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Di-*tert*-butylchloro(*N*,*N*-dimethyldithiocarbamato-κ^2S,S')tin(IV)

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.008 Å
Disorder in main residue
R factor = 0.032
wR factor = 0.092
Data-to-parameter ratio = 15.6

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

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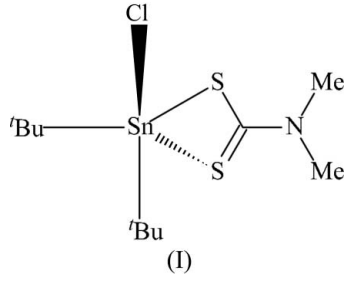
In the title compound, $[Sn(C_4H_9)_2(C_3H_6NS_2)Cl]$, the Sn^{IV} atom has a distorted trigonal bipyramidal geometry that is defined by Cl, two S and two C atoms. The dithiocarbamate chelates to the Sn atom in an anisobidentate manner, and the double-bonded S atom occupies one of the two axial sites.

Comment

The chemistry of organotin compounds has been studied extensively due to their biological activity (Crowe *et al.*, 1988). In our previous work, tribenzyltin dithiocarbamates and dibenzyltin unsaturated alkylphosphates were synthesized, and their biological activity studied (Yin, Ma & Zhang, 1998; Yin, Zhang & Ma, 1998). However, their crystal structures are little known (Yin *et al.*, 1999, 2000). In order to study the coordination at tin in diorganoyltin dithiolate complexes, we have prepared the title compound, (I), and determined its structure (Fig. 1)



(I)

The Sn^{IV} atom is five-coordinated in a distorted trigonal–bipyramidal geometry, the Cl and double-bonded S atoms occupying axial sites (Table 1). The atoms of the equatorial belt (S2, C4, C8 and Sn1) are rather distorted from coplanarity, with a mean deviation of 0.3819 (8) Å.

Experimental

Di-*tert*-butyltin dichloride and sodium *N*,*N*-dimethyldithiocarbamate were prepared according to literature methods (Nai *et al.*, 1961; Xie *et al.*, 1992). The anhydrous sodium salt of *N*,*N*-dimethyldithiocarbamate (1.0 mmol) was added to a dichloromethane solution (20 ml) of di-*tert*-butyltin dichloride (1.0 mmol) and stirred for 14 h at 303 K. The precipitated sodium chloride was removed by filtration and the filtrate was concentrated to about 10 ml under reduced pressure. Diethyl ether (5 ml) and hexane (5 ml) were added to this solution, and a precipitate formed immediately. The products were recrystallized from dichloromethane–hexane (1:1 *v*/*v*) to give colorless crystals (yield 76%; m.p. 405 K). Analysis calculated for $C_{11}H_{24}ClNS_2Sn$: C 34.00, H 6.22, N 3.60%; found: C 34.21, H 6.08, N 3.72%.

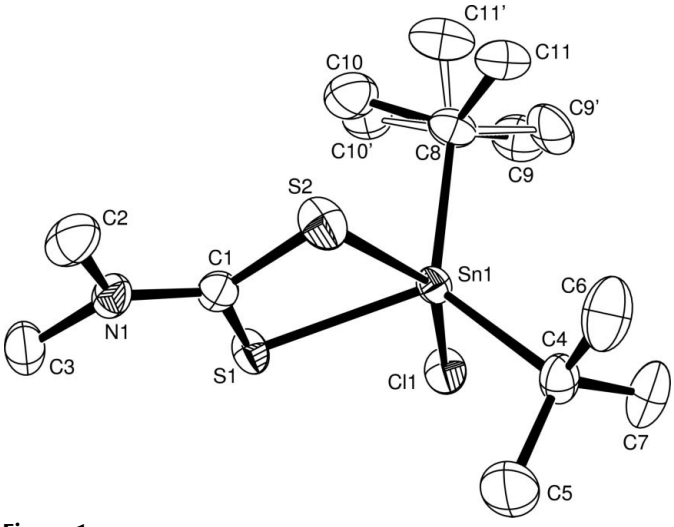


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity. Both disorder components are shown.

Crystal data

$[Sn(C_4H_9)_2(C_3H_6NS_2)Cl]$
M_r = 388.61
Monoclinic, $P2_1/n$
a = 8.3002 (15) Å
b = 16.453 (3) Å
c = 13.053 (2) Å
β = 107.391 (2)°
V = 1701.0 (5) Å^3
Z = 4
D_x = 1.517 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3825 reflections
θ = 2.6–27.1°
μ = 1.88 mm^{-1}
T = 298 (2) K
Block, colorless
0.33 × 0.31 × 0.29 mm

Data collection

Siemens SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.545, T_{max} = 0.580
8742 measured reflections
2987 independent reflections
2376 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 25.0°
$h = -9 \rightarrow 9$
$k = -19 \rightarrow 19$
$l = -13 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.092
S = 1.00
2987 reflections
191 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0554P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −1.25 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—Cl1	2.4904 (12)	Sn1—C4	2.191 (4)
Sn1—S1	2.4866 (11)	Sn1—C8	2.191 (18)
Sn1—S2	2.7418 (12)	Sn1—C8′	2.209 (10)
Cl1—Sn1—S1	84.83 (4)	S1—Sn1—C4	118.33 (12)
Cl1—Sn1—S2	153.06 (4)	S1—Sn1—C8	115.3 (8)
Cl1—Sn1—C4	97.85 (14)	S2—Sn1—C4	93.67 (13)
Cl1—Sn1—C8	98.9 (10)	S2—Sn1—C8	94.3 (9)
S1—Sn1—S2	68.28 (4)	C4—Sn1—C8	124.8 (9)

One of the *tert*-butyl groups (C8–C11) is disordered over two positions. The two site-occupancy factors were refined and converged to 0.35 (2) and 0.65 (2) in the final cycles of refinement. All H atoms were positioned geometrically and refined as riding on their parent atoms, with C—H distances of 0.96 Å and with $U_{iso}(H) = 1.5U_{eq}(C)$. The deepest density hole in the final difference map was 0.92 Å from atom Sn1.

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

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