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Redetermination of the borax structure from laboratory X-ray data at 145 K

Graeme J. Gainsford,* Tim Kemmitt and Caleb Higham

Industrial Research Limited, PO Box 31-310, Lower Hutt, New Zealand
Correspondence e-mail: g.gainsford@irl.cri.nz

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Key indicators: single-crystal X-ray study; $T = 145$ K; mean $\sigma(\text{O}-\text{B}) = 0.001$ Å;
 R factor = 0.025; wR factor = 0.077; data-to-parameter ratio = 15.5.

The title compound, sodium tetraborate decahydrate (mineral name: borax), $\text{Na}_2[\text{B}_4\text{O}_5(\text{OH})_4] \cdot 8\text{H}_2\text{O}$, has been studied previously using X-ray [Morimoto (1956). *Miner. J.* **2**, 1–18] and neutron [Levy & Lisensky (1978). *Acta Cryst.* **B34**, 3502–3510] diffraction data. The structure contains tetraborate anions $[\text{B}_4\text{O}_5(\text{OH})_4]^{2-}$ with twofold rotation symmetry, which form hydrogen-bonded chains, and $[\text{Na}(\text{H}_2\text{O})_6]$ octahedra that form zigzag chains $[\text{Na}(\text{H}_2\text{O})_{4/2}(\text{H}_2\text{O})_{2/1}]$. The O–H bond distances obtained from the present redetermination at 145 K are shorter than those in the neutron study by an average of 0.127 (19) Å.

Related literature

For previous studies of the borax structure, see: Morimoto (1956); Levy & Lisensky (1978). For other structures listed in the Cambridge Structural Database (Allen, 2002) that contain the $[\text{B}_4\text{O}_5(\text{OH})_4]^{2-}$ anion, see: Wang *et al.* (2004); Pan *et al.* (2007). For related structures, see: Yi *et al.* (2005). For comparative studies of hydrogen bonds obtained from X-ray and neutron data, see: Allen (1986); Smrčok *et al.* (2006).

Experimental

Crystal data

| | |
|----------------------------------------------------------------------------|-----------------------------------|
| $\text{Na}_2[\text{B}_4\text{O}_5(\text{OH})_4] \cdot 8\text{H}_2\text{O}$ | $V = 1473.06$ (10) Å ³ |
| $M_r = 381.38$ | $Z = 4$ |
| Monoclinic, $C2/c$ | Mo $K\alpha$ radiation |
| $a = 11.8843$ (5) Å | $\mu = 0.22$ mm ⁻¹ |
| $b = 10.6026$ (4) Å | $T = 145$ (2) K |
| $c = 12.2111$ (5) Å | $0.65 \times 0.36 \times 0.26$ mm |
| $\beta = 106.790$ (2)° | |

Data collection

| | |
|----------------------------------------------------------|----------------------------------------|
| Bruker–Nonius APEX2 CCD area-detector diffractometer | 8429 measured reflections |
| Absorption correction: multi-scan (SADABS; Bruker, 2006) | 2275 independent reflections |
| $T_{\min} = 0.813$, $T_{\max} = 0.94$ | 2137 reflections with $I > 2\sigma(I)$ |
| | $R_{\text{int}} = 0.018$ |

Refinement

| | |
|---------------------------------|-----------------------------------------------------|
| $R[F^2 > 2\sigma(F^2)] = 0.025$ | 147 parameters |
| $wR(F^2) = 0.076$ | All H-atom parameters refined |
| $S = 1.08$ | $\Delta\rho_{\text{max}} = 0.37$ e Å ⁻³ |
| 2275 reflections | $\Delta\rho_{\text{min}} = -0.22$ e Å ⁻³ |

Table 1

Selected bond lengths (Å).

| | | | |
|-----------------------|------------|--------------------|------------|
| Na1–O8 ⁱ | 2.3815 (6) | B1–O1 | 1.4657 (7) |
| Na1–O6 ⁱⁱ | 2.3979 (5) | B1–O2 | 1.4902 (8) |
| Na1–O7 | 2.4121 (6) | B1–O3 | 1.5075 (8) |
| Na2–O7 ⁱⁱⁱ | 2.4041 (6) | B2–O2 | 1.3655 (8) |
| Na2–O9 | 2.4214 (6) | B2–O3 ⁱ | 1.3757 (8) |
| Na2–O6 | 2.4441 (6) | B2–O5 | 1.3784 (8) |
| B1–O4 | 1.4451 (8) | | |

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

Table 2

Hydrogen-bond geometry (Å, °).

| $D-H \cdots A$ | $D-H$ | $H \cdots A$ | $D \cdots A$ | $D-H \cdots A$ |
|----------------------------------------------|------------|--------------|--------------|----------------|
| O5–H5 ^{iv} ···O3 ^{iv} | 0.836 (15) | 1.895 (15) | 2.7300 (7) | 176.3 (15) |
| O4 ^v –H4···O9 ^{vi} | 0.828 (14) | 2.049 (14) | 2.8658 (8) | 168.4 (12) |
| O6–H6A ^{vii} ···O5 ^{viii} | 0.868 (16) | 1.978 (16) | 2.8323 (8) | 167.9 (14) |
| O6 ^{ix} –H6B···O4 ^v | 0.846 (16) | 2.040 (15) | 2.8624 (8) | 163.9 (16) |
| O7 ^{vii} –H7A···O2 | 0.827 (16) | 1.989 (16) | 2.8135 (8) | 174.1 (12) |
| O7–H7B···O4 | 0.816 (15) | 2.135 (15) | 2.9233 (8) | 162.3 (14) |
| O8 ^v –H8A···O1 ^x | 0.866 (13) | 1.936 (13) | 2.7865 (6) | 167.0 (14) |
| O8 ^v –H8B···O5 ^{xi} | 0.855 (15) | 2.341 (14) | 3.1320 (8) | 154.2 (12) |
| O9–H9A ^{xii} ···O3 | 0.843 (16) | 2.253 (16) | 3.0894 (8) | 171.7 (15) |
| O9–H9B ^{xiii} ···O8 ^{xiii} | 0.849 (17) | 2.069 (16) | 2.9034 (8) | 167.4 (15) |

Symmetry codes: (iv) $x, -y + 1, z + \frac{1}{2}$; (v) $-x + 1, y, -z + \frac{1}{2}$; (vi) $x + \frac{1}{2}, y - \frac{1}{2}, z$; (vii) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$; (viii) $-x + \frac{1}{2}, -y + \frac{1}{2}, -z + 1$; (ix) $-x + 1, -y + 1, -z + 1$; (x) $x + 1, y, z$; (xi) $x + \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$; (xii) $x - \frac{1}{2}, y + \frac{1}{2}, z$; (xiii) $x, -y + 1, z - \frac{1}{2}$.

Data collection: APEX2 (Bruker, 2006); cell refinement: APEX2; data reduction: APEX2; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997) and Mercury (Macrae *et al.*, 2006); software used to prepare material for publication: SHELXL97 and PLATON (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2174).

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supporting information

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S1. Comment

The crystal structure of the title compound was previously studied by Morimoto (1956) using X-ray and later by Levy & Lisensky (1978, hereafter LL) using neutron diffraction data.

There are 8 other compounds with free tetraborate anions $[\text{B}_4\text{O}_5(\text{OH})_4]^{2-}$ reported in the Cambridge Structural Database [C.S.D., version 5.29 with November 2007 updates (Allen, 2002)] with most containing protonated amine-based cations, *e.g.* DALQEN (Wang *et al.*, 2004) and SIBDIR (Pan *et al.*, 2007). The tetraborate anion in borax has 2-fold symmetry with the axis passing through O1 (Fig. 1) as is observed in five of the related structures. Both Na1 and Na2 cations are on special positions (centre of symmetry and 2-fold axis, respectively) so that they elegantly bind *via* shared water molecules in a typical zigzag cationic chain $[\text{Na}(\text{H}_2\text{O})_{4/2}(\text{H}_2\text{O})_{2/1}]$ parallel to the *c* axis (*e.g.* DARNOA, Yi *et al.*, 2005), as shown in Figure 2. As is found through a C.S.D. search of similar $\text{Na}^+/\text{H}_2\text{O}$ cation chains, the Na–O distances to the bridging water molecules are longer than those to non-bridging water molecules, where the *trans* related Na–O distances belong to non-bridging water molecules.

The results of the present study and the LL model are essentially superimposable, but do reflect expected differences associated with the H atom positions: The systematic pairwise study (Allen, 1986) gave a difference for O–H (X-ray versus neutron) of -0.155 (10) Å, while a more recent study of levoglucosan (Smrčok *et al.*, 2006) averaged at -0.016 (6) Å. The mean O–H distance here (0.843 (17) Å) is significantly shorter than for the neutron set (0.97 (1) Å). As the O···O distances involved in the hydrogen bonding are very similar for both studies (Table 1), the observed H···O distances are correspondingly longer here than in the LL model. We also note that average Na–O distances are marginally longer (0.006 (6) Å) and the B–O distances marginally shorter (-0.005 (2) Å) in the LL model, *e.g.* Na–O6, B1–O2 are 2.458 (3), 1.500 (2) Å compared with 2.4441 (6), 1.5075 (8) Å, respectively, in the present study. These latter differences are barely significant given that the neutron data set was collected at 296.5 K.

Cell cohesion is provided by strong O–H···O hydrogen bonds of two types: (1) tetraborate anions "head to tail" link *via* the O5–H and O2 atoms (entry 1, Table 1) to form anionic chains as also seen in DALQEN (Wang *et al.*, 2004); (2) the anionic and cationic chains crosslink through the water & tetraborate strong O–H···O hydrogen bond interactions (entries 2–10; see also Fig. 2 and diagrams in the LL study).

S2. Experimental

To a tetrahydrofuran (thf) solution (90 ml) of sodium tetrahydridoborate (0.31 g, 8.4 mmol) was added 0.5 g (4.2 mmol) of diaminomethane dihydrochloride. After 24 h, the solvent was removed and the remaining product dissolved in water. Methanol was added and the solution was left in a refrigerator. A small clump of colourless crystals of the title compound appeared after several days in the bottom of the flask.

S3. Refinement

A total of 13 reflections (below $50^\circ/2\theta$) were not collected. In the present re-determination the same atomic labels and atomic coordinates have been used as in the previous studies (Morimoto, 1956; Levy & Lisensky, 1978). The positions of the H atoms were fully refined with isotropic thermal parameters for each H atom.

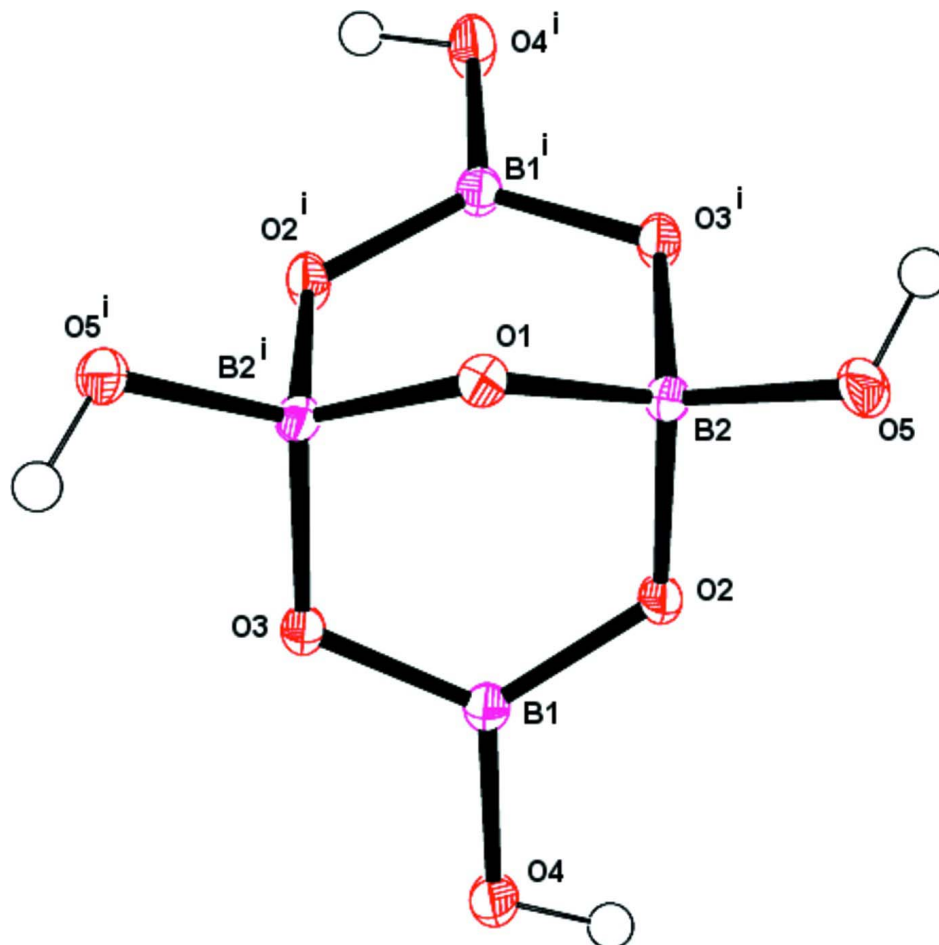


Figure 1

Molecular structure of the tetraborate anion shown with displacement ellipsoids at the 50% probability level. [Symmetry code: i) $-x, y, 1/2 - z$.]

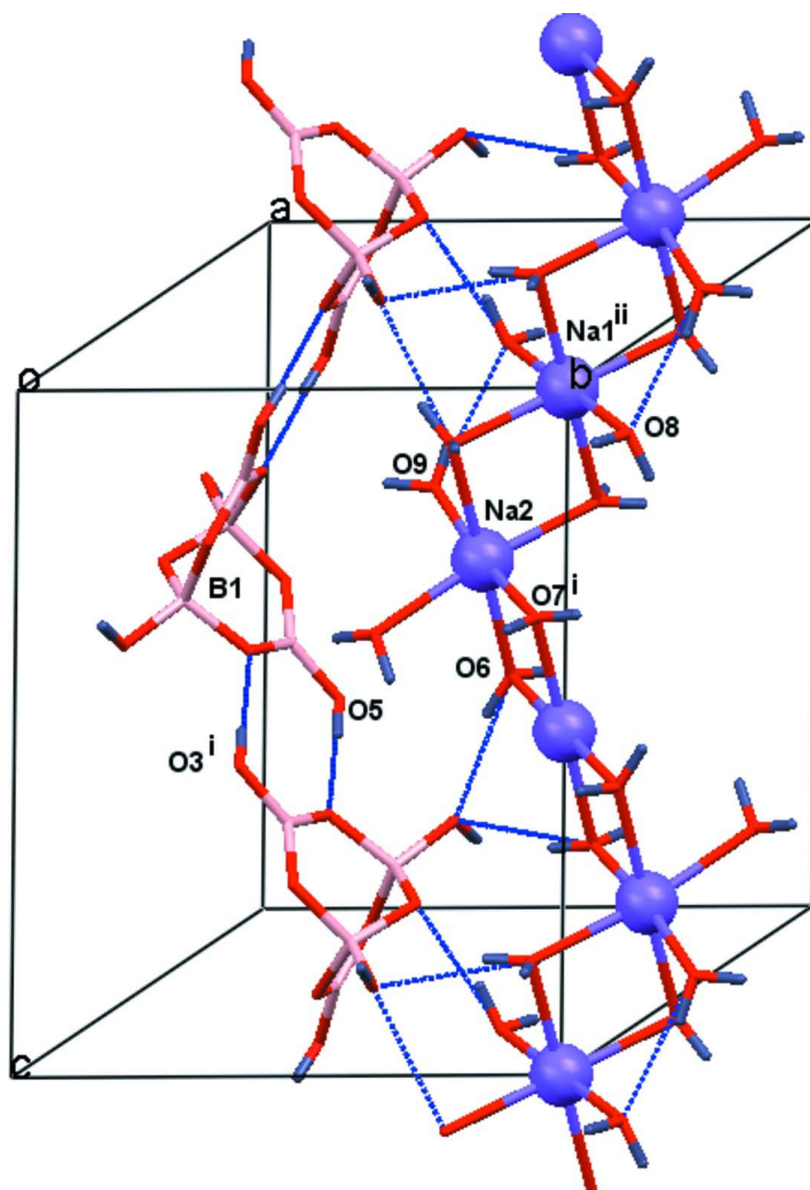


Figure 2

Part of the crystal structure showing the zigzag $[\text{Na}(\text{H}_2\text{O})_{4/2}(\text{H}_2\text{O})_{2/1}]$ chain, the hydrogen bonded tetraborate chain and some interlinking hydrogen bonds, shown as dashed lines. For clarity, only selected atoms and one of each chain is shown. [Symmetry codes: i) $-x, y + 1/2, -z$; ii) $x, 1 + y, z$.]

sodium tetraborate decahydrate

Crystal data

$\text{Na}_2[\text{B}_4\text{O}_5(\text{OH})_4] \cdot 8\text{H}_2\text{O}$

$M_r = 381.38$

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 11.8843\ (5)\ \text{\AA}$

$b = 10.6026\ (4)\ \text{\AA}$

$c = 12.2111\ (5)\ \text{\AA}$

$\beta = 106.790\ (2)^\circ$

$V = 1473.06\ (10)\ \text{\AA}^3$

$Z = 4$

$F(000) = 792$

$D_x = 1.720\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 6688 reflections

$\theta = 2.6\text{--}31.9^\circ$
 $\mu = 0.22\text{ mm}^{-1}$
 $T = 145\text{ K}$

Prism, colourless
 $0.65 \times 0.36 \times 0.26\text{ mm}$

Data collection

Bruker–Nonius APEX2 CCD area-detector
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 Detector resolution: $8.192\text{ pixels mm}^{-1}$
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2006)
 $T_{\min} = 0.813$, $T_{\max} = 0.94$

8429 measured reflections
 2275 independent reflections
 2137 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.018$
 $\theta_{\max} = 33.2^\circ$, $\theta_{\min} = 2.6^\circ$
 $h = -17 \rightarrow 16$
 $k = -15 \rightarrow 15$
 $l = -16 \rightarrow 17$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.025$
 $wR(F^2) = 0.076$
 $S = 1.08$
 2275 reflections
 147 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 All H-atom parameters refined
 $w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 0.3901P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.37\text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.22\text{ e \AA}^{-3}$

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. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|-------------|-------------|----------------------------------|
| Na1 | 0.0000 | 0.0000 | 0.0000 | 0.01676 (10) |
| Na2 | 0.0000 | 0.84795 (4) | 0.2500 | 0.01796 (11) |
| B1 | 0.08552 (6) | 0.34499 (6) | 0.21553 (5) | 0.01044 (13) |
| B2 | 0.09847 (6) | 0.45643 (6) | 0.39269 (6) | 0.01154 (13) |
| O1 | 0.0000 | 0.26659 (6) | 0.2500 | 0.01094 (13) |
| O2 | 0.15546 (4) | 0.41927 (4) | 0.31574 (4) | 0.01276 (11) |
| O3 | 0.01964 (4) | 0.43573 (4) | 0.12445 (4) | 0.01339 (11) |
| O4 | 0.16140 (4) | 0.27014 (5) | 0.16772 (4) | 0.01570 (11) |
| O5 | 0.16369 (4) | 0.51522 (5) | 0.49130 (4) | 0.01815 (12) |
| O6 | 0.12357 (5) | 0.84607 (5) | 0.44846 (5) | 0.01851 (12) |
| O7 | 0.12296 (5) | 0.00117 (5) | 0.19548 (5) | 0.01799 (12) |
| O8 | 0.11919 (5) | 0.16556 (5) | 0.46252 (5) | 0.02067 (12) |
| O9 | 0.11746 (5) | 0.70654 (6) | 0.17227 (5) | 0.02187 (12) |

| | | | | |
|-----|-------------|-------------|-------------|------------|
| H4 | 0.7717 (12) | 0.2622 (11) | 0.2876 (12) | 0.032 (3)* |
| H5 | 0.1187 (13) | 0.4667 (13) | 0.0305 (12) | 0.040 (3)* |
| H6A | 0.3089 (13) | 0.3828 (14) | 0.0413 (12) | 0.042 (4)* |
| H6B | 0.8662 (14) | 0.2018 (16) | 0.4941 (13) | 0.051 (4)* |
| H7A | 0.3098 (13) | 0.4817 (11) | 0.3051 (12) | 0.030 (3)* |
| H7B | 0.1304 (12) | 0.0776 (14) | 0.2014 (12) | 0.039 (3)* |
| H8A | 0.9099 (12) | 0.1906 (13) | 0.1075 (11) | 0.036 (3)* |
| H8B | 0.8131 (12) | 0.1365 (12) | 0.0352 (11) | 0.034 (3)* |
| H9A | 0.4018 (13) | 0.1300 (15) | 0.3385 (12) | 0.046 (4)* |
| H9B | 0.6140 (15) | 0.2331 (15) | 0.1058 (14) | 0.053 (4)* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|------------|--------------|------------|---------------|--------------|---------------|
| Na1 | 0.0165 (2) | 0.01812 (19) | 0.0158 (2) | 0.00057 (13) | 0.00485 (15) | 0.00047 (13) |
| Na2 | 0.0179 (2) | 0.0201 (2) | 0.0176 (2) | 0.000 | 0.00773 (16) | 0.000 |
| B1 | 0.0100 (3) | 0.0121 (3) | 0.0096 (3) | 0.0012 (2) | 0.0035 (2) | -0.00028 (19) |
| B2 | 0.0108 (3) | 0.0133 (3) | 0.0111 (3) | -0.0014 (2) | 0.0040 (2) | -0.0014 (2) |
| O1 | 0.0117 (3) | 0.0104 (3) | 0.0111 (3) | 0.000 | 0.0037 (2) | 0.000 |
| O2 | 0.0103 (2) | 0.0168 (2) | 0.0122 (2) | -0.00223 (15) | 0.00493 (16) | -0.00376 (15) |
| O3 | 0.0103 (2) | 0.0180 (2) | 0.0130 (2) | 0.00309 (15) | 0.00519 (16) | 0.00559 (15) |
| O4 | 0.0119 (2) | 0.0209 (2) | 0.0142 (2) | 0.00489 (17) | 0.00365 (17) | -0.00434 (16) |
| O5 | 0.0132 (2) | 0.0275 (3) | 0.0150 (2) | -0.00595 (18) | 0.00593 (19) | -0.00949 (18) |
| O6 | 0.0146 (2) | 0.0219 (2) | 0.0186 (2) | -0.00109 (18) | 0.00414 (19) | 0.00229 (18) |
| O7 | 0.0151 (3) | 0.0160 (2) | 0.0232 (3) | 0.00012 (17) | 0.0061 (2) | 0.00003 (17) |
| O8 | 0.0185 (3) | 0.0242 (3) | 0.0184 (3) | 0.00116 (19) | 0.0039 (2) | 0.00608 (19) |
| O9 | 0.0217 (3) | 0.0196 (2) | 0.0229 (3) | -0.00047 (19) | 0.0041 (2) | -0.00352 (19) |

Geometric parameters (Å, °)

| | | | |
|-----------------------------------------|-------------|----------------------------------------|-------------|
| Na1—O8 ⁱ | 2.3815 (6) | Na2—O6 ⁱⁱ | 2.4441 (6) |
| Na1—O8 ⁱⁱ | 2.3815 (6) | Na2—O6 | 2.4441 (6) |
| Na1—O6 ⁱⁱⁱ | 2.3979 (5) | B1—O4 | 1.4451 (8) |
| Na1—O6 ^{iv} | 2.3979 (5) | B1—O1 | 1.4657 (7) |
| Na1—O7 ^v | 2.4121 (6) | B1—O2 | 1.4902 (8) |
| Na1—O7 | 2.4121 (6) | B1—O3 | 1.5075 (8) |
| Na2—O7 ^{vi} | 2.4041 (6) | B2—O2 | 1.3655 (8) |
| Na2—O7 ^{vii} | 2.4041 (6) | B2—O3 ⁱⁱ | 1.3757 (8) |
| Na2—O9 | 2.4214 (6) | B2—O5 | 1.3784 (8) |
| Na2—O9 ⁱⁱ | 2.4214 (6) | | |
| O8 ⁱ —Na1—O8 ⁱⁱ | 180.00 (2) | O9—Na2—O6 ⁱⁱ | 81.696 (19) |
| O8 ⁱ —Na1—O6 ⁱⁱⁱ | 90.45 (2) | O9 ⁱⁱ —Na2—O6 ⁱⁱ | 97.72 (2) |
| O8 ⁱⁱ —Na1—O6 ⁱⁱⁱ | 89.55 (2) | O7 ^{vi} —Na2—O6 | 88.29 (2) |
| O8 ⁱ —Na1—O6 ^{iv} | 89.55 (2) | O7 ^{vii} —Na2—O6 | 92.34 (2) |
| O8 ⁱⁱ —Na1—O6 ^{iv} | 90.45 (2) | O9—Na2—O6 | 97.72 (2) |
| O6 ⁱⁱⁱ —Na1—O6 ^{iv} | 180.00 (3) | O9 ⁱⁱ —Na2—O6 | 81.70 (2) |
| O8 ⁱ —Na1—O7 ^v | 91.717 (19) | O6 ⁱⁱ —Na2—O6 | 179.07 (3) |

| | | | |
|-----------------------------------------|--------------|-----------------------------------------------|---------------|
| O8 ⁱⁱ —Na1—O7 ^v | 88.283 (19) | O4—B1—O1 | 111.72 (5) |
| O6 ⁱⁱⁱ —Na1—O7 ^v | 89.177 (19) | O4—B1—O2 | 110.91 (5) |
| O6 ^{iv} —Na1—O7 ^v | 90.823 (19) | O1—B1—O2 | 109.42 (4) |
| O8 ⁱ —Na1—O7 | 88.283 (19) | O4—B1—O3 | 107.71 (5) |
| O8 ⁱⁱ —Na1—O7 | 91.717 (19) | O1—B1—O3 | 108.56 (5) |
| O6 ⁱⁱⁱ —Na1—O7 | 90.823 (19) | O2—B1—O3 | 108.43 (5) |
| O6 ^{iv} —Na1—O7 | 89.177 (19) | O2—B2—O3 ⁱⁱ | 122.44 (6) |
| O7 ^v —Na1—O7 | 180.000 (16) | O2—B2—O5 | 117.78 (6) |
| O7 ^{vi} —Na2—O7 ^{vii} | 94.98 (3) | O3 ⁱⁱ —B2—O5 | 119.78 (6) |
| O7 ^{vi} —Na2—O9 | 172.90 (2) | B1 ⁱⁱ —O1—B1 | 110.90 (6) |
| O7 ^{vii} —Na2—O9 | 81.05 (2) | B2—O2—B1 | 116.59 (5) |
| O7 ^{vi} —Na2—O9 ⁱⁱ | 81.05 (2) | B2 ⁱⁱ —O3—B1 | 120.25 (5) |
| O7 ^{vii} —Na2—O9 ⁱⁱ | 172.90 (2) | Na1 ^{vi} —O6—Na2 | 90.952 (19) |
| O9—Na2—O9 ⁱⁱ | 103.49 (3) | Na2 ^{viii} —O7—Na1 | 91.581 (19) |
| O7 ^{vi} —Na2—O6 ⁱⁱ | 92.34 (2) | Na2 ^{viii} —O7—H7B | 134.8 (10) |
| O7 ^{vii} —Na2—O6 ⁱⁱ | 88.29 (2) | Na1—O7—H7B | 96.4 (10) |
| O4—B1—O1—B1 ⁱⁱ | -172.74 (6) | O7 ^{vi} —Na2—O6—Na1 ^{vi} | -0.355 (19) |
| O2—B1—O1—B1 ⁱⁱ | 64.05 (4) | O7 ^{vii} —Na2—O6—Na1 ^{vi} | 94.56 (2) |
| O3—B1—O1—B1 ⁱⁱ | -54.12 (3) | O9—Na2—O6—Na1 ^{vi} | 175.84 (2) |
| O3 ⁱⁱ —B2—O2—B1 | -4.91 (9) | O9 ⁱⁱ —Na2—O6—Na1 ^{vi} | -81.56 (2) |
| O5—B2—O2—B1 | 174.23 (5) | O6 ⁱⁱ —Na2—O6—Na1 ^{vi} | -132.911 (15) |
| O4—B1—O2—B2 | -156.88 (5) | O8 ⁱ —Na1—O7—Na2 ^{viii} | -89.93 (2) |
| O1—B1—O2—B2 | -33.19 (7) | O8 ⁱⁱ —Na1—O7—Na2 ^{viii} | 90.07 (2) |
| O3—B1—O2—B2 | 85.06 (6) | O6 ⁱⁱⁱ —Na1—O7—Na2 ^{viii} | 179.640 (19) |
| O4—B1—O3—B2 ⁱⁱ | 136.95 (6) | O6 ^{iv} —Na1—O7—Na2 ^{viii} | -0.360 (19) |
| O1—B1—O3—B2 ⁱⁱ | 15.82 (7) | O7 ^v —Na1—O7—Na2 ^{viii} | -122 (44) |
| O2—B1—O3—B2 ⁱⁱ | -102.97 (6) | | |

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

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------------------------------------|------------|-------------|-------------|---------------|
| O5—H5 ^{ix} ...O3 ^{ix} | 0.836 (15) | 1.895 (15) | 2.7300 (7) | 176.3 (15) |
| O4 ^x —H4...O9 ^{xi} | 0.828 (14) | 2.049 (14) | 2.8658 (8) | 168.4 (12) |
| O6—H6A ^{xii} ...O5 ^{xiii} | 0.868 (16) | 1.978 (16) | 2.8323 (8) | 167.9 (14) |
| O6 ^{xiv} —H6B...O4 ^x | 0.846 (16) | 2.040 (15) | 2.8624 (8) | 163.9 (16) |
| O7 ^{xii} —H7A...O2 | 0.827 (16) | 1.989 (16) | 2.8135 (8) | 174.1 (12) |
| O7—H7B...O4 | 0.816 (15) | 2.135 (15) | 2.9233 (8) | 162.3 (14) |
| O8 ^x —H8A...O1 ^{xv} | 0.866 (13) | 1.936 (13) | 2.7865 (6) | 167.0 (14) |
| O8 ^x —H8B...O5 ^{xvi} | 0.855 (15) | 2.341 (14) | 3.1320 (8) | 154.2 (12) |
| O9—H9A ^{xii} ...O3 | 0.843 (16) | 2.253 (16) | 3.0894 (8) | 171.7 (15) |
| O9—H9B ^{xvii} ...O8 ⁱⁱⁱ | 0.849 (17) | 2.069 (16) | 2.9034 (8) | 167.4 (15) |

Symmetry codes: (iii) $x, -y+1, z-1/2$; (ix) $x, -y+1, z+1/2$; (x) $-x+1, y, -z+1/2$; (xi) $x+1/2, y-1/2, z$; (xii) $-x+1/2, y+1/2, -z+1/2$; (xiii) $-x+1/2, -y+3/2, -z+1$; (xiv) $-x+1, -y+1, -z+1$; (xv) $x+1, y, z$; (xvi) $x+1/2, -y+1/2, z-1/2$; (xvii) $x-1/2, y+1/2, z$.