

SHORT-FORMAT PAPERS

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Structure of Dilithium Selenate(VI)

BY FRANZ PERTLIK

*Institut für Mineralogie und Kristallographie der Universität Wien, Dr Karl Lueger-Ring 1,
A-1010 Vienna, Austria*

AND ARMIN H. FUITH

Institut für Experimentalphysik der Universität Wien, Strudlhofgasse 4, A-1090 Vienna, Austria

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Abstract. Li_2SeO_4 , $M_r = 156.83$, rhombohedral, $R\bar{3}$, $a = 13.967$ (1), $c = 9.342$ (1) Å, $V = 1578.2$ (1) Å³, $Z = 18$, $D_m = 2.96$ (6), $D_x = 2.970$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 10.2$ mm⁻¹, $F(000) = 1296.0$, room temperature. $R = 0.032$ for 1387 independent observed reflections and 65 variables. Li_2SeO_4 is isostructural with phenakite, Be_2SiO_4 . Single crystals up to 7 mm in length were synthesized under hydrothermal conditions.

Experimental. The compounds $M\text{LiSO}_4$ ($M = \text{K}, \text{Rb}, \text{Cs}, \text{NH}_4$) undergo incommensurate–commensurate phase transitions and ferroelectric as well as ferroelastic phases are known. Similar properties are presumed for the selenate analogues. Therefore the system $\text{NH}_4\text{LiSeO}_4\text{–H}_2\text{O}$ was investigated to synthesize the selenate analogue of $\beta\text{-NH}_4\text{LiSO}_4$ (Dollase, 1969). Preparation was carried out by a modified temperature-gradient method, with the flow of the solution guided by proper baffles (*cf.* Wyslouzil, Schranz, Fuith & Warhanek, 1986; Schranz, Parlinski, Warhanek & Zabinska, 1987). Volume of the Teflon-lined autoclaves: 20 ml; temperature range: 450–500 K; saturation vapour pressure.

Under the given conditions the reaction products are crystals of Li_2SeO_4 , exclusively. The crystals are elongated parallel to [001] up to 7 mm and their diameter is up to 2 mm. To prove their identity with the compound Li_2SeO_4 , prepared by Pistorius (1967), as well as to verify the structure type, a structure determination was performed. D_m was determined by flotation.

Synthetic crystal, $0.08 \times 0.08 \times 0.50$ mm; Stoe AED-2 four-circle diffractometer, graphite-mono-

chromatized Mo $K\alpha$ radiation: lattice parameters from 26 reflections, $2\theta \leq 40^\circ$. Intensity measurements: $2\theta \leq 70^\circ$; $2\theta/\omega$ -scan mode, minimum of 40 steps increased for (α_1, α_2) splitting, step width 0.03° , time per step between 0.5 and 1.5 s. Four standard reflections showed no significant variation of intensities during measuring time. Empirical absorption corrections (four ψ scans: transmission factors from 0.13 to 0.26). 2515 measured reflections ($h: -23 \rightarrow 23, k: 0 \rightarrow 23, l: -15 \rightarrow 0$); $R_{\text{int}} = 0.046$; 1547 unique reflections; $R = 0.032$ for 1387 reflections with $F_o > 2\sigma(F_o)$, and $wR = 0.028$, $w = [\sigma(F_o)]^{-2}$; $R = 0.037$ for all 1547 unique reflections; number of parameters refined: 65; max. $\Delta/\sigma < 10^{-3}$; max. and min. heights in final difference Fourier map: 0.8 and $-1.5 \text{ e } \text{Å}^{-3}$. The value of g for the isotropic secondary extinction (Zachariasen, 1967) is $3.14 (17) \times 10^{-6}$.

All calculations were performed with the program system *STRUCSY* (Fa. Stoe & Cie, Darmstadt). The transformed atomic coordinates for phenakite (Zachariasen, 1971) were used in a starting set of full-matrix least-squares calculations on F with complex scattering functions for neutral atoms (*International Tables for X-ray Crystallography*, 1974). Structure parameters are listed in Table 1,* some important interatomic distances and bond angles are summarized in Table 2.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51304 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement factors ($\text{\AA}^2 \times 10^4$)
$$U_{\text{eq}} = \frac{1}{3}[U_{33} + \frac{2}{3}(U_{11} + U_{22} - U_{12})].$$

	x	y	z	U_{eq}
Li(1)	9797 (4)	1917 (4)	845 (5)	202
Li(2)	9840 (4)	1917 (4)	4141 (5)	197
Se	9854 (1)	1978 (1)	7495 (1)	118
O(1)	1104 (2)	2156 (2)	7504 (2)	185
O(2)	-45 (2)	3196 (1)	7512 (2)	171
O(3)	9189 (2)	1282 (2)	8928 (2)	178
O(4)	9223 (2)	1297 (2)	6046 (2)	179

Table 2. Some interatomic distances (\AA) and bond angles ($^\circ$) with e.s.d.'s in parentheses

Li(1)—O(1)	1.931 (5)	O(1)—O(2)	3.164 (3)	108.4 (2)
O(2)	1.968 (6)	O(3)	3.347 (2)	117.2 (2)
O(3)	1.990 (5)	O(3')	3.131 (3)	106.6 (2)
O(3')	1.975 (6)	O(2)—O(3)	3.139 (3)	104.9 (2)
		O(3')	3.225 (3)	109.7 (2)
		O(3)—O(3')	3.245 (4)	109.9 (2)
Li(2)—O(1)	1.946 (5)	O(1)—O(2)	3.218 (3)	112.1 (2)
O(2)	1.932 (6)	O(4)	3.334 (2)	116.3 (2)
O(4)	1.979 (5)	O(4')	3.163 (3)	108.9 (2)
O(4')	1.942 (6)	O(2)—O(4)	3.117 (3)	105.7 (2)
		O(4')	3.047 (3)	103.7 (2)
		O(4)—O(4')	3.201 (4)	109.4 (2)
Se—O(1)	1.636 (2)	O(1)—O(2)	2.650 (3)	108.2 (1)
O(2)	1.636 (2)	O(3)	2.673 (3)	109.3 (1)
O(3)	1.642 (1)	O(4)	2.654 (3)	108.5 (1)
O(4)	1.636 (1)	O(2)—O(3)	2.680 (3)	109.7 (1)
		O(4)	2.692 (3)	110.8 (1)
		O(3)—O(4)	2.692 (2)	110.4 (1)

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Structure of Tetrakis(trimethylphosphine)cobalt(I) Tetraphenylborate

BY L. C. ANANIAS DE CARVALHO, YOLANDE PÉRÈS, MICHÈLE DARTIGUENAVE* AND YVES DARTIGUENAVE

Laboratoire de Chimie de Coordination du CNRS, 205 route de Narbonne, 31400-Toulouse, France

AND ANDRÉ L. BEAUCHAMP*

Département de Chimie, Université de Montréal C.P. 6128, Succ. A, Montréal, Québec, H3C 3J7, Canada

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Abstract. $[\text{Co}\{\text{P}(\text{CH}_3)_3\}_4]\text{B}(\text{C}_6\text{H}_5)_4$, $M_r = 682.48$, monoclinic, $P2_1/c$, $a = 16.190$ (9), $b = 12.422$ (4), $c = 20.900$ (10) \AA , $\beta = 111.87$ (5) $^\circ$, $V = 3900.7$ \AA^3 , $Z = 4$, $D_x = 1.162$ Mg m^{-3} , $\lambda(\text{Mo K}\alpha) = 0.71069$ \AA , $\mu = 0.63$ mm^{-1} , $F(000) = 1456$, $T = 296$ K, final $R = 0.037$ for 2167 unique observed reflections. The unit cell contains BPh_4^- anions and $[\text{Co}(\text{PMe}_3)_4]^+$ cations. Co has a distorted tetrahedral coordination ($\text{P—Co—P} = 101.1\text{--}125.4^\circ$, $\sigma = 0.1^\circ$), and the Co—P distances range from 2.193 (2) to 2.252 (2) \AA .

* To whom correspondence should be addressed.

Related literature. The crystals of the title compound correspond to Li_2SeO_4 as described by Pistorius (1967) based on a powder diffraction diagram. They are representatives of the phenakite-type structure (cf. Wyckoff, 1965). Recently Zemmann (1986) pointed out a very short interpolyhedral O(2)—O(2) distance of 2.75 \AA in phenakite, Be_2SiO_4 . The analogous distance in Li_2SeO_4 is 3.128 (3) \AA . The reasons are the average cation—oxygen distances in the XO_4 tetrahedra: Be—O (1.645 and 1.646 \AA) is shorter than Li—O (1.966 and 1.950 \AA), and Si—O (1.632 \AA) is shorter than Se—O (1.638 \AA).

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