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# Structure of Caesium Triiodostannate(II)

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**Abstract.** $Cs[SnI_3]$, $M_r = 632{\cdot}3$, orthorhombic, *Pnam*, $a = 10{\cdot}328$ (2), $b = 17{\cdot}677$ (3), $c = 4{\cdot}765$ (2) Å, $V = 869{\cdot}94$ Å$^3$, $Z = 4$, $D_m = 4{\cdot}80$, $D_c = 4{\cdot}83$ Mg m$^{-3}$, $\mu$(Mo $K\alpha$) = 16·515 mm$^{-1}$, $F(000) = 1052$. The final $R = 0{\cdot}051$ for 570 observed [$|F_o| > 4\sigma(F_o)$] reflections. Sn is at the centre of a distorted octahedron of I atoms [Sn–I = 2·941 (3), 3·197 (2) 3·227(2), 3·469 (3) Å]. The octahedra form double chains along **c** which are linked by Cs. $Cs[SnI_3]$ is isomorphous with $Rb[PbI_3]$ and $Cs[PbI_3]$.

**Introduction.** Only a few halogenostannates(II) have been characterized by diffraction methods, and a systematic comparison of crystal structures of these compounds is not yet possible. Trying to prepare new halogenostannates(II) and to investigate their structure, we obtained $Cs[SnI_3]$ as an example with the anionic moiety $[SnI_3]^-$. Only some results by J. Watts on the structure of this compound have been reported (Scaife, Weller & Fisher, 1974). We have independently determined the structure of $Cs[SnI_3]$.

$Cs[SnI_3]$ was obtained as yellow, long, thin needles from the reaction of $SnI_2$ with CsI in 2·5 *M* HCl. The product is easily oxidized and for the diffraction study the crystals were sealed in thin-walled glass capillaries. Preliminary precession and Weissenberg photographs indicated an orthorhombic lattice with systematic absences (0*kl*, $k + l = 2n + 1$, and *h*0*l*, $h = 2n + 1$) consistent with the space groups *Pnam* and $Pna2_1$. In accordance with the structure of $Cs[PbI_3]$ (Møller, 1959) and $Rb[PbI_3]$ (Haupt, Huber & Preut, 1974), *Pnam* was adopted. The choice was confirmed by the successful analysis.

The intensity measurements were made on a Hilger & Watts four-circle diffractometer controlled by a PDP-8I computer (Mo $K\alpha$ radiation, $\lambda = 0{\cdot}70926$ Å, graphite monochromator and scintillation counter). Cell dimensions and the orientation matrix were determined by least squares from the angular settings of 21 reflections. A complete set of 746 symmetry-independent reflections were collected in the range $2 \le 2\theta \le 30{\cdot}9°$ in an $\omega$–$2\theta$ scan mode with 70 steps [scan width $\Delta 2\theta = (1{\cdot}34 + 0{\cdot}34 \text{ tg } \theta)°$ from background to background], an $\omega$ step rate of 0·01° s$^{-1}$ and a $2\theta$ step rate of 0·02° s$^{-1}$. Backgrounds were measured at each end of the scan range for 7 s. Four standard reflections showed no significant change in intensity. The intensities were corrected for background and then reduced after correction for the Lorentz effect. No absorption correction was made. After averaging of the equivalent reflections the data set contained 673 independent reflections. The final *R* is based on 570 reflections with $|F_o| > 4\sigma(F_o)$.

The structure was solved by Patterson and Fourier methods with *SHELX* (Sheldrick, 1976) and refined by least-squares methods. The refinement converged to *R*

Table 1. *Positional parameters* (×10$^4$) *of the atoms in* $Cs[SnI_3]$

Numbers in parentheses here and throughout the paper give the e.s.d.'s on the least significant digits.

| | *x* | *y* | *z* |
|---|---|---|---|
| Cs | 4144 (2) | 8300 (1) | 2500 |
| Sn | 1574 (2) | 634 (1) | 2500 |
| I(1) | 2956 (2) | 2089 (1) | 2500 |
| I(2) | 1662 (2) | 5008 (1) | 2500 |
| I(3) | 310 (2) | 8815 (1) | 2500 |

Table 2. *Distances* (Å) *and angles* (°) *in* $Cs[SnI_3]$ (*see Fig.* 2 *for numbering of atoms*)

| | | | |
|---|---|---|---|
| Sn–I(1) | 2·941 (3) | I(1)–I(2)″ | 4·400 (3) |
| Sn–I(2)″ | 3·197 (2) | I(1)–I(3)′ | 4·427 (3) |
| Sn–I(2)‴ | 3·197 (2) | I(2)″–I(3) | 4·461 (3) |
| Sn–I(3) | 3·469 (3) | I(2)″–I(3)′ | 4·304 (3) |
| Sn–I(3)′ | 3·227 (2) | I(3)–I(3)′ | 4·861 (2) |
| Sn–I(3)‴ | 3·227 (2) | Cs–I(2)″ | 3·934 (2) |
| | | Cs–I(3) | 4·064 (3) |

| | | | |
|---|---|---|---|
| I(1)–Sn–I(2)″ | 91·50 (7) | I(2)″–Sn–I(3)‴ | 176·81 (8) |
| I(1)–Sn–I(2)‴ | 91·50 (7) | I(2)‴–Sn–I(3) | 83·90 (6) |
| I(1)–Sn–I(3) | 173·08 (8) | I(2)‴–Sn–I(3)′ | 176·81 (8) |
| I(1)–Sn–I(3)′ | 91·63 (7) | I(2)‴–Sn–I(3)‴ | 84·14 (6) |
| I(1)–Sn–I(3)‴ | 91·63 (7) | I(3)–Sn–I(3)′ | 93·03 (6) |
| I(2)″–Sn–I(2)‴ | 96·36 (7) | I(3)–Sn–I(3)‴ | 93·03 (6) |
| I(2)″–Sn–I(3) | 83·90 (6) | I(3)′–Sn–I(3)‴ | 95·19 (6) |
| I(2)″–Sn–I(3)′ | 84·14 (6) | | |

Symmetry code: (′) $-x, -y, \frac{3}{4}$; (″) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{3}{4}$; (‴) I(2)″ and I(3)′ at $z = \frac{3}{4} - 1$.

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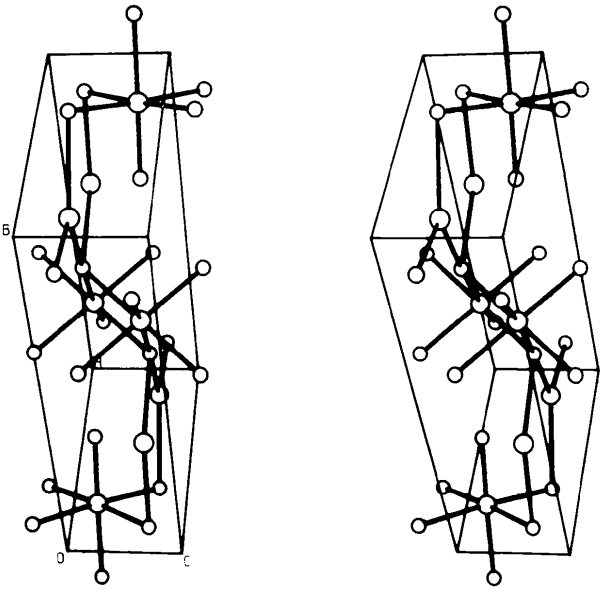


Fig. 1. Unit cell of $Cs[SnI_3]$.

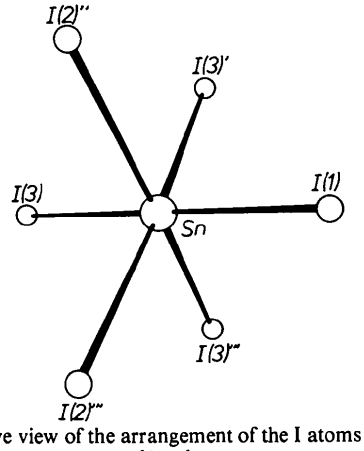


Fig. 2. Perspective view of the arrangement of the I atoms about Sn in $Cs[SnI_3]$.

$= \sum |F_o - F_c| / \sum |F_o| = 0{\cdot}051$.* Scattering factors were taken from Cromer & Mann (1968) and Doyle & Turner (1968). The final parameters of the atoms are given in Table 1, distances and angles in Table 2. A *PLUTO* (Motherwell & Clegg, 1978) stereoplot of the arrangement of the atoms in the unit cell is shown in Fig. 1. Fig. 2 is a perspective view of the octahedral coordination of one Sn by the six nearest I atoms. The numbering scheme is also given.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34985 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

**Discussion.** $Cs[SnI_3]$ is isomorphous with $Cs[PbI_3]$ (Møller, 1959) and $Rb[PbI_3]$ (Haupt, Huber & Preut, 1974). The structure is characterized by parallel double chains of distorted $SnI_6$ octahedra. This kind of coordination has already been recognized (Scaife, Weller & Fisher, 1974). The double chains are linked by Cs. Each octahedron (Fig. 2) shares opposite edges [I(3)'–I(2)'' and I(2)'''–I(3)'''] of the equatorial plane with two neighbouring octahedra, thereby forming a chain along **c**, and it shares two edges [I(3)'–I(3) and I(3)'''–I(3)] with two octahedra of a parallel chain, thereby forming the double chain.

The Sn–I distances differ depending on the function of the I atoms in the double chain. Distances between Sn and linking I atoms within the equatorial plane are 3·227 (2) Å when the I atoms [I(3)' and I(3)'''] are part of an octahedron of a parallel chain, but 3·197 (2) Å when this is not the case [I(2)'' and I(2)''']. The longest Sn–I distance [3·469 (3) Å] is observed between Sn and the axial I(3), which is part of the equatorial plane of the parallel chain, while the distance to I(1), *trans* to I(3), and not involved in linking octahedra, is the shortest at 2·941 (3) Å. (The corresponding values found by J. Watts are 3·19, 3·23, 3·46, 2·96 Å.) It is remarkable that, unlike in other halogenostannates(II), in $Cs[SnI_3]$ the inert 5*s* pair is not stereochemically active.

The Cs atoms are situated at the centre of a nearly equilateral triangle of I atoms, each linking three double chains. Three I atoms above and three below this triangular plane supplement the coordination number of Cs to nine.

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