

tation of the two diffraction patterns relative to each other. It is clear from the continuous nature of the streak connecting the 003 reflexion in the wet and dry states that there is no intermediate stage in the drying process of these crystals, such as that observed for haemoglobin (Perutz, 1946). The shrinkage process here merely seems

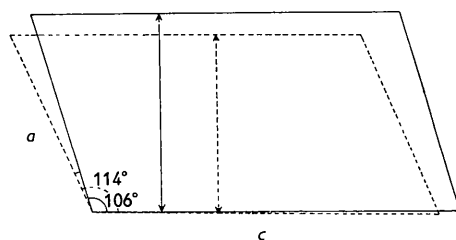


Fig. 2. Relationship of wet and dry unit cells. Full lines: wet cell; broken lines: dry cell.

to involve the relative and continuous movement of the molecules from one state to the other.

Reflexions in the dried crystal can be observed to spacings of about 3 Å, indicating that shrinkage can take place in an ordered manner provided it proceeds sufficiently slowly.

Fig. 2 shows the relationship of the wet to the dry unit cell. The small angular shift of the *c* axis is consistent with elongated molecules lying with their major axes approximately parallel to *c* (Carlisle & Scouloudi, 1951); hence the shortening of *b* from 38.80 to 30.08 Å would suggest a movement of the elongated molecules perpendicular to their lengths.

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**Use of a fluorescent source in an X-ray diffractometer.** By R. A. COYLE and R. I. GARROD, *Aeronautical Research Laboratories, P.O. Box 4331, Melbourne, Australia*

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Weiss, DeMarco & Weremchuk (1954) have described a method for converting a Norelco X-ray spectrograph to a diffractometer in which a fluorescent X-ray source is used. With suitable geometry, the half breadth of the 110 reflexion from an annealed iron specimen (Co radiation) was found to be only about 20% larger than the value obtained with a standard Norelco diffractometer (22' and 18' respectively), whilst the background was reduced by a factor ~ 25. If these results were applicable generally, the method would be an attractive alternative to a conventional X-ray source and crystal monochromator for applications requiring high resolution and a low background, since the use of a monochromator leads to complexity in mechanical design if focusing conditions are to be satisfied for a range of X-radiations.

Calculations, however, suggest that favourable comparison in line breadths between the fluorescent and normal methods is likely to be obtained only when the specimen itself produces appreciable broadening. For example, applying the convolution methods developed by Alexander (1950, 1954) to the experimental conditions specified by Weiss *et al.* leads to values for *instrumental* broadening of 6' ( $\alpha_1$ ) for the standard diffracto-

meter and 27' (unresolved  $\alpha_1\alpha_2$  doublet) for the fluorescent arrangement. It would therefore appear that the values obtained experimentally by Weiss *et al.* were determined largely by (i) the specimen and (ii) instrumental factors, in the standard and fluorescent methods respectively.

This impression has been confirmed by measurements in these Laboratories on suitable reflexions from a 'standard' silicon specimen and annealed powdered iron, using the following experimental methods:

- (A) A fluorescent source and a slit system identical with that described by Weiss *et al.*; goniometer radius 14 cm.
- (B) Normal X-ray source and standard Philips (Eindhoven) goniometer; 1° divergence slit, 0.1 mm. receiving slit, 17 cm. radius.
- (C) Normal X-ray source, quartz crystal monochromator, Philips goniometer; 1° divergence slit, 0.2 mm. receiving slit, 11 cm. radius.

The half breadths observed are summarized in Table 1, together with the equivalent calculated values for instrumental broadening.

Table 1. Half breadths for reflexions from silicon and iron obtained by three diffractometer methods

(Values of half breadths are in minutes of arc)

Specimen	Radiation	2θ(°)	Method A		Method B		Method C	
			Obs.	Calc.	Obs.	Calc.	Obs.	Calc.
Silicon	Co	33	24*	25*	—	—	—	—
Silicon	Co	55	—	—	6½	6	9	10
Silicon	Cu	56	—	—	7	6	10	10
Iron	Co	52	30*	27*	12	6	12	9

\* Unresolved  $\alpha_1\alpha_2$ ; otherwise  $\alpha_1$  alone.

For the silicon specimen, with negligible natural broadening, the observed and calculated breadths are in excellent agreement. Correction curves (Alexander, 1954) indicate a natural breadth of 4–5' for the iron specimen.

The predominant source of broadening in the fluorescent method is the effective source width (1 mm.). It does not appear practicable in the present method to reduce this appreciably and still maintain sufficient intensity for accurate line profile determinations (although a development of the fluorescent X-ray tube outlined by Guinier (1950) might perhaps be more suitable for this purpose). Nevertheless, where poor resolution can be

tolerated, the method does appear to have a number of attractive features.

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## International Union of Crystallography

### Commission on Crystallographic Apparatus: Recommendations on Goniometer Heads\*

The Commission wishes to draw the attention of all apparatus manufacturers to the great importance of making interchangeable goniometer heads, so that one sample can be rapidly and precisely moved from one goniometer to another, regardless of manufacture.

The Commission recommends for general adoption the dimensions and design data recently approved by the

American Crystallographic Association. The chief features and dimensions to be adopted imperatively are given in Fig. 1.

The Commission notes that most cameras and goniometers in use today require that the head be usable with an inclination of plus or minus twenty degrees ( $\pm 20^\circ$ ) of both arcs inside a cylinder of fifty (50.0) millimetre diameter. Since this limits the maximum radius of the lower arc, a shorter standard height is shown to be *permissible only* on eucentric goniometer heads (i.e. those having a fixed common intersection of principal axis and the axes of both arcs and providing translations and elevations only above the arcs).

Correspondence concerning this note should be addressed to the Chairman of the Commission (Prof. A. GUINIER, Conservatoire des Arts et Métiers, 292 Rue St Martin, Paris 3, France).

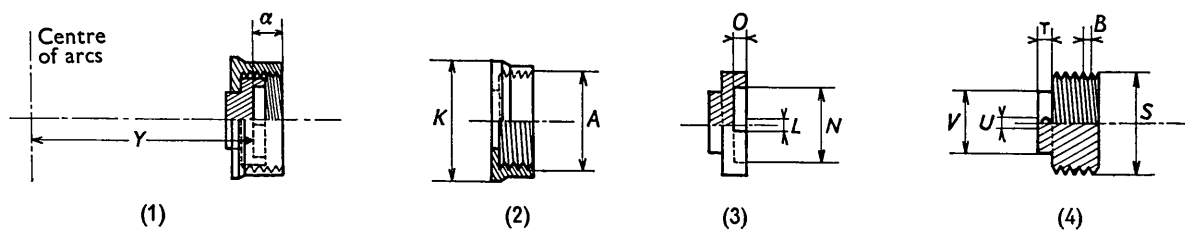


Fig. 1. Description, dimensions and tolerances of goniometer heads (excerpts from the specifications adopted by the American Crystallographic Association).

	Inches	Millimetres
Y Above male fitting (4)	$\left\{ \begin{array}{l} 2.518 \pm 0.001 \\ [1.929 \pm 0.001]^* \end{array} \right.$	63.96 $\pm$ 0.03
$\alpha$ Overlap of nut		7.14 (7.50 maximum)
A Pitch diameter for loose nut	1.144	29.06
K Outside diameter of knurl	1.375 (1.400 maximum)	34.92 (35.56 maximum)
L Slot for pin	0.1250 +0.0005, -0.0000	3.18 +0.01, -0.00
N Inside diameter of cup	0.8660 +0.0005, -0.0000	22.00 +0.01, -0.00
O Depth of cup	0.155 maximum	3.94 maximum
B Single thread pitch	0.0394 (60° vee threads)	1.00 (60° vee threads)
S Pitch diameter of fitting	1.138	28.91
T Height of stud	0.157 minimum	3.99 minimum
U Diameter of pin	0.1245 +0.0000, -0.0005	3.16 +0.00, -0.01
V Diameter of stud	0.865 +0.0000, -0.0005	21.97 +0.00, -0.01

\* Alternative height to centre of arcs *permissible only* on eucentric goniometer heads.