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Bis[μ-pyruvic acid benzoylhydrazonato(2−)]-bis[bis(4-chlorobenzyl)ethanoltin(IV)]

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The Sn atom in the title complex, $[Sn_2(C_7H_6Cl)_4(C_{10}H_8N_2O_3)_2(C_2H_6O)_2]$, has a distorted pentagonal–bipyramidal configuration, being coordinated by three O atoms and one N atom from the pyruvic acid benzoylhydrazone ligands, one O atom of an ethanol molecule and two axial C atoms from *trans* 4-chlorobenzyl groups, thus forming a dimeric molecule, which has crystallographic $\overline{1}$ symmetry. Intramolecular hydrogen bonds contribute to the stability and compactness of the crystal structure.

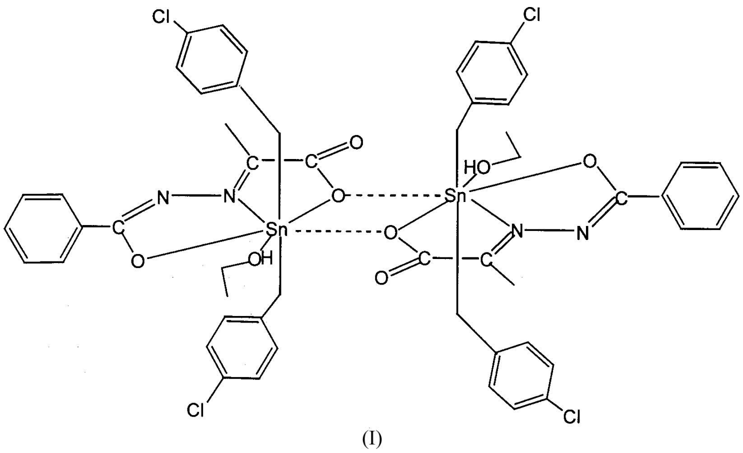
Key indicators

Single-crystal X-ray study
$T = 298$ K
Mean σ(C–C) = 0.07 Å
R factor = 0.034
wR factor = 0.106
Data-to-parameter ratio = 14.8

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) (Fig. 1), the Sn atom exists in a distorted pentagonal–bipyramidal coordination environment in which one ethanol molecule, one tridentate and one monodentate pyruvic acid benzoylhydrazone ligands, and two *trans* 4-chlorobenzyl groups coordinate to each Sn center. Atoms O1, O1^i, O4, O3 and N1 are coplanar within 0.062 Å [symmetry code: (i) $-x + 1, -y + 2, -z + 2$] and form the equatorial plane. The axial angle C11—Sn1—C18 deviates from the ideal value of 180° (Table 1). 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. The Sn1–O1^i bond length is longer than Sn1—O1, but comparable with values found in related seven-coordinate diorganotin systems (Gielen *et al.*, 1998; Yin *et al.*, 2003). The structure of (I) can thus be described as a dimer, formed through weak Sn–O bonding interactions, with crystallographically imposed $\overline{1}$ symmetry.



(I)

The C4—O3 bond length 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), the C4—N2 and C2—N1 bonds should be classified

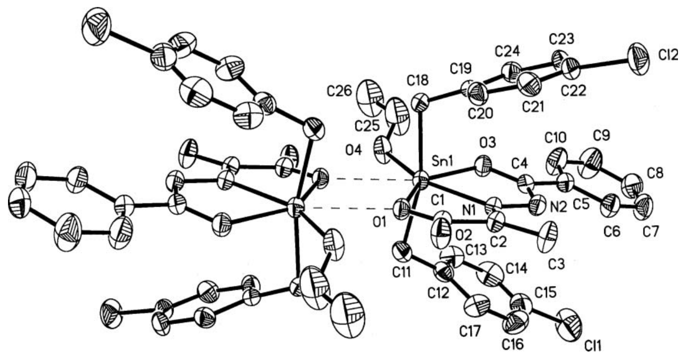


Figure 1
The structure of the title complex, showing 30% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity. Unlabeled atoms are related by the symmetry operation ($-x+1$, $-y+2$, $-z+2$).

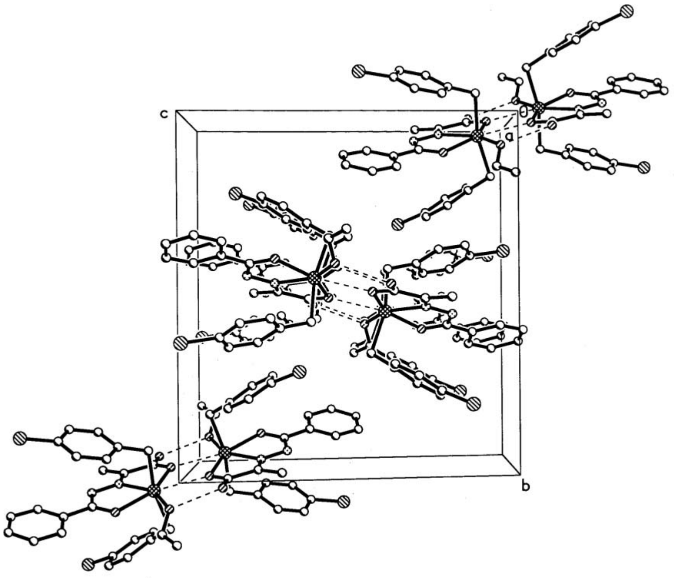


Figure 2
The crystal packing of the title complex, viewed along the *b* axis. H atoms have been omitted.

as C═N double bonds. The N1—N2 bond length 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 an *O*,*N*,*O*-tridentate chelate ligand in the deprotonated enol form.

Each Sn atom is also coordinated by an ethanol molecule, the Sn—O4 bond distance being relatively longer than those in the analogous compounds (Yin *et al.*, 2003; Gielen *et al.*, 1998), because of the formation of intradimer hydrogen bonds. These hydrogen bonds contribute to the stability and compactness of the crystal structure (Fig. 2).

Experimental

Pyruvic acid benzoylhydrazone (1 mmol) and sodium ethoxide (1 mmol) were added to dry benzene (20 ml) in a Schlenk flask and stirred for 0.5 h. Di-*p*-chlorobenzyltin 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 colorless crystals suitable for X-ray diffraction were obtained (m.p. 483 K). Elemental analysis calculated for $C_{52}H_{52}Cl_4N_4O_8Sn_2$: C 50.32, H 4.19, N 4.52; found: C 49.97, H 4.31, N 4.57%.

Crystal data

$[Sn_2(C_7H_6Cl)_4(C_{10}H_8N_2O_3)_2(C_2H_6O)_2]$
$M_r = 1240.16$
Monoclinic, $P2_1/n$
$a = 10.4162$ (14) Å
$b = 16.693$ (2) Å
$c = 15.536$ (2) Å
$\beta = 97.474$ (2)°
$V = 2678.4$ (6) Å^3
$Z = 2$
$D_x = 1.538$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 5918 reflections
$\theta = 2.3$–27.3°
$\mu = 1.19$ mm^{-1}
$T = 298$ (2) K
Block, colorless
0.53 × 0.47 × 0.34 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.572$, $T_{max} = 0.688$
13835 measured reflections
4723 independent reflections
3591 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 25.0°$
$h = -12 \rightarrow 12$
$k = -19 \rightarrow 19$
$l = -12 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.106$
$S = 1.00$
4723 reflections
319 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 2.1458P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.65$ e Å^{-3}
$\Delta\rho_{min} = -0.54$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—C18	2.148 (5)	Sn1—O1^i	2.682 (2)
Sn1—O3	2.148 (3)	N1—C2	1.283 (6)
Sn1—C11	2.151 (5)	N1—N2	1.372 (5)
Sn1—N1	2.235 (4)	N2—C4	1.318 (6)
Sn1—O1	2.353 (3)	O3—C4	1.293 (5)
Sn1—O4	2.394 (4)		
C18—Sn1—O3	92.91 (16)	N1—Sn1—O1	69.61 (12)
C18—Sn1—C11	165.72 (19)	C18—Sn1—O4	87.67 (17)
O3—Sn1—C11	96.52 (17)	C11—Sn1—O4	83.9 (2)
C18—Sn1—N1	97.67 (16)	C18—Sn1—O1^i	83.63 (12)
O3—Sn1—N1	70.74 (13)	O3—Sn1—O1^i	153.83 (9)
C11—Sn1—N1	95.55 (18)	O1—Sn1—O1^i	65.95 (10)
C11—Sn1—O1	92.10 (17)	O4—Sn1—O1^i	76.14 (10)

Symmetry code: (i) $-x+1, -y+2, -z+2$.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H1···O2^i	0.85 (6)	1.77 (6)	2.619 (5)	173 (6)

Symmetry code: (i) $-x+1, -y+2, -z+2$.

Acta Cryst. (2005). E**61**, m1248–m1250 Hong *et al.* • $[Sn_2(C_7H_6Cl)_4(C_{10}H_8N_2O_3)_2(C_2H_6O)_2]$ **m1249**

H atoms attached to C atoms were all 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 coordinates of the H atom bonded to O4 were refined, giving an O—H distance of 0.90 (7) Å.

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*.

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