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Bis(4-methylpyridinium) pentachloridoantimonate(III)

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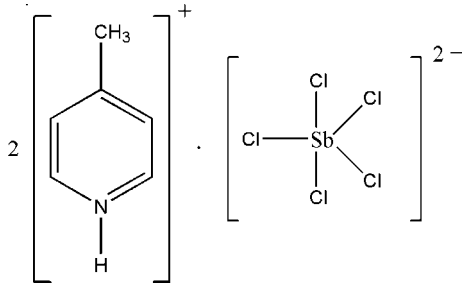
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.006 Å; R factor = 0.027; wR factor = 0.071; data-to-parameter ratio = 17.7.

The asymmetric unit of the title compound, $(C_6H_8N)_2[SbCl_5]$, consists of an $SbCl_5$ anion linked to two 4-methylpyridinium cations by two N—H···Cl hydrogen bonds. The bond lengths and angles around the Sb^{III} ion describe a disorted square-pyramidal coordination geometry. In the crystal structure, weak π–π stacking interactions occur between inversion-related pyridine rings [$Cg1\cdots Cg1$ = 3.788 (2) Å and $Cg2\cdots Cg2$ = 3.522 (2) Å, where $Cg1$ and $Cg2$ are the centroids of the two pyridine rings].

Related literature

For related literature, see: Andras *et al.* (1993); Battaglia *et al.* (1983); Biradha & Zaworotko (1998); Clegg *et al.* (2000); Goher & Mautner (1999); James *et al.* (1999); Jin *et al.* (2000); Johnson *et al.* (1984); Leonard *et al.* (1999); Mayr *et al.* (1993); Ohms *et al.* (1985).



Experimental

Crystal data

$(C_6H_8N)_2[SbCl_5]$
M_r = 487.29
Triclinic, $P\overline{1}$
a = 8.9815 (13) Å
b = 10.4501 (16) Å
c = 10.7488 (16) Å
α = 97.639 (2)°
β = 110.180 (3)°
γ = 98.242 (2)°
V = 918.9 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.22 mm^{-1}
T = 273 (2) K
0.39 × 0.29 × 0.17 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.467, T_{max} = 0.686
4810 measured reflections
3246 independent reflections
2966 reflections with $I > 2\sigma(I)$
R_{int} = 0.012

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.071
S = 1.06
3246 reflections
183 parameters
$\Delta\rho_{max}$ = 0.61 e Å^{-3}
$\Delta\rho_{min}$ = −0.62 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sb1—Cl2	2.4268 (10)	Sb1—Cl5	2.6555 (9)
Sb1—Cl1	2.5173 (9)	Sb1—Cl4	2.7649 (10)
Sb1—Cl3	2.6177 (9)		
Cl2—Sb1—Cl1	90.58 (4)	Cl3—Sb1—Cl5	177.67 (3)
Cl2—Sb1—Cl3	88.40 (3)	Cl2—Sb1—Cl4	90.26 (3)
Cl1—Sb1—Cl3	90.59 (3)	Cl1—Sb1—Cl4	178.98 (3)
Cl2—Sb1—Cl5	89.28 (3)	Cl3—Sb1—Cl4	88.86 (3)
Cl1—Sb1—Cl5	89.22 (3)	Cl5—Sb1—Cl4	91.36 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···Cl3	0.86	2.55	3.272 (5)	142
N2—H2···Cl5	0.86	2.43	3.212 (6)	151

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: LH2475).

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supplementary materials

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Bis(4-methylpyridinium) pentachloridoantimonate(III)

W.-J. Feng, X.-J. Ma, F. Zhang, Y.-C. Yuan and Z.-M. Jin

Comment

The crystal structure of 4-methylpyridine has already been reported (Ohms *et al.*, 1985). It is an important solvent and intermediate in organic synthesis and it is present in many complexes (Leonard *et al.*, 1999; Biradha & Zaworotko, 1998; Jin *et al.*, 2000). It can be coordinated to a metal ions (Battaglia *et al.*, 1983; Johnson *et al.*, 1984; Andras *et al.*, 1993; Goher & Mautner 1999; James *et al.*, 1999; Mayr *et al.*, 1993; Clegg *et al.*, 2000). Herein present here the crystal structure of the title compound.

As shown in Fig. 1, there are two 4-methylpyridinium (HMP) cations and one $(SbCl_5)^{2-}$ anion in the formula unit. The anion is linked to one HMP cation (N1/C1–C6) by an N1—H1···Cl3 hydrogen bond, and to the other HMP cation (N2/C7–C12) by an N2—H2···Cl5 hydrogen bond. The dihedral angle between the two pyridine rings in the formula unit is 17.18 (3)°. The geometry of $SbCl_5$ anion is a distorted square pyramid. The four atoms Cl1, Cl3, Cl4 and Cl5 form the basal plane, while Cl2 is the apex of the pyramid.

Symmetry related $SbCl_5$ anions are linked into dimers *via* weak coordinated bonds of the type Sb1···Cl4($-x + 1, -y + 1, -z + 1$) [3.271 (10) Å] (Fig. 2), which play a role in the stabilization of the crystal structure. In addtion, there are weak π···π stacking between symmetry related pyridine rings with a centroid to centroid distance of 3.522 (2) and 3.788 (2) Å and a interplanar distances of 3.434 and 3.431 Å resulting in offset angles of 25.1° and 12.8 (Fig. 3). These interactions further stablize the crystal structure.

Experimental

Antimony trichloride, hydrochloride acid and 4-methylpyridine in a 1:2:2 molar ratio were mixed and dissolved in sufficient acetone to heat to a temperature at which a clear solution resulted. Crystals of the title compound were formed by gradual evaporation of acetone over a period of three days at 298 K.

Refinement

All H atoms were placed in calculated positions and allowed to ride on their parent atoms at distances of 0.86 Å for (N—H), 0.93 Å for aromatic groups and 0.96 Å for methyl, with $U_{iso}(H) = 1.2–1.5\ U_{eq}(C)$.

Figures

Fig. 1. The molecular structure shown with 30% probability displacement ellipsoids. Hydrogen bonds are illustrated as dashed lines.

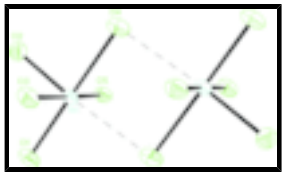

Fig. 2. A dimer unit of $SbCl_5$ in the crystal structure. Atoms which are not labeled are obtained by symmetry operation of (−*x* + 1, −*y* + 1, −*z* + 1). Weak coordinated Sb1···Cl4 bonds are illustrated by dashed lines.

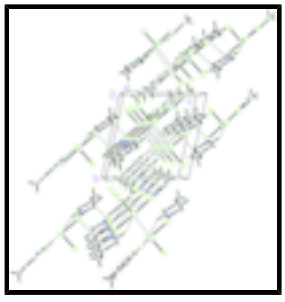

Fig. 3. The packing of the title compound viewed down along the *a* axis. Hydrogen bonds and weak Sb1···Cl4 interactions are illustrated by dashed lines.

Bis(4-methylpyridinium) pentachloridoantimonate(III)

Crystal data

$(C_6H_8N)_2[SbCl_5]$	$V = 918.9\ (2)\ Å^3$
$M_r = 487.29$	$Z = 2$
Triclinic, $P\overline{1}$	$F_{000} = 476$
Hall symbol: -P 1	$D_x = 1.761\ Mg\ m^{-3}$
$a = 8.9815\ (13)$ Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 10.4501\ (16)$ Å	$\mu = 2.22\ mm^{-1}$
$c = 10.7488\ (16)$ Å	$T = 273\ (2)$ K
$\alpha = 97.639\ (2)^o$	Block, colourless
$\beta = 110.180\ (3)^o$	0.39 × 0.29 × 0.17 mm
$\gamma = 98.242\ (2)^o$	

Data collection

Bruker SMART CCD area-detector diffractometer	3246 independent reflections
Radiation source: fine-focus sealed tube	2966 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.012$
$T = 273(2)$ K	$\theta_{max} = 25.0^o$
φ and ω scans	$\theta_{min} = 2.0^o$
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$h = -10 \rightarrow 10$
$T_{min} = 0.467$, $T_{max} = 0.686$	$k = -11 \rightarrow 12$
4810 measured reflections	$l = -12 \rightarrow 11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.027$	?
$wR(F^2) = 0.071$	$w = 1/[\sigma^2(F_o^2) + (0.0397P)^2 + 0.2418P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
3246 reflections	$\Delta\rho_{max} = 0.61$ e Å^{-3}
183 parameters	$\Delta\rho_{min} = -0.62$ e Å^{-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.

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.2694 (4)	0.1629 (3)	0.6001 (3)	0.0670 (8)
H1	0.3330	0.2002	0.5653	0.080*
C1	−0.0441 (7)	−0.0211 (4)	0.7670 (5)	0.111 (2)
H1A	−0.1456	0.0069	0.7346	0.167*
H1B	0.0022	0.0025	0.8639	0.167*
H1C	−0.0618	−0.1150	0.7395	0.167*
C2	0.0697 (5)	0.0451 (3)	0.7094 (4)	0.0599 (9)
C3	0.0245 (4)	0.1371 (3)	0.6302 (4)	0.0573 (8)
H3	−0.0767	0.1588	0.6126	0.069*
C4	0.1285 (4)	0.1961 (3)	0.5777 (3)	0.0582 (8)
H4	0.0994	0.2597	0.5261	0.070*
C5	0.3177 (5)	0.0745 (5)	0.6741 (5)	0.0774 (11)
H5	0.4183	0.0531	0.6873	0.093*
C6	0.2201 (6)	0.0152 (4)	0.7305 (4)	0.0742 (11)
H6	0.2547	−0.0460	0.7838	0.089*
N2	0.8711 (4)	0.5762 (3)	0.1493 (3)	0.0655 (8)
H2	0.7973	0.5395	0.1746	0.079*
C7	1.2405 (6)	0.7603 (5)	0.0351 (5)	0.0882 (13)
H7A	1.3359	0.7254	0.0728	0.132*
H7B	1.2638	0.8535	0.0698	0.132*
H7C	1.2069	0.7446	−0.0615	0.132*
C8	1.1073 (4)	0.6939 (3)	0.0731 (3)	0.0546 (8)

C9	1.1411 (4)	0.6212 (3)	0.1731 (3)	0.0565 (8)
H9	1.2471	0.6117	0.2162	0.068*
C10	1.0231 (4)	0.5636 (3)	0.2096 (4)	0.0575 (8)
H10	1.0480	0.5146	0.2775	0.069*
C11	0.8306 (4)	0.6439 (4)	0.0511 (4)	0.0626 (9)
H11	0.7232	0.6506	0.0095	0.075*
C12	0.9468 (5)	0.7039 (4)	0.0112 (4)	0.0647 (9)
H12	0.9186	0.7516	−0.0577	0.078*
Sb1	0.56843 (2)	0.360071 (18)	0.368247 (19)	0.04399 (9)
Cl1	0.83975 (11)	0.34203 (10)	0.35602 (11)	0.0707 (3)
Cl2	0.43481 (12)	0.18074 (9)	0.17427 (10)	0.0691 (2)
Cl3	0.59349 (11)	0.18528 (10)	0.52378 (10)	0.0643 (2)
Cl4	0.27301 (11)	0.38111 (10)	0.38662 (10)	0.0670 (2)
Cl5	0.53816 (10)	0.52942 (9)	0.20178 (10)	0.0613 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0599 (19)	0.0698 (19)	0.0691 (19)	−0.0025 (16)	0.0302 (16)	0.0061 (16)
C1	0.165 (5)	0.072 (3)	0.121 (4)	−0.015 (3)	0.102 (4)	0.009 (3)
C2	0.083 (3)	0.0412 (16)	0.062 (2)	0.0044 (16)	0.041 (2)	0.0033 (15)
C3	0.0500 (19)	0.0549 (19)	0.066 (2)	0.0136 (15)	0.0217 (16)	0.0028 (16)
C4	0.067 (2)	0.0500 (18)	0.0543 (19)	0.0111 (16)	0.0174 (17)	0.0149 (15)
C5	0.059 (2)	0.085 (3)	0.085 (3)	0.025 (2)	0.022 (2)	0.006 (2)
C6	0.094 (3)	0.065 (2)	0.072 (2)	0.037 (2)	0.030 (2)	0.023 (2)
N2	0.0598 (19)	0.0619 (18)	0.077 (2)	0.0050 (15)	0.0344 (17)	0.0043 (16)
C7	0.082 (3)	0.087 (3)	0.105 (3)	0.002 (2)	0.050 (3)	0.025 (3)
C8	0.056 (2)	0.0495 (17)	0.0576 (19)	0.0065 (15)	0.0235 (16)	0.0058 (15)
C9	0.0467 (18)	0.0585 (19)	0.059 (2)	0.0160 (15)	0.0127 (16)	0.0076 (16)
C10	0.060 (2)	0.0580 (19)	0.060 (2)	0.0198 (16)	0.0229 (17)	0.0190 (16)
C11	0.047 (2)	0.064 (2)	0.065 (2)	0.0127 (17)	0.0095 (17)	0.0005 (18)
C12	0.070 (3)	0.062 (2)	0.055 (2)	0.0154 (19)	0.0125 (18)	0.0158 (17)
Sb1	0.03552 (13)	0.04786 (14)	0.05226 (15)	0.01193 (9)	0.01773 (10)	0.01444 (10)
Cl1	0.0467 (5)	0.0858 (6)	0.0981 (7)	0.0261 (4)	0.0359 (5)	0.0417 (6)
Cl2	0.0676 (6)	0.0604 (5)	0.0681 (5)	0.0170 (4)	0.0123 (4)	0.0064 (4)
Cl3	0.0558 (5)	0.0732 (5)	0.0761 (6)	0.0246 (4)	0.0290 (4)	0.0306 (5)
Cl4	0.0491 (5)	0.0798 (6)	0.0834 (6)	0.0194 (4)	0.0276 (4)	0.0396 (5)
Cl5	0.0471 (5)	0.0627 (5)	0.0748 (6)	0.0161 (4)	0.0181 (4)	0.0233 (4)

Geometric parameters (Å, °)

N1—C4	1.311 (5)	C7—C8	1.501 (5)
N1—C5	1.322 (5)	C7—H7A	0.9600
N1—H1	0.8600	C7—H7B	0.9600
C1—C2	1.502 (5)	C7—H7C	0.9600
C1—H1A	0.9600	C8—C9	1.372 (5)
C1—H1B	0.9600	C8—C12	1.389 (5)
C1—H1C	0.9600	C9—C10	1.344 (5)
C2—C6	1.379 (6)	C9—H9	0.9300

C2—C3	1.380 (5)	C10—H10	0.9300
C3—C4	1.365 (5)	C11—C12	1.364 (5)
C3—H3	0.9300	C11—H11	0.9300
C4—H4	0.9300	C12—H12	0.9300
C5—C6	1.352 (6)	Sb1—Cl2	2.4268 (10)
C5—H5	0.9300	Sb1—Cl1	2.5173 (9)
C6—H6	0.9300	Sb1—Cl3	2.6177 (9)
N2—C11	1.325 (5)	Sb1—Cl5	2.6555 (9)
N2—C10	1.329 (5)	Sb1—Cl4	2.7649 (10)
N2—H2	0.8600		
C4—N1—C5	122.6 (3)	H7A—C7—H7B	109.5
C4—N1—H1	118.7	C8—C7—H7C	109.5
C5—N1—H1	118.7	H7A—C7—H7C	109.5
C2—C1—H1A	109.5	H7B—C7—H7C	109.5
C2—C1—H1B	109.5	C9—C8—C12	117.2 (3)
H1A—C1—H1B	109.5	C9—C8—C7	120.4 (3)
C2—C1—H1C	109.5	C12—C8—C7	122.3 (3)
H1A—C1—H1C	109.5	C10—C9—C8	120.8 (3)
H1B—C1—H1C	109.5	C10—C9—H9	119.6
C6—C2—C3	117.6 (3)	C8—C9—H9	119.6
C6—C2—C1	122.0 (4)	N2—C10—C9	120.5 (3)
C3—C2—C1	120.3 (4)	N2—C10—H10	119.8
C4—C3—C2	119.9 (3)	C9—C10—H10	119.8
C4—C3—H3	120.1	N2—C11—C12	119.8 (3)
C2—C3—H3	120.1	N2—C11—H11	120.1
N1—C4—C3	119.8 (3)	C12—C11—H11	120.1
N1—C4—H4	120.1	C11—C12—C8	120.2 (3)
C3—C4—H4	120.1	C11—C12—H12	119.9
N1—C5—C6	119.7 (4)	C8—C12—H12	119.9
N1—C5—H5	120.1	Cl2—Sb1—Cl1	90.58 (4)
C6—C5—H5	120.1	Cl2—Sb1—Cl3	88.40 (3)
C5—C6—C2	120.3 (4)	Cl1—Sb1—Cl3	90.59 (3)
C5—C6—H6	119.8	Cl2—Sb1—Cl5	89.28 (3)
C2—C6—H6	119.8	Cl1—Sb1—Cl5	89.22 (3)
C11—N2—C10	121.5 (3)	Cl3—Sb1—Cl5	177.67 (3)
C11—N2—H2	119.2	Cl2—Sb1—Cl4	90.26 (3)
C10—N2—H2	119.2	Cl1—Sb1—Cl4	178.98 (3)
C8—C7—H7A	109.5	Cl3—Sb1—Cl4	88.86 (3)
C8—C7—H7B	109.5	Cl5—Sb1—Cl4	91.36 (3)
C6—C2—C3—C4	−0.9 (5)	C12—C8—C9—C10	−0.6 (5)
C1—C2—C3—C4	−179.6 (3)	C7—C8—C9—C10	178.7 (4)
C5—N1—C4—C3	−0.9 (5)	C11—N2—C10—C9	0.8 (5)
C2—C3—C4—N1	1.6 (5)	C8—C9—C10—N2	−0.1 (5)
C4—N1—C5—C6	−0.4 (6)	C10—N2—C11—C12	−0.8 (5)
N1—C5—C6—C2	1.0 (6)	N2—C11—C12—C8	0.1 (5)
C3—C2—C6—C5	−0.4 (6)	C9—C8—C12—C11	0.6 (5)
C1—C2—C6—C5	178.3 (4)	C7—C8—C12—C11	−178.7 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···Cl3	0.86	2.55	3.272 (5)	142
N2—H2···Cl5	0.86	2.43	3.212 (6)	151

Fig. 1

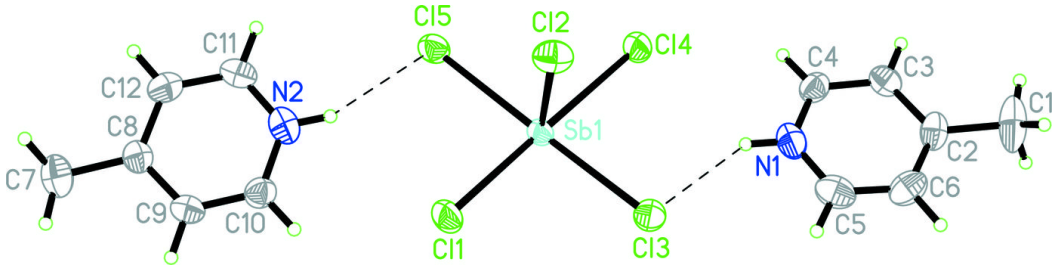

Fig. 2

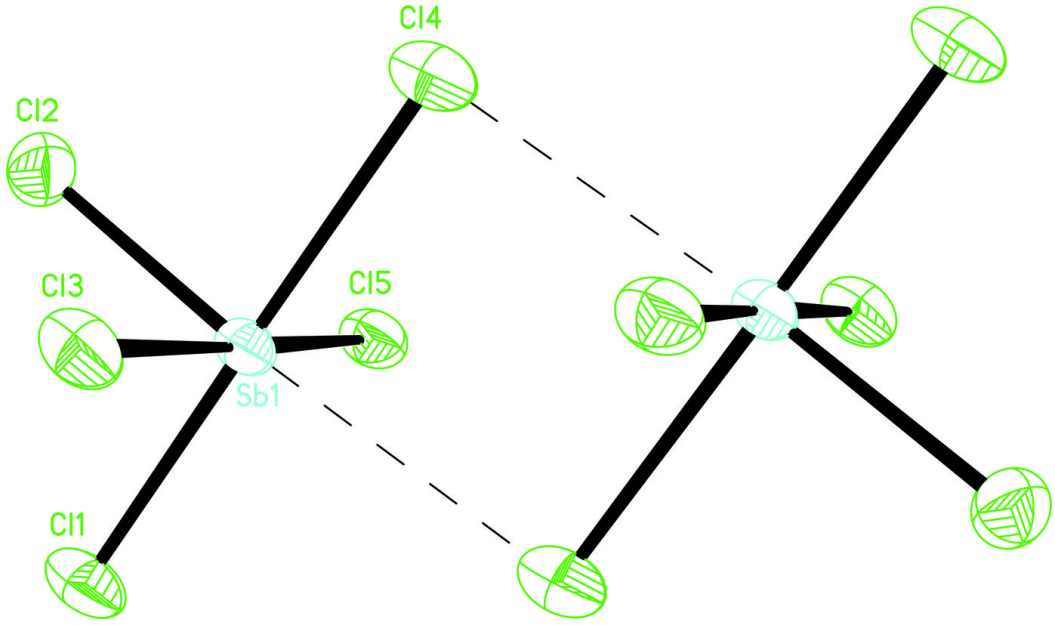

Fig. 3