

Dipotassium tetrahydroxidopentaoxidotetraborate monohydrate

M. K. Dhatchaiyini,^a M. NizamMohideen,^{b*} G. Rajasekar^a and A. Bhaskaran^{a*}

^aPG & Research Department of Physics, Presidency College (Autonomous), Chennai 600 005, Tamil Nadu, India, and

^bPG & Research Department of Physics, The New College (Autonomous), Chennai 600 014, Tamil Nadu, India.

*Correspondence e-mail: mnizam.new@gmail.com, abhaskaran_68@yahoo.co.in

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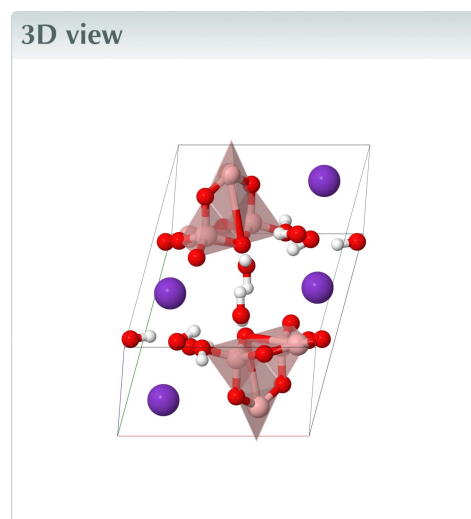
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In the tetraborate anion of the title compound, $K_2[B_4O_5(OH)_4] \cdot H_2O$, the bridging B—O bond lengths of the tetrahedral BO_4 and the trigonal-planar BO_3 units are slightly longer than the corresponding terminal B—OH bond lengths. The crystal structure is stabilized by intermolecular $O—H \cdots O$, $O—H \cdots O_{water}$ and $O_{water}—H \cdots O$ hydrogen bonds, generating a three-dimensional network. The two potassium cations both show a coordination number of 9.



Structure description

Inorganic borates exhibit promising applications as non-linear optical materials, birefringent materials, ferroelectric and piezoelectric materials, or host materials for luminescence (Berger, 1950; Heller *et al.*, 1986; Becker, 1998; Chen *et al.*, 1999, 2007*a,b*, 2010, 2012; Yu *et al.*, 2011; Wu *et al.*, 2012; Strauss *et al.*, 2016). In general, boron atoms in borates can be coordinated by either three or four oxygen atoms, forming trigonal-planar BO_3 or tetrahedral BO_4 groups, respectively. These groups may condense with each other through common oxygen atoms to give polyborate anionic groups that can adopt different coordination modes to bind to metal cations. The crystal chemistry of the resultant borates is rich, including rings, loops, infinite chains, sheets or three-dimensional networks (Burns *et al.*, 1995). Against this background, we report herein on the crystal structure of $K_2[B_4O_5(OH)_4] \cdot H_2O$ (I). This monohydrate is closely related to the corresponding dihydrate $K_2[B_4O_5(OH)_4] \cdot 2H_2O$ (Marezio *et al.*, 1963).

Fig. 1. shows the asymmetric unit of (I). It features two K^+ cations, one $[B_4O_5(OH)_4]^{2-}$ tetraborate anion and one water molecule of crystallization. The anion comprises of two tetrahedral BO_4 (B2, B4) and two trigonal-planar BO_3 (B1, B3) units, fused to a double ring *via* the central tetrahedra. Both BO_4 and BO_3 groups are rather regular; the O—B—O angles in the tetrahedra cover the range between $106.7(2)$ and $111.51(14)^\circ$, and those

Table 1
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O9—H9 \cdots O8 ⁱ	0.83 (2)	2.02 (2)	2.8385 (18)	173 (2)
O4—H4 \cdots O6 ⁱⁱ	0.83 (2)	2.03 (2)	2.8338 (18)	164 (3)
O2—H2 \cdots O9 ⁱⁱⁱ	0.80 (2)	2.19 (2)	2.957 (2)	161 (3)
O10—H10A \cdots O6	0.86 (2)	1.82 (2)	2.6704 (18)	175 (3)
O10—H10B \cdots O1 ^{iv}	0.85 (2)	2.10 (2)	2.850 (2)	146 (3)
O7—H7 \cdots O10 ^v	0.83 (2)	2.00 (2)	2.823 (2)	169 (3)

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

in the triangles between 116.26 (18) and 124.20 (17)°, with average O—B—O angles of 109.4 and 119.9°, respectively. The B—O bond lengths in the tetrahedra range from 1.441 (2) to 1.512 (2) Å, and those in the trigonal-planar units between 1.356 (2) and 1.390 (2) Å. The average B—O bond lengths (1.478 and 1.370 Å, respectively) are in good agreement with the data reviewed by Hawthorne *et al.* (1996), Chen *et al.* (2017) or Zobetz (1982).

All terminal O atoms (O2, O4, O7, O9) in the anion carry an additional hydrogen atom, and are active in intermolecular O—H \cdots O hydrogen bonding (Table 1, Fig. 2), generating centrosymmetric hydrogen-bonded dimers with a cyclic $R_2^2(8)$ ring motif. The crystal packing further comprises O—H \cdots O_{water} and O_{water}—H \cdots O hydrogen bonds whereby the water molecule (O10) interacts with O1 and O6 of the anion to form $R_2^2(12)$ ring motifs. Taking all the hydrogen-bonding interactions together, a three-dimensional network arises. The two unique potassium cations are situated in between the anionic network, with K—O distances ranging from 2.731 (2) to 3.269 (2) Å, and with a coordination number of 9 for both cations.

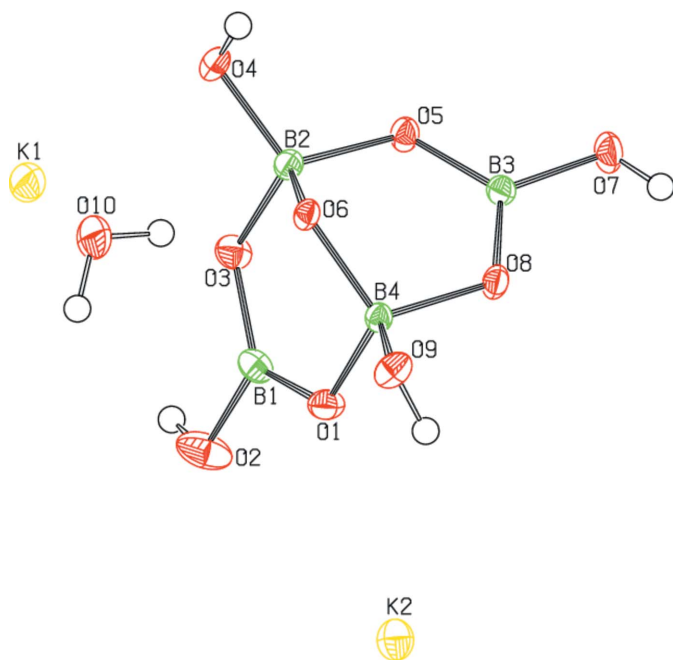


Figure 1
View of the asymmetric unit of (I) with the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

Table 2
Experimental details.

Crystal data	$K_2[B_4O_5(OH)_4]\cdot H_2O$
Chemical formula	287.49
M_r	Triclinic, $P\bar{1}$
Crystal system, space group	296
Temperature (K)	7.1850 (6), 7.8479 (7), 8.9932 (8)
a, b, c (Å)	68.572 (1), 88.393 (2), 74.975 (1)
α, β, γ (°)	454.66 (7)
V (Å ³)	2
Z	Cu $K\alpha$
Radiation type	9.68
μ (mm ⁻¹)	0.15 × 0.15 × 0.10
Crystal size (mm)	
Data collection	
Diffractometer	Bruker APEX3 CMOS
Absorption correction	Multi-scan (SADABS; Bruker, 2014)
T_{min}, T_{max}	0.428, 0.755
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	12205, 1782, 1685
R_{int}	0.048
$(\sin \theta/\lambda)_{max}$ (Å ⁻¹)	0.618
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.029, 0.073, 1.08
No. of reflections	1782
No. of parameters	169
No. of restraints	8
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å ⁻³)	0.26, -0.41

Computer programs: APEX3 and SAINT (Bruker, 2014), SHELXS97 (Sheldrick, 2008), SHELXL2014/7 (Sheldrick, 2015), ORTEP-3 for Windows (Farrugia, 2012), PLATON (Spek, 2009) and publCIF (Westrip, 2010).

Synthesis and crystallization

Potassium carbonate (13.8 g) and boric acid (6.1 g) were mixed in the molar ratio 1:1 to prepare an aqueous solution of potassium borate. By continuous stirring, the solution achieved super saturation conditions. Crystallization from this solution yielded good-quality crystals. In order to ensure the purity of the product, recrystallization was carried out for

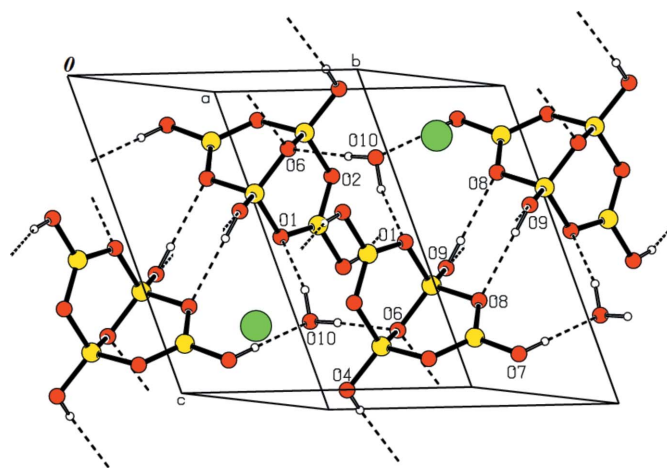


Figure 2
The crystal packing of the title compound, stabilized by medium-strong O—H \cdots O, O—H \cdots O_{water} and O_{water}—H \cdots O hydrogen bonds (shown as dashed lines; see Table 1 for numerical details).

several times by using double-distilled water to get high-quality crystals. The crystals were grown by slow and controlled evaporation of the solvent in a constant temperature bath at 313 K. The period of growth ranged from 30 to 40 days. In this way, crystals of $\text{K}_2[\text{B}_4\text{O}_5(\text{OH})_4]\cdot\text{H}_2\text{O}$ with dimensions up to $7 \times 9 \times 6 \text{ mm}^3$ could be obtained.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2019). 4, x190128 [https://doi.org/10.1107/S2414314619001287]

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Crystal data

$K_2(B_4O_5(OH)_4) \cdot H_2O$

$M_r = 287.49$

Triclinic, $P\bar{1}$

$a = 7.1850$ (6) Å

$b = 7.8479$ (7) Å

$c = 8.9932$ (8) Å

$\alpha = 68.572$ (1)°

$\beta = 88.393$ (2)°

$\gamma = 74.975$ (1)°

$V = 454.66$ (7) Å³

$Z = 2$

$F(000) = 288$

$D_x = 2.100$ Mg m⁻³

Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å

Cell parameters from 9535 reflections

$\theta = 5.3$ – 72.9 °

$\mu = 9.68$ mm⁻¹

$T = 296$ K

Block, colourless

$0.15 \times 0.15 \times 0.10$ mm

Data collection

Bruker APEX3 CMOS

diffractometer

Radiation source: micro-focus sealed tube

ω and ϕ scan

Absorption correction: multi-scan

(*SADABS*; Bruker, 2014)

$T_{\min} = 0.428$, $T_{\max} = 0.755$

12205 measured reflections

1782 independent reflections

1685 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.048$

$\theta_{\max} = 72.5$ °, $\theta_{\min} = 5.3$ °

$h = -8 \rightarrow 8$

$k = -9 \rightarrow 9$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.029$

$wR(F^2) = 0.073$

$S = 1.08$

1782 reflections

169 parameters

8 restraints

Hydrogen site location: difference Fourier map

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0364P)^2 + 0.3324P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.26$ e Å⁻³

$\Delta\rho_{\min} = -0.41$ e Å⁻³

Special details

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

Refinement. The O-bound hydrogen atoms were located in a difference Fourier map and were refined with distance restraints of O—H = 0.85 (2) Å.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
K1	-0.09251 (7)	0.38216 (6)	0.80053 (5)	0.02243 (14)
K2	0.20676 (6)	1.09022 (6)	0.19990 (5)	0.02022 (13)
O1	0.30645 (19)	0.80307 (18)	0.52405 (15)	0.0163 (3)
O2	0.0185 (2)	0.7652 (2)	0.44414 (18)	0.0278 (4)
O3	0.11229 (19)	0.64763 (19)	0.72235 (16)	0.0170 (3)
O4	0.2514 (2)	0.44850 (18)	0.98578 (16)	0.0165 (3)
O5	0.20264 (19)	0.78980 (17)	0.89638 (15)	0.0147 (3)
O6	0.45190 (18)	0.59912 (16)	0.78995 (15)	0.0118 (3)
O7	0.2574 (2)	1.07780 (19)	0.88399 (17)	0.0193 (3)
O8	0.40972 (19)	0.93391 (17)	0.70523 (15)	0.0148 (3)
O9	0.64504 (19)	0.74772 (18)	0.58708 (16)	0.0167 (3)
O10	0.5427 (3)	0.2666 (2)	0.75091 (19)	0.0276 (4)
B1	0.1486 (3)	0.7371 (3)	0.5679 (3)	0.0154 (4)
B2	0.2586 (3)	0.6167 (3)	0.8510 (2)	0.0125 (4)
B3	0.2906 (3)	0.9315 (3)	0.8275 (2)	0.0127 (4)
B4	0.4565 (3)	0.7673 (3)	0.6519 (2)	0.0111 (4)
H9	0.620 (3)	0.843 (3)	0.504 (2)	0.030 (7)*
H4	0.351 (3)	0.420 (4)	1.043 (3)	0.036 (8)*
H2	-0.076 (3)	0.735 (4)	0.482 (4)	0.049 (9)*
H10A	0.508 (5)	0.375 (3)	0.759 (3)	0.051 (9)*
H10B	0.547 (5)	0.285 (4)	0.652 (2)	0.049 (9)*
H7	0.329 (4)	1.148 (4)	0.842 (4)	0.050 (9)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.0295 (3)	0.0193 (2)	0.0218 (2)	-0.00895 (17)	0.00811 (18)	-0.01020 (17)
K2	0.0186 (2)	0.0179 (2)	0.0239 (2)	-0.00297 (16)	0.00074 (17)	-0.00873 (16)
O1	0.0168 (7)	0.0199 (6)	0.0121 (6)	-0.0072 (5)	0.0003 (5)	-0.0041 (5)
O2	0.0174 (8)	0.0495 (10)	0.0191 (8)	-0.0102 (7)	-0.0008 (6)	-0.0146 (7)
O3	0.0147 (6)	0.0228 (7)	0.0161 (6)	-0.0085 (5)	0.0008 (5)	-0.0077 (5)
O4	0.0201 (7)	0.0119 (6)	0.0162 (7)	-0.0069 (5)	0.0018 (6)	-0.0022 (5)
O5	0.0163 (6)	0.0130 (6)	0.0173 (6)	-0.0057 (5)	0.0063 (5)	-0.0078 (5)
O6	0.0130 (6)	0.0082 (5)	0.0137 (6)	-0.0025 (5)	0.0019 (5)	-0.0039 (5)
O7	0.0246 (7)	0.0152 (7)	0.0239 (7)	-0.0077 (6)	0.0081 (6)	-0.0125 (6)
O8	0.0194 (7)	0.0102 (6)	0.0169 (6)	-0.0060 (5)	0.0059 (5)	-0.0062 (5)
O9	0.0136 (7)	0.0144 (6)	0.0188 (7)	-0.0023 (5)	0.0055 (5)	-0.0037 (5)
O10	0.0435 (9)	0.0161 (7)	0.0268 (8)	-0.0075 (6)	0.0089 (7)	-0.0127 (6)
B1	0.0142 (10)	0.0179 (10)	0.0155 (10)	-0.0022 (8)	0.0007 (8)	-0.0091 (8)
B2	0.0131 (10)	0.0117 (9)	0.0127 (9)	-0.0040 (7)	0.0011 (8)	-0.0039 (7)
B3	0.0145 (10)	0.0107 (9)	0.0122 (9)	-0.0018 (7)	-0.0003 (8)	-0.0044 (7)
B4	0.0113 (9)	0.0105 (9)	0.0124 (9)	-0.0031 (7)	0.0023 (8)	-0.0051 (7)

Geometric parameters (Å, °)

K1—O3	2.7315 (14)	O4—B2	1.441 (2)
K1—O5 ⁱ	2.7608 (14)	O4—K1 ⁱ	2.7783 (14)
K1—O4 ⁱ	2.7782 (14)	O4—K2 ^x	2.8624 (13)
K1—O2 ⁱⁱ	2.8174 (16)	O4—H4	0.825 (17)
K1—O7 ⁱⁱⁱ	2.8759 (15)	O5—B3	1.363 (2)
K1—O9 ^{iv}	2.9741 (14)	O5—B2	1.512 (2)
K1—O10 ^{iv}	3.0799 (18)	O5—K1 ⁱ	2.7608 (14)
K1—O6 ^{iv}	3.2608 (13)	O5—K2 ^{ix}	2.8982 (14)
K1—O4	3.2684 (14)	O6—B4	1.451 (2)
K2—O10 ^v	2.7938 (16)	O6—B2	1.471 (2)
K2—O8 ^{vi}	2.8461 (14)	O6—K1 ^{xi}	3.2608 (13)
K2—O4 ^{vii}	2.8624 (13)	O7—B3	1.383 (2)
K2—O7 ^{viii}	2.8877 (15)	O7—K1 ^{xii}	2.8759 (15)
K2—O5 ^{ix}	2.8982 (14)	O7—K2 ^{xiii}	2.8877 (15)
K2—O3 ^{ix}	2.9142 (15)	O7—H7	0.834 (18)
K2—O1	2.9274 (13)	O8—B3	1.372 (3)
K2—O9 ^{vi}	3.0031 (14)	O8—B4	1.508 (2)
K2—O2	3.2690 (18)	O8—K2 ^{vi}	2.8461 (14)
O1—B1	1.356 (3)	O9—B4	1.453 (2)
O1—B4	1.497 (2)	O9—K1 ^{xi}	2.9741 (14)
O2—B1	1.390 (2)	O9—K2 ^{vi}	3.0031 (14)
O2—K1 ⁱⁱ	2.8174 (16)	O9—H9	0.825 (17)
O2—H2	0.804 (17)	O10—K2 ^v	2.7939 (16)
O3—B1	1.361 (3)	O10—K1 ^{xi}	3.0799 (18)
O3—B2	1.495 (2)	O10—H10A	0.856 (17)
O3—K2 ^{ix}	2.9142 (15)	O10—H10B	0.850 (17)
O3—K1—O5 ⁱ	124.63 (4)	B1—O3—K2 ^{ix}	107.19 (11)
O3—K1—O4 ⁱ	83.56 (4)	B2—O3—K2 ^{ix}	98.46 (10)
O5 ⁱ —K1—O4 ⁱ	52.02 (4)	K1—O3—K2 ^{ix}	90.33 (4)
O3—K1—O2 ⁱⁱ	102.87 (5)	B2—O4—K1 ⁱ	99.00 (10)
O5 ⁱ —K1—O2 ⁱⁱ	127.95 (5)	B2—O4—K2 ^x	167.17 (11)
O4 ⁱ —K1—O2 ⁱⁱ	167.06 (5)	K1 ⁱ —O4—K2 ^x	90.48 (4)
O3—K1—O7 ⁱⁱⁱ	91.40 (4)	B2—O4—K1	91.71 (10)
O5 ⁱ —K1—O7 ⁱⁱⁱ	87.71 (4)	K1 ⁱ —O4—K1	109.46 (4)
O4 ⁱ —K1—O7 ⁱⁱⁱ	121.96 (4)	K2 ^x —O4—K1	76.91 (3)
O2 ⁱⁱ —K1—O7 ⁱⁱⁱ	69.61 (4)	B2—O4—H4	105 (2)
O3—K1—O9 ^{iv}	72.91 (4)	K1 ⁱ —O4—H4	83 (2)
O5 ⁱ —K1—O9 ^{iv}	117.88 (4)	K2 ^x —O4—H4	84 (2)
O4 ⁱ —K1—O9 ^{iv}	77.28 (4)	K1—O4—H4	158 (2)
O2 ⁱⁱ —K1—O9 ^{iv}	93.72 (4)	B3—O5—B2	118.48 (15)
O7 ⁱⁱⁱ —K1—O9 ^{iv}	154.34 (4)	B3—O5—K1 ⁱ	135.06 (11)
O3—K1—O10 ^{iv}	149.75 (4)	B2—O5—K1 ⁱ	97.85 (10)
O5 ⁱ —K1—O10 ^{iv}	74.23 (4)	B3—O5—K2 ^{ix}	112.27 (11)
O4 ⁱ —K1—O10 ^{iv}	94.59 (4)	B2—O5—K2 ^{ix}	98.67 (9)
O2 ⁱⁱ —K1—O10 ^{iv}	74.14 (4)	K1 ⁱ —O5—K2 ^{ix}	85.04 (4)

O7 ⁱⁱⁱ —K1—O10 ^{iv}	114.49 (4)	B4—O6—B2	111.10 (13)
O9 ^{iv} —K1—O10 ^{iv}	77.23 (4)	B4—O6—K1 ^{xi}	95.42 (10)
O3—K1—O6 ^{iv}	108.12 (4)	B2—O6—K1 ^{xi}	153.42 (10)
O5 ⁱ —K1—O6 ^{iv}	75.04 (4)	B3—O7—K1 ^{xii}	122.95 (11)
O4 ⁱ —K1—O6 ^{iv}	55.28 (4)	B3—O7—K2 ^{xiii}	132.83 (12)
O2 ⁱⁱ —K1—O6 ^{iv}	111.80 (4)	K1 ^{xii} —O7—K2 ^{xiii}	83.18 (4)
O7 ⁱⁱⁱ —K1—O6 ^{iv}	159.03 (4)	B3—O7—H7	110 (2)
O9 ^{iv} —K1—O6 ^{iv}	44.97 (3)	K1 ^{xii} —O7—H7	95 (2)
O10 ^{iv} —K1—O6 ^{iv}	49.71 (4)	K2 ^{xiii} —O7—H7	106 (2)
O3—K1—O4	45.69 (3)	B3—O8—B4	118.94 (14)
O5 ⁱ —K1—O4	85.16 (4)	B3—O8—K2 ^{vi}	111.12 (11)
O4 ⁱ —K1—O4	70.54 (5)	B4—O8—K2 ^{vi}	97.89 (10)
O2 ⁱⁱ —K1—O4	121.96 (4)	B4—O9—K1 ^{xi}	108.04 (10)
O7 ⁱⁱⁱ —K1—O4	65.58 (4)	B4—O9—K2 ^{vi}	92.77 (10)
O9 ^{iv} —K1—O4	111.87 (4)	K1 ^{xi} —O9—K2 ^{vi}	84.16 (4)
O10 ^{iv} —K1—O4	159.30 (4)	B4—O9—H9	99.7 (15)
O6 ^{iv} —K1—O4	123.31 (3)	K1 ^{xi} —O9—H9	152.0 (16)
O10 ^v —K2—O8 ^{vi}	68.19 (4)	K2 ^{vi} —O9—H9	98.6 (18)
O10 ^v —K2—O4 ^{vii}	126.34 (5)	K2 ^v —O10—K1 ^{xi}	85.71 (5)
O8 ^{vi} —K2—O4 ^{vii}	78.89 (4)	K2 ^v —O10—H10A	155 (2)
O10 ^v —K2—O7 ^{viii}	74.82 (4)	K1 ^{xi} —O10—H10A	72 (2)
O8 ^{vi} —K2—O7 ^{viii}	100.77 (4)	K2 ^v —O10—H10B	85 (2)
O4 ^{vii} —K2—O7 ^{viii}	71.12 (4)	K1 ^{xi} —O10—H10B	99 (2)
O10 ^v —K2—O5 ^{ix}	126.21 (4)	H10A—O10—H10B	108 (2)
O8 ^{vi} —K2—O5 ^{ix}	165.60 (4)	O1—B1—O3	124.20 (17)
O4 ^{vii} —K2—O5 ^{ix}	90.65 (4)	O1—B1—O2	116.26 (18)
O7 ^{viii} —K2—O5 ^{ix}	84.92 (4)	O3—B1—O2	119.54 (18)
O10 ^v —K2—O3 ^{ix}	154.22 (5)	O1—B1—K2	52.28 (9)
O8 ^{vi} —K2—O3 ^{ix}	118.33 (4)	O3—B1—K2	162.79 (13)
O4 ^{vii} —K2—O3 ^{ix}	78.91 (4)	O2—B1—K2	66.91 (11)
O7 ^{viii} —K2—O3 ^{ix}	124.27 (4)	O1—B1—K2 ^{ix}	123.52 (12)
O5 ^{ix} —K2—O3 ^{ix}	49.06 (3)	O3—B1—K2 ^{ix}	51.40 (9)
O10 ^v —K2—O1	59.69 (4)	O2—B1—K2 ^{ix}	95.47 (12)
O8 ^{vi} —K2—O1	71.95 (4)	K2—B1—K2 ^{ix}	114.13 (6)
O4 ^{vii} —K2—O1	144.45 (4)	O4—B2—O6	111.51 (14)
O7 ^{viii} —K2—O1	133.55 (4)	O4—B2—O3	108.42 (14)
O5 ^{ix} —K2—O1	113.64 (4)	O6—B2—O3	110.00 (15)
O3 ^{ix} —K2—O1	97.25 (4)	O4—B2—O5	110.66 (15)
O10 ^v —K2—O9 ^{vi}	108.20 (4)	O6—B2—O5	109.36 (14)
O8 ^{vi} —K2—O9 ^{vi}	48.87 (4)	O3—B2—O5	106.78 (14)
O4 ^{vii} —K2—O9 ^{vi}	75.56 (4)	O4—B2—K1 ⁱ	55.65 (9)
O7 ^{viii} —K2—O9 ^{vi}	138.95 (4)	O6—B2—K1 ⁱ	133.31 (11)
O5 ^{ix} —K2—O9 ^{vi}	119.08 (4)	O3—B2—K1 ⁱ	116.64 (12)
O3 ^{ix} —K2—O9 ^{vi}	70.02 (4)	O5—B2—K1 ⁱ	55.37 (8)
O1—K2—O9 ^{vi}	70.06 (4)	O4—B2—K2 ^{ix}	103.66 (11)
O10 ^v —K2—O2	71.45 (5)	O6—B2—K2 ^{ix}	144.84 (11)
O8 ^{vi} —K2—O2	115.02 (4)	O3—B2—K2 ^{ix}	56.29 (8)
O4 ^{vii} —K2—O2	161.79 (4)	O5—B2—K2 ^{ix}	55.77 (8)

O7 ^{viii} —K2—O2	115.01 (4)	K1 ⁱ —B2—K2 ^{ix}	68.57 (4)
O5 ^{ix} —K2—O2	73.42 (4)	O5—B3—O8	122.62 (16)
O3 ^{ix} —K2—O2	83.88 (4)	O5—B3—O7	118.18 (18)
O1—K2—O2	43.80 (4)	O8—B3—O7	119.19 (17)
O9 ^{vi} —K2—O2	104.13 (4)	O5—B3—K2 ^{vi}	129.93 (12)
B1—O1—B4	118.49 (15)	O8—B3—K2 ^{vi}	47.92 (9)
B1—O1—K2	106.22 (11)	O7—B3—K2 ^{vi}	92.06 (11)
B4—O1—K2	132.16 (10)	O6—B4—O9	111.38 (14)
B1—O2—K1 ⁱⁱ	126.73 (13)	O6—B4—O1	110.21 (14)
B1—O2—K2	90.06 (12)	O9—B4—O1	109.12 (15)
K1 ⁱⁱ —O2—K2	81.83 (4)	O6—B4—O8	107.72 (14)
B1—O2—H2	109 (2)	O9—B4—O8	109.87 (14)
K1 ⁱⁱ —O2—H2	105 (2)	O1—B4—O8	108.48 (14)
K2—O2—H2	149 (2)	O6—B4—K2 ^{vi}	99.24 (10)
B1—O3—B2	117.57 (15)	O9—B4—K2 ^{vi}	61.95 (9)
B1—O3—K1	121.32 (11)	O1—B4—K2 ^{vi}	150.20 (12)
B2—O3—K1	114.18 (10)	O8—B4—K2 ^{vi}	56.04 (8)
B4—O1—B1—O3	3.3 (3)	B3—O5—B2—O4	145.94 (16)
K2—O1—B1—O3	-159.13 (16)	K1 ⁱ —O5—B2—O4	-6.65 (15)
B4—O1—B1—O2	-176.73 (16)	K2 ^{ix} —O5—B2—O4	-92.79 (13)
K2—O1—B1—O2	20.86 (19)	B3—O5—B2—O6	22.7 (2)
B4—O1—B1—K2	162.41 (17)	K1 ⁱ —O5—B2—O6	-129.86 (12)
B4—O1—B1—K2 ^{ix}	66.24 (18)	K2 ^{ix} —O5—B2—O6	144.00 (11)
K2—O1—B1—K2 ^{ix}	-96.17 (11)	B3—O5—B2—O3	-96.26 (18)
B2—O3—B1—O1	-1.4 (3)	K1 ⁱ —O5—B2—O3	111.15 (12)
K1—O3—B1—O1	-150.56 (14)	K2 ^{ix} —O5—B2—O3	25.01 (14)
K2 ^{ix} —O3—B1—O1	108.18 (18)	B3—O5—B2—K1 ⁱ	152.59 (16)
B2—O3—B1—O2	178.60 (16)	K2 ^{ix} —O5—B2—K1 ⁱ	-86.14 (5)
K1—O3—B1—O2	29.4 (2)	B3—O5—B2—K2 ^{ix}	-121.27 (15)
K2 ^{ix} —O3—B1—O2	-71.82 (19)	K1 ⁱ —O5—B2—K2 ^{ix}	86.14 (5)
B2—O3—B1—K2	-73.6 (5)	B2—O5—B3—O8	8.0 (2)
K1—O3—B1—K2	137.3 (4)	K1 ⁱ —O5—B3—O8	147.81 (13)
K2 ^{ix} —O3—B1—K2	36.0 (5)	K2 ^{ix} —O5—B3—O8	-106.03 (16)
B2—O3—B1—K2 ^{ix}	-109.58 (15)	B2—O5—B3—O7	-171.96 (15)
K1—O3—B1—K2 ^{ix}	101.26 (11)	K1 ⁱ —O5—B3—O7	-32.2 (2)
K1 ⁱⁱ —O2—B1—O1	62.0 (2)	K2 ^{ix} —O5—B3—O7	73.99 (17)
K2—O2—B1—O1	-17.83 (16)	B2—O5—B3—K2 ^{vi}	-51.8 (2)
K1 ⁱⁱ —O2—B1—O3	-118.01 (17)	K1 ⁱ —O5—B3—K2 ^{vi}	87.94 (18)
K2—O2—B1—O3	162.16 (16)	K2 ^{ix} —O5—B3—K2 ^{vi}	-165.90 (8)
K1 ⁱⁱ —O2—B1—K2	79.83 (12)	B4—O8—B3—O5	-4.2 (2)
K1 ⁱⁱ —O2—B1—K2 ^{ix}	-166.25 (9)	K2 ^{vi} —O8—B3—O5	-116.66 (16)
K2—O2—B1—K2 ^{ix}	113.93 (5)	B4—O8—B3—O7	175.75 (15)
K1 ⁱ —O4—B2—O6	128.59 (12)	K2 ^{vi} —O8—B3—O7	63.32 (18)
K2 ^x —O4—B2—O6	-94.2 (5)	B4—O8—B3—K2 ^{vi}	112.44 (15)
K1—O4—B2—O6	-121.41 (13)	K1 ^{xii} —O7—B3—O5	-77.59 (19)
K1 ⁱ —O4—B2—O3	-110.17 (13)	K2 ^{xiii} —O7—B3—O5	36.5 (2)
K2 ^x —O4—B2—O3	27.0 (6)	K1 ^{xii} —O7—B3—O8	102.43 (17)

K1—O4—B2—O3	-0.16 (13)	K2 ^{xiii} —O7—B3—O8	-143.50 (13)
K1 ⁱ —O4—B2—O5	6.62 (14)	K1 ^{xii} —O7—B3—K2 ^{vi}	144.00 (8)
K2 ^x —O4—B2—O5	143.8 (5)	K2 ^{xiii} —O7—B3—K2 ^{vi}	-101.93 (13)
K1—O4—B2—O5	116.63 (12)	B2—O6—B4—O9	-177.72 (14)
K2 ^x —O4—B2—K1 ⁱ	137.2 (5)	K1 ^{xi} —O6—B4—O9	3.91 (14)
K1—O4—B2—K1 ⁱ	110.00 (6)	B2—O6—B4—O1	-56.45 (18)
K1 ⁱ —O4—B2—K2 ^{ix}	-51.57 (8)	K1 ^{xi} —O6—B4—O1	125.17 (12)
K2 ^x —O4—B2—K2 ^{ix}	85.6 (5)	B2—O6—B4—O8	61.73 (17)
K1—O4—B2—K2 ^{ix}	58.43 (6)	K1 ^{xi} —O6—B4—O8	-116.65 (12)
B4—O6—B2—O4	178.63 (13)	B2—O6—B4—K2 ^{vi}	118.90 (12)
K1 ^{xi} —O6—B2—O4	-5.0 (3)	K1 ^{xi} —O6—B4—K2 ^{vi}	-59.48 (6)
B4—O6—B2—O3	58.31 (18)	K1 ^{xi} —O9—B4—O6	-4.49 (16)
K1 ^{xi} —O6—B2—O3	-125.31 (19)	K2 ^{vi} —O9—B4—O6	-89.24 (13)
B4—O6—B2—O5	-58.66 (18)	K1 ^{xi} —O9—B4—O1	-126.38 (11)
K1 ^{xi} —O6—B2—O5	117.7 (2)	K2 ^{vi} —O9—B4—O1	148.87 (11)
B4—O6—B2—K1 ⁱ	-118.89 (15)	K1 ^{xi} —O9—B4—O8	114.80 (12)
K1 ^{xi} —O6—B2—K1 ⁱ	57.5 (3)	K2 ^{vi} —O9—B4—O8	30.05 (13)
B4—O6—B2—K2 ^{ix}	-1.1 (3)	K1 ^{xi} —O9—B4—K2 ^{vi}	84.75 (6)
K1 ^{xi} —O6—B2—K2 ^{ix}	175.28 (8)	B1—O1—B4—O6	25.8 (2)
B1—O3—B2—O4	-151.09 (16)	K2—O1—B4—O6	-177.26 (9)
K1—O3—B2—O4	0.22 (18)	B1—O1—B4—O9	148.38 (15)
K2 ^{ix} —O3—B2—O4	94.41 (13)	K2—O1—B4—O9	-54.66 (19)
B1—O3—B2—O6	-28.9 (2)	B1—O1—B4—O8	-91.93 (18)
K1—O3—B2—O6	122.39 (12)	K2—O1—B4—O8	65.03 (19)
K2 ^{ix} —O3—B2—O6	-143.42 (11)	B1—O1—B4—K2 ^{vi}	-145.0 (2)
B1—O3—B2—O5	89.65 (19)	K2—O1—B4—K2 ^{vi}	12.0 (3)
K1—O3—B2—O5	-119.05 (12)	B3—O8—B4—O6	-30.2 (2)
K2 ^{ix} —O3—B2—O5	-24.85 (14)	K2 ^{vi} —O8—B4—O6	89.31 (12)
B1—O3—B2—K1 ⁱ	148.80 (13)	B3—O8—B4—O9	-151.68 (15)
K1—O3—B2—K1 ⁱ	-59.89 (12)	K2 ^{vi} —O8—B4—O9	-32.19 (14)
K2 ^{ix} —O3—B2—K1 ⁱ	34.30 (10)	B3—O8—B4—O1	89.11 (18)
B1—O3—B2—K2 ^{ix}	114.50 (15)	K2 ^{vi} —O8—B4—O1	-151.40 (11)
K1—O3—B2—K2 ^{ix}	-94.20 (8)	B3—O8—B4—K2 ^{vi}	-119.49 (15)

Symmetry codes: (i) $-x, -y+1, -z+2$; (ii) $-x, -y+1, -z+1$; (iii) $x, y-1, z$; (iv) $x-1, y, z$; (v) $-x+1, -y+1, -z+1$; (vi) $-x+1, -y+2, -z+1$; (vii) $x, y+1, z-1$; (viii) $x, y, z-1$; (ix) $-x, -y+2, -z+1$; (x) $x, y-1, z+1$; (xi) $x+1, y, z$; (xii) $x, y+1, z$; (xiii) $x, y, z+1$.

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O9—H9 \cdots O8 ^{vi}	0.83 (2)	2.02 (2)	2.8385 (18)	173 (2)
O4—H4 \cdots O6 ^{xiv}	0.83 (2)	2.03 (2)	2.8338 (18)	164 (3)
O2—H2 \cdots O9 ^{iv}	0.80 (2)	2.19 (2)	2.957 (2)	161 (3)
O10—H10A \cdots O6	0.86 (2)	1.82 (2)	2.6704 (18)	175 (3)
O10—H10B \cdots O1 ^v	0.85 (2)	2.10 (2)	2.850 (2)	146 (3)
O7—H7 \cdots O10 ^{xii}	0.83 (2)	2.00 (2)	2.823 (2)	169 (3)

Symmetry codes: (iv) $x-1, y, z$; (v) $-x+1, -y+1, -z+1$; (vi) $-x+1, -y+2, -z+1$; (xii) $x, y+1, z$; (xiv) $-x+1, -y+1, -z+2$.