### THE DEVELOPMENT OF THE CURVED QUARTZ CRYSTAL X-RAY SPECTROGRAPH AND A DETERMINATION OF THE GRATING CONSTANT OF QUARTZ

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### Abstract

The development of the curved crystal type of high luminosity x-ray spectrograph is traced from the original suggestion by Gouy in 1916 through the work of Cauchois in 1932 and 1933. The construction of a small experimental spectrograph of the curved crystal type is described. The use of quartz instead of mica or gypsum which are customarily used and the improvement of the spectra obtained when quartz is used are discussed.

The theory of the curved crystal spectrograph has been extended in two ways - (1) to cover the doubling of spectral lines which arises due to reflections from opposite surfaces of the crystal and (2) to cover the vertical separation of spectral lines reflected from opposite sides of atomic reflecting planes. This last effect is traced to a non-parallelism between the reflecting planes and the axis of curvature of the crystal.

By means of the curved crystal spectrograph the grating constant for the basal planes of quartz was determined. This value was found to be  $d = 5.393 \pm .002$  Å

### Historical Summary

From the very beginnings of x-ray spectroscopy the need of methods for increasing spectral intensities was keenly felt. There are many reasons why this should be so. In the first place all serious spectroscopic work in x-rays must, in the present state of our knowledge, make use of selective scattering from crystals and this is a somewhat inefficient process. In addition, a further limitation is imposed which may be expressed mathematically by the Bragg equation,  $n\lambda = 2d\sin\theta$ . This equation states that a given wavelength will be selectively scattered in a given order only if incident upon the crystal at a specified angle. Fortunately the Bragg equation is not strictly true and in reality a wavelength  $\lambda$  will be selectively reflected with intensity appreciably different from zero if incident within a finite but very small range of angles in the neighborhood of Q. As a result with the use of a flat crystal only a very small portion of the crystal can be effective if sharp spectra are to be obtained. Over the inefficiency of the scattering process we have little or no control but we are able as a result of methods developed within recent years to increase the portion of a crystal which may be offective in the selective scattering of x-rays. One of these methods forms the subject of this paper.

Before discussing the various methods used in developing high luminosity spectrographs it might be well to point out another reason why the need for these spectrographs is so acute. Much of the interesting x-ray work today concerns the scattering of x-rays from gases, liquids and solids; the structure of the Compton line and related problems. In all of this work the beam of x-rays incident on the spectrograph has already suffered a diminution of intensity as a result of scattering se that the further diminution necessitated by the very process of spectrum formation may cause the entire disappearance of any contrast between the spectral distribution of radiation and the general background.

The term "focussing" will be used in this paper to denote a true accumulation of intensity at a point. It is essential that this be clear at the outset since workers in x-rays are familiar with another type of "focussing" spectrograph, that of Bragg, in which a focussing condition was used to average out irregularities of reflection from a crystal face but in which there was no actual piling up of intensity at any point.

We shall concern ourselves chiefly with that type of high luminosity spectrograph in which focussing is obtained as a result of reflections from atomic planes of a crystalline lamina which has been bent into the form of a circular cylinder. Presently, however,

we shall discuss a very ingenious high luminosity spectrograph, that of DuMond and Kirkpatrick, in which many individual plane crystalline surfaces properly distributed and oriented play the same roles as the atomic planes of the curved crystal type.

The first mention of the use of a curved crystal in x-ray spectroscopy was, to our knowledge. made by M. de Broglie and Lindemann" in 1914. They did not, however, investigate the focussing properties of a curved crystal but merely made the suggestion to indicate an alternative technique to the oscillating crystal method. Gouy in 1916 made the first analysis of the focussing of x-rays by curved crystals. He discussed the formation of real images by crystalline lamina bent into the shape of circular cylinders. In spite of this early start in the theory of high luminosity spectrographs no progress toward the actual realization of such a spectrograph was made until the construction of the multicrystal spectrograph by Dumond and Kirkpatrick.

In an early paper<sup>(3)</sup> in the Review of Scientific Instruments DuMond and Kirkpatrick described the design of the multicrystal spectrograph. It is an interesting observation that at the time of the publication of this paper (Pebruary 1930) DuMond had the complete idea of the curved crystal spectrograph two years before it was brought to practical realization by Mlle. Cauchois. The authors first discuss in general the focussing of x-rays by crystals. Their analysis is essentially as follows: 4

We may consider first the case in which the reflecting crystal planes are parallel to the surface of the crystal and ask whether it is possible to bend the crystal into conformity with some simple smooth surface in such a way that monochromatic x-rays from a point source at A will after reflection from the crystal converge to a point image at B.



FIG. I

There are two conditions which must be satisfied for Bragg reflection from such a surface - (1) the angles of incidence and reflection (measured with the surface) must be equal at each point of the surface and (2) the angle of deviation between the incident and reflected beams must be a constant for all points of the surface, the constant of course depending on the particular wavelength under consideration. The first condition dictates the direction of the surface element at each point of the reflecting surface while the second specifies the position of each surface element. In general these two conditions are not satisfied at the same time over a finite area of surface so that by simply bending a crystal it is impossible to obtain perfect focussing of x-rays.

Perfect focussing can be obtained, as DuMond has shown, if the crystal is both ground and bent in a specified way. Consider the two possibilities below.





FIG. 3

In Figure 2 the reflecting planes were before grinding parallel to the surface of the crystal. After

grinding and bending the perpendiculars to the reflecting planes intersect at C while the surface of the crystal conforms to a circle with center at O. In Figure 3 the reflecting planes were before grinding perpendicular to the surface of the crystal. After grinding and bending the reflecting planes would intersect at C if prolonged while the surface again coincides with a circle of radius OC and center at O. In both cases, as is apparent from the diagrams, the two conditions for x-ray reflection are simultaneously satisfied at all points of the surface.

Because they thought it impossible to grind and bend a crystal with the accuracy required for spectrographic work and because they wished to be able to explain their results on the broadening of the Compton line with certainty DuMond and Kirkpatrick constructed the now well-known multicrystal spectrograph. The principle of the multicrystal spectrograph is essentially that of the perfect focussing type of curved crystal spectrograph shown in Figure 3. The plane surfaces of fifty individual crystals take the place of the internal reflecting planes. Each crystal surface is confronted with a brass wedge making in reality fifty Seeman type spectrographs distributed along the arc of a circle and so oriented that their surfaces if prolonged would intersect at a point on the same circle.

The DuMond-Kirkpatrick spectrograph marked a

long step forward in spectroscopic technique. It increased the luminosity obtainable from an x-ray spectrograph by a factor of fifty. Its construction must have required as much ingenuity as its adjustment required patience. The chief disadvantage of the multicrystal spectrograph is that it requires for complete effectiveness a quite extended source of x-radiation.

In order to still further increase the luminosity obtainable and in order to circumvent the necessity for a very extended source of x-rays two different types of curved crystal spectrographs have been developed within recent years. These two types are fundamentally those discussed by DuMond in 1930 with the exception that perfect focussing has been sacrificed for ease of construction. Figure 4 shows the curved crystal spectrograph as developed by Johann in 1931; Figure 5 that developed by Cauchois (3,(4),(7),(8). in 1932. In both types if the aperture of the crystal



FIG. 4

is small, that is, if the position of the crystal surface does not deviate much from its position for perfect focussing then sharp spectral lines can be obtained. The Cauchois type of spectrograph is of greater universal applicability and will be dealt with exclusively in this paper.

### Principles of the Transmission Type of Curved Crystal Spectrograph

Cauchois has discussed the simple theory of the curved crystal spectrograph as regards focussing, luminosity, dispersion and defects of construction.



FIG. 6

Consider a crystal bent along the arc of a circle with center at C. Let I, T and M (Figure 6) be three points on the crystal. X-rays of a given wavelength  $\lambda$  will be reflected from a set of trans-verse planes of the crystal (not necessarily perpendicular to the surface of the crystal) in such a way that each ray after reflection will make a constant angle 0 (Bragg angle) with its reflecting plane. If the reflecting planes make an angle  $\epsilon$  with the normals to the crystal surface then the reflected x-rays will make with these normals constant angles u where  $u = |\Theta \pm \epsilon|$ 

Let us inquire after the equation of the geometrical path of a single reflected ray MD. Let the coordinates of M be (x,y). Then

 $x_o = R \cos \phi$  $y_o = R \sin \phi$ 

The slope of MD is given by

y - y = m(x - x)

 $m = \tan(\phi + u)$ 

The equation of MD is

y - R sin  $\phi = \tan (\phi + u) \left[ x - R \cos \phi \right]$  (1)

We wish now the envelope of the reflected rays as  $\phi$  varies. We obtain this by differentiating equ. (1) with respect to  $\phi$  and solving for x and y from (1) and the derived equation (2).

-  $R \cos \phi = \sec^2(\phi + u) \left[ x - R \cos \phi \right] + \tan(\phi + u) R \sin \phi$  (2)

This gives for the envelope in parametric form

- $x = R \sin u \sin (\phi + u)$  (3)
- $y = -R \sin u \cos (\not y + u)$  (4)

a circle of radius R sin u with center at C.

or

As  $\omega \to 0$  ( $\phi \to 90^{\circ}$ ), that is, for small crystal apertures equations (3) and (4) reduce to

<b>x</b> = R	sin u	cos u	(5)	
y = R	sin <sup>2</sup> u		(6)	
<b>x</b> <sup>2</sup> +(	y - R/2	$\left(\frac{1}{2}\right)^2 = \frac{R^2}{4}$	(7)	

Thus for a vanishingly small crystal aperture the reflected x-rays of a given wavelength (constant u) intersect at a point (x,y) specified by equations 5 and 6 and as  $\lambda$  varies the point of intersection moves along a circle (equation 7) with center at (0,R/2) and with radius R/2. This circle with a radius of curvature one half that of the crystal and tangent to the crystal is, then, the focal circle. The focussing is perfect only for zero aperture - it then becomes identical with the DuMond case, Figure 3 - but for all useful apertures the focussing though not exact can be made as sharp as desired by keeping the size of the aperture within well defined limits.

Just how serious the defect of focussing due to the finite size of the aperture is can be arrived at by simple reasoning. Let CD (Figure 7) be the reflected ray through the point of tangency of the crystal and focal circle and let M be one extremity of the crystal.



### FIG. 7

If the DuMond conditions were satisfied, that is, if the crystal conformed to the arc CN instead of the arc CM while at the same time the crystal planes intersected on the circle CNO, then all reflected rays of a given wavelength would be focussed at D. In the present case, however, the ray reflected at M will intersect the focal circle at E as is geometrically obvious.

### MO = R

#### $NO = R \cos \omega$

 $MN = R(1 - \cos \omega) = R(1 - 1 + \omega/2 + ---) = R\omega/2$ 

For  $\omega$  small LN = MN tan u = R $\omega^2/2$  tan u

Let the length of the aperture be  $a = 2MC = 2\omega R$ 

 $\omega^2 = a^2/4R^2$ 

### $LN = DE = 1 = a^2 / 8R \tan u$ (8)

Thus the width of the spectral line as a result of imperfect focussing is directly proportional to the square of the linear aperture of the crystal and inversely proportional to the radius of curvature of the crystal.

The luminosity of the transmission type of curved crystal spectrograph is to us its most interesting feature and it would be well to form some idea of the order of magnitude of the increase in luminosity over that obtainable from a single flat crystal spectrograph. If the path traversed by the x-rays from source to photographic film is the same for the two types of spectrographs then the luminosity of the curved crystal spectrograph will be roughly proportional to the aperture of the crystal while that of the flat crystal spectrograph will be proportional to the length of crystal effective in selectively reflecting x-rays. This latter length is from Figure 8 seen to be equal to 1/sin @ where 1 is either the width of the slit or the width of the spectral line and 9 is the Bragg We can write then angle.

## Lexa

### $L_r \propto 1/\sin \theta$

In comparing luminosities we are interested





in the energy which can be concentrated into a given region in a given time. We must therefore compare the luminosities of the two spectrographs when they are producing spectral lines of the same width. We have already derived an expression for the width of the spectral lines formed by a curved crystal spectrograph when only the effect of finite aperture is taken into account. This was

 $1 = a^2/8R \tan u$ 

The ratio of the luminosities is thus given

by

$$P = L_c/L_r = a/1 \sin \Theta = 8R/a \sin \Theta/\tan u$$
$$= 8R/a \sin \Theta/\tan (\epsilon \pm \Theta)$$
$$= 4 \sin \Theta/[\omega \tan (\epsilon \pm \Theta)]$$
(9)

and the second second

where 
$$\omega$$
 is half the angular aperture of the crystal.  
If we use a possible set of values for  $\omega$ ,  $\epsilon$   
and  $\Theta$  such as  $\omega_3$   $3^\circ$ ,  $\epsilon = 3^\circ$ ,  $\Theta = 11^\circ$  we obtain a multi-

plication of intensity by a factor of 100. In terms of exposure times this would mean that if to produce a satisfactory spectral photograph required one hour with a flat crystal spectrograph, a similar photograph could be obtained with a curved crystal spectrograph in 36 seconds.

We can also very simply obtain an expression for the dispersion of a curved crystal spectrograph. The linear separation on a photographic film between spectral lines associated with wavelengths  $\lambda_{,}$  and  $\lambda_{z}$ and Bragg angles  $\Theta_{,n}$  and  $\Theta_{2n}$  in the nth order is evidently given by

$$\Delta = R(\Theta_{n} - \Theta_{n}) \tag{10}$$

where R is again the radius of curvature of the crystal. The Bragg equation is

$$n\lambda = 2d \sin \Theta_{n}$$

$$n d\lambda = 2d \cos \Theta_{n} d\Theta_{n}$$

$$d\lambda/\lambda = \cot \Theta_{n} d\Theta_{n} = \cot \Theta_{n} \Delta/R$$

$$\Delta = R d\lambda/\lambda \tan \Theta_{n} \qquad (11)$$

It is often desirable to be able to compute the grating constant d for a given set of planes from separations between two spectral lines either of different wavelengths in the same order or of the same wavelength in different orders. All of the formulae for accomplishing this which have come to our attention in the literature of the curved crystal spectrograph

have been derived on the assumption of separations between lines small enough to permit approximations to be made. Since formulae in which no approximations have been made may be useful to others as they have been to us we state them here.

To find d from the separations between lines of wavelength  $\lambda$  in orders n, and n, consider the relations

 $n_{\lambda} = 2d \sin \Theta_{\lambda}$  $n_{\lambda} \lambda = 2d \sin \Theta_{\lambda}$  $\Theta_{\lambda} = \Theta_{\lambda} = \delta/R$ 

where  $\delta$  is the linear separation between the lines on the photographic film. By simple manipulations it is possible to obtain the result

$$d = \frac{\lambda}{2} \sqrt{n_{i}^{2} + \left(\frac{n_{2} - \varkappa, \cos \frac{s}{R}}{sin \frac{s}{R}}\right)^{2}} (12a)$$
$$d = \frac{n}{2} \sqrt{\lambda_{i}^{2} + \left(\frac{\lambda_{2} - \lambda, \cos \frac{s}{R}}{sin \frac{s}{R}}\right)^{2}} (12b)$$

where  $\delta$  is now the linear separation between two spectral lines in the same order n but of different wavelengths  $\lambda_i$  and  $\lambda_i$ .

### Description of the Apparatus

Similarly

In November 1933 at the suggestion of Dr. DuMond I undertook an investigation of the curved crystal spectrograph for the purpose of studying the advantages which it offered and extending, if possible, its effectiveness and range of applicability. We designed for this investigation an experimental spectrograph of small radius of curvature which was constructed in the machine shop of this Institute by Mr. S. A. Macallister.

The design of the spectrograph is shown on Plates One and Two. Plate Three shows a photograph of the instrument. The crystal holder consists of two brass plates, the surfaces of contact having been turned to a radius of eight inches. The aperture in the crystal holder through which the radiation passes is rectangular in shape being 13/16 inches long and 7/16 inches wide. The crystal is curved by being imprisoned between the two plates which form the crystal holder. The crystal holder has been built so that it may be turned upside down without changing any other adjustments. The ability to invert the crystal is important in the measurement of the angle between the crystal planes and the normal to the crystal surface.

The film holder consists also of two brass lamina, the surfaces of contact being, in this case, turned to a radius of four inches. The film is, like the crystal, given its proper curvature by being imprisoned between the two lamina. The opening in the front plate of the film holder through which the





## PLATE ONE

## CRYSTAL HOLDER





FILM HOLDER

# CROSS-SECTION THROUGH AXIS





radiation passes is approximately 5 1/2 inches long and 3/4 inches wide. This opening is covered with black paper when the spectrograph is in use. Both the crystal holder and the film holder may be rotated about a common axis which is the axis of curvature of the film holder.

The spectrograph is equipped with a divided circle concentric with the axis about which the crystal and film holders rotate. The circle is graduated in half degrees. There are in addition two verniers, one fixed to the crystal holder and one to the film holder which enable the position of each to be read directly to one minute of arc.

The spectrograph is mounted on a tripod equipped with levelling screws. When in use the instrument is covered with a lead box pierced only by a rectangular opening in front of the crystal.

### Crystals Employed

Our original intention was to use the spectrograph with mica as the crystal. Mica is the natural crystal to use since it can be given a fairly small radius of curvature up to a considerable thickness. Our experience with mica, however, was far from encouraging. Although we obtained spectra of great intensity as was to be expected in no case

did we obtain spectral lines which were straight and in many cases our lines showed well defined breaks along their length.

Early in the summer of 1934 we decided to abandon the use of mica and attempt the use of quartz. Quartz had not, to our knowledge, been used in a curved crystal spectrograph up to that time. Quartz possesses the distinct advantage that it has a small effective grating constant parallel to its basal planes giving greater dispersion and better resolution. Its chief disadvantage for this type of spectrograph is that it does not cleave easily in thin sheets making it necessary to grind the crystal down to the thickness required.

We prepared the crystal for use by first cutting a large crystal of quartz about 5 inches long and 1 1/2 inches thick into thin slabs parallel to the trigonal axis. We had computed that in order to allow bending to a radius of curvature of 8 inches the crystal must be no thicker than 0.4 mm. To allow a factor of safety we ground the crystal until it was 0.21 mm. thick. By measurements with a micrometer caliper we determined that the two surfaces of the crystal were parallel with a maximum deviation of 0.01 mm.

Late in June or early in July 1934 we obtained our first spectral photograph with quartz. We have reported this work elsewhere" together with one of our early photographs.

Our experience with quartz has been uniformly good. The spectra we obtained have been sharp and clear and the spectral lines straight. Its luminosity, also, has been up to our expectations. We have obtained a clear though somewhat underexposed photograph of the Compton lines of Mo K< radiation scattered from carbon in a period of 10 hours. A similar photograph on the multicrystal spectrograph would probably have required a few hundred hours.

Recently Erik Carlsson Ingelstam working in Upsala has brought to our attention that he had used quartz in a curved crystal spectrograph<sup>(\*\*)</sup> late in 1933.

### Extensions of the Theory of the Curved Crystal Spectrograph

From the very beginnings of our work with a quartz crystal we encounterd two effects which are not treated elsewhere and for which we sought an explanation. The first of these effects gives rise to a doubling of all single spectral lines. We sought the explanation for this doubling of spectral lines in the supposition that x-ray reflection takes place for the most part from the surface layers of a crystal. Surface reflection is even more pronounced in our case since the crystal surfaces were ground and were not subsequently polished. We can calculate in the case of thin crystals the separations which should exist on the photographic film for rays reflected from opposite surfaces of a crystal.

We shall consider first that the reflecting planes are normal to the surface of the crystal.



FIG. 9

Because of the method of clamping the crystal both its inner and outer surfaces are given curvatures with centers at 0. The focal cylinders for the two crystal surfaces are not, however, coincident since from the geometry of the focussing effect the focal cylinder is tangent to the crystalline lamina and passes through its center of curvature. The intersections of the focal cylinders with a plane perpendicular to the axis are shown in Figure 9. By construction the photographic film is constrained to lie on the surface represented by the full circle in Figure 9 with the result that x-rays reflected near the outer surface will not be focussed as sharply as those reflected in the neighborhood of the inner surface.

The separation on the photographic film between rays reflected from the outer and inner surfaces may be simply calculated. When the Bragg angle  $\Theta$  is not too large we may approximate DE (Figure 9) by BC. To the same order of approximation BC = t tan  $\Theta$ , where t is the thickness of the crystal, so that we may write

linear separation =  $\int = t \tan \Theta$  (13) It is easily seen that if the reflecting planes are not normal to the surface of the crystal but make a small angle  $\epsilon$  with the normal then we must replace  $\Theta$  in equation 13 by  $u = |\Theta \pm \epsilon|$  with the general result

$$\int = t \tan u \tag{14}$$

The actual separations follow, within the limits of observational error, equation 14. The value of  $\epsilon$  for the OOl planes was in our case 2°41'. The separations in the third, sixth and ninth orders for Mo K $\propto$ , are tabulated below.

## 001 Third Order

	$u = 8^{\circ} 40^{\circ}$	$u = 14^{\circ}2^{\circ}$
	not resolved	.0053 cm. .0054 .0043 .0036
Nean (meas.)	4 at	.0047 cm.
Calculated	.0032 cm.	.0053 cm.

### 001 Sixth Order

	$u = 20^{\circ} 30^{\circ}$	$u = 25^{\circ} 52^{\circ}$
		al an
	.00828 cm.	.01010 cm.
	.00789	.01000
	.00848	.01029
	.00787	.01017
	.00894	.01191
	.00848	.01161
	.00926	.01139
Mean (meas.)	.0085 cm.	.0108 cm.
Calculated	.0079 cm.	.0102 cm.

## 001 Ninth Order

	$u = 33^{\circ} 31^{\circ}$	u = 38°53'
×		
	.0139 cm.	.0157 cm.
	.0144	.0161
	.0151	.0160
3	.0137	.0163
	.0142	.0157
	.0151	.0182
	.0158	.0167
	.0149	.0176
о. Ж	.0143	.0177
Mean (meas.)	.0146 cm.	.0167 cm.
Calculated	.0139 cm.	.0169 cm.



X 9



001 SIXTH ORDER X 9



001 NINTH ORDER X 9

## PLATE FOUR

On Plate Four are reproductions of spectral photographs in the third, sixth and ninth orders which have been enlarged to show the doubling of the lines.

The second of the two effects which we noted and analyzed is that of the vertical separation between lines which are due to reflections from opposite sides of the same set of atomic planes. Reflecting from opposite sides of a set of planes is a useful procedure in determining grating constants and it was in the course of such work that we found that our lines were displaced vertically with respect to each other. A glance at Plate Five will make our meaning clear. Where the ends of the lines are visible it is apparent that the lines reflected from one side of the atomic planes are shifted down from the horizontal center line while those reflected from the other side of the atomic planes are shifted up.

It is possible to account for this effect by assuming that the reflecting planes instead of being parallel to the axis of the spectrograph (axis of curvature of the crystal) make a small angle  $\prec$  with this axis or in other words that the normals to the reflecting planes make a small angle  $\prec$  with a plane perpendicular to the axis of the spectrograph. Consider a set of cartesian axes with origin at the center of the crystal surface. Let the XY plane be

perpendicular to the axis of the spectrograph with the Y axis normal to the crystal surface and passing through its center of curvature. In this system of coordinates the normal to the reflecting plane at the center of the crystal will make an angle  $\prec$  with the X axis.

As an approximation we may consider a single ray incident in the XY plane and meeting the crystal at the origin of coordinates. We wish to calculate the angular amount by which the reflected ray deviates from the XY plane. Let  $\mathcal{S}$  be the angle between the incident ray and the negative X axis. The reflected ray will make general angles  $\mathcal{F}, \mathcal{S}, \mathcal{E}$  with the three axes.





The approximations following eqn. (16) are not good enough, and lead in eqn (18) to an erroneous result, as will be seen if the case of  $\theta = 90^{\circ}$  is considered. The following work has been done to clear this up.

From Fig. 10-a cor (90°-0) = sin & = cors cord, and cord (180°-20) = - cord = corx cord (5+4). Eliminating B between these two equations will give us a relation between 4 and 0, a, X. Substitution of this new equation in eqn. (16) will lead to the correct result. (All angles are taken positive, as in the fig (10-a)) The expression for cor4 becomes

 $\cos \psi = -\frac{\cos 2\theta \sin \theta}{\cos x \cos \alpha} + \left[1 - \frac{\sin^2 \theta}{\cos^2 \alpha}\right]^2 \left[1 - \frac{\cos^2 2\theta}{\cos^2 x}\right]^2$ 

After substituting this into eqn (16) we have, rigorously:

sind = -coo 20 sind + coo sin 20 [1 - sin 2]<sup>1/2</sup>[1 - sin 2]<sup>1/2</sup> + sin x sin x  
Retaining only 2nd order terms, expanding, we have:  
sin 0 = - coo 20 sin 0 + coo 0 sin 20 - coo sin 20 [
$$\frac{x^2}{2}$$
 +  $\frac{\chi^2}{2}$ ] + dx  
Solving for a we find:

Fig 10-a

0

Russell Yost dr 9/19/40

29-a

Direction cosines of incident ray

-  $\cos\beta$ ,  $\sin\beta$ , 0.

Direction cosines of normal to reflecting plane

-  $\cos \alpha$ , 0,  $\sin \alpha$ .

Direction cosines of reflected ray.

cos 8, cos 8, cos €.

The angle between the reflected ray and the normal to the crystal plane is 90° - 0 where 0 is the Bragg angle.

 $\therefore \cos (90^{\circ} - \Theta) = -\cos \propto \cos 7 + \sin \propto \cos 6$  $\sin \Theta = -\cos \propto \cos 7 + \sin \propto \cos 6$ (15)

If we denote by X the angle between the reflected ray and the XY plane and by  $\psi$  the angle between the projection of the reflected ray on the XY plane and the X axis, we can write

 $\cos \delta = \cos x \cos \psi$ 

so that equation 15 becomes

 $\sin \theta = -\cos \alpha \cos \chi \cos \psi + \sin \alpha \sin \chi$  (16) See inserted sheet. To a first approximation

 $\cos x = 1$   $\psi = 90^\circ + \Theta$   $\cos \psi = -\sin \Theta$ 

 $\therefore$  sin  $\theta = \cos \alpha$  sin  $\theta + \sin \alpha$  sin X

 $(1 - \cos \alpha)/\sin \alpha = \sin \chi/\sin \theta$  (17)

$$\tan 1/2 \propto = \sin X / \sin \theta$$
 (18)

Substituting equation 17 in equation 16 we obtain

\_ \_ \_ \_ \_ \_ \_ \_ \_ \_ \_ \_

 $\sin \Theta (1 - 1 + \cos \alpha) = -\cos \alpha \cos x \cos \psi$  $\sin \Theta = -\cos x \cos \psi$  But  $\psi = 90^{\circ} + 0$ ; where  $\Theta$ 'is the Bragg angle as measured (projection on XV plane).

 $\therefore \cos \psi = -\sin \theta'$ 

giving  $\sin \Theta = \cos \chi \sin \Theta'$ 

The vertical separations between the K $\ll$  lines reflected in the first three orders from opposite sides of the basal planes of quartz can be seen in the spectral photographs of Plate Five. Measurements on these separations indicate an error in orientation of 2°52' ( $\propto = 2^{\circ}52^{\circ}$ ). The corrections to be applied to the measured Bragg angle are in this case small. In the sixth order cos X = .99995 giving

sin 0 = (.99995) sin 0'

In the ninth order the correction is slightly larger.

If the angle  $\prec$  is large or if the focal spot of the x-ray tube is not in the plane passing through the center of the crystal and perpendicular to its axis of curvature then the resulting lines are not parallel to the axis of the spectrograph and are somewhat diffuse.

### Determination of the Grating Constant of Quartz

In order to test the precision with which measurements can be made by means of a curved crystal spectrograph we undertook a determination of the interplanar distance for the basal planes of quartz. To determine this distance it is, of course, necessary to



OOI THIRD ORDER



001 SIXTH ORDER



OOI NINTH ORDER

## PLATE FIVE

X

measure the Bragg angle for reflections from these planes.

The Bragg angle was obtained by measuring the separation between the Ka lines of Molybdenum reflected from opposite sides of the basal planes. This separation gives twice the Bragg angle or 20. In order to reduce the errors incident to measuring on a photographic film the film holder was turned between exposures from opposite sides of the atomic planes so that the lines on the developed film were separated by only a millimeter or two. The angle through which the film holder was turned was read on the divided circle and vernier. The error in reading the vernier was minimized by setting on predetermined angles. Because of the greater precision in setting than in reading a vernier it is believed that in no case was an error greater than 30" made in determining the angle through which the film holder was turned.

This procedure of turning the film holder through the greater portion of the angle to be measured has the tremendous advantage that it becomes unnecessary to know with a great deal of precision the radius of curvature of the crystal but only that the radius of curvature of the crystal is twice as large as that of the film. This last condition can be accurately realized in the construction of the instrument.

Because of the crystal structure of quartz the first order of reflection which appears is the

third, the second is the sixth, the third is the ninth, and so on. Measurements of the interplanar distance were made in the third, sixth and ninth orders with the following results:

Third Order	Sixth Order	Ninth Order
d 5.3922 Å 5.3922 5.3949 5.3946 5.3940 5.3922 5.3958	d 5.3922 Å 5.3928 5.3928 5.3915 5.3909 5.3928 5.3928 5.3935	d 5.39244 Å 5.39217 5.39181 5.39286 5.39244 5.39217 5.39334
5.3946 Mean 5.3938 Å	Nean 5.3924 Å	5.39253 5.39199 Mean
		5.39235 A

The average deviation from the mean in the case of the third order is .022%, in the sixth order .013% and in the ninth order .0056%. After making corrections for the fact that the atomic planes are tilted away from the axis of the spectrograph by  $2^{\circ}52^{\circ}$  the results in the three orders are

Third Order d = 5.3938 Å Sixth Order d = 5.3927 Å Ninth Order d = 5.3927 Å The weighted mean of these re-

The weighted mean of these results is

d = 5.3929 A

Because of the procedure of turning the film

holder through the greater part of the angle to be measured all errors due to stretching of the film, reading of the comparator and inaccuracies in our knowledge of the exact radius of curvature of the crystal have been reduced to insignificance. By far the greatest error is made in setting and reading the vernier on the film holder. This error, in our work, should not exceed 30" in any single determination of the Bragg angle. The error in d, therefore, in the sixth order should not exceed 2 parts in 5000 and in the ninth order should not exceed 1 part in 5000.

We can, then, with great certainty state our result as

 $d = 5.393 \pm .002 \text{ Å}$ 

WycKoff<sup>('')</sup> on the basis of the results of a number of observers gives the value of this interplanar distance as 5.393 Å with the last figure doubtful. One of the most recent determinations is that of Harrington<sup>('')</sup> who finds d = 5.393  $\pm$  .006 Å.

#### Conclusions

Our work with the curved crystal spectrograph has led us to believe that it is an instrument of great versatility in x-ray spectroscopic work. When equipped with a precision divided circle it may be used for accurate determinations of grating constants and wavelengths. Its luminosity also measures up to our expectations - we have stated above that we were able to obtain in 10 hours a Compton photograph which would require an exposure of about 300 hours even on the multicrystal spectrograph.

One serious drawback appears to stand in the way of a general use of the curved crystal spectrograph. Recent preliminary tests by myself on uranium K radiation and by Mr. Maxwell Kelch on tungsten K radiation seem to indicate that this type of spectrograph of ordinary dimensions cannot profitably be used with hard x-rays. We feel that the trouble lies in the inability to use thick enough crystals to scatter selectively an appreciable fraction of the incident radiation.

In conclusion I wish to thank Dr Jesse W. M. DuMond at whose suggestion this work was begun and who has advised and helped me on the many problems which were encountered during its progress.