

Yong-Xin Li, Ru-Fen Zhang and
Chun-Lin Ma*Department of Chemistry, Liaocheng University,
Liaocheng 252059, People's Republic of China

Correspondence e-mail: macl@lctu.edu.cn

Key indicators

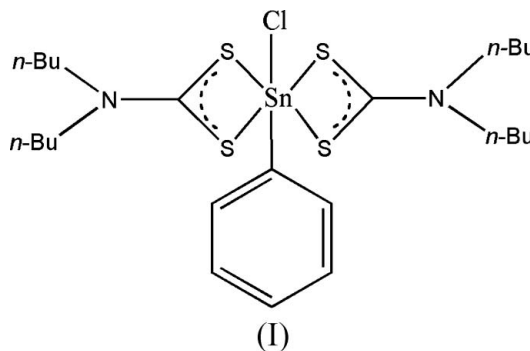
Single-crystal X-ray study
 $T = 273$ K
Mean $\sigma(\text{C}-\text{C}) = 0.020$ Å
 R factor = 0.075
 wR factor = 0.254
Data-to-parameter ratio = 19.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Chlorobis(*N,N*-di-*n*-butyldithiocarbamato)-
phenyltin(IV)The title complex, $[\text{Sn}(\text{C}_6\text{H}_5)(\text{C}_9\text{H}_{18}\text{NS}_2)_2\text{Cl}]$, contains two bidentate dithiocarbamate groups, resulting in a distorted SnS_4CCl octahedral geometry for the Sn atom.

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Comment

In recent years, dithiocarbamate complexes have been extensively studied (Burgmayer & Templeton, 1985). Such groups usually bond to the metal in a bidentate fashion (Mak *et al.*, 1985); typical examples range from transition metal complexes such as $\text{Co}(\text{S}_2\text{CNMe}_2)\text{NO}$, where the Co atom is at the centre of a square pyramid (Alderman *et al.*, 1962), and $\text{Cu}_4[\text{S}_2\text{CNET}_2]_4$ (Hesse, 1963), containing a Cu_4 tetrahedron, to main group compounds such as $\text{C}_4\text{H}_9\text{Sn}(\text{S}_2\text{CNET}_2)_2$, where the geometry around the tin atom is pentagonal bipyramidal (Morris & Schlemper, 1979). We report here the structure of the title complex, (I), (Fig. 1).The hexacoordinate Sn atom in (I) is situated at the centre of a distorted octahedral arrangement of four S atoms from the two chelating dithiocarbamate groups, a chloride ion and a benzene C atom. As with other dithiocarbamate complexes, the MS_2C ring is almost planar (Khan *et al.*, 1987). Selected geometrical values are listed in Table 1.The chelating ligands result in acute S—Sn—S angles (Table 1) which are consistent with those in related complexes (Bell *et al.*, 1989). The two dithiocarbamate ligands in (I) show different behaviour in their bonding to Sn. The Cl ligand shows distinctly different Sn—S bond lengths [$\text{Sn1}-\text{S1} = 2.514$ (3), $\text{Sn1}-\text{S2} = 2.627$ (3) Å]. The longer Sn—S bond is associated with the shorter adjacent C—S bond [$\text{C1}-\text{S1} = 1.745$ (11), $\text{C1}-\text{S2} = 1.712$ (11) Å]. This corresponds to the asymmetrical bonding mode described by Bell *et al.* (1989). The difference in Sn—S bond lengths for the other (C10) ligand is much less pronounced.

The C—N bond lengths in both ligands indicate substantial double-bond character, which would reflect a significant

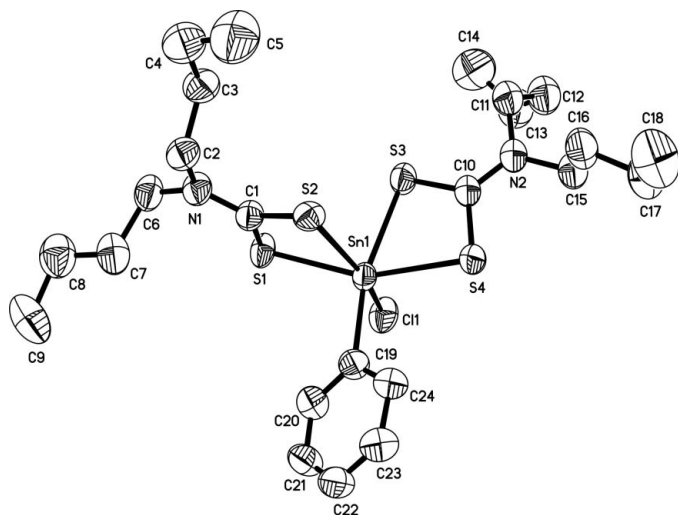


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids (H atoms omitted for clarity).

contribution to the structure made by the canonical form discussed by Casas *et al.* (1989).

Experimental

The synthesis was carried out under a nitrogen atmosphere. Potassium *N,N*-dibutylthiocarbamate (0.243 g, 1 mmol) was suspended in 20 ml benzene and phenyltin trichloride (0.150 g, 0.5 mmol) was added to the mixture. After 12 h at room temperature the solution was filtered and the solvent was gradually removed by evaporation under vacuum until a solid product was obtained. The solid was then recrystallized from diethyl ether and colourless crystals of (I) suitable for X-ray diffraction were obtained. Yield = 0.258 g (80.7%), m.p. 375 K, analysis, calculated for $C_{24}H_{41}ClN_2S_4Sn$: C 45.04, H 6.46, N 4.38; found: C 44.59, H 6.47, N 4.35%.

Crystal data

$[Sn(C_6H_5)(C_9H_{18}NS_2)_2Cl]$
 $M_r = 639.97$
Monoclinic, $P2_1/n$
 $a = 9.2011$ (19) Å
 $b = 10.933$ (2) Å
 $c = 31.164$ (6) Å
 $\beta = 92.821$ (3)°
 $V = 3131.1$ (11) Å³
 $Z = 4$

$D_x = 1.358$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 3244 reflections
 $\theta = 2.3$ – 20.0°
 $\mu = 1.18$ mm⁻¹
 $T = 273$ (2) K
Block, colorless
 $0.39 \times 0.28 \times 0.17$ mm

Data collection

Siemens SMART CCD diffractometer
 φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{min} = 0.656$, $T_{max} = 0.824$
15968 measured reflections

5498 independent reflections
2914 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.050$
 $\theta_{max} = 25.0^\circ$
 $h = -10 \rightarrow 10$
 $k = -10 \rightarrow 13$
 $l = -37 \rightarrow 37$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.075$
 $wR(F^2) = 0.254$
 $S = 1.03$
5498 reflections
289 parameters
H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.1431P)^2 + 1.7378P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 1.30$ e Å⁻³
 $\Delta\rho_{min} = -0.93$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Sn1—C19	2.157 (12)	C1—S1	1.745 (11)
Sn1—Cl1	2.456 (3)	C1—S2	1.707 (11)
Sn1—S1	2.514 (3)	C10—S3	1.715 (12)
Sn1—S4	2.566 (3)	C10—S4	1.737 (12)
Sn1—S3	2.569 (3)	C1—N1	1.312 (13)
Sn1—S2	2.627 (3)	C10—N2	1.308 (14)
S4—Sn1—S3	69.40 (10)	S1—Sn1—S2	69.67 (9)

All H atoms were positioned geometrically (C—H = 0.96–0.97 Å) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(\text{carrier})$ or $U_{iso}(H) = 1.5U_{eq}(\text{methyl carrier})$. The highest peak is located 1.05 Å from atom C7.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL.

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