

Aminoguanidinium(2+) amino- guanidinium(1+) hexachloro- antimonate(III) at 295 and 92 K

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The crystal and molecular structure of the title compound, $(\text{CH}_7\text{N}_4)(\text{CH}_8\text{N}_4)[\text{SbCl}_6]$, has been determined at 295 and 92 K. It is composed of isolated $[\text{SbCl}_6]^{3-}$ octahedra and aminoguanidinium mono- and dications. One of four of the crystallographically inequivalent aminoguanidinium cations is disordered at both temperatures. Two crystallographically inequivalent $[\text{SbCl}_6]^{3-}$ octahedra were found to possess three significantly longer Sb—Cl bonds than three other octahedra. The shorter bonds are in the range 2.456 (2)–2.577 (2) Å, whereas the longer ones are between 2.705 (2) and 2.931 (2) Å. Each short Sb—Cl bond is located *trans* to a long bond. It is argued that this deformation is caused by N—H...Cl hydrogen bonds.

Comment

Alkylammonium halogenoantimonates(III) are a group of organic–inorganic salts. They are characterized by a general formula $R_a\text{Sb}_b\text{X}_c$ (R = organic cation, X = Cl, Br or I). Their anionic sublattices are generally built from distorted $[\text{SbX}_6]^{3-}$ octahedra, which are isolated or joined with each other by corners, edges or faces, typically forming more complex polyanions (Sobczyk *et al.*, 1997). Organic cations fill the voids in the anionic sublattice and are typically bound to the anionic sublattice by N—H...X hydrogen bonds.

Many salts of this class are characterized by the presence of phase transitions, some of them to polar phases (Jakubas, 1986). The mechanisms of these transformations are often associated with a decrease in rotational freedom of the organic cations on lowering the temperature (Jakubas, 1990). The slowing down of reorientational motions of the cations leads to instabilities in the crystal lattice, resulting in a lowering of the symmetry. This is often associated with changes in the hydrogen-bonding scheme. Generally, in this class of chloroantimonates(III), the environment of the Sb^{III} atom is almost always significantly distorted. The Sb—Cl bond lengths differ from each other sometimes by as much as *ca* 0.9 Å (Bujak & Zaleski, 1998). In a series of our earlier work we correlated the differences in the Sb—Cl bond lengths to: (i) primary deformation resulting from the tendency of $[\text{SbCl}_6]^{3-}$ octahedra to

share Cl atoms with each other, resulting in the formation of polyanionic units; and (ii) secondary deformation resulting from the presence of N—H...Cl hydrogen bonds which additionally distort $[\text{SbCl}_6]^{3-}$ octahedra, shifting Cl atoms in the direction of a positive charge located on the cation.

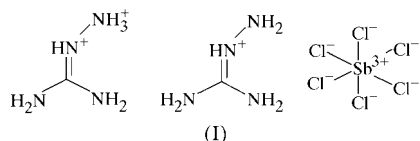
We have also noticed that significant changes in Sb—Cl bond lengths take place with decreasing temperature. The largest changes are associated with changes in the hydrogen-bonding scheme across a phase transition [0.326 (4) Å; Zaleski & Pietraszko, 1996]. Smaller changes [0.049 (2) Å; Bujak & Zaleski, 1998] are correlated with a decrease in the donor/acceptor distances within hydrogen bonds with decreasing temperature.

The present work results from our earlier investigations in which chloroantimonates(III) with guanidinium cations were characterized by the presence of solid–solid phase transitions correlated with changes in the molecular motion of the organic sublattice. The structure, phase transitions and molecular motion in these crystals have been studied by differential scanning calorimetry, dielectric, pyroelectric, IR, NMR and X-ray diffraction methods (Zaleski, 1995, and references therein). Recently, we have extended those studies to N,N,N',N' -tetramethylguanidinium derivatives. By changing the ratio of SbCl_3 to N,N,N',N' -tetramethylguanidine between 1:10 and 10:1, we were able to obtain only one salt of RSbCl_4 stoichiometry (Bujak *et al.*, 1999). In the crystal lattice, the $[\text{SbCl}_6]^{3-}$ octahedra share two *cis* edges with two other neighbours, forming an infinite one-dimensional zigzag chain. The N,N,N',N' -tetramethylguanidinium cations are located between the inorganic chains and bound to the anions by N—H...Cl hydrogen bonds. We have observed significant changes in the Sb—Cl bond lengths and Cl—Sb—Cl angles on decreasing the temperature from 295 to 92 K. These were correlated with the changes in the donor/acceptor distances of N—H...Cl hydrogen bonds to the Cl atoms in question.

Aminoguanidine is a closely related derivative of guanidine. It was selected for the multiple possibilities it holds for creating intermolecular hydrogen-bond networks. The aminoguanidinium cation can exist in either a monocationic or a dicationic form (Bukvetskii *et al.*, 1990). In each form, its structure may be represented by four resonance tautomers (Koskinen *et al.*, 1996). Until now, only one salt containing an aminoguanidinium monocation has been reported for the whole family of halogenoantimonates(III) and halogenobismuthates(III) $(\text{CH}_7\text{N}_4)_3[\text{BiCl}_6]$ (Davidovich *et al.*, 1995). Its structure consists of isolated $[\text{BiCl}_6]^{3-}$ anions and aminoguanidinium $(\text{CH}_7\text{N}_4)^+$ cations connected to an anionic sublattice through N—H...Cl hydrogen bonds.

The structure was solved in both Pc and $P2/c$ space groups. The refinement in the $P2/c$ space led to a much higher R factor (*ca* 13%) than in the Pc group. Additionally, displacement parameters for several N atoms had non-positive values. We therefore selected the Pc space group. The asymmetric unit of aminoguanidinium(2+) aminoguanidinium(1+) hexachloroantimonate(III), (I), consists of two significantly distorted $[\text{SbCl}_6]^{3-}$ octahedra and four aminoguanidinium cations (Fig. 1). Two of the cations are monocations and the two

others dications. The dications (AG1 and AG2), in contrast with the monocations (AG3 and AG4), possess an additional H atom located on the terminal ammonium group.



One of the monocations (AG4) is disordered. At 295 K, the disorder is realized by the presence of two positions for C4 and N15, with occupancy factors of 0.6 (for C41 and N151) and 0.4 (for C42 and N152) (Fig. 2). At 92 K, only atom N15 is split between two positions, N151 and N152, with occupancy factors of 0.75 and 0.25, respectively. The C4 atom at 92 K only shows an enlarged displacement ellipsoid. All H atoms of the ordered cations were located from a difference Fourier synthesis. The N—H bond lengths were set to an idealized distance of 0.90 Å and were included as riding atoms. The CN_3 guanidinium moiety in each cation at both temperatures is nearly planar, with an average deviation of *ca* 0.01 (1) Å from the mean plane. The terminal N atom of the NH_3^+ ammonium groups deviates from the CN_3 guanidine plane by 0.24 (1) and 0.64 (1) Å for AG1 and AG2 at 295 K, and by 0.30 (1) and 0.74 (1) Å, respectively, at 92 K. In the case of the AG3 and AG4 cations, they are almost planar, with an average deviation of 0.01 (1) Å at 295 K and 0.02 (2) Å at 92 K for all non-H atoms of the CN_3 plane. The NH_3^+ groups are twisted about C—N bonds by 12.7 (9)° in the case of AG1 and -33.4 (9)° in the case of AG2 at 295 K, and 15.4 (6) and -39.0 (5)° at 92 K, respectively. It should be noted that the dications differ in the conformation of the NH_3^+ group, which is rotated by roughly 45° in AG1 with respect to the AG2 cation. Analogous

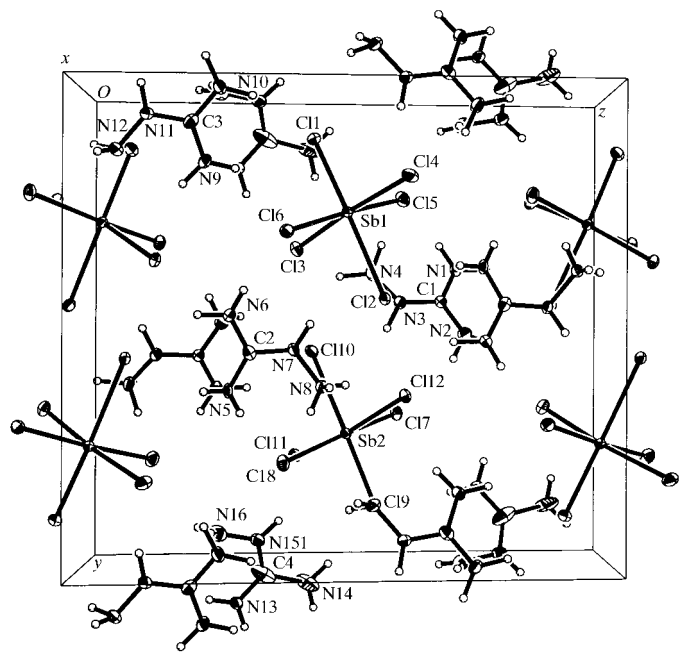


Figure 1

Packing diagram of (I) at 92 K viewed along the *x* axis. Only one position of the disordered N15 atom is shown for clarity. Displacement ellipsoids are plotted at the 50% probability level.

differences between the aminoguanidinium cation in the monocationic (Bryden, 1957; Koskinen *et al.*, 1996; Akella & Keszler, 1994) and dicationic (Ross *et al.*, 1998; Ross *et al.*, 1999) forms have been observed previously.

There are two crystallographically inequivalent $[\text{SbCl}_6]^{3-}$ anions in the crystal structure. In each of the octahedra are observed three shorter and three longer Sb—Cl bonds distributed mutually in a *trans* fashion. At 295 K, the three shorter Sb—Cl bonds are in the range 2.469 (2)–2.577 (2) Å for Sb1 and between 2.456 (2) and 2.518 (2) Å for the Sb2 octahedron. The three longer Sb—Cl distances range from 2.705 (2) to 2.902 (2) Å for the Sb1 octahedron and from 2.807 (2) to 2.931 (2) Å for Sb2 (Table 1). The average Sb—Cl bond lengths are 2.665 (12) Å for Sb1 and 2.675 (12) Å for Sb2. These are similar to the value of 2.643 (6) Å [2.652 (6) Å when corrected for thermal motion] found in undistorted octahedral $[\text{SbCl}_6]^{3-}$ (Schroeder & Jacobson, 1973). They differ from the value characteristic of this reference structure by 0.022 (18) Å for Sb1 and by 0.032 (18) Å for the Sb2 octahedron. The largest deviations are 0.259 (8) and 0.288 (8) Å for Sb1 and Sb2, respectively. The Cl—Sb—Cl angles involving Cl atoms mutually *cis* to each other range from 82.65 (5) to 93.61 (6)° for Sb1 and from 83.32 (5) to 97.50 (6)° for the Sb2 octahedron (Table 1).

At 92 K, small changes are observed in the lengths of the Sb—Cl bonds and Cl—Sb—Cl angles. The shortening of one Sb—Cl bond length leads to the elongation of the corresponding *trans* Sb—Cl bond. The values of shortening/elongation in appropriate pairs of Sb—Cl bonds are of comparable magnitude. The largest changes of the Sb—Cl bond lengths are observed for the *trans* pair consisting of the Sb1—Cl5 and Sb1—Cl6 bonds [average 0.033 (6) Å] in the Sb1 octahedron and the Sb2—Cl11 and Sb2—Cl12 *trans* pair [average 0.030 (6) Å] in the Sb2 octahedron (Tables 1 and 3). In comparison with Sb—Cl bond lengths of the reference octahedron, the deviation from 2.643 (6) Å is larger at 92 K than at room temperature. The changes in the Sb—Cl bond lengths in the two crystallographically independent octahedra are comparable at both temperatures, whereas the Cl—Sb—Cl angles show significant differences. The largest change in the Cl—Sb—Cl bond angles for the Sb1 octahedron amounts to 0.72 (8)° (Cl2—Sb1—Cl4), whereas for the second one it is much larger, 3.77 (8)° (Cl8—Sb2—Cl10).

All the aminoguanidinium cations are joined to $[\text{SbCl}_6]^{3-}$ octahedra through N—H...Cl hydrogen bonds. Each amino-

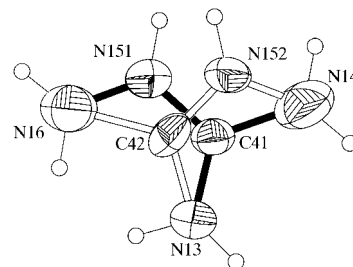


Figure 2

Type of disorder of the AG4 aminoguanidinium(1+) cation at 295 K. Displacement ellipsoids are plotted at the 50% probability level.

guanidinium group forms several hydrogen bonds. Tables 2 and 4 present hydrogen bonds with H...Cl distances shorter than *ca* 2.6 Å. The geometries of the hydrogen bonds at both temperatures are comparable. The N...Cl distances for the shortest N—H...Cl hydrogen bonds are in the range 3.118 (6)–3.444 (6) Å at room temperature and decrease to 3.100 (4)–3.353 (4) Å at 92 K. Among these N—H...Cl interactions, the largest changes in N...Cl distances and N—H...Cl angles are noted for Cl2, Cl8, Cl10 and Cl12. They are reflected in changes of the Cl—Sb—Cl angles: Cl2—Sb1—Cl4 0.72 (8)°, Cl8—Sb2—Cl10 3.77 (8)°, Cl9—Sb2—Cl10 2.06 (11)° and Cl8—Sb2—Cl12 1.69 (8)°.

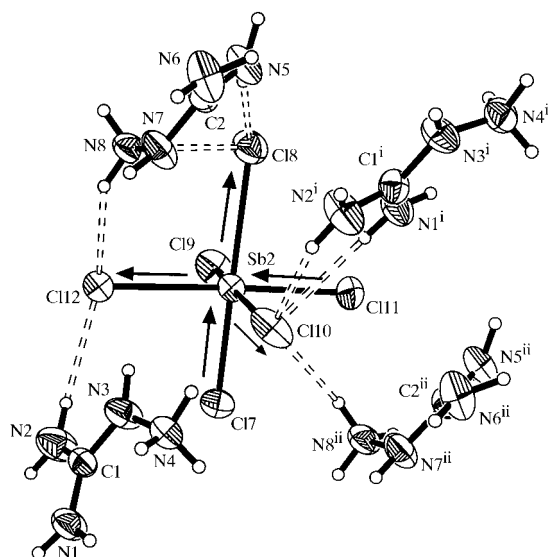


Figure 3 Hydrogen-bonding scheme (indicated by dashed lines) involving the Sb2 octahedron in the structure of (I) at 295 K. Arrows denote relative shortening/elongation of appropriate Sb—Cl bonds in relation to undistorted octahedral [SbCl₆]³⁻ (Schroeder & Jacobson, 1973). Displacement ellipsoids are plotted at the 50% probability level. [Symmetry codes: (i) *x*, 1 - *y*, *z* - ½; (ii) *x* - 1, *y*, *z*.]

In Fig. 3, the hydrogen-bonding scheme to the Sb2 octahedron is depicted. Arrows denote relative elongation or shortening of Sb—Cl bonds in comparison with the reference structure. It should be noted that the hydrogen bonds are formed to Cl atoms of the elongated Sb—Cl bonds. The H⁺...Cl⁻ interactions to the Cl8, Cl10 and Cl12 atoms lead to a shift of the lone electron pair at Sb^{III} in the direction of the positive charge located on cations, resulting in elongation of the appropriate Sb—Cl bonds and shortening of Sb—Cl bonds located in the *trans* position. The analogous situation is observed in the other octahedron.

Experimental

Compound (I) was prepared by the reaction of aminoguanidinium hydrocarbonate and antimony trichloride (molar ratio from 1:1 to 4:1) in an aqueous solution of concentrated hydrochloric acid. The solution obtained was evaporated slowly at constant room temperature, resulting in single crystals of suitable dimensions for the X-ray structure analysis.

Compound (I) at 295 K

Crystal data

(CH₇N₄)(CH₈N₄)[SbCl₆]
M_r = 485.67
 Monoclinic, *Pc*
a = 7.522 (2) Å
b = 13.856 (3) Å
c = 15.544 (3) Å
 β = 94.75 (3)°
V = 1614.5 (6) Å³
Z = 4
D_x = 1.998 Mg m⁻³
D_m = 1.99 (1) Mg m⁻³

D_m measured by flotation method
 CCl₄/CHBr₃
 Mo Kα radiation
 Cell parameters from 40 reflections
 θ = 7–14°
 μ = 2.695 mm⁻¹
T = 295 (2) K
 Irregular, colourless
 0.40 × 0.40 × 0.30 mm

Data collection

Kuma KM-4 diffractometer
 ω-θ scans
 Absorption correction: ψ scan (Sheldrick, 1990)
T_{min} = 0.368, *T_{max}* = 0.446
 5690 measured reflections
 2867 independent reflections (plus 2823 Friedel-related reflections)

5052 reflections with *I* > 2σ(*I*)
 θ_{max} = 25.06°
h = -8 → 8
k = 0 → 16
l = -18 → 18
 2 standard reflections every 50 reflections
 intensity decay: 1.0%

Table 1

Selected geometric parameters (Å, °) for (I) at 295 K.

Sb1—Cl1	2.469 (2)	Sb2—Cl7	2.456 (2)
Sb1—Cl2	2.902 (2)	Sb2—Cl8	2.931 (2)
Sb1—Cl3	2.532 (2)	Sb2—Cl9	2.463 (2)
Sb1—Cl4	2.802 (2)	Sb2—Cl10	2.874 (2)
Sb1—Cl5	2.577 (2)	Sb2—Cl11	2.518 (2)
Sb1—Cl6	2.705 (2)	Sb2—Cl12	2.807 (2)
Cl1—Sb1—Cl2	173.19 (6)	Cl7—Sb2—Cl8	179.34 (5)
Cl1—Sb1—Cl3	93.59 (6)	Cl7—Sb2—Cl9	90.77 (6)
Cl1—Sb1—Cl4	91.53 (5)	Cl7—Sb2—Cl10	84.28 (5)
Cl1—Sb1—Cl5	91.59 (6)	Cl7—Sb2—Cl11	92.51 (6)
Cl1—Sb1—Cl6	92.15 (6)	Cl7—Sb2—Cl12	86.63 (5)
Cl2—Sb1—Cl3	90.68 (5)	Cl8—Sb2—Cl9	89.37 (6)
Cl2—Sb1—Cl4	84.13 (5)	Cl8—Sb2—Cl10	95.50 (5)
Cl2—Sb1—Cl5	93.61 (5)	Cl8—Sb2—Cl11	86.85 (5)
Cl2—Sb1—Cl6	82.65 (5)	Cl8—Sb2—Cl12	93.99 (5)
Cl3—Sb1—Cl4	174.79 (5)	Cl9—Sb2—Cl10	170.96 (7)
Cl3—Sb1—Cl5	91.31 (6)	Cl9—Sb2—Cl11	89.36 (6)
Cl3—Sb1—Cl6	88.54 (5)	Cl9—Sb2—Cl12	97.50 (6)
Cl4—Sb1—Cl5	89.49 (6)	Cl10—Sb2—Cl11	83.32 (5)
Cl4—Sb1—Cl6	90.33 (5)	Cl10—Sb2—Cl12	89.77 (5)
Cl5—Sb1—Cl6	176.25 (5)	Cl11—Sb2—Cl12	173.10 (5)

Table 2

Hydrogen-bonding geometry (Å, °) for (I) at 295 K.

<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—H12...Cl4 ⁱ	0.90	2.41	3.310 (5)	173
N1—H11...Cl10 ⁱⁱ	0.90	2.51	3.323 (5)	150
N2—H22...Cl12	0.90	2.43	3.291 (5)	159
N2—H21...Cl10 ⁱⁱ	0.90	2.44	3.266 (5)	152
N4—H41...Cl2 ⁱ	0.90	2.33	3.182 (5)	158
N4—H42...Cl5	0.90	2.41	3.183 (5)	144
N5—H52...Cl8	0.90	2.35	3.227 (5)	164
N5—H51...Cl2 ⁱⁱⁱ	0.90	2.58	3.303 (5)	138
N6—H62...Cl6	0.90	2.41	3.238 (5)	154
N7—H7...Cl2	0.90	2.49	3.195 (5)	136
N8—H83...Cl8	0.90	2.22	3.118 (6)	174
N8—H82...Cl12	0.90	2.47	3.223 (5)	142
N8—H81...Cl10 ^v	0.90	2.63	3.374 (6)	141
N11—H111...Cl4 ^v	0.90	2.60	3.444 (6)	157

Symmetry codes: (i) *x* - 1, *y*, *z*; (ii) *x*, 1 - *y*, ½ + *z*; (iii) *x*, 1 - *y*, *z* - ½; (iv) 1 + *x*, *y*, *z*; (v) *x*, -*y*, *z* - ½.

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.024$
 $wR(F^2) = 0.065$
 $S = 1.097$
 5690 reflections
 329 parameters
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.03P)^2 + 1.1741P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.002$
 $\Delta\rho_{\max} = 0.59 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.36 \text{ e } \text{Å}^{-3}$
 Extinction correction: *SHELXL97* (Sheldrick, 1997)
 Extinction coefficient: 0.0045 (2)
 Absolute structure: (Flack, 1983)
 Flack parameter = -0.04 (2)

Compound (I) at 92 K

Crystal data

$(\text{CH}_7\text{N}_4)(\text{CH}_8\text{N}_4)[\text{SbCl}_6]$
 $M_r = 485.67$
 Monoclinic, *Pc*
 $a = 7.426$ (2) Å
 $b = 13.836$ (3) Å
 $c = 15.403$ (3) Å
 $\beta = 95.27$ (3)°
 $V = 1575.9$ (6) Å³
 $Z = 4$

$D_x = 2.047 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 40 reflections
 $\theta = 7\text{--}14^\circ$
 $\mu = 2.761 \text{ mm}^{-1}$
 $T = 92.0$ (5) K
 Irregular, colourless
 $0.40 \times 0.40 \times 0.30 \text{ mm}$

Data collection

Kuma KM-4 diffractometer
 ω - θ scans
 Absorption correction: ψ scan (Sheldrick, 1990)
 $T_{\min} = 0.360$, $T_{\max} = 0.437$
 5579 measured reflections
 2804 independent reflections (plus 2775 Friedel-related reflections)

5334 reflections with $I > 2\sigma(I)$
 $\theta_{\max} = 25.06^\circ$
 $h = -8 \rightarrow 8$
 $k = -16 \rightarrow 0$
 $l = -18 \rightarrow 17$
 2 standard reflections every 50 reflections
 intensity decay: 1.1%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.019$
 $wR(F^2) = 0.048$
 $S = 1.093$
 5579 reflections
 319 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0216P)^2 + 2.2178P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.48 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.56 \text{ e } \text{Å}^{-3}$
 Absolute structure: (Flack, 1983)
 Flack parameter = -0.026 (15)

Table 3

Selected geometric parameters (Å, °) for (I) at 92 K.

Sb1—Cl1	2.4633 (11)	Sb2—Cl7	2.4511 (12)
Sb1—Cl2	2.9108 (11)	Sb2—Cl8	2.9380 (13)
Sb1—Cl3	2.5067 (12)	Sb2—Cl9	2.4508 (11)
Sb1—Cl4	2.8253 (13)	Sb2—Cl10	2.8995 (12)
Sb1—Cl5	2.5472 (13)	Sb2—Cl11	2.4888 (12)
Sb1—Cl6	2.7405 (13)	Sb2—Cl12	2.8379 (12)
Cl1—Sb1—Cl2	173.85 (4)	Cl7—Sb2—Cl8	177.50 (3)
Cl1—Sb1—Cl3	92.89 (4)	Cl7—Sb2—Cl9	90.08 (4)
Cl1—Sb1—Cl4	92.07 (4)	Cl7—Sb2—Cl10	83.03 (3)
Cl1—Sb1—Cl5	91.35 (4)	Cl7—Sb2—Cl11	92.50 (4)
Cl1—Sb1—Cl6	91.70 (4)	Cl7—Sb2—Cl12	85.21 (4)
Cl2—Sb1—Cl3	90.06 (4)	Cl8—Sb2—Cl9	87.49 (4)
Cl2—Sb1—Cl4	84.85 (3)	Cl8—Sb2—Cl10	99.28 (3)
Cl2—Sb1—Cl5	93.95 (3)	Cl8—Sb2—Cl11	86.89 (4)
Cl2—Sb1—Cl6	83.00 (3)	Cl8—Sb2—Cl12	95.68 (3)
Cl3—Sb1—Cl4	174.79 (3)	Cl9—Sb2—Cl10	168.90 (4)
Cl3—Sb1—Cl5	91.69 (4)	Cl9—Sb2—Cl11	89.08 (4)
Cl3—Sb1—Cl6	88.18 (4)	Cl9—Sb2—Cl12	97.76 (4)
Cl4—Sb1—Cl5	89.78 (4)	Cl10—Sb2—Cl11	82.56 (3)
Cl4—Sb1—Cl6	90.09 (4)	Cl10—Sb2—Cl12	90.35 (3)
Cl5—Sb1—Cl6	176.95 (3)	Cl11—Sb2—Cl12	172.78 (3)

Table 4

Hydrogen-bonding geometry (Å, °) for (I) at 92 K.

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
N1—H12...Cl4 ⁱ	0.90	2.40	3.300 (4)	176
N1—H11...Cl10 ⁱⁱ	0.90	2.46	3.279 (3)	152
N2—H22...Cl12	0.90	2.45	3.267 (3)	151
N2—H21...Cl10 ⁱⁱⁱ	0.90	2.44	3.255 (3)	151
N4—H41...Cl2 ⁱ	0.90	2.26	3.129 (4)	164
N4—H42...Cl5	0.90	2.35	3.152 (4)	148
N5—H52...Cl8	0.90	2.31	3.192 (3)	168
N5—H51...Cl2 ⁱⁱⁱ	0.90	2.44	3.269 (3)	153
N6—H62...Cl6	0.90	2.33	3.206 (4)	166
N7—H7...Cl2	0.90	2.42	3.172 (4)	142
N8—H83...Cl8	0.90	2.27	3.100 (4)	153
N8—H82...Cl12	0.90	2.33	3.176 (4)	157
N8—H81...Cl10 ^{iv}	0.90	2.51	3.270 (4)	142
N11—H111...Cl4 ^v	0.90	2.49	3.353 (4)	160

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

For both compounds, data collection: *Kuma Diffraction Software* (Kuma, 1996); cell refinement: *Kuma Diffraction Software*; data reduction: *Kuma Diffraction Software* (Kuma, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BJ1014). Services for accessing these data are described at the back of the journal.

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