# PRECISION MEASUREMENTS OF THE GLANCING-ANGLE OF REFLECTION FROM CALCITE FOR SILVER ( $K\alpha_1$ ) X-RAYS BY THE "METHOD OF DISPLACEMENT."<sup>1</sup>

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#### Abstract

**Part I. Method of displacement.**—This method permits the determination of glancing-angles in terms of *linear measurements only*. It consists in letting a properly limited beam of x-rays, after reflection, fall on a photographic plate placed successively in two parallel positions which differ in distance from the crystal by a known amount. The theory of the method and its practical application to the present case are briefly discussed with references to Uhler's paper.

**Part II. Apparatus and Adjustments.**—A new high precision spectrometer, including a specially designed bearing on which the crystal rotates and a unique form of plate holder using air pressure to ensure the flatness of the photographic plate, is fully described. A *special* form of hot filament *cathode* which gives an approximately *linear source* of x-rayshas been originated. Precision methods for adjusting and testing the reliability of the instrument are discussed at length. It is believed that the instrument is free from any defects that might cause a constant error of as much as 0.04" in the glancing-angle.

**Part III. Experimental prodedure.**—The cleavage planes of a nearly perfect specimen of Iceland spar were used as grating. The bearing of its perfection on the widths of the spectral images is discussed. *The coefficient of expansion of calcite* normal to the cleavage planes was computed from the most reliable data obtainable. A mean value which is sufficiently precise, over a sixteen degree range, to reduce values of glancing-angles to  $18^{\circ}$ C within 0.008'' is  $1.02_{\circ}(10)^{-5}$  per °C. Attention is called to the fact that the commonly used value  $1.04(10)^{-5}$  is not a sufficient approximation.

Distances between spectral images on each negative were measured on a Gaertner measuring engine, the errors of which were carefully investigated. The "displacements" of the photographic plate were equal to fixed intervals marked on a measuring bar and calibrated against the screw of the same measuring engine. The accuracy of these measurements and the effect of their errors on the glancing-angle are discussed.

The experimental procedure and precautions used in taking and measuring spectrograms are fully described. A complete protocol of observations is given for one plate. Evidence for the accuracy of the measurements is presented and discussed.

**Part IV. Results.**—The weighted mean value of the glancing-angle for the  $K\alpha_1$ line of silver from the cleavage planes of calcite at 18°C is 5°17′13.81″±0.06″ giving a wave-length, if log  $2d_{18}$ =0.7823350, of 0.558238A. This result is compared with those of other observers. It differs from Kellström's value quoted by the Int. Crit. Tables by more than ten times the combined probable errors. The only probable source of an adequate constant error in either result seems to lie in the crystal. Compton's measurements of the density of calcite are invoked to prove that different specimens of Iceland spar do differ enough in density to account for more than the discrepancy, if changes in density and grating space are interdependent.

<sup>1</sup> The final numerical results published in this paper have already been given by us in a letter to the Phys. Rev. [2] **35**, 564 (1930) and Nature **125**, 461 (1930).

# PART I. THEORY OF THE METHOD OF DISPLACEMENT

VERY precise methods have been developed for measuring the glancingangles at which characteristic x-rays are reflected by a crystal. In most cases where the highest precision has been attained, the results are based on measurements with a divided circle. In 1917 H. S. Uhler,<sup>2</sup> of this laboratory, suggested the so-called "Method of Displacement," by which glancing-angles are computed from linear measurements only. The method was given a preliminary trial, at the time, by Uhler and one of us<sup>3</sup> and gave results of greater accuracy than any hitherto reported. The present work was undertaken for the purpose of developing this method to the high degree of precision of which it seemed capable. Unforeseen difficulties delayed the work until divided circle methods had been developed to give a precision that we could not expect to surpass. However, glancing-angles are of such importance that, it seemed to us, their values should be checked by an independent method of equal reliability, and herefore the work was continued.

The method is a photographic one and "consists in taking one exposure when the plate is at a certain distance from the crystal and then a second exposure when it is at a different distance from the reflector. The displacement of the spectral image, corresponding to some one wave-length, is a function of the distance through which the plate has been *translated* parallel to the collimation line. The form of the function and the details of the calculation of the glancing-angle depend respectively upon the value of the constant angle between the normal to the plate and the collimation line, and upon whether the measurements are absolute or are based upon adjacent images pertaining to known wave-lengths. The interval of translation may be determined with ease and great accuracy, whereas only an approximate value of the distance between the plate and the axis of rotation is required in any case."<sup>2</sup> It involves the fundamental assumption that the plate, in each of its chosen positions, intersects a region of the reflected beam in which the intensity is symmetrically distributed about a line which makes an angle with the axis of the incident beam equal to twice the glancing-angle. Such a region can be obtained approximately, in the case of a perfectly selective reflector, if the incident beam completely fills, and is limited by, two narrow slits whose long axes are parallel to the axis of rotation of the reflector; provided that the aperture of the beam in the plane of the long axes of the slits is sufficiently limited. The region then lies between the Bragg foci of the two slits. The condition that the reflector be *perfectly selective* is not fulfilled by real crystals, but it will be shown later that the crystal used in the present work reflected a beam of the required symmetry.

The function by which the glancing-angle can be computed from the measured data is easily derived from the geometry of Fig. 1. This figure is drawn in a plane which is perpendicular to the mutually parallel long axes of slits  $S_1$  and  $S_2$ , axis of rotation and reflecting planes of the crystal. For simplicity the axis of rotation is assumed to intersect the line of centers of

<sup>2</sup> H. S. Uhler, Phys. Rev. [2] 11, 1 (1918).

<sup>3</sup> H. S. Uhler and C. D. Cooksey, Phys. Rev. [2] 10, 645 (1917).

the slits and to lie in the mean reflecting plane of the crystal, but the final result is independent of these assumptions. O is the point of intersection of the axis of rotation with the axis of the incident beam XX'. ORR' and OLL'represent reflected rays on either side of the direct beam and inclined to it at twice the Bragg angle of reflection,  $\theta$ . LR and L'R' are the paralle<sup>1</sup> traces of the photographic plate in its two positions and intersecting the reflected rays at the points L, R and L', R' between the Bragg foci of the slits. The central ray, XX', intersects the plate at the points C and C' and makes the angle  $\omega$  with the normal to the plate, OPP'. The dotted lines LL'' and RR''are drawn parallel to XX'.



Fig. 1. Geometry of the "Method of Displacement."

If  $x \equiv R'R''$ ,  $y \equiv L'L''$ , and  $D \equiv CC'$ , the angles  $\theta$  and  $\omega$  are given by

$$\tan 2\theta = \frac{2xy}{D(x+y)} \left[ 1 - \frac{D^2(y-x)^2}{4x^2y^2} \right]^{1/2}$$
(1)

$$\sin \omega = \frac{D(y-x)}{2xy} \tag{2}$$

If the plate is translated in the direction XX', the quantities x and y are respectively equal to the distances between corresponding spectral images on each side of the image of the direct beam, and D is the distance the plate has been displaced. But if the direction of translation is inclined to XX', the images of the undeviated beam taken in the two positions of the plate will not be superposed, and their separation must be measured and used in computing x and y from the separation of the spectral images. If is not difficult, in practice, to make this inclination so small that D may be taken as the distance the plate has been displaced in its line of motion without sensible error, but a corrected value of D can always be computed from this distance and the separation of the central images.

The quantity within the brackets in Eq. (1) is  $\cos^2 \omega$ , and therefore this term has very little effect on the value of  $\theta$ , if  $\omega$  is small. For small values of  $\omega$ , the effect of this factor will be a maximum for glancing-angles in the

neighborhood of 22°.5. Even if  $\omega$  is as great as 3.5′, the omission of  $\cos^2 \omega$  only causes an error of 0.03″ at such glancing-angles, and at 5°, the region under investigation, the error is only 0.01″. When  $\cos \omega$  and the central image displacement can both be neglected, the glancing-angle may be computed with sufficient accuracy from

$$\tan 2\theta = \frac{L'R' - LR}{2D} \,. \tag{3}$$

Thus the result depends on the measurement of intervals—LR and L'R'—each of which separates spectral images taken with the plate in a single position. This is an advantage if the spectral images corresponding to different positions of the plate have not the same width, and it is desired to use parallel hairs in the eye-piece of the measuring microscope.

A very full discussion of the geometry of image formation as applied to plane space-gratings, treated as perfectly selective reflectors, is to be found in Uhler's paper.<sup>2</sup> We therefore omit a detailed consideration of the effects of extreme rays and of inaccuracies in adjustment, and will merely cite these effects and the precautions taken to eliminate them in a later part of this paper.

### PART II. APPARATUS AND ADJUSTMENTS

The work was begun on an apparatus, designed by one of us,<sup>4</sup> but the results obtained lacked the accuracy of which the method was capable. Investigation showed the following defects: the apparatus as a whole was not sufficiently rigid; the plate-holder did not make the photographic plate flat and could not be displaced without fortuitous rotations; various parts of the apparatus were subject to distortion by small changes of temperature; and the axis about which the crystal rotated was indefinite. It was decided to build a new spectrometer which would be subject in use to no distortions causing a motion of one part relative to another of as much as  $0.25\mu$ . We are deeply indebted to Mr. Paul M. Mueller, then in charge of the Gauge Department of the Pratt and Whitney Machine Tool Co. of Hartford, Conn., for the design of a new spectrometer and plate-holder, which were built by that company under his constant supervision. The bearing for rotating the crystal was designed by him, and the unique form of plate-holder finally adopted was his original idea. We are also grateful to the Pratt and Whitney Co. for their very careful construction of the instrument.

Fig. 2 shows a view of the essential parts of the apparatus as set up for taking spectrograms. It is a tracing made from a photograph, and the wooden measuring stick, one-half meter long and divided in centimeters, fastened to base A, shows the scale in one direction. The radius of the divided circle table L is 13 cm, and the webbing of the castings varies in thickness from 1 to 1.5 cm. The whole assemblage weighs about 200 kg and rests on a stone-topped brick pier.

The primary base A is a standard Pratt and Whitney measuring engine bed, made of cast iron and aged by exposure to the weather for several years,

<sup>4</sup> C. D. Cooksey, Phys. Rev. [2] 16, 305 (1920).

after having been machined. The maximum vertical depth of the casting is 25 cm. The upper surface is 146 cm long and consists of two parallel ways or rails separated by a *T*-slot. The farther way is flat; the nearer is, in cross-section, an inverted 90° "V", truncated. After strains had been relieved by aging, the ways were scraped straight to conform to a "master." This base is supported on three leveling screws, furnished with lock nuts, one of which is near the forward end and directly under the center line between the ways. The bracket M which carries the forward slit  $S_1$  fits the track and, when in use, is locked directly over this foot.



Fig. 2. Spectrometer assemblage.

The secondary base B is an iron casting in the form of an "I" beam, reenforced with heavy vertical cross webs. It is 56 cm long, 11.5 cm wide at top and bottom, and 16.5 cm high. The bottom surface was machined and scraped to fit the rails of base A accurately, so that B may be considered an integral part of A when locked in place. Thus, any bending stress due to a load on B will be resisted by a section of cast iron 41.5 cm deep. The position of B relative to A may be adjusted and fixed by means of the screw and clamp shown at the left of B. The upper surface of B consists of a track, a vertical cross-section of which is shown in Fig. 3.\* The straightness and rigid-



Fig. 3. Cross-section of ways on base B.

\* Ways of this type were chosen because they have a vertical axis of symmetry and therefore permit the reversal of the "master" during the process of scraping the ways straight. ity of these ways are essential to the success of the displacement method, as it is along them that the plate-holder carriage is moved. The scraping of the bearing surfaces of this track was done with extreme care. When assembled the track on B is very nearly parallel to that on A.

The plate-holder carriage F (the forward end of which is also shown in Fig. 4) rests on the track on B. It has bearing surfaces 15.5 cm long which were scraped to fit this track with great accuracy. It is provided with a slow motion adjusting screw I, and may be locked in position by a clamp operated by a level seen just to the right of the letter F. A portion of its upper surface was scraped flat and in the assemblage is parallel to the upper surfaces of the track on B, and serves as a seat for the measuring bar H. The forward end of the carriage is a vertical "goose-neck" having a horizontal hardened steel plate bolted to it. This plate has a "V" groove milled across its upper surface and serves as a support for the plate-holder J. The carriage with the bar and plate-holder weighs 15.1 kg.

H is a standard 12 in. Pratt and Whitney steel measuring bar, adapted for our use. The lower and back surfaces are scraped flat. In the front surface are thirteen hardened steel plugs with their outer ends ground and polished so that their surfaces lie all in one plane, parallel to the opposite surface of the bar. The plugs are numbered from left to right, beginning with zero and each has a very fine line, ruled with a diamond. The lines are accurately parallel and are approximately perpendicular to the lower surface of the bar. The distances between successive rulings are very accurately 1 "U. S. inch" each. The bar is held in place on the carriage by a tapered dowel pin which fits in a reamed hole at the left end of the bar. Two horizontal set screws (one of which is shown at V in Fig. 4) near the right hand end of the bar bear against a stud in the carriage which passes through an oversized hole in the bar. This permits the adjustment of the plane of the ruled plug surfaces parallel to the line of motion of the carriage.

The axis marked XX' in Figs. 1, 2, and 4 is defined as a line parallel to the line of motion of F and intersecting Y, the axis of rotation of the crystal, at the fixed elevation of the center of the slit  $S_2$ .

The microscope bracket D clamps to a short hollow cylinder which is bolted to a scraped surface on the central web of base B. The microscope is clamped in an eccentric collar E in the upper end of the bracket, and can be adjusted to any desired position. It is of 75-power and has a micrometer eye-piece. The movable reference lines ("crosshairs") are two fine diamond rulings on a glass plate and intersect at an angle of one radian. When the microscope is in proper adjustment the bisector of this angle is parallel to the vertical fiducial lines on the plugs of the measuring bar. An axial illuminator can be mounted on the forward end of the objective housing.

The bearing mechanism for rotating the crystal, and the slit  $S_2$ , are supported on a base which somewhat resembles a sled, the two runners of which embrace the lower edges of the base B. The nearer of these runners is shown in Fig. 2 and is labeled C. This base can rotate about an accurately fitting horizontal trunion bolt which passes transversely through the runners and B.

The lock nut and washers of the nearer end of this trunion are partly visible behind the wheel K. The base is adjusted and locked in position by means of two set screws in the left hand end of each runner; they bear against studs screwed into B through oversized holes in the runners. This mounting is thus a sort of cantilever and permits a necessary adjustment of the axis of rotation of the crystal with respect to the rest of the instrument.

Two vertical posts, one of which is shown, bolt to the forward end of the sled and have bolted to their upper ends a steel cross piece N, which in turn carries the adjustable mounting for the slit  $S_2$ . In the center of the sled is a scraped seat, with a large hole bored through it, to which is bolted the bearing for rotating the crystal.

The parts B, C, D, F, J, and M are made of cast iron. After being machined, but before the final finishing was done, they were artificially aged. The aging process was apparently effective, for none of the parts has shown any noticeable warping during several years.

A section of the plate-holder J in a plane parallel to that of XY is shown at (a) in Fig. 4 in position on the carriage F, and in perspective with the cover partly removed at (b). The holder is supported and positioned by two cone-pointed bolts, one of which is shown at T, and the round-ended screw U. The cone points fit in the slot milled across the steel plate which is bolted to F, and the screw U bears against a plane surface milled on F. The cone of one of the bolts is not coaxial with the bolt, thus permitting a small rotation of the plate-holder about a vertical axis. Adjustment about the line of the slot is made with the screw U. The center of gravity of the plate-holder is far enough forward of the screws T to keep the holder in position when the carriage is moved. The plate-holder is so compact that the distance from the center of the plate to the surface of the track below is only about 5.5 cm.

The plate-holder is of special design to ensure the flatness of the sensitized surface of the photographic plate. This surface is forced against two parallel flat surfaces on the cover, just to the left of OO, by thin walled rubber tubes RR' inflated with air at about two atmospheres pressure. The two surfaces OO are an integral part of a sufficiently large body of cast iron to prevent bending under this stress.

The cover of the plate-holder is one casting in the form of two V-sections joined together at both ends; these are reenforced by seven cross webs on each side, which terminate in bosses through which the fourteen screws QQhold the cover to the back. Each of the V-sections is of the nature of a cantilever, such that the pressure on the surfaces OO causes a tension in the screws QQ and a pressure across the surfaces of contact between the cover and back of the plate-holder at PP. The back of the plate-holder offers about five centimeters depth of cast iron to resist both these stresses. The opposing surfaces of the cover and back, as well as the surfaces OO, were scraped plane to a high degree of precision. It had been the original intention of the designer to finish these surfaces by lapping, but it was feared that the slight peening action of the abrasive would only warp them. The edges of the surfaces were left sharp so that in working the two parts of the holder into contact dust particles would be removed.



Fig. 4. Cross-section and perspective of plate-holder.

A strip of black paper, fitted into slots in the V-sections, covers the opening between the sections and serves as a light seal. A centimeter scale, ruled on the surface S along one side of the opening, facilitates the positioning of a lead screen when it is desired to shield a portion of the plate from x-rays. The photographic plates were cut in strips 25 cm long and 3.4 cm wide, dimensions such that the extreme edges of the strips extended slightly beyond the boundaries of the surfaces OO. This was to prevent irregularities in the edges of the gelatin from interfering with the proper seating of the plate. The mahogany strip W, backed by a piece of velvet, and having two longitudinal grooves 6 mm wide milled in its front, affords support for the rubber tubes and distributes the pressure in them uniformly to the back of the plate-holder. The portions of the rubber tubes which press against the photographic plate are thinner than the remainder. We are indebted to Mr. G. W. Patterson of the Seamless Rubber Co. of this city for these tubes. Air is admitted to the tubes through a nozzle shown just below the letter J, Fig. 2. The tube R' is shown inflated in the section. When the tubes are deflated. There is ample clearance between the plate and the surfaces OOto prevent scratching of the gelatin while the cover is being worked into place. The plate-holder assemblage weighs 5.6 kg.



Fig. 5. Cone bearing.

The cone bearing for rotating the crystal is shown in Fig. 5. The flange on the seasoned cast iron sleeve B was scraped to fit its supporting seat on base C, Fig. 2, and the sleeve passes through the hole in this seat. The steel spindle Ais a right circular cone supported by a hardened steel foot C; the lower surface of C is spherical and rests on the upper plane surface of D, also of hardened steel. The lower part of D is a stud which fits tightly in the sleeve F, and the latter has a sliding fit in B and a long enough bearing to keep D from rocking. The stud rests on the screw cap E, which is threaded to B with a fine thread and serves to adjust the bearing. By using a long tommy in the holes provided in E, the spindle and all its superstructure can easily be raised by as little as  $2.5\mu$  at a time. As the tangent of the semi-apical angle of the cone is slightly less than 0.06, an accurate means is at hand for adjusting the oil film separating the surfaces of A and B to the minimum thickness required for lubrication. If this adjustment was correctly made at one angular position of the cone, it was found to be correct for all others, showing that the cone and its bearing fitted accurately. This corroborated what had already been inferred from the usual test with prussian blue. When the spindle was properly adjusted, a diminution in the thickness of the oil film of  $0.3\mu$  would cause the oil to cease acting as a lubricant. Therefore the cone may be considered to rotate in *B* about a practically *fixed* axis.

An essential feature of the design of the sleeve B is that portions of its bearing surfaces are relieved to insure the proper distribution of the oil and to prevent particles of foreign matter from getting between the bearing surfaces. This is done by removing one half the bearing area of B by three grooves, similar to rifling, but running parallel to the elements of the cone. The edges of these grooves were left sharp. This bearing was originally set up four years ago with "3 in 1" oil for lubricant, and the above tests and adjustment made. Since that time, no oil has been added, nor has any adjustment been found necessary by later tests. The oil shows no sign of having evaporated, or of becoming sticky after lying idle for six months at a stretch. It is doubtful if expensive watch oil would have been more satisfactory. If such a bearing was being built for continuous use, it would be well to replace the steel bearing surface of D by one of agate.

The spindle A is bolted to the divided circle table L, Fig. 2, and can be rotated by a tangent screw, the divided head of which appears at K in the figure. Screwed to the upper surface of this table and coaxial with it, is a circular brass plate with three radial slots 120° apart which serve to position the crystal holder or an auxiliary slit.

The bilateral slits,  $S_1$  and  $S_2$ , have gold jaws 2.4 mm thick, 2.5 mm wide, and 1 cm high, inserted in brass plates which slide in dovetails on the slit frames. The opposing surfaces of the jaws were carefully scraped flat. The slit widths can be varied by means of cams acting above their centers; but during the taking of spectrograms one jaw of each slit is held in place by two dowel pins, and the other jaw is clamped tight against platinum shims placed between the opposing surfaces above and below the limited portion of the slit used. The slit  $S_2$  is at a fixed height above the supporting bar N, but  $S_1$ can be raised or lowered. Both slits can be rotated about horizontal axes through their centers and about vertical axes, and can be translated at right angles to the plane XY.

The crystal holder assemblage is shown mounted on the table L. Its base is a brass disk with three cone feet which fit the slots provided for it. On this base is mounted a rectangular plate with lateral adjustment. The plate supports, in a vertical sleeve with locking set screw, a sliding post which carries at its upper end the outer of a pair of coaxial cylinders. The inner cylinder can be rotated with respect to the outer by a tangent screw. The crystal is fixed by soft wax with its working face against the surface of a slotted brass plate, which bolts inside the inner cylinder in such a manner that the axis of the latter lies approximately in the face of the crystal. Thus, the lateral and rotational adjustments of the crystal are independent. The crystal holder can be replaced by an auxiliary slit (not shown) later referred to as  $S_3$ . The later is so adjusted that, when in place, its long axis coincides with the axis of rotation Y, and the plane through its upper corners contains the axis XX'.

The x-ray tube is mounted on a bakelite stand in a rectangular box of 5 mm lead sheet. The box is supported on two pieces of channel steel 31 cm deep which rest on the same pier as the spectrometer. The tube used in these experiments was entirely constructed in this laboratory and was of Coolidge type with 15 cm bulb. The target was a button of silver welded, to insure good thermal contact, to a water-cooled copper anode. The x-rays were taken at an angle of about 3.5° to the plane of the target. The cathode, heated by current from a storage battery, was designed to bring the cathode rays to an approximately line focus on the target. With this in view, the filament was wound in a spiral groove on a tapered mandrel, just as for a fine focus Coolidge filament; but, instead of being annealed between spherical surfaces, it was placed between cylindrical surfaces of 19 mm radius during annealing. This gave a focal spot on the target which was longer in one direction than in a direction at right angles; but, due to the magnetic field of the heating current, its long axis was inclined at an angle (roughly 45°) to the axis of the cylindrical filament. This is a drawback if it is desired to change the strength of the filament current after the tube has once been aligned with respect to the spectrometer. We hope to overcome this difficulty by the use of a noninductive filament. Such a filament should have a greater thermionic emission at its center, owing to the elimination of the center lead, and should give an excellent focal spot distribution. We have succeeded in winding such a double filament, but have not had an opportunity to mount and try it.

The cooling water was circulated by a pump through a Ford automobile radiator and its temperature measured just before and just after passing through the anode. By regulating the speed of a fan blowing on the radiator, the temperature, and therefore the length of the anode could be held constant, thus keeping the focal spot on the line through the narrow slits used.

The source of potential for the tube was a 10 K.V.A. transformer connected to the 220 volt, 60 cycle, city supply. It was controlled by a variable inductive reactance in the primary and had a mechanical rectifier in the secondary, utilizing about one half of each side of the wave.

For precision measurements with the above described apparatus, it is essential that the plate-holder, when the carriage F is moved along its ways, should have rectilinear motion only; and that there should be no appreciable motion of the microscope relative to the crystal. It is also important to have the planes of the opposing faces of the slit jaws parallel to the plane XY, and the reflecting planes of the crystal approximately parallel to the axis of rotation. These adjustments were made with the aid of several steel mirrors of different thickness, each having two opposite reflecting surfaces accurately plane and parallel; and an auto-collimating device for detecting and measuring any rotation of the mirrors. The auto-collimator consisted of a 43 cm focal length objective with illuminated cross-hairs in its focal plane. The  $90^{\circ}$  cross-hairs, together with their reflected image, were viewed with a 20power micrometer microscope. The sensitivity of this arrangement was 7, micrometer divisions per second of rotation of the mirror, and one-third of this rotation could be detected. For detecting small motions of one part of the spectrometer relative to another, two steel balls were fastened to one part, a third ball to another, and on a glass or hardened steel plate resting on these three balls was mounted a mirror facing the auto-collimator. When the balls bore on hardened surfaces, scrupulously clean, a relative motion of 0.01u could be detected by this device.

Making use of this tilting mirror, it was found that the largest relative motion between the spectrometer microscope and the axis Y, due to moving the carriage F with the plate-holder along the ways, was the entirely negligible amount of  $0.06\mu$ . The carriage, when displaced the full length of the ways, rotated about horizontal and vertical axes perpendicular to its line of motion by the insignificant amounts of one and two seconds respectively, as measured with the auto-collimator. A sensitive spirit level showed no greater rotation abouts its line of motion.

These tests having been made, the sled C was adjusted, with the aid of the auto-collimator and mirrors until the Y-axis was perpendicular to XX'. A micrometer microscope, giving 45° deviation, was then clamped to the carriage F and adjusted until the axis of its objective coincided with the line XX', thus supplying a means for bringing the centers of the slits  $S_1$  and  $S_2$  into this line.

The measuring bar was adjusted by a dial indicator until the scraped surface on the side opposite the plugs was parallel to XX' to within 3 seconds of arc. Since the surfaces of the plugs had all been ground and polished into one plane parallel to this scraped surface, this adjustment brought their surfaces parallel to XX', and insured all of the diamond scratches being in focus without parallax as they came opposite the microscope, once this was focused on one of them.

By means of a mirror wrung to the vertical scraped surface of H, another on the divided circle table with its surfaces parallel to the axis Y, and mirrors clamped between the jaws of the slits  $S_1$  and  $S_2$ , the auto-collimator could be set with its axis normal to the plane XY, and the slits adjusted until the surfaces of their mirrors were parallel to this plane. The slit surfaces were thus adjusted parallel to Y within 30" and to XX' within 15". In a similar manner the plate-holder was adjusted so that the plane of the photographic plate would be approximately perpendicular to the axis XX'. The crystal, which had a cleavage face giving fair optical reflection, was similarly adjusted so that this face was parallel to the axis Y and as nearly as possible in this axis, the latter adjustment being made with a high power microscope. The various mirrors were brought opposite the auto-collimator by sliding the carriage B, and slit bracket M along the tracks on A. Wherever possible, the foregoing tests were checked with a spirit level sensitive to 5 seconds of arc per mm displacement of the bubble and, in some cases, with one of five times this sensitivity.

The greatest care was used to test the flatness of the opposing surfaces of the two parts of the plate-holder, not only when the plate-holder was disassembled and free from stress, but also when it was assembled and the air pressure applied.

The front portion, mounted on the carriage F in such a manner that it could be moved parallel to the surfaces OO, was examined with an interferometer using parallel and approximately monochromatic green light. The high portions of the scraping marks on these surfaces were so nearly continuous, and so well polished from working the two parts of the plate-holder together during assemblage, that the fringes were entirely unambiguous and could be counted. After allowing for the slight departure of the motion of F from a straight line, the surfaces OO were found to be flat to one fringe.\*

The back of the plate-holder was similarly mounted on the carriage F, but the scraping marks were not close enough together to give useful fringes. However, with mirrors wrung to its surfaces in various places, it was tested both with the interferometer and the auto-collimator, and no evidence of an appreciable deviation from flatness was observed. This, together with the usual test for the fit of two scraped surfaces, convinced us that these surfaces were flat enough when separate and not under stress. Incidentally, the rectilinear motion of the carriage F was checked when these observations were made.

The plate-holder was then assembled with a photographic plate of "parallel plate" glass 1.5 mm thick in place. Tilting mirrors with short lever arms showed that contact between the surfaces OO and the gelatin surface was approximately complete at a pressure of 25 lb in<sup>-2</sup>, because an increase to 30 did not diminish the separation of these surfaces by as much as  $0.1\mu$ . A pressure of 12 was required to expand the rubber tubes. The photographic plate was then replaced by a strip of "parallel plate" glass of the same thickness, without gelatin, and this was examined with the interferometer, the air pressures varying from 20 to 30. The fringe system showed very little change after a pressure of 25 was reached, showing approximately complete contact at this pressure. Though the surface of "parallel plate" glass is not optically perfect, the interferometer could be set so that there were not more than one or two fringes in a distance of 3 cm at any part of the strip. The number of fringes per cm was determined for every centimeter of the useful length of the plate, as the plate holder moved parallel to the plane OO in front of the interferometer. This procedure was repeated with several other strips of "parallel plate." The data thus obtained gave no indication of a curvature of the surfaces OO which could cause a constant error in measurements of glancing-angles of as much as 0.04". The gelatin surface of ordinary commercial photographic plates, when examined with the interferometer, showed very rapidly changing local curvatures many times greater than any constant curvature that could be introduced by the plate-holder. It is in-

\* The perfection of the spectrometer and plate holder is largely due to the painstaking care with which the scraped surfaces were fitted by Mr. Moody of the Pratt and Whitney Company.

teresting to note, in this connection, that when a strip of gelatin was removed from the plate, the fringe system associated with the remaining gelatin was usually continuous with that associated with the glass. This showed that the upper surface of the gelatin conformed pretty generally to the irregularities of the glass surface on which it was coated.

The spectrometer, having been adjusted and tested in the manner described, was next aligned with the source of x-rays. In order to obtain maximum intensity through two very narrow slits, it is necessary that the line of centers of the slits should pass through the center of the most intense region of the focal spot on the target. As the authors have previously shown,<sup>5</sup> the focal spot is striated when the hot cathode is a spiral filament, being approximately a projection of the turns of the filament on the target. By taking x-ray photographs through  $S_1$  when  $S_3$  was in place and again through horizontal slits substituted for  $S_1$  and  $S_2$ , it was possible to adjust the tube and spectrometer so that the latter was approximately level, and the center line of  $S_1$  and  $S_2$  passed through the center of the most intense region of the focal spot. That this line coincided with the axis XX', as previously defined, was also checked photographically with the aid of  $S_3$ .

### PART III. EXPERIMENTAL PROCEDURE

The crystal used was a calcite rhomb  $12 \times 10 \times 7$  mm cleaved from a block of Iceland spar obtained from the "Marsh Collection," Yale University. This specimen is denoted by Ca 1. The largest cleavage face was used for reflection, and, as far as could be found by examinations with reflected light and with a microscope, the portion on which the x-rays fell was a plane. After mounting in the crystal holder with soft wax, this face was adjusted parallel to, and into coincidence with, the axis of rotation within 25'' and  $10\mu$  respectively. A tilt of 25'' would cause an error in the glancing angle under consideration of 0.0004''.

Due to refraction, no crystal is a perfectly selective reflector, and therefore a monochromatic beam, upon reflection, will diverge beyond the limits of the rectangular region discussed by Uhler.<sup>2</sup> A sufficient measure of this divergence was obtained, without using a double crystal method, by comparing the angular width of the incident beam, as calculated from the slit widths, with the angle through which the crystal could be turned while still reflecting some energy of the wave-length under consideration. The limits of this angle were determined by finding the angular settings at which the crystal ceased to give an image on the photographic plate, during an exposure time a little too short to permit the formation of a perceptible image due to white radiation. This angle was approximately 46.8", with an extreme variation of less than 10 percent. As the beam was limited by the slits to an angular width of 26", the divergence due to the crystal was 20.8."

Table I gives a comparison between line widths calculated from the above angle and average line widths measured on the spectrograms, at three different distances from the crystal denoted by the plug numbers on the mea-

<sup>b</sup> C. D. Cooksey and D. Cooksey, Science 58, 382 (1926).

suring bar. The plate intersected the reflected beam near the Bragg focus of  $S_2$  and  $S_1$  in the first and third positions respectively and about half way between these foci in the second position. Considering the difficulty of de-

TABLE I. Line widths at various positions of plate.

Plug No.	0	6	11
Calculated	0.0249 mm	0.0400 mm	0.0535 mm
Measured	0.0276 "	0.0395 "	0.0519 "

termining line widths, the above figures are in fair agreement and give a reasonable indication of the perfection of the crystal. In practice it was found that a properly filled out image was obtained when the crystal was rotated through an angle of 18" symmetrically about the best setting; no additional width accrued due to energy from the wave-length under investigation through using a larger angle.

Uhler<sup>2</sup> has pointed out that oblique rays in a plane parallel to the axis of rotation of the crystal broaden the image asymmetrically. During the taking of spectrograms, the incident beam was limited by the following conditions: the widths of the two slits  $S_1$  and  $S_2$  were fixed by strips of platinum foil 0.02 mm thick, their lengths were limited to 3.2 and 2.5 mm respectively, and their distances from the crystal were 35.5 and 4.5 cm respectively. Limited thus, the maximum effect of oblique rays on the glancing-angle is the negligible amount of 0.02'' for an eleven inch displacement.

In order to reduce the values of the glancing-angle obtained from the spectrograms to their values at a standard temperature, it is necessary to know the coefficient of expansion of calcite along the normal to its cleavage planes; i.e., the coefficient  $a_{18}$  in the formula  $\sin\theta_{18} = \sin\theta_t [1 + a_{18}(t-18)]$ . W. Stenström<sup>6</sup> gives the generally quoted approximate value of  $1.04(10)^{-5}$  per degree centigrade, which is not precise enough to be used to correct data of the accuracy attained today. In obtaining this value, he assumed an angle of 45° between the optic axis and the normal to a cleavage plane, and 2.62  $(10)^{-5}$  and  $-0.54(10)^{-5}$  per degree centigrade respectively for the coefficients parallel and perpendicular to the optic axis. We have recalculated the coefficient using the following constants: the axial angle of the crystal is 101° 55.0′ ± 0.2′ at 22°C as given by H. N. Beets;<sup>7</sup> the mean coefficients of expansion parallel and perpendicular to the optic axis are  $(\alpha_{\parallel}+2 \beta_{\parallel}t)$  and  $(\alpha_{\perp}+2 \beta_{\perp}t)$  respectively where:

$\alpha_{\parallel} = +2.513  53  (10)^{-5}$	$\beta_{\parallel} = +0.001 \ 18 \ (10)^{-5}$
$\alpha_{\perp} = -0.557 \ 82 \ (10)^{-5}$	$\beta_{\perp} = +0.000 \ 138 \ (10)^{-5}$

in degrees centigrade as given by Benoît.<sup>8</sup> The value of the coefficient  $a_{18}$  corresponding to these constants is:

<sup>6</sup> W. Stenström, Dissertation, Lund (1919) p. 38.

<sup>7</sup> H. N. Beets, Phys. Rev. [2] 25, 621 (1925).

<sup>8</sup> R. Benoît, Travaux et Memoires du Bureau International des Poids et Mesures 6, 190 (1888).

$$a_{18} = 1.02_3(10)^{-5}$$

which, if used over a sixteen degree interval, will give the correction of the glancing-angle for the  $K\alpha_1$  line of sliver to 18°C within 0.008″.

Table II gives  $\log_{10}[1+a_{18} (t-18)]$  for the interval 10°C to 26°C.

TABLE II. Temperature correction for calcite.

t°C	$\log [1+a_{18}(t-18)]$	<i>t</i> °C	$\log [1+a_{18}(t-18)]$
10	0.999 964 46	18	0.000 000 00
11	968 90	19	004 44
12	973 34	20	008 88
13	977 79	21	013 32
14	982 23	22	017 76
15	986 67	23	022 21
16	991 12	24	026 65
17	995 56	25	031 09
18	0.000 000 00	26	035 53

A Gaertner measuring engine was used to measure the spectrograms and the distances between the fiducial lines on the measuring bar. The pitch of the screw is approximately 0.5 mm, and the least count of the divided head is  $1\mu$ . The errors of the screw were investigated by measuring, on successive parts of the screw, two overlapping 9 mm distances between fine lines on a glass plate manufactured by the Zeiss Company. As both these distances were within less than two divisions of a whole number of turns at all stations, errors of one turn did not enter. The settings were determined with parallel hairs in the eye piece of a three hundred power microscope. The algebraic sums of the residuals from the mean of the 9 mm lengths for successive intervals were plotted against the corresponding 9 mm stations on the screw. The errors so determined all lay within  $0.1\mu$  of a smooth curve. The errors in one turn were found at four stations, equally distributed along the screw, by measuring a 0.1 mm interval on the glass plate for successive fifths of the head. These errors are nearly sinusoidal and therefore are eliminated in the mean of three measurements started from three stations on the head, each of which is separated from the others by one third of a turn. As only relative linear measurements are involved in the displacement method, an absolute calibration of the screw was not made, although its units are termed millimeters throughout this paper.

For comparing the measuring bar with the screw the spectrometer microscope was used. The reliability of setting on the lines was found from the maximum variation between settings occurring in each of 143 groups, each group consisting of two to four settings on a line. The maximum variation of setting in one group was  $0.9\mu$ , and the average about  $0.25\mu$ . Table III gives the frequency of occurrence of the variations of different amount, from  $0.1\mu$  to the maximum, for all the groups.

TABLE III. Frequency of occurrence of large and small variations in setting.

Frequency of occurrence of a											
certain variation	25	28	35	23	14	6	8	1	1	2	0
Amount of variation in a group $\mu$	.0	. 1	.2	.3	.4	. 5	.6	.7	.8	.9	1.0

The maximum amount that any setting differed from the mean in any of the 143 groups was  $0.5\mu$ . Thus it appears that, when the plate-holder is displaced by a certain interval on the bar, the chance will be very small that the actual displacement will differ from the measured value of the interval by as much as  $1\mu$ , in spite of the fact that the end positions of the interval are determined only once. Such an error in the displacement would cause an error of 0.07'' in the glancing-angle under consideration.

The following procedure was adopted for obtaining spectrograms.  $8 \times 10$  plates were cut in strips of the correct size and "normalized,"\* the edge strips being discarded; after drying, the edges of the gelatin were carefully bevelled by cutting with a sharp knife blade, inclined at a slight angle with the plane of the plate, to remove irregular frills. A plate having been placed in the plate-holder, and the rubber tubes inflated, the holder was mounted on its carriage, with a lead screen so placed as to shield the plate from the lower half of the direct beam. Then the carriage was moved to that one of the desired positions which was most remote from the crystal and adjusted until, when locked, the setting of the microscope cross-hairs on the corresponding fiducial line of the bar appeared exact. After the lapse of at least twelve hours, the setting of the microscope was again scrutinized, but in no case was it necessary to readjust it.

With thermal equilibrium thus assured, the spectrogram was taken. First, with the divided circle set so that the crystal holder did not intercept the incident beam, a short exposure was made to record the upper half of the central image. Then the spectral line was taken on both sides of the direct beam, equal exposures being given for each of five angular positions of the crystal symmetrically spaced about the best setting on each side and covering a range of 18"; this was repeated for each desired position of the plate-holder. The tangent screw was disengaged from the circle table before making an exposure to avoid unnecessary stress on the cone bearing, and the microscope setting was checked before and after the set of exposures for each spectral image. During each exposure, the temperature of the crystal was recorded from the reading on a calibrated mercury thermometer having five divisions per degree centigrade; this thermometer was so held by a clamp that its bulb was three centimeters from the crystal. After the last spectral image exposure the plate-holder was moved (when necessary) to that station which had been nearest the crystal, and a short exposure was made for the central image with the lead screen moved, and the crystal turned so as not to interfere with the direct beam. The plate-holder was then given a small transverse displacement, and the central image again taken for convenience in measuring the separation of the portions of the central images taken with the holder in its two extreme positions.

During exposures, the current through the x-ray tube was approximately 4 m.a.; and the filament current was regulated so as to maintain a brush

\* By normalizing we mean soaking the unexposed plate in water for thirty minutes, then in strong ethyl alcohol for twenty minutes and finally leaving it to attain equilibrium in a humid atmosphere. The reasons for subjecting the plates to this process are fully discussed in another paper.<sup>14</sup> discharge, with only occasional sparking, between the blunt points of a six inch spark gap in parallel with the tube. No attempt was made to get a more exact measure of the potential, but these operating conditions assured spectral images of ample density with exposures of moderate duration.

Table IV gives the detailed data recorded during the taking of a typical spectrogram, plate AG2, and Fig. 6 is a drawing to scale of this spectrogram.

TABLE	I٧	Γ.	Typical	exposure	record.
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Plate AG2. Line Ag  $K\alpha_1$ . Crystal Ca 1.  $S_1$ —axis 35.5 cm. Plate—axis 32.7, 4.8, and 20 cm.  $S_1$  and  $S_2$ —0.02 mm. Spark gap 6 in. Tube current 4 m.a. Rheostat button 3. Filament current 3.7 amps. Set up 5 P.M. May 3, 1928. Exposures started May 4, 9:15 A.M., finished 11:45 A.M.

Exp. No.	Micr. set on plug	Cry sett Deg.	stal ing Div.	Tempe Crystal °C	rature Anode °C	Air pressure plate-holder lb./sq. in.	Exp. time min.
1	11	out*					2
2	11	343.0 " "	$6.0 \\ 7.0 \\ 8.0 \\ 9.0 \\ 10.0$	24.7 24.7 24.7 24.9 24.9	28.0 27.2 29.0 28.2 28.0	27.2 27.2	6 6 6 6 6
3	11	152.0 "	$\begin{array}{r} 439.0 \\ 440.0 \\ 441.0 \\ 442.0 \\ 443.0 \end{array}$	25.0 25.1 25.2 25.2 25.2	$28.0 \\ 28.0 \\ 28.0 \\ 28.1 \\ 28.1 \\ 28.1$	27.1 27.0	6 6 6 6 6
4	0	152.0 " "	$\begin{array}{r} 439.0 \\ 440.0 \\ 441.0 \\ 442.0 \\ 443.0 \end{array}$	25.3 25.2 25.3 25.3 25.3 25.3	27.8 27.4 27.7 28.0 28.1	27.0 26.9	3 3 3 3 3
5	0	343.0 " "	6.0 7.0 8.0 9.0 10.0	25.3 25.4 25.4 25.4 25.4 25.4	28.128.028.128.228.3	26.9 26.9	3 3 3 3 3
6	6	343.0 " "	6.0 7.0 8.0 9.0 10.0	25.5 25.5 25.5 25.5 25.5 25.6	28.2 27.9 28.2 28.3 28.5	26.9 26.9	5 5 5 5 5
7	6	152.0 " "	$\begin{array}{r} 439.0 \\ 440.0 \\ 441.0 \\ 442.0 \\ 443.0 \end{array}$	25.6 25.6 25.6 25.6 25.7	28.6 28.5 28.7 28.7 28.8	26.9 26.6	5 5 5 5 5
8	0	out*					1
9	0	out* (pla	ite-holder di	splaced sidev	ways 2 mr	n)	1

Remarks: Developed 4 min at 77°F, fixed 20 min, washed 30 min, rinsed in distilled water 10 min, and soaked in alcohol 20 min at 77°F, then dried in a humid atmosphere. \* Turned to give direct transmission.

After the plates were thoroughly dry, rulings were scribed at intervals on the gelatin surface (the dotted lines in Fig. 6) along a line through the centers of, and normal to, the spectral images, and all measurements were made along this line except those of the central image separation. If the spectral images were not filled out and black, they were discarded. Table

				1)	77 D	
		Top Up		_	Top Down	
А	162.2487	162.0819	161.9182	23.7314	23.5627	23.4006
	92	.0793	89	.7270	20	.3982
	78	.0809	67	.7290	26	.4004
Меап	162.2486	162.0807	161.9179	23.7291	23.5624	23.3997
	(20.2°C)	(20.0°C)	(20.0°C)	(20.0°C)	(20.4°C)	(20.2°C)
В	138.5332	138.3640	138.1996	47.4476	47.2782	47.1170
	14	50	.2004	78	95	57
	33	48	.2000	93	89	63
Mean	138.5326	138.3646	138.2000	47.4482	47.2789	47.1163
	(20.1°C)	(20.0°C)	(20.0°C)	(20.0°C)	(20.2°C)	(20.2°C)
С	110.0780	109.9086	109.7470	75.9033	75.7361	75.5720
	64	.9104	84	40	61	13
	77	.9104	76	28	41	16
Меап	110.0774	109.9098 (20.1°C)	109.7477 (19.8°C)	75.9034 (20.1°C)	75.7354 (20.4°C)	75.5716 (20.3°C)
D	92.1552	91.9880	91.8245	93.8269	93.6567	93.4956
	69	74	40	78	84	52
	55	78	51	70	79	56
Mean	92.1559 (19.9°C)	91.9877	91.8245 (19.8°C)	93.8272 (20.0°C)	93.6577 (20.4°C)	93.4955
E	63.7039	63.5360	63.3716	122.2768	122.1082	121.9471
	60	59	20	68	77	79
	59	60	29	88	81	80
Mean	63.7053	63.5360	63.3722	122.2775	122.1080	121.9477
	(19.8°C)	(20.2°C)	(19.6°C)	(20.2°C)	(20.4°C)	(20.4°C)
F	39.9994	39.8311	39.6623	145.9828	145.8144	145.6543
	90	18	37	10	66	.6498
	70	03	48	12	31	.6523
Mean	39.9985	39.8311	39.6636	145.9817	145.8147	145.6521
	(19.6°C)	(20.2°C)	(19.6°C)	(20.4°C)	(20.4°C)	(20.4°C)
L	102.8114	102.6425	102.4800	83.1689 .1710	83.0037	82.8415
Mean	102.8114	102.6425	102.4800	83.1699	83.0037	82.8415
Ν	101.1121	100.9447	100.7813	84.8668 86	84.7000	84.5386
Mean	101.1121	100.9447	100.7813	84.8677	84.7000	84.5386
L	108.1553	107.9860	107.8243	82.8453	82.6748	82.5122
М	106.4640	106.2956	106.1320	84.5346	84.3655	84.2016

TABLE V. Plate AG2. Engine settings in millimeters.

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V gives the details of the measurements of the spectrogram illustrated. Before the value of the glancing-angle can be computed, the measured distances must be increased by the following quantities:  $a \equiv$  screw correction;  $b \equiv$  correction for difference between temperatures of plate when taken and



Fig. 6. Facsimile of spectrogram AG2.

when measured;  $c \equiv$  lateral shift of plate-holder during displacemente as indicated by central image separation. These corrections together with the corrected values of the distances between spectral images and their corresponding displacements are given in Table VI. The values of the angle

Dist Fiş	ance g. 6	a× (10)⁴mm	Mean Temp Diff. δ°C	o. b× (10)⁴mm	c× (10)⁴mm	Image separation corrected	Displacement mm
1.0	Up	+6.8	5.05	0.7	72	F2 16406	
A-C	Down	+0.7	5.05	-9.1	-13	52.10400	279.44590
	Up	+1.3	5 05	-9.7	172	52 16279	$\pm 0.00042$
D-F	Down	+4.7	5.05		+75	52.10578	
B C	Up	+4.0	5 25	5.6	10	28 45062	
B-C	Down	+1.6	5.35	-5.0	-40	28.45002	152.42506
	Up	+0.3	5 25	5 6	1.40	20 45471	$\pm 0.00020$
D-E	Down	+1.0	5.55	-5.0	+40	28.45471	

TABLE VI. Plate AG2. Corrections to measurements.

Differential thermal coefficient glass plate-screw 3.7 (10)<sup>-6</sup> per °C.

 $\omega$  between the normal to the plate and the line XX', computed by Eq. (2) from the data corresponding to the two different displacements, differ in magnitude and sign; but the more precise optical tests had shown very conclusively that the maximum rotation of the plate-holder, for any displacement along the tracks, was less than 2". Such differences are, there fore, probably due to residual fortuitous variations in position of a developed image with respect to its corresponding latent image,<sup>14</sup> and the fact that commercial plates cannot be forced flat.

Table VII is given as an illustration of the accuracy with which the spectrograms could be measured. As shown in Table V, the distances be

- <sup>9</sup> A. Leide, Dissertation, Lund (1925).
- <sup>10</sup> A. P. Weber, Zeits. f. wiss. Photo. 23, 149 (1925).

<sup>11</sup> K. Lang, Ann. d. Physik [4] 75, 489 (1924).

12 G. Kellström, Zeits. f. Physik 41, 516 (1927).

tween pairs of lines were measured first with the plate in one position on the engine, and again when it was reversed ("top up", "top down"). The numbers tabulated are the differences between the means of these measurements in each position. The first three plates had lines corresponding to

Spectro- gram	Spectro- gram	Differences		Spectro- gram	Differ	ences
	11 in. displ.	6 in. displ.		11 in. displ.	6 in. displ.	
AG1 AG2	0.25μ 0.20μ	0.29µ 0.44µ	AG3 AG4	0.56μ 0.81μ	0.04μ	

TABLE VII. Difference between measurements direct and reversed.

both 11 and 6 in. displacements, but the fourth had only the former. The measurements on plate AG4 were repeated. If the new measurements are averaged with the first set, the corresponding difference is 0.72 instead of  $0.81\mu$ . If it is assumed that the measurements can be in error by as much as  $0.81\mu$  in the case of the 11 in. displacement, the resulting glancing-angle would be in error by 0.29''. The maximum effect on this angle due to the uncertainty of setting on the bar is 0.07''. Therefore it seems evident that there is a negligible chance that values of the glancing-angle obtained from different 11 in. displacements can fluctuate, due to errors of measurement, by as much as 0.36'', assuming of course good lines on the spectrograms. Remeasurements made after a lapse of three months on another plate, AG5, by an independent observer gave glancing-angle values differing for the 5 and 6 in. displacements by 0.13'' and 0.00'' respectively.

 TABLE VIII. Effect of method of measurement. Plate No. AG9, 4 in. displacement, taken at 22.1° C. glancing-angle at 18° C.

			and the second
Measured at ° C.	Uncorrected for temp. of meas.	Corrected for temp. of meas.	Description of method of measurement
20.5	5° 17′ 14.54″	5° 17′ 14.42″	Deep focus objective; 57° cross-hairs; 18 X.
21.2	5° 17′ 14.41″	5° 17′ 14.34″	Same objective; parallel hairs separated to straddle width of remote lines; 18 X.
19.7	5° 17′ 14.65″	5° 17′ 14.48″	Shallow focus; parallel hairs same; 20 X.
Remote 17.0 Near 19.0	5° 17′ 14.95″	5° 17′ 14.43″	Same objective, but parallel hairs suitably adjusted to measure separation of remote and near lines independently; 20 X.

Spectral images taken with the plate in a remote position are broader than those taken with it near the crystal; therefore, if parallel hairs of fixed separation are used in the measuring microscope, their adjustment will not 106

be suitable for both types of images. Plate AG9 was measured under varying conditions of magnification, type of eye-piece and temperature, and Table VIII gives the glancing-angles thus obtained. It will be observed that the second column shows excellent agreement for different methods of measurement, especially when it is borne in mind that for such a short displacement a variation in the image separation of  $0.2\mu$  would change the glancing-angle by 0.20'', and that the near and remote lines were of different width.

# PART IV. RESULTS

The final results are given in Table IX. They were obtained from eight spectrograms, six of which were taken with the plate-holder in three, the remainder in only two, positions, but that nearest the crystal was the same for all. All the plates had been cut from the central portions of large plates; AG1 to AG4 from one Eastman "40," AG5 to AG7 from another, and AG9

Spectro- gram	Displ't inches	Taken at ° C	Glancing- angle at 18° C	Mean values, probable errors of mean and of one determination respectively
AG1 "2 "3 "4	11 11 11 11	25.2 25.1 25.0 25.2	5° 17′ 13.68″ 14.00 13.53 13.95	$\theta = 5^{\circ} \ 17' \ 13.79''$ $R = \pm 0.075$ $r = \pm 0.15$
" 1 " 2 " 3 " 5 " 6 " 7 " 5 " 6 " 7 " 9	6 6 6 6 5 5 5 4	25.4 25.3 21.4 21.8 21.7 21.4 21.7 21.4 21.7 21.9 22.1	$\begin{array}{c} 13.35\\ 13.78\\ 13.92\\ 13.03\\ 13.93\\ 14.13\\ 14.15\\ 13.43\\ 14.10\\ 14.42\\ \end{array}$	$\theta = 5^{\circ} \ 17' \ 13.82''$ $R = \pm 0.092$ $r = \pm 0.29$

TABLE IX. Final data and results. Line; Ag (Ka1). Crystal; Calcite (Ca 1).

from an Eastman "Universal." If the values of the glancing-angle corresponding to eleven inch displacements are given twice the weight of the others, the following values are obtained:

$$\theta = 5^{\circ}17'13.81''(\pm 0.06'')$$
 at  $18^{\circ}C.^{1}$ 

 $\lambda = 0.558238A^{1}(\log 2d_{18} = 0.7823350)$ 

 $\pm$  0.000002.

For comparison, the most reliable published results that we have seen are given with ours in Table X. In cases where the probable errors were not published we have calculated them from the data given. Omitting Kellström's second order value and weighting the others according to their probable errors,

 $\theta = 5^{\circ}17'14.66''(\pm 0.036'')$  at  $18^{\circ}C$ .

Orders	Clansing angle	Probab	le error of	Av. deviation	Source
used	Glancing-angle –	Mean	One det'n	from mean	Source
1 1 & 2 1	5° 17′ 13.05″ 7.8 13.1	0.4" 0.7 0.6	0.8" 2.0 1.5	1.0" 2.2 1.8	A. Leide <sup>9</sup> A. P. Weber <sup>10</sup> K. Lang <sup>11</sup>
1 2	15.2 (10 37 9.8)	$\begin{array}{c} 0.045\\ 0.36\end{array}$	0.12 0.72	0.13 0.8 11 in. displ't 0.2	G. Kellström <sup>12</sup>
1	13.81	0.06	0.13	Other displ't 0.34	Present work

TABLE X. Comparison with results of others.

### DISCUSSION

Of the values given in the table, Kellström's first order determination has the smallest probable error and is the one quoted in the International Critical Tables. The value he gives for the wave-length determined from second order reflection would predict a first order glancing-angle (5° 17'  $14.58'' \pm 0.18''$ ) approximately midway between his measured first order and ours and with four times his first order probable error. The fact that our value of the glancing-angle differs from his two values, and the latter differ among themselves, by about three times the sum of the respective probable errors would lead one to suspect that any one or all of the values may be affected by a constant error. From the point of view of agreement, the two sensibly identical values of Leide and Lang, large though their probable errors may be, favor our value rather than those of Kellström. The "Method of Displacement" may be subject to an error sufficient to account for our disagreement with Kellström's accepted value if the underlying assumption regarding the symmetry of the reflected beam is not fulfilled. However, the mean value of the glancing-angle computed from the eleven inch displacements only, where the plate was near a Bragg focus in both its positions, is sensibly the same as that from other displacements, where the plate intersected an intermediate portion of the beam in one position (Table IX). This fact confirms our belief that the reflected beam possessed the required symmetry, in spite of the fact that the crystal was not a perfectly selective reflector. The exhaustive tests to which the apparatus was subjected failed to show any defect which could cause an appreciable constant error. The only remaining source of such an error seems to be the crystal. The discrepancy between the results would be accounted for by a difference in grating space of seven parts in a hundred thousand between the crystals used. We have made use of only one specimen of Iceland spar while Kellström has used two (Pk 1 and Pk 2), although most of his data were obtained from Pk 1. He states that both crystals gave the same result within his limits of error. However, in the one case in which he used Pk 2 for a second order determination of the wave-length considered here his result is identical with that of our eleven inch displacements. We have been unable

to find any statement by Leide or Lang giving information concerning the particular specimens of calcite used by them. DeFoe and Compton<sup>13</sup> have measured the density of specimens of calcite, obtained from various sources, within a probable error of  $(10)^{-4}$  gm cm<sup>-3</sup>. They give values for two specimens from Iceland which differ by thirteen times this amount. indicating a real difference in density, even though they allow only half weight to the value for one of these specimens in computing the mean density of all the specimens. If all variations in density are reflected in differences of grating space, a difference in density of  $6 \times (10)^{-4}$  gm cm<sup>-3</sup> (less than half that found by DeFoe and Compton) between Kellström's crystal and ours would be sufficient to account for the difference in the values of the glancing-angle under consideration. The discrepancy might therefore be explained in this way, though it is doubtful that the observed differences in density are really due to variations in grating space.

Displacements	11 in.	6 in.	5 in.	4 in.
Average variations due to:				
Variations in setting on bar	0.034"	0.06″	0.07″	0.10'
Unflatness of gelatin	0.17	0.31	0.37	0.50
Motions of images after exposure				
if plates are:				
normalized	0.25	0.46	0.56	0.69
not normalized	1.00	1.84	2.24	2.76
Sum of average variations.				
If plates are:				
normalized	0 45	0.83	1 00	1 29
not normalized	1 20	2 21	2 68	3 36
not normanzed	1.20	2.21	2.00	5.50
Observed average variations	0.20	0.33	0.30	
Observed maximum variations	0.47	1.10	0.72	

TABLE XI. Average variations in measured values of glancing-angle due to various causes.

It is of interest to consider the effect on the final result of various causes of uncertainty in the directly measured quantities. The average difference between successive settings on the bar was  $0.25\mu$ , therefore any displacement of the plate-holder is subject to an average uncertainty of  $0.5\mu$  from this cause. Irregularities in the surface of the gelatin would cause the effective displacement to differ from the actual. An interferometer investigation of the gelatin surface of the developed spectrograms, when held in the plateholder, showed that these irregularities could cause an average error in the displacement of  $2.5\mu$ . Our work on the unreliability of position of photographic images<sup>14</sup> showed that the average error in the measured value of a fixed distance, as recorded on photographic plates, was  $0.7\mu$  when the plates had been normalized and  $2.8\mu$  when they had not. These errors, of course, include both the error of setting and that due to motion of the image. Though the images of the template slits<sup>14</sup> could be set on more accurately than

<sup>13</sup> O. K. DeFoe and A. H. Compton, Phys. Rev. [2] 25, 618 (1925).

<sup>14</sup> D. Cooksey and C. D. Cooksey, Phys. Rev. preceding article.

spectral images, we shall assume that these figures include the average variations that might be expected in the remeasurement of a spectrogram. The effect of the average variation due to each of these causes on glancing-angle values, calculated for various displacements, is shown in Table XI.

These statistics not only emphasize the advisability of normalizing photographic plates before using them as accurate recorders of distances, but suggest that, even for small glancing-angles, the accuracy of the displacement method would be improved by the use of emulsions coated on good *plate* glass. With the accuracy of setting on spectral images limited as it is, there would seem to be no necessity for resorting to more accurate methods of measuring the displacement. Even though the apparatus is massive and so has a large thermal capacity, there is little doubt that better precautions for maintaining it at a constant temperature would have a beneficial effect. However, the manipulation in the present work was carried out with special reference to making the effects of temperature changes fortuitous.

In conclusion we wish to express our great appreciation of the encouraging and helpful interest shown, during the whole progress of this work, by Professor Uhler and Professor Emeritus Hastings of this department. We are also grateful to Dr. Albert W. Hull of the Research Staff of the General Electric Company for advice in connection with x-ray tubes and filaments, and to Professors McKeehan and Uhler for helpful suggestions in connection with the presentation of this investigation. In directing the construction and exhaustion of x-ray tubes, we have been greatly aided by experience gained in our long association with Mr. Alfred Greiner, formerly connected with this laboratory.