Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis(4-benzylpiperidine-1-carbodithioato-κ^2S,S')-dimethyltin(IV)

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Received 9 November 2006
Accepted 22 December 2006

Key indicators

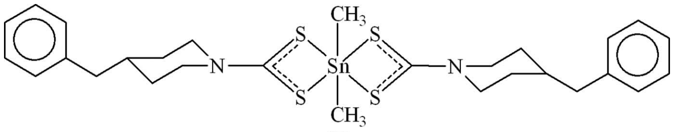
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.020
wR factor = 0.051
Data-to-parameter ratio = 14.9

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

The Sn atom in the title compound, $[Sn(CH_3)_2(C_{13}H_{16}NS_2)_2]$, has a highly distorted octahedral geometry that may be best described as skew-trapezoidal planar for thiocarboxylates. The ligands are asymmetrically coordinated to the Sn atom, with shorter Sn—S bond lengths that are very close to the sum of the covalent radii of Sn and S, while the longer Sn—S distances are significantly less than the sum of their van der Waals radii.

Comment

The increasing industrial use of organotin(IV) compounds containing an Sn—S bond, especially as stabilizers of polyvinyl chlorides (Tarassoli *et al.*, 2006), and the recognition of the importance of this bond for the biological properties of organotin compounds (Barbieri *et al.*, 1989), have together spurred the study of thiolates (Casas *et al.*, 1997, 1999). We present here the crystal structure of the title compound, (I).



(I)

The structure of (I) is composed of a discrete monomeric molecule (Fig. 1), in which the Sn atom exists in a skew–trapezoidal planar geometry. The equatorial plane is defined by four S atoms from two chelating thiocarboxylate ligands. The Sn atom is 0.016 (1) Å out of the least-squares plane formed by the four S atoms, and on the same side as atom C28. The two remaining octahedral sites are occupied by two methyl groups which lie over the weaker Sn—S bonds and define a C27—Sn—C28 angle of 135.64 (7)°. The angle between the Sn—C27 bond and the least-squares plane is 69.92 (5)°, and that between the Sn—C28 bond and the least-squares plane is 65.71 (5)°.

The degrees of asymmetry in the modes of coordination of each thiocarboxylate ligand, while comparable, are not equivalent. The first ligand forms Sn—S1 and Sn—S2 bond distances of 2.5190 (4) and 2.9779 (5) Å, respectively, while the other forms Sn—S3 and Sn—S4 bonds of 2.5240 (4) and 2.9715 (4) Å, respectively; the shorter bond lengths are close to the sum of the covalent radii of Sn and S and the longer distances are significantly less than the sum of their van der Waals radii (4.0 Å) (Ma *et al.*, 2003).

The C—S bond distances [S1—C13 = 1.7548 (16) Å and S3—C26 = 1.7496 (16) Å] for the S atoms bound strongly to the Sn centre are longer than the C—S bonds [S2—C13 =

1.6902 (16) Å and S4—C26 = 1.6990 (15) Å] involving the S atoms forming the weaker bonds to the Sn atom. The geometry of complex (I) is similar to those usually observed for $R_2Sn(S_2CNR'_2)$ compounds (Tiekink, 1992, 1993).

Experimental

To a solution of 4-benzylpiperidine-1-carbodithioic acid (0.5 g, 1.99 mmol) in dry methanol (40 ml), a solution of dimethyltin(IV) chloride (0.218 g, 0.995 mmol) in methanol (40 ml) was added dropwise and the mixture was stirred vigorously for 3 h. The resulting white solid was separated off and the filtrate was allowed to evaporate to obtain white crystals.

Crystal data

$[Sn(CH_3)_2(C_{13}H_{16}NS_2)_2]$
$M_r = 649.60$
Triclinic, $P\bar{1}$
$a = 9.8937$ (6) Å
$b = 11.4658$ (7) Å
$c = 14.2179$ (8) Å
$\alpha = 99.811$ (1)°
$\beta = 108.859$ (1)°
$\gamma = 96.158$ (1)°
$V = 1480.99$ (15) Å^3
$Z = 2$
$D_x = 1.457$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.16$ mm^{-1}
$T = 100$ (1) K
Block, colourless
0.49 × 0.43 × 0.41 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
$T_{min} = 0.540$, $T_{max} = 0.620$
11332 measured reflections
6972 independent reflections
6620 reflections with $I > 2\sigma(I)$
$R_{int} = 0.013$
$\theta_{max} = 28.3°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.020$
$wR(F^2) = 0.051$
$S = 1.04$
6972 reflections
468 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0252P)^2 + 0.4409P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.53$ e Å^{-3}
$\Delta\rho_{min} = -0.43$ e Å^{-3}

All H atoms were located in a difference Fourier map and refined isotropically.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT-Plus* (Bruker, 2000); data reduction: *XPREP* (Bruker, 2000); program(s) used to solve structure: *DIRDIF99* (Beurskens *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

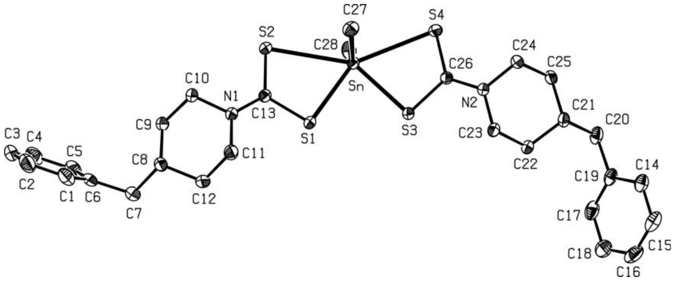


Figure 1
A perspective drawing of the title compound. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

Zia-ur-Rehman and NM are grateful to the Higher Education Commission of Pakistan for financial support.

References

Barbieri, R., Silvestri, A., Lo Giudice, M., Ruisi, R. & Musmeci, M. T. (1989). *J. Chem. Soc. Dalton Trans.* pp. 519–525.
Beurskens, P. T., Beurskens, G., de Gelder, R., García-Granda, S., Israel, R., Gould, R. O. & Smits, J. M. M. (1999). *The DIRDIF99 Program System*. Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.
Bruker, (2000). *SMART* (Version 5.624), *SAINT-Plus* (Version 6.02a) and *XPREP* (Version 5.1 for Windows NT). Bruker AXS Inc., Madison, Wisconsin, USA.
Casas, J. S., Castineiras, A., Martinez, E. G., Gonzalez, A. S., Sanchez, A. & Sordo, J. (1997). *Polyhedron*, **16**, 795–800.
Casas, J. S., Castineiras, A., Martinez, E. G., Rodriguez, P. R., Russo, U., Sanchez, A., Gonzalez, A. S. & Sord, J. (1999). *J. Appl. Organomet. Chem.* **13**, 69–77.
Ma, C., Zhang, J. & Zhang, R. (2003). *Can. J. Chem.* **81**, 1070–1075.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2001). *SADABS*. Version 2.03. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Tarassoli, A., Asadi, A. & Hitchcock, P. B. (2006). *J. Organomet. Chem.* **691**, 1631–1636.
Tiekink, E. R. T. (1992). *Main Group. Met. Chem.* **15**, 161–186.
Tiekink, E. R. T. (1993). *Main Group. Met. Chem.* **16**, 129–130.

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