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Redetermination of $CaB_8O_{11}(OH)_4$ at low temperature

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(O–B) = 0.003 Å
R factor = 0.034
wR factor = 0.075
Data-to-parameter ratio = 11.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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The structure of $CaB_8O_{11}(OH)_4$ (calcium octaborate tetrahydroxide) [Zayakina & Brovkin (1978). *Kristallografiya*, **23**, 1167–1170] has been redetermined at 120 (2) K with improved precision. The O—H···O hydrogen-bonding arrangement has been established, based on freely refined H-atom positions.

Comment

During the investigation of templated boroarsenate frameworks, single crystals of the known (Zayakina & Brovkin, 1978) title compound, (I) (Fig. 1), were obtained from a molten salt reaction of $CaCl_2$, H_3BO_3 and $NH_4(H_2AsO_4)$. This redetermination at 120 (2) K offers a significantly better structural model and the H-atom positions and hydrogen-bonding scheme have been established. There is also an isostructural strontium material, strontioborite, reported by Brovkin *et al.* (1975).

The structure of (I) can be described in terms of linked triple six-rings of stoichiometry $B_6O_{12}H$ with a pendant $H_3B_2O_5$ group, as shown in Fig. 2. The three-coordinate O8 species (Table 1) is a distinctive feature of these units. Each of these triple-six-ring units have six O atoms that do not contribute to the ring formation. One of these forms a hydroxide grouping, four link to further similar units to form a sheet in the *bc* plane and the last bridges to an $H_3B_2O_5$ unit that is located outside the plane. The triple six-ring unit has two of the rings in the *bc* plane, while the third is below this plane. The out-of-plane ring has the hydroxide group

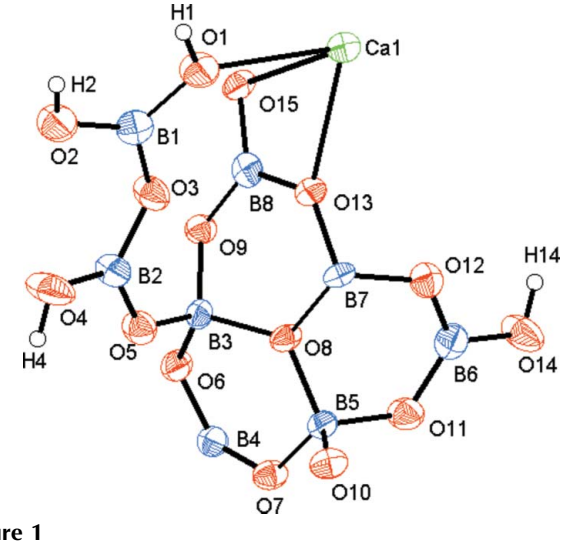

Figure 1
The asymmetric unit of (I), showing 50% probability displacement ellipsoids and arbitrary spheres for the H atoms. The mixture of trigonal (B1, B2, B4, B6 and B8) and tetrahedral (B3, B5 and B7) B atoms and the three-coordinate O8 species are evident.

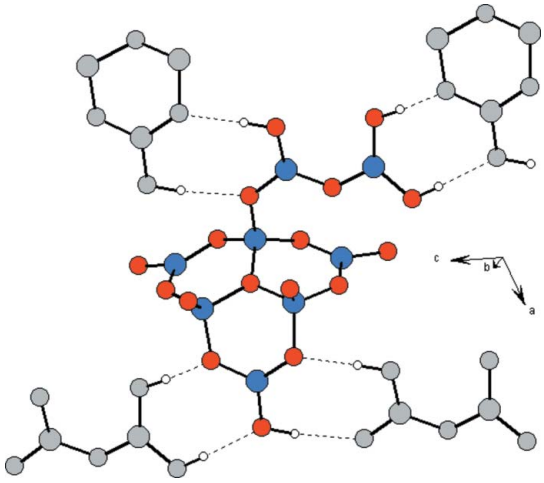

Figure 2
View of the borate unit in (I). Colour key: B blue, O red, and H white. Dotted lines signify hydrogen bonds.

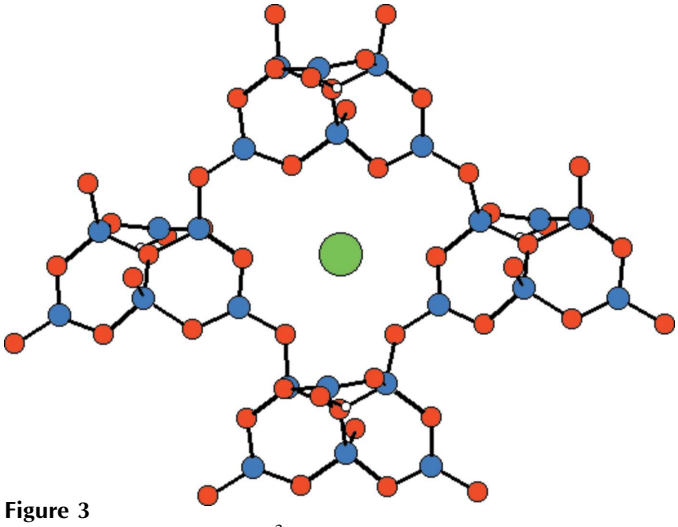

Figure 3
Detail of (I), showing the Ca^{2+} ion within its 18-atom ring. The $H_3B_2O_5$ units above and below the plane have been removed for clarity. Colour key: Ca green, other atom colours as in Fig. 2.

attached, forming, along with the pendant $H_3B_2O_5$ unit, an extensive hydrogen-bonding network between the borate sheets (Table 2). There are six distinct hydrogen bonds per unit, with O···O distances ranging from 2.585 (3) to 2.917 (4) Å. This network connects four adjacent $B_8O_{11}(OH)_4$ units to a central unit, as shown in Fig. 2.

The calcium ion sits in the centre of an 18-atom ring formed by four of the triple six-ring units (Fig. 3). Nine O atoms coordinate to the calcium cation, with Ca—O distances ranging from 2.482 (2) to 2.634 (2) Å (Table 1). Six of these Ca—O bonds arise from the 18-atom ring, and two $H_3B_2O_5$ units that occur above and below the plane complete the Ca nine-coordination.

Experimental

Compound (I) was prepared using a molten salt technique. A typical reaction involved grinding H_3BO_3 (0.4637 g, 7.5 mmol), $NH_4(H_2AsO_4)$ (1.1923 g, 7.5 mmol) and $CaCl_2$ (0.5549 g, 5 mmol) in a pestle and mortar before placing the powder in a 23 ml Parr Teflon-lined steel autoclave and heating to 513 K for 120 h. The product was washed with hot water to dissolve any remaining borate flux, leaving a white powder containing many colourless crystals of (I) in moderate yield (34% based on Ca). The material appears completely air- and water-stable.

Crystal data

$CaB_8O_{11}(OH)_4$	D_x = 2.131 Mg m^{-3}
M_r = 370.59	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 2430 reflections
a = 7.481 (6) Å	θ = 2.9–27.5°
b = 8.2693 (12) Å	μ = 0.64 mm^{-1}
c = 9.859 (3) Å	T = 120 (2) K
β = 108.76 (6)°	Plate, colourless
V = 577.5 (5) Å^3	0.06 × 0.06 × 0.01 mm
Z = 2	

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer	2611 independent reflections
φ and ω scans	2430 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	R_{int} = 0.059
T_{min} = 0.732, T_{max} = 0.994	θ_{max} = 27.5°
13192 measured reflections	$h = -9 \rightarrow 9$
	$k = -10 \rightarrow 10$
	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0314P)^2 + 0.2076P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.034	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.075	$\Delta\rho_{max}$ = 0.32 e Å^{-3}
S = 1.05	$\Delta\rho_{min}$ = −0.40 e Å^{-3}
2611 reflections	Absolute structure: Flack (1983), 1196 Friedel pairs
233 parameters	Flack parameter: 0.03 (3)
All H-atom parameters refined	

Table 1
Selected geometric parameters (Å, °).

Ca1—O1	2.619 (3)	Ca1—O7^{iii}	2.5610 (19)
Ca1—O13	2.5329 (18)	Ca1—O10^{iv}	2.621 (2)
Ca1—O15	2.634 (2)	Ca1—O2^{ii}	2.626 (3)
Ca1—O9^{i}	2.4806 (18)	Ca1—O6^{i}	2.6320 (18)
Ca1—O4^{ii}	2.528 (3)		
B5—O8—B7	116.33 (19)	B7—O8—B3	120.73 (18)
B5—O8—B3	122.88 (18)		

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

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O14—H14···O5^{ii}	0.98 (4)	1.93 (4)	2.900 (3)	173 (3)
O1—H1···O14^{v}	0.86 (3)	1.95 (3)	2.817 (3)	177 (3)
O2—H2···O11^{v}	0.87 (4)	1.72 (4)	2.585 (3)	172 (4)
O2—H2···O7^{v}	0.87 (4)	2.50 (4)	2.917 (4)	110 (3)
O4—H4···O12^{vi}	0.90 (4)	1.81 (4)	2.695 (3)	171 (4)
O4—H4···O13^{vi}	0.90 (4)	2.27 (4)	2.751 (3)	113 (3)

Symmetry codes: (ii) $x+1, y, z$; (v) $x-1, y, z-1$; (vi) $x-1, y, z$.

The H atoms were found in a difference map and their positions and U_{iso} values were freely refined.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; method used to solve structure: coordinates taken from Zayakina & Brovkin (1978); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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supporting information

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calcium octaborate tetrahydroxide

Crystal data

$CaB_8O_{11}(OH)_4$
$M_r = 370.59$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 7.481$ (6) Å
$b = 8.2693$ (12) Å
$c = 9.859$ (3) Å
$\beta = 108.76$ (6)°
$V = 577.5$ (5) Å^3
$Z = 2$
$F(000) = 368$
$D_x = 2.131$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2430 reflections
$\theta = 2.9$–$27.5°$
$\mu = 0.64$ mm^{-1}
$T = 120$ K
Plate, colourless
0.06 × 0.06 × 0.01 mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
Radiation source: Bruker-Nonius FR591 rotating anode
10cm confocal mirrors monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
$T_{min} = 0.732$, $T_{max} = 0.994$
13192 measured reflections
2611 independent reflections
2430 reflections with $I > 2\sigma(I)$
$R_{int} = 0.059$
$\theta_{max} = 27.5°$, $\theta_{min} = 3°$
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.075$
$S = 1.05$
2611 reflections
233 parameters
1 restraint
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0314P)^2 + 0.2076P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.40$ e Å^{-3}
Absolute structure: Flack (1983), 1196 Friedel pairs
Absolute structure parameter: 0.03 (3)

Special details

Experimental. *SADABS* was used to perform the Absorption correction Parameter refinement on 11680 reflections reduced *R*(int) from 0.1212 to 0.0551 Ratio of minimum to maximum apparent transmission: 0.736941 The given Tmin and Tmax were generated using the *SHELX* SIZE command

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
B1	0.1035 (4)	0.2765 (4)	0.2720 (3)	0.0113 (5)
B2	0.0908 (4)	0.2695 (4)	0.5216 (3)	0.0114 (6)
B3	0.3787 (4)	0.1696 (3)	0.7189 (3)	0.0086 (6)
B4	0.4671 (4)	0.1318 (3)	0.9806 (3)	0.0095 (6)
B5	0.5905 (4)	0.3827 (3)	0.9050 (3)	0.0092 (6)
B6	0.8931 (4)	0.3814 (4)	0.8517 (3)	0.0121 (6)
B7	0.5944 (4)	0.3830 (3)	0.6449 (3)	0.0087 (5)
B8	0.4913 (4)	0.1220 (3)	0.5148 (3)	0.0099 (6)
O1	0.2331 (2)	0.3060 (2)	0.20108 (18)	0.0141 (4)
O2	−0.0882 (2)	0.2699 (3)	0.2085 (2)	0.0166 (4)
O3	0.1776 (2)	0.2518 (2)	0.41723 (18)	0.0137 (4)
O4	−0.0921 (3)	0.3203 (3)	0.48254 (19)	0.0202 (5)
O5	0.1892 (2)	0.2394 (2)	0.66179 (18)	0.0116 (4)
O6	0.3931 (2)	0.0722 (2)	0.84528 (18)	0.0105 (4)
O7	0.5296 (2)	0.2872 (2)	1.00614 (16)	0.0102 (3)
O8	0.5214 (2)	0.3078 (2)	0.75752 (16)	0.0092 (3)
O9	0.4256 (2)	0.0600 (2)	0.61924 (18)	0.0094 (4)
O10	0.5179 (2)	0.5466 (2)	0.89685 (18)	0.0108 (4)
O11	0.7977 (2)	0.3809 (2)	0.94971 (17)	0.0124 (4)
O12	0.8015 (2)	0.4038 (2)	0.70949 (18)	0.0117 (4)
O13	0.5652 (2)	0.2750 (2)	0.52450 (17)	0.0101 (4)
O14	1.0848 (3)	0.3566 (3)	0.9032 (2)	0.0190 (4)
O15	0.4999 (2)	0.0372 (2)	0.39748 (18)	0.0107 (4)
Ca1	0.60200 (6)	0.29083 (6)	0.27828 (5)	0.01030 (12)
H1	0.191 (5)	0.320 (4)	0.109 (3)	0.029 (9)*
H2	−0.120 (5)	0.300 (5)	0.119 (4)	0.047 (11)*
H4	−0.127 (6)	0.337 (5)	0.560 (4)	0.061 (14)*
H14	1.130 (6)	0.314 (5)	0.827 (4)	0.054 (12)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
B1	0.0134 (12)	0.0077 (14)	0.0125 (13)	0.0017 (11)	0.0038 (10)	−0.0002 (11)
B2	0.0106 (12)	0.0130 (14)	0.0108 (13)	0.0025 (12)	0.0037 (10)	0.0016 (11)
B3	0.0082 (13)	0.0079 (14)	0.0096 (13)	−0.0014 (10)	0.0028 (11)	−0.0012 (10)
B4	0.0081 (13)	0.0114 (13)	0.0081 (14)	0.0012 (11)	0.0015 (11)	0.0006 (11)
B5	0.0096 (13)	0.0100 (13)	0.0077 (13)	−0.0015 (11)	0.0025 (11)	−0.0010 (11)
B6	0.0111 (14)	0.0117 (14)	0.0125 (14)	−0.0002 (11)	0.0024 (11)	−0.0006 (11)
B7	0.0104 (14)	0.0091 (13)	0.0061 (13)	−0.0001 (11)	0.0017 (11)	−0.0018 (11)
B8	0.0073 (13)	0.0107 (13)	0.0105 (14)	0.0027 (10)	0.0010 (10)	0.0022 (11)
O1	0.0138 (8)	0.0197 (10)	0.0082 (8)	0.0015 (8)	0.0025 (7)	0.0029 (8)
O2	0.0120 (8)	0.0257 (12)	0.0108 (9)	0.0016 (8)	0.0021 (7)	0.0009 (8)
O3	0.0088 (8)	0.0214 (10)	0.0105 (9)	0.0030 (7)	0.0027 (7)	0.0016 (7)
O4	0.0140 (9)	0.0376 (13)	0.0091 (9)	0.0075 (9)	0.0039 (7)	0.0012 (8)
O5	0.0101 (9)	0.0132 (9)	0.0120 (9)	0.0016 (6)	0.0039 (7)	0.0000 (6)

O6	0.0125 (9)	0.0101 (9)	0.0089 (9)	−0.0027 (7)	0.0035 (7)	−0.0001 (7)
O7	0.0130 (8)	0.0085 (7)	0.0094 (7)	0.0003 (9)	0.0040 (6)	−0.0004 (8)
O8	0.0136 (8)	0.0068 (8)	0.0076 (8)	−0.0032 (8)	0.0037 (6)	−0.0010 (7)
O9	0.0113 (9)	0.0088 (9)	0.0086 (8)	−0.0010 (7)	0.0038 (7)	−0.0001 (7)
O10	0.0139 (9)	0.0093 (8)	0.0087 (9)	0.0015 (7)	0.0029 (7)	0.0003 (7)
O11	0.0119 (9)	0.0159 (9)	0.0089 (9)	0.0013 (8)	0.0026 (7)	0.0003 (7)
O12	0.0114 (9)	0.0120 (9)	0.0115 (9)	0.0001 (7)	0.0035 (7)	0.0013 (7)
O13	0.0112 (8)	0.0093 (9)	0.0099 (8)	−0.0012 (8)	0.0038 (6)	−0.0022 (7)
O14	0.0095 (9)	0.0366 (12)	0.0108 (9)	0.0043 (8)	0.0030 (7)	0.0018 (8)
O15	0.0123 (9)	0.0107 (8)	0.0082 (8)	−0.0013 (7)	0.0017 (7)	−0.0013 (7)
Ca1	0.0114 (2)	0.0098 (2)	0.0096 (2)	0.0003 (2)	0.00327 (17)	−0.0004 (2)

Geometric parameters (Å, º)

B1—O2	1.370 (3)	B8—O15	1.372 (3)
B1—O3	1.374 (3)	B8—O13	1.372 (3)
B1—O1	1.387 (3)	B8—O9	1.375 (3)
B2—O4	1.363 (3)	O1—H1	0.86 (3)
B2—O5	1.364 (3)	O2—H2	0.87 (4)
B2—O3	1.390 (3)	O4—H4	0.90 (4)
B3—O6	1.458 (3)	O14—H14	0.98 (4)
B3—O9	1.460 (3)	O2—Ca1^{iii}	2.626 (3)
B3—O5	1.465 (3)	O4—Ca1^{iii}	2.528 (3)
B3—O8	1.526 (3)	O6—Ca1^{iv}	2.6320 (18)
B4—O6	1.361 (3)	O7—Ca1^{v}	2.5610 (19)
B4—O7	1.363 (4)	O9—Ca1^{iv}	2.4806 (18)
B4—O10^{i}	1.372 (3)	O10—B4^{vi}	1.372 (3)
B5—O10	1.452 (3)	O10—Ca1^{ii}	2.621 (2)
B5—O7	1.455 (3)	O15—B7^{iv}	1.453 (3)
B5—O11	1.469 (3)	Ca1—O1	2.619 (3)
B5—O8	1.511 (3)	Ca1—O13	2.5329 (18)
B6—O12	1.361 (3)	Ca1—O15	2.634 (2)
B6—O11	1.374 (3)	Ca1—O9^{ii}	2.4806 (18)
B6—O14	1.374 (4)	Ca1—O4^{vii}	2.528 (3)
B7—O13	1.444 (3)	Ca1—O7^{viii}	2.5610 (19)
B7—O15^{ii}	1.453 (3)	Ca1—O10^{iv}	2.621 (2)
B7—O12	1.483 (3)	Ca1—O2^{vii}	2.626 (3)
B7—O8	1.519 (3)	Ca1—O6^{ii}	2.6320 (18)
O2—B1—O3	118.8 (2)	O9^{ii}—Ca1—O4^{vii}	76.20 (7)
O2—B1—O1	125.2 (2)	O9^{ii}—Ca1—O13	66.86 (6)
O3—B1—O1	116.0 (2)	O4^{vii}—Ca1—O13	65.85 (8)
O4—B2—O5	120.6 (2)	O9^{ii}—Ca1—O7^{viii}	114.83 (6)
O4—B2—O3	119.2 (2)	O4^{vii}—Ca1—O7^{viii}	131.92 (7)
O5—B2—O3	120.2 (2)	O13—Ca1—O7^{viii}	162.18 (6)
O6—B3—O9	105.4 (2)	O9^{ii}—Ca1—O1	81.81 (7)
O6—B3—O5	110.0 (2)	O4^{vii}—Ca1—O1	145.73 (7)
O9—B3—O5	113.5 (2)	O13—Ca1—O1	81.49 (8)

O6—B3—O8	110.3 (2)	O7^{viii}—Ca1—O1	81.26 (8)
O9—B3—O8	109.42 (19)	O9^{ii}—Ca1—O10^{iv}	154.72 (6)
O5—B3—O8	108.3 (2)	O4^{vii}—Ca1—O10^{iv}	129.02 (7)
O6—B4—O7	122.0 (2)	O13—Ca1—O10^{iv}	118.17 (6)
O6—B4—O10^{i}	124.7 (2)	O7^{viii}—Ca1—O10^{iv}	52.31 (6)
O7—B4—O10^{i}	113.3 (2)	O1—Ca1—O10^{iv}	74.93 (7)
O10—B5—O7	110.5 (2)	O9^{ii}—Ca1—O2^{vii}	111.21 (6)
O10—B5—O11	111.5 (2)	O4^{vii}—Ca1—O2^{vii}	64.27 (8)
O7—B5—O11	108.7 (2)	O13—Ca1—O2^{vii}	128.64 (8)
O10—B5—O8	108.9 (2)	O7^{viii}—Ca1—O2^{vii}	68.41 (8)
O7—B5—O8	110.6 (2)	O1—Ca1—O2^{vii}	149.66 (6)
O11—B5—O8	106.6 (2)	O10^{iv}—Ca1—O2^{vii}	85.45 (7)
O12—B6—O11	121.5 (2)	O9^{ii}—Ca1—O6^{ii}	53.88 (6)
O12—B6—O14	121.4 (2)	O4^{vii}—Ca1—O6^{ii}	98.03 (7)
O11—B6—O14	117.1 (2)	O13—Ca1—O6^{ii}	120.74 (6)
O13—B7—O15^{ii}	112.0 (2)	O7^{viii}—Ca1—O6^{ii}	63.58 (6)
O13—B7—O12	106.6 (2)	O1—Ca1—O6^{ii}	89.61 (7)
O15^{ii}—B7—O12	111.4 (2)	O10^{iv}—Ca1—O6^{ii}	115.40 (6)
O13—B7—O8	110.7 (2)	O2^{vii}—Ca1—O6^{ii}	78.02 (7)
O15^{ii}—B7—O8	108.32 (19)	O9^{ii}—Ca1—O15	117.30 (6)
O12—B7—O8	107.76 (19)	O4^{vii}—Ca1—O15	92.26 (7)
O15—B8—O13	113.8 (2)	O13—Ca1—O15	52.77 (6)
O15—B8—O9	124.5 (2)	O7^{viii}—Ca1—O15	117.66 (7)
O13—B8—O9	121.7 (2)	O1—Ca1—O15	74.82 (7)
B1—O1—H1	118 (2)	O10^{iv}—Ca1—O15	66.02 (6)
Ca1—O1—H1	108 (2)	O2^{vii}—Ca1—O15	118.11 (7)
B1—O2—Ca1^{iii}	139.51 (16)	O6^{ii}—Ca1—O15	163.61 (6)
B1—O2—H2	111 (3)	O9^{ii}—Ca1—B8	91.19 (7)
Ca1^{iii}—O2—H2	105 (3)	O4^{vii}—Ca1—B8	80.44 (8)
B1—O3—B2	128.9 (2)	O13—Ca1—B8	26.40 (7)
B2—O4—Ca1^{iii}	139.90 (17)	O7^{viii}—Ca1—B8	141.02 (7)
B2—O4—H4	110 (3)	O1—Ca1—B8	74.02 (9)
Ca1^{iii}—O4—H4	105 (3)	O10^{iv}—Ca1—B8	91.80 (7)
B2—O5—B3	127.0 (2)	O2^{vii}—Ca1—B8	130.46 (8)
B4—O6—B3	122.2 (2)	O6^{ii}—Ca1—B8	143.67 (7)
B4—O6—Ca1^{iv}	135.38 (16)	O15—Ca1—B8	26.64 (7)
B3—O6—Ca1^{iv}	95.79 (13)	O9^{ii}—Ca1—B4^{viii}	136.70 (7)
B4—O7—B5	123.3 (2)	O4^{vii}—Ca1—B4^{viii}	137.46 (8)
B4—O7—Ca1^{v}	98.58 (15)	O13—Ca1—B4^{viii}	142.12 (7)
B5—O7—Ca1^{v}	134.77 (15)	O7^{viii}—Ca1—B4^{viii}	26.00 (7)
B5—O8—B7	116.33 (19)	O1—Ca1—B4^{viii}	75.61 (9)
B5—O8—B3	122.88 (18)	O10^{iv}—Ca1—B4^{viii}	26.36 (7)
B7—O8—B3	120.73 (18)	O2^{vii}—Ca1—B4^{viii}	76.60 (9)
B8—O9—B3	119.6 (2)	O6^{ii}—Ca1—B4^{viii}	89.16 (7)
B8—O9—Ca1^{iv}	137.40 (16)	O15—Ca1—B4^{viii}	91.85 (7)
B3—O9—Ca1^{iv}	102.32 (14)	B8—Ca1—B4^{viii}	116.57 (8)
B4^{vi}—O10—B5	120.5 (2)	O9^{ii}—Ca1—B3^{ii}	27.05 (6)
B4^{vi}—O10—Ca1^{ii}	95.64 (15)	O4^{vii}—Ca1—B3^{ii}	82.49 (7)

B5—O10—Ca1^{ii}	142.95 (15)	O13—Ca1—B3^{ii}	93.55 (7)
B6—O11—B5	121.7 (2)	O7^{viii}—Ca1—B3^{ii}	90.83 (7)
B6—O12—B7	122.5 (2)	O1—Ca1—B3^{ii}	89.77 (7)
B8—O13—B7	125.3 (2)	O10^{iv}—Ca1—B3^{ii}	141.33 (7)
B8—O13—Ca1	98.42 (15)	O2^{vii}—Ca1—B3^{ii}	91.50 (7)
B7—O13—Ca1	136.19 (15)	O6^{ii}—Ca1—B3^{ii}	27.57 (6)
B6—O14—H14	110 (2)	O15—Ca1—B3^{ii}	144.13 (7)
B8—O15—B7^{iv}	122.8 (2)	B8—Ca1—B3^{ii}	118.24 (8)
B8—O15—Ca1	93.93 (15)	B4^{viii}—Ca1—B3^{ii}	115.83 (8)
B7^{iv}—O15—Ca1	138.97 (15)		

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+2; (ii) −*x*+1, *y*+1/2, −*z*+1; (iii) *x*−1, *y*, *z*; (iv) −*x*+1, *y*−1/2, −*z*+1; (v) *x*, *y*, *z*+1; (vi) −*x*+1, *y*+1/2, −*z*+2; (vii) *x*+1, *y*, *z*; (viii) *x*, *y*, *z*−1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O14—H14···O5^{vii}	0.98 (4)	1.93 (4)	2.900 (3)	173 (3)
O1—H1···O14^{ix}	0.86 (3)	1.95 (3)	2.817 (3)	177 (3)
O2—H2···O11^{ix}	0.87 (4)	1.72 (4)	2.585 (3)	172 (4)
O2—H2···O7^{ix}	0.87 (4)	2.50 (4)	2.917 (4)	110 (3)
O4—H4···O12^{iii}	0.90 (4)	1.81 (4)	2.695 (3)	171 (4)
O4—H4···O13^{iii}	0.90 (4)	2.27 (4)	2.751 (3)	113 (3)

Symmetry codes: (iii) *x*−1, *y*, *z*; (vii) *x*+1, *y*, *z*; (ix) *x*−1, *y*, *z*−1.