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Bis(2-methylpiperidinium) pentachloridoantimonate(III)

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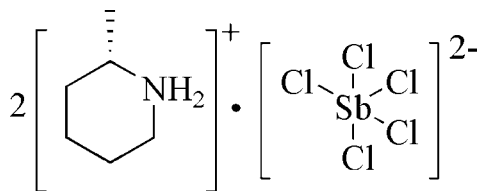
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.033; wR factor = 0.065; data-to-parameter ratio = 24.1.

The asymmetric unit of the title compound, $(\text{C}_6\text{H}_{14}\text{N})_2[\text{SbCl}_5]$, contains one cation and half of the anion on a special position (specifically, the Sb^{III} ion and three chloride anions are situated on a mirror plane). In the $[\text{SbCl}_5]^{2-}$ unit, the Sb^{III} ion is coordinated by five chloride anions [$\text{Sb}-\text{Cl} = 2.3721$ (11)– 2.6656 (12) Å] in a distorted square-pyramidal geometry. However, one chloride anion from a neighbouring $[\text{SbCl}_5]^{2-}$ unit provides a short $\text{Sb}\cdots\text{Cl}$ contact of 3.3600 (12) Å and completes the Sb coordination environment up to an elongated octahedron. In the crystal, $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds link cations and anions into columns propagating along [100].

Related literature

For the crystal structure of bis(4-benzylpiperidinium) pentachloridoantimonate(III), see: Marsh (1995). For background to ferroelectric metal-organic frameworks, see: Fu *et al.* (2009); Ye *et al.* (2006); Zhang *et al.* (2008, 2010).



Experimental

Crystal data

 $(\text{C}_6\text{H}_{14}\text{N})_2[\text{SbCl}_5]$
 $M_r = 499.36$

 Orthorhombic, $Pnma$
 $a = 7.5995$ (15) Å

 $b = 23.165$ (5) Å

 $c = 11.453$ (2) Å

 $V = 2016.2$ (7) Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 2.03$ mm⁻¹
 $T = 293$ K

 $0.28 \times 0.25 \times 0.21$ mm

Data collection

Rigaku Mercury70 CCD diffractometer

 Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)

 $T_{\text{min}} = 0.421$, $T_{\text{max}} = 0.558$

19754 measured reflections

2361 independent reflections

 1938 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.061$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.065$
 $S = 1.08$

2361 reflections

98 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.58$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.52$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1D}\cdots\text{Cl3}^{\text{i}}$	0.90	2.40	3.226 (3)	153
$\text{N1}-\text{H1E}\cdots\text{Cl1}^{\text{ii}}$	0.90	2.48	3.373 (3)	173

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

Data collection: *SCXmini Benchtop Crystallography System Software* (Rigaku, 2006); cell refinement: *SCXmini Benchtop Crystallography System Software*; data reduction: *SCXmini Benchtop Crystallography System Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *SHELXL97*.

This work was supported by Southeast University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5279).

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supplementary materials

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Bis(2-methylpiperidinium) pentachloridoantimonate(III)

Qian Xu

Comment

As a contribution to a search for new ferroelectric materials (Fu *et al.*, 2009; Ye *et al.*, 2006; Zhang *et al.*, 2008; Zhang *et al.*, 2010), we have synthesized the title compound, (I). Herewith we present its crystal structure.

The asymmetric unit of (I), $2(\text{C}_6\text{H}_{14}\text{N})^+[\text{SbCl}_5]^{2-}$, contains one cation and one-half of the anion in a special position (Fig. 1). The Sb1 atoms coordinated in a slightly distorted square-pyramidal geometry by five Cl atoms and distance of the top Cl2 and Sb1 is 2.3721 (11) Å much shorter than the mean values of other Sb—Cl[2.636 (11) Å]. The bond angles around the Sb1 are in the range 84.93 (2)–91.454 (19)° and correspond to those observed in the related compound $2(\text{C}_{12}\text{H}_{18}\text{N})^+[\text{SbCl}_5]^{2-}$ (Marsh, 1995).

In the crystal structure, intermolecular N—H···Cl hydrogen bonds (Table 1) link cations and anions into columns propagated in [100] (Fig. 2). In the title compound, no dielectric anomalies were observed in the range from 190 K to its melting point, which is more than 357 K.

Experimental

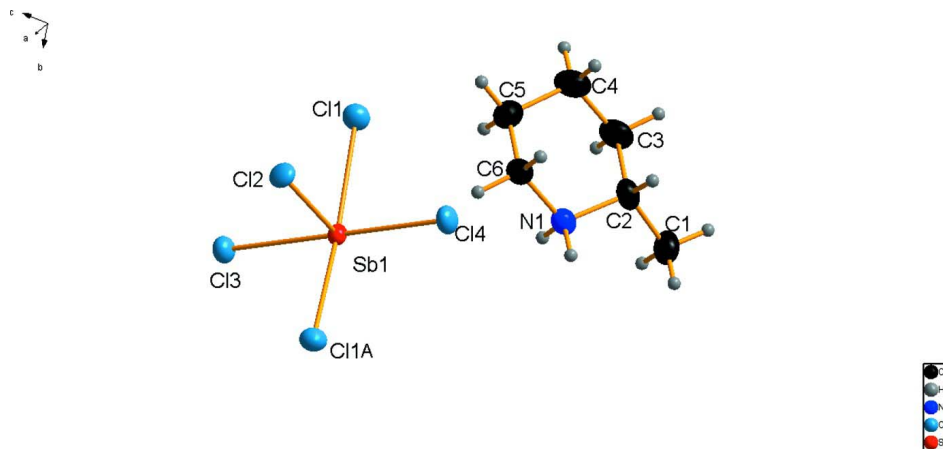
The mixture of SbCl_3 (1.1 g, 5 mmol) and 2-methylpiperidine (1.05 g, 10 mmol) in hydrochloric acid solution was stirred for several minutes at room temperature. Colourless crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of the solution at room temperature over 2 weeks.

Refinement

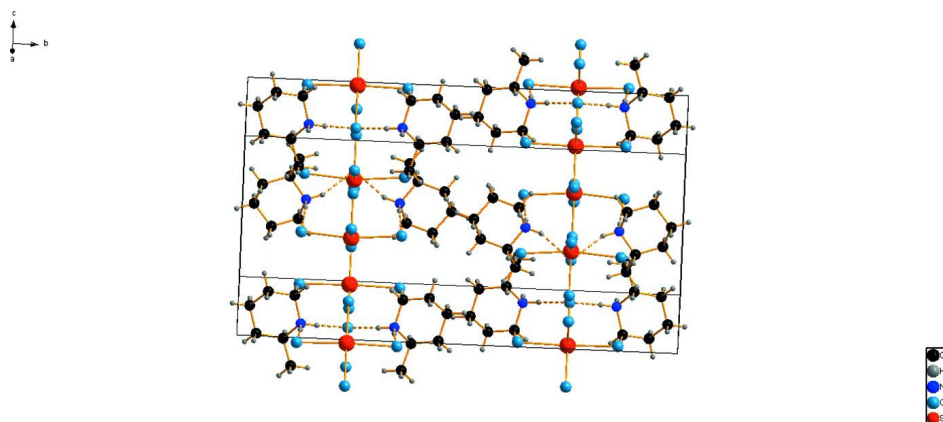
All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H = 0.93–0.98 Å and N—H = 0.90 Å, and with $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5 U_{\text{iso}}(\text{C}, \text{N})$.

Computing details

Data collection: *SCXmini Benchtop Crystallography System Software* (Rigaku, 2006); cell refinement: *SCXmini Benchtop Crystallography System Software* (Rigaku, 2006); data reduction: *SCXmini Benchtop Crystallography System Software* (Rigaku, 2006); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).


Figure 1

The molecular structure of the (I), with the displacement ellipsoids drawn at the 30% probability level [symmetry code: (A) $x, 0.5 - y, z$].


Figure 2

A portion of the packing diagram with hydrogen bonds shown as dashed lines.

Bis(2-methylpiperidinium) pentachloridoantimonate(III)

Crystal data

$(C_6H_{14}N)_2[SbCl_5]$

$M_r = 499.36$

Orthorhombic, $Pnma$

Hall symbol: $-P\ 2ac\ 2n$

$a = 7.5995\ (15)\ \text{\AA}$

$b = 23.165\ (5)\ \text{\AA}$

$c = 11.453\ (2)\ \text{\AA}$

$V = 2016.2\ (7)\ \text{\AA}^3$

$Z = 4$

$F(000) = 1000$

$D_x = 1.645\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2361 reflections

$\theta = 2.2\text{--}27.5^\circ$

$\mu = 2.03\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Block, colourless

$0.28 \times 0.25 \times 0.21\ \text{mm}$

Data collection

Rigaku Mercury70 CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

Absorption correction: multi-scan

(*CrystalClear*; Rigaku, 2005)

$T_{\min} = 0.421, T_{\max} = 0.558$

19754 measured reflections
 2361 independent reflections
 1938 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.061$

$\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 3.2^\circ$
 $h = -9 \rightarrow 9$
 $k = -30 \rightarrow 30$
 $l = -14 \rightarrow 14$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.065$
 $S = 1.08$
 2361 reflections
 98 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0181P)^2 + 1.8699P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.58 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.52 \text{ e } \text{\AA}^{-3}$

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	0.8199 (5)	0.1245 (2)	-0.1957 (3)	0.0703 (12)
H1A	0.7817	0.1631	-0.2129	0.105*
H1B	0.7841	0.0991	-0.2575	0.105*
H1C	0.9457	0.1238	-0.1887	0.105*
C2	0.7387 (5)	0.10494 (17)	-0.0830 (3)	0.0554 (10)
H2	0.6106	0.1081	-0.0900	0.066*
C3	0.7834 (6)	0.04338 (18)	-0.0511 (4)	0.0750 (13)
H3A	0.7347	0.0177	-0.1098	0.090*
H3B	0.9103	0.0388	-0.0519	0.090*
C4	0.7143 (7)	0.0260 (2)	0.0672 (4)	0.0866 (15)
H4A	0.7536	-0.0128	0.0855	0.104*
H4B	0.5866	0.0259	0.0659	0.104*
C5	0.7785 (6)	0.06724 (18)	0.1599 (4)	0.0707 (12)
H5A	0.7284	0.0566	0.2348	0.085*
H5B	0.9055	0.0647	0.1661	0.085*
C6	0.7275 (5)	0.12723 (16)	0.1310 (3)	0.0552 (10)
H6A	0.7741	0.1534	0.1895	0.066*
H6B	0.6002	0.1306	0.1314	0.066*
N1	0.7977 (4)	0.14369 (12)	0.0122 (3)	0.0499 (7)
H1D	0.7632	0.1799	-0.0044	0.060*
H1E	0.9160	0.1435	0.0147	0.060*
Cl1	0.73805 (11)	0.13613 (4)	0.45405 (8)	0.0508 (2)

C12	0.58634 (15)	0.2500	0.61136 (10)	0.0463 (3)
C13	1.05345 (15)	0.2500	0.57047 (11)	0.0489 (3)
C14	0.48085 (16)	0.2500	0.31467 (11)	0.0529 (3)
Sb1	0.76256 (3)	0.2500	0.44041 (2)	0.03097 (9)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.070 (3)	0.095 (3)	0.046 (2)	-0.005 (2)	-0.001 (2)	-0.007 (2)
C2	0.047 (2)	0.065 (2)	0.054 (2)	0.0012 (18)	-0.0107 (18)	-0.0133 (18)
C3	0.090 (3)	0.048 (2)	0.088 (3)	-0.004 (2)	0.006 (3)	-0.017 (2)
C4	0.111 (4)	0.051 (3)	0.098 (4)	-0.014 (3)	0.008 (3)	-0.005 (3)
C5	0.077 (3)	0.071 (3)	0.064 (3)	-0.007 (2)	-0.003 (2)	0.012 (2)
C6	0.065 (2)	0.051 (2)	0.050 (2)	-0.0070 (19)	0.0054 (19)	-0.0070 (17)
N1	0.0522 (17)	0.0429 (17)	0.0547 (18)	0.0026 (14)	-0.0054 (15)	-0.0050 (14)
Cl1	0.0496 (5)	0.0453 (5)	0.0576 (5)	0.0051 (4)	0.0006 (4)	-0.0039 (4)
Cl2	0.0521 (7)	0.0532 (7)	0.0336 (6)	0.000	0.0144 (5)	0.000
Cl3	0.0438 (6)	0.0535 (7)	0.0493 (7)	0.000	-0.0117 (6)	0.000
Cl4	0.0481 (7)	0.0584 (8)	0.0523 (7)	0.000	-0.0189 (6)	0.000
Sb1	0.02942 (15)	0.03721 (16)	0.02627 (14)	0.000	0.00072 (12)	0.000

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

C1—C2	1.500 (5)	C5—C6	1.480 (5)
C1—H1A	0.9600	C5—H5A	0.9700
C1—H1B	0.9600	C5—H5B	0.9700
C1—H1C	0.9600	C6—N1	1.511 (5)
C2—N1	1.481 (4)	C6—H6A	0.9700
C2—C3	1.511 (6)	C6—H6B	0.9700
C2—H2	0.9800	N1—H1D	0.9000
C3—C4	1.509 (6)	N1—H1E	0.9000
C3—H3A	0.9700	Cl1—Sb1	2.6491 (10)
C3—H3B	0.9700	Cl2—Sb1	2.3721 (11)
C4—C5	1.510 (6)	Cl3—Sb1	2.6656 (12)
C4—H4A	0.9700	Cl4—Sb1	2.5802 (12)
C4—H4B	0.9700	Sb1—Cl1 ⁱ	2.6491 (10)
C2—C1—H1A	109.5	C4—C5—H5A	109.5
C2—C1—H1B	109.5	C6—C5—H5B	109.5
H1A—C1—H1B	109.5	C4—C5—H5B	109.5
C2—C1—H1C	109.5	H5A—C5—H5B	108.1
H1A—C1—H1C	109.5	C5—C6—N1	110.3 (3)
H1B—C1—H1C	109.5	C5—C6—H6A	109.6
N1—C2—C1	109.0 (3)	N1—C6—H6A	109.6
N1—C2—C3	109.0 (3)	C5—C6—H6B	109.6
C1—C2—C3	113.6 (3)	N1—C6—H6B	109.6
N1—C2—H2	108.4	H6A—C6—H6B	108.1
C1—C2—H2	108.4	C2—N1—C6	113.8 (3)
C3—C2—H2	108.4	C2—N1—H1D	108.8
C4—C3—C2	113.0 (4)	C6—N1—H1D	108.8

C4—C3—H3A	109.0	C2—N1—H1E	108.8
C2—C3—H3A	109.0	C6—N1—H1E	108.8
C4—C3—H3B	109.0	H1D—N1—H1E	107.7
C2—C3—H3B	109.0	Cl2—Sb1—Cl4	89.56 (5)
H3A—C3—H3B	107.8	Cl2—Sb1—Cl1	84.93 (2)
C3—C4—C5	110.5 (4)	Cl4—Sb1—Cl1	88.54 (2)
C3—C4—H4A	109.6	Cl2—Sb1—Cl1 ⁱ	84.93 (2)
C5—C4—H4A	109.6	Cl4—Sb1—Cl1 ⁱ	88.542 (19)
C3—C4—H4B	109.6	Cl1—Sb1—Cl1 ⁱ	169.47 (4)
C5—C4—H4B	109.6	Cl2—Sb1—Cl3	90.40 (4)
H4A—C4—H4B	108.1	Cl4—Sb1—Cl3	179.96 (4)
C6—C5—C4	110.6 (4)	Cl1—Sb1—Cl3	91.454 (19)
C6—C5—H5A	109.5	Cl1 ⁱ —Sb1—Cl3	91.454 (19)
N1—C2—C3—C4	53.2 (5)	C4—C5—C6—N1	-56.7 (5)
C1—C2—C3—C4	175.0 (4)	C1—C2—N1—C6	-178.6 (3)
C2—C3—C4—C5	-55.2 (6)	C3—C2—N1—C6	-54.0 (4)
C3—C4—C5—C6	56.5 (5)	C5—C6—N1—C2	57.2 (4)

Symmetry code: (i) $x, -y+1/2, z$.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1D \cdots Cl3 ⁱⁱ	0.90	2.40	3.226 (3)	153
N1—H1E \cdots Cl1 ⁱⁱⁱ	0.90	2.48	3.373 (3)	173

Symmetry codes: (ii) $x-1/2, y, -z+1/2$; (iii) $x+1/2, y, -z+1/2$.