Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Phenyltris(pyrimidine-2-thiolato)tin(IV)

Yong-Xin Li, Ru-Fen Zhang and Chun-Lin Ma*

Department of Chemistry, Liaocheng University, Liaocheng 252059, People's Republic of China

Correspondence e-mail: liyongxin_duan_0@yahoo.com

Received 21 February 2006
Accepted 29 March 2006

Key indicators

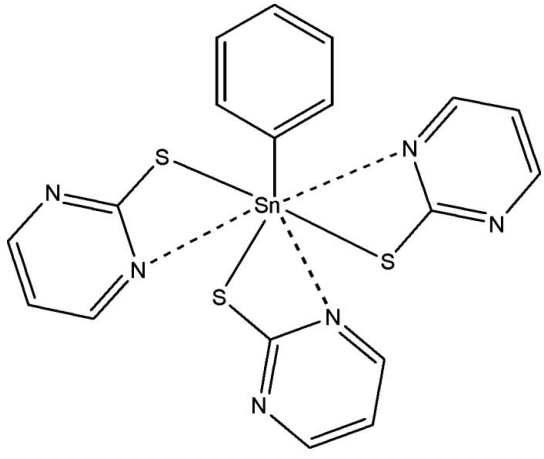
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.023
wR factor = 0.058
Data-to-parameter ratio = 14.5

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

The seven-coordinate Sn atom in the monomeric title compound, $[Sn(C_6H_5)(C_4H_3N_2S)_3]$, has distorted pentagonal–bipyramidal geometry. The axial C—Sn—S angle is 154.10 (7)°. Thiolate S and phenyl C atoms define the axial positions. Two S atoms and three N atoms define the pentagonal equatorial plane.

Comment

Investigation of organotin(IV) complexes has been focused on acquiring well defined solid-state structures to understand their versatile coordination chemistry. This work has examined organotin(IV) derivatives from heterocyclic thionates containing one or more N atoms and an adjacent, exocyclic thioketo group (Ma *et al.*, 2005). In our earlier work, we studied the coordination chemistry of four ligands of this type which contain at least one deprotonated heterocyclic thioamide group $(RN—C—S)^-$ and found that the S rather than the N atom coordinates to the Sn atom (Ma *et al.*, 2003). Within this context, we have undertaken the structural study of the title compound, (I), which is reported in this paper.



(I)

As shown in Fig. 1, the title complex has three bidentate SPym (2-mercaptopyridyl) ligands which form a distorted pentagonal bipyramid around tin, with two S atoms and three N atoms in the pentagonal equatorial plane and C and one S atom in axial positions. The Sn—C(Ph), Sn—S and Sn—N bond lengths (see Table 1) are all close to those found in $MeSn(SPy)_3$ [Sn—C 2.121 (3) Å, Sn—S 2.4805 (10)–2.5858 (10) Å and Sn—N 2.416 (3)–2.577 (3) Å] and $PhSn(SPy)_3$ [Sn—C 2.139 (5) Å, Sn—S 2.491 (2)–2.576 (2) Å and Sn—N 2.419 (4)–2.453 (5) Å] (Huber *et al.*, 1997). The

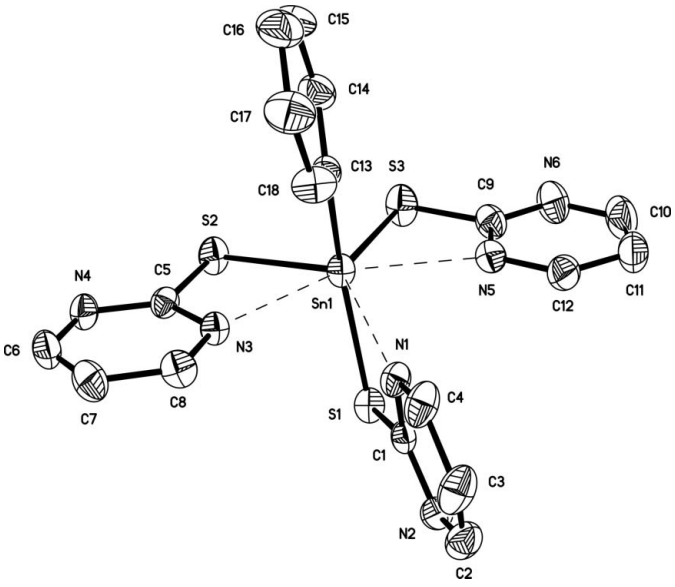


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity.

Sn—S bond lengths are slightly shorter than those in $PhSn(SPy)_3$, while the Sn—N bond lengths are slightly longer than those in $PhSn(SPy)_3$. The distortion of the pentagonal–bipyramidal geometry is evident considering the angle C13—Sn1—S1 of 154.10 (7)°. No directional intermolecular interactions were found.

Experimental

The reaction was carried out under a nitrogen atmosphere. Pyrimidine-2-thiol (0.168 g, 1.5 mmol) was added to sodium ethoxide (0.102 g, 1.5 mmol) dissolved in 30 ml of benzene and stirred for 10 min. Phenyltin trichloride (0.150 g, 0.5 mmol) was then added and the reaction mixture was kept at 313 K for 12 h. After cooling to room temperature, the solution was filtered, and the filtrate solvent was removed under vacuum. The residue was recrystallized from diethyl ether, and colorless crystals suitable for X-ray diffraction study were obtained (yield 0.212 g, 81%; m.p. 415 K). Analysis calculated for $C_{18}H_{14}N_6S_3Sn$: C 40.85, H 2.67, N 15.88%; found: C 40.87, H 2.63, N 15.91%.

Crystal data

$[Sn(C_6H_5)(C_4H_3N_2S)_3]$
M_r = 529.22
Monoclinic, $P2_1/c$
a = 16.242 (4) Å
b = 9.468 (2) Å
c = 14.111 (3) Å
β = 106.959 (3)°
V = 2075.6 (8) Å^3
Z = 4
D_x = 1.694 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 1.55 mm^{-1}
T = 298 (2) K
Block, colorless
0.48 × 0.39 × 0.32 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.523, T_{max} = 0.637
10588 measured reflections
3665 independent reflections
3127 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.058
S = 1.00
3665 reflections
253 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0258P)^2 + 1.3494P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.58 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—C13	2.139 (3)	Sn1—S2	2.5522 (8)
Sn1—S1	2.4556 (9)	Sn1—N1	2.618 (2)
Sn1—N3	2.458 (2)	N1—C1	1.339 (3)
Sn1—N5	2.498 (2)	N3—C5	1.349 (3)
Sn1—S3	2.5297 (8)	N5—C9	1.343 (4)
C13—Sn1—S1	154.10 (7)	C13—Sn1—S3	104.03 (8)
C13—Sn1—N3	84.38 (9)	C13—Sn1—S2	99.86 (7)
C13—Sn1—N5	92.13 (9)	C13—Sn1—N1	92.53 (9)
N3—Sn1—N5	148.77 (7)		

All H atoms were positioned geometrically and treated as riding atoms, with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

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 thank the National Natural Science Foundation of China (No. 20271025) for financial support.

References

Huber, F., Schmiedgen, R., Schurmann, M., Barbieri, R., Ruisi, G. & Silvestri, A. (1997). *Appl. Organomet. Chem.* **11**, 869–888.
Ma, C. L., Jiang, Q. & Zhang, R. F. (2003). *J. Organomet. Chem.* **678**, 148–155.
Ma, C. L., Tian, G. R. & Zhang, R. F. (2005). *Polyhedron*, **24**, 1773–1780.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.