

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)

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

	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).

This work was sponsored by grants from FAPESP, CNPq, FINEP and CAPES, which are hereby gratefully acknowledged.

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Acta Cryst. (1996). **C52**, 1629–1631

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

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(Received 11 April 1995; accepted 19 February 1996)

Abstract

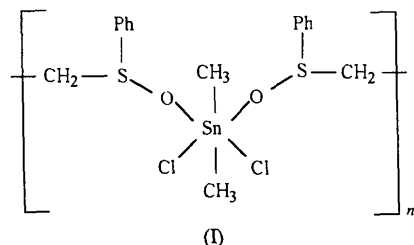
The coordination of the Sn atom in the title compound, $[\text{SnCl}_2(\text{CH}_3)_2(\text{C}_{14}\text{H}_{14}\text{O}_2\text{S}_2)]_n$, is octahedral, with chlorides at *cis* [Sn—Cl 2.482 (1) and 2.486 (1) Å] and

methyl groups at *trans* positions [Sn—C 2.101 (4) and 2.105 (5) Å]. Completing the coordination, there are two different sulfoxide groups in a *cis* orientation [Sn—O 2.397 (3) and 2.404 (3) Å]. Each sulfoxide is bound to two Sn atoms, yielding infinite ribbons.

Comment

Organotin halides have an extremely rich and variable chemistry and a number of compounds contain special ligands with biological significance (Lee, Gabe, Khoo, Eng & Smith, 1990). Organotin halides with sulfoxide groups have been studied recently in order to clarify their structural features (de Sousa, Filgueiras, Darsenbourg & Reibenspies, 1992).

The structure determination of [SnCl₂(CH₃)₂(C₁₄H₁₄O₂S₂)]_n, (I), revealed the occurrence of infinite chains along the crystallographic *a* axis. The Sn⁴⁺ cations are connected by *meso*-1,2-bis(phenylsulfinyl)ethane molecules, which act as bidentate *O*:*O'* chelating ligands, as shown in Fig. 1.



Each *meso*-1,2-bis(phenylsulfinyl)ethane molecule has an inversion centre at the C—C (ethane) midpoint. The bond distance and angle values for *meso*-1,2-bis(phenylsulfinyl)ethane are comparable with those determined for a platinum complex (Francisco, Gambardella, Rodrigues, de Sousa & Filgueiras, 1995).

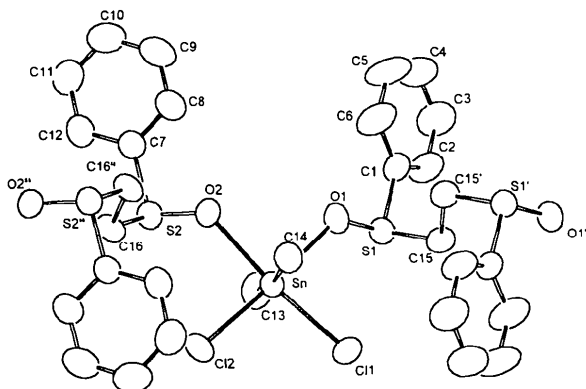


Fig. 1. An ORTEP (Johnson, 1965) view of part of the infinite chain [SnCl₂(CH₃)₂(C₁₄H₁₄O₂S₂)]_n. Displacement ellipsoids are plotted at the 50% probability level.

Experimental

Single crystals of [SnCl₂(CH₃)₂(C₁₄H₁₄O₂S₂)]_n were obtained by a 1:1 reaction of *meso*-(PhSOCH₂)₂ with Me₂SnCl₂ in CH₂Cl₂.

Crystal data

[SnCl₂(CH₃)₂(C₁₄H₁₄O₂S₂)]
M_r = 498.03
 Monoclinic
*P*2₁/*c*
a = 16.445 (2) Å
b = 6.3075 (6) Å
c = 21.092 (3) Å
 β = 112.02 (1)°
V = 2028.2 (7) Å³
Z = 4
D_r = 1.631 Mg m⁻³

Data collection

Enraf-Nonius CAD-4
 diffractometer
 $\omega/2\theta$ scans
 Absorption correction:
 ψ scan (North, Phillips
 & Matthews, 1968)
T_{min} = 0.832, *T_{max}* =
 0.999
 4230 measured reflections
 4104 independent reflections

Refinement

Refinement on *F*²
R(*F*) = 0.0334
 $\omega R(F^2)$ = 0.0836
S = 1.038
 4104 reflections
 214 parameters
 H atoms riding
 $w = 1/[\sigma^2(F_o^2) + (0.0531P)^2 + 1.2731P]$
 where $P = (F_o^2 + 2F_c^2)/3$

Mo *K* α radiation
 λ = 0.71073 Å
 Cell parameters from 25
 reflections
 θ = 10.00–18.10°
 μ = 1.734 mm⁻¹
T = 293 (2) K
 Prismatic
 0.4 × 0.2 × 0.1 mm
 Colorless

3168 observed reflections
 $[I > 2\sigma(I)]$
R_{int} = 0.0462
 θ_{max} = 26.29°
 h = -20 → 18
 k = -7 → 0
 l = 0 → 26
 3 standard reflections
 frequency: 120 min
 intensity decay: 8.5%

$(\Delta/\sigma)_{max}$ = -0.007
 $\Delta\rho_{max}$ = 0.724 e Å⁻³
 $\Delta\rho_{min}$ = -0.576 e Å⁻³
 Extinction correction: none
 Extinction coefficient: none
 Atomic scattering factors
 from *International Tables
 for Crystallography* (1992,
 Vol. C, Tables 4.2.6.8 and
 6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
Sn1	0.25785 (2)	0.19297 (4)	0.01932 (1)	0.0384 (1)
C11	0.40228 (7)	0.0193 (2)	0.07883 (6)	0.0552 (3)
C12	0.17348 (8)	0.0135 (2)	0.07984 (7)	0.0620 (3)
O1	0.3283 (2)	0.3846 (6)	-0.0440 (2)	0.0542 (8)
S1	0.39773 (7)	0.2832 (2)	-0.06527 (5)	0.0444 (2)
C1	0.3896 (3)	0.4170 (8)	-0.1417 (2)	0.045 (1)
C2	0.4268 (4)	0.329 (1)	-0.1828 (3)	0.066 (1)
C3	0.4191 (4)	0.428 (1)	-0.2434 (3)	0.084 (2)
C4	0.3745 (5)	0.610 (1)	-0.2621 (3)	0.085 (2)
C5	0.3378 (5)	0.698 (1)	-0.2211 (3)	0.097 (2)
C6	0.3434 (4)	0.604 (1)	-0.1608 (3)	0.078 (2)
O2	0.1278 (2)	0.3869 (6)	-0.0468 (2)	0.0544 (8)
S2	0.03389 (7)	0.3082 (2)	-0.07375 (6)	0.0456 (2)
C7	-0.0239 (3)	0.4923 (8)	-0.1403 (2)	0.046 (1)
C8	0.0225 (4)	0.6531 (8)	-0.1547 (2)	0.059 (1)
C9	-0.0213 (4)	0.797 (1)	-0.2057 (3)	0.074 (2)
C10	-0.1085 (5)	0.782 (1)	-0.2403 (3)	0.085 (2)
C11	-0.1535 (4)	0.620 (1)	-0.2247 (4)	0.101 (3)
C12	-0.1123 (3)	0.471 (1)	-0.1758 (3)	0.077 (2)
C13	0.2191 (3)	-0.0200 (9)	-0.0635 (3)	0.065 (1)
C14	0.2879 (3)	0.4639 (7)	0.0819 (2)	0.050 (1)
C15	0.5009 (3)	0.3826 (7)	-0.0067 (2)	0.0424 (9)
C16	-0.0097 (3)	0.3862 (7)	-0.0104 (2)	0.0425 (9)

Table 2. Selected geometric parameters (Å, °)

Sn1—C14	2.101 (4)	S1—C1	1.780 (4)
Sn1—C13	2.105 (5)	S1—C15	1.796 (4)
Sn1—O1	2.397 (3)	O2—S2	1.515 (3)
Sn1—O2	2.404 (3)	S2—C7	1.795 (5)
Sn1—Cl2	2.482 (1)	S2—C16	1.807 (4)
Sn1—Cl1	2.486 (1)	C15—C15 ⁱ	1.511 (9)
O1—S1	1.517 (3)	C16—C16 ⁱⁱ	1.500 (9)
C14—Sn1—C13	165.2 (2)	O2—Sn1—Cl1	172.23 (8)
C14—Sn1—O1	83.7 (2)	Cl2—Sn1—Cl1	98.73 (4)
C13—Sn1—O1	85.3 (2)	S1—O1—Sn1	121.8 (2)
C14—Sn1—O2	84.2 (2)	O1—S1—C1	105.0 (2)
C13—Sn1—O2	85.2 (2)	O1—S1—C15	105.8 (2)
O1—Sn1—O2	86.03 (1)	C1—S1—C15	100.8 (2)
C14—Sn1—Cl2	95.34 (1)	S2—O2—Sn1	127.6 (2)
C13—Sn1—Cl2	94.7 (2)	O2—S2—C7	104.3 (2)
O1—Sn1—Cl2	175.04 (8)	O2—S2—C16	105.4 (2)
O2—Sn1—Cl2	89.03 (8)	C7—S2—C16	99.3 (2)
C14—Sn1—Cl1	94.94 (1)	C15 ⁱ —C15—S1	113.0 (4)
C13—Sn1—Cl1	94.2 (2)	C16 ⁱⁱ —C16—S2	112.1 (4)
O1—Sn1—Cl1	86.21 (8)		

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

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, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965).

This work was sponsored by grants from FAPESP, CNPq, FINEP and CAPES, which are hereby gratefully acknowledged.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1084). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). **C52**, 1631–1634

Phenylimidopentachloroniobate(V) Dianion with *N*-Allylphenylammonium Counterion

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(Received 11 December 1995; accepted 6 March 1996)

Abstract

Bis[phenyl(2-propenyl)ammonium] pentachloro(phenylimido)niobate(V), $(C_9H_{12}N)_2[NbCl_5(C_6H_5N)]$, is a product of the reaction of niobium pentachloride and *N*-allylphenylamine. The coordinated secondary amine loses its allyl group and forms an imino bond to the metal. Unreacted *N*-allylphenylamine behaves as a proton acceptor and is a counterion in the solid state. In the crystalline state, the asymmetric unit consists of two octahedral pentachloro(phenylimido)niobate(V) dianions and four *N*-allylphenylammonium cations.

Comment

Several routes exist for synthesizing alkylimido complexes of early transition metals: (i) azobenzene has been reported to react metathetically with $[Nb_2Cl_6-(Me_2S)_3]$ to form a monomeric phenylimido complex (Cotton, Duraj & Roth, 1984) and (ii) *tert*-butylamide lithium together with $LiNMe_2$ reacts with niobium and tantalum pentahalides to form tris(dimethylamido)-*tert*-butylimido complexes (Nugent & Harlow, 1978). It is also known that even osmium tetroxide can be used as a metal source and oxoimino derivatives are formed when osmium(VIII) oxide is allowed to react with imido selenium reagents (Chong, Oshima & Sharpless, 1977). Many of the known niobium and tantalum-imido complexes are cyclopentadienyl derivatives, for example, half-sandwich monocyclopentadienyl-methylimido complexes (Williams *et al.*, 1992) and *ansa*-metallocene derivatives with butylimido ligands (Chernega, Malcolm & Suárez, 1995).

The title complex, (I), was prepared by the direct reaction of *N*-allylphenylamine and niobium pentachloride in toluene. The pentachloro(phenylimido)niobium dianion is formed when *N*-allylphenylamine loses its allylic group. Some of the *N*-allylphenylamine molecules form ammonium ions by behaving as proton acceptors and these exist as counterions in the solid state. Two pentachloro(phenylimido)niobium dianions and four *N*-allylphenylammonium ions form the asymmetric unit.