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Use of a two-circle device to obtain three-dimensional neutron diffraction data.

By B. T. M. Willis, *Atomic Energy Research Establishment, Harwell, Didcot, Berks., England*

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It has been customary in neutron diffraction work to use a pillar-shaped crystal to obtain two-dimensional F^2 data, the long axis of the crystal corresponding to the zone axis of the reflexions to be examined (Bacon & Pease, 1953; Atoji & Rundle, 1958). As this long axis is normal to the plane containing the incident and reflected beams, a maximum counting rate is achieved while the possibility of extinction errors is minimized. The purpose of this note is to point out that, by mounting the crystal on a two-circle device placed on the central table of the spectrometer, three-dimensional data (or two-dimensional data about an arbitrary zone axis) can be obtained from a pillar-shaped crystal with approximately the same accuracy as for the conventional two-dimensional arrangement. The method has been used successfully in this laboratory for several months.

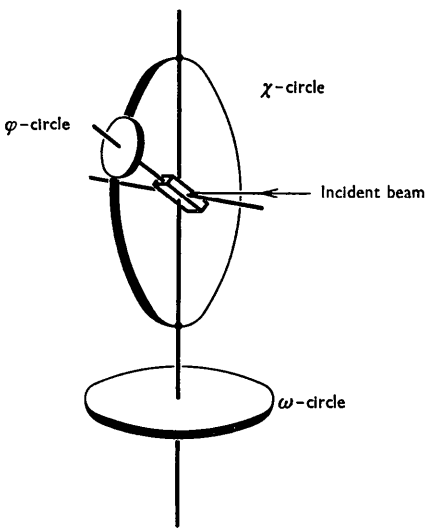

Fig. 1. Diagram of the φ-, χ- and ω-circles.

The device constitutes the φ- and χ-circles of a three-circle diffractometer (Furnas & Harker, 1955). χ is a vertical circle and φ a circle which rotates as a whole about the horizontal χ-axis (Fig. 1). The χ-circle and counter move independently round the third circle, ω, which is the large horizontal circle of the diffractometer. The crystal, mounted on a goniometer head attached to the φ-circle, is located at the common point of intersection of the φ-, χ- and ω-axes.

By appropriate adjustments of the three circles, the normal to any (*hkl*) plane is brought into the Bragg reflecting position in the equatorial plane, and the integrated intensity is then measured in the normal way by moving the counter round to the $2\theta_{hkl}$ position. The stereogram in Fig. 2 illustrates the adjustments required. OP represents the (*hkl*) normal initially, RO the incident neutron beam and $\chi O\chi'$ the χ-axis, set at an arbitrary angle to RO: it is required to move the pole P to P', where OP' is in the equatorial plane and at an angle $\pi/2-\theta_{hkl}$ to RO. Initially, the φ-circle is moved round the χ-circle to make the φ-axis vertical. The φ-axis is then rotated through the angle PQ, moving P to Q, where Q is the point of intersection of the two small circles of centre O, radius OP and centre χ', radius $\chi'P'$. Finally, the χ-axis is rotated through the angle QP',

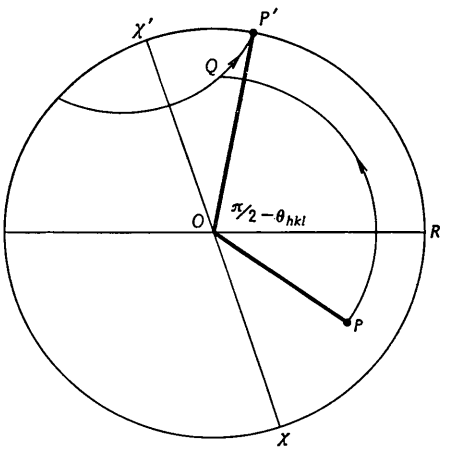

Fig. 2. Stereogram illustrating the rotations about the φ- and χ-axes necessary to bring the (*hkl*) normal, OP, to the reflecting position, OP', in the equatorial plane. RO is the incident beam direction, $\chi O\chi'$ is the χ-axis and the φ-axis is initially vertical.

moving Q to P'. Clearly, the circle settings are not unique but depend on the arbitrary ω setting of the χ-circle. By mounting the crystal with its long direction along the φ-axis and by choosing the χ-axis perpendicular to the (*hkl*) normal in its reflecting position, OP', the path length of the beam inside the crystal is a minimum (Fig. 3). The ω setting is then θ_{hkl}, and the φ and χ settings are given by

$$\tan\varphi = \frac{V}{abc}\left[\frac{k/b - l\cos\alpha/c}{h\sin^2\alpha/a + k(\cos\alpha\cos\beta-\cos\gamma)/b + l(\cos\gamma\cos\alpha-\cos\beta)/c}\right]$$

and

$$\sin\chi = ld_{hkl}/c.$$

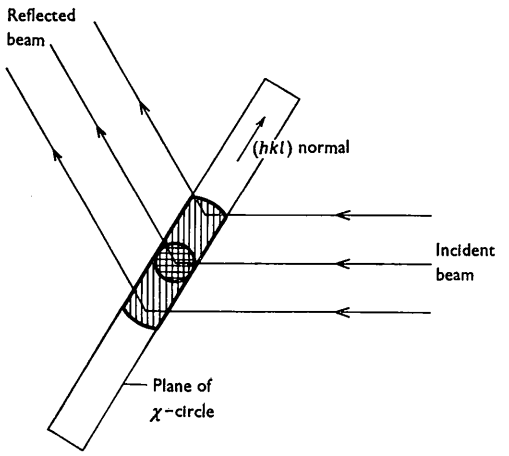

Fig. 3. Diagram showing the path of the beam inside the crystal. The cross-hatched region represents the crystal at $\chi=0$ with its long axis vertical; the vertically shaded region represents the crystal after it has been moved round the χ-circle to bring the (*hkl*) normal into the horizontal plane.

V is the volume of the unit cell and it is assumed that the c-axis of the crystal is along the φ-axis, that $\varphi=0$ corresponds to the a^*-axis in the plane of the χ-circle and that $\chi=0$ corresponds to the φ-axis vertical.

Thus by attaching a two-circle device, representing the χ- and φ-circles, to the existing ω-circle of a neutron diffraction spectrometer, it is possible to extend collection of F^2 data to three dimensions. In essence, the method depends on the principle that a three-circle instrument not only allows any (*hkl*) plane to be brought into the reflecting position, but also allows rotation of the crystal about the normal to this plane to make the path length a minimum.

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Crystallographic data for certain amidinium carboxylates.

By OLGA KENNARD and JAMES WALKER,
National Institute for Medical Research, Mill Hill, London, England

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During an investigation of the mechanism of salt formation between carboxylic acids and substances containing an unsubstituted amidinium group the crystallographic constants of a number of these salts were determined (Kennard & Walker, 1954). Unit-cell dimensions were obtained from oscillation and Weissenberg photographs ($\lambda=1{\cdot}5418$ Å), except for the last two compounds listed below, where the θ method (Weisz, Cochran & Cole, 1948) was used. Densities were determined with an accuracy of $\pm 0{\cdot}5\%$ by centrifuging the crystals in a continuous-gradient density column (Low & Richards, 1952). The three-dimensional structure analysis of S-methylthiuronium *p*-chlorobenzoate is being reported elsewhere (Kennard & Walker, 1961).

Benzamidine benzoate
$C_6H_5.C(:NH).NH_2$, $C_6H_5.CO_2H$

Benzamidine benzoate was prepared from benzamidine hydrochloride and sodium benzoate in aqueous solution (*cf.* Pinner, 1892). It crystallized from water as flat plates elongated along [001], with marked striations in this direction. The striations were traces of an excellent cleavage plane.

The refractive index for white light was $1{\cdot}680\pm 5$ with the electric vector vibrating in the direction of elongation of the plates, and $1{\cdot}630\pm 5$ at right angles to this direction.

Orthorhombic

$a=28{\cdot}9(4)$, $b=35{\cdot}8(6)$, $c=9{\cdot}9(5)$ Å, $U=10326$ Å^3, $D_m=1{\cdot}25$ g.cm.$^{-3}$, $Z=32$, $D_x=1{\cdot}25$ g.cm.$^{-3}$.

Space group $Ccc2$ with additional non-space-group absences. Absent spectra: hkl when $h+k$ odd, but very few weak reflexions of the type $h+l$ odd or $k+l$ odd were observed. The $hk0$ reflexions were with a few exceptions absent unless $h+k=4n$. The $0kl$ reflexions were absent unless $k=4n$, $l=2n$, and the $h0l$ reflexions if $h=4n+1$ or $l=2n+1$.

3,5-Dibromobenzamidine benzoate
$C_6H_3Br_2.C(:NH).NH_2$, $C_6H_5.CO_2H$

3,5-Dibromobenzamidine benzoate was prepared from 3,5-dibromobenzamidine hydrochloride and sodium benzoate; it was recrystallized from water and had m.p. 228–229° (decomp.). (Found: C, 42·0; H, 2·9; N, 6·8. $C_7H_6Br_2N_2$, $C_7H_6O_2$ requires C, 42·0; H, 3·0; N, 7·0%). The crystals were needle-shaped with diagonal extinction; they had faint striations and imperfect cleavage parallel to the needle axis.

Triclinic

$a=15{\cdot}21$, $b=9{\cdot}64$, $c=12{\cdot}34$ Å,
$\alpha=110$, $\beta=110$, $\gamma=100{\cdot}7°$,
$U=1501$ Å^3, $D_m=1{\cdot}755$, $Z=4$, $D_x=1{\cdot}77$ g.cm.$^{-3}$.

Space group $P1$ or $P\bar{1}$. No absences.

3,5-Dibromobenzamidine 3,5-dibromobenzoate
$C_6H_3Br_2.C(:NH).NH_2$, $C_6H_3Br_2.CO_2H$

3,5-Dibromobenzamidine 3,5-dibromobenzoate was obtained from 3,5-dibromobenzamidine hydrochloride and