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Lithium Sulphate – a Redetermination

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Abstract. Li_2SO_4, monoclinic, $P2_1/c$, $a=8\cdot45$ (1), $b=4\cdot95$ (1), $c=8\cdot21$ (1) Å, $\beta=107\cdot5$ (2)°, $Z=4$, $D_x=2\cdot23$ g cm^{-3}. Crystals were grown from a melt of Li_2SO_4. The redetermination confirms the work of Albright [*Z. Kristallogr.* (1932). **84**, 150–158]. The lithium atom is tetrahedrally coordinated with a Li–O distance of 1·96 Å (mean) and there is a S–O distance of 1·472 Å (mean) in the SO_4^{2-} tetrahedra.

Introduction. The crystals were grown by heating $Li_2SO_4 . H_2O$ in a platinum crucible to remove water and then to fusion. The crystals obtained on cooling are hygroscopic and were protected by a coating of shellac; they are generally of poor quality.

The intensities and cell dimensions were obtained with a Stoe–Weissenberg diffractometer (graphite monochromator, ω-scan, no attenuators, Mo $K\alpha$, $\lambda=0\cdot7107$ Å; data collected for layers $h0l$ to $h5l$ to a maximum 2θ value of 90°). Because of difficulty of finding suitable crystals, no crossing data were collected and, consequently, the anisotropic temperature factors are not known absolutely. Corrections for Lorentz and polarization factors were applied, but none were made for absorption. 1319 reflexions were considered to be observed with $I/\sigma(I)>3\cdot0$.

Systematic absences $h0l$, $l\neq2n$ and $0k0$, $k\neq2n$ confirm space group $P2_1/c$. [The a and c axes of Albright (1932) have been exchanged to give the standard space-group setting.] Unit-cell constants were obtained from the reflecting position of high-angle reflexions on the diffractometer and their errors estimated from the accuracy of measurement.

Trial refinement of the S and O positions of Albright gave an R index of 0·17 and a difference Fourier synthesis showed the lithium atoms, also fairly close to Albright's positions. Final least-squares refinement gave $R=0\cdot081$, with a weighting scheme of the form:

$$\sigma^2(F)=(6200+F+0\cdot0011F^2)/40$$

to compensate for the slight F and F^2 dependence of the average Δ^2 *versus* F curve. Atomic coordinates and temperature factors are given in Table 1 together with those of Albright (1932), and bond lengths and angles are in Table 2. A list of structure factors is obtainable as Supplementary Publication No. SUP 30026 (7 pp).*

Discussion. This structure determination was undertaken as part of a study of lattice energies of sulphate crystals (*e.g.* Jenkins, 1972), in which anomalous results were found for Li_2SO_4 that might have been due to incorrect atomic positions. The structure was originally determined (Albright, 1932) by trial-and-error methods without refinement, but this work shows that the positions were broadly correct. The unit-cell lengths agree well with those reported by Swanson, McMurdie, Morris & Evans (1968), namely $a=8\cdot474$ (1), $b=4\cdot9533$ (3), $c=8\cdot2414$ (4) Å and $\beta=107\cdot98$ (5)°. In the present determination S–O is 1·472 Å (mean) and Li–O is 1·96 Å (mean) which may be compared with the corresponding S–O distances 1·402 Å (Na_2SO_4), 1·491 Å (K_2SO_4), 1·502 Å (Rb_2SO_4) and 1·493 Å (Cs_2SO_4), and corresponding M–O distances 2·32 Å (M = Na), 2·66 Å (M = K), 2·88 Å (M = Rb) and 3·04 Å (M = Cs) in other sulphates (data from Wyckoff, 1966). The latter figures suggest an 'ionic' radius for the oxygen of a sulphate group of 1·34 Å (mean), by subtracting standard ionic radii (Shannon & Prewitt, 1969) for the alkali metals from these S–O distances. This is al-

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Table 1. *Atomic coordinates and temperature factors*

Standard errors in the last figures quoted as given by the final round of least-squares analysis are shown in parentheses. The temperature factors ($\times 10^4$) were calculated using the expression

$$\exp\left[-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}+U_{33}l^2c^{*2}+2U_{12}hka^*b^*+2U_{23}klb^*c^*+2U_{13}hla^*c^*)\right].$$

Present work ($\times 10^4$) Albright (1932) ($\times 10^3$)

	x	y	z	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Li(1)	3815 (11)	5689 (23)	1993 (11)	375	582	205	20 (4)	18 (6)	15 (3)	3 (4)	6 (3)	1 (4)
Li(2)	1287 (12)	5776 (23)	4357 (11)	125	582	455	22 (4)	20 (6)	16 (3)	−4 (4)	7 (3)	−1 (4)
S	2517 (1)	642 (3)	3111 (1)	250	61	319	11·2 (3)	8·7*	6·7 (3)	−0·5 (4)	2·3 (3)	−0·1 (4)
O(1)	2291 (4)	− 656 (9)	4634 (4)	250	− 42	492	23 (1)	15 (2)	9 (1)	−6 (1)	−5 (1)	−2 (1)
O(2)	1049 (4)	65 (9)	1634 (4)	99	− 42	186	15 (1)	30 (3)	9 (1)	−1 (1)	0 (1)	−3 (1)
O(3)	3979 (4)	− 509 (9)	2750 (4)	401	− 42	280	15 (1)	20 (2)	20 (1)	0 (1)	7 (1)	−5 (1)
O(4)	2743 (5)	3573 (9)	3380 (5)	250	367	319	25 (2)	8 (2)	15 (1)	3 (1)	9 (1)	0 (1)

* Not refined to remove scale factor/temperature factor degeneracy.

Table 2. *Interatomic distances and angles*

Standard errors in the last figures quoted in parentheses. For shifted atoms the transformation applies to the second atom.

S–O(1)	1·468 (4) Å	O(1)–S–O(2)	108·8 (2)°
S–O(2)	1·479 (3)	O(1)–S–O(3)	109·6 (2)
S–O(3)	1·470 (4)	O(2)–S–O(4)	110·4 (2)
S–O(4)	1·471 (5)	O(3)–S–O(3)	108·0 (2)
		O(2)–S–O(4)	110·7 (2)
		O(3)–S–O(4)	109·5 (3)
Li(1)–O(1′) (x, ½−y, z−½)	1·98 (1)	O(1′) –Li–O(3′)	106·3 (5)
		O(1′) –Li–O(3″)	115·7 (5)
		O(1′) –Li–O(4)	106·6 (5)
Li(1)–O(3′) (x, 1+y, z)	1·98 (1)	O(3′) –Li–O(3″)	106·7 (5)
		O(3′) –Li–O(4)	109·1 (5)
Li(1)–O(3″) (1−x, ½+y, ½−z)	1·91 (1)	O(3″)–Li–O(4)	112·2 (5)
Li(1)–O(4)	1·96 (1)		
Li(2)–O(1″) (x, 1+y, z)	1·94 (1)	O(1″)–Li–O(2′)	104·2 (5)
		O(1″)–Li–O(2″)	124·8 (6)
Li(2)–O(2′) (x, ½−y, ½+z)	1·98 (1)	O(1″)–Li–O(4)	104·2 (5)
		O(2′) –Li–O(2″)	89·8 (5)
Li(2)–O(2″) (−x, ½+y, ½−z)	1·93 (1)	O(2′) –Li–O(4)	121·1 (5)
		O(2″)–Li–O(4)	114·2 (5)
Li(2)–O(4)	1·99 (1)		

most indistinguishable from the standard ionic radius for O^{2-} of 1·35–1·38 Å depending on coordination (Shannon & Prewitt, 1969).

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