

**Xiao-Niu Fang,* Yan Sui,
 Shao-Ming Ying, Ya-Ping Xu and
 Xue-Feng Guo**

JiangXi Province Key Laboratory of Coordination
 Chemistry, College of Chemistry & Chemical
 Engineering, JingGangShan University, 343009
 Ji'an, JiangXi, People's Republic of China

Correspondence e-mail: fangxiaoniui@163.com

Key indicators

Single-crystal X-ray study
 T = 296 K
 Mean $\sigma(C-C)$ = 0.006 Å
 Disorder in main residue
 R factor = 0.032
 wR factor = 0.093
 Data-to-parameter ratio = 15.1

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

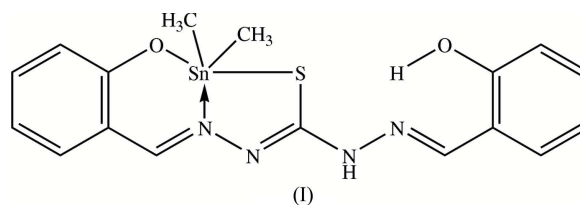
**[1,5-Bis(2-hydroxybenzylidene)thiocarbono-
 hydrazide(2-)- κ^3S,O,N]dimethyltin(IV)**

Received 22 July 2006
 Accepted 27 July 2006

In the title compound, $[Sn(CH_3)_2(C_{15}H_{12}N_4O_2S)]$, there are two independent molecules in the asymmetric unit which have almost identical geometries. It is a pentacoordinated Sn^{IV} complex, with distorted trigonal-bipyramidal geometry. 1,5-Bis(2-hydroxybenzylidene)thiocarbonohydrazide acts as a tridentate ligand, coordinating through the deprotonated phenolate O atom, imino N atom and thiolate S atom. Strong intermolecular N—H...N hydrogen bonds lead to the formation of dimers; the dimers are held together by van der Waals forces.

Comment

In recent years, organotin complexes have attracted more and more attention, partly owing to their proven or potential pharmaceutical value, the versatile molecular structure and supermolecular architecture exhibited by these complexes (Ma *et al.*, 2005) and, importantly, their good antitumor activities (Barbieri *et al.*, 2001). Thiosemicarbazones and their metal complexes are widely known for their wide range of biological applications, such as antiviral, antibacterial, anti-malarial, antifungal and antitumoral activities (Casas *et al.*, 2000). In the context of our continuing interest in the structural and biological properties of organotin compounds (Fang *et al.*, 2001, 2005), we report here the crystal structure of the title compound, (I). The structure is similar to that of structures previously reported (Ng *et al.*, 1994; Sousa *et al.*, 2001).



The title compound, (I), has two independent molecules in the asymmetric unit, with almost identical geometries. It is a pentacoordinated Sn^{IV} complex, with a distorted trigonal-bipyramidal geometry. 1,5-Bis(2-hydroxybenzylidene)thiocarbonohydrazide acts as a tridentate ligand, coordinating through the deprotonated phenolate O atom, imino N atom and thiolate S atom. In the coordination polyhedron, the O and S atoms (from the ligand) occupy the axial positions [the O4—Sn2—S1 bond angle is 158.67 (8)°, and O2—Sn1—S2 is 154.8 (2)°]. The two C atoms (from two methyl groups) and the imino N atom (from the ligand) occupy the equatorial plane; these equatorial atoms and the Sn atom are nearly coplanar, with mean deviations of 0.005 (5) and 0.023 (5) Å for Sn1 and Sn2, respectively, and the dihedral angle between the two planes is 50.2 (3)°.

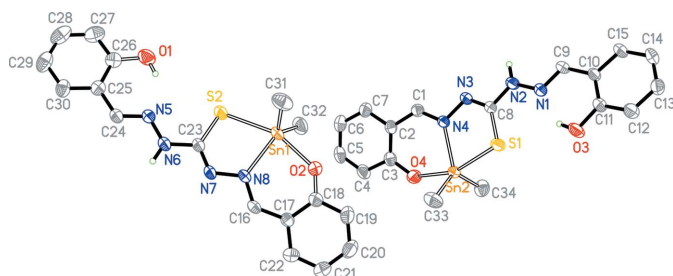


Figure 1
The asymmetric unit of (I), showing 30% probability displacement ellipsoids. All the H atoms on carbon and the disordered atom O2A have been omitted for clarity.

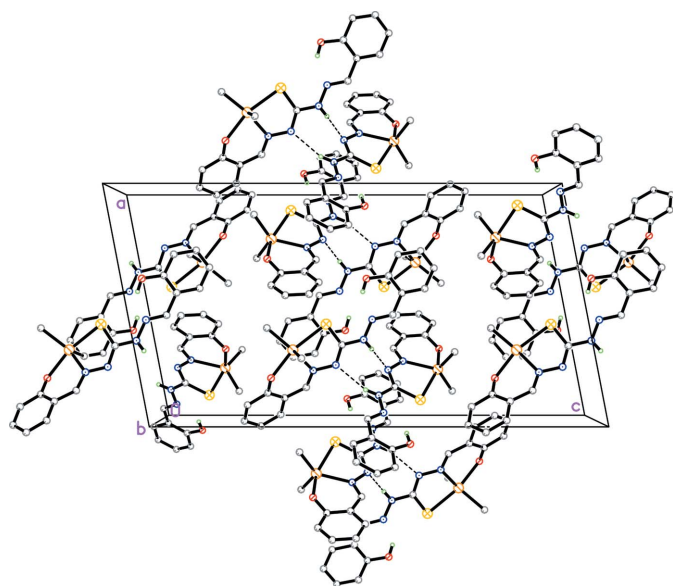


Figure 2
A packing diagram of (I), viewed along the *b* axis; hydrogen bonds are shown as dashed lines. All the H atoms on carbon and the disordered atom O2A have been omitted for clarity.

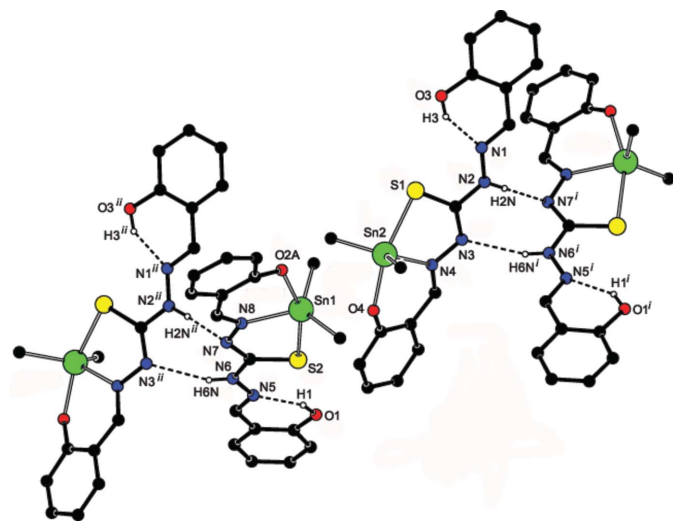


Figure 3
The hydrogen bonds in the packing of (I). Hydrogen bonds are shown as dashed lines. The disordered atom O2 has been omitted for clarity. [Symmetry codes: (i) $x, -y - \frac{1}{2}, z - \frac{1}{2}$; (ii) $x, -y - \frac{1}{2}, z - \frac{3}{2}$.]

All the bond lengths in (I) are within the expected range and in agreement with literature values (Ng *et al.*, 1994; Sousa *et al.*, 2001). The average C—S and Sn—S bond lengths are 1.726 (4) and 2.56 (3) Å, respectively; these values indicate that the ligand exists in the thiolate form, as shown in Fig. 1. This is not the same as the uncoordinated ligands, which exist in the thione form (Fang *et al.*, 2006; Zhang *et al.*, 2003).

Strong intermolecular N—H···N hydrogen bonds (as shown in Table 2, and Figs. 2 and 3) lead to the formation of dimers, which are held together by van der Waals forces.

Experimental

The title compound, (I), was prepared by the reaction of dimethyltin oxide (0.165 g, 1 mmol) with 1,5-bis(2-hydroxybenzylidene)thio-carbonohydrazide (0.314 g, 1 mmol) in acetonitrile (20 ml) at boiling point for about 5 h. The solvent was removed by vacuum distillation in a rotary evaporator and a yellow solid product was obtained. When the solid was recrystallized from methanol, single crystals suitable for X-ray diffraction analysis precipitated after several days (yield 80%; m.p. 441 K). MS (*m/e*) = 462.014, in accordance with the formula $C_{17}H_{18}N_4O_2SSn$.

Crystal data

[$Sn(CH_3)_2(C_{15}H_{12}N_4O_2S)$]
 $M_r = 461.10$
Monoclinic, $P2_1/c$
 $a = 14.2026$ (8) Å
 $b = 10.5032$ (6) Å
 $c = 26.3135$ (14) Å
 $\beta = 100.869$ (1)°
 $V = 3854.8$ (4) Å³

$Z = 8$
 $D_x = 1.589$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 1.45$ mm⁻¹
 $T = 296$ (2) K
Block, yellow
 $0.39 \times 0.18 \times 0.16$ mm

Data collection

Bruker APEX-II area-detector diffractometer
 φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2004)
 $T_{min} = 0.75, T_{max} = 0.80$

23464 measured reflections
7047 independent reflections
5441 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.038$
 $\theta_{max} = 25.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.032$
 $wR(F^2) = 0.093$
 $S = 1.01$
7047 reflections
466 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.054P)^2 + 1.068P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.82$ e Å⁻³
 $\Delta\rho_{min} = -0.77$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Sn1—O2	2.091 (7)	Sn2—C33	2.110 (4)
Sn1—C32	2.108 (4)	Sn2—C34	2.110 (4)
Sn1—C31	2.110 (5)	Sn2—N4	2.218 (3)
Sn1—N8	2.193 (3)	Sn2—S1	2.5393 (10)
Sn1—S2	2.5805 (10)	S1—C8	1.722 (4)
Sn2—O4	2.095 (3)	S2—C23	1.729 (3)
O4—Sn2—S1	158.67 (8)	C32—Sn1—C31	125.23 (19)
O2—Sn1—S2	154.8 (2)	C3—O4—Sn2	132.6 (2)
C8—S1—Sn2	95.30 (12)	C23—S2—Sn1	94.52 (12)
C33—Sn2—C34	126.6 (2)	C18—O2—Sn1	132.9 (6)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H3 \cdots N1	0.82	1.87	2.589 (4)	146
O1—H1 \cdots N5	0.82	1.92	2.636 (4)	146
N2—H2N \cdots N7 ⁱ	0.86	2.10	2.949 (4)	171
N6—H6N \cdots N3 ⁱⁱ	0.86	2.33	3.155 (4)	161

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

In the structure of (I), there are two independent molecules in the asymmetric unit and atom O2 in one of these molecules is disordered. The site-occupation factors of the disordered atoms O2 and O2A refined to 0.372 (13) and 0.628 (13), respectively. All H atoms were positioned in idealized locations and refined as riding on their carrier atoms, with C—H distances of 0.93 (aryl) and 0.96 Å (methyl), N—H = 0.86 Å and O—H = 0.82 Å; $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C,N})$ for the H atoms of aryl and NH groups, and $1.5U_{\text{eq}}(\text{C,O})$ for other H atoms.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *APEX2*; program(s) used to refine structure: *APEX2*; molecular graphics: *APEX2*; software used to prepare material for publication: *APEX2*.

We gratefully acknowledge financial support from the Natural Science Foundation of JiangXi Province (No. 0620029), and the Science and Technology Bureau of Ji'an City (grant No. 200528).

References

- Barbieri, F., Sparatore, F., Cagnoli, M., Bruzzo, C., Novelli, F. & Alama, A. (2001). *Chemico-Biol. Interact.* **134**, 27–39.
- Bruker (2004). *APEX2*. Version 1.22. Bruker AXS Inc., Madison, Wisconsin, USA.
- Casas, J. S., Garcia-Tasende, M. S. & Sordo, J. (2000). *Coord. Chem. Rev.* **209**, 197–261.
- Fang, X.-N., Luo, Q.-Y., Zeng, X.-R., Sui, Y. & Li, X.-F. (2005). *Acta Cryst.* **E61**, m2604–m2606.
- Fang, X.-N., Song, X.-Q. & Xie, Q. L. (2001). *J. Organomet. Chem.* **619**, 43–48.
- Fang, X.-N., Xu, Y.-P., Guo, X.-F. & Zeng, X.-R. (2006). *Acta Cryst.* **E62**, o1052–o1054.
- Ma, C.-N., Zhang, Q.-F., Zhang, R.-F. & Qiu, L.-L. (2005). *J. Organomet. Chem.* **690**, 3033–3043.
- Ng, S. W., Kumar Das, V. G., Luo, B. S. & Malk, T. C. W. (1994). *Z. Kristallogr.* **209**, 961–964.
- Sousa, G. F., Francisco, R. H. P., Gambadella, M. T. P., Santos, R. H. A. & Avras, A. (2001). *J. Braz. Chem. Soc.* **12**, 722–728.
- Zhang, A., Zhang, L.-X., Hou, Y. L., Xiong, Y., Li, X.-J. & Yu, K.-B. (2003). *Chin. J. Org. Chem.* **23**, 281–285.