

Table 3. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
N3—H3N...O9	0.77 (10)	2.785 (7)	2.09 (10)	152 (8)
N5—H5N...N3	0.76 (7)	2.892 (7)	2.61 (10)	104 (8)
N5—H5N...O9	0.76 (7)	2.750 (6)	2.03 (7)	158 (9)
O2—H2P...O7	1.27 (7)	3.326 (6)	2.74 (9)	106 (4)
O10—H2P...O4	1.23 (7)	3.422 (7)	2.90 (9)	104 (4)
O11—H1O1...O5	0.73 (7)	2.748 (6)	2.07 (8)	154 (9)
O12—H1O2...O13	0.96 (8)	2.748 (7)	1.79 (8)	176 (7)
O12—H2O2...O9	0.84 (8)	3.548 (8)	2.88 (9)	138 (7)
O12—H2O2...O10	0.84 (8)	2.991 (7)	2.18 (8)	164 (8)
O13—H1O3...O2	0.92 (9)	3.583 (6)	2.93 (8)	129 (6)
O13—H1O3...O3	0.92 (9)	2.829 (6)	1.95 (9)	161 (8)
O14—H2O4...O5	0.81 (9)	2.80 (1)	2.03 (9)	160 (7)
O15—H2O5...O14	0.80 (9)	2.81 (1)	2.05 (9)	159 (8)
O13—H2O3...O8 ⁱ	0.84 (7)	2.730 (6)	1.91 (8)	165 (8)
O3—H1P...O11 ⁱ	0.93 (8)	2.532 (7)	1.61 (9)	171 (8)
N1—H1N...O8 ⁱⁱ	0.95 (10)	2.916 (8)	2.02 (10)	155 (9)
N6—H6N...O8 ⁱⁱ	0.96 (10)	2.831 (7)	1.88 (10)	172 (8)
N1—H1N...O10 ⁱⁱ	0.95 (10)	3.390 (7)	2.59 (10)	141 (8)
O15—H1O5...O6 ⁱⁱ	0.88 (9)	2.78 (1)	1.92 (9)	166 (7)
O16—H1O6...O2 ⁱⁱ	1.24 (7)	2.881 (7)	1.66 (6)	165 (5)
N2—H2N...O1 ⁱⁱⁱ	0.89 (6)	2.759 (5)	1.89 (6)	164 (8)
N4—H4N...O1 ⁱⁱⁱ	0.80 (10)	2.771 (6)	2.00 (10)	163 (9)
O11—H2O1...O16 ^{iv}	0.82 (10)	2.72 (1)	1.97 (9)	152 (8)
O14—H1O4...O12 ^v	0.76 (9)	2.830 (8)	2.18 (8)	144 (9)

Symmetry codes: (i) $x, 1 + y, z$; (ii) $x, y, z - 1$; (iii) $x - 1, y - 1, z - 1$; (iv) $x, y - 1, z$; (v) $1 + x, y, z$.

Program(s) used to solve structure: *SHELX76* (Sheldrick, 1976). Program(s) used to refine structure: *SHELX76*. Molecular graphics: *ORTEP* (Johnson, 1965), *PLUTO* (Motherwell & Clegg, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983), *ABRAHAMS* (Gilli, 1977).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PT1022). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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catena-Poly[*cis*-dichloro-*trans*-dimethyltin(IV)- μ -*meso*-1,2-bis(propylsulfinyl)ethane-O:O']

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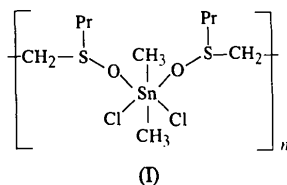
Abstract

The structure of the title compound, $[\text{SnCl}_2(\text{CH}_3)_2(\text{C}_8\text{H}_{18}\text{O}_2\text{S}_2)]_n$, has been determined and revealed the occurrence of infinite chains of tin complexes bridged by disulfoxide derivatives. The Sn^{IV} atom is hexacoordinate in a distorted octahedral environment, with Cl atoms at distances of 2.521 (1) and 2.515 (1) Å, and methyl carbanions at distances of 2.110 (4) and 2.111 (4) Å. Two different disulfoxide groups, with Sn—O distances of 2.316 (3) and 2.320 (3) Å, complete the coordination. NMR spectroscopy indicated the occurrence of discrete binuclear molecules in solution.

Comment

Dihalide-organotin compounds form adducts with a number of ligands and exhibit an important variety of structural features depending on the binding mode of the ligand. A number of these adducts of organotin and sulfoxide derivatives have been studied due to their potential application in biological systems (de Sousa, Filgueiras, Darenbourg & Reibenspies, 1992).

It was observed for the title compound, (I), in the solid phase, that the chelating $\{(C_3H_7)SOCH_2\}_2$ ligand bonds through both O atoms and forms bridges between the tin cations generating chains along the n glide. This kind of chain is also observed when a phenyl group replaces the propyl group (Carvalho, Francisco, Gambardella, de Sousa & Filgueiras, 1996), but is not always the case. For example, it was verified that the structure of $\{[Sn(C_6H_5)_3Cl]\{rac-[(C_3H_7)SOCHCHSO(C_3H_7)]\}\}$ occurs as discrete binuclear molecules, with two tin cations bridged by one $\{(C_3H_7)SOCH\}_2$ ligand (Filgueiras, Holland, Johnson & Raithby, 1982).



A $CH_3-Sn-CH_3$ bond angle of 128.7° was calculated from ^{119}Sn NMR spectroscopy in solution (Lockhart & Manders, 1986) and suggests trigonal-bipyramidal geometry for the Sn atom in a binuclear complex with the $\{(C_3H_7)SOCH_2\}_2$ ligand bridging the two cations (de Sousa *et al.*, 1995).

The isotropic displacement parameters equivalent to the best refined anisotropic displacement parameters (Hamilton, 1959) are given in Table 1 for the title compound, (I), in the solid state. The asymmetric part of the polymer is shown in Fig. 1. In the structure of (I), the C3 atom is bonded to atom C4' [which is related to atom C4 by the n glide and translated -1 along z ($\frac{1}{2} + x, \frac{1}{2} - y, z - \frac{1}{2}$)]. Similarly, atom C4 is bonded to C3' [related by $(x - \frac{1}{2}, \frac{1}{2} - y, \frac{1}{2} + z)$]. The packing mode is shown in Fig. 2.

The bond distances and angles around the Sn^{IV} atom are comparable to those observed for other compounds with *trans* dimethyl carbanions, *cis* dichlorides and

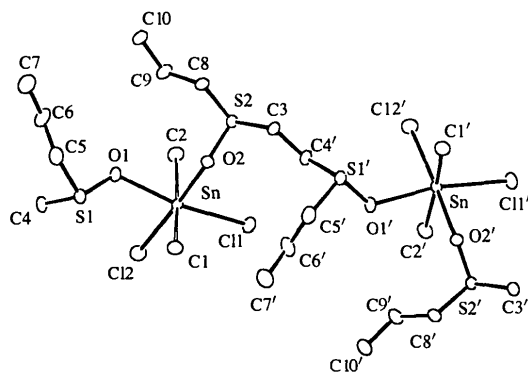


Fig. 1. An ORTEP (Johnson, 1965) partial view of the $[SnCl_2(CH_3)_2\{(C_3H_7)SOCH_2\}_2]_n$ chain shown with 20% probability displacement ellipsoids.

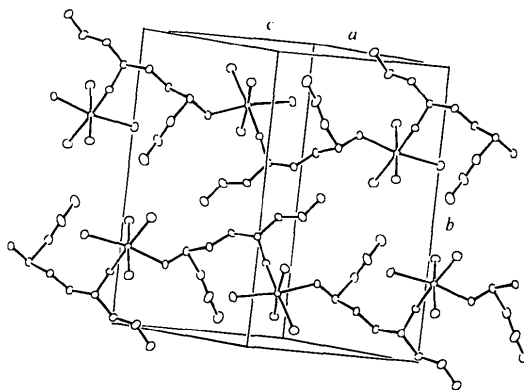


Fig. 2. A view of the chain packing of (I).

cis di-O ligands (Lockhart & Manders, 1987). For example, bond angles $CH_3-Sn-CH_3$ of $172.5(1)$, $Cl-Sn-Cl$ of $97.82(5)$ and $O-Sn-O$ of $85.0(1)^\circ$ have been observed for $Me_2SnCl_2 \cdot 2DMSO$ (Aslanov, Ionov, Attiya, Permin & Petrosyan, 1978).

Experimental

Single crystals of $[SnCl_2(CH_3)_2\{(C_3H_7)SOCH_2\}_2]_n$ were obtained by the 1:1 reaction of *meso*-($C_3H_7SOCH_2$)₂ with $(CH_3)_2SnCl_2$ in dichloromethane.

Crystal data

$[SnCl_2(CH_3)_2(C_8H_{18}O_2S_2)]$
 $M_r = 430.03$
 Monoclinic
 $P2_1/n$
 $a = 10.4375(7) \text{ \AA}$
 $b = 15.835(1) \text{ \AA}$
 $c = 11.0997(8) \text{ \AA}$
 $\beta = 104.151(6)^\circ$
 $V = 1778.86(2) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.606 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation
 $\lambda = 0.7107 \text{ \AA}$
 Cell parameters from 25 reflections
 $\theta = 10.0-18.1^\circ$
 $\mu = 1.962 \text{ mm}^{-1}$
 $T = 298 \text{ K}$
 Prismatic
 $0.30 \times 0.30 \times 0.15 \text{ mm}$
 Colorless

Data collection

Enraf-Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
 $T_{min} = 0.613$, $T_{max} = 1.000$
 3788 measured reflections
 3598 independent reflections

3038 observed reflections
 $[F > 3\sigma(F)]$
 $R_{int} = 0.0201$
 $\theta_{max} = 26.3^\circ$
 $h = -13 \rightarrow 12$
 $k = 0 \rightarrow 19$
 $l = 0 \rightarrow 13$
 3 standard reflections
 frequency: 120 min
 intensity decay: 2.1%

Refinement

Refinement on F^2
 $R(F) = 0.0325$
 $wR(F^2) = 0.0927$
 $S = 1.098$

$(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 1.52 \text{ e \AA}^{-3}$
 $\Delta\rho_{min} = -0.82 \text{ e \AA}^{-3}$
 Extinction correction: none

3030 reflections
167 parameters
H atoms refined as riding atoms
 $w = 1/[\sigma^2(F_o^2) + (0.0573P)^2 + 1.8856P]$
where $P = (F_o^2 + 2F_c^2)/3$

Atomic scattering factors
from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1065). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	x	y	z	U_{eq}
Sn	0.16188 (2)	0.20067 (1)	0.38845 (2)	0.0363 (1)
Cl1	0.3826 (1)	0.17381 (8)	0.3414 (1)	0.0639 (3)
Cl2	0.1764 (1)	0.08925 (8)	0.5527 (1)	0.0691 (3)
O1	-0.0363 (3)	0.2487 (2)	0.4224 (3)	0.0529 (7)
S1	-0.1648 (1)	0.19913 (1)	0.4040 (1)	0.0480 (3)
C1	0.2429 (5)	0.2952 (3)	0.5187 (5)	0.059 (1)
C2	0.0602 (5)	0.1177 (3)	0.2486 (4)	0.055 (1)
C3	0.2083 (4)	0.3828 (3)	0.0603 (4)	0.0476 (9)
C4	-0.1701 (4)	0.1696 (3)	0.5591 (4)	0.050 (1)
C5	-0.2929 (5)	0.2772 (3)	0.3646 (5)	0.058 (1)
C6	-0.2850 (6)	0.3486 (4)	0.4530 (6)	0.081 (2)
C7	-0.3910 (8)	0.4145 (5)	0.4017 (8)	0.103 (2)
O2	0.1234 (3)	0.2970 (2)	0.2266 (3)	0.0463 (6)
S2	0.1819 (1)	0.38357 (6)	0.21418 (9)	0.0432 (2)
C8	0.0457 (5)	0.4549 (3)	0.1895 (4)	0.054 (1)
C9	0.0073 (7)	0.4680 (3)	0.3130 (6)	0.074 (2)
Cl0	-0.0968 (7)	0.5346 (4)	0.3039 (6)	0.077 (2)

Table 2. Selected geometric parameters (\AA , $^\circ$)

Sn—Cl1	2.520 (1)	S1—C5	1.795 (5)
Sn—Cl2	2.515 (1)	S1—C4	1.798 (4)
Sn—O1	2.320 (3)	C4—C3 ⁱ	1.519 (6)
Sn—O2	2.316 (3)	O2—S2	1.520 (3)
Sn—C1	2.110 (4)	S2—C8	1.783 (4)
Sn—C2	2.111 (4)	S2—C3	1.796 (4)
O1—S1	1.523 (3)	C3—C4 ⁱⁱ	1.519 (6)
Cl1—Sn—Cl2	97.80 (5)	O2—Sn—C2	83.1 (2)
Cl1—Sn—O1	170.37 (8)	C1—Sn—C2	172.3 (2)
Cl1—Sn—O2	86.82 (8)	Sn—O1—S1	126.9 (2)
Cl1—Sn—C1	91.6 (2)	O1—S1—C5	104.9 (2)
Cl1—Sn—C2	93.6 (1)	O1—S1—C4	103.8 (2)
Cl2—Sn—O1	90.58 (8)	C5—S1—C4	102.6 (2)
Cl2—Sn—O2	173.13 (8)	S1—C4—C3 ⁱ	111.8 (3)
Cl2—Sn—C1	93.3 (2)	Sn—O2—S2	131.9 (2)
Cl2—Sn—C2	91.5 (1)	O2—S2—C8	105.2 (2)
O1—Sn—O2	85.3 (1)	O2—S2—C3	103.8 (2)
O1—Sn—C1	83.1 (2)	C8—S2—C3	99.2 (2)
O1—Sn—C2	90.9 (2)	S2—C3—C4 ⁱⁱ	109.5 (3)
O2—Sn—C1	91.6 (2)		

Symmetry codes: (i) $x - \frac{1}{2}, \frac{1}{2} - y, \frac{1}{2} + z$; (ii) $\frac{1}{2} + x, \frac{1}{2} - y, z - \frac{1}{2}$.

During the refinement process, anisotropic displacement parameters were assigned to all non-H atoms. The H-atom positions were calculated; they were refined as riding atoms and assigned isotropic displacement parameters of 0.076\AA^2 .

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *SDP* (Frenz, 1978). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965).

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catena-Poly[*cis*-dichloro-*trans*-di-methyltin(IV)- μ -*meso*-1,2-bis(phenyl-sulfinyl)ethane-*O*:*O'*]

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Abstract

The coordination of the Sn atom in the title compound, $[\text{SnCl}_2(\text{CH}_3)_2(\text{C}_{14}\text{H}_{14}\text{O}_2\text{S}_2)]_n$, is octahedral, with chlorides at *cis* [Sn—Cl 2.482 (1) and 2.486 (1) Å] and