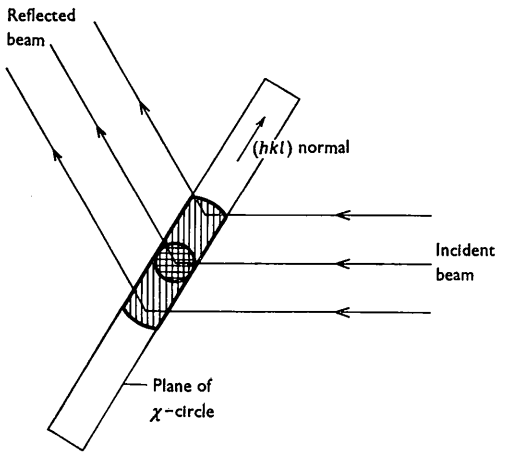

Fig. 3. Diagram showing the path of the beam inside the crystal. The cross-hatched region represents the crystal at $\chi=0$ with its long axis vertical; the vertically shaded region represents the crystal after it has been moved round the χ-circle to bring the (*hkl*) normal into the horizontal plane.

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

sodium 3,5-dibromobenzoate; it was recrystallized from propan-1-ol–water (2:3) and had m.p. 265° (decomp.). (Found: C, 30·4; H, 1·8; N, 5·0. $C_7H_6Br_2N_2$, $C_7H_4Br_2O_2$ requires C, 30·1; H, 1·8; N, 5·0%).

The crystals were columnar with marked striations along the [001] axis which is also the direction of elongation. Cleavage was perfect parallel to (100). Refractive index for polarized white light travelling along [010] was $1·615 \pm 5$ parallel to [001] and $1·750 \pm 5$ parallel to [100].

Orthorhombic

$a = 23·8$, $b = 32·1$, $c = 4·85$ Å, $U = 3705$ Å^3,
$D_m = 2·10$ g.cm.$^{-3}$, $Z = 8$, $D_x = 2·00$ g.cm.$^{-3}$.

S-Methylthiuronium benzoate
$CH_3S.C(:NH).NH_2$, $C_6H_5.CO_2H$

S-Methylthiuronium benzoate gave columnar crystals (Walker, 1949) with marked striations in the direction of elongation [001]. Cleavage was perfect parallel to (100).

Orthorhombic

$a = 9·52$, $b = 20·29$, $c = 5·61$ Å, $U = 1084$ Å^3,
$D_m = 1·28$ g.cm.$^{-3}$, $Z = 4$, $D_x = 1·30$ g.cm.$^{-3}$.

Space group $P2_12_12_1$. Absences observed: $h00$ when h odd, $0k0$ when k odd, $00l$ when l odd.

S-Methylthiuronium *p*-iodobenzoate
$CH_3S.C(:NH).NH_2$, $C_6H_4I.CO_2H$

S-Methylthiuronium *p*-iodobenzoate was obtained from S-methylthiuronium sulphate and sodium *p*-iodobenzoate; it was recrystallized from water and had m.p. 220° (decomp.). (Found: C, 32·1; H, 3·2; N, 8·2. $C_2H_6N_2S$, $C_7H_5IO_2$ requires C, 32·0; H, 3·3; N, 8·3%).

The crystals were prismatic or needle-shaped and cleavage was not pronounced. Interpenetrating twins were fairly common producing a pseudo-symmetry plane perpendicular to [100].

Monoclinic

$a = 9·40$, $b = 5·61$, $c = 23·55$ Å, $\beta = 101·5°$, $U = 1217$ Å^3,
$D_m = 1·82$ g.cm.$^{-3}$, $Z = 4$, $D_x = 1·85$ g.cm.$^{-3}$.

Space group $P2_1/c$ or $P2/c$. Absent reflexions: $h0l$ if l odd; $0k0$: only the second order observed.

S-Methylthiuronium *p*-bromobenzoate
$CH_3S.C(:NH).NH_2$, $C_6H_4Br.CO_2H$

S-Methylthiuronium *p*-bromobenzoate was similarly prepared from S-methylthiuronium sulphate and sodium *p*-bromobenzoate; it had m.p. 214° (decomp.). (Found: C, 37·3; H, 3·9; N, 9·5. $C_2H_6N_2S$, $C_7H_5BrO_2$ requires C, 37·2; H, 3·8; N, 9·6%).

The majority of crystals were tabular but a few prisms were also observed. Cleavage was imperfect parallel to (100). Frequent twinning as in isomorphous iodo-compound was observed.

Monoclinic

$a = 9·505 \pm 5$, $b = 5·61 \pm 1$, $c = 22·556 \pm 10$ Å,
$\beta = 103·22 \pm 5°$, $U = 1171$ Å^3, $D_m = 1·625$ g.cm.$^{-3}$,
$Z = 4$, $D_x = 1·651$ g.cm.$^{-3}$.

Space group $P2_1/c$ from systematic absences and from the appearance of the Patterson projection on $(0kl)$ which contained no prominent peaks related to a twofold axis.

S-Methylthiuronium *p*-chlorobenzoate
$CH_3S.C(:NH).NH_2$, $C_6H_4Cl.CO_2H$

S-Methylthiuronium *p*-chlorobenzoate was prepared in an analogous manner; it had m.p. 210–211° (decomp.). (Found: C, 44·1; H, 4·8; N, 11·2. $C_2H_6N_2S$, $C_7H_5ClO_2$ requires C, 43·8; H, 4·5; N, 11·4%).

It is isomorphous with the bromo-compound.

Monoclinic

$a = 9·505 \pm 5$, $b = 5·61 \pm 1$, $c = 22·176 \pm 10$ Å,
$\beta = 103·22 \pm 5°$, $U = 1151$ Å^3, $D_m = 1·41$ g.cm.$^{-3}$,
$Z = 4$, $D_x = 1·422$ g.cm.$^{-3}$.

Space group $P2_1/c$ from systematic absences.

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Données cristallographiques sur le diphényl méthane.

Par M. J. HOUSTY, *Laboratoire de Minéralogie et de Rayons X, Faculté des Sciences, Université de Bordeaux, France*

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Le diphényl méthane cristallise sous forme de cristaux monocliniques, incolores, allongés suivant la direction [010]. Ces cristaux sont obtenus par cristallisation à une température légèrement inférieure à la température de fusion (25 °C.) dans un bain thermostaté au 1/50ème de degré centigrade.

Le diphényl méthane étant volatil à la température ordinaire nous avons enfermé le cristal dans un tube de verre scellé.

L'étude radiocristallographique a été faite à l'aide d'une chambre de Bragg et d'un rétigraphe de de Jong, en utilisant la radiation Cu $K\alpha$. Nous avons ainsi déterminé les paramètres de la maille monoclinique:

$a = 8,95 \pm 0,04$, $b = 6,22 \pm 0,03$, $c = 20,50 \pm 0,05$ Å;
$\beta = 120° \pm 1°$.

Nombre de molécules par maille: 4.

La densité observée de 1,008 g.cm.$^{-3}$ est en bon accord avec celle calculée 1,017 g.cm.$^{-3}$ à partir des données cristallographiques.

Groupe spatial $P2_1/c$ ou $P2/c$.

Une étude plus poussée de cette structure est en cours.