

O(2)	0.181 (1)	0.4004 (6)	0.036 (1)	0.119 (5)
O(3)	0.2558 (8)	0.4644 (6)	-0.161 (1)	0.118 (4)
O(4)	0.200 (1)	0.3539 (5)	-0.193 (1)	0.228 (7)
O(5)	0.5683 (7)	0.6076 (5)	-0.175 (1)	0.083 (3)
O(6)	0.4791 (9)	0.5932 (5)	0.054 (1)	0.092 (4)
O(7)	0.3822 (7)	0.6441 (5)	-0.142 (1)	0.105 (4)
O(8)	0.5261 (7)	0.7015 (4)	-0.027 (2)	0.121 (5)
N(1)	0.2452 (6)	0.6623 (4)	0.4047 (10)	0.037 (3)
N(2)	0.2492 (7)	0.5430 (4)	0.2084 (10)	0.045 (3)
N(3)	0.1016 (6)	0.5655 (4)	0.5968 (10)	0.044 (3)
N(4)	0.3802 (7)	0.5435 (4)	0.5480 (10)	0.040 (3)
N(5)	0.1840 (6)	0.4515 (4)	0.437 (2)	0.053 (3)
C(1)	0.1614 (9)	0.6894 (6)	0.289 (1)	0.039 (4)
C(2)	0.178 (1)	0.6582 (6)	0.134 (1)	0.060 (4)
C(3)	0.162 (1)	0.5804 (6)	0.121 (1)	0.058 (4)
C(4)	0.223 (1)	0.7016 (6)	0.550 (1)	0.047 (4)
C(5)	0.108 (1)	0.6908 (7)	0.628 (1)	0.051 (4)
C(6)	0.1043 (10)	0.6233 (6)	0.707 (1)	0.052 (4)
C(7)	0.3638 (9)	0.6777 (5)	0.354 (1)	0.048 (4)
C(8)	0.4582 (8)	0.6534 (5)	0.458 (2)	0.048 (4)
C(9)	0.4757 (8)	0.5765 (5)	0.470 (1)	0.043 (4)
C(10)	0.1836 (7)	0.3938 (5)	0.435 (2)	0.047 (3)
C(11)	0.1814 (9)	0.3187 (5)	0.435 (2)	0.087 (4)

Table 2. Selected geometric parameters (\AA , $^\circ$)

Cu(1)—N(1)	2.061 (7)	Cu(1)—N(4)	2.121 (8)
Cu(1)—N(2)	2.035 (8)	Cu(1)—N(5)	2.099 (7)
Cu(1)—N(3)	2.046 (8)		
N(1)—Cu(1)—N(2)	89.6 (3)	N(2)—Cu(1)—N(4)	108.7 (3)
N(1)—Cu(1)—N(3)	95.6 (3)	N(2)—Cu(1)—N(5)	85.1 (5)
N(1)—Cu(1)—N(4)	94.5 (3)	N(3)—Cu(1)—N(4)	106.9 (3)
N(1)—Cu(1)—N(5)	171.3 (4)	N(3)—Cu(1)—N(5)	84.7 (4)
N(2)—Cu(1)—N(3)	143.5 (3)	N(4)—Cu(1)—N(5)	93.8 (3)

Table 3. Hydrogen-bonding geometry (\AA , $^\circ$)

D—H...A	D—H	H...A	D...A	D—H...A
N(2)—H(1)...O(6)	0.91	2.30	3.19 (1)	165
N(2)—H(2)...O(2)	0.91	2.42	3.25 (1)	153
N(3)—H(3)...O(1 ⁱ)	0.91	2.33	3.16 (1)	152
N(3)—H(4)...O(1 ⁱⁱ)	0.91	2.31	3.20 (1)	165
N(4)—H(5)...O(7 ⁱⁱⁱ)	0.91	2.49	3.36 (1)	162
N(4)—H(6)...O(6 ⁱⁱⁱ)	0.91	2.30	3.12 (1)	150
C(3)—H(12)...O(1 ⁱ)	0.98	2.45	3.40 (2)	161
C(4)—H(13)...O(7 ⁱⁱⁱ)	0.98	2.54	3.49 (2)	163
C(7)—H(19)...O(6)	0.98	2.48	3.40 (1)	157
C(11)—H(27)...O(8 ^{iv})	0.98	2.53	3.36 (1)	142

Symmetry codes: (i) $-x, 1-y, \frac{1}{2}+z$; (ii) $x, y, 1+z$; (iii) $1-x, 1-y, \frac{1}{2}+z$; (iv) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}+z$.

The polarity (for the crystal used for data collection) was determined by a parallel refinement of the mirror image. The residuals were slightly higher for the opposite polarity: $R = 0.0396$ and $wR = 0.0340$. One of the perchlorate ions shows marked anisotropy consistent with some possible unresolved disorder; this is not uncommon in structures with such small, almost spherical, anions.

Data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1992). Cell refinement: *MSCIAFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1210). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). **C52**, 1959–1961

Bis(2-hydroxybenzoato-*O,O'*)dimethyltin

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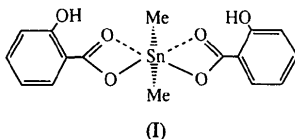
Abstract

The Sn atom in $[\text{Sn}(\text{C}_7\text{H}_5\text{O}_3)_2(\text{CH}_3)_2]$ is in a skew trapezoidal bipyramidal geometry with two types of Sn—O bond distances of approximately 2.1 and 2.5 \AA . The methyl substituents lie over the weaker Sn...O interactions. The weakly bonded O atoms are involved in intramolecular hydrogen-bonding contacts with the hydroxyl groups. In addition, there is a close intermolecular contact between one of the weakly associated O atoms and a symmetry-related hydroxyl group.

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Comment

Interest in salicylate compounds of organotin systems arises, in part, as a result of their antitumour activity (Boualam, Willem, Biesemans & Gielen, 1991). In the title compound, (I), the Sn atom is six-coordinate, existing in a skew trapezoidal bipyramidal geometry. The Sn—O(1,4) bond distances are significantly shorter than the Sn—O(2,5) distances and the methyl groups are orientated so as to lie over the weaker Sn···O interactions; the methyl groups define an angle at Sn of 138.2(2)°. The Sn atom lies 0.0101(3) Å above the O₄ plane in the direction of the C(16) atom. The structural motif found here resembles that found for a number of compounds of general formula R₂Sn(O₂CR')₂ (Tiekink, 1994).



Close intramolecular O···H—O interactions involving the weakly coordinating carboxylate O atoms are noted (the hydroxyl H atoms were located from a difference map); O(2)···O(3) 2.631(4), O(2)···H(3O) 1.75 Å and O(2)···H(3O)—O(3) 163°, and O(5)···O(6) 2.612(4), O(5)···H(6O) 1.72 Å and O(5)···H(6O)—O(6) 176°. In the lattice, there is a contact of 3.395(3) Å between the Sn and O(3ⁱ) atoms, however, this separation is too long to be considered a significant interaction [symmetry code: (i) $-x, -y, -z$]. A hydrogen-bonding contact between the O(2) and O(3ⁱ) atoms is noted; O(2)···O(3ⁱ) 2.822(4), O(2)···H(3Oⁱ) 2.58 Å and O(2)···H—O(3ⁱ) 96°.

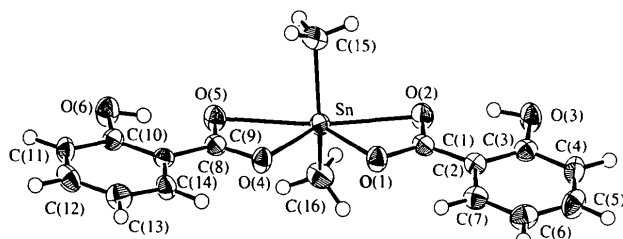


Fig. 1. The structure of the title compound drawn with 50% probability displacement ellipsoids (ORTEP; Johnson, 1976).

Experimental

Crystals of (I) were prepared from the metathetical reaction of sodium salicylate and Me₂SnCl₂ (2:1) in methanol solution under reflux conditions for 2 h. The solid obtained after removal of the solvent was washed with petroleum ether (333–353 K). The compound was extracted with CHCl₃ which on concentration at room temperature gave colourless crystals (84% yield, m.p. 483–484 K).

Crystal data

[Sn(C₇H₅O₃)₂(CH₃)₂]
M_r = 422.99
 Monoclinic
*P*2₁/*n*
a = 7.471(4) Å
b = 21.635(4) Å
c = 10.346(3) Å
 β = 104.32(3)°
V = 1620.2(9) Å³
Z = 4
D_x = 1.734 Mg m⁻³

Mo *K*α radiation
 λ = 0.7107 Å

Cell parameters from 25 reflections

θ = 42.0–47.8°

μ = 1.604 mm⁻¹

T = 193 K

Plate

0.34 × 0.23 × 0.04 mm

Colourless

Data collection

Rigaku AFC-6R diffractometer

$\omega/2\theta$ scans

Absorption correction:
 analytical (De Meulenaer & Tompa, 1965)

T_{min} = 0.660, *T_{max}* = 0.845

4157 measured reflections

3877 independent reflections (including systematic absences)

3768 observed reflections

All unique data used

R_{int} = 0.0317

θ_{max} = 27.50°

h = 0 → 9

k = 0 → 28

l = -13 → 13

3 standard reflections

monitored every 400

reflections

intensity decay: 3.03%

Refinement

Refinement on *F*²

R(F) = 0.064

*wR(F*²) = 0.085

S = 1.267

3768 reflections

208 parameters

H-atom parameters not refined

Weighting scheme based on measured e.s.d.'s;

w = 1/ $\sigma^2(F_o)$

(Δ/σ)_{max} = 0.0006

$\Delta\rho_{max}$ = 1.18 e Å⁻³

[near C(13)]

$\Delta\rho_{min}$ = -1.21 e Å⁻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 (Å²)

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

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
Sn	0.17576 (4)	0.05856 (1)	0.34951 (3)	0.02693 (7)
O(1)	0.1635 (4)	-0.0384 (1)	0.3691 (3)	0.0341 (8)
O(2)	0.0633 (4)	-0.0138 (1)	0.1580 (3)	0.0355 (8)
O(3)	-0.0610 (4)	-0.0994 (1)	-0.0225 (3)	0.0401 (9)
O(4)	0.2803 (4)	0.0548 (1)	0.5585 (3)	0.0321 (7)
O(5)	0.2769 (4)	0.1530 (1)	0.5019 (3)	0.0338 (8)
O(6)	0.4210 (5)	0.2361 (1)	0.6806 (3)	0.0426 (9)
C(1)	0.0949 (6)	-0.0552 (2)	0.2460 (4)	0.031 (1)
C(2)	0.0526 (5)	-0.1202 (2)	0.2145 (4)	0.028 (1)
C(3)	-0.0263 (6)	-0.1387 (2)	0.0825 (4)	0.031 (1)
C(4)	-0.0713 (6)	-0.2003 (2)	0.0552 (5)	0.037 (1)
C(5)	-0.0407 (7)	-0.2429 (2)	0.1560 (5)	0.043 (1)
C(6)	0.0398 (7)	-0.2257 (2)	0.2874 (5)	0.040 (1)
C(7)	0.0845 (6)	-0.1649 (2)	0.3149 (4)	0.034 (1)
C(8)	0.3167 (5)	0.1120 (2)	0.5900 (4)	0.029 (1)
C(9)	0.4107 (5)	0.1272 (2)	0.7300 (4)	0.028 (1)
C(10)	0.4602 (5)	0.1885 (2)	0.7663 (4)	0.029 (1)
C(11)	0.5577 (6)	0.2013 (2)	0.8976 (4)	0.034 (1)

C(12)	0.6018 (6)	0.1549 (2)	0.9887 (4)	0.038 (1)
C(13)	0.5530 (6)	0.0940 (2)	0.9544 (4)	0.038 (1)
C(14)	0.4555 (6)	0.0807 (2)	0.8255 (4)	0.032 (1)
C(15)	0.4117 (7)	0.0772 (2)	0.2815 (5)	0.043 (1)
C(16)	-0.0952 (6)	0.0913 (2)	0.3179 (4)	0.040 (1)

Acta Cryst. (1996). **C52**, 1961–1963

N,N'-Bis[*trans*-2-phenyl-5-(triphenylstannylmethoxymethyl)-1,3-dioxan-5-yl]ethanediamide

Table 2. Selected geometric parameters (Å, °)

Sn—O(1)	2.112 (3)	O(2)—C(1)	1.256 (5)
Sn—O(2)	2.503 (3)	O(3)—C(3)	1.353 (5)
Sn—O(4)	2.111 (3)	O(4)—C(8)	1.292 (5)
Sn—O(5)	2.577 (3)	O(5)—C(8)	1.254 (5)
Sn—C(15)	2.092 (5)	O(6)—C(10)	1.343 (5)
Sn—C(16)	2.092 (4)	C(1)—C(2)	1.460 (6)
O(1)—C(1)	1.301 (5)	C(8)—C(9)	1.482 (6)
O(1)—Sn—O(2)	56.0 (1)	Sn—O(5)—C(8)	82.2 (2)
O(1)—Sn—O(4)	82.9 (1)	O(1)—C(1)—O(2)	118.0 (4)
O(1)—Sn—O(5)	137.9 (1)	O(1)—C(1)—C(2)	119.8 (4)
O(1)—Sn—C(15)	106.5 (2)	O(2)—C(1)—C(2)	122.2 (4)
O(1)—Sn—C(16)	106.7 (2)	C(1)—C(2)—C(3)	120.4 (4)
O(2)—Sn—O(4)	138.9 (1)	C(1)—C(2)—C(7)	121.0 (4)
O(2)—Sn—O(5)	166.1 (1)	C(3)—C(2)—C(7)	118.6 (4)
O(2)—Sn—C(15)	90.0 (1)	O(3)—C(3)—C(2)	123.5 (4)
O(2)—Sn—C(16)	88.0 (2)	O(3)—C(3)—C(4)	116.8 (4)
O(4)—Sn—O(5)	55.0 (1)	O(4)—C(8)—O(5)	119.8 (4)
O(4)—Sn—C(15)	102.9 (2)	O(4)—C(8)—C(9)	118.6 (4)
O(4)—Sn—C(16)	105.7 (1)	O(5)—C(8)—C(9)	121.6 (4)
O(5)—Sn—C(15)	84.8 (1)	C(8)—C(9)—C(10)	120.1 (4)
O(5)—Sn—C(16)	87.3 (2)	C(8)—C(9)—C(14)	120.4 (4)
C(15)—Sn—C(16)	138.2 (2)	C(10)—C(9)—C(14)	119.5 (4)
Sn—O(1)—C(1)	101.5 (3)	O(6)—C(10)—C(9)	123.3 (4)
Sn—O(2)—C(1)	84.6 (2)	O(6)—C(10)—C(11)	117.7 (4)
Sn—O(4)—C(8)	102.9 (2)		

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Abstract

The title compound, μ -{*N,N'*-bis[*trans*-5-(methoxymethyl)-2-phenyl-1,3-dioxan-5-yl]ethanediamide(2-)}-bis(triphenyltin), [Sn₂(C₆H₅)₆(C₂₆H₃₀N₂O₈)], was isolated from the products of the (triphenylstannyl)-methylation of a 1:1 mixture of stereoisomers of *N,N'*-bis(5-hydroxymethyl-2-phenyl-1,3-dioxan-5-yl)ethanediamide. The molecule lies about an inversion centre and the unique Sn atom has distorted tetrahedral geometry, with Sn—C_{alkyl} and Sn—C_{aryl} distances of 2.154 (2) and 2.138 (2)–2.147 (3) Å, respectively, and C—Sn—C angles in the range 105.5 (1)–113.2 (1)°.

Comment

The crystal structure of a 1:1 mixture of stereoisomers of *N,N'*-bis(5-hydroxymethyl-2-phenyl-1,3-dioxan-5-yl)ethanediamide [(*Ia*) and (*Ib*)] has been reported recently (Ross, Wardell, Low & Ferguson, 1996). Reaction of Ph₃SnCH₂I with this 1:1 mixture in the presence of NaH in dimethylformamide solution was found to give a product mixture which exhibited four ¹¹⁹Sn NMR chemical shift values in the region -145.2 to -139.8 p.p.m. (*i.e.* the region expected for compounds of the type Ph₃SnCH₂OR) (Cox, Doidge-Harrison, Howie & Wardell, 1991). Chromatographic separation of the product mixture resulted in the isolation of the crystalline title compound, (II) [NMR (CDCl₃): δ¹¹⁹Sn -139.8 p.p.m.]. In order to both characterize this product unambiguously and determine its stereochemistry, the crystal structure determination of (II) was carried out.

Analysis showed compound (II) (Fig. 1) to be clearly derived from the *trans* stereoisomer, (*Ia*), of the (*Ia*)/(*Ib*) mixture, which had axial N—H moieties. The molecule of (II) lies about an inversion centre and the conformation is stabilized by intramolecular N—H...O bifurcated hydrogen bonding (Fig. 1 and Table 2). There are

Data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSCIAFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation (1992). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1090). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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