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Chlorodiethyl[4-(4-nitrophenyl)piperazine-1-carbodithioato]tin(IV)

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Key indicators

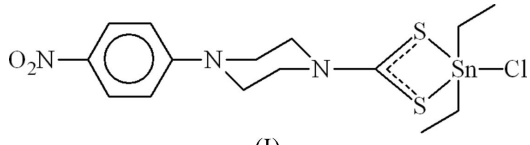
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.024
wR factor = 0.062
Data-to-parameter ratio = 15.3

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

The molecule of the title compound, $[Sn(C_2H_5)_2(C_{11}H_{12}N_3O_2S_2)Cl]$, features an asymmetrically chelating thiocarboxylate ligand. The Sn atom is five-coordinate within a C_2ClS_2 donor set that is best described as trigonal bipyramidal with S and Cl atoms in axial positions, defining a bond angle of 156.58 (2)°.

Comment

Complexing agents with a dithio functional group have been widely used in industry as rodent repellents, vulcanization additives in the manufacture of rubber, additives in lubricants and in agriculture as fungicides on almond trees, stone fruits and vegetables.



(I)

As in a similar compound in the literature (Stiefel & Matsumoto, 1995), the Sn atom is five-coordinate. The geometry of the complex (I) is approximately trigonal bipyramidal, with atoms C12, S1 and C14 occupying the equatorial positions. The sum of the equatorial angles (359.27°) at the tin atom involving the two coordinated C atoms and one S atom [S1—Sn—C12 = 119.25 (6)°, S1—Sn—C14 = 116.36 (7)° and C12—Sn—C14 = 123.66 (9)°] deviates by only 0.73° from 360°, so atoms C12, S1, C14 and Sn are approximately coplanar; the Sn atom is displaced by 0.111 (1) Å from the least-squares plane formed by S1, C12 and C14, and is on the same side as Cl.

The Cl atom occupies approximately one axial position of the trigonal bipyramid; the angle between the Sn—Cl bond and the equatorial plane is 83.66 (9)°. Conversely, because of

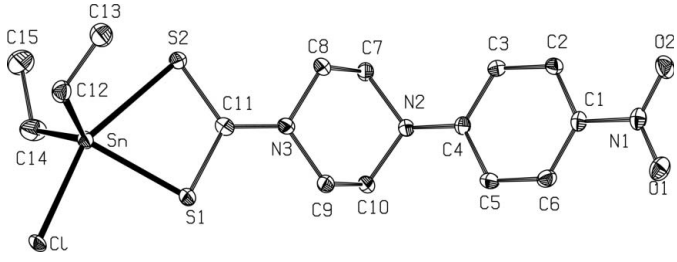


Figure 1
The molecular structure of the title compound. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level. H atoms have been omitted for clarity.

the constraint of the chelate [the S1—Sn—S2 angle is only 69.59 (2)°, the angle between the Sn—S2 bond and the equatorial plane is 72.10 (9)°]; atom S2 cannot exactly occupy the second *trans* axial position of the trigonal bipyramid, the angle Cl—Sn—S2 being 156.58 (2)°.

The S—C bond lengths [S1—C11 = 1.752 (3) Å and S2—C11 = 1.716 (2) Å] appear to be characteristic of the thiocarboxylate group and these distances are all intermediate between the values expected for single and double bonds (Tiekink, 1992).

Experimental

To a solution of 4-(4-nitrophenyl)piperazine-1-carbodithioic acid (0.3 g, 1.059 mmol) in dry methanol (50 ml) was added diethyltin(IV) chloride (0.262 g, 1.059 mmol), dissolved in methanol (30 ml), dropwise and the mixture was stirred vigorously for 3 h. The resulting yellow solid was separated and the filtrate was allowed to evaporate, yielding yellow crystals.

Crystal data

$[Sn(C_2H_5)_2(C_{11}H_{12}N_3O_2S_2)Cl]$	$Z = 4$
$M_r = 494.65$	$D_x = 1.735$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 14.1638$ (9) Å	$\mu = 1.73$ mm^{-1}
$b = 10.5851$ (7) Å	$T = 100$ (1) K
$c = 14.0247$ (9) Å	Trianglar block, yellow
$\beta = 115.799$ (1)°	0.49 × 0.42 × 0.35 mm
$V = 1893.1$ (2) Å^3	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	16982 measured reflections
	4661 independent reflections
φ and ω scans	4332 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)	$R_{int} = 0.021$
	$\theta_{max} = 28.3°$
$T_{min} = 0.430$, $T_{max} = 0.549$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0298P)^2 + 2.2398P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.024$	
$wR(F^2) = 0.062$	
$S = 1.07$	$(\Delta/\sigma)_{max} < 0.001$
4661 reflections	$\Delta\rho_{max} = 1.25$ e Å^{-3}
305 parameters	$\Delta\rho_{min} = -0.39$ e Å^{-3}
All H-atom parameters refined	

The final difference Fourier map was essentially featureless, except for one peak of 1.25 (9) e Å^{-3} within 1.0 Å of Cl. All H atoms were freely refined, with C—H = 0.87 (3)–1.01 (4) Å.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT-Plus* (Bruker, 2000); data reduction: *SAINT-Plus* and *XPREP* (Bruker, 2000); program(s) used to solve structure: *DIRDIF99* (Beurskens *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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