0·27 (2), close to those observed for the title compound.

The numerical details concerning the three-dimensional network of hydrogen bridges interconnecting the NH_3 radicals, the water molecules and the external oxygen atoms of the P_4O_{12} ring anions are reported in Table 3.

References

AVERBUCH-POUCHOT, M. T., DURIF, A. & GUITEL, J. C. (1988). *Acta Cryst.* C**44**, 1189–1191.

AVERBUCH-POUCHOT, M. T., DURIF, A. & GUITEL, J. C. (1989). In preparation.

BAGIEU-BEUCHER, M., DURIF, A. & GUITEL, J. C. (1988). *Acta Cryst.* C**44**, 2063–2065.

Enraf–Nonius (1977). *Structure Determination Package.* Enraf–Nonius, Delft, The Netherlands.

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

JASKÓLSKI, M. (1987). *Acta Cryst.* C**43**, 1761–1763.

MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1977). *MULTAN77. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

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Structure of Hydroxotris(2,4,6-trimethylphenyl)antimony Dichloroacetate

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Abstract. $[Sb(OH)\{C_6H_2(CH_3)_3\}_3]C_2HCl_2O_2$, $M_r = 624{\cdot}26$, monoclinic, $P2_1/n$, $a = 8{\cdot}974\,(5)$, $b = 13{\cdot}307\,(6)$, $c = 23{\cdot}995\,(12)$ Å, $\beta = 95{\cdot}78\,(6)°$, $V = 2850{\cdot}8$ Å^3, $Z = 4$, $D_x = 1{\cdot}455$ Mg m^{-3}, $\lambda(\text{Ag } K\alpha) = 0{\cdot}5608$ Å, $\mu = 0{\cdot}62$ mm^{-1}, $F(000) = 1272$, $T = 291\,(1)$ K. Final $R = 0{\cdot}032$ for 3821 unique observed X-ray diffractometer data. Sb has a distorted trigonal bipyramidal environment with three equatorial C(mesityl) and two apical O atoms, one from a hydroxo group and the other from a unidentately bonded carboxylate group. One of the equatorial angles C(21)—Sb—(1)—C(31) is reduced to 114·3 (1)°, probably due to steric hindrance by mesityl groups and ligand.

Introduction. Only two X-ray diffraction studies of hydroxotriorganoantimony compounds have been reported: tris(2,6-dimethylphenyl)hydroxystibonium iodide (Ferguson, Harris & Khan, 1987) and hydroxotris(2,4,6-trimethylphenyl)antimony 1-adamantylcarboxylate (Westhoff, Huber & Preut, 1988), and the question which parameters determine the stability of such compounds in the solid state still waits to be answered. In this respect the existence of intermolecular hydrogen bonds demands special consideration and it is remarkable that in the carboxylate compound mentioned above no hydrogen bond was found. Since the bulkiness of the mesityl and adamantyl groups might prevent the formation of hydrogen bonds it seemed worthwhile to study similar compounds. We now describe the structure of a second hydroxotris(2,4,6-trimethylphenyl)antimony carboxylate, which actually contains an intermolecular hydrogen bond.

Experimental. The title compound was prepared from $SbMes_3(OH)_2$ (Mes = 2,4,6-trimethylphenyl) and dichloroacetic acid in acetone. Crystals obtained by recrystallization from methanol. Approximate dimensions 0·60 × 0·29 × 0·11 mm. $\omega/2\theta$ scan. Scan speed: 2·2–10·0° min^{-1} in θ. Nonius CAD-4 diffractometer, graphite-monochromated Ag $K\alpha$ radiation; lattice parameters from least-squares fit with 25 reflexions up to $2\theta = 29{\cdot}0°$, six standard reflexions recorded every 2·5 h, only random deviations; 11474 reflexions measured, $1 \le \theta \le 20°$, $-10 \le h \le 10$, $0 \le k \le 16$, $-29 \le l \le 29$; after averaging 5407 unique reflexions, $R_{\text{int}} = 0{\cdot}019$, 3821 with $I \ge 1{\cdot}96\sigma(I)$; Lorentz–polarization correction, absorption correction *via* ψ-scans; max./min. transmission 1·00/0·94; systematic absences ($h0l$) $h + l = 2n + 1$, ($0k0$) $k = 2n + 1$ conform to space group $P2_1/n$; structure solution *via* direct methods, ΔF syntheses and full-matrix least-squares refinement with anisotropic temperature factors for all non-H atoms and a common isotropic temperature factor for H atoms, which were placed in geometrically calculated positions (C—H 0·95 Å); H atoms of the methyl substituents of the phenyl rings were placed such that one H atom lies in the plane of the corresponding ring; refinement on F with 3821 reflexions and 317 refined parameters; $w = 4F_o^2/$

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$[\sigma^2(F_o^2) + (0{\cdot}045F_o^2)^2]$; $S = 1{\cdot}14$, $R = 0{\cdot}032$, $wR = 0{\cdot}042$, max. $\Delta/\sigma = 0{\cdot}01$, no extinction correction, largest peak in final ΔF map $= \pm 0{\cdot}4\,(2)\,\mathrm{e\,Å^{-3}}$; complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974); programs: *SDP* (Frenz, 1981), *MULTAN*80 (Main *et al.*, 1980).

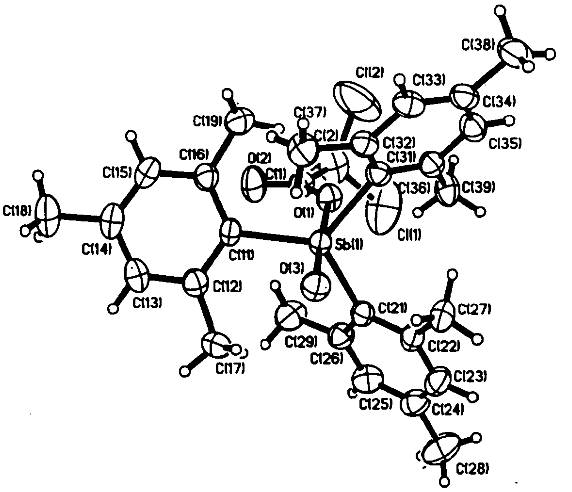

Fig. 1. General view of the molecule, showing atom numbering.

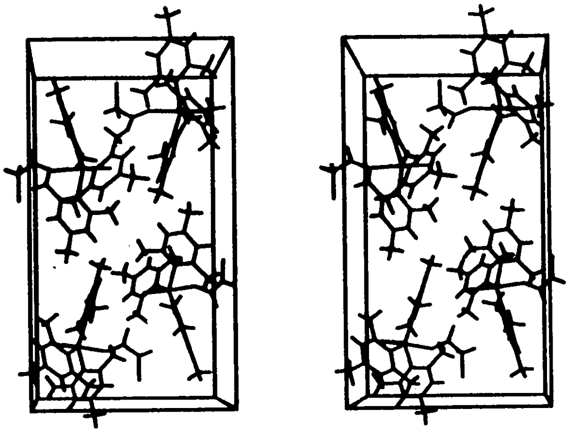

Fig. 2. Stereoscopic view of the unit cell (*b* horizontal, *c* vertical).

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

$$U_{\mathrm{eq}} = (1/6\pi^2)\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Sb(1)	0·19183 (3)	0·26912 (2)	0·66545 (1)	31
Cl(1)	0·0330 (3)	−0·0537 (1)	0·56867 (7)	117
Cl(2)	−0·1905 (2)	−0·0359 (2)	0·6456 (1)	141
O(1)	0·0849 (3)	0·1143 (2)	0·6527 (1)	41
O(2)	0·1554 (4)	−0·0023 (3)	0·7173 (1)	61
O(3)	0·2829 (3)	0·4041 (2)	0·6729 (1)	41
C(1)	0·0907 (5)	0·0273 (3)	0·6737 (2)	41
C(2)	−0·0002 (6)	−0·0527 (4)	0·6389 (2)	67
C(11)	0·2843 (4)	0·2226 (3)	0·7465 (2)	33
C(12)	0·4399 (4)	0·2301 (3)	0·7599 (2)	39
C(13)	0·4993 (5)	0·1998 (3)	0·8130 (2)	44
C(14)	0·4125 (5)	0·1677 (3)	0·8531 (2)	45
C(15)	0·2594 (5)	0·1637 (3)	0·8393 (2)	45
C(16)	0·1936 (4)	0·1902 (3)	0·7865 (2)	38
C(17)	0·5491 (5)	0·2687 (4)	0·7207 (2)	56
C(18)	0·4830 (6)	0·1373 (4)	0·9108 (2)	68
C(19)	0·0236 (5)	0·1812 (4)	0·7768 (2)	53
C(21)	0·3023 (4)	0·2366 (3)	0·5926 (2)	36
C(22)	0·2866 (4)	0·3048 (3)	0·5477 (2)	39
C(23)	0·3578 (5)	0·2839 (3)	0·5008 (2)	47
C(24)	0·4471 (5)	0·1998 (4)	0·4971 (2)	50
C(25)	0·4616 (5)	0·1348 (3)	0·5421 (2)	49
C(26)	0·3901 (5)	0·1512 (3)	0·5898 (2)	41
C(27)	0·1956 (5)	0·4004 (3)	0·5469 (2)	46
C(28)	0·5266 (7)	0·1806 (5)	0·4456 (2)	77
C(29)	0·4156 (5)	0·0736 (4)	0·6355 (2)	56
C(31)	−0·0316 (4)	0·3251 (3)	0·6480 (2)	33
C(32)	−0·0756 (4)	0·4010 (3)	0·6833 (2)	37
C(33)	−0·2090 (4)	0·4520 (3)	0·6677 (2)	41
C(34)	−0·2946 (4)	0·4322 (3)	0·6183 (2)	39
C(35)	−0·2507 (4)	0·3552 (3)	0·5852 (2)	43
C(36)	−0·1209 (4)	0·2999 (3)	0·5985 (2)	37
C(37)	0·0100 (5)	0·4322 (4)	0·7381 (2)	50
C(38)	−0·4359 (5)	0·4919 (4)	0·6014 (2)	57
C(39)	−0·0886 (5)	0·2166 (3)	0·5588 (2)	49

Table 2. *Bond distances* (Å) *and bond angles* (°) *with standard deviations*

Sb(1)–O(1)	2·280 (3)	C(1)–O(1)	1·261 (5)
Sb(1)–O(3)	1·975 (3)	C(1)–O(2)	1·211 (5)
Sb(1)–C(11)	2·128 (4)	Cl(1)–C(2)	1·740 (6)
Sb(1)–C(21)	2·139 (4)	Cl(2)–C(2)	1·746 (5)
Sb(1)–C(31)	2·140 (3)	C(1)–C(2)	1·536 (7)
O(1)–Sb(1)–O(3)	177·5 (1)	C(21)–Sb(1)–C(31)	114·3 (1)
O(1)–Sb(1)–C(11)	88·9 (1)	C(1)–O(1)–Sb(1)	140·9 (3)
O(1)–Sb(1)–C(21)	86·0 (1)	O(2)–C(1)–O(1)	129·9 (4)
O(1)–Sb(1)–C(31)	85·1 (1)	O(2)–C(1)–C(2)	115·2 (4)
O(3)–Sb(1)–C(11)	93·6 (1)	O(1)–C(1)–C(2)	114·9 (4)
O(3)–Sb(1)–C(21)	92·1 (1)	C(1)–C(2)–Cl(1)	113·4 (4)
O(3)–Sb(1)–C(31)	94·1 (1)	C(1)–C(2)–Cl(2)	109·4 (3)
C(11)–Sb(1)–C(21)	121·2 (1)	Cl(1)–C(2)–Cl(2)	110·8 (3)
C(11)–Sb(1)–C(31)	123·5 (1)		

Discussion. The structure of the title compound is shown in Fig. 1 and in a stereoview in Fig. 2. Positional parameters and equivalent values of the anisotropic temperature factors are given in Table 1, bond lengths and angles in Table 2.* As in $[\mathrm{Sb(OH)Mes_3}]$-$\mathrm{C_{11}H_{15}O_2}$ (Westhoff *et al.*, 1988) the coordination around the central Sb is a distorted trigonal bipyramid but in the title compound there is no interaction between Sb and the second O of the carboxylate group. The missing interaction is probably a result of hydrogen-bond formation (see below), which causes atom O(2) to be directed towards a neighbouring molecule. The distortion is probably caused by the bulkiness of the mesityl groups and of the dichloroacetate ligand.

H of the hydroxyl group, which is clearly represented by $\nu(\mathrm{OH})$ at 3490 cm^{-1} in the IR spectrum, was not localized. The short intermolecular O(3)···O(2) ($\frac{1}{2}-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$) distance of 2·915 (6) Å can be

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and a complete list of bond distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51372 (42 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

correlated with a hydrogen bond between these atoms. The existence of a hydrogen bond is also indicated by the position and broadness of υ(OH).

The bond distance between Sb and O(1) of the unidentately bonded carboxylate group is 2·280 (3) Å indicating an appreciably high ionic character. A similar situation was found in $(SbPh_4)CHO_2$, the analogous Sb—O distance being 2·223 Å (Bone & Sowerby, 1979). The Sb—O(H) bond of 1·975 (3) Å is significantly shorter than the Sb—O bonds in $Sb(OH)Ph_4$ (2·048 Å; Beauchamp, Bennett & Cotton, 1969) or in $Sb(OMe)Ph_4$ [2·061 (7) Å] and $Sb(OCH_3)_2Ph_3$ [mean value: 2·033 (8) Å; Kei-wei Shen, McEwen, La Placa, Hamilton & Wolf, 1968] which are considered to be essentially covalent.

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References

BEAUCHAMP, A. L., BENNETT, M. J. & COTTON, F. A. (1969). *J. Am. Chem. Soc.* **91**, 297–301.

BONE, S. P. & SOWERBY, D. B. (1979). *J. Chem. Res.* (*M*), pp. 1029–1068.

FERGUSON, G., HARRIS, G. S. & KHAN, A. (1987). *Acta Cryst.* C**43**, 2078–2081.

FRENZ, B. A. (1981). *Enraf–Nonius Structure Determination Package*, 4th ed., version 18. Enraf–Nonius, Delft, The Netherlands.

International Tables for X-ray Crystallography (1974). Vol. IV, Tables 2.2B and 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

KEI-WEI SHEN, MCEWEN, W. E., LA PLACA, S. J., HAMILTON, W. C. & WOLF, A. P. (1968). *J. Am. Chem. Soc.* **90**, 1718–1723.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

WESTHOFF, T., HUBER, F. & PREUT, H. (1988). *J. Organomet. Chem.* **348**, 185–191.

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Structures of Bis[aquadimethyl(2,6-pyridinedicarboxylato)tin(IV) hydrate] and Bis[aquadi-*n*-butyl(2,6-pyridinedicarboxylato)tin(IV)]

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Abstract. (I) $\{[Sn(CH_3)_2(C_7H_3NO_4)(H_2O)].H_2O\}_2$, $M_r = 699{\cdot}79$, monoclinic, $P2_1/c$, $a = 11{\cdot}576\,(6)$, $b = 13{\cdot}266\,(7)$, $c = 7{\cdot}747\,(5)$ Å, $\beta = 92{\cdot}67\,(5)°$, $V = 1188\,(1)$ Å^3, $Z = 2$, $D_x = 1{\cdot}956$ Mg m^{-3}, λ(Ag $K\alpha$) = 0·56087 Å, $\mu = 1{\cdot}1$ mm^{-1}, $F(000) = 688$, $T = 291\,(1)$ K, final $R = 0{\cdot}040$ for 2118 unique observed diffractometer data [$F \geq 2{\cdot}0\sigma(F)$]. (II) $[Sn(n\text{-}C_4H_9)_2(C_7H_3NO_4)(H_2O)]_2$, $M_r = 832{\cdot}08$, tetragonal, $P4_2/n$, $a = 17{\cdot}684\,(12)$, $c = 11{\cdot}148\,(12)$ Å, $V = 3486\,(5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}585$ Mg m^{-3}, λ(Ag $K\alpha$) = 0·56087 Å, $\mu = 0{\cdot}78$ mm^{-1}, $F(000) = 1680$, $T = 291\,(1)$ K, final $R = 0{\cdot}055$ for 2410 unique observed diffractometer data [$F \geq 2{\cdot}0\sigma(F)$]. In both compounds Sn has a distorted pentagonal bipyramidal environment, the alkyl groups being in the apical positions. Both carboxylate groups of the tridentate ligand are unidentate. Two molecular units are linked *via* two asymmetric Sn—O(carboxylate)—Sn bridges to form a planar Sn—O—Sn—O ring. Four O atoms (2 × bridging, 1 × water, 1 × carboxylate) and one N atom form the equatorial plane. Linking of the dimeric units by hydrogen bonds is effected by the water of hydration in the Me_2Sn complex, and by the coordinated water molecule in the Bu_2Sn complex.

Introduction. $Ph_2Sn(H_2O)L$ (H_2L = 2,6-pyridinedicarboxylic acid) has a polymeric chain structure in the crystalline state, the chains being formed by bidentate carboxylate groups and intrachain hydrogen bridges and being linked by interchain hydrogen bridges. Aside from this particular structural feature this compound earns attention since it adds one more example to the few organotin compounds with heptacoordinated Sn and since it proved to possess high *in vitro* activity against various leukemic and tumor cells (Gielen *et al.*, 1987). We now report on the structures of two

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