

Mostafa M. Amini,^a
Shabnam Hossein Abadi,^a
Mahdi Mirzaee,^a Shi-Yao Yang^b
and Seik Weng Ng^{c*}

^aDepartment of Chemistry, Shahid Beheshti University, Tehran, Iran, ^bDepartment of Chemistry, Xiamen University, Xiamen 361005, People's Republic of China, and ^cDepartment of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

Key indicators

Single-crystal X-ray study
T = 298 K
Mean $\sigma(\text{C}-\text{C}) = 0.013 \text{ \AA}$
R factor = 0.059
wR factor = 0.129
Data-to-parameter ratio = 22.6

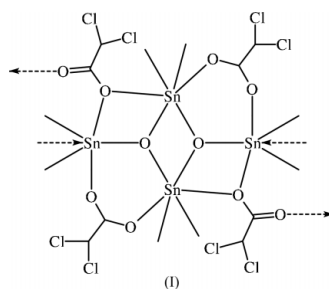
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Bis[1,1,3,3-tetramethyl-1,3-bis(dichloroacetato)distannoxane]

In centrosymmetric octamethyl- $1\kappa^2\text{C}, 2\kappa^2\text{C}, 3\kappa^2\text{C}, 4\kappa^2\text{C}$ -tetrakis- μ -dichloroacetato- $1:2\kappa^2\text{O}, \text{O}'; 2:3\kappa^2\text{O}, \text{O}'; 3:4\kappa^2\text{O}, \text{O}'; 1:4\kappa^2\text{O}$ -bis- μ_3 -oxo- $1:2:3:\kappa^3\text{O}; 1:3:4\kappa^3\text{O}$ -tetratin(IV), $[[(\text{CH}_3)_2\text{SnO}_2\text{CCHCl}_2]_2\text{O}]_2$, two Sn atoms are six-coordinate in a C_2SnO_4 skew-trapezoidal bipyramidal geometry [C—Sn—C 145.7 (4)°]. The other two Sn atoms are five-coordinate [C—Sn—C 154.3 (4)°], but the geometry is better regarded as a *trans*- C_2SnO_4 octahedron owing to a long intermolecular Sn—O bond [Sn—O 2.846 (6) Å], which links the molecules into a linear chain.

Comment

An earlier study reported the synthesis of dimethylphenyltin trifluoroacetate by the reaction of dimethylphenyltin iodide with silver trifluoroacetate (Amini *et al.*, 2002a). The analogous reaction with silver benzoate led to tin–phenyl cleavage, affording bis(1,1,3,3-tetramethyl-1,3-dibenzoatodistannoxane) (Amini *et al.*, 2002b); a similar cleavage is invoked to account for the formation of the title distannoxane, (I) (Fig. 1), from the reaction with silver dichlorobenzoate.



The structures of tetraorganodicarboxylatodistannoxanes are classified into four types (Ng *et al.*, 1991), with the type reported for bis(tetramethyldibenzoatodistannoxane) being the most common (Haiduc & Edelmann, 1999). In this centrosymmetric type, one carboxylato anion bridges the Sn—O—Sn unit through its $-\text{CO}_2$ constituent. The other anion uses only one O atom, the single-bond carboxyl O atom, to bridge the second Sn—O—Sn unit. In contrast, the double-bonded carbonyl O atom in the title distannoxane is rotated about the carbon–carbon single bond in order to interact with the five-coordinate Sn2 atom [C—Sn—C 154.3 (3)°] of an adjacent molecule, so that the geometry is instead better regarded as a *trans*- C_2SnO_4 octahedron arising from this somewhat long [Sn2—O4ⁱⁱ 2.846 (6) Å; symmetry code (ii) 2 - x, 1 - y, 2 - z] interaction. This interaction leads to the formation of a chain structure. The other $(\text{CH}_3)_2\text{Sn}$ is more bent. The O5, O1, O1ⁱ and O2ⁱ [symmetry code (i) 1 - x,

Received 29 August 2003
Accepted 3 September 2003
Online 11 September 2003

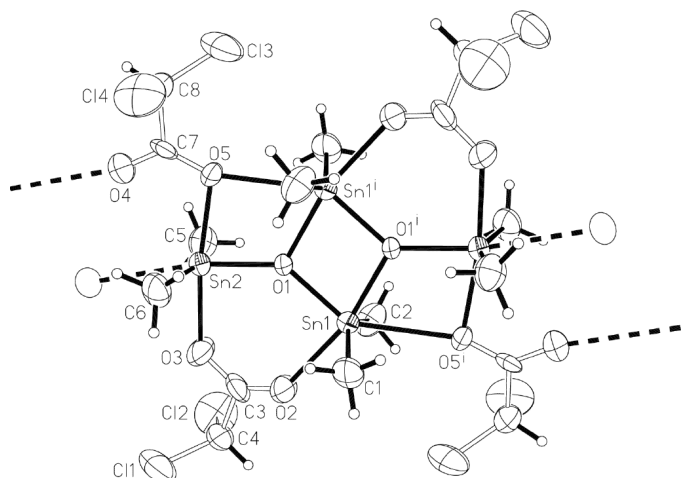


Figure 1
ORTEP (Johnson, 1976) plot of bis[1,1,3,3-tetramethyl-1,3-bis(dichloroacetato)distannoxane]; displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii. [Symmetry code: (i) $1 - x, 1 - y, 1 - z$.]

$1 - y, 1 - z$] atoms constitute an approximate trapezoid, and the methyl groups are skewed over the long ($O5-O2^i$) edge [$C-Sn-C$ 145.7 (4) $^\circ$].

Experimental

Dimethylphenyltin iodide was synthesized using iodine to cleave the tin–aryl bond of dimethyldiphenyltin (Davison & Rakita, 1970). The iodide (0.37 g, 1 mmol) and silver dichloroacetate (0.23 g, 1 mmol), when reacted in ethanol, gave a precipitate of silver iodide, which was removed by filtration. Evaporation of the solvent gave an oily material, which was purified by crystallization from chloroform to furnish colorless crystals, m.p. 548–550 K.

Crystal data

$[Sn_4(C_2HO_2Cl_2)_4(CH_3)_8O_2]$	$Z = 1$
$M_r = 1138.74$	$D_x = 2.164 \text{ Mg m}^{-3}$
Triclinic, $P1$	Mo $K\alpha$ radiation
$a = 8.9026$ (5) \AA	Cell parameters from 3478 reflections
$b = 9.6131$ (6) \AA	$\theta = 2.5\text{--}28.3^\circ$
$c = 11.2145$ (7) \AA	$\mu = 3.48 \text{ mm}^{-1}$
$\alpha = 91.674$ (1) $^\circ$	$T = 298$ (2) K
$\beta = 113.129$ (1) $^\circ$	Parallelepiped, colorless
$\gamma = 96.651$ (1) $^\circ$	$0.29 \times 0.18 \times 0.12 \text{ mm}$
$V = 873.73$ (9) \AA^3	

Data collection

Bruker SMART APEX area-detector diffractometer	3977 independent reflections
φ and ω scans	3218 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.037$
$T_{\text{min}} = 0.540, T_{\text{max}} = 0.659$	$\theta_{\text{max}} = 28.3^\circ$
7696 measured reflections	$h = -11 \rightarrow 11$
	$k = -12 \rightarrow 12$
	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.059$	$w = 1/[\sigma^2(F_o^2) + (0.0466P)^2]$
$wR(F^2) = 0.129$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{\text{max}} = 0.001$
3977 reflections	$\Delta\rho_{\text{max}} = 0.98 \text{ e \AA}^{-3}$
176 parameters	$\Delta\rho_{\text{min}} = -1.07 \text{ e \AA}^{-3}$

Table 1

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

Sn1—C1	2.088 (8)	Sn2—C5	2.062 (8)
Sn1—C2	2.076 (8)	Sn2—C6	2.099 (8)
Sn1—O1	2.041 (4)	Sn2—O1	2.034 (4)
Sn1—O1 ⁱ	2.118 (5)	Sn2—O3	2.207 (6)
Sn1—O2	2.330 (6)	Sn2—O4 ⁱⁱ	2.846 (6)
Sn1—O5 ⁱ	2.696 (5)	Sn2—O5	2.234 (5)
C1—Sn1—C2	145.7 (4)	C5—Sn2—O3	87.9 (3)
C1—Sn1—O1	106.6 (3)	C5—Sn2—O4 ⁱⁱ	77.2 (3)
C1—Sn1—O1 ⁱ	98.7 (3)	C5—Sn2—O5	94.9 (3)
C1—Sn1—O2	84.0 (3)	C6—Sn2—O1	102.5 (3)
C1—Sn1—O5 ⁱ	80.6 (3)	C6—Sn2—O3	89.0 (3)
C2—Sn1—O1	105.4 (3)	C6—Sn2—O4 ⁱⁱ	77.2 (3)
C2—Sn1—O1 ⁱ	100.0 (3)	C6—Sn2—O5	91.7 (3)
C2—Sn1—O2	84.4 (3)	O1—Sn2—O3	93.7 (2)
C2—Sn1—O5 ⁱ	80.7 (3)	O1—Sn2—O4 ⁱⁱ	170.2 (2)
O1—Sn1—O1 ⁱ	77.2 (2)	O1—Sn2—O5	78.2 (2)
O1—Sn1—O2	89.4 (2)	O3—Sn2—O4 ⁱⁱ	76.5 (2)
O1—Sn1—O5 ⁱ	144.0 (2)	O3—Sn2—O5	171.8 (2)
O1 ⁱ —Sn1—O2	166.6 (2)	O4 ⁱⁱ —Sn2—O5	111.6 (2)
O1 ⁱ —Sn1—O5 ⁱ	66.9 (2)	Sn1—O1—Sn1 ⁱ	102.8 (2)
O2—Sn1—O5 ⁱ	126.6 (2)	Sn1—O1—Sn2	136.1 (2)
C5—Sn2—C6	154.3 (4)	Sn1 ⁱ —O1—Sn2	121.0 (2)
C5—Sn2—O1	103.2 (3)	Sn1 ⁱ —O5—Sn2	93.8 (2)

Symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $2 - x, 1 - y, 2 - z$.

The H atoms were placed in calculated positions and were allowed to ride on their parent atoms [$C-H$ 0.96 \AA and $U(H) = 1.3U_{\text{eq}}(C)$ for the methyl H atoms; $C-H$ 0.98 \AA and $U(H) = 1.5U_{\text{eq}}(C)$ for the methine H atom]. The torsion angles were refined for the methyl groups. The deepest hole is about 1 \AA from atom H8.

Data collection: SMART (Bruker, 2001); cell refinement: SMART; data reduction: SAINT (Bruker, 2001); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEPII (Johnson, 1976); software used to prepare material for publication: SHELXL97.

The authors thank the Vice President's Office of Research Affairs of Shahid Beheshti University, Xiamen University and the University of Malaya for supporting this work.

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