research communications

Crystal structure of a new hybrid antimony–halide-based compound for possible non-linear optical applications

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The hybrid title compound, *catena*-poly[[[bis(1,4-diazoniabicyclo[2.2.2]octane)[tetraachloridoantimonate(III)]-μ-chlorido-[tetrachloridoantimonate(III)]-μ-chlorido]] monohydrate], $\{(C_6H_{14}N_2)_2[Sb_2Cl_{10}]\cdot H_2O\}_n$, is self-assembled into alternating organic and inorganic layers parallel to the *bc* plane. The anionic inorganic layer consists of infinite zigzag chains of corner-sharing $[SbCl_6]^{3-}$ octahedra running along the *b* axis. The organic part is made up of 1,4-diazoniabicyclo[2.2.2]octane dications (dabcoH$_2$$^{2+}$). The water molecules in the structure connect inorganic and organic layers. Hydrogen-bonding interactions between the ammonium groups, water molecules and Cl atoms ensure the structure cohesion.

1. Chemical context

Organic–inorganic hybrid structures with the general formula $\{(R_a)^{n+}M_bX_{3b+na}\}$ (where R is an organic cation; M is any trivalent metal and X is Cl, Br or/and I) are able to combine desirable characteristics from both types of constituents into a molecular scale composite. These hybrids have been extensively studied for their excitonic and magneto-optical properties. In recent years, a significant number of organic–inorganic hybrid materials based on antimony–halide units have been studied. Six-coordinate antimony halides can arrange themselves in three-, two- or one-dimensional networks through sharing halides in the SbX_6 octahedra, separated by organic cations (Ben Rhaiem *et al.*, 2013; Leblanc *et al.*, 2012; Piecha *et al.*, 2012; Bujack & Angel, 2005, 2006; Bujack & Zaleski, 2004). One-dimensional extended chains can be formed by one, two or three bridging halides and combinations thereof. The use of one bridging halide leads to two types of chains; if the two bridging halides connecting the central octahedron to its neighbours are related *cis*, a zigzag pattern is obtained; if they are *trans*, the chain is linear.
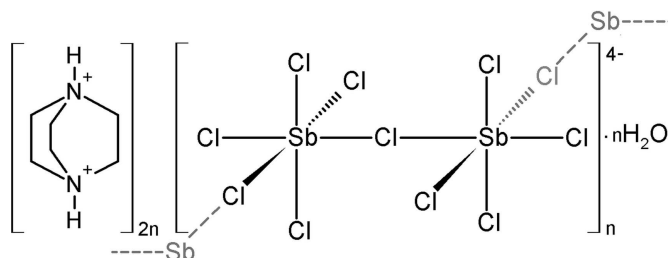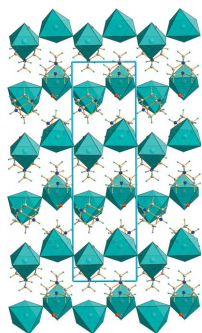
2. Structural commentary

The asymmetric unit of the new chloridoantimonate(III) compound, $(C_6H_{14}N_2)_2[Sb_2Cl_{10}]\cdot H_2O$, (I), consists of two

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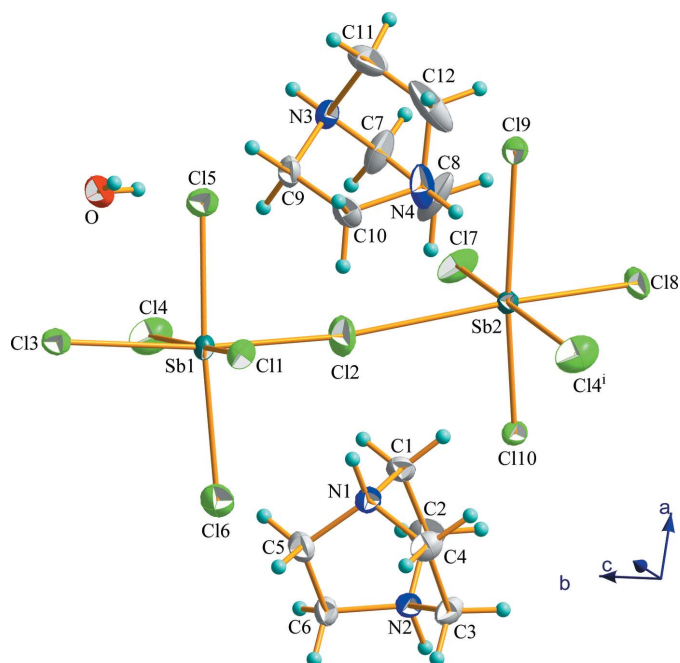
Figure 1
The asymmetric unit of (I) completed by Cl4^i, showing the atomic numbering scheme. Displacement ellipsoids are shown at 30% probability level. [Symmetry code: (i) $x, y − 1, z$.]

symmetry-independant $(dabcoH_2)^{2+}$ dications, a corner sharing bi-octahedron decachloridodiantimonate(III) anion and one crystallization water molecule. The cations are labeled Cat1 (containing atoms N1 and N2) and Cat2 (containing N3 and N4) and the atomic numbering scheme is shown in Fig. 1.

The structure of the title compound, (I), is self-assembled into an alternating organic and inorganic layered structure. The anionic layer consists of infinite zigzag chains of corner-sharing $[SbCl_6]^{3−}$ octahedra running along the b axis. Thus, (I) can be classified among the one-dimensional hybrid structures. The organic part is made up of $(dabcoH_2)^{2+}$ cations located in the holes around the corner-sharing octahedra. The layers are stacked along the a axis and water molecules connect the organic and inorganic components (Fig. 2).

The inorganic structural unit part of (I) is build up by two Sb atoms in an octahedral coordination ($[Sb1Cl_6]^{3−}$ and $[Sb2Cl_6]^{3−}$) joined by the Cl2 ion. Both octahedra are severely distorted with Sb—Cl bond lengths lying in the range of 2.5233 (18)–3.073 (2) Å for the bridging ones and 2.4277 (15)–2.8233 (17) Å for the terminal ones. The two bridging halides (Cl2 and Cl4) connecting the central octahedron to its neighbours are related *cis*, leading to zigzag chain of corner-sharing $[SbCl_6]^{3−}$ octahedra running along the b axis (Fig. 3).

It is worth noting that at room temperature the DABCO molecule crystallizes in the hexagonal system ($P6_3/m$) (Nimmo & Lucas, 1976). In our case, Cat2 seems to be more distorted than Cat1. In fact, the highest absolute value of the N—C—C—N torsion angle of 7.80 (14)° proves that both $(dabcoH_2)^{2+}$ cations exhibit deviations from ideal D_{3h} symmetry. The observed lowering symmetry (hexagonal to orthorhombic) is
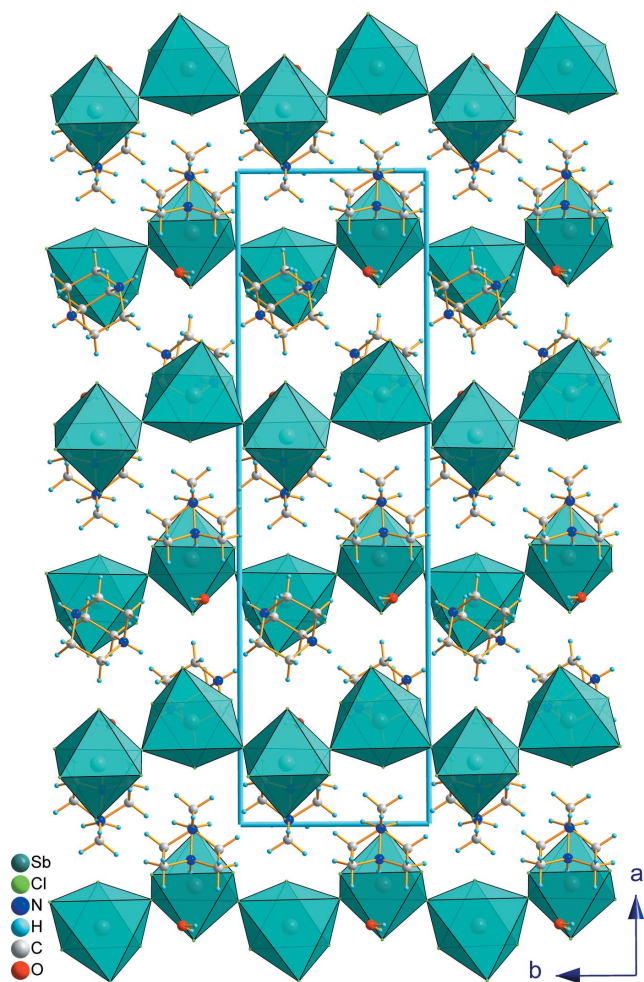
Figure 2
The organic–inorganic layered structure of (I), projected along the c axis, showing the zigzag chains of corner-sharing $[SbCl_6]^{3−}$ octahedra.
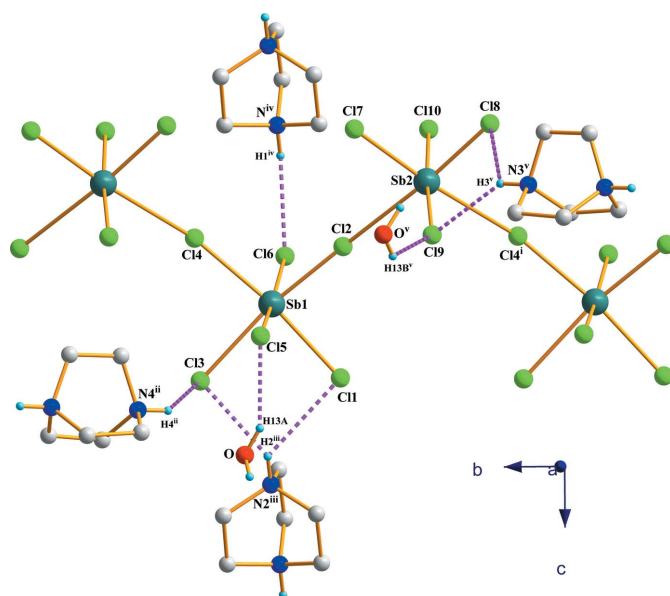
Figure 3
A magnified view of the hydrogen bonding of the inorganic chain in (I). H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry codes: (i) $x, y − 1, z$; (ii) $x, y + 1, z$; (iii) $−x + 1, −y + 1, z + \frac{1}{2}$; (iv) $−x + 1, −y + 1, z − \frac{1}{2}$; (v) $−x + \frac{3}{2}, y − \frac{1}{2}, z − \frac{1}{2}$.]

Table 1
Hydrogen-bond geometry (Å, °).

$D—H\cdots A$	$D—H$	$H\cdots A$	$D\cdots A$	$D—H\cdots A$
$N1—H1\cdots Cl6^i$	0.95	2.67	3.391 (6)	134
$N2—H2\cdots Cl1^{ii}$	0.88	2.78	3.378 (4)	126
$N2—H2\cdots Cl3^{ii}$	0.88	2.62	3.281 (6)	133
$N2—H2\cdots O^{ii}$	0.88	2.46	3.040 (7)	124
$N3—H3\cdots Cl8^{iii}$	0.89	2.82	3.418 (7)	126
$N3—H3\cdots Cl9^{iii}$	0.89	2.38	3.132 (9)	143
$N4—H4\cdots Cl3^{iv}$	0.87	2.66	3.303 (6)	131
$N4—H4\cdots O^{iv}$	0.87	2.30	3.026 (8)	143
$O—H13A\cdots Cl5$	0.84	2.43	3.185 (7)	151
$O—H13B\cdots Cl9^{iii}$	0.83	2.66	3.210 (5)	126

Symmetry codes: (i) $-x + 1, -y + 1, z + \frac{1}{2}$; (ii) $-x + 1, -y + 1, z - \frac{1}{2}$; (iii) $-x + \frac{3}{2}, y + \frac{1}{2}, z + \frac{1}{2}$; (iv) $x, y - 1, z$.
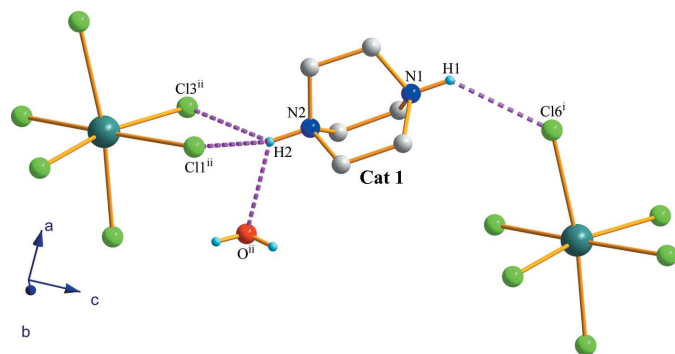
probably due to the distortion of the $(dabcoH_2)^{2+}$ cation and can be related to the complex hydrogen-bond network linking the molecular components (cations, anions and water molecules).

The studied compound crystals are transparent and the structure is noncentrosymmetric ($Pna2_1$). These are two indispensable conditions making this phase a potential promising candidate for non-linear optical (NLO) behaviour as is the case for the well-known $KTiOPO_4$ (KTP) and equivalent efficient NLO materials.
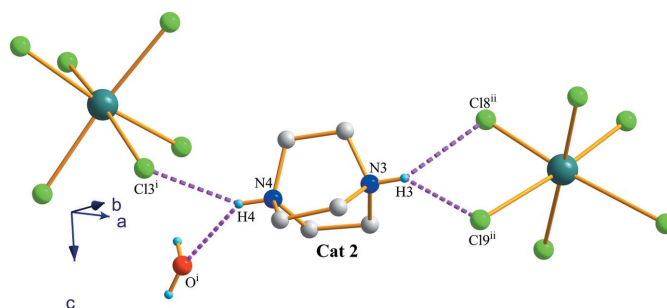
3. Supramolecular features

As shown in Fig. 3, every bi-octahedron unit is linked to four $(dabcoH_2)^{2+}$ cations and two water molecules *via* hydrogen bonds (Table 1): on one side Cat1 *via* $Cl6\cdots H1^{iv}—N1^{iv}$ and Cat 2 by $Cl8\cdots H3^v—N3^v$, $Cl9\cdots H3^v—N3^v$ [symmetry codes: (iv) $-x + 1, -y + 1, -z - \frac{1}{2}$; (v) $-x + \frac{3}{2}, y - \frac{1}{2}, z - \frac{1}{2}$] and the other side Cat1 *via* $Cl1\cdots H2^{iii}—N2^{iii}$, $Cl3\cdots H2^{iii}—N2^{iii}$ and Cat2 by $Cl3\cdots H4^{ii}—N4^{ii}$ [symmetry codes: (ii) $x, y + 1, z$; (iii) $-x + 1, -y + 1, z + \frac{1}{2}$]. The water molecules are linked by $Cl5\cdots H13A—O$ and $Cl9\cdots H13B^v—O^v$ [symmetry code: (v) $-x + \frac{3}{2}, y - \frac{1}{2}, z - \frac{1}{2}$].

Using ammonium groups, both cations (Cat1 and Cat2) are linked to the anionic chains by hydrogen bonds *via* halogenous octahedral vertices. As shown in Fig. 4, Cat1 is linked by N1—

Figure 5
The hydrogen-bonding environment of Cat 2 in (I). Only H atoms involved in hydrogen bonding have been represented. [Symmetry codes: (i) $x, y - 1, z$; (ii) $-x + \frac{3}{2}, y + \frac{1}{2}, z + \frac{1}{2}$.]

$H1\cdots Cl6^i$ hydrogen bond and three interactions between N2—H2 group, both vertices $Cl1^{ii}—Sb1^{ii}$, $Cl3^{ii}—Sb1^{ii}$ and O atom of the water molecule [symmetry codes: (i) $-x + 1, -y + 1, z + \frac{1}{2}$; (ii) $-x + 1, -y + 1, z - \frac{1}{2}$]. On the other hand, each ammonium group of Cat 2 interacts by two hydrogen bonds. N4—H4 to $Cl3^i—Sb1^i$ and the O atom and N3—H3 group to both $Cl8^{ii}—Sb2^{ii}$ and $Cl9^{ii}—Sb2^{ii}$ vertices (Fig. 5) [symmetry codes: (i) $x, y - 1, z$; (ii) $-x + \frac{3}{2}, y + \frac{1}{2}, z + \frac{1}{2}$].

As can be seen in Fig. 6, the water molecule plays an important role in the structure connectivity. It is establishing four hydrogen links joining Cat1 by $O\cdots H2^{ii}—N2^{ii}$, Cat2 through $O\cdots H4^i—N4^i$ and two $[SbCl_6]^{3-}$ octahedra *via* $O—H13A\cdots Cl5$ and $O—H13B\cdots Cl9^{iii}$ [symmetry codes: (i) $x, y + 1, z$; (ii) $-x + 1, -y + 1, z + \frac{1}{2}$; (iii) $-x + \frac{3}{2}, y + \frac{1}{2}, z + \frac{1}{2}$].

4. Database survey

A search of the Cambridge Structural Database (Version 5.36; Groom & Allen, 2014) gave 184 hits for organic–inorganic hybrid materials based on antimony chloride units. For this class of compounds with $(dabcoH_2)^{2+}$ cations, there is only one zero-dimensional compound, $(C_6H_{14}N_2)_2[Sb_2Cl_{10}]\cdot 2H_2O$

Figure 4
The hydrogen-bonding environment of Cat 1 in (I). Only H atoms involved in hydrogen bonding have been represented. [Symmetry codes: (i) $-x + 1, -y + 1, z + \frac{1}{2}$; (ii) $-x + 1, -y + 1, z - \frac{1}{2}$.]
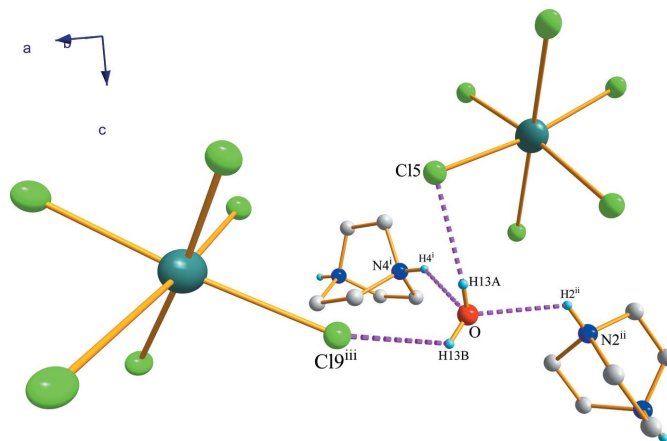

Figure 6
Water-molecule hydrogen-bonding interactions in (I). C—H bonds have been omitted for clarity. [Symmetry codes: (i) $x, y + 1, z$; (ii) $-x + 1, -y + 1, z + \frac{1}{2}$; (iii) $-x + \frac{3}{2}, y + \frac{1}{2}, z + \frac{1}{2}$.]

containing isolated $[Sb_2Cl_{10}]^{4-}$ double octahedra, $(dabcoH_2)^{2+}$ cations and water molecules (Ben Rhaiem *et al.*, 2013). Indeed, this compound is a pseudo-polymorph over the title compound. For similar one-dimensional compounds with *N,N*-dimethylethylenediammonium cations, $[(CH_3)_2NH(CH_2)_2\text{-}NH_3]^{2+}$, see: Bujack & Angel (2006). For two-dimensional compounds with $[\{Sb_2Cl_9\}_n]^{3n-}$ polyanionic layers, see: Bujack & Angel (2005); Bujack & Zaleski (2004).

5. Synthesis and crystallization

A mixture of SbCl₃ (1 mmol) and DABCO (0.5 mmol) was dissolved in a hydrochloric aqueous solution and stirred for several minutes at 353 K. Colourless crystals suitable for X-ray diffraction analysis were obtained by slow evaporation at room temperature after two weeks.

6. Refinement

Data collection and structure refinement details are summarized in Table 2. H atoms were localized from geometrical constraint conditions using adequate AFIX and DFIX *SHELXL* (Sheldrick, 2008) options and parameters were refined with a common isotropic displacement parameter. Water H atoms were found in difference Fourier maps and O—H distances were refined using DFIX and DANG soft restraints. The Flack parameter was refined despite the low Friedel pair coverage because the structure contains a sufficient number of relatively strong anomalous scatterers.

Table 2
Experimental details.

Crystal data	
Chemical formula	$(C_6H_{14}N_2)_2[Sb_2Cl_{10}]\cdot H_2O$
M_r	844.40
Crystal system, space group	Orthorhombic, $Pna2_1$
Temperature (K)	298
a, b, c (Å)	29.122 (3), 8.4029 (10), 11.358 (2)
V (Å³)	2779.4 (7)
Z	4
Radiation type	Mo $K\alpha$
μ (mm⁻¹)	2.92
Crystal size (mm)	$0.13 \times 0.06 \times 0.02$
Data collection	
Diffractometer	Enraf–Nonius CAD-4
Absorption correction	ψ scan (North *et al.*, 1968)
T_{min}, T_{max}	0.358, 0.555
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7000, 3492, 2988
R_{int}	0.041
$(\sin \theta/\lambda)_{max}$ (Å⁻¹)	0.638
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.026, 0.070, 1.09
No. of reflections	3492
No. of parameters	273
No. of restraints	5
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å⁻³)	0.72, −0.62
Absolute structure	Flack (1983), 66 Friedel pairs
Absolute structure parameter	−0.01 (3)

Computer programs: *CAD-4 EXPRESS* (Duisenberg, 1992), *XCAD4* (Harms & Wocadlo, 1995), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *DIAMOND* (Brandenburg, 2006) and *publCIF* (Westrip, 2010).

References

Ben Rhaiem, T., Boughzala, H. & Driss, A. (2013). *Acta Cryst.* E**69**, m330.

Brandenburg, K. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.

Bujack, M. & Angel, R. J. (2005). *J. Solid State Chem.* **178**, 2237–2246.

Bujack, M. & Angel, R. J. (2006). *J. Phys. Chem. B*, **110**, 10322–10331.

Bujack, M. & Zaleski, J. (2004). *J. Solid State Chem.* **177**, 3202–3211.

Duisenberg, A. J. M. (1992). *J. Appl. Cryst.* **25**, 92–96.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Groom, C. R. & Allen, F. H. (2014). *Angew. Chem. Int. Ed.* **53**, 662–671.

Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.

Leblanc, N., Mercier, N., Allain, M., Toma, O., Auban-Senzier, P. & Pasquier, C. (2012). *J. Solid State Chem.* **195**, 140–148.

Nimmo, J. K. & Lucas, B. W. (1976). *Acta Cryst.* B**32**, 348–353.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Piecha, A., Bialonska, A. & Jakubas, R. (2012). *J. Mater. Chem.* **22**, 333–335.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Crystal structure of a new hybrid antimony–halide-based compound for possible non-linear optical applications

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Computing details

Data collection: *CAD-4 EXPRESS* (Duisenberg, 1992); cell refinement: *CAD-4 EXPRESS* (Duisenberg, 1992); data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

catena-poly[[[bis(1,4-diazoniabicyclo[2.2.2]octane) [tetraachloridoantimonate(III)]-*μ*-chlorido-[tetrachloridoantimonate(III)]-*μ*-chlorido]] monohydrate]

Crystal data

$(C_6H_{14}N_2)_2[Sb_2Cl_{10}] \cdot H_2O$	$D_x = 2.018$ Mg m^{-3}
$M_r = 844.40$	Melting point: 594 K
Orthorhombic, $Pna2_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2c -2n	Cell parameters from 3492 reflections
$a = 29.122$ (3) Å	$\theta = 2.4$–27.0°
$b = 8.4029$ (10) Å	$\mu = 2.92$ mm^{-1}
$c = 11.358$ (2) Å	$T = 298$ K
$V = 2779.4$ (7) Å^3	Prism, colourless
$Z = 4$	$0.13 \times 0.06 \times 0.02$ mm
$F(000) = 1640$	

Data collection

Enraf–Nonius CAD-4 diffractometer	3492 independent reflections
Radiation source: fine-focus sealed tube	2988 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.041$
non–profiled $\omega/2\theta$ scans	$\theta_{max} = 27.0°$, $\theta_{min} = 2.3°$
Absorption correction: ψ scan (North *et al.*, 1968)	$h = -37 \rightarrow 1$
	$k = -10 \rightarrow 10$
$T_{min} = 0.358$, $T_{max} = 0.555$	$l = -1 \rightarrow 14$
7000 measured reflections	2 standard reflections every 120 min
	intensity decay: −1%

Refinement

Refinement on F^2	5 restraints
Least-squares matrix: full	Primary atom site location: structure-invariant direct methods
$R[F^2 > 2\sigma(F^2)] = 0.026$	
$wR(F^2) = 0.070$	Secondary atom site location: difference Fourier map
$S = 1.09$	
3492 reflections	Hydrogen site location: inferred from neighbouring sites
273 parameters	

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0316P)^2 + 2.3277P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.72$ e Å^{-3}

$\Delta\rho_{min} = -0.62$ e Å^{-3}

Extinction correction: *SHELXL97* (Sheldrick, 2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$

Extinction coefficient: 0.00168 (12)

Absolute structure: Flack (1983), 66 Friedel pairs

Absolute structure parameter: −0.01 (3)

Special details

Experimental. Absorption correction: North *et al.* (1968) Number of psi-scan sets used was 6 Theta correction was applied. Averaged transmission function was used. No Fourier smoothing was applied.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 > 2sigma(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*	$U_{iso}*/U_{eq}$
Sb1	0.593657 (12)	0.74481 (3)	0.49688 (3)	0.02787 (11)
Sb2	0.657146 (11)	0.24077 (3)	0.22050 (3)	0.02842 (11)
Cl1	0.57361 (5)	0.54093 (18)	0.66622 (15)	0.0452 (4)
Cl2	0.61712 (8)	0.5151 (2)	0.3671 (2)	0.0760 (6)
Cl3	0.57606 (5)	0.97355 (17)	0.67100 (15)	0.0437 (3)
Cl4	0.61408 (7)	0.9753 (2)	0.3459 (2)	0.0765 (6)
Cl5	0.67713 (6)	0.75095 (17)	0.56999 (19)	0.0522 (4)
Cl6	0.50864 (7)	0.7328 (2)	0.3883 (2)	0.0619 (5)
Cl7	0.69068 (7)	0.4519 (2)	0.09980 (17)	0.0645 (5)
Cl8	0.68555 (6)	0.0345 (2)	0.08973 (16)	0.0562 (5)
Cl9	0.74068 (5)	0.19561 (18)	0.34215 (18)	0.0430 (3)
Cl10	0.58708 (5)	0.25828 (14)	0.1013 (2)	0.0478 (4)
N1	0.49311 (19)	0.2444 (5)	0.5900 (5)	0.0390 (12)
H1	0.5113 (15)	0.2480 (6)	0.659 (6)	0.047*
N2	0.44602 (17)	0.2354 (5)	0.4084 (5)	0.0378 (11)
H2	0.4294 (4)	0.2318 (5)	0.3440 (16)	0.045*
N3	0.72003 (16)	0.3837 (5)	0.7294 (5)	0.0389 (11)
H3	0.7352 (10)	0.475 (6)	0.7261 (5)	0.047*
N4	0.6767 (2)	0.1317 (6)	0.7367 (7)	0.068 (2)
H4	0.6616 (14)	0.044 (8)	0.7384 (7)	0.082*
C1	0.5235 (2)	0.2498 (6)	0.4859 (7)	0.0455 (16)
H1A	0.5447	0.1609	0.4882	0.055*
H1B	0.5412	0.3475	0.4867	0.055*
C2	0.4952 (2)	0.2417 (8)	0.3748 (8)	0.059 (2)
H2A	0.5010	0.3347	0.3264	0.071*
H2B	0.5033	0.1476	0.3299	0.071*
C3	0.4365 (2)	0.0911 (7)	0.4788 (6)	0.0481 (17)

H3A	0.4434	−0.0030	0.4326	0.058*
H3B	0.4043	0.0880	0.5001	0.058*
C4	0.4656 (2)	0.0921 (7)	0.5887 (6)	0.0479 (15)
H4A	0.4860	0.0010	0.5891	0.057*
H4B	0.4461	0.0865	0.6579	0.057*
C5	0.4622 (2)	0.3851 (8)	0.5871 (6)	0.0510 (16)
H5A	0.4802	0.4821	0.5888	0.061*
H5B	0.4423	0.3844	0.6556	0.061*
C6	0.4333 (2)	0.3800 (6)	0.4754 (6)	0.0467 (17)
H6A	0.4010	0.3773	0.4953	0.056*
H6B	0.4391	0.4742	0.4283	0.056*
C7	0.7097 (3)	0.3302 (8)	0.6095 (6)	0.063 (2)
H7A	0.7381	0.3127	0.5668	0.075*
H7B	0.6924	0.4116	0.5685	0.075*
C8	0.6829 (4)	0.1808 (11)	0.6134 (8)	0.092 (4)
H8A	0.6532	0.1972	0.5767	0.110*
H8B	0.6989	0.0981	0.5702	0.110*
C9	0.6768 (2)	0.4080 (7)	0.7935 (7)	0.0473 (15)
H9A	0.6577	0.4839	0.7516	0.057*
H9B	0.6831	0.4505	0.8712	0.057*
C10	0.6519 (2)	0.2512 (6)	0.8042 (8)	0.0479 (18)
H10A	0.6502	0.2195	0.8862	0.057*
H10B	0.6208	0.2617	0.7744	0.057*
C11	0.7484 (3)	0.2641 (9)	0.7941 (10)	0.081 (3)
H11A	0.7525	0.2964	0.8754	0.097*
H11B	0.7784	0.2535	0.7577	0.097*
C12	0.7221 (3)	0.1056 (10)	0.7878 (11)	0.107 (4)
H12A	0.7391	0.0303	0.7399	0.129*
H12B	0.7190	0.0612	0.8662	0.129*
O	0.65412 (17)	0.8095 (7)	0.8407 (6)	0.0637 (14)
H13A	0.662 (3)	0.760 (8)	0.780 (4)	0.070*
H13B	0.674 (2)	0.788 (9)	0.890 (5)	0.070*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sb1	0.0343 (2)	0.02365 (18)	0.0257 (2)	−0.00135 (12)	0.00135 (16)	−0.00099 (18)
Sb2	0.03007 (18)	0.02876 (19)	0.0264 (2)	0.00021 (12)	−0.00028 (17)	−0.0021 (2)
Cl1	0.0497 (9)	0.0439 (7)	0.0421 (8)	−0.0053 (6)	−0.0007 (7)	−0.0001 (7)
Cl2	0.0915 (15)	0.0703 (11)	0.0662 (14)	0.0140 (11)	0.0062 (12)	−0.0308 (11)
Cl3	0.0418 (8)	0.0419 (7)	0.0473 (8)	0.0013 (6)	−0.0043 (7)	0.0077 (7)
Cl4	0.0833 (14)	0.0808 (13)	0.0655 (14)	−0.0231 (11)	−0.0066 (13)	0.0276 (12)
Cl5	0.0457 (8)	0.0532 (9)	0.0578 (12)	−0.0055 (7)	0.0018 (8)	0.0039 (8)
Cl6	0.0538 (10)	0.0626 (11)	0.0695 (14)	−0.0031 (8)	0.0168 (10)	0.0005 (10)
Cl7	0.0750 (12)	0.0718 (11)	0.0466 (10)	−0.0361 (9)	−0.0079 (10)	0.0184 (9)
Cl8	0.0638 (11)	0.0643 (9)	0.0406 (9)	0.0285 (8)	−0.0105 (8)	−0.0222 (8)
Cl9	0.0429 (7)	0.0501 (7)	0.0360 (7)	0.0012 (7)	0.0007 (7)	−0.0113 (7)
Cl10	0.0447 (8)	0.0333 (7)	0.0652 (12)	0.0041 (5)	−0.0225 (8)	−0.0034 (7)

N1	0.044 (3)	0.038 (3)	0.035 (3)	0.001 (2)	−0.007 (2)	0.000 (2)
N2	0.039 (3)	0.036 (2)	0.038 (3)	0.0019 (18)	−0.007 (2)	0.003 (2)
N3	0.046 (3)	0.033 (2)	0.038 (3)	−0.0052 (18)	0.004 (3)	−0.003 (2)
N4	0.089 (5)	0.028 (2)	0.088 (5)	−0.010 (3)	0.049 (4)	0.000 (3)
C1	0.034 (3)	0.041 (3)	0.062 (5)	−0.004 (2)	0.004 (3)	0.003 (3)
C2	0.045 (4)	0.089 (6)	0.043 (4)	−0.001 (3)	0.003 (3)	−0.001 (4)
C3	0.057 (4)	0.031 (2)	0.057 (4)	−0.008 (2)	−0.021 (4)	0.009 (3)
C4	0.053 (4)	0.046 (3)	0.044 (4)	−0.011 (3)	−0.002 (3)	0.013 (3)
C5	0.055 (4)	0.051 (3)	0.047 (4)	0.012 (3)	0.000 (3)	−0.009 (3)
C6	0.057 (4)	0.032 (3)	0.051 (4)	0.010 (3)	−0.014 (3)	−0.004 (3)
C7	0.110 (7)	0.051 (3)	0.026 (3)	−0.031 (4)	0.009 (4)	−0.004 (3)
C8	0.133 (9)	0.087 (6)	0.054 (5)	−0.071 (6)	0.028 (6)	−0.035 (5)
C9	0.052 (4)	0.038 (3)	0.052 (4)	0.002 (3)	0.008 (3)	−0.015 (3)
C10	0.047 (4)	0.051 (4)	0.046 (4)	0.004 (3)	0.017 (3)	0.004 (3)
C11	0.056 (5)	0.111 (7)	0.075 (7)	0.033 (5)	−0.011 (5)	−0.004 (5)
C12	0.103 (8)	0.071 (5)	0.148 (10)	0.047 (5)	0.079 (7)	0.060 (6)
O	0.055 (3)	0.067 (3)	0.069 (4)	0.017 (2)	−0.010 (3)	−0.016 (3)

Geometric parameters (Å, º)

Sb1—Cl2	2.5233 (18)	C1—H1B	0.9700
Sb1—Cl5	2.5695 (18)	C2—H2A	0.9700
Sb1—Cl1	2.6411 (17)	C2—H2B	0.9700
Sb1—Cl4	2.654 (2)	C3—C4	1.507 (9)
Sb1—Cl6	2.768 (2)	C3—H3A	0.9700
Sb1—Cl3	2.8051 (17)	C3—H3B	0.9700
Sb2—Cl8	2.4277 (15)	C4—H4A	0.9700
Sb2—Cl7	2.4457 (17)	C4—H4B	0.9700
Sb2—Cl10	2.4532 (16)	C5—C6	1.521 (9)
Sb2—Cl9	2.8233 (17)	C5—H5A	0.9700
Sb2—Cl2	3.073 (2)	C5—H5B	0.9700
Sb2—Cl4[i]	2.9291 (19)	C6—H6A	0.9700
N1—C1	1.477 (9)	C6—H6B	0.9700
N1—C5	1.487 (8)	C7—C8	1.480 (10)
N1—C4	1.510 (7)	C7—H7A	0.9700
N1—H1	0.9496	C7—H7B	0.9700
N2—C3	1.478 (7)	C8—H8A	0.9700
N2—C6	1.481 (7)	C8—H8B	0.9700
N2—C2	1.483 (9)	C9—C10	1.509 (7)
N2—H2	0.8784	C9—H9A	0.9700
N3—C7	1.465 (8)	C9—H9B	0.9700
N3—C9	1.469 (8)	C10—H10A	0.9700
N3—C11	1.494 (9)	C10—H10B	0.9700
N3—H3	0.8858	C11—C12	1.538 (11)
N4—C10	1.456 (8)	C11—H11A	0.9700
N4—C12	1.462 (12)	C11—H11B	0.9700
N4—C8	1.471 (11)	C12—H12A	0.9700
N4—H4	0.8565	C12—H12B	0.9700

C1—C2	1.508 (11)	O—H13A	0.84 (2)
C1—H1A	0.9700	O—H13B	0.82 (2)
Cl2—Sb1—Cl5	87.02 (7)	C1—C2—H2B	110.0
Cl2—Sb1—Cl1	89.38 (7)	H2A—C2—H2B	108.4
Cl5—Sb1—Cl1	89.25 (6)	N2—C3—C4	109.8 (5)
Cl2—Sb1—Cl4	96.90 (9)	N2—C3—H3A	109.7
Cl5—Sb1—Cl4	88.98 (6)	C4—C3—H3A	109.7
Cl1—Sb1—Cl4	173.38 (6)	N2—C3—H3B	109.7
Cl2—Sb1—Cl6	87.36 (7)	C4—C3—H3B	109.7
Cl5—Sb1—Cl6	172.33 (7)	H3A—C3—H3B	108.2
Cl1—Sb1—Cl6	95.92 (6)	C3—C4—N1	108.1 (5)
Cl4—Sb1—Cl6	86.51 (6)	C3—C4—H4A	110.1
Cl2—Sb1—Cl3	170.28 (7)	N1—C4—H4A	110.1
Cl5—Sb1—Cl3	86.05 (5)	C3—C4—H4B	110.1
Cl1—Sb1—Cl3	83.72 (5)	N1—C4—H4B	110.1
Cl4—Sb1—Cl3	89.79 (6)	H4A—C4—H4B	108.4
Cl6—Sb1—Cl3	100.12 (5)	N1—C5—C6	109.3 (5)
Cl8—Sb2—Cl7	92.23 (8)	N1—C5—H5A	109.8
Cl8—Sb2—Cl10	89.35 (6)	C6—C5—H5A	109.8
Cl7—Sb2—Cl10	88.82 (7)	N1—C5—H5B	109.8
Cl8—Sb2—Cl9	84.83 (5)	C6—C5—H5B	109.8
Cl7—Sb2—Cl9	91.59 (6)	H5A—C5—H5B	108.3
Cl10—Sb2—Cl9	174.17 (6)	N2—C6—C5	108.3 (5)
Cl4^i—Sb2—Cl10	87.62 (5)	N2—C6—H6A	110.0
Cl2—Sb2—Cl10	86.49 (6)	C5—C6—H6A	110.0
Cl4^i—Sb2—Cl9	91.63 (5)	N2—C6—H6B	110.0
Cl2—Sb2—Cl9	99.35 (5)	C5—C6—H6B	110.0
Cl8—Sb2—Cl4^i	84.24 (6)	H6A—C6—H6B	108.4
Cl4^i—Sb2—Cl2	98.36 (5)	N3—C7—C8	109.9 (6)
Cl2—Sb2—Cl7	84.89 (6)	N3—C7—H7A	109.7
Cl2—Sb2—Cl8	174.98 (6)	C8—C7—H7A	109.7
Cl4^i—Sb2—Cl7	175.00 (6)	N3—C7—H7B	109.7
C1—N1—C5	108.7 (5)	C8—C7—H7B	109.7
C1—N1—C4	109.6 (5)	H7A—C7—H7B	108.2
C5—N1—C4	110.6 (5)	N4—C8—C7	109.3 (6)
C1—N1—H1	109.3	N4—C8—H8A	109.8
C5—N1—H1	109.3	C7—C8—H8A	109.8
C4—N1—H1	109.3	N4—C8—H8B	109.8
C3—N2—C6	110.4 (5)	C7—C8—H8B	109.8
C3—N2—C2	110.4 (5)	H8A—C8—H8B	108.3
C6—N2—C2	110.1 (5)	N3—C9—C10	109.3 (5)
C3—N2—H2	108.6	N3—C9—H9A	109.8
C6—N2—H2	108.6	C10—C9—H9A	109.8
C2—N2—H2	108.6	N3—C9—H9B	109.8
C7—N3—C9	109.1 (5)	C10—C9—H9B	109.8
C7—N3—C11	111.4 (6)	H9A—C9—H9B	108.3
C9—N3—C11	108.9 (6)	N4—C10—C9	108.7 (5)

C7—N3—H3	109.1	N4—C10—H10A	109.9
C9—N3—H3	109.1	C9—C10—H10A	109.9
C11—N3—H3	109.1	N4—C10—H10B	109.9
C10—N4—C12	110.1 (7)	C9—C10—H10B	109.9
C10—N4—C8	111.6 (7)	H10A—C10—H10B	108.3
C12—N4—C8	108.0 (8)	N3—C11—C12	106.5 (7)
C10—N4—H4	109.0	N3—C11—H11A	110.4
C12—N4—H4	109.0	C12—C11—H11A	110.4
C8—N4—H4	109.0	N3—C11—H11B	110.4
N1—C1—C2	110.0 (5)	C12—C11—H11B	110.4
N1—C1—H1A	109.7	H11A—C11—H11B	108.6
C2—C1—H1A	109.7	N4—C12—C11	109.8 (6)
N1—C1—H1B	109.7	N4—C12—H12A	109.7
C2—C1—H1B	109.7	C11—C12—H12A	109.7
H1A—C1—H1B	108.2	N4—C12—H12B	109.7
N2—C2—C1	108.3 (6)	C11—C12—H12B	109.7
N2—C2—H2A	110.0	H12A—C12—H12B	108.2
C1—C2—H2A	110.0	H13A—O—H13B	105 (4)
N2—C2—H2B	110.0		

Symmetry code: (i) *x*, *y*−1, *z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···Cl6[ii]	0.95	2.67	3.391 (6)	134
N2—H2···Cl1[iii]	0.88	2.78	3.378 (4)	126
N2—H2···Cl3[iii]	0.88	2.62	3.281 (6)	133
N2—H2···O[iii]	0.88	2.46	3.040 (7)	124
N3—H3···Cl8[iv]	0.89	2.82	3.418 (7)	126
N3—H3···Cl9[iv]	0.89	2.38	3.132 (9)	143
N4—H4···Cl3[i]	0.87	2.66	3.303 (6)	131
N4—H4···O[i]	0.87	2.30	3.026 (8)	143
O—H13*A*···Cl5	0.84	2.43	3.185 (7)	151
O—H13*B*···Cl9[iv]	0.83	2.66	3.210 (5)	126

Symmetry codes: (i) *x*, *y*−1, *z*; (ii) −*x*+1, −*y*+1, *z*+1/2; (iii) −*x*+1, −*y*+1, *z*−1/2; (iv) −*x*+3/2, *y*+1/2, *z*+1/2.