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## Redetermination of [( $\mu$ -OEt)<sub>2</sub>{SnCl<sub>3</sub>(EtOH)}<sub>2</sub>]

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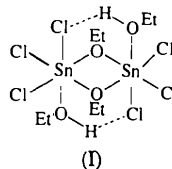
(Received 9 January 1996; accepted 27 February 1996)

## Abstract

The structure of neutral di- $\mu$ -ethanolato-bis[trichloro(ethanol)tin], [Sn<sub>2</sub>Cl<sub>6</sub>(C<sub>2</sub>H<sub>5</sub>O)<sub>2</sub>(C<sub>2</sub>H<sub>6</sub>O)<sub>2</sub>], has been re-determined and shows the dimer lying across a crystallographic inversion centre, giving an edge-shared bioctahedral structure, with the Sn atoms coordinated by an EtOH molecule, three terminal Cl<sup>-</sup> ligands and two doubly bridging ethoxide ligands, resulting in a precisely planar Sn<sub>2</sub>O<sub>2</sub> core, with bond distances Sn—Cl 2.3441 (11)–2.3947 (12), Sn—O(1) 2.164 (3), Sn—O(2) 2.092 (3) and Sn—O(2') 2.084 (3) Å. The octahedral arrangement at each Sn atom is distorted due to hydrogen-bonding interactions between the axially coordinated EtOH molecule and an adjacent chloride ligand, H(1)···Cl(2') and H(1')···Cl(2) 2.14 Å.

## Comment

During the course of our work on Group 16 complexes of tin(IV) halides, we obtained the title compound, (I), through reaction of SnCl<sub>4</sub> with an EtOH impurity in one of the dithioether ligands being studied. We report here a redetermination of the structure of (I).



The structure of (I) shows (Fig. 1) the dinuclear molecule lying across a crystallographic inversion centre, with each Sn<sup>IV</sup> centre coordinated by a neutral EtOH ligand, three terminal Cl<sup>-</sup> ligands and two  $\mu$ -OEt<sup>-</sup> ligands, giving a distorted edge-shared bioctahedral arrangement, with bond distances Sn(1)—Cl(1) 2.3441 (11), Sn(1)—Cl(2) 2.3947 (12), Sn(1)—Cl(3) 2.3504 (14), Sn(1)—O(1) 2.164 (3), Sn(1)—O(2) 2.092 (3) and Sn(1)—O(2') 2.084 (3) Å [symmetry code: (i) 1 - x, -y, 2 - z]. Although not significantly different from that originally reported (*R* = 0.079; Webster & Collins, 1974), this is a more accurate structure determination (*R* = 0.0247). Fur-

thermore, we have been able to locate the hydroxy H atom of the EtOH ligand and hence establish hydrogen-bonding between atoms H(1) and Cl(2<sup>i</sup>) [H(1)··Cl(2<sup>i</sup>) 2.14 Å and O(1)—H(1)··Cl(2<sup>i</sup>) 161.7°] and between H(1<sup>i</sup>) and Cl(2). The hydrogen bonding occurs above and below the Sn<sub>2</sub>O<sub>2</sub> ring and probably helps to stabilize it.

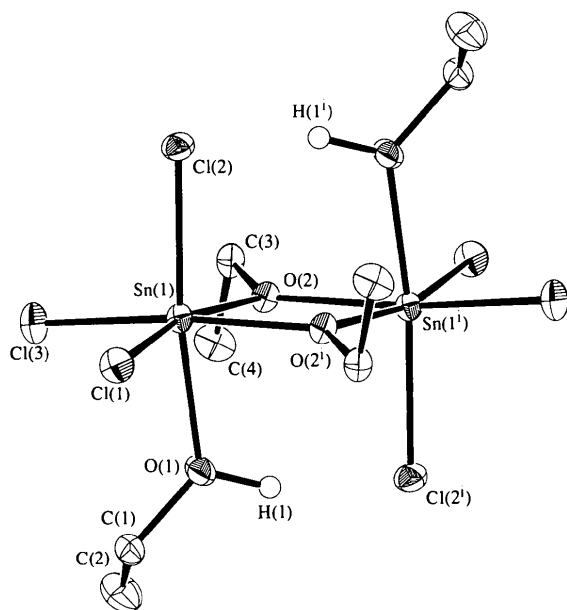


Fig. 1. A view of the structure of  $[(\mu\text{-OEt})_2\{\text{SnCl}_3(\text{EtOH})\}_2]$  with the atomic numbering scheme adopted. Displacement ellipsoids are shown at the 40% probability level and H atoms (except the hydroxy H atoms) have been omitted for clarity. The symmetry code is as given in Table 2.

The O(2)—Sn(1)—O(2<sup>i</sup>) and Sn(1)—O(2)—Sn(1<sup>i</sup>) angles involved in the planar Sn<sub>2</sub>O<sub>2</sub> core have values of 71.50(12) and 108.50(12)°, respectively. Thus, the strain of the four-membered ring is accommodated by very acute angles at the Sn atom, with those around the O atoms remaining close to tetrahedral. Very similar geometric parameters have been observed in related dinuclear species, *e.g.*  $[(\mu\text{-OMe})_2\{\text{SnCl}_3(\text{MeOH})\}_2]$  [Sn—Cl 2.341–2.385 Å, Sn—O( $\mu$ -methoxide) 2.085–2.101 Å and Sn—O(methanol) 2.117–2.172 Å] (Reuter & Schroder, 1992; Sinsel, Dou & Weiss, 1992) and  $[(\mu\text{-O}^i\text{Pr})_2\{\text{Sn}^i(\text{PrOH})(\text{O}^i\text{Pr})_3\}_2]$  [Sn—O(2-propanol) 2.142(6) Å, Sn—O(terminal isopropoxide) 1.923(7), 1.964(7) and 2.098(4) Å, and Sn—O( $\mu$ -isopropoxide) 2.080(4) and 2.091(4) Å] (Hampden-Smith, Wark, Rheingold & Huffman, 1991). Both of these species also show a distorted edge-shared bioctahedral structure. Several other examples of tin alkoxide compounds have been structurally characterized, *e.g.*  $[\text{Sn}(\text{O}^i\text{Bu})_4]$  [Sn—O 1.946(2) and 1.949(2) Å] (Hampden-Smith, Wark, Rheingold & Huffman, 1991). Examples involving Sn<sub>2</sub>O<sub>2</sub> cores with bridging alkoxide ligands similar to (I) have also been reported, *e.g.*

$[\text{Sn}(\text{acac})(\text{O}^i\text{Pr})_3]_2$  (acac is acetylacetonate) (Chandler, Fallon, Koplick & West, 1987) and  $[\text{Sn}(\text{OC}_6\text{H}_3\text{Ph-C}_6\text{H}_4)\text{Cl}_2]_2$ , involving cyclometallated 2,6-diphenylphenoxide ligands (Smith, Visciglio, Fanwick & Rothwell, 1992).

## Experimental

Crystals of the title compound were obtained by recrystallization from CHCl<sub>3</sub>. The selected crystal was mounted on a glass fibre.

### Crystal data

$[\text{Sn}_2\text{Cl}_6(\text{C}_2\text{H}_5\text{O})_2(\text{C}_2\text{H}_6\text{O})_2]$   
 $M_r = 632.36$   
 Monoclinic  
 $P2_1/n$   
 $a = 9.653(3) \text{ \AA}$   
 $b = 7.472(3) \text{ \AA}$   
 $c = 14.627(2) \text{ \AA}$   
 $\beta = 107.08(2)^\circ$   
 $V = 1008.5(5) \text{ \AA}^3$   
 $Z = 2$   
 $D_x = 2.082 \text{ Mg m}^{-3}$   
 $D_m$  not measured

Mo  $K\alpha$  radiation  
 $\lambda = 0.71073 \text{ \AA}$   
 Cell parameters from 25 reflections  
 $\theta = 15.55\text{--}20.85^\circ$   
 $\mu = 3.276 \text{ mm}^{-1}$   
 $T = 150.0 \text{ K}$   
 Plate-like needle  
 $0.70 \times 0.20 \times 0.10 \text{ mm}$   
 Colourless

### Data collection

Rigaku AFC-7S diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction:  
 $\psi$  scan (North, Phillips & Mathews, 1968)  
 $T_{\min} = 0.749$ ,  $T_{\max} = 1.000$   
 2043 measured reflections  
 1925 independent reflections

1428 observed reflections  
 $[I > 3\sigma(I)]$   
 $R_{\text{int}} = 0.021$   
 $\theta_{\max} = 24.97^\circ$   
 $h = 0 \rightarrow 11$   
 $k = 0 \rightarrow 8$   
 $l = -17 \rightarrow 16$   
 3 standard reflections monitored every 150 reflections  
 intensity decay: none

### Refinement

Refinement on  $F$   
 $R = 0.0247$   
 $wR = 0.0291$   
 $S = 1.32$   
 1428 reflections  
 91 parameters  
 H-atom parameters not refined  
 $w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{\max} < 0.0001$   
 $\Delta\rho_{\max} = 0.66 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.78 \text{ e \AA}^{-3}$   
 Extinction correction: none  
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}$
Sn(1)	0.54473(3)	0.05148(4)	0.89943(2)	0.01863(9)
Cl(1)	0.4304(1)	−0.0177(2)	0.73860(8)	0.0281(3)
Cl(2)	0.4460(1)	0.3461(2)	0.89678(8)	0.0276(3)
Cl(3)	0.7680(1)	0.1417(2)	0.88063(8)	0.0313(3)

O(1)	0.6238 (4)	-0.2211 (5)	0.9172 (2)	0.0286 (9)
O(2)	0.6164 (3)	0.0576 (4)	1.0490 (2)	0.0194 (8)
C(1)	0.7100 (6)	-0.3169 (7)	0.8662 (3)	0.030 (1)
C(2)	0.8524 (6)	-0.3712 (9)	0.9331 (4)	0.043 (2)
C(3)	0.7432 (5)	0.1563 (7)	1.1058 (3)	0.024 (1)
C(4)	0.8710 (5)	0.0337 (8)	1.1409 (4)	0.032 (1)

Table 2. Selected geometric parameters (Å, °)

Sn(1)—Cl(1)	2.3441 (11)	Sn(1)—O(2')	2.084 (3)
Sn(1)—Cl(2)	2.3947 (12)	O(1)—C(1)	1.459 (6)
Sn(1)—Cl(3)	2.3504 (14)	O(2)—C(3)	1.461 (5)
Sn(1)—O(1)	2.164 (3)	C(1)—C(2)	1.491 (7)
Sn(1)—O(2)	2.092 (3)	C(3)—C(4)	1.501 (7)
Cl(1)—Sn(1)—Cl(2)	96.80 (4)	Cl(3)—Sn(1)—O(2')	164.21 (8)
Cl(1)—Sn(1)—Cl(3)	97.10 (4)	O(1)—Sn(1)—O(2)	84.16 (12)
Cl(1)—Sn(1)—O(1)	87.63 (9)	O(1)—Sn(1)—O(2')	82.11 (12)
Cl(1)—Sn(1)—O(2)	165.43 (9)	O(2)—Sn(1)—O(2')	71.50 (12)
Cl(1)—Sn(1)—O(2')	95.49 (8)	Sn(1)—O(1)—C(1)	129.4 (3)
Cl(2)—Sn(1)—Cl(3)	96.22 (5)	Sn(1)—O(2)—Sn(1')	108.50 (12)
Cl(2)—Sn(1)—O(1)	172.73 (9)	Sn(1)—O(2)—C(3)	124.8 (2)
Cl(2)—Sn(1)—O(2)	90.27 (9)	Sn(1')—O(2)—C(3)	125.5 (2)
Cl(2)—Sn(1)—O(2')	91.71 (9)	O(1)—C(1)—C(2)	110.6 (4)
Cl(3)—Sn(1)—O(1)	88.91 (10)	O(2)—C(3)—C(4)	110.7 (4)
Cl(3)—Sn(1)—O(2)	94.76 (9)		

Symmetry code: (i) 1 - x, -y, 2 - z.

Data collection used a Rigaku AFC-7S four-circle diffractometer equipped with an Oxford Cryosystems low-temperature attachment (Cosier & Glazer, 1986) operating at 150 K. Data collection and cell refinement were performed using *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988); *TEXSAN* (Molecular Structure Corporation, 1995) was used for data reduction. As there were no identifiable faces, the data were corrected for absorption using  $\psi$  scans. The structure was solved by direct methods (Sheldrick, 1990), which located the positions of all non-H atoms for one half molecule in the asymmetric unit. This is related to the other half of the neutral dinuclear species by a crystallographic inversion centre. Refinement was achieved with iterative cycles of least-squares refinement and difference Fourier synthesis (*TEXSAN*). All non-H atoms were refined anisotropically and H atoms [except for H(1)] were included in fixed calculated positions with C—H = 0.96 Å and  $U_{\text{iso}}(\text{H}) = U_{\text{eq}}(\text{parent C atom})$ . Atom H(1) was located in the difference map and its coordinates fixed.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1062). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Trimethyl[3-(triphenylstannyl)propyl]-ammonium Iodide

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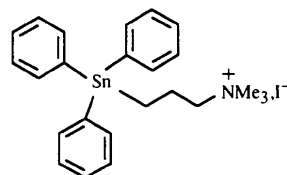
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### Abstract

The title compound, [Sn(C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>(C<sub>6</sub>H<sub>15</sub>N)]I, exists as an ionic species with discrete cations and anions. Both the Sn- and N-atom centres in the cation have tetrahedral geometries.

### Comment

The title compound, (I), is an ionic species with discrete cations and anions. All I···C distances are > 3.83 Å, I···N > 4.41 Å, and I···I > 5.09 Å. The geometries about both the Sn and N atoms in the title compound are essentially tetrahedral with C—Sn—C and C—N—C valence angles ranging from 106.2 (3) to 113.3 (3)°, and from 106.7 (5) to 111.6 (5)°, respectively. The



(I)

Sn—C bond lengths fall in a narrow band, between 2.135 (8) and 2.145 (7) Å, and are unexceptional. The aliphatic chain has an extended all-*anti* conformation with torsion angles Sn—C1—C2—C3 = 162.9 (5) and C1—C2—C3—N = 179.0 (6)°. The structure of only