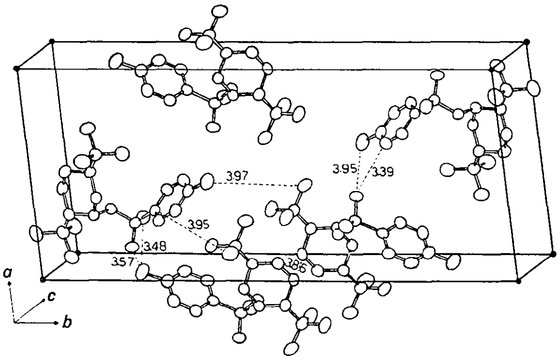

Fig. 6. *ORTEP* plot (Johnson, 1967) of the crystal structure of BCT, showing some short intermolecular distances.

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The Crystal Structure of Orthorhombic Antimony Trioxide, Sb_2O_3

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The crystal structure of orthorhombic Sb_2O_3 has been reinvestigated with three-dimensional single-crystal X-ray diffractometer data. The crystals are orthorhombic, space group *Pccn*, with $a = 4{\cdot}911$, $b = 12{\cdot}464$ and $c = 5{\cdot}412$ Å. The structure was refined by full-matrix least-squares with 340 independent reflexions to $R = 0{\cdot}032$. The idealized geometry of the antimony coordination can be described as a tetrahedron with oxygens at three corners, at the approximately equal distances 1·98, 2·02 and 2·02 Å, and the lone pair of electrons of antimony at the fourth corner. The coordination polyhedra are joined by sharing corners to form double infinite chains with the lone pairs pointing out from the chains.

Introduction

The structures of solid α- and β-Bi_2O_3 have recently been redetermined and refined (Malmros, 1970; Aurivillius & Malmros, 1972). Within this research programme, and also in order to obtain accurate antimony-(III)-oxygen distances, a refinement of orthorhombic Sb_2O_3 has been undertaken. The results of this investigation are in good agreement with the early structure determination reported by Buerger & Hendricks (1938).

Experimental

Orthorhombic Sb_2O_3 was prepared from a hydrochloric acid solution of $SbCl_3$ and a boiling sodium carbonate solution as described by Debray (1866). Single crystals suitable for X-ray work were obtained by subliming the oxide at 600 °C in nitrogen (Bloom & Buerger, 1937).

Weissenberg photographs taken with Cu $K\alpha$ radiation indicated orthorhombic symmetry and showed systematic extinctions unique for space group *Pccn* (No. 56).

The unit-cell dimensions were determined by least-squares refinement of 31 lines in the Cu $K\alpha_1$ (λ= 1·54051 Å) powder pattern collected at room temperature in a Guinier–Hägg camera equipped with a quartz monochromator and with KCl as internal standard (a = 6·2929 Å). The least-squares program *PIRUM* by Werner (1970) was used. The results were a = 4·911 (1), b = 12·464 (2), c = 5·412 (1) Å and V = 331·3 Å^3. Standard deviations of the least significant digits are given in parentheses. With $Z=4$ the calculated density is 5·844 g cm^{-3}, compared with 5·778 g cm^{-3} observed by Boullay (1830).

Intensities for a colourless, prismatic (110) crystal, elongated along **c**, of approximate dimensions 0·02 × 0·04 × 0·12 mm were collected at room temperature, 22 °C, on an Enraf–Nonius computer-controlled four-circle diffractometer, CAD4, with equatorial geometry. Graphite-monochromatized Mo $K\alpha$ radiation (λ= 0·71069 Å) with a take-off angle of 5° was used. The crystal was mounted with the c axis tilted approximately 13° from the φ axis of the goniometer. The crystal orientation was determined, after centring 14 reflexions in the detector aperture, by least-squares determination of the matrix relating the indices of reflexion to the orthogonal goniometer-head coordinate system.

The ω–2θ scan technique was used with an ω range of 1·00° and an aperture 4·5 mm wide and 6·0 mm high, corresponding to an angle in the equatorial plane as seen from the crystal of 1·5°. The distance X-ray source to crystal was 216 mm. A minimum net count of 3000 was attained within a maximum measuring time of 5 min. The scan speed was calculated from the net intensity in a fast prescan. The scan was continuous and the background counts were taken at each end of the scan interval with stationary crystal–stationary counter for $\frac{1}{4}$ of the time spent on the scan. The measurements were repeated and the corresponding counts were added in the output from the diffractometer.

Two octants of reciprocal space out to $(\sin\theta)/\lambda$= 0·70 were examined, part of one octant being measured twice. The total number of reflexions was 1195 of which 403 were considered not above background by giving net counts less than 10 in the fast prescan of 9 s. Another 59 intensities were considered unobserved, being weaker than $3\sigma(I)$, where I is the intensity and $\sigma(I)$ its standard deviation. I was calculated from $I=[T-k(B_1+B_2)]/t$ and $\sigma(I)$ from $\sigma^2(I)=[T+k^2(B_1+B_2)]/t^2$, where T, B_1 and B_2 are the peak counts and the two background counts respectively. The factor k is the ratio of scan time to total background measuring time, *i.e.* in this experiment $k=2$, and t is a factor proportional to the measuring time. The remaining 733 intensities were corrected for Lorentz, polarization and absorption effects with the program *DATAPC* which is a modification by the author of a program originally written by Coppens, Leiserowitz & Rabinovich (1965). The polarization factor used was the one for ideally mosaic crystals, $p=(1+\cos^2 2\theta_M \cos^2 2\theta)/(1+\cos^2 2\theta_M)$. The linear absorption coefficient for Mo $K\alpha$ radiation is 150 cm^{-1}. The transmission factors were in the range 0·53–0·73.

Two control reflexions, 240 and 080, were measured at 30 min intervals to check for crystal decomposition and radiation stability. The mean decrease in intensity over the whole exposure time, 54 h, was 4 % for both control reflexions. Maximum intensity variations over the data collection period, 90 h, were 7 and 8 % respectively corresponding to approximately 7 and $8\sigma(I)$. All intensities and their variances were scaled according to these control reflexions.

The structure refinement was started with the atomic coordinates given by Buerger & Hendricks (1938) (*cf.* Table 1) and isotropic temperature factors. Antimony and one of the oxygen atoms occupy the general eightfold point position, whereas the other oxygen atom is located on a twofold axis in a fourfold point position. The least-squares program used was *LINUS* by Coppens & Hamilton (1970). The function minimized was $\sum w_i(|F_o|-|F_c|)^2$ where the weights, w_i, were calculated from $w_i^{-1}=\sigma^2(F_o^2)/4F_o^2+cF_o^2$. The constant c was chosen so as to given the most constant averages of $w_i(|F_o|-|F_c|)^2$ over ranges of F and $\sin\theta$. A value of c = 0·00025 was used. The scattering factors were those of Doyle & Turner (1968) for neutral atoms antimony and oxygen. An anomalous dispersion correction for antimony (Cromer & Liberman, 1970) was included. The process of refinement can be followed in Table 2. The 733 observed reflexions refined to $R=\sum||F_o|-|F_c||/\sum|F_o|$ = 0·039 with all atoms anisotropic and an isotropic correction for secondary extinction. The extinction parameter was g = 3·2 (3) . 10^3. The structure factor of the reflexion suffering most from extinction was corrected by 29 %. After averaging of all equivalent reflexions the final R was 0·032 for 340 reflexions. The shifts in the last cycle of refinement

Table 1. *Fractional coordinates of orthorhombic* Sb_2O_3 *according to Buerger & Hendricks* (1938)

	x	y	z
Sb	0·044	0·128	0·179
O(1)	0·25	0·25	0·029
O(2)	0·147	0·058	−0·139

Table 2. *Summary of the refinement*

Parameters refined	Number of observations (m)	Number of parameters refined (n)	S	R_w	R	$\sigma(z)$ for O(1)
(a) overall scale factor, atomic coordinates, isotropic temperature factors	733	11	3·13	0·071	0·060	0·0024
(b) same as (a) but anisotropic temperature factor for Sb.	733	16	2·51	0·057	0·048	0·0019
(c) same as (b) but also isotropic extinction correction	733	17	2·05	0·046	0·040	0·0016
(d) same as (c) but anisotropic temperature factors for O(1) and O(2)	733	25	2·00	0·045	0·039	0·0014
(e) all equivalent reflexions averaged.	340	24	1·59	0·037	0·032	0·0015

$S=[\sum w(|F_o|-|F_c|)^2/(m-n)]^{1/2}$, $R_w=(\sum w(|F_o|-|F_c|)^2/\sum w|F_o|^2)^{1/2}$, $R=\sum||F_o|-|F_c||/\sum|F_o|$, $\sigma(z)$ for O(1) is the standard deviation of fractional coordinate z for atom O(1).

Table 3. *Final positional and thermal parameters*

The form of the temperature factor is $\exp\,[-(h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+2hk\beta_{12}+2kl\beta_{23}+2hl\beta_{13})]$. The β_{ij}'s have been multiplied by 10^4. Least-squares standard deviations are given in parentheses.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Sb	0·04149 (11)	0·12745 (4)	0·17845 (9)	122·1 (2·5)	17·4 (0·4)	62·0 (1·9)	9·7 (0·8)	7·4 (1·6)	5·1 (0·7)
O(1)	0·25	0·25	0·0229 (15)	250 (43)	15 (4)	40 (24)	−5 (12)	0	0
O(2)	0·1520 (15)	0·0591 (4)	−0·1446 (10)	121 (26)	17 (3)	73 (19)	−17 (8)	−42 (19)	10 (7)

were for all parameters less than 0·01 times their standard deviations. At this stage a difference synthesis showed residuals near the antimony position ranging from −1·9 to 1·7 e Å^{-3}. The largest residuals elsewhere were 1·1 e Å^{-3}. The final positional and thermal parameters and their estimated standard deviations are given in Tables 3 and 4. Observed and calculated structure factors are listed in Table 5. Some selected distances and angles are given in Table 6. These were calculated by program *ORFFE* (Busing & Levy, 1964), using the complete variance–covariance matrix to determine standard deviations.

Table 4. *Root-mean-square amplitudes along the principal axes of the thermal vibration ellipsoids*

	R_1(Å)	R_2(Å)	R_3(Å)
Sb	0·0928 (17)	0·1070 (16)	0·1334 (13)
O(1)	0·077 (24)	0·108 (17)	0·175 (16)
O(2)	0·082 (17)	0·095 (14)	0·152 (12)

Discussion

The structure consists of infinite chains of composition Sb_2O_3 running parallel to **c**. Part of such a chain is shown in Fig. 1. Three oxygen atoms are coordinated all to one side of antimony, with a stereochemically active lone pair of electrons completing a somewhat distorted tetrahedron. Three-valent antimony with this one-sided coordination also occurs in compounds such as cubic Sb_2O_3 (Bozorth, 1923) and SbF_3 (Edwards, 1970). Every oxygen is shared between two antimony atoms and the three antimony–oxygen distances, 2·023 (4), 2·019 (6) and 1·977 (7) Å, are very similar. In fact, assuming an underestimation of the standard

Table 5. *Observed and calculated structure factors*

The columns list h, F_o and F_c.

h	F_o	F_c
K= 0 L= 0		
2	230	235
4	92	94
K= 0 L= 2		
0	169	192
1	34	35
2	158	160
3	115	116
4	46	46
5	122	123
K= 0 L= 4		
0	59	63
1	42	42
2	43	43
3	122	124
5	114	118
K= 0 L= 6		
0	143	158
1	20	32
2	104	109
3	40	39
4	48	50
K= 1 L= 0		
1	113	113
3	113	118
5	108	108
K= 1 L= 1		
1	124	119
2	63	65
3	38	38
4	112	114
6	76	78
K= 1 L= 2		
0	176	173
1	19	20
2	132	131
3	83	84
4	57	59
5	59	58
K= 1 L= 3		
1	194	190
2	26	28
3	83	83
4	19	18
5	26	26
K= 1 L= 4		
0	146	151
2	129	130
3	23	26
4	49	51
K= 1 L= 5		
1	90	97
2	23	24
3	58	60
4	54	54
K= 1 L= 6		
0	53	59
1	30	33
2	34	33
3	56	57
4	19	17
K= 1 L= 7		
2	40	41
K= 2 L= 0		
4	17	19
K= 2 L= 1		
1	293	279
2	77	77
3	102	107
4	66	67
5	37	36
6	49	49
K= 2 L= 2		
0	24	21
1	36	31
2	19	20
K= 2 L= 3		
1	62	59
2	127	128
3	24	24
4	133	134
6	102	100
K= 2 L= 4		
0	27	25
2	17	12
3	18	12
K= 2 L= 5		
1	107	106
2	64	67
3	74	77
4	88	90
5	21	21
K= 2 L= 7		
1	113	123
K= 3 L= 0		
1	67	69
3	126	130
5	82	81
K= 3 L= 1		
1	96	93
2	106	107
3	59	60
4	104	106
6	75	76
K= 3 L= 2		
0	124	121
1	58	55
2	136	133
3	56	56
4	59	59
5	59	58
K= 3 L= 3		
1	180	170
2	19	17
3	107	106
4	28	27
5	26	23
K= 3 L= 4		
0	148	143
1	31	30
2	110	110
4	57	57
K= 3 L= 5		
1	105	102
2	42	43
3	61	58
4	47	47
K= 3 L= 6		
0	44	46
1	30	25
2	32	35
3	60	59
K= 3 L= 7		
2	44	45
K= 4 L= 0		
0	296	308
2	255	260
4	86	88
K= 4 L= 1		
1	37	35
2	15	14
K= 4 L= 2		
0	208	199
1	55	54
2	121	121
3	119	118
4	59	59
5	102	102
K= 4 L= 3		
2	16	10
K= 4 L= 4		
0	56	56
1	60	56
2	33	32
3	112	112
4	29	24
5	111	111
K= 4 L= 5		
1	16	12
K= 4 L= 6		
0	136	132
2	110	113
3	38	43
K= 5 L= 0		
1	10	10
3	121	126
5	109	113
K= 5 L= 1		
1	86	84
2	65	65
3	40	42
4	89	88
6	67	66
K= 5 L= 2		
0	199	189
1	46	43
2	116	116
3	62	60
4	35	36
5	70	69
K= 5 L= 3		
1	155	149
2	17	16
3	84	82
5	23	25
K= 5 L= 4		
0	175	168
2	113	112
4	47	46
5	31	28
K= 5 L= 5		
1	84	82
2	27	27
3	51	50
4	42	44
K= 5 L= 6		
1	20	16
2	48	47
3	60	59
K= 6 L= 0		
0	14	15
2	35	35
K= 6 L= 1		
1	249	246
2	39	40
3	116	119
4	67	68
5	34	33
6	50	48
K= 6 L= 2		
0	28	27
1	16	15
K= 6 L= 3		
1	43	42
2	67	64
3	33	31
4	132	124
K= 6 L= 4		
1	13	16
K= 6 L= 5		
1	108	105
2	64	62
3	56	54
4	77	77
K= 6 L= 7		
1	114	113
K= 7 L= 0		
1	39	41
3	72	73
5	85	87
K= 7 L= 1		
1	72	73
2	96	98
3	54	54
4	94	95
6	71	64
K= 7 L= 2		
0	138	137
2	88	86
3	53	54
4	43	40
5	45	42
K= 7 L= 3		
1	145	143
3	99	98
4	26	27
5	23	19
K= 7 L= 4		
0	117	112
2	94	93
3	20	19
4	37	39
K= 7 L= 5		
1	97	94
2	46	44
3	53	53
4	41	42
K= 7 L= 6		
0	35	33
1	26	20
2	32	24
3	41	40
K= 8 L= 0		
0	219	231
2	176	182
4	91	94
K= 8 L= 1		
1	24	23
K= 8 L= 2		
0	122	121
1	64	63
2	115	115
3	96	95
4	42	42
5	82	80
K= 8 L= 3		
2	21	17
4	17	16
K= 8 L= 4		
0	20	20
1	45	44
2	43	42
3	103	102
4	20	19
K= 8 L= 5		
2	17	10
K= 8 L= 6		
0	118	113
2	97	92
K= 9 L= 0		
1	53	55
3	90	92
5	97	98
K= 9 L= 1		
1	48	48
2	61	62
3	43	44
4	57	57
K= 9 L= 2		
0	136	134
1	18	17
2	94	95
3	67	66
4	38	38
5	55	53
K= 9 L= 3		
1	98	96
3	78	78
5	28	23
K= 9 L= 4		
0	126	127
2	105	103
3	22	18
4	38	37
K= 9 L= 5		
1	63	62
2	33	34
3	35	38
K= 9 L= 6		
0	52	49
1	23	25
2	29	32
K= 10 L= 0		
0	56	59
K= 10 L= 1		
1	172	174
2	33	33
3	93	95
4	52	51
5	25	26
K= 10 L= 2		
5	26	21
K= 10 L= 3		
1	31	33
2	70	68
3	21	22
4	102	101
K= 10 L= 4		
3	18	20
K= 10 L= 5		
1	86	83
2	48	47
3	47	47
K= 10 L= 6		
0	22	21
K= 11 L= 0		
3	56	57
5	61	60
K= 11 L= 1		
1	62	63
2	58	58
4	86	86
K= 11 L= 2		
0	90	91
1	34	18
2	63	64
3	38	25
4	25	26
5	38	37
K= 11 L= 3		
1	122	123
2	21	19
3	63	64
K= 11 L= 4		
0	88	88
2	61	61
K= 11 L= 5		
1	78	77
K= 12 L= 0		
0	155	164
2	133	137
4	54	55
K= 12 L= 1		
1	20	20
2	18	15
3	23	19
K= 12 L= 2		
0	102	104
1	25	25
2	69	70
3	72	71
4	44	38
K= 12 L= 3		
1	17	13
K= 12 L= 4		
1	34	34
2	24	19
3	73	71
K= 12 L= 5		
2	19	12
K= 13 L= 0		
1	38	39
3	83	85
K= 13 L= 1		
1	35	36
2	34	31
3	26	25
4	43	42
K= 13 L= 2		
0	81	80
1	28	29
2	78	78
3	44	44
4	40	35
K= 13 L= 3		
1	68	69
3	50	49
K= 13 L= 4		
0	97	99
1	23	16
2	78	77
K= 14 L= 0		
0	28	30
K= 14 L= 1		
1	109	111
2	32	32
3	79	79
4	34	32
K= 14 L= 3		
1	28	26
2	59	60
K= 15 L= 0		
1	25	25
3	33	30
K= 15 L= 1		
1	42	46
2	44	42
K= 15 L= 2		
0	47	47
2	45	45
3	23	23
K= 15 L= 3		
1	92	92
K= 16 L= 0		
0	122	126
2	81	84
K= 16 L= 1		
1	22	25
K= 16 L= 2		
0	63	63
2	56	56
K= 17 L= 0		
1	29	29
K= 17 L= 1		
1	28	26

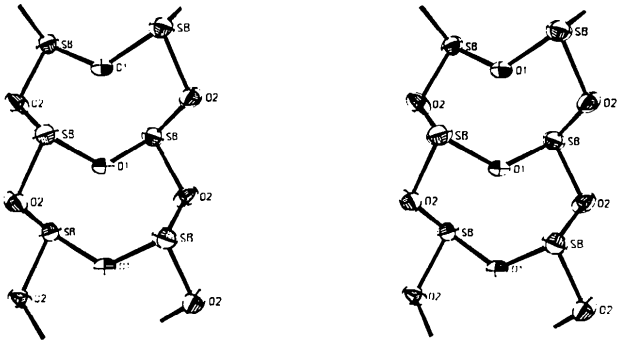

Fig. 1. A stereoscopic view down [100] of part of an infinite chain in orthorhombic Sb_2O_3, drawn by program *ORTEP* (Johnson, 1965). The thermal vibration ellipsoids have been scaled to include 95% probability.

Table 6. *Interatomic distances and bond angles*

All distances shorter than 3·0 Å are included. The superscripts refer to atoms at the following equivalent positions: x, y, z; (i) $\frac{1}{2}-x, \frac{1}{2}-y, z$; (ii) $-x, -y, -z$; (iii) $\frac{1}{2}-x, y, \frac{1}{2}+z$ and (iv) $\frac{1}{2}-x, y, z-\frac{1}{2}$, where x, y, z refer to the coordinates listed in Table 4. Standard deviations are given in parentheses.

(*a*) Distances

Sb—O(1)	2·023 (4) Å
Sb—O(1^{iii})	2·619 (6)
Sb—O(2)	2·019 (6)
Sb—O(2^{ii})	2·518 (6)
Sb—O(2^{iii})	1·977 (7)
O(1)–O(1^{iii}), O(1^{iv})	2·706 (1)
O(1)–O(2)	2·592 (6)
O(2)–O(2^{ii})	2·617 (12)
O(2)–O(2^{iii}), O(2^{iv})	2·872 (5)

The shortest antimony–antimony distance is Sb–Sb^{iii} of 3·394 (1) Å.

(*b*) Angles

O(1)–Sb—O(2)	79·8 (3)°
O(1)–Sb—O(2^{ii})	98·1 (2)
O(2)–Sb—O(2^{ii})	91·9 (2)
Sb—O(1)–Sb^{i}	130·8 (4)
Sb—O(2)–Sb^{iii}	116·2 (3)

deviations by a factor of two the differences are not significant at the 5% level. The weighted mean Sb–O bond distance is 2·013 (6) Å.

The antimony atom has two next-nearest oxygen neighbours at distances 2·619 (6) and 2·518 (6) Å in the same chain and in a neighbouring chain, respectively. The latter distance probably results from an antimony–oxygen interaction and this would thus contribute to the van der Waals forces to hold the structure together. The interlayer bonding is however weak and accounts well, as pointed out by Buerger & Hendricks, for the perfect prismatic cleavage of the crystals.

The structure can also be discussed with respect to the volume per anion. It has recently been shown by Andersson, Åström, Galy & Meunier (1973) that for many solid oxides, or oxide fluorides of Sb^{3+}, Pb^{2+}, Bi^{3+} and Te^{4+} the volume of the lone pair and its cation is of the size of that of an anion. If simply the unit-cell volume of orthorhombic Sb_2O_3 is divided by its contents of antimony lone pairs and oxygens the resulting volume per anion is 16·6 Å^3. When comparing with the corresponding figure 17·3 Å^3 for cubic Sb_2O_3 one can conclude that the orthorhombic polymorph should be the high-pressure form. The magnitude of these figures also compares well with the corresponding figures 16·6, 16·6 and 16·7 Å^3 for SbF_3 (Edwards, 1970) and *L*- and *M*-SbOF (Åström, 1972; Åström & Andersson, 1973), respectively.

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