Short Communications

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 *words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible. Publication will be quicker if the contributions are without illustrations.*

The space groups and unit-cell dimensions of 2,4,6-tribromoaniline and 2,4,6-trichloroaniline.

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Crystals of 2,4,6-tribromoaniline are orthorhombic, space group $P2_12_12_1$, with $a=13\cdot46$, $b=14\cdot69$, $c=4\cdot27$ Å, $Z=4$. Crystals of 2,4,6-trichloroaniline are monoclinic, space group either $P2_1/m$ or $P2_1$, with $a=15\cdot88$, $b=3\cdot86$, $c=13\cdot38$ Å, $\beta=111°45'$, $Z=4$.

In connection with some X-ray work being done on 2,4,6-tribromobenzonitrile and 2,4,6-trichlorobenzonitrile, the space groups and unit cells of 2,4,6-tribromoaniline and 2,4,6-trichloroaniline were determined.

From oscillation, Weissenberg, and precession photographs (Mo $K\alpha$, $\lambda=0\cdot7107$ Å), crystals of 2,4,6-tribromoaniline were found to be orthorhombic with cell dimensions $a=13\cdot462\pm0\cdot008$, $b=14\cdot692\pm0\cdot014$, $c=4\cdot266\pm0\cdot005$ Å. The systematic extinctions, ($h00$, $h=2n+1$; $0k0$, $k=2n+1$; $00l$, $l=2n+1$), indicate that the space group is $P2_12_12_1$. If $Z=4$, the calculated density is $2\cdot596$ g.cm^{-3}; the experimental density is $2\cdot578$ g.cm^{-3} (Jaeger, 1907).

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Needle-like crystals of 2,4,6-trichloroaniline were mounted in capillaries to prevent sublimation. Precession photographs taken with Mo $K\alpha$ radiation ($\lambda=0\cdot7107$ Å) indicate that the cell is monoclinic with the unique axis as the needle axis. A unit cell with the dimensions $a=15\cdot875\pm0\cdot006$, $b=3\cdot863\pm0\cdot005$, $c=13\cdot381\pm0\cdot005$ Å, $\beta=111°45'$ was chosen. A rough experimental density of $1\cdot6$ g.cm^{-3} was obtained; for $Z=4$, the calculated density is $1\cdot712$ g.cm^{-3}. The systematic extinctions, ($0k0$, $k=2n+1$), indicate that the space group is either $P2_1/m$ or $P2_1$. No further X-ray work on these compounds is planned.

References

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Absorption correction in the Weissenberg methods.

By A. Santoro and M. Zocchi, *Institute for Materials Research, National Bureau of Standards, Washington, D.C., U.S.A.*

A procedure is described for calculating the direction cosines of the incident and diffracted beams in a reference system attached to the crystal for the general case of the Weissenberg method.

Methods have been described for the calculation of the absorption correction in the equi-inclination and normal-beam techniques (Wells, 1960; Coppens, Leiserowitz & Rabinovich, 1965; Wuensch & Prewitt, 1965). However in some cases (Santoro & Zocchi, 1966) it is preferable to use the Weissenberg method with arbitrary values of the angle μ (*International Tables for X-ray Crystallography*, 1959), and therefore it seems useful to extend the treatment of the absorption correction to the general case.

In what follows we will only describe a procedure for calculating the direction cosines of the incident and diffracted beams in a reference system attached to the crystal; from this point on the calculation of the transmission factor for each reflection can be performed as suggested, for example, by Busing & Levy (1957) or by Wuensch & Prewitt (1965).

It has been shown (Santoro & Zocchi, 1966) that, for any Weissenberg method, a reciprocal lattice point is in reflecting position when:

$$zs(n-2v_0) + 2\left[1 - s^2\left(\frac{n}{2} - v_0\right)^2\right]^{\frac{1}{2}}(y\cos\omega - x\sin\omega) + d^{*2} = 0, \quad (1)$$

where ω is the rotation angle, v_0 is the index of the reciprocal layer under examination, s is the period on the rotation axis, x,y,z are the coordinates of the point for $\mu=\omega=0°$ with respect to a Cartesian system X,Y,Z, attached to the laboratory, and defined in the given reference,

$d^{*2}=x^2+y^2+z^2$, and $n=(2\sin v)/s$ (*International Tables*).

From equation (1) we obtain:

$$\tan\frac{\omega}{2} = \frac{x\pm[x^2-(A+y)(A-y)]^{\frac{1}{2}}}{A-y}, \quad (2)$$

where

$$A = \frac{zs\,(n-2v_0)+d^{*2}}{2\left[1-s^2\left(\dfrac{n}{2}-v_0\right)^2\right]^{\frac{1}{2}}} \; .$$

The direction cosines of a diffracted beam are given by:

$$\varepsilon_1 = \frac{x_1}{B}, \qquad \varepsilon_2 = \frac{y_1+1}{B}, \qquad \varepsilon_3 = \frac{z_1}{B}$$

where x_1, y_1, z_1 are given by:

$$\{x_1 y_1 z_1\} = (d_{ij})\{xyz\}$$

and are the coordinates of the point when it is in reflecting position,

$$(d_{ij}) = \begin{pmatrix} \cos\omega & \sin\omega & 0 \\ -\sin\omega\cos\mu & \cos\omega\cos\mu & \sin\mu \\ \sin\omega\sin\mu & -\cos\omega\sin\mu & \cos\mu \end{pmatrix}$$

where ω is obtained from equation (2), and $B = x_1^2 + (y_1+1)^2 + z_1^2 = 1$, since the radius of the reflection sphere is equal to one.

Let us define now a reference system X', Y', Z', attached to the crystal, so that, for $\mu = \omega = 0°$, and after the crystal has been oriented, it is coincident with X, Y, Z.

It follows immediately that the direction cosines of the incident beam in the system X', Y', Z' are:

$$\gamma_i = \Sigma_j d_{2i}$$

and those of the diffracted beam in the same system are:

$$\delta_i = \Sigma_j d_{ij}\varepsilon_j \; .$$

In some cases it is convenient to choose X', Y', Z' non-coincident with X, Y, Z under the above conditions; in such cases, the two systems are related by rotations on the two arcs of the goniometer head and on the dial, *i.e.* they are related by the transformation

$$\{xyz\} = (S_{ij})\{x_0 y_0 z_0\} \; .$$

References

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Alkaline earth silicides, germanides and stannides with CrB structure type.

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Five new alkaline earth compounds: SrGe, SrSn, BaSi, BaGe and BaSn are found to crystallize with the CrB ($B33$) structure type. It is shown that a relationship exists between the c/b axial ratio, the y parameter of the Si, Ge or Sn positions and the chain angle α of the Si–Si, Ge–Ge or Sn–Sn chains. The new compounds are discussed under the aspect of the general valence equation.

In continuation of investigations on monosilicides and monogermanides with CrB- and FeB-type structure (Schob & Parthé, 1965; Hohnke & Parthé, 1966) we found it of interest to ascertain whether these structure types occur also with silicides, germanides and stannides of the alkaline earth elements. Since these compounds contain no transition elements and have one component to the right of the Zintl line it is possible to apply the general valence equation (Mooser & Pearson, 1956; Pearson, 1964; Kjekshus, 1964; Hulliger & Mooser, 1965; Parthé, 1966). With an electron to anion ratio of 6 and under the assumption that the cation–cation bonds are negligible the general valence equation suggests the presence of anion–anion chains. Such anion–anion chains are the particular structural feature not only of the CrB and FeB structure types but also of the MoB and the monoclinic LiAs structure types.

Five alkaline earth compounds with group 4B elements are already known. All of them crystallize in the orthorhombic CrB structure type: CaSi (Hellner, 1950), CaGe (Eckerlin, Meyer & Wölfel, 1955), CaSn (Eckerlin, Meyer & Wölfel, 1955), SrSi (Rocktäschel & Weiss, 1962) and also BaPb (Sands, Wood & Ramsey, 1959). After our investiga-

tions were completed, we received information on two additional compounds: BaSi and BaGe (Burnashowa & Gladyshevskii, 1966) which crystallize also in the CrB structure type.

Samples of CaSi, CaPb, SrSi, SrGe, SrSn, SrPb, BaSi, BaGe and BaSn were prepared by arc melting the elements under argon atmosphere. The arc melting procedure was done with great care to avoid losses of the materials by vaporization. Weight losses were checked and proved not to exceed an average of 5 %; thus the reacted samples were assumed not to be far off stoichiometry. Since the reactions were violent and complete, it was not necessary to homogenize the alloys. The materials were crystallized in the shape of platelets; all of them proved to react violently with moisture, and powder patterns (Cr$K\alpha$ radiation) were therefore taken in glass capillaries under dried ligroin.

The powder diffraction patterns of CaSi, SrSi, SrGe, SrSn, BaSi, BaGe and BaSn could be indexed orthorhombic with the lattice constants given in Table 1. The extinctions of hkl with $h+k = 2n+1$ and $h0l$ with $l = 2n+1$ led to the possible space groups: $Cmc2_1$ (C_{2v}^{12}), $C2cm$ (C_{2v}^{16}) and $Cmcm$ (D_{2h}^{17}). The similarity of the diffraction patterns of