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Dichlorodiethylbis(pyridine-*N*)tin(IV)

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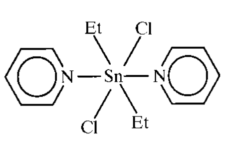
In the title complex, $[Sn(C_2H_5)_2Cl_2(C_5H_5N)_2]$, the Sn atom lies on an inversion centre and is octahedrally coordinated by two Cl atoms, two ethyl C atoms and two pyridine N atoms in an all-*trans* configuration. The dihedral angle between the pyridine ring and the SnNCl plane is 22.4 (2)°.

Comment

The antitumoural activity of diorganotin(IV) dihalide complexes with N-donor ligands (Yang & Guo, 1999) arouses interest in their structures. Surprisingly, despite the wide use of pyridine (py) as a ligand (Thornton, 1990), the only py complexes of this kind that have previously been characterized structurally are the methyl derivatives, $[SnMe_2X_2(py)_2]$ (X = Cl, Br; Aslanov *et al.*, 1978). In view of this, and bearing in mind that spectral data for $[SnEt_2Cl_2(py)_2]$ suggest an all-*trans* octahedral structure (Van den Berghe *et al.*, 1969) and that the Cambridge Structural Database contains no all-*trans* complexes of $SnEt_2Cl_2$ with N-donor ligands (Allen & Kennard, 1993), we have synthesized and determined the crystal and molecular structures of the title compound, (I).



(I)

The molecular structure and atomic numbering scheme of (I) are shown in Fig. 1. The Sn atom is octahedrally coordinated by two Cl atoms, two ethyl C atoms and two py N atoms in an all-*trans* configuration with the Sn atom at a centre of symmetry. Comparison of the Sn—C, Sn—Cl and Sn—N bond lengths with those of similar $[SnR_2Cl_2L_2]$ complexes (as noted above, no data are available for other all-*trans* $[SnEt_2Cl_2L_2]$ complexes) shows that the Sn—N bond is the longest so far reported, and that the Sn—C and Sn—Cl bond lengths are also near the upper ends of the corresponding ranges (Table 2).

The pyridine ring exhibits negligible deviation from the best least-squares plane. The bond lengths in the ring are slightly different from those found in $[SnMe_2X_2(py)_2]$ (Aslanov *et al.*, 1978), but the large s.u.'s of the latter preclude detailed comparison. The dihedral angle between the py ring and the SnNCl plane is 22.4 (2)°. In the pyrazole complexes mentioned above, and in those of some of its derivatives, the orientation of the ring with respect to the SnNCl plane is essentially determined by the formation of intramolecular N—H···Cl hydrogen bonds. Since the C1—H and C5—H groups contiguous to the py N atom in $[SnEt_2Cl_2(py)_2]$ are positionally equivalent to the N—H groups in pyrazole, it is possible that in this case ring orientation may be determined by C—H···Cl interactions [C5···Cl 3.507 (4), H5···Cl 2.86 Å and C5—H5···Cl 128°; C1···Cl^{i} 3.460 (4), H1···Cl^{i} 2.83 Å and C1—H1···Cl^{i} 126°; symmetry code: (i) $-x$, $-y$, $-z$] (Steiner, 1998).

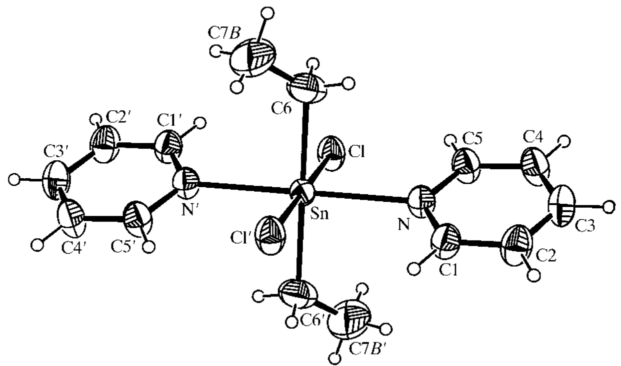


Figure 1
*ORTEP*III (Burnett & Johnson, 1996) diagram of (I), showing the numbering system. Atoms represented as displacement ellipsoids are drawn at the 30% probability level.

With regard to the molecular packing, the crystal may be described as composed of two kinds of molecule layers, *A* and *B*. The N—Sn—N axes of all the molecules have the same orientation, but the molecules of type *A* layers are rotated 90° about this axis with respect to the type *B* layers. Type *A* layers comprise molecules lying at the vertices and centres of the *ab* faces of the unit cell, and type *B* layers comprise molecules lying at the centre of the cell and the centres of the sides oriented in the *c* direction. Intermolecular C—H···Cl interactions [C2···Cl^{ii} 3.719 (5), H2···Cl^{ii} 3.00 Å and C2—H2···Cl^{ii} 136°; symmetry code: (ii) $x + \frac{1}{2}$, $y - \frac{1}{2}$, z] can contribute to the stability of this molecular packing.

Experimental

The title complex was prepared by addition of a solution of py in CH_2Cl_2 to a solution of diethyltin dichloride in the same solvent. After 3 d of stirring, the solution was concentrated, and upon cooling, crystals suitable for X-ray analysis were obtained.

Crystal data

$[Sn(C_2H_5)_2Cl_2(C_5H_5N)_2]$
M_r = 405.91
Monoclinic, $C2/c$
a = 13.9828 (3) Å
b = 9.6029 (2) Å
c = 14.0432 (2) Å
β = 113.7640 (13)°
V = 1725.78 (6) Å^3
Z = 4

D_x = 1.562 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 39 reflections
θ = 3–23°
μ = 1.780 mm^{-1}
T = 293 (2) K
Block, colourless
0.35 × 0.20 × 0.15 mm

Data collection

Siemens CCD diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.550$, $T_{\max} = 0.766$
5824 measured reflections
2128 independent reflections
1459 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.031$
$\theta_{\max} = 28.25°$
$h = -7 \rightarrow 18$
$k = -12 \rightarrow 12$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.077$
$S = 0.999$
2128 reflections
99 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0354P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.602$ e Å^{-3}
$\Delta\rho_{\min} = -0.309$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn—C6	2.151 (4)	Sn—N	2.411 (3)
Sn—N$^{\rm i}$	2.410 (3)	Sn—Cl	2.591 (1)
C6$^{\rm i}$—Sn—N	91.9 (2)	C6—Sn—Cl	89.1 (1)
C6—Sn—N	88.1 (2)	N$^{\rm i}$—Sn—Cl	89.6 (1)
C6$^{\rm i}$—Sn—Cl	90.9 (1)	N—Sn—Cl	90.5 (1)
Cl—Sn—C6—C7*A*	−161.1 (12)	Cl—Sn—C6—C7*B*	127.6 (7)

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

Table 2
Comparison of bond lengths (Å) in [SnR_2Cl$_2L_2$] complexes.

R	*L*	Sn—C	Sn—Cl	Sn—N	Ref.
Methyl	Pyridine	2.15 (2)	2.570 (1)	2.39 (2)	*a*
	Imidazole	2.110 (3)	2.5955 (7)	2.312 (2)	*b*
	N-Methylimidazole	2.118 (5)	2.571 (3)	2.329 (5)	*c*
	2-Chloroimidazole	2.134 (4)	2.591 (2)	2.380 (4)	*d*
	Indazole	2.12 (1)	2.590 (3)	2.377 (6)	*e*
	3-Methyladenine	2.121 (4)	2.596 (1)	2.384 (3)	*f*
	Pyrazole	2.114 (13)	2.570 (3)	2.338 (6)	*g*
	4-Bromopyrazole	2.129 (5)	2.563 (2)	2.359 (4)	*h*
	4-Methylpyrazole	2.128 (6)	2.572 (2)	2.351 (5)	*i*
	3,5-Dimethylpyrazole	2.11 (1)	2.581 (2)	2.379 (6)	*j*
		2.12 (1)			
	3,4,5-Trimethylpyrazole	2.128 (5)	2.563 (4)	2.38 (2)	*k*
		2.125 (5)	2.596 (5)	2.37 (2)	
Ethyl	Pyridine	2.151 (4)	2.591 (1)	2.410 (3)	*l*
				2.411 (3)	
Vinyl	Pyrazole	2.103 (13)	2.565 (2)	2.322 (5)	*m*
Butyl	Pyrazole	2.131 (5)	2.592 (4)	2.329 (5)	*n*
		2.149 (5)	2.587 (4)	2.388 (5)	
Phenyl	Pyrazole	2.146 (7)	2.526 (2)	2.315 (6)	*o*
		2.140 (8)	2.536 (2)	2.341 (6)	
	Thiazole	2.154 (8)	2.508 (3)	2.368 (8)	*p*
		2.158 (8)	2.569 (3)	2.370 (8)	

References: (*a*) Aslanov *et al.* (1978); (*b*) García-Martínez *et al.* (1990); (*c*) Bardi *et al.* (1984); (*d*) Casellato *et al.* (1992); (*e*) Alvarez Boo *et al.* (1997); (*f*) Hazell *et al.* (1997); (*g*) Valle *et al.* (1987); (*h*) Casellato *et al.* (1995); (*i*) Gioia Lobbia *et al.* (1996); (*j*) Graziani *et al.* (1982); (*k*) Calogero *et al.* (1996); (*l*) this work; (*m*) Peruzzo *et al.* (1989); (*n*) Sánchez-González *et al.* (1992); (*o*) Casas *et al.* (1996); (*p*) Alvarez Boo *et al.* (1996).

H atoms were refined using a riding model (HFIX 43 for aromatic, HFIX 23 for methylene and HFIX 137 for methyl). Disorder of the methyl group (C7) was modelled by refinement of the occupancy factor at values of 0.30 and 0.70. The C6—C7 distance was weakly restrained to be close to 1.5 Å.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL*97 (Sheldrick, 1997).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1442). Services for accessing these data are described at the back of the journal.

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