

structure determination of $B_{48}B_2C_2$ and $B_{48}B_2N_2$ (Ploog, Schmidt, Amberger, Will & Kossobutzki, 1972; Ploog, 1974). It can be explained by the fact that the 2(b) sites are not fully occupied in these crystals, all of which were prepared as thermodynamically unstable compounds far below their melting point by chemical vapour deposition. The bond distance of 1.67 Å between the carbon atom in 2(b) and the icosahedral boron atom B(2) is comparable to that found in $B_{48}B_2C_2$ (1.63 Å) and in the rhombohedral boron carbide $B_{13}C_2$ (1.61 Å, Ploog, Kossobutzki & Will, 1973).

Since conclusions regarding transfer of charge, as derived from even the best X-ray data, must be regarded with reserve, the interpretation of the data of Amberger & Polborn on the basis of an occupation of position 2(b) by carbon atoms is much more straightforward. Thus, the tetragonal icosahedral framework of this boron-rich Ti boride again is stabilized by the atoms in 2(b), namely carbon, and the Ti atoms in 2(a) merely fill interstitials. The structure chemical composition of this tetragonal Ti boride, derived from the refinement procedure, is then given by $(B_{12})_4C_{1.68}Ti_{1.78}$.

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Crystal structure of β -Li₂SO₄. By A. G. NORD, *Departments of Inorganic and Structural Chemistry, Arrhenius Laboratory, University of Stockholm, S-104 05 Stockholm 50, Sweden*

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The crystal structure of β -Li₂SO₄ ($P2_1/a$) has been refined to $R=0.039$ on the basis of 2027 independent diffractometer-measured reflexions corrected for absorption. The sulphate tetrahedra are nearly regular with S-O distances in the region 1.470 (1)–1.478 (1) Å.

The present work was undertaken as part of a programme to determine the dimensions of the sulphate ion as accurately as possible (*e.g.* Nord, 1974). The results here are almost consistent with an independent investigation by Alcock, Evans & Jenkins (1973). Since they reported an R value of 0.081 and approximately four times greater values for the estimated standard deviations of the atomic parameters, interatomic distances, and angles, the publication of the present study seems worthwhile.

Crystals of β -Li₂SO₄ were grown from the melt. The lattice parameters, based on a refinement of 32 single-indexed reflexions measured from a Guinier powder photograph (Cu $K\alpha_1$ radiation, 25°C), are: $a=8.239$ (1), $b=4.954$ (1), $c=8.474$ (1) Å, $\beta=107.98$ (3)°, $V=328.9$ Å³, $Z=4$, $d_{calc}=2.220$ g cm⁻³. The space group is $P2_1/a$ (No. 14). A small irregularly shaped single crystal, covered with a thin layer of shellac to prevent hygroscopic decomposition, was used for the collection of single-crystal data on a Siemens AED diffractometer (Mo $K\alpha$ radiation, graphite monochromator,

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θ -2 θ scan up to $\theta=50^\circ$). Corrections for Lp and absorption effects [$\mu(\text{Mo } K\alpha)=7.92$ cm⁻¹] were applied to the 2027 independent non-extinct reflexions.

The final refinements were carried out on an IBM 360/75 computer by means of the full-matrix least-squares program LALS. Hughes's (1941) weighting function was used. However, zero weights were assigned to 320 reflexions for which $\sigma(I)/I > \frac{1}{2}$; these were nevertheless included in all other calculations. The atomic scattering factors for Li⁺, S⁰, and O⁻ were taken from *International Tables for X-ray Crystallography* (1968). The final atomic parameters are given in Table 1. $R(\text{unweighted})=0.039$. It is evident upon inspection of the structure factor list* that extinction effects are quite negligible.

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31494 (5 pp.). Copies may be obtained from The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. Atomic coordinates ($\times 10^4$) and temperature factors ($\times 10^4$) for β -Li₂SO₄ ($P2_1/a$)

General equivalent positions 4(e): $\pm(x, y, z)$; $\pm(\frac{1}{2}+x, \frac{1}{2}-y, z)$. Estimated standard deviations are given in parentheses. The anisotropic temperature factors are of the form $\exp[-(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + B_{12}hk + B_{13}hl + B_{23}kl)]$.

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Li(1)	1986 (3)	5693 (4)	3806 (3)	58 (3)	117 (6)	47 (3)	-7 (4)	36 (4)	3 (3)
Li(2)	4356 (3)	5755 (4)	1287 (3)	58 (3)	151 (6)	49 (3)	-4 (4)	45 (4)	-32 (4)
S	3110 (1)	640 (1)	2516 (1)	31 (1)	71 (1)	28 (1)	-5 (1)	19 (1)	-5 (1)
O(1)	4636 (1)	-644 (2)	2299 (1)	37 (2)	121 (2)	60 (2)	19 (2)	34 (2)	-36 (2)
O(2)	1636 (1)	58 (2)	1053 (1)	37 (2)	201 (2)	34 (2)	-34 (2)	2 (2)	-3 (2)
O(3)	2747 (1)	-507 (2)	3981 (1)	78 (2)	117 (2)	33 (2)	-45 (2)	51 (2)	-2 (2)
O(4)	3389 (1)	3572 (2)	2739 (1)	64 (2)	69 (2)	63 (2)	-11 (2)	66 (2)	-11 (2)

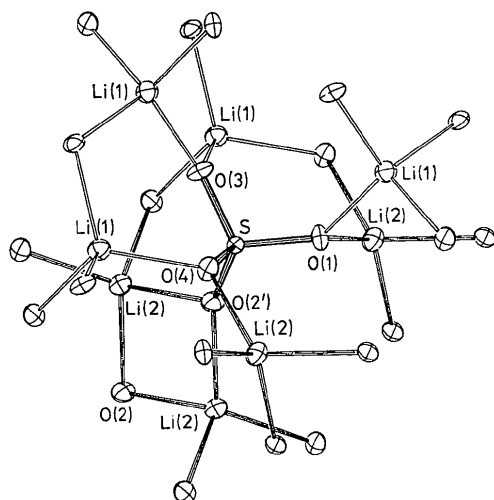


Fig. 1. An ORTEP diagram of part of the β -Li₂SO₄ structure, with one sulphate ion connected to eight LiO₄ tetrahedra. The edge shared between two LiO₄ tetrahedra, O(2)–O(2'), is 2.737 (2) Å. The angle O(2)–Li(2)–O(2') is 88.6 (1)°.

The crystal structure is built up of SO₄ and LiO₄ tetrahedra. The SO₄ tetrahedra are almost regular with the S–O distances in the range 1.470 (1) to 1.478 (1) Å. There are two different kinds of LiO₄ tetrahedra. One of these, around Li(1), is almost regular with O–Li(1)–O angles within 106.5 (1)–115.2 (1)°; the other, around Li(2), is somewhat more distorted with O–Li(2)–O angles within 88.6 (1)–124.6 (1)°. All the LiO₄ tetrahedra are more nearly regular than suggested by Albright (1932), who deduced the lithium atom positions solely from spatial considerations. Some interatomic distances and angles are given in Table 2.

Each oxygen in the structure is linked to one sulphur and two lithium atoms, *i.e.* each SO₄ tetrahedron is connected with eight LiO₄ tetrahedra. Mostly the tetrahedra share corners; however, two adjacent Li(2)O₄ tetrahedra share an edge as shown in Fig. 1. The edge-sharing O–O distance is only 2.737 (2) Å, in contrast with 3.105 (1)–3.467 (1) Å for the other O–O distances within the LiO₄ tetrahedra. The angle O(2)–Li(2)–O(2') (*cf.* Fig. 1) is 88.6 (1)°. Further details of this work are available from the author.

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Refinement of the 2H GaS β -type. By A. KUHN and A. CHEVY, *Laboratoire de Luminescence II, Université Pierre et Marie Curie, Equipe de recherche associée au CNRS, 4 place Jussieu, 75230 Paris Cedex 05, France* and R. CHEVALIER, *Laboratoire de Minéralogie–Cristallographie, Université Pierre et Marie Curie, Laboratoire associé au CNRS, 4 place Jussieu, 75230 Paris Cedex 05, France*

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GaS β , hexagonal, space group $P6_3/mmc$, $a = 3.587$ (3), $c = 15.492$ (7) Å, $Z = 4$. The mean atomic distances in the layers are: Ga–Ga = 2.447 (9), Ga–S = 2.334 (4) and S–S = 4.599 (18) Å. The mean atomic distances between the layers are: S–S = 3.768 (11) and Ga–S = 4.223 (14) Å. The interlayer S–S distance is more than twice the van der Waals radius.

Introduction

GaS crystallizes only in the 2H β structure, as determined by Hahn (1953), Hahn & Frank (1955) and Terhell & Lieth

Table 2. Interatomic distances (Å) and angles (°) in the β -Li₂SO₄ structure

The distances marked with an asterisk have been corrected for thermal vibrations according to the riding-motion model (Busing & Levy, 1964).

	This work	This work	Alcock, Evans & Jenkins (1973)
S–O(1)	1.470 (1)	1.474*	1.468 (4)
–O(2)	1.471 (1)	1.476*	1.479 (3)
–O(3)	1.478 (1)	1.482*	1.470 (4)
–O(4)	1.473 (1)	1.476*	1.471 (5)
Average	1.473	1.477*	1.472
Li(1)–O(1) ($x - \frac{1}{2}, \frac{1}{2} - y, z$)		1.963 (3)	1.98 (1)
–O(3) ($x, 1 + y, z$)		1.975 (2)	1.98 (1)
–O(3) ($\frac{1}{2} - x, \frac{1}{2} + y, 1 - z$)		1.914 (3)	1.91 (1)
–O(4) (x, y, z)		1.975 (2)	1.94 (1)
Average		1.957	1.95
Li(2)–O(1) ($x, 1 + y, z$)		1.962 (2)	1.94 (1)
–O(2) ($\frac{1}{2} - x, \frac{1}{2} + y, -z$)		1.929 (3)	1.93 (1)
–O(2) ($\frac{1}{2} + x, \frac{1}{2} - y, z$)		1.990 (2)	1.98 (1)
–O(4) (x, y, z)		1.980 (2)	1.99 (1)
Average		1.965	1.96
O(1)–S–O(2)		109.0 (1)	108.8 (2)
O(1)–S–O(3)		110.0 (1)	109.6 (2)
O(1)–S–O(4)		109.7 (1)	110.4 (2)
O(2)–S–O(3)		107.7 (1)	108.0 (2)
O(2)–S–O(4)		110.9 (1)	110.7 (2)
O(3)–S–O(4)		109.6 (1)	109.5 (3)

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(1971). Because an exact knowledge of the interatomic distances is important for band-structure calculations (Bourdon, 1976), we thought that a structure determination of higher accuracy would be of interest.