

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1188). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(1,1'-dimethyl-5-nitro-2,2'-biimidazol-3'-ium) *trans*-Tetrachlorodimethylstannate

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Abstract

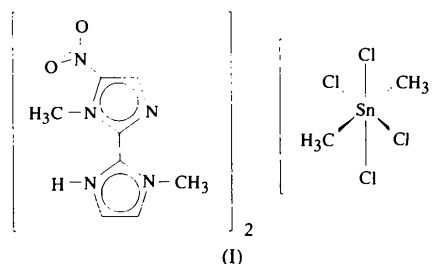
The crystal structure of the title compound, $2C_8H_{10}N_5 \cdot O_2 \cdot [SnMe_2Cl_4]^{2-}$, has been determined. The crystal consists of 1,1'-dimethyl-5-nitro-2,2'-biimidazol-3'-ium cations, in which the angle between the two imidazole rings is $65.0(3)^\circ$, and tetrachlorodimethylstannate(IV) anions, in which the Sn atom has a *trans*-octahedral

environment. Hydrogen bonds between N—H groups and Cl atoms interconnect anions and cations.

Comment

The hydrolysis of dichlorodimethyltin in the presence of donor bases leads to the formation of the tetrachlorodimethylstannate(IV) ion (Nasser, Hossain, Van der Helm & Zuckerman, 1984; Valle, Sánchez Gonzalez, Ettore & Plazzogna, 1988). The structure of this ion, like those of other organotin anions (Johnson, Polborn & North, 1991), is influenced by the presence of hydrogen bonds formed between the anion and cation. This effect was first observed for $[SnMe_2Cl_4]^{2-}$ in the pyridinium salt (Smart & Webster, 1976).

As part of our continuing structural studies on halodiorganotin anions (García Martínez, Sánchez Gonzalez, Castiñeiras, Casas & Sordo, 1994), we have synthesized the title compound, (I). We report here the crystal structure of (I), which includes hydrogen bonds between the anions and cations.



The structure and atomic numbering scheme of (I) are shown in Fig. 1. The crystal consists of 1,1'-dimethyl-5-nitro-2,2'-biimidazol-3'-ium cations and tetrachlorodimethylstannate(IV) anions. Each $[SnMe_2Cl_4]^{2-}$ unit forms N—H...Cl bonds with two neighbouring cations (Fig. 2); the parameters of the hydrogen bonds (Table 2) are in good agreement with the previously reported range for hydrogen bonds between this anion and N—H groups (Smart & Webster, 1976; Nasser, Hossain, Van der Helm & Zuckermann, 1984; Valle, Sánchez Gonzalez, Ettore & Plazzogna 1988).

In the cation, both imidazole rings are planar [for ring A (N11—C12—N13—C14—C15), $\chi^2 = 6.7$; for ring B (N21—C22—N23—C24—C25), $\chi^2 = 7.3$], but have slightly different interatomic distances and angles. In particular, protonation of N23 makes C22—N23—C24 slightly wider than C12—N13—C14; it also appears to make N23—C22 and N23—C24 slightly longer than N13—C12 and N13—C14, although the observed bond lengthening is at the limit of experimental accuracy. The dihedral angle between the rings [$65.0(3)^\circ$] is greater than that in 2,2'-biimidazole (Cromer, Ryan & Storm, 1987) or 1,1'-dimethyl-4,5'-dinitro-2,2'-biimidazole (Casas, Castiñeiras, Martínez, Sordo & Varela, 1995). The angle between ring A and the nitro group [$14.9(3)^\circ$]

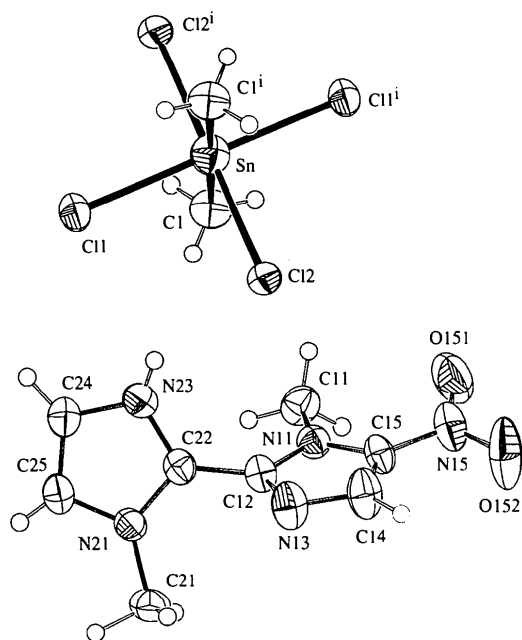


Fig. 1. PLATON (Spek, 1990) drawing showing the atom-numbering scheme and the coordination geometry about the Sn atom in the anion [symmetry code: (i) $-x, -y, -z$]. Displacement ellipsoids are drawn at the 50% probability level.

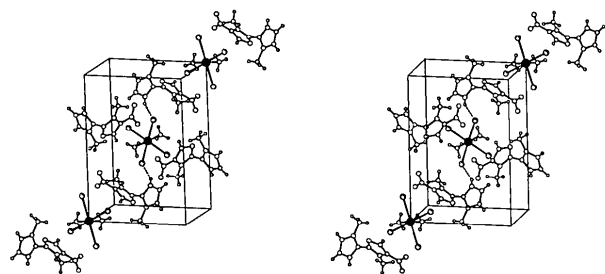


Fig. 2. Stereoview of the unit-cell packing showing the hydrogen bonding. The origin of the unit cell lies at the lower left corner, with a pointing downwards, b upwards and c from left to right.

is close to that found for the 5-NO₂ group in 4,4',5,5'-tetranitro-2,2'-biimidazole (Cromer & Storm, 1990).

The tetrachlorodimethylstannate(IV) anion has a centrosymmetric *trans*-octahedral structure. The Sn—C bond distances are similar to those found in this anion in the above-mentioned compounds. As in these structures, the greatest environmental effect is to lengthen the Sn—Cl distances as a result of either hydrogen bonds or other electrostatic interactions, such as Cl \cdots S (Matsubayashi, Ueyama & Tanaka, 1985).

Experimental

The title compound was prepared by the slow addition of an ethanolic solution of SnMe₂Cl₂ (3 mmol) to an acidified (HCl, 6 mmol) solution of *N,N'*-dimethyl-5-nitro-2,2'-biimidazole (3 mmol) in the same solvent. Crystals were obtained by partial elimination of the solvent.

Crystal data

$(C_8H_{10}N_5O_2)_2-$
[SnCl₄(CH₃)₂]
 $M_r = 706.97$
Monoclinic
 $P2_1/n$
 $a = 9.184(2) \text{ \AA}$
 $b = 14.793(1) \text{ \AA}$
 $c = 10.495(2) \text{ \AA}$
 $\beta = 97.22(1)^\circ$
 $V = 1414.6(6) \text{ \AA}^3$
 $Z = 2$
 $D_x = 1.66 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 7.1\text{--}13.2^\circ$

$\mu = 1.329 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Prismatic

$0.25 \times 0.25 \times 0.25 \text{ mm}$

Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer

$\omega/2\theta$ scans

Absorption correction: refined from ΔF (DIFABS; Walker & Stuart, 1983)

$T_{\min} = 0.77$, $T_{\max} = 0.98$

4889 measured reflections

4397 independent reflections

2515 observed reflections

$[I > 3\sigma(I)]$

$R_{\text{int}} = 0.025$

$\theta_{\text{max}} = 31^\circ$

$h = 0 \rightarrow 13$

$k = 0 \rightarrow 21$

$l = -15 \rightarrow 15$

2 standard reflections

frequency: 120 min

intensity decay: none

Refinement

Refinement on F

$R = 0.038$

$wR = 0.042$

$S = 1.506$

2515 reflections

209 parameters

$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 0.449 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.381 \text{ e \AA}^{-3}$

Extinction correction:

Zachariasen (1963)

Extinction coefficient:

1.01×10^{-7}

Atomic scattering factors

from *International Tables*

for *X-ray Crystallography*

(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

	$B_{\text{eq}} = (4/3)\sum_i \sum_j \beta_{ij} a_i \cdot a_j$			
	x	y	z	B_{eq}
Sn	0	0	0	2.411 (6)
Cl1	0.0562 (2)	-0.08083 (9)	-0.2095 (1)	3.65 (2)
Cl2	-0.0699 (1)	-0.15880 (8)	0.0841 (1)	3.34 (2)
O151	0.3873 (6)	0.2877 (3)	0.0221 (4)	6.0 (1)
O152	0.5154 (5)	0.1780 (4)	-0.0375 (4)	7.4 (1)
N11	0.3536 (4)	0.1931 (3)	0.2477 (4)	2.83 (7)
N13	0.5000 (5)	0.0810 (3)	0.3291 (4)	4.0 (1)
N15	0.4473 (5)	0.2153 (4)	0.0398 (4)	4.3 (1)
N21	0.2621 (4)	0.0805 (3)	0.5274 (4)	2.89 (7)
N23	0.3539 (5)	0.2143 (3)	0.5512 (4)	3.46 (9)
C1	0.2228 (5)	-0.0193 (4)	0.0761 (5)	3.7 (1)
C11	0.2341 (6)	0.2602 (4)	0.2367 (5)	3.8 (1)
C12	0.3929 (5)	0.1372 (3)	0.3485 (4)	2.93 (9)
C14	0.5322 (6)	0.1016 (4)	0.2094 (5)	4.2 (1)
C15	0.4452 (5)	0.1703 (4)	0.1601 (5)	3.12 (9)
C21	0.2171 (6)	-0.0086 (4)	0.4719 (5)	3.9 (1)
C22	0.3330 (5)	0.1446 (3)	0.4704 (4)	2.82 (9)
C24	0.2916 (6)	0.1947 (4)	0.6606 (5)	3.8 (1)
C25	0.2357 (6)	0.1118 (4)	0.6456 (5)	3.7 (1)

Table 2. Selected geometric parameters (Å, °)

Sn—C11	2.611 (1)	N15—C15	1.431 (7)	
Sn—C12	2.618 (1)	N21—C21	1.479 (7)	
Sn—C1	2.121 (5)	N21—C22	1.334 (6)	
O151—N15	1.208 (8)	N21—C25	1.374 (6)	
O152—N15	1.218 (8)	N23—C22	1.334 (6)	
N11—C11	1.473 (7)	N23—C24	1.377 (7)	
N11—C12	1.356 (7)	C12—C22	1.459 (7)	
N11—C15	1.364 (6)	C14—C15	1.355 (8)	
N13—C12	1.323 (7)	C24—C25	1.331 (8)	
N13—C14	1.361 (7)			
C11—Sn—C11'	180.0	C21—N21—C22	125.8 (5)	
C11—Sn—C12	87.5 (4)	C21—N21—C25	125.7 (4)	
C11—Sn—C12'	92.5 (4)	C22—N21—C25	108.5 (4)	
C11—Sn—C1	88.4 (2)	C22—N23—C24	109.0 (4)	
C11—Sn—C1'	91.6 (2)	N11—C12—N13	113.2 (4)	
C12—Sn—C12'	180.0	N11—C12—C22	123.1 (4)	
C12—Sn—C1	91.0 (2)	N13—C12—C22	123.5 (4)	
C12—Sn—C1'	89.0 (2)	N13—C14—C15	109.5 (5)	
C1—Sn—C1'	180.0	N11—C15—N15	123.9 (4)	
C11—N11—C12	126.6 (4)	N11—C15—C14	108.3 (4)	
C11—N11—C15	129.1 (4)	N15—C15—C14	127.9 (5)	
C12—N11—C15	104.4 (4)	N21—C22—N23	107.7 (4)	
C12—N13—C14	104.7 (4)	N21—C22—C12	126.9 (4)	
O151—N15—O152	124.3 (5)	N23—C22—C12	125.1 (4)	
O151—N15—C15	119.5 (5)	N23—C24—C25	106.9 (5)	
O152—N15—C15	116.2 (6)	N21—C25—C24	108.0 (5)	
D—H...A	D—H	H...A	D...A	D—H...A
N23—H23...C12''	0.76 (7)	2.52 (7)	3.191 (5)	147 (6)

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

The Sn and Cl atoms were located in a Patterson map. The remaining atoms (including H atoms) were obtained from difference Fourier syntheses. Refinement was by full-matrix least-squares methods with anisotropic displacement parameters for non-H atoms and isotropic displacement parameters for H atoms, B_{iso} being fixed at 4.0 \AA^2 and not refined.

Data collection: *CAD-4 software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 software*. Data reduction: *SDP-VAX* (Frenz, 1986). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1986). Program(s) used to refine structure: *SDP-VAX*. Molecular graphics: *PLATON* (Spek, 1990), *SCHAKAL* (Keller, 1988).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1160). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bromotriphenyltin–Triphenylphosphine Oxide (1/1)

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Abstract

The Sn atom in bromo(triphenyl)(triphenylphosphine oxide-*O*)tin, $[\text{SnBr}(\text{C}_6\text{H}_5)_3(\text{C}_{18}\text{H}_{15}\text{OP})]$, is five-coordinate in a *trans*-trigonal bipyramidal environment.

Comment

Unlike the isomorphous pair of triphenylphosphine oxide complexes of triphenyltin chloride (Ng & Kumar Das, 1992) and triphenyllead bromide (Epply, Ealy, Yoder, Spencer & Rheingold, 1992), the isoelectronic triphenyltin bromide complex, (I), crystallizes in a polar space group, which permitted the determination of its absolute structure. The five-coordinate complex adopts

