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Bis(4-fluorobenzyl)tin(IV) ester of pyruvic acid salicylhydrazone

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In the title complex, bis[μ-salicylaldehyde (1-carboxylatoethylidene)hydrazonato(2−)]bis[ethanolbis(4-fluorobenzyl)tin(IV)], $[Sn_2(C_7H_6F)_4(C_{10}H_8N_2O_4)_2(C_2H_6O)_2]$, each central Sn^{IV} atom has a distorted pentagonal–bipyramidal configuration, being coordinated by three O atoms and one N atom from the pyruvic acid salicylhydrazone ligands, one O atom from an ethanol molecule and two axial C atoms from *trans* 4-fluorobenzyl groups, thus forming a dimeric molecule, which sits on a crystallographic center of symmetry. Intramolecular hydrogen bonds contribute to the stability and compactness of the crystal structure.

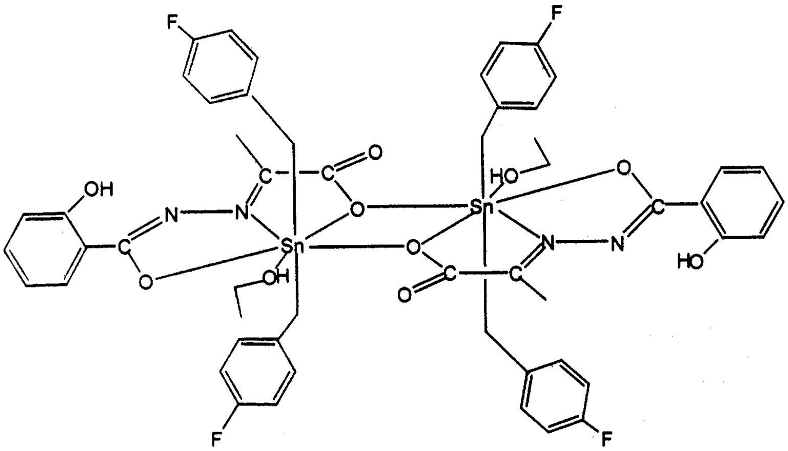
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.029
wR factor = 0.080
Data-to-parameter ratio = 13.7

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), each Sn atom is in a distorted pentagonal–bipyramidal coordination environment, in which one ethanol molecule, two tridentate pyruvic acid salicylhydrazone ligands and two *trans* p-fluorobenzyl groups coordinate to each Sn center. Atoms O1, O5, O1^i, O3 and N1 are coplanar within 0.0346 Å [symmetry code: (i) $-x+1, -y, -z$] and form the equatorial plane. The axial C11—Sn1—C18 angle is 165.71 (13)°, which deviates from the ideal value of 180°. Atom O1 of the carboxylate residue also binds another Sn atom, generating an Sn_2O_2 four-membered ring. The structure of this complex can thus be described as a dimer, with crystallographically imposed $\bar{1}$ symmetry. The formation of the dimer leads to a short interaction between atoms O1 and O1^i, because the interaction of two monomers surpasses the repulsive effect of the two O atoms.



(I)

The C—O bond length [C4—O3 = 1.281 (4) Å] lies between a double- (1.224 Å) and single-bond (1.430 Å) length. Compared to the length of a C=N double bond (1.270 Å) and

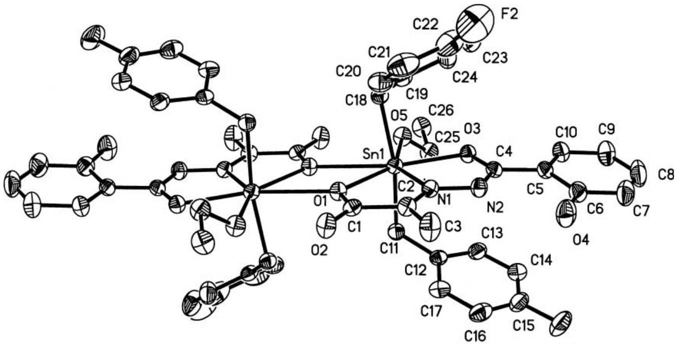


Figure 1
The structure of the title complex, showing 30% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity. Unlabeled atoms are related by the symmetry operation $1 - x$, $-y$, $-z$.

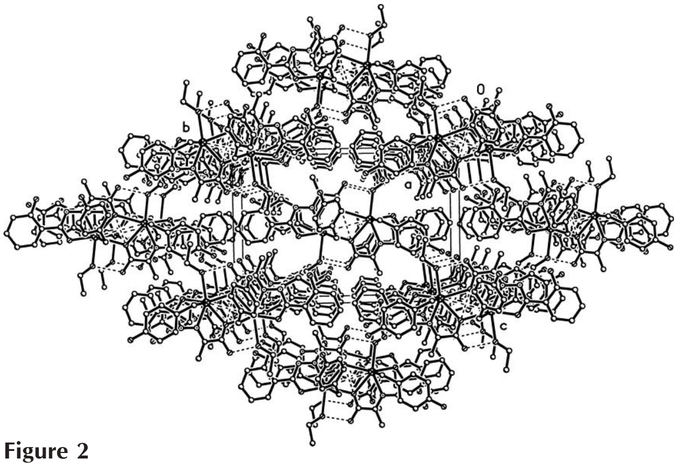


Figure 2
Crystal packing of the title complex, showing the hydrogen-bonded interactions as dashed lines. H atoms have been omitted.

a C—N single bond (1.470 Å), both the C4—N2 [1.331 (4) Å] and C2—N1 [1.281 (4) Å] bonds should be classified as C=N double bonds. The N1—N2 bond [1.369 (4) 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 O, N and O atoms in the deprotonated enol form. In this molecule, the phenol O atoms do not participate in coordination to the Sn atoms.

Each Sn atom is also coordinated by an ethanol molecule, the Sn—O bond distance being 2.417 (2) Å, which is longer than those found in analogous compounds (Yin *et al.*, 2003; Parulekar *et al.*, 1989), due to the formation of intradimer hydrogen bonds, *viz*. O2···O5^{i} (or O2^{i}···O5) of 2.625 (4) Å. There are also strong intramolecular O—H···N hydrogen bonds (O···N = 2.577 Å) involving atom N2 and the phenol O atom. These hydrogen bonds contribute to the crystal stability and compactness.

Experimental

Pyruvic acid salicylhydrazone (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*-fluorobenzyltin 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. 407 K). Elemental analysis calculated for $C_{52}H_{52}F_4N_4O_{10}Sn_2$: C 51.73, H 4.31, N 4.64%; found: C 51.81, H 4.26, N 4.67%.

Crystal data

$[Sn_2(C_7H_6F)_4(C_{10}H_8N_2O_4)_2(C_2H_6O)_2]$
$M_r = 1206.36$
Monoclinic, $P2_1/n$
$a = 11.063\ (2)$ Å
$b = 18.313\ (3)$ Å
$c = 12.615\ (2)$ Å
$\beta = 92.578\ (2)^\circ$
$V = 2553.2\ (8)$ Å^3
$Z = 2$
$D_x = 1.569$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5043 reflections
$\theta = 2.4\text{–}26.9^\circ$
$\mu = 1.05$ mm^{-1}
$T = 298\ (2)$ K
Block, colorless
0.38 × 0.35 × 0.31 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.676$, $T_{max} = 0.721$
13036 measured reflections
4506 independent reflections
3421 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 25.0^\circ$
$h = -6 \rightarrow 13$
$k = -21 \rightarrow 21$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.080$
$S = 1.00$
4506 reflections
329 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0422P)^2 + 0.5048P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.54$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—O3	2.171 (2)	N1—N2	1.369 (4)
Sn1—N1	2.253 (3)	N2—C4	1.331 (4)
Sn1—O1	2.363 (2)	O1—C1	1.281 (4)
Sn1—O5	2.417 (2)	O2—C1	1.226 (4)
Sn1—O1^{i}	2.681 (2)	O3—C4	1.281 (4)
N1—C2	1.281 (4)	O4—C6	1.353 (5)
C18—Sn1—C11	165.71 (13)	C11—Sn1—O5	87.91 (11)
C18—Sn1—O3	93.38 (11)	N1—Sn1—O5	147.87 (9)
C18—Sn1—N1	99.22 (12)	O1—Sn1—O5	143.27 (8)
O3—Sn1—N1	70.49 (9)	C18—Sn1—O1^{i}	83.62 (10)
C18—Sn1—O1	89.69 (11)	O3—Sn1—O1^{i}	156.91 (7)
O3—Sn1—O1	139.18 (8)	O1—Sn1—O1^{i}	63.85 (8)

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

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···N2	0.82	1.86	2.577 (4)	146
O5—H1···O2^{i}	0.839 (19)	1.81 (2)	2.625 (4)	163 (4)

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

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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 O4—H4 distance was fixed at 0.82 Å; the coordinates of the H atom bonded to O5 was refined, giving an O—H distance of 0.839 (19) 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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