

A SIMPLIFIED X-RAY SPECTROMETER

by

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TABLE OF CONTENTS

	Page
INTRODUCTION	1
MATERIALS AND METHODS	4
THE X-RAY SPECTROMETER	4
The Collimator	6
The Crystal	6
The Ionization Chamber	9
The Electrometer	11
CONSTRUCTION DETAIL	11
The Collimator	11
Crystal and Mounting	13
The Ionization Chamber	13
The PP-54 D.C. Amplifier	15
CHARACTERISTIC EXPERIMENTAL CURVES	20
The Measurement of X-Ray Line Spectra	20
The Measurement of Total Radiation as a Function of ⁴⁰ Excitation Voltage	23
The Measurement of Ionization Current as a Function of Ionization Chamber Potential	23
LIMITATIONS OF METHOD	29
CONCLUSION	30
ACKNOWLEDGEMENTS	31
LITERATURE CITED	32

INTRODUCTION

The wide application of X-rays in recent years has made it desirable that students acquire a first-hand knowledge of these rays by actual laboratory experience. This necessitates the use of instruments which are as yet not available to the general laboratory. There is, for example, no simple inexpensive X-ray spectrometer available for laboratory use. It is the purpose of this paper to describe such a spectrometer, giving construction details, characteristic experimental curves, and a full discussion of the limitations of such a simplified spectrometer.

X-rays may be observed by three different methods. First, since they affect a photographic plate, they may be studied photographically. Many history-making discoveries have been made by photographic methods, especially in the field of medicine. The spectrograph is an instrument used to study X-rays photographically and can be used to make both qualitative and quantitative measurements.

Second, X-rays may be observed by their production of fluorescence in certain substances. A fluoroscope is an application of this property of X-rays.

Third, since X-rays ionize molecules of any gas which they strike, X-rays may be observed by means of the ionization which they produce. The ionization chamber is a device for determining the number of particles which are ionized. It consists

essentially of a metal container having a positive potential in which is placed an insulated wire or collecting rod having a negative potential. When X-rays enter the chamber, they produce ions which will migrate either to the walls of the ionization chamber or to the collecting rod, depending upon their charge. Results of experiments show that the polarity of the ionization chamber and the collecting rod is of no significance. The resulting current is then measured by means of some sensitive measuring device such as a quadrant electrometer or an FP-54 amplifier. The magnitude of this current offers a measure of the relative intensity of X-rays by means of an ionization chamber.

Early attempts to produce diffraction and interference with X-rays were only moderately successful. Haga and Wind (4) succeeded in producing some diffraction by using a v-shaped slit. In 1909, Walter and Pohl (16) performed a similar experiment but reported little evidence for diffraction. In 1912, Sommerfeld, using accurate photographic measurements, succeeded in producing definite diffraction patterns. From these patterns he calculated the wave length of the observed X-rays as 4×10^{-9} cm. In recent years, several workers have been very successful in producing clear photographs showing diffraction of X-rays. This has been due largely to the fact that these workers had learned how to produce a more nearly monochromatic X-ray beam.

Long before success had been attained by the use of slits, Laue (8) suggested that the orderly arrangement of atoms in a

crystal might act as a grating. He argued that since the calculated distance between atomic planes was only a few wave lengths, a crystal might produce interference. In his experiment, X-rays, after being collimated by two circular holes, passed through a crystal of zinc blende on to a photographic plate. The result was a remarkable diffraction pattern which consisted of a central spot surrounded by symmetrically arranged smaller spots.

W. L. Bragg (1) immediately interpreted Laue's diffraction patterns as the result of reflection from parallel atomic planes within the crystal. Each spot, except the center, was considered the result of reflection of the incident beam by some plane of atoms. From the arrangement of the spots in the Laue pattern, it is possible to determine the arrangement of atoms within the crystal.

It is logical to assume that a crystal would have atomic planes parallel to the cleavage surface. Working on this assumption, W. H. Bragg (1) tried his historic experiment of reflecting X-rays from the cleavage surface of the crystal and found a spot on his photographic plate at the angle of reflection, proving conclusively that crystals reflect X-rays. Roentgen had tried a similar experiment in 1895, but because of underexposure his results were negative. Bragg then replaced the photographic plate of the original experiment with an ionization chamber which he mounted on the arm of a spectrometer. A crystal on the prism table served as a refractor

for the X-rays which were detected by a sensitive electrometer. By this method, it was possible to observe X-rays through their ionizing effect.

MATERIALS AND METHODS

The materials required to convert an optical spectrometer into an X-ray spectrometer are (a) a student optical spectrometer, (b) a diffracting crystal, such as calcite or rock salt, (c) an ionization chamber of medium size, (d) sheets of lead for shielding, and (e) a rectangular block to serve as a crystal mounting.

The following auxiliary equipment is also needed to operate the spectrometer: (a) an X-ray tube having a sufficiently high voltage to excite the K lines of the target, and (b) an electrometer, either electrostatic or vacuum tube, having a sensitivity of at least 15,000 millimeters per volt.

THE X-RAY SPECTROMETER

An X-ray spectrometer consists essentially of three parts: (a) a series of lead slits which limit the X-rays to a narrow beam and which is called a collimator, (b) a diffracting crystal, such as rock salt or calcite, and (c) an ionization chamber for locating and determining the relative intensity of the beam after diffraction by the crystal.

Figure 1 illustrates the relative position of the three essential parts. In the figure, S_1 and S_2 are very narrow

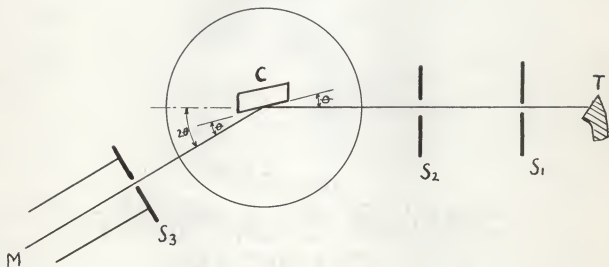


Fig. 1. Relative position of collimator, crystal, and ionization chamber.

lead slits to define the beam to a narrow pencil. The angle θ refers to the angle between the parallel atomic planes of the crystal and the incident X-ray beam. The reflected X-ray beam proceeds at an angle 2θ , and the opening of the ionization chamber must be placed along the line CM to receive it. The entrance slit to the ionization chamber may be much wider than the slits of the collimator.

The Collimator

The purpose of the collimator is to define the X-ray beam to a narrow pencil. It consists of a system of lead slits, of which the last one is adjustable. Lead is used because of its opaqueness to X-rays. Ordinarily only two slits are needed, but in case of long collimators, three or four may be necessary to counteract the effect of scattering from the walls of the collimator. It must be emphasized that these slits must be narrow in order to produce sharp spectra.

The Crystal

The purpose of the crystal is to reflect the collimated beam of X-rays. Only a few crystals are suitable for X-ray work. For general work, either calcite or rock salt is utilized. Of these two, calcite is more commonly used since rock salt is difficult to obtain in pieces free from imperfections. Bragg and Bragg (1) state that in general crystals are imperfect and appear to consist of a multitude of small more perfect crystals

put together in an imperfect manner. As a result, reflection from the crystal face takes place not in a straight line but in a manner analogous to the reflection of light from a slightly rippling surface of water. A good crystal must be found simply by trial and error. Siegbahn (12) has compiled a list of crystals which have been used with varying degrees of success. His list includes rock salt, calcite, gypsum, mica, sugar, quartz, potassium ferrocyanide, and carborundum.

In the spectroscopy of X-rays, the distance between atomic planes parallel to the cleavage surface is of primary interest. Bragg and Bragg (1) analyzed the structure of rock salt and determined the distance between its atoms. Probably the most accurate determination of this distance was that of Mosley (9), who obtained the value of 2.814×10^{-8} cm.

Siegbahn (12) made accurate measurements of this spacing for calcite on the basis of the accepted value for rock salt and obtained the value of 3.92904×10^{-8} cm.

When a monochromatic beam of X-rays falls on the face of a crystal, the crystal acts merely as an absorber or scatterer unless the Bragg relation, $n \lambda = 2d \sin \theta$, is satisfied. In the latter case, a strong reflection or scattering in a preferred direction takes place.

Bragg's law can be derived in a simple manner from Fig. 2. The points 1, 2, 3, etc., represent the position of the ions in the crystal. Consider a beam of X-rays striking the crystal at an angle ϕ . Let hg be the reflected wave front. For

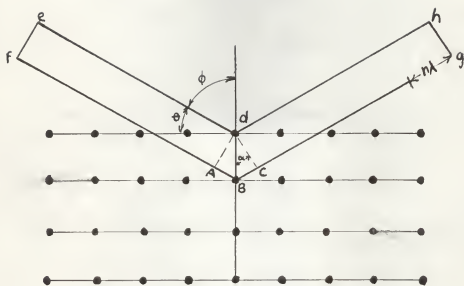


Fig. 2. Reflection of X-rays from successive parallel atomic planes.

constructive interference between the rays edh and fBg , the path difference must be an integral number of wave lengths. It is obvious that this path difference is equal to AB plus BC . But $AB = BC = d \sin \theta$; therefore,

$$n \lambda = 2d \sin \theta$$

where n is 1, 2, 3, etc., λ is the wave length, and d is the distance between adjacent layers of atoms in the crystal.

The above formula is not exactly correct since the X-rays are refracted slightly as they enter the crystal. It can be shown that the correct form is

$$n \lambda = 2d \sin \theta \left[1 + \frac{\mu^2 - 1}{\sin^2 \theta} \right]^{\frac{1}{2}}$$

where μ is the index of refraction. The deviation of this from the original Bragg formula is very small since the quantity $(1 - \mu)$ is of the order of 10^{-5} or 10^{-6} .

From the above relations, it is seen that reflection can occur only at certain very definite angles in the case of monochromatic X-rays. These angles represent the various orders of spectra in which n assumes values of 1, 2, 3, etc. In the case of a beam of X-rays which contains many wave lengths, such as a beam coming directly from the target of a tube, reflection occurs at any angle for which there is a component wave length that satisfies the Bragg relation.

The Ionization Chamber

The ionization chamber serves to locate the X-ray beam and to measure its relative intensity after reflection by the

crystal. Ionization chambers for spectrometers are frequently made several feet long in order to obtain large ionization currents. Shorter chambers are more convenient as they can be attached directly to the spectrometer and require no special rotating mechanism.

Another means of obtaining large ionization currents is through the use of various gases within the chamber. Bragg and Bragg (1) found that sulphur dioxide was one of the best absorbers for medium hard X-rays, being about ten times as efficient as air. For harder X-rays, methyl bromide is more efficient. Siegbahn (12) suggested methyl iodide for the hardest X-rays.

If these gases are used, the insulated part of the chamber must be made of quartz or sulphur; ebonite and amber are made conducting by the chemical action of these gases. An ordinary metal chamber with bolted ends will leak considerably, and provisions must be made for constant refilling. Duane and Blake (3) designed an ionization chamber which appears to work satisfactorily. Their chamber consisted of a sealed glass vessel in which the insulated electrode was held by a quartz joint. Such a chamber has a fixed gas content for a considerable time, and results obtained at different times are comparable. It is, however, expensive and difficult to construct.

In an elementary student X-ray spectrometer, such as the one described in this paper, it seemed desirable to use air in the ionization chamber.

The Electrometer

Every X-ray spectrometer requires some device for measuring the ionization current. Since the current which is measured is very small, a sensitive instrument is necessary. Sensitive electrometers are generally used. More recently, however, vacuum tube amplifiers using special high insulation tubes have proved very satisfactory. In this study such an amplifier was used. A discussion of the circuit and characteristics of the amplifier is found in another part of this paper.

CONSTRUCTION DETAIL

In converting an optical spectrometer into an X-ray spectrometer, the following changes were made: (a) the collimator lens and brass slit were replaced by a system of parallel lead slits; (b) a refracting crystal was substituted for the prism; and (c) an ionization chamber was substituted for the telescope.

The Collimator

In these experiments the original collimator tube of the optical spectrometer was used. The collimator lens and the adjustable brass slit were replaced by a system of four parallel slits spaced at approximately equal intervals. Only the last of these slits was adjustable. Originally only two slits were used, but these proved inadequate as there was some evidence of scattering by the brass walls of the collimator tube.

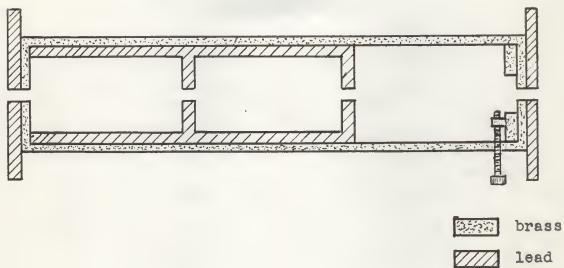


Fig. 3. Detail of collimator.

After the two middle slits were added, the beam appeared to be satisfactory. Detail of the collimator is illustrated in Fig. 3.

Crystal and Mounting

The crystal was mounted by cementing it to a rectangular block of brass, with the cleavage face of the crystal vertical. The brass block was held in place by the prism clamp.

The Ionization Chamber

The ionization chamber (Fig. 4) was made out of brass tubing with a lead slit facing the crystal. This shielding, however, proved inadequate; and it was necessary to shield the entire chamber with lead. Bakelite was used as insulation for the lead wire from the collector, and a copper guard ring was used to reduce the leakage. Since this was to be a simple elementary form of X-ray spectrometer, it was decided to use air at atmospheric pressure as the ionizing gas. This greatly simplified the construction of the chamber as it was not necessary to construct an air-tight container and did not require a special window for the admission of the X-rays. This, as already has been pointed out, involves a sacrifice of sensitivity since other gases, such as methyl iodide, argon, and xenon, are much better absorbers than air.

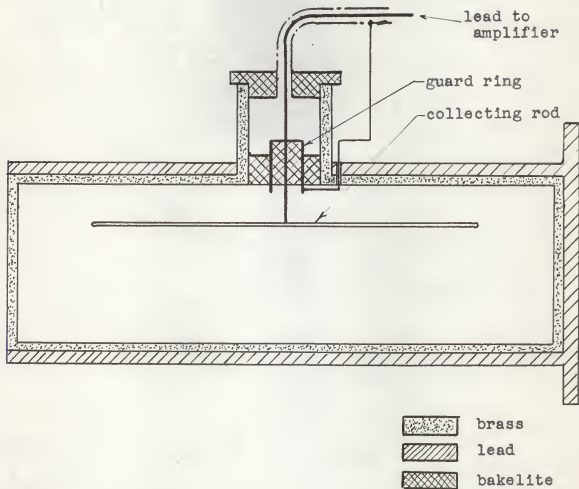


Fig. 4. Detail of ionization chamber.

The FP-54 D.C. Amplifier

In this study the ionization current was measured by means of an FP-54 D.C. amplifier. The FP-54 tube is specially constructed with an unusually high input resistance. This tube may be used as a D.C. amplifier of any small current applied across a fixed high resistance between the grid and filament.

Various circuits have been designed using these so-called "electrometer" tubes. The circuit used in this case was a modified Barth circuit (Fig. 5). In this circuit, small changes in the filament current are balanced out and therefore cause no change in plate current. Figure 5 is a schematic wiring diagram for the modified Barth circuit.

The single-tube electrometer circuit may be considered a Wheatstone bridge in which R_p and R_n form two of the resistance arms and the filament-to-plate resistance and the filament-to-space-charge-grid resistance form the other arms. Obviously, the condition for zero galvanometer current is

$$R_p I_p = R_n I_s$$

in which R_p and I_p represent the plate resistance and current respectively, and R_n and I_s represent the resistance and current in the space-charge grid circuit. R_d is not included in R_n and may be considered a part of the tube resistance. In order for the above condition to remain satisfied when the battery voltage changes or the filament emission changes for any reason, it is further necessary that

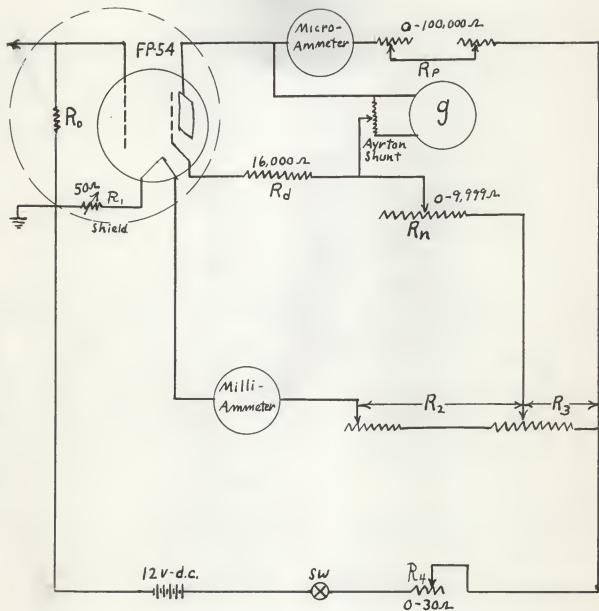


Fig. 5. Modified Barth single-tube electrometer circuit.

$$\frac{dI_p}{dI_f} = \frac{R_1}{R_2} \frac{dI_s}{dI_f}$$

For these conditions to be satisfied, it would be necessary that the I_p - I_f and I_s - I_f curves be straight lines intersecting on the I_f axis. Naturally no tube has these characteristics; but over short ranges, the tangents of the I_p and I_s curves do satisfy this condition and it is possible to adjust the resistances of the circuit so that this can be made to occur at roughly the rated filament current.

The balancing procedure, as suggested by Strong (14), is as follows:

With the galvanometer shunted to one tenth or one hundredth of its full sensitivity and R_p adjusted so that the galvanometer reads zero when I_f is near its rated value, I_f is slowly varied by means of the rheostat R_4 . With the galvanometer connected so that a positive deflection is caused by a decrease in the plate current, the deflection should pass through a maximum value of I_f . If the galvanometer deflection goes off the scale before the maximum is reached, it may be brought back by an adjustment of R_p . If the value I_f for maximum is not within a few percent of the rated value for the tube, the adjustment of R_2 - R_3 and R_p will bring the balance point to a different value I_f . The adjustment is finally made with the galvanometer at full sensitivity.

In practice, it was found that a slight modification of this method was more practical. A series of I_p - R_4 curves was plotted for different values of R_2 - R_3 from the data in Table 1 (Fig. 6). It is obvious that the value of R_4 corresponding to the horizontal portion of curve d is the one which gives the most stable circuit.

Table 1. Balancing curves for FP-54 D.C. amplifier.

R_4^*	Values of $R_2-R_3^*$			
	69	70	71	72
	a	b	c	d
67	--	15.5	35.1	--
68	--	22.0	42.4	2.5
69	0.5	26.4	45.9	12.0
70	5.6	29.7	46.2	18.2
71	14.0	29.9	44.0	20.3
72	20.0	28.3	38.8	21.2
73	26.4	24.7	35.5	22.7
74	28.5	22.7	27.8	22.5
75	33.0	19.2	20.0	22.4
76	40.5	17.8	17.0	22.8
77	--	17.1	12.0	25.5
78	--	19.9	11.6	28.6
79	--	28.5	16.0	39.5
80	--	48.0	--	--

*All resistance readings are dial settings.

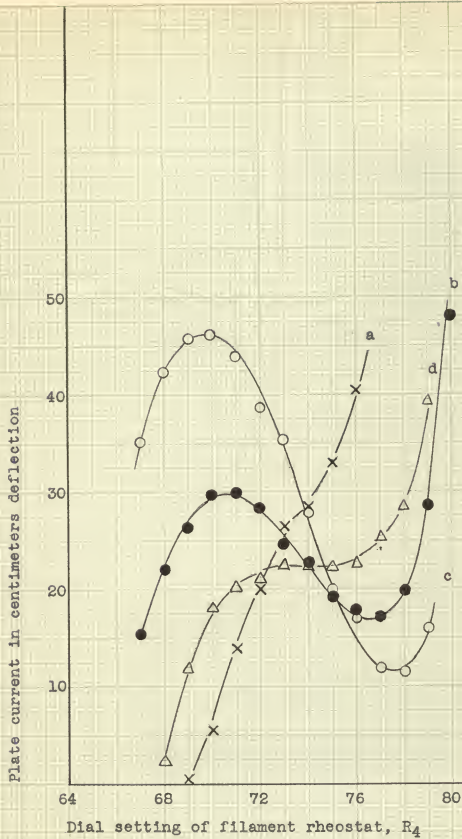


Fig. 6. Balancing curves for FP-54 D.C. amplifier.

The sensitivity of the FP-54 D.C. amplifier compares favorably with that of the best electrometers. A typical circuit has a sensitivity of 250,000 millimeters per volt with a galvanometer period of five seconds. A Compton quadrant electrometer, which is one of the most sensitive electrometers, has a sensitivity of 20,000 millimeters per volt and a period of fifteen seconds.

The above sensitivity is, of course, a maximum and was obtained by using extremely high input resistance and operating the tube in an evacuated chamber. In this experiment, such refinements were not considered necessary. The tube was operated in air and the input resistance was well below the suggested maximum of 10^{12} ohms. The sensitivity under these conditions was measured as 18,000 millimeters per volt which is roughly equal to that of the Compton electrometer.

CHARACTERISTIC EXPERIMENTAL CURVES

In this study, the following kinds of measurements were made: (a) the measurement of X-ray line spectra, (b) the measurement of total radiation as a function of the excitation voltage, and (c) the measurement of ionization current as a function of ionization chamber potential.

The Measurement of X-Ray Line Spectra

In order to measure X-ray line spectra, it was first necessary to determine the true zero setting of the spectrometer.

To determine this setting, the crystal was removed, the ionization chamber and collimator were then lined up, and readings were taken in the region of the expected zero (Table 2). These readings were plotted, using current as ordinate and angle setting as abscissa (Fig. 7). The peak of the curve is, of course, the true zero setting. The scale was then re-set at this true zero setting to eliminate the necessity of making corrections on all subsequent readings.

Table 2. Readings to determine the zero setting of spectrometer.

:Spectrometer setting :		Galvanometer deflection :	
:	359° 0'	:	5 cm.
:	359 12	:	21
:	359 24	:	31
:	359 36	:	44
:	359 48	:	78
:	0 0	:	70
:	0 12	:	38
:	0 24	:	29
:	0 36	:	12
:	0 48	:	4
:	1 0	:	2

Having determined the zero setting, steps were taken as follows:

1. The calcite crystal was placed so that its face cut the exact center of the prism table.
2. The ionization chamber was moved through small angles and the crystal set at half the prism angle. (This angle was obtained by sighting through the ionization chamber and rotating the crystal until the glow of the X-ray tube was seen by reflection from the crystal face.)

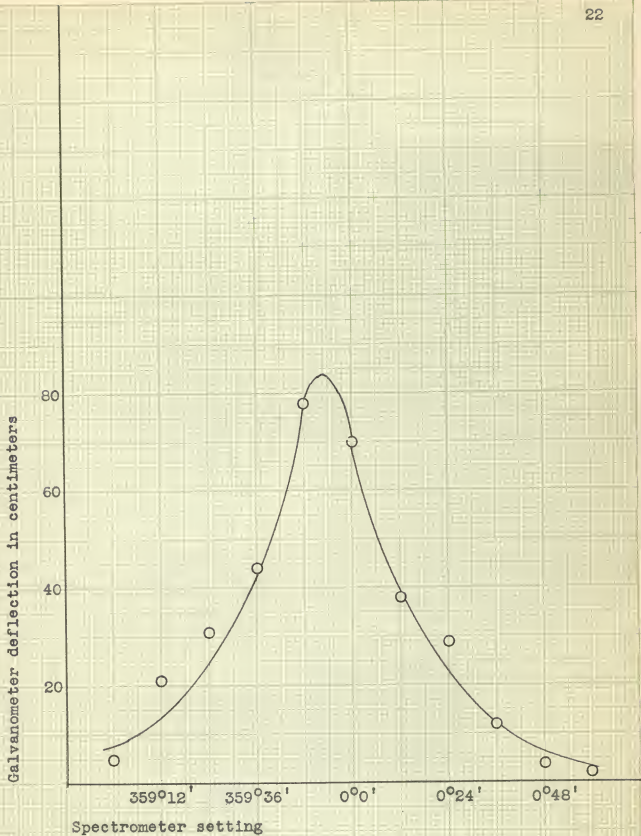


Fig. 7. Readings to determine the zero setting of the spectrometer.

3. This was repeated for many successive small angles in the vicinity of the expected lines. These data were tabulated in Table 3 and shown in Fig. 8. Fig. 8 illustrates the appearance of the K lines of the target with the increase in excitation voltage. It also shows the appearance of the first and second orders of the K lines in the 100 K.V. reading. The second order line appears at twice the angle for the first order line, which is in agreement with the Bragg formula.

The Measurement of Total Radiation as a Function of the Excitation Voltage

The purpose of this measurement was to determine the relation between the intensity of the total radiation and the voltage on the target of the tube. The crystal was removed, and the spectrometer was set at zero. The total radiation, as indicated by the deflection of the galvanometer in the FP-54 circuit, as a function of excitation voltage is tabulated in Table 4, and shown in Fig. 9.

The Measurement of Ionization Current as a Function of Ionization Chamber Potential

It was the purpose of this part of the experiment to find the voltage necessary to produce a saturation current. In this measurement the spectrometer was again set at zero and the ionization chamber potential varied from zero to 180 volts. The excitation voltage was kept constant. These data are recorded in Table 5, and illustrated in Fig. 10.

Table 3. Intensity of diffracted X-rays as a function of the angle of incidence of the X-ray beam for various excitation potentials (tungsten target).

Angle of incidence ; of X-ray beam		Galvanometer deflection (cm.)			
Degrees	Minutes	70 K.V. ; 7 M.A.	80 K.V. ; 7 M.A.	90 K.V. ; 7 M.A.	100 K.V. ; 5 M.A.
1	0	0	1.6	5	20
1	15	0	1.5	2.5	5
1	30	.1	1.4	3	4
1	45	.2	2	4	4
1	48	--	--	--	3.6
1	51	--	--	--	3.8
1	54	--	--	--	4.4
1	57	--	--	--	5
2	0	1	2.4	4	10
2	3	--	--	--	12
2	6	--	--	--	14
2	9	--	--	--	14
2	12	--	--	--	11
2	15	2	3.2	5	9
2	30	2	3	4	6
2	45	2	3.2	3.8	8
3	0	2	2.6	3.6	8
3	15	2	2.2	2.8	7
3	30	2.2	2.8	3	12
3	45	2.5	4	8	12
4	0	3	4	15	26
4	15	--	--	--	28
4	30	2	3.7	12	26
5	0	2	3	6	11
5	30	1.8	3	4	4
6	0	1.6	2.4	3	3
7	30	1.2	2	3	--

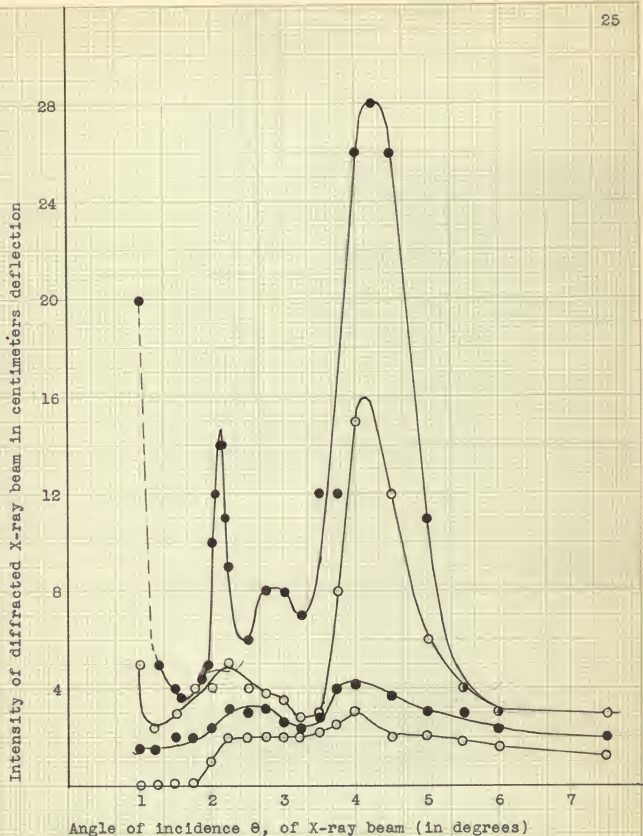


Fig. 8. Intensity of diffracted X-rays as a function of the angle of incidence of the X-ray beam for various excitation potentials (tungsten target).

Table 4. Readings showing relation between total radiation and excitation voltage.

Excitation voltage (kilovolts)	Total radiation (centimeters)
40	4
50	13
60	21
70	32
80	47
90	74
100	106
110	151

Table 5. Readings showing relation between ionization current and ionization chamber potential.

Ionization chamber voltage	Deflection (Cm.)
22.5	10
45	16
67.5	18
90	18
112.5	18
120	17
130	18
140	18
150	18
160	18
170	18
180	18

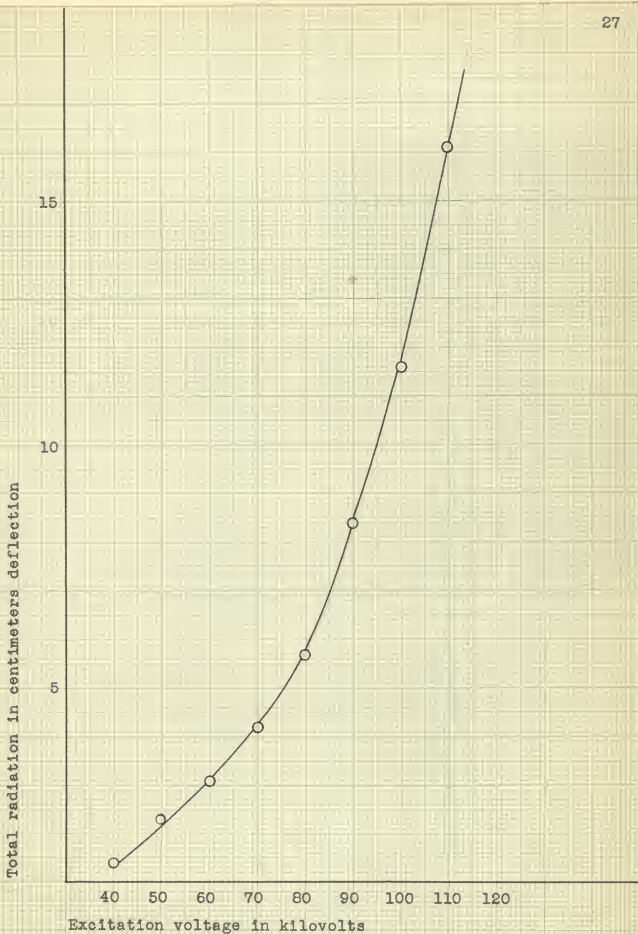


Fig. 9. Total radiation as a function of excitation voltage.

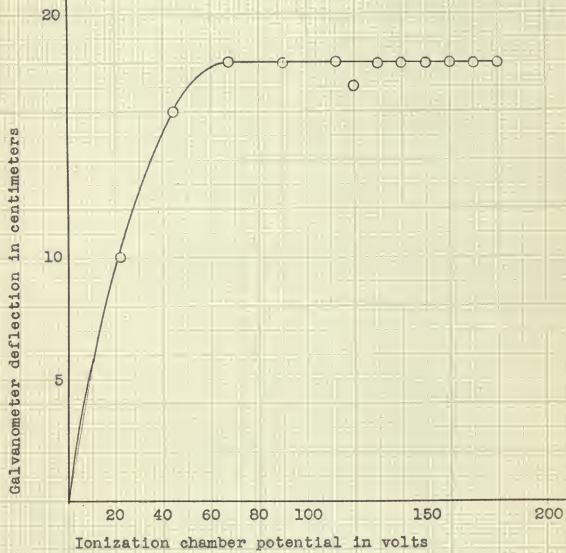


Fig. 10. Ionization current as a function of ionization chamber potential.

LIMITATIONS OF METHOD

The accuracy of this method of studying X-rays is limited by several factors. Some of the most prominent of these are as follows:

1. The slit must be sufficiently wide to produce a reasonable deflection. This will have the effect of broadening the peaks.

2. The method of setting the crystal, which was by sighting through the ionization chamber and rotating the crystal until the reflection of the red glow of the filament of the tube was seen, was by its nature rather inaccurate. Better results could be obtained by employing some mechanical device which would rotate the prism table with half the velocity of the ionization chamber.

3. The FP-54 d.c. amplifier is a very sensitive current measuring device, but unfortunately it is rather unstable and requires constant attention and frequent balancing.

4. Electromagnetic fields have an effect on the amplifier and must be taken into account. It is difficult to shield the entire PF-54 amplifier circuit and the lead wires from electromagnetic induction.

5. Unless a very constant source of potential is provided for the X-ray tube, the shift in the threshold of general radiation emitted, and the change in intensity of the general radiation may cause fluctuations in the ionization chamber.

CONCLUSION

Although this particular type of apparatus is not as accurate as some of the more elaborate ones, it, nevertheless, affords a reasonably accurate method of studying X-rays. The cost of making the change is very low, and is certainly justified by the variety of measurements possible.

Greater accuracy could be obtained by using some mechanical device for turning the crystal through half the angle of the ionization chamber. Also, some form of X-ray shielding should be used in the part of the amplifier lead nearest to the X-ray tube.

It would be desirable to use higher voltage than that used in the experiments. The apparatus used was limited to about 110 kilovolts. Better results could be obtained with higher voltages, especially when desirable to study the X-rays produced by K electrons.

ACKNOWLEDGMENTS

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