

Structure of Bis(2-chloroethyl)ammonium Hexachlorostannate

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Abstract. $[(\text{ClCH}_2\text{CH}_2)_2\text{NH}_2]_2\text{SnCl}_6$, $M_r = 617.48$, monoclinic, $C2/c$, $a = 15.67$ (2), $b = 13.54$ (2), $c = 13.24$ (2) Å, $\beta = 127.9$ (2)°, $V = 2217$ Å³, $Z = 4$, $D_x = 1.85$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 23.70$ cm⁻¹, $F(000) = 1208$, $T = 295$ K, $R = 0.058$, $wR = 0.071$ for 1231 unique reflections. The structure consists of discrete $(\text{ClCH}_2\text{CH}_2)_2\text{NH}_2^+$ cations and octahedral SnCl_6^{2-} anions with the Sn sitting on the twofold axis.

Experimental. The compound, the hydrolysis product of *N*-methyl-*N,N*-bis(2-chloroethyl)ammonium hexachlorostannate (Pettit & Rawson, 1981), was recrystallized from a 70/30% mixture of acetonitrile and diethyl ether to form clear platelet-shaped crystals. $\text{Cu } K\alpha$ Weissenberg photographs were taken to obtain the symmetry and unit cell. Symmetry and systematic extinctions (hkl , $h + k$ odd and $h0l$, l odd) indicated monoclinic Cc or $C2/c$ as the choices for the space group. Successful refinement in the centrosymmetric $C2/c$ proved this to be the correct space group. A 0.1–0.3 mm crystal was mounted in a low- X -ray-absorption glass capillary for data collection on a Picker diffractometer with Enraf–Nonius X -ray tube and generator using a $2.0^\circ \theta$ – 2θ scan with β -filtered $\text{Mo } K\alpha$ radiation. Cell dimensions were determined by least-squares refinement of 12 reflections in the range $22 < 2\theta < 32^\circ$. One standard reflection (200) was monitored every 30 reflections and showed no systematic variation. Intensities of 2262 reflections were measured ($2\theta < 50^\circ$, $-18 \leq h \leq 14$, $0 \leq k \leq 16$, $0 \leq l \leq 15$) with 1231 unique reflections having $F < 4\sigma(F)$. Fourier series absorption corrections (Walker & Stuart, 1983) were made after the structure was solved and refined isotropically. The Sn and two Cl atoms were located using heavy-atom Patterson methods, and subsequent cycles of structure-factor calculation and difference synthesis were used to locate the remainder of the non-H atoms. These atoms were refined anisotropically,

Table 1. Positional parameters and equivalent isotropic thermal parameters (Å²)

$$B_{\text{eq}} = (4/3)[a^2\beta(1,1) + b^2\beta(2,2) + c^2\beta(3,3) + ab(\cos\gamma)\beta(1,2) + ac(\cos\beta)\beta(1,3) + bc(\cos\alpha)\beta(2,3)].$$

	x	y	z	B_{eq}
Sn*	0.0	0.0842 (1)	0.250	2.58 (2)
Cl(1)	0.1881 (2)	0.0848 (3)	0.3273 (2)	3.74 (6)
Cl(2)	0.0446 (2)	0.2148 (3)	0.4040 (3)	4.28 (8)
Cl(3)	0.0478 (2)	-0.0419 (3)	0.4082 (3)	4.81 (9)
Cl(4)	0.0901 (2)	0.5303 (3)	0.1992 (4)	5.5 (1)
Cl(5)	0.1423 (3)	0.3324 (4)	0.0044 (3)	5.7 (1)
N	0.2852 (6)	0.3819 (9)	0.2967 (9)	3.2 (2)
C(2)	0.2030 (8)	0.361 (1)	0.318 (1)	3.9 (3)
C(3)	0.3150 (9)	0.294 (1)	0.255 (1)	4.4 (3)
C(4)	0.1660 (9)	0.454 (1)	0.341 (1)	4.5 (3)
C(5)	0.2174 (9)	0.245 (1)	0.131 (1)	4.7 (3)

* Sn is in special position 4(e) (0, y , $\frac{1}{2}$).

Table 2. Bond lengths (Å) and bond angles (°)

Atom X is related to atom X' by a twofold rotation axis through the Sn atom.

Sn—Cl(1)	2.455 (3)	N—C(2)	1.51 (2)
Sn—Cl(2)	2.455 (4)	N—C(3)	1.50 (2)
Sn—Cl(3)	2.436 (4)	C(2)—C(4)	1.50 (2)
Cl(4)—C(4)	1.80 (1)	C(3)—C(5)	1.54 (2)
Cl(5)—C(5)	1.78 (1)		
Cl(1)—Sn—Cl(2)	89.7 (1)	Cl(1)—Sn—Cl(1')	179.6 (1)
Cl(1)—Sn—Cl(3)	89.3 (1)	Cl(2)—Sn—Cl(3')	178.2 (1)
Cl(2)—Sn—Cl(3)	90.6 (1)	Cl(2)—N—C(3)	115 (1)
Cl(1)—Sn—Cl(2')	90.0 (1)	N—C(2)—C(4)	111 (1)
Cl(1)—Sn—Cl(3')	91.0 (1)	N—C(3)—C(5)	114 (1)
Cl(2)—Sn—Cl(2')	87.8 (1)	Cl(4)—C(4)—C(2)	111 (1)
Cl(3)—Sn—Cl(3')	91.1 (1)	Cl(5)—C(5)—C(3)	112 (1)

ically, with H atoms included but not refined, to $R = 0.058$ and $wR = 0.071$ based on 96 least-squares parameters, and $w^{-1} = \sigma^2(F_o) + 0.00040|F_o|^2$, $\sum w(|F_o| - |F_c|)^2$ minimized. The goodness of fit was 2.07. The H atoms were included with C—H and N—H distances fixed at 0.95 Å and isotropic thermal parameters set at 30% larger than the atoms to which they were bonded. The final shift to e.s.d. ratio was less than 0.030. The largest residual on the final difference map was $0.997 \text{ e } \text{Å}^{-3}$. All data reduction, structure solution and refinement, and graphics were executed using *MolEN* (Enraf–Nonius, 1990) on a VAX 6410 computer. Scattering factors and

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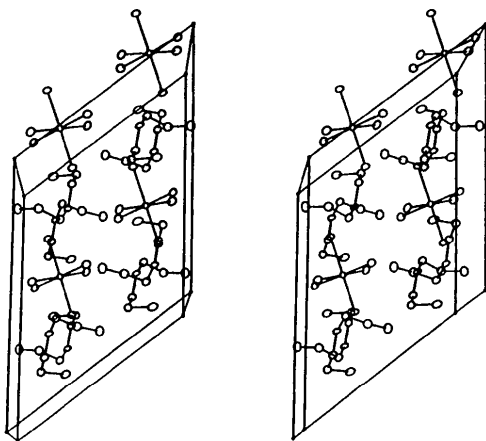


Fig. 1. Stereoscopic view of the unit cell looking down the b axis with the c axis horizontal and the a axis vertical.

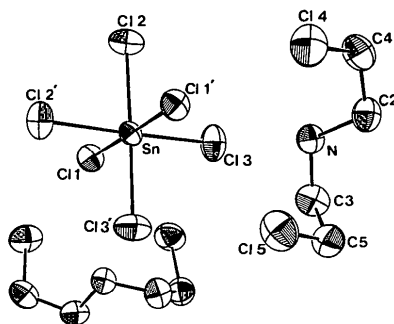


Fig. 2. Illustration of the structure of one formula unit of the title compound and the atom-numbering scheme. H atoms have been omitted.

anomalous-dispersion corrections, provided by *MolEN*, were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV) and Cromer & Mann (1968). The final equivalent isotropic thermal and positional parameters of non-H atoms are listed in Table 1.* Bond lengths and angles are presented in Table 2. All bond lengths and angles are in a normal range. Packing of the ions in a unit cell is shown in Fig. 1. Fig. 2 shows one formula unit including the atom-numbering scheme.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54990 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH0578]

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[2-(Bromodimethylstannyl)ethyl]diphenylphosphine Sulfide

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Abstract. $C_{16}H_{20}BrPSSn$, $M_r = 473.96$, orthorhombic, $P2_12_12_1$, $a = 10.535(2)$, $b = 10.936(2)$, $c = 16.500(3)$ Å, $V = 1901.0(6)$ Å³, $Z = 4$, $D_x = 1.656$ Mg m⁻³, $F(000) = 928$, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 3.61$ mm⁻¹, $T = 291(1)$ K, final $R = 0.032$ for 2900 unique observed [$F \geq 4.0\sigma(F)$] diffractometer data. The atoms bound to Sn form a distorted trigonal bipyramid with S and Br in the apical [Sn—S 2.872(2), Sn—Br 2.6504(9) Å, S—Sn—Br 175.09(4)°] and the C atoms in the equatorial posi-

tions [Sn—C 2.126(7), 2.155(6), 2.158(5) Å, C—Sn—C 120.4(2), 119.1(2), 119.3(3)°; C—Sn—Br 91.6(1), 93.7(2), 96.0(2)°; C—Sn—S 84.5(1), 85.8(2), 88.5(2)°]. The atoms bound to P form a slightly distorted tetrahedron with bond angles in the range 106.3(3) to 112.5(2)°. The five-membered ring has a half-chair conformation; the atoms P, S, Sn and C(3) are nearly coplanar. The molecules are separated by normal van der Waals contacts.