Acta Cryst. (1961). **14**, 113

A Quickly Adjustable Vertical Guinier Camera

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(*Received* 22 *January* 1960 *and in revised form* 21 *March* 1960)

A Guinier camera for use with a vertical X-ray tube is described which can be readjusted quickly and easily for radiations of different wavelength. All critical distances and angles are defined by exchangeable spacers that are adjusted once for all. Experience over a period of three years has shown that this method of positioning is sufficiently accurate to make visual or photographic final adjustments after each change of wavelength unnecessary.

This camera was designed for a laboratory in which studies on widely different materials—from plastics to ceramics, ferro-electrics and metals—are conducted at one X-ray source (Philips–Norelco). The varying character of the work necessitated frequent shifts between different radiations. Under such conditions it was clear that, to be used to full capacity, a Guinier camera (Guinier, 1939) would have to meet the following requirements:

(i) vertical position at the X-ray tube (most commercial cameras are designed for horizontal position, with the exception of that described by Hofmann & Jagodzinski (1955));
(ii) quick adjustability for different radiations;
(iii) good reproducibility and high accuracy of adjustments;
(iv) all adjustments to be sufficiently easy and foolproof to allow operation by unskilled personnel; and
(v) no geometrical interference with apparatus at neighbouring tube windows (a back-reflexion Debye and a Laue camera at adjacent windows, and a high-angle diffractometer at the opposite window).

The shortage of skilled laboratory technicians gave paramount importance to condition (iv).

Although similar conditions probably prevail in many industrial laboratories, none of the existing cameras was found to satisfy them all. This may be part of the reason for the comparatively limited use of the Guinier technique in industrial work. Experience in this laboratory has shown that the above requirements can be met, and that an easily manipulated Guinier camera can be teamed up very profitably with a diffractometer for many applications, especially in the fields of corrosion research and ceramics.

In existing cameras, settings for different wavelengths are realized in three ways: by changing pertinent distances and angular settings, with the curvature of the monochromator crystal kept constant (Guinier, 1939); by adjusting the curvature of the monochromator crystal (de Wolff, 1948), or by exchanging the whole monochromator unit (Hofmann & Jagodzinski, 1955), so that the distance between the source and the camera can be kept constant. The first of these was adopted here, since it appeared to combine cheapness with the possibility of a fool-proof construction.

The prime consideration, ease and accuracy of adjustments on change of radiation, was met by defining all critical distances and angles by exchangeable gauges. All these are combined in essentially two units, the exit plate of the monochromator (Fig. 1, item 4), and the pillar of the cassette support (11). Readjustment for a different radiation is made by simply exchanging these two pieces. The whole operation takes only a few minutes.

As seen from Fig. 1, the apparatus is built onto an ordinary Philips camera support (17)—partly to exploit the vertical movement already incorporated in this part, and partly to enable the whole camera to be removed, if necessary, without disturbing more than the vertical adjustment. However, a more accurate base (of dove-tail section) is provided (16), which also decreases the take-off angle to 3° instead of the original 6°, in order to reduce the apparent focal-line width.

A change of wavelength will affect the following settings in the *monochromator unit* (cf. Fig. 2):

f, the focal distance towards the X-ray source;
θ, the angle of reflexion of the monochromator; and
h_s, the position of the exit slit of the monochromator compartment.

The *film holder* must be displaced in such a way that the focal spot of the monochromator falls on the film at F', and that the beam channel PF' of the camera (as defined by the positions of the sample holder, P, the stray radiation shield and the beam stop) will be collinear with the new direction of the beam. This implies changing the following settings:

f', the distance between the centre of the monochromator and the centre of the camera;
h_c, the vertical position of the cassette; and
α, the angular position of the cassette.

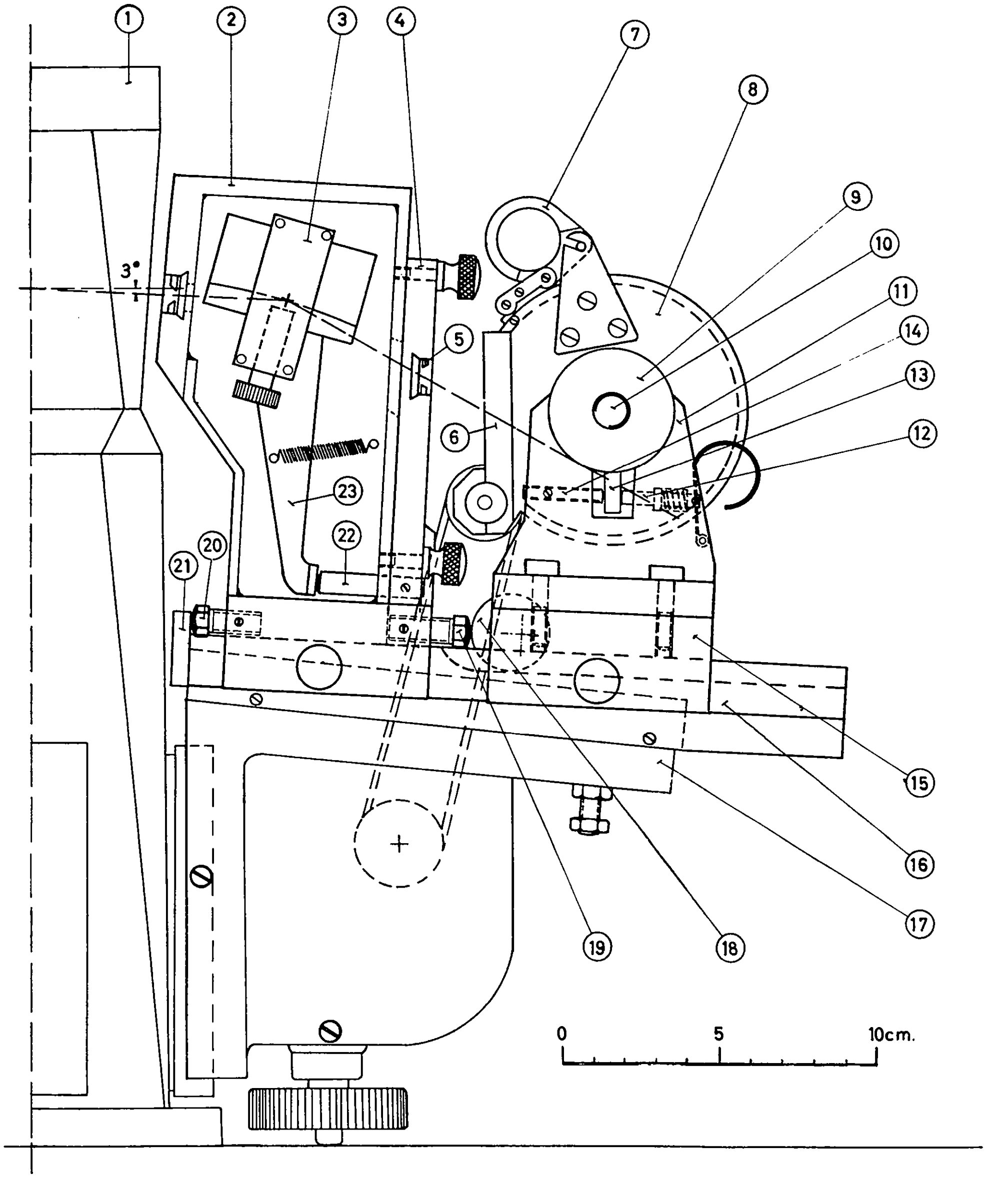

Fig. 1. Side view of Guinier camera: 1 X-ray tube. 2 monochromator housing, with cover removed. 3 quartz monochromator and holder. 4 exchangeable exit plate. 5 exit slit. 6 rotating-sample holder. 7 film-loading clamp (tightens lead rubber strip that keeps film in place). 8 cassette. 9 nut. 10 cassette mounting pin. 11 pillar. 12, 13, 14 cassette tilt adjustment. 15 cassette base. 16 dove-tail base. 17 Philips Norelco camera support. 18, 19 cam and adjustable stop for cassette. 20, 21 end stop. 22, 23 monochromator tilt adjustment.

The distance f is adjusted by means of screw (20), Fig. 1, against the end stop (21) of the dove-tail base. Screw (20) is adjusted and locked in position for the wavelength for which f is smallest; the additional displacements necessary for other wavelengths are defined by metal inserts, the widths of which are fixed according to the focal lengths of the monochromator for different radiations as specified by the supplier.

The other settings are combined in the exit plate (4) of the monochromator and in the pillar (11) of the camera support. The exit plate carries the exit slit (5) that defines the width of the beam striking the sample, adjusted once for all in the vertical position appropriate to the wavelength marked on the plate. It also carries the tilt adjusting screw (22) for the monochromator, which rotates the crystal about an axis passing through the vortex of its reflecting surface (corresponding to point M in Fig. 2) by acting on the spring-loaded lever (23). Finally, the plate carries screw (19), which defines the distance f' between the monochromator and the cassette. To avoid jarring when the camera body is replaced after loading, contact with this screw is made by the cam (18) being slowly turned to its outermost position.

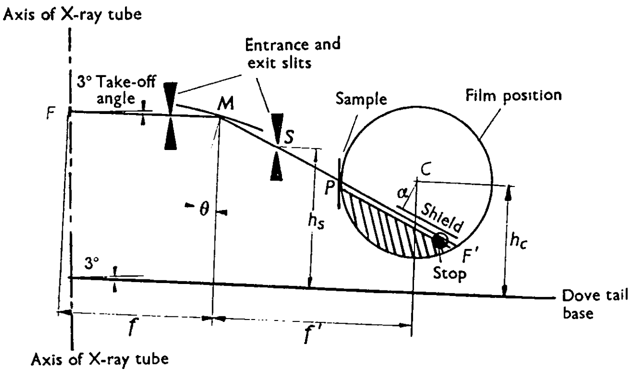

Fig. 2. Geometry of Guinier camera.

The vertical adjustment of the cassette is made by substituting the appropriate pillar (11). This pillar is made from a short piece of *L*-bar. Its shorter leg is screwed on top of the dove-tail rider (15). Its longer, upright leg has a bore with an excentric bushing that can be rotated and locked for fine adjustment of h_c, and which receives the central mounting pin (10) of the cassette. To define the proper angular setting of the cassette (α in Fig. 2), a peg (13) on its face is clamped between the adjustable stop (14) and a spring-loaded locating pin (12). The camera is secured in position by tightening the knurled nut (9) on its mounting pin.

The film is held in place by a strip of lead-rubber which is tightened by the clamp (7). The sample is mounted on a turntable (6) driven at its periphery by a worm which in turn is driven, *via* a rubber belt, by the camera drive housed in the original support. The distance between the sample and the centre of the film circle being one of the critical sources of error, it is important that the turntable should have no more axial play than necessary to allow smooth motion, and that the sample disks bearing the sample proper should all be of precisely equal thickness. The sample disks are circular brass plates with a central opening of 6 mm. diameter. The sample is mounted on a piece of adhesive tape stuck to the outer surface of the disk so that the powder faces toward the film. In this way, metal-to-metal contact is assured in the positioning of the sample disk and the diffracted radiation does not have to pass the tape (at high diffraction angles, quite long paths through the tape result when the sample is mounted on the off-camera side of the tape, giving rise to a considerable loss of intensity for soft radiations).

The radiations ordinarily employed are Cu and Fe $K\alpha$. Pillars and gauges for Co and Mn radiation were made in connexion with work on the cation distribution in ferrites. For Cr radiation, an auxiliary base is used which puts the cassette in a position below and beyond the ordinary base. This auxiliary base simply clamps on to the ordinary one. However, exposure times with Cr radiation become very long due to air absorption. The use of a hydrogen or helium bag has been contemplated, but not tried so far.

The camera has now been in continuous use for three years, being manipulated, after the first adjustment, by unskilled personnel only. Despite frequent shifts between different radiations, only one major readjustment has been necessary (after moving the whole X-ray unit within the laboratory). Thanks to the combination of sample stage and cassette into one integral unit and to the accurate workmanship of the turntable, no change of calibration has been observed so far. The films are measured to 0·01 mm., giving $\sin^2\theta$ to five decimals, of which the fifth is uncertain by ± 2 to ± 5 (the camera radius is 40 mm.). Hägg's (1949) procedure for eliminating errors due to uneven film shrinkage by printing a scale (division: 0·1 mm.) on the film prior to development is generally followed. As an additional safeguard, an internal standard can be used when accurate cell parameters are to be determined, as advocated by Westman & Magnéli (1957). For quick identification work *d* scales are used. A limited number of these scales, for Cu, Fe and Cr $K\alpha$ radiations, may be obtained on request from the author.

In the design of this camera, the author has drawn freely on experience gained at the Institute of Chemistry, University of Uppsala, with Guinier cameras of fixed, horizontal type, designed by Prof. G. Hägg. The author is indebted to Prof. Hägg for the loan of his drawings, from which some details of the cassette and rotating-sample stage, and the entrance and exit slits of the monochromator have been copied.

The camera described here has been designed and tested while the author was associated with the Research and Development Department of Telefonaktiebolaget L. M. Ericsson, Stockholm.

The author is grateful to Mr B. Engelbrand of Telefon–AB L. M. Ericsson, Stockholm, for his skilful draughtsmanship. The apparatus was constructed in the company's workshop. The primary adjustments, testing, and the supervision of the further operation of the camera were in the hands of the author's colleague, Mr M. Möller, to whom the author is indebted for his painstaking accuracy. Finally, the author wishes to record his gratitude to tekn. Dr C. Jacobaeus, technical director and director of research and development of Telefon–AB L. M. Ericsson, for the facilities placed at his disposal and for permission to publish this note.

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