Acta Cryst. (1992). C**48**, 2125–2126

Structure of Dichlorobis(2-chloroimidazole)dimethyltin(IV)

By U. Casellato

Istituto di Chimica e Tecnologia dei Radioelementi, CNR, Padua, Italy

R. Graziani

Dipartimento di Chimica Inorganica, Università di Padova, Padua, Italy

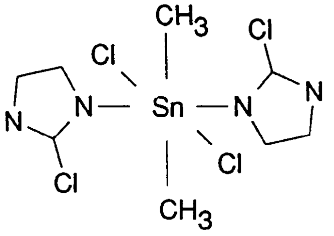
and A. Sánchez González

Departamento de Química Inorgánica, Universidad de Santiago de Compostela, Santiago de Compostela, Spain

(*Received* 26 *February* 1992; *accepted* 3 *April* 1992)

Abstract. $[SnCl_2(CH_3)_2(C_3H_3ClN_2)_2]$, monoclinic, $P2_1/a$, $a = 7.408$ (3), $b = 14.318$ (5), $c = 7.188$ (3) Å, $\beta = 112.59$ (4)°, $V = 703.9$ (5) Å^3, $Z = 2$, $D_x = 2.003$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 2.572$ mm^{-1}, $F(000) = 412$, $R = 0.037$ for 1488 reflections with $I > 3\sigma(I)$. The title compound crystallizes as centrosymmetric *trans* octahedral (OC-6-12)-$[SnMe_2Cl_2(C_3H_3ClN_2)_2]$ molecules. The crystal packing is stabilized by a complex network of intermolecular hydrogen bonds between the coordinated Cl atoms and the N—H groups of the organic ligands of the neighbouring units.

Introduction. With reference to the possible antitumour activity of dialkyltin(IV) dihalide complexes with azoles (Crowe, Smith & Atassi, 1984; Sánchez González, Casas, Sordo, Russo, Lareo & Regueiro, 1990), $[SnMe_2Cl_2(C_3H_3ClN_2)_2]$ was prepared by reacting $[SnMe_2Cl_2]$ with 2-chloroimidazole.



Experimental. The ligand 2-chloroimidazole was prepared according to the literature (Takeuchi, Kirk & Cohen, 1979). Crystals of the complex were obtained by diffusion of cyclohexane into a 1:2 solution of $[SnMe_2Cl_2]$ and $C_3H_3ClN_2$ in dichloromethane. A crystal of maximum dimension 0.2 mm was used for the X-ray work.

Unit-cell parameters were determined by least-squares analysis of 30 carefully measured angle settings, on a Philips 1100 four-circle diffractometer. Intensity data were collected with the θ–2θ scan technique in the range $2 < \theta < 25°$. The intensities of two standard reflections monitored at constant intervals showed that the crystal is stable under irradiation. The intensities were corrected for Lp and for absorption (North, Phillips & Mathews, 1968). From 1853 reflections recorded over the range of indices $-9 \leq h \leq 9$, $0 \leq k \leq 18$, $0 \leq l \leq 9$, 1488 with $I > 3\sigma(I)$ were obtained and used in the calculations.

The structure was solved by the heavy-atom method and final scale factor, atomic coordinates and anisotropic thermal parameters were obtained by full-matrix least-squares refinement. Minimization of the function $\sum w(F)^2$ (with $w = 1$) using 91 variables affords the residual R factor of 0.037. H atoms were introduced in calculated positions with fixed temperature factors (C—H = 1.08 Å, $U_{iso} = 0.07$ Å^2). The maximum feature in the final difference electron density map was 2.4 e Å^{-3}.

All calculations were performed on a MicroVAX using the *SHELX* program package (Sheldrick, 1976). Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Fractional atomic coordinates with equivalent isotropic thermal parameters are given in Table 1.*

Discussion. Interatomic distances and angles are listed in Table 2. A drawing of the structure with the numbering scheme is shown in Fig. 1. The molecular complex is *trans* octahedral with molecular symmetry C_i, the Sn atoms being located on the crystallo-

---

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55345 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1002]

0108-2701/92/122125-02$06.00

Table 1. *Fractional coordinates with equivalent isotropic thermal parameters* ($\text{Å}^2 \times 10^2$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Sn	0.0	0.0	0.0	2.47 (1)
Cl(1)	0.0273 (2)	0.2445 (1)	0.2642 (2)	4.29 (5)
Cl(2)	−0.2321 (2)	0.0193 (1)	0.1903 (2)	3.61 (4)
N(1)	−0.0198 (6)	0.1612 (3)	−0.0867 (6)	2.9 (1)
N(2)	−0.0286 (7)	0.3148 (3)	−0.0962 (8)	3.7 (2)
C(1)	−0.0040 (8)	0.2386 (3)	0.0186 (8)	3.0 (2)
C(2)	−0.0515 (9)	0.2863 (4)	−0.2855 (9)	4.0 (2)
C(3)	−0.0474 (8)	0.1906 (4)	−0.2801 (8)	3.5 (2)
C(4)	0.2490 (7)	0.0221 (4)	0.2720 (7)	3.5 (2)

Table 2. *Bond distances* (Å) *and angles* (°)

Sn—Cl(2)	2.591 (2)	Sn—C(4)	2.134 (4)
Sn—N(1)	2.380 (4)	N(1)—C(1)	1.321 (6)
N(1)—C(3)	1.391 (7)	C(1)—N(2)	1.337 (7)
C(1)—Cl(1)	1.692 (6)	N(2)—C(2)	1.368 (9)
C(2)—C(3)	1.371 (8)		
Cl(2)···H(N^{i})	2.74	Cl(2)···N(2^{i})	3.37 (1)
Cl(2)···H(N^{ii})	2.72	Cl(2)···N(2^{ii})	3.36 (1)
Cl(2)—Sn—C(4)	90.9 (1)	Cl(2)—Sn—N(1)	92.8 (1)
C(4)—Sn—N(1)	92.3 (2)	Sn—N(1)—C(1)	132.9 (3)
Sn—N(1)—C(3)	121.7 (3)	C(1)—N(1)—C(3)	105.4 (4)
N(1)—C(1)—Cl(1)	125.9 (4)	N(2)—C(1)—Cl(1)	122.3 (4)
N(1)—C(1)—N(2)	111.7 (5)	C(1)—N(2)—C(2)	107.8 (4)
N(2)—C(2)—C(3)	106.1 (5)	C(2)—C(3)—N(1)	108.9 (5)

Symmetry code: (i) $-x-\frac{1}{2}, y-\frac{1}{2}, -z$; (ii) $x-\frac{1}{2}, -y+\frac{1}{2}, z$.

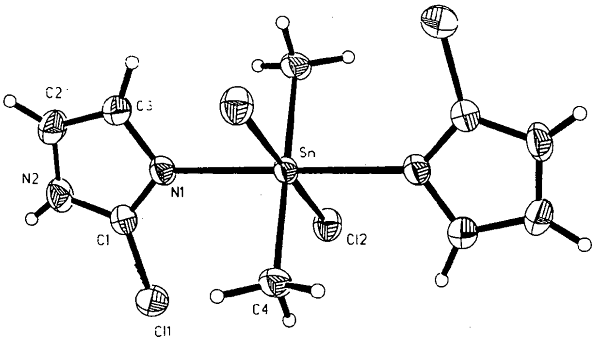


Fig. 1. The crystal structure of $[SnMe_2Cl_2(C_3H_3ClN_2)_2]$.

graphic inversion centres at 0,0,0 and $\frac{1}{2},\frac{1}{2},0$ of equipoint 2(*a*). Coordination bond lengths are normal and the Sn—N bond distance of 2.380 (4) Å can be compared with the corresponding values in imidazole and pyrazole *trans*-$[SnMe_2Cl_2L_2]$ complexes: 2.379 (6) Å (L = 3,5-dimethylpyrazole; Graziani, Casellato, Ettorre & Plazzogna, 1982), 2.338 (6) Å (L = pyrazole; Valle, Ettorre, Peruzzo & Plazzogna, 1987), 2.312 (2) Å (L = imidazole; García Martínez, Sánchez Gonzáles, Macías, Castaño, Casas & Sordo, 1990) and 2.329 (5) Å (L = *N*-methylimidazole; Bardi, Piazzesi, Ettorre & Plazzogna, 1984). It is worth noting that the Sn—N distances for the ligands bearing a Cl atom or a CH_3 group near to the coordination site are 0.04–0.07 Å longer than for the other ligands.

Steric hindrance between Cl(1) and the coordinated Cl(2) atom and C(4) methyl group can probably be ascribed to the fact that the N(1)—Sn—Cl(2) and N(1)—Sn—C(4) bond angles are slightly larger [92.8 (1) and 92.3 (2)°] than the C(4)—Sn—Cl(2) angle [90.9 (1)°].

This is supported by the more evident asymmetry observed in bond angles at N(2), where the Sn—N(1)—C(1) angle [132.9 (3)°], which is contiguous to Cl(1), is over 10° larger than the corresponding Sn—N(1)—C(3) angle [121.7 (3)°], and also at the C(1) level, where the Cl(1)—C(1)—N(1) angle [125.9 (4)°] is significantly larger than the corresponding Cl(1)—C(1)—N(2) angle [122.3 (4)°]. The Sn—N—C angles differ by 3.2° in $[SnMe_2Cl_2(mim)_2]$, (mim = *N*-methylimidazole), while they are essentially equal in $[SnMe_2Cl_2(im)_2]$.

Atoms in the imidazole ligands are coplanar within 0.018 Å, and the ring, which is rigorously normal to the $SnCl_2C_2$ plane, is inclined by 45° with respect to both $SnCl_2N_2$ and SnC_2N_2 planes in order to minimize repulsion forces between Cl(1) and the coordinated groups. Corresponding dihedral angles in complexes where the imidazole or pyrazole ligands have less bulky substituents, or in complexes with imidazole or pyrazole themselves, are in the range 24–35°.

As shown in Table 2 each coordinated Cl atom is hydrogen bonded with the H—N atoms of two neighbouring molecules, each imidazole ligand making two bifurcated hydrogen bonds, so that the entire structure is involved in a complex network of intermolecular hydrogen bonds of the type Cl···H—N which give stability to the crystal packing.

References

Bardi, R., Piazzesi, A. M., Ettorre, R. & Plazzogna, G. (1984). *J. Organomet. Chem.* **270**, 171–176.

Crowe, A. J., Smith, P. J. & Atassi, G. (1984). *Inorg. Chim. Acta*, **93**, 179–184.

García Martínez, E., Sánchez González, A., Macías, A., Castaño, M. V., Casas, J. S. & Sordo, J. (1990). *J. Organomet. Chem.* **385**, 329–334.

Graziani, R., Casellato, U., Ettorre, R. & Plazzogna, G. (1982). *J. Chem. Soc. Dalton Trans.* pp. 805–808.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Sánchez González, A., Casas, J. S., Sordo, J., Russo, U., Lareo, M. I. & Regueiro, B. J. (1990). *J. Inorg. Biochem.* **39**, 227–235.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Takeuchi, Y., Kirk, K. L. & Cohen, L. A. (1979). *J. Org. Chem.* **44**, 4240–4242.

Valle, G., Ettorre, R., Peruzzo, V. & Plazzogna, G. (1987). *J. Organomet. Chem.* **326**, 169–172.