THE CONSTRUCTION OF A CURVED CRYSTAL X-RAY SPECTROMETER

A Thesis

Submitted in Partial Fulfilment of the Requirements

for the Degree of

Master of Science

by

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SUMMARY

A twelve inch curved crystal x-ray spectrometer was constructed. With it the x-ray spectrum of radioactive sources could be scanned with a scintillation counter or recorded on a photographic film.

The instrument has a relative line width of about 0.65% and a transmission of around 1×10^{-7} at 20 Kev.

ACKNOWLEDGEMENTS

The author wishes to express his appreciation of Prof. J.S.Foster,F.R.S. Director of the Radiation Laboratory, for suggesting the problem and for his help in carrying it to a successful conclusion. Thanks are also due to Dr. W.M. Martin and Dr. R.E.Bell for their help. The cooperation of the laboratory staff, especially Mr. Doig and Mr. Belley, is gratefully acknowledged.

I INTRODUCTION AND HISTORY

The problem involved in this thesis is the construction of a curved crystal x-ray spectrometer which would be useful in the study of radioactive decay of elements produced by bombardment in a cyclotron.

This type of x-ray spectrometer is the latest step in the evolution of energy measurement using crystal diffraction. Starting with the famous work of von Laue this method was further developed by Bragg in his single and double crystal spectrometers. During this time de Broglie,[1] Gouy[2] and Darbord[3] suggested the possibility of curving a crystal to obtain focusing. But the first workable idea was advanced by Dumond and Kirkpatrick[4] and the first two working models were built independently by Johann[5] and Cauchois[6] who used the same principle, in slightly different form, of focusing by a curved crystal.

All instruments built since then have used the same basic principle, although the mechanics of applying it have followed different and ingenious paths. A list of some of these methods is given in the Bibliography[7] and a system developed in this laboratory is outlined in Appendix I. This principle is explained in section 2. The advantage of the focusing effect is that greater intensity can be obtained than by simply reflecting a parallel beam.

The disadvantage of this machine as applied to nuclear physics is its low transmission. Sources of the order of 100 millicuries are required to get good results for high energy gamma-rays. While the low energy gamma-rays and x-rays can be measured with an activity of the order of a microcurie.

The instrument has several advantages over the other means of measuring gamma-rays:

(a) The energy of the gamma-ray is measured directly as compared with the indirect means of the beta-ray spectrometers.

(b) Its resolution is in general much higher than other types of gamma-ray detectors.

(c) Sources are very easily mounted in the instrument.

The main factor to take into account is transmission when using this type of instrument, with sources prepared by cyclotron bombardment. Since the transmission varies with the square of the wavelength, the spectrometer is only useable for relatively long wavelengths. Therefore, the instrument is most suitable for measurements in the x-ray region and this fits in with the study of the electron capture activities produced by the cyclotron. Thus, the instrument was built to study the x—rays and low energy gamma-rays produced in radioactive decay. It was designed to have sufficient resolution to resolve the x-rays of two neighbouring elements and high enough transmission to be useful with cyclotron produced sources.

A second factor, that entered into its design, was versatility. Using the principle as explained in section two, the instrument can be set up in three ways. From diagram IB it can be seen that a source placed at EFG will cause x—rays to be brought to a focus on the circle DSAB at an angle with respect to the crystal planes. Equally a source placed at D on the focal circle will have a virtual image at S' and a counter placed at EFG will detect a line whenever the point D makes an angle θ corresponding to a Bragg angle for its radiation.

Therefore the instrument was set up to use a film detector on the focal circle, a scintillation counter on the focal circle, or a scintillation counter at EFG. The film is useful for getting low energy x-rays or no page 3

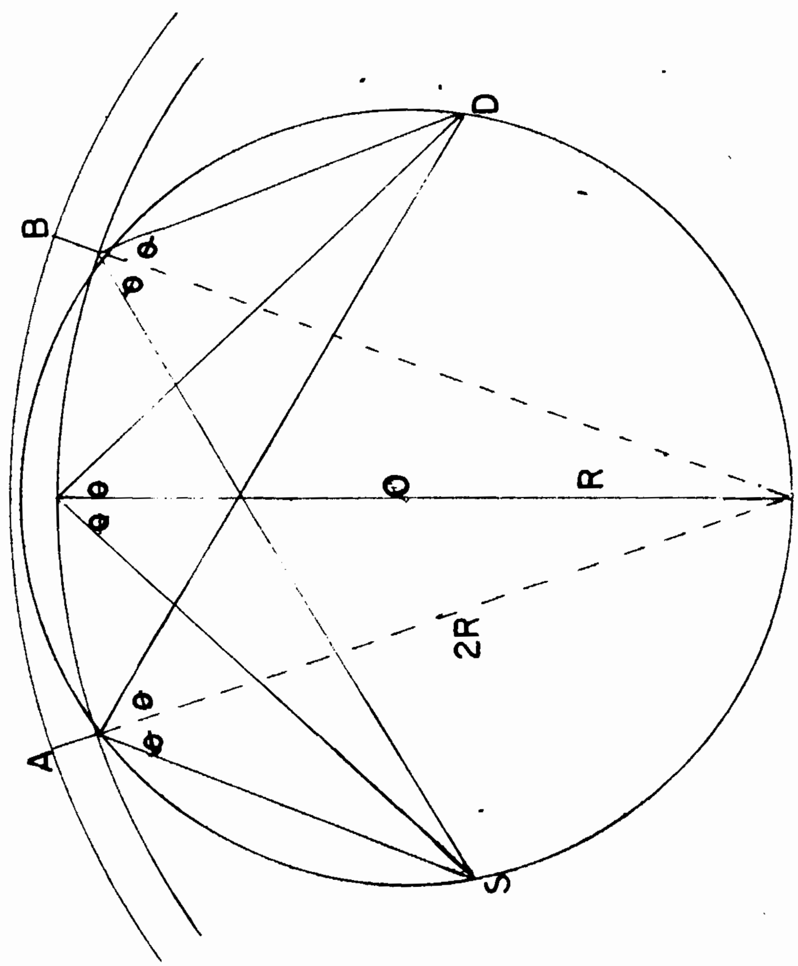
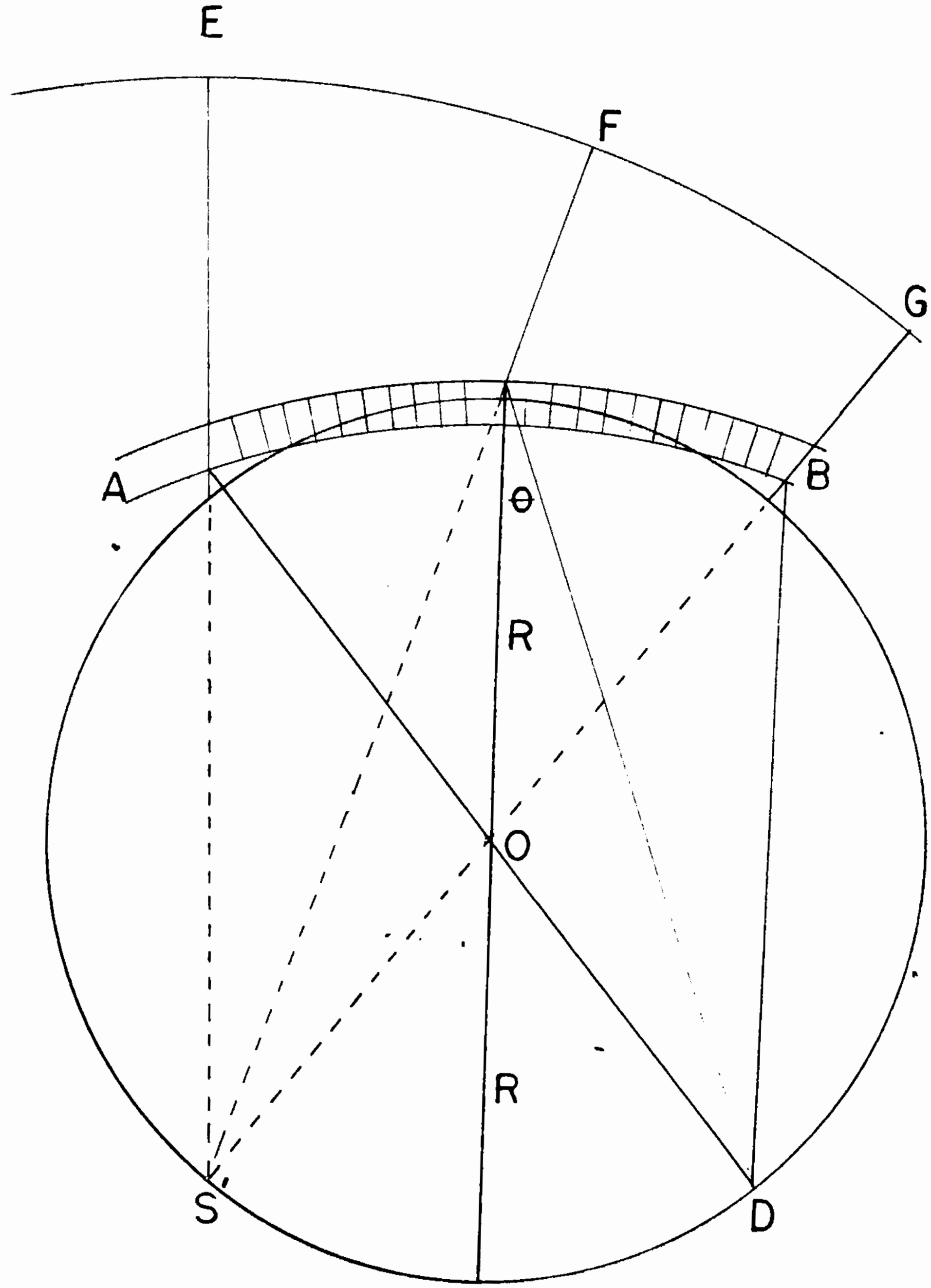


DIAGRAM IA

DIAGRAM. 1B



integrating the radiation to get weak lines. The scintillation counter on the focal circle requires an extended source and therefore the source is easy to mount. A source placed on the focal circle has the advantage of higher transmission.

In summary then the instrument is limited by its low transmission. It has enough resolution to separate the K alpha lines of two neighbouring elements, and is useful in the study of X-rays and low energy gamma-rays with emphasis on decay by electron capture.

II THEORY

This instrument works on a principle similar to the Rowland Grating. From diagram 1A it is seen that if a crystal AB is bent with a radius 2R, a source placed at S on the focal circle of radius R will have its radiation diffracted to the point D on the focal circle from all points on the crystal, if the Bragg condition is fulfilled.

From diagram 1B it can be seen that a source placed at D gives a beam which is diffracted from the crystal AB as if it had come from the point S'. That is, it has a virtual image at S'. Or a broad source at EFG has its radiation brought to a focus at D.

The first system, as in diagram 1A is only used for long wavelengths. The second system can be used for higher energies up to about 5 Mev. As was mentioned in the introduction the second method can be used several ways. If the source is placed at EFG, we can either place a photographic plate on the focal circle or the focal circle can be scanned with a counter. If a source is placed at D, it can be moved along the focal circle and the peaks detected with a counter at EFG.

The photographic plate method allows a large portion of the spectrum to be surveyed at one time. The two counter methods give increased detecting efficiency and are therefore more useful with cyclotron produced targets. Source preparation is easy for a distributed source while the line source increases the solid angle and hence the transmission of the instrument.

The mathematics of this instrument is worked out in detail by Miss Cauchois.[(6)] It is obvious the line will be formed at an angle relative to the geometric centre according to the Bragg relation

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

1

This angle is changed according to the position of the planes of the crystal relative to the surface of the mica.

If the planes are at an angle α to the centre line R, the focus will be at an angle $\alpha \pm \theta$.

The linear dispersion of the instrument is $\frac{ds}{d\lambda}$.

Since

$$s \doteq 2R\sin\theta \qquad 2$$

and from formula 1 the dispersion is

$$\frac{ds}{d\lambda} = \frac{nR}{d} \qquad 3$$

(See diagram 2).

The line width is dependent on three factors: (a) the aperture of the crystal, (b) the thickness of the crystal and (c) the perfection of the crystal structure.

The geometrical line width was worked out by Miss Cauchois to be

$$L = (E + \frac{o^2}{8R}) \tan u \qquad 4$$

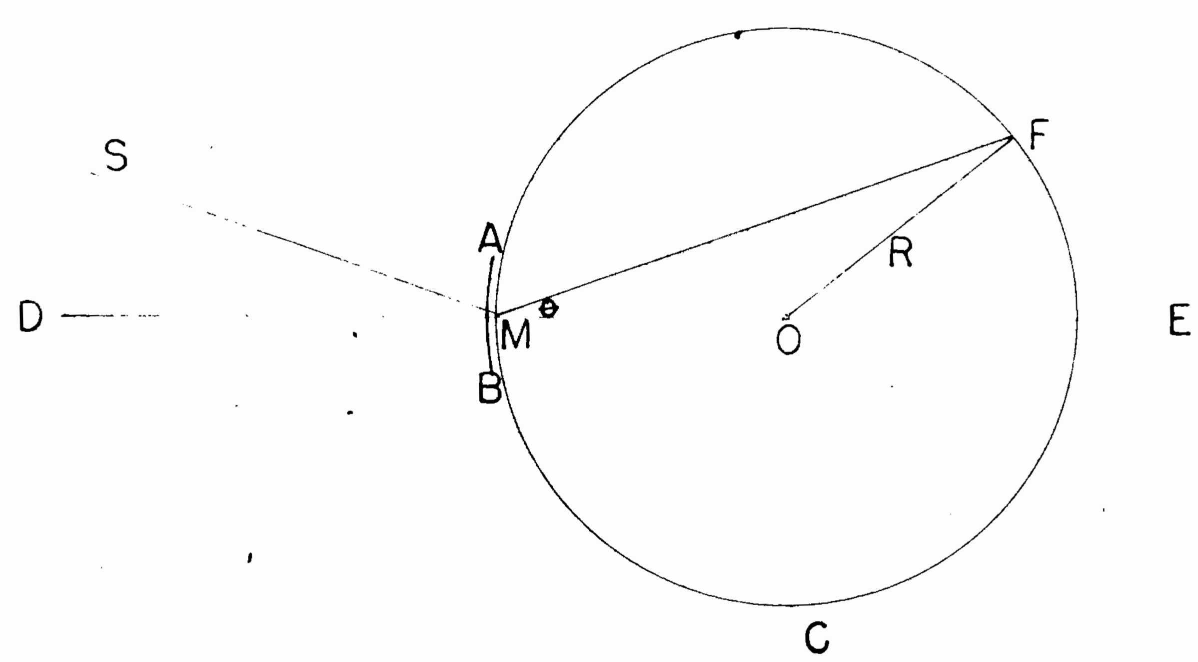
where E is the thickness of the crystal, o is the linear opening of the crystal, and

$$u = \alpha + \theta \qquad 5$$

This formula neglects the effect of the crystal structure which is a very complex problem, even for an unstressed crystal.

The position of the source for the best background condition was investigated by P. Marmier, J. P. Blaser, P. Preiswerk and S. Scherrer (8). They found this was best when the detector is 3 times as far from the crystal as the source.

DIAGRAM 2



III THE CONSTRUCTION OF THE APPARATUS

(a) General Description

The instrument was made as flexible as possible. The general idea was to have a slit moving on the focal circle, a crystal clamp fixed on the focal circle, and a broad source or detector which moves so as to make the same angle with the crystal planes as the slit.

This is illustrated in diagram 2. The slit is at F moving on the focal circle MCF. The crystal clamp is at AB. The broad source or detector, (hereafter called the source), must rotate about the centre of the crystal clamp aperture, point M, so as to keep the angles DMS and OMF equal. This will be true if the crystal planes used are normal to the surface of the crystal clamp. If they are not normal, the angle FMO must be the angle between the slit and the crystal planes used.

For the purpose of a description of the apparatus it is convenient to divide it into three parts. These parts are the slit (or photographic film holder), the mica clamp and the source. The requirements to be filled by these are discussed in the following sections.

Crystal clamp

The crystal clamp is the most important feature of the instrument as its accuracy together with the quality of the mica crystal determine the sharpness of the focus of the instrument. The position of the mica clamp must be known accurately since this determines the position of the focal circle.

The slit

The slit limits the radiation passing through it and must therefore be made of a material which strongly absorbs x-rays. Its width must be variable so as to allow differing amounts of a spectral line to be examined. Further, rotation about a line drawn perpendicular to the generator of the focal circle must be provided. This is necessary since the planes of the mica crystal are not easily set in the clamp with an accuracy better than two degrees relative to this generator. The slit must move so as to remain accurately on the focal circle. Finally, since it will be in the vicinity of high energy gamma-rays it must have a large amount of shielding around it. If a film or plate holder is used in place of a slit, it must have the curvature of the focal circle and it must be placed accurately on the focal circle.

<u>The source</u>

The source is the least critical part of the instrument. If its centre moves away from the Bragg angle, at which it is supposed to be located, by even a relatively large amount, most of the source will still be radiating onto the mica crystal and therefore it will be at the Bragg angle for some part of the crystal. The reader is reminded that the term source is here used to represent either the source or the detector, whichever is located at position S of diagram 2. The main requirement is to keep its centre at about the Bragg angle so that the detected intensity will be maximum. It is also necessary to vary the source radially with respect to the crystal in order to get a full range of energies. Finally, it will require a large amount of shielding, since intense radioactive sources will increase the background.

(b) <u>Details of Construction</u>

These requirements were fulfilled in the following ways.

<u>The crystal clamp</u>

The crystal clamps were cut to a radius of twelve inches and then ground to a good surface. This surface was coated with 0.01" of chromium. It was then lapped to a fine finish by the Optics Laboratory of the National Research Council.

The crystal clamps were bolted on a base plate and accurately located relative to the centre of the focal circle.

The slit

The one inch slit was made of lead bonded to a brass backing. It was then milled to as sharp an edge as would be taken by the lead. These lead knife edges were then mounted in a groove, and held in place by screws. The screws could be loosened to allow adjustment of the slit width and position.

In order to get rotation of the slit, it was decided to fasten it solidly to a cylindrical lead shield in which the counter or radioactive source could be placed. Then to rotate the slit, the whole piece of apparatus was rotated. This allowed the use of a thin crystal counter whose orientation relative to the slit did not need to be changed once it was adjusted. Also, if a line radioactive source was used, it could be repeatedly keyed into the right position. This cylinder was fitted into a plate with a cylindrical groove and was then held in place by two U-shaped clamps. One of the clamps was fitted with two screws working in opposite directions against a projection fastened to the lead, in order to give the required rotational adjustment. This setup is shown in plate 1.

The motion cf the slit had to satisfy two conditions. It must move along the focal circle, and it must be pointed at the centre of the crystal at all times since shielding must be placed in front of it.

The slit was driven by an arm from O to F in diagram 2. This is No. 25 in diagram 3.The slit and castle were placed on a plate (No. 19 of diagram 3) which was pivoted at F and M of diagram 2. Since the distance FM is not constant the sliding pivot at M allows the plate to slide as it rotates. The sliding pivot is provided by a fork (No. 21) moving along the axle (No. 20). The arm (No. 25) is pivoted about the axle (No. 23) at the

1. Aluminum Base
2. Source Drive Gear
3. Driven Source Gear
4. Lower Source Shaft Bearing and Bearing Block
5. Lower Main Bearing & Bearing Mount
6. Upper Source Shaft Bearing & Bearing Block
7. Main Gear
8. Slit Driving Arm
9. Source Carriage
10. Adjustable Source Mount
11. Radial Source Adjusting Screw
12. Back of Source Holder
13. Collimator Holders
14. Collimators
15. Top Source Holer
16. Bottom Source Holder
17. Mica Clamp
18. Collimator Adjust
19. Slit Carriage
20. Source Shaft
21. Slit Carriage Guide
22. Base Plate
23. Main Shaft
24. Slit Carriage Pivot
25. Slit Carriage Driving Arm
26. Radial Slit Adjust
27. Slit Mount
28. Slit Rack
29. Counter Shield
30. Slit
31. Slit Horizontal Rotation Adjust
32. Counter Shield Clamps
33. Counter
34. Slit Carriage Legs

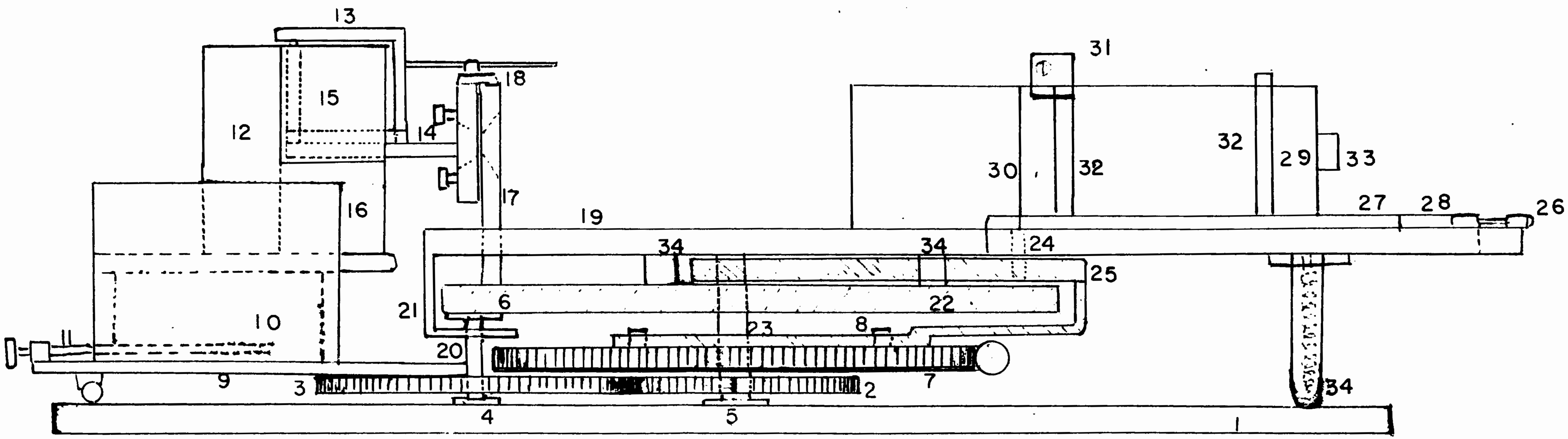


DIAGRAM 3

centre of the focal circle. The pivot on the focal circle is provided by a bearing whose centre is 6" $\pm$ 0.005" from the axle (No. 23). The plate holding the slit is then pivoted by an axle through this bearing.

The slit carriage (No. 19) had to carry the rather large lead shield. This meant that it projected beyond the edge of the base plate (No. 22). At the same time, this large weight had to move accurately enough to keep the slit on the focal circle. To prevent any play of the slit carriage, a good friction contact between the carriage and the base plate was necessary. This was supplied by three iron legs fastened to the carriage. To move this large weight as easily as possible and to keep as much warping out of the plate as possible, two legs (No. 34) were put under the projecting end of the carriage. These legs rolled on steel balls. To allow for vertical play the balls were spring loaded and the tension on the balls was adjustable.

When the apparatus is in operation, the slit (No. 30) and the shield (No. 29) and slit mount (No. 27) must be adjustable relative to the slit carriage, in order to place the slit directly over the pivot (No. 29). If the slit is not over this pivot, it will move off the focal circle as the arm (No. 25) rotates. To make this adjustment, the slit mount was placed in a rack (No. 28). The rack and slit assembly could be adjusted radially along the slit carriage (No. 19) by screw (No. 26). The slit mount could be adjusted across the rack, transverse to the slit carriage, to give the two dimensional motion relative to the carriage required to adjust the slit directly over the pivot (No. 24).

The drive of the arm (No. 25) is used to measure the Bragg angle. This angle must be measured to within one minute. From diagram 2 it is seen that the angle FOE is twice the angle FME for all positions of F. This means a reduction of two by driving the arm (No. 25) of diagram 2. Therefore, to provide a slit rotation of say one degree for a complete rotation of the control dial, a gear reduction ratio of 180 was necessary. This was obtained by two reductions, a worm gear and worm system with a reduction of 100:1 driven by a helical gear system giving a reduction of 1.8:1. This gives the total required reduction from the control dial to the slit of 360:1. The control dial was divided into 120 divisions. Therefore, one division corresponds to a slit rotation of half a minute.

The shielding around the slit was made in three pieces. The main piece is a lead tube eight inches long and four inches in diameter, with a two inch diameter hole through the centre of it. The slit is fastened to one end of this tube. The hole in the centre of this tube may contain either a radioactive source or a counter. This arrangement is No. 29 in diagram 3. Two blocks, four inches wide and two inches thick, may be placed in front of the slit. Each block has a groove cut in it one inch wide. The groove makes an angle with the surface of the block, which corresponds to the half angle that the slit subtends at the crystal. These blocks supply good shielding of the slit from radiation leaving the source. They are shown as No. 30 in diagram 3. This means that the slit has at least one inch of lead in all directions, except that of the crystal.

<u>The source</u>

The source is made up of five parts, the drive, the carriage, the mount, the shielding, and the collimators. The drive is done in co-ordination with the slit drive. The main shaft of the source drive must be such as to change the angles SMD and FME at the same rate. This was done by a gear train driven by the main shaft No. 23 diagram 3. Since this angular change is twice that of the source a reduction of two to one was made by means of two spur gears (No. 2 and No. 3 of diagram 3). The shaft (No. 20) of the gear (No. 3) was fastened under the centre of the crystal clamps. A plate (No. 9) was fastened to this gear. The other end of the plate was supported by two rollers. In the centre of the plate a groove was cut and the source mount (No. 10) was keyed into this groove. The source mount is adjustable along the groove by means of a screw fixed to the source carriage. It may also be adjusted in height by means of four screws.

The source shielding and holder are combined. They are made of three pieces of lead. (See Nos. 12, 15 and 16). Pieces No. 12 and No. 16 are rectangular blocks. Piece No. 15 has a groove cut in it to hold the source. The collimators (No. 14) project into this groove. The blocks give two inches of lead shielding around the source.

The action of the collimators is shown in diagram 4. The source is at DN, the mica clamp is at G and the slit at F. It can be seen that with this arrangement the source is completely visible to the slit. The radiation will go directly from the source to the slit swamping out any radiation reflected from the mica. By putting in the collimators KL, SM, and HJ the direct radiation is blocked out at this angle and this allows smaller Bragg angles to be measured.

The collimators have to be fastened at the source and pointed at the virtual image of the slit. The collimators were fastened to one side of a U-shaped clamp (No. 13). These clamps were pinned to the source holder just above the position of the source. Rods attached to the clamps were pivoted above the crystal. The pivots were made adjustable to get the best position of the collimators.

The collimators themselves were silver. The absorption of X-rays in the wavelength region considered was found to be better for silver than for lead and the silver has greater rigidity.

Three collimators are used and, at a distance of four inches from the mica, they allow the minimum Bragg angle to be 2.25° which corresponds

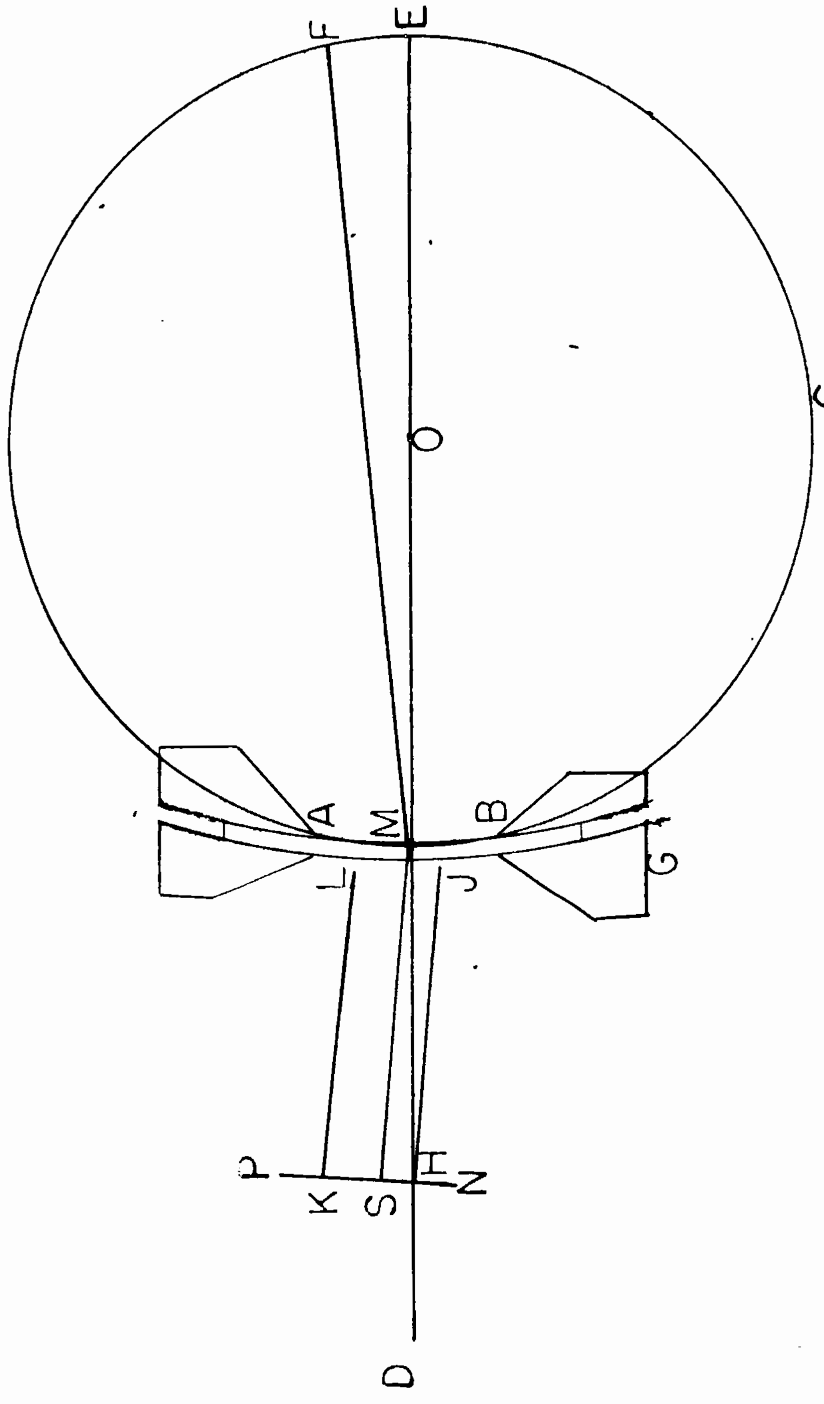


DIAGRAM 4

to an energy of about 60 Kev. Higher energies and smaller angles may be achieved by moving the source further away from the mica. This set up is shown in plate No. 1.

The detector

The instrument has been tested only for the case of a broad source and a line detector. The detector is a wedge shaped crystal of Na I (Tl) held in an aluminum can (see diagram 5).

The crystal is thick enough to stop 99.9% of incident 100 Kev. x-rays. For improved reflectivity, the inside of the can was painted with a mixture of magnesium oxide and water glass.

The aluminum window in front of the NaI was made 1 thousandth of an inch thick. This absorbs 5% of the radiation at an energy of about 15 Kev.

The phototube is an RCA 6199. It is followed by a cathode follower whose circuit is shown in diagram 6. The cathode follower feeds the pulses from the crystal into a single channel pulse height analyser. This instrument allows a window to be set about the peak to be studied, thereby rejecting many background counts.

The output pulses of the pulse height analyzer are counted with a manually operated scaler.

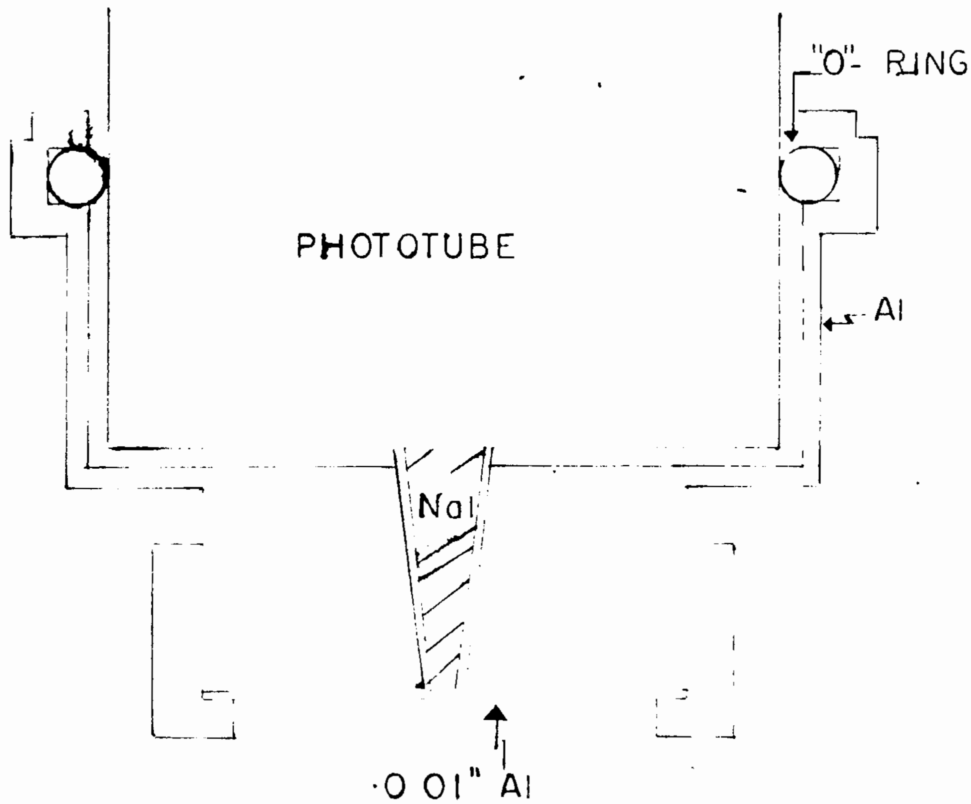


DIAGRAM 5

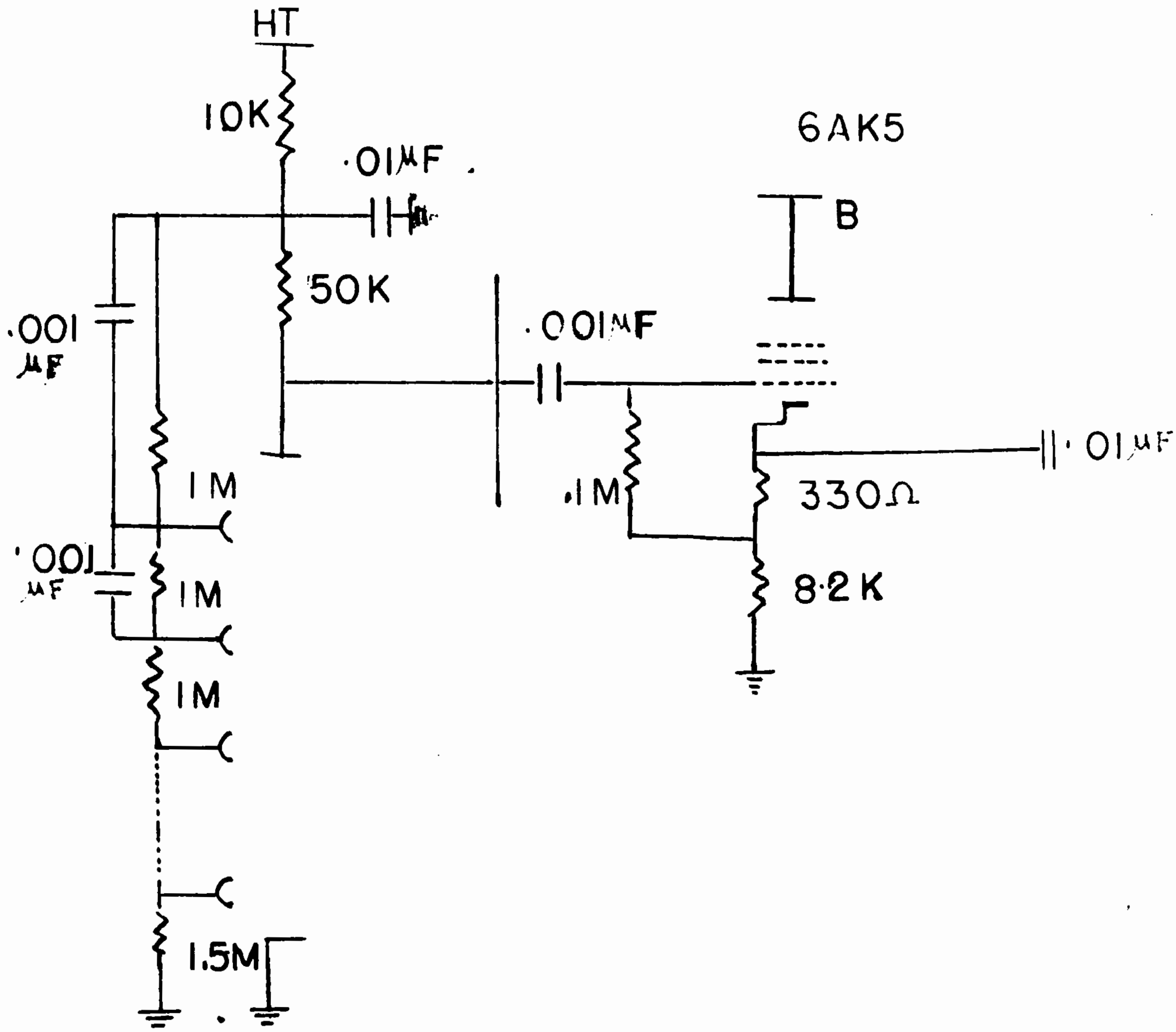

HT
10K
·01μF
6AK5
B
50K
·001
μF
·001μF
1M
·1M
330Ω
·001
μF
1M
8·2K
1M
1.5M
·01μF

DIAGRAM. 6

IV ADJUSTMENT OF THE INSTRUMENT

A mica crystal was used in the instrument. Its crystal structure must be determined in order to place the mica in the blocks at the correct angle. This can be found with relative ease using optical methods. The mica was placed in a polarizing microscope and rotated until extinction was reached. The position of the planes to be used in the spectrometer are then either parallel or perpendicular to this extinction position. This involves four possible positions of the mica in the clamp. These positions were tried until the correct one was found.

Care was taken to ensure that the surface of the mica was flat. This was done by stripping a thin layer off the surface of the piece. Also all dust and small specks of dirt were removed from the clamp and the mica before clamping.

Five adjustments are needed to bring the instrument to focus. The first adjustment is to align the source, the slit pivot and the centre of the crystal at an angle of ten degrees with the axis of the instrument. The other adjustments are, the angle of the slit relative to the generator of the focal cylinder, the position of the collimators, and the two adjustments to the slit for placing it on the focal circle.

The slit was placed on the $K\alpha_1$ line of silver and the angle of the slit was varied until maximum counting rate was reached in a scintillation crystal behind the slit. This position was then checked photographically and the two coincided.

The collimators were aligned by irradiating a rhodium foil with an x-ray machine and placing the slit on the peak due to the $K\alpha_1$ fluorescence. The collimator pivot above the mica was then varied by steps of $\frac{1}{32}$" and the counts recorded. This was repeated for each of the three collimators (Table 1).

TABLE 1

POSITIONING OF THE COLLIMATORS

Position $\frac{1}{32}$" units	Centre Collimator Counts/Min.	Right Collimator Counts/Min.	Left Collimator Counts/Min.
0	2462	2640	1650
1	2496	2656	1580
2	2656	2896	1690
3	3214	2782	1958
4	3600	2800	2004
5	4128	2942	2232
6	4434	3078	2462
7	4218	3058	2464
8	3752	3284	2238
9	2348	3368	
10	3460	3056	
11		2790	
12		2474	

The values in table 1 are plotted in graph 1. It can be seen that the positioning of these collimators is not too critical. A variation of one sixty-fourth of an inch would have only a small effect.

Two adjustments were incorporated to place the slit over the pivot on the focal circle. One of these adjustments moved the slit in a radial direction with respect to the centre of the mica. The other moved the slit at right angles to the first direction. These two variables are called the radial and transverse adjustments respectively. The effect of each adjustment on the resolution and the energy indicated by the instrument were determined.

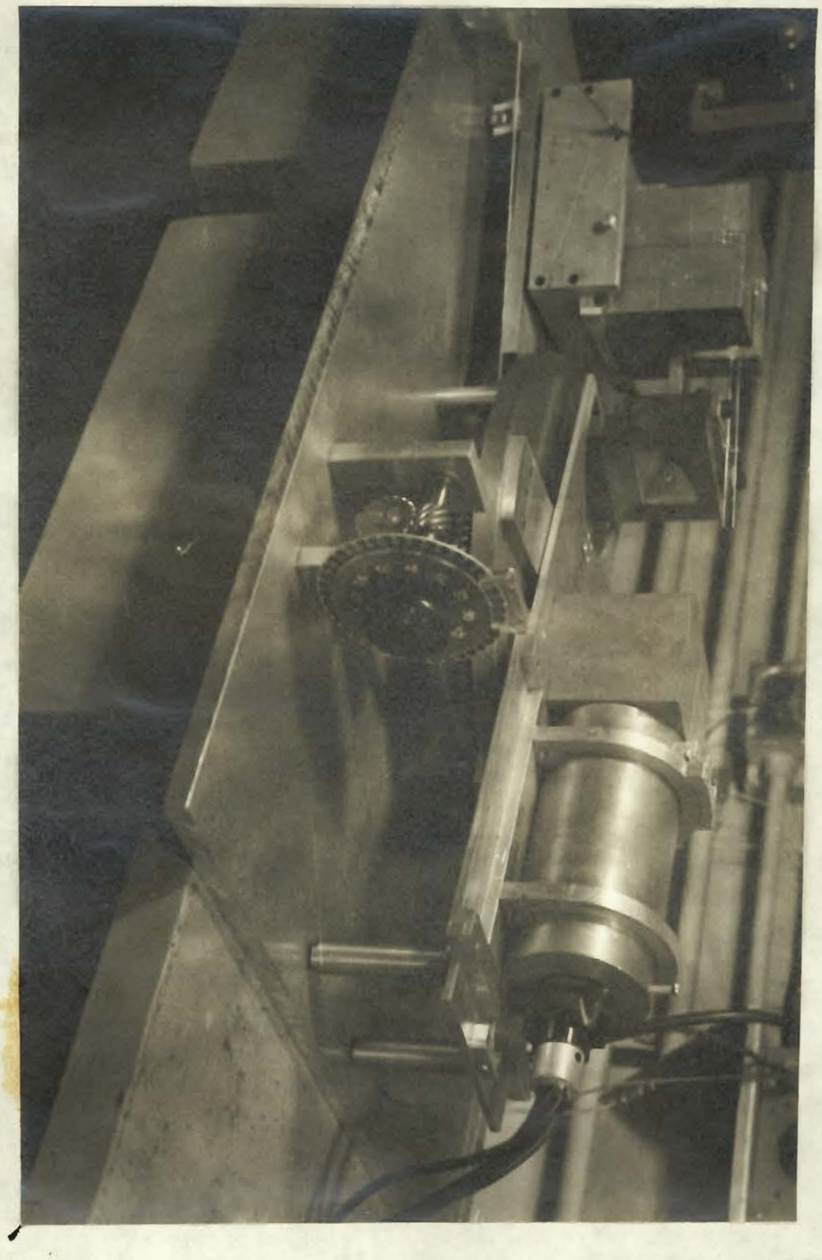
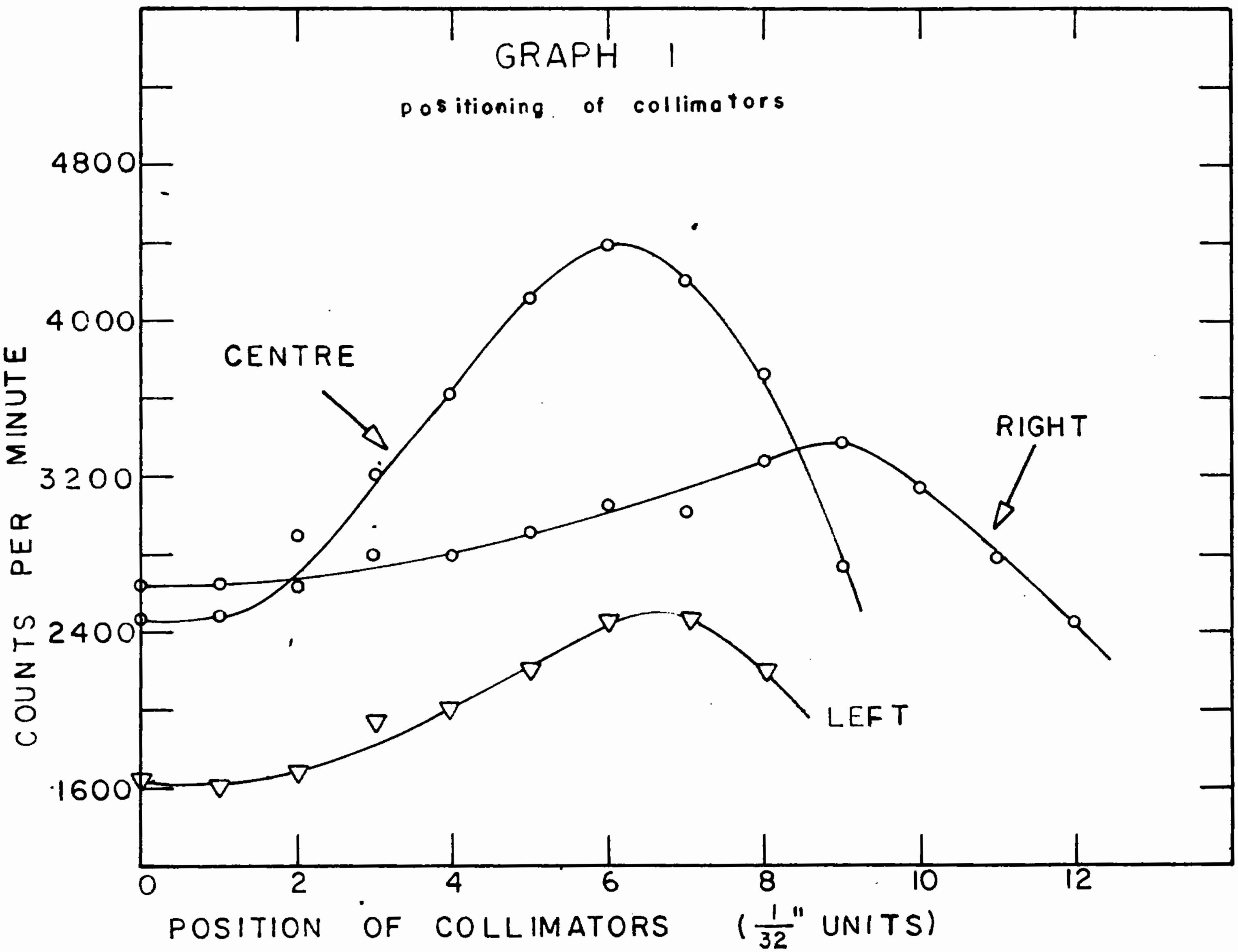


PLATE. I


GRAPH I
positioning of collimators
4800
4000
3200
2400
1600
COUNTS PER MINUTE
CENTRE
RIGHT
LEFT
0
2
4
6
8
10
12
POSITION OF COLLIMATORS ($\frac{1}{32}$" UNITS)

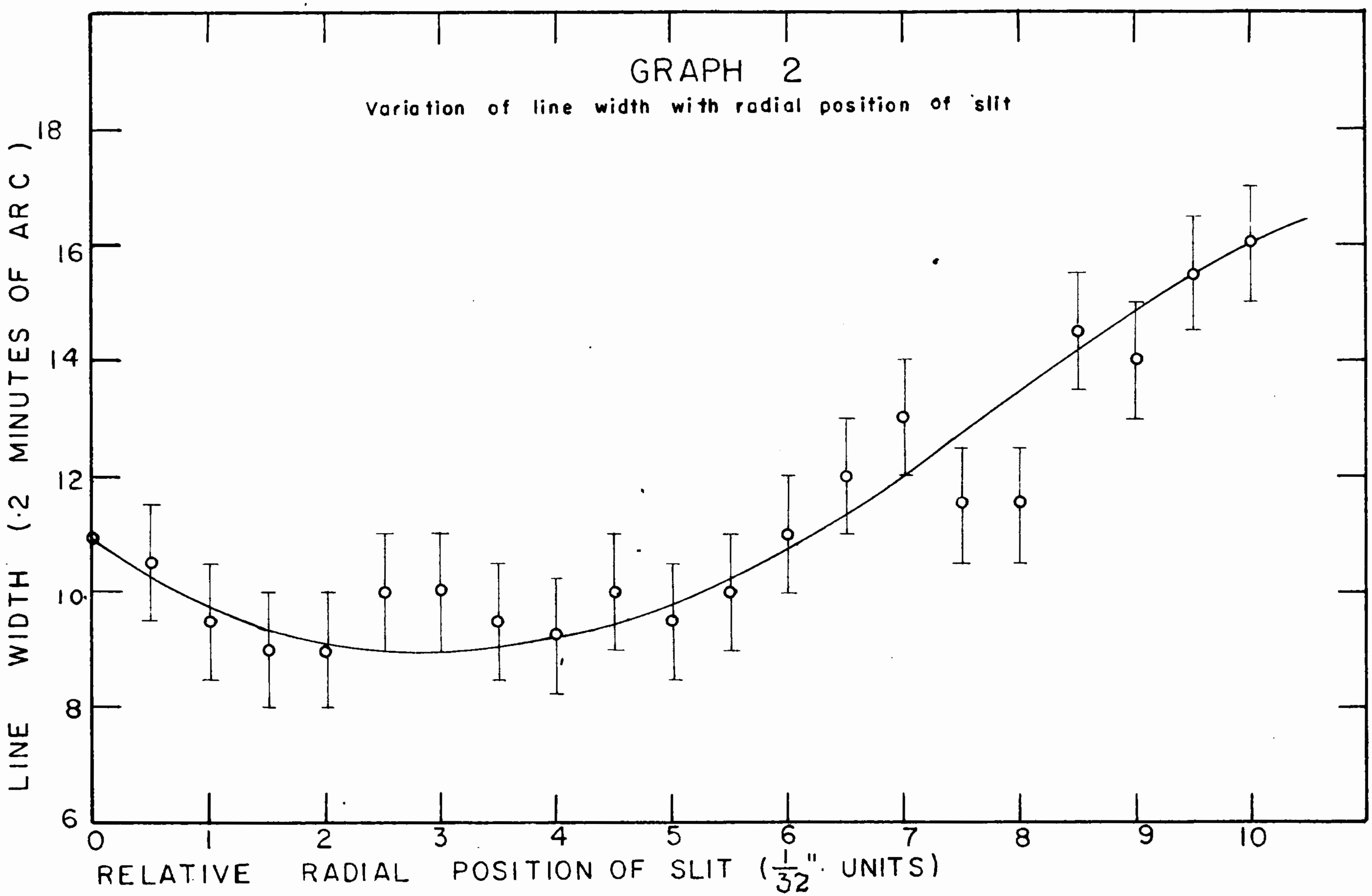
The line width as a function of the radial adjustment was determined by measuring the width at half height of the silver $K\alpha_1$ x-ray line for various radial positions. The $K\alpha_1$ radiation was produced by bombarding a 0.004" silver foil with x-rays of a higher energy from an x-ray tube. The slit was moved through 0.313" at intervals of 1/2 turns on the 10-32 screw of the radial adjustment. The line was scanned at each adjustment with a 0.001" slit. The 1/2 width was plotted in units of 0.2 minutes of arc.

The resulting curve is shown in graph 2. It can be seen that there is a broad minimum between one and six turns. This is a distance of about 0.156". From the geometry of the instrument, the line should be about 50% wider at 6 turns away from the minimum. Graph 2 bears out this expectation.

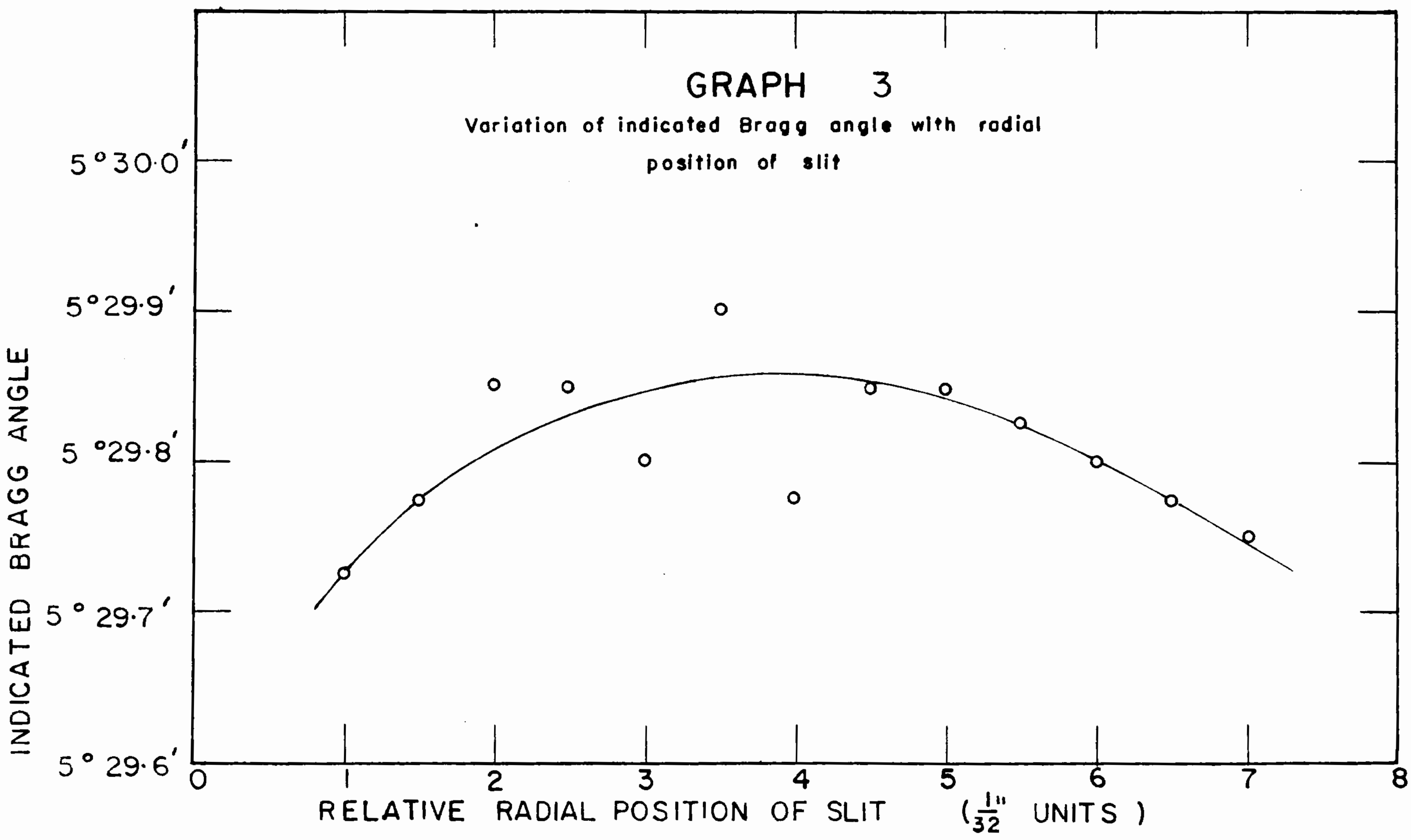
The Bragg angle as indicated by the instrument was plotted as a function of the radial adjustment using a method similar to the previous one. The only difference was the slit width which was opened to 0.002". The result is shown in graph 3. It can be seen that there appears to be a weak extremum near the centre of the variation. This is about the same point as the minimum in the line width curve. Therefore the slit was set at this position.

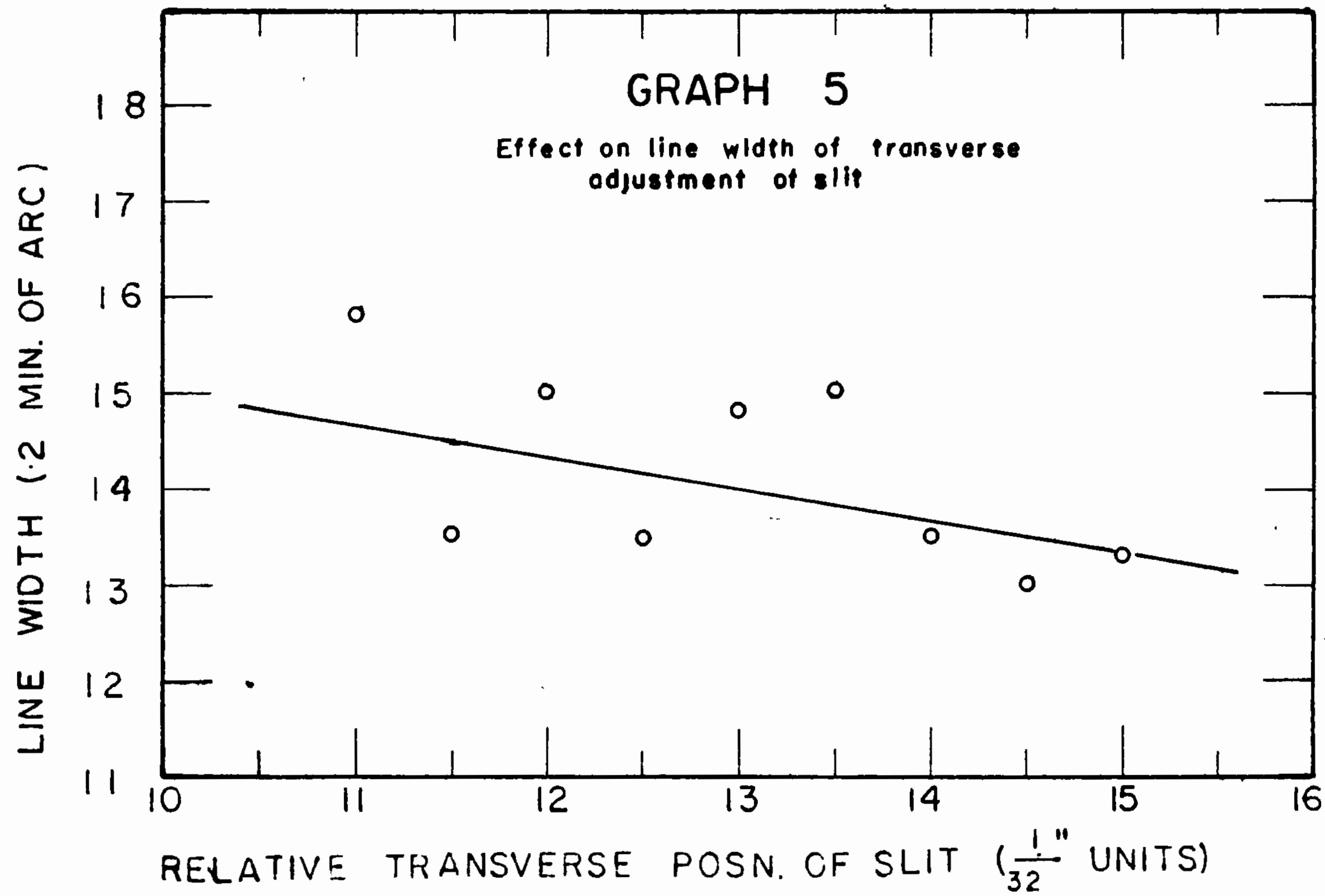
The line width and the Bragg angle as indicated by the instrument for the silver $K\alpha_1$ line were plotted as a function of the transverse adjustment. The results are shown in graphs 4 and 5. It is seen that the Bragg angle displays a small slope while the line width curve remains relatively constant over the whole range.

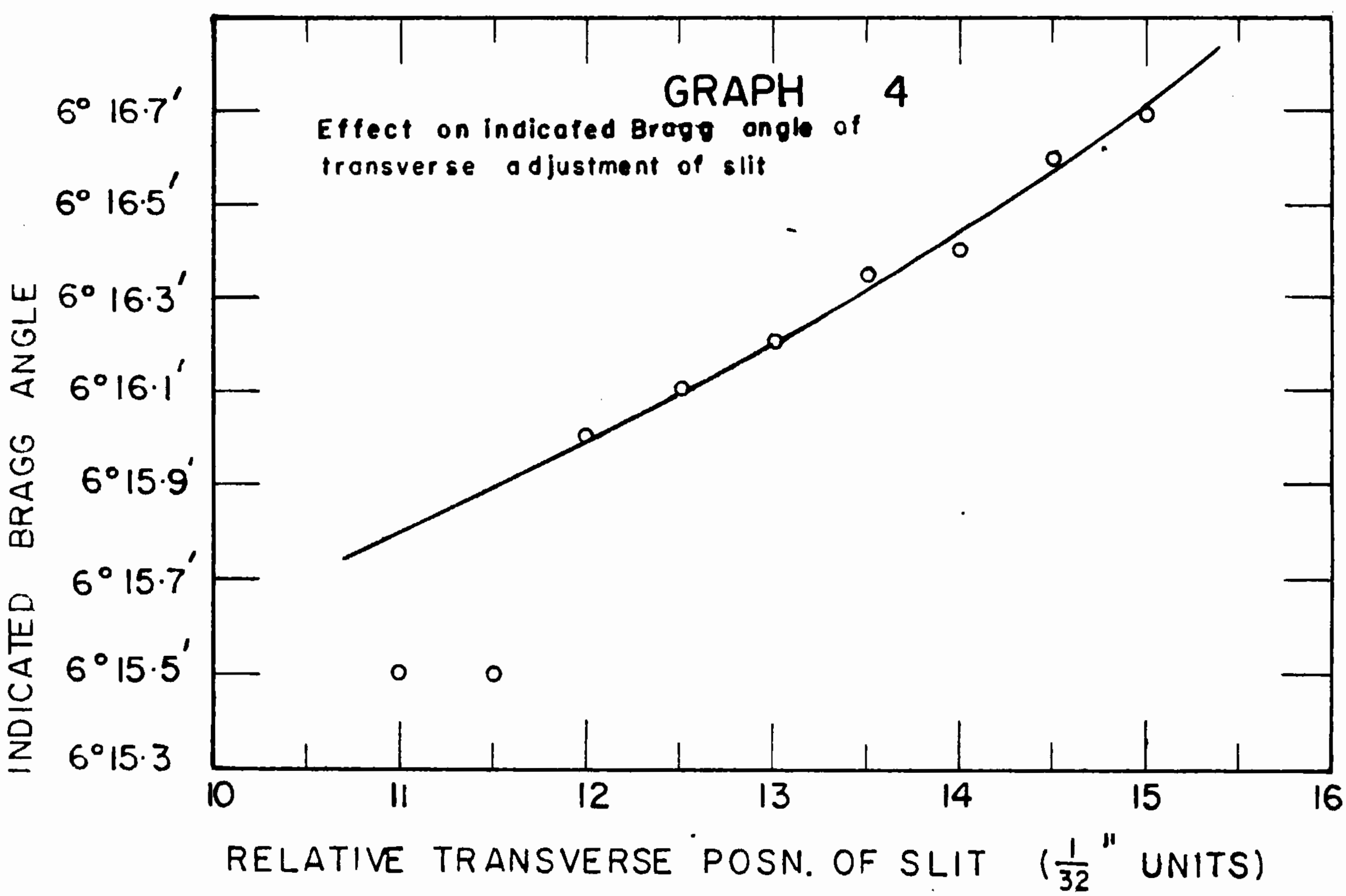
Therefore the instrument is relatively insensitive to all these adjustments. This means that it is easily brought into focus.



GRAPH 2
Variation of line width with radial position of slit
LINE WIDTH (·2 MINUTES OF ARC)
18
16
14
12
10
8
6
0
1
2
3
4
5
6
7
8
9
10
RELATIVE RADIAL POSITION OF SLIT ($\frac{1}{32}$" UNITS)


GRAPH 3
Variation of indicated Bragg angle with radial position of slit
INDICATED BRAGG ANGLE
5°30.0′
5°29.9′
5°29.8′
5°29.7′
5°29.6′
0
1
2
3
4
5
6
7
8
RELATIVE RADIAL POSITION OF SLIT ($\frac{1''}{32}$ UNITS)


GRAPH 5
Effect on line width of transverse adjustment of slit
LINE WIDTH (·2 MIN. OF ARC)
18
17
16
15
14
13
12
11
10
11
12
13
14
15
16
RELATIVE TRANSVERSE POSN. OF SLIT ($\frac{1}{32}$" UNITS)


GRAPH 4
Effect on indicated Bragg angle of transverse adjustment of slit
INDICATED BRAGG ANGLE
6° 16·7′
6° 16·5′
6° 16·3′
6° 16·1′
6° 15·9′
6° 15·7′
6° 15·5′
6° 15·3
10
11
12
13
14
15
16
RELATIVE TRANSVERSE POSN. OF SLIT ($\frac{1}{32}$" UNITS)

V RESULTS

Resolution

The line width was measured for a small source which used only a small area of the crystal and for a large source using the whole aperture of the mica. The line width using the small aperture and a tin target as measured from graph 6 was found to be 0.63%. The line width for a large source was found to be 0.67%. The target for the large source was rhodium and the resultant profile of the line is shown in graph 7. The line width as determined geometrically was found to be about 0.003". This corresponds to a line width of 0.23%. The discrepancy may be explained as due mainly to imperfections in the crystal structure and faults in the surface of the mica and the crystal clamp.

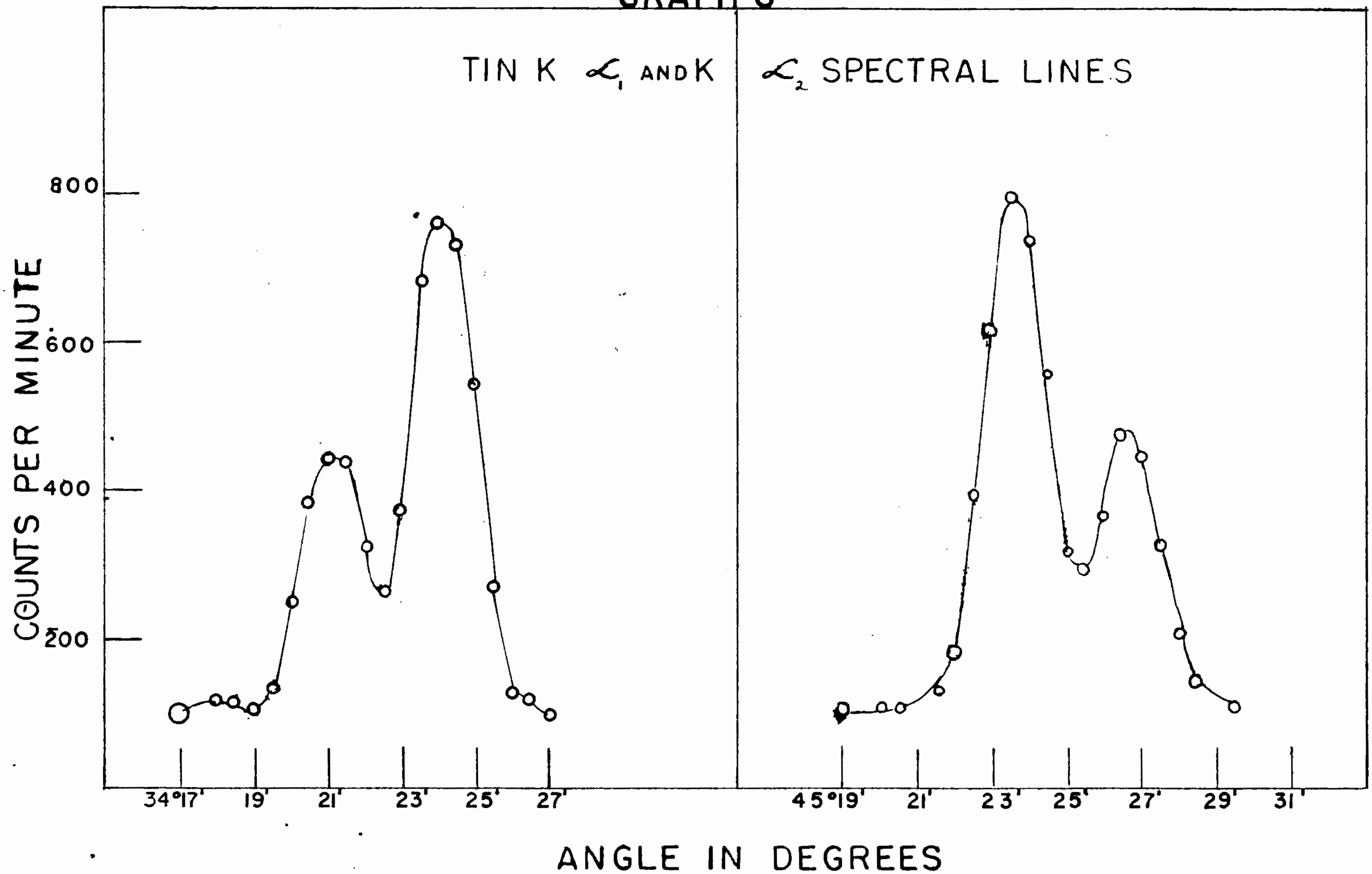
Measurements, as found in "X-rays in Theory and Experiment" by Compton and Allison, have shown that the crystal structure of calcite broadens the line structure by more than one minute of angle. The $K\alpha_1$ line of tin in graph 6 is 2.1 minute wide as compared with geometrical width of 0.9 minute. The total broadening is then 1.2 minutes greater than the geometrical broadening. The structure of the mica would be thought worse than that of calcite and this broadening is therefore not unreasonable. The line width from the photographic plate seems to be of the same order as that from using the counter as a detector. It can be seen from the microphotometer trace that the two $K\alpha$ lines are just barely resolved (Plate 2).

The wave length measurement

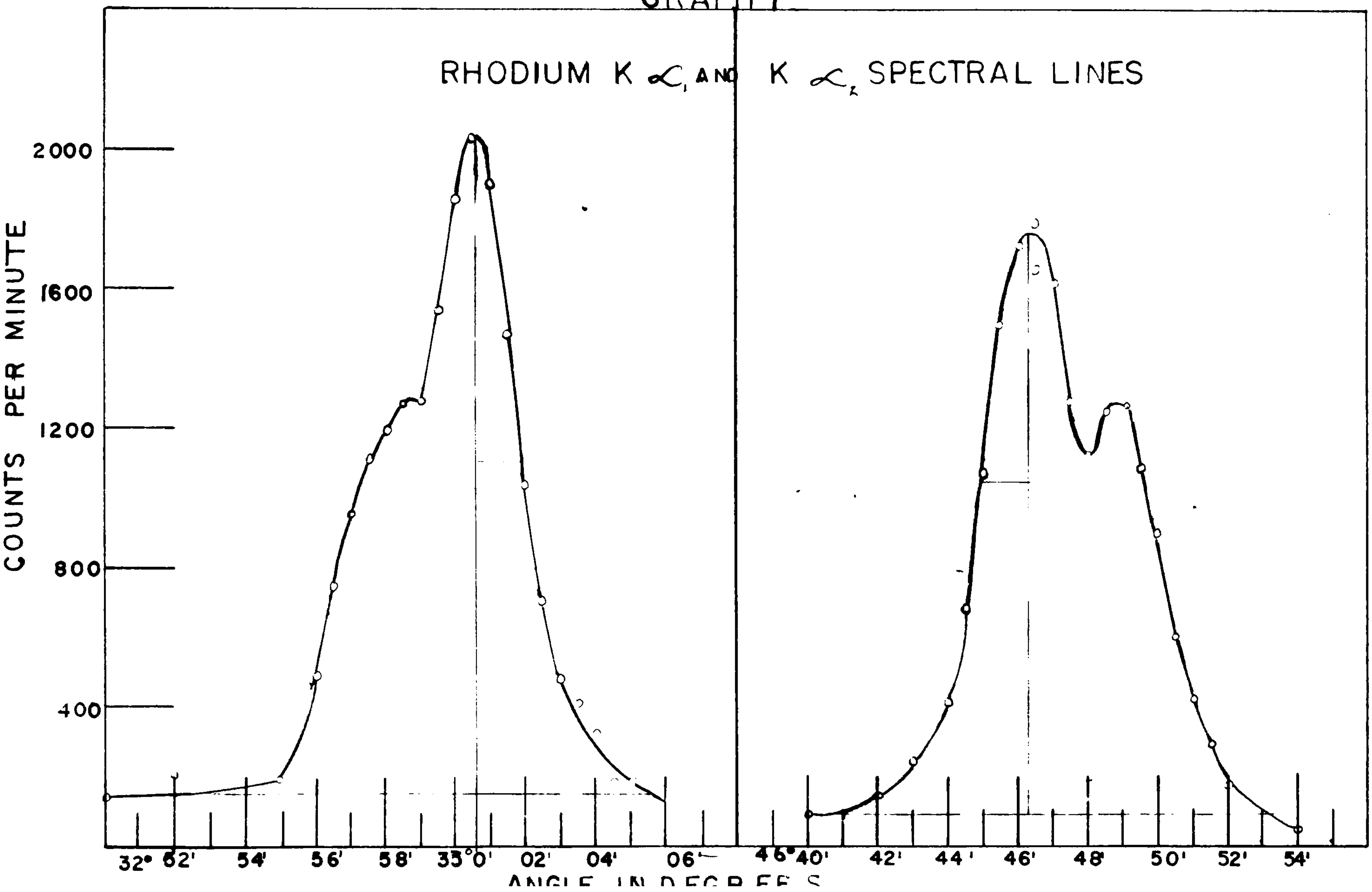
The wave length of the $K\alpha_1$ line for tin, rhodium and silver calculated from graphs 6, 7 and 10 respectively and from the known crystal spacing of 2.554 AU.

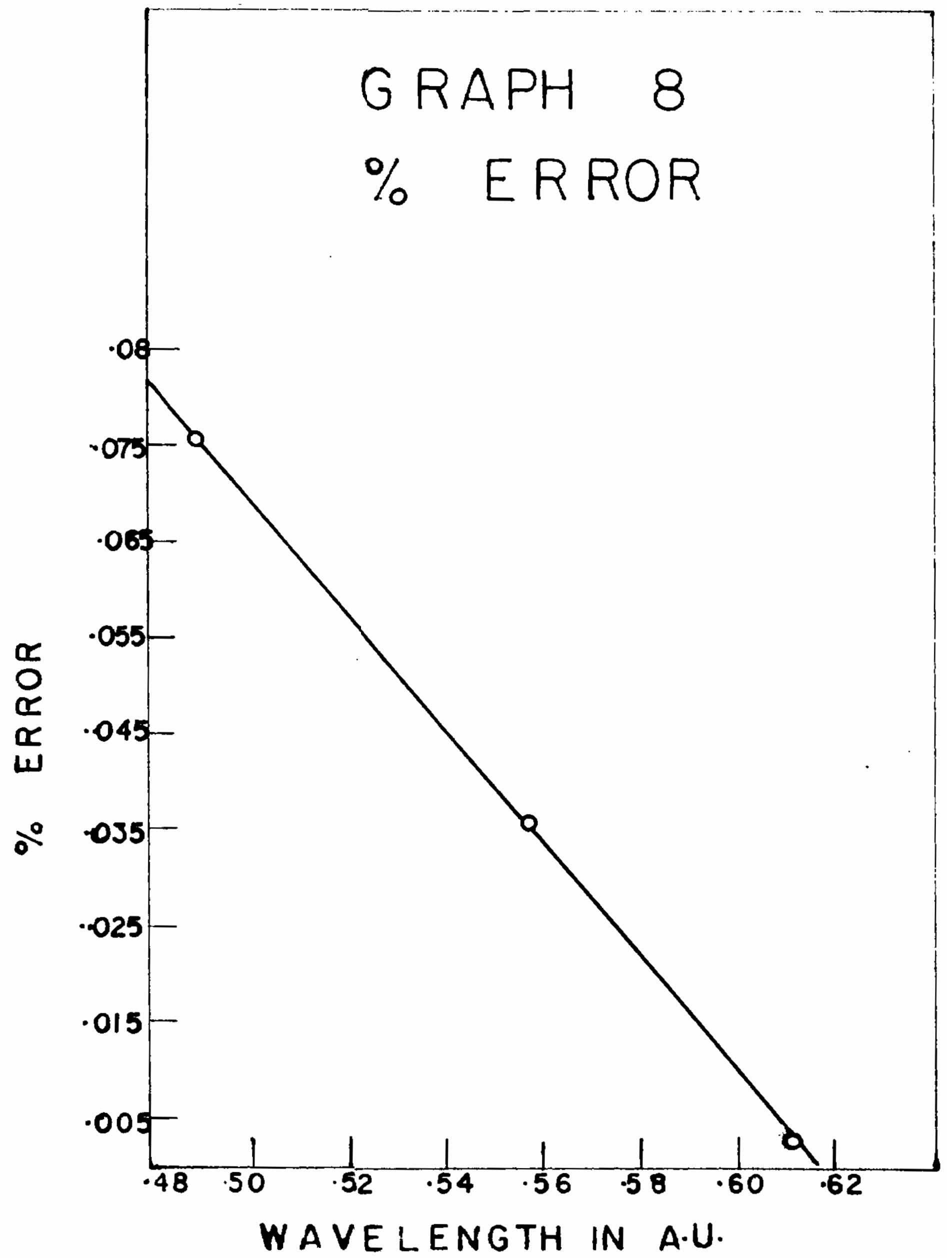
The wave lengths are shown in table 2.

GRAPH 6


TIN K α_1 AND K α_2 SPECTRAL LINES
COUNTS PER MINUTE
800
600
400
200
34°17'
19'
21'
23'
25'
27'
45°19'
21'
23'
25'
27'
29'
31'
ANGLE IN DEGREES

GRAPH 7


RHODIUM K α_1 AND K α_2 SPECTRAL LINES
COUNTS PER MINUTE
2000
1600
1200
800
400
32° 52'
54'
56'
58'
33° 0'
02'
04'
06'
46° 40'
42'
44'
46'
48'
50'
52'
54'
ANGLE IN DEGREES


GRAPH 8
% ERROR
·08
·075
·065
·055
·045
·035
·025
·015
·005
% ERROR
·48
·50
·52
·54
·56
·58
·60
·62
WAVELENGTH IN A·U·

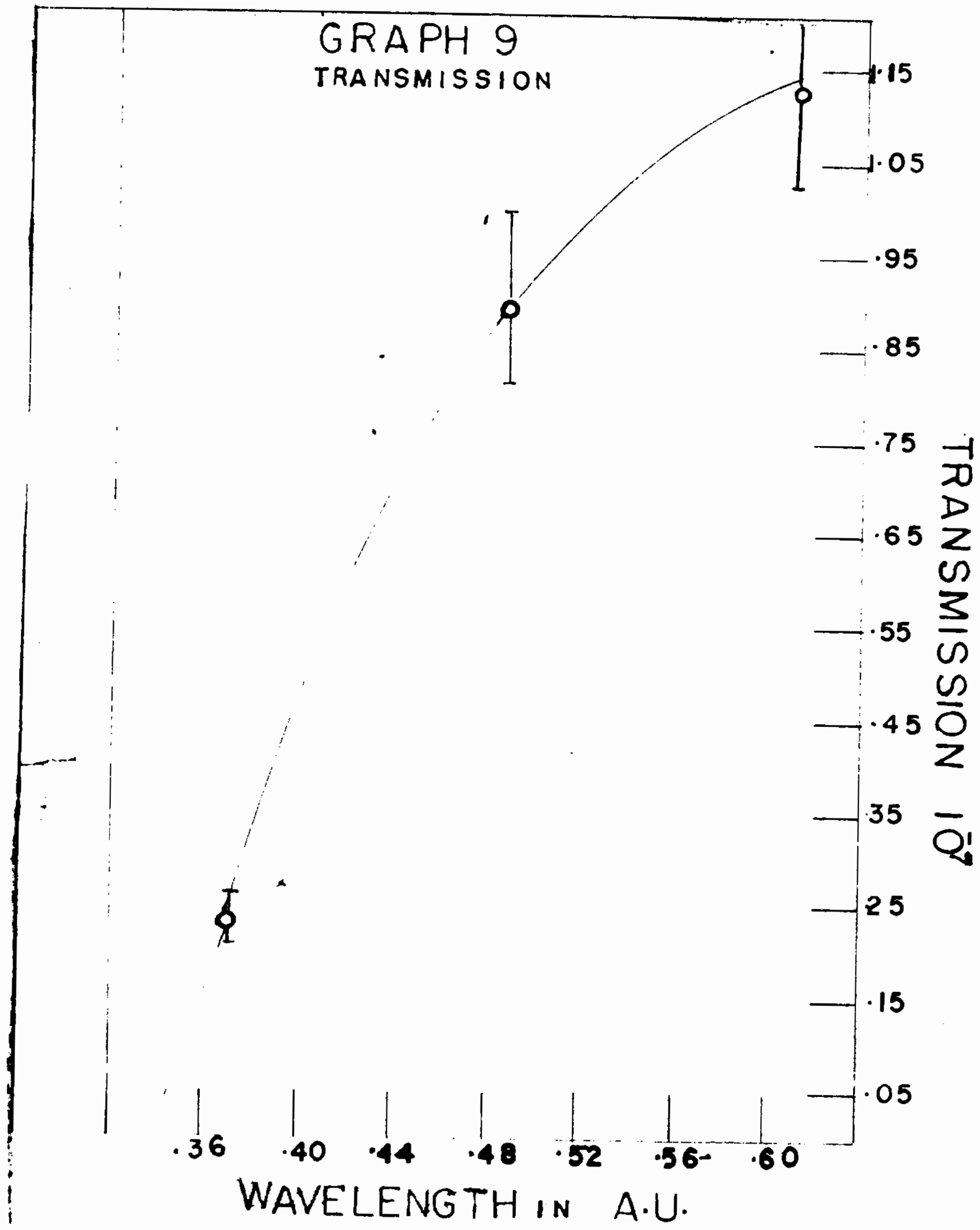

GRAPH 9
TRANSMISSION
1·15
1·05
·95
·85
·75
·65
·55
·45
·35
·25
·15
·05
TRANSMISSION 10^{-7}
·36
·40
·44
·48
·52
·56
·60
WAVELENGTH IN A·U·

TABLE 2

Element	Wavelength Measured AU	Wavelength Accepted AU	Difference AU	Estimated Probable Error AU	% Error %
Rh	0.61204	0.61202	-0.00002	0.00015	0.003
Ag	0.55804	0.55824	0.0002	0.00015	0.036
Sn	0.48924	0.48961	0.00037	0.00015	0.076

The measurement of the wavelength is shown to be in good agreement with the accepted values. It may be noted that the difference in the case of tin is more than twice the estimated probable error in the wavelength. This can be a systematic error of the gear mechanism. If we plot the percentage error as a function of the wavelength (graph 8) we see that it is a straight line. It would take more measurement to prove this linear relationship over the whole range. In any case it is seen that the maximum error is 0.076%. This shows that the gear reduction train is accurate enough.

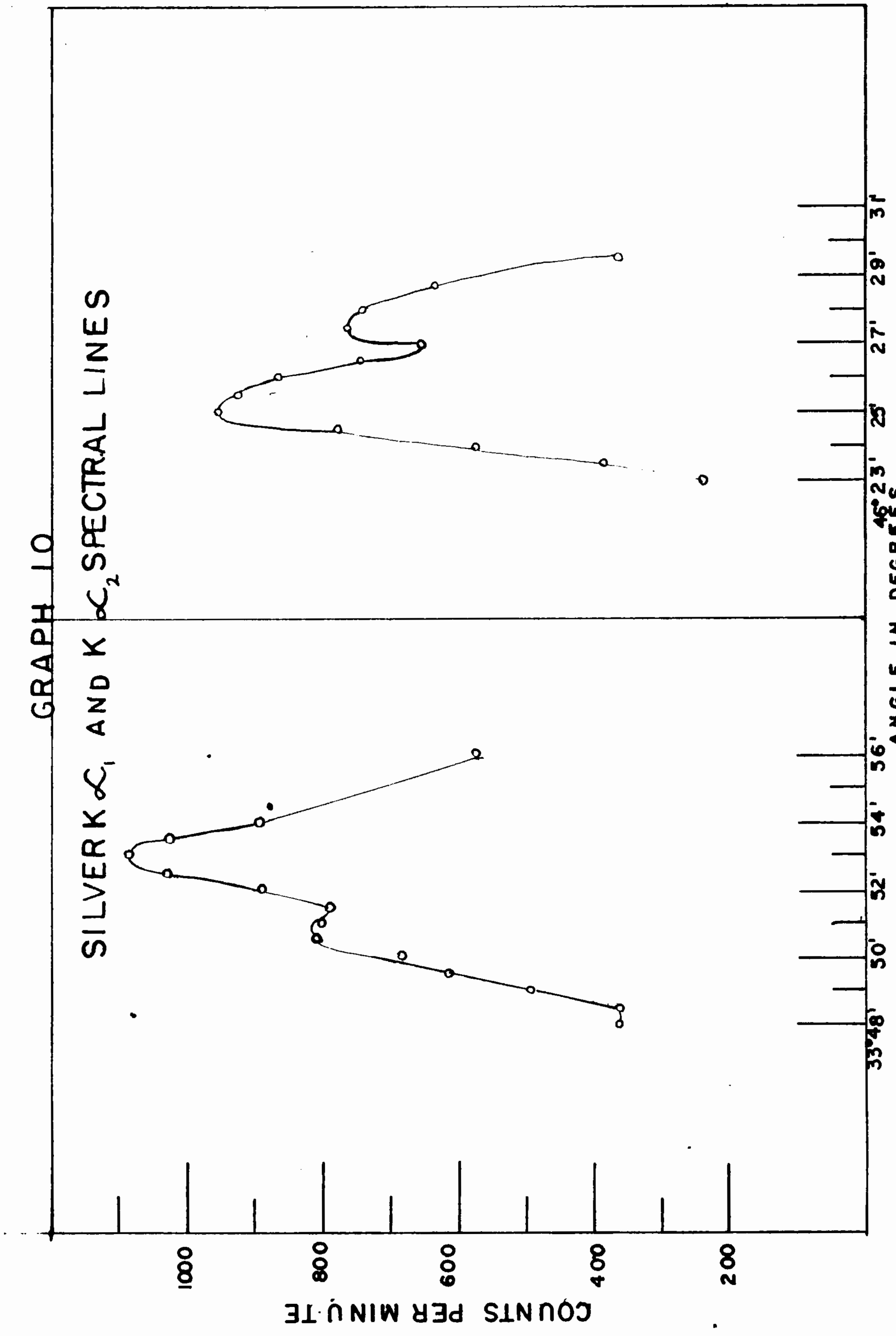
A photograph of the K lines of Ag was taken for diffraction from one side of the crystal planes only (plate 2). Thus the wavelengths cannot be measured absolutely. However, the separation between the lines was measured and checked against the accepted values (table 3).

TABLE 3

SEPARATION OF K - SERIES LINES OF SILVER

Line	Separation cm	Separation AU	Accepted Separation AU	Error %
$K\alpha_1 - K\beta_1$	0.397_1	0.0665	0.0665	0
$K\alpha_2 - K\beta_1$	0.372_5	0.0624	0.062	0.66

This shows that these results are almost as good as the counter results.


GRAPH 10
SILVER K α_1 AND K α_2 SPECTRAL LINES
COUNTS PER MINUTE
1000
800
600
400
200
ANGLE IN DEGREES
33°48'
50'
52'
54'
56'
46°23'
25'
27'
29'
31'

The transmission

The transmission of the instrument was measured for the $K\alpha$ doublet of three elements. These elements were rhodium, tin and lanthanum. The slit was set wide enough to straddle the doublet, and its length reduced to 1/2". The slit was turned to look directly at the source through the mica in the clamp and the counting rate was measured. Then it was moved onto the peak and the counting rate measured again. The counting rate was also taken on the peak with a piece of mica in front of the slit, the mica being of the same type as is used in the instrument. From this the percentage of the direct beam lost due to the absorption of the mica in the clamp was determined. Then from the known distance from the source to the counter the activity at the source was calculated. The measurements were used to calculate the transmission, table 4.

TABLE 4

THE TRANSMISSION

Element	Counting From Source Counts/Min.	Counting On Peak Without Mica Absorber Counts/Min.	Counting On Peak With Mica Absorber Counts/Min.	Transmission $\times 10^{-7}$
Rh	3.18×10^4	1264	1002	1.04
Sn	4.38×10^4	1386	1206	0.89
La	5.76×10^4	439	392	0.23

Width of slit	0.02"	Thickness of bent crystal	0.0175"
Height of slit	0.5"		
Diameter of source	0.187"	Thickness of mica absorber	0.0133"
Distance of source from counter	16.44"		

The calculation of the transmission is as follows. The area of the counter is 0.01 square inches at a distance of 16.44". This gives a geometrical counting efficiency of 0.294×10^{-5}. The detector counts both the $K\alpha$ and the $K\beta$ lines when it is looking directly at the source. The $K\alpha$ x-rays form 5/6 of the total number of x-rays. Therefore the counting efficiency becomes 0.353×10^{-5}. A piece of the same type of mica which is used to diffract the x-rays was placed in the beam going to the line. From this the absorption coefficient for this wavelength was calculated.

$$I = I_0 e^{-\mu x}$$

Where: I is the measured intensity with absorber

I_0 is the measured intensity without absorber

μ is the linear absorption coefficient

x is the thickness of the mica

For rhodium x-rays the absorption coefficient would be:

$$\mu = \frac{-1}{x} \ln \frac{I}{I_0}$$

$$= \frac{-1}{0.0133} \ln \frac{1002}{1206}$$

$$= 13.75 \text{ inches}^{-1}$$

From this, the intensity with no mica in the clamp would be:

$$I'_0 = I' e^{\mu x}$$

$$= 3.18 \times 10^4 e^{13.75 \times 0.0175}$$

$$4.04 \times 10^4 \text{ counts/min.}$$

Using the previously calculated value of the geometrical efficiency the counting rate at the source is 1.12×10^{10} counts/min. Therefore the transmission is

$$\frac{\text{Counts/minute at the line}}{\text{Counts/minute at the source}} = \frac{1.26}{1.12} \times 10^{-7}$$

This gives a value of 1.12×10^{-7}. The other values are calculated in the same way.

There are three main causes of error in these measurements. They are counting, scattering of the x-ray beam and variations in the intensity of the x-ray beam.

The number of counts taken for each element were all greater than 1000. This meant that the probable error was less than 3% from this cause.

The x-rays from the x-ray machine could have been scattered into the slit from various parts of the source holder and collimators although care was taken to keep this as low as possible. This would make the direct counting rate high and therefore increase the transmission.

The most likely cause of error would be in the measurement of the current in the x-ray tube. This was not measured to better than $\pm$ 10%. Fluctuations were noticed in this when the peaks were scanned.

The last factor overshadows the others. However, it is thought the measurements were accurate to within $\pm$ 15%.

These values are plotted against wavelength in graph 9. It can be seen that the transmission increases with wavelength. The range is not long enough and the points are not accurate enough to determine what sort of a functional relation exists.

For a photographic film, the important quantity would be the number of events at a source necessary to give a line on the film. Two exposures were made. The first one was forty minutes in which the line was just visible. The second was for six hours and is shown in plate 2. The number of counts per minute under this line was then measured with the scintillation counter. Since silver-rays have only a slightly shorter wavelength than rhodium the transmission values of rhodium were used to calculate the total counts at the source to produce the line.

The total number of counts under the silver peak was 3.25×10^4 counts/min. assuming the counter is 100% efficient. This was measured from graph 10. Therefore the number of counts to produce the line on the film

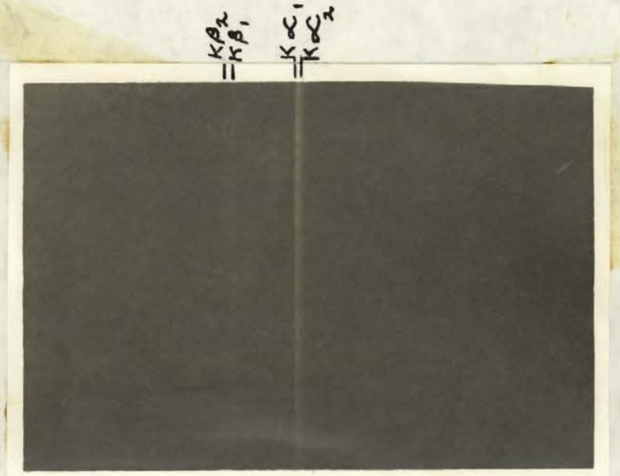


PLATE 2

would be 1.47×10^6. This would represent a total number of events at the source of 1.47×10^{13} for the forty-five minutes' exposure. The six hours' exposure which gave a good dark line would require eight times this number of counts.

The film used in this experiment was not the most sensitive. However, the most sensitive film available would be better by about a factor of three. Therefore the source would have to have 5×10^{12} events to be useable with the film. These figures would have the same errors as the previous ones, mainly due to fluctuations in the x-ray tube current.

The decay of cerium

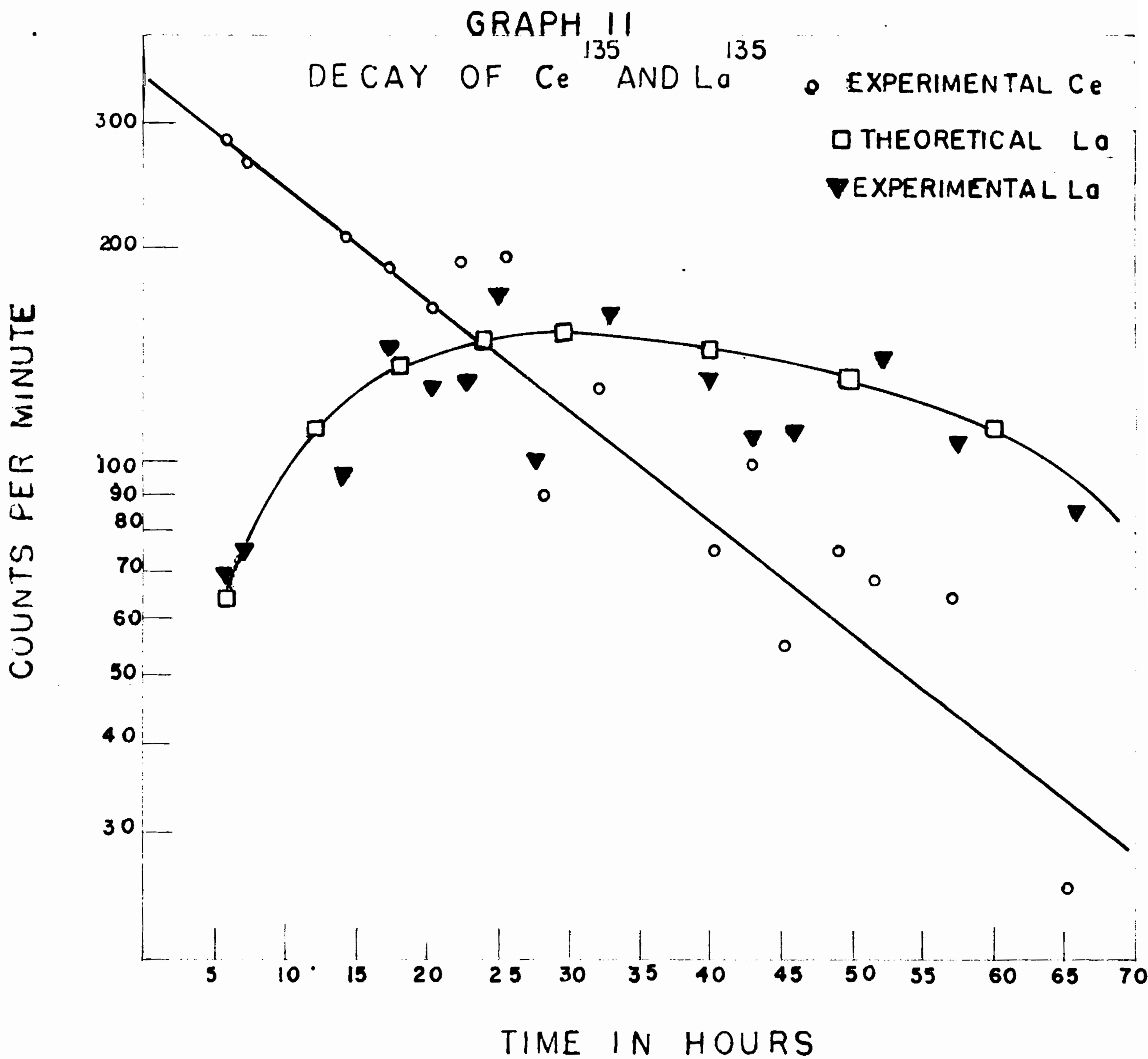
A lanthanum oxide target was bombarded in the cyclotron with 55 mev. protons for 8 hours. The beam current was one micro-ampere. The cerium was chemically separated from lanthanum and placed in the instrument and the resultant x-ray spectrum due to the decay of cerium was studied.

Three peaks were found with energies corresponding to the x-rays of cerium, lanthanum and barium.

The cerium x-rays were too weak to allow the study of their half life. It was noted that they were strong enough to register on the machine but were gone by nine hours after the separation. These x-rays were probably due to the conversion of isomeric states in Ce^{135} and Ce^{137}.

The decay of lanthanum x-rays was followed for 70 hours and plotted (graph 11). This decay showed the half life of 19 ± 2 hours. This half life is in good agreement with published values.

The decay of the barium x-rays was followed for 70 hours. These showed a growth and a decay. The half life of La^{135} has been reported as 19.5 hours. A half life of 24 hours for this decay has been tentatively set from measurements in this laboratory. Therefore using Ce^{135} decay curve as the


GRAPH II
DECAY OF Ce 135 AND La 135
EXPERIMENTAL Ce
THEORETICAL La
EXPERIMENTAL La
COUNTS PER MINUTE
300
200
100
90
80
70
60
50
40
30
5
10
15
20
25
30
35
40
45
50
55
60
65
70
TIME IN HOURS

standard the expected counting rate for La^{135} was calculated. The experimental values are shown as they fall around this curve in graph 11. It can be seen that there is good agreement, and the half life is therefore consistent with previously measured values.

VI CONCLUSION

In conclusion it can be said that the instrument has a relative line width of $\sim$ 0.65% with a mica sheet 0.0175" thick. This resolution may be increased by reducing the aperture of the mica and also by making it thinner. However, for most purposes this is entirely adequate since the $K\alpha$ lines of two neighbouring elements may be separated with two per cent relative line width.

From the measurements of the wavelengths of the 3 $k\alpha$ lines of Ag, Sn and Rh, the accuracy of the instrument was found to be less than 0.1%. This accuracy is entirely adequate for the setting of the slit on a line using the dial. It is noted that there is possibly a linear error in the gear train which is however too small to hinder the usefulness of the instrument.

The transmission of the instrument for a broad source seems to vary around the value of 0.5×10^{-7} for a 25 Kev. x-ray. This could be improved by using a thicker mica crystal which would of course reduce the resolution. But since a relative line width of 2% would be adequate, thicker mica would be quite useful. However, it is difficult to get a piece of thick mica of reasonable uniformity. This difficulty might be overcome by laminating several pieces.

The greatest single improvement to this transmission could be effected by interchanging the positions of the source and the detector. The interchange would increase the transmission by a factor of ten.

Another way to increase the transmission would be to increase the area of the mica and reduce the radius of curvature. These two factors would give considerably greater transmission perhaps by as much as a factor of five or six while still keeping the resolution at useable values.

Thus the instrument has shown itself capable of studying the decay of sources which can give counting rates of the order of 1.5×10^{6} counts per second and have radiation energies up to about 100 Kev. This is particularly applicable to the study of decay by electron capture of cyclotron produced sources.

APPENDIX I

In many cases it would be useful to have the instrument measure the energy directly, instead of the Bragg angle. This is particularly so where a scintillation detector is used, since the output of the detector tends to be proportional to the energy. If the movement of the apparatus is proportional to the wavelength, then it would be difficult to drive a window around a photopeak to keep in step with a drive in the instrument. Therefore, a design was thought of which would make the instrument move directly as the energy.

The design had also incorporated several other features. The source and detector, in an instrument such as this, must be heavily shielded against radiation. This shielding gives them a large mass and makes them difficult to move accurately. The accuracy of movement is most important for the slit since it must be kept on the focal circle. The clamp is relatively light and therefore easily moved. The movement of the source does not have to be of the same order of accuracy as the clamp and the slit. Since the machine can be set up with any two of the three moving, the slit would be best kept steady while the clamp and the source move. These features have been included in a movement thought of here.

The movement is shown schematically in diagram 7. The clamp is shown at AMB. The focal circle is shown as AMBCFE. The focus or slit is at F. The diameter of the focal circle normal to the centre of the surface of the crystal lies along ME. The crystal planes used for the Bragg diffractions lie along the line GMH. It is to be noted that the angle EFM is a right angle regardless of the position of any of the components. The angle that we wish to measure is HMF. It can be seen that if the point E is forced to move along the direction PF and the point M along the direction JK, the slit will always be on the focal

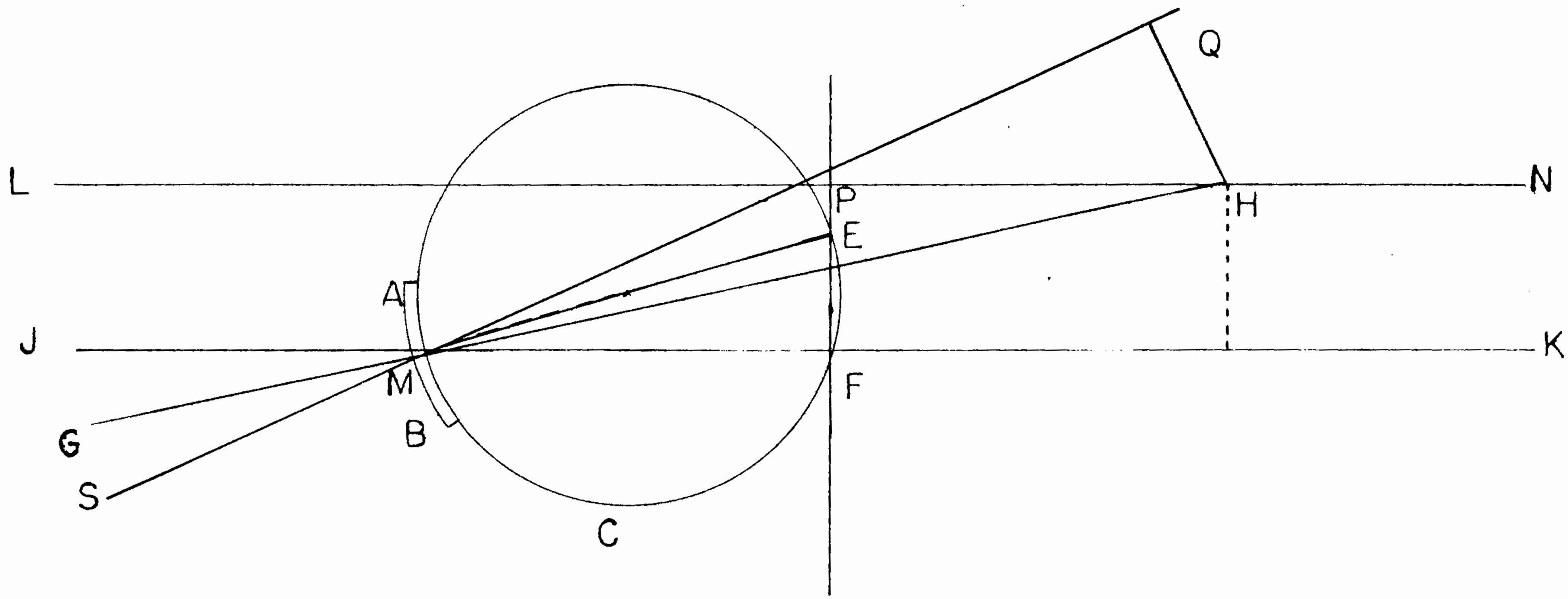


DIAGRAM 7

circle of the crystal AB. The source must be forced to move so as to keep the angles GMS and HMF equal.

Now the Bragg condition states

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

where n is the order of the diffraction, λ the wavelength, d the crystal spacing and θ the Bragg angle. Now λ is proportional to 1/E therefore n/E is proportional to $2d/\csc\theta$. So E is proportional to the $\csc\theta$. In diagram 7 if the point H is forced to move along a direction LM and the distance HM is measured we see that HM equals $PF\csc\theta$ and therefore is proportional to E.

The source S may be driven by pivoting the arm SQ at M and then driving SQ by the arm HQ, where HQ equals PF and the angle MQH is always kept a right angle.

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