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N-Methylpiperazinediium penta-chloridoantimonate(III) monohydrate

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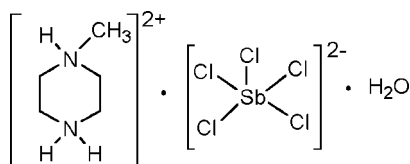
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Key indicators: single-crystal X-ray study; $T = 273$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.024; wR factor = 0.054; data-to-parameter ratio = 20.0.

The asymmetric unit of the title compound, $(\text{C}_5\text{H}_{14}\text{N}_2)\text{[SbCl}_5\text{]}\cdot\text{H}_2\text{O}$, consists of an *N*-methylpiperazinediium cation, a pentachloridoantimonate anion with the Sb^{III} ion in a slightly distorted square-pyramidal coordination environment, and one solvent water molecule. The crystal structure is stabilized by intermolecular $\text{N}-\text{H}\cdots\text{Cl}$, $\text{O}-\text{H}\cdots\text{Cl}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For related literature, see: Baker & Williams (1978); Bujak & Zaleski (1999); Clemente & Marzotto (2003); Feng *et al.* (2007); Knodler *et al.* (1988); Linden *et al.* (1999); Marsh (1995).



Experimental

Crystal data

$(\text{C}_5\text{H}_{14}\text{N}_2)\text{[SbCl}_5\text{]}\cdot\text{H}_2\text{O}$
 $M_r = 419.20$
 Monoclinic, $P2_1/c$
 $a = 9.600$ (4) Å
 $b = 7.934$ (3) Å
 $c = 19.966$ (6) Å
 $\beta = 106.765$ (16)°

$V = 1456.1$ (9) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 2.79$ mm⁻¹
 $T = 273$ (2) K
 $0.33 \times 0.18 \times 0.14$ mm

Data collection

Bruker SMART CCD diffractometer
 Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
 $T_{\text{min}} = 0.460$, $T_{\text{max}} = 0.696$

7167 measured reflections
 2577 independent reflections
 2400 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.024$
 $wR(F^2) = 0.054$
 $S = 1.07$
 2577 reflections

129 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.35$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.48$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Sb1—Cl2	2.4110 (10)	Sb1—Cl4	2.7446 (11)
Sb1—Cl3	2.4623 (10)	Sb1—Cl5	2.9112 (11)
Sb1—Cl1	2.5538 (11)		
Cl2—Sb1—Cl3	89.29 (4)	Cl1—Sb1—Cl4	174.79 (3)
Cl2—Sb1—Cl1	90.76 (4)	Cl5—Sb1—Cl1	92.15(4)
Cl3—Sb1—Cl1	93.67 (4)	Cl5—Sb1—Cl2	84.50(4)
Cl2—Sb1—Cl4	91.71 (4)	Cl5—Sb1—Cl3	171.54(4)
Cl3—Sb1—Cl4	90.95 (4)	Cl5—Sb1—Cl4	83.52(4)

Table 2

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>
O1—H1F \cdots Cl1 ⁱ	0.82	2.57	3.346 (4)	159
O1—H1G \cdots Cl4	0.82	2.51	3.223 (5)	147
N1—H1A \cdots Cl5 ⁱⁱ	0.90	2.43	3.179 (4)	141
N1—H1B \cdots O1 ⁱⁱⁱ	0.90	1.91	2.801 (5)	168
N2—H2 \cdots Cl5	0.91	2.28	3.133 (5)	157

Symmetry codes: (i) $x, y + 1, z$; (ii) $-x + 2, y - \frac{1}{2}, -z + \frac{3}{2}$; (iii) $x, -y + \frac{3}{2}, z - \frac{1}{2}$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINTE* (Bruker, 2000); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2556).

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supplementary materials

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***N*-Methylpiperazinediium pentachloridoantimonate(III) monohydrate**

C. Shen-Tu, H. Y. Li, X. J. Ma, W. Huang and Z. M. Jin

Comment

Halogenoantimonates constitute a group of salts in which a number of compounds have been reported (*e.g.* Feng *et al.*, 2007; Bujak & Zaleski, 1999; Knodler *et al.*, 1988; Baker & Williams, 1978 and see: Clemente & Marzotto (2003); Marsh *et al.* (1995) for corrected space groups of some of these types of compounds). In our laboratory, a compound containing pentachloridoantimonate has been synthesized, its crystal structure is reported herein.

As shown in Fig. 1, an ion pair consisting of *N*-methylpiperazinium and $(\text{SbCl}_5)^{2+}$, and one water molecule comprise the formula unit. In the selected asymmetric unit The SbCl_5 anion is linked to *N*-methylpiperazinium and the water molecule by $\text{N—H}\cdots\text{Cl}$ and $\text{O—H}\cdots\text{Cl}$ hydrogen bonds.

The Sb atom is coordinated by five Cl atoms, with Sb—Cl distances ranging from 2.4110 (10) to 2.9112 (11) Å. The Sb—Cl distances are slightly different to the values of 2.499 (4)–2.768 (4) Å reported by Bujak & Zaleski (1999). In the title compound the difference between the longest bond (Sb1—Cl5) and shortest bond (Sb1—Cl2) is *ca* 0.50 Å. The slight deformation of the square-pyramidal coordination environment may be attributed to the presence of relatively strong $\text{N—H}\cdots\text{Cl}$ hydrogen bonds. The atoms Cl1/Cl3/Cl4/Cl5 form the basal plane, while atom Cl2 is the apical atom. The structure of the anion is similar to that of the $(\text{TiCl}_5)^{2-}$ anion (Linden *et al.*, 1999).

The six-membered piperazine ring is in chair conformation. The crystal structure is stabilized by $\text{N—H}\cdots\text{Cl}$, $\text{O—H}\cdots\text{Cl}$ and $\text{N—H}\cdots\text{O}$ hydrogen bonds, and by weak $\text{C—H}\cdots\text{Cl}$ and $\text{C—H}\cdots\text{O}$ hydrogen bonds (Fig. 2).

Experimental

SbCl_3 , *N*-methylpiperazine and 30% aqueous HCl in a molar ratio of 1:1:1 were mixed and dissolved in sufficient ethanol by heating to 373 K forming a clear solution. The reaction mixture was cooled slowly to room temperature, crystals of the title compound were formed, collected and washed with dilute aqueous HCl.

Refinement

H atoms were included in calculated positions with $\text{O—H} = 0.82$, $\text{N—H} = 0.90 - 0.91$ and $\text{C—H} = 0.96 - 0.97$ Å and included in the riding-model approximation with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N}, \text{O})$ or $1.5U_{\text{eq}}(\text{C})$ for methyl H atoms.

Figures

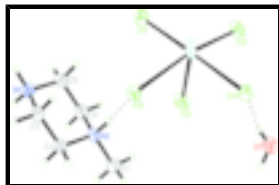


Fig. 1. The asymmetric unit of the title compound with atom labels, and 30% probability displacement ellipsoids. Hydrogen bonds are illustrated as dashed lines.

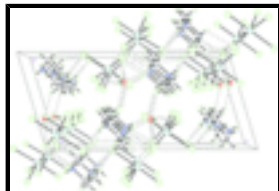


Fig. 2. The packing viewed approximately along the *b* axis. Hydrogen bonds are drawn as dashed lines.

N-Methylpiperazinediium pentachloridoantimonate(III) monohydrate

Crystal data

(C₅H₁₄N₂)[SbCl₅]·H₂O

M_r = 419.20

Monoclinic, *P*2₁/*c*

Hall symbol: -*P* 2ybc

a = 9.600 (4) Å

b = 7.934 (3) Å

c = 19.966 (6) Å

β = 106.765 (16)°

V = 1456.1 (9) Å³

Z = 4

*F*₀₀₀ = 816

D_x = 1.912 Mg m⁻³

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 3256 reflections

θ = 2.1–25.0°

μ = 2.79 mm⁻¹

T = 273 (2) K

Block, brown

0.33 × 0.18 × 0.14 mm

Data collection

Bruker SMART CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

T = 273(2) K

φ and ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2000)

*T*_{min} = 0.460, *T*_{max} = 0.696

7167 measured reflections

2577 independent reflections

2400 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.021

θ_{max} = 25.0°

θ_{min} = 2.1°

h = -11→11

k = -9→9

l = -16→23

Refinement

Refinement on *F*²

Least-squares matrix: full

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$R[F^2 > 2\sigma(F^2)] = 0.024$	$w = 1/[\sigma^2(F_o^2) + (0.0213P)^2 + 0.8984P]$
$wR(F^2) = 0.054$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{\max} = 0.002$
2577 reflections	$\Delta\rho_{\max} = 0.35 \text{ e } \text{\AA}^{-3}$
129 parameters	$\Delta\rho_{\min} = -0.48 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL, $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.0050 (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 $F^2 > 2\sigma(F^2)$ 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	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.6846 (3)	1.0018 (3)	0.96651 (12)	0.0597 (6)
H1F	0.6811	1.0770	0.9378	0.072*
H1G	0.7246	0.9149	0.9607	0.072*
N1	0.8129 (3)	0.3813 (3)	0.60274 (12)	0.0430 (6)
H1A	0.8580	0.2814	0.6042	0.052*
H1B	0.7664	0.4045	0.5577	0.052*
N2	0.7418 (2)	0.6714 (3)	0.67442 (12)	0.0386 (5)
H2	0.7903	0.6427	0.7192	0.046*
C1	0.6710 (4)	0.8384 (4)	0.67596 (18)	0.0546 (8)
H1C	0.7433	0.9196	0.6988	0.082*
H1D	0.5999	0.8284	0.7011	0.082*
H1E	0.6242	0.8747	0.6290	0.082*
C2	0.9227 (3)	0.5143 (4)	0.63140 (17)	0.0432 (7)
H2A	0.9879	0.5239	0.6024	0.052*
H2B	0.9800	0.4835	0.6783	0.052*
C3	0.8497 (3)	0.6806 (4)	0.63348 (16)	0.0434 (7)
H3A	0.9230	0.7645	0.6544	0.052*
H3B	0.8006	0.7163	0.5861	0.052*
C4	0.6329 (3)	0.5373 (4)	0.64511 (18)	0.0490 (8)
H4A	0.5765	0.5684	0.5981	0.059*
H4B	0.5665	0.5278	0.6735	0.059*
C5	0.7048 (3)	0.3696 (4)	0.64308 (18)	0.0524 (8)

supplementary materials

H5A	0.7533	0.3331	0.6904	0.063*
H5B	0.6315	0.2864	0.6216	0.063*
Sb1	0.82184 (2)	0.48123 (2)	0.906548 (10)	0.03847 (9)
Cl1	0.71738 (9)	0.22687 (12)	0.83017 (4)	0.0586 (2)
Cl2	0.63328 (9)	0.65958 (12)	0.83543 (5)	0.0628 (2)
Cl3	0.67147 (9)	0.43460 (13)	0.98606 (5)	0.0607 (2)
Cl4	0.95679 (9)	0.75059 (10)	0.98509 (4)	0.0497 (2)
Cl5	0.98660 (8)	0.58815 (10)	0.81238 (4)	0.04343 (18)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0592 (15)	0.0667 (16)	0.0495 (14)	0.0137 (11)	0.0096 (12)	0.0028 (11)
N1	0.0458 (14)	0.0450 (14)	0.0374 (13)	0.0000 (11)	0.0106 (11)	-0.0041 (11)
N2	0.0367 (13)	0.0475 (14)	0.0324 (13)	0.0034 (11)	0.0114 (10)	0.0027 (11)
C1	0.053 (2)	0.0537 (19)	0.061 (2)	0.0157 (16)	0.0214 (17)	0.0051 (17)
C2	0.0366 (16)	0.0524 (18)	0.0428 (18)	-0.0024 (13)	0.0150 (14)	-0.0043 (14)
C3	0.0448 (17)	0.0477 (17)	0.0432 (17)	-0.0071 (14)	0.0213 (14)	-0.0033 (14)
C4	0.0324 (15)	0.063 (2)	0.052 (2)	-0.0043 (14)	0.0140 (14)	-0.0037 (16)
C5	0.0505 (19)	0.0527 (19)	0.059 (2)	-0.0117 (15)	0.0233 (16)	-0.0005 (17)
Sb1	0.03744 (13)	0.04726 (14)	0.03172 (13)	0.00487 (8)	0.01155 (9)	0.00274 (8)
Cl1	0.0575 (5)	0.0664 (5)	0.0521 (5)	-0.0089 (4)	0.0163 (4)	-0.0123 (4)
Cl2	0.0541 (5)	0.0779 (6)	0.0553 (5)	0.0234 (4)	0.0140 (4)	0.0178 (4)
Cl3	0.0572 (5)	0.0802 (6)	0.0548 (5)	0.0108 (4)	0.0323 (4)	0.0108 (5)
Cl4	0.0565 (5)	0.0523 (4)	0.0457 (4)	0.0030 (4)	0.0232 (4)	-0.0021 (4)
Cl5	0.0432 (4)	0.0520 (4)	0.0346 (4)	0.0007 (3)	0.0104 (3)	-0.0005 (3)

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

O1—H1F	0.8219	C2—H2A	0.9700
O1—H1G	0.8128	C2—H2B	0.9700
N1—C2	1.485 (4)	C3—H3A	0.9700
N1—C5	1.488 (4)	C3—H3B	0.9700
N1—H1A	0.9000	C4—C5	1.505 (4)
N1—H1B	0.9000	C4—H4A	0.9700
N2—C4	1.488 (4)	C4—H4B	0.9700
N2—C1	1.493 (4)	C5—H5A	0.9700
N2—C3	1.496 (3)	C5—H5B	0.9700
N2—H2	0.9100	Sb1—Cl2	2.4110 (10)
C1—H1C	0.9600	Sb1—Cl3	2.4623 (10)
C1—H1D	0.9600	Sb1—Cl1	2.5538 (11)
C1—H1E	0.9600	Sb1—Cl4	2.7446 (11)
C2—C3	1.500 (4)	Sb1—Cl5	2.9112 (11)
H1F—O1—H1G	116.3	C2—C3—H3A	109.2
C2—N1—C5	111.3 (2)	N2—C3—H3B	109.2
C2—N1—H1A	109.4	C2—C3—H3B	109.2
C5—N1—H1A	109.4	H3A—C3—H3B	107.9
C2—N1—H1B	109.4	N2—C4—C5	111.5 (2)

C5—N1—H1B	109.4	N2—C4—H4A	109.3
H1A—N1—H1B	108.0	C5—C4—H4A	109.3
C4—N2—C1	111.7 (2)	N2—C4—H4B	109.3
C4—N2—C3	109.8 (2)	C5—C4—H4B	109.3
C1—N2—C3	111.1 (2)	H4A—C4—H4B	108.0
C4—N2—H2	108.0	N1—C5—C4	110.9 (3)
C1—N2—H2	108.0	N1—C5—H5A	109.5
C3—N2—H2	108.0	C4—C5—H5A	109.5
N2—C1—H1C	109.5	N1—C5—H5B	109.5
N2—C1—H1D	109.5	C4—C5—H5B	109.5
H1C—C1—H1D	109.5	H5A—C5—H5B	108.0
N2—C1—H1E	109.5	Cl2—Sb1—Cl3	89.29 (4)
H1C—C1—H1E	109.5	Cl2—Sb1—Cl1	90.76 (4)
H1D—C1—H1E	109.5	Cl3—Sb1—Cl1	93.67 (4)
N1—C2—C3	110.5 (2)	Cl2—Sb1—Cl4	91.71 (4)
N1—C2—H2A	109.5	Cl3—Sb1—Cl4	90.95 (4)
C3—C2—H2A	109.5	Cl1—Sb1—Cl4	174.79 (3)
N1—C2—H2B	109.5	Cl5—Sb1—Cl1	92.15 (4)
C3—C2—H2B	109.5	Cl5—Sb1—Cl2	84.50 (4)
H2A—C2—H2B	108.1	Cl5—Sb1—Cl3	171.54 (4)
N2—C3—C2	112.0 (2)	Cl5—Sb1—Cl4	83.52 (4)
N2—C3—H3A	109.2		
C5—N1—C2—C3	-55.7 (3)	C1—N2—C4—C5	179.5 (3)
C4—N2—C3—C2	-56.1 (3)	C3—N2—C4—C5	55.8 (3)
C1—N2—C3—C2	179.8 (3)	C2—N1—C5—C4	55.9 (3)
N1—C2—C3—N2	56.2 (3)	N2—C4—C5—N1	-56.3 (4)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
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Fig. 1

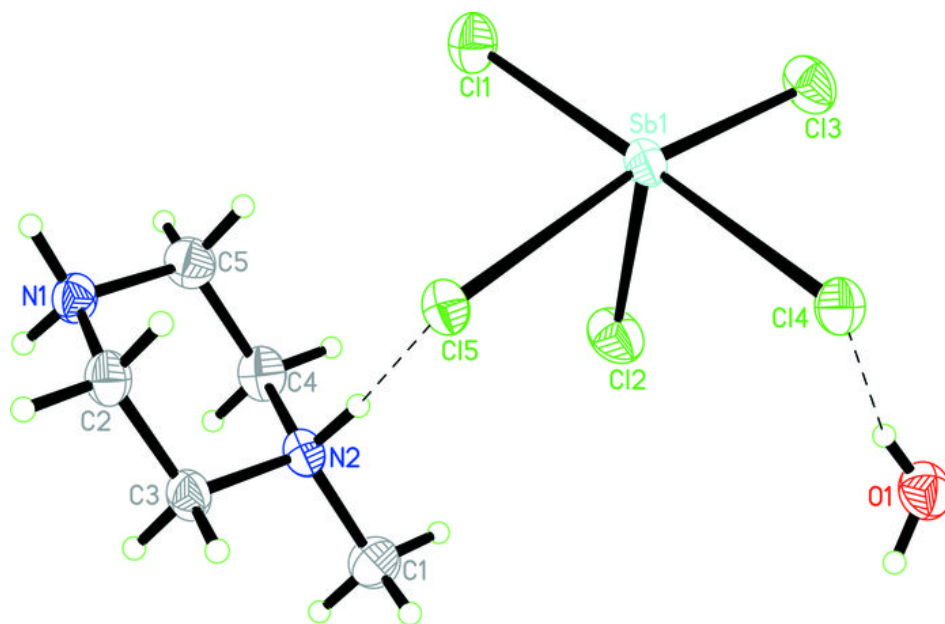


Fig. 2

