

Structure of Bis(2-methoxycarbonylethyl-*C',O*)tin Dichloride

BY SEIK WENG NG

Institute of Advanced Studies, University of Malaya, 59100 Kuala Lumpur, Malaysia

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**Abstract.**  $[\text{SnCl}_2(\text{C}_4\text{H}_7\text{O}_2)_2]$ , monoclinic,  $M_r = 363.79$ ,  $P2_1/n$ ,  $a = 7.984(2)$ ,  $b = 16.019(2)$ ,  $c = 10.172(2)$  Å,  $\beta = 94.27(1)^\circ$ ,  $V = 1297.3(4)$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.863$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 23.84$  cm<sup>-1</sup>,  $F(000) = 712$ ,  $T = 298$  K,  $R = 0.023$  for 2443 reflections. The  $-\text{CH}_2\text{CH}_2\text{C}(\text{O})\text{OCH}_3$  group chelates through the carbonyl O atom to Sn, which has a distorted *trans*-octahedral geometry.

**Experimental.** Crystals of the title compound (Harrison, King & Healy, 1979) were grown from its solution in dimethylformamide, and a crystal of approximate dimensions  $0.20 \times 0.25 \times 0.25$  mm was chosen for data collection. Unit-cell dimensions were obtained from 25 reflections in the  $\theta$  range  $13\text{--}15^\circ$ . Intensity data were collected on an Enraf-Nonius CAD-4 diffractometer to  $2\theta_{\text{max}} = 55^\circ$  ( $h = 0\text{--}10$ ,  $k = 0\text{--}20$ ,  $l = -13\text{--}13$ ) using an  $\omega\text{--}2\theta$  scan. Of 3183 data measured, 2773 were unique with 2443 satisfying the  $I > 3\sigma(I)$  criterion. Three reflections ( $\bar{1}, 11, 0$ ,  $\bar{1}17$  and  $\bar{5}30$ ) monitored hourly showed a decrease of 3.3% for the 24.5 h of data collection. The data were corrected for decay (minimum/maximum corrections 1.0000/1.0168, average correction 1.0077).  $R_{\text{int}} = 0.021$ . The structure was solved by the heavy-atom method and refined isotropically. An empirical  $\theta$ -dependent absorption correction (Walker & Stuart, 1983) was applied (minimum/maximum corrections 0.8487/1.1122, average correction 0.9927). Non-H atoms were then refined anisotropically. H-atoms were located and were refined with  $B = 5$  Å<sup>2</sup>. Full-matrix least-squares refinement based on  $F$  converged at  $R = 0.023$ ,  $wR = 0.028$   $\{w = [\sigma(F)]^2 +$

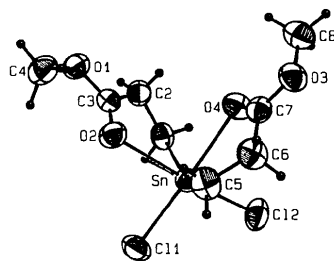


Fig. 1. Atomic labelling scheme for  $[\{\text{CH}_3\text{OC}(\text{O})\text{CH}_2\text{CH}_2\}_2\text{SnCl}_2]$ . Thermal ellipsoids are drawn at the 50% probability level.

Table 1. Positional parameters and equivalent isotropic thermal parameters (Å<sup>2</sup>)

$$B_{\text{eq}} = (4/3)[a^2B_{1,1} + b^2B_{2,2} + c^2B_{3,3} + ab(\cos\gamma)B_{1,2} + ac(\cos\beta)B_{1,3} + bc(\cos\alpha)B_{2,3}]$$

	x	y	z	$B_{\text{eq}}$
Sn	0.01028 (3)	0.17900 (1)	0.31811 (2)	3.335 (4)
Cl(1)	-0.2443 (1)	0.09896 (7)	0.3133 (1)	5.40 (2)
Cl(2)	-0.0657 (1)	0.28078 (6)	0.4778 (1)	5.48 (2)
O(1)	0.3427 (3)	-0.0042 (2)	0.1423 (2)	4.44 (5)
O(2)	0.1158 (3)	0.0742 (2)	0.1568 (2)	4.49 (5)
O(3)	0.3652 (3)	0.3730 (2)	0.2015 (3)	4.65 (5)
O(4)	0.2782 (3)	0.2605 (2)	0.3043 (2)	4.49 (5)
C(1)	0.1791 (4)	0.0981 (2)	0.4273 (3)	3.99 (7)
C(2)	0.3121 (4)	0.0653 (2)	0.3434 (4)	4.32 (7)
C(3)	0.2459 (4)	0.0453 (2)	0.2060 (3)	3.74 (6)
C(4)	0.2866 (5)	-0.0243 (3)	0.0068 (4)	5.17 (8)
C(5)	-0.0244 (5)	0.2528 (3)	0.1463 (4)	5.00 (8)
C(6)	0.0881 (5)	0.3278 (2)	0.1476 (4)	4.91 (8)
C(7)	0.2528 (4)	0.3158 (2)	0.2249 (3)	3.88 (6)
C(8)	0.5288 (5)	0.3639 (3)	0.2723 (4)	5.48 (9)

Table 2. Bond distances (Å) and angles ( $^\circ$ )

Sn—Cl(1)	2.4004 (9)	O(2)—C(3)	1.211 (4)
Sn—Cl(2)	2.4104 (10)	O(3)—C(7)	1.316 (4)
Sn—O(2)	2.536 (2)	O(3)—C(8)	1.451 (5)
Sn—O(4)	2.519 (2)	O(4)—C(7)	1.206 (4)
Sn—C(1)	2.122 (4)	C(1)—C(2)	1.506 (5)
Sn—C(5)	2.111 (4)	C(2)—C(3)	1.492 (5)
O(1)—C(3)	1.311 (4)	C(5)—C(6)	1.500 (6)
O(1)—C(4)	1.454 (5)	C(6)—C(7)	1.493 (5)
Cl(1)—Sn—Cl(2)	96.87 (4)	Sn—O(2)—C(3)	107.5 (2)
Cl(1)—Sn—O(2)	87.41 (6)	Sn—O(4)—C(7)	108.5 (2)
Cl(1)—Sn—O(4)	175.54 (6)	Sn—C(1)—C(2)	111.4 (2)
Cl(1)—Sn—C(1)	100.9 (1)	Sn—C(5)—C(6)	113.6 (3)
Cl(1)—Sn—C(5)	102.9 (1)	C(3)—O(1)—C(4)	116.8 (3)
Cl(2)—Sn—O(2)	175.18 (6)	C(7)—O(3)—C(8)	116.2 (3)
Cl(2)—Sn—O(4)	86.82 (7)	C(1)—C(2)—C(3)	112.9 (3)
Cl(2)—Sn—C(1)	104.0 (1)	O(1)—C(3)—O(2)	123.0 (3)
Cl(2)—Sn—C(5)	98.9 (1)	O(1)—C(3)—C(2)	114.3 (3)
O(2)—Sn—O(4)	89.02 (9)	O(2)—C(3)—C(2)	122.6 (3)
O(2)—Sn—C(1)	72.9 (1)	C(5)—C(6)—C(7)	113.9 (3)
O(2)—Sn—C(5)	82.2 (1)	O(3)—C(7)—O(4)	123.3 (3)
O(4)—Sn—C(1)	80.6 (1)	O(3)—C(7)—C(6)	113.6 (3)
O(4)—Sn—C(5)	73.9 (1)	O(4)—C(7)—C(6)	123.0 (3)
C(1)—Sn—C(5)	144.5 (2)		

$(0.02F)^2 + 1]^{-1}$  (Killeen & Lawrence, 1969) for the 178 variables;  $S = 0.505$ ;  $(\Delta/\sigma)_{\text{max}} \leq 0.05$  for all atoms;  $(\Delta\rho)_{\text{min/max}} = -0.20(4)/0.28(4)$  e Å<sup>-3</sup>. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.3.1 and 2.2B). Computations were performed with the *MolEN* package (Fair, 1990) on a DEC MicroVAX II minicomputer. The atomic coordinates are

listed in Table 1\* and selected bond distances and angles in Table 2. The molecule is depicted in Fig. 1.

**Related literature.** The refinement is an improvement of the reported refinement for which  $R = 0.048$  for 2388  $I \geq 3\sigma(I)$  data (Harrison, King & Healy, 1979).

\* Lists of structure factors, anisotropic thermal parameters and calculated H-atom positional parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55636 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: OH1001]

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## Structure of Bis(triphenyltin) Succinate–[Bis(triphenyltin) Succinate Bis(*N,N*-dimethylformamide)] (1/1)

BY SEIK WENG NG

*Institute of Advanced Studies, University of Malaya, 59100 Kuala Lumpur, Malaysia*

AND V. G. KUMAR DAS

*Department of Chemistry, University of Malaya, 59100 Kuala Lumpur, Malaysia*

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**Abstract.** [Sn<sub>4</sub>(C<sub>4</sub>H<sub>4</sub>O<sub>4</sub>)<sub>2</sub>(C<sub>3</sub>H<sub>7</sub>NO)<sub>2</sub>(C<sub>6</sub>H<sub>5</sub>)<sub>12</sub>],  $M_r = 1778.38$ , triclinic,  $P\bar{1}$ ,  $a = 12.1759$  (7),  $b = 12.8661$  (9),  $c = 14.3293$  (9) Å,  $\alpha = 79.041$  (5),  $\beta = 71.665$  (5),  $\gamma = 69.467$  (5)°,  $V = 1987.4$  (2) Å<sup>3</sup>,  $Z = 1$ ,  $D_x = 1.486$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 13.03$  cm<sup>-1</sup>,  $F(000) = 892$ ,  $T = 298$  K,  $R = 0.026$  for 5135 reflections. The carbonyl O atoms of the centrosymmetric bis(triphenyltin) succinate.2DMF [Sn—O = 2.145 (3), Sn←O<sub>DMF</sub> = 2.404 (3) Å] molecule are linked datively to the Sn atoms [Sn—O = 2.444 (2) Å] of two centrosymmetric bis(triphenyltin) succinate [Sn—O = 2.112 (3) Å] molecules to form a three-dimensional network. The coordination environment at each of the Sn atoms is a *trans*-trigonal bipyramid.

**Experimental.** The compound, m.p. 430 K, was obtained as an adventitious product on reacting (C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>SnOH with (CH<sub>3</sub>)<sub>2</sub>NCS<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>H in hot ethanol. As the condensation performed in an earlier attempt yielded the stannyl ester (CH<sub>3</sub>)<sub>2</sub>NCS<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>Sn(C<sub>6</sub>H<sub>5</sub>)<sub>3</sub> (Ng & Kumar Das, 1991), the anomalous occurrence of the —O<sub>2</sub>CCH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>— and (CH<sub>3</sub>)<sub>2</sub>NCHO components in the present compound

must be attributed to a transformation of either the parent acid or its stannyl salt, induced by an undefined impurity in the solvent used in the experiment. A parallel can be drawn to the well documented susceptibility of a number of ethylenebis(dithiocarbamate) salts to oxidation and hydrolysis. Aeration of a suspension of manganese ethylene-1,2-bis(dithiocarbamate) at pH 6, for example, results in the formation of the break-down products, ethylenethiourea, 5,6-dihydro-3*H*-imidazo-1,2,4-dithiazole-3-thione, ethylenediamine, carbon disulfide and elemental sulfur (Vonk, 1975).

A crystal measuring approximately 0.11 × 0.18 × 0.22 mm was used in the study. Cell dimensions were calculated from the 25 most intense reflections in the 13 ≤  $\theta$  ≤ 15° shell. Data were collected on an Enraf-Nonius CAD-4 diffractometer using  $\omega$ -2 $\theta$  scans to  $2\theta_{\text{max}} = 50^\circ$  ( $h$  0 to 14,  $k$  -15 to 15,  $l$  -17 to 17). Of 7350 data measured, 6622 were unique with 5135 obeying  $I \geq 3\sigma(I)$ . Crystal decay was monitored by three reflections (247, 273 and 455). A linear decay correction was applied owing to a decrease in their intensities of 6.5% during the 61 h of data collection (minimum/maximum corrections 1.00002/1.03396;