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The crystal structure of indium (I) iodide.

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Crystals of InI were grown by heating indium with a slight excess of iodine in an evacuated tube at 300° C. The vapor condensed on a cooler surface outside the furnace as brownish-red plates of the {010} form. From Weissenberg and powder photographs taken with Cu $K\alpha$ and Cr $K\alpha$ radiation an orthorhombic unit cell was found with dimensions:

$$a = 4\cdot75,\ b = 12\cdot76,\ c = 4\cdot91\ \text{Å}\ (\text{each}\ \pm0\cdot2\%)\,.$$

For $Z = 4$, the density is calculated as 5·39 g.cm.$^{-3}$ compared with 5·32 g.cm.$^{-3}$ measured by Klemm & Dierks (1934). The absences correspond to space groups *Cmcm*, *Cmc*2 and *C*2*cm*. Agreement with the intensities is found with the TlI-type structure (Helmholz, 1936), space group $Cmcm\text{–}D_{2h}^{17}$ with the atoms in positions 4(*c*) (*International Tables*, 1952):

$$\pm(0, y, \tfrac{1}{4})+(0, 0, 0;\ \tfrac{1}{2}, \tfrac{1}{2}, 0)\,.$$

The atomic parameters were determined by sharpened (F_o-F_c) syntheses for the projection along [001], computed only along the y axis. The $hk0$ intensities were estimated visually and corrected for Lorentz and polarization factors and for absorption. For allowed reflections the intensity is independent of h for constant k and l, except for the factors dependent on θ. Sets of corrected intensities with $l = 0$ and with the same value of k were plotted versus $\sin^2\theta$, and sharpened F^2 values, corresponding to point atoms at rest, were read from these curves at an arbitrary $\sin^2\theta$. This procedure was adopted to improve the accuracy of the absorption corrections. The best fit was obtained with $y = 0\cdot398\pm0\cdot001$ for indium and $y = 0\cdot145\pm0\cdot001$ for iodine. The standard deviations are according to the method of Cox & Cruickshank (1948).

Each atom has one neighbor of the opposite kind at 3·23 Å and four more at 3·46 Å. Two additional neighbors at 3·95 Å can hardly be considered nearest neighbors. The shortest In–In and I–I distances are 3·58 and 4·44 Å, respectively. The ionic radius of In$^+$, if this compound can be considered a salt, may be estimated as 1·32 Å if one uses the polymorphism of TlI (Helmholz, 1936; Barth, 1927) to calculate the distance In and I would have in the CsCl-type structure if it existed. One obtains essentially the same result using either Pauling's (1942) or Zachariasen's (1950) scheme for correcting for coordination if one uses the corresponding course for the radius of I$^-$.

The compound InBr has this same structure (Stephenson & Mellor, 1950). From the distances in this compound one can calculate in the same way essentially the same value for the radius by either scheme.

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The length of the uranyl ion in uranyl carbonate.[*]

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Christ, Clark & Evans (1955) have recently published the structure of rutherfordine, the naturally occurring mineral UO_2CO_3. We had simultaneously been investigating some synthetic UO_2CO_3. These synthetic crystals were made by Dr B. Thamer of this laboratory by the method of Miller, Pray & Munger (1949).

The synthetic crystals occur as small, very thin laths elongated along [001] with the (010) face dominant. Weissenberg photographs with Cu radiation gave the following cell dimensions:

$$a = 4\cdot85\pm0\cdot01,\ b = 9\cdot22\pm0\cdot02,\ c = 4\cdot30\pm0\cdot01\ \text{Å}\,.$$

These values are in good agreement with those reported by Christ *et al.* (1955).

The $hk0$ projection is particularly well suited for a determination of the length of the U–O bond. A crystal $52\times2\cdot3\times18\cdot4$ microns was selected for measurement of the $hk0$ intensities. Using Cu radiation all $hk0$ reflections were measured with a Geiger counter, and the average value of symmetry-related reflections was obtained. Thirty crystallographically different reflections were measured. Absorption corrections were computed by the graphical method of Howells (1950).

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A five-parameter least-squares refinement was made. The variables were the scale factor, two isotropic temperature factors (one for uranium and one for the light atoms), the x parameter of the fourfold carbonate oxygens, and the y parameter of the uranyl oxygens (see Christ *et al.*, 1955). The McWeeny (1951) form factors were used for carbon and oxygen. For uranium, the form factor given in the *Internationale Tabellen* was used after subtracting 5·6 electrons to correct for anomalous dispersion. The observations were given a weight proportional to $1/\sigma_I^{\frac{1}{2}}$, where σ_I is the standard deviation of the intensity measurements. The 020 reflection was given zero weight because of obvious extinction effects. The calculations were made on an IBM 701.

The results of the least-squares refinement are given below:

$$x = 0{\cdot}011 \pm 0{\cdot}018,\; y = 0{\cdot}932 \pm 0{\cdot}010;$$
$$B_U = 0{\cdot}51 \pm 0{\cdot}20,\; B_{O,C} = 1{\cdot}65 \pm 0{\cdot}86\ \text{Å}^2\,.$$

For this projection $R = 7{\cdot}3\%$ with 020 omitted.

The significant result is the value of the y coordinate of the uranyl oxygen. Assuming that the uranyl ion is linear, the U–O distance is 1·67±0·09 Å. This distance is shorter than any previously observed. Zachariasen (1954) has published a survey of the uranyl and similar type compounds and has given an empirical curve relating bond strength to bond length. In the plane of the carbonate groups (see Christ *et al.*, 1955) there are four U–O distances of 2·52 Å, each with a bond strength of 0·38, and two distances of 2·44 Å having a bond strength of 0·46. The remaining two bonds must have a strength of 1·78 in order to achieve a total of 6·0 valence electrons. A distance of 1·68 Å is empirically predicted. This distance compares very favorably with the observed value of 1·67±0·09 Å.

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Crystallographic data for rhodanine, $C_3H_3S_2NO$.*

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Rhodanine was first prepared by Nencki (1877). Feigl & Pollack (1926) reported the water-insoluble silver and cuprous salts. Dubsky, Sindelar & Cernak (1938) and Feigl (1949) have studied the salt-forming properties of the molecule but these studies have not succeeded in clarifying the stereochemistry of rhodanine and its metallic salts. An attempt to prove the structures of these compounds by X-ray methods has been initiated in this laboratory.

Rhodanine crystals grown from a solution in hot ethanol were hexagonal plates with well formed {010} and {101} faces. Oscillation and Weissenberg photographs were taken about the three crystallographic axes with nickel-filtered Cu $K\alpha$ radiation and gave the monoclinic unit cell dimensions:

$$a = 10{\cdot}02 \pm 0{\cdot}10,\; b = 7{\cdot}67 \pm 0{\cdot}08,\; c = 7{\cdot}28 \pm 0{\cdot}07\ \text{Å}\,,\quad \beta = 102^\circ\ 38'\,.$$

There are 4 molecules per unit cell. Density: calculated 1·61 g.cm.$^{-3}$; observed (flotation) 1·55 g.cm.$^{-3}$.

Reflections of type ($h0l$) were observed only when $h+l = 2n$ and reflections of the type ($0k0$) only when $k = 2n$. These data fix the space group as C_{2h}^5–$P2_1/n$.

The indices of refraction, measured by means of standard refraction liquids, were:

$$n_\alpha = 1{\cdot}585 \pm 0{\cdot}003,\; n_\beta = 1{\cdot}78 \pm 0{\cdot}01,\; n_\gamma = 1{\cdot}81 \pm 0{\cdot}01\,.$$

The powder pattern was run using nickel-filtered Cu $K\alpha$ radiation and Eastman Kodak No-Screen X-ray film. The intensities of the most intense lines, estimated visually by comparison with standard intensity strips prepared by timed exposures of one reflection from a single crystal, are summarized in Table 1.

Table 1. *Powder diffraction data*

d (Å)	I/I_0	d (Å)	I/I_0
3·25	0·23	1·82	0·09
3·04	0·09	1·70	1·00
2·48	0·26	1·30	0·34
2·20	0·34	1·25	0·26
2·00	0·44	1·20	0·23
1·95	0·13		

The intensities of 926 unique (hkl) reflections have been estimated visually by comparison with the standard intensity strips and a three-dimensional Patterson synthesis has been prepared. Attempts to solve this structure have thus far proven unsuccessful. However, work is continuing towards the solution of this problem.

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