SOME OBSERVATIONS WITH A
CURVED CRYSTAL X-RAY SPECTROMETER

THESIS

Submitted by

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CHAPTER 1
INTRODUCTION

I. HISTORICAL

The study of X-ray spectra by means of crystalline diffraction had been carried on for many years before the identification of electro-magnetic radiation from radio-active nuclei by Ellis in 1922. The principal method used was the rotating crystal technique developed by Bragg\(^1\) and others, and this was applied to the study of the \(\gamma\)-rays emitted in the decay of radium (B+C) and radiothorium by Frilley\(^2\) and Thibaud.\(^3\) However, this method required very intense line sources, and it was to a large extent superseded by magnetic spectrometer methods, in which the internal conversion electrons associated with a \(\gamma\)-ray are examined. In addition to involving the use of considerably weaker sources, these methods were capable of making accurate measurements of higher energy \(\gamma\)-rays than had been possible by crystalline diffraction.

The curved crystal focusing X-ray spectrometer technique was first suggested by Dumond\(^4\) in 1930 and realized practically by Cauchois,\(^5\) Johann,\(^6\) and Johannson\(^7\) between 1931 and 1933. This technique considerably improves the collecting power of a crystal spectrometer. For the same resolving power, the exposure times can be reduced by a factor of about 50
compared with those required by the conventional Bragg spectrometer. It has been extensively used in the study of weak X-ray lines, principally by groups working in Paris \(^{(8-11)}\) and Uppsala \(^{(12-16)}\).

Very little attention was paid to the adaptation of this method to the investigation of \(\gamma\)-ray spectra until 1947. In that year Dumond \(^{(17)}\) constructed a curved crystal spectrometer of 2 metres radius of curvature with a view to making accurate determinations of the wavelengths of high energy \(\gamma\)-rays by direct crystalline diffraction. Since 1948 he has published several papers \(^{(18-22)}\) on the precision measurement of \(\gamma\)-ray wavelengths chiefly in the region above 150 KeV, and he has succeeded in measuring the 1.31 MeV \(\gamma\)-ray from Co\(^{60}\). This is the most energetic \(\gamma\)-ray whose wavelength has so far been measured by a direct crystal method \(^{(23)}\).

In 1949, another curved crystal spectrometer was constructed in Zurich by Marmier and his colleagues \(^{(24)}\). This has been used for identification of radio-active elements by means of their characteristic X-rays, but so far no measurements on \(\gamma\)-rays from radio-active sources have been reported.

This year another instrument, constructed in the University of California by Browne and Perlman \(^{(25)}\), has been used to examine the low energy \(\gamma\)-radiations emitted in the decay of Am\(^{241}\). The report on this
instrument has only been published in abstract form and as yet very few details of it are available.

II. OUTLINE OF RESEARCH

The object of the present research, started in 1948, was to construct a curved crystal spectrometer and to investigate the scope of such an instrument when applied to the study of $\gamma$-ray spectra, particularly in the region below 100 KeV, which is least suited to $\beta$-ray spectrometer methods. The chief difficulty encountered in this work has been the need for very intense sources and long exposure times, and considerable attention has been paid to attempting to reduce the exposures required.

Two instruments have been constructed in the course of this research. The first has a radius of curvature of 46.40 cm. and a dispersion on the focusing circle of 11 X.U. per mm. at small Bragg angles. This instrument is capable of giving accurate measurements of the wavelengths of low energy $\gamma$-rays correct to 0.2 X.U., corresponding to an accuracy of about 1 part in 1500 as regards energy measurements in the region of 40-50 KeV. It has been used to investigate the $\gamma$-ray spectrum of RaD in which Tsien\(^{(26)}\) reported the existence of several low energy $\gamma$-rays, in addition to the well-known 46.7 KeV $\gamma$-ray. In the course of this work the $L$ X-ray spectrum emitted in the decay of
RaD has also been examined and the wavelength of the 46.7 KeV $\gamma$-ray accurately determined.

The second instrument, designed as a result of experience gained in these experiments, has a radius of curvature of 20 cm. and a correspondingly lower dispersion of 25 X.U. per mm. at small Bragg angles. This spectrometer has a larger solid angle of collection from the same size of source than the first and it has been possible to reduce the exposure times by a factor of between 2 and 3, depending on the position of the source. While this instrument is better adapted to the discovery of new $\gamma$-rays, where high resolution or accurate determination of wavelengths is required the larger spectrometer is superior.

The two instruments have been used to examine the $\gamma$-ray spectrum of Pa$^{233}$, in which several low energy $\gamma$-rays were reported by Keller and Cork.$^{(27)}$ A recent investigation by Elliot and Underhill$^{(28)}$ failed to observe any $\gamma$-rays below 76 KeV. Of the three reported by Keller and Cork in this region, two have been observed in the present experiments.
Fig. (1)
CHAPTER 2

THEORY OF CURVED CRYSTAL SPECTROMETER

In order to help the understanding of the operation of the spectrometers described in this thesis, the relevant theory will be discussed in this chapter. It is largely based on the early papers of Dumond (4) and Cauchois (29) which give a full account of the mode of operation of transmission-type curved crystal X-ray spectrometers.

I. GENERAL THEORY

Fundamentally the problem consists of applying the principle of the Rowland concave grating to the case of X-ray spectroscopy.

Consider the two-dimensional case as shown in fig. (1). A is a point source of composite X-rays and B is the point image of a narrow wavelength band $\Delta \lambda$ of wavelength $\lambda$ in the spectrum of source A. The problem is to find a surface $CC'$ such that a flexible crystal conforming to it would, by Bragg reflection over an extended arc, take X-radiation from A and focus it selectively at B. This imposes two conditions at every point of the surface:

(a) At all points on the surface the angles of incidence and reflection referred to the atomic planes must be equal.
Fig. (2). The exact solution of Dumond.

(a) Reflection case. (b) Transmission case.
(b) At all points on the surface the angle of deviation $\phi$ of the reflected beam must be constant for any given wavelength $\lambda$ and is given by

$$\phi = 2 \arcsin \frac{n\lambda}{2d}$$

where $d$ is the spacing of the crystal planes used for reflection.

Condition (a) dictates only the direction of the atomic reflection planes and does not impose any condition on the boundary surface, while condition (b) dictates the position of every point on the surface but places no condition on the atomic reflecting planes. Thus, by suitably profiling the surface and simultaneously bending the crystal, it is possible to satisfy both conditions and theoretically obtain perfect focusing.

Fig. (2) shows the realisation of a crystal bent to satisfy these conditions. In fig. 2(a), the reflection case, the boundary of the crystal coincides with part of the circumference of a circle of centre O, while the reflecting planes are bent so that they coincide with concentric circles centred on the circumference at C. For all points on the surface condition (a) is fulfilled as arc SC = arc CI, and, as PC is normal to the reflecting planes, these arcs measure the angles of incidence and reflection. Condition (b) is also fulfilled as SI measures twice
the angle of deviation of the beam for all points on the surface.

Fig. 2(b) shows the corresponding transmission case, in which the neutral axis of the bent crystal coincides with the focusing circle. The short reflecting planes traversing the crystal, if produced, would meet at C diametrically opposite to C₁. The transmission case corresponds to a virtual source at V which is realised physically by the extended source as shown.

From considerations of geometry it may be seen that the reflection case is better adapted to long wavelengths and large Bragg angles, and the transmission case to short wavelengths and small Bragg angles. The present work was primarily concerned with γ-rays of wavelengths less than 1 Å and so transmission instruments were constructed.

The exact solution described above was employed by Dumond in his 2-metre spectrometer, using a suitably profiled curved crystal of quartz, but in general an approximate solution suggested by Cauchois has been used. She pointed out that condition (b), governing the position of the atomic planes, is less stringent than condition (a), governing the direction of the atomic planes. If a spectrometer is constructed conforming to the latter condition only, a geometrical aberration is introduced, causing an increase in line
Fig. (3). Principle of Cauchois transmission spectrometer.
width, but this is very small provided that the aperture of the crystal is not too large.

For reasons to be discussed later, the present spectrometers employ as curved crystals sheets of mica, and it would be extremely difficult to profile the surface of this crystal to satisfy the exact focusing conditions. The approximate focusing case has therefore been used, and the associated theory will now be considered in more detail.

II. THE CAUCHOIS TRANSMISSION SPECTROMETER

The previous theory considered the case where the reflecting planes used were normal to the crystal slab, so that, when the crystal was curved, they all pointed towards the centre of curvature of the crystal. In this section, the more general case of oblique planes inclined at an angle $\alpha$ to the normals will be considered.

In fig. (3), $CC'$ is an extremely thin crystal bent parallel to its cleavage planes, so as to assume a circular section of radius of curvature $R$ and centre of curvature $O$.

The transverse atomic planes are inclined at an angle $\alpha$ to the normals to the crystal slab, and when produced meet at $P$.

Let $ACI$ be a ray of wavelength $\lambda$, from an extended source, incident on the atomic plane at $C$ at an angle $\phi$
such that the Bragg reflection condition \(n\lambda = 2d \sin \phi\) is satisfied. Only rays of the wavelength satisfying this condition will be reflected at this angle \(\phi\).

Let \(u = \angle OCI = \phi - \alpha\)

and \(\theta = \angle D\bar{O}C\)

Consider \(O\) as centre of co-ordinates

\(C\) will be the point

\[x = -R \sin \theta\]
\[y = R \cos \theta\]

\(CI\) will make an angle \((90^\circ + u + \theta)\) with the \(x\)-axis.

The equation of \(CI\) is

\[(y - R \cos \theta) - (x + R \sin \theta) \tan (90^\circ + u + \theta) = 0\]

or

\[(y - R \cos \theta) \tan (u + \theta) + (x + R \sin \theta) = 0 - - - -(1)\]

It is required to find the locus of the intersection of rays from the source striking the crystal \(CC'\) at this angle \(\phi\) at positions corresponding to different values of \(\theta\). Differentiate (1) with respect to \(\theta\) and solve for \(x\) and \(y\) using the equation obtained and equation (1).

This gives

\[x = R \sin u \cos (u + \theta)\]
\[y = R \sin u \sin (u + \theta)\]

If the aperture \(CC'\) is limited, then the locus of intersection is the arc \(FF'\) of a circle of radius \(R \sin u\) and centre \(O\), the intersecting rays being tangents to the circle.
If the aperture is small enough, so that \( \Theta \approx 0^\circ \), then the rays intersect at the point

\[
\begin{align*}
x &= R \sin u \cos u \\
y &= R \sin^2 u
\end{align*}
\]

The locus of points for different values of \( \phi \) (or \( u \)) and so corresponding to rays of different wavelengths will be a circle of equation

\[
x^2 + (y - \frac{R}{2})^2 = \frac{R^2}{4}
\]

whose centre has co-ordinates

\[
\begin{align*}
x &= 0 \\
y &= \frac{R}{2}
\end{align*}
\]

i.e. \( 0^\circ \) of fig. (3)

This circle is known as the focusing circle, and, if we have a composite source of X-rays, rays of different wavelengths will be focused along the circumference of this circle.

III. **LINE WIDTH DUE TO GEOMETRICAL ABERRATIONS**

So far only the two-dimensional case has been considered, but in order to determine the geometrical line width the three-dimensional case will now be considered.

The width of the line may be due to any one of four effects:

(a) The finite thickness of the crystal.

(b) The width of the crystal aperture, i.e., the approximation \( \Theta = 0^\circ \) being not strictly true.

(c) The height of the crystal aperture.
(d) Misorientation of the crystal, i.e. the normals to the crystal planes not lying in the same plane as the cross-section of the cylindrical surface.

Cauchois pointed out that in the transmission case there is a focusing effect through the thickness of the curved crystal. This is due to the fact that when the crystal is curved, the grating spacing is increased in the region of the crystal outside the neutral axis, and so the Bragg angle at a specified wavelength is decreased. The opposite effect occurs in the region inside the neutral axis. This change of Bragg angle is such that the rays have the exact convergence to give perfect focusing from front to back. The analysis of this problem by Cauchois is only true for planes normal to the crystal, i.e., the angle $\alpha = 0^\circ$. If the planes are oblique, exact focusing is not obtained but the contribution to the line width is extremely small.

There remain the three other causes of the line width. To determine the magnitude of these effects, the three-dimensional case, as shown in fig (4), must be considered. In the following analysis, it will be assumed, for the sake of clarity, that the reflecting planes are normal to the crystal slab, i.e., $\alpha = 0$.

In fig. (4):

$O$ is the centre of curvature of the crystal.

The semi-aperture of the crystal ED subtends an
Fig. (4). 3-dimensional geometry used in calculation of line width.
angle $\omega$ at $0$.

ORD is a part of the focusing circle.

SC'R is a ray incident on the crystal at C'.

This ray strikes the crystal planes at the Bragg angle $\phi$ and is reflected and cuts the focusing circle at the point $R$.

Take $0$ as the centre of co-ordinates.

$C'$ is the point

\[
\begin{align*}
X &= R \cos \omega \\
Y &= R \sin \omega \\
Z &= Rm
\end{align*}
\]

where $m = \frac{h}{2R}$ and $h$ = height of crystal aperture

Let the normal to the crystal planes be inclined at an angle $\delta$ to the cross-section of the cylindrical surface. The direction cosines of this normal at $C'$ will be given by

\[
\alpha = -\cos \delta \sin \omega \\
\beta = \cos \delta \cos \omega \\
\gamma = \sin \delta
\]

The ray $C'R$ makes an angle of $(\frac{\pi}{2} - \phi)$ with this normal. This ray cuts the focusing circle at $R$ given by

\[
\begin{align*}
x &= R \sin^2 \theta \\
y &= R \cos \theta \sin \theta \\
z &= 0
\end{align*}
\]

Let $\xi$, $\eta$, and $\zeta$ be the direction cosines of this ray.

\[
\alpha \xi' + \beta \eta' + \gamma \zeta' = \cos(\frac{\pi}{2} - \phi) = \sin \phi
\]

\[
\frac{x - X}{\xi'} + \frac{y - Y}{\eta'} + \frac{z - Z}{\zeta'} = 1
\]

\[
\xi'^2 + \eta'^2 + \zeta'^2 = 1
\]
Eliminate \( \xi \), \( \eta \), and \( \xi \)

\[ (\alpha + \beta + \gamma)^2 = \sin^2 \phi \left[ \left( (x - x)^2 + (y - y)^2 + (z - z)^2 \right) \right] \]

\[ \left[ \cos \delta \sin \theta \cos (\omega + \Theta) - m \sin \delta \right]^2 = \sin^2 \phi \left[ 1 + m^2 + \sin^2 \Theta - 2 \sin \Theta \sin (\omega + \Theta) \right] \]

If \( \omega = 0 \) and \( m = 0 \), the value of \( \Theta = \Theta_0 \)
corresponding to the central ray is given by

\[ \sin \Theta_0 = \frac{\sin \phi}{\cos \delta} \]

If \( \omega \), \( m \), and \( \delta \) are small, then \( \epsilon = \Theta - \Theta_0 \) is an
infinitesimal quantity of the second order and is given
by

\[ \Theta - \Theta_0 = \epsilon = \frac{m^2}{2} \frac{\sin \phi}{\cos^2 \phi} + m \delta \frac{1}{\cos \phi} + \frac{\omega^2}{2} \tan \phi \]

Let \( \ell = \epsilon R = \) line width on the focusing circle.

Let \( \sigma \) = linear aperture of crystal = \( 2 R \omega \)
and \( h \) = height of crystal aperture = \( 2 R m \)

Hence \( \ell = \frac{\tan \phi}{8R} \left[ \sigma^2 + \frac{h^2}{\cos^2 \phi} \right] + \frac{h}{2} \frac{\delta}{\cos^2 \phi} \)

In the case where the crystal planes are inclined
at an angle \( \alpha \) to the normals to the crystal slab, the
analysis is very similar, if instead of \( \phi \) the angle
\( u = \phi - \alpha \) is used.

The result in this case, to the same degree of
approximation, is given by

\[ \ell = \frac{\tan u}{8R} \left[ \sigma^2 + \frac{h^2}{\cos^2 \phi} \right] + \frac{h}{2} \frac{\delta}{\cos^2 \phi} \]

In the case of the large spectrometer the values
of the variables in this equation were:

- Maximum value used for \( u = (\phi - \alpha) = 8^\circ \)
- \( R = 46.4 \) cm.
- \( \sigma = 2.5 \) cm.
- \( h = 0.65 \) cm. and \( \delta < 4^\circ \)
Fig. (5). The two methods of using a transmission-type curved crystal spectrometer.
This would result in a theoretical line width of
\[ l = 0.0024 + 0.0002 + 0.001 = 0.0027 \text{ cm}. \]

The largest contribution to this expression comes from the term involving the width of the crystal aperture, so if sharper lines are required, the width of the crystal aperture should in the first instance be reduced.

In this spectrometer the value of the line width obtained was 0.016 cm., which is considerably larger than the theoretical value. This is due to the lack of perfect crystalline structure in the mica. Consequently, the advantage to be gained by using Dumond's exact solution would not have been very great, even if it had been practicable to profile the mica. In his spectrometer, Dumond used a crystal of quartz, which has more perfect crystalline properties and in which, therefore, a greater advantage is obtained by using the exact solution.

IV. OPTIMUM POSITION OF SOURCE

In the design of his 2-metre spectrometer, Dumond pointed out that there were two possible methods of using a transmission-type curved crystal spectrometer. These are shown in fig. (5), reproduced from his paper. Either an extended source can be placed at A or a very concentrated source at R. In the first case the spectrum is recorded by a photographic emulsion placed
on the focusing circle, and in the second case by an ion-chamber or counter placed as shown, the counting rate being plotted as the sample is moved round the circle. If the source is placed at R, then the solid angle into which each atom of the source can radiate a specified wavelength is that angle subtended by the whole crystal aperture. This angle is considerably greater than that into which each atom from A can radiate in the correct direction for reflection of the required wavelength. If the two cases are compared in which the spectrum is explored, firstly with an extended source and a counter moving round the focusing circle, and secondly with an extended counter and a source, of the same total activity, moving round the focusing circle, then the counting rate for a γ-ray of given wavelength would, in the second case, be very much larger. However, if the instrument is designed to be used photographically, then a considerable range of wavelengths can be examined on one plate, and to a certain extent the advantage gained by using the second arrangement is lost. In addition, if a very concentrated source is being used, in the case of low energy γ-rays the self-absorption of the source may become appreciable, particularly if the L X-ray spectrum of the element, excited by internal conversion processes, is being examined. If high energy γ-rays, above 150 KeV, are being examined, the photographic
method tends to lose its advantage, as in general self-absorption of the source is inappreciable and the sensitivity of a photographic emulsion falls off rapidly.

The present work has been entirely concerned with $\gamma$-rays of energy less than 100 KeV and so all spectra have been recorded photographically, no attempt having been made to use the Dumond modification, though its advantages in the higher energy region, above 200 KeV, are evident.
CHAPTER 3
DESIGN AND CONSTRUCTION OF 46.40 cm. SPECTROMETER

I. THE SPECTROMETER

Choice of Radius of Curvature of Crystal

In a curved crystal spectrometer, it is possible to increase the dispersion and resolving power of the instrument by increasing the radius of curvature of the crystal used. In general, however, the improvement thus gained results in a loss of luminosity and hence increased exposure times are required. Any instrument must be designed as a compromise between resolving power and luminosity. In the present case it was decided to construct an instrument of fairly high resolving power and to determine whether it had sufficient luminosity for useful work with γ-ray sources. It was decided to make the radius of curvature approximately 50 cm. and, as a result of constructional details discussed later the actual radius used was 46.40 cm. It was estimated that, using atomic planes of grating spacing less than 3 Å, this would be sufficient to resolve lines differing in wavelength by 1.5 X.U.

Relative Positions of Source and Crystal

In order to cover a large range of Bragg angles, or to use different sets of planes if these are available, either the source or the crystal must be
Fig. (6). Constructional details of 46.40 cm. spectrometer.
capable of being moved. Since, in the case of radioactive sources, heavy lead collimation is usually required to prevent fogging of the photographic plate due to extraneous high energy $\gamma$-rays, it was decided to mount the crystal so that it was capable of rotation.

**Constructional Details**

The basic details of the spectrometer as finally constructed are shown in fig. (6). The blocks in which the crystal was clamped, (H and H') are mounted on a male cone F, which is rigidly attached to a large plate A which runs on a ball-race. In this plate there was a circular hole already cut whose centre was 23.20 cm. from the axis of rotation of the cone. It was decided to use the centre of this hole as the centre of the focusing circle, and this required that the radius of curvature of the crystal would be 46.40 cm.

Referring to fig. (7), it can be seen that if N is the centre of the camera, to ensure that this point always lies on the focusing circle, the distances OP and ON must be equal for all positions of the camera. The base plate B (fig. 6), on which the camera was mounted, swivels on a small ball-race C whose centre of rotation is O, the centre of the focusing circle. The distance from O to the centre of the camera was made exactly 23.20 cm., thus satisfying the condition
To make absolute measurements of wavelengths it is only necessary to measure the angle \( u \) of fig. (7), provided that the lattice spacing \( d \) of the planes used and the inclination \( \alpha \) of these planes to the direction PO are known. To measure this angle it is necessary to realise the direction of PN physically. This is done by means of the bar D which is attached to the female cone E. The centre of the camera is located on this bar, and, as the distance PN changes for different positions of the camera, the point N was capable of sliding along the bar, being located by a ball-bearing running in a groove cut in the bar. The direction of the groove was such that it pointed to the centre of the female cone E. This cone bears directly on the male cone F whose axis passes through the centre of the crystal (point P of fig. 7). As the axes of rotation of both cones will coincide this ensures that the direction of the groove is in fact PN.

The angle is measured by means of a reading microscope (not shown in fig. 6) rigidly attached to the bar D, which reads, through the totally reflecting prism G, a scale S which was centred on the plate. The scale was obtained from Messrs. Hilger and Watts, London, and is graduated in thirds of a degree to a guaranteed accuracy of 5" of arc. By means of a scale in the eyepiece of the reading microscope it is
Fig. (8). The adjusting mechanism for levelling and centring the crystal.
possible to read positions on the scale correct to 10" of arc. The scale was centred on the plate A by observing the difference in angle between two fixed reading microscopes, approximately 180° apart and adjusting the position of the scale so that on rotation of the plate this difference remained constant. The accuracy of the adjustment is determined by the reading accuracy of the scale and so all angles measured on the scale are correct to within 10" of arc.

The crystal clamping blocks H and H', together with the adjusting mechanism necessary for levelling and centring the crystal, are mounted on top of the cone E. These are shown in more detail in fig. (8). The crystal clamping blocks are mounted on the plate J and the centring process is carried out by means of the three screws K bearing on this plate, which is clamped by the screws L when in the correct position. This method ensures that, during the centring process, no additional strains are placed on the crystal clamping blocks, thus distorting the curvature of the crystal. The levelling of the crystal is carried out by means of the screws M, one of which fits into a groove so that there is no tendency for the clamping blocks to rotate independently of the whole system. In fig. (8) the ball-bearing which is the third point of support for the levelling system is shown in section for the sake of clarity.
Fig. (10). The 46.40 cm. spectrometer.
Fig. (9). Details of camera support.

Fig. (10), photograph of completed apparatus, is reproduced on the previous page. The camera shown is the one constructed for holding 3 in. by 1 in. photographic plates.
The details of the camera support attached to the base plate are shown in fig. (9). The centre of the film is located directly above the centre of the ball-bearing B and the bar D is spring-loaded as shown so that it is always in contact with the ball B. The spectrometer was initially designed for use with X-ray film, and the camera consisted of an aluminium frame bent into a radius of curvature of 23.20 cm., into which fitted a piece of X-ray film 5" by 1". The camera was maintained in position and at the correct radius of curvature by fitting into slots in the two pieces of perspex R and S, which were spaced by pieces of perspex fitting over the brass supporting columns T. When it was decided to use β-sensitive plates, another simple aluminium plate-holder was designed which fitted on to the columns T, so that the plate was always tangential to the focusing circle at the point corresponding to the centre of the plate.

A view of the completed apparatus is shown in fig. (10). The reading microscope not shown in fig. (6) can be seen, and also the lead collimator used during experiments on the γ-rays emitted by RaD.

II. THE CRYSTAL CLAMPING BLOCKS

The crystal used in the spectrometer was a piece of muscovite mica 2½" square by 0.5 mm. thick. In order to bend it to the correct radius of curvature it was
Fig. (11). The crystal clamping blocks.
clamped between two hardened steel blocks, on one of which had been profiled a concave circular cylindrical surface of 46.40 cm. radius and on the other a corresponding convex surface. The blocks were 3\" square by approximately 1\½\" thick. To allow the $\gamma$-rays to pass through, a window was cut in the blocks, the sides being tapered as shown in fig. (11) to prevent the reflection of rays from them fogging the film when the camera was set at small Bragg angles. The size of the window was limited by the necessity of maintaining the correct radius of curvature in that part of the mica covering the aperture, and therefore receiving no support from the blocks. Optical testing showed that the size chosen, 1\" by 1\½\", was sufficiently small to ensure this.

As the radius of curvature of the surfaces to be profiled on the blocks was too large to be generated conveniently on an available lathe, it was decided to use a method suggested by Dumond, Lind and Cohen\(^{(30)}\) for precision grinding of circular cylindrical surfaces of large radii of curvature by means of a surface grinder. The procedure used in grinding the present blocks was practically identical with that fully described in their paper. It might have been simpler to generate the surfaces by some other method, but it was thought that the experience gained in using this method would prove useful if in the future a
spectrometer of larger radius of curvature, giving higher resolution, was constructed.

It is important in this work to obtain as near as possible an optical finish on the surfaces of the blocks and so, after being ground, they were lapped, using cast-iron laps ground to the same radius of curvature. The lapping process was carried out using a simple machine designed to keep the generators of the surfaces parallel, similar to the one described by Dumond and his colleagues in the paper referred to above.

Initially blocks were constructed of brass but the resolution obtained during X-ray tests on the spectrometer was not as good as had been expected. This was thought to be due to the difficulty of obtaining a good finish on a soft material like brass and so it was decided to construct the blocks of hardened steel. The steel used was KE 595/371, supplied by Messrs. Kayser Ellison, Ltd. It was chosen since it had the property of changing its dimensions only very little during the hardening process, thus making it possible to grind the surface to very near its finished size before hardening.

During the lapping process several compounds were tried, to determine the best procedure for lapping this steel with cast-iron blocks. It was found most satisfactory to use Carborundum Lapping Compound,
grade H 240 WP, followed by grade H 500 WP for initial breaking down of the surfaces. This was followed by grade 2A 700 WP to produce the first polish, and the final polish was obtained using either Dragon's Blood or 'Silvo'.

The grinding and lapping procedure used gave a concave and convex block with surfaces of the same radius of curvature, no allowance being made for the thickness of the crystal to be used. The crystal was 0.5 mm. thick and the convex block alone determined the radius of curvature of the crystal, since a thin rubber gasket was placed between the crystal and the concave clamping block.

**Testing of Blocks**

As a result of the lapping process, a good mirror finish was obtained on both blocks and the curvature of the concave block was tested by a Foucault knife-edge test. The radius of curvature was found to be $46.40 \pm 0.03$ cm.

The crystal was clamped between the blocks, care being taken to exclude dust particles, and the pressure on the clamping bolts was adjusted so as to obtain as narrow a line image as possible. The quality of this image was not sufficiently good for a knife-edge test, due to reflections from the back surface and the doubly refracting properties of the mica. An object and image test showed that the radius of curvature of the
mica across the crystal aperture was $46.40 \pm 0.1$ cm. Sections of the crystal aperture were next tested individually and all were found to have this radius of curvature, within the experimental error. The performance of the crystal was then tested using X-rays and found to be satisfactory.
Fig. (12). Geometrical arrangement of X-ray tube and spectrometer, such that no direct radiation from the anti-cathode falls on the crystal.
CHAPTER 4
Preliminary Experiments with X-rays

I. Quality of Focus Obtained

After the construction of the apparatus, a series of tests was carried out to determine the focusing qualities of the instrument. These experiments were mainly performed using the (201) planes of a sheet of mica to examine the fluorescent K X-rays emitted by silver.

The general arrangement used is shown schematically in fig. (12). It is a Phillips Metallex X-ray tube which has a tungsten anti-cathode. Immediately in front of the body of the tube there was a cap (C), whose purpose was to hold an aluminium filter to remove the soft X-radiation, if this was required. In the present experiments this filter was replaced by a thin piece of silver 28 mgm/cm² thick, which was used as a silver radiator. Two positions of the X-ray tube were used in these experiments. In the first case, the relative positions of the X-ray tube and the spectrometer were such that only radiation from the silver scatterer, and no direct radiation from the anti-cathode, fell on the crystal. In the second case, the tube was rotated so that radiation from both the silver scatterer and the anti-cathode fell on the crystal. In this case, in addition to the silver
Fig. (13). Photographs showing performance of 46.40 cm. spectrometer.

(a) Silver K spectrum obtained with arrangement shown in fig. (12).
(b) Silver K spectrum with tungsten K spectrum in second order, obtained by rotating the tube so that radiation from the anti-cathode falls on the crystal.
K-spectrum, there was obtained the tungsten K-spectrum and a certain amount of continuous spectrum.

Considerable attention was paid to the placing of lead shielding in order to prevent fogging of the film caused by stray primary radiation. The X-ray tube itself was enclosed in a box covered with $\frac{1}{8}$" sheet lead, the radiation being emitted through an aperture in the side of the box. This was found to be sufficient to prevent fogging of the film if the geometrical arrangement shown in fig. (12) was being used, but if, as in later tests, the scatterer was placed nearer the crystal, additional shielding was required.

The performance of the spectrometer using the final sample of mica is shown in fig. (13). The first spectrum is that of the silver fluorescent radiation alone and shows the complete resolution of the silver K\(\alpha\) doublet. The exposure in this case was 10 minutes with 1 mA passing through the tube at 70 KV. The second spectrum shows, in addition to the silver K\(\alpha\) doublet, the tungsten K\(\alpha\) doublet in the second order, the whole superimposed on continuous radiation from the anti-cathode. It was during tests similar to these that the performance of the brass clamping blocks was found to be disappointing and it was decided to change over to hardened steel blocks. The photographs reproduced are those obtained using the spectrometer.
in its final form.

After the general performance, as indicated by these tests, had been found to be satisfactory, there remained three main problems to be considered before attempting to carry out experiments using γ-ray sources. These were:

a) The choice of the most suitable crystal to use as regards intensity of reflection consistent with good resolution.

b) The choice of the most suitable type of detector.

c) The estimation of the exposures that would be required with radio-active sources.

These problems will now be considered in some detail.

II. CHOICE OF CRYSTAL

The crystals that have been most frequently used in transmission-type curved crystal spectrometers have been muscovite mica and quartz. Quartz has the more perfect crystal structure and so would be expected to produce sharper lines. However, the reflecting power of a crystal depends to a large extent on its mosaic structure, and, using the available sets of planes of mica, it is possible to get greater intensity than with those of quartz. As the sources used in this work are very weak compared with X-ray sources, it was decided to use mica because of its higher reflecting
power. The difference in line widths obtained using mica and quartz is very small, and Cauchois\(^{(31)}\) has stated that, if great care is taken in the selection of the crystal, it is possible to obtain sharper lines using mica.

**Properties of Muscovite Mica, \(\text{KAl}_2(\text{AlSi}_3)\text{O}_{10}(\text{OH})_2\)**

Muscovite mica is known to be a monoclinic crystal, its structure having been worked out by Jackson and West\(^{(32)}\) and by Maugin.\(^{(33)}\) The nomenclature used to describe various planes is different in these two papers, and will be discussed in an Appendix. For present purposes the nomenclature used will be that of Maugin, which has been used by Cauchois and other workers in curved crystal spectroscopy.

Mica has two sets of planes which are suitable for use in curved crystal transmission spectrographs. These are the \((\bar{2}01)\) planes of grating spacing \(d = 2.65\ \text{Å}\) and the \((100)\) planes of grating spacing \(d = 2.61\ \text{Å}\). The angles of inclination of these sets of planes to the normals to the cleavage surfaces are \(4^\circ 56'\) for the \((\bar{2}01)\) planes and \(10^\circ 10'\) for the \((100)\) planes.

The \((\bar{2}01)\) planes of mica have a high reflecting power in both the first and second order, and so are useful when measurements are required of high energy \(\gamma\)-rays, or when high resolution is required. The possibility of having reflections from two orders may lead to difficulties in the interpretation of the
spectra obtained, but a method of overcoming these difficulties will be discussed in section IV of this chapter.

The (100) planes have a very high reflecting power in the first order and no second order reflections have been observed using them. The reflecting power is approximately twice as great as that of the (201) planes, and so in general they have been used for experiments with γ-ray sources, although the line-width obtained using them is slightly greater than in the case of the (201) planes.

There is a considerable variation in the properties of different samples of mica, and so a large number of samples were investigated in order to find the most suitable crystal for use in the present spectrometer.

Most Suitable Type of Mica

It was reported by Preiswerk (34) that there was a large variation of the reflecting power of the (100) planes in different samples of mica. This variation would be of great interest in the present case, and so experiments were carried out to investigate it.

The technique used was as follows. Laue photographs of different samples of mica of equal thickness were taken under identical conditions of exposure and development. The relative intensities of the spots corresponding to the (100) planes were examined visually. By this method, very little
Fig. (14). Laue photograph of Australian mica.

Fig. (15). Laue photograph showing twinning effect.
variation in the reflecting power of the (100) planes could be detected in the samples tested.

A typical Laue photograph is shown in fig. (14).

With some samples of mica a twinning effect was observed in some of the spots, which in general indicates that there is a slip of the atomic planes at some point in the thickness of the mica. An extreme example of this twinning effect is shown in fig. (15). These samples of mica were found to be useless in the curved crystal spectrometer, and latterly this was used as a criterion for rejection.

As several samples of mica showed equally good properties as demonstrated by a Laue photograph, these were tested in the spectrometer, using the silver Kα doublet as a resolution test. The samples of mica used in these tests all conformed to the following specifications:

a) They showed no twinning effects in Laue photographs.

b) They had good splitting properties, so that it was possible to obtain a truly parallel sheet of mica.

c) They contained few air inclusions.

Before describing these tests, the method employed to orientate the crystal between the clamping blocks will be discussed, since misorientation can contribute considerably to the line width, and hence
impair the resolution.

**Orientation of Mica**

The most accurate method of determining the direction of the (100) and (201) planes is by reference to a Laue diagram with the crystal in position between the clamping blocks, but this is a very tedious procedure when a large number of samples is being investigated, and so a simpler method was used. In mica, the normals to the (100) planes are parallel to the optic axis (Winchell\(^{35}\)), whose direction can be determined by means of a polarisation microscope, correct to less than half a degree. The samples of mica to be investigated were all examined in a polarisation microscope and a fine line inscribed on them indicating the direction of the optic axis. This line was used in orientating the crystal between the blocks, and it was estimated that this adjustment was correct to within half a degree. A misorientation of this magnitude would contribute 0.003 cm. to the line width, which would be unimportant in resolution tests using the silver Kα doublet.

**Resolution Tests**

The samples of mica were examined using the silver Kα doublet, and the most suitable types of mica were found to be a piece of Bengal Ruby Mica obtained from Messrs. Mica and Micanite Ltd., and a piece of Australian mica obtained from Professor Tolansky. There
was little to choose between the two samples when using the (201) planes, but the Australian mica was clearly superior for use with the (100) planes, and so it has been used in all subsequent work with this spectrometer.

In these experiments, there was more indication of the variation of intensity of reflection between various samples of mica, as reported by Preiswerk. This was due to the fact that some samples produced diffuse lines, many times wider than the sharp ones produced by the Australian mica. This defocusing effect causes less contrast between the line and the background and consequently an apparent loss of intensity even though the total blackening is the same.

**Final Orientation of Mica**

It is possible to orientate the crystal of mica more accurately by use of a Laue diagram than by reference to its optic axis, and so, after the most suitable crystal had been selected, this adjustment was improved. A base line, exactly parallel to the cross-section of the curved surface, was drawn on a piece of X-ray film placed at a distance of 20 cm. from the crystal. A Laue photograph was taken with the crystal between the clamping blocks. In the correct adjustment, the line joining the spots corresponding to the (100) and (201) planes would be parallel to the
base line on the film, and successive adjustments were made until this condition was satisfied. By this method it was possible to adjust the mica so that the orientation was correct to less than 10' of arc, and hence the contribution to the line width due to misorientation of the mica was practically negligible.

**Australian Mica Used in the Spectrometer**

This type of mica is mined in the Rex Mine in the Harts Range of Central Australia. Tolansky, who has investigated the optical properties of a large number of different samples of mica\(^{(36)}\) found this type to be far superior to any previous samples he had tested. It has very good splitting properties and is fairly free from air inclusions.

**List of Types of Mica Tested**

The samples of mica tested for use in the spectrometer were:

- Australian
- Bengal Ruby
- Madras Ruby, A, B, and C qualities
- Madagascar Amber
- First and Second Quality Spotted
- Madras Clear Green
- East Africa Clear Green
- Tanganyika Ruby
- and several samples of unknown origin, already in this Department.

III. CHOICE OF DETECTOR

The apparatus was initially designed for use with X-ray film, but various types of photographic plates were investigated in order to determine the most suitable type to use.

The first experiments were to compare the relative merits of different types of X-ray film. The tests were carried out using the silver fluorescent radiation. Very little difference could be detected between the speed of double-coated Kodirex and Ilfex. If Ilford Red Seal film was used with normal intensifying screens, there was an increase in speed of a factor of slightly less than 2 but in addition a considerable broadening of the spectral line, even when the intensifying screens were clamped tightly against the film. If fluorazure intensifying screens were used with Red Seal film, the increase of speed over Kodirex was by a factor of approximately 3, but there was a still further broadening of the spectral line, so that, using the (100) planes, the silver K\(\alpha\) doublet was no longer resolved.

Kodirex X-ray film was next compared with the \(\beta\)-sensitive 200\(\mu\) thick nuclear emulsions produced by
Kodak and Ilford. No difference in speed could be detected between the Kodak NT4 and the Ilford G5 emulsions, but the G5 plates were found to be more reliable as regards processing, and so they have been used throughout this work. As regards blackening, these plates were found to be 8 times as fast as Kodirex film at 22 KeV and 16 times as fast at 60 KeV. These tests were carried out using silver Kα X-rays and tungsten Kα X-rays and determining the time required to produce a line just visible to the eye. Kodirex X-ray film is only 40 μ thick as compared with 200 μ for the nuclear emulsion. It was thought that this might explain the difference in speed, and so Ilford and Kodak were asked if it was possible to produce X-ray plates with emulsions thicker than 100 μ, but so far have been unable to do so. Ilford produced one batch of plates 300 μ thick, but it was found impossible to process these satisfactorily.

In all measurements where photographic recording has been used, Ilford G5 200 μ thick β-sensitive plates have been used, and their properties will be considered in more detail in the next section. The processing of these plates is rather tedious, taking at least 10 hours, so, in experiments concerned with the adjustment of the spectrometer using X-rays, Kodirex film was used, since this could be processed in 20 minutes.
Experiments were also carried out using an argon-filled Geiger-Muller counter of G.E.C. type G.M.4, to see whether a counter would have any advantage over a photographic plate. The actual arrangement used is described in detail in section V of this chapter, and only the results will be considered here. It was found that a counting rate of approximately 300 a minute above a background of 60 a minute could be obtained on a line which took 2 minutes to photograph on a G5 emulsion. For practical purposes with γ-ray sources, exposures of at least 20 hours are required, and so the counting rate would only be 1 in 2 minutes. The sensitivity of the G.M.4 type counter, estimated from its dimensions and filling, is approximately ½% in this region, and so, even with a 100% efficient γ-ray counter, the counting rate would only be 100 a minute. The development of scintillation counters has made it possible to obtain γ-ray counters with an efficiency of approximately 50% in this region, but it was decided that for the present experiments, in which considerable ranges of wavelengths were examined, photographic plates would be more suitable.

IV. PROPERTIES OF G5 200 α/β-SENSITIVE EMULSIONS

The β-sensitive emulsions are generally used for the study of nuclear events within the emulsions. They have been developed to record the individual
Fig. (16). 50 KeV electron track.
tracks of electrons, which, on passing through the emulsion, give rise to tracks of a number of grains depending on their energy. A typical example is shown in fig. (16), corresponding to an electron of 50 KeV. Since these emulsions are sensitive enough to produce several grains for a low energy electron, it was thought that they would also be suitable for measurements of γ-rays by means of the photo-electrons produced in the emulsion. In the present work they have been used to measure the total blackening produced by a large number of γ-rays incident on the emulsion, and have been found to be several times faster than Kodirex X-ray film.

Due to the thickness of these emulsions, a special temperature cycle development technique has been devised by Dilworth and Occhialini(37) to ensure that uniform development is obtained throughout the thickness of the emulsions. The development times used in this technique are those most suited for the production of electron tracks in the emulsion. Since the present work is concerned with total blackening, tests were carried out to determine the optimum development times to produce maximum contrast between line and background. A series of identical exposures was taken, using a silver radiator, and developed for 20, 30, 35, 40, 45 and 55 minutes. It was found that the optimum development time was 35 minutes, any increase in this
time leading to a brownish stain in the emulsion.

The three standard developers used in this type of work are Amidol, Ilford ID 19 and Kodak "Elon" developer. It was found that, during the development process, a black surface stain was formed on the plate. "Elon" left the plates most nearly free from this surface stain, and it has been used throughout this work.

**Removal of Surface Stain**

There are three methods of removing the surface stain.

a) Rub the surface lightly when in the stop-bath.

b) Rub the surface with alcohol when the processing and drying have been completed.

c) After the plate has been thoroughly dried, bleach the surface by immersing for a few seconds in slightly acid $\text{KMnO}_4$, place in a 5% sodium bisulphite stop-bath, and fix, wash and dry as before.

Normally the first method was used, as it tended to leave fewer marks on the plate than the second. The third method, while very effective in removing the surface stain, is difficult to control, and in general bleaches to a certain extent at least the top 20 μ of the plate. This method was never used when any intensity measurements were being made, as it would tend to falsify the results, as different radiations
penetrate to different depths in the emulsion.

**Intensifying Solutions**

Several different types of intensifier were used to see whether they would bring up lines that were not visible to the eye. Very little success was obtained, though, for lines which were already visible, the contrast between line and background was enhanced. The most successful solution for this purpose was Uranium Intensifier.

**Grain Counts**

The number of grains produced by electrons of various energies in a $\beta$-sensitive/emulsion has been determined experimentally by Zajac and Ross.\(^{(38)}\) They have also shown that, in the case of a G5 emulsion, though the actual number of grains produced by an electron of given energy is different, the ratio of the number of grains at different energies in the two emulsions remains constant for electrons below 100 KeV.

Experiments were carried out to attempt to identify a weakly exposed line by determining the total number of tracks per field of view at points along the emulsion. It was found possible to identify a visible line by this method, but not a line that could not be seen by the eye.

Since the length of the electron track depends on the energy of the photo-electron caused by a $\gamma$-ray, it
EXPOSURE TIME

Fig. (17). Graph of blackening against exposure time. (The scale of blackening is such that unity corresponds to unit exposure time.)
is possible to determine approximately the energy of the γ-ray by measuring this length. This method would prove useful in the case of confusion between first and second order spectra.

**Linearity Test of Blackening**

An experiment was carried out to investigate the logarithmic linearity of the graph of exposure against density of blackening, using the Kα radiation from a silver scatterer. Owing to fluctuations in the production of X-rays by the tube, a Geiger counter was placed in the direct beam, to monitor the exposures. A series of exposures was taken on one plate, successive exposures corresponding to an increase in the total count in the monitor by a factor of two.

The resultant lines were microphotometered and a graph was plotted of the blackening produced against the exposure time. The blackening has been measured by the total area under the line in the microphotometer trace, and not by its height. The exposure time scale is such that the time required to produce a just visible line is taken as unity. The graph, shown in fig. (17), is linear for normal exposures, but at high exposures there is a saturation effect corresponding to the case when two or more grains lie directly beneath each other, or when an electron passes through an already developable grain. This curve has been plotted using an energy of 22.5 KeV, and so is only
strictly true in that region, but can, in a first approximation, be used in other regions. However, if very accurate intensity measurements are required, a separate curve should be plotted in the appropriate energy region.

**Variation of Sensitivity with Energy**

The variation of the sensitivity of a G5 emulsion with energy has been calculated on the assumption that the blackening produced is proportional to the total number of grains developed.

Consider a γ-ray of energy E incident normally on the surface of the emulsion. This photon can be absorbed either by photo-electric absorption in the K or the L shell of one of the constituent atoms, or by the Compton effect. In the analysis of the present problem, it was found that the effect due to Compton scattering was negligible compared with that due to photo-electric absorption.

Consider the case of a photon absorbed in the K shell of a silver atom.

Let \( \mu_n \) be the corresponding linear absorption coefficient.

Let \( \Sigma \mu_n \) be the total linear absorption coefficient of the emulsion.

The probability that an incident photon will be absorbed in this shell during its passage through an emulsion of thickness x is given by
Fig. (18). Grain-energy relationship of Zajac and Ross used in calculating sensitivity graph.
Absorption in this shell will produce a definite number of grains due to:

a) The photo-electron ejected by the photon, and

b) The de-excitation of the resultant ionised atom leading to the production of further grains due to the Auger electrons or the absorption of the characteristic X-radiation. The latter is assumed to be completely absorbed in the emulsion.

In calculating the number of grains produced due to these effects, the grain-energy relationship determined by Zajac and Ross has been used. This relationship is shown in fig. (18), plotted on a logarithmic scale.

Let \( g_n \) be the number of grains produced due to these effects by absorption in the shell of absorption coefficient \( \mu_n \).

The average number of grains produced per incident photon will be

\[
\rho_n = \frac{\mu_n g_n}{\varepsilon \mu_n} \left( 1 - e^{-\varepsilon \mu_n x} \right)
\]

If the effects due to all possible modes of photo-electric absorption are considered, the total number of grains produced per incident photon will be

\[
g = \sum \rho_n g_n = \frac{\varepsilon}{\varepsilon \mu_n} \left( 1 - e^{-\varepsilon \mu_n x} \right)
\]

The value of \( g \) has been calculated for values of the energy of the incident photon between 10 KeV and
Fig. (20). Spectrum of continuous radiation from a tungsten anti-cathode X-ray tube, showing sudden change in sensitivity of emulsion at 25.5 KeV.
Fig. (19). Variation of sensitivity of 200 μ G5 emulsion with energy of photon incident normal to its surface.
100 KeV, in the case of a 200 μ thick G5 emulsion. The graph obtained is shown in fig. (19). This graph shows a sudden change at 25.5 KeV, corresponding to the silver absorption edge. This change in sensitivity can be seen in fig. (20), which shows the continuous spectrum from a tungsten anti-cathode X-ray tube. The ratio of the intensities of the blackening on either side of this edge is 1.36:1, as compared with the calculated value of 1.40:1.

The experimental verification of this graph has not been carried out in great detail, but results given in Chapter 5 justify its use for present purposes.

**Line Width due to Action of Photographic Plate**

The photo-electric absorption in the emulsion for X-rays of energy greater than 25.5 KeV takes place principally in the K shell of a silver atom. This absorption ionises the atom, with the resultant emission of a silver K characteristic X-ray or an Auger electron. Silver Kα X-rays have an appreciable range in the emulsion, being reduced to $\frac{1}{e}$ of their intensity in a distance of 100 μ. Since these photons are emitted isotropically, this could give rise to an appreciable line width. An attempt was made to evaluate this effect, but the calculation is very complex, requiring a triple numerical integration, and the actual magnitude of the effect has not been
Fig. (21). Tapering of the jaws of the slit.
(Slit width 8 times scale of spectrometer)
determined. However, it was seen that this effect would contribute approximately 0.08 mm. to the line width, and hence place a limitation on the sharpness of lines obtainable using a 200 μm emulsion to record γ-ray spectra.

V. EXPERIMENTS WITH A GEIGER-MULLER COUNTER

The object of the experiments with a Geiger counter was twofold; firstly to investigate how useful the counter technique would be in curved crystal spectroscopy, and secondly to estimate the exposure times that would be required using radio-active sources.

The counter was placed directly behind a brass slit on the focusing circle. As, in this investigation, the radiation from a silver scatterer was used, the jaws of the slit were tapered as indicated in fig. (21) so as to prevent absorption in the edges of the slit. The exact tapering is important in the case of slit widths of 1/10 mm. or less, since penetration through the thin part of the tapered jaws limits the minimum width of slit that can be used.

In all work with counters, it is essential to shield the counter from scattered radiation. This was done by surrounding the counter with lead, and it was found necessary to connect the lead shielding directly to the same earth point as the counter body.
Fig. (22). Comparison of resolution obtained with counter and with photographic recording.

(a) Graph of counting rate against position using slit width of 1/10 mm.
(b) Microphotometer trace obtained with slit width of 1/10 mm.
Otherwise, there was a large increase in background counting rate, due to pick-up caused by corona discharges from the H.T. set supplying the X-ray tube. Consequently, before commencing an experiment, the background counting rate with the window of the counter shielded with lead was checked before and after the X-ray tube was switched on.

As the X-ray tube H.T. supply was derived from unstabilised mains, and the current through the tube varied considerably during experiments, the output of the tube was monitored, using a second counter. Variation of the H.T. voltage on the tube causes a change in the distribution of the continuous spectrum emitted by the tube, and so the counting rate observed is not exactly proportional to the output of a given energy. However, the variation of the tube voltage was small, 2%, compared with the variation in the current, 20%, and so the total counts recorded by the monitor gave a sufficiently accurate measure of the output for present purposes. All counting experiments were timed by means of the monitoring counter.

A series of experiments was performed using a slit length of 2 cm. and various slit widths. A graph of counting rate against position on the focusing circle is shown in fig. (22 a). This was obtained with a slit width of 1/10 mm., and each point corresponds to a direct beam count of 10,000 recorded in a time of
approximately 2 minutes. The resolution obtained in this experiment is as good as that obtained using photographic recording, with the resultant spectrum analysed by a microphotometer with a 1/10 mm. slit width. For comparison, the photographic spectrum obtained in this way is shown in fig. (22 b).

In the counting experiment described above, the counting rate on the peak of the line was 608 per 10,000 direct beam counts (or 2 minutes). To obtain a clearly visible photographic line, the exposure required corresponded to 20,000 direct counts (or 4 minutes). The photographic plate used records a spectrum simultaneously along its whole useful length, which is usually about 6 cm. To examine this length of plate with a counter of slit width 1/10 mm. would require 600 separate counts, corresponding to a total counting time of approximately 1200 minutes, or 20 hours, as compared with 4 minutes for the complete photographic recording.

For practical purposes with γ-ray sources, photographic exposures of at least 20 hours are required, and so the use of a G.M.4 counter is obviously impracticable. The development of scintillation counters has made it possible to obtain an efficiency of 50% in this region, compared with approximately ½% for the G.M.4 counter. Thus, with a γ-ray source producing a photographic line in 20 hours, a peak
counting rate of 50 a minute could be obtained, but, if weaker lines were being examined, considerable attention would have to be paid to the reduction of the background counting rate. In the present work it was decided to use photographic recording to explore the potentialities of the method, and no attempt has been made to develop a suitable scintillation counter for use with the spectrometer.

Estimation of Source Strength Required

In order to make an approximate estimate of the exposures that would be required to radio-active sources, experiments were carried out to determine the reflecting power of the crystal, using the radiation from a silver scatterer. The number of photons falling on the crystal in a given time was compared with the number reflected into the Kα lines in an equal interval of time, both measurements being made with the same G.M.4 counter. The size of the silver scatterer used in these experiments was 2 cm. by 1 cm., and it was placed 12" from the crystal, since these were the expected values of area and position of a radio-active source. In order to ensure that only silver radiation was being observed, the Ross method of balanced filters was employed, using rhodium and molybdenum absorbers.

It was found that 1 photon was reflected into the Kα lines for $10^4$ falling on the crystal. It required
600 counts to produce a visible line, so, estimating the efficiency of the counter as $\frac{1}{4}\%$, this would correspond to $12 \times 10^4$ photons falling on the photographic plate. The crystal had an effective area of 1" by 4", placed 12" from the source, so, making allowance for the solid angle effect, an exposure of 1 hour would be required to a source of 65 mC of this radiation. In practice, using a source of RaD 2.5 cm. by 1 cm., placed 14" from the crystal, the exposure required to produce a visible line corresponding to the 46.7 KeV γ-ray was found to be approximately 40 mC-hours of the radiation.

Throughout the discussion of the experiments with radio-active sources, exposures will be referred to in terms of mC-hours of the radiation. An exposure of 1 mC-hour of the radiation corresponds to an exposure of 1 hour to a source which emits $3.7 \times 10^7$ photons per second of the given radiation.
CHAPTER 5

INVESTIGATION OF RaD Χ-RAY SPECTRUM

I. INTRODUCTION

Until 1939 the decay of RaD → RaE was assumed to be by a single mode of β-disintegration leading to an excited state of the RaE nucleus. The γ-ray of 46.7 KeV emitted in the de-excitation of the RaE nucleus had been observed in emission and also by means of the photo-electrons produced by its internal conversion in the L, M and N shells of the RaE atom. The continuous β-ray spectrum had an end point of approximately 16 KeV(39) and there was no reported evidence of another continuous β-spectrum corresponding to the ground-ground state transition.

There are, however, considerable difficulties concerning the intensities of the RaD radiation. It is fairly well established that the unconverted γ-radiation has an intensity of $2.8 \pm 0.6 \%$. (40-42) The intensity of the internal conversion lines of this γ-ray has been measured by several workers and a large divergence of results obtained. The most recent work by Butt and Brodie(43) and Cranberg(44) indicates that the total intensity of these lines is of the order of $70 \pm 5\%$. This value leaves a discrepancy of about 25% of the disintegrations to be accounted for.

In addition to the 46.7 KeV γ-ray, 6 other low
<table>
<thead>
<tr>
<th>Quantum energy (KeV)</th>
<th>46.7</th>
<th>42.6</th>
<th>37±0.5</th>
<th>31.3±0.8</th>
<th>23.2±0.6</th>
<th>16.1±0.4</th>
<th>7.3±0.7</th>
</tr>
</thead>
<tbody>
<tr>
<td>Emission intensity (%)</td>
<td>2.8±0.6</td>
<td>0.2±0.1</td>
<td>0.2±0.1</td>
<td>0.4±0.2</td>
<td>~1</td>
<td>~0</td>
<td>~10</td>
</tr>
</tbody>
</table>

Table I. Reported γ-radiations from RaD.

Fig. (23). Possible decay scheme for RaD.

| Tsien et al. (26) | 100 | 7 | 7 | 14 | 33 | 0 | 300 |
| Amaldi et al. (47) | 100 | 20 |
| Salgueiro (48) | 100 | — | — | 5 |
| Frilley (49) | 100 | — | Observed, but intensities not given |
| Curran et al. (50) | 100 | — | — | 12 | 250 |
| Cranberg (44) | 100 | — | Possible 2.5 |
| Cork et al. (51) | 100 | No γ-ray of intensity > 1 |
| Pringle (52) | 100 | No γ-ray of intensity > 3 |

(— indicates γ-ray not observed but within range of experiment.)

Table II. Summary of measurements of intensities of RaD γ-rays.
energy γ-rays have been reported, principally by Tsien(26) and his colleagues in Paris. Table I gives their results as summarised by Feather(45) in a review article in 1949. Feather pointed out that, within the limits of experimental error

\[ 16.1 + 31.3 = 46.7 \]

\[ 16.1 + 7.3 + 23.2 = 46.7 \]

He suggested that five of these γ-rays might arise from the unique excitation of the 46.7 KeV level in the RaE nucleus and the subsequent decay by cascade processes, such as indicated in fig. (23).

The spin and parity assigned to the 46.7 KeV level in this scheme is based on the β-disintegration from RaD to this level being allowed. The spin and parity assigned to the ground states are based on the ground to ground state transition being second forbidden as suggested by Feather and Richardson.(46)

Some of these γ-rays have been observed by other workers in this field and a summary of results obtained is shown in Table II. The intensity of the radiation, if observed, is given normalised to an intensity of 100 for the 46.7 KeV γ-ray. The exact value of the energy measured in various cases differs, and the values used are those given by Feather in his summary.

It can be seen from this table that there is considerable doubt as to the existence of these γ-rays,
or, if they do exist, as regards their relative intensities. The object of the present investigation was to attempt to prove the existence of these γ-rays, and measure their energies and relative intensities, or to place a limit on their possible intensities in emission. In the course of this work the wavelength of the 46.7 keV γ-ray was measured correct to 1 part in 2,000, and some observations were made on the L X-ray spectrum emitted by the RaE (or Bi) atom.

II. THE SOURCE AND COLLIMATING SYSTEM

The source of RaD used in these experiments was obtained from the Radio-Chemical Centre at Amersham. It consisted of approximately 100 mC of RaD deposited anodically on a platinum foil 2.5 cm. by 1 cm. by 0.010" thick. The weight of inactive deposit was 2.1 mgm. Since the source had a very high specific activity, it was sealed off, using a sheet of aluminium 0.1 mm. thick, so as to prevent the loss of any of the radio-active deposit. This thickness of aluminium was sufficient to prevent the escape of the α-particles from Po produced as a result of the decay of RaE, but had a negligible effect on the γ-rays emitted by RaD.

In addition to the 46.7 KeV γ-ray emitted by the source, there is the high energy β-ray spectrum from RaE, which is in equilibrium with the RaD. As β-rays
Fig. (24). Lead collimator used with RaD source.
falling on a crystal tend to destroy its crystalline properties, it was necessary to ensure that these were either deflected or absorbed before reaching the mica. No suitable magnetic field was available for deflecting $\beta$-rays of energies up to 1.2 MeV, and so it was decided to absorb them by using filter paper of thickness 0.45 $\text{gm/cm}^2$. This was sufficient to remove the highest energy $\beta$-rays from the source, and, in subsequent intensity measurements, corrections have been made for the absorption of the $\gamma$-rays in this paper.

In order to prevent undue fogging of the photographic plate, caused by $\gamma$-rays direct from the source, a collimating system was constructed as shown in fig. (24). The lead baffles were 1/8 in. thick, and had slots in them 1 in. by 1/4 in., corresponding to the size of the crystal aperture. These baffles could be replaced by others containing slots of smaller dimensions if it was not necessary to use the whole of the source. The paper to absorb the $\beta$-rays was placed in front of the source, which was sunk into a lead container so that the $\gamma$-rays escaped only in a forward direction. The collimator as a whole was lined with thin aluminium, covered with paper to absorb as much as possible of the lead L X-rays caused by $\gamma$-rays striking the lead walls of the collimator.

As the source was too strong for a normal
Fig. (25). Pin-hole photograph of RaD source.
radiograph, a pin-hole photograph of it was taken. This showed (fig, (25) ), that the source was not uniformly deposited over the whole area of the foil. Consequently, in intensity measurements, it is necessary to use the whole area of the source, and this limits the range of wavelengths that can be examined at one setting. This range will be calculated in the next section.

III. INTENSITY MEASUREMENTS WITH A CURVED CRYSTAL SPECTROMETER

When a curved crystal spectrometer is being used to examine simultaneously a large range of wavelengths corresponding to a large variation in Bragg angles, it is not possible to use the same portion of the source and the crystal for all wavelengths. In the present case, where the source is not deposited uniformly it is better if possible to use the whole source and different parts of the crystal. The reflecting power of different parts of the crystal was examined during the adjustment of the spectrometer and was found to be constant within experimental error.

To calculate the angular range over which the whole source can be used in a given case, consider the geometrical arrangement shown in fig. (26).

$S_1S_2$ is the source whose centre point is $S_0$

$C_1C_2$ is the crystal whose centre point is $C_0$
Fig. (26). Geometry for calculation of angular range over which the whole source can be used.
0 is the centre of the focusing circle.

Let \( C_0 \hat{OC}_0 = \delta \)

The atomic planes are inclined at an angle \( \alpha \) to the normals to the crystal slab. In particular the atomic plane at \( C_0 \) will make an angle \( \alpha \) to the direction \( \hat{OC}_0 \).

\( V_1 \) is the virtual source, corresponding to the minimum value of the Bragg angle using the whole source. Only those rays emitted from the source in the direction of \( V_1 \) will be reflected at this Bragg angle.

Consider 0 as the centre of co-ordinates.

Let \( \theta_m = \) angle the ray \( S_0 C_0 \) makes with the crystal planes.

\[ \text{half} \]

Let \( b = \) breadth of source.

Let \( d = \) distance of source from crystal = \( C_0 S_0 \)

Let \( r = \) radius of the focusing circle.

\( S_0 \) is point \([-d \sin(\alpha + \theta_m), r + d \cos(\alpha + \theta_m)]\)

\( S_1 \) is point \([-d \sin(\alpha + \theta_m) - b \cos(\alpha + \theta_m), r + d \cos(\alpha + \theta_m) - b \sin(\alpha + \theta_m)]\)

\( S_2 \) is point \([-d \sin(\alpha + \theta_m) + b \cos(\alpha + \theta_m), r + d \cos(\alpha + \theta_m) + b \sin(\alpha + \theta_m)]\)

\( C_1 \) is point \((-r \sin \delta, r \cos \delta)\)

\( C_2 \) is point \((r \sin \delta, r \cos \delta)\)

Equation of \( S_1 C_1 V_1 \) is

\[ \frac{y - r \cos \delta}{x + r \sin \delta} = m_1 \]

where \( m_1 = \frac{r - r \cos \delta + d \cos (\alpha + \theta_m) - b \sin (\alpha + \theta_m)}{r \sin \delta - d \sin (\alpha + \theta_m) - b \cos (\alpha + \theta_m)} \)

which can be evaluated in a given case.

The second point of intersection of this line with
the circle will be \( V_1 \) of co-ordinates

\[
\begin{align*}
x_1 &= \frac{r}{1 + m^2} \left[ \left( l - m^2 \right) \sin \delta - 2 m \cos \delta \right] \\
y_1 &= \frac{r}{1 + m^2} \left[ \left( l - m^2 \right) \cos \delta + 2 m \sin \delta \right]
\end{align*}
\]

\[
\sin \phi_1 = \frac{x_1}{r}
\]

Hence the value of \( \phi_1 \) can be calculated.

Similarly the value of \( \phi_2 \), corresponding to position of \( V_2 \).

The maximum aperture angle \( \theta_2 - \theta_1 \) = angular separation of \( V_1 \) and \( V_2 \) with respect to crystal \( \frac{1}{2} (\phi_2 - \phi_1) \).

For the present spectrometer and source this has been evaluated and found to be \( 4^\circ 9' \), when \( m = 5\frac{1}{2}^\circ \).

The position at which the straight through beam cuts the focusing circle, and which therefore places a limit on the value of the minimum Bragg angle that can be used for a given value of \( \Theta_m \), can also be calculated if required by finding the intersection of the ray \( S_2 O_1 \) with the focusing circle.

**Variation of Sensitivity of Apparatus with Energy**

All intensity measurements must be corrected for the variations in the sensitivity of the apparatus with energy. This is due to the variation in the sensitivity of the photographic plate and to the absorption of the \( \gamma \)-rays during their passage from source to plate.

The variation of sensitivity of the G5 emulsion was calculated in Chapter 4, and the graph deduced
Fig. (27). Variation of sensitivity of apparatus with energy.

Table III. Relative intensities of uranium L X-rays compared with values obtained by Allison.

<table>
<thead>
<tr>
<th>L X-ray</th>
<th>$\alpha_1$</th>
<th>$\alpha_2$</th>
<th>$\beta_1$</th>
<th>$\beta_2$</th>
<th>$\beta_3$</th>
<th>$\beta_4$</th>
<th>$\gamma_1$</th>
<th>$\gamma_2$</th>
<th>$\gamma_3$</th>
<th>$\gamma_4$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ewan</td>
<td>103</td>
<td>12</td>
<td>49.4</td>
<td>27</td>
<td>3.9</td>
<td>3.7</td>
<td>6.5</td>
<td>11</td>
<td>1.8</td>
<td>1.5</td>
</tr>
<tr>
<td>Allison</td>
<td>100</td>
<td>11</td>
<td>49.4</td>
<td>28</td>
<td>4.2</td>
<td>4.1</td>
<td>6.4</td>
<td>12</td>
<td>1.5</td>
<td>1.4</td>
</tr>
</tbody>
</table>
there has been used. The absorption corrections are due to self-absorption in the source, absorption in the aluminium covering the source, in the paper present to absorb the $\beta$-rays, in the mica, and in the air path of 32 in. from source to photographic plate. The values of the absorption coefficients have been calculated from tables given by Compton and Allison\(^{(53)}\) with the exception of that of mica, for which the value of $\mu = 36.56 \lambda^{2.56}$, determined experimentally by Williams,\(^{(54)}\) has been used.

The graph of variation of the sensitivity of the apparatus with energy is shown in fig. (27). It can be seen that the sensitivity falls off rapidly below 25 KeV, chiefly due to the increasingly large effect of the absorption in the mica.

The validity of this graph in the low energy region has been tested using the fluorescent radiation excited by bombarding a thick film of Uranium Oxide with $\alpha$-rays. In this case, the intensities observed were corrected for self-absorption in the Uranium Oxide film, absorption in the mica and the air path, and for the variation in sensitivity of the photographic plate. The relative intensities of the lines, as measured in the spectrometer and corrected in this way, are shown in Table III, compared with values determined by Allison.\(^{(55)}\) The values given by Allison were normalised to an intensity of 100 for the $\alpha_1$ X-ray.
Since in the present investigation the intensity of the $\beta$, X-ray was most accurately determined by the microphotometer, the results have been normalised so that its intensity agrees with that given by Allison. The agreement obtained between the two sets of results is satisfactory and justifies the use of this graph for present purposes in this energy region.

IV. EXPOSURES FOR 46.7 KeV AND OTHER $\gamma$-RAYS

In the following experiments, unless otherwise stated, the spectrometer was set for a central ray striking the (100) planes at an angle of $5^{1/2}_0$ and the angular range covered using the whole source was $4^0 9'$.

The first series of exposures was made to identify the 46.7 KeV $\gamma$-ray. It was found to be clearly visible in 18 hours and just visible in 14 hours. Since the source strength was 100 mC and this $\gamma$-ray is emitted 2.8 times per 100 disintegrations, this corresponds to an exposure time of approximately 40 mC-hours of the radiation, which is fairly close to the estimate of 65 mC-hours found in Chapter 4 using silver K radiation.

The next series of experiments was designed to identify the 23.2 KeV $\gamma$-ray reported by Tsien to be $1/3$ the intensity of the 46.7 KeV ray. Exposures of 3 days, 7 days, and 14 days were given, but neither the 23.2 KeV nor any other $\gamma$-ray, except the 46.7 KeV
\( \gamma \)-ray, was visible.

Finally, three plates were exposed to attempt to observe the other lines, or to put an upper limit on the possible intensities of these lines. Each of the three plates was exposed for 700 hours. Before performing these experiments, the effects of long exposures were considered, and these will be discussed in the next section.

After the first exposure the plate was moved slightly and a calibration line, corresponding to 1/50 of the exposure, was placed on the plate to the high energy side of the 46.7 KeV line by exposing for a further 14 hours. Similar calibration lines of 1/30 and 1/20 of the main exposure were also put on the plate. There was no visible evidence of any line in the region from 46.7 KeV to 20 KeV although all three calibration lines were visible.

In the second exposure the position of the camera was altered so that, in addition to the 46.7 KeV line, the L \( \gamma \) X-rays emitted by the RaE atom should be visible, in order to show that the spectrometer was effective over the whole range from 46.7 KeV to 16 KeV. In this case, the calibration lines were put on as before, but only on the top half of the plate, the lower half being masked off with lead. The L \( \gamma \) X-rays were clearly visible, but again there was no evidence of any lines in the region 46.7 KeV to
16 KeV.

Microphotometer analysis of these plates showed that in both cases the 14 hour calibration line was of rather higher intensity than expected, but the variation could be accounted for by the possible errors in the microphotometer traces. As, in both cases, the calibration line was 3 mm. from the main line, it was decided to carry out a third exposure to make sure that there was no $\gamma$-ray in that region.

In this case, the spectrometer was set so as to cover the range up to 62 KeV. Again an exposure of 700 hours was given, but no visible line produced, other than the 46.7 KeV line.

These three plates were microphotometered using a technique described in Section VI, and the quantitative limit put on the possible existence of the various $\gamma$-rays will be discussed later in the light of the microphotometer analysis.

V. EFFECTS OF LONG EXPOSURES

Temperature Changes

During the exposures of 700 hours (i.e. 29 days approximately) there could be fluctuations of temperature in the room of up to $20^\circ$C, corresponding to the difference of temperature between the day, when the heating in the building was full on, and the night, when there was no heating at all. The effect of these
temperature changes on the width of the spectral lines observed with the spectrometer was calculated, since increase in line width would tend to diminish the contrast between line and background.

The effects of variation of the radius of curvature of the steel blocks, change in the position of the camera support, and change in the grating spacing of the mica were all found to be negligible for temperature changes of 20°C. There is also the effect of the variation of the pressure of the bolts clamping the blocks together, which is more difficult to determine. However, it was noticed when the blocks were being clamped together that this adjustment was not extremely critical, so it was expected that this effect would also be negligible. Observations taken during a series of exposures gave very little indication of an increase in line width due to temperature effects.

**Increase of Background**

During the course of a month's exposure it was expected that the background fogging of the photographic plate would increase considerably. However, the collimating system already described proved very efficient in reducing the background radiation and the increase was not very marked. Moreover, the technique of superposing the calibration lines on top of the background showed that the masking effect of the
background was very small indeed, since a visible 46.7 KeV line was produced in 14 hours on a plate exposed for 700 hours.

**Latent Image Fading**

In certain types of photographic plate, if the plate is left for a considerable time before developing, the latent image fades a little, and consequently the image finally produced is not as intense as it should be. If this were to happen in the case of the G5 emulsion used, it would tend to prevent the appearance of weak lines. Before the third of the 700 hour exposures, a plate was exposed to the 46.7 KeV line for exactly 2 days and then laid aside for the duration of the exposure. During the exposure the spectrometer remained in the same setting, and afterwards another plate from the same batch was exposed for exactly 2 days and then the two 2-day exposures were developed together. The intensities of the lines were then examined in the microphotometer and were found to be 128 and 130 in arbitrary units, indicating that, within the experimental error, there was no evidence of fading.

**Ghost Images**

Although no lines other than the 46.7 keV were obtained on these plates, it was felt advisable to investigate the possibility of ghost images being produced in the vicinity of the main line. With this
in view, the silver $K\alpha$ doublet was over-exposed by a factor of 400 and no visible evidence was found for the existence of ghost lines. Therefore, in the case of the 46.7 KeV line, which is at the most over-exposed by a factor of 50, there is very little possibility of ghost images.

VI. MICROPHOTOMETER ANALYSIS OF PLATES

The plates were examined on one or other of two microphotometers at the Royal Observatory, Blackford Hill, Edinburgh. One of these instruments was a Kipp microphotometer, designed with an optical system containing a microscope objective of 4 mm focal length to project the slit on to the photographic plate, and another to receive the light. The plates used in our experiments were all 200$\mu$m thick, and these objectives had insufficient depth of focus, so they were replaced by 16 mm objectives and the calibration of the height and width of the slit were adjusted accordingly.

The second instrument was a Hilger and Watts non-recording microphotometer. It used objectives of 1" focal length and so there was no difficulty as regards depth of focus. It was found to be well suited to the analysis of these plates and, apart from the first 700 hour exposure plate and one L X-ray plate, all plates have been analysed using this instrument or a similar one which has since been purchased for this
Physics Department.

The G5 emulsions were found to have poor qualities as regards microphotometer analysis, as the random fluctuations in the background are considerable, and so tend to mask weak lines. However, the spectral lines extend across 2 cm. of the plate and it is possible to take a series of independent runs and average them. This considerably reduces the random fluctuations and consequently increases the probability of detecting a weak line. Great care has to be taken in this case to ensure that the plate is truly lined up, so that points in each run exactly correspond.

The first 700 hour plate was analysed using the Kipp microphotometer as a non-recording instrument. The slit width used was 200 $\mu$ and readings were taken every 0.2 mm. The trace obtained is shown in Trace I(a) (see end). There is very little evidence of any $\gamma$-rays of energy lower than 46.7 KeV and the calibration lines corresponding to 1/50, 1/30, and 1/20 the intensity of the main line show quite distinctly. The rise to the high energy side of the main line is due to the effects of the extreme edge of the straight through beam, and the drop at about 19 KeV is due to a noticeably transparent spot on the plate. The quality of this trace was not very good, showing several features which might represent a weak line, and so the second 700 hour exposure was given to see if these features
were also present in its trace.

The second and third plates were analysed on the Hilger and Watts microphotometer, using a 100μ slit width and taking readings every 0.1 mm. These traces are also shown in Trace I(b) and (c). Trace I(b) is of much better quality than Trace I(a), and there is no indication of any γ-ray lines below 46.7 KeV, though the calibration lines are visible, and also the first of the L X-rays. In Trace I(c), corresponding to the plate which covers the region above as well as below the main line, again there is no indication of any other line.

These traces have been used to help to place a quantitative limit on the intensity of possible low energy γ-rays, and this will be discussed in the next section.

VII. QUANTITATIVE LIMIT OF EXISTENCE OF γ-RAYS

In the experiments described, the whole source was used in the energy region from 40 KeV to 18 KeV, and only 0.92 of the area of the whole source at 46.7 KeV. This means that the intensity obtained at 46.7 KeV is slightly less than would have been the case if the whole source had been used. In this analysis, no allowance has been made for this effect, due to the non-uniform distribution of the source. Consequently, the limit which will be placed on the possible
<table>
<thead>
<tr>
<th>$\gamma$-ray (KeV)</th>
<th>46.7</th>
<th>42.6</th>
<th>37</th>
<th>31.3</th>
<th>23.2</th>
<th>16.1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Intensity</td>
<td>100</td>
<td>&lt;1.5</td>
<td>&lt;1.5</td>
<td>&lt;1.5</td>
<td>&lt;2.5</td>
<td>&lt;6</td>
</tr>
</tbody>
</table>

Table IV. Limit on existence of low energy $\gamma$-rays reported by Tsien.
intensity of lines below 46.7 KeV is rather higher than
would have been possible if the whole source could have
been used over the whole range.

It is estimated from examination of the micro-
photometer traces that a line of 2/3 the intensity of
the 14 hour exposure would have been visible above the
random fluctuations in the background. This would
correspond to an intensity of 1/75 of the main line.
Using the sensitivity graph already calculated for
this experiment, the quantitative limits that can be
set on the existence of the γ-rays that were reported
by Tsien are shown in Table IV.

The discussion of these results will be given in
Section X of this chapter.

VIII. INVESTIGATION OF THE L X-RAY SPECTRUM EMITTED
BY RaE

Examination of the L X-ray spectrum can give
useful information regarding the relative ionisations
of the L_1, L_II and L_III levels after the internal
conversion of the 46.7 KeV γ-ray. Cranberg,(44) by
measuring the relative intensities of the L conversion
lines, found that the ratio of initial ionisation was
100:9:2. Coster and Kronig(56) pointed out that, in
the ionised atom, it was possible to have Auger
transitions in which a vacancy in the L_1 level was
filled to produce a vacancy in the L_III level and
another in either the $M_V$ or $M_V$ level. They estimated that about 80% of the $L_I$ ionisation was transferred to the $L_{III}$ level by this means. Kinsey$^{(57)}$ placed this on a more quantitative basis and deduced values of the fluorescent yields of each of the $L$ levels for several of the heavy elements, on the assumption that only transfer of ionisation from $L_I$ to $L_{III}$ was possible. It is therefore of theoretical interest to determine the relative fluorescent yields of the levels experimentally and the present investigation was carried out with this in view.

In the present experiments, the central setting of the spectrometer was 8.5° so that the complete $L$ X-ray spectrum could be examined in one setting, using the whole source. Three exposures, each of 350 hours, were given to the $L$ X-rays. In the first two the $\beta_1$ and $\beta_2$ lines were not sufficiently well resolved to allow of measurements of their relative intensities. In the third the effective width of the source was cut down to $\frac{1}{4}$" but again the resolution was not good enough. The three plates were examined on the microphotometer and the resulting traces averaged. Since the first two plates were exposed to the whole source and the third to only half of it, in the averaging process the first two were weighted by a factor of two with respect to the third. The portions of the spectrum in the $L\alpha$, $\beta$, and $\gamma$ regions are shown in Trace II (see
<table>
<thead>
<tr>
<th>L X-ray</th>
<th>Transition</th>
<th>RaE</th>
<th>Bi</th>
<th>Bi\textsuperscript{(a)}</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\alpha_1$</td>
<td>L\textsubscript{III} - N\textsubscript{V}</td>
<td>145</td>
<td>135</td>
<td>130</td>
</tr>
<tr>
<td>$\alpha_2$</td>
<td>L\textsubscript{III} - M\textsubscript{IV}</td>
<td>18</td>
<td>17</td>
<td>15</td>
</tr>
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(a) Values deduced by interpolation from tables given by Compton and Allison.

Table V. Observed intensities of L X-rays from RaE compared with those of bismuth.

L Shell 54% Absorption
L Shell 22.7% photons, K Shell 26.8% photons
3.65% Y

Total 77% ± 5%
In the smoothed microphotometer trace there is evidence of the $\text{L}_2^\alpha$ line which could not be seen visually. The $\text{L}_1^\alpha$ line can be seen to have doublet structure. This is due to the presence of a satellite line $\text{L}_2^\alpha$ due to the double ionisation of the atom following the emission of an Auger electron in a transition of the type suggested by Coster and Kronig.

In the $\text{L}_\beta$ region of the spectrum the existence of the $\beta_1/\beta_2$ doublet is only shown by the shape of the spectral line and its increase in width. The $\text{L}_\beta_2$ line, which was not visible to the eye, can be seen in the trace. In the $\text{L}_\gamma$ region the four principal lines can be clearly seen.

The intensities of these lines have been measured and corrected for the variation of sensitivity of the apparatus with energy. The corrected values are given in Table V, normalised to a total intensity of 100 for the $\beta_1/\beta_2$ doublet, since this corresponded to the most accurate intensity measurement. For comparison, the $\text{L}$ X-ray spectrum of Bi, measured by the same spectrometer, with the intensities corrected by a similar sensitivity graph, is also shown. No direct results are available on the relative intensities of the Bi $\text{L}$ X-ray spectrum, but in the last column are given values deduced by interpolation from those given for some of the heavy elements by Compton and Allison. (53a)
It can be seen that the relative intensities of radiations from the same initial levels are the same in the X-ray and RaE cases. Using this fact the unresolved $\beta_1$ and $\beta_2$ lines have been allocated intensities of 67 and 33 respectively.

From the table the ratio of the intensities of $L_I$, $L_{II}$ and $L_{III}$ fluorescence has been deduced and found to be 65:41:100. The values for the intensities of fluorescence found theoretically by Kinsey for Bi were 0.090, 0.041, and 0.192 for the $L_I$, $L_{II}$, and $L_{III}$ levels respectively. The present determination shows that the yields from the $L_I$ and $L_{II}$ levels are not in the ratio of 2:1 predicted by Kinsey, but that there is a greater yield from the $L_{II}$ level.

Kinsey, in the derivation of the intensities quoted, only made allowance for changes of ionisation from the $L_I$ to $L_{III}$ levels as suggested by Coster and Kronig. However, it was pointed out by Cooper, who listed all the possible types of Coster-Kronig transitions, that for Bi it is energetically possible to have a transition of the type $L_I\rightarrow L_{II}$, ejecting an electron from the $N_{IV}$ level. The results of the present experiment indicate that this process, in the case of the RaE atom, is in competition with the $L_I\rightarrow L_{III}$ transition.*

Fig. (28). One of the plates used in measuring the wavelength of the 46.7 KeV $\gamma$-ray.
IX. MEASUREMENT OF WAVELENGTH OF 46.7 KeV $\gamma$-RAY

The spectrometer has been used to measure the wavelength of the 46.7 KeV $\gamma$-ray. This was done using the (201) planes to reflect the $\gamma$-ray in the second order and the position on the plate was calibrated using silver K X-rays in the first order and tungsten K X-rays in the second order. Two independent plates were taken, one of which is reproduced in fig. (28).

The relative positions of the peaks of the lines were measured using the Hilger and Watts microphotometer at four different vertical positions on each plate. The measurements on each plate were treated independently by the method of least squares and the values $\lambda_1$ and $\lambda_2$ obtained were

$$\lambda_1 = 265.98 \pm 0.1 \text{ X.U.}$$

and

$$\lambda_2 = 265.92 \pm 0.15 \text{ X.U.}$$

These values are in good agreement with previous determinations of 266 X.U.\(^{(60,61)}\)

If these values of $\lambda$ are converted into terms of energy using the most recent values of the fundamental constants given by Dumond and Cohen\(^{(62)}\) the corresponding energies are

$$E_1 = 46.513 \pm 0.02 \text{ KeV}$$

$$E_2 = 46.523 \pm 0.03 \text{ KeV}$$

the mean value being 46.52 $\pm$ 0.02 KeV.

This differs considerably from the value of 46.7 KeV generally quoted for this $\gamma$-ray. However,
this discrepancy can be almost entirely accounted for by the changes in the values of the fundamental constants used. The previous determinations of the energy of this $\gamma$-ray have been made by $\beta$-ray spectrometer methods. The most reliable measurement is probably that of Curtiss (65) who obtained a value for $H_\rho$ of 594.3 gauss-cm. for conversion of the $\gamma$-ray in the $L_I$ level. If this value is converted into energy units using the formula

$$E = m_0 c^2 \left( \sqrt{\frac{(H_\rho)^2}{(m_0 c^2)^2} + 1} - 1 \right)$$

the value obtained is $E = 30.17$ KeV using Dumond's values of the atomic constants.

The ionisation potential of the $L_I$ level is 16.38 KeV, according to the values given by Slack (64) in 1952. The corresponding value of the energy of the $\gamma$-ray determined by Curtiss will therefore be 46.55 KeV, which agrees, within the experimental errors of the two experiments, with the present determination of $46.52 \pm 0.02$ KeV.

X. CONCLUSIONS

The experiments described have been successful in identifying the 46.7 KeV $\gamma$-ray in an exposure corresponding to 40 mC-hours of the radiation. Due to the high internal conversion of this $\gamma$-ray, it required an exposure of 14 hours, using a source of RaD of total activity 100 mC. This order of exposure is rather
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(a) Coincides with Auger line.
(b) Observed with one source only.

Table VI. Summary of experiments capable of identifying low energy γ-rays from RaD. The intensities are normalised to 100 for the 46.7 KeV γ-ray which is emitted $2.8 \pm 0.6$ times per 100 disintegrations.
excessive if weak low energy \( \gamma \)-rays are to be identified and so it was decided to construct the small spectrometer described in the next chapter, to attempt to reduce the exposure times required.

In addition to identifying this \( \gamma \)-ray and measuring accurately the energy as \( 46.52 \pm 0.02 \) KeV, a careful search was carried out in an attempt to identify the low energy \( \gamma \)-rays reported by Tsien. No evidence has been discovered for their existence in emission, and a quantitative limit has been placed on their possible intensities, based on the present experiments.

The previous results concerned with the low energy \( \gamma \)-rays from RaD are summarised in Table VI, giving, in addition to the intensities observed, an indication of the method used. It can be seen that, apart from the results of Cranberg on the 31.3 KeV line, no internal conversion electrons have been observed corresponding to any of the low energy \( \gamma \)-rays, though Cork and his colleagues carried out a careful investigation in order to identify these. Cranberg is very doubtful about the existence of the line he observed, pointing out that it was only observed using one source, and its intensity was of the same order as the errors in intensity measurements.

The evidence based on these experiments is that these \( \gamma \)-rays are very weak in internal conversion, and
the present experiments indicate that they are also very weak in emission. It seems very doubtful, therefore, that the 25% discrepancy between the total number of conversion electrons plus emitted photons and the total number of disintegrations can be explained by a cascade process based on the low energy $\gamma$-rays reported by Tsien.
CHAPTER 6

20 cm. CURVED CRYSTAL SPECTROMETER

I. INTRODUCTION

As the exposure times that were required with the large spectrometer were rather long, it was decided to construct a spectrometer of smaller radius of curvature and so reduce the exposures required. A smaller spectrometer will not give as high resolution, but it would be very useful for preliminary identification of lines. If accurate measurements were required, these could then be examined on the large spectrometer.

The radius chosen for the small spectrometer was 20 cm. By reducing the radius, in addition to decreasing the dispersion of the focusing circle, the magnitude of the geometrical aberrations is increased. The largest of these, that due to the width of the crystal aperture, is given by \( \frac{\sigma^2}{8R} \tan u \), where \( \sigma \) is the width of the crystal aperture, \( R \) the radius of curvature of the crystal and \( u \) depends on the angle of setting. In the present instrument it was decided to reduce the width of the crystal aperture from 1 in. to 3/4 in., which compensates to a large extent for the effect of the reduction of the radius of curvature. The value of the aberrations in this case will give a contribution to the line width of .0026 cm. for \( u = \)}
Fig. (29). Constructional details of 20 cm. spectrometer.
\[ \phi - \alpha = 8^\circ. \]

Since the purpose of this instrument was the identification of lines, as opposed to their accurate measurement, it was decided to make the apparatus of much simpler construction, and to take measurements by comparison with X-rays. The constructional details of the instrument will be described in the next section.

II. CONSTRUCTIONAL DETAILS

The constructional details of this spectrometer are shown in fig. (29).

The crystal clamping blocks H are mounted on a rigid bar A, and the camera support on another rigid bar B. Both these bars pivot at their ends about the ball race C, the centre of which defines the centre of the focusing circle. The crystal clamping blocks are mounted on a sliding piece E, so that a radial adjustment can be made in the position on the crystal. The groove in which E moves is cut so that its centre line points directly to the centre of the focusing circle and ensures that, once set, the crystal will always be tangential to a circle of centre O. In this spectrometer, no adjustment was designed to ensure that the crystal was tangential to the focusing circle. Instead, the machining and assembly of the blocks H and the sliding piece E were carried out in
Fig. (30). The plate-holder.

Fig. (31). The 20 cm. spectrometer.
such a way that this was automatically ensured. The camera was also mounted on a movable block F, so that its radial position could be adjusted. The details of the camera support are shown in fig. (30). It consists of a perspex frame attached to the block F, the photographic plate being held in position by the two spring clips shown. As an approximate indication of the relative settings of the crystal and camera, a protractor P was mounted on bar A and the angle was read by means of a pointer attached to bar B. When the relative positions of the two bars were fixed, it was possible to clamp them using the clamping circle G.

For a radius of curvature of the surfaces of 20 cm., the clamping blocks can be prepared more easily on a lathe than by the method of Dumond, Lind and Cohen. The steel used was the same as that used for the larger instrument. After as good a surface as possible had been obtained on the lathe, the blocks were again lapped until a good mirror finish was obtained. The concave one was then tested optically and found to have a radius of curvature of $20.00 \pm 0.02$ cm.

A photograph of the completed spectrometer is shown in fig. (31).
Fig. (32). Photographs showing performance of 20 cm. spectrometer.

(a) Silver K spectrum.

(b) Portion of that spectrum magnified 6 times.
III. PERFORMANCE OF SPECTROMETER

The crystal used with this spectrometer was a piece of Australian mica 2½" square by 0.5 mm. thick, cut from the same sheet as the crystal used in the large spectrometer. The orientation of the mica was again carried out by reference to a Laue diagram with the mica held between the clamping blocks. In this case the measured radius of curvature of the mica, after clamping, was found to be 20.0 ± 0.1 cm.

The resolving power of the spectrometer was then tested using the K X-rays from a silver scatterer. The radial positions of the crystal and camera were adjusted until the sharpest lines were obtained. It was found that, using the (201) planes, the silver Kα doublet was clearly resolved, the line width being 0.012 cm. when the whole crystal aperture was used. With the (100) planes the line observed was clearly a doublet, but the resolution was not as clear as in the previous case. The spectrum obtained using the (201) planes is shown in fig. (32).

After the best adjustment had been obtained with X-rays, tests were carried out to determine what length of exposures would be required with radioactive sources. The RaD source described in the previous chapter was used, and it was found that, using the same collimating system as previously, the exposure required to produce a visible line
corresponding to the 46.7 keV $\gamma$-ray was 7 hours, half the time that had been required with the large spectrometer.

The exposure required with a curved crystal spectrometer depends on the distance of the source from the crystal as well as on the radius of curvature of the crystal. In the experiments with RaD, the source was 14" from the crystal, but, if this distance had been reduced, the advantage gained by using a small spectrometer would have been greater.

This small spectrometer has been used in conjunction with the large one to examine the $\delta$-ray spectrum emitted by the 27.4 day activity of Pa$^{233}$. These experiments will be described in the next chapter.
Fig. (33). Level scheme suggested by Keller and Cork for $^{233}$U nucleus.
CHAPTER 7

STUDY OF DISINTEGRATION OF Pa\textsuperscript{233}

I. INTRODUCTION

The existence of a 27.4 day activity in protactinium was first suggested by Meitner, Strassman and Hahn\textsuperscript{(69)} in 1938. There was some doubt as to whether the activity did in fact come from protactinium, but finally, in 1941, it was assigned to the isotope 233 of protactinium by Seaborg and other workers.\textsuperscript{(70)} The $\beta$-spectrum was investigated by Haggstrom\textsuperscript{(71)} and Levy\textsuperscript{(72)} and they estimated that the continuum had an end point of approximately 200 KeV, although this was very difficult to measure accurately since it was heavily masked by conversion electrons.

Recently, in a thorough study of the $\beta$-spectrum, Keller and Cork\textsuperscript{(27)} have identified 46 conversion lines, confined to energies below 411 KeV. They assigned these conversion lines to 13 $\gamma$-rays, and on this basis proposed the level scheme shown in fig. (33). In this scheme all the $\gamma$-rays are accounted for, and, to make the scheme self-consistent, they postulated a 17.4 KeV $\gamma$-ray, though this had not been observed.

Still more recently, a report by Elliot and Underhill\textsuperscript{(28)} became available. They have also studied the internal conversion lines of Pa\textsuperscript{233} and observed lines corresponding to 9 different $\gamma$-rays.
Fig. (35). Comparison of the two level schemes proposed.

(a) The level scheme of Keller and Cork inverted.

(b) The level scheme of Elliot and Underhill.
Fig. (34). Decay scheme for Pa$^{233}$, proposed by Elliot and Underhill.
They found a very intense γ-ray at 310 KeV and, by measuring co-incidences, found that the three γ-rays of 76, 88, and 105 KeV, as well as about 75% of the continuum, were correlated with this level. They also examined the intensities of the γ-rays in emission by investigating the secondary electron spectrum ejected by the γ-radiation from a thin silver radiator. On the basis of these results, they proposed the decay scheme shown in fig. (34). To account for the intensities observed, they proposed 6 partial β-spectra leading to the required levels as shown. They did not report the existence of any γ-rays of energy lower than 76 KeV, but their proposed scheme could incorporate the low energy γ-rays of 58, 40, and 29 KeV reported by Keller and Cork. In fact, if the level scheme proposed by Keller and Cork is inverted and the energies correspondingly re-allocated, their scheme is very similar to that of Elliot and Underhill, as demonstrated in fig. (35).

The existence of the low energy γ-rays would require a redistribution of the intensities given by Elliot and Underhill, and perhaps remove the necessity for some of the partial β-spectra proposed. The present investigation was undertaken to examine the low energy region of the spectrum, and to attempt to identify some of the γ-rays reported by Keller and Cork, using a curved crystal spectrometer.
Fig. (36). Lead collimator used with the Pa$^{233}$ source.
II. THE SOURCE AND COLLIMATING SYSTEM

The source was prepared by the slow neutron bombardment of Th$^{232}$ in the A.E.R.E. pile, followed by the 23 minute $\beta$-decay to Pa$^{233}$. It was deposited on a piece of platinum 0.010" thick at the Radio-Chemical Centre, Amersham and ignited to remove the impurities. At the commencement of the present experiments it had an activity of approximately 130 mC, deposited over an area of 1 sq.cm. The whole source was covered with aluminium 0.1 mm. thick, to prevent loss of any of the deposit. Again a pin-hole photograph of the source showed the distribution over the platinum foil to be non-uniform. It was therefore necessary to use the whole source over the complete range in which any intensity measurements were being made.

Since both spectrometers were now available, a special lead collimator was designed, as shown in fig. (36), to enable both to view the source simultaneously. In this case, the lead baffles were made 1 cm. thick, with apertures 3/4 in. by 1/4 in. The ends of the collimator were machined so that they were exactly perpendicular to the central lines down the two sections which meet at the centre of the source as shown. This arrangement was used to enable accurate measurements to be made of the angle between the central ray and the crystal planes.

In the decay of Pa$^{233}$, the highest energy electrons
emitted in any appreciable quantity are those due to the L conversion of the 411 KeV γ-ray. These electrons were absorbed in 0.25gm/cm² of filter paper, placed immediately behind the first lead baffle. In intensity measurements, allowance was made for absorption in this paper, and sensitivity graphs were calculated similar to that used in the RaD experiments.

In the present experiments, the source had an effective width of 0.92 cm. and was placed 20 cm. from each crystal. The straight through beam, which limits the highest energy of γ-rays that can be observed, would in each case strike the plate at a position corresponding to reflection of a γ-ray of 95 KeV in the first order. As a result, the highest energy range that could be covered using the whole source was from 95 KeV to 14 KeV with the large spectrometer, and from 95 KeV to 16 KeV with the small spectrometer.

III. EXPOSURES TO Pa²³³

Since the half-life of Pa²³³ is 27.4 days, only a limited number of exposures could be given to the source. The maximum exposure given was 28 days, since very little more information could be gained by exposing for longer than one half-life.

The small spectrometer was set so that the central ray made an angle of 4.8° with the atomic planes, thus
covering the energy range from 90 KeV to 16 KeV using the whole source. Because of the lower dispersion of this spectrometer, this region occupies less than 4 cm. on the photographic plate, and so could be covered in one setting of the plate. The straight through beam struck the plate at a point corresponding to a $\gamma$-ray of 95 KeV, but it was found that scattering of the beam from the glass backing of the plate fogged an additional centimetre of the plate. In the later exposures, the straight through beam was screened off with lead, but this did not remove all the fogging, though it reduced it considerably. Initially, exposures of 1 week each were given using the (100) planes and the ($\overline{2}01$) planes, and then exposures of 2 weeks and 4 weeks using the (100) planes. In the 4 weeks exposure the 28.9 KeV and 88 KeV lines are clearly visible, though the latter is masked to a certain extent by the fogging and does not show up in the microphotometer trace. The 40.6 KeV line is just visible, though it is also masked by the straight through beam. The four plates were microphotometered, and the traces corresponding to the 2 and 4 weeks exposures are shown in Trace III (see end). These will be discussed in the next section.

Because of its greater resolving power, the large spectrometer was used to examine the uranium L X-ray region, to attempt to identify the 17.4 KeV $\gamma$-ray
Fig. (37). Plate obtained in the 20 day exposure to $\text{Pa}^{233}$, using the $(\overline{2}01)$ planes of the large spectrometer.
postulated by Keller and Cork. It was expected that this would be difficult to observe, due to the presence of the strong Lβ X-ray line of uranium at 17.3 KeV. For these experiments, the central setting was 7.5°, thus covering the region from the Lα X-rays to the Lβ X-rays using the whole source. Exposures of 1 week each were given to the (201) and (100) planes, and all the principal X-rays were observed. There was no decisive evidence for the existence of the 17.4 KeV γ-ray, though its presence would be masked by the heavy exposure of the Lβ X-rays. After these first exposures, the setting of the spectrometer was altered so that the range from 90 KeV to 14 KeV was covered using the whole source. Exposures of 10 days to the (100) planes and 20 days to the (201) planes were given, care being taken to shield off the direct beam. The plate obtained in the 20 day exposure using the (201) planes is reproduced in fig. (37). The uranium Kα X-rays and the platinum Kα X-rays reflected in the second order can be seen.

IV. DISCUSSION OF PHOTOGRAPHS OBTAINED

In the exposures described above, no calibration lines were put on the plate, since there were the K and L X-ray spectra of uranium of known wavelengths, and, in addition, the K X-ray spectrum of platinum excited by the intense bombardment of the backing of
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</tbody>
</table>

Table VII. L X-rays observed in microphotometer traces.

(With the exception of "a" and "c", the L X-rays are those of uranium.)
the source.

In Trace III (a), corresponding to the 4 weeks exposure, the 28.9 KeV $\gamma$-ray can be clearly seen, and there is evidence for the existence of the 40.6 KeV $\gamma$-ray. The 88 KeV $\gamma$-ray, though it could be seen visually by shining an intense light through the plate, is obscured in the trace by the effect of the straight through beam. The L X-ray spectrum in this trace is heavily over-exposed, and the total width of the lines is thus increased. However, the weaker lines are brought up, and in addition some of the platinum L X-rays are visible. A list of the observed L X-rays and their identification is given in Table VII opposite. Trace III (b), corresponding to the 2 weeks exposure, is essentially the same as III (a), except that the lines are of weaker intensity.

One of the differences between the two level schemes proposed for Pa$^{233}$ is that that of Elliot and Underhill has two levels separated by 12 KeV, and so could accommodate a 12 KeV $\gamma$-ray. A careful search was carried out in this region, but, apart from the slight irregularities in the two curves at the point A, corresponding to 12.1 KeV, no evidence could be found for the existence of this $\gamma$-ray. It should, however, be pointed out that, in the exposures corresponding to these traces, only half of the source was being used and the absorption in the mica would considerably
<table>
<thead>
<tr>
<th>L X-ray</th>
<th>Transition</th>
<th>Pa$^{233}$</th>
<th>U</th>
<th>U(a)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$\alpha_3$</td>
<td>L$_{III}$ - M$_I$</td>
<td>2.8</td>
<td>3.4</td>
<td></td>
</tr>
<tr>
<td>$\alpha_2$</td>
<td>L$<em>{III}$ - M$</em>{IV}$</td>
<td>11</td>
<td>12</td>
<td>11</td>
</tr>
<tr>
<td>$\alpha_1$</td>
<td>L$_{III}$ - M$_V$</td>
<td>100</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>$\gamma$</td>
<td>L$_{II}$ - M$_I$</td>
<td>1.5</td>
<td>1.0</td>
<td></td>
</tr>
<tr>
<td>$\beta_6$</td>
<td>L$_{III}$ - N$_I$</td>
<td>1.5</td>
<td>1.6</td>
<td></td>
</tr>
<tr>
<td>$\beta_2$</td>
<td>L$_{III}$ - N$_V$</td>
<td>26</td>
<td>26</td>
<td>28</td>
</tr>
<tr>
<td>$\beta_4$</td>
<td>L$<em>I$ - M$</em>{II}$</td>
<td>26</td>
<td>3.6</td>
<td>4.1</td>
</tr>
<tr>
<td>$\beta_5$</td>
<td>L$<em>{III}$ - O$</em>{IV}$</td>
<td>6.0</td>
<td>6.3</td>
<td>6.4</td>
</tr>
<tr>
<td>$\beta_3$</td>
<td>L$<em>{II}$ - M$</em>{IV}$</td>
<td>85</td>
<td>48</td>
<td>49.4</td>
</tr>
<tr>
<td>$\beta_9$</td>
<td>L$<em>I$ - M$</em>{III}$</td>
<td>28</td>
<td>3.8</td>
<td>4.2</td>
</tr>
<tr>
<td>$\beta_{10}$</td>
<td>L$_I$ - M$_V$</td>
<td>2.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$\gamma_1$</td>
<td>L$<em>{II}$ - N$</em>{IV}$</td>
<td>20</td>
<td>11</td>
<td>12</td>
</tr>
<tr>
<td>$\gamma_2$</td>
<td>L$<em>I$ - N$</em>{II}$</td>
<td>11</td>
<td>1.7</td>
<td>1.5</td>
</tr>
<tr>
<td>$\gamma_3$</td>
<td>L$<em>I$ - N$</em>{III}$</td>
<td>10</td>
<td>1.4</td>
<td>1.4</td>
</tr>
<tr>
<td>$\gamma_6$</td>
<td>L$<em>{II}$ - O$</em>{IV}$</td>
<td>3.7</td>
<td>2.0</td>
<td>2.2</td>
</tr>
<tr>
<td>$\gamma_4$</td>
<td>L$<em>I$ - O$</em>{III}$</td>
<td>3.0</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

(a) Values obtained by Allison.

Table VIII. Relative intensities of L X-rays emitted in the decay of Pa$^{233}$, compared with those of uranium.

$^{+4}$

<table>
<thead>
<tr>
<th>Level</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>L$_{II}$</td>
<td>147.3 : 144.5 : 150.4</td>
</tr>
<tr>
<td>L$_{IV}$</td>
<td>110.2 : 61.5 : 64.6</td>
</tr>
<tr>
<td>L$_{III}$</td>
<td>80 : 10.5 : 11.2</td>
</tr>
</tbody>
</table>


reduce the intensity of such a line.

In fig. (37), corresponding to the 20 day exposure on the large spectrometer using the (201) planes, the platinum Kα doublet and the uranium Kα doublet reflected in the second order can be clearly seen. The uranium Kα X-ray corresponds to an energy of 98.4 KeV, which is the highest energy line yet measured by this instrument. The 28.9 KeV line in the first order and the 88 KeV line in the second order are just visible in the original plate, though not in the reproduction. In the photograph corresponding to the 10 day exposure using the (100) planes, the 28.9 KeV line can again be seen, but no other γ-rays were observed. In this case, the γ-ray line falls in exactly the same position as previously, and so it is unlikely that the line at 28.9 KeV observed using the (201) planes is a second order reflection of the 58 KeV line, which has not been observed on any of the plates.

The intensities of the L X-ray lines have been measured and are given in Table VIII. They are compared with those of the fluorescent X-ray spectrum measured with the large spectrometer, and also with the values determined by Allison.\(^{(53a)}\) In this case, the ratio of the fluorescent yields of the L\(_I\), L\(_{II}\), and L\(_{III}\) levels is 0.54:0.75:1, as compared with the theoretical value of 0.144:0.053:0.225 deduced by Kinsey. However, since,
at the present time, the multipolarity of the $\gamma$-rays emitted in the decay of $^{233}$Pa is not known, very little interpretation of the measured value is possible.

From measurements of the positions of the lines, the wavelengths of the $\gamma$-rays observed were determined, using the known values for the uranium K and L X-rays, and the platinum K X-rays. Using the atomic constants given by Dumond and Cohen, the energies have been calculated, and were found to be $28.75 \pm 0.05$ KeV, $40.6 \pm 0.1$ KeV, $87.6 \pm 0.15$ KeV.

V. DISCUSSION OF RESULTS

The $28.7$ KeV and the $87.6$ KeV $\gamma$-rays from $^{233}$Pa have been clearly identified, and there is strong evidence in the microphotometer trace for the existence of the $40.6$ KeV $\gamma$-ray. The present experiments have failed to identify the $58$ KeV $\gamma$-ray, though this could be due to under-exposure. The $75$ KeV $\gamma$-ray coincides with the $K\beta_2$ doublet from the platinum on which the source was mounted to allow for removal of the impurities by ignition at a high temperature. The intensity of this doublet relative to the $K\alpha$ lines cannot be determined with sufficient accuracy to give conclusive evidence for the existence or non-existence of this $\gamma$-ray. It is difficult to form a good estimate of the relative intensities of the observed lines from the present experiments, but approximate
<table>
<thead>
<tr>
<th>$\gamma$-RAY (KEV)</th>
<th>EWAN QUANTA</th>
<th>ELLIOT AND UNDERHILL QUANTA</th>
<th>CONVERSION ELECTRONS</th>
</tr>
</thead>
<tbody>
<tr>
<td>28.7</td>
<td>0.60</td>
<td></td>
<td></td>
</tr>
<tr>
<td>40.6</td>
<td>0.40</td>
<td></td>
<td></td>
</tr>
<tr>
<td>76</td>
<td></td>
<td>8</td>
<td>20</td>
</tr>
<tr>
<td>87.8</td>
<td>15</td>
<td>10</td>
<td>24</td>
</tr>
<tr>
<td>105</td>
<td></td>
<td>$\leq 9$</td>
<td>11</td>
</tr>
<tr>
<td>$K\alpha_1$</td>
<td>60</td>
<td></td>
<td>3</td>
</tr>
<tr>
<td>$K\alpha_2$</td>
<td>30</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$K\beta^*$</td>
<td></td>
<td>30</td>
<td></td>
</tr>
<tr>
<td>298</td>
<td></td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>310</td>
<td>100</td>
<td>100</td>
<td>25</td>
</tr>
<tr>
<td>339</td>
<td>10</td>
<td>10</td>
<td>2.5</td>
</tr>
<tr>
<td>398</td>
<td></td>
<td>0.2</td>
<td>0.05</td>
</tr>
<tr>
<td>415</td>
<td>12</td>
<td>$\leq 1.3$</td>
<td>0.4</td>
</tr>
<tr>
<td>474</td>
<td>$\leq 0.4$</td>
<td>0.07</td>
<td>$\sim 0.01$</td>
</tr>
</tbody>
</table>

Table IX. Intensities of $\gamma$-rays observed by Ewan and by Elliot and Underhill.
values have been obtained.

From comparison of the exposure times that were required to produce a visible Lβ, X-ray line and the 28.9 KeV line, and making allowance for the change of sensitivity with energy, it was estimated that one 28.9 KeV photon was emitted for every 150 L X-ray photons. From the relative intensities given by Elliot and Underhill for photons and internal conversion electrons, it has been calculated that there are approximately 56 L-ionisations per 100 disintegrations. Using Kinsey's corrected value of 52% for the L fluorescent yield for uranium, this corresponds to 29 L X-rays, and consequently the intensity of the 28.9 KeV γ-ray is approximately 0.20 photons per 100 disintegrations.

The 40.6 KeV γ-ray has an observed intensity of approximately half that of the 28.9 KeV γ-ray, and so, after allowance has been made for the sensitivity variation, it has an estimated intensity of approximately 0.13 photons per 100 disintegrations.

The 88 KeV γ-ray is about half the intensity of the uranium \( Kα_2 \) line, and so corresponds to 5 photons per 100 disintegrations, using the Elliot and Underhill intensities.

Table IX gives a list of the γ-rays observed by Elliot and Underhill, with their values for the relative both intensities/in emission, and as internal conversion.
electrons. The results obtained in the present experiments for the low energy $\gamma$-rays, normalised to the same scale, are also shown. The intensities of the 28.9 KeV and 40.6 KeV $\gamma$-rays are low in emission, but in general low energy $\gamma$-rays are highly internally converted, and so their total intensity may be considerably greater.

The results of the present experiments indicate that, if the Elliot and Underhill decay scheme is to be adopted, a re-allocation of intensities will be required. The extent of this and the possibility of its removing the necessity for some of the partial $\beta$-spectra postulated cannot be estimated until the relative intensities of conversion of these $\gamma$-rays have also been determined.
CHAPTER 8
CONCLUSIONS

The initial object of the research described in this thesis was to construct a curved crystal spectro-meter and investigate the uses of such an instrument when applied to the study of low energy γ-rays emitted by radio-active sources. It has been found possible to obtain a visible line with the 46.40 cm. instrument in an exposure time of approximately 50 mC-hours for a radiation of energy less than 50 KeV. For the 20 cm. spectrometer, the corresponding exposure required is approximately 25 mC-hours. However, in the second case, the resolution is only 3 X.U., compared with 1.6 X.U. with the larger instrument, using first order reflection. The exposures quoted above were obtained using the (100) planes of a carefully selected crystal of mica and Ilford G5 200 μβ-sensitive emulsions to record the spectral lines. The range of energies of radiation which has been observed is from 12 KeV to 98.4 KeV, the latter using the (Z01) planes of mica reflecting in the second order. The lower limit is set by absorption in the crystal and in the air path between the source and the photographic plate, and so, if it is required to measure lower energy radiations, it would be advisable to use a thin crystal of mica and place the whole apparatus in a vacuum. The upper
limit is set by the fogging of the photographic plate by the straight through beam. With the simple collimating system at present used, it would be possible to measure \( \gamma \)-rays of energies up to 160 KeV, but, if higher energy \( \gamma \)-rays were to be measured, considerable attention would have to be paid to the design of the collimating system. However, since photographic recording has lost its main advantages in this region, it would be advisable to use the Dumond modification described in Section IV of Chapter 2.

The exposures quoted above are those for the amount of the radiation emitted of a given energy. Low energy \( \gamma \)-rays are in general highly internally converted. For example, the 46.7 KeV \( \gamma \)-ray is present in emission only 2.8 times per 100 disintegrations, and so an exposure of 14 hours was required, using a source of total activity 100 mC. The weakest line that has been observed is the 40.6 KeV \( \gamma \)-ray from Pa\(^{233}\), whose estimated intensity was 0.13 per 100 disintegrations, although, in the experiments on RaD, a \( \gamma \)-ray of intensity 0.06 per 100 disintegrations could have been observed. While these exposures are practicable if intense sources are available, it would considerably improve the usefulness of the apparatus if they could be further reduced, and possible methods of doing this will now be discussed.

The three principal variables which determine the
exposures required are:

(a) The type of crystal and the planes used.
(b) The type of detector used.
(c) The radius of curvature of the crystal.

In addition, the perfection of bending of the crystal will influence the focusing, and hence the contrast between the line and background. The results obtained with the hardened steel clamping blocks have been highly satisfactory, and little improvement can be expected in this direction.

In the experiments described, the (100) planes of a piece of Australian mica were used because of their high reflecting power consistent with good resolution. Comparison tests were carried out with other samples of mica, and it seems unlikely that any great degree of improvement will be gained by using a different sample of mica. The most promising line of advance in the choice of crystal would appear to be in the use of monocrystals of aluminium, prepared by a method described by Tiedema.\(^{(73)}\) Recently Cauchois\(^{(74)}\) used these in an X-ray spectrometer, and found the reflecting power four times as great as that of mica, with very little loss of resolution.

If photographic recording is being used, there seems very little hope of improving on the use of 200 \(\mu\beta\)-sensitive emulsions, though the sensitivity of these emulsions may be further improved by the
manufacturers. If the thickness of the emulsion is increased, the absorption of a \( \gamma \)-ray is increased, and so the total blackening, but the background stain is also increased and there is very little improvement in the contrast between a line and the background. The use of a scintillation counter instead of a photographic plate would probably prove useful for examination of a limited region of a spectrum. However, the size of pulse obtained with low energy \( \gamma \)-rays is comparatively small, and so, if this type of technique was being used, considerable attention would have to be paid to the removal of background pulses due to noise in the photomultiplier tube.

Very little is to be gained by reducing the radius of curvature of the crystal below 20 cm., since the resolving power of the instrument is considerably reduced. If a further reduction in resolving power can be tolerated, it would probably be better to use a plastically bent crystal, such as rock salt, which gives very intense reflections, bent to a greater radius of curvature. However, if rock salt is being used, great care must be taken to prevent the production of ghost images and the consequent misinterpretation of spectral lines.

The experiments on the L X-ray spectra of bismuth and uranium indicate that the sensitivity graphs calculated for the spectrometers are fairly reliable
in the region below 20 KeV. However, observations on the relative intensities of the K and L X-ray lines emitted in the decay of Pa$^{233}$ suggest that the sensitivity of the apparatus falls off more rapidly than shown by the graph. The experimental examination of the calibration curve will therefore have to be extended to the higher energy region if accurate measurements of relative intensities of $\gamma$-rays over a large range of energies are required.

Although more development work along the lines indicated above might further reduce the exposures required to radio-active sources, the results of the experiments on RaD and Pa$^{233}$ described in this thesis show that the curved crystal spectrometer technique can make a useful contribution to the study of low energy $\gamma$-radiations.
Fig. (38). Lattice of mica, used by Cauchois, projected on the (010) plane.

Fig. (39). Lattice proposed by Jackson and West, projected on the (010) plane. The unit cell has constants \( a = 5.18 \, \text{Å}, \quad b = 9.02 \, \text{Å}, \quad c = 20.04 \, \text{Å}, \)

\[ \text{and} \quad \beta = 95^\circ 30' \]
Mica is known to be a monoclinic crystal, though the exact structure of the unit cell varies with different types. Throughout this thesis, the nomenclature used to designate the crystal planes has been that of Cauchois. This is based on a unit cell as shown in fig. (38) of constants

\[
\begin{align*}
a &= 5.32 \text{ Å} \\
b &= 8.94 \text{ Å} \\
c &= 10.06 \text{ Å} \\
\beta &= 100^\circ 10' 
\end{align*}
\]

The planes used in the present experiments are shown in red. They are the \((\overline{2}01)\) in both the first and second orders, and the \((200)\) in the first order. In this thesis, the \((200)\) has been designated \((100)\) for the sake of consistency with the nomenclature used by other workers in the field of curved crystal spectroscopy.

The structure of the unit cell proposed by Jackson and West is shown in fig. (39). In this case, the monoclinic angle is \(95^\circ 30'\) and the planes corresponding to those used are the \((200)\) in the first and second orders, and the \((\overline{2}02)\) in the first order.

The exact values of the lattice spacing and the monoclinic angle vary slightly among different types.
of mica, and so, if absolute measurements are being made, these have to be determined for the sample of mica used. However, in the experiments described in this thesis, all measurements have been made by reference to X-ray lines of known wavelengths, so this has been unnecessary.
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(Abbreviations are as listed in Physics Abstracts, January, 1950)