SHORT-FORMAT PAPERS

The Structure of the Triple Cation Salt Li₂NaK(SO₄)₂

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Abstract. Dilithium sodium potassium disulfate, M_r = 268·09, orthorhombic, $P2_12_12_1$, $a = 4·905$ (4), $b = 7·753$ (6), $c = 18·923$ (16) Å, $V = 720$ (1) Å³, $Z = 4$, $D_x = 2·473$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0·71069$ Å, $\mu = 13·00$ cm⁻¹, $F(000) = 528$, $T = 138$ (2) K, $R = 0·022$ for 845 unique, observed reflections. Cell constants at 298 (5) K were also measured: $a = 4·946$ (2), $b = 7·814$ (2), $c = 19·019$ (16) Å, $V = 735·0$ (7) Å³. All atoms occupy general positions in the cell and show no evidence of disorder. Sulfate anions are normal: S—O lengths average 1·476 (6) Å. The lithium cations are surrounded by an approximate tetrahedral arrangement of oxygen atoms: Li—O range 1·90–1·99 Å, O—Li—O range 99–120°. The Na⁺ and K⁺ ions show close contacts to six and eight oxygen atoms respectively but the arrangement is irregular: Na—O range 2·38–2·65 Å, K—O range 2·68–3·04 Å.

Experimental. Li₂NaK(SO₄)₂ was crystallized by Kitahama & Frech (1985) and possesses interesting spectroscopic and conductive properties. Colorless columnar parallelepipeds, 0·18 × 0·18 × 0·25 mm, Nonius CAD-4, LT cell parameters from 62 high-angle reflections (2θ > 18°) measured at ±2θ, RT cell parameters from 44 high-angle reflections measured at ±2θ, θ/2θ scans, Gaussian integration absorption corrections, transmission coefficient range 0·81–0·79, $2·4 \leq 2\theta \leq 53°$, $0 \leq h \leq 6$, $0 \leq k \leq 9$, $0 \leq l \leq 23$, three standard reflections fluctuated 0·4%, 950 total reflections, 882 unique, no averaging, 845 observed with $I_o \geq 2\sigma(I)$, direct-methods *MULTAN*76 (Main, Lessinger, Woolfson, Germain & Declercq, 1976), refinement *via SHELX*76 (Sheldrick, 1976) on *F*'s minimizing $\sum w(F_o - |F_c|)^2$, all atoms anisotropic. For ob-

served reflections $R = 0·022$, $wR = 0·031$, $S = 1·34$, $w = (\sigma_F)^{-2}$. Including weak reflections: $R = 0·024$, $wR = 0·032$. $(\Delta/\sigma)_{max} < 0·01$, $(\Delta\rho)_{max} = 0·37$, $(\Delta\rho)_{min} = -0·39$ e Å⁻³. No correction for extinction. Neutral-atom scattering factors and corrections for anomalous dispersion from *International Tables for X-ray Crystallography* (1974). Table 1† lists atomic coordinates. A view of the cell is shown in Fig. 1.

Related literature. For comparison with other Group Ia sulfates see Alcock, Evans & Jenkins (1973), Zilber, Tordjman & Guitel (1980), Zilber, Durif & Averbuch-Pouchot (1980) and Okada & Ossaka (1980). The

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters*

$$U_{eq} = \tfrac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{eq}(\text{Å}^2)$
K(1)	0·00163 (13)	0·05485 (8)	0·07186 (3)	0·0108 (2)
S(1)	0·49000 (15)	−0·54748 (9)	0·55847 (3)	0·0061 (2)
O(11)	0·1948 (4)	−0·5368 (3)	0·57576 (11)	0·0095 (6)
O(12)	0·5183 (5)	−0·5666 (3)	0·48144 (10)	0·0117 (6)
O(13)	0·6291 (5)	−0·38919 (25)	0·58122 (11)	0·0102 (6)
O(14)	0·6060 (5)	−0·6994 (3)	0·59449 (11)	0·0095 (6)
S(2)	0·96643 (15)	0·46739 (9)	0·79270 (4)	0·0061 (2)
O(21)	0·8674 (5)	0·3098 (3)	0·82857 (10)	0·0107 (6)
O(22)	1·2661 (4)	0·4672 (3)	0·79036 (12)	0·0119 (6)
O(23)	0·8515 (4)	0·4721 (3)	0·72076 (11)	0·0115 (6)
O(24)	0·8680 (5)	0·6206 (3)	0·83158 (10)	0·0096 (6)
Na(1)	0·5149 (3)	0·24714 (15)	0·72572 (6)	0·0116 (3)
Li(1)	0·4650 (11)	−0·1010 (6)	0·36036 (23)	0·011 (1)
Li(2)	0·4941 (12)	0·2550 (6)	0·4103 (3)	0·013 (1)

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$\bullet = Li^+$
$\bullet = Na^+$
$\bullet = K^+$

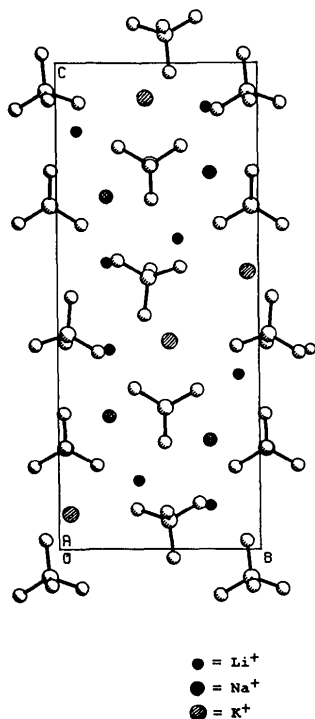Fig. 1. *PLUTO* diagram (Motherwell & Clegg, 1978) of the unit cell of $Li_2NaK(SO_4)_2$ viewed along the *x* crystallographic axis.

crystallization and Raman scattering studies of this compound have already been published (Kitahama & Frech, 1985).

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Tricarbonyl(η^2-cyclenphosphoranido)cobalt(I)

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Abstract. Tricarbonyl(octahydro-2a,4a,6a,8a-tetraaza-8b-phosphapentaleno[6,1-*cd*]pentalene-*N,P*)cobalt(I), [Co(CO)$_3$(C$_8$H$_{16}$N$_4$P)], $M_r = 342 \cdot 18$, orthorhombic, $Pca2_1$, $a = 15 \cdot 599$ (4), $b = 7 \cdot 445$ (2), $c = 12 \cdot 216$ (3) Å, $V = 1418 \cdot 7$ (7) Å^3, $Z = 4$, $D_x = 1 \cdot 602$ g cm^{-3}, λ(Mo $K\alpha$) = $0 \cdot 71069$ Å, μ(Mo $K\alpha$) = $13 \cdot 28$ cm^{-1}, $F(000) = 704$, $T = 295$ K, final $R = 0 \cdot 035$ for 927 observed reflections with $I > 3\sigma(I)$. The cyclenphosphoranido ligand coordinates to the Co atom through P and one of its N atoms with Co–P and Co–N bonds of $2 \cdot 195$ (2) and $2 \cdot 043$ (6) Å, respectively. Three carbonyl groups complete the coordination at the Co atom with Co–C bonds ranging from $1 \cdot 763$ (9) to $1 \cdot 801$ (8) Å.

Experimental. Crystals of the title compound are yellow plates. Unit-cell parameters by least-squares fit of 15 reflections in the range $14 < 2\theta < 24°$. Crystal $0 \cdot 30 \times 0 \cdot 24 \times 0 \cdot 06$ mm, Syntex $P2_1$ automatic diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, $\theta/2\theta$ scanning mode, 1324 independent reflections in the range $3 < 2\theta < 50°$, *hkl* range: $h\ 0\to18$, $k\ 0\to8$, $l\ 0\to14$, 927 observed reflections with $I > 3\sigma(I)$, $\sigma(I)$ from counting statistics; three standard reflections (200, 020 and 002) remeasured after every 100 reflections did not show any significant change in intensity during data collection. Lorentz–polarization correction, no absorption or extinction correction. Co position located by the heavy-atom method; its *z* coordinate was arbitrarily fixed at $\frac{1}{2}$ to specify the origin. Remaining non-H atoms located by difference Fourier methods.

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