Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CR1185). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). C**51**, 2559–2561

[4-Chloro-3-(2-nitrophenylthio)butyl]triphenylstannane

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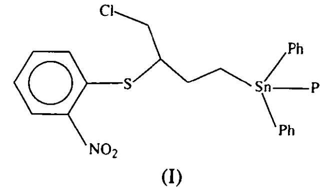
(*Received* 30 *March* 1995; *accepted* 19 *June* 1995)

Abstract

The Sn atom in $[Sn(C_6H_5)_3(C_{10}H_{11}ClNO_2S)]$ has slightly distorted tetrahedral geometry; a weak intramolecular S···O interaction exists as shown by the S···O distance of 2.610 (5) Å and the C—S···O angle of 177.5 (3)°.

Comment

The crystal structure determination of the addition product of $Ph_3SnCH_2CH_2CH{:}CH_2$ and 2-$O_2NC_6H_4SCl$ reveals it to be $Ph_3SnCH_2CH_2CH(SC_6H_4NO_2\text{-}2)CH_2Cl$, (I), the anti-Markownikov adduct. This corrects an earlier assignment of the structure, based on the ^1H NMR spectrum, as $Ph_3SnCH_2CH_2CH(Cl)CH_2SC_6H_4NO_2$-2 (Wigzell & Wardell, 1982). The geometry about the Sn atom in (I) is slightly distorted tetrahedral, with C—Sn—C valence angles ranging from 107.2 (1) to 115.5 (2)°. There are no short Sn···Cl or Sn···S contacts.



(I)

As found for a number of aryl and alkyl 2-nitroaryl sulfides, there is a weak intramolecular S···O interaction within (I) in the solid state; the nitro group is nearly coplanar with the atoms in the SC_6H_4 moiety: the O(10)—N(8)—C(7)—C(6) torsion angle is 3.6 (7)°. The S(5)···O(10) separation in (I) is 2.610 (5) Å, which is less than the sum of the van der Waals radii (3.25 Å). The C(4)—S(5)···O(10) angle is 177.5 (3)°. Crystallographically determined values of S···O distances and C—S···O angles in other 2-nitroaryl sulfides are: 2.656 (1) Å and 171.7° in 2-$O_2NC_6H_4SC_6H_4NO_2$-2 (Kucsman, Kapovits, Parkanyi, Argay & Kalman, 1984); 2.715 (8) Å and 178.2 (3)° in $Ph_3SnCHClCH_2SC_6H_3Me$-4-NO_2-2 and 2.655 (5) Å and 172.7 (3)° in $Ph_3SnCH(SCN)CH_2SC_6H_4NO_2$-2 (Howie, Wardell, Zanetti, Cox & Doidge-Harrison, 1992). As shown by electron diffraction, the S···O interactions in 2-nitroaryl sulfides can persist in the gas phase; for example, values of S···O and C—S···O in gaseous 2-$O_2NC_6H_4SMe$ were determined to be 2.769 (9) Å and

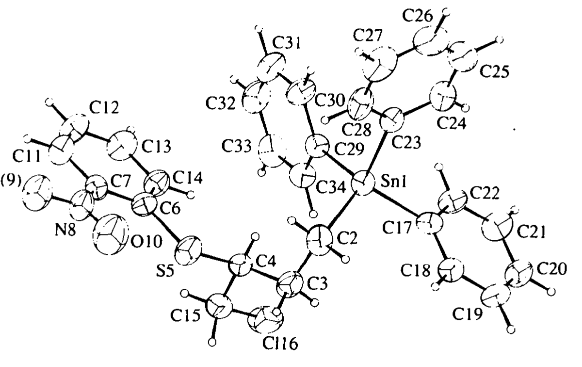


Fig. 1. View of a molecule of (I) indicating non-H-atom labelling. Displacement ellipsoids are at the 50% probability level and H atoms are drawn as small circles of arbitrary radii.

Acta Crystallographica Section C
ISSN 0108-2701 ©1995

173.9 (15)°, respectively (Schultz, Hargittai, Kapovits & Kucsman, 1987).

There is a staggered arrangement of atoms about the C(4)—C(15) bond: the Cl(16)—C(15)—C(4)—S(5) torsion angle is 175.5 (3)°.

Experimental

Compound (I) was prepared from $Ph_3SnCH_2CH_2CH{:}CH_2$ and $2\text{-}O_2NC_6H_4SCl$ and was recrystallised from ethanol (Wigzell & Wardell, 1982).

Crystal data

[Sn(C$_6$H$_5$)$_3$(C$_{10}$H$_{11}$ClNO$_2$S)]
$M_r = 594.72$
Monoclinic
$P2_1/c$
$a = 23.382$ (6) Å
$b = 10.160$ (3) Å
$c = 11.265$ (5) Å
$\beta = 92.78$ (3)°
$V = 2673$ (2) Å^3
$Z = 4$
$D_x = 1.48$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 13.03$–14.86°
$\mu = 1.158$ mm^{-1}
$T = 296$ K
Hexagonal
1.0 × 0.5 × 0.125 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scans (Frenz, 1983)
$T_{\min} = 0.967$, $T_{\max} = 0.999$
4720 measured reflections
4197 independent reflections

2680 observed reflections [$I > 3\sigma(I)$]
$R_{\rm int} = 0.014$
$\theta_{\max} = 24.0°$
$h = -26 \rightarrow 26$
$k = -11 \rightarrow 0$
$l = 0 \rightarrow 12$
4 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F
$R = 0.035$
$wR = 0.042$
$S = 0.934$
2680 reflections
259 parameters
H-atom parameters not refined
Weights: five-term Chebychev polynomial (Carruthers & Watkin, 1979)
$(\Delta/\sigma)_{\max} = 0.012$

$\Delta\rho_{\max} = 0.68$ e Å^{-3}
$\Delta\rho_{\min} = -0.65$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)
Absolute configuration: Flack (1983) parameter = 0; Rogers (1981) parameter = 1.0

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Sn(1)	0.18338 (1)	0.01966 (3)	0.11660 (3)	0.0558
Cl(16)	0.34310 (9)	−0.0683 (2)	0.4020 (2)	0.1013
S(5)	0.37237 (7)	−0.0908 (1)	0.0228 (1)	0.0735
O(9)	0.4899 (2)	0.0007 (5)	−0.2444 (5)	0.0993
O(10)	0.4311 (3)	−0.1177 (6)	−0.1669 (6)	0.1214
N(8)	0.4553 (2)	−0.0169 (5)	−0.1713 (4)	0.0691
C(2)	0.2369 (2)	−0.1416 (5)	0.0639 (6)	0.0745
C(3)	0.2858 (3)	−0.1780 (5)	0.1485 (6)	0.0733
C(4)	0.3343 (2)	−0.0751 (5)	0.1584 (5)	0.0615
C(6)	0.4067 (1)	0.0638 (2)	0.0042 (3)	0.0601
C(7)	0.4434 (1)	0.0810 (3)	−0.0880 (3)	0.0593
C(11)	0.4690 (1)	0.2026 (3)	−0.1051 (3)	0.0737
C(12)	0.4578 (2)	0.3071 (3)	−0.0300 (3)	0.0826
C(13)	0.4212 (2)	0.2900 (3)	0.0622 (3)	0.0757
C(14)	0.3956 (1)	0.1683 (3)	0.0793 (2)	0.0655
C(15)	0.3756 (3)	−0.0946 (6)	0.2625 (6)	0.0823
C(17)	0.1367 (1)	−0.0392 (3)	0.2672 (2)	0.0593
C(18)	0.1273 (2)	−0.1712 (2)	0.2921 (3)	0.0730
C(19)	0.0961 (2)	−0.2063 (2)	0.3891 (4)	0.0853
C(20)	0.0742 (2)	−0.1093 (4)	0.4612 (3)	0.0775
C(21)	0.0836 (2)	0.0228 (3)	0.4363 (3)	0.0779
C(22)	0.1148 (2)	0.0578 (2)	0.3393 (3)	0.0655
C(23)	0.1246 (1)	0.0642 (3)	−0.0294 (2)	0.0576
C(24)	0.0683 (1)	0.0995 (4)	−0.0107 (3)	0.0752
C(25)	0.0300 (1)	0.1206 (4)	−0.1072 (4)	0.0913
C(26)	0.0480 (2)	0.1063 (5)	−0.2222 (3)	0.0965
C(27)	0.1043 (2)	0.0710 (5)	−0.2409 (2)	0.0906
C(28)	0.1426 (1)	0.0500 (4)	−0.1445 (3)	0.0754
C(29)	0.2278 (1)	0.1984 (2)	0.1643 (2)	0.0521
C(30)	0.2249 (2)	0.3052 (3)	0.0870 (2)	0.0675
C(31)	0.2536 (2)	0.4212 (3)	0.1172 (3)	0.0826
C(32)	0.2852 (2)	0.4304 (3)	0.2245 (3)	0.0815
C(33)	0.2882 (1)	0.3236 (3)	0.3018 (3)	0.0759
C(34)	0.2595 (1)	0.2076 (3)	0.2716 (2)	0.0628

Table 2. *Selected geometric parameters* (Å, °)

Sn(1)—C(2)	2.162 (5)	O(9)—N(8)	1.196 (6)
Sn(1)—C(17)	2.146 (2)	O(10)—N(8)	1.173 (6)
Sn(1)—C(23)	2.140 (2)	N(8)—C(7)	1.404 (5)
Sn(1)—C(29)	2.147 (2)	C(2)—C(3)	1.498 (9)
Cl(16)—C(15)	1.799 (7)	C(3)—C(4)	1.543 (7)
S(5)—C(4)	1.811 (5)	C(4)—C(15)	1.497 (8)
S(5)—C(6)	1.782 (2)		
C(2)—Sn(1)—C(17)	109.2 (2)	C(3)—C(4)—C(15)	114.3 (5)
C(2)—Sn(1)—C(23)	107.9 (2)	S(5)—C(6)—C(7)	119.8 (2)
C(17)—Sn(1)—C(23)	109.5 (1)	S(5)—C(6)—C(14)	120.2 (2)
C(2)—Sn(1)—C(29)	115.5 (2)	N(8)—C(7)—C(6)	124.0 (3)
C(17)—Sn(1)—C(29)	107.2 (1)	N(8)—C(7)—C(11)	115.9 (3)
C(23)—Sn(1)—C(29)	107.3 (1)	Cl(16)—C(15)—C(4)	112.4 (4)
C(4)—S(5)—C(6)	105.3 (2)	Sn(1)—C(17)—C(18)	121.3 (2)
O(9)—N(8)—O(10)	120.3 (5)	Sn(1)—C(17)—C(22)	118.7 (2)
O(9)—N(8)—C(7)	121.1 (5)	Sn(1)—C(23)—C(24)	121.1 (2)
O(10)—N(8)—C(7)	118.6 (4)	Sn(1)—C(23)—C(28)	118.8 (2)
Sn(1)—C(2)—C(3)	116.5 (4)	Sn(1)—C(29)—C(30)	119.7 (2)
C(2)—C(3)—C(4)	114.5 (4)	Sn(1)—C(29)—C(34)	120.3 (2)
S(5)—C(4)—C(3)	105.6 (4)	C(4)—S(5)—O(10)	177.5 (3)
S(5)—C(4)—C(15)	108.9 (4)		
Cl(16)—C(15)—C(4)—S(5)	175.5 (3)	O(10)—N(8)—C(7)—C(6)	3.6 (7)

The weighting scheme was derived empirically using a five-term Chebychev polynomial in $|F_o|$ (Carruthers & Watkin, 1979). Each phenyl group was idealized and refined as a rigid group. H-atom parameters were chosen so that C—H = 1.00 Å and $U_{\rm iso} = 0.05$ Å^2. All calculations were performed on a Viglen Genie 486 PC and latterly on a Viglen Genie P590 PC.

Data collection: *SDP* (Frenz, 1983). Data reduction: *SDP*. Program(s) used to solve structure: *SIR*88 (Burla *et al.*, 1989). Program(s) used to refine structure: *CRYSTALS/PC* (Watkin, Carruthers & Betteridge, 1985). Molecular graphics: *CAMERON* (Pearce, Watkin & Prout, 1992). Software used to prepare material for publication: *CRYSTALS*/PC.

Thanks are due to Dr D. C. Povey, Department of Chemistry, University of Surrey, for collection of the reflection data.

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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Acta Cryst. (1995). C**51**, 2561–2563

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

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(*Received* 18 *January* 1995; *accepted* 10 *April* 1995)

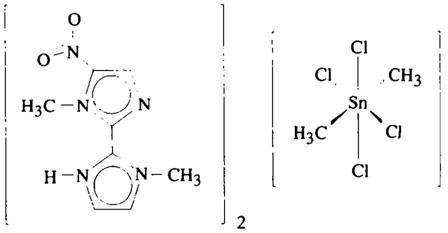
Abstract

The crystal structure of the title compound, $2C_8H_{10}N_5O_2^+.[SnMe_2Cl_4]^{2-}$, 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)°, and tetrachlorodimethylstannate(IV) anions, in which the 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, Ettorre & 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 Martinez, 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.



(I)

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, Ettorre & Plazzogna 1988).

In the cation, both imidazole rings are planar [for ring *A* (N11—C12— N13—C14—C15), χ^2 = 6.7; for ring *B* (N21—C22—N23—C24—C25), χ^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)°] 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)°]