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Dibutylchloro[4-(4-nitrophenyl)piperazine-1-carbodithioato-κ^2S,S']tin(IV)

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Key indicators

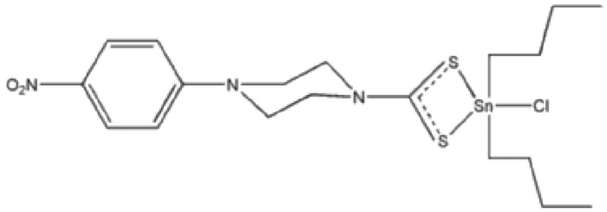
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.025
wR factor = 0.059
Data-to-parameter ratio = 14.6

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

The title complex, $[Sn(C_4H_9)_2(C_{11}H_{12}N_3O_2S_2)Cl]$, features an asymmetrically chelating thiocarboxylate ligand. There are two molecules in the asymmetric unit. The Sn atom is five-coordinated within a C_2ClS_2 donor set that is best described as trigonal–bipyramidal with one S atom and the Cl atom in axial positions, subtending bond angles of 155.67 (2) and 155.11 (2)°, respectively.

Comment

Increasing industrial use of organotin(IV) compounds containing the Sn—S bond, especially as stabilizers of polyvinyl chlorides (Tarassoli *et al.*, 2006), and recognition of the importance of this bond for the biological properties of organotin compounds (Barbieri *et al.*, 1989), have together stimulated the study of thiolates (Casas *et al.*, 1997, 1999). There are two molecules in the asymmetric unit of the title complex, (I).



(I)

The configuration about the Sn atom is five-coordinated distorted trigonal–bipyramidal with atoms S11, C12 and C16 occupying equatorial positions for molecule 1, and S22, C212 and C216 for molecule 2. The sums of the ligand–Sn–ligand angles in the trigonal girdle of the molecules are 359.47 and 358.70°, respectively. The Sn atoms show no large deviations from the equatorial planes, *viz.* 0.0935 (1) and 0.1468 (1) Å, respectively. The distortion is also illustrated by the τ descriptor for five-coordination: 0.46 and 0.45 (Addison *et al.*, 1984); for ideal trigonal-pyramidal coordination τ is 1. Atoms Cl1 and Cl2 each occupy one of the axial positions of the trigonal bipyramid. As a result of being part of the chelate ring the S11—Sn1—S12 and S21—Sn2—S22 angles are not 90° but only 69.13 (2) and 68.94 (2)°, so atoms S12 and S22 do not occupy exactly the corresponding axial positions *trans* to Cl1 and Cl2. The Cl1—Sn1—S12 angle is 155.67 (2) and Cl2—Sn2—S21 is 155.11 (2)°. The Sn1—S12 and Sn2—S21 bonds are markedly elongated [2.7088 (5) and 2.7315 (5) Å], compared with the Sn1—S11 and Sn2—S22 bonds [2.4690 (5) and 2.4720 (7) Å]. This indicates that the thiocarboxylate coordination is unsymmetrical.

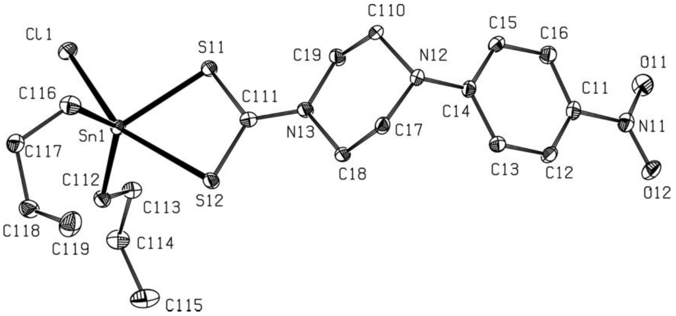


Figure 1
The structure of molecule 1 of the title compound. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level. H atoms have been omitted to improve clarity.

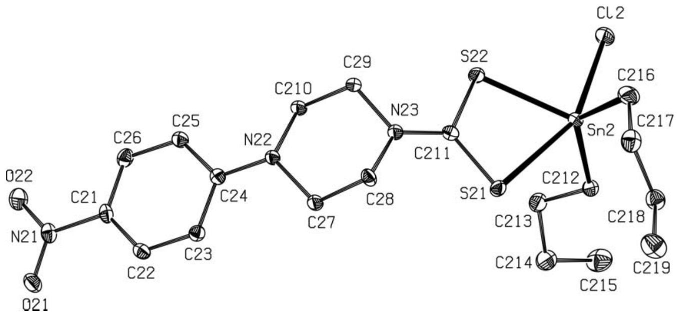


Figure 2
The structure of molecule 2 of the title compound. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level. H atoms have been omitted to improve clarity.

Experimental

To a solution of 4-(4-nitrophenyl)piperazine-1-carbodithioic acid (0.3 g, 1.059 mmol) in dry methanol (40 ml) was added dibuthyltin(IV) chloride (0.161 g, 0.5295 mmol), dissolved in methanol (40 ml), dropwise and the mixture was stirred vigorously for 3 h. The resulting yellow solid was separated and the filtrate was allowed to evaporate to obtain yellow crystals.

Crystal data

$[Sn(C_4H_9)_2(C_{11}H_{12}N_3O_2S_2)Cl]$
M_r = 550.76
Triclinic, $P\bar{1}$
a = 9.7151 (7) Å
b = 14.978 (1) Å
c = 16.152 (1) Å
α = 76.553 (1)°
β = 88.464 (1)°
γ = 87.508 (1)°
V = 2283.4 (3) Å^3
Z = 4
D_x = 1.602 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 1.44 mm^{-1}
T = 100 (1) K
Block, yellow
0.51 × 0.21 × 0.18 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.510, T_{max} = 0.772
21022 measured reflections
10881 independent reflections
9782 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 28.3°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.059
S = 1.05
10881 reflections
745 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0255P)^2 + 1.1175P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.94 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}

A difference Fourier synthesis resulted in the location of all the H atoms, and their coordinates and isotropic displacement parameters were refined [C—H = 0.91 (3)–1.03 (3) 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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