References

Candlish, E. F. K., Michael, T. K., Neal, J. A., Lingafelter, E. C. & Rose, N. J. (1978). *Inorg. Chem.* **17**, 1383–1394.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 99–103 and 149. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Lind, M. D., Hamor, J. J., Hamor, T. A. & Hoard, J. L. (1964). *Inorg. Chem.* **3**, 34–43.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Passer, E., White, J. G. & Cheng, K. L. (1977). *Inorg. Chim. Acta*, **24**, 13–23.

Pozhidaev, A. I., Polynova, T. N., Porai-Koshits, M. A. & Neronova, N. N. (1973). *Zh. Strukt. Khim.* **14**, 570–571.

Richards, S., Pedersen, B., Silverton, J. V. & Hoard, J. L. (1964). *Inorg. Chem.* **3**, 27–33.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Solans, X., Font-Altaba, M., Oliva, J. & Herrera, J. (1983). *Acta Cryst.* C**39**, 435–438.

Stezowski, J. J., Countryman, R. & Hoard, J. L. (1973). *Inorg. Chem.* **12**, 1749–1754.

Acta Cryst. (1985). C**41**, 1022–1024

Tris(trimethylammonium) Nonachlorodiantimonate(III), $[NH(CH_3)_3]_3[Sb_2Cl_9]$

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Abstract. $M_r = 742{\cdot}94$, monoclinic, Pc, $a = 10{\cdot}085\ (3)$, $b = 9{\cdot}072\ (4)$, $c = 15{\cdot}459\ (6)$ Å, $\beta = 90{\cdot}17\ (3)°$, $V = 1414\ (2)$ Å^3, $Z = 2$, $D_x = 1{\cdot}744\ (2)$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71069$ Å, $\mu = 27{\cdot}8$ cm^{-1}, $F(000) = 720$, $T = 296$ K, final $R = 0{\cdot}034$ for 2384 independent observed reflections. The structure contains $SbCl_3$ pyramids with Sb—Cl bonds ranging from 2·401 (3) to 2·473 (3) Å. The Cl^- anions interconnect the $SbCl_3$ groups into a two-dimensional network by forming bridges with Sb—Cl lengths ranging from 2·883 (3) to 3·316 (3) Å. The cations are connected by N—H···Cl hydrogen bonds to the Sb_2Cl_9 layers. One hydrogen bond is bifurcated and very weak.

Introduction. This work is part of our study of the crystal structures of alkylammonium metal halogenides. In the particular case of Sb^{III} chlorides and bromides the following ions can be obtained: $[SbX_4]^-$, $[SbX_5]^{2-}$, $[SbX_6]^{3-}$ and the enneahalide $[Sb_2X_9]^{3-}$. In these compounds the metal shows a tendency towards distorted octahedral coordination with some rather long Sb—X bonds, which is attributed to the aspherical distribution of lone-pair electrons at Sb^{III}. This work describes the crystal structure determination of $[NH(CH_3)_3]_3[Sb_2Cl_9]$.

Experimental. Crystals by slow evaporation of an aqueous solution of $[NH(CH_3)_3]Cl$ and $SbCl_3$. Crystal 0·2 × 0·2 × 0·6 mm. Precession photographs indicated monoclinic symmetry. Cell constants from setting angles of 15 reflections with $6 < \theta < 10°$. Syntex $P2_1$ diffractometer, Nb filter. Hemisphere up to $2\theta = 50°$, $(\sin\theta/\lambda)_{\max} = 0{\cdot}59$ Å^{-1}, range of hkl: $h\ 0 \rightarrow 11$, $k \pm 10$, $l \pm 18$, $\omega/2\theta$ scan. Total number of reflections 6757; 2465 independent. Three standard reflections every 60 reflections remained stable. Background corrections by profile analysis (Blessing, Coppens & Becker, 1974). Absorption correction, transmission range 0·57–0·62. Averaging of equivalent reflections, $R_{\rm int} = 0{\cdot}035$. 2384 reflections with $I > 0$ used. Weighting scheme $w(I) = 1/[\sigma^2(I) + (0{\cdot}03I)^2]$. Structure determination by Patterson and Fourier methods. H atoms at N were calculated but not refined; those at C could not be determined. Refinement on F. Extinction coefficient $g = 11{\cdot}2\ (2) \times 10^{-3}$ (Larson, 1969). $(\Delta/\sigma)_{\max} = 0{\cdot}1$. Final difference Fourier map peaks $< 0{\cdot}35$ e Å^{-3}. Final $R = 0{\cdot}034$, $wR = 0{\cdot}029$, $S = 1{\cdot}21$. Scattering factors and f' from *International Tables for X-ray Crystallography* (1974); use of f'' did not distinguish between enantiomorphs, consequently f'' was not used in final refinement. Calculations with *XRAY* system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972).

Discussion. The positional parameters are reported in Table 1,[†] bond lengths and angles in Table 2. A view of the structure is shown in Fig. 1. The structure contains $SbCl_3$ pyramids with Sb—Cl bond lengths ranging from 2·401 (3) to 2·473 (3) Å. The average Cl—Sb—Cl angle is 89·6 (1)°. The Cl^- anions form bridges with

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[†] Lists of structure factors, anisotropic thermal parameters, and bond distances and angles in the cations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42133 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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considerably longer Sb—Cl distances [2·883 (3) to 3·316 (3) Å] between the $SbCl_3$ subunits. Each Sb atom accepts three long Sb—Cl contacts which are, however, still much shorter than the sum of the van der Waals radii of Sb and Cl (4·0 Å according to Pauling, 1960). The six Cl atoms bonded to each Sb atom form a distorted octahedron, with the short and long contacts in *trans* positions. As a consequence, the $SbCl_3$ subunit is considerably deformed compared with gaseous $SbCl_3$ (Konaka & Kimura, 1973) which has an Sb—Cl bond length of 2·333 (3) Å and a bond angle of 97·2 (9)°. A coordination very similar to that in $[NH(CH_3)_3]_3[Sb_2Cl_9]$ is found in the crystal structure of α-$Cs_3Sb_2Cl_9$ (Kihara & Sudo, 1971) and β-$Cs_3Sb_2Cl_9$ (Kihara & Sudo, 1974). In the crystal structure of $SbCl_3$, on the other hand, no coordinative SbCl bonds are shorter than 3·457 (1) Å; thus an almost undistorted $SbCl_3$ molecule is observed with bond lengths between 2·340 (2) and 2·368 (1) Å (Lipka, 1979). Almost undistorted $SbCl_3$ subunits are also observed in many other $SbCl_3$ adducts (Demaldè, Mangia, Nardelli, Pelizzi & Vidoni Tani, 1972; Lipka & Mootz, 1978). The $Sb_2Cl_9^{3-}$ unit forms an infinite two-dimensional network in the *bc* plane (Fig. 2). The cations are connected by N—H···Cl hydrogen bonds (Table 3) to these layers.

Table 1. *Positional parameters and equivalent values of the anisotropic thermal parameters*

$U_{eq} = \frac{1}{3}$ trace **U**.

	x	y	z	U_{eq}(Å^2)
Sb(1)	0·0	0·21025 (6)	0·0	0·0452 (4)
Sb(2)	0·30281 (5)	0·71061 (6)	0·17241 (3)	0·0433 (4)
Cl(1)	0·4202 (3)	0·5155 (3)	0·2456 (2)	0·063 (1)
Cl(2)	−0·1191 (3)	0·0385 (3)	−0·0892 (2)	0·065 (2)
Cl(3)	−0·1274 (3)	0·4131 (3)	−0·0638 (2)	0·070 (2)
Cl(4)	0·1518 (3)	−0·0526 (3)	0·0570 (2)	0·087 (2)
Cl(5)	0·4266 (3)	0·6605 (3)	0·0424 (2)	0·062 (1)
Cl(6)	−0·1700 (3)	0·1766 (3)	0·1118 (2)	0·067 (2)
Cl(7)	0·4774 (2)	0·8882 (3)	0·2146 (2)	0·065 (1)
Cl(8)	0·1196 (3)	0·4841 (3)	0·1190 (2)	0·069 (2)
Cl(9)	0·1862 (2)	0·7738 (3)	0·3591 (2)	0·074 (2)
N(1)	−0·1482 (8)	0·6728 (9)	0·1431 (6)	0·066 (5)
N(2)	0·4489 (8)	0·8069 (9)	0·4802 (8)	0·080 (6)
N(3)	0·1508 (11)	−0·1971 (11)	−0·1498 (7)	0·101 (9)
C(11)	−0·242 (1)	0·564 (1)	0·1787 (11)	0·100 (9)
C(12)	−0·114 (1)	0·779 (2)	0·2097 (7)	0·100 (9)
C(13)	−0·205 (1)	0·746 (1)	0·0653 (7)	0·089 (8)
C(21)	0·511 (1)	0·961 (1)	0·4778 (10)	0·098 (9)
C(22)	0·534 (1)	0·695 (1)	0·4372 (7)	0·093 (8)
C(23)	0·422 (1)	0·759 (1)	0·5720 (9)	0·104 (9)
C(31)	0·129 (2)	−0·344 (2)	−0·1139 (11)	0·15 (1)
C(32)	0·296 (2)	−0·183 (1)	−0·1666 (12)	0·13 (1)
C(33)	0·088 (3)	−0·160 (3)	−0·2334 (13)	0·25 (2)
H(1)*	−0·070	0·624	0·125	0·10
H(2)	0·368	0·813	0·449	0·10
H(3)	0·112	−0·134	−0·108	0·11

* H atoms not refined.

Table 2. *Bond distances* (Å) *and angles* (°) *in the* $Sb_2Cl_9^{3-}$ *network, and hydrogen-bond details*

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

Sb(1)—Cl(2)	2·401 (3)	Sb(2)—Cl(1)	2·410 (3)
Sb(1)—Cl(3)	2·450 (3)	Sb(2)—Cl(5)	2·412 (3)
Sb(1)—Cl(6)	2·457 (3)	Sb(2)—Cl(7)	2·473 (3)
Sb(1)—Cl(4)	2·966 (3)	Sb(2)—Cl(8)	2·883 (3)
Sb(1)—Cl(8)	3·316 (3)	Sb(2)—Cl(9)	3·172 (3)
Sb(1)—Cl(9^{i})	2·884 (3)	Sb(2)—Cl(4^{ii})	3·178 (3)
Cl(2)—Sb(1)—Cl(3)	89·74 (9)	Cl(1)—Sb(2)—Cl(7)	90·35 (9)
Cl(2)—Sb(1)—Cl(6)	88·53 (10)	Cl(1)—Sb(2)—Cl(8)	85·69 (9)
Cl(2)—Sb(1)—Cl(4)	84·59 (10)	Cl(1)—Sb(2)—Cl(9)	83·61 (8)
Cl(2)—Sb(1)—Cl(8)	170·21 (8)	Cl(1)—Sb(2)—Cl(4^{ii})	173·71 (9)
Cl(2)—Sb(1)—Cl(9^{i})	85·67 (8)	Cl(5)—Sb(2)—Cl(7)	88·44 (9)
Cl(3)—Sb(1)—Cl(6)	90·60 (9)	Cl(5)—Sb(2)—Cl(8)	87·70 (9)
Cl(3)—Sb(1)—Cl(4)	173·22 (9)	Cl(5)—Sb(2)—Cl(9)	170·52 (8)
Cl(3)—Sb(1)—Cl(8)	81·36 (9)	Cl(5)—Sb(2)—Cl(4^{ii})	84·70 (9)
Cl(3)—Sb(1)—Cl(9^{i})	90·00 (8)	Cl(7)—Sb(2)—Cl(8)	174·47 (8)
Cl(6)—Sb(1)—Cl(4)	92·97 (10)	Cl(7)—Sb(2)—Cl(9)	84·72 (8)
Cl(6)—Sb(1)—Cl(8)	87·50 (8)	Cl(7)—Sb(2)—Cl(4^{ii})	92·71 (9)
Cl(6)—Sb(1)—Cl(9^{i})	174·16 (8)	Cl(8)—Sb(2)—Cl(9)	98·65 (8)
Cl(4)—Sb(1)—Cl(8)	104·54 (8)	Cl(8)—Sb(2)—Cl(4^{ii})	90·87 (9)
Cl(4)—Sb(1)—Cl(9^{i})	85·87 (8)	Cl(9)—Sb(2)—Cl(4^{ii})	102·14 (8)
Cl(8)—Sb(1)—Cl(9^{i})	98·33 (7)		
Cl(1)—Sb(2)—Cl(5)	89·90 (9)		
Sb(1)—Cl(4)—Sb(2^{iii})	162·96 (12)	Sb(1^{iv})—Cl(9)—Sb(2)	160·13 (9)
Sb(1)—Cl(8)—Sb(2)	157·62 (10)		

Hydrogen bonding

N—H···Cl	N—H (Å)	H···Cl (Å)	N···Cl (Å)	N—H—Cl (°)
N(1)—H(1)···Cl(8)	0·95	2·29	3·220 (9)	165
N(2)—H(2)···Cl(9)	0·95	2·33	3·254 (10)	166
N(3)—H(3)···Cl(4)	0·95	2·69	3·455 (11)	139
N(3)—H(3)···Cl(2)	0·95	2·82	3·588 (11)	138

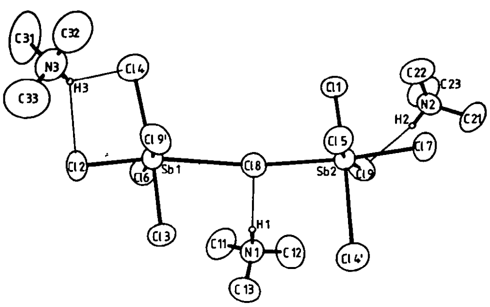

Fig. 1. Fragment of the structure, showing the atomic numbering scheme, Sb coordination and hydrogen bonding. The thermal ellipsoids are the 50% probability surfaces.

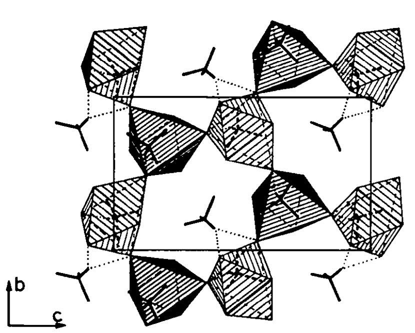

Fig. 2. Schematic view of the structure along **a**, showing the two-dimensional network. The $SbCl_6$ octahedra are represented by their surfaces, the $NH(CH_3)_3$ tetrahedra by bonds.

Two cations show N—H···Cl contacts of about 3·20 Å corresponding to hydrogen bonds of intermediate strength. These cations point with their methyl groups towards the next $Sb_2Cl_9^{3-}$ layers and are expected to constitute the bonding in the **a** direction by electrostatic interactions. The third cation which occupies the free space in a twelve-membered $(\text{—Sb—Cl—})_6$ ring shows a very weak bifurcated hydrogen bond. The large thermal parameters show this cation to be disordered.

References

BLESSING, R. H., COPPENS, P. & BECKER, P. (1974). *J. Appl. Cryst.* **7**, 488–492.

DEMALDÉ, A., MANGIA, A., NARDELLI, M., PELIZZI, G. & VIDONI TANI, M. E. (1972). *Acta Cryst.* **B28**, 147–150.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

KIHARA, K. & SUDO, T. (1971). *Z. Kristallogr.* **134**, 142–144.

KIHARA, K. & SUDO, T. (1974). *Acta Cryst.* **B30**, 1088–1093.

KONAKA, S. & KIMURA, M. (1973). *Bull. Chem. Soc. Jpn*, **46**, 404–407.

LARSON, A. C. (1969). *Crystallographic Computing*, edited by F. R. AHMED, pp. 291–294. Copenhagen: Munksgaard.

LIPKA, A. (1979). *Acta Cryst.* **B35**, 3020–3022.

LIPKA, A. & MOOTZ, D. (1978). *Z. Anorg. Allg. Chem.* **440**, 217–236.

PAULING, L. (1960). *The Nature of the Chemical Bond*, p. 260. Ithaca: Cornell Univ. Press.

STEWART, J. M., KRUGER, G. J., AMMON, H. L., DICKINSON, C. W. & HALL, S. R. (1972). The *XRAY* system–version of June 1972. Tech. Rep. TR-192. Computer Science Center, Univ. of Maryland, College Park, Maryland.

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Structure of (6,13-Diacetyl-5,12-dimethyl-1,4,8,11-tetraazacyclotetradeca-4,6,11,13-tetraenato)nickel(II) 7,7,8,8-Tetracyano-*p*-quinodimethane (1/1), $C_{16}H_{22}N_4NiO_2.C_{12}H_4N_4$*

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Abstract. $M_r = 565{\cdot}3$, monoclinic, $P2_1/n$, $a = 7{\cdot}111\,(1)$, $b = 14{\cdot}191\,(3)$, $c = 13{\cdot}062\,(4)$ Å, $\beta = 93{\cdot}21\,(2)°$, $V = 1316{\cdot}0\,(5)$ Å^3, $Z = 2$, $D_x = 1{\cdot}43$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 0{\cdot}776$ mm^{-1}, $F(000) = 588$, $T = 293$ K, final $R = 0{\cdot}045$ for 1405 observed reflections. The nickel(II) ion lies in a square-planar environment surrounded by the four N atoms of the tetradentate macrocyclic ring [Ni—N distances are 1·828 (3) and 1·887 (3) Å]. The structure consists of mixed stacks of neutral DADMTANi and TCNQ molecules. The molecules stack alternately in infinite columns, parallel to the *a* axis, the spacing and dihedral angle between the mean planes being 3·45 (5) Å and 6·1 (5)° respectively. The electrical conductivity of the title complex measured along the shortest axis of the crystal is 0·01 Ω^{-1} m^{-1}, which is larger than the value obtained for iodine-doped DADMTANi.

Introduction. Much attention has been focused on the study of materials that, by virtue of their crystalline packing arrangements, exhibit anisotropy in certain intensive variables including electrical, optical and magnetic behaviour (Ferraro, 1982). Among these systems are macrocyclic complexes that exhibit upon doping an increase in their electrical conductivity (Petersen, Schramm, Stojakovic, Hoffman & Marks, 1977; Phillips & Hoffman, 1977) and a great variety of complexes formed with TCNQ whose properties vary from insulators to conductors. Most of these complexes are purely organic; few examples are reported that contain an inorganic complex (Inoue & Inoue, 1983).

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