Both are slightly longer than those of the corresponding tpp complex (Johnson & Scheidt, 1978). The Mo atom is displaced 0·13 Å out of the plane defined by the four porphyrinato N atoms toward the terminal oxo ligand. The Mo—N bond distance is 2·111 (4) Å, also slightly longer than the value found in the tpp complex.

The porphyrinato core is planar within 0·06 Å, but is slightly domed. The bond lengths and angles are normal compared with those reported for the planar porphyrins. The two porphyrin rings are twisted by 22·3° with respect to one another. The interplanar distance between mean nitrogen planes is 3·65 Å. The interplanar distance between the mean porphyrin planes is 3·54 Å, considerably shorter than that in the tpp complex (3·85 Å) where the approach of the two porphyrin cores is hindered by the bulky phenyl groups. As shown in Fig. 1, all peripheral ethyl groups are oriented outside, permitting better π–π interaction between the two porphyrin cores.

When we attempted to crystallize $[Mo(oep)]_2$ from toluene/heptane, the title compound crystallized in space group $C2/m$ (C_{2h}^3) of the monoclinic system with $a = 20·13$ (8), $b = 14·327$ (6), $c = 24·155$ (10) Å, $\beta = 103·54$ (1)°, $V = 6773$ Å^3. However, owing to severe disordering of solvent molecules, this structure did not refine well and was abandoned.

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Tris(2,6-dimethylphenyl)hydroxystibonium Iodide

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Abstract. $[Sb(C_8H_9)_3(OH)]I$, $M_r = 581·14$, monoclinic, $P2_1/c$, $a = 10·584$ (2), $b = 11·184$ (3), $c = 19·292$ (4) Å, $\beta = 94·60$ (2)°, $V = 2276$ (2) Å^3, $Z = 4$, $D_x = 1·70$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 25·7$ cm^{-1}, $F(000) = 1136$, $T = 294$ K, $R = 0·023$ for 4167 reflections with $I > 3\sigma(I)$. The structure contains discrete hydrogen-bonded units, $(Me_2C_6H_3)_3\overset{+}{Sb}—O—H\cdots I^-$, with O⋯I 3·315 (2), Sb—O 1·907 (2), mean Sb—C 2·120 (3) Å, C—Sb—C 114·3–115·6 (1), O—Sb—C 97·4–106·9 (1)°. The infrared spectrum is also in accord with a stibonium salt structure. The coordination at Sb is distorted tetrahedral due to repulsions between the bulky 2,6-dimethylphenyl ligands and with the iodide ion.

Introduction. Whereas the products of partial hydrolysis of triaryl- and trialkylarsenic dihalides, the so-called 'hydroxyhalides' [*e.g.* $Ph_3AsOHCl$], have been structurally characterized (Ferguson & Macaulay, 1968), structural information on the corresponding antimony compounds is lacking and some doubt has been expressed about their existence (Long, Doak & Freedman, 1964; Doak, Long & Freedman, 1965). In this paper we report the first structure determination of an R_3SbOHI compound, namely tris(2,6-dimethylphenyl)hydroxystibonium iodide $[(Me_2C_6H_3)_3Sb(OH)I]$.

Experimental. Tris(2,6-dimethylphenyl)hydroxystibonium iodide crystallized as orange plates from a solution of tris(2,6-dimethylphenyl)antimony diiodide dissolved in aqueous (10%) methanol, m.p. 487–489 K. (Found: C, 49·4; H, 4·8%. Calc. for $C_{24}H_{28}IOSb$: C, 49·6; H, 4·9%.) Accurate cell dimensions and crystal

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orientation matrix determined on a CAD-4 diffractometer by a least-squares treatment of the setting angles of 25 reflections in the range $10 < \theta < 15°$. Crystal dimensions 0·10 × 0·25 × 0·25 mm; intensities of reflections with indices h 0 to 13, k 0 to 14, l −24 to 24, with $2 < 2\theta < 54°$ measured; ω–2θ scans; ω-scan width (0·60 + 0·35tanθ)°, graphite-monochromatized Mo $K\alpha$ radiation; intensities of three reflections measured every 2 h showed no evidence of crystal decay. 5671 reflections measured, 4966 unique, 4167 with $I > 3\sigma(I)$ labelled observed and used in structure solution and refinement; $R_{\text{int}} = 0{\cdot}009$. Data corrected for Lorentz, polarization and absorption effects (max. and min. transmission factors 0·742, 0·403, Gaussian integration). Space group $P2_1/c$ uniquely from systematic absences $h0l$, $l = 2n + 1$; $0k0$, $k = 2n + 1$. The coordinates of the Sb and I atoms were determined from a detailed analysis of the three-dimensional Patterson function and the coordinates of the remaining non-H atoms found *via* the heavy-atom method. Refinement was by full-matrix least-squares calculations on F, initially with isotropic and then with anisotropic thermal parameters. At an intermediate stage in the refinement, difference maps showed maxima in positions consistent with the expected locations of the H atoms; one of the methyl groups had the H atoms distributed over two locations corresponding to a 60° rotation about the exocyclic C—C bond. In the final rounds of calculations the H atoms were positioned on geometrical grounds (C—H 0·95 Å) and included (as riding atoms) in the structure-factor calculations with an overall B_{iso} of 4·0 Å^2; the hydroxyl H was positioned from a difference map [O—H 0·91 (2) Å]. The final cycle of refinement included 244 variable parameters, $R = 0{\cdot}023$, $wR = 0{\cdot}037$, goodness-of-fit 1·43, $w = 1/[\sigma^2F_o + 0{\cdot}04(F_o)^2]$. Max. shift/e.s.d. = 0·009; density in final difference map ±0·46 e Å^{-3}; no chemically significant features. Scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). All calculations were performed on a PDP11/73 computer using *SDP-Plus* (B. A. Frenz & Associates Inc., 1983). Fig. 1 is a view of the salt prepared using *ORTEP*II (Johnson, 1976). Atomic coordinates and details of molecular geometry are given in Tables 1 and 2.* The infrared spectrum was recorded in the region 4000–650 cm^{-1} using a Perkin–Elmer 1330 infrared spectrophotometer; the compound was studied as mulls in hydrocarbon oil (Nujol) and fluorocarbon oil (Voltalef 3S). $\nu_{\max}$ (cm^{-1}) 2940*s,br*, 1565*m*, 1550*w*, 1445*s*, 1370*w*, 1240*w*, 1160*w*, 1150*w*, 1025*w*, 965*w*, 790*m*, 775*m*, 660*m*.

Discussion. Crystals of the title compound contain discrete hydrogen-bonded $(Me_2C_6H_3)_2\overset{+}{Sb}$—OH···I$^-$ units (Fig. 1) separated by normal van der Waals distances. The O(H)···I separation [3·315 (2) Å] is shorter than the sum of the van der Waals radii (3·55 Å) and lies at the shorter end of normal O(H)···I$^-$ distances [typical O(H)···I distances are *e.g.* 3·39 (2) and 3·47 (2) (Trotter & Fawcett, 1966), 3·44 (2) (Wetherington & Moncrieff, 1975), 3·45 (1) Å (Codding & Kerr, 1981)]. The Sb—O distance [1·907 (2) Å] is significantly shorter than the 2·048 (7) Å reported for the Sb—OH bond in penta-coordinate Ph_4Sb—OH (Beauchamp, Bennett & Cotton, 1969) and is only 0·013 Å longer than that [1·894 (5) Å] found in $(2,4,6\text{-}Me_3C_6H_2)_3SbO.HO_3SC_6H_5$ (Huber, Westhoff & Preut, 1987) where appreciable double-bond character was ascribed to the Sb—O bond. Our unequivocal location (Fig. 2) of the hydroxyl H 0·91 Å from the O atom in $(2,6\text{-}Me_2C_6H_3)_3SbOHI$ establishes the structure as a stibonium salt $R_3\overset{+}{Sb}$—O—H···I$^-$ rather than as a hydrogen-bonded adduct $R_3SbO\cdots HI$.

The coordination at Sb is distorted tetrahedral, with mean Sb—C 2·120 (3) Å. The phenyl rings adopt a fairly regular propeller conformation, the angles between the phenyl rings and the appropriate O—Sb—C planes being in the range 41 to 52°. The C—Sb—C angles are greater than tetrahedral, 114·3 to 115·6 (1)°, mean 115·0°, and the O—Sb—C angles are all less than tetrahedral, 97·4 to 106·9 (1)°; the smallest O—Sb—C angle is *trans* to the iodide ion (torsion angle C31—Sb—O—H 156°). Repulsions between the bulky 2,6-dimethylphenyl ligands and with the iodide ion are undoubtedly the cause of these distortions. Similar distortions were found in $(2,4,6\text{-}Me_3C_6H_2)_3SbO.HO_3SC_6H_5$ (Huber *et al.*, 1987) with C—Sb—C 110·0 to 119·1 (3)°, O—Sb—C 99·8 to 105·7 (2)°. By contrast, the C—Sb—C angles in tris(2,6-dimethylphenyl)stibine [98·7 (2), 109·5 (2), 106·0 (2)°] (Sobolev, Romm, Belsky, Syutkina &

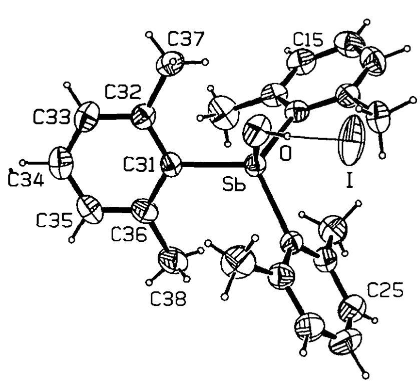

Fig. 1. A view of $(2,6\text{-}MeC_6H_3)_2Sb$—O—H···I with our numbering scheme. The ellipsoids are at the 50% probability level.

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* Lists of structure factors, anisotropic thermal parameters, calculated H coordinates and mean-plane data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44252 (55 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional and thermal parameters and e.s.d.'s*

	x	y	z	B_{eq}(Å^2)
I	0·27553 (2)	0·73903 (2)	0·49085 (1)	5·640 (5)
Sb	0·28922 (2)	0·44834 (1)	0·30790 (1)	2·462 (3)
O	0·2551 (2)	0·6076 (2)	0·3364 (1)	3·80 (4)
C11	0·4827 (3)	0·4498 (2)	0·2860 (1)	2·73 (5)
C12	0·5702 (3)	0·4946 (3)	0·3380 (2)	3·11 (5)
C13	0·6959 (3)	0·5004 (3)	0·3236 (2)	4·10 (7)
C14	0·7351 (3)	0·4613 (3)	0·2609 (2)	4·32 (7)
C15	0·6471 (3)	0·4134 (3)	0·2114 (2)	3·80 (6)
C16	0·5207 (3)	0·4055 (3)	0·2221 (2)	3·26 (5)
C17	0·5359 (3)	0·5308 (3)	0·4091 (2)	3·94 (6)
C18	0·4309 (3)	0·3503 (4)	0·1668 (2)	4·99 (7)
C21	0·2487 (2)	0·3341 (3)	0·3911 (1)	2·83 (5)
C22	0·1423 (3)	0·3603 (3)	0·4273 (2)	3·53 (6)
C23	0·1225 (3)	0·2901 (4)	0·4849 (2)	4·63 (7)
C24	0·2050 (4)	0·1983 (4)	0·5056 (2)	5·33 (8)
C25	0·3048 (3)	0·1719 (3)	0·4683 (2)	4·74 (7)
C26	0·3306 (3)	0·2380 (3)	0·4092 (2)	3·18 (5)
C27	0·0458 (3)	0·4538 (3)	0·4054 (2)	4·90 (8)
C28	0·4383 (3)	0·2009 (3)	0·3693 (2)	4·26 (7)
C31	0·1570 (2)	0·4433 (2)	0·2197 (1)	2·58 (5)
C32	0·1534 (3)	0·5425 (2)	0·1754 (1)	2·94 (5)
C33	0·0607 (3)	0·5448 (3)	0·1200 (2)	3·73 (6)
C34	−0·0248 (3)	0·4526 (3)	0·1090 (2)	4·18 (7)
C35	−0·0171 (3)	0·3540 (3)	0·1522 (2)	3·70 (6)
C36	0·0734 (3)	0·3461 (3)	0·2073 (2)	3·10 (5)
C37	0·2451 (3)	0·6456 (3)	0·1837 (2)	4·16 (6)
C38	0·0798 (3)	0·2342 (3)	0·2513 (2)	4·66 (7)

Anisotropically refined atoms are given in the form of the equivalent thermal parameter defined as: $\frac{4}{3}[a^2B_{11} + b^2B_{22} + c^2B_{33} + ab\cos\gamma B_{12} + ac\cos\beta B_{13} + bc\cos\alpha B_{23}]$.

Table 2. *Molecular dimensions*

(*a*) Bond lengths (Å)

Sb	O	1·907 (2)	C23	C24	1·385 (5)
Sb	C11	2·123 (3)	C24	C25	1·357 (5)
Sb	C21	2·122 (3)	C25	C26	1·405 (5)
Sb	C31	2·115 (2)	C26	C28	1·485 (5)
C11	C12	1·401 (4)	C31	C32	1·398 (4)
C11	C16	1·417 (4)	C31	C36	1·410 (4)
C12	C13	1·382 (4)	C32	C33	1·391 (4)
C12	C17	1·502 (4)	C32	C37	1·508 (4)
C13	C14	1·382 (5)	C33	C34	1·377 (5)
C14	C15	1·387 (4)	C34	C35	1·381 (5)
C15	C16	1·374 (4)	C35	C36	1·376 (4)
C16	C18	1·504 (4)	C36	C38	1·511 (4)
C21	C22	1·402 (4)	O···	I	3·315 (2)
C21	C26	1·408 (4)	HO···	I	2·43
C22	C23	1·391 (5)	HO	O	0·91
C22	C27	1·499 (5)			

(*b*) Bond angles (°)

O	Sb	C11	104·9 (1)	C21	C22	C27	124·2 (3)
O	Sb	C21	106·9 (1)	C23	C22	C27	118·4 (3)
O	Sb	C31	97·4 (1)	C22	C23	C24	121·1 (3)
C11	Sb	C21	114·3 (1)	C23	C24	C25	120·6 (3)
C11	Sb	C31	115·2 (1)	C24	C25	C26	121·7 (3)
C21	Sb	C31	115·6 (1)	C21	C26	C25	116·6 (3)
Sb	C11	C12	117·0 (2)	C21	C26	C28	124·6 (3)
Sb	C11	C16	121·1 (2)	C25	C26	C28	118·8 (3)
C12	C11	C16	122·0 (3)	Sb	C31	C32	117·0 (2)
C11	C12	C13	117·7 (3)	Sb	C31	C36	121·5 (2)
C11	C12	C17	123·7 (3)	C32	C31	C36	121·5 (2)
C13	C12	C17	118·5 (3)	C31	C32	C33	117·8 (3)
C12	C13	C14	121·5 (3)	C31	C32	C37	123·8 (2)
C13	C14	C15	119·5 (3)	C33	C32	C37	118·3 (3)
C14	C15	C16	122·0 (3)	C32	C33	C34	121·2 (3)
C11	C16	C15	117·2 (3)	C33	C34	C35	119·9 (3)
C11	C16	C18	123·7 (3)	C34	C35	C36	121·4 (3)
C15	C16	C18	119·1 (3)	C31	C36	C35	118·0 (3)
Sb	C21	C22	118·0 (2)	C31	C36	C38	123·0 (2)
Sb	C21	C26	119·3 (2)	C35	C36	C38	119·0 (3)
C22	C21	C26	122·6 (3)	I···	HO	O	163
C21	C22	C23	117·3 (3)				

Guryanova, 1981) and in tri-*p*-tolylstibine [97·3 (1)°] (Sobolev, Romm, Belsky & Guryanova, 1979) are smaller than the C—Sb—C angles found here, consistent with the effects of lone-pair:bond-pair repulsion being greater than those of bond-pair:bond-pair repulsion. The angles of the 2,6-dimethylphenyl moieties also show effects of intramolecular overcrowding. Thus the methyl C atoms are displaced out of the relevant phenyl-ring planes by 0·054 to 0·124 (3) Å, and are also bent away from the Sb atom [angles of the type C11—C12—C17 *etc.* have a mean value of 123·9 (2)°]. As expected, the bond lengths [mean aromatic C—C 1·390, mean Csp^2—C(methyl) 1·502 Å] show no effects of overcrowding.

The infrared spectrum of $(Me_2C_6H_3)_3Sb(OH)I$ is of interest in that it differs significantly from that of triarylarsenic hydroxyhalides in the region of OH stretching. A single strong, broad peak with maximum at 2940 cm^{-1} is present in the antimony compound whereas the arsenic compounds display strong multiple absorption at lower frequency, between 2750 and 2000 cm^{-1}. The crystal-structure analysis of the arsenic compounds (Ferguson & Macaulay, 1968) indicated the presence of strong hydrogen bonds and that the compounds are essentially adducts of triphenylarsine oxide and hydrogen halide (*e.g.* $Ph_3AsO\cdots HCl$). The infrared spectrum of $(Me_2C_6H_3)_3Sb(OH)I$ in contrast bears a close resemblance to the spectra of compounds containing the hydroxytriphenylarsonium ion, $Ph_3As(OH)^+$ [*e.g.* $Ph_3As(OH)^+.ClO_4^-$], all of which display a single strong, broad absorption band in the range 2800–3000 cm^{-1} (Harris & Inglis, 1967), in accord with the stibonium salt formulation found from our X-ray analysis.

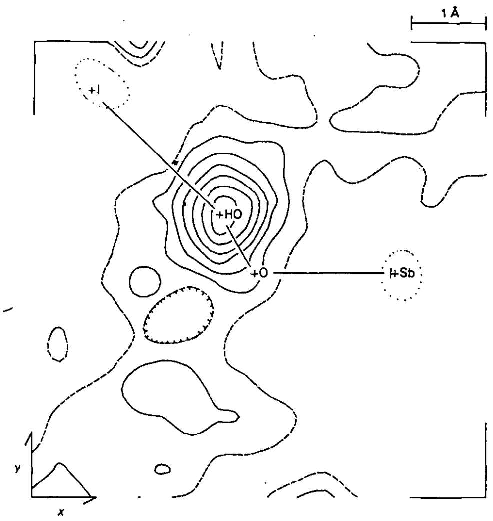

Fig. 2. A section of the difference map in the Sb,O,I plane showing the hydroxyl H atom. The contour levels are at approximately 0·1 e Å^{-3}; the zero contour is broken.

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Structure of Tetraaquatrinitratoneodymium–4,4′-Bipyridyl–Water (1/2/1)

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Abstract. $[Nd(NO_3)_3(H_2O)_4].2C_{10}H_8N_2.H_2O$, $M_r = 732{\cdot}71$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}137\ (7)$, $b = 15{\cdot}954\ (14)$, $c = 24{\cdot}688\ (27)$ Å, $U = 2811{\cdot}1$ Å^3, $Z = 4$, $D_m = 1{\cdot}70$, $D_x = 1{\cdot}73$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu = 19{\cdot}40$ cm^{-1}, $F(000) = 1468$, room temperature, final $R = 0{\cdot}044$ for 2146 $[I > 4\sigma(I)]$ observed reflections. The asymmetric unit contains one molecule of tetraaquatrinitratoneodymium(III), two molecules of 4,4-bipyridyl and one molecule of water. These latter three solvent molecules remain unconnected to the metal complex but all molecules form a three-dimensional network of hydrogen bonds. In the tetraaquatrinitratoneodymium(III) complex, the metal atom is ten coordinate being bonded to three bidentate nitrate ions and four water molecules. The geometry is of a standard type for molecules of the type *M*(bidentate)$_3$(monodentate)$_4$; that is a 4*A*,6*B*-expanded dodecahedron. Nd—O bond lengths to the nitrate ligands are consistently longer [2·558 (8)–2·716 (9) Å] than those to the water molecules [2·419 (8)–2·462 (8) Å].

Introduction. Numerous compounds of the type $Ln(NO_3)_3(4\text{-bipy})_x(H_2O)_n$ (4-bipy = 4,4′-bipyridyl) have been reported (Czakis-Sulikowska & Radwanska-Doczckalska, 1975); seven classes of adducts have so far been characterized and are listed in Table 1. At least one crystal structure in each category has been determined and all of these compounds have been found to be isomorphous with at least one or more lanthanide element.

In five of the seven categories, 4-bipy remains uncoordinated, the metal-coordination sphere being solely made up of nitrate ions and water molecules with

Table 1. *Summary of crystal structures of type* $Ln(NO_3)_y(H_2O)_x$ *with* (4-*bipy*)

Structure type	Compound	Space group	Coordination number	Cell dimensions (Å; °)	Reference
(1)	$[La_2(NO_3)_6(H_2O)_7].4(4\text{-bipy})$	$Cmc2_1$	10,11	24·52, 16·67, 13·25	(*a*)
(2)	$[Ho(NO_3)_3(H_2O)_3].2(4\text{-bipy})$	*Pbca*	9	7·73, 43·77, 15·67	(*a*)
(3)	$[Y(NO_3)_3(H_2O)_3].2(4\text{-bipy})$	$P2_1/c$	9	7·91, 20·81, 16·56	(*a*)
(4)	$[4\text{-bipyH}]^+.[Ce(NO_3)_4(H_2O)_2(4\text{-bipy})]$	$P2_12_12_1$	11	7·31, 18·59, 19·95	(*d*)
(4)	$[4\text{-bipyH}]^+.[Nd(NO_3)_4(H_2O)_2(4\text{-bipy})]$	$P2_12_12_1$	11	7·21, 18·51, 19·93	(*b*)
(5)	$2[4\text{-bipyH}]^+.[Nd_2(NO_3)_8(H_2O)_4(4\text{-bipy})]^{2}.3(4\text{-bipy})$	$P2_1/c$	10	18·72, 10·72, 18·03; 94·4	(*c*)
(6)	$[Ho(NO_3)_3(H_2O)_3].2(4\text{-bipy})$	$P2_1/c$	9	15·83, 21·44, 15·70; 100·4	(*c*)
(7)	$[Y(NO_3)_3(H_2O)_4].2(4\text{-bipy})$	$P\bar{1}$	10	15·18, 12·75, 8·16; 79·7, 78·5, 95·1	(*b*)
(8)	$[Nd(NO_3)_3(H_2O)_4].2(4\text{-bipy}).H_2O$	$P2_12_12_1$	10	7·14, 15·95, 24·69	(*e*)

References: (*a*) Weakley (1982), (*b*) Al-Rasoul & Weakley (1982), (*c*) Weakley (1984), (*d*) Bukowska-Strzyzewska & Tasik (1978), (*e*) this work.

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