SHORT-FORMAT PAPERS

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Refinement of the Structure of Boroleucite, $K(BSi_2O_6)$

By Dušan Mikloš, Ľubomír Smrčok, Slavomil Ďurovič, Dalma Gyepesová and Milan Handlovič

Institute of Inorganic Chemistry, Slovak Academy of Sciences, CS – 842 36 Bratislava, ČSFR

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Abstract. Potassium borosilicate, $KBSi_2O_6$, $M_r = 202.09$, cubic, $I\bar{4}3d$, $a = 12.618$ (4) Å, $V = 2009.0$ Å^3, $Z = 16$, $D_x = 2.67$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 14.7$ cm^{-1}, $F(000) = 1600$, $T = 293$ K, $R = 0.038$, $wR = 0.051$ for all 528 independent reflections. The model given by Ihara & Kamei [*Yogyo Kyokai Shi* (1980), **88**, 32–35] and tetrahedral coordination of B atoms, have been confirmed. The structure is similar to that of cubic leucite [Peacor (1968). *Z. Kristallogr.* **127**, 213–224] and consists of a framework of tetrahedra linked into four- and six-membered rings. The K atom is placed in a tunnel along [111]. The refinement of the occupation factors of K, Si and B atoms yields nearly ideal values corresponding to the chemical formula.

Experimental. The title compound was prepared in the powder form and named boroleucite by Voldán (1979). Details of the synthesis of the single crystals used in this work are published elsewhere (Figusch, 1981). A crystal was ground to a sphere of diameter 0.4 mm, and used for data collection on a Syntex $P2_1$ diffractometer. Unit-cell parameters were determined from least squares on 15 reflections; $16.51 < 2\theta < 46.67°$. Intensity measurements were by ω–2θ scans from [2θ(Mo $K\alpha_1$) − 1] to [2θ(Mo $K\alpha_2$) + 1]°, $0 < 2\theta < 77°$, with variable scan speed 4.88–29.30° min^{-1}; $(\sin\theta/\lambda)_{\max} = 0.8772$ Å^{-1}. Two standards (413 and 022) measured after every 100 reflections showed no appreciable trends. 2751 reflections were measured ($R_{\rm int} = 0.013$); 528 were independent (h = 2–22, k = 1–15, l = 1–12) and used to refine the structure. An absorption correction based on ψ scans of seven reflections with $11.17 < 2\theta < 34.27°$ was applied. No extinction correction was made. Initial calculations were performed on a NOVA 1200 computer with the *XTL/E-XTL* system (Syntex, 1976), and on a PC with the *SDS* system (Petříček & Malý, 1988).

All atoms were refined anisotropically. For the K atom in 16(c) the occupancy factor a and x, β_{11} and β_{12} were refined with $y = z = x$, $\beta_{22} = \beta_{33} = \beta_{11}$, $\beta_{13} = \beta_{23} = \beta_{12}$. For the (Si, B) site in 48(e) all parameters of Si were refined. For the B atom the same parameters as for Si were assumed except the occupation factor, $a_B = 1.0 - a_{Si}$. For the O atoms in 48(e) the occupancy factor was not refined. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). $(\Delta/\sigma)_{\max} = 0.00$; $-0.88 < \Delta\rho < 0.80$ e Å^{-3}; $R = 0.038$, $wR = 0.051$, $S = 1.33$. The quantity minimized was $w(\Delta F)^2$, where $w = [\sigma(F_o)^2 + 0.03F_o^2]^{-1}$.

Atomic parameters are given in Table 1.* Bond distances and angles are given in Table 2. Fig. 1 is a view of the structure.

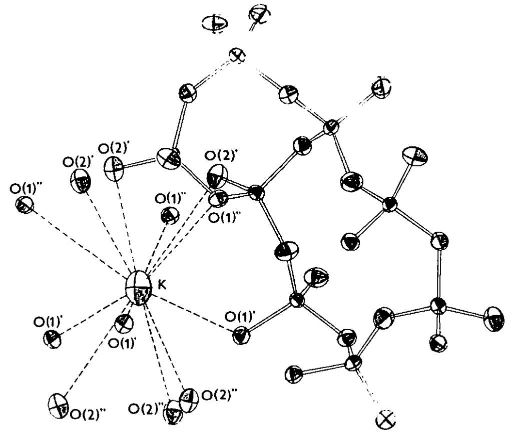

Fig. 1. A view of a structure fragment of boroleucite (Johnson, 1976) showing a four- and a six-membered ring of (Si,B)O_4 tetrahedra and the surrounding of the K atom with O atoms qualitatively labeled according to different K—O distances (see Table 2).

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55201 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA0017]

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Table 1. *Atomic occupation factors* (*a*), *positional parameters and equivalent isotropic temperature parameters* (Å^2)

$$B_{eq} = (4/3)\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	a	*x*	*y*	*z*	B_{eq}
K	0.327 (3)	0.36685 (10)	0.36685	0.36685	3.98 (3)
Si	0.653 (10)	0.37690 (7)	0.34345 (5)	0.08183 (6)	1.11 (1)
B	0.347	0.37690	0.34345	0.08183	1.11
O(1)	1.0	0.2908 (1)	0.4251 (1)	0.1200 (1)	1.50 (3)
O(2)	1.0	0.4871 (1)	0.3927 (1)	0.1092 (2)	2.11 (3)

Table 2. *Selected bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

(Si,B)O$_4$ tetrahedra			
(Si,B)—O(1)	1.573 (2)	O(1)—(Si,B)—O(1^{i})	111.24 (9)
—O(1^{i})	1.567 (2)	—O(2)	106.7 (1)
—O(2)	1.562 (2)	—O(2^{ii})	108.0 (1)
—O(2^{ii})	1.554 (2)	O(1^{i})—(Si,B)—O(2)	109.1 (1)
		—O(2^{ii})	109.6 (1)
O(1)—O(1^{i})	2.592 (2)	O(2)—(Si,B)—O(2^{ii})	112.3 (1)
—O(2)	2.515 (2)		
—O(2^{ii})	2.530 (2)	(Si,B)—O(1)—(Si,B^{iii})	126.9 (1)
O(2)—O(1^{i})	2.549 (3)	(Si,B)—O(2)—(Si,B^{iv})	144.2 (1)
—O(2)	2.588 (3)		
O(1^{i})—O(2^{ii})	2.550 (2)		
Shortest contacts (< 3.5 Å) of K atoms			
K—O(1^{v})	2.800 (2) × 3	K—O(1)	3.341 (2) × 3
—O(2^{ii})	3.291 (2) × 3	—O(2^{v})	3.405 (2) × 3

Symmetry code: (i) $\frac{1}{4}+z, \frac{3}{4}-y, \frac{1}{4}-x$; (ii) $\frac{3}{4}-y, -\frac{1}{4}+x, \frac{1}{4}-z$; (iii) $\frac{1}{4}-z, \frac{3}{4}-y, -\frac{1}{4}+x$; (iv) $\frac{1}{4}+y, \frac{3}{4}-x, \frac{1}{4}-z$; (v) $x, 1-y, \frac{1}{2}-z$.

Related literature. Structure analysis results for K(BSi$_2$O$_6$) were published by Kamei, Tsuda, Fukunaga, Yoshida & Ihara (1979) and Ihara & Kamei (1980). They refined the structure to $R = 0.0945$ using isotropic temperature factor coefficients (no occupancy factors were refined).

The authors are indebted to Dr J. Voldán for initiating this work and to Dr V. Figusch for supplying the crystals.

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Carbonyl(1-methyl-η^5-cyclopentadienyl)bis(triphenylphosphine)manganese Dichloromethane Solvate

By Arnold L. Rheingold,* Corbet B. White and Brian S. Haggerty

Department of Chemistry and Biochemistry, University of Delaware, Newark, DE 19716, *USA*

and Michael R. Terry and Gregory L. Geoffroy

Department of Chemistry, Pennsylvania State University, University Park, PA 16802, *USA*

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Abstract. [Mn(C$_{18}$H$_{15}$P)$_2$(C$_6$H$_7$)(CO)]·CH$_2$Cl$_2$, M_r = 771.53, triclinic, $P\bar{1}$, a = 9.875 (3), b = 14.794 (5), c = 14.917 (5) Å, α = 71.99 (2), β = 70.41 (2), γ = 74.67 (2)°, V = 1920.8 (9) Å^3, Z = 2, D_x = 1.334 g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, μ = 5.80 cm^{-1}, F(000) = 800, T = 296 K, $R_{(F)}$ = 0.0514 for 4733 observed reflections and 379 parameters. The structure around the central Mn atom has the expected three-legged piano-stool arrangement. The Cp′-ring methyl group is positioned between the two triphenylphosphine groups and nearly eclipses the carbonyl group (Cp′ = η^5-C$_5$H$_4$CH$_3$).

Experimental. An orange–red crystal from dichloromethane (0.41 × 0.41 × 0.52 mm) was used for data collection on a Nicolet $R3m$ diffractometer with graphite-monochromated radiation and ω scans. Lattice parameters were determined from least-squares fit of 25 reflections ($20 \leq 2\theta \leq 25°$). No absorption correction was applied (μ = 5.80 cm^{-1}).

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* Author to whom correspondence should be addressed.

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