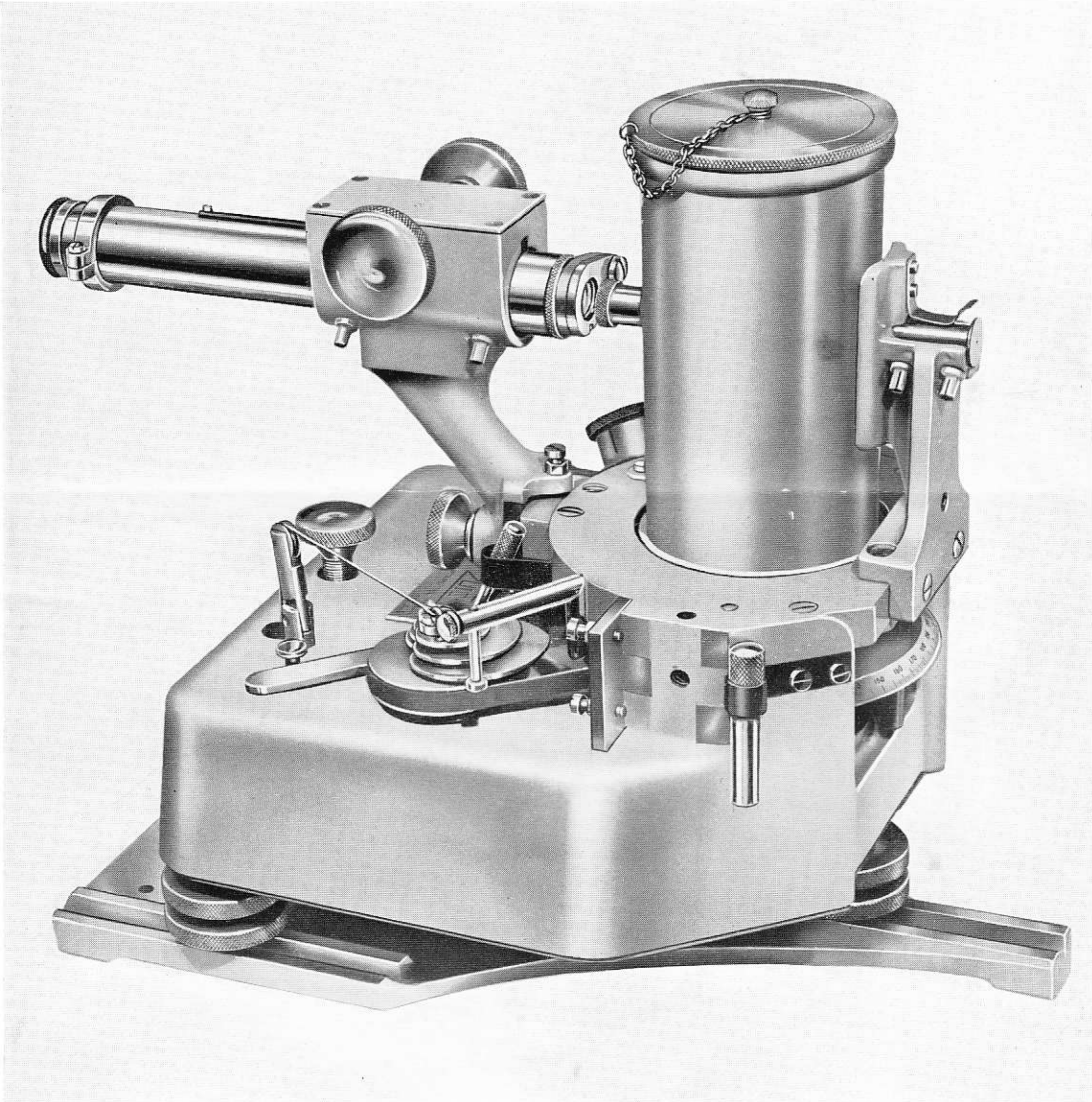
UNICAM INSTRUMENTS LTD.

CAMBRIDGE

S.25

SINGLE CRYSTAL GONIOMETER



UNICAM INSTRUMENTS LTD., YORK STREET, CAMBRIDGE, ENGLAND

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GENERAL DESCRIPTION OF THE STANDARD INSTRUMENT

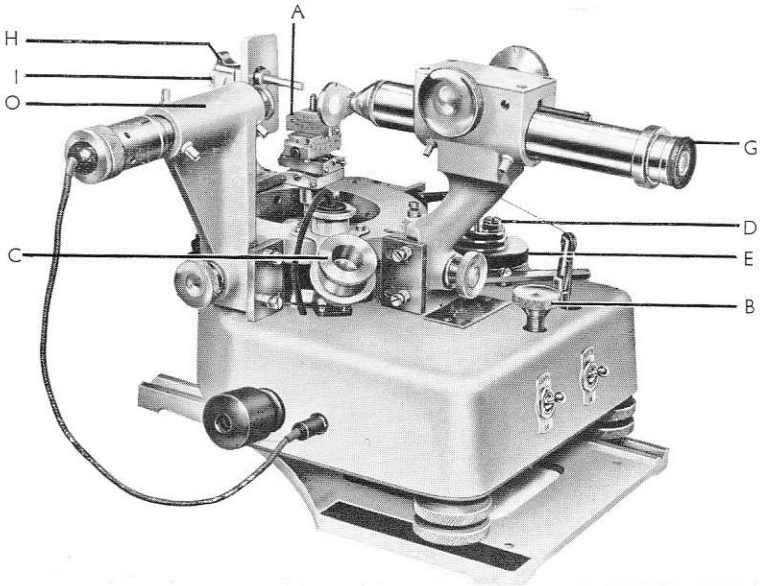


FIG. 1

This instrument has been designed primarily as an oscillation or rotation X-ray goniometer. The goniometer head A (Fig. 2) is fitted with two slides and two arcs and can be detached from the supporting spindle by turning a screw. The arcs are graduated in 2° intervals with a vernier and are rotated by a worm operated by a key. The main spindle can be raised or lowered by the screw B (Fig. 1) on the base of the instrument. A 6″ diameter silver bronze circle, graduated in degrees and read by a vernier to 3′ is mounted in the base and is coupled by a spline to the spindle. A small lamp illuminates the scale and it is read by a lens, C. The synchronous electric motor used for oscillating or rotating the spindle is mounted in the base. The shaft of this motor projects through the base and on it is mounted a set of heart-shaped cams D (Figs 1 and 3) and a pulley E. The cams are cut accurately by a mechanical generator to such a form that the crystal oscillates at a strictly uniform velocity through 5°, 10° or 15°. Which of these cams is in action depends on the position to which a small follower is set in the arm F (Fig. 3). Alternatively the crystal may be rotated continuously by a belt which passes round the pulley E and also round a similar pulley which is on the axis of the spindle. The belt is loose when oscillations of the crystal are taking place but is pressed against the pulleys by a small jockey pulley when complete rotation of the crystal is necessary. The arm F must, of course, be pulled off the spindle to allow the crystal to rotate continuously in the same direction.

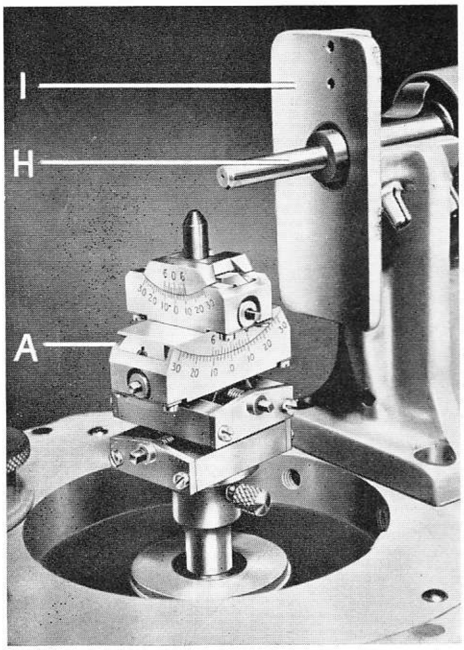


FIG. 2

A tele-microscope G (Fig. 1) is provided for orientating the crystal on the goniometer head. When used as a microscope it has a focal length of 2 inches and by moving a front lens it can be converted into a telescope. There are three positions on the base at which the tele-microscope may be fixed; one in which the axis of the tele-microscope is parallel to the collimator (as shown in Fig. 1) and two in which it is perpendicular to that direction, one to the right and one to the left (as shown in Fig. 3). The tele-microscope is carefully adjusted before despatch but if it should get out of adjustment it can be re-aligned by means of several adjusting screws.

The X-ray collimators H (Figs. 1, 2 and 3) are of two types each having three cylindrical apertures and the same external dimensions. In type 1 the hole furthest from the crystal is 1 mm. in diameter, the middle hole is $\frac{1}{2}$ mm. in diameter and the one nearest to the crystal is again 1 mm. in diameter. The middle hole is not midway between the outer apertures but is nearer to the crystal than the mid-point of the collimator. In this way, the intensity of X-ray beam is as great as possible consistent with a given size of X-ray spot on the film. The aperture near the crystal does not limit the size of X-ray beam but catches any radiation scattered from the middle aperture. In type 2 the diameters of the aperture in the collimator are $\frac{1}{2}$, $\frac{1}{4}$ and $\frac{1}{2}$ mm. respectively and their relative positions are the same as in type 1. The collimator is carried on the support I (Figs. 1, 2 and 3) which is screwed to the base.

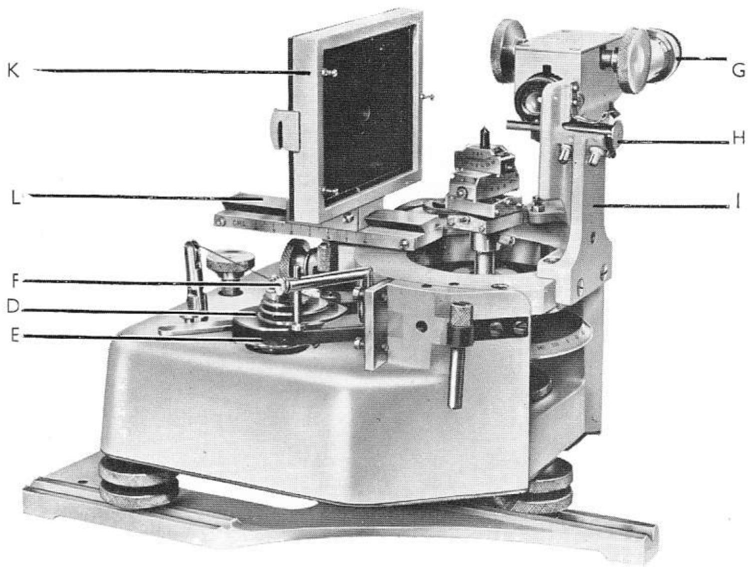


FIG. 3

The cylindrical camera J (Fig. 4) is of radius 30.0 mm.* and takes a film 12.4×17.5 cm. It is supported on the base by hardened steel pieces which have been machined to bring the axis of the cylinder co-axial with the spindle. There are two holes in the side of the camera diametrically opposite to one another. Into the larger hole the collimator projects and into the other fits a small brass tube with a flange. This brass tube prevents X-rays or light rays from fogging the film at the point where the direct beam escapes from the camera. The film is punched by a special punch with one small hole. The brass tube is pushed into this hole and a pair of tweezers slipped over the film to hold the brass tube. It is then a simple matter, after a little experience, to make the brass tube enter the appropriate hole in the camera and so hold the film in its correct position as well as protect it from fogging at that place. The ends of the film are also protected from light fogging by rings which have bevelled grooves to fit over the film, or films, if more than one is used at a time.

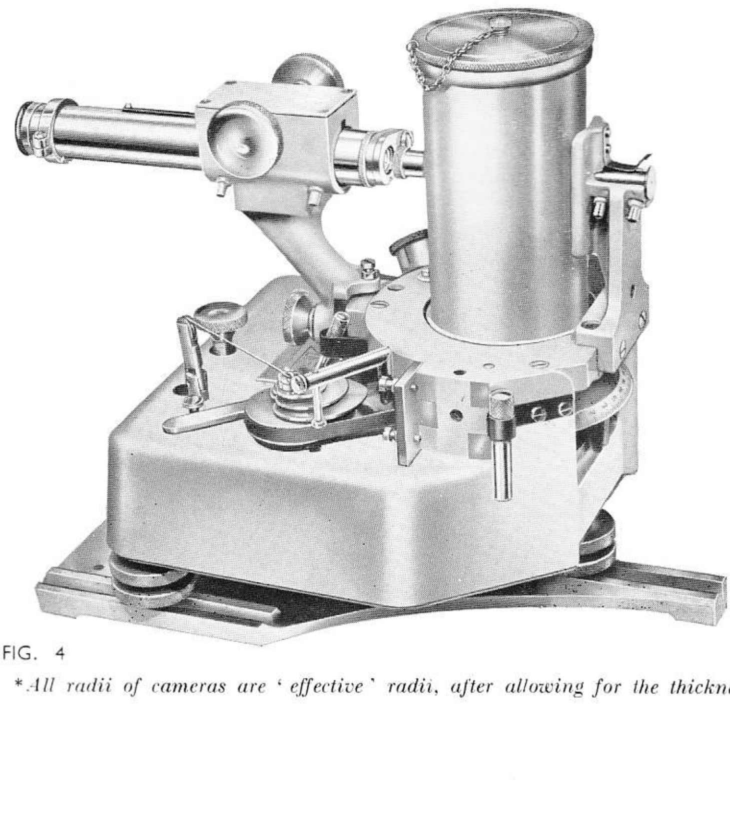


FIG. 4

ACCESSORIES

The components so far described constitute the standard instrument but a number of additional parts are available which greatly extend its usefulness. With the standard instrument the following types of photograph can be taken on the cylindrical camera: oscillation, rotation, Laue, powder, preferred orientation. However, Laue photographs are in general best taken on flat cameras and 30 mm. is too small a radius for many powder photographs.

* *All radii of cameras are 'effective' radii, after allowing for the thickness of X-ray film and black paper.*

FLAT PLATE CAMERAS

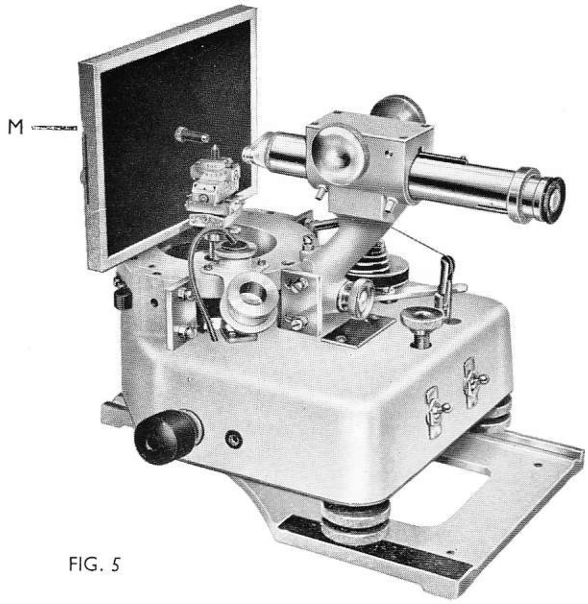


FIG. 5

1. TRANSMISSION OR SIDE-REFLECTION (S.26)

The flat plate camera K (Fig. 3) takes a film or plate, $3\frac{1}{4}'' \times 4\frac{1}{4}''$, which is mounted behind black paper and pressed against a frame adjusted to be accurately perpendicular to the X-ray beam. The frame of the camera is carried in a slide L and it may be set at any distance from the crystal between 40 mm. and 100 mm. The slide is screwed on to the base in place of the tele-microscope and may therefore be set either parallel or perpendicular to the X-ray beam. In the second position a side-reflection photograph would be obtained. Such a camera is almost essential in the study of the symmetry of crystals because only on a flat plate may the presence of the axes and planes of symmetry be recognised easily. It also has great value for taking oscillation photographs when a large crystal to film distance is necessary and only the part of the record near the centre is of interest.

2. BACK REFLECTION CAMERA (S.31)

The back reflection camera M (Fig. 5) takes a flat film $6\frac{1}{2}'' \times 4\frac{3}{4}''$ which must be punched so as to make it possible to pass the X-ray collimators through it. The frame of the camera is mounted in a special support which replaces the usual collimator holder. The crystal to film distance is 30 mm. This camera is invaluable when a reflection is required at normal incidence from a thick crystal specimen. It may be used for the study of crystallographic orientation or symmetry when the transmission method cannot be employed owing to the absorption of the specimen. It may also be used for the study of metal blocks which are so large that only a back reflection method is applicable.

3. RADIAL CAMERAS 5·73 cm. and 11·46 cm. (S.26A and B)

These cameras N, O (Figs. 6 and 7) are hemi-cylindrical and take films size 17.5×12.4 cm. Special punches are required to make the exit apertures in the correct position in each film. These cameras are mounted in the same way as the tele-microscope. Light tight end-pieces are provided to prevent fogging. These cameras are useful when only the X-rays scattered forwards and sideways are of interest and when the spots occur too close together on a camera of radius 30.0 mm. Such cameras therefore have special application to many biological crystals and others having large cell dimensions.

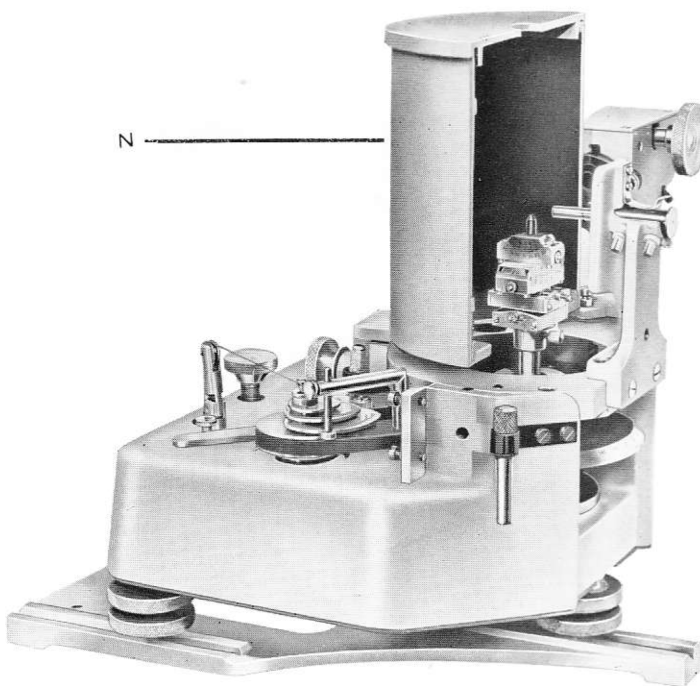


FIG. 6

4. OPTICAL COLLIMATOR (S.27)

An optical collimator O (Fig. 1) provided with its own lamp, which is fed with current from the transformer in the base of the instrument, can be mounted in any one of the positions normally occupied by the tele-microscope. The axis of the collimator may therefore be set in a position either perpendicular to the axis of the telescope or parallel to it. In the former arrangement (see Fig. 1) the whole instrument constitutes a single circle optical goniometer and can be used for morphological studies of crystals, or for orientating a specimen prior to taking an X-ray photograph. In the arrangement with the axes of the collimator and telescope parallel to one another any observations in polarised light which might be required can be made with the addition of polaroids.

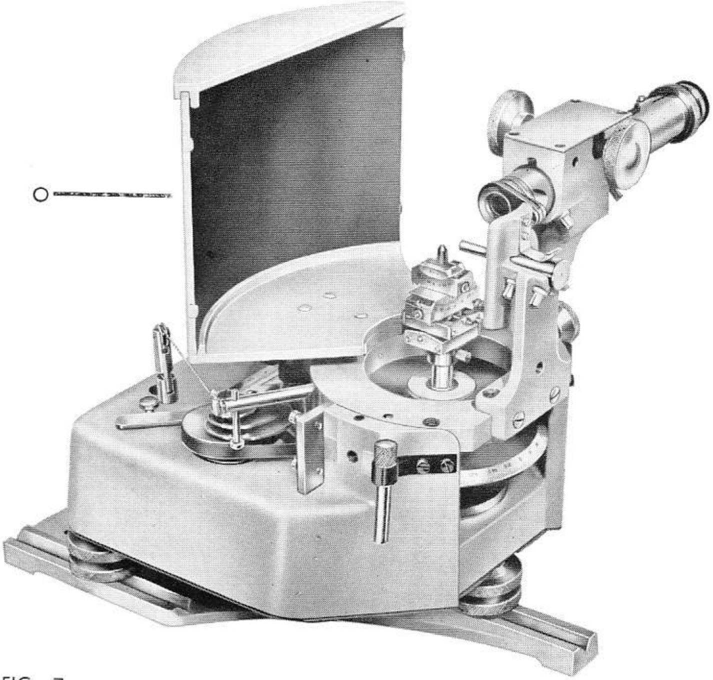


FIG. 7

5. POLARISING ATTACHMENT (S.27A)

A special accessory designed by Dr. M. F. Perutz is also available for observations in polarised light. This provides the means for the orientation of single crystals optically and is particularly useful for crystals having poorly developed edges and faces.

6. FILM PUNCHES (S.28)

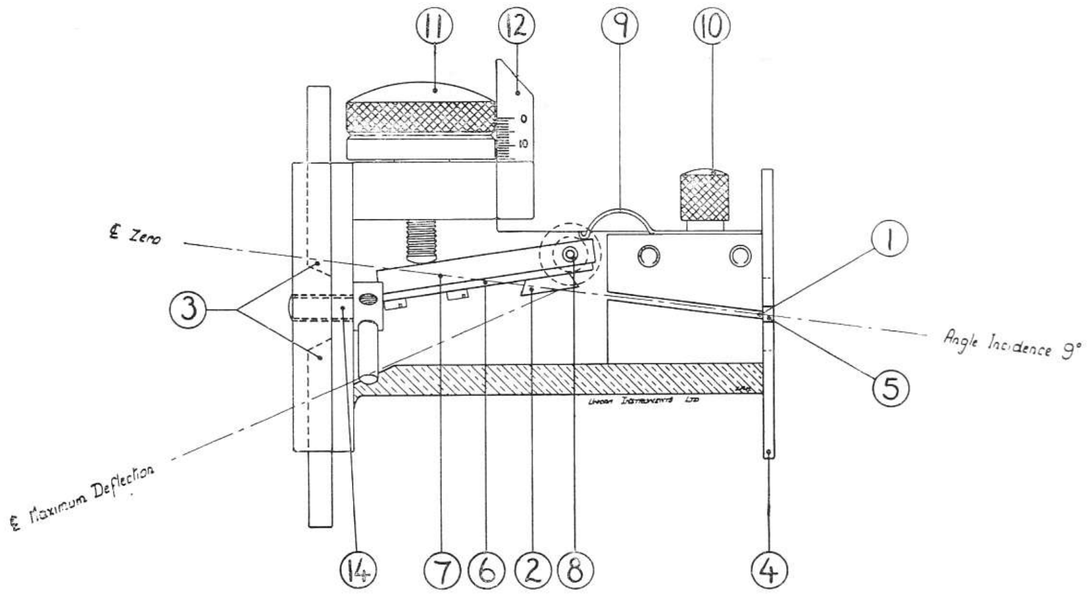
A film punch is necessary for all cameras except the flat-plate camera (S.26). A hole must be punched accurately in the film to locate it in the camera and also to allow the passage of the direct X-ray beam. A short metal tube is pressed into the hole in the film and into a corresponding hole in the camera. A flange on the tube prevents fogging by either stray light or X-rays at the point of entry. The punches carry locating pegs to position the film accurately for the appropriate camera and operation is very simple.

7. TWEEZERS (S.29)

Cameras of diameter 60 mm. and less are too small to load conveniently without tweezers. A scissor-like instrument consisting of two pieces of brass pivoted about their centre and fitted with accurately designed end-pieces makes camera loading a comparatively simple operation with a little experience.

The fullest possible instructions are included in the operating manual supplied with each instrument.

S.30 MONOCHROMATOR FOR X-RAYS



This instrument, which is a development of a design by Dr. I. Frankuchen, consists essentially of a slit (1) which limits the beam of X-rays falling on a crystal (2) from which the monochromatised beam is reflected through the adjustable slits (3). The monochromator is supported on the X-ray tube by a circular plate (4) which has a rectangular aperture (5). The crystal is attached by wax to a thin plate (6) which is fixed to a brass plate (7) pivoted on steel points (8). A spring (9) is fastened by a screw (10) to the fixed blocks forming the slit (1) and presses the plate (7) into contact with the micrometer screw (11). A head on this screw, which is divided into four parts, enables the angular setting of the crystal to be read off against an index (12), graduated in degrees. The slits (3) can be set to any desired positions and clamped in position by turning the screw (14).

The instrument is adjusted in the following way :—
The crystal is attached to the pivoted brass plate (7)—care being taken to ensure that when the head of the micrometer screw is set to zero the crystal does not intercept more than half of the direct beam passing down the slit (1). The monochromator is now mounted on the X-ray tube so that (with the crystal in zero setting) the maximum amount of radiation, as judged by a fluorescent screen, emerges from it. The pivoted plate is now rotated by turning the micrometer screw (11) until the monochromatised beam is reflected with its maximum intensity. This adjustment may also be made using a fluorescent screen. The setting of the micrometer head does not in general have to be very exact because of the divergence of the beam of X-rays incident to the crystal. The slits (3) are now set to transmit only the monochromatic component of the radiation and are then clamped in position. The plate (4) can be made to any design to suit the type of tube which is in use, but the drawing shows the circular plate normally supplied.

For work requiring a beam which is as parallel as possible, a perfect crystal is necessary. Calcite is perhaps the easiest material to obtain in good crystals though it is not such a good reflector as boric acid, pentaerythritol or di-benzalazine. When an oscillation or Weissenberg photograph is required with monochromatic X-rays it is best to use the strongest reflection almost irrespective of the perfection of the crystal. For this purpose graphite and the three crystals mentioned above as alternatives to calcite are suitable.

SPECIMEN PHOTOGRAPHS

(a) Oscillation Photograph

This is an oscillation photograph of dibromo cholesteryl chloride taken with copper characteristic radiation. The angle of oscillation was 15°. The photograph shows well developed layer lines and the characteristic appearance obtained when the X-rays travel parallel, or nearly parallel, to a long crystallographic axis. The readily adjustable goniometer head makes it easy to obtain straight layer lines.

(b) Laue Photograph

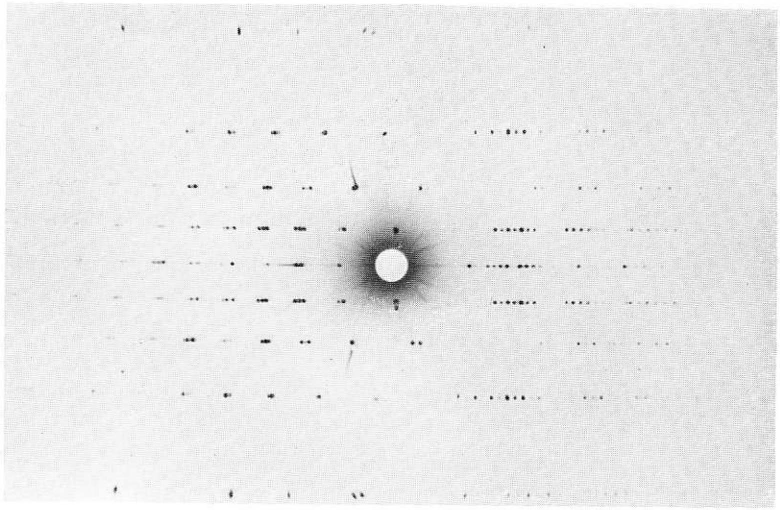
This is a Laue photograph of gypsum taken with the X-rays travelling parallel to a two fold axis. The characteristic ellipses arranged with this symmetry may easily be seen. The sharpness of the spots indicates the good collimation of the X-ray beam and the perfection of the crystal. The divided circle of the goniometer as well as the adjustable goniometer head are essential for obtaining a photograph showing the exact symmetry of the crystal.

(c) Back-reflection Laue Photograph

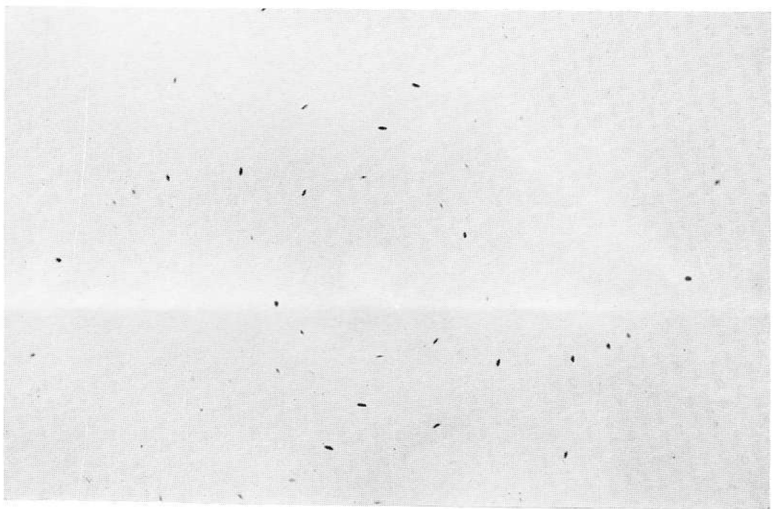
This is a back reflection Laue photograph of quartz. An X-cut oscillator plate was set normal to the X-ray beam and the photograph shows a two-fold axis almost coinciding with the direction of the beam. Such a photograph can readily be used for checking the accuracy with which such an oscillator plate has been cut.

Charts for the interpretation of X-ray photographs

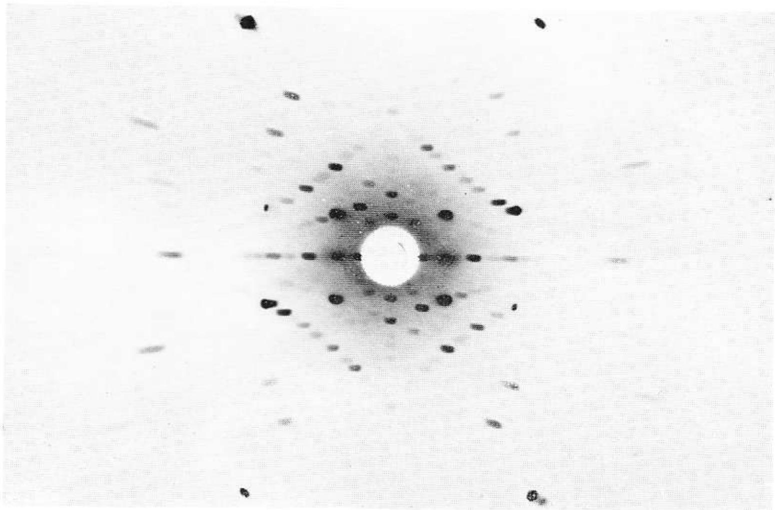
Bernal charts for a cylindrical camera of 30.0 mm. radius are available printed on transparent paper. These give ξ, ζ, values by super-position on the photograph. A chart, also giving ξ, ζ, values, but suitable for a flat plate or film placed 4 cm. from the crystal in the direction of the direct beam is also available.



(a)



(b)



(c)

UNICAM INSTRUMENTS

SINGLE CRYSTAL GONIOMETER

Instrument and Accessories

SPECIFICATION

CATALOGUE NUMBERS

S.25	Single Crystal Goniometer (normally fitted for 200/240 v. 50 c/s. but available on request for other voltages and frequencies), complete with tele-microscope, 6 cm. diameter cylindrical camera and two X-ray collimators, $\frac{1}{2}$ and $\frac{1}{4}$ mm. apertures.
S.26	Plate Camera.
S.26A	5.73 cm. radius Radial Camera.
S.26B	11.46 cm. radius Radial Camera.
S.27	Optical Collimator.
S.27A	Perutz Polarising Attachment.
S.28	Film Punch for S.25 Cylindrical Camera.
S.28A	Film Punch for S.26A Radial Camera.
S.28B	Film Punch for S.26B Radial Camera.
S.28C	Film Punch for S.31 Back Reflection Camera.
S.29	Tweezers.
S.30	Monochromator for X-rays.
S.31	Back Reflection Camera.
S.33	Base and Rails for S.25 Goniometer.
S.34	Spare interchangeable Goniometer Head.

OVERALL DIMENSIONS

Length 14.5″ (37 cm.); Width 8″ (20.1 cm.); Height 11.5″ (29.21 cm.); Weight 27 lb. (13 kilos); Minimum height of beam centre line from table 7.75″ (19.68 cm.).

Finish: Light pearl grey glossy enamel and bright nickel.

WHEN ORDERING:

(1) Please quote catalogue numbers.

(2) Please give voltage and frequency of mains supply.

Full quotations will be sent by return.

As a result of design improvement, current production models may differ in minor detail from this description.

Price List on request

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