The sodium cation is octahedrally coordinated with outer O atoms from oxalate groups which thus bridge sodium and aluminium coordination octahedra. The Na—O bond distances range from 2·386 (3) to 2·439 (3) Å and *cis*-O—Na—O bond angles range from 70·3 (1) to 106·9 (1)°. Distortion of the octahedron around the Na atom is larger than that of the octahedron around the Al atom. However, the structure of sodium tris(oxalato)aluminate(III) pentahydrate (Bulc, Golič & Šiftar, 1982) exhibits four different coordination geometries around Na^+ cations compared with only one in this determination.

There are two independent tetraethylammonium cations which display bond lengths and angles similar to those found in previously determined structures (McCool, Marsh, Ingle & Gagne, 1981). The $N(C_2H_5)_4^+$ ions have no contacts with any other atoms shorter than 3·1 Å. They lie in the holes of the net of aluminium and sodium octahedra.

The structure is supplemented with disordered water molecules placed on two positions [O(20), O(21)] with population parameters 0·53 (3) and 0·51 (4). The distances O(21)···O(1) 2·92 (2) and O(20)···O(2) 2·77 (1) Å are consistent with O—H···O hydrogen bonding to oxalate groups. Disorder of water molecules was found in previous structure determinations of metal(III) oxalate complexes (Taylor, 1978; Bulc, Golič & Šiftar, 1984; Merrachi, Mentzen & Chassagneux, 1986).

The crystal structure determination of the title compound confirms the expected octahedral configuration around the metal(III) atom. The sodium cation does not influence the structure configuration nor the water molecule disorder. Its sole influence is on the water content in the compound.

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Structure of Copper Tris(ethylenediammonium) Bis(*cyclo*-tetraphosphate) Tetradecahydrate

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Abstract. $Cu[C_2H_{10}N_2]_3[P_4O_{12}]_2.14H_2O$, $M_r = 1133·87$, monoclinic, $P2_1/n$, $a = 13·162$ (8), $b = 13·301$ (8), $c = 12·308$ (8) Å, $\beta = 106·69$ (1)°, $V = 2064$ (4) Å^3, $Z = 2$, $D_x = 1·824$ Mg m^{-3}, $\lambda(Ag\ K\alpha) = 0·5608$ Å, $\mu = 0·517$ mm^{-1}, $F(000) = 1178$, $T = 295$ K, final $R = 0·037$ for 3480 independent reflexions. The P_4O_{12} ring anion, one of the ethyleneammonium groups and three water molecules alternate in planes $z \simeq 0·25$ and 0·75. The condensed phosphoric group has no internal symmetry. The centrosymmetrical distorted $Cu(H_2O)_6$ octahedron, one centrosymmetrical ethylenediammonium group and one water molecule alternate in planes $z \simeq 0·0$ and 0·50. One of the ethylenediammonium groups has its two inner carbon atoms statistically distributed.

Introduction. A systematic investigation of the system $H_4P_4O_{12}$–$M^{II}O$–ethylenediamine–H_2O has recently

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Table 1. *Unit-cell dimensions and cell volumes for the series of isotypic salts* $M^{II}[NH_3(CH_2)_2NH_3]_3(P_4O_{12})_2.14H_2O$

	a(Å)	b(Å)	c(Å)	β(°)	V(Å³)
Ni	12·916 (8)	13·189 (8)	12·363 (8)	106·94 (1)	2014·7 (4·4)
Cd	13·193 (8)	13·309 (8)	12·466 (8)	107·13 (1)	2091·8 (4·4)
Mn	13·098 (8)	13·278 (8)	12·424 (8)	107·15 (1)	2064·6 (4·4)
Zn	12·902 (8)	13·187 (8)	12·303 (8)	106·04 (1)	2011·7 (4·4)
Mg	13·101 (8)	13·292 (8)	12·465 (8)	108·20 (1)	2062·1 (4·4)
Co	13·146 (8)	13·296 (8)	12·258 (8)	106·77 (1)	2051·4 (4·4)
Cu	13·162 (8)	13·301 (8)	12·308 (8)	106·69 (1)	2064·0 (4·4)

been undertaken. Up to now four new types of compounds have been characterized in this system: (*a*) ethylenediammonium *cyclo*-tetraphosphate; $[NH_3(CH_2)_2NH_3]_2P_4O_{12}.H_2O$ (Averbuch-Pouchot, Durif & Guitel, 1989); (*b*) calcium ethylenediammonium *cyclo*-tetraphosphate, $Ca[NH_3(CH_2)_2NH_3]P_4O_{12}.15/2H_2O$ (Averbuch-Pouchot, Durif & Guitel, 1988); (*c*) strontium ethylenediammonium *cyclo*-tetraphosphate, $Sr[NH_3(CH_2)_2NH_3]P_4O_{12}.5H_2O$ (Bagieu-Beucher, Durif & Guitel, 1988); and (*d*) a series of isotypic compounds: $M^{II}[NH_3(CH_2)_2NH_3]_3[P_4O_{12}]_2.14H_2O$, for M^{II} = Ni, Cd, Mn, Zn, Mg, Co, Cu. The structure type corresponding to this series, determined using the copper salt, is the object of the present article.

Table 1 reports the unit-cell dimensions measured for this series of isotypic compounds.

Experimental. The title compound is readily obtained by slowly adding the stoichiometric amount of diphosphorus pentoxide to an aqueous solution of ethylenediamine added with copper hydroxycarbonate and kept at low temperature ($T < 278$ K). The reaction is

$$P_4O_{10} + 2H_2O \rightarrow H_4P_4O_{12}$$

$$2H_4P_4O_{12} + 3NH_2(CH_2)_2NH_2 + \tfrac{1}{2}CuCO_3.Cu(OH)_2 \rightarrow$$

$$Cu[NH_3(CH_2)_2NH_3]_3(P_4O_{12})_2.14H_2O + \tfrac{1}{2}CO_2.$$

After some days of evaporation at room temperature thick diamond-like plates of the title compound appear in the solution.

Crystal size: 0·35 × 0·35 × 0·38 mm. Density not measured. Philips PW 1100 diffractometer, graphite monochromator. Systematic absences: $h0l$ ($h + l = 2n$), $0k0$ ($k = 2n$). 20 reflexions ($10 < \theta < 13°$) for refining unit cell. $\omega/2\theta$ scan. 4957 reflexions collected: ($3 < \theta < 25°$), $\pm h$, k, l, $h_{max} = 18$, $k_{max} = 19$, $l_{max} = 16$. Scan width: 1·20°, scan speed 0·03° s^{-1}, total background measuring time: 10 s. Two intensity and orientation reflexions (040 and $0\bar{4}0$) measured every 3 h with no significant variation. Lorentz and polarization corrections, no absorption correction. 4741 independent reflexions ($R_{int} = 0·02$).

Table 2. *Final atomic coordinates and* B_{eq}*'s for* $Cu[NH_3(CH_2)_2NH_3]_3(P_4O_{12})_2.14H_2O$ *with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j \mathbf{a}_i.\mathbf{a}_j\beta_{ij}.$$

	x	y	z	B_{eq}(Å²)
Cu	0·000	0·000	0·000	2·09 (1)
P(1)	0·37328 (7)	0·12931 (7)	0·98129 (7)	1·67 (1)
P(2)	0·33930 (7)	0·34713 (7)	0·99553 (7)	1·64 (1)
P(3)	0·49916 (7)	0·37894 (7)	0·87022 (7)	1·62 (1)
P(4)	0·48721 (6)	0·16357 (7)	0·81223 (7)	1·70 (1)
O(*E*11)	0·4136 (2)	0·1220 (2)	0·1066 (2)	2·32 (5)
O(*E*12)	0·3013 (2)	0·0513 (2)	0·9146 (2)	2·39 (5)
O(*L*12)	0·3143 (2)	0·2353 (2)	0·9439 (2)	1·83 (4)
O(*L*41)	0·4768 (2)	0·1398 (2)	0·9375 (2)	1·91 (4)
O(*E*21)	0·3517 (2)	0·3478 (2)	0·1183 (2)	2·23 (5)
O(*E*22)	0·2420 (2)	0·9135 (2)	0·5804 (2)	2·23 (5)
O(*L*23)	0·4533 (2)	0·3692 (2)	0·9779 (2)	1·86 (4)
O(*E*31)	0·1158 (2)	0·1246 (2)	0·4178 (2)	2·31 (5)
O(*E*32)	0·0522 (2)	0·9642 (2)	0·7038 (2)	2·29 (5)
O(*L*34)	0·4531 (2)	0·2803 (2)	0·7971 (2)	1·85 (4)
O(*E*41)	0·0935 (2)	0·6069 (2)	0·7748 (2)	2·38 (5)
O(*E*42)	0·6015 (2)	0·1549 (2)	0·8201 (2)	2·41 (5)
O(*W*1)	0·4368 (2)	0·6301 (2)	0·5133 (2)	3·18 (6)
O(*W*2)	0·1979 (2)	0·5126 (2)	0·2709 (2)	2·74 (5)
O(*W*3)	0·3609 (2)	0·4297 (2)	0·5639 (2)	2·45 (5)
O(*W*4)	0·3067 (2)	0·2437 (2)	0·4562 (3)	3·36 (6)
O(*W*5)	0·4102 (2)	0·4865 (3)	0·3408 (2)	3·11 (6)
O(*W*6)	0·4500 (2)	0·2378 (3)	0·3301 (2)	3·61 (7)
O(*W*7)	0·1774 (3)	0·2439 (3)	0·1560 (3)	5·46 (8)
N(1)	0·1329 (2)	0·3900 (2)	0·6988 (3)	2·16 (6)
N(2)	0·1812 (2)	0·1155 (2)	0·6574 (2)	1·97 (5)
C(1)	0·1371 (3)	0·2054 (3)	0·7003 (3)	1·82 (6)
C(2)	0·1776 (3)	0·2994 (3)	0·6585 (3)	1·97 (6)
N(3)	0·6446 (3)	0·9669 (3)	0·5298 (3)	3·12 (7)
C(3)	0·018 (1)	0·551 (1)	−0·007 (2)	3·6 (4)
C(4)	0·4563 (4)	0·9739 (4)	0·5195 (5)	3·3 (1)

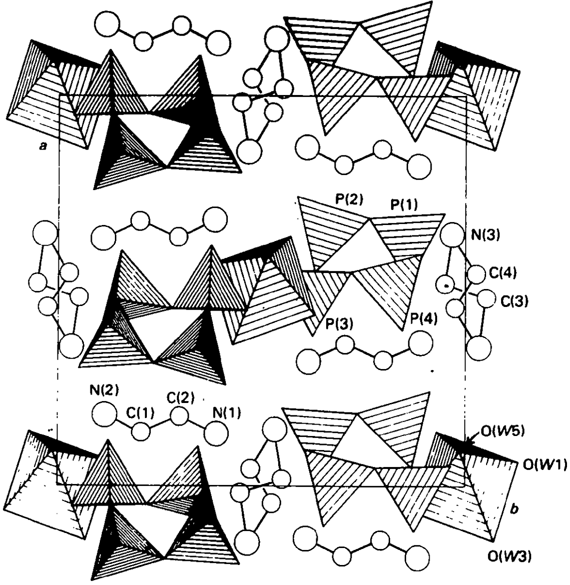

Fig. 1. Projection along the *c* axis of the atomic arrangement of the title compound. Water molecules not belonging to the $Cu(H_2O)_6$ polyhedron have been omitted. The various PO_4 tetrahedra are simply denoted by the name of the central phosphorus.

Crystal structure solved by direct methods with *MULTAN77* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1977); anisotropic full-matrix least-squares refinement (on *F*) for non-hydrogen atoms, isotropic for H atoms. Unit weights. Final refinement with 3480 reflexions ($I > 4\sigma_I$). Final $R = 0·037$ ($wR = 0·041$). No extinction correction. $S = 1·590$. Max. $\Delta/\sigma = 0·15$ [β_{33} of C(4)]. Max. peak

Table 3. *Main interatomic distances* (Å) *and bond angles* (°) *in the atomic arrangement of* $Cu[NH_3(CH_2)_2NH_3]_3(P_4O_{12})_2.14H_2O$ *with e.s.d's in parentheses*

P_4O_{12} ring anion

$P(1)O_4$ tetrahedron

P(1)	O(*E*11)	O(*E*12)	O(*L*12)	O(*L*41)
O(*E*11)	1·483 (2)	2·578 (3)	2·546 (3)	2·464 (4)
O(*E*12)	120·7 (2)	1·484 (2)	2·473 (3)	2·536 (3)
O(*L*12)	110·7 (1)	106·0 (1)	1·611 (3)	2·508 (3)
O(*L*41)	105·6 (1)	110·1 (1)	102·3 (1)	1·609 (3)

$P(2)O_4$ tetrahedron

P(2)	O(*L*12)	O(*E*21)	O(*E*22)	O(*L*23)
O(*L*12)	1·614 (2)	2·545 (3)	2·476 (3)	2·501 (3)
O(*E*21)	111·0 (1)	1·472 (3)	2·555 (3)	2·487 (4)
O(*E*22)	105·7 (1)	119·2 (2)	1·490 (2)	2·532 (3)
O(*L*23)	102·0 (1)	107·8 (1)	109·8 (1)	1·604 (3)

$P(3)O_4$ tetrahedron

P(3)	O(*L*23)	O(*E*31)	O(*E*32)	O(*L*34)
O(*L*23)	1·611 (3)	2·457 (4)	2·551 (3)	2·518 (3)
O(*E*31)	105·3 (1)	1·479 (2)	2·574 (3)	2·562 (3)
O(*E*32)	110·7 (1)	120·3 (2)	1·489 (2)	2·446 (3)
O(*L*34)	103·0 (1)	112·2 (1)	104·3 (2)	1·607 (2)

$P(4)O_4$tetrahedron

P(4)	O(*L*41)	O(*L*34)	O(*E*41)	O(*E*42)
O(*L*41)	1·617 (3)	2·503 (3)	2·546 (3)	2·486 (4)
O(*L*34)	101·6 (1)	1·613 (2)	2·483 (3)	2·522 (3)
O(*E*41)	110·5 (1)	106·8 (1)	1·479 (2)	2·576 (3)
O(*E*42)	106·5 (1)	109·0 (1)	120·7 (2)	1·483 (3)

P(1)—P(2)	2·945 (1)	P(2)—P(1)—P(4)	90·38 (4)
P(1)—P(4)	2·929 (1)	P(1)—P(2)—P(3)	88·11 (4)
P(2)—P(3)	2·975 (1)	P(2)—P(3)—P(4)	89·47 (4)
P(3)—P(4)	2·945 (1)	P(1)—P(4)—P(3)	88·96 (4)

P(1)—O(*L*12)—P(2)	131·8 (1)
P(1)—O(*L*41)—P(4)	130·4 (1)
P(2)—O(*L*23)—P(3)	135·4 (1)
P(3)—O(*L*34)—P(4)	132·3 (1)

CuO_6 octahedra

Cu—O(*W*1)	1·947 (3)	(× 2)	O(*W*1)—Cu—O(*W*3)	93·8 (1)
Cu—O(*W*3)	2·382 (3)	(× 2)	O(*W*1)—Cu—O(*W*5)	90·9 (1)
Cu—O(*W*5)	1·985 (2)	(× 2)	O(*W*3)—Cu—O(*W*5)	90·3 (1)

Ethylenediammonium groups

N(1)—C(2)	1·487 (5)	N(1)—C(2)—C(1)	110·2 (3)
C(2)—C(1)	1·507 (5)	C(2)—C(1)—N(2)	109·3 (3)
C(1)—N(2)	1·491 (5)		
N(3)—C(3)	1·62 (2)	N(3)—C(3)—C(3)	100 (1)
C(3)—C(3)	1·43 (3)		
N(3)—C(4)	1·513 (6)	N(3)—C(4)—C(4)	106·9 (4)
C(4)—C(4)	1·533 (8)		

Hydrogen bonds

O(N)—H···O	O(N)—H	H···O	O(N)—H—O	O(N)—O
O(*W*1)—H(1*W*1)···O(*W*7)	0·76 (4)	1·92 (4)	169 (5)	2·663 (4)
O(*W*1)—H(2*W*1)···O(*W*6)	0·73 (4)	1·99 (4)	170 (5)	2·714 (4)
O(*W*2)—H(1*W*2)···O(*E*42)	0·81 (6)	1·91 (6)	175 (5)	2·717 (4)
O(*W*2)—H(2*W*2)···O(*E*11)	0·87 (6)	1·96 (6)	160 (5)	2·795 (4)
O(*W*3)—H(1*W*3)···O(*E*12)	0·82 (5)	1·95 (5)	166 (5)	2·751 (4)
O(*W*3)—H(2*W*3)···O(*E*32)	0·77 (4)	2·04 (5)	164 (5)	2·795 (3)
O(*W*4)—H(1*W*4)···O(*E*31)	0·84 (5)	2·22 (6)	138 (5)	2·892 (4)
O(*W*4)—H(1*W*4)···O(*E*42)	0·84 (5)	2·40 (5)	136 (5)	3·054 (4)
O(*W*4)—H(2*W*4)···O(*W*3)	0·82 (5)	1·98 (5)	175 (5)	2·801 (4)
O(*W*5)—H(1*W*5)···O(*W*2)	0·80 (4)	1·90 (4)	179 (4)	2·700 (4)
O(*W*5)—H(2*W*5)···O(*E*32)	0·73 (7)	2·20 (7)	174 (6)	2·929 (4)
O(*W*6)—H(1*W*6)···O(*W*4)	0·94 (5)	1·89 (5)	155 (4)	2·766 (5)
O(*W*6)—H(2*W*6)···O(*E*21)	0·74 (6)	2·25 (5)	157 (6)	2·845 (4)
O(*W*7)—H(1*W*7)···O(*E*21)	0·77 (5)	2·07 (6)	170 (6)	2·828 (5)
O(*W*7)—H(2*W*7)···O(*E*42)	0·77 (5)	2·06 (5)	176 (5)	2·838 (5)
N(1)—H(1N1)···O(*E*11)	0·90 (4)	1·89 (3)	170 (4)	2·787 (4)
N(1)—H(2N1)···O(*E*12)	0·85 (4)	1·98 (4)	176 (4)	2·827 (4)
N(1)—H(3N1)···O(*E*22)	0·95 (4)	1·85 (4)	161 (4)	2·762 (4)
N(2)—H(1N2)···O(*E*41)	0·90 (4)	1·96 (4)	167 (4)	2·843 (4)
N(2)—H(2N2)···O(*E*31)	0·95 (4)	1·89 (4)	169 (4)	2·828 (4)
N(2)—H(3N2)···O(*E*32)	0·83 (4)	1·99 (4)	165 (4)	2·796 (4)
N(3)—H(1N3)···O(*W*4)	0·83 (5)	2·09 (5)	155 (4)	2·867 (5)
N(3)—H(2N3)···O(*W*2)	0·90 (4)	1·96 (4)	173 (4)	2·859 (4)
N(3)—H(3N3)···O(*E*22)	1·00 (6)	1·84 (6)	157 (5)	2·787 (5)

Table 3 (*cont.*)

H(1*W*1)—O(*W*1)—H(2*W*1)	108 (5)
H(1*W*2)—O(*W*2)—H(2*W*2)	100 (6)
H(1*W*3)—O(*W*3)—H(2*W*3)	105 (5)
H(1*W*4)—O(*W*4)—H(2*W*4)	105 (5)
H(1*W*5)—O(*W*5)—H(2*W*5)	120 (6)
H(1*W*6)—O(*W*6)—H(2*W*6)	110 (5)
H(1*W*7)—O(*W*7)—H(2*W*7)	108 (6)

height in final difference-Fourier synthesis 0·460 e Å^{-3}. Final R value = 0·052 for the complete set of 4741 independent reflexions. H atoms located by difference-Fourier map and refined isotropically. Scattering factors for neutral atoms and f', f'' from *International Tables for X-ray Crystalllography* (1974).

Enraf–Nonius (1977) *SDP* employed for all calculations. Computer used: MicroVAX 2.

Discussion. Table 2 reports the final atomic coordinates of the non-hydrogen atoms.* The atomic arrangement can be described as a succession of two types of layers, perpendicular to the b axis (Fig. 1). In the first type corresponding to $z \simeq 0{\cdot}0$ and 0·5, the $Cu(H_2O)_6$ octahedra, one ethylenediammonium group and one water molecule [O(*W*2)] alternate. In the second type with $z \simeq 0{\cdot}25$ and 0·75, the P_4O_{12} ring anion, one ethylenediammonium group and three water molecules [O(*W*4), O(*W*6), O(*W*7)] alternate.

The copper coordination comprises a centrosymmetrical distorted octahedron of six water molecules (Table 3). The P_4O_{12} ring anion comprising four independent PO_4 tetrahedra has no internal symmetry. Its main geometrical features reported in Table 3 are not significantly different from what is commonly observed for inorganic condensed phosphates. The environments of the two independent ethylenediammonium groups are very different. The first, made of N(1), N(2), C(1) and C(2), is pseudo-centrosymmetric, almost planar, and perpendicular to the planes $z \simeq 0{\cdot}25$ and 0·75 corresponding to the second type of layers described earlier. The second, located around an inversion centre, is disordered and assumes two different centrosymmetric forms. These two forms share common terminal N atoms but the inner C—C bonds are different, crossing on the inversion centre. The occupancies for the two configurations have been refined and are respectively 0·77 (1) and 0·23 (1) for the C(4)—C(4) and C(3)—C(3) bonds. A similar type of disorder has recently been observed for the two central carbon atoms of a putrescinium group in the crystal structure of putrescinium dinitrate (Jaskólski, 1987). In this case the two occupancy rates are 0·73 (2) and

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51368 (31 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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.

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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, $\beta = 95{\cdot}78\,(6)^\circ$, $V = 2850{\cdot}8$ 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 $2\theta = 29{\cdot}0^\circ$, six standard reflexions recorded every 2·5 h, only random deviations; 11474 reflexions measured, $1 \leq \theta \leq 20^\circ$, $-10 \leq h \leq 10$, $0 \leq k \leq 16$, $-29 \leq l \leq 29$; after averaging 5407 unique reflexions, $R_{\text{int}} = 0{\cdot}019$, 3821 with $I \geq 1{\cdot}96\sigma(I)$; 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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