

C(13)	0.0215 (6)	0.1101 (3)	0.4635 (4)	0.066 (2)
C(14)	0.1230 (5)	0.0922 (3)	0.5495 (4)	0.066 (2)
C(15)	-0.0217 (4)	0.0689 (2)	0.6482 (3)	0.042 (1)
C(16)	0.1055 (4)	0.0710 (2)	0.6456 (4)	0.051 (1)
C(17)	0.2068 (4)	0.0511 (3)	0.7383 (4)	0.071 (2)
C(18)	0.1814 (4)	0.0301 (3)	0.8251 (4)	0.074 (2)
C(19)	0.0534 (4)	0.0289 (3)	0.8218 (3)	0.059 (1)
C(100)	-0.544 (1)	0.1173 (7)	0.0665 (8)	0.133 (4)
C(101)	-0.4995 (8)	0.1172 (6)	-0.018 (1)	0.134 (4)
C(102)	-0.531 (1)	0.179 (1)	-0.0815 (8)	0.151 (5)
C(103)	-0.605 (2)	0.239 (1)	-0.051 (2)	0.267 (9)
C(104)	-0.646 (3)	0.225 (1)	0.031 (3)	0.45 (2)
C(105)	-0.615 (1)	0.175 (1)	0.077 (1)	0.210 (7)

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

Sn—Cl(1)	2.428 (1)	N(1)—C(8)	1.332 (5)
Sn—Cl(2)	2.430 (1)	N(1)—C(12)	1.358 (5)
Sn—Cl(3)	2.400 (1)	N(2)—C(15)	1.357 (5)
Sn—N(1)	2.281 (3)	N(2)—C(19)	1.329 (5)
Sn—N(2)	2.283 (3)	C(1)—C(2)	1.466 (6)
Sn—C(1)	2.175 (5)		
Cl(1)—Sn—Cl(2)	100.91 (5)	Cl(3)—Sn—C(1)	174.6 (1)
Cl(1)—Sn—Cl(3)	90.04 (5)	N(1)—Sn—N(2)	72.7 (1)
Cl(1)—Sn—N(1)	164.14 (9)	N(1)—Sn—C(1)	92.3 (2)
Cl(1)—Sn—N(2)	92.29 (9)	N(2)—Sn—C(1)	90.1 (2)
Cl(1)—Sn—C(1)	92.9 (1)	Sn—N(1)—C(8)	125.5 (3)
Cl(2)—Sn—Cl(3)	89.00 (5)	Sn—N(1)—C(12)	115.5 (3)
Cl(2)—Sn—N(1)	93.56 (9)	C(8)—N(1)—C(12)	118.9 (4)
Cl(2)—Sn—N(2)	165.57 (9)	Sn—N(2)—C(15)	115.5 (3)
Cl(2)—Sn—C(1)	95.0 (2)	Sn—N(2)—C(19)	125.6 (3)
Cl(3)—Sn—N(1)	83.71 (9)	C(15)—N(2)—C(19)	118.5 (4)
Cl(3)—Sn—N(2)	85.16 (9)	Sn—C(1)—C(2)	117.4 (3)

The values of T obtained from *DIFABS* (Walker & Stuart, 1983) have been normalized by multiplying them by $1/T(m)$, where $T(m)$ was the maximum value of T obtained by the program. It is recognized that the range of T values thus obtained may not be as great as might be expected from crystals of this shape.

Data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSCIAFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

The award of a Commonwealth Postgraduate Research Award (VJH) and the Australian Research Council are acknowledged.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1046). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(diethylammonium) Tetrachloro-divinylstannate(IV)

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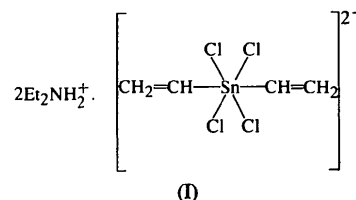
(Received 12 June 1995; accepted 10 July 1995)

Abstract

The Sn atom in the anion of the title compound, $(\text{C}_4\text{H}_{12}\text{N})_2[\text{SnCl}_4(\text{C}_2\text{H}_3)_2]$, exists in a distorted *trans*- C_2SnCl_4 octahedral geometry. Differences in the Sn—Cl separations within the anion are related to hydrogen bonding between the Cl(2) atoms of the anion and the diethylammonium cations. The average Sn—Cl distance in this and related compounds is correlated with the Lewis acidity of the diorganotin moiety.

Comment

The Sn atom in the anion of $(\text{H}_2\text{NEt}_2)_2[(\text{vin})_2\text{SnCl}_4]$, (I), where vin is vinyl, is located on a centre of symmetry and the unit cell comprises two anions and four cations. The Sn atom exists in a distorted C_2Cl_4 octahedral geometry. The independent Sn—Cl bond distances of 2.583 (2) and 2.602 (2) \AA arise as a result of a hydrogen-bonding contact between atoms H(10b) and Cl(2) at a distance of 2.41 \AA ; the Cl(2)··N(10) distance is 3.199 (6) \AA and the angle at H(10b) is 138 $^\circ$.



Similar *trans*- C_2SnCl_4 geometries have been observed previously in the structures of the $[\text{Ph}_2\text{SnCl}_4]^{2-}$ (Teoh, Teo, Yeap & Declercq, 1992) and $[\text{Me}_2\text{SnCl}_4]^{2-}$ (Valle, González, Ettore & Plazzogna, 1988) anions. The average Sn—Cl bond distances in the three structures follow the expected trends in the Lewis acidities

of the diorganotin(IV) moieties. The average Sn—Cl bond distances in the Ph_2Sn [2.583 (1) Å] and $(vin)_2Sn$ [2.593 (2) Å] complexes are equivalent, as both involve C_{sp^2} atoms, and are shorter than the distance found in the Me_2Sn compound [2.628 (1) Å].

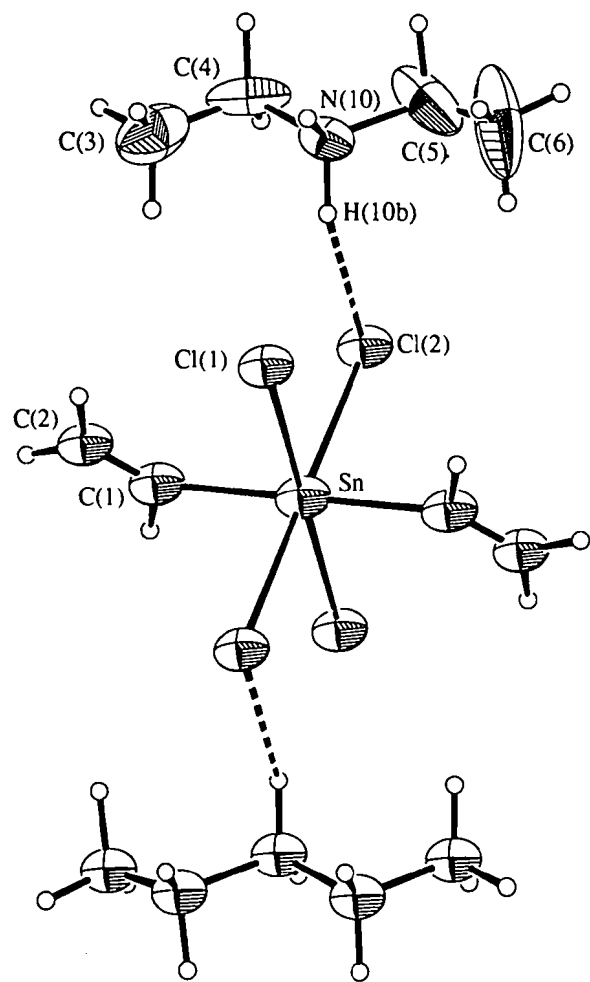


Fig. 1. The molecular structure of $(H_2NEt_2)_2[vin_2SnCl_4]$ showing 20% probability ellipsoids (ORTEP; Johnson, 1976).

Experimental

Crystals of $(H_2NEt_2)_2[(vin)_2SnCl_4]$ were isolated from the reaction mixture of $[(vin)_2SnCl_2]$ and $Na_2S_2CNEt_2$ (1/1) in hot ethanol solution (m.p. 375–377 K).

Crystal data

$(C_4H_{12}N)_2[SnCl_4(C_2H_3)_2]$

$M_r = 462.88$

Monoclinic

$P2_1/n$

$a = 7.765$ (5) Å

$b = 13.805$ (2) Å

$c = 10.292$ (3) Å

$\beta = 105.10$ (3)°

$V = 1065.2$ (6) Å³

$Z = 2$

$D_x = 1.443$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.7107$ Å

Cell parameters from 25 reflections

$\theta = 15.6$ – 18.9 °

$\mu = 1.692$ mm⁻¹

$T = 293$ K

Plate

$0.35 \times 0.24 \times 0.05$ mm

Colourless

Data collection

Rigaku AFC-6R diffractometer

$R_{int} = 0.0814$

$\theta_{max} = 27.5$ °

$\omega/2\theta$ scans

Absorption correction:

refined from ΔF (Walker & Stuart, 1983)

$T_{min} = 0.90$, $T_{max} = 1.00$

2735 measured reflections

2559 independent reflections

1151 observed reflections

$[I \geq 2.0\sigma(I)]$

$h = 0 \rightarrow 10$

$k = 0 \rightarrow 17$

$l = -13 \rightarrow 12$

3 standard reflections

monitored every 400

reflections

intensity decay: 6.47%

Refinement

Refinement on F

$R = 0.0465$

$wR = 0.0362$

$S = 1.839$

1151 reflections

88 parameters

H-atom parameters not

refined

Weighting scheme based

on measured e.s.d.'s;

$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{max} = 0.0130$

$\Delta\rho_{max} = 0.44$ e Å⁻³

$\Delta\rho_{min} = -0.45$ e Å⁻³

Atomic scattering factors

from *International Tables*

for *X-ray Crystallography*

(1974, Vol. IV)

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

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

	x	y	z	U_{eq}
Sn	0	0	0	0.0647 (2)
Cl(1)	-0.2024 (3)	-0.0808 (2)	-0.2115 (2)	0.0909 (7)
Cl(2)	0.0262 (3)	-0.1673 (2)	0.1201 (2)	0.1026 (8)
N(10)	-0.685 (1)	-0.1817 (5)	-0.6002 (7)	0.094 (3)
C(1)	-0.234 (1)	0.0287 (9)	0.060 (1)	0.117 (5)
C(2)	-0.278 (2)	0.061 (1)	0.141 (1)	0.178 (7)
C(3)	-0.444 (2)	-0.167 (1)	-0.700 (2)	0.187 (8)
C(4)	-0.519 (2)	-0.1247 (8)	-0.602 (1)	0.152 (6)
C(5)	-0.777 (2)	-0.141 (1)	-0.508 (1)	0.203 (8)
C(6)	-0.928 (2)	-0.181 (2)	-0.507 (2)	0.27 (1)

Table 2. Selected geometric parameters (Å, °)

Sn—Cl(1)	2.583 (2)	N(10)—C(5)	1.44 (1)
Sn—Cl(2)	2.602 (2)	C(1)—C(2)	1.09 (1)
Sn—C(1)	2.10 (1)	C(3)—C(4)	1.42 (2)
N(10)—C(4)	1.51 (2)	C(5)—C(6)	1.30 (2)
Cl(1)—Sn—Cl(2)	88.40 (6)	Sn—C(1)—C(2)	141 (1)
Cl(1)—Sn—C(1)	86.9 (3)	N(10)—C(4)—C(3)	107 (1)
Cl(2)—Sn—C(1)	89.7 (3)	N(10)—C(5)—C(6)	116 (1)
C(4)—N(10)—C(5)	112 (1)		

The values of T obtained from *DIFABS* (Walker & Stuart, 1983) have been normalized by multiplying them by $1/T(m)$, where $T(m)$ was the maximum value of T obtained by the program. It is recognized that the range of T values thus obtained may not be as great as might be expected from crystals of this shape.

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The award of a Commonwealth Postgraduate Research Award (VJH) and the Australian Research Council are acknowledged.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1047). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Comment

As part of a wide ranging study of metal alkoxide complexes, which are soluble analogues of and precursors to metal oxides, we have been interested in oxoalkoxides, species intermediate between homoleptic alkoxides and oxides. For molybdenum and tungsten, neutral and anionic complexes have been crystallographically characterized containing two (Abbott, Cotton & Falvello, 1990; Clegg, Errington, Fraser & Richards, 1993), four (Kang, Liu, Shaikh, Nicholson & Zubieta, 1989), five (Chen, Ma, Liu & Zubieta, 1989), six (Chisholm, Foltling, Huffman & Kirkpatrick, 1984) and eight (Liu & Zubieta, 1989) metal atoms and oxo-alkoxide stoichiometric ratios ranging from 5:4 to 16:1, the oxo ligands being in excess in most cases.

In addition to these, structures are known for a number of molybdenum and tungsten complexes with the simple empirical formula $[MO(OR)_4]$. Monomeric five-coordinate species are found for $[WO(OMe)_4]$ and $[WO(OEt)_4]$ (Kucheiko, Turova, Kozlova & Zhadanov, 1985), and for $[MoO\{OC(CF_3)_3\}_4]$ (Johnson, Taylor & Waugh, 1980) and $[WO(OC_6H_3^iPr-2,6)_4]$ (Gibson & McPartlin, 1996) with bulky substituents. Dimeric molecules with pairs of bridging alkoxide ligands giving octahedral coordination geometry are reported for $[MoO(OMe)_4]_2$ (Kessler, Mironov, Turova, Yanovsky & Struchkov, 1993), $[WO(OMe)_4]_2$ and $[WO(OC_6H_{11})_2]_2$ (Clegg, Errington, Kraxner & Redshaw, 1992). There is evidence from NMR measurements that many species of this kind are dynamic in solution, existing in both monomeric and dimeric forms (Clegg, Errington, Kraxner & Redshaw, 1992), and $[WO(OMe)_4]$ has been crystallographically characterized in both forms. The steric bulk of the alkoxide ligand substituents appears to be only one factor affecting the relative stability of the monomers and dimers.

In the course of our work, we prepared what appeared to be a second crystalline form of $[MoO(OMe)_4]$, previously shown to exist as a dimer (Kessler *et al.*, 1993). This was also found to be dimeric with alkoxide bridges, but initial refinement was unsatisfactory, with poor resolution and unacceptable geometry for one terminal alkoxide ligand in the asymmetric unit (half a dimer) of each of the two crystallographically independent molecules. Modelling of this as a disordered overlap of alkoxide and chloride ligands is successful, with no constraints or restraints necessary on the geometrical parameters. The disorder appears to be restricted to one pair of symmetry-related ligands in each molecule and is of unequal proportions in the two molecules. Molecule 1 contains only 15(2)% chloro ligands, while molecule 2 contains 34(3)%, giving an overall proportion of essentially 3:1 for the disorder, (I). The chloro ligands are due to incomplete substitution in the $[MoOCl_4]$ starting material. On purely X-ray crystallographic grounds, we are, of course, unable

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A 3:1 Disordered Solid Solution of Di- μ -methoxy-*O*:*O*-bis[trimethoxooxo-molybdenum(VI)] and Di- μ -methoxy-*O*:*O*-bis[chlorodimethoxooxomolybdenum(VI)]

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Abstract

The title structure, $[Mo_2(CH_3O)_8(O)_2]_{0.75} \cdot [Mo_2Cl_2(CH_3O)_6(O)_2]_{0.25}$, contains two crystallographically independent centrosymmetric molecules, each of which has a major $[MoO(OMe)_4]_2$ disorder component and a minor $[MoOCl(OMe)_3]_2$ disorder component, with a *cis* arrangement of the chloro and oxo ligands. The oxo ligands lie in the equatorial plane of the metal atoms and bridging ligands. The methoxy bridges are markedly asymmetric as a consequence of the strong *trans* influence of the terminal oxo ligands compared with the terminal methoxy ligands. Coordination of the Mo atoms is distorted octahedral.