Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Bis(3-chlorobenzyl)tin(IV) ester of pyruvic acid 4-hydroxybenzoylhydrazone

Min Hong, Handong Yin,* Daqi Wang and Gang Li

College of Chemistry and Chemical Engineering, Liaocheng University, Shandong 252059, People's Republic of China

Correspondence e-mail: handongyin@lctu.edu.cn

Received 14 April 2005
Accepted 21 April 2005
Online 30 April 2005

The Sn atom in the title complex, bis[μ-pyruvic acid 4-hydroxybenzoylhydrazonato(2−)]bis[bis(3-chlorobenzyl)-ethanoltin(IV)], $[Sn_2(C_8H_6Cl)_4(C_{10}H_8N_2O_4)_2(C_2H_6O)_2]$, is six-coordinate with a distorted octahedral geometry, three O atoms and one N atom forming the equatorial plane. Through additional weak Sn—O bonding and hydrogen bonds, two molecules form a dimer, which has crystallographic $\overline{1}$ symmetry. These discrete dimers are connected by hydrogen-bonding interactions into a three-dimensional supramolecular network.

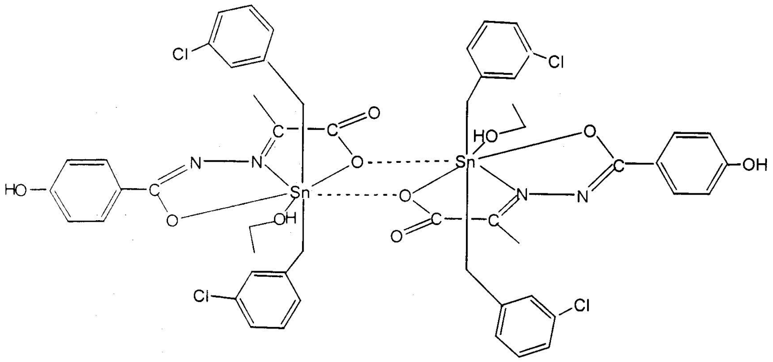
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.030
wR factor = 0.083
Data-to-parameter ratio = 14.4

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

Comment

In the title complex, (I), the Sn atom exists in a distorted octahedral coordination environment in which one ethanol molecule, one tridentate pyruvic acid 4-hydroxybenzoylhydrazone ligand, and two *trans* 3-chlorobenzyl groups coordinate to each Sn center. Atoms O1, O3, N1 and O5, which form the equatorial plane, are coplanar within 0.0917 Å and the axial angle C11—Sn1—C18 [160.40 (14)°] deviates from the linear value of 180°, indicating that the Sn atom has a distorted octahedral configuration. Atom O1 of the carboxylate residue also binds more weakly to the other Sn atom, Sn^i, generating an Sn_2O_2 four-membered ring [symmetry code: (i) $-x$, $-y$, $-z$]. The $Sn1—O1^i$ distance of 2.764 Å is longer than Sn1—O1 [2.357 (2) Å], but comparable with the values found in related seven-coordinate diorganotin systems (Gielen *et al.*, 1998; Yin *et al.*, 2003).



(I)

The structure of the title complex can be described as a dimer, formed through weak Sn—O bonding interactions, with crystallographically imposed $\overline{1}$ symmetry, and the coordination geometry of tin can be also described as a *trans*-C_2SnO_4N pentagonal bipyramid with the two 3-chlorobenzyl groups occupying *trans* positions [C11—Sn1—C18 = 160.40 (14)°]. The formation of the dimer leads to a short distance between O and O^i, which does not represent a direct bond.

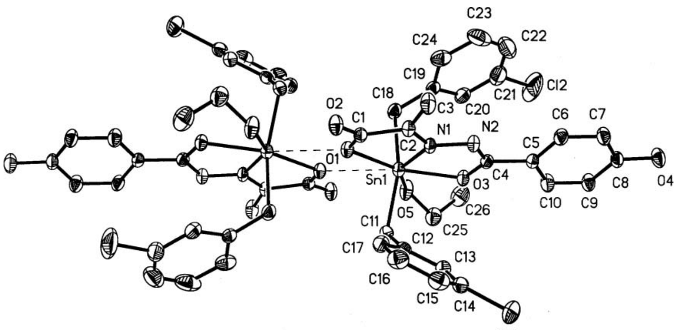


Figure 1
The structure of the title complex, showing 30% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity. Unlabeled atoms are related by the symmetry code ($-x$, $-y$, $-z$).

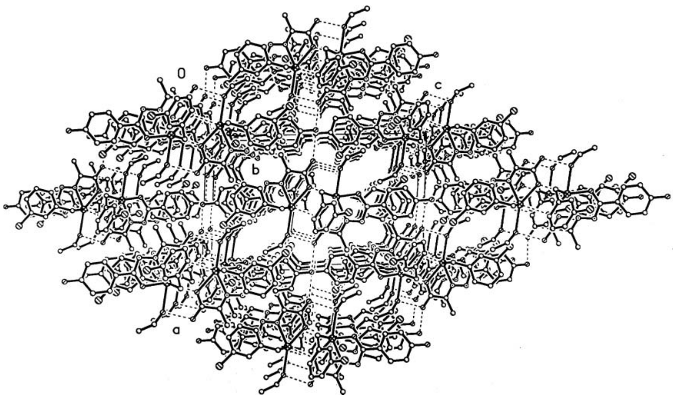


Figure 2
A packing diagram, showing the three-dimensional structure of the title complex formed *via* hydrogen-bonding (dashed lines) interactions.

The C4—O3 bond length [1.293 (4) Å] lies between double- (1.224 Å) and single-bond (1.430 Å) lengths. Compared with the length of a C═N double bond (1.270 Å) and a C—N single bond (1.470 Å), both the C4—N2 [1.333 (4) Å] and C2—N1 [1.293 (4) Å] bonds should be classified as C═N double bonds. The N1—N2 bond [1.368 (3) Å] falls within the normal range of N—N single bonds (Yang *et al.*, 1999; He *et al.*, 2002). These data indicate that the Schiff base ligand forms a C═N—N═C conjugated system, which is introduced into the inner coordination sphere and functions as a tridentate chelate with the O, N and O atoms in the deprotonated enol form. In this molecule, the phenol O atoms do not participate in the coordination to the Sn atoms.

Each Sn atom is also coordinated by an ethanol molecule, the Sn—O bond distance being 2.424 (3) Å, which is longer than those in analogous compounds (Yin *et al.*, 2003; Gielen *et al.*, 1998) due to the formation of intradimer hydrogen bonds [O2···O5^{i} (or O2^{i}···O5) of 2.696 (4) Å]. Neighboring molecules are held together by interdimer O—H···O hydrogen bonds [O4···O2^{ii} = 2.577 Å; symmetry code: (ii) $x-\frac{1}{2}$, $-y+\frac{1}{2}$, $z+\frac{1}{2}$]. These hydrogen bonds contribute to the crystal stability and compactness and result in a three-dimensional dimeric supramolecular network arrangement (Fig. 2).

Experimental

Pyruvic acid 4-hydroxybenzhydrazone (1 mmol) and sodium ethoxide (1 mmol) were added to dry benzene (20 ml) in a Schlenk flask and stirred for 0.5 h. Bis(3-chlorobenzyl)tin dichloride (1 mmol) was then added and the reaction mixture was stirred for 12 h at 313 K and then filtered. The solvent was gradually removed by evaporation under vacuum until a solid product was obtained. The solid was then recrystallized from ethanol and light-yellow crystals suitable for X-ray diffraction were obtained (m.p. 472 K). Elemental analysis calculated for $C_{52}H_{52}Cl_4N_4O_{10}Sn_2$: C 48.84, H 3.39, N 4.75%; found: C 49.02, H 3.31, N 4.67%.

Crystal data

$C_{52}H_{52}Cl_4N_4O_{10}Sn_2$
$M_r = 1272.16$
Monoclinic, $P2_1/n$
$a = 11.887$ (2) Å
$b = 11.775$ (2) Å
$c = 19.293$ (3) Å
$\beta = 92.171$ (2)°
$V = 2698.4$ (8) Å^3
$Z = 2$

$D_x = 1.566$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6599 reflections
$\theta = 2.4$–28.0°
$\mu = 1.18$ mm^{-1}
$T = 298$ (2) K
Block, light yellow
0.48 × 0.43 × 0.27 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.601$, $T_{max} = 0.741$
13 866 measured reflections

4753 independent reflections
3808 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 25.0°$
$h = -14 \rightarrow 7$
$k = -13 \rightarrow 14$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.083$
$S = 1.01$
4753 reflections
329 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0403P)^2 + 2.5821P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.81$ e Å^{-3}
$\Delta\rho_{min} = -0.70$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—O3	2.137 (2)	N1—C2	1.293 (4)
Sn1—N1	2.228 (3)	N1—N2	1.368 (3)
Sn1—O1	2.357 (2)	N2—C4	1.333 (4)
Sn1—O5	2.424 (3)	O3—C4	1.293 (4)
Sn1—O1^{i}	2.764 (2)		
O3—Sn1—C11	93.70 (11)	O3—Sn1—O1	140.44 (8)
C11—Sn1—C18	160.40 (14)	C11—Sn1—O1	90.32 (11)
O3—Sn1—N1	71.17 (9)	N1—Sn1—O1	69.67 (8)
C18—Sn1—N1	94.47 (13)	O3—Sn1—O5	77.14 (9)

Symmetry code: (i) $-x$, $-y$, $-z$.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O2^{ii}	0.82	1.97	2.767 (4)	165
O5—H1···O2^{i}	0.77 (5)	2.00 (5)	2.696 (4)	151 (5)

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

H atoms attached to C atoms were positioned geometrically and treated as riding on their parent atoms, with aromatic C—H distances of 0.93 Å, methylene C—H distances of 0.97 Å and methyl C—H distances of 0.96 Å. The $U_{\rm iso}$(H) values were set at $1.5U_{\rm eq}$(C) for the methyl H atoms and at $1.2U_{\rm eq}$(C) for the other C-bound H atoms. The fixed O—H distance was O4—H4 = 0.82 Å. The coordinates of the H atom bonded to atom O5 were refined freely.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

The authors acknowledge the financial support of the Shandong Province Science Foundation and the State Key Laboratory of Crystal Materials, Shandong University, People's Republic of China.

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