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Bis(4-methylpiperidinium) hexachlorido-stannate(IV)

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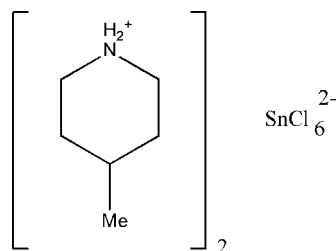
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 Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.018; wR factor = 0.041; data-to-parameter ratio = 17.7.

The crystal structure of the title compound, $(\text{C}_6\text{H}_{14}\text{N})_2[\text{SnCl}_6]$, is built of 4-methylpiperidinium cations, occupying special positions on the mirror plane, and hexachloridostannate(IV) anions on a special position of $2/m$ symmetry. The ions are linked *via* $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds into chains running along the b axis.

Related literature

For related literature, see: Shahzadi, Ali & Fettouhi (2006); Shahzadi, Ali, Bhatti *et al.* (2006).



Experimental

Crystal data

$(\text{C}_6\text{H}_{14}\text{N})_2[\text{SnCl}_6]$	$V = 1064.0(9) \text{ \AA}^3$
$M_r = 531.75$	$Z = 2$
Orthorhombic, $Pnmm$	Mo $K\alpha$ radiation
$a = 13.123(5) \text{ \AA}$	$\mu = 1.95 \text{ mm}^{-1}$
$b = 7.722(5) \text{ \AA}$	$T = 100(2) \text{ K}$
$c = 10.500(5) \text{ \AA}$	$0.25 \times 0.25 \times 0.25 \text{ mm}$

Data collection

Bruker APEX CCD area-detector diffractometer	1153 independent reflections
Absorption correction: none	1055 reflections with $I > 2\sigma(I)$
7975 measured reflections	$R_{\text{int}} = 0.050$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.018$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.040$	$\Delta\rho_{\text{max}} = 0.78 \text{ e \AA}^{-3}$
$S = 1.04$	$\Delta\rho_{\text{min}} = -0.29 \text{ e \AA}^{-3}$
1153 reflections	
65 parameters	

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N}\cdots\text{Cl1}$	0.88 (3)	2.63 (3)	3.258 (3)	129 (2)
$\text{N1}-\text{H2N}\cdots\text{Cl2}^{\text{i}}$	0.84 (3)	2.72 (2)	3.413 (2)	141.6 (5)
$\text{N1}-\text{H2N}\cdots\text{Cl2}^{\text{ii}}$	0.84 (3)	2.72 (2)	3.413 (2)	141.6 (5)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: YA2069).

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supporting information

Acta Cryst. (2008). E64, m573 [doi:10.1107/S1600536808007368]

Bis(4-methylpiperidinium) hexachloridostannate(IV)

Saira Shahzadi, Hamid Nawaz Khan, Saqib Ali and Madeleine Helliwell

S1. Comment

We report here the crystal structure of the title compound (I) as shown in Fig. 1. The Sn1—Cl distances span the range of 2.417 (1)–2.431 (1) Å; the N1—C1 bond is 1.500 (2) Å. The N—H···Cl bonds link the ions into chains along the *b* axis (Table 1, Fig. 2).

S2. Experimental

The 4-methyl-1-piperidine carbodithioic acid (3.0 g, 17.1 mmol) and tin tetrachloride pentahydrate (5.99 g, 17.1 mmol) were added to 100 ml of dry methanol in round bottom flask and stirred for 6 h. The resulting clear solution was evaporated at room temperature. Colourless crystals of the title compound were obtained after recrystallization in chloroform and *n*-hexane (1:1). Yield: 64%. m.p. 228°C.

S3. Refinement

H atoms bonded to C1—C3 were included in riding motion approximation in calculated positions with C—H distances of 0.99 Å and U_{iso} 1.2 times those of the parent atoms; those bonded to C4 and N1 were located in a difference Fourier map and refined isotropically with U_{iso} 1.2 times those of the parent atoms (C4 - H distances 0.93 (2) and 0.95 (3) Å and N1 - H 0.84 (3) and 0.88 (3) Å).

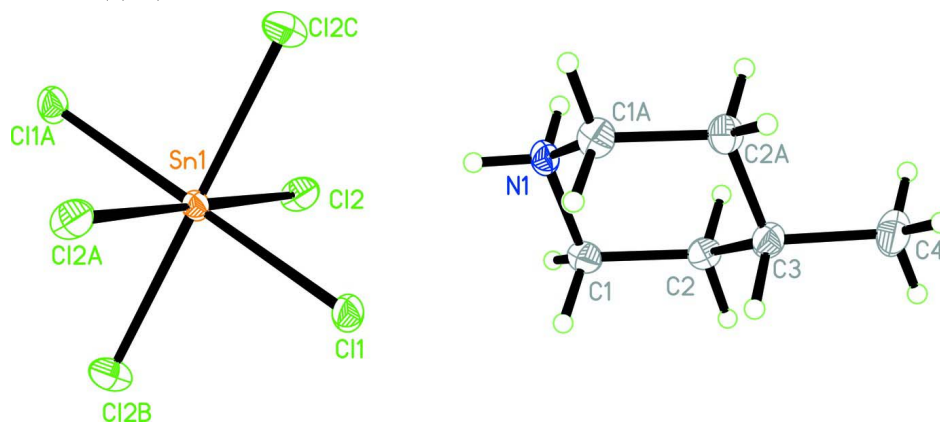


Figure 1

Structure of (I) with displacement ellipsoids drawn at the 50% probability level. The unlabelled atoms of the 4-methylpiperidinium cation are symmetry related (symmetry code $x, y, -z$). The unlabelled Cl atoms are symmetry related to Cl1 (symmetry code $-x, 2 - y, -z$) and Cl2 (symmetry codes $-x, 2 - y, -z$; $-x, 2 - y, z$; $x, y, -z$).

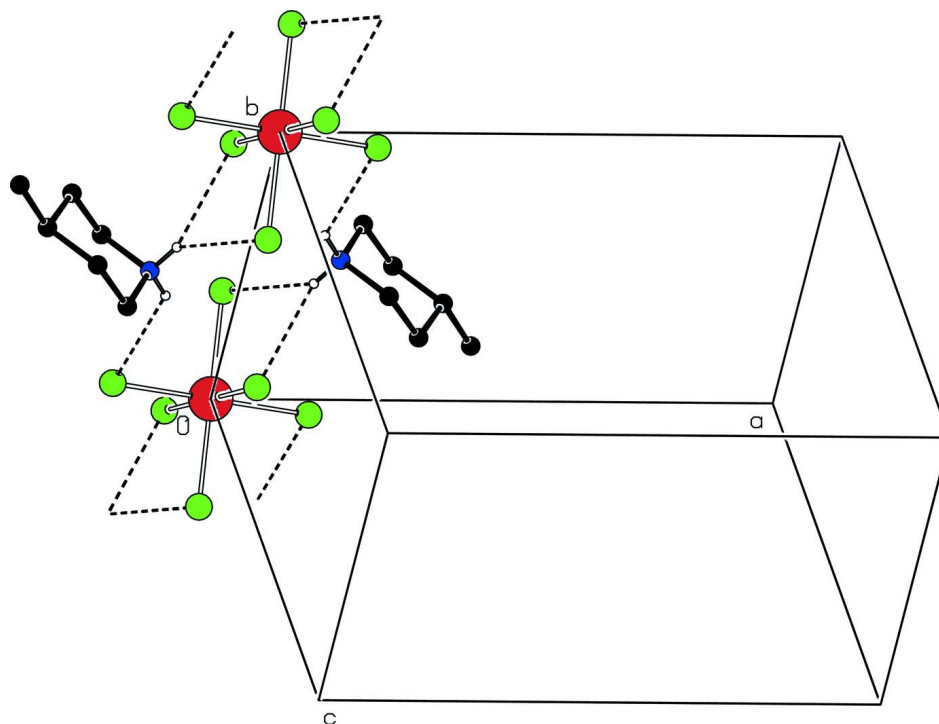


Figure 2

Fragment of the crystal packing of (I) showing chain along the *b* axis, viewed approximately along the diagonal of the *bc*-plane; H-bonds are shown as dashed lines.

Bis(4-methylpiperidinium) hexachloridostannate

Crystal data

$(C_6H_{14}N)_2[SnCl_6]$

$M_r = 531.75$

Orthorhombic, *Pnmm*

Hall symbol: $-P22n$

$a = 13.123 (5) \text{ \AA}$

$b = 7.722 (5) \text{ \AA}$

$c = 10.500 (5) \text{ \AA}$

$V = 1064.0 (9) \text{ \AA}^3$

$Z = 2$

$F(000) = 532$

$D_x = 1.660 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71069 \text{ \AA}$

Cell parameters from 3718 reflections

$\theta = 2.5\text{--}26.3^\circ$

$\mu = 1.95 \text{ mm}^{-1}$

$T = 100 \text{ K}$

Pyramidal, colourless

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

Data collection

Bruker APEX CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

7975 measured reflections

1153 independent reflections

1055 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.050$

$\theta_{\text{max}} = 26.3^\circ$, $\theta_{\text{min}} = 2.5^\circ$

$h = -16 \rightarrow 16$

$k = -9 \rightarrow 9$

$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.018$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.040$	$w = 1/[\sigma^2(F_o^2) + (0.0164P)^2]$
$S = 1.04$	where $P = (F_o^2 + 2F_c^2)/3$
1153 reflections	$(\Delta/\sigma)_{\max} < 0.001$
65 parameters	$\Delta\rho_{\max} = 0.78 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\min} = -0.29 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Sn1	0.0000	1.0000	0.0000	0.01428 (9)
Cl1	0.18119 (5)	0.94377 (8)	0.0000	0.02127 (15)
Cl2	-0.02412 (3)	0.77582 (6)	0.15973 (4)	0.02279 (12)
N1	0.16716 (17)	0.5226 (3)	0.0000	0.0187 (5)
H1N	0.128 (2)	0.615 (4)	0.0000	0.022*
H2N	0.130 (2)	0.434 (4)	0.0000	0.022*
C1	0.23016 (14)	0.5228 (2)	0.11919 (17)	0.0202 (4)
H1C	0.1850	0.5171	0.1946	0.024*
H1D	0.2701	0.6313	0.1242	0.024*
C2	0.30167 (13)	0.3686 (2)	0.11902 (17)	0.0194 (4)
H2A	0.2611	0.2607	0.1235	0.023*
H2B	0.3456	0.3736	0.1956	0.023*
C3	0.36885 (19)	0.3640 (3)	0.0000	0.0194 (6)
H3	0.4130	0.4696	0.0000	0.023*
C4	0.4374 (2)	0.2055 (4)	0.0000	0.0271 (7)
H4A	0.4780 (15)	0.200 (3)	-0.0726 (19)	0.032*
H4B	0.399 (2)	0.102 (4)	0.0000	0.032*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.01250 (13)	0.01126 (13)	0.01907 (14)	-0.00031 (9)	0.000	0.000
Cl1	0.0136 (3)	0.0153 (3)	0.0349 (4)	0.0005 (2)	0.000	0.000

C12	0.0214 (2)	0.0228 (2)	0.0242 (3)	-0.00568 (18)	-0.00453 (18)	0.00756 (19)
N1	0.0165 (11)	0.0122 (12)	0.0274 (13)	0.0012 (9)	0.000	0.000
C1	0.0205 (10)	0.0199 (10)	0.0203 (10)	-0.0007 (8)	-0.0011 (7)	-0.0040 (8)
C2	0.0189 (10)	0.0200 (10)	0.0192 (10)	0.0010 (8)	-0.0028 (8)	-0.0011 (8)
C3	0.0151 (13)	0.0189 (14)	0.0241 (15)	0.0003 (10)	0.000	0.000
C4	0.0239 (16)	0.0321 (18)	0.0252 (17)	0.0102 (13)	0.000	0.000

Geometric parameters (Å, °)

Sn1—C11	2.4170 (11)	C1—H1C	0.9900
Sn1—C11 ⁱ	2.4170 (11)	C1—H1D	0.9900
Sn1—C12 ⁱⁱ	2.4310 (11)	C2—C3	1.530 (2)
Sn1—C12 ⁱⁱⁱ	2.4310 (11)	C2—H2A	0.9900
Sn1—C12 ⁱ	2.4310 (11)	C2—H2B	0.9900
Sn1—C12	2.4310 (11)	C3—C4	1.519 (4)
N1—C1 ⁱⁱⁱ	1.500 (2)	C3—C2 ⁱⁱⁱ	1.530 (2)
N1—C1	1.500 (2)	C3—H3	1.0000
N1—H1N	0.88 (3)	C4—H4A	0.931 (19)
N1—H2N	0.84 (3)	C4—H4B	0.95 (3)
C1—C2	1.516 (2)		
C11—Sn1—C11 ⁱ	180.000 (5)	N1—C1—C2	109.83 (15)
C11—Sn1—C12 ⁱⁱ	89.990 (19)	N1—C1—H1C	109.7
C11 ⁱ —Sn1—C12 ⁱⁱ	90.010 (19)	C2—C1—H1C	109.7
C11—Sn1—C12 ⁱⁱⁱ	90.010 (19)	N1—C1—H1D	109.7
C11 ⁱ —Sn1—C12 ⁱⁱⁱ	89.990 (19)	C2—C1—H1D	109.7
C12 ⁱⁱ —Sn1—C12 ⁱⁱⁱ	180.0	H1C—C1—H1D	108.2
C11—Sn1—C12 ⁱ	89.990 (19)	C1—C2—C3	112.08 (16)
C11 ⁱ —Sn1—C12 ⁱ	90.010 (19)	C1—C2—H2A	109.2
C12 ⁱⁱ —Sn1—C12 ⁱ	87.24 (5)	C3—C2—H2A	109.2
C12 ⁱⁱⁱ —Sn1—C12 ⁱ	92.76 (5)	C1—C2—H2B	109.2
C11—Sn1—C12	90.010 (19)	C3—C2—H2B	109.2
C11 ⁱ —Sn1—C12	89.990 (19)	H2A—C2—H2B	107.9
C12 ⁱⁱ —Sn1—C12	92.76 (5)	C4—C3—C2 ⁱⁱⁱ	111.10 (15)
C12 ⁱⁱⁱ —Sn1—C12	87.24 (5)	C4—C3—C2	111.10 (15)
C12 ⁱ —Sn1—C12	180.0	C2 ⁱⁱⁱ —C3—C2	109.6 (2)
C1 ⁱⁱⁱ —N1—C1	113.1 (2)	C4—C3—H3	108.3
C1 ⁱⁱⁱ —N1—H1N	108.7 (8)	C2 ⁱⁱⁱ —C3—H3	108.3
C1—N1—H1N	108.7 (8)	C2—C3—H3	108.3
C1 ⁱⁱⁱ —N1—H2N	108.6 (10)	C3—C4—H4A	112.1 (14)
C1—N1—H2N	108.6 (10)	C3—C4—H4B	111.2 (18)
H1N—N1—H2N	109 (3)	H4A—C4—H4B	105.6 (17)
C1 ⁱⁱⁱ —N1—C1—C2	-56.8 (2)	C1—C2—C3—C4	-178.20 (18)
N1—C1—C2—C3	55.7 (2)	C1—C2—C3—C2 ⁱⁱⁱ	-55.1 (2)

Symmetry codes: (i) $-x, -y+2, -z$; (ii) $-x, -y+2, z$; (iii) $x, y, -z$.

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1N \cdots C11	0.88 (3)	2.63 (3)	3.258 (3)	129 (2)
N1—H2N \cdots C12 ^{iv}	0.84 (3)	2.72 (2)	3.413 (2)	142 (1)
N1—H2N \cdots C12 ^v	0.84 (3)	2.72 (2)	3.413 (2)	142 (1)

Symmetry codes: (iv) $-x, -y+1, -z$; (v) $-x, -y+1, z$.