

Bis(dimethylammonium) tetrachlorido-dimethylstannate(IV)

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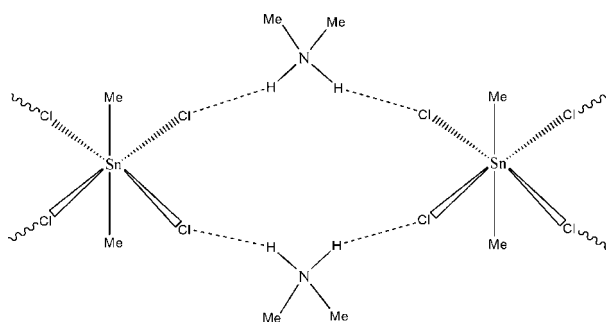
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Key indicators: single-crystal X-ray study; $T = 297$ K; mean $\sigma(\text{N}-\text{C}) = 0.004$ Å; R factor = 0.023; wR factor = 0.060; data-to-parameter ratio = 29.2.

Regular crystals of the title compound, $(\text{C}_2\text{H}_8\text{N})_2[\text{Sn}(\text{CH}_3)_2\text{Cl}_4]$, were obtained by reacting SnMe_2Cl_2 with $(\text{CH}_3)_2\text{NH}\cdot\text{HCl}$ in ethanol in a 1:1 ratio. The Sn atom lies on a center of symmetry and is six-coordinated. It has a distorted octahedral SnC_2Cl_4 environment with the Cl atoms in *cis* positions. The Cl atoms are connected to dimethylammonium cations through $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds, forming an infinite chain extending parallel to [010].

Related literature

For background to organotin(IV) chemistry, see: Gielen *et al.* (1996); Evans & Karpel (1985); Crowe *et al.* (1994); Diasse-Sarr *et al.* (1997); Diop *et al.* (2002, 2003). For related compounds, see: Valle *et al.* (1985); Casas *et al.* (1996); Diop *et al.* (2011).



Experimental

Crystal data

$(\text{C}_2\text{H}_8\text{N})_2[\text{Sn}(\text{CH}_3)_2\text{Cl}_4]$
 $M_r = 382.75$
 Triclinic, $P\bar{1}$
 $a = 6.6162$ (9) Å
 $b = 7.3703$ (11) Å

$c = 8.4555$ (12) Å
 $\alpha = 109.625$ (14)°
 $\beta = 98.345$ (12)°
 $\gamma = 92.812$ (12)°
 $V = 382.13$ (9) Å³

$Z = 1$
 Mo $K\alpha$ radiation
 $\mu = 2.34$ mm⁻¹

$T = 297$ K
 $0.5 \times 0.3 \times 0.2$ mm

Data collection

Oxford Diffraction Xcalibur
 Sapphire2 diffractometer
 Absorption correction: multi-scan
 (*CrysAlis CCD*; Oxford
 Diffraction, 2009)
 $T_{\min} = 0.352$, $T_{\max} = 0.652$

3329 measured reflections
 1871 independent reflections
 1839 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.018$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.023$
 $wR(F^2) = 0.060$
 $S = 1.06$
 1871 reflections

64 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.56$ e Å⁻³
 $\Delta\rho_{\min} = -0.43$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1E}\cdots\text{Cl1}$	0.9	2.31	3.201 (2)	169
$\text{N1}-\text{H1D}\cdots\text{Cl2}^i$	0.9	2.37	3.229 (2)	160

 Symmetry code: (i) $x, y + 1, z$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2009).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BR2163).

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supplementary materials

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Bis(dimethylammonium) tetrachloridodimethylstannate(IV)

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Comment

As some compounds belonging to organotin family have been screened and found to be very more active than *cis* platin towards some kinds of cancer, many groups have been involved in the seek of new organotin compounds (Gielen, 1996; Crowe, 1994). In another hand the various applications of compounds of this family have been outlined (Evans & Karpel, 1985). In our group we have yet published some papers in this field (Diop *et al.* 2002; Diop *et al.* 2003; Diasse-Sarr *et al.* 1997). In this paper we have initiated the study of the interactions between $(\text{CH}_3)_3\text{NH}\cdot\text{Cl}$ and SnMe_2Cl_2 which has yielded $[(\text{CH}_3)_2\text{NH}_2^+]_2[\text{SnMe}_2\text{Cl}_4^{2-}]$, X-ray structure determination of which has been carried out.

In the $[\text{SnMe}_2\text{Cl}_4]^{2-}$ anion the tin atom, which lies on a center of symmetry, is coordinated to the two methyl groups and four Cl atoms (Fig 1) in an octahedral geometry with *trans* methyl groups.

The Sn—C bond distances (2.116 Å) are practically equal to those found in other octahedral dimethyltin(IV) diaquo-dichloro complexes $\text{SnMe}_2(\text{H}_2\text{O})_2\text{Cl}_2$ (2.112 Å) reported by Valle *et al.* (1985) and longer than those in $[\text{Hthiamine}][\text{SnMe}_2(\text{H}_2\text{O})_2\text{Cl}_2]\text{Cl}$ (2.092 Å and 2.084 Å) reported by Casas *et al.* (1996).

The Cl—Sn—Cl and Cl—Sn—CH₃ angles being very near to 90° indicates an almost perfect octahedron. The interactions between $[(\text{CH}_3)\text{NH}_2^+]$ and anion are hydrogen bonds type. The C—N—C angles of the cation is close to 109°, in agreement with the expected sp^3 hybridation. The interactions between $[(\text{CH}_3)\text{NH}_2^+]$ and anion imply hydrogen bonds.

Experimental

The title compound has been obtained as white crystalline solid by reacting dimethylammonium chloride (Merck) with dimethyltin dichloride (Aldrich) in ethanol (1/1 ratio, mp: 190°). After a slow solvent evaporation colourless crystals suitable for X-ray work were obtained. All the chemicals were used without any further purification.

Figures

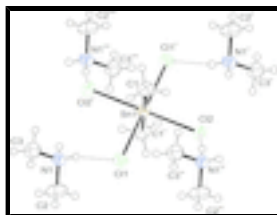


Fig. 1. Molecular packing around one anion implying hydrogen bonds (dashed lines) with the atom numbering used and 50% probability displacement ellipsoids. Symmetry operations : ['] -x, -y, -z; ["] x, y+1, z, [""'] -x, -y, -z.

Bis(dimethylammonium) tetrachloridodimethylstannate(IV)

Crystal data

$(C_2H_8N)_2[SnCH_3)_2Cl_4]$	$Z = 1$
$M_r = 382.75$	$F(000) = 190$
Triclinic, PT	$D_x = 1.663 \text{ Mg m}^{-3}$
Hall symbol: $-P 1$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 6.6162 (9) \text{ \AA}$	Cell parameters from 3675 reflections
$b = 7.3703 (11) \text{ \AA}$	$\theta = 2.9\text{--}31.3^\circ$
$c = 8.4555 (12) \text{ \AA}$	$\mu = 2.34 \text{ mm}^{-1}$
$\alpha = 109.625 (14)^\circ$	$T = 297 \text{ K}$
$\beta = 98.345 (12)^\circ$	Fragment of rounded block, colourless
$\gamma = 92.812 (12)^\circ$	$0.5 \times 0.3 \times 0.2 \text{ mm}$
$V = 382.13 (9) \text{ \AA}^3$	

Data collection

Oxford Diffraction Xcalibur Sapphire2 diffractometer	1871 independent reflections
Radiation source: sealed X-ray tube graphite	1839 reflections with $I > 2\sigma(I)$
Detector resolution: $8.3622 \text{ pixels mm}^{-1}$	$R_{\text{int}} = 0.018$
4 stepped ω -scans over 115 deg. with kappa -79 deg. (chi -58.3 deg.), phi 0, 90, 180, 270 deg. step 1 deg., exposure time 45 s detector distance 50 mm detector angle 30 deg.	$\theta_{\text{max}} = 28.3^\circ$, $\theta_{\text{min}} = 4.1^\circ$
Absorption correction: multi-scan (<i>Crys.Alis CCD</i> ; Oxford Diffraction, 2009)	$h = -8 \rightarrow 7$
$T_{\text{min}} = 0.352$, $T_{\text{max}} = 0.652$	$k = -9 \rightarrow 9$
3329 measured reflections	$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.023$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.060$	H-atom parameters constrained
$S = 1.06$	$w = 1/[\sigma^2(F_o^2) + (0.0362P)^2 + 0.2233P]$
1871 reflections	where $P = (F_o^2 + 2F_c^2)/3$
64 parameters	$(\Delta/\sigma)_{\text{max}} < 0.001$
0 restraints	$\Delta\rho_{\text{max}} = 0.56 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.43 \text{ e \AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.2689 (4)	0.3818 (4)	0.5967 (3)	0.0404 (5)
H1A	0.2902	0.2502	0.5847	0.061*
H1B	0.1366	0.3849	0.5343	0.061*
H1C	0.275	0.4566	0.715	0.061*
Cl1	0.25609 (10)	0.40746 (9)	0.20155 (7)	0.04289 (14)
Cl2	0.64584 (8)	0.16207 (8)	0.39707 (7)	0.03603 (12)
Sn1	0.5	0.5	0.5	0.02900 (8)
C2	0.2993 (5)	0.8184 (6)	0.0309 (4)	0.0621 (8)
H2A	0.4272	0.764	0.0166	0.093*
H2B	0.3073	0.9423	0.0167	0.093*
H2C	0.1905	0.7333	-0.0527	0.093*
C3	0.0570 (4)	0.9131 (4)	0.2302 (4)	0.0479 (6)
H3A	0.0331	0.9174	0.3406	0.072*
H3B	-0.0493	0.8274	0.1442	0.072*
H3C	0.0563	1.0407	0.2239	0.072*
N1	0.2581 (3)	0.8422 (3)	0.2023 (3)	0.0381 (4)
H1D	0.3576	0.9263	0.28	0.046*
H1E	0.2622	0.7276	0.2183	0.046*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0393 (11)	0.0380 (11)	0.0450 (12)	-0.0035 (9)	0.0167 (10)	0.0128 (10)
Cl1	0.0486 (3)	0.0369 (3)	0.0360 (3)	-0.0001 (2)	-0.0059 (2)	0.0094 (2)
Cl2	0.0387 (3)	0.0282 (2)	0.0402 (3)	0.00715 (19)	0.0087 (2)	0.0093 (2)
Sn1	0.03107 (11)	0.02496 (11)	0.03074 (11)	-0.00012 (7)	0.00626 (7)	0.00940 (8)
C2	0.0604 (18)	0.078 (2)	0.0417 (14)	-0.0007 (16)	0.0195 (13)	0.0098 (14)
C3	0.0387 (12)	0.0550 (15)	0.0478 (14)	0.0036 (11)	0.0083 (10)	0.0151 (12)
N1	0.0382 (10)	0.0390 (10)	0.0351 (9)	0.0007 (8)	0.0029 (7)	0.0120 (8)

Geometric parameters (\AA , $^\circ$)

C1—Sn1	2.116 (2)	C2—H2A	0.96
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supplementary materials

C1—H1A	0.96	C2—H2B	0.96
C1—H1B	0.96	C2—H2C	0.96
C1—H1C	0.96	C3—N1	1.475 (3)
Cl1—Sn1	2.6441 (7)	C3—H3A	0.96
Cl2—Sn1	2.6297 (7)	C3—H3B	0.96
Sn1—C1 ⁱ	2.116 (2)	C3—H3C	0.96
Sn1—Cl2 ⁱ	2.6297 (7)	N1—H1D	0.9
Sn1—Cl1 ⁱ	2.6441 (7)	N1—H1E	0.9
C2—N1	1.468 (4)		
Sn1—C1—H1A	109.5	Cl1 ⁱ —Sn1—Cl1	180
Sn1—C1—H1B	109.5	N1—C2—H2A	109.5
H1A—C1—H1B	109.5	N1—C2—H2B	109.5
Sn1—C1—H1C	109.5	H2A—C2—H2B	109.5
H1A—C1—H1C	109.5	N1—C2—H2C	109.5
H1B—C1—H1C	109.5	H2A—C2—H2C	109.5
C1 ⁱ —Sn1—C1	180.00 (13)	H2B—C2—H2C	109.5
C1 ⁱ —Sn1—Cl2 ⁱ	90.42 (7)	N1—C3—H3A	109.5
C1—Sn1—Cl2 ⁱ	89.58 (7)	N1—C3—H3B	109.5
C1 ⁱ —Sn1—Cl2	89.58 (7)	H3A—C3—H3B	109.5
C1—Sn1—Cl2	90.42 (7)	N1—C3—H3C	109.5
Cl2 ⁱ —Sn1—Cl2	180	H3A—C3—H3C	109.5
C1 ⁱ —Sn1—Cl1 ⁱ	90.43 (8)	H3B—C3—H3C	109.5
C1—Sn1—Cl1 ⁱ	89.57 (8)	C2—N1—C3	112.7 (2)
Cl2 ⁱ —Sn1—Cl1 ⁱ	89.90 (2)	C2—N1—H1D	109.1
Cl2—Sn1—Cl1 ⁱ	90.10 (2)	C3—N1—H1D	109.1
C1 ⁱ —Sn1—Cl1	89.57 (8)	C2—N1—H1E	109.1
C1—Sn1—Cl1	90.43 (8)	C3—N1—H1E	109.1
Cl2 ⁱ —Sn1—Cl1	90.10 (2)	H1D—N1—H1E	107.8
Cl2—Sn1—Cl1	89.90 (2)		

Symmetry codes: (i) $-x+1, -y+1, -z+1$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1E \cdots Cl1	0.9	2.31	3.201 (2)	169
N1—H1D \cdots Cl2 ⁱⁱ	0.9	2.37	3.229 (2)	160

Symmetry codes: (ii) $x, y+1, z$.

Fig. 1

