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The Crystal Structure of the Antimony Phosphate, $SbO(H_2PO_4).H_2O$

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The crystal structure of the antimony phosphate $SbO(H_2PO_4).H_2O$ has been determined from X-ray single crystal film data and refined to a final R-value of 0.056 (1380 independent reflections). The crystals are monoclinic (space group $P2_1/c$). The unit cell contains four formula units and has the dimensions $a = 9.120$ Å, $b = 10.730$ Å, $c = 5.790$ Å, and $\beta = 95.5°$.

The antimony atom has the usual one-sided coordination to four oxygen atoms and the SbO_4 polyhedron can be described as a distorted trigonal bipyramid with a lone pair of electrons in one of the equatorial corners.

The fundamental structural element of the crystal structure is an infinite layer of the composition $SbO(H_2PO_4)$ parallel to the bc plane. Each layer is built up of SbO_4 polyhedra and PO_4 tetrahedra that share corners only. The water molecules are situated between the layers.

Several studies on the crystal chemistry of antimony(III) compounds, *e.g.* Sb_2O_3 (*orth.*),[1] SbOF,[2,3] $Sb_4O_5Cl_2$,[4,5] $SbPO_4$,[6] $Sb_4O_4(OH)_2(NO_3)_2$[7] have been completed or are in progress in this laboratory. In connection with these it was of interest to investigate the coordination of the metal atom in the salt described as $SbPO_4.2H_2O$ in *Gmelins Handbuch*.[8]

The results of this study show that the formula of the salt should be written as $SbO(H_2PO_4).H_2O$ instead of $SbPO_4.2H_2O$.

EXPERIMENTAL

Crystal preparation. An excess of $(NH_4)_2HPO_4$ solution was added to an acid water solution of $SbCl_3$ and the mixture was filtered. Tabular (100) colourless crystals separated in the filtrate within a day.

Crystal data and space group. Preliminary Weissenberg exposures of the layers $hk0$ and $hk1$ showed the crystals to be monoclinic. The only systematic absences found were: $h0l$ with $l = 2n+1$ and $0k0$ with $k = 2n+1$ which are characteristic of the space group $P2_1/c$ (No. 14). The dimensions of the unit cell were determined from powder photographs taken in a Guinier-Hägg focusing camera with Cu$K\alpha_1$ radiation ($\lambda = 1.54051$ Å) and potassium chloride (cubic, $a = 6.2929$ Å) as an internal standard. Refinement of the cell parameters was performed with a least-squares program.

The density observed by measuring the loss of weight in benzene was in fair agreement with the calculated value for four formula units $SbO(H_2PO_4).H_2O$ per unit cell. Some crystal data are presented in Table 1.

Table 1. Crystal data for $SbO(H_2PO_4).H_2O$. The estimated standard deviations are given within brackets.

Space group $P2_1/c$ (No. 14)

Unit cell dimensions:

$a = 9.1199(10)$ Å	$D_m = 2.86$ g cm^{-3}
$b = 10.7301(8)$ Å	$Z = 4$
$c = 5.7899(5)$ Å	Formula weight =
$\beta = 95.49(1)°$	252.75 g mol^{-1}
$V = 564.0$ Å^3	$D_x = 2.82$ g cm^{-3}

Collection and reduction of intensity data. A crystal with the dimensions $0.01 \times 0.07 \times 0.12$ mm^3 was chosen for the single crystal work. The rotation axis [001] chosen coincides with the dimension 0.12 mm of the crystal. Intensity data were collected for the reflections $hk0$ to $hk7$ (1380 independent reflections) with an integrating Weissenberg camera using Zr-filtered Mo$K\alpha$ radiation. The multiple film technique (3 films) with thin (0.05 mm) steelfoils between the films was used. The relative intensities of the reflections were measured by means of a microdensitometer (Enraf-Nonius). Corrections for Lorentz and polarization effects were performed and an absorption correction was applied. The linear absorption coefficient was 44.2 cm^{-1}.

Table 2. Analysis of the weights used in the last cycle of the least-squares refinement. The averages $\overline{w\Delta^2}$ are normalized. w = weighting factor. $\Delta = |F_o| - |F_c|$.

F_o-interval	Number of independent reflections	$\overline{w\Delta^2}$	sin θ-interval	Number of independent reflections	$\overline{w\Delta^2}$
0.0 – 28.2	138	1.06	0.000 – 0.302	255	0.99
28.2 – 32.7	138	1.49	0.302 – 0.380	245	0.64
32.7 – 36.6	138	0.93	0.380 – 0.435	214	0.80
36.6 – 40.3	138	0.99	0.435 – 0.479	188	0.71
40.3 – 44.8	138	0.76	0.479 – 0.516	141	0.85
44.8 – 49.6	138	0.98	0.516 – 0.548	115	1.35
49.6 – 55.7	138	0.81	0.548 – 0.577	83	1.12
55.7 – 66.0	138	0.91	0.577 – 0.603	50	1.49
66.0 – 80.7	138	0.92	0.603 – 0.628	30	0.85
80.7 – 168.1	138	1.16	0.628 – 0.650	36	1.21

Table 3a. Positional and thermal parameters obtained in the least-squares refinement. Estimated standard deviations are given within brackets. The anisotropic thermal parameters for the antimony atom are based on the expression exp $[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2\ hk\beta_{12} + 2\ hl\beta_{13} + 2\ kl\beta_{23})]$.

Atom	x	y	z	B (Å^2)
Sb	0.46144(7)	0.16360(6)	0.24294(11)	[0.72]
P	0.74434(30)	0.40469(26)	0.28149(47)	0.81(3)
O(1)	0.5917(9)	0.3464(9)	0.2586(15)	1.69(12)
O(2)	0.8376(10)	0.3608(9)	0.0921(16)	1.77(14)
O(3)	0.8268(11)	0.3722(10)	0.5257(18)	2.08(15)
O(4)	0.7316(9)	0.5484(8)	0.2823(15)	1.39(12)
O(5)	0.3547(8)	0.2507(7)	0.4753(13)	0.91(9)
O(6)	0.1384(13)	0.4079(11)	0.1916(20)	2.53(18)

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Sb	0.00247(5)	0.00152(3)	0.00569(14)	0.00067(7)	0.00047(5)	0.00009(9)

Table 3b. The r.m.s. components (Å) of the thermal displacement along the principal axes of the ellipsoid of thermal vibration for Sb.

$R(1) = 0.114(2)$	$R(2) = 0.079(2)$	$R(3) = 0.098(1)$

Computer programs. The following crystallographic computer programs were used in this work:

1. PIRUM: Indexing of powder photographs and refinement of unit cell parameters, written by P. E. Werner, Stockholm, Sweden.
2. DRF: Data reduction and Fourier calculations, written by A. Zalkin, Berkeley, USA. Modified by R. Liminga and J.-O. Lundgren, Uppsala, Sweden.
3. DATAP2: Absorption correction, written by P. Coppens, L. Leiserowitz and D. Rabinowich, Rehovoth, Israel. Modified by O. Olofsson and M. Elfström, Uppsala, Sweden.
4. LALS: Full matrix least-squares refinement of atomic parameters, written by P. K. Gantzel, R. A. Sparks and K. N. Trueblood, Los Angeles, USA. Modified by A. Zalkin, J.-O. Lundgren, R. Liminga and C.-I. Brändén, Uppsala, Sweden.
5. DISTAN: Calculation of interatomic distances and bond angles, written by A. Zalkin.
6. ORFFE: Crystallographic function and error program, written by W. R. Busing, K. O. Martin and H. A. Levy, Oak Ridge, USA.

All programs are modified and adopted for the UNIVAC 1108 computer in Lund by G. Malmros, C. Svensson and C. Särnstrand, Lund, Sweden.

Table 4. Observed and calculated structure factors for $SbO(H_2PO_4).H_2O$. The columns are h, $|F_o|$, and $|F_c|$, respectively. Each group is headed by k and l common to the group.

K=-10 L= 0

0	53	51
1	46	40

K=-9 L= 0

0	36	26

K=-8 L= 0

3	57	54
4	45	45
7	64	60
8	65	63

K=-7 L= 0

0	81	91
1	77	84
2	58	64
3	44	47
4	37	42

K=-6 L= 0

1	34	33
2	34	41
3	42	44
4	56	58
5	64	67
6	65	67
7	63	63
8	58	60
9	54	54

K=-5 L= 0

0	63	67
1	71	73
2	66	67
3	58	57
8	40	34

K=-4 L= 0

2	36	36
5	42	40

K=-3 L= 0

0	98	100
0	26	18
1	44	42
2	43	43
4	47	44
5	30	29

K=-2 L= 0

1	52	48
2	30	32
4	73	71
5	116	114
6	86	87
7	51	54
8	48	48
9	57	60
10	46	49

K=-1 L= 0

0	98	97
1	129	137
2	159	158
3	111	109
4	45	49
6	37	24
8	58	58
9	42	43
11	45	45
12	63	60
13	59	58

K=0 L= 0

1	39	36
2	55	54
3	72	71
4	81	80
5	121	124
6	100	102
7	86	86
8	84	89
9	56	56
10	36	33

K=1 L= 0

0	168	168
1	61	59
2	49	45
3	122	119
4	51	45
7	40	39
10	45	42

K=2 L= 0

1	111	111
2	112	107
3	41	41
6	31	33
8	31	26
9	33	30

K=-1 L= 0

2	107	99
3	105	111
4	65	65
5	92	88
6	102	104
7	101	102
8	103	103
9	67	67
10	38	39
11	39	38

K= 0 L= 0

2	56	57
3	144	155
4	76	76
5	35	35
7	42	41
10	88	90
11	74	75
12	43	39
13	48	45
14	53	55
15	46	44

K= 1 L= 0

1	73	77
2	66	67
3	38	37
4	40	34

K= 2 L= 0

0	26	18
1	44	42
2	43	43
4	47	44
5	30	29

K= 3 L= 0

1	52	48
2	30	32
4	73	71
5	116	114
6	86	87
7	51	54
8	48	48
9	57	60
10	46	49

K= 4 L= 0

0	98	97
1	129	137
2	159	158
3	111	109
4	45	49
6	37	24
8	58	58
9	42	43
11	45	45
12	63	60
13	59	58

K= 5 L= 0

1	39	36
2	55	54
3	72	71
4	81	80
5	121	124
6	100	102
7	86	86
8	84	89
9	56	56
10	36	33

K= 6 L= 0

0	168	168
1	61	59
2	49	45
3	122	119
4	51	45
7	40	39
10	45	42

K= 7 L= 0

1	57	53
2	27	20
3	30	28

K=-10 L= 1

10	46	46

K=-9 L= 1

11	35	34
12	35	33
13	28	41

K=-8 L= 1

-9	53	49
1	17	16
2	50	51
3	60	61
4	53	53
5	59	62
6	68	63
7	52	49
8	30	33
9	24	25
10	29	25

K=-7 L= 1

-8	36	33
2	50	51
3	60	61
4	53	53
5	59	62
6	68	63
7	52	49
8	30	33
9	24	25
10	29	25

K=-6 L= 1

-7	22	23
1	72	69
2	50	47
5	19	16
6	19	17
8	31	27
10	24	19
11	24	24

K=-5 L= 1

-6	50	50
2	20	21
3	19	22
4	49	53
6	30	33
7	32	34

K=-4 L= 1

-5	81	85
4	116	121
-3	43	42
3	111	112
4	104	101
5	83	83
6	121	120
7	101	97
8	57	58
9	48	47
10	35	39
11	31	30

K=-3 L= 1

-4	74	77
3	96	94
-2	161	171
-1	129	137
1	155	166
2	139	145
3	77	75
4	81	78
5	52	46
6	26	28
8	59	56
9	55	53
10	40	37
11	59	59
12	68	69
13	55	47

K=-2 L= 1

-3	33	30
-8	30	25
-4	39	37
-3	78	78
-2	118	106
-1	43	45
1	103	90
2	108	95
3	57	54
4	24	22
8	36	34

K=-1 L= 1

-2	32	29
-8	47	48
-14	41	38
-13	56	55
-12	58	60
-11	43	44
-10	31	32
-9	47	49
-8	36	37
-7	28	24
-6	47	43
-5	42	42
-4	72	70
-3	107	105
-2	112	121
-1	91	101
0	73	91
1	100	109
2	112	109
3	74	72
4	28	29
5	42	38
6	27	30
7	36	34
8	53	50
9	38	35
10	46	46
11	52	55
12	55	55

K= 0 L= 1

-14	36	36
-13	46	55
-12	66	66
-11	59	56
-10	35	37
-9	53	52
-8	50	49
-7	43	40
-6	74	77
-5	56	55
-4	74	77
-3	96	94
-2	161	171
-1	129	137
1	155	166
2	139	145
3	77	75
4	81	78
5	52	46
6	26	28
8	59	56
9	55	53
10	40	37
11	59	59
12	68	69
13	55	47

K= 1 L= 1

-14	26	19
-13	32	26
-12	27	26
-11	23	18
-10	21	18
-9	26	23
-8	22	23
-7	21	15
-6	21	15
-4	20	17
-3	54	53
-2	74	74
-1	54	55
0	54	53
1	72	69
2	50	47
5	19	16
6	19	17
8	31	27
10	24	19
11	24	24

K= 2 L= 1

-13	51	52
-12	55	47
-10	36	34
-9	48	38
-6	36	37
-5	46	45
-4	45	42
-3	91	92
-2	99	105
-1	46	49
0	73	75
1	96	101
2	57	56
3	42	42
4	33	31
8	45	48
9	36	30
11	60	53
12	49	45

K= 3 L= 1

-11	39	36
-10	53	49
-9	52	57
-8	69	70
-7	75	73
-6	69	73
-5	84	84
-4	69	70
-3	46	44
-2	38	37
0	33	33
1	41	43
2	63	63
3	82	87
5	74	76
6	72	75
7	58	57
8	47	49
9	41	41

K= 4 L= 1

-3	89	91
-2	76	74
-1	106	104
0	105	112
1	74	74
2	79	80
3	76	75
4	48	47
5	28	24
9	50	45
10	53	52
11	43	41
12	37	38
13	40	41
14	35	36

K= 5 L= 1

-12	32	24
-8	43	46
-7	58	56
-6	22	18
-4	62	61
-3	42	37
2	35	32
3	71	72
4	24	20
6	57	53
7	50	47

K= 6 L= 1

-15	32	29
-14	32	32
-11	34	34
-10	27	22
-8	24	19
-7	21	14
-5	30	26
-4	70	67
-3	50	49
-2	37	31
-1	89	86
0	76	74
1	36	35
2	57	58
3	56	50
4	27	22
6	29	28
9	28	28
10	31	32

K= 7 L= 1

-11	33	28
-10	41	43
-9	48	51
-8	47	48
-7	57	57
-6	60	61
-5	48	49
-4	44	46
-3	35	34
0	18	20
1	45	45
2	46	47
3	53	55
4	64	66
5	54	55
6	48	49
7	47	48
8	34	35

K= 8 L= 1

-1	83	83
0	76	76
1	57	59
2	53	54
3	53	49
4	40	35
9	44	42

K= 9 L= 1

-2	49	46
-1	63	68
0	56	57
1	26	28
3	25	24
9	37	32

K= 4 L= 7

-10	31	28
-6	25	22
-5	40	40
-4	46	49
-3	40	41
-2	51	51
-1	63	64
0	49	50
1	37	39
2	38	39
3	29	32
4	22	19

K= 5 L= 1

-8	42	40
-7	64	63
-6	40	41
-4	60	58
-3	72	73
2	32	24
3	75	74
4	43	41
6	47	47
7	63	59
8	36	37

K= 5 L= 2

-10	53	55
-9	64	67
-8	83	90
-7	99	101
-6	69	70
-5	76	75
-4	96	96
-3	60	58
-2	43	40
-1	37	32
1	42	41
2	50	48
3	97	90
4	86	83
5	70	66
6	94	91
7	93	94
8	76	75
9	57	61
10	36	34

K= 5 L= 3

-12	32	24
-8	43	46
-7	58	56
-6	22	18
-4	62	61
-3	42	37
2	35	32
3	71	72
4	24	20
6	57	53
7	50	47

K= 5 L= 4

-12	31	24
-11	29	27
-10	41	38
-9	60	63
-8	68	69
-7	74	75
-6	89	90
-5	74	73
-4	59	56
-3	47	46
1	28	25
2	53	50
3	67	61
4	84	84
5	82	82
6	69	70
7	64	64
8	52	52
9	33	34
10	31	27

K= 5 L= 5

-8	37	39
-7	42	39
-5	27	16
-4	49	46
-3	26	27
2	42	41
3	37	39

K= 3 L= 2

-7	36	35
-5	40	39
-3	21	19
-1	34	30
0	31	24
3	50	46
5	42	38
7	30	25

K= 3 L= 3

-10	49	50
-9	67	70
-8	78	81
-7	79	84
-6	99	103
-5	101	107
-4	89	91
-3	91	93
-2	56	56
1	56	56
2	98	99
3	89	92
4	110	109
5	106	111
6	81	82
7	77	79
8	71	72
9	44	46
10	36	32

K= 3 L= 4

-13	40	42
-12	37	37
-11	52	49
-10	50	49
-9	26	20
-6	29	26
-5	50	48
-4	77	76
-3	89	91
-2	76	74
-1	106	104
0	105	112
1	74	74
2	79	80
3	76	75
4	48	47
5	28	24
9	50	45
10	53	52
11	43	41
12	37	38
13	40	41
14	35	36

K= 4 L= 4

-15	34	35
-14	40	42
-13	40	40
-12	37	37
-11	52	49
-10	50	49
-9	26	20
-6	29	26
-5	50	48
-4	77	76
-3	89	91
-2	76	74
-1	106	104
0	105	112
1	74	74
2	79	80
3	76	75
4	48	47
5	28	24
9	50	45
10	53	52
11	43	41
12	37	38
13	40	41
14	35	36

K= 4 L= 5

-10	35	37
-9	64	67
-8	83	90
-7	99	101
-6	69	70
-5	76	75
-4	96	96
-3	60	58
-2	43	40
-1	37	32
1	42	41
2	50	48
3	97	90
4	86	83
5	70	66
6	94	91
7	93	94
8	76	75
9	57	61
10	36	34

K= 6 L= 4

-15	32	29
-14	32	32
-11	34	34
-10	27	22
-8	24	19
-7	21	14
-5	30	26
-4	70	67
-3	50	49
-2	37	31
-1	89	86
0	76	74
1	36	35
2	57	58
3	56	50
4	27	22
6	29	28
9	28	28
10	31	32

K= 6 L= 5

-10	39	35
-6	28	23
-5	50	49
-4	64	63
-3	61	61
-2	66	66
-1	83	83
0	76	76
1	57	59
2	53	54
3	53	49
4	40	35
9	44	42

K= 6 L= 6

-14	26	19
-11	40	32
-10	30	23
-5	28	26
-3	26	27
2	42	41
3	37	39

Table 4. Continued.

6	38	38	-15	33	25	-4	52	53	2	27	24	3	59	60	5	38	36	-4	45	46	1	47	51
7	43	37	-14	33	35	-3	29	25	3	28	27	4	54	56				-3	42	42	2	47	44
			-13	44	45	2	42	45	4	26	25	5	58	58	K= 11 L=		2	-2	44	45			
K=	5 L=	6	-12	41	38	3	60	60				6	54	60	-9	43	48	-1	57	56	K= 14 L=		7
-11	33	31	-11	30	26	4	32	30	K=	8 L=	5	7	46	52	-8	49	53	0	48	50	-3	43	46
-10	42	43	-10	32	28	5	40	33	-11	48	49	8	47	37	-7	57	61	1	39	41	-2	41	39
-9	55	56	-9	28	27	6	52	49	-10	43	35				-6	72	76	2	42	41	-1	32	28
-8	61	62	-7	25	22	7	28	33	-5	60	57	K=	9 L=	6	-5	72	74	3	39	33	0	39	37
-7	50	53	-5	19	19				-4	59	59	-6	33	26	-4	53	50	4	24	17	1	40	41
-6	51	50	-4	43	41	K=	7 L=	6	-3	39	42	4	26	24	-2	32	25						
-5	53	53	-3	82	83	-11	29	30	-2	54	56	5	23	18	2	33	32	K=	13 L=	1	K=	15 L=	1
-4	43	43	-2	80	85	-10	45	48	-1	83	88				3	45	44	-7	38	38	-8	42	39
-3	35	31	-1	63	64	-9	47	46	0	77	77	K=	9 L=	7	4	63	64	-6	44	43	-7	33	38
-2	20	17	0	72	76	-8	32	33	1	44	44	-11	37	28	5	78	78	-5	46	42	-6	47	53
0	31	28	1	78	80	-7	40	42	2	38	39	-10	33	30	6	67	68	-4	44	43	-5	62	60
1	35	34	2	51	52	-6	59	60	3	53	53	-9	43	39	7	53	53	-3	43	41	-4	34	39
2	46	47	3	26	21	-5	40	39	4	45	42	-8	47	49	8	48	49	3	38	37	4	42	45
3	50	50	7	30	31	-3	26	27	9	47	45	-7	47	47	9	39	42	4	52	47	5	57	62
4	50	50	8	40	38	-2	29	31	10	41	47	-6	44	40				5	48	44	6	48	48
5	51	54	9	28	29	0	33	32				-5	39	42	K=	11 L=	3	6	41	38	9	43	41
6	55	57	10	39	35	1	40	40	K=	8 L=	6	-4	37	38	-9	30	25	7	40	39			
7	52	55	11	43	42	2	24	21	-1	35	34	-3	28	24	-6	27	24				K=	15 L=	3
8	42	45	12	39	38	3	27	28				1	28	27	-5	30	28	K=	13 L=	2	-10	38	36
9	28	28				4	55	56	K=	8 L=	7	2	47	45	4	27	25	-8	45	45	-9	40	40
			K=	7 L=	1	5	52	52	-6	29	22	3	45	47	5	29	25	-7	47	49	-8	35	36
K=	5 L=	7	-8	43	45	6	33	34	-5	45	46	4	41	42				-6	37	39	-7	33	39
-8	38	38	-7	64	62	7	36	36	-4	46	46	5	50	47	K=	11 L=	4	-4	41	42	-6	51	51
-5	24	23	-6	55	53	8	40	45	-3	27	31	6	43	47	-10	42	43	-3	42	43	-5	48	51
-4	27	29	-5	43	41	9	30	34	-2	48	49	7	38	37	-9	50	51	2	35	29	-4	33	31
2	36	36	-4	71	75				-1	69	71	8	29	27	-8	50	51	3	52	49	1	35	30
3	29	27	-3	73	77	K=	7 L=	7	0	44	48				-7	51	55	6	40	44	2	29	23
6	36	35	-2	31	27	-8	37	32	1	32	29	K=	10 L=	1	-6	54	57	7	45	50	3	31	30
			2	45	39	-7	40	32	2	37	35	-5	29	26	-5	49	51				4	50	49
K=	6 L=	1	3	82	84	-6	35	25	3	39	37	-4	37	40	-4	42	41	K=	13 L=	3	5	48	53
-2	46	41	4	71	70	-5	27	27	9	33	39	-3	55	55	-3	31	32	-8	38	35	6	39	42
0	30	29	5	37	37	-4	36	37	10	39	36	-2	68	70	-2	26	26	-7	36	35	7	33	34
1	26	23	6	59	57	-3	20	20				-1	68	70	0	26	19	-6	39	37	8	33	36
2	37	36	7	64	62	1	29	24	K=	9 L=	1	0	64	62	1	39	37	-5	45	42	9	32	37
			8	39	36	2	36	36	-11	38	33	1	66	68	2	35	35	-4	34	36			
K=	6 L=	2				3	33	33	-10	42	39	2	73	69	3	46	42	-3	33	30	K=	15 L=	5
-13	45	54	K=	7 L=	2	4	32	30	-9	46	49	3	50	51	4	55	54	2	38	33	-6	43	48
-12	57	61	-10	51	54	5	35	28	-8	62	63	4	31	30	5	54	56	3	40	40	-5	38	36
-9	47	52	-9	66	71	6	34	31	-7	80	82				6	50	53	4	39	40	4	53	47
-8	27	28	-8	52	57				-6	79	84	K=	10 L=	2	7	52	51	5	38	39	5	47	45
-5	32	28	-7	45	46	K=	8 L=	1	-5	64	69	-3	47	48	8	40	46	6	39	37			
-4	36	34	-6	82	83	-12	43	41	-4	77	80	-2	76	79	9	39	37	7	34	32	K=	16 L=	1
-3	84	87	-5	92	89	-11	60	60	-3	82	79	-1	50	50				-1	37	36			
-2	167	162	-4	30	29	-10	63	62	-2	45	41	0	38	39	K=	11 L=	6	0	48	40			
-1	133	129	-3	34	32	-9	48	51	1	34	29	1	78	73	-10	32	32	-8	43	48	1	39	37
0	113	110	-2	69	66	-8	91	89	2	58	57	2	60	61	-9	39	39	-7	44	43			
1	167	159	-1	33	34	-7	71	72	3	77	78	3	33	33	-8	42	42	-5	26	24	K=	16 L=	2
2	120	117	1	73	64	-6	48	51	4	82	81	8	38	38	-7	53	49	-4	48	44	-1	43	49
3	56	52	2	58	56	-1	101	106	5	72	75				-6	55	54	-3	27	29	0	57	59
4	27	27	4	67	63	0	127	136	6	81	80	K=	10 L=	3	-5	39	43	2	36	36			
8	59	56	5	102	96	1	77	79	7	76	79	-12	34	33	-4	27	25	3	51	46	K=	16 L=	3
9	46	44	6	60	60	2	60	59	8	60	60	-5	32	30	1	28	22	6	44	44	-4	29	24
11	49	50	7	44	47	3	78	75	9	45	41	-4	37	35	2	35	30	7	40	44	-3	31	26
12	55	58	8	68	64	4	81	76	10	37	37	-3	56	54	3	40	40	8	30	29	-2	39	33
			9	63	64	5	42	41	11	39	31	-2	72	71	4	47	52				-1	39	36
K=	6 L=	3				9	43	41				-1	53	54	5	50	52	K=	13 L=	6	0	32	34
-3	32	26	K=	7 L=	3	10	69	66	K=	9 L=	2	0	56	55	6	40	41	-9	23	26	1	34	34
-2	20	19	-9	29	30	11	56	52	-5	36	37	1	69	68	7	36	34	-8	32	37	2	32	28
2	35	31	-8	51	48	12	45	40	-1	28	22	2	54	51	8	34	32	-7	37	34			
4	22	20	-7	53	52	13	50	39	5	34	33	3	39	37	9	22	24	-6	29	22	K=	16 L=	4
			-6	45	44	14	47	42				4	33	30				-5	24	25	-4	34	29
K=	6 L=	4	-5	47	43				K=	9 L=	3				K=	12 L=	1	-4	35	32	-2	26	27
-16	28	30	-4	61	62	K=	8 L=	2	-10	36	38	K=	10 L=	4	0	37	31	2	34	31	-1	37	41
-15	32	33	-3	62	60	-3	29	31	-9	43	46	-3	43	44				3	36	34	0	41	39
-14	47	44	2	60	58	-1	52	52	-8	63	66	-2	63	62	K=	12 L=	2	4	30	23	1	30	27
-13	47	52	3	66	65	0	67	64	-7	72	76	-1	54	53	-4	51	55	5	28	27	2	31	23
-12	41	47	4	50	47	3	28	27	-6	70	67	0	49	48	-3	66	67	6	39	37			
-11	28	27	5	43	47				-5	68	66	1	60	58	-2	51	55	7	35	30	K=	16 L=	6
-10	29	24	6	50	48	K=	8 L=	3	-4	71	71	2	42	43	-1	71	74				-2	35	28
-9	45	45	7	52	48	-14	34	41	-3	60	56				0	80	84	K=	14 L=	1	-1	30	42
-8	25	27	8	34	35	-13	37	35	-2	35	33	K=	10 L=	5	1	55	60	-3	51	51	0	40	34
-7	18	15				-12	36	40	1	27	26	-4	39	39	2	55	60	-2	68	71			
-6	22	24	K=	7 L=	4	-11	60	59	2	60	57	-3	48	47	3	59	61	-1	58	59	K=	17 L=	4
-5	33	31	-11	29	28	-10	50	51	3	78	78	-2	51	50				0	41	43	-7	45	43
-4	62	60	-10	40	50	-9	57	55	4	64	65	-1	49	50	K=	12 L=	3	1	63	61	-6	36	36
-3	101	101	-9	46	47	-8	81	77	5	66	67	0	44	46	0	29	30	2	74	70	-5	36	35
-2	119	115	-8	38	40	-7	67	63	6	78	77	1	50	47				3	46	42	-4	31	33
-1	75	79	-7	53	55	-6	48	50	7	61	65	2	50	45	K=	12 L=	4				3	37	37
0	80	78	-6	85	84	-1	103	105	8	46	48				-13	39	32	K=	14 L=	3	4	34	35
1	117	114	-5	75	73	0	111	116	9	39	37	K=	10 L=	6	-12	36	33	-9	37	31	5	34	37
2	98	99	-4	28	24	1	54	54				-4	31	25	-11	39	36	-4	30	27	6	40	41
3	48	49	-3	18	13	2	53	50	K=	9 L=	4	-3	50	47	-10	33	33	-3	57	55	7	31	34
4	23	19	-2	33	31	3	76	72	-6	32	27	-2	40	41	-5	30	32	-2	66	68			
5	23	17	-1	20	19	4	60	57	-2	24	19	-1	28	27	-4	40	40	-1	43	44	K=	18 L=	4
7	34	35	0	27	25	9	44	44	1	32	30	0	37	37	-3	46	47	0	39	43	-3	25	34
8	46	46	1	47	41	10	59	62	4	20	20	1	46	44	-2	59	61	1	64	64	-2	30	37
9	29	29	3	33	32	11	40	45	5	26	21	2	36	33	-1	72	74	2	65	55	-1	28	33
10	28	30	4	79	80	12	34	33							0	68	72	3	30	30	0	37	35
11	42	46	5	82	81	13	42	38	K=	9 L=	5	K=	10 L=	7	1	56	57	8	38	34	1	32	36
12	46	53	6	50	51	14	41	38	-10	42	36	-4	34	28	2	41	42				2	28	30
13	37	41	7	38	37				-9	43	45	-3	46	43	3	35	35	K=	14 L=	4	3	31	26
14	32	28	8	44	43	K=	8 L=	4	-8	50	57	-2	41	40	4	30	27	0	26	22			
			9	43	46	-5	34	31	-7	60	64	-1	34	32	9	36	36						
K=	6 L=	5	10	26	24	-4	25	25	-6	49	54	0	44	40	10	30	37	K=	14 L=	5			
-2	24	21				-3	25	27	-5	44	50	1	40	37	11	29	33	-4	39	33			
1	24	26	K=	7 L=	5	-2	37	32	-4	57	56	2	27	25	12	32	30	-3	49	52			
			-8	47	46	-1	27	22	-3	40	41							-2	54	52			
K=	6 L=	6	-7	46	45	0	26	26	1	28	31	K=	11 L=	1	K=	12 L=	6	-1	39	39			
-16	35	25	-5	40	42	1	35	32	2	60	53	-5	43	36	-5	30	33	0	43	39			

STRUCTURE DETERMINATION

From a two-dimensional Pattersson projection $P(uv)$ and a Harker section $P(u\frac{1}{2}w)$ the antimony atoms were found to occupy the four-fold point position 4(*e*) in $P2_1/c$. A least-squares refinement based solely on the deduced antimony atom position was performed. Subsequent three-dimensional electron density difference syntheses revealed the positions of all other non-hydrogen atoms, *viz.* one phosphorus and six oxygen atoms. All atoms occupy the general point position 4(*e*).

A full matrix least-squares refinement was now performed with eight inter-layer scale factors and isotropic temperature factors for all atoms. The refinement converged to an *R*-value of 0.064 ($R=\sum||F_o|-|F_c||/\sum|F_o|$). After the absorption correction was applied the *R*-value was reduced to 0.058. An anisotropic temperature factor was introduced for the antimony atom and the positional and thermal parameters were refined together with an overall scale factor. The final *R*-value was 0.056 (1380 reflections). Only observed reflections were included in the calculations. The contributions of the antimony and phosphorus atoms were corrected for anomalous dispersion in a least-squares refinement. This gave no significant decrease in the *R*-factor or in the standard deviations as compared with the calculations based on the uncorrected values. No correction was applied for extinction as nothing in the intensity data indicates the presence of such effects. The atomic scattering factors used were those given by Cromer and Waber [9] for neutral antimony, and those by Hanson *et al.*[10] for neutral phosphorus and oxygen. Several weighting schemes were tried. That recommended by Hughes,[11] which gave the best result from the weight analysis (*cf.* Table 2), was used in the last refinement. The final atomic fractional coordinates and thermal parameters are given in Table 3. Observed and calculated structure factors are listed in Table 4.

Table 5. Interatomic distances (Å) and bond angles (°) in the structure of $SbO(H_2PO_4).H_2O$ (*cf.* Table 3 and Fig. 2).
The superscripts (*i*) – (*iii*) are used to indicate the following equivalent sites in the structure:

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

where *x*,*y*,*z*, are the atomic coordinates given in Table 3.

The phosphate tetrahedron			
P – O(1)	1.520(9)	∠O(1) – P – O(2)	112.3(5)
P – O(2)	1.525(10)	∠O(1) – P – O(3)	110.0(5)
P – O(3)	1.576(11)	∠O(1) – P – O(4)	110.0(5)
P – O(4)	1.546(9)	∠O(2) – P – O(3)	109.0(5)
		∠O(2) – P – O(4)	110.9(5)
		∠O(3) – P – O(4)	104.3(5)
The SbO_4 polyhedron			
Sb – O(1)	2.291(10)	∠O(1) – Sb – O(4)	156.3(3)
Sb – O(4)	2.145(9)	∠O(1) – Sb – O(5)	81.7(3)
Sb – O(5)	1.970(8)	∠O(1) – Sb – O(5^i)	81.2(3)
Sb – O(5^i)	1.977(8)	∠O(4) – Sb – O(5)	82.2(3)
		∠O(4) – Sb – O(5^i)	82.8(3)
		∠O(5) – Sb – O(5^i)	94.4(3)
The water oxygen atom			
O(6) – O(2^{iii})	2.794(15)	∠O(2^{iii}) – O(6) – O(3^{ii})	108.7(5)
O(6) – O(3^{ii})	2.872(16)		
O(6) – O(5)	2.969(14)		
O(6) – O(5^i)	2.971(14)		
O(6) – O(2^{ii})	2.996(15)		

DESCRIPTION AND DISCUSSION OF THE STRUCTURE

Six oxygen positions were found. Four of them [O(1) – O(4)], grouped around the phosphorus atom, form the phosphate tetrahedron (*cf.* Tables 3 and 5 for the atom notations). The distances P – O and the angles O – P – O (Table 5) are normal (*cf. International Tables* [12]).

One of the other two oxygen atoms, O(5), is directly bound to two antimony atoms. Thus it cannot belong to a water molecule, but it could be an ion O^{2-} or OH^-. The last oxygen atom, O(6), is situated at hydrogen bond distance from the nearest oxygen atom, $O(2^{iii})$, and is certainly a water oxygen. The temperature factors of the atoms O(5) and O(6) (*cf.* Table 3) also reflect their different situations.

That atom O(5) is an oxygen ion is supported by the following considerations. Since the X-ray data did not show the positions of the hydrogen atoms, it was difficult to distinguish O^{2-} and OH^-. To establish the nature of the oxygen atom, a calculation was made based on the principle of local neutralization of charge. This procedure makes it possible to recognize O^{2-}, OH^- and H_2O in crystal structures determined by X-ray diffraction methods. The valence sum $\sum v$ of possible bonds to O(5) was calculated according to Donnay and Allman.[13] Values of 2.0, 1.0, and 0.0 for $\sum v$ indicate, respectively, the presence of O^{2-}, OH^-, and H_2O. In the present case $\sum v$ for O(5) was found to be 1.9. It should thus be O^{2-}.

If O(5) is an oxide ion it follows that the phosphate tetrahedron must be an $H_2PO_4^-$ group and thus the structural formula should be written $SbO(H_2PO_4).H_2O$ instead of $SbPO_4.2H_2O$.

The antimony atom is coordinated to four oxygen atoms, O(1), O(4), O(5), and $O(5^i)$. The coordination polyhedron can be described as a distorted trigonal bipyramid with the unshared electron pair of the antimony atom occupying one of the equatorial corners. This type of coordination polyhedron has been found in several structures containing Sb^{3+}, Te^{4+}, or Pb^{2+}.[14] The distances and angles within the SbO_4 polyhedron are similar to those in related structures and to the calculated values given by Andersson *et al.*[14] A comparison of some distances and angles in the SbO_4 polyhedra in structures containing trivalent antimony is given in Table 6.

Table 6. Distances (Å) and angles (°) within the SbO_4 polyhedra (*cf.* Fig. 3) in some compounds containing antimony(III).
d_1 is the average of the axial distances Sb – O(1) and Sb – O(4), d_2 is the average of the equatorial distances Sb – O(5) and $Sb - O(5^i)$, α is the angle O(1) – Sb – O(4), and β is the angle $O(5) - Sb - O(5^i)$.

	$SbO(H_2PO_4).H_2O$	$SbPO_4$[5]	$\beta - Sb_2O_4$[14]	$SbNbO_4$[15]	L – SbOF[2,a]	M – SbOF[3,a]
d_1	1.97	2.01	2.02	2.02	1.97	2.00
d_2	2.22	2.18	2.22	2.23	2.16	2.19
α	156.3	164.8	148.1	150.7	142.9	145.0
β	94.4	87.9	87.9	92.1	95.9	93.7

[a] SbO_3F polyhedra instead of SbO_4 polyhedra.

The fundamental structural element in $SbO.(H_2PO_4).H_2O$ is an infinite layer of the composition $SbO(H_2PO_4)$ parallel to the *bc* plane. The water molecules are situated between the $SbO.(H_2PO_4)$ layers. Each layer consists of SbO_4 polyhedra and PO_4 tetrahedra sharing corners only. The interrelationships of the polyhedra are shown in Figs. 1a, 1b, and 1c. As can be seen in the figures, every SbO_4 polyhedron shares two oxygen atoms [O(5) and $O(5^i)$] with two

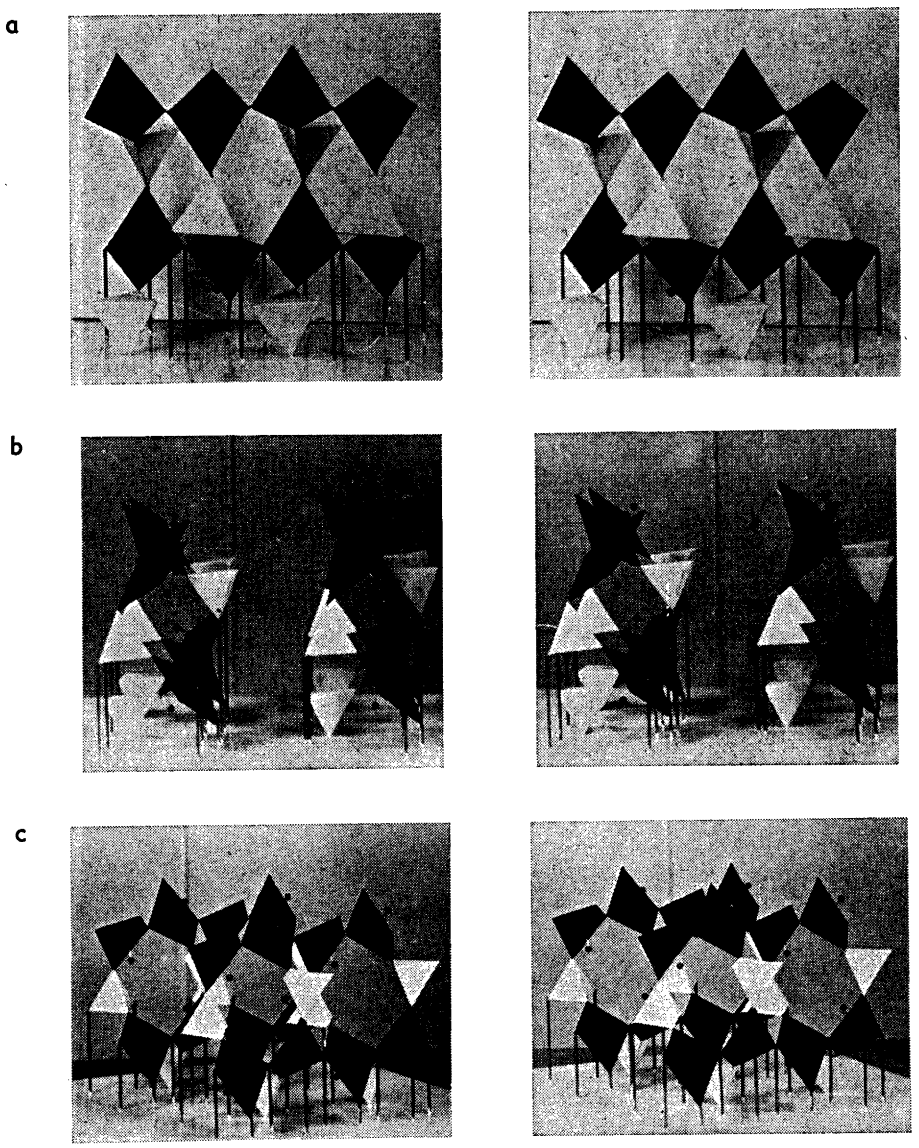

Fig. 1. Stereo views of the structure of $SbO(H_2PO_4).H_2O$. (Black polyhedra = SbO_4, white polyhedra = PO_4). a. The structure viewed along the x-axis. Only one layer is shown. b. The structure viewed along the z-axis. c. The structure viewed along [101].

other SbO_4 polyhedra and two oxygen atoms [O(1) and O(4)] with two PO_4 tetrahedra. The PO_4 tetrahedron shares two oxygen atoms [O(1) and O(4)] with two SbO_4 polyhedra. The other two oxygen atoms [O(2) and O(3)] are OH-groups. These two corners of the tetrahedron are directed out from the layer.

The layers are probably held together by weak hydrogen bonds involving the water molecules. Some possible hydrogen bonds are indicated by dashed-dotted lines in Fig. 2.

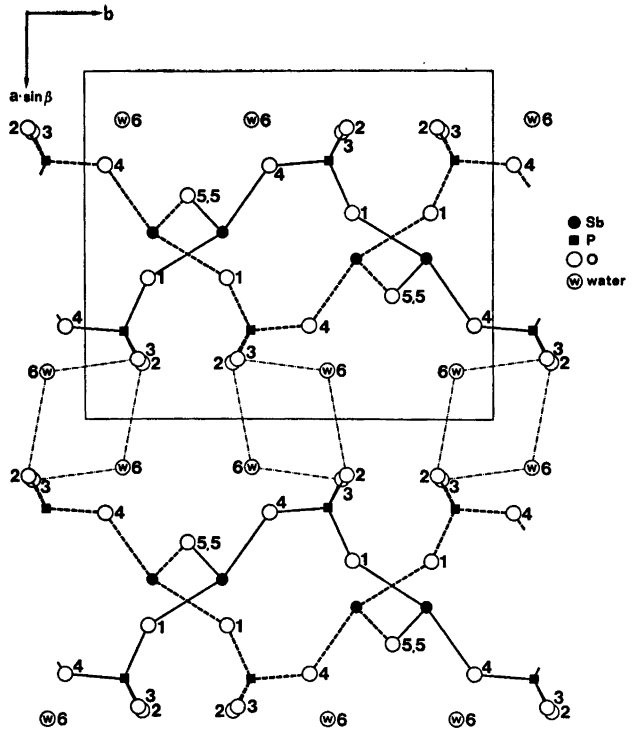

Fig. 2. Projection of the structure of $SbO(H_2PO_4).H_2O$ along [001]. The numbering of the oxygen atoms is given in the figure (*cf.* Table 3).

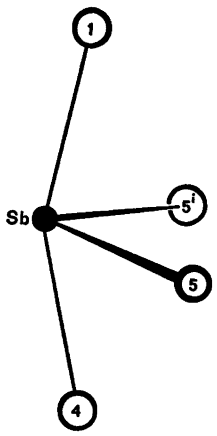

Fig. 3. The SbO_4 polyhedra. The notations of the oxygen atoms are given in the figure (*cf.* Tables 3 and 6).

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