SHORT STRUCTURAL PAPERS

The Structure of Antimony(III) Phosphite

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Abstract. $Sb_2(HPO_3)_3$, $P\bar{1}$, $a = 9.182$ (1), $b = 8.353$ (1), $c = 7.220$ (1) Å, $\alpha = 68.21$ (1), $\beta = 79.52$ (1), $\gamma = 66.47$ (1)° at 297 K, $Z = 2$, $D_o = 3.33$ (4) at 296 K, $D_c = 3.41$ Mg m^{-3}. $R = 0.020$ for 2150 reflexions. The structure consists of isolated HPO_3 tetrahedra and Sb atoms coordinated to four O atoms.

Introduction. The title compound was studied as part of an investigation of phosphites. Reaction of antimony trioxide with a concentrated aqueous solution of phosphorous acid (3.5 mmol Sb_2O_3, 30–150 mmol H_3PO_3, and water to 15 ml) gave a voluminous precipitate from which, after standing for more than one year at 298 K, brownish crystals of the title compound were obtained (Vojtíšek, 1979), with well developed faces {100} and {011} of maximum prism dimensions $0.3 \times 0.3 \times 0.5$ mm. 4428 intensities were collected ($\omega/2\theta$ scan) in the range $3 \leq 2\theta \leq 55°$ from a crystal of volume 0.0157 mm^3 on a Stoe four-circle diffractometer with graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0.71069$ Å). 2163 reflexions were unique; those with $F < 2\sigma(F)$ were treated as unobserved. The remaining 2150 reflexions were corrected for Lp and absorption effects ($\mu = 6.24$ mm^{-1}).

The cell dimensions were determined by least squares from the 2θ angles of 40 reflexions measured on the same instrument. The positions of the non-H atoms were obtained by direct methods and refined by least squares to $R = \sum ||F_o| - |F_c||/\sum |F_o| = 0.056$ with isotropic temperature factors. Further cycles with anisotropic temperature factors gave $R = 0.020$ and $R_w = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2} = 0.019$ with

weights based on counting statistics. A difference map showed no electron density maximum > 1.1 eÅ^{-3}. The H atoms were placed in optimum tetrahedral positions with P–H $= 1.38$ Å and $U = 0.02$ Å^2. The final R and R_w values were 0.020 and 0.019. A final difference map showed no residual electron density > 0.7 eÅ^{-3}. Scattering factors for neutral atoms were taken from *International Tables for X-ray Crystallography* (1974). All calculations were performed with *SHELX* 76 (Sheldrick, 1976), modified for use on a small computer (Data General Nova 3).

Table 1. *Atomic coordinates* ($\times 10^4$) *with e.s.d.'s in parentheses, and equivalent temperature factors*

$$B_{eq} = 4|V^2 \det(\beta_{ij})|^{1/3}.$$

	x	y	z	B (Å^2)
Sb(1)	7329 (1)	4922 (1)	11 (1)	0.97
Sb(2)	4188 (1)	8438 (1)	3172 (1)	0.91
P(1)	6402 (1)	1092 (1)	2373 (1)	0.91
H(1)	7820	62	1645	1.58
O(11)	5071 (3)	584 (3)	2050 (4)	1.40
O(12)	6728 (3)	566 (3)	4572 (3)	1.12
O(13)	5985 (3)	3148 (3)	1248 (4)	1.26
P(2)	616 (1)	2166 (1)	2276 (1)	1.00
H(2)	299	1534	4305	1.58
O(21)	2003 (3)	625 (3)	1788 (4)	1.71
O(22)	915 (3)	3906 (3)	1968 (4)	1.48
O(23)	−798 (3)	2612 (3)	1044 (4)	1.37
P(3)	2609 (1)	5004 (1)	4558 (1)	0.98
H(3)	1127	5003	5363	1.58
O(31)	2574 (3)	5908 (3)	2261 (4)	1.34
O(32)	3679 (3)	2979 (3)	5140 (4)	1.55
O(33)	3036 (3)	6139 (4)	5424 (4)	1.72

Discussion. Final atomic parameters are given in Table 1.* The more important interatomic distances, bond angles and the symmetry code are given in Table 2. A projection of the structure in the XY plane is depicted in Fig. 1. Sb(1), Sb(2), Sb(1^i) and Sb(2^i) form a slightly distorted parallelogram. The Sb—O distances show that eight out of nine phosphite O atoms are relatively strongly coordinated to two Sb atoms. O(33), with the shortest P—O distance, is uncoordinated. The coordination polyhedron of Sb is trigonal bipyramidal with one of the equatorial corners occupied by the lone pair of electrons. The means of the shorter equatorial
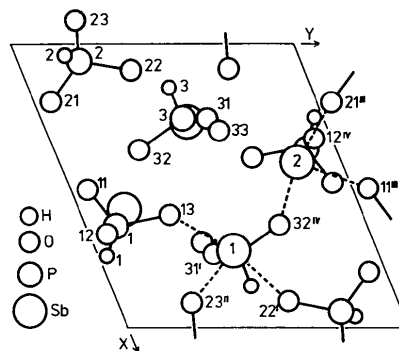
* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35847 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Fig. 1. Projection of the structure in the XY plane.

distances and the angles are 2·02 Å and 86·0°; the means of the longer axial distances and the angles are 2·18 Å and 159·4°. The other O atoms are at distances > 2·47 Å. The distances and angles in the bipyramids agree with literature data (Bovin, 1976).

The average P—O distance, 1·52 (2) Å, and the average O—P—O angle, 111 (3)°, also agree with literature data, e.g. for $Na_2HPO_3.5H_2O$ (Brodalla, Goeters, Kniep, Mootz & Wunderlich, 1978), $CuHPO_3.2H_2O$ (Handlovič, 1969) or $MgHPO_3.6H_2O$ (Corbridge, 1956). The longest intramolecular O—O distance is 2·569 Å [O(11)—O(12)], the shortest intermolecular O—O distance is 2·631 Å [O(13)—O(31^i)].

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Table 2. *Interatomic distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Sb(1)—Sb(1^i)	4·230 (1)	Sb(2)—Sb(1^i)	4·916 (1)
—Sb(2)	4·240 (1)	—Sb(2^v)	5·065 (1)
Sb(1)—O(31^i)	1·980 (2)	O(31^i)—Sb(1)—O(23^{ii})	87·0 (1)
—O(23^{ii})	1·996 (2)	O(13)—Sb(1)—O(31^i)	79·2 (1)
—O(13)	2·143 (3)	—O(23^{ii})	84·7 (1)
—O(22^i)	2·233 (3)	—O(22^i)	158·8 (1)
Sb(2)—O(12^{iv})	2·000 (2)	O(12^{iv})—Sb(2)—O(11^{iii})	85·1 (1)
—O(11^{iii})	2·091 (3)	O(32^{iv})—Sb(2)—O(12^{iv})	83·9 (1)
—O(32^{iv})	2·161 (2)	—O(11^{iii})	82·9 (1)
—O(21^{iii})	2·192 (2)	—O(21^{iii})	160·6 (1)
P(1)—O(11)	1·522 (3)	O(11)—P(1)—O(12)	114·4 (1)
—O(12)	1·534 (3)	—O(13)	106·8 (1)
—O(13)	1·520 (2)	O(12)—P(1)—O(13)	112·6 (2)
P(2)—O(21)	1·506 (3)	O(21)—P(2)—O(22)	116·0 (2)
—O(22)	1·514 (3)	—O(23)	107·7 (2)
—O(23)	1·550 (3)	O(22)—P(2)—O(23)	110·8 (1)
P(3)—O(31)	1·546 (2)	O(31)—P(3)—O(32)	110·7 (2)
—O(32)	1·516 (4)	—O(33)	109·0 (2)
—O(33)	1·506 (4)	O(32)—P(3)—O(33)	114·7 (2)

Symmetry code

(i)	$1 - x, 1 - y, \bar{z}$		(ii)	$1 + x, y, z$
(iii)	$x, 1 + y, 1 - z$		(iv)	$1 - x, 1 - y, 1 - z$
(v)	$1 - x, 2 - y, 1 - z$			

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