

(2-Chlorobenzyl)tris(2-pyridinethiolato)tin(IV)

 Markus Schürmann,^{a*} Friedo Huber^a and Renato Barbieri^b

^aLehrstuhl für Anorganische Chemie II, Universität Dortmund, Otto-Hahn-Straße 6, D-44227 Dortmund, Germany, and ^bDipartimento di Chimica Inorganica, Università di Palermo, Via delle Scienze, 90128 Palermo, Italy
Correspondence e-mail: msch@platon.chemie.uni-dortmund.de

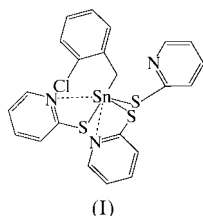
Received 7 June 2000

Accepted 23 October 2000

In the title compound, (2-chlorobenzyl)tris(pyridine-2-thiolato)- $\kappa^2N,S;\kappa^2N,S;\kappa S$ -tin(IV), [Sn(C₇H₆Cl)(C₅H₄NS)₃], two of the three pyridine-2-thiolato ligands (SPy) are bidentate and one is monodentate. The bonding C atom of the 2-chlorobenzyl group, the S atom of the monodentate SPy and the S and N atoms of the two bidentate SPy ligands form a distorted octahedron around the Sn atom. The three S atoms and the N atom of one of the bidentate SPy ligands occupy the equatorial positions, while the N atom of the second bidentate SPy ligand and the C(CH₂) atom are axial. The axial N—Sn—C angle of 157.9 (1)° demonstrates the heavy distortion of the octahedron.

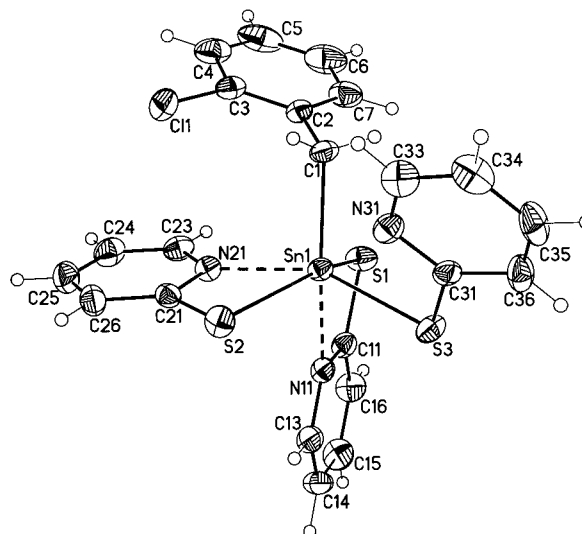
Comment

In mono- and diorganotin(IV) complexes of 2-mercaptopyridine (HSPy), such as RSn(SPy)₃ [R is Me or Ph (Huber *et al.*, 1997), or *p*-tolyl (Schürmann & Huber, 1994)], Ph₂Sn(SPy)₂ (Schmiedgen *et al.*, 1993) or Ph₂SnCl(SPy) (Schmiedgen, Huber, Preut *et al.*, 1994), SPy acts as a bidentate ligand and thus, the Sn atom achieves coordination numbers of 7, 6 and 5, respectively. In Sn(SPy)₄-HSPy, the Sn atom was found not to be eight-coordinate, as might have been expected, but only six-coordinate, two SPy ligands being bidentate and the other two monodentate (Damude *et al.*, 1990). A study of the structure of the title compound, (I), showed that bidentate and monodentate SPy ligands are also present in this compound, the Sn atom, in contrast with other RSn(SPy)₃ compounds, being six-coordinate.



(I)

All three SPy ligands of (I) are bonded *via* S to Sn. The Sn—S bond distances are comparable with the sums of the covalent


Figure 1

The molecular view of (I) showing the atom labelling scheme. Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as small spheres of arbitrary radii.

radii (2.42 Å; Bondi, 1964) and correspond to Sn—S bond distances observed in similar compounds. These were discussed in a previous paper on the structure of *n*-BuSn(SPy)₃ (SPy is 2-mercaptopyrimidine; Schmiedgen, Huber & Schürmann, 1994).

Each of the three Sn—N distances in (I) [2.303 (4)–3.113 (4) Å] is distinctly shorter than the sum of the appropriate van der Waals distances (3.75 Å; Bondi, 1964). However, the Sn—N distances of two of the SPy ligands (average value 2.315 Å) are shorter than in comparable compounds [*p*-TolSn(SPy)₃ 2.466 Å (Schürmann & Huber, 1994), MeSn(SPy)₃ 2.483 Å and PhSn(SPy)₃ 2.432 Å (Huber *et al.*, 1997), and Ph₂Sn(SPy)₂ 2.667 Å (Schmiedgen *et al.*, 1993)] and indicate, when compared with the sum of the covalent radii (2.15 Å; Bondi, 1964), the existence of Sn—N coordination and the bidentate nature of two of the SPy ligands. The Sn—N distance of the third ligand [3.113 (4) Å] is distinctly longer, thus excluding appreciable coordinative interaction. Accordingly, this ligand shows the shortest of the three Sn—S distances. Therefore, the coordination polyhedron around Sn can be described as a distorted octahedron.

The S—Sn—N bite angles [64.42 (9) and 63.20 (8)°] of the two bidentate ligands are similar to the appropriate angles in Sn(SPy)₄-HSPy [64.2 (2) and 65.1 (2)°; Damude *et al.*, 1990] and tend to be larger than in RSn(SPy)₃ and Ph₂Sn(SPy)₂, which contain only bidentate SPy ligands. Intermolecular distances shorter than the sum of the van der Waals radii were not found.

Experimental

Compound (I) was prepared from *o*-ClC₆H₄CH₂SnCl₃ (2.10 g, 6 mmol) in CHCl₃ (100 ml) and NaSPy (2.40 g, 18 mmol) in MeOH (40 ml) at room temperature. Single crystals of (I) (m.p. 446 K) were obtained from a solution in CHCl₃ containing some petroleum ether

at 243 K. The compound was characterized by elemental analysis and by IR, Mössbauer, NMR and mass spectroscopies (Huber *et al.*, 1997).

Crystal data

[Sn(C ₇ H ₆ Cl)(C ₅ H ₄ NS) ₃]	Z = 2
$M_r = 574.71$	$D_x = 1.648 \text{ Mg m}^{-3}$
Triclinic, <i>P</i> 1	Mo $K\alpha$ radiation
$a = 7.725 (3) \text{ \AA}$	Cell parameters from 46 reflections
$b = 10.321 (3) \text{ \AA}$	$\theta = 7.8\text{--}14.8^\circ$
$c = 15.548 (5) \text{ \AA}$	$\mu = 1.503 \text{ mm}^{-1}$
$\alpha = 80.26 (2)^\circ$	$T = 170 (1) \text{ K}$
$\beta = 77.18 (2)^\circ$	Plate, light yellow
$\gamma = 74.82 (2)^\circ$	$0.50 \times 0.14 \times 0.05 \text{ mm}$
$V = 1158.4 (7) \text{ \AA}^3$	

Data collection

Siemens <i>R3m/V</i> diffractometer	$R_{\text{int}} = 0.030$
$\omega/2\theta$ scans	$\theta_{\text{max}} = 25.05^\circ$
Absorption correction: ψ scan (<i>XEMP</i> in <i>P3 Software</i> ; Siemens, 1989)	$h = -9 \rightarrow 9$
$T_{\text{min}} = 0.501$, $T_{\text{max}} = 0.928$	$k = -12 \rightarrow 12$
5674 measured reflections	$l = -18 \rightarrow 18$
4098 independent reflections	6 standard reflections
3216 reflections with $I > 2\sigma(I)$	every 300 reflections
	intensity decay: 1.6%

Refinement

Refinement on F^2	Only H-atom U s refined
$R[F^2 > 2\sigma(F^2)] = 0.033$	$w = 1/[\sigma^2(F_o^2) + (0.0356P)^2]$
$wR(F^2) = 0.073$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.936$	$(\Delta/\sigma)_{\text{max}} = 0.001$
4098 reflections	$\Delta\rho_{\text{max}} = 0.54 \text{ e \AA}^{-3}$
273 parameters	$\Delta\rho_{\text{min}} = -0.46 \text{ e \AA}^{-3}$

All H atoms were placed in calculated positions using a riding model and refined with common isotropic displacement parameters for the different C—H types [C—H_{methylene} = 0.99 Å and $U_{\text{iso}} = 0.031 (9) \text{ \AA}^2$, and C—H_{aromatic} = 0.95 Å and $U_{\text{iso}} = 0.039 (3) \text{ \AA}^2$].

Data collection: *P3 Software* (Siemens, 1989); cell refinement: *P3 Software*; data reduction: *XDISK* in *P3 Software*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to

Table 1

Selected geometric parameters (Å, °).

Sn1—C1	2.205 (4)	Sn1—S1	2.6264 (13)
Sn1—N11	2.303 (4)	Sn1—S2	2.5362 (13)
Sn1—N21	2.324 (3)	Sn1—S3	2.4931 (13)
Sn1—N31	3.113 (4)	Sn1—Cl1	4.2240 (17)
C1—Sn1—N11	157.88 (13)	S2—Sn1—S1	144.61 (4)
N21—Sn1—S3	154.28 (9)		

refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 1990).

We thank the Fonds der Chemischen Industrie for financial support.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: AV1054). Services for accessing these data are described at the back of the journal.

References

- Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
 Damude, L. C., Dean, P. A. W., Manivannan, V., Srivastava, R. S. & Vittal, J. J. (1990). *Can. J. Chem.* **68**, 1323–1331.
 Huber, F., Schmiedgen, R., Schürmann, M., Barbieri, R., Ruisi, G. & Silvestri, A. (1997). *Appl. Organomet. Chem.* **11**, 869–888.
 Schmiedgen, R., Huber, F. & Preut, H. (1993). *Acta Cryst.* **C49**, 1735–1737.
 Schmiedgen, R., Huber, F., Preut, H., Ruisi, G. & Barbieri, R. (1994). *Appl. Organomet. Chem.* **8**, 397–407.
 Schmiedgen, R., Huber, F. & Schürmann, M. (1994). *Acta Cryst.* **C50**, 391–394.
 Schürmann, M. & Huber, F. (1994). *Acta Cryst.* **C50**, 206–209.
 Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
 Sheldrick, G. M. (1997a). *SHELXL97*. University of Göttingen, Germany.
 Sheldrick, G. M. (1997b). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
 Siemens (1989). *P3 Software*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Spek, A. L. (1990). *Acta Cryst.* **A46**, C-34.