Dibutylbis(3-methylsulfanyl-1,2,4-thiadiazole-5-thiolato)tin(IV)

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In the title complex, $[Sn(C_4H_9)_2(C_3H_3N_2S_3)_2]$, if weak $Sn\cdots N$ interactions are included, the central Sn^{IV} atom is situated in a skew-trapezoidal bipyramidal geometry, with the basal plane defined by two symmetrically chelating 3-methylmercapto-5-mercapto-1,2,4-thiadiazole ligands. The apical positions are occupied by two *n*-butyl groups. The complex shows a three-dimensional network structure assembled by intermolecular $S\cdots S$ and $C\cdots S$ non-bonded interactions.

Comment

From Fig. 1, it can be seen that, if weak $Sn\cdots N$ interactions are included, the central Sn^{IV} atom in the title compound, (I), is situated in a skew-trapezoidal bipyramidal geometry, with the basal plane defined by two symmetrically chelating 3-methylmercapto-5-mercapto-1,2,4-thiadiazole ligands. The apical positions are occupied by two *n*-butyl groups. The coordination geometry of the Sn atom can also be described as distorted *trans*-octahedral, with atoms N1, N3, S2 and S5 occupying the equatorial positions, and atoms C7 and C11 occupying the axial positions. The molecular structure consists of a monomer with a hexa-coordinated Sn atom surrounded by two S atoms and two N atoms of the ligand, and two *n*-butyl groups.

(I)
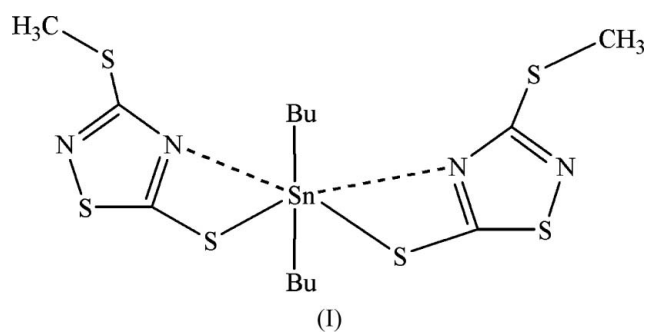
The bite angles S2—Sn1—N1 and S5—Sn1—N3 of (I) [60.95 (7) and 61.22 (8)°, respectively] can be reconciled with a skew-trapezoidal bipyramidal geometry, although this geometry can also be considered as distorted *trans* octahedral. The structure of (I) is close to those we have reported previously for diorganotin derivatives with 2-mercapto-4-methylpyrimidine (Ma *et al.*, 2005). There is a good correspondence in their structural parameters: the Sn—S distances lie in the range 2.477–2.526 Å and the Sn—N distances in the range 2.650–2.933 Å.

From Fig. 2 it can be seen that the complex forms a three-dimensional network structure assembled by intermolecular $S2\cdots S2^i$ and $S4\cdots C10^{ii}$ weak non-bonded interactions

between adjacent 3-methylmercapto-5-mercapto-1,2,4-thia-diazole groups [symmetry code: (ii) $1 + x$, $y - 1$, z]. The S4···C10^{ii} distance is 3.414 (6) Å. The S2···S2^{i} distance of 3.324 (3) Å is significantly shorter than the sum of the van der Waals radii of two S atoms (3.7 Å; Barone *et al.*, 2001).

Experimental

3-Methylmercapto-5-mercapto-1,2,4-thiadiazole (2 mmol) was added to a solution of sodium ethoxide (2 mmol) in ethanol (20 ml) and the mixture was stirred for 30 min. Bu$_2$SnCl$_2$ (1 mmol) was then added to the mixture and the reaction was continued for 12 h at 318 K. After cooling to room temperature, the mixture was filtered. The solvent of the filtrate was gradually removed by evaporation under vacuum until a solid product was obtained. This solid was then recrystallized from diethyl ether–dichloromethane (1:1) and colourless crystals of (I) suitable for X-ray diffraction were obtained (m.p. 354 K). Analysis, calculated for C$_{14}$H$_{14}$N$_4$S$_6$Sn: C 30.06, H 4.32, N 10.01%; found: C 30.13, H 4.29, N 9.96%.

Crystal data

[Sn(C$_4$H$_9$)$_2$(C$_3$H$_3$N$_2$S$_3$)$_2$]	$Z = 2$
$M_r = 559.42$	$D_x = 1.598$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 9.386$ (4) Å	Cell parameters from 4037
$b = 10.059$ (5) Å	reflections
$c = 13.561$ (6) Å	$\theta = 2.2$–$25.0°$
$\alpha = 77.549$ (6)°	$\mu = 1.64$ mm^{-1}
$\beta = 82.877$ (6)°	$T = 298$ (2) K
$\gamma = 68.646$ (6)°	Block, colourless
$V = 1162.9$ (10) Å^3	$0.42 \times 0.29 \times 0.25$ mm

Data collection

Siemens SMART CCD area-detector diffractometer	4037 independent reflections
	3253 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.019$
Absorption correction: multi-scan	$\theta_{max} = 25.0°$
(*SADABS*; Sheldrick, 1996)	$h = -10 \rightarrow 11$
$T_{min} = 0.545$, $T_{max} = 0.684$	$k = -11 \rightarrow 11$
6113 measured reflections	$l = -14 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0337P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.034$	$+ 0.7694P]$
$wR(F^2) = 0.082$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
4037 reflections	$\Delta\rho_{max} = 0.69$ e Å^{-3}
230 parameters	$\Delta\rho_{min} = -0.41$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Sn1—S2	2.5060 (14)	Sn1—N1	2.762 (3)
Sn1—S5	2.5182 (15)	Sn1—N3	2.735 (3)
C7—Sn1—C11	133.81 (17)	S2—Sn1—S5	93.08 (5)
C7—Sn1—S2	104.50 (12)	C7—Sn1—N1	81.94 (13)
C11—Sn1—S2	105.46 (13)	C11—Sn1—N1	82.83 (15)
C7—Sn1—S5	104.84 (12)	S2—Sn1—N1	60.96 (7)
C11—Sn1—S5	107.76 (14)	S5—Sn1—N1	153.98 (7)

All H atoms were positioned geometrically and treated as riding on their parent atoms, with methylene C—H distances of 0.97 Å, and methyl and thiadiazole C—H distances of 0.96 Å. The U_{iso}(H) values were set at $1.5U_{eq}$(C) for methyl H atoms and at $1.2U_{eq}$(C) for the other H atoms.
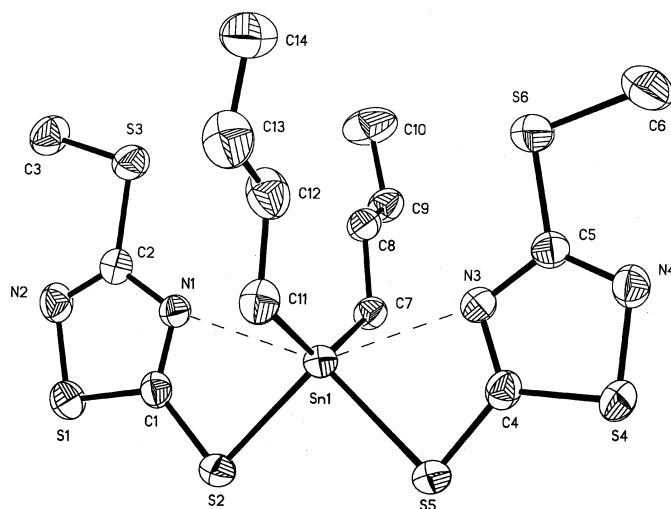
Figure 1
The structure of the title complex, showing 30% probability displacement ellipsoids. H atoms have been omitted.
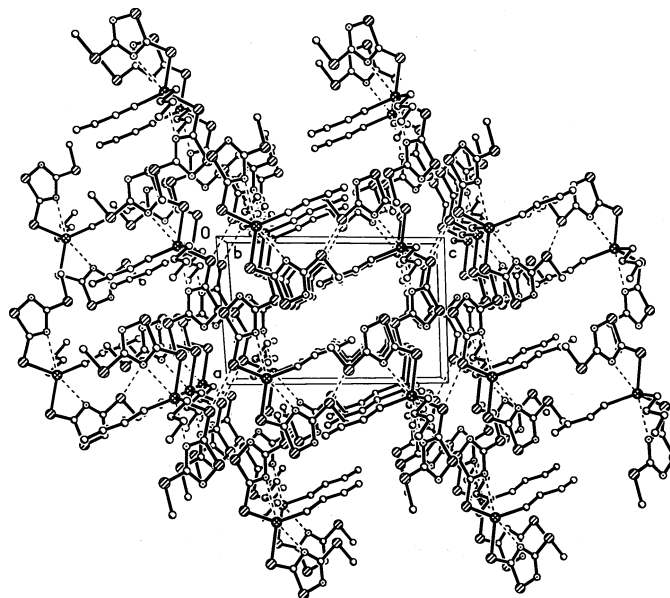
Figure 2
The crystal packing of the title complex, showing the weak S···S and C···S interactions (dashed lines).

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*.

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