SHORT-FORMAT PAPERS

Contributions intended for publication under this heading should follow the format given in the Checklist for Authors [*Acta Cryst.* (1985). C**41**, 1–4].

Acta Cryst. (1993). C**49**, 78–79

Structure of $2CaO.B_2O_3$

BY YANGYANG JI, JINGKUI LIANG AND SISHEN XIE

Institute of Physics, Chinese Academy of Sciences, Beijing 100080, *People's Republic of China*

AND NEIJUE ZHU AND YAN LI

Institute of Chemistry, Chinese Academy of Sciences, Beijing 100080, *People's Republic of China*

(*Received* 18 *November* 1991; *accepted* 13 *February* 1992)

Abstract. Diboron dicalcium pentaoxide, $B_2Ca_2O_5$, $M_r = 181.777$, monoclinic, $P2_1/c$, $a = 7.234$ (3), $b = 5.181$ (1), $c = 11.524$ (3) Å, $\beta = 92.94$ (3)°, $V = 431.41$ Å^3, $Z = 4$, $D_x = 2.80$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 24.97$ cm^{-1}, $F(000) = 360$, room temperature, $R = 0.037$ for 1840 observed reflections. There are two BO_3 groups in $2CaO.B_2O_3$ and the Ca ions are bonded in two different ways by six and seven O atoms, respectively.

Experimental. The crystals were prepared by the flux-growth method. Starting materials were $CaCO_3$ (99.9%), B_2O_3 (99.99%) and $BaCO_3$ (99.99%), with $BaCO_3$ and B_2O_3 added in excess as fluxes. The mixture was thoroughly ground and melted, then slowly cooled at a rate of 2 K h^{-1}. Colourless $2CaO.B_2O_3$ crystals were obtained.

A crystal of size $0.30 \times 0.20 \times 0.15$ mm was mounted on a Nicolet $R3$ single-crystal X-ray diffractometer. The unit-cell parameters were obtained by least-squares refinement of 25 strong centred reflections ($8 \le 2\theta \le 28°$). The intensity data were collected at room temperature with graphite-monochromated Mo $K\alpha$ radiation, using ω–2θ scans. A total of 2267 reflections were measured for $2\theta \le 45°$ ($0 \le h \le 8$, $0 \le k \le 6$, $-14 \le l \le 14$). A periodic check of two standard reflections showed no significant intensity variations. Intensity data were corrected for Lorentz and polarization effects, and an empirical absorption correction (North, Phillips & Mathews, 1968) was applied; the transmission factors varied between 0.250 and 0.148. 1840 unique reflections with $F > 2.5\sigma(F)$ were used in the subsequent analysis. The structure was solved by direct methods (Sheldrick, 1990) and subsequent $\Delta\rho$ maps. Least-squares refinement on F used the program *SHELX*76 (Sheldrick, 1976). The refinement converged at $R = 0.0372$, $wR = 0.0617$ $\{w = 1/[\sigma^2(F) + 0.0005F^2]\}$, $S = 2.10$. $(\Delta/\sigma)_{max} = 0.02$. Minimum and maximum heights in the final $\Delta\rho$ map were -1.24 and 1.05 e Å^{-3}. All calculations were carried out on an Eclipse-S140 computer. Atomic scattering factors and f' and f'' values were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atomic coordinates are listed in Table 1. Selected bond lengths and angles are given in Table 2.* The molecular configuration is shown in Fig. 1 and the unit-cell contents in Fig. 2. The Ca atoms are bonded by six or seven O atoms; the coordination polyhedra are illustrated in Fig. 3.

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$U_{eq} = (U_{11} + U_{22} + U_{33})/3$.

	x	y	z	U_{eq}
Ca(1)	0.6233 (1)	0.1008 (1)	0.3361 (1)	0.0087 (1)
Ca(2)	0.8820 (1)	0.5650 (1)	0.1331 (1)	0.0088 (1)
O(1)	0.6084 (2)	0.3069 (3)	0.1417 (1)	0.0124 (3)
O(2)	0.6460 (2)	−0.1447 (3)	0.1572 (1)	0.0109 (3)
O(3)	1.0922 (2)	−0.0802 (3)	0.1747 (1)	0.0129 (3)
O(4)	1.1437 (2)	0.3218 (3)	0.0710 (1)	0.0112 (3)
O(5)	0.7276 (3)	0.0917 (3)	−0.0155 (1)	0.0150 (3)
B(1)	0.6550 (3)	0.0760 (4)	0.0955 (2)	0.0093 (4)
B(2)	1.1631 (3)	0.0634 (4)	0.0888 (2)	0.0095 (4)

Related literature. The CaO–B_2O_3 binary phase diagram was reported by Carlson (1932). There are four

* Lists of structure factors, anisotropic thermal parameters, and bond distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55188 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB0264]

0108-2701/93/010078-02$06.00

Table 2. *Selected bond distances* (Å) *and angles* (°)

B(1)—O(1)	1.359 (2)	B(1)—O(2)	1.349 (2)
B(1)—O(5)	1.410 (2)	B(2)—O(3)	1.360 (2)
B(2)—O(4)	1.360 (2)	B(2)—O(5′)	1.433 (2)
Ca(1)—Ca(2)	3.900 (1)	Ca(1)—O(1)	2.480 (1)
Ca(1)—O(2)	2.436 (1)	Ca(1)—O(1′)	2.289 (1)
Ca(1)—O(2′)	2.358 (1)	Ca(1)—O(3′)	2.648 (1)
Ca(1)—O(4′)	2.427 (1)	Ca(1)—O(5′)	2.429 (1)
Ca(2)—O(1)	2.395 (1)	Ca(2)—O(4)	2.413 (1)
Ca(2)—O(2′)	2.303 (1)	Ca(2)—O(3″)	2.337 (1)
Ca(2)—O(3′)	2.418 (1)	Ca(2)—O(4′)	2.422 (1)

O(1)—B(1)—O(2)	121.3 (2)	O(2)—B(1)—O(5)	123.8 (2)
O(1)—B(1)—O(5)	114.6 (2)	O(3)—B(2)—O(4)	127.4 (2)
O(3)—B(2)—O(5′)	111.8 (2)	O(4)—B(2)—O(5′)	123.5 (2)
O(1)—Ca(1)—O(2)	57.4 (1)	O(1′)—Ca(1)—O(1)	112.9 (1)
O(1′)—Ca(1)—O(2)	80.1 (1)	O(2′)—Ca(1)—O(1)	77.9 (1)
O(2′)—Ca(1)—O(2)	114.3 (1)	O(2′)—Ca(1)—O(1′)	75.9 (1)
O(3′)—Ca(1)—O(1)	71.7 (1)	O(3′)—Ca(1)—O(2)	101.5 (1)
O(3′)—Ca(1)—O(1′)	175.0 (1)	O(3′)—Ca(1)—O(2′)	107.3 (1)
O(4′)—Ca(1)—O(1)	130.7 (1)	O(4′)—Ca(1)—O(2)	89.2 (1)
O(4′)—Ca(1)—O(1′)	92.9 (1)	O(4′)—Ca(1)—O(2′)	151.1 (1)
O(4′)—Ca(1)—O(3′)	82.5 (1)	O(5′)—Ca(1)—O(1)	110.6 (1)
O(5′)—Ca(1)—O(2)	155.0 (1)	O(5′)—Ca(1)—O(1′)	92.9 (1)
O(5′)—Ca(1)—O(2′)	80.6 (1)	O(5′)—Ca(1)—O(3′)	53.8 (1)
O(5′)—Ca(1)—O(4′)	84.1 (1)	O(2′)—Ca(2)—O(1)	75.0 (1)
O(1)—Ca(2)—O(4)	112.5 (1)	O(3″)—Ca(2)—O(1)	78.9 (1)
O(2′)—Ca(2)—O(4)	167.1 (1)	O(3″)—Ca(2)—O(2′)	96.9 (1)
O(3″)—Ca(2)—O(4)	95.0 (1)	O(3′)—Ca(2)—O(4)	87.8 (1)
O(3′)—Ca(2)—O(1)	158.3 (1)	O(3′)—Ca(2)—O(3″)	92.2 (1)
O(3′)—Ca(2)—O(2′)	86.7 (1)	O(4′)—Ca(2)—O(4)	82.0 (1)
O(4′)—Ca(2)—O(1)	98.8 (1)	O(4′)—Ca(2)—O(3″)	175.2 (1)
O(4′)—Ca(2)—O(2′)	86.5 (1)		
O(4′)—Ca(2)—O(3′)	91.4 (1)		

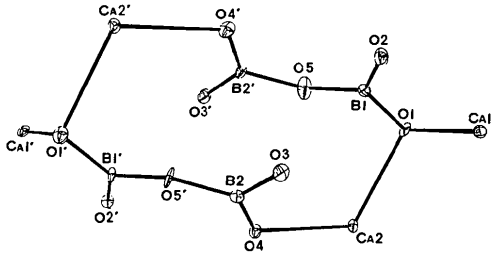

Fig. 1. A view of the molecule with the atom-numbering scheme.

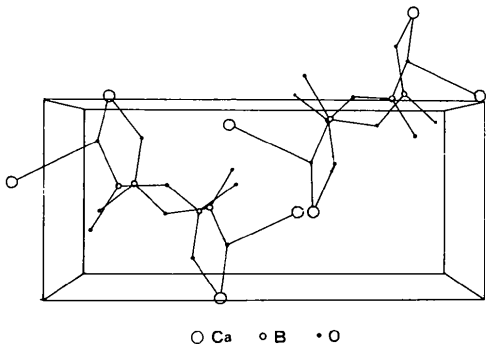

Fig. 2. Stereoscopic view of the unit cell.

compounds $CaO.2B_2O_3$, $CaO.B_2O_3$, $2CaO.B_2O_3$ and $3CaO.B_2O_3$ in the system. The structure of $CaO.B_2O_3$ has been determined by Marezio, Plettinger & Zachariasen (1963) and the electron-density

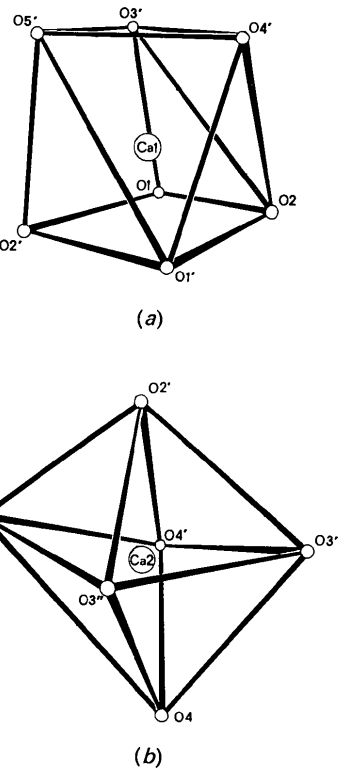

Fig. 3. O-ion coordination polyhedron (*a*) around Ca(1) and (*b*) around Ca(2).

distribution in $CaO.B_2O_3$ has been reported by Kirfel (1987). The structure of $3CaO.B_2O_3$ was described by Vegas (1985). The structure of $2CaO.B_2O_3$ has not been investigated previously, although powder diffraction data of this compound were given by Schäfer (1968) who reported a monoclinic structure with space group $P2_1/a$, $a = 11.497$, $b = 5.157$, $c = 7.200$ Å, $\beta = 92.91°$, $V = 426.89$ Å^3.

References

CARLSON, E. T. (1932). *Bur. Stand. J. Res.* **9**, 830.

KIRFEL, A. (1987). *Acta Cryst.* B**43**, 333–343.

MAREZIO, M., PLETTINGER, H. A. & ZACHARIASEN, W. H. (1963). *Acta Cryst.* **16**, 390–392.

NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

SCHÄFER, U. L. (1968). *Neues Jahrb. Mineral. Monatsh.* pp. 75–80. (Joint Committee on Powder Diffraction Standards, Powder Diffraction File, Inorganic Volume, Set 22, p. 139.)

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

SHELDRICK, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

VEGAS, A. (1985). *Acta Cryst.* C**41**, 1689–1690.