

The Crystal Structure of the Double Lithium Antimony(V)oxide LiSbO_3

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In the review of antimonates given by Wells¹ it is pointed out that all antimonates, which do not contain water, are best described as complex oxides all based on octahedral SbO_6 coordination. When one of us in trying to prepare a double lithium antimony(III) oxide halogenide found that this gave crystals evidently of LiSbO_3 , it was therefore decided to continue the investigation on lithium antimonate by Schrewelius². Schrewelius had to base his investigation on powder photographs from which he could find probable positions of the antimony atoms but had to leave the question of the oxygen and lithium positions open. It thus seemed of interest to see if LiSbO_3 would contain isolated antimonate groups or rather be described as a double oxide.

PREPARATION

Schrewelius² prepared LiSbO_3 by heating LiSb(OH)_6 and by heating lithium carbonate and antimony(III)oxide in various proportions at various temperatures. This did not give crystals sufficiently large for single crystal photographs.

In this investigation LiSbO_3 was prepared by heating a mixture of Sb_2O_3 and a large amount of LiBr or LiCl in a porcelain boat in a tube heated at temperatures between 550° and 800° C and in a stream of air or oxygen for about three hours. The melt was then extracted with water, and the residue of LiSbO_3 filtered and dried. The best way to prepare crystals of sufficient size was to heat a mixture of Sb_2O_3 and a large amount of LiCl in an open porcelain crucible at 750° – 800° C with a stream of oxygen passing over the surface of the melt. Portions of LiCl were poured in now and then as LiCl evaporated fairly rapidly. The time was about 24 hours. The residue of LiSbO_3 was then made free from LiCl by dissolving the latter in water. The crystals were needle-shaped.

ANALYSES

LiSbO_3 is very difficult to dissolve in acids, so it was dissolved and pentavalent antimony determined in the following way. Approximately 200 mg of the sample was mixed with 100 ml concentrated hydrochloric acid and 2 g potassium iodide in a distilling flask through which was lead a stream of carbon dioxide. The distilling flask was heated and the liberated iodine received in a flask containing potassium iodide. The time of distillation was 5–9 hours. The iodine was titrated with sodium thiosulfate. In the distilling flask, the remaining trivalent antimony was precipitated as sulfide, which as a control

was dissolved in hydrochloric acid and titrated with potassium bromate according to Smith and May³ with naphthol blue-black as indicator. The thiosulfate and the bromate titration gave practically the same result. To free the filtrate from iodide it was run through a cation exchange column containing Dowex 50. Lithium and potassium were then extracted with hydrochloric acid. From this mixture lithium chloride was extracted with absolute alcohol and ether according to Palkin⁴ and then converted to the sulfate and weighed as Li_2SO_4 .

	Antimony %	Lithium %
Calc. for LiSbO_5 :	68.91	3.93
Found:	68.5, 68.7, 68.9	3.94, 3.90, 3.88

UNIT CELL AND SPACE GROUP

Single crystals were selected and set and rotation and Weissenberg photographs were taken round two of the axes with Cu-radiation. The following Weissenberg photographs were taken: $hk0$, hkl , $hk2$, $0kl$, $1kl$, and $2kl$. The crystals proved to be orthorhombic with the c -axis coinciding with the needle axis. The cell dimensions were more accurately determined from powder photographs of which a section is given in Table 1. The values from the present investigation are compared with those by Schrewelius².

Present investigation	Schrewelius
$a = 4.883 \text{ kX} = 4.893 \text{ \AA}$	$a = 4.89 \text{ kX}$
$b = 8.474 \text{ kX} = 8.491 \text{ \AA}$	$b = 8.46 \text{ kX}$
$c = 5.172 \text{ kX} = 5.183 \text{ \AA}$	$c = 5.18 \text{ kX}$
$V = 214.010 \text{ kX}^3 = 215.335 \text{ \AA}^3$	

with an accuracy of about $\pm 0.05 \%$.

If 4 formula units are assumed per unit cell the density would be $d_{\text{calc}} = 5.45$. $d_{\text{obs}} = 5.25, 5.26, 5.35$. (The density of the crystals was determined from the loss of weight in benzene).

In the photographs the following reflections are systematically absent

$$\begin{aligned} 0kl \text{ with } k + l &= 2n + 1 \\ h0l \text{ with } l &= 2n + 1 \\ hk0 \text{ with } h + k &= 2n + 1 \end{aligned}$$

This is characteristic of the space group No. 52, $Pncn-D_{2h}^6$. (Space group number according to International Tables⁵.)

PATTERSON SYNTHESSES TO FIND THE ANTIMONY POSITIONS

Schrewelius² has, from powder data, placed the four antimony atoms of the cell in the fourfold position 4 (c): $0, y, \frac{1}{4}; 0, \bar{y}, \frac{3}{4}; \frac{1}{2}, \frac{1}{2} + y, \frac{3}{4}; \frac{1}{2}, \frac{1}{2} - y, \frac{1}{4}$ with $y = 0.20$.

In this investigation it seemed best to start with Patterson syntheses to find the positions of the antimony atoms. The values of the intensities were estimated visually from the Weissenberg photographs (double films) and then corrected for the Lorentz and polarization factors using the curves given by Kaan and Cole⁶. These corrected intensities were used instead of the F^2 values.

Table 1. A section of a powder photograph of LiSbO_3 . CrK α -radiation. $\lambda = 2.2863$ kX = 2.2909 Å. For lines, where a_1 and a_2 reflections are separated the given $\sin^2\Theta_{\text{obs}}$ are the weighted averages. Notations: v st = very strong, st = strong, m = medium, w = weak, v w = very weak.

<i>hkl</i>	$10^4\sin^2\Theta$		<i>I</i>
	calc	obs	
123	5 673	5 681	m
232	5 784		
033	6 035	6 043	m
321	6 149	6 158	st
060	6 551	6 557	m
330	6 570		
133	6 583	6 579	v w
213	6 771	6 769	w
302	6 886		
152	7 051		
242	7 058		
331	7 058	7 070	v st
312	7 068		
251	7 230		
223	7 317		
161	7 587	7 589	w
322	7 614		
004	7 817		
143	7 857	7 862	w
233	8 227	8 222	m
341	8 332	8 335	m++
104	8 365		
062	8 505	8 506	m
332	8 524		
024	8 545	8 538	m
114	8 547		
252	8 696		
260	8 743	8 741	w+
400	8 768	8 768	m++
053	8 946		
162	9 053	9 044	v w
261	9 231		
071	9 405	9 403	m+
411	9 439		
170	9 464	9 461	w
350	9 481	9 475	m++
153	9 494		
420	9 496		
243	9 501		
313	9 511		

In space group No. 52, $Pncn-D_{2h}^6$ we have the following equivalent positions:

- 8 (e) $x, y, z; \bar{x}, y, \frac{1}{2}-z; \frac{1}{2}-x, \frac{1}{2}-y, z; \frac{1}{2} + x, \frac{1}{2}-y, \frac{1}{2}-z;$
 $\bar{x}, \bar{y}, \bar{z}; x, \bar{y}, \frac{1}{2} + z; \frac{1}{2} + x, \frac{1}{2} + y, \bar{z}; \frac{1}{2}-x, \frac{1}{2} + y, \frac{1}{2} + z$
- 4 (d) $\frac{1}{2}, \frac{1}{2}, z; \frac{3}{4}, \frac{3}{4}, \bar{z}; \frac{1}{4}, \frac{3}{4}, \frac{1}{2} + z; \frac{3}{4}, \frac{1}{4}, \frac{1}{2}-z$
- 4 (c) $0, y, \frac{1}{4}; 0, \bar{y}, \frac{3}{4}; \frac{1}{2}, \frac{1}{2} + y, \frac{3}{4}; \frac{1}{2}, \frac{1}{2}-y, \frac{1}{4}$
- 4 (b) $0, \frac{1}{2}, 0; 0, \frac{1}{2}, \frac{1}{2}; \frac{1}{2}, 0, 0; \frac{1}{2}, 0, \frac{1}{2}$
- 4 (a) $0, 0, 0; 0, 0, \frac{1}{2}; \frac{1}{2}, \frac{1}{2}, 0; \frac{1}{2}, \frac{1}{2}, \frac{1}{2}$

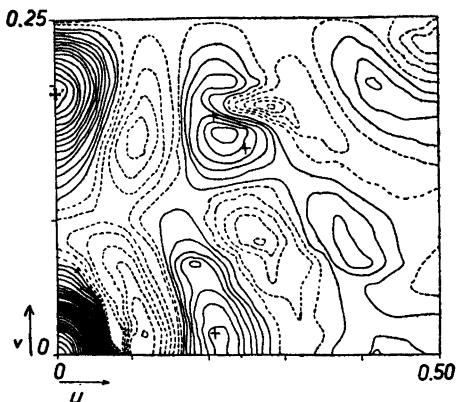


Fig. 1. $P(uvp)$. Contours at an interval of 100 arbitrary units. Negative values dotted. + Sb-Sb vectors and + Sb-O vectors calculated with the final parameters.

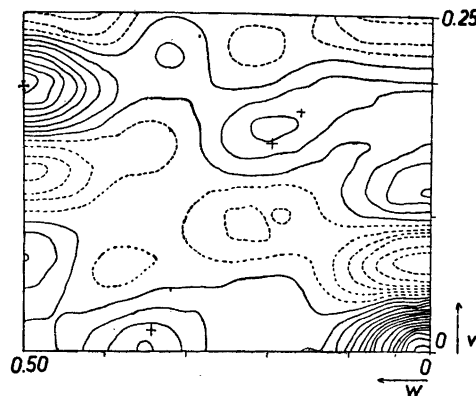


Fig. 2. $P(pvw)$. Contours at an interval of 100 arbitrary units. Negative values dotted. + Sb-Sb vectors and + Sb-O vectors calculated with the final parameters.

In the Patterson space $P(uvw)$ the fourfold positions give the following vectors

I	within 4 (a) and 4 (b) of weight 4	$(0, 0, \frac{1}{2}); (\frac{1}{2}, \frac{1}{2}, 0); (\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$
II	within 4 (c) A of weight 2	$\pm (0, 2y, \frac{1}{2}); \pm (\frac{1}{2}, \frac{1}{2} + 2y, 0)$
	B of weight 4	$(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$
III	within 4 (d) A of weight 2	$\pm (\frac{1}{2}, \frac{1}{2}, 2z); \pm (\frac{1}{2}, 0, \frac{1}{2} + 2z)$
	B of weight 4	$(0, \frac{1}{2}, \frac{1}{2})$

The calculated $P(uvp)$ -projection is given in Fig. 1 and the $P(pvw)$ -projection in Fig. 2. The positions 4 (a) and 4 (b) would require a maximum in $P(pvw)$ at $v = 0$ $w = \frac{1}{2}$, which does not exist, so that these positions can be excluded. As the maximum next in height to that at the origin and of approximately half of its height is situated at $u = 0$ for $P(uvp)$ and at $w = \frac{1}{2}$ for $P(pvw)$ it seems probable that antimony is placed in 4 (c).

From $P(uvp)$ we thus have $2y = v = 0.1950, 0.8050, 1.1950 \dots$ which gives $y = 0.0975, 0.4025, 0.5975 \dots$

From $P(pvw)$ we have $2y = v = 0.1938, 0.8062, 1.1938 \dots$ which gives $y = 0.0969, 0.4031, 0.5969 \dots$

The mean value is $y = 0.0972, 0.4028, 0.5972 \dots$ where we choose $y = 0.0972$. We can see that $y = 0.20$ given by Schrewelius² does not fall in the series of possible y -values.

The lower maxima in the Patterson projections might indicate Sb-O vectors, but it seems safer not to make any suggestions of the oxygen positions at this stage. (The subsequently calculated interatomic vectors are marked in Figs. 1 and 2 and we see that they coincide with some of the lower maxima.)

FOURIER SYNTHESSES

The best way to refine the antimony parameter and try to find the oxygen positions seemed to be by Fourier syntheses. These syntheses have been calculated with $F_{\text{obs}} = \sqrt{\frac{I}{Lp}}$ where F is the structure factor, I is the estimated intensity, Lp are the combined Lorentz and polarization factors. In all syntheses the value of $F(000)$ has been omitted.

The $\rho(XYp)$ - and $\rho(pYZ)$ -projections were calculated. Minor maxima were obtained that seemed to indicate quite probable oxygen positions. In order to refine the oxygen positions we applied the bounded projection method (Booth⁷). When the difference between the bounding planes is a half only the zero zone and the odd zones are included. We must, however, be careful that the bounding planes do not pass too near the atoms we want to locate.

For this reason we calculated $B_{Z=-\frac{1}{4}}^{Z=+\frac{1}{4}}$ and $B_{X=0}^{X=\frac{1}{2}}$ as $\rho(XYp)$ and $\rho(pYZ)$ indicating that the twelve oxygen atoms are situated in 8 (e) with approximately $x = 0.21$, $y = 0.08$, $z = -0.09$ and in 4 (d) with approximately $z = 0.03$. To include as many reflections as possible in the determinations of the antimony parameter we also calculated $B_{Z=0}^{Z=\frac{1}{2}}$. In space group No. 52, $Pncn-D_{2h}^6$ we have the following expressions for the bounded projections:

$$B_{Z=-\frac{1}{4}}^{Z=+\frac{1}{4}} = \frac{2}{A} \sum_{h=0}^{\infty} \sum_{k=0}^{\infty} F(hk0) \cos 2\pi h X \cos 2\pi k Y \pm \frac{2}{A} \sum_{h=0}^{\infty} \sum_{k=0}^{\infty} \sum_{l=1}^{\infty} \frac{4}{\pi l} F(hkl) \sin 2\pi h X \sin 2\pi k Y$$

where the signs are — for $l = 4n + 1$ and + for $l = 4n + 3$.

$$B_{Z=0}^{Z=\frac{1}{2}} = \frac{2}{A} \sum_{h=0}^{\infty} \sum_{k=0}^{\infty} F(hk0) \cos 2\pi h X \cos 2\pi k Y - \frac{2}{A} \sum_{h=0}^{\infty} \sum_{k=0}^{\infty} \sum_{l=1}^{\infty} \frac{4}{\pi l} F(hkl) \cos 2\pi h X \sin 2\pi k Y$$

$$B_{X=0}^{X=\frac{1}{2}} = \frac{2}{A} \left\{ \sum_{k=0}^{\infty} \sum_{l=0}^{\infty} F(0kl) \cos 2\pi k Y \cos 2\pi l Z - \sum_{k=0}^{\infty} \sum_{l=0}^{\infty} F(0kl) \sin 2\pi k Y \sin 2\pi l Z \right\} \\ - \frac{2}{A} \sum_{h=1}^{\infty} \sum_{k=0}^{\infty} \sum_{l=0}^{\infty} \frac{4}{\pi h} F(hkl) \cos 2\pi k Y \sin 2\pi l Z - \frac{2}{A} \sum_{h=1}^{\infty} \sum_{k=0}^{\infty} \sum_{l=0}^{\infty} \frac{4}{\pi h} F(hkl) \sin 2\pi k Y \cos 2\pi l Z$$

For $B_{Z=-\frac{1}{4}}^{Z=\frac{1}{4}}$ and $B_{Z=0}^{Z=\frac{1}{2}}$ only the Weissenberg photographs $hk0$ and $hk1$ were used and were correlated after correction for the Lorentz and polarization factors by the aid of the Weissenberg photographs $0kl$, $1kl$ and $2kl$. For $B_{X=0}^{X=\frac{1}{2}}$ the Weissenberg photographs $0kl$ and $1kl$ were used and similarly correlated



Fig. 3. The bounded projection $B_{z=0}^{z=+\frac{1}{2}}$. Contours at an interval of 50 arbitrary units. Negative values dotted. The final O positions are marked + and the Li positions +. The bounding planes pass through the centers of the Sb and Li atoms.

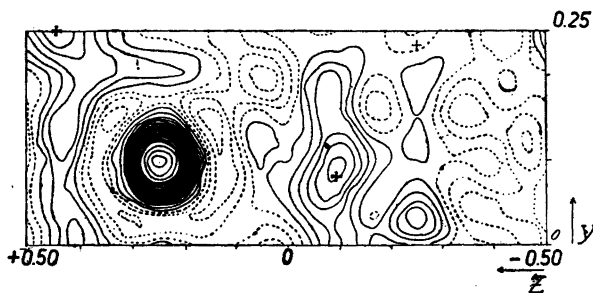


Fig. 4. The bounded projection $B_{x=0}^{x=\frac{1}{2}}$. Contours at an interval of 50 arbitrary units. Negative values dotted. The final O positions are marked + and the Li positions +. The bounding planes pass through the centers of the Sb and Li atoms.

by means of the Weissenberg photographs $hk0$, $hk1$ and $hk2$. For $B_{z=0}^{z=+\frac{1}{2}}$ (Fig. 3) and $B_{x=0}^{x=\frac{1}{2}}$ (Fig. 4) only very few reflections were included from the higher zones and to these reflections only oxygen atoms contribute.

We arrived at the following set of parameters:

$$\begin{array}{l}
 \text{Sb in 4(c) with } y = 0.0971 \quad \text{from } B_{z=0}^{z=\frac{1}{2}} \\
 \text{O}_1 \text{ in 4(d) with } z = 0.444 \quad \text{from } B_{x=0}^{x=\frac{1}{2}} \\
 \text{O}_2 \text{ in 8(e) with } x = 0.209 \quad \left. \begin{array}{l} \\ \\ \end{array} \right\} \text{from } B_{z=0}^{z=+\frac{1}{2}} \\
 \quad \quad \quad y = 0.0728 \quad \left. \begin{array}{l} \\ \\ \end{array} \right\} \\
 \quad \quad \quad y = 0.0895 \quad \left. \begin{array}{l} \\ \\ \end{array} \right\} \text{from } B_{x=0}^{x=\frac{1}{2}} \\
 \quad \quad \quad z = 0.907 \quad \left. \begin{array}{l} \\ \\ \end{array} \right\} \\
 \quad \quad \quad \text{mean value of } y = 0.081 \pm 0.009
 \end{array}$$

The two y -values for O_2 give an approximate estimation of the accuracy of the oxygen parameters, which is perhaps not larger than ± 0.02 and equivalent to about $\pm 0.15 \text{ \AA}$.

These oxygen positions give a distorted octahedral coordination around antimony with Sb-O distances of about 2.0 \AA , indicating that they seem to be quite probable.

THE LITHIUM POSITIONS

As the contribution from the lithium atoms to the $F(hkl)$ values is very low, we had to locate them from space considerations. The sum of the ionic radii are Li-O 2.10 kX (Goldschmidt) 2.00 kX (Pauling). The minimum distance Li-Sb was assumed to be the sum of the atomic radii, which are 3.08 kX when the coordination number is 8 (Goldschmidt). (Values of radii are taken from Internationale Tabellen⁸.)

Spheres of appropriate radii, inside of which no lithium centres could occur, were drawn around the oxygen and antimony centres. Cuts were made parallel with the xy -plane for $0 \leq z \leq 0.5$. As the four lithium atoms ought to be situated in one of the fourfold positions, it was easily found that it must be $4(c)$ with approximately $y = 0.75$. In this position lithium is surrounded by six oxygen atoms 2 from O_1 and 4 from O_2 in a distorted octahedron. The more exact y -parameter was calculated assuming lithium equidistant from the four nearest O_2 atoms. This gave $y = 0.730$.

The atomic coordinates of $LiSbO_3$ are (the equivalent positions are given on p. 1023):

Sb in $4(c)$	$y = 0.097_1$		
Li in $4(c)$	$y = 0.73_0$		
O_1 in $4(d)$	$z = 0.44_4$		
O_2 in $8(e)$	$x = 0.20_9$	$y = 0.08_1$	$z = 0.90_7$

Table 2. Calculated and observed F -values of $hk0$ for a Weissenberg photograph of $LiSbO_3$. $CuK\alpha$ -radiation.

k	$0k0$		$1k0$		$2k0$		$3k0$		$4k0$		$5k0$		$6k0$	
	F_c	F_o	F_c	F_o	F_c	F_o	F_c	F_o	F_c	F_o	F_c	F_o	F_c	F_o
0			0		104	111	0		185	187	0		143	133
1	0		134	87	0		104	105	0		159	186	0	
2	45	62	0		47	48	0		39	57	0		57	97
3	0		-35	28	0		-36	33	0		-35	30		
4	-105	125	0		-111	108	0		-101	114	0			
5	0		-162	154	0		-138	124	0		-173	152		
6	-174	168	0		-86	88	0		-150	176				
7	0		-66	68	0		-44	67						
8	17		0		19		0							
9	0		99	84	0		102	106						
10	143	117	0		150	117								

Table 3. Calculated and observed F -values of $hk1$ for a Weissenberg photograph of $LiSbO_3$. $CuK\alpha$ -radiation.

k	0k1		1k1		2k1		3k1		4k1		5k1		6k1	
	F_c	F_o	F_c	F_o	F_c	F_o	F_c	F_o	F_c	F_o	F_c	F_o	F_c	F_o
0			0		0		0		0		0		0	
1			2		-63	48	-4		-62	56	6		-59	76
2	0		-98	103	-10		-108	154	12		-93	113	-12	
3	-82	107	-35	39	-121	103	25		-95	106	-12			
4	0		-66	63	-9		-77	77	12		-62	78		
5	-6	14	0		-12		-3		-10		5			
6	0		54	32	-1		53	28	1		55	39		
7	95	76	-3		97	81	4		98	103				
8	0		104	82	5		111	124						
9	64	54	21		87	100								
10	0		17	17										

Table 4. Calculated and observed F -values of $0kl$ for a Weissenberg photograph of $LiSbO_3$. $CuK\alpha$ -radiation.

k	0k0		0k1		0k2		0k3		0k4		0k5		0k6	
	F_c	F_o	F_c	F_o	F_c	F_o	F_c	F_o	F_c	F_o	F_c	F_o	F_c	F_o
0			0		-71	95	0		77	92	0		-100	98
1	0		-41	62	0		45	54	0		-50	44	0	
2	26	35	0		-28	33	0		18	36	0		-29	28
3	0		-71	66	0		110	96	0		-74	69	0	
4	-59	70	0		64	55	0		-54	55	0		64	49
5	0		-3		0		6		0		-10			
6	-97	109	0		58	57	0		-62	53	0			
7	0		72	72	0		-73	60	0		79	70		
8	14		0		-14		0		23					
9	0		51	49	0		-79	71						
10	-79	69	0		-84	65								

Table 5. Calculated and observed F -values of $1kl$ for a Weissenberg photograph of $LiSbO_3$. $CuK\alpha$ -radiation.

k	1k0		1k1		1k2		1k3		1k4		1k5		1k6	
	F_c	F_o	F_c	F_o	F_c	F_o	F_c	F_o	F_c	F_o	F_c	F_o	F_c	F_o
0					23	37	0		17	22	0		1	
1					-58	63	6		57	66	4		-61	58
2	0		-65	76	3		71	78	-1		-67	64	-6	
3	-18	10	-24	28	18	14	-1		-18	14	12		18	
4	0		-45	46	-1		50	43	2		-46	50	6	
5	-76	84	1		70	61	5		-70	66	5			
6	0		36	32	-17		-36	20	-14		36	32		
7	-34	43	-2		30	32	-4		-30	38				
8	0		70	76	-3		-73	59						
9	50	47	14		-50	40	1							

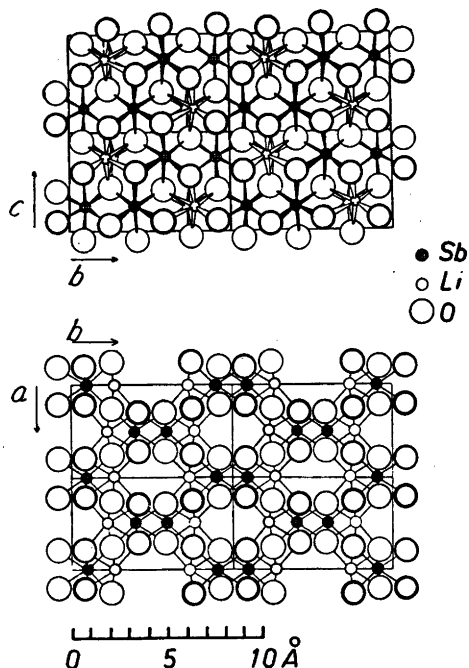


Fig. 5. Projections of the structure of LiSbO_3 . In each projection four adjacent unit cells. The lines between the circles represent metal-oxygen contacts. The difference in thickness of the circles indicate the difference in height above the plane of projection.

with an accuracy of about ± 0.001 for Sb and ± 0.02 for O. As the Li-parameter is dependent of the O-parameters, its accuracy apparently is approximately the same as for O.

F -values were then calculated as

$$F_{\text{calc}} = \text{const} \left[A_{\text{Sb}} + \frac{f_{\text{O}}}{f_{\text{Sb}}} \sum_1^2 A_{\text{O}_i} + \frac{f_{\text{Li}}}{f_{\text{Sb}}} A_{\text{Li}} \right]$$

where f_{Sb} , f_{O} and f_{Li} are the atomic scattering factors for Sb, O and Li with due consideration taken to their variation with θ . The calculated and observed F -values are given in tables 2 (for $hk0$), 3 (for $hk1$), 4 (for $0kl$) and 5 (for $1kl$).

The reliability factor $R = \frac{\sum |F_o| - |F_c|}{\sum |F_o|}$ varies for these zones between 0.13 and 0.19 when only the observed reflections have been included and between 0.15 and 0.28 when the absent reflections have been included assuming F_o zero.

DISCUSSION OF THE STRUCTURE

Projections of the structure of LiSbO_3 are given in Fig. 5, where four adjacent unit cells are given, so that the building principle of the structure will be more obvious. The interatomic distances are given in Table 6.

We can see that the oxygen atoms form a distorted hexagonal close packing, with antimony and lithium occupying some of the octahedral holes. The

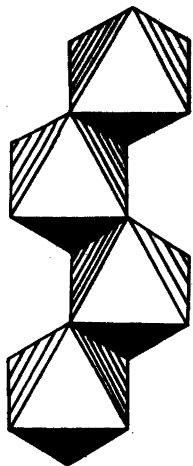


Fig. 6. A string of staggered octahedra with shared edges, representing the antimony oxygen configuration in LiSbO_3 . The octahedra are idealized as regular polyhedra.

shortest O—O distance is 2.6_1 \AA , which seems quite reasonable. The distances between antimony and the six nearest oxygen atoms vary between 2.0_0 and 2.0_5 \AA , which is in agreement with distances reported from other antimonates. In this investigation, however, these distances are probably not accurate to more than $\pm 0.2\text{--}0.3 \text{ \AA}$. Each oxygen octahedron around antimony shares two edges with two other antimony octahedra, thus forming a string of staggered octahedra (Fig. 6) along the *c*-axis, which coincides with the needle axis of the crystal. This type of string is also found in MnSb_2O_8 (Brandt⁹), which is isomorphous with the mineral columbite (Strudivant¹⁰). The strings are coupled together in a way different from columbite by sharing corners. The

Table 6. Interatomic distances in LiSbO_3 . Å units. The numerals $2 \times$ and $4 \times$ before a distance means that it occurs two or four times.

	Distorted Sb-O octahedron			
Sb-6O	$2 \times 2.0_0$	$2 \times 2.0_4$	$2 \times 2.0_5$	
O—O edges	2.6_1	$2 \times 2.6_4$	$2 \times 2.7_7$	$2 \times 2.8_8$
	$2 \times 2.9_9$	$2 \times 3.1_3$	3.1_6	
O—O diagonals	$2 \times 4.0_2$	4.0_5		
	Distorted Li-O octahedron			
Li-6O	$2 \times 2.0_1$	$4 \times 2.0_7$		
O—O edges	2.6_1	$2 \times 2.7_7$	$4 \times 2.8_0$	$2 \times 2.8_8$
	$2 \times 3.1_0$	3.2_3		
O—O diagonals	4.0_0	4.1_0	4.1_1	
$\text{O}_1\text{-2Sb (Sb-2O}_1)$	$2 \times 2.0_4$			
$\text{O}_1\text{-2Li (Li-2O}_1)$	$2 \times 2.0_1$			
$\text{O}_2\text{-2Sb (Sb-4O}_2)$	2.0_0	2.0_5	$(2 \times 2.0_0, 2 \times 2.0_5)$	
$\text{O}_2\text{-2Li (Li-4O}_2)$	$2 \times 2.0_7$	$(4 \times 2.0_7)$		
Sb-2Sb within a string of octahedra	3.06			
Sb-2Sb (4Sb) between two strings	$2 \times 3.56 (2 \times 4.89)$			
Li-2Li within a string of octahedra	2.4			
Li-Li (4Li) between two strings	$4.6_3 (2 \times 5.1_5, 5.2_6)$			
Sb-3Li (9Li) [Li-3Sb(9Sb)]	$2 \times 2.9_7, 3.1_1 (2 \times 3.6_3, 4 \times 3.7_3)$			

oxygen O_2 forms the edges, which are shared within the string and the oxygen O_1 forms the corners, which are shared amongst the strings.

The distances between lithium and the six nearest oxygen atoms vary between 2.0₁ and 2.0₇ Å, which is in agreement with the sum of the ionic radii. One oxygen octahedron around lithium shares faces with two other lithium octahedra, thus forming a string along the *a*-axis. The Li—Li distance of 2.4₇ Å within the string seems very short compared with the atomic diameter of 3.04 Å, but is in good agreement with the Li—Li distance of 2.48 Å in $LiOH \cdot H_2O$ (Pepinsky¹¹). The lithium octahedron also shares one edge with an adjacent antimony octahedron and two corners with other antimony octahedra. It therefore seems more appropriate to describe $LiSbO_3$ as a double lithium antimony(V) oxide than a lithium antimonate. The structure of $LiSbO_3$ seems to be different from other known double oxides of the type $M'SbO_3$.

SUMMARY

$LiSbO_3$ has been investigated by X-ray crystallographic methods. It is orthorhombic, space group No. 52, $Pnca-D_{2h}^6$ with 4 formula units in the unit cell. The cell dimensions are given on p. 1022, the atomic coordinates on p. 1027, and the interatomic distances in Table 6. Antimony and oxygen positions have been determined from bounded projections of the electron density. Lithium positions have been determined from space considerations.

Projections of the structure are given in Fig. 5. The oxygen atoms form a distorted hexagonal close packing with antimony and lithium in some of the octahedral holes. Antimony and oxygen form a staggered chain of octahedra (Fig. 6) along the *c*-axis, which coincides with the needle axis of the crystal. The structure of $LiSbO_3$ seems to be different from other known double oxides of the type $M'SbO_3$.

It seems more appropriate to describe $LiSbO_3$ as a double lithium antimony (V)oxide than a lithium antimonate.

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