# metal-organic papers

# Bis[1,1,3,3-tetramethyl-1,3-bis(dichloro-acetato)distannoxane]

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In centrosymmetric octamethyl-$1\kappa^2C,2\kappa^2C,3\kappa^2C,4\kappa^2C$-tetrakis-$\mu$-dichloroacetato-$1:2\kappa^2O,O';2:3\kappa^2O,O';3:4\kappa^2O,O'$;-$1:4\text{-}\kappa^2O$-bis-$\mu_3$-oxo-$1:2:3:\kappa^3O;1:3:4\kappa^3O$-tetratin(IV), $[[(CH_3)_2\text{-}SnO_2CCHCl_2]_2O]_2$, 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.*, 2002*a*). The analogous reaction with silver benzoate led to tin–phenyl cleavage, affording bis(1,1,3,3-tetramethyl-1,3-dibenzoatodistannoxane) (Amini *et al.*, 2002*b*); a similar cleavage is invoked to account for the formation of the title distannoxane, (I) (Fig. 1), from the reaction with silver dichlorobenzoate.



(I)
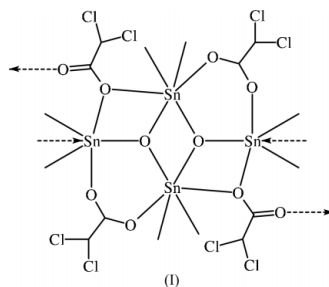
The structures of tetraorganodicarboxylatodistannoxanes are classified into four types (Ng *et al.*, 1991), with the type reported for bis(tetramethyldibenzoatodistannoxane) being the most common (Haiduc & Edelmann, 1999). In this centrosymmetric type, one carboxylato anion bridges the Sn—O—Sn unit through its —$CO_2$ 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—O4$^{ii}$ 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 $(CH_3)_2$Sn is more bent. The O5, O1, O1$^i$ and O2$^i$ [symmetry code (i) $1 - x$,
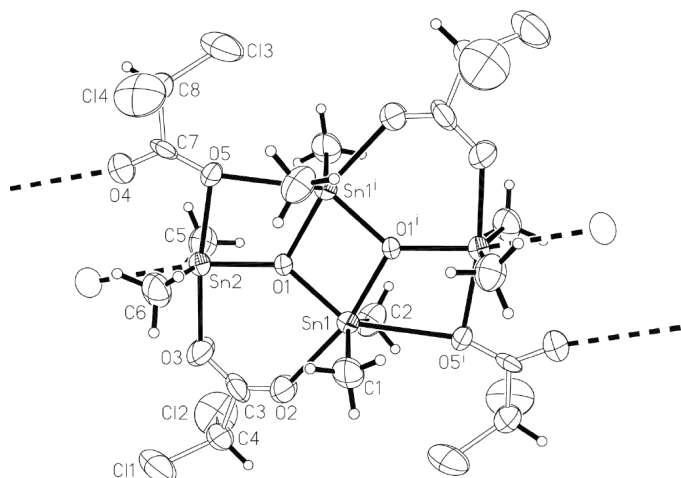
**Figure 1**
*ORTEP* (Johnson, 1976) plot of bis[1,1,3,3-tetramethyl-1,3-bis(dichloroacetato)distannoxane]; displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii. [Symmetry code: (i) $1 - x$, $1 - y$, $1 - z$.]

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

## 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$_2$HO$_2$Cl$_2$)$_4$(CH$_3$)$_8$O$_2$] | $Z = 1$ |
| $M_r = 1138.74$ | $D_x = 2.164$ Mg m$^{-3}$ |
| Triclinic, $P\bar{1}$ | Mo $K\alpha$ radiation |
| $a = 8.9026$ (5) Å | Cell parameters from 3478 |
| $b = 9.6131$ (6) Å | reflections |
| $c = 11.2145$ (7) Å | $\theta = 2.5$–28.3° |
| $\alpha = 91.674$ (1)° | $\mu = 3.48$ mm$^{-1}$ |
| $\beta = 113.129$ (1)° | $T = 298$ (2) K |
| $\gamma = 96.651$ (1)° | Parallelepiped, colorless |
| $V = 873.73$ (9) Å$^3$ | $0.29 \times 0.18 \times 0.12$ mm |

*Data collection*

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

*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 $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.08$ | $(\Delta/\sigma)_{max} = 0.001$ |
| 3977 reflections | $\Delta\rho_{max} = 0.98$ e Å$^{-3}$ |
| 176 parameters | $\Delta\rho_{min} = -1.07$ e Å$^{-3}$ |

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| 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 Å and $U$(H) = 1.3$U_{eq}$(C) for the methyl H atoms; C—H 0.98 Å and $U$(H) = 1.5$U_{eq}$(C) for the methine H atom]. The torsion angles were refined for the methyl groups. The deepest hole is about 1 Å from atom H8.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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