metal-organic papers

[3-Methoxy-2-oxidobenzaldehyde (2-thienyl-carbonyl)hydrazonato]dimethyltin(IV)

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.019 Å
Disorder in main residue
R factor = 0.067
wR factor = 0.236
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.

In the molecule of the title complex, [Sn(CH$_3$)$_2$-(C$_{13}$H$_{10}$N$_2$O$_3$S)], the Sn atom is in a distorted trigonal–bipyramidal geometry. There are two molecules in the asymmetric unit. A centrosymmetric arrangement leads to an Sn$_2$O$_2$ core through weak Sn$\cdots$O interactions and causes the formation of a dimer. Intermolecular C—H$\cdots$O hydrogen bonds seem to be effective in the stabilization of the crystal structure.

Comment

In the molecule of the title complex, (I), the Sn atom adopts a distorted trigonal–bipyramidal geometry (Table 1 and Fig. 1). The Schiff base ligand coordinated to the Sn atom is tridentate *via* the azomethine N atom, and the hydroxy and carbonyl O atoms. A centrosymmetric arrangement leads to an Sn$_2$O$_2$ core through weak Sn$\cdots$O [2.995 (8) Å] interactions and causes the formation of a dimer.

(I)
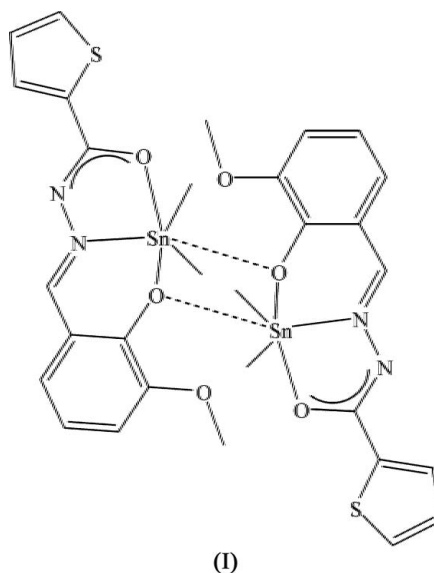
The distorted trigonal–bipyramidal geometry may be best described by the C15—Sn1—N1, C14—Sn1—C15, O1—Sn1—N1 and N1—Sn1—O3 angles (Table 1). The distorted geometry can also be seen in the deviation of the O1—Sn1—O3 angle from 180°.

The C—N—N—C chain shows conjugation, as evidenced by the intermediate values for the bond lengths (Table 1). The Sn1—N1 bond length is close to the sum of the non-polar covalent radii (2.15 Å; Sanderson, 1967), indicating a strong Sn—N interaction.

Intermolecular C—H$\cdots$O hydrogen bonds (Table 2) seem to be effective in the stabilization of the crystal structure.
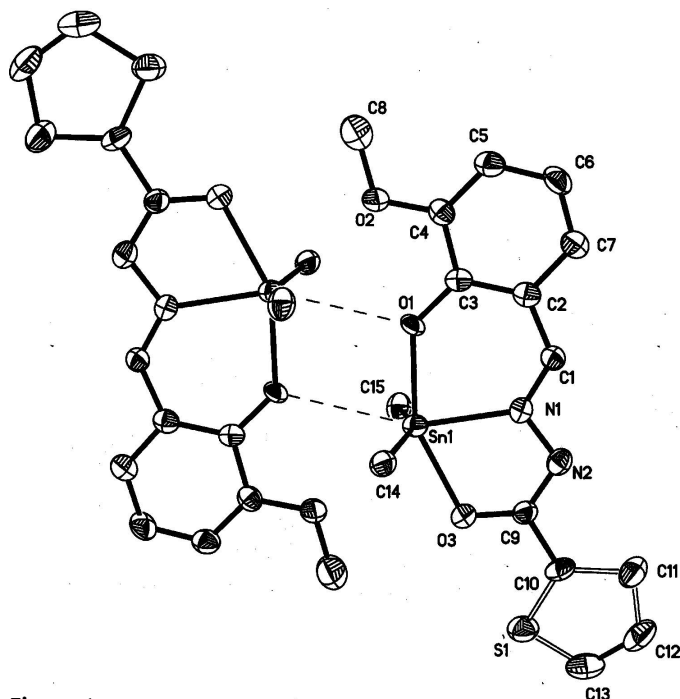
Figure 1
The hydrogen-bonded (dashed lines) dimer of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms have been omitted for clarity. Only one disorder component is shown.
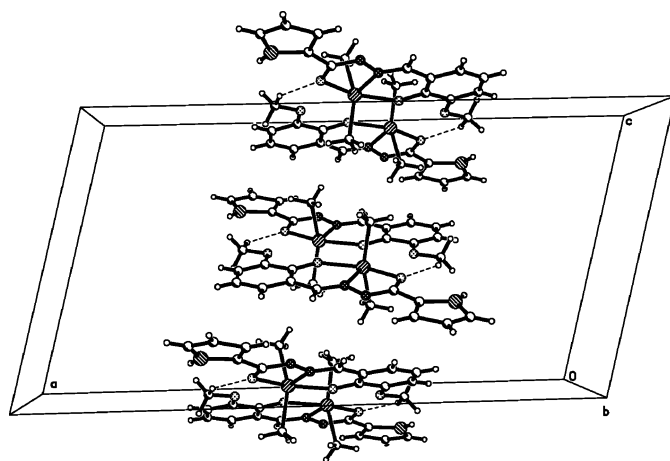

Figure 2
Partial packing diagram of (I). Intermolecular C—H···O hydrogen bonds are shown as dashed lines. Only one disorder component is shown.

Experimental

The reaction was carried out under a nitrogen atmosphere using standard Schlenk techniques. o-Vanillin-2-thiophenylhydrazone (0.2516 g, 1.0 mmol) was added to a mixture of ethanol and benzene (1:3 v/v, 30 ml) with sodium ethoxide (0.068 g, 1.0 mmol). It was stirred for 0.5 h, and then $(CH_3)SnCl_2$ (0.2197 g, 1.0 mmol) was added and stirring continued for an additional 10 h under reflux. After cooling to room temperature, the mixture was filtered and evaporated. The solid product was then recrystallized from dichloromethane/hexane (1:1 v/v) (yield 0.265 g, 62.6%; m.p. 515–516 K). Analysis calculated: C 42.55, H 3.78, N 6.62%; found: C 42.43, N 3.56, N 6.53%.

Crystal data

$[Sn(CH_3)_2(C_{13}H_{10}N_2O_3S)]$	$Z = 8$
$M_r = 423.08$	$D_x = 1.727$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
$a = 25.777$ (11) Å	$\mu = 1.71$ mm^{-1}
$b = 9.670$ (4) Å	$T = 298$ (2) K
$c = 13.476$ (6) Å	Block, colorless
$\beta = 104.318$ (6)°	$0.48 \times 0.23 \times 0.07$ mm
$V = 3255$ (2) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	8215 measured reflections
φ and ω scans	2858 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	2074 reflections with $I > 2\sigma(I)$
$T_{min} = 0.494$, $T_{max} = 0.890$	$R_{int} = 0.044$
	$\theta_{max} = 25.0°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.145P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.067$	$+ 47.0333P]$
$wR(F^2) = 0.236$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} < 0.001$
2858 reflections	$\Delta\rho_{max} = 2.02$ e Å^{-3}
199 parameters	$\Delta\rho_{min} = -0.89$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Sn1—C14	2.078 (12)	N1—C1	1.279 (14)
Sn1—C15	2.099 (12)	N1—N2	1.385 (13)
Sn1—O1	2.153 (7)	N2—C9	1.303 (15)
Sn1—O3	2.200 (8)	O1—C3	1.326 (13)
Sn1—N1	2.210 (9)	O3—C9	1.281 (14)
Sn1—O1^i	2.995 (8)		
C14—Sn1—C15	150.0 (5)	O1—Sn1—N1	82.4 (3)
C14—Sn1—O1	94.0 (4)	O3—Sn1—N1	71.4 (3)
C15—Sn1—O1	96.4 (5)	C14—Sn1—O1^i	77.3 (4)
C14—Sn1—O3	91.6 (4)	C15—Sn1—O1^i	78.0 (4)
C15—Sn1—O3	91.5 (5)	O1—Sn1—O1^i	75.7 (3)
O1—Sn1—O3	153.6 (3)	O3—Sn1—O1^i	130.7 (3)
C14—Sn1—N1	107.2 (4)	N1—Sn1—O1^i	157.9 (3)
C15—Sn1—N1	102.1 (4)		

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

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8A···O3^i	0.96	2.29	3.09 (2)	140

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

H atoms were positioned geometrically, with C—H = 0.93 and 0.96 Å for aromatic and methyl H atoms, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C)$, where $x = 1.2$ for aromatic and $x = 1.5$ for methyl H atoms. The title molecule was refined with statistical disorder, with the site occupancies of 0.67 (3) (for S1, C11 and H11) and 0.33 (3) (for S1′, C11′ and H11′). The highest peak is located 1.18 Å from atom O1.

Data collection: *SMART* (Bruker, 1996); cell refinement: *SAINT* (Bruker, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics:

References

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