Lai-Jin Tian,* Wu-Tao Mao, Yu-Xi Sun and Xi-Cheng Liu

Department of Chemistry, Qufu Normal University, Qufu 273165, People's Republic of China

Correspondence e-mail: laijintian@163.com

Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.009 Å
R factor = 0.042
wR factor = 0.094
Data-to-parameter ratio = 19.9

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

Bromo(N,N-dimethyldithiocarbamato-κ²S,S')-diphenyltin(IV)

The Sn atom in the title compound, $[SnBr(C_6H_5)_2(C_3H_6NS_2)]$, adopts a distorted $SnBrC_2S_2$ trigonal–bipyramidal environment.

Comment

The structures of some chloro-diorgano-tin N,N-dialkyl-dithiocarbamates, $R_2Sn(S_2CNR'_2)Cl$ have shown that the Sn atom exists in a distorted $SnClC_2S_2$ trigonal–bipyramidal environment (Tiekink, 1992; Tiekink *et al.*, 1999; Yin *et al.*, 2001). In the bromine-containing title compound, (I), the Sn atom is also five-coordinate and possesses a distorted trigonal–bipyramidal geometry (Fig. 1 and Table 1). The equatorial plane is defined by two C atoms (C4 and C10) of the phenyl groups and the more tightly held S1 atom from the asymmetrically chelating dithiocarbamate ligand, and the axial positions are occupied by the less tightly held atom S2 and atom Br1. The tin atom lies 0.1475 (5) Å out of the SC_2 trigonal plane in the direction of the Br atom. The dithiocarbamate ligand chelates in an asymmetric manner to the Sn atom with a difference in the Sn—S bond lengths of some 0.214 Å; the average Sn—S bond distance is 2.5605 (16) Å, which is similar to those found in $Ph_2Sn(S_2CNEt_2)Cl$ (Dakternieks *et al.*, 1992), $Ph_2Sn(S_2CNCy_2)Cl$ (Basu Baul & Tiekink, 1993), $Ph_2Sn(S_2CNEtCy)Cl$ (Hall & Tiekink, 1995) and $(C_6H_5-CH_2)_2Sn(S_2CNMe_2)Cl$ (Yin *et al.*, 2001). The thiocarbamate C—S bond lengths suggest that the bonding of this group is localized.

(I)
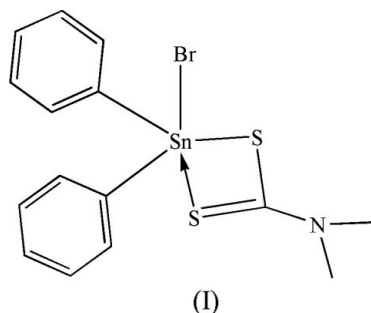
Experimental

A solution of $NaS_2CNMe_2\cdot2H_2O$ (0.36 g, 2 mmol) in 30 ml of hot acetone was added dropwise to a solution of diphenyltin dibromide (0.87 g, 2 mmol) in 30 ml of the same solvent. The reaction mixture was stirred for about an hour under reflux, and then cooled to about 273 K. The NaBr formed was removed by filtration, and the solvent was evaporated under reduced pressure. The solid obtained was recrystallized from trichloromethane–hexane (1:1 v/v) and crystals of

metal-organic papers

(I) were obtained from dichloromethane–hexane (1:1 v/v) by slow evaporation at 298 K (yield 76.3%, m.p. 406–407 K). Analysis found: C 38.12, H 3.27, N 2.83%; calculated for $C_{15}H_{16}BrNS_2Sn$: C 38.09, H 3.41, N 2.96%.

Crystal data

[SnBr(C₆H₅)₂(C₃H₆NS₂)]
$M_r = 473.01$
Monoclinic, $C2/c$
$a = 17.560 (8)$ Å
$b = 9.133 (3)$ Å
$c = 21.838 (10)$ Å
$\beta = 91.772 (6)°$
$V = 3501 (3)$ Å³

$Z = 8$
$D_x = 1.795$ Mg m⁻³
Mo $K\alpha$ radiation
$\mu = 3.97$ mm⁻¹
$T = 295 (2)$ K
Block, colorless
$0.14 \times 0.10 \times 0.10$ mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2002)
$T_{min} = 0.606$, $T_{max} = 0.692$

9614 measured reflections
3634 independent reflections
2687 reflections with $I > 2\sigma(I)$
$R_{int} = 0.042$
$\theta_{max} = 26.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.094$
$S = 1.04$
3634 reflections
183 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.029P)^2 + 3.0728P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.61$ e Å⁻³
$\Delta\rho_{min} = -0.64$ e Å⁻³

Table 1
Selected geometric parameters (Å, °).

Sn1—C10	2.124 (5)	Sn1—S2	2.6674 (16)
Sn1—C4	2.132 (5)	C1—S1	1.744 (5)
Sn1—S1	2.4537 (15)	C1—S2	1.701 (6)
Sn1—Br1	2.6098 (11)		
C10—Sn1—C4	111.87 (19)	S1—Sn1—Br1	87.02 (5)
C10—Sn1—S1	113.58 (14)	C10—Sn1—S2	97.45 (15)
C4—Sn1—S1	133.21 (14)	C4—Sn1—S2	93.80 (14)
C10—Sn1—Br1	98.05 (15)	S1—Sn1—S2	70.05 (5)
C4—Sn1—Br1	97.21 (14)	Br1—Sn1—S2	156.05 (4)

H atoms were placed at calculated positions and were included in the refinement in the riding-model approximation, with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic H atoms, and C—H = 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms.

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHEL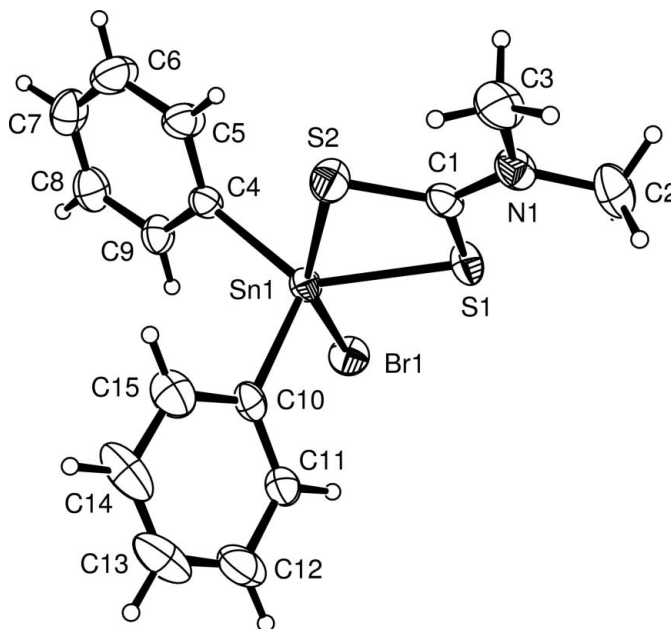XS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

Figure 1
The structure of (I), with displacement ellipsoids drawn at the 30% probability level (arbitrary spheres for the H atoms).

References

Basu Baul, T. S. & Tiekink, E. R. T. (1993). *Main Group Met. Chem.* **16**, 201–207.
Bruker (2002). *SADABS, SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Dakternieks, D., Zhu, H., Masi, D. & Mealli, C. (1992). *Inorg. Chem.* **31**, 3601–3606.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Hall, V. J. & Tiekink, E. R. T. (1995). *Main Group Met. Chem.* **18**, 217–223.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Tiekink, E. R. T. (1992). *Main Group Met. Chem.* **15**, 161–186.
Tiekink, E. R. T., Hall, V. J. & Buntine, M. A. (1999). *Z. Kristallogr.* **214**, 124–134.
Yin, H.-D., Wang, C.-H., Ma, C.-L., Wang, Y. & Zhang, R.-F. (2001). *Chin. J. Org. Chem.* **21**, 1117–1121.