Fig. 2. Stereoscopic diagram of the crystal packing (c axis vertical, view down a axis).

hydrogen bonding is important to stabilize the crystal. The bridged-hydroxo groups are hydrogen-bonded to the nitrate ion. The distances $O(3)-H\cdots O(4) = 3\cdot20$ (2) and $O(3)-H\cdots O(5) = 2\cdot94$ (2) Å [transformation on O(4) and O(5): $-x$, $1-y$, $-z$] and the angles $Pt-O(3)\cdots O(4) = 83\cdot7$ (4), $Pt'-O(3)\cdots O(4) = 111\cdot1$ (4), $Pt-O(3)\cdots O(5) = 116\cdot6$ (4) and $Pt'-O(3)\cdots O(5) = 113\cdot0$ (4)°, close to the tetrahedral value.

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1H+,1'H+-2,2'-Bipyridinium Di-μ-chloro-bis[dichloro(phenyl)antimonate(III)]

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Abstract. $[C_{10}H_{10}N_2][Sb_2Cl_6(C_6H_5)_2]$, $M_r = 768\cdot6$, monoclinic, $I2/c$, $a = 14\cdot095$ (9), $b = 11\cdot649$ (5), $c = 15\cdot954$ (8) Å, $\beta = 98\cdot07$ (6)°, $V = 2593\cdot6$ Å^3, $Z = 4$, $D_x = 1\cdot968$ Mg m^{-3}, Ag $K\alpha$, $\lambda = 0\cdot56087$ Å, $\mu = 1\cdot4$ mm^{-1}, $F(000) = 1480$, $T = 291$ (1) K, final $R = 0\cdot028$ for 2072 $[I > 3\sigma(I)]$ unique diffractometer data. Two $C_6H_5SbCl_2$ units are linked by two slightly unsymmetrically bridging Cl atoms to form the anionic moiety $(C_6H_5)_2Sb_2Cl_6$ with each Sb having distorted square-planar (pseudo-octahedral) coordination neglecting a very weak intermolecular Sb$\cdots$Cl contact. Phenyl groups are in apical positions. No bonding contacts exist to the bipyridinium cation.

Introduction. The first structural proposal made for halomonophenylantimonates(III) was based on Mössbauer data, but no decision was possible between monomeric units $[C_6H_5SbX_3]^-$ with pseudo trigonal-

bipyramidal structure and dimers $[(C_6H_5)_2Sb_2X_6]^{2-}$ ($X = Cl$, Br, I) with a pseudo-octahedral environment for Sb (Alonzo, Bertazzi, Di Bianca & Gibb, 1981). We subsequently established that in the bromo derivative [with $(C_6H_5)_4As^+$ as counter cation] the anion is a bridged dimer $[(C_6H_5)_2Sb_2Br_6]^{2-}$ (Preut, Huber, Alonzo & Bertazzi, 1986). We then synthesized a new chloromonophenylantimonate(III) compound with 2,2'-bipyridinium as cation which contains an analogous dimeric anion in the solid state.

Experimental. $[bpyH_2][(C_6H_5)_2Sb_2Cl_6]$ (bpy = 2,2'-bipyridine) was prepared from a solution of $[NH_4]-[C_6H_5SbCl_5]$ in $1M$ HCl, which was reduced by adding an excess of solid Na_2SO_3 in small amounts under vigorous stirring. The title compound precipitated on addition of bpy. It was recrystallized from water/methanol acidified with HCl. Colourless plates, m.p. 411–413 K. Crystal size $0.03 \times 0.26 \times 0.35$ mm; $\omega/2\theta$ scan, scan speed between 0.87 and 2.5° min^{-1} in ω; Nonius CAD-4 diffractometer, graphite-monochromated Ag $K\alpha$ radiation; lattice parameters from least-squares fit using 20 reflexions up to $2\theta = 42.4°$; four standard reflexions recorded every 3 h, only random deviations; 3216 reflexions, $1 \le \theta \le 22°$, max. $(\sin\theta)/\lambda = 0.67$ Å^{-1}, $-23 \le h \le 23$, $0 \le k \le 15$, $0 \le l \le 21$; 2072 unique reflexions with $I > 3\sigma(I)$ (1018 unobserved) ($R_{int} = 0.027$) used for structure deter-

mination; Lorentz–polarization correction and absorption correction *via* ψ scans; max./min. transmission 1.00/0.82; systematic absences hkl $h+k+l = 2n+1$, $h0l$ $l = 2n+1$ conform to space groups $I2/c$ and Ic, $N(z)$ test indicates centrosymmetric space group. Structure solution *via* Patterson function, ΔF syntheses and full-matrix least-squares refinement on F with anisotropic temperature factors for all non-H atoms in space group $I2/c$; 146 refined parameters; H atoms in geometrically calculated positions (C—H 0.95 Å), $w^{-1} = [\sigma^2(I) + (0.04F_o^2)^2]^{1/2}$, $S = 1.75$, $R = 0.028$, $wR = 0.047$, max. $\Delta/\sigma < 0.00$, largest peak in final ΔF map ± 0.5 (2) e Å^{-3}; complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974); programs: *Enraf–Nonius Structure Determination Package* (Frenz, 1981), *ORTEP* (Johnson, 1976).

Fig. 1. General view of part of the crystal structure showing the numbering scheme.

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å$^2 \times 10^3$)

$$U_{eq} = (\tfrac{1}{3}\pi^2)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Sb(1)	0.16278 (2)	0.10397 (3)	0.20651 (2)	30
Cl(1)	0.2027 (1)	−0.0991 (1)	0.2056 (1)	47
Cl(2)	0.0027 (1)	0.0685 (2)	0.1302 (1)	53
Cl(3)	0.3668 (1)	0.1323 (1)	0.2890 (1)	42
N(1)	0.0557 (3)	0.5471 (4)	0.3314 (3)	35
C(1)	0.2161 (4)	0.1321 (4)	0.0872 (3)	29
C(2)	0.1885 (4)	0.0544 (4)	0.0212 (3)	32
C(3)	0.2231 (5)	0.0669 (6)	−0.0553 (4)	41
C(4)	0.2846 (5)	0.1574 (6)	−0.0661 (3)	39
C(5)	0.3125 (5)	0.2312 (5)	−0.0010 (4)	43
C(6)	0.2789 (4)	0.2170 (5)	0.0765 (3)	35
C(11)	0.0848 (4)	0.5387 (6)	0.4139 (4)	46
C(12)	0.0766 (5)	0.6310 (7)	0.4666 (4)	56
C(13)	0.0404 (5)	0.7312 (6)	0.4326 (5)	58
C(14)	0.0104 (5)	0.7395 (6)	0.3454 (5)	51
C(15)	0.0167 (4)	0.6453 (5)	0.2951 (4)	35

Table 2. *Bond distances* (Å) *and angles* (°) *in* $[C_{10}H_{10}N_2][(C_6H_5)_2Sb_2Cl_6]$

C—C bond distances are in the range 1.354 (7) to 1.402 (7) Å [mean value 1.379 (8) Å] and C(15)—N(1) 1.362 (7), C(11)—N(1) 1.326 (8) Å.

Bond angles C—C—C (in the rings) are in the range 119.1 (6) to 120.5 (5)° [mean value 119.8 (5)°] and C(14)—C(15)—N(1) 118.7 (6), C(15)—N(1)—C(11) 122.3 (5), N(1)—C(11)—C(12) 120.4 (6)°.

Sb(1)—Cl(1)	2.432 (2)	Sb(1)—Cl(3^i)	3.103 (1)
Sb(1)—Cl(2)	2.444 (1)	Sb(1)—C(1)	2.167 (5)
Sb(1)—Cl(3)	3.007 (1)	C(15)—C(15^{ii})	1.451 (8)

Cl(1)—Sb(1)—Cl(2)	91.75 (6)	Cl(2)—Sb(1)···Cl(2^{ii})	72.98 (4)
Cl(1)—Sb(1)—Cl(3)	84.35 (5)	Cl(3)—Sb(1)—Cl(3^i)	90.26 (4)
Cl(1)—Sb(1)—Cl(3^i)	174.41 (5)	Cl(3)—Sb(1)—C(1)	86.5 (1)
Cl(1)—Sb(1)—C(1)	91.9 (1)	Cl(3)—Sb(1)···Cl(2^{ii})	110.94 (4)
Cl(1)—Sb(1)···Cl(2^{ii})	93.64 (5)	Cl(3^i)—Sb(1)—C(1)	86.3 (1)
Cl(2)—Sb(1)—Cl(3)	174.59 (5)	Cl(3^i)—Sb(1)···Cl(2^{ii})	89.67 (4)
Cl(2)—Sb(1)—Cl(3^i)	93.53 (5)	C(1)—Sb(1)···Cl(2^{ii})	162.1 (1)
Cl(2)—Sb(1)—C(1)	89.9 (1)		

Symmetry code: (i) $\tfrac{1}{2}-x$, $\tfrac{1}{2}-y$, $\tfrac{1}{2}-z$; (ii) $-x, y, \tfrac{1}{2}-z$.
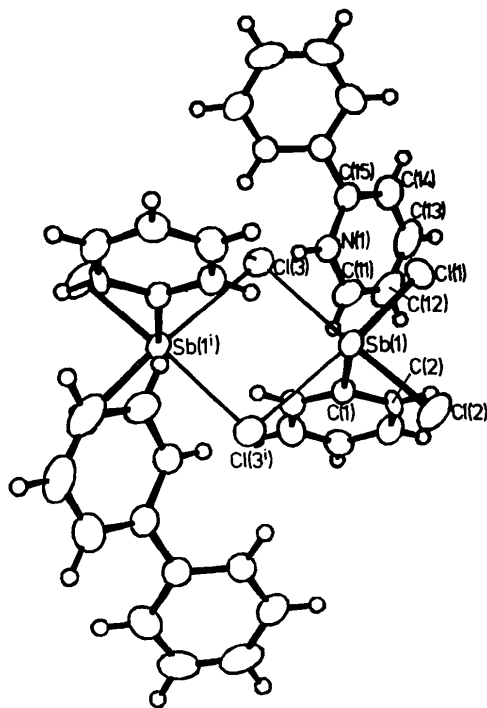
Discussion. The structure of the title compound is shown in Fig. 1. Positional parameters and equivalent values of the anisotropic temperature factors are given in Table 1,* bond lengths and angles in Table 2. The unit cell contains four $(C_6H_5)_2Sb_2Cl_6$ moieties and four $C_{10}H_{10}N_2$ moieties. The arrangement shown in Fig. 1 resides on a center of symmetry. In this arrangement the $C_{10}H_{10}N_2$ moieties have point symmetry 2. In $(C_6H_5)_2Sb_2Cl_6$ two $C_6H_5SbCl_2$ units are linked by two bridging Cl atoms. The two non-equivalent Sb—Cl bond distances in the bridge are significantly different [difference 0·096 (1) Å] and more than 0·563 (1) Å longer than the terminal Sb—Cl bonds. The atoms bound to the Sb atom form a distorted square pyramid with C(phenyl) in the apical position. Aside from these five bonds at the Sb atom there exists an intermolecular Sb···Cl(2) $(\bar{x}, y, \frac{1}{2}-z)$ contact [3·756 (1) Å], which is somewhat shorter than the sum of the corresponding van der Waals radii of 3·87 Å (Bondi, 1964). The distance between Sb and the Cl atom which makes this intermolecular contact is significantly longer than the other terminal Sb—Cl bond lengths, and apparently does not seriously influence the

coordination spheres around the two Sb atoms of the anionic unit. *Trans* to each phenyl ligand there is ample space for the lone pair at antimony. If the inter-molecular Sb···Cl contact is not considered, the atoms around Sb form a distorted pseudo-octahedron. A similar geometry is found in the anion of $[(C_6H_5)_4As]$-$[(C_6H_5)_2Sb_2Br_4]$, in which two $C_6H_5SbBr_2$ units are linked *via* two unsymmetrically bridging Br atoms with Sb—Br bond distances of 2·957 (1) and 3·187 (1) Å (Preut, Huber, Alonzo & Bertazzi, 1986). The ring atoms of the bipyridinium unit do not deviate significantly from planarity and have a torsion angle $C(14)—C(15)—C(15^{ii})—C(14^{ii})$ of 34·96 (2)°.

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Structure of [V(CO)₃(PMe₃)₄][V(CO)₆]*

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Abstract. Tricarbonyltetrakis(trimethylphosphine)-vanadium(1+) hexacarbonylvanadate(1−), [V(CO)₃{(CH₃)₃P}₄][V(CO)₆], $M_r = 658·3$, monoclinic, $P2_1/n$, $a = 12·901$ (3), $b = 16·137$ (3), $c = 15·519$ (3) Å, $\beta = 95·96$ (1)°, $V = 3213·3$ Å³, $Z = 4$, $D_x = 1·361$ Mg m⁻³, $\lambda(Cu K\alpha_1) = 1·54056$ Å, $\mu = 7·22$ mm⁻¹, $F(000) = 1360$, $T = 295$ K, $R = 0·105$ for 773 observed reflections. The structure consists of pairs of ions: $V(CO)_6^-$ which is approximately octahedral, and $V(CO)_3(PMe_3)_4^+$, a hepta-coordinated vanadium cation having the configuration of a monocapped octahedron in which a PMe₃ ligand caps a face defined by three carbonyl groups.

Introduction. The title compound was prepared during an attempted synthesis of *fac*-V(CO)₃(PMe₃)₃ (McCall, Morton & Preston, 1985), and is an example of a comparatively rare (Drew, 1977) hepta-coordinated vanadium compound. These compounds usually have structures based on the pentagonal bipyramid (Drew, 1977, 1982), but the title compound has a structure based on a monocapped octahedron.