

Note

# Synthesis, Characterization and Crystal Structure of $(\text{PhCH}_2)_2\text{Sn}(\text{S}_2\text{CNEt}_2)_2$ and $(\text{PhCH}_2)_2\text{Sn}(\text{S}_2\text{CNC}_4\text{H}_8)_2$

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Two new dibenzyltin bisdithiocarbamates  $(\text{PhCH}_2)_2\text{Sn}(\text{S}_2\text{CNEt}_2)_2$  (**1**) and  $(\text{PhCH}_2)_2\text{Sn}(\text{S}_2\text{CNC}_4\text{H}_8)_2$  (**2**) were synthesized by the reaction of dibenzyltin dichloride with dithiocarbamates and characterized by elemental analysis, IR,  $^1\text{H}$  NMR and MS spectra. The crystal structures were determined by X-ray single crystal diffraction analysis. In both complexes, the tin atom is six-coordinated in a distorted octahedral configuration. In the crystals of **1**, the molecular packing in unit cell reveals that the two adjacent molecules are symmetrically linked to each other in dimers by two  $\text{Sn}\cdots\text{S}$  interactions of 0.3816 nm. In the crystals of **2**, the molecules are packed in the unit cell in one-dimensional chain structure linked by weaker intermolecular  $\text{S}\cdots\text{S}$  contacts.

**Keywords** dibenzyltin derivative, dithiocarbamate, crystal structure,  $\text{Sn}\cdots\text{S}$ ,  $\text{S}\cdots\text{S}$  interaction

## Introduction

Organotin(IV) complexes with carboxylates were widely used as biocides, fungicides and in industry as homogeneous catalysts.<sup>1-5</sup> More recently, pharmaceutical properties of alkyltin(IV) complexes with *N,N*-dialkyl dithiocarbamates have been investigated because of their antitumour activity.<sup>6-8</sup> Crystallographic studies have revealed that the coordination at the tin atom depends not only on stereochemistry of R group, but also on whether the 1,1-dithiolates behave as monodentate or bidentate ligands and whether the complexes are monomeric or oligomeric. In order to explore the relationships between biological activity and structure, we report the synthesis and the crystal structures of two new dibenzyltin(IV) complexes with dithiocarbamate.

## Experimental

### General procedure

The reaction was carried out under nitrogen atmosphere using standard Schlenk techniques. The solvent was dried over phosphorus(V) oxide prior to use. IR spectra were

recorded with a Nicolet-460 spectrophotometer, and samples were as KBr discs.  $^1\text{H}$  NMR spectra were recorded on a Jeol-FX-90Q NMR spectrometer, and chemical shifts are given relative to  $\text{Me}_4\text{Si}$  in  $\text{CDCl}_3$  solvent. Elemental analyses were performed in a Perkin-Elmer II elemental analyzer, and tin was estimated as  $\text{SnO}_2$ . The mass spectra were recorded on an HP-5988A spectrometer operating at 70 eV.

### Synthesis of dibenzyltin(IV) dithiocarbamate

$(\text{PhCH}_2)_2\text{Sn}(\text{S}_2\text{CNEt}_2)_2$  (**1**)    Anhydrous sodium salt of *N,N*-dialkyl diethyldithiocarbamate (376 mg, 2.2 mmol) were added to 30 mL of dichloromethane solution of  $(\text{PhCH}_2)_2\text{SnCl}_2$  (372 mg, 1.0 mmol). The reaction mixture was stirred at 30 °C for 10 h. The precipitated sodium chloride was removed by filtration and the filtrate was concentrated to about 5 mL under reduced pressure. Ethyl ether (5 mL) and hexane (5 mL) were added to this solution, immediately a precipitate was formed. The products were recrystallized from dichloromethane-ethyl ether solution to give colorless crystals 544 mg, yield 91%, m.p. 105–108 °C; UV-vis ( $\text{CHCl}_3$ )  $\lambda_{\text{max}}$ : 223, 255, 276 nm;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 90 MHz)  $\delta$ : 1.13 (t,  $J = 7.9$  Hz, 12H,  $\text{CH}_3$ ), 2.91 (t,  $J_{\text{Sn-H}} = 88.1$  Hz, 4H,  $\text{PhCH}_2\text{Sn}$ ), 3.76 (q,  $J = 7.9$  Hz, 8H,  $\text{NCH}_2$ ), 7.09–7.17 (m, 10H,  $\text{PhH}$ ); IR (KBr)  $\nu$ : 3022 (w,  $\text{Ph-H}$ ), 2974, 2860 (m,  $\text{C-H}$ ), 1498 (s,  $\text{C-N}$ ), 1115, 987 (s,  $\text{CS}_2$ ), 569 (s,  $\text{Sn-C}$ ), 446 (s,  $\text{Sn-S}$ )  $\text{cm}^{-1}$ ; MS (70 eV)  $m/z$  (%): 507 ( $[\text{M} - \text{Bz}]^+$ , 10), 450 ( $[\text{M} - \text{S}_2\text{CN}(\text{C}_2\text{H}_5)_2]^+$ , 15), 416 ( $[\text{M} - 2\text{Bz}]^+$ , 62), 268 ( $[\text{SnS}_2\text{CN}(\text{C}_2\text{H}_5)_2]^+$ , 31), 116 ( $[\text{SCN}(\text{C}_2\text{H}_5)]^+$ , 18), 91 ( $\text{PhCH}_2^+$ , 100). Anal. calcd for  $\text{C}_{24}\text{H}_{34}\text{N}_2\text{S}_4\text{Sn}$ : C 48.25, H 5.74, N 4.69, Sn 19.87; found C 48.12, H 5.54, N 4.62, Sn 19.98.

$(\text{PhCH}_2)_2\text{Sn}(\text{S}_2\text{CNC}_4\text{H}_8)_2$  (**2**)    This complex was similarly prepared as **1** with anhydrous sodium salt of tetrahydropyrroldithiocarbamate (372 mg, 2.2 mmol) and  $(\text{PhCH}_2)_2\text{SnCl}_2$  (372 mg, 1.0 mmol) to give colorless crystals

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522 mg, yield 88%, m.p. 134–136 °C; UV-vis ( $\text{CHCl}_3$ )  $\lambda_{\text{max}}$ : 221, 265, 280 nm;  $^1\text{H NMR}$  ( $\text{CDCl}_3$ , 90 MHz)  $\delta$ : 1.75 (t,  $J = 8.0$  Hz, 8H,  $\text{CH}_2$ ), 3.80 (t,  $J = 8.0$  Hz, 8H,  $\text{NCH}_2$ ), 2.90 (t,  $J_{\text{Sn-H}} = 84.9$  Hz, 4H,  $\text{PhCH}_2\text{Sn}$ ), 7.05–7.24 (m, 10H,  $\text{PhH}$ ); IR (KBr)  $\nu$ : 3024 (w,  $\text{Ph-H}$ ), 2966, 2855 (m,  $\text{C-H}$ ), 1480 (s,  $\text{C-N}$ ), 1125, 994 (s,  $\text{CS}_2$ ), 563 (s,  $\text{Sn-C}$ ), 445 (s,  $\text{Sn-S}$ )  $\text{cm}^{-1}$ ; MS (70 eV)  $m/z$  (%): 503 ( $[\text{M} - \text{Bz}]^+$ , 20), 448 ( $[\text{M} - \text{S}_2\text{CN}(\text{CH}_2\text{CH}_2)_2]^+$ , 10), 412 ( $[\text{M} - 2\text{Bz}]^+$ , 58), 266 ( $[\text{SnS}_2\text{CN}(\text{CH}_2\text{CH}_2)_2]^+$ , 27), 114 ( $[\text{SCN}(\text{CH}_2\text{CH}_2)_2]^+$ , 13), 91 ( $\text{PhCH}_2^+$ , 100). Anal. calcd for  $\text{C}_{24}\text{H}_{30}\text{N}_2\text{S}_4\text{Sn}$ : C 48.57, H 5.10, N 4.72, Sn 20.00; found C 48.23, H 4.94, N 4.70, Sn 20.31.

### Crystallographic measurements

Two colorless block crystals with dimensions 0.40 mm  $\times$  0.40 mm  $\times$  0.25 mm (**1**) and 0.60 mm  $\times$  0.50 mm  $\times$  0.40 mm (**2**) were mounted in a glass fiber, respectively. All measurements were made on a Bruker Smart-1000 CCD diffractometer with graphite monochromated  $\text{Mo K}\alpha$  ( $\lambda = 0.071073$  nm) radiation. The structures were solved by direct method and difference Fourier map using SHELXL-97 program, and refined by full-matrix least-squares on  $F^2$ . All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were located at calculated positions and refined isotropically.

## Results and discussion

### IR, UV, $^1\text{H NMR}$ and MS spectra

The assignment of IR bands of complexes **1** and **2** has been made by comparison with the IR spectra of related dibenzyltin(IV) complexes with dithiocarbamate ligands, sodium dithiocarbamate and dibenzyltin dichloride. The new absorption bands appear at  $446 \text{ cm}^{-1}$  and  $445 \text{ cm}^{-1}$ , which are the characteristic vibrations of  $\text{Sn-S}$  bond.<sup>7</sup>

One obvious feature of the IR spectra is the similarity of the stretching bands arising from the dithiocarbamate ligand. The relatively high value ( $1498, 1480 \text{ cm}^{-1}$ ) for  $\nu(\text{C-N})$  is similar to that reported for analogous tin complexes.<sup>7,8</sup> This suggests that the dithiocarbamate ligands of the two complexes be linked to Sn atom in a bidentate fashion.

In IR spectra, the important bands arising from  $\nu(\text{CS}_2)_{\text{asym}}$  and  $\nu(\text{CS}_2)_{\text{sym}}$  appear at  $1115$  and  $1125 \text{ cm}^{-1}$ , and  $987 \text{ cm}^{-1}$  and  $994 \text{ cm}^{-1}$ , respectively. The  $\Delta\nu$  values [ $\nu(\text{CS}_2)_{\text{asym}} - \nu(\text{CS}_2)_{\text{sym}}$ ] are  $128$  and  $131 \text{ cm}^{-1}$ , which are much smaller than the  $\Delta\nu^*$  for the  $\text{R}_2\text{NCS}_2\text{R}'$ ,<sup>9</sup> but it is larger than the  $\Delta\nu'$  for the corresponding sodium dithiocarbamate.<sup>10</sup> This shows that the dithiocarbamate groups in the complexes are coordinated to tin atom in an anisobidentate fashion.<sup>11,12</sup>

The  $^1\text{H NMR}$  spectra of complexes **1** and **2** show that the

chemical shifts of the protons on the benzyl group exhibit two signals at about  $\delta 7.05\text{--}7.24$  as multiplet, and  $\delta 2.91$  and  $\delta 2.90$  as triplet which is caused by the tin ( $^{119}\text{Sn}$ )-hydrogen couplings, the spin-spin coupling constant  $J_{\text{Sn-H}} = 88.1$  and  $84.9$  Hz. The proton signals of methylene connected directly with nitrogen appear at  $\delta 3.76$  and  $\delta 3.80$ . They shift downfields  $\delta 0.4$  and  $\delta 0.6$  as compared with those of the salts of appropriate acid. It is shown that the coordinated dithiocarbamate group is more electronegative than that of non-coordinated dithiocarbamate group.<sup>10</sup>

From the UV spectra of complexes **1** and **2**, it can be seen that the band at  $\lambda_{\text{max}} = 223$  and  $221$  nm which belongs to strong absorption, is a K band due to  $\pi\text{-}\pi^*$  transition of dithiocarbamate  $\text{N-C-S}$ . The band at  $\lambda_{\text{max}} = 255$  and  $265$  nm which belongs to medium absorption due to  $\pi\text{-}\pi^*$  transition of  $\text{S-C-S}$  radical is a hypsochromic shift about  $23$  and  $28$  nm as compared with that of the appropriate salts of dithiocarbamate acid.<sup>10</sup> The band at  $\lambda_{\text{max}} = 276$  and  $280$  nm which belongs to a weak absorption is attributed to electron-transfer transition  $n\text{-}\pi^*$  in  $\text{CS}_2$  radical, which exhibits a hypsochromic shift by  $25$  and  $33$  nm, as compared with the corresponding band of salts of dithiocarbamate acid.

In the mass spectra of complexes **1** and **2**, the molecular ions were not detected. The  $[\text{M} - \text{Bz}]^+$ ,  $[\text{M} - 2\text{Bz}]^+$ ,  $[\text{SnS}_2\text{CN}(\text{C}_2\text{H}_5)_2]^+$  and  $[\text{SnS}_2\text{CN}(\text{CH}_2\text{CH}_2)_2]^+$  ion peaks were the main tin-containing fragments. The most abundant ions were  $\text{PhCH}_2^+$  ( $m/z = 91$ ). Ions whose mass were larger than parent species were not detected, nor were there fragments containing more than one tin atoms. This indicates that inter-molecular  $\text{Sn}\cdots\text{S}$  or  $\text{S}\cdots\text{S}$  bond has been broken up, and the complexes take only monomeric structure in gaseous state.

### Molecular structure of complexes **1** and **2**

#### Complex **1**

Crystal data for complex **1** are listed in Table 1. The molecular structure and molecular packing in the unit cell are shown in Fig. 1 and Fig. 2. Table 2 gives the atomic coordinates and equivalent isotropic thermal parameters. The selected bond lengths and angles are listed in Table 4.

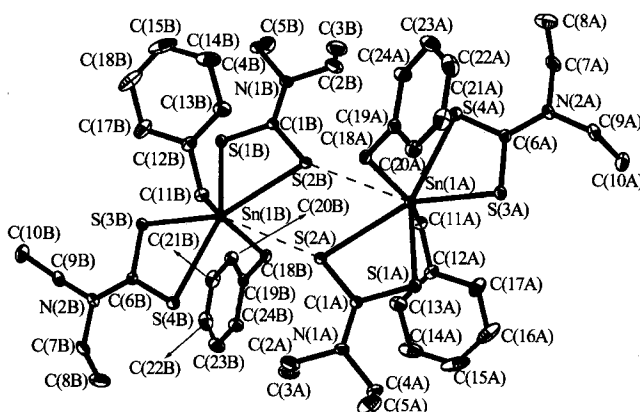


Fig. 1 Molecular structure of complex **1**.

**Table 1** Crystallographic data of complexes **1** and **2**

	<b>1</b>	<b>2</b>
Molecular formula	C <sub>24</sub> H <sub>34</sub> N <sub>2</sub> S <sub>4</sub> Sn	C <sub>24</sub> H <sub>30</sub> N <sub>2</sub> S <sub>4</sub> Sn
Formular weigh	597.46	593.43
Crystal system	Monoclinic	Orthorhombic
Space group	<i>P2(1)/c</i>	<i>Pccn</i>
Unit cell dimensions	<i>a</i> = 1.6052(2) nm <i>b</i> = 1.2217(2) nm <i>c</i> = 1.5661(2) nm $\beta$ = 113.474(2)°	<i>a</i> = 2.1653(3) nm <i>b</i> = 0.9938(7) nm <i>c</i> = 1.1976(9) nm $\beta$ = 90°
Volume (nm <sup>3</sup> )	2.8171(7)	2.5774(6)
<i>Z</i>	4	4
<i>D<sub>c</sub></i> (g/cm <sup>3</sup> )	1.409	1.529
<i>F</i> (000)	1224	1208
Scan range $\theta$ (°)	2.17–25.03	1.88–25.02
Total/unique/ <i>R<sub>int</sub></i>	14260/4983/0.0278	12543/2278/0.0849
$\mu$ (mm <sup>-1</sup> )	1.217	1.330
<i>R</i> <sub>1</sub> / <i>wR</i> <sub>2</sub>	0.0313/0.0708	0.0338/0.0849
Largest difference	463/–757	572/–1328
Peak and hole (e/nm <sup>3</sup> )		

**Table 2** Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters ( $\times 10^5$  nm<sup>2</sup>) of complex **1**

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U<sub>eq</sub></i>	Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U<sub>eq</sub></i>
Sn(1)	6983(1)	–150(1)	5955(1)	42(1)	C(10)	10468(4)	–2016(6)	8443(4)	86(2)
N(1)	5948(2)	3142(2)	6274(2)	62(1)	C(11)	6489(3)	–788(4)	6963(3)	56(1)
N(2)	9390(2)	–2378(3)	6842(2)	57(1)	C(12)	6570(3)	–13(3)	7708(3)	57(1)
S(1)	7325(1)	1825(1)	6487(1)	49(1)	C(13)	5853(5)	615(4)	7689(4)	81(1)
S(2)	5408(1)	1290(1)	5332(1)	52(1)	C(14)	5974(8)	1356(5)	8413(6)	124(3)
S(3)	8676(1)	–417(1)	6828(1)	55(1)	C(15)	6787(10)	1449(7)	9132(6)	156(5)
S(4)	7599(1)	–2365(1)	5901(1)	60(1)	C(16)	7494(7)	832(9)	9162(5)	146(3)
C(1)	6184(2)	2182(3)	6048(2)	43(1)	C(17)	7399(5)	100(6)	8461(4)	97(2)
C(2)	4994(3)	3555(5)	5820(5)	98(2)	C(18)	6737(3)	–65(4)	4489(3)	54(1)
C(3)	4470(6)	3178(6)	6316(7)	124(2)	C(19)	7551(2)	–226(3)	4269(2)	49(1)
C(4)	6600(3)	3888(4)	6965(3)	75(1)	C(21)	8168(3)	611(5)	4404(3)	68(1)
C(5)	6976(6)	4721(6)	6529(5)	121(2)	C(21)	8915(3)	488(6)	4190(4)	87(2)
C(6)	8622(2)	–1801(3)	5644(2)	46(1)	C(22)	9048(4)	–481(7)	3838(4)	98(2)
C(7)	9402(4)	–3534(4)	6584(4)	76(1)	C(23)	8455(4)	–1328(6)	3693(4)	88(2)
C(8)	9607(8)	–3655(7)	5732(6)	121(3)	C(24)	7704(3)	–1200(4)	3908(3)	70(1)
C(9)	10217(3)	–1910(4)	7435(3)	64(1)					

In complex **1**, the tin atom is six-coordinated [Sn(1)—S(1) 0.2539(5), Sn(1)—S(2) 0.2910(8), Sn(1)—S(3) 0.2531(1), Sn(1)—S(4) 0.2893(9), Sn(1)—C(11) 0.2175(4), Sn(1)—C(18) 0.2171(4) nm], with a distorted octahedron configuration. Two tin atoms are so close that the distance between Sn(1) and S(2) atom of the other (centrosymmetrically-related) molecule is only 0.3816 nm (the sum of the Van Der Waals radii for Sn and S is 0.40 nm), which suggests that there is a very weak interaction between them.<sup>13</sup> The dimeric structure contrasts with the monomeric structure of <sup>1</sup>Bu<sub>2</sub>Sn(S<sub>2</sub>CNEt<sub>2</sub>)<sub>2</sub>,<sup>14</sup> (PhCH<sub>2</sub>)<sub>2</sub>Sn-

(S<sub>2</sub>CNC<sub>5</sub>H<sub>10</sub>)<sub>2</sub><sup>15</sup> and (PhCH<sub>2</sub>)<sub>2</sub>Sn(S<sub>2</sub>CNC<sub>4</sub>H<sub>8</sub>O)<sub>2</sub>,<sup>16</sup> for which no intermolecular contacts of <0.4 nm were found.

The geometry is loosely based on an octahedron, with atoms S(1), S(2), S(3) and S(4), occupying equatorial positions. As an indication the sum of the equatorial angles (359.8°) at the tin atom by the four coordinated sulfur atoms [S(1)-Sn(1)-S(3) 84.2(4)°, S(4)-Sn(1)-S(3) 65.7(2)°, S(4)-Sn(1)-S(2) 144.7(7)°, S(1)-Sn(1)-S(2) 65.2(8)°] deviates at most by 0.2° from the 360°, so the atoms S(1), S(2), S(3), S(4) and Sn(1) are almost in the same plane.

## Complex 2

Crystal data for complex 2 are listed in Table 1. The molecular structure and molecular packing in the unit cell are

shown in Fig. 3 and Fig. 4. Table 3 gives the atomic coordinates and equivalent isotropic thermal parameters. The selected bond lengths and angles are listed in Table 5.

**Table 3** Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters ( $\times 10^5 \text{ nm}^2$ ) of complex 2

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}$	Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}$
Sn(1)	2500	2500	7672(1)	42(1)	C(5)	3503(3)	7499(5)	7271(4)	60(1)
N(1)	3256(2)	6394(3)	6597(2)	47(1)	C(6)	3427(2)	1977(6)	8243(4)	55(1)
S(1)	2798(1)	5219(1)	8392(1)	51(1)	C(7)	3927(2)	2761(4)	7705(4)	55(1)
S(2)	2707(1)	4113(1)	6092(1)	49(1)	C(8)	4218(2)	3820(6)	8234(5)	72(1)
C(1)	2949(2)	5359(4)	7007(3)	42(1)	C(9)	4675(3)	4563(7)	7674(8)	97(2)
C(2)	3440(3)	6539(6)	5419(3)	61(1)	C(10)	4838(3)	4214(8)	6610(8)	103(2)
C(3)	3911(3)	7644(6)	5452(5)	76(2)	C(11)	4557(3)	3173(8)	6097(7)	92(20)
C(4)	3739(3)	8481(6)	6426(5)	82(2)	C(12)	4108(2)	2464(5)	6622(5)	66(1)

**Table 4** Selected bond distances (nm) and angles ( $^\circ$ ) of complex 1

Sn(1)—C(11)	0.2175(4)	Sn(1)—S(4)	0.2893(9)	N(2)—C(7)	0.1472(6)
Sn(1)—C(18)	0.2171(4)	N(1)—C(1)	0.1322(4)	N(2)—C(9)	0.1464(5)
Sn(1)—S(1)	0.2539(5)	N(1)—C(2)	0.1496(6)	S(1)—C(1)	0.1737(3)
Sn(1)—S(2)	0.2910(8)	N(1)—C(4)	0.1481(5)	S(2)—C(1)	0.1699(3)
Sn(1)—S(3)	0.2531(1)	N(2)—C(6)	0.1332(4)	S(3)—C(6)	0.1741(4)
S(4)—C(6)	0.1693(3)	C(2)—C(3)	0.1429(9)	Sn(1)⋯S(2A)	0.3816
C(18)-Sn(1)-C(11)	145.0(5)	S(3)-Sn(1)-S(4)	65.7(2)	C(1)-S(2)-Sn(1)	81.7(2)
C(18)-Sn(1)-S(3)	106.3(4)	S(1)-Sn(1)-S(4)	149.9(6)	C(6)-S(3)-Sn(1)	93.0(7)
C(11)-Sn(1)-S(3)	100.6(6)	C(18)-Sn(1)-S(2)	81.3(4)	C(6)-S(4)-Sn(1)	92.1(9)
C(18)-Sn(1)-S(1)	103.0(9)	C(11)-Sn(1)-S(2)	86.6(1)	C(12)-C(11)-Sn(1)	113.8(3)
C(11)-Sn(1)-S(1)	101.3(1)	S(3)-Sn(1)-S(2)	149.5(1)	C(19)-C(18)-Sn(1)	115.5(3)
S(3)-Sn(1)-S(1)	84.2(4)	S(1)-Sn(1)-S(2)	65.2(8)	S(1)-C(1)-S(2)	118.7(2)
C(18)-Sn(1)-S(4)	86.2(3)	S(4)-Sn(1)-S(2)	144.7(7)	S(4)-C(6)-S(3)	119.0(1)
C(11)-Sn(1)-S(4)	85.0(2)	C(1)-S(1)-Sn(1)	93.0(2)	C(1)-N(1)-C(2)	121.3(3)

**Table 5** Selected bond distances (nm) and angles ( $^\circ$ ) of complex 2

Sn(1)—S(1A)	0.2908(6)	S(1)—C(6A)	0.2183(4)	Sn(1)—S(2)	0.2521(4)
Sn(1)—S(1)	0.2908(6)	S(1)—C(6)	0.2183(4)	Sn(1)—S(2A)	0.2521(4)
N(1)—C(1)	0.1319(5)	N(1)—C(5)	0.1464(5)	N(1)—C(2)	0.1473(5)
S(1)—C(1)	0.1697(4)	S(2)—C(1)	0.1734(4)	S(1)⋯S(2A)	0.2972
S(1)⋯S(1A)	0.3330	C(2)—C(3)	0.1500(7)	C(3)—C(4)	0.1481(8)
S(1A)-Sn(1)-S(1)	145.5(3)	S(1A)-Sn(1)-S(2A)	65.93(3)	S(1)-Sn(1)-S(2)	65.9(3)
S(1A)-Sn(1)-S(2)	148.5(2)	S(1)-Sn(1)-S(2A)	148.5(2)	S(2)-Sn(1)-S(2A)	82.6(7)
S(1)-C(1)-S(2)	120.0(2)	N(1)-C(1)-S(2)	118.3(3)	N(1)-C(1)-S(1)	121.7(3)
C(1)-S(1)-Sn(1)	80.1(8)	C(1)-S(2)-Sn(1)	91.9(2)	C(7)-C(6)-Sn(1)	114.3(3)
C(1)-N(1)-C(5)	124.3(3)	S(1)-Sn(1)-C(6)	85.6(6)	S(2)-Sn(1)-C(6)	102.8(9)
S(1A)-Sn(1)-C(6A)	85.6(6)	S(2A)-Sn(1)-C(6)	104.3(1)	S(2A)-Sn(1)-C(6A)	102.8(9)
S(1A)-Sn(1)-C(6)	83.6(9)	S(2)-Sn(1)-C(6A)	104.3(1)	C(6A)-Sn(1)-C(6)	143.5(3)
S(1)-Sn(1)-C(6A)	83.6(9)	N(1)-C(2)-C(3)	103.3(4)	C(1)-N(1)-C(2)	124.7(3)

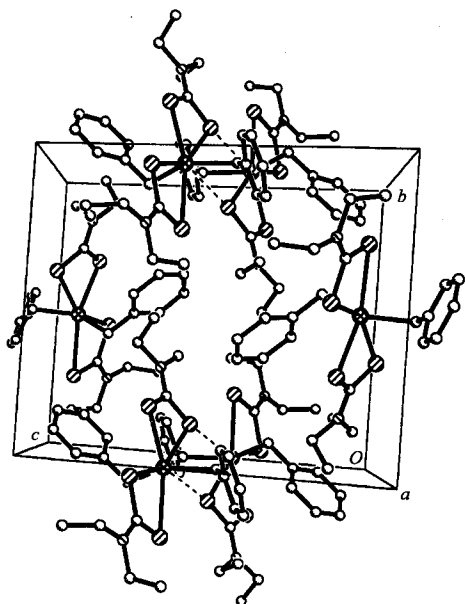


Fig. 2 Projection of the unit cell of complex 1.

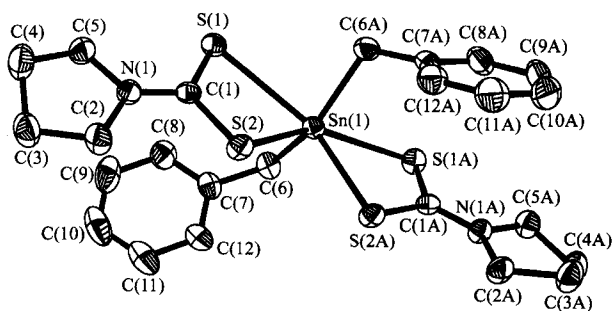


Fig. 3 Molecular structure of complex 2.

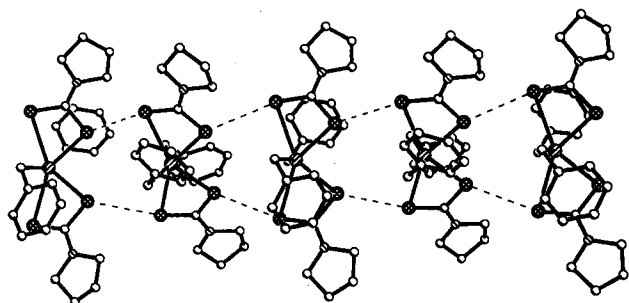


Fig. 4 Perspective view showing the one-dimensional S...S interaction network of complex 2.

In complex 2, the tin atom also is six-coordinated [Sn(1)—S(1) 0.2908(6), Sn(1)—S(1A) 0.2908(6), Sn(1)—S(2) 0.2521(1), Sn(1)—S(2A) 0.2521(1), Sn(1)—C(6) 0.2183(4), Sn(1)—C(6A) 0.2183(4) nm], with a distorted octahedron configuration. An additional feature is noted in this structure, however, which is the presence of a close intermolecular S...S interaction of 0.2972

and 0.3330 nm (the sum of the Van Der Waals radii for S and S is 0.370 nm), the structure is thus described as a one-dimensional chain structure linked by weaker intermolecular S...S contacts. This interaction is very weak so that the deviation from the common structural type is minimal. This phenomenon was not observed in the complexes  $t\text{Bu}_2\text{Sn}(\text{S}_2\text{CNEt}_2)_2$ ,<sup>13</sup>  $(\text{PhCH}_2)_2\text{Sn}(\text{S}_2\text{CNC}_5\text{H}_{10})_2$ ,<sup>14</sup> and  $(\text{PhCH}_2)_2\text{Sn}(\text{S}_2\text{CNC}_4\text{H}_8\text{O})_2$ .<sup>15</sup>

The geometry is loosely based on an octahedron for complex 2, with atoms S(1), S(2), S(1A) and S(2A), occupying equatorial positions. As an indication the sum of the equatorial angles ( $360.06^\circ$ ) at the tin atom by the four coordinated sulfur atoms [S(1)—Sn(1)—S(1A)  $145.53(4)^\circ$ , S(1A)—Sn(1)—S(2A)  $65.93(3)^\circ$ , S(2A)—Sn(1)—S(2)  $82.67(5)^\circ$ , S(1)—Sn(1)—S(2)  $65.93(3)^\circ$ ] deviates at most by  $-0.06^\circ$  from the  $360^\circ$ , so the atoms S(1), S(2), S(3), S(4), and Sn(1) are in the same plane.

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