

Bis(2-amino-6-methylpyridinium) di- μ -chlorido- μ -oxido-bis[dichlorido-antimonate(III)]

Wen-Jie Feng,^a Hai-Bin Wang,^b Xin-Jü Ma,^a Hai-Yan Li^a and Zhi-Min Jin^{a*}

^aCollege of Pharmaceutical Sciences, Zhejiang University of Technology, Hangzhou 310014, People's Republic of China, and ^bCollege of Chemical Engineering and Material Sciences, Zhejiang University of Technology, Hangzhou 310014, People's Republic of China

Correspondence e-mail: apharm@sina.com

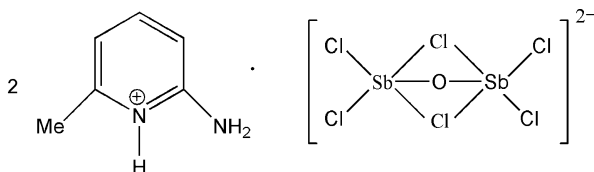
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.011$ Å; R factor = 0.047; wR factor = 0.095; data-to-parameter ratio = 16.5.

The title compound, $(\text{C}_6\text{H}_9\text{N}_2)_2[\text{Sb}_2\text{Cl}_6\text{O}]$, consists of one triple-bridged $[\text{Sb}_2\text{Cl}_6\text{O}]^{2-}$ anion and two 2-amino-6-methylpyridinium cations that undergo aminium-iminium tautomerism. The counter-ion pair are linked together by $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds.

Related literature

For related literature, see: Albrecht *et al.* (2003); Jin *et al.* (2001, 2002, 2000, 2005); Krizanovic *et al.* (1993); Lah *et al.* (2002); Luque *et al.* (1997); Hall & Sowerby (1979); Navarro *et al.* (1985); Qin *et al.* (1999); Ren *et al.* (2002); Rivas *et al.* (2003); Yip *et al.* (1999); Inuzuka & Fujimoto (1986, 1990).



Experimental

Crystal data

$(\text{C}_6\text{H}_9\text{N}_2)_2[\text{Sb}_2\text{Cl}_6\text{O}]$
 $M_r = 690.52$
 Monoclinic, $P2_1/n$
 $a = 10.0746$ (10) Å
 $b = 15.1972$ (16) Å
 $c = 15.4631$ (14) Å
 $\beta = 101.597$ (6)°

$V = 2319.2$ (4) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 3.03$ mm⁻¹
 $T = 298$ (2) K
 $0.30 \times 0.30 \times 0.30$ mm

Data collection

Bruker APEX area-detector diffractometer

Absorption correction: multi-scan (SADABS; Bruker, 2000)
 $T_{\text{min}} = 0.419$, $T_{\text{max}} = 0.419$

12055 measured reflections
 4137 independent reflections

3876 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.095$
 $S = 1.24$
 4137 reflections
 250 parameters
 8 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.86$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.50$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Sb1—O1	1.964 (4)	Sb2—O1	1.977 (4)
Sb1—Cl2	2.4221 (18)	Sb2—Cl4	2.418 (2)
Sb1—Cl1	2.4783 (18)	Sb2—Cl5	2.426 (2)
Sb1—Cl3	2.8123 (16)		
O1—Sb1—Cl2	90.35 (12)	Cl1—Sb1—Cl3	168.84 (6)
O1—Sb1—Cl1	89.82 (12)	O1—Sb2—Cl4	89.39 (12)
Cl2—Sb1—Cl1	90.96 (7)	O1—Sb2—Cl5	90.13 (12)
O1—Sb1—Cl3	79.04 (12)	Cl4—Sb2—Cl5	91.95 (10)
Cl2—Sb1—Cl3	88.44 (6)	Sb1—O1—Sb2	116.72 (18)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1N \cdots O1	0.84 (2)	2.08 (3)	2.907 (8)	169 (3)
N2—H2A \cdots Cl4	0.84 (2)	2.59 (6)	3.329 (8)	148 (3)
N2—H2B \cdots Cl3 ⁱ	0.84 (2)	2.67 (6)	3.439 (8)	157 (3)
N3—H3N \cdots Cl6	0.85 (2)	2.30 (5)	3.127 (8)	167 (4)
N4—H4A \cdots Cl6	0.85 (2)	2.66 (3)	3.362 (8)	141 (5)
N4—H4B \cdots Cl1 ⁱⁱ	0.84 (2)	2.62 (3)	3.432 (8)	162 (5)

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

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; 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: AT2304).

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Bis(2-amino-6-methylpyridinium) di- μ -chlorido- μ -oxido-bis[dichloridoantimonate(III)]

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Comment

There are numerous examples of 2-amino-substituted pyridine compounds reported previously (Navarro Ranninger *et al.*, 1985; Krizanovic *et al.*, 1993; Luque *et al.*, 1997; Qin *et al.*, 1999; Yip *et al.*, 1999; Lah *et al.*, 2002; Ren *et al.*, 2002; Rivas *et al.*, 2003; Jin *et al.*, 2000, 2001, 2002, 2005; Albrecht *et al.*, 2003). Among them, the tautomerism phenomenon (Scheme 1) of 2-amino-*X*-methylpyridine [2AXMP; *X* indicates the methyl position] has been proved by X-ray diffraction, such as 2 A6MP-hydrochloric acid (2/2; Jin *et al.*, 2005) and 2 A6MP-neoabietic acid (1/1; Jin *et al.*, 2000). All the above studies provide important references to further research into 2-aminopyridines. Now, the title compound, (I), has been synthesized, as a continue part of research.

As shown in Fig. 1, there are two crystallographically independent 2-amino-6-methylpyridinium (HAMP) cations and a $(\text{Sb}_2\text{Cl}_6\text{O})^{2-}$ anion in the formula unit. The anion is linked to one HAMP (N1/N2/C1/C6) by N1—H1N \cdots O1 and N2—H2A \cdots Cl4 hydrogen bonds, and to the other HAMP(N3/N4/C7/C12) by N3—H3N \cdots Cl6 hydrogen bond.

In the structure, two 2-amino-6-methylpyridine molecules are protonated, and show aminium-iminium tautomerism phenomenon (Inuzuka & Fujimoto, 1986 and 1990). Features of the iminium tautomer are most clearly observed in (I), suggesting that the imine tautomer play a important role in the structure. In cation HAMP (N1/N2/C1/C6), the N2—C1 bond [1.329 (8) Å] is a little but significantly shorter than the N1—C1 [1.353 (8) Å] and N1—C5 [1.353 (8) Å] bonds, coincident with the iminium tautomer (Table 1). Moreover, the existence of the iminium tautomer is testified by the fact that the C1—C2 [1.389 (9) Å] and C3—C4 [1.387 (10) Å] bonds are longer than the C2—C3 [1.343 (9) Å] and C4—C5 [1.350 (9) Å] bonds. Similar features are provided with HAMP (N3/N4/C7/C12). In the HAMP (N3/N4/C7/C12), the N4 and three H atoms of N3 and N4 are on the same plane with the matrix heterocycle. But, there is a slight difference in the cation HAMP (N1/N2/C1/C6). H2A of N2 deviates from the matrix plane at a distance of 0.2507 (8) Å, due to N2—H2A \cdots Cl4 hydrogen bond (Fig. 1).

The structure of the $(\text{Sb}_2\text{Cl}_6\text{O})^{2-}$ anion resembles a anionic antimony(III) oxide chloride reported previously (Hall & Sowerby, 1979). It has a discrete dimeric structure resulting from the sharing of a common face between two pseudo-octahedral units. The triple bridge involves one oxygen (O1) and two chlorine atoms (Cl3 and Cl6). The structure is based on two SbOCl_4 pseudo-octahedral units. The lone pair of electrons of Sb in each case occupies the sixth octahedral position which is *trans* to the bridging oxygen atom. Bonds to terminal chlorine atoms are of normal length and are substantially shorter than those involving in bridging (Table. 1). Bridging is asymmetrical at Cl3 but both Cl6 and O1 form symmetrical bridges. The distance between Sb1 and Sb2 is of 3.530 (8) Å, a value well within the sum of van der Waals' radius (4.4 Å).

In the structure, the $\text{Sb}_2\text{Cl}_6\text{O}$ entities link to their neighbouring ones through coordinated bonds of Sb1—Cl5 ($-x + 1/2, y + 1/2, -z + 1/2$) [3.530 (8) Å] in chains (Fig. 2). The chains link to their antiparallel neighbouring ones by Sb2—Cl3 ($-x, -y + 2, -z$) coordinated bonds [3.481 (8) Å]. Every two corresponding $\text{Sb}_2\text{Cl}_6\text{O}$ entities in the two antiparallel neighbouring chains submit centrosymmetrically. The whole crystal structure is formed by extensive network of moderate inter-

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molecular hydrogen bonds of N4—H4A···Cl6, N2—H2B···Cl3 ($-x + 1, -y + 2, -z$), N4—H4B···Cl2 ($-x + 1, -y + 2, -z$) and N4—H4b···Cl1 ($-x + 1, -y + 2, -z$), including the above three strong intramolecular hydrogen bonds and coordinated Sb—Cl bonds (Fig. 3).

Experimental

The preparation of the single-crystal of (I): dissolving 1.5 g antimony trichloride in 10 ml absolute acetone to form a solution, then adding 1 ml hydrochloride acid and 2 ml 2-amino-6-methylpyridine to the solution. Keeping stir and heating, until it became a clear solution, then the reaction system was cooled slowly to room temperature. Crystals of (I) were formed by gradual evaporation of the acetone over a period of three days at 300 K. Analysis calculated for $\text{Sb}_2\text{Cl}_6\text{O}(\text{C}_6\text{H}_9\text{N}_2)_2$: C 20.33, H 2.42, N 7.90%. Found: C 20.28, H 2.45, N 7.97%.

Refinement

H atoms attaching to N atoms were deduced from difference Fourier maps, and incorporated in refinement freely. Others were placed in calculated positions and allowed to ride on their parent atoms at distances of 0.93Å for aromatic group and 0.96Å for methyl, with $U_{\text{iso}}(\text{H}) = 1.2-1.5 U_{\text{eq}}(\text{C})$.

Figures

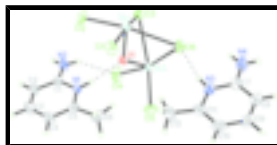


Fig. 1. The molecular structure of (I), 30% probability displacement ellipsoids is shown. Hydrogen bonds are illustrated as dashed lines.

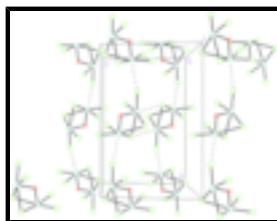


Fig. 2. The packing diagram of $\text{Sb}_2\text{Cl}_6\text{O}$ anions viewed down along the c axis.

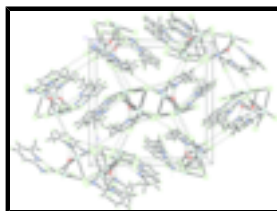


Fig. 3. The packing diagram of (I) viewed down along the a axis. Hydrogen bonds are illustrated by dashed lines.

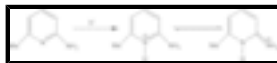


Fig. 4. The tautomerism in the 2-amino-6-methylpyridinium cation.

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Crystal data

$(\text{C}_6\text{H}_9\text{N}_2)_2[\text{Sb}_2\text{Cl}_6\text{O}]$

$F_{000} = 1320.0$

$M_r = 690.52$	$D_x = 1.978 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
Hall symbol: -P 2yn	$\lambda = 0.71073 \text{ \AA}$
$a = 10.0746 (10) \text{ \AA}$	Cell parameters from 5386 reflections
$b = 15.1972 (16) \text{ \AA}$	$\theta = 2.2\text{--}24.9^\circ$
$c = 15.4631 (14) \text{ \AA}$	$\mu = 3.03 \text{ mm}^{-1}$
$\beta = 101.597 (6)^\circ$	$T = 298 (2) \text{ K}$
$V = 2319.2 (4) \text{ \AA}^3$	Block, colourless
$Z = 4$	$0.30 \times 0.30 \times 0.30 \text{ mm}$

Data collection

Bruker APEX area-detector diffractometer	4154 independent reflections
Radiation source: fine-focus sealed tube	3876 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.026$
$T = 298(2) \text{ K}$	$\theta_{\text{max}} = 25.2^\circ$
φ and ω scans	$\theta_{\text{min}} = 1.9^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$h = -12 \rightarrow 8$
$T_{\text{min}} = 0.419$, $T_{\text{max}} = 0.419$	$k = -18 \rightarrow 18$
12055 measured reflections	$l = -17 \rightarrow 18$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.048$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.095$	$w = 1/[\sigma^2(F_o^2) + (0.03P)^2 + 5.7532P]$
$S = 1.24$	where $P = (F_o^2 + 2F_c^2)/3$
4137 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
250 parameters	$\Delta\rho_{\text{max}} = 0.86 \text{ e \AA}^{-3}$
8 restraints	$\Delta\rho_{\text{min}} = -0.50 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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.

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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	$U_{\text{iso}}^*/U_{\text{eq}}$
Sb1	0.31762 (4)	0.91360 (3)	0.22353 (3)	0.03853 (13)
Sb2	0.11872 (4)	1.06417 (3)	0.10221 (3)	0.03951 (13)
Cl5	0.1190 (2)	1.20568 (13)	0.17485 (18)	0.0883 (7)
Cl4	0.2383 (2)	1.12353 (18)	-0.00553 (15)	0.0899 (8)
Cl6	0.0414 (2)	0.96108 (14)	0.24743 (13)	0.0676 (5)
Cl2	0.52714 (18)	0.90105 (13)	0.17034 (12)	0.0612 (5)
Cl3	0.17591 (17)	0.88522 (11)	0.04966 (10)	0.0502 (4)
Cl1	0.44172 (18)	0.96909 (13)	0.36800 (11)	0.0607 (5)
O1	0.2946 (4)	1.0349 (2)	0.1794 (3)	0.0369 (9)
N1	0.5322 (5)	1.1474 (3)	0.1987 (4)	0.0428 (12)
N3	-0.0424 (6)	1.0719 (4)	0.3982 (4)	0.0530 (14)
N2	0.5688 (7)	1.0906 (5)	0.0664 (4)	0.0653 (18)
C1	0.6139 (6)	1.1355 (4)	0.1402 (4)	0.0440 (15)
C5	0.5662 (7)	1.1973 (4)	0.2724 (4)	0.0476 (16)
N4	-0.2302 (7)	0.9894 (6)	0.3401 (5)	0.079 (2)
C7	-0.1687 (7)	1.0454 (5)	0.4007 (5)	0.0534 (17)
C11	0.0303 (8)	1.1289 (5)	0.4562 (5)	0.063 (2)
C6	0.4618 (8)	1.2076 (5)	0.3281 (5)	0.065 (2)
H6A	0.3829	1.1737	0.3032	0.098*
H6B	0.4374	1.2685	0.3301	0.098*
H6C	0.4981	1.1871	0.3868	0.098*
C2	0.7428 (7)	1.1720 (5)	0.1600 (5)	0.0546 (17)
H2	0.8037	1.1630	0.1230	0.065*
C8	-0.2278 (8)	1.0787 (5)	0.4684 (5)	0.062 (2)
H8	-0.3149	1.0619	0.4729	0.075*
C3	0.7781 (8)	1.2206 (5)	0.2336 (5)	0.067 (2)
H3	0.8647	1.2446	0.2473	0.081*
C4	0.6893 (8)	1.2359 (5)	0.2898 (5)	0.067 (2)
H4	0.7142	1.2723	0.3388	0.080*
C9	-0.1572 (9)	1.1350 (6)	0.5269 (5)	0.072 (2)
H9	-0.1967	1.1576	0.5716	0.086*
C10	-0.0270 (10)	1.1603 (5)	0.5221 (6)	0.079 (3)
H10	0.0211	1.1985	0.5640	0.094*
C12	0.1683 (9)	1.1504 (6)	0.4414 (7)	0.090 (3)
H12A	0.1840	1.1196	0.3902	0.135*
H12B	0.2347	1.1329	0.4920	0.135*
H12C	0.1750	1.2126	0.4323	0.135*
H1N	0.458 (3)	1.121 (3)	0.191 (3)	0.024 (14)*
H4A	-0.195 (6)	0.970 (5)	0.299 (4)	0.08 (3)*
H3N	-0.008 (6)	1.046 (4)	0.359 (3)	0.046 (19)*

H2A	0.487 (3)	1.076 (4)	0.052 (4)	0.06 (2)*
H2B	0.616 (5)	1.085 (5)	0.028 (3)	0.07 (3)*
H4B	-0.312 (3)	0.975 (5)	0.337 (4)	0.08 (3)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sb1	0.0418 (2)	0.0367 (2)	0.0381 (2)	0.00126 (17)	0.01051 (18)	0.00583 (17)
Sb2	0.0305 (2)	0.0408 (2)	0.0460 (3)	0.00311 (17)	0.00485 (17)	-0.00017 (18)
Cl5	0.0748 (14)	0.0531 (11)	0.128 (2)	0.0129 (10)	-0.0002 (13)	-0.0301 (12)
Cl4	0.0681 (13)	0.1222 (19)	0.0832 (15)	0.0132 (13)	0.0242 (11)	0.0587 (14)
Cl6	0.0643 (12)	0.0844 (13)	0.0647 (12)	-0.0134 (10)	0.0381 (10)	-0.0141 (10)
Cl2	0.0497 (10)	0.0758 (12)	0.0620 (11)	0.0222 (9)	0.0209 (8)	0.0095 (9)
Cl3	0.0538 (10)	0.0551 (9)	0.0421 (9)	-0.0001 (8)	0.0110 (7)	-0.0087 (7)
Cl1	0.0562 (11)	0.0833 (13)	0.0413 (9)	-0.0084 (9)	0.0067 (8)	-0.0003 (9)
O1	0.031 (2)	0.033 (2)	0.044 (2)	-0.0005 (17)	0.0028 (17)	0.0111 (18)
N1	0.036 (3)	0.042 (3)	0.051 (3)	-0.005 (2)	0.012 (3)	-0.003 (3)
N3	0.052 (4)	0.058 (4)	0.052 (4)	0.000 (3)	0.019 (3)	-0.010 (3)
N2	0.056 (4)	0.085 (5)	0.058 (4)	-0.020 (4)	0.018 (3)	-0.024 (4)
C1	0.046 (4)	0.040 (3)	0.047 (4)	-0.005 (3)	0.012 (3)	-0.006 (3)
C5	0.052 (4)	0.042 (3)	0.050 (4)	0.002 (3)	0.014 (3)	-0.008 (3)
N4	0.051 (4)	0.118 (6)	0.071 (5)	-0.021 (4)	0.019 (4)	-0.035 (5)
C7	0.045 (4)	0.067 (5)	0.050 (4)	0.003 (3)	0.015 (3)	0.002 (4)
C11	0.076 (5)	0.047 (4)	0.068 (5)	-0.008 (4)	0.020 (4)	0.003 (4)
C6	0.069 (5)	0.070 (5)	0.063 (5)	0.008 (4)	0.029 (4)	-0.018 (4)
C2	0.043 (4)	0.069 (5)	0.055 (4)	-0.010 (3)	0.016 (3)	-0.013 (4)
C8	0.061 (5)	0.071 (5)	0.060 (5)	0.009 (4)	0.022 (4)	0.001 (4)
C3	0.053 (4)	0.084 (6)	0.068 (5)	-0.027 (4)	0.018 (4)	-0.018 (4)
C4	0.070 (5)	0.071 (5)	0.059 (5)	-0.023 (4)	0.013 (4)	-0.026 (4)
C9	0.093 (6)	0.076 (6)	0.058 (5)	0.013 (5)	0.041 (5)	-0.002 (4)
C10	0.107 (7)	0.063 (5)	0.071 (6)	-0.013 (5)	0.031 (5)	-0.022 (4)
C12	0.087 (6)	0.077 (6)	0.113 (8)	-0.032 (5)	0.037 (6)	-0.016 (5)

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

Sb1—O1	1.964 (4)	N4—H4A	0.85 (2)
Sb1—Cl2	2.4221 (18)	N4—H4B	0.84 (2)
Sb1—Cl1	2.4783 (18)	C7—C8	1.399 (10)
Sb1—Cl3	2.8123 (16)	C11—C10	1.355 (10)
Sb2—O1	1.977 (4)	C11—C12	1.490 (11)
Sb2—Cl4	2.418 (2)	C6—H6A	0.9600
Sb2—Cl5	2.426 (2)	C6—H6B	0.9600
Sb2—Sb1	3.355 (2)	C6—H6C	0.9600
N1—C1	1.353 (8)	C2—C3	1.343 (9)
N1—C5	1.353 (8)	C2—H2	0.9300
N1—H1N	0.836 (19)	C8—C9	1.340 (11)
N3—C7	1.343 (9)	C8—H8	0.9300
N3—C11	1.351 (9)	C3—C4	1.387 (10)
N3—H3N	0.85 (2)	C3—H3	0.9300

supplementary materials

N2—C1	1.329 (8)	C4—H4	0.9300
N2—H2A	0.835 (19)	C9—C10	1.383 (12)
N2—H2B	0.837 (19)	C9—H9	0.9300
C1—C2	1.389 (9)	C10—H10	0.9300
C5—C4	1.350 (9)	C12—H12A	0.9600
C5—C6	1.496 (9)	C12—H12B	0.9600
N4—C7	1.323 (9)	C12—H12C	0.9600
O1—Sb1—C12	90.35 (12)	N3—C11—C12	115.9 (7)
O1—Sb1—C11	89.82 (12)	C10—C11—C12	126.2 (8)
C12—Sb1—C11	90.96 (7)	C5—C6—H6A	109.5
O1—Sb1—C13	79.04 (12)	C5—C6—H6B	109.5
C12—Sb1—C13	88.44 (6)	H6A—C6—H6B	109.5
C11—Sb1—C13	168.84 (6)	C5—C6—H6C	109.5
O1—Sb2—C14	89.39 (12)	H6A—C6—H6C	109.5
O1—Sb2—C15	90.13 (12)	H6B—C6—H6C	109.5
C14—Sb2—C15	91.95 (10)	C3—C2—C1	118.9 (6)
Sb1—O1—Sb2	116.72 (18)	C3—C2—H2	120.5
C1—N1—C5	123.6 (5)	C1—C2—H2	120.5
C1—N1—H1N	119 (4)	C9—C8—C7	119.2 (8)
C5—N1—H1N	117 (4)	C9—C8—H8	120.4
C7—N3—C11	124.3 (6)	C7—C8—H8	120.4
C7—N3—H3N	114 (4)	C2—C3—C4	122.0 (7)
C11—N3—H3N	122 (5)	C2—C3—H3	119.0
C1—N2—H2A	121 (4)	C4—C3—H3	119.0
C1—N2—H2B	121 (4)	C5—C4—C3	119.0 (7)
H2A—N2—H2B	117 (3)	C5—C4—H4	120.5
N2—C1—N1	119.6 (6)	C3—C4—H4	120.5
N2—C1—C2	122.6 (6)	C8—C9—C10	121.4 (7)
N1—C1—C2	117.8 (6)	C8—C9—H9	119.3
C4—C5—N1	118.6 (6)	C10—C9—H9	119.3
C4—C5—C6	124.3 (6)	C11—C10—C9	119.6 (8)
N1—C5—C6	117.1 (6)	C11—C10—H10	120.2
C7—N4—H4A	124 (4)	C9—C10—H10	120.2
C7—N4—H4B	121 (4)	C11—C12—H12A	109.5
H4A—N4—H4B	115 (3)	C11—C12—H12B	109.5
N4—C7—N3	118.9 (7)	H12A—C12—H12B	109.5
N4—C7—C8	123.6 (7)	C11—C12—H12C	109.5
N3—C7—C8	117.5 (7)	H12A—C12—H12C	109.5
N3—C11—C10	118.0 (7)	H12B—C12—H12C	109.5
C12—Sb1—O1—Sb2	127.71 (19)	N2—C1—C2—C3	176.4 (8)
C11—Sb1—O1—Sb2	-141.33 (19)	N1—C1—C2—C3	-3.1 (10)
C13—Sb1—O1—Sb2	39.36 (17)	N4—C7—C8—C9	179.8 (8)
C14—Sb2—O1—Sb1	-128.3 (2)	N3—C7—C8—C9	0.3 (11)
C15—Sb2—O1—Sb1	139.7 (2)	C1—C2—C3—C4	-0.6 (13)
C5—N1—C1—N2	-175.3 (7)	N1—C5—C4—C3	-2.4 (11)
C5—N1—C1—C2	4.3 (9)	C6—C5—C4—C3	179.3 (7)
C1—N1—C5—C4	-1.5 (10)	C2—C3—C4—C5	3.4 (13)
C1—N1—C5—C6	177.0 (6)	C7—C8—C9—C10	-0.6 (12)

C11—N3—C7—N4	179.8 (8)	N3—C11—C10—C9	-1.5 (12)
C11—N3—C7—C8	-0.6 (11)	C12—C11—C10—C9	179.3 (8)
C7—N3—C11—C10	1.2 (11)	C8—C9—C10—C11	1.3 (14)
C7—N3—C11—C12	-179.5 (7)		

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>
N1—H1N...O1	0.84 (2)	2.08 (3)	2.907 (8)	169 (3)
N2—H2A...C14	0.84 (2)	2.59 (6)	3.329 (8)	148 (3)
N2—H2B...C13 ⁱ	0.84 (2)	2.67 (6)	3.439 (8)	157 (3)
N3—H3N...C16	0.85 (2)	2.30 (5)	3.127 (8)	167 (4)
N4—H4A...C16	0.85 (2)	2.66 (3)	3.362 (8)	141 (5)
N4—H4B...C11 ⁱⁱ	0.84 (2)	2.62 (3)	3.432 (8)	162 (5)

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

Fig. 1

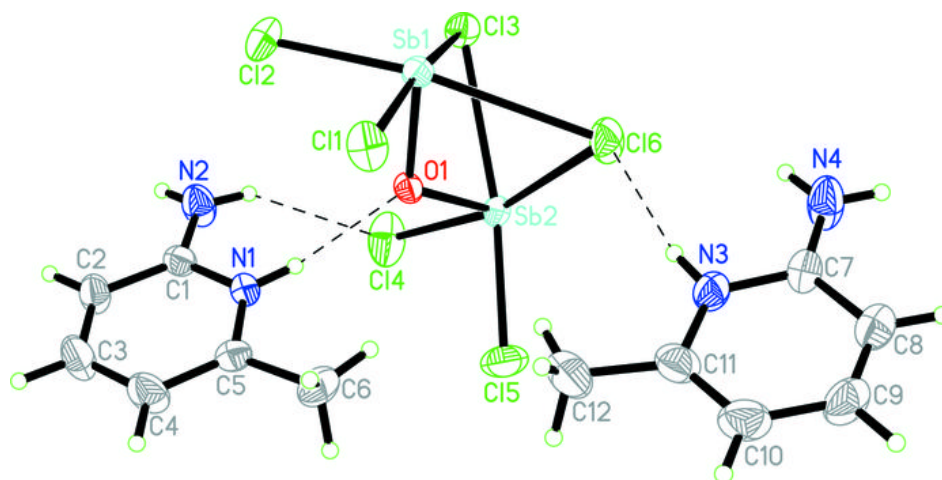


Fig. 2

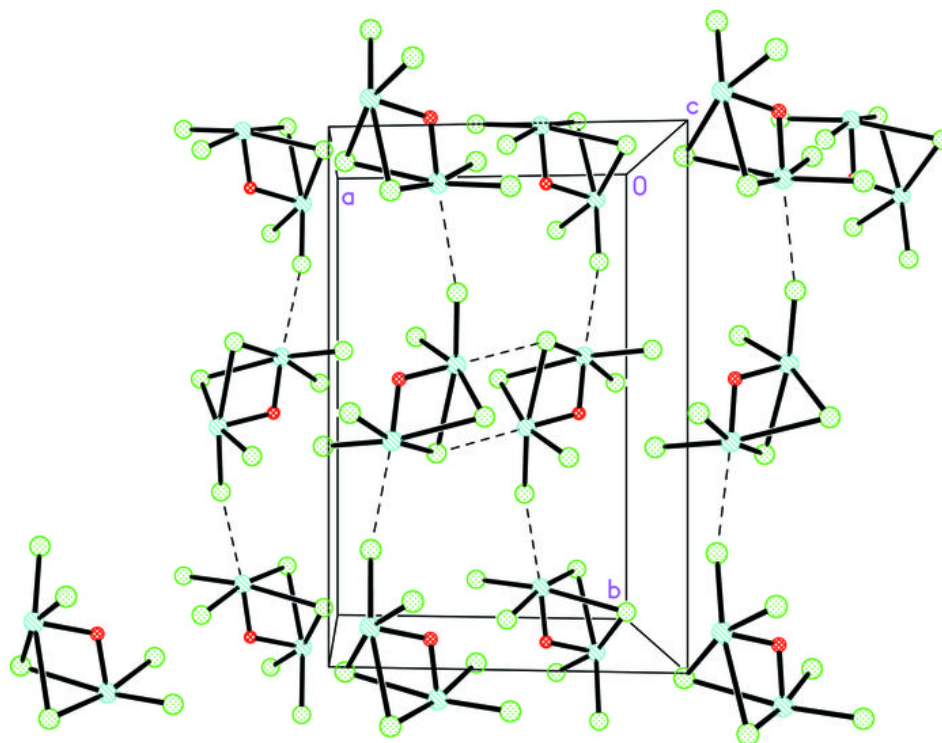


Fig. 3

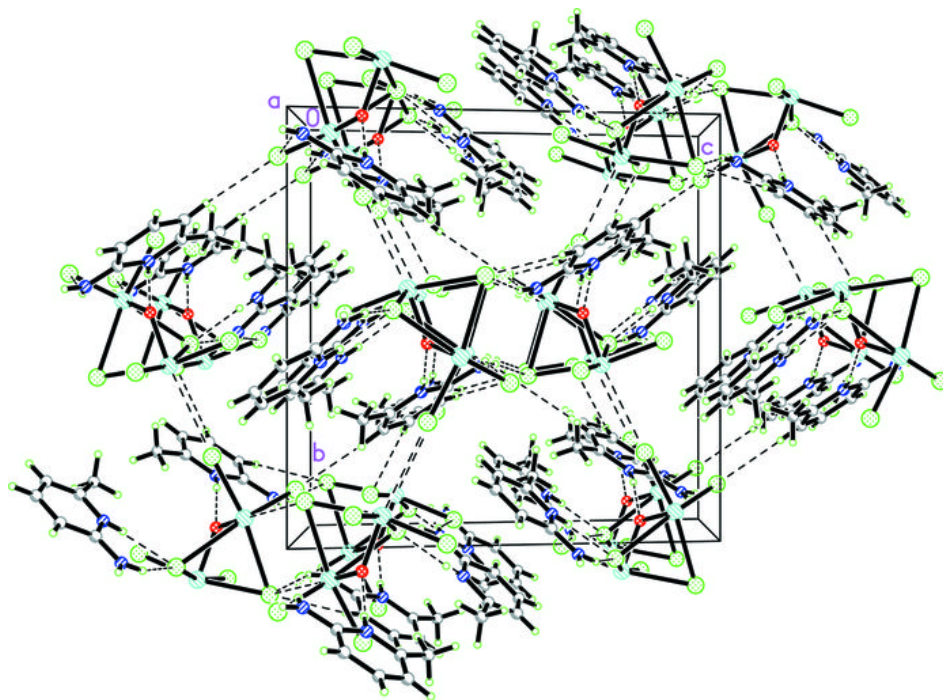


Fig. 4

