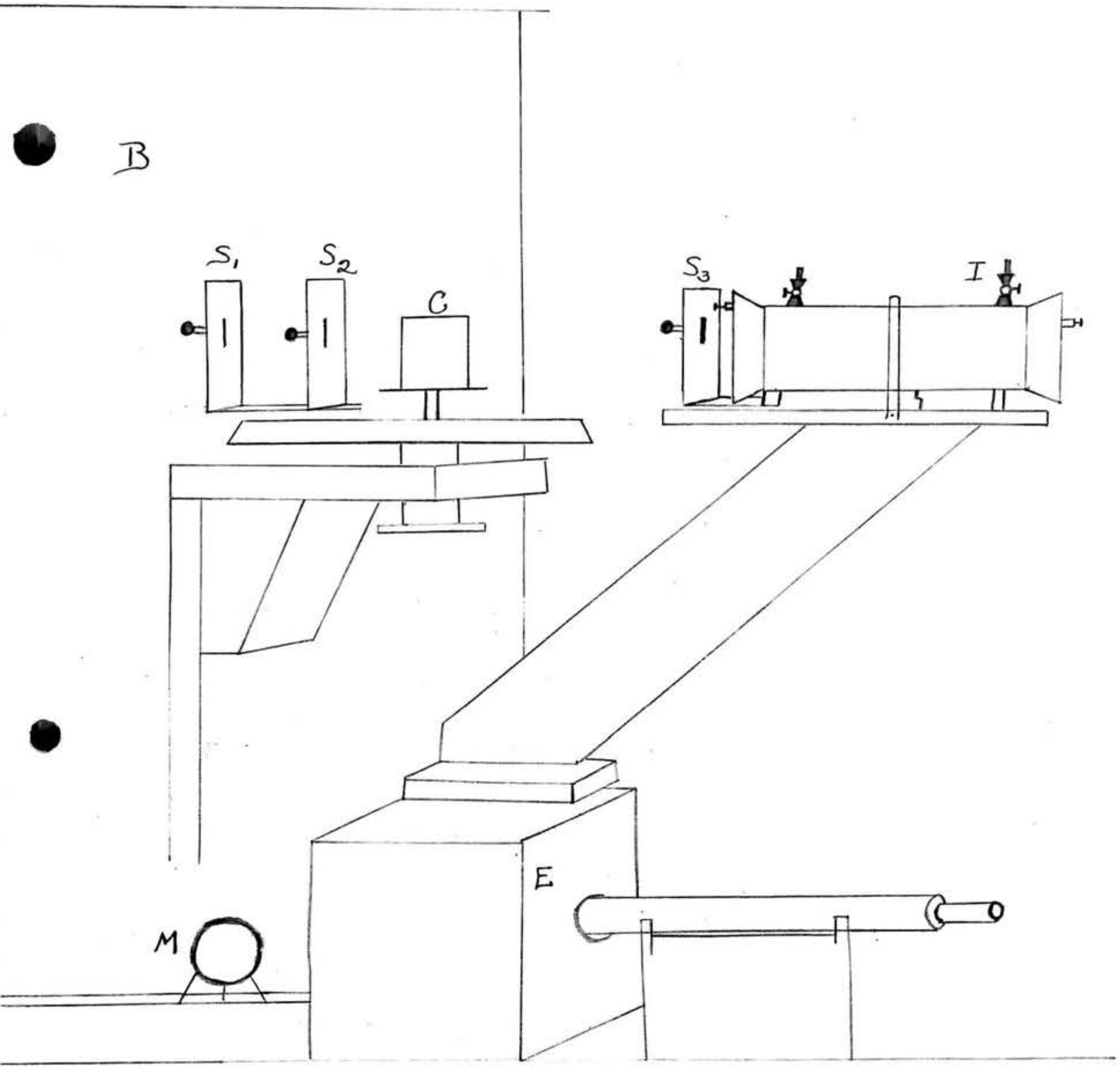
It may be considered that gamma rays which come from radio-active substances are X-rays of extremely short wave-length, and some work has been done to prove this. Without doubt all forms of radiation, from the long Hertz waves of wireless telegraphy down through heat, light, ultra violet light, and X-rays to the ~~infinitely~~ very short gamma rays, are essentially similar in character.

Prof. W. H. Bragg and his son W. L. Bragg made a thorough study of the reflection phenomena of X-rays and their work is very important. Moseley and Darwin, working at the same time, made experiments on similar lines. The instrument they used was called an X-ray Spectrometer. It was invented by the Braggs in 1913.

The X-ray spectrometer (See Fig. I) consists of an X-ray tube enclosed in a wooden box lined with lead, two slits for controlling the rays, a crystal mounted upon a revolving table which is arranged with a vernier so that angles can be read, an adjustable slit, and an ionization chamber having its front faced with lead and containing a small opening covered by a thin sheet of aluminum. In the chamber is an electrode (placed so that the rays will not hit it) in contact with a Wilson tilted electroscope. The chamber also has a vernier connected with it and this works on the same scale as the one with the table.

The X-ray bulb is mounted so that it can be adjusted to any position. The first is used to allow a fine pencil of rays to escape from the box, and the second one makes them of a certain width and prevents outside radiation from interfering. The crystal is used to reflect the rays into the ionization chamber. The opening in the front of the chamber is made quite large, but the width of the pencil of rays is controlled by the adjustable slit. Sulphur dioxide is the gas generally used in the chamber because it absorbs the X-rays strongly and yields a large ionization current. The chamber is in-

Figure I.



B is a box containing an X-ray bulb.
S₁ and S₂ are adjustable slits which direct a beam of X rays on to the face of the crystal C.
S₃ is a slit which controls the beam passing into the ionization chamber I.
E is a metal box containing the tilted electroscope which records the beam.
M is a mirror for illuminating the electroscope.

sulated from the earth and is raised to a high potential. The electrode is charged by the ions created in the gas by the X-rays. When the current of ions flows in the ionization chamber it causes the charged gold leaves to collapse. The rate of collapse of the leaves shows the intensity of the ionization.

There are three principal methods of making observations with the spectrometer. The crystal may be given a fixed position, the ionization chamber moved and the rate of collapse of the leaves for each position recorded. A curve is used to show the range of the radiations obtained at the different angles the primary rays make with the face of the crystal. A large crystal will make it possible to map out quite a long spectrum. Moseley used a photographic plate in place of the ionization chamber and it has proved to be a great help in mapping the spectra for observation.

In the second method both crystal and chamber are moved and the rate of the chamber is made twice that of the crystal. Reflection takes place at that part of the crystal face which is near the axis. This method is used for observing the spectra emitted by different anticathodes and for finding the constants of crystals.

A third method has the ionization chamber in a fixed position and the crystal is made to revolve. This makes it possible for each wavelength, in the incident radiation, to be reflected in order.

When it comes to choosing a method of observation, the one that will give intensity and sharpness of definition or resolving power is the one to be desired.

The X-ray spectrometer is used to study the properties of X-rays in general; to study X-rays sent out by anticathodes of various metals; to analyze the structure of cyrstals; and to observe the absorption power of different elements and compounds.

The work on crystal-reflection has shown that X-rays and light rays are identical. There is no difference to be considered beyond that of wave length. It has been found that X-rays, gamma rays, and light rays on striking a metal plate cause electrons to be ejected from it. Experiments have shown that the intensity of the incident rays does not affect the speed, but merely the number of electrons sent out; that the speed is controlled by the quality of the incident ray, but not at all by the metal; that the secondary electrons tend to continue in the line of flight of the original rays; and that there is a selective emission of electrons for certain wave lengths.

W. H. Bragg and W. L. Bragg, using an X-ray tube with a platinum anticathode, discovered three very homogeneous components in the radiation given off, which could be reflected only at definite angles from the crystals. Moseley using the same kind of a tube, somewhat later, found five types of monochromatic radiation. The homogenous components show strong reflection peaks superimposed on the general reflection. The general reflection is made up of radiation of indefinite wave-length, analagous to white light, and reflected from the crystal at all angles of incidence. Each component has a definite absorption coefficient in aluminum and can be recognized when reflected from many crystals. Rays of definite quality are reflected from a crystal only when the crystal is set at the right angle. Experiments show that since the reflection angle of each set of rays is sharply defined, the waves must occur in trains of great length. A succession of irregular spaced pulses could not give the observed effect.

X-rays reflected from crystals have the general properties of ordinary X-rays. However they differ in one important particular, that of penetrating power. This is due to the fact that the different constituents of the incident beam are not reflected equally by the crystal. The result is that the two differ in their average hardness. It has been found that the reflected rays may be considerably harder than the incident rays. Experiments show the same components in the incident and reflected radiations, but they are in different porportion in the two.

A substance can be stimulated to the emission of its characteristic X-rays by bombarding it with cathode rays of sufficient velocity. An anticathode of large atomic weight gives the largest quantity of rays. It is best to have a metal of high melting point so that it will not fuse when it is made the target of the sharply focused cathode rays. Various experiments show that anticathodes of different metals give reflected rays of different homogeneity. The wave length of a homogeneous beam of X-rays can be found accurately in terms of the spacing of the elements of a crystal. A number of investigators have brought out the fact that the wave length of a beam increases as the atomic weight of the metal of the anticathode diminishes and the rate of increase is almost in proportion to the square root of the atomic weight.

It has been found that the special rays are characteristic of the anticathode and not the crystal. Experiments performed with a particular bulb, and a large number of different crystals, showed the same spectrum. When tubes with different anticathodes were used with a certain crystal the spectrum changed entirely.

It has been discovered that crystals containing only elements of small atomic weight are the most efficient reflectors. The diamond, for example, is one which gives splendid results.

The reflected rays, when tried with various metals, showed approximately the same absorption coefficient that Barkla obtained for characteristic X-radiation and this established the fact that they were of the same nature.

Metals possess the power of absorbing rays from radiators of various other metals. The absorbing powers of a metal decline steadily with the increase in atomic weight of the radiator. If the metal of the radiator has a higher atomic weight than the absorbing metal it will excite the characteristic X-rays of the absorbing metal. Therefore, certain metals which do not give out rays penetrating enough fail to excite the characteristic rays of all metals.

The quality of a homogeneous beam may be defined by its absorption coefficient in some standard substance. Aluminum, on account of its low atomic weight is found to be a good substance to use as a standard. Barkla has done a great amount of work along this line, and has prepared a table giving the absorption coefficients in aluminum of the radiations characteristic of a large number of metals. The absorption coefficients are found to be independent of temperature and of chemical association.

If the masses of various substances are equal, their atomic weights cause but little variance in the scattering of X-rays of very short wave-length. When the radiation is of long wave-length the heavier elements scatter much more, mass for mass, than the lighter elements. The scattering of X-rays increases with wave-length, slightly in light elements, and more strongly in heavy elements.

The X-ray spectrometer has also been used to compare the range of Alpha and Beta rays in different substances. It was observed in the case of both these rays that the stopping power of an atom increases with its mass, but not so rapidly as its mass. It appears to be in porportion to the cube root.

The reflection phenomena, observed in the use of the X-ray spectrometer, lead to definite knowledge of crystal structure.

There are two methods by which X-rays may be made to help determine crystal structure.

The Laue transmission method makes use of the heterogeneous X-rays which make up the greater part of the output from an ordinary tube. The crystal acts somewhat the same as crossed transmission gratings. The structure of the crystal controls the pattern of the diffracted spots. Each spot on the photograph is referred to its proper reflecting plane within the crystal. It then yields information as to the position of these planes and the relative number of atoms which they contain.

The Bragg spectrometer method employs the homogeneous X-rays and uses the crystal as a reflection grating. The structure of the crystal is shown in the distribution and intensity of the spectrum lines among the various orders. Results obtained by the Lane method are verified and extended to other crystals by measuring the glancing angle at which one of the homogeneous beams of X-rays from a platinum anticathode is reflected from the various faces of the crystals. From these measurements the distance between successive reflecting planes is obtained. When this distance is determined the position of the heavier atoms can be estimated. The positions of the lighter atoms can be found by examination of the relative intensities of the reflected beam of homogeneous radiation in the different orders. While the Bragg method gives the data by which the dimensions of the lattices of crystals can be compared the Laue method gives information concerning the nature of the lattices only. A great many crystals have been examined by the X-ray spectrometer and it has proved itself a powerful instrument for determining crystal-structure.

The work on crystals with X-rays has an important bearing on chemistry. In the study of various forms of crystals it is possible to note the arrangements of atoms in the molecule and to observe the changes that take place when one atom is substituted for another. Thus a field of investigation is opened which makes possible the study of the fundamental causes of replacement, double decomposition, chemical affinity, the chemical activity of the various elements, and many related phenomena. A great amount of interesting and important work should be done along this line.

Further light will undoubtedly be thrown upon the subject of allotropism, which has been a mystery to the chemical investigator. It is thought that the allotropic forms assumed by certain substances must be due to atomic arrangement. The X-ray spectrometer should play a large part in the solution of this problem.

Crystal analysis may be applied to the science of metallurgy. It will be possible to classify the composition of the materials dealt with so as to represent each quality present. The actual knowledge of certain compounds is very limited, especially those containing silicates and aluminates. This field may be opened up and some of the great problems of scientific metallurgy solved by a complete knowledge of the crystal.

The X-ray spectrometer will give us the power to study quantitatively the motion of the atoms with heat. We should be able to acquire more knowledge about the mechanism of evaporation, liquefaction, condensation, etc. We should be able to develop new and definite conceptions as to the nature of the chemical forces causing absorption,capillary attraction, and like phenomena.

Very little work has been done in using the X-rays to find out the nature of the atomic structure from which they come.

A great amount of work remains to be done in the study of X-rays themselves. For example, the field of the gamma ray is particularly undeveloped.

Experienced investigators will find a powerful ally in the X-ray spectrometer, in their attempts to learn more about the electron in matter. It should be possible to determine the distribution of electrons in the atom. A great many important phenomena may be explained when this possibility becomes realized. It will be a test of many old theories and the cause of many new ones.

It has been shown that there is an immense field of work ahead, which may well occupy the activities of many observers for many years to come. It is to be regretted that Moseley, who lost his life at the Dardanelles, is not to be among those to continue the research. He did much important work with the X-ray spectrometer and his loss is keenly felt.

During the past four years progress has been necessarily very slow, but on the return of peace once more research will be pushed forward with renewed vigor.

Bibliography.

Beyond the Atom
by John Cox M. A.,
Cambridge University Press 1913.

The Electron Theory
by E. E. Fournier D'Albe,
Longmans, Green and Co. 1916.

The Nature of Matter and Electricity
by Daniel F. Comstock and Leonard T. Troland,
D. Van Nostrand Co. 1917.

The Corpuscular Theory of Matter
by J. J. Thomson,
Scribners 1907.

X-Rays
by C. W. C. Kaye,
Longmans, Green and Co. 1914.

X-Rays and Crystal Structure
by W. H. Bragg and W. L. Bragg,
G. Bell and Sons, Ltd. 1915.

Modern Electrical Theory
by Norman R. Campbell,
Cambridge University Press, 1913. 2nd Edition.

The Theory of Ionization of Gases by Collision
by John S. Townsend,
Constable and Co. Ltd. 1910.

I have read carefully all of the books mentioned above and have found a great amount of material in connection with the subject in all of them. X-Rays by C. W. C. Kaye and X-Rays and Crystal Structure, by W. H. Bragg and W. L. Bragg were especially rich in material and I made an exhaustive study of them.

Reflection of X-rays by Crystals,
by W. H. Bragg and W. L. Bragg,
Paper before Royal Society, July 1, 1913.
1 hour, 15 mins.

X-rays and Crystals by W. H. Bragg,
Nature, July 10, 1913.
1 hour.

Spectrum of Röutgen Rays,
by J. Herweg.
Paper before Deutscher Physikal Gesellshaft
July 15, 1913.
45 mins.

Reflection by Crystals by W. H. Bragg,
Paper before Royal Society, Sept. 22, 1913.
45 mins.

Structure of Some Crystals as Indicated by Their Diffraction of X-rays,
by W. L. Bragg,
Paper before Royal Society, Sept. 22, 1913.
45 mins.

Reflection of γ-rays from Crystals
by E. Rutherford and E. N. Audrade.
Nature. Oct. 30, 1913.
1 hour 15 mins.

Mica X-ray Spectrometer by M. Siegbalm,
Physical Review, Sept. 1916.
1 hour.

Recent Work on X-rays and Its Bearing on Chemistry,
by W. H. Bragg,
Paper before Chemical Society, March 1916.
2 hours.

X-rays and Crystal Structure by W. H. Bragg,
Paper before Royal Society, July 13, 1915.
1 hour.

Constitution and Fundamental Properties of Solids and Liquids
by I. Langmuir,
Before American Chem. Society, Nov. 1916.
1 hour.

Influence of Temperature in X-ray Interference
by M. V. Laue
Annallen der Physik, Jan. 1914.
1 hour.

Reflection of X-rays,
by H. G. J. Moseley and C. G. Darwin.
Paper in Philosophical Magazine, July 1913.
1 hour, 30 mins.

III

Absorption and Scattering of X-rays,
by C. G. Barkla and Margaret P. White,
Paper in Philosophical Magazine, 1917.
2 hours

The Scattering of X-rays and Atomic Structure,
by C. G. Barkla and J. G. Dunlop,
Paper in Philosophical Magazine, 1916.
1 hour, 30 mins.

I looked over about fifty Science Abstracts for material on the subject, spending from five to fifteen minutes on each one.