The Crystal Structure of Tin(II) Iodide

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CONTRADICTORY reports on tin(II) iodide[1,2] have prompted a thorough investigation of the compound. Pure tin(II) iodide was prepared as brilliant red needles by heating under reflux a critical concentration of iodine in 2N-HCl with tin; the reaction mixture was cooled slowly under an atmosphere of nitrogen.

Crystal data: $\mathrm{SnI_2}$, M = 372·5, monoclinic, $a = 14{\cdot}29$, $b = 4{\cdot}53$, $c = 10{\cdot}72$ Å, $\beta = 92°$, $U = 695{\cdot}5$ Å^3, $D_m = 5{\cdot}28$, $Z = 6$, $D_c = 5{\cdot}33$. Space group *Cm* (C_s^3, No. 8). Cu-K_α radiation, oscillation, rotation, and Weissenberg photographs about *b*.

With programmes made available by Daly, Stephens, and Wheatley,[3] the structure was determined from 235 independent reflections measured on a Hilger Watt Y-190 single-crystal linear diffractometer with Mo-K_α filtered radiation. Statistical analysis of the *h0l* intensities indicated a non-centric distribution, *i.e.*, space-group *Cm* rather than *C*2 or *C*2/*m*. A three-dimensional Patterson synthesis confirmed the arrangement of all atoms in layers parallel to 010 and separated by *b*/2, as indicated already by the correspondence of the *hkl* and *h*, *k* + 2,*l* reflections. The requirements of space-group and cell-content are met by nine independent atoms (3Sn, 6I) lying in special positions on the mirror plane at $y = 0$, with a symmetry-related set at $y = \frac{1}{2}$. The approximate positions of four of the nine independent atoms were determined from the zero layer of the Patterson synthesis. The remaining positions were located from Fourier and difference maps, by using a fast structure factor routine and an overall isotropic temperature factor. Least-squares refinement (intensities weighted according to Cruickshank[4]) with individual isotropic temperature factors brought *R* down to a present value of 0·102.

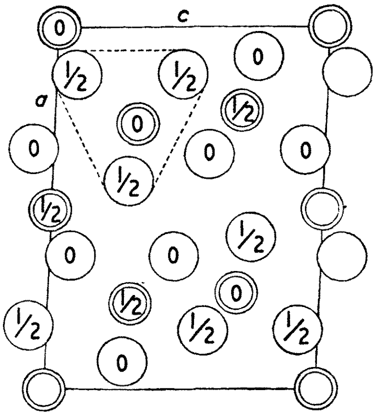

FIGURE. *Tin*(II) *iodide*; *projection down* b. *Iodine atoms are represented by single circles, tin atoms by smaller double circles. One* $(\mathrm{SnI_3})_n^{n-}$ *prism is outlined. The* y *co-ordinates of the atoms are indicated in the centres of the circles as fractions of* b.

The structure is best illustrated by a projection down *b* (see Figure). It may be thought of in terms of infinite $(\mathrm{SnI_3})_n^{n-}$ trigonal prisms parallel to *b*, having $y_\mathrm{I} = \frac{1}{2}$, $y_\mathrm{Sn} = 0$ (one of these outlined in the Figure) or $y_\mathrm{I} = 0$, $y_\mathrm{Sn} = \frac{1}{2}$. The co-ordination of the tin atoms in these prisms is seven-fold, as each has a close iodine neighbour (in the same layer) in an adjacent prism in addition to six (in layers above and below) in its own prism. The co-ordination is similar to that in $SnCl_2$[5] ($PbCl_2$ structure), generally regarded as nine-fold, except that two of the halogen atoms in the same layer are very much more distant.

The resulting prism pairs are connected to adjacent pairs by other tin atoms which are six-fold co-ordinated by iodine in a distorted octahedral arrangement. There are twice as many seven-co-ordinate as six-co-ordinate tin atoms. A six-co-ordinate tin of the $y = 0$ layer has been chosen as the origin to facilitate comparison with related structures.

Deletion of the six-co-ordinate tin atoms leaves an AB_3 structure, almost identical with the $PuBr_3$ structure found in lanthanide and actinide bromides and iodides.[6] The unit cell of PuI_3 closely resembles that of SnI_2, having 0, $\frac{1}{2}$ layering, and similar cell dimensions $a = 14{\cdot}0$, $b = 4{\cdot}30$, $c = 9{\cdot}91$.

There are also certain similarities with the structure of $Cu(OH)IO_3$,[7] if one deletes the OH and half of the Cu to give the same stoicheiometry, although the layering in $Cu(OH)IO_3$ is not uniformly 0, $\frac{1}{2}$.

The SnI_2 structure, unlike that of SnI_4 contains no discrete molecular units. The solid might be formulated as $Sn(SnI_3)_2$.

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