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[2-(Butylamino)cyclopent-1-ene-1-carbodithioato]chlorodiphenyltin(IV)

Muhammad Hanif,[a] Saqib Ali,[a]* Mukhtiar Hussain,[a] Hussain Moazam Bhatti,[a] Lydia Karmazin Brelot[b] and Helen Stoeckli-Evans[b]

[a]Department of Chemistry, Quaid-i-Azam University, Islamabad, Pakistan, and [b]Institute of Microtechnology, University of Neuchâtel, Rue Emile-Argand 11, CH-2009 Neuchâtel, Switzerland

Correspondence e-mail: drsa54@yahoo.com

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Key indicators

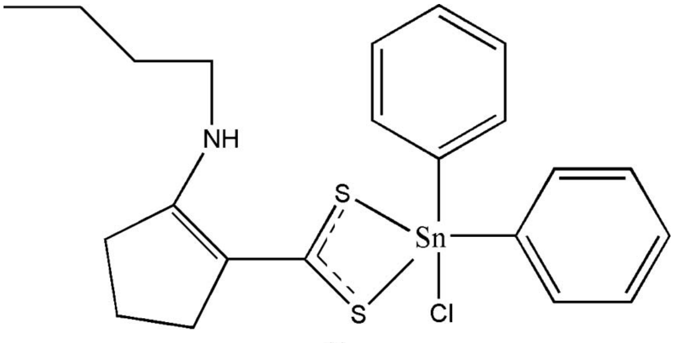
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.007 Å
R factor = 0.027
wR factor = 0.089
Data-to-parameter ratio = 17.2

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

In the title complex, $[Sn(C_6H_5)_2(C_{10}H_{16}NS_2)Cl]$, the Sn atom has a distorted trigonal–bipyramidal geometry, with an axial S—Sn—Cl bond angle of 156.87 (3)°. Intramolecular hydrogen bonding is present between an S atom and the H atom of the amine group. The molecules are linked by an intermolecular C—H···Cl hydrogen bond to form chains extending in the *c* direction.

Comment

Metal complexes of 2-amino-1-cyclopentene-1-dithiocarboxylic acid (ACDA) and its *N*– and *S*-alkyl derivatives have received considerable attention due to their good chelating properties (Amado & Ribeiro-Claro, 2004). The bonding in metal complexes takes place either from the amino N atom and the deprotonated thiol S atom (*N*,*S*-coordination) or from the dithiocarboxylic acid group (*S*,*S*-coordination). The latter pattern can be isobidentate or anisobidentate and this pattern is more frequently observed for main group metals. In recent years, a number of organotin complexes of ACDA and its derivatives have been synthesized and characterized by X-ray crystallographic analysis (Tarassoli *et al.*, 2006; Barroso-Flores *et al.*, 2004). In continuation of our interest in the synthesis and crystal structures of organotin(IV) derivatives of *S*-donor ligands (Ali, Ahmad, Sadiq-Ur-Rehman *et al.*, 2005; Ali, Ahmad, Shahzadi *et al.*, 2005), we report here the synthesis and molecular structure of the title compound, (I).



(I)

The molecular structure of compound (I) is shown in Fig. 1, and selected geometric parameters are listed in Table 1. The coordination geometry about the Sn atom is pentacoordinate distorted trigonal–bipyramidal, with the C atoms of the two phenyl rings attached to atom Sn1 and atom S2 occupying the equatorial positions, while atoms Cl1 and S1 occupy the apical positions. The sum of the equatorial angles is 358.3°, showing

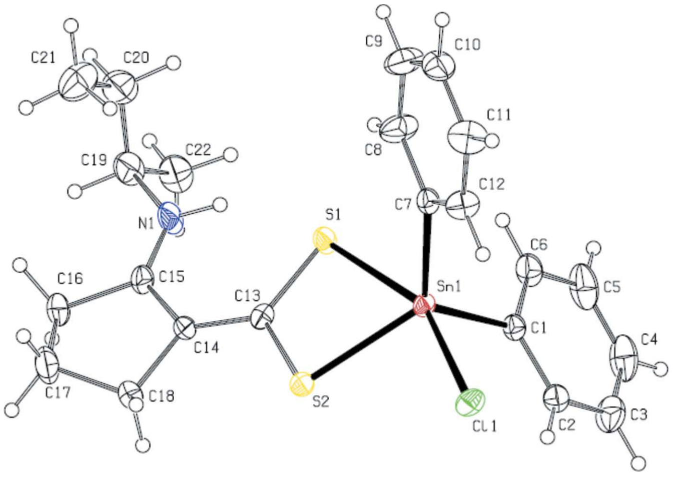


Figure 1
The molecular structure of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

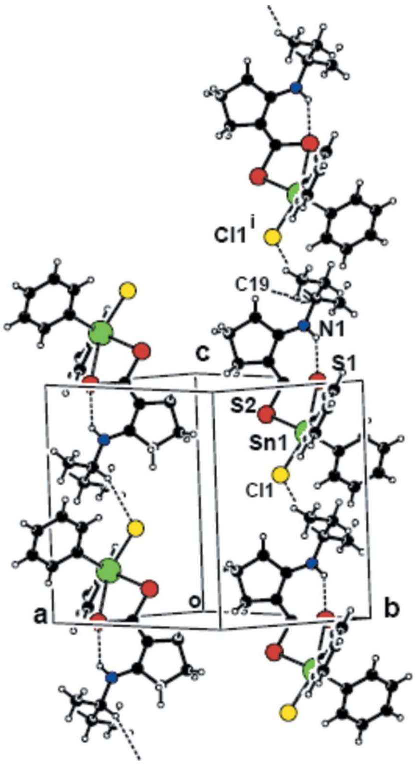


Figure 2
A crystal packing diagram of compound (I), showing the intra- and intermolecular hydrogen bonding as dotted lines. [Symmetry code: (i) *x*, *y*, 1 + *z*.]

some distortion from the ideal bond angle sum of 360° (Shahzadi *et al.*, 2006). Furthermore, the shorter Sn1—S1 bond length is very close to the sum of the covalent radii of Sn and S, and the longer Sn1—S1 distance is significantly less than the sum of their van der Waals radii (4.0 Å; Ma *et al.*, 2003). The Sn—C distances [Sn1—C1 = 2.134 (4) Å and Sn1—C2 = 2.143 (4) Å] are similar to those found in earlier reported structures (Ali, Ahmad, Sadiq-Ur-Rehman *et al.*, 2005; Ali, Ahmad, Shahzadi *et al.*, 2005). The Sn—Cl bond lengths [Sn1—Cl1 = 2.4562 (9) Å] lie in the range of the normal covalent radii (2.37–2.60 Å;).

The distance between atom S1 and the H atom of the amine group (S1$\cdots$H1 = 2.31 Å) is close to the reported value for N—H$\cdots$S hydrogen bonds (2.4 Å; Ueyama *et al.*, 1998). This suggests an intramolecular hydrogen bond between atom S1 and the H atom of the amine group (Fig. 2).

In the crystal structure of compound (I), molecules are linked into chains extending in the *c* direction *via* a C19—H19$\cdots$Cl1^i hydrogen bond; details are given in Table 2 and Fig. 2.

Experimental

The ligand was synthesized by a reported method (Bordas *et al.*, 1972). The title compound was prepared by the reaction of the ligand acid (1.0 g, 4.6 mmol) with diphenyltin(IV) dichloride (0.6 g, 4.65 mmol) in dry methanol (50 ml). The mixture was refluxed for 2 h. The resulting yellow solid was filtered off and washed with methanol. The solid was then recrystallized from dichloromethane (yield 65%; m.p. 470 K).

Crystal data

$[Sn(C_6H_5)_2(C_{10}H_{16}NS_2)Cl]$
M_r = 522.70
Triclinic, $P\bar{1}$
a = 9.4881 (9) Å
b = 11.0779 (10) Å
c = 12.1896 (12) Å
α = 106.457 (11)°
β = 104.555 (11)°
γ = 96.053 (11)°
V = 1167.57 (19) Å^3
Z = 2
D_x = 1.487 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 1.39 mm^{-1}
T = 173 (2) K
Block, yellow
0.50 × 0.40 × 0.40 mm

Data collection

Stoe IPDS diffractometer
φ scans
Absorption correction: multi-scan (*MULABS* in *PLATON*; Spek, 2003)
T_{min} = 0.607, T_{max} = 0.812 (expected range = 0.428–0.573)
8018 measured reflections
4133 independent reflections
3825 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 25.9°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.089
S = 1.16
4133 reflections
240 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0353P)^2 + 2.4792P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.98 e Å^{-3}
$\Delta\rho_{min}$ = −1.17 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—Sn1	2.134 (4)	S2—Sn1	2.4538 (10)
C7—Sn1	2.143 (4)	Cl1—Sn1	2.4562 (9)
S1—Sn1	2.7139 (9)		
C1—Sn1—C7	121.10 (14)	S2—Sn1—Cl1	87.67 (3)
C1—Sn1—S2	116.28 (11)	C1—Sn1—S1	92.31 (10)
C7—Sn1—S2	120.92 (10)	C7—Sn1—S1	95.10 (10)
C1—Sn1—Cl1	97.18 (10)	S2—Sn1—S1	69.22 (3)
C7—Sn1—Cl1	97.93 (10)	Cl1—Sn1—S1	156.87 (3)

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Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···S1	0.88	2.31	3.045 (3)	141
C19—H19*A*···Cl1^{i}	1.00	2.83	3.737 (5)	152

Symmetry code: (i) $x, y, z+1$.

The measured fraction $\theta_{\max}$ of the diffractometer is limited to 93% for triclinic systems. This appears to have no effect on the final bond distances and angle. The H atoms were included in calculated positions and treated as riding atoms, with N—H = 0.88 Å and C—H = 0.95–1.00 Å, and with $U_{\rm iso}$(H) = 1.2 or 1.5 times $U_{\rm eq}$(C,N). The deepest hole is located 0.87 Å from atom Sn1.

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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[2-(Butylamino)cyclopent-1-ene-1-carbodithioato]chlorodiphenyltin(IV). Corrigendum

Muhammad Hanif,[a] Saqib Ali,[a]* Mukhtiar Hussain,[a] Moazzam H. Bhatti,[b] Lydia Karmazin Brelot[c] and Helen Stoeckli-Evans[c]

[a]Department of Chemistry, Quaid-i-Azam University, Islamabad, Pakistan, [b]Department of Chemistry, Allama Iqbal Open University, Islamabad, Pakistan, and [c]Institute of Microtechnology, University of Neuchâtel, Rue Emile-Argand 11, CH-2009 Neuchâtel, Switzerland

Correspondence e-mail: drsa54@yahoo.com

In the paper by Hanif, Ali, Hussain, Bhatti, Karmazin Brelot & Stoeckli-Evans [*Acta Cryst.* (2007), E**63**, m143–m145], the name and address of the fourth author are given incorrectly. The correct author list is shown opposite.

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