Cu–O(2') angle expected for a tetragonal pyramidal Cu coordination and the 120° Cu–O(2)–Cu' angle expected for a normal sp^2 hybridized oxygen coordination. These angles cause the O(2)–O(2') distance to be much less than the Cu–Cu' distance across the bridge, 2·38 (1) and 3·34 (3) Å respectively. Distances and angles involving the chloride ions are distorted by the geometrical requirements of the bridge (Sager, Williams & Watson, 1967). Those for the pyridine ring appear normal. The DMSO molecule is coordinated through oxygen to the Copper(II) ion as expected (Cotton, Francis & Horrocks, 1960). The resulting Cu–O(1) distance of 2·279 (6) Å is normal for apical coordination. This coordination causes a slight lengthening of the S–O bond in the DMSO group to 1·546 (6) Å over that found in free DMSO (Thomas, Shoemaker & Eriks, 1966), 1·531 (8) Å and is consistent with a slight decrease in $d \rightarrow p\pi$ bonding from sulfur to oxygen caused by coordination (Bennett, Cotton, Weaver, Williams & Watson, 1967).

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Refinement of the Crystal Structure of Lithium Chromate

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Abstract. Li_2CrO_4 crystallizes with the phenacite structure (type $S1_3$), rhombohedral, $R\bar{3}$ (C_{3i}^2), $a = 14·005(10)$, $c = 9·405(5)$ Å, $Z = 18$ (hexagonal cell), $D_m = 2·426$, $D_c = 2·427$ g cm^{-3}. The sample was prepared by heating $Li_2Cr_2O_7(H_2O)_2$ and recrystallizing from the melt.

Introduction. A cylindrical crystal 0·15 mm in diameter and 0·20 mm long was sealed inside a quartz capillary. The measured density was taken from Hartford, Costa & Moore (1952). The unit-cell parameters and intensities were measured on a Syntex $P\bar{1}$ diffractometer with graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0·71069$ Å) and a θ–2θ scan. No systematic absences were found for the rhombohedral setting. A total of 2751 intensity measurements in the region $2\theta \le 55°$ were reduced to 825 by averaging reflexions related by the threefold axis. Of these, 152 were less than 3σ, the standard error derived from counting statistics. A cylindrical absorption correction ($\mu = 3·01$ mm^{-1}) was applied and the intensities were reduced to structure factors by applying Lorentz and polarization correc-

tions. We assumed that Li_2CrO_4 has the phenacite structure (Martin & Durif, 1964) which we refined by least-squares calculations using the local program *CUDLS* to give R_1 (all reflexions) $= 0·075$ and R_2 (weighted) $= 0·054$. In the final round an extinction correction, $F^* = F[1 + 0·12 \times 10^{-6}\beta(2\theta)F^2]^{-1/2}$ (Larson, 1967), was applied and the reflexions were weighted by the function $\omega = (151·2 - 2·18F + 0·00844F^2)^{-1/2}$ except that for the 97 reflexions for which $I_o < 3\sigma$ and $F_c < F_o$, ω was set equal to zero. The atomic parameters are given in Table 1.†

Discussion. The phenacite structure ($S1_3$ type) was first proposed for Li_2CrO_4 by Martin & Durif (1964) on the basis of powder diagrams. Their cell constants agree with ours given above.

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† A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31045 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Atomic coordinates (hexagonal setting)* ($\times 10^4$) *and anisotropic thermal parameters* ($\times 10^3$)

The expression used for the temperature factors is: $\exp[-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}+U_{33}l^2c^{*2}+2U_{12}hka^*b^*+2U_{13}hla^*c^*+2U_{23}klb^*c^*)]$. Figures in parentheses are the standard errors in the last decimal place quoted.

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cr	4563 (1)	3194 (1)	5838 (1)	8·0 (6)	5·8 (6)	3·3 (4)	4·1 (5)	−0·5 (4)	0·1 (4)
O(1)	5618 (3)	4462 (4)	5812 (5)	12 (2)	12 (2)	13 (2)	1 (2)	1 (2)	3 (2)
O(2)	3424 (4)	3276 (4)	5856 (5)	10 (2)	13 (2)	18 (2)	7 (2)	1 (2)	0 (2)
O(3)	4627 (4)	2576 (4)	7303 (5)	22 (3)	15 (2)	9 (2)	12 (2)	3 (2)	6 (2)
O(4)	4573 (4)	2511 (4)	4405 (5)	25 (3)	18 (2)	8 (2)	14 (2)	0 (3)	−2 (2)
Li(1)	4581 (12)	3149 (12)	9196 (12)	25 (7)	18 (7)	20 (6)	10 (6)	2 (6)	8 (5)
Li(2)	4541 (11)	3135 (10)	2522 (10)	17 (6)	15 (6)	13 (5)	2 (5)	−3 (5)	−4 (4)

Table 2. *Bond lengths* (Å) *and angles* (°) *in* Li_2CrO_4

			O(2)	O(3)	O(4)
Cr–O(1)	1·647 (4)	1·655*	107·6 (3)	108·8 (2)	110·4 (2)
Cr–O(2)	1·656 (7)	1·662*		109·5 (3)	109·6 (3)
Cr–O(3)	1·653 (5)	1·660*			110·9 (3)
Cr–O(4)	1·657 (5)	1·665*			
av. (Cr–O)	1·653	1·660*			

		O(2)	O(3)	O(3′)
Li(1)–O(1)	1·94 (2)	111 (1)	119 (1)	109 (1)
Li(1)–O(2)	1·94 (2)		108 (1)	103 (1)
Li(1)–O(3)	1·97 (1)			107 (1)
Li(1)–O(3′)	1·97 (1)			

		O(2)	O(4)	O(4′)
Li(2)–O(1)	1·95 (1)	108 (1)	106 (1)	117 (1)
Li(2)–O(2)	1·98 (2)		110 (1)	106 (1)
Li(2)–O(4)	1·97 (1)			111 (1)
Li(2)–O(4′)	1·98 (1)			
av. (Li–O)	1·96			

* Corrected for riding motion.

Although the $S1_3$ structure has been known for a long time (Bragg, 1926; Zachariasen, 1926) and some 18 compounds are known to crystallize with it (*Crystal Data Determinative Tables*, 1973), only one accurate determination of an $S1_3$ type crystal, Li_2BeF_4, has been reported (Burns & Gordon, 1966).

The bond lengths and angles in Li_2CrO_4 are given in Table 2. The thermal parameters suggest that the CrO_4 group is librating with an r.m.s. amplitude of about $3\frac{1}{2}°$, very similar to the libration of the BeF_4 group observed in Li_2BeF_4. The individual bond lengths do not differ significantly from the average values of Li–O = 1·96, Cr–O = 1·653 Å although the angles deviate by as much as 6° from tetrahedral. The Cr–O distances are close to the weighted average [1·650(7) Å] of all Cr–O distances in alkali chromates-(VI) (Löfgren, 1974). The O atoms each bond to one Cr and two Li atoms in a plane, the average Li–O–Li angle being 110° and Cr–O–Li being 125°.

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