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The chloro(diethylamino)dimethyltin dimer

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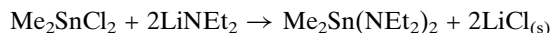
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The title compound, di- μ -diethylamido-*N,N*-bis[chlorodimethyltin(IV)], consists of discrete $[\text{Sn}_2\text{Cl}_2(\text{CH}_3)_4(\text{C}_4\text{H}_{10}\text{N})_2]$ dimer molecules, with Sn atoms linked by bridging diethylamido groups. The coordination geometry about the metal atom is distorted trigonal bipyramidal, with the two methyl C atoms and one N atom in the equatorial plane, and the Cl and second N atom in axial positions.

Comment

In the course of our studies on diethylaminodimethylstannane(IV), $[\text{Me}_2\text{Sn}(\text{NEt}_2)]$, as a novel molecular precursor for the chemical vapour deposition (CVD) of SnO_2 thin films (Barreca *et al.*, 1999), we have observed that defects of LiNEt_2 in the synthesis of

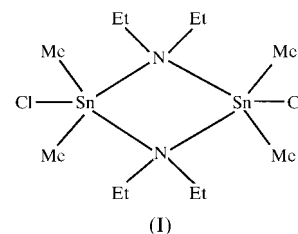


induce the formation of diethylaminodimethyltin chloride, $\text{Me}_2\text{Sn}(\text{NEt}_2)\text{Cl}$, in accordance with literature data (Davies & Kennedy, 1970, and references therein). In this work, the latter compound was reported to be unstable, so its complete characterization was not performed. Nevertheless, two different dimeric structures were proposed for it. In order to clarify the question, we have decided to verify the possibility of obtaining $\text{Me}_2\text{Sn}(\text{NEt}_2)\text{Cl}$ by the 1:1 reaction of Me_2SnCl_2 and LiNEt_2 :



and to undertake a structure determination of the obtained product. Operating under controlled atmosphere conditions, we obtained suitable crystals for X-ray diffraction of the title compound, (I). The structure consists of discrete dimers with an approximate binary symmetry. Two Sn atoms are bridged by two diethylamino N atoms, forming an irregular square. The coordination geometry about the Sn atom is distorted

trigonal bipyramidal, with the two methyl C atoms and one N atom in the equatorial plane, and the Cl and second N atom in axial positions. The Sn—N bond distances are different, the shorter ones are for the equatorial positions [Sn1—N1_{eq} 2.125 (8) and Sn2—N2_{eq} 2.136 (7) Å], while the longer ones are for the axial positions [Sn1—N2_{ax} 2.419 (7) and Sn2—N1_{ax} 2.444 (7) Å]. The latter distances are comparable to the values of 2.459 (15) and 2.453 (14) Å found for the axial Sn—N bond distances in the dimeric structure of $[\text{Me}_2\text{Cl}(\text{N}_2\text{C}_3\text{H}_3)\text{Sn}(\text{CH}_2)\text{Sn}(\text{N}_2\text{C}_3\text{H}_3)\text{ClMe}_2]$ with trigonal bipyramidal coordination geometry (Austin *et al.*, 1987). The Sn—C(methyl) bond distances present the usual values found in similar derivatives (Chivers *et al.*, 1989). The two axial Sn—Cl bond distances of 2.525 (2) and 2.543 (3) Å are comparable with the averaged value of 2.54 (2) Å reported for the two axial bonds in the $\text{SnMe}_2\text{Cl}_3^-$ anion (Einstein & Penfold, 1968).



Experimental

The reaction between Me_2SnCl_2 (3.5 g, 16 mmol) and LiNEt_2 (1.25 g, 16 mmol) in a 1:1 molar ratio was carried out in *n*-hexane (Davies & Kennedy, 1970, and references therein). The suspension was stirred overnight. The LiCl was separated by filtration and the *n*-hexane solution was concentrated under vacuum, giving a colourless crystalline product suitable for a single-crystal X-ray analysis. ¹H NMR signals [Bruker BHZ 200/52 spectrometer; positive δ values (p.p.m.) downfield from internal SiMe_4 , in C_6D_6]: 2.95 (quartet, 4H, NCH_2CH_3), 1.04 (triplet, 6H, NCH_2CH_3), 0.31 (singlet, 6H, SnCH_3).

Crystal data

$[\text{Sn}_2\text{Cl}_2(\text{CH}_3)_4(\text{C}_4\text{H}_{10}\text{N})_2]$

$M_r = 512.68$

Monoclinic, $C2/c$

$a = 16.015$ (3) Å

$b = 10.219$ (3) Å

$c = 24.275$ (5) Å

$\beta = 93.94$ (3)°

$V = 3963.4$ (16) Å³

$Z = 8$

$D_x = 1.718$ Mg m⁻³

Mo $K\alpha$ radiation

Cell parameters from 30

reflections

$\theta = 10\text{--}14^\circ$

$\mu = 2.780$ mm⁻¹

$T = 293$ (2) K

Prism, white

$0.50 \times 0.40 \times 0.24$ mm

Data collection

Philips PW1100 four-circle diffractometer

Profile fitted $\theta/2\theta$ scans (Lehman & Larsen, 1974)

Absorption correction: ψ scan (North *et al.*, 1968)

$T_{\min} = 0.276$, $T_{\max} = 0.513$

4818 measured reflections

3183 independent reflections

3144 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.031$

$\theta_{\text{max}} = 25.03^\circ$

$h = -19 \rightarrow 18$

$k = 0 \rightarrow 12$

$l = 0 \rightarrow 28$

3 standard reflections

frequency: 200 min

intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.056$
 $wR(F^2) = 0.140$
 $S = 1.209$
 3114 reflections
 171 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0514P)^2 + 78.9387P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.003$
 $\Delta\rho_{\max} = 1.03 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -1.27 \text{ e } \text{\AA}^{-3}$

Table 1

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

Sn1—C2	2.109 (11)	Sn2—C3	2.106 (10)
Sn1—C1	2.115 (11)	Sn2—C4	2.125 (10)
Sn1—N1	2.125 (8)	Sn2—N2	2.136 (7)
Sn1—N2	2.419 (7)	Sn2—N1	2.444 (7)
Sn1—Cl1	2.543 (3)	Sn2—Cl2	2.525 (2)
C2—Sn1—C1	125.9 (6)	C3—Sn2—N2	119.3 (4)
C2—Sn1—N1	115.1 (4)	C4—Sn2—N2	110.8 (4)
C1—Sn1—N1	119.0 (5)	N2—Sn2—N1	77.9 (3)
N1—Sn1—N2	78.6 (2)	N1—Sn2—Cl2	173.25 (18)
N2—Sn1—Cl1	173.06 (19)	Sn1—N1—Sn2	96.5 (3)
C3—Sn2—C4	129.7 (5)	Sn2—N2—Sn1	96.9 (2)

The H atoms of the C atoms were introduced at calculated positions in their described geometries and during refinement were

allowed to ride on the attached C atom with fixed isotropic displacement parameters ($1.2U_{\text{eq}}$ of the parent C atom).

Data collection: *FEBO* (Belletti, 1993); cell refinement: *FEBO*; data reduction: *FEBO*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); software used to prepare material for publication: *PARST94* (Nardelli, 1995).

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