Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis(ethylenediammonium) tetradecaborate

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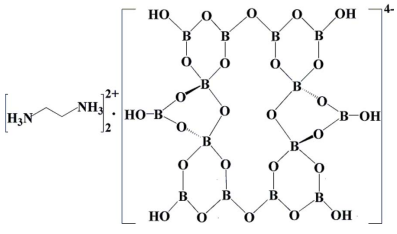
Received 25 February 2010; accepted 5 March 2010

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.043; wR factor = 0.107; data-to-parameter ratio = 11.7.

The title compound, $2C_2H_{10}N_2^{2+}\cdot B_{14}O_{20}(OH)_6^{4-}$, consists of a centrosymmetric tetradecaborate anion and two ethylenediammonium cations. The anions are interconnected through strong O—H···O hydrogen bonds into a three-dimensional supramolecular network with channels along [100], [010], [001] and [111]. The diprotonated cations reside in the channels and interact with the inorganic framework by extensive N—H···O hydrogen bonds.

Related literature

For general background to the structures and applications of inorganic borates, see: Burns *et al.* (1995); Chen *et al.* (1995); Grice *et al.* (1999); Touboul *et al.* (2003); Wang *et al.* (2007). For some typical examples of organically templated non-metal borates, see: Li *et al.* (2008); Liu *et al.* (2006); Pan *et al.* (2007); Wang *et al.* (2004). For two typical examples of crystalline aluminoborates, see: Wang *et al.* (2008*a*,*b*).



Experimental

Crystal data

$2C_2H_{10}N_2^{2+}\cdot B_{14}H_6O_{26}^{4-}$
M_r = 697.63
Triclinic, $P\overline{1}$
a = 8.4849 (3) Å
b = 8.8387 (3) Å
c = 10.0406 (2) Å
α = 95.085 (2)°
β = 96.942 (3)°
γ = 116.856 (4)°
V = 658.08 (3) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.16 mm^{-1}
T = 293 K
0.28 × 0.13 × 0.04 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.956, T_{max} = 0.994
5101 measured reflections
2541 independent reflections
2033 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.107
S = 1.03
2541 reflections
217 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1F···O8^{i}	0.82	2.11	2.909 (2)	165
O8—H8A···O9^{ii}	0.82	1.83	2.6433 (19)	176
O13—H13A···O7^{iii}	0.82	1.79	2.6030 (18)	172
N1—H1C···O13^{iv}	0.89	1.87	2.755 (2)	172
N1—H1D···O10^{v}	0.89	2.04	2.919 (2)	168
N1—H1E···O2^{v}	0.89	2.09	2.892 (2)	150
N2—H2C···O6^{vi}	0.89	1.89	2.777 (2)	174
N2—H2D···O1^{vii}	0.89	2.18	2.926 (2)	141
N2—H2E···O5^{iii}	0.89	2.08	2.951 (2)	168

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT-Plus* (Bruker, 2007); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXTL*.

This work was supported by the National Natural Science Foundation of China (No. 20901043), the Young Scientist Foundation of Shandong Province (No. BS2009CL041) and the Qingdao University Research Fund (No. 063-06300522).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2288).

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supporting information

Acta Cryst. (2010). E**66**, o798–o799 [doi:10.1107/S1600536810008494]

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S1. Comment

Borate materials have attracted considerable attention in the past decades owing to their fascinating structural diversities and promising applications in mineralogy, luminescence and nonlinear optical properties (Burns *et al.*, 1995; Chen *et al.*, 1995; Grice *et al.*, 1999; Touboul *et al.*, 2003; Wang *et al.*, 2007). From a structural chemistry point of view, the ability of boron to adopt both BO_4 and BO_3 coordination modes, coupled with the tendency of such units to polymerize into a large range of polyanions, has made inorganic borates into a rapidly growing family. To date, borate materials with various alkali metal, alkaline earth metal, rare earth and transition metal, traditionally prepared under high temperature/pressure solid-state conditions, have been extensively studied. In contrast, the template synthesis of nonmetal borates is still a relatively undeveloped area. Recently, solvothermal method has been proved to be very effective in isolating such borates by employing various organic molecules as templates or structure-directing agents (Li *et al.*, 2008; Liu *et al.*, 2006; Pan *et al.*, 2007; Wang *et al.*, 2004). Our interest is to explore the introduction of aluminium into borate system, constructing novel microporous aluminoborate materials templated by organic agents with different shape and size (Wang *et al.*, 2008a, b). Interestingly, the title compound was obtained, which is a new organically templated nonmetal tetradecaborate.

As shown in Fig. 1, the asymmetric unit of the title compound consists of one $[B_7O_{10}(OH)_3]^{2-}$ anionic unit and one $[C_2H_{10}N_2]^{2+}$ cation. The anionic unit is composed of two BO_4 tetrahedra [B3 and B5], two BO_3 [B2 and B6] and three $BO_2(OH)$ [B1, B4 and B7] trigonal units, which forms three classic B_3O_3 cycles linked by two common BO_4 tetrahedra. Two such $[B_7O_{10}(OH)_3]^{2-}$ units are further jointed together through the exocyclic O atoms [O4 and O4^{i}, symmetry code: (i) -x, -y, 2-z], generating the FBBs (Fundamental Building Blocks), a large isolated $[B_{14}O_{20}(OH)_6]^{4-}$ polyanion. Thus, the borate FBBs, featuring one cyclic 8-membered ring (MR) and six 3-MRs, is made up of four BO_4 and ten BO_3 and $BO_2(OH)$ units. The B—O bond distances lie in the range 1.337 (3)–1.386 (3) Å for the BO_3 triangles (av. 1.361 Å) and 1.430 (3)–1.489 (3) Å for the BO_4 tetrahedra (av. 1.466 Å), in good agreement with those reported previously for other borate materials. The O—B—O bond angles of the BO_4 tetrahedra lie in the range of 106.7 (2)–112.6 (2)° and those of the BO_3 triangles span from 115.4 (2) to 123.4 (2)°; the averages for the corresponding angles are very close to 109.5 and 120°, respectively.

The FBBs, $[B_{14}O_{20}(OH)_6]^{4-}$, are connected with each other through strong intermolecular O—H···O hydrogen bonds (Table 1), forming a three-dimensional framework with channels along [100], [010], [001] and [111] directions. The diprotonated $[C_2H_{10}N_2]^{2+}$ cations reside in the channels, interacting with the framework through N—H···O hydrogen bonds (Fig. 2).

S2. Experimental

A mixture of H_3BO_3 (0.217 g), Al_2O_3 (0.104 g), ethylenediamine (0.42 ml), pyridine (5.0 ml) and H_2O (0.90 ml) was sealed in a Teflon-lined steel autoclave, heated at 443 K for 10 d, and then cooled to room temperature. The colorless prism-shaped crystals were separated from the solution by filtration, washed with distilled water and dried in air.

S3. Refinement

All H atoms were positioned geometrically and treated as riding atoms, with O—H = 0.82, N—H = 0.89 and C—H = 0.97 Å and with U_{iso}(H) = 1.2U_{eq}(C,N) or 1.5U_{eq}(O).

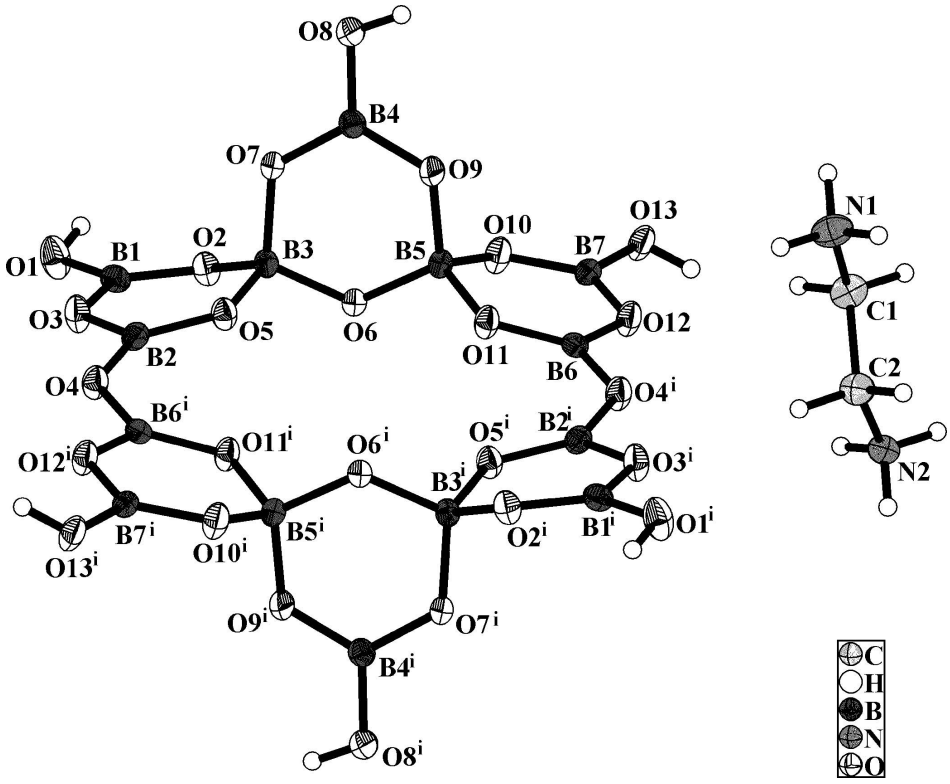

Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (i) -x, -y, 2-z.]

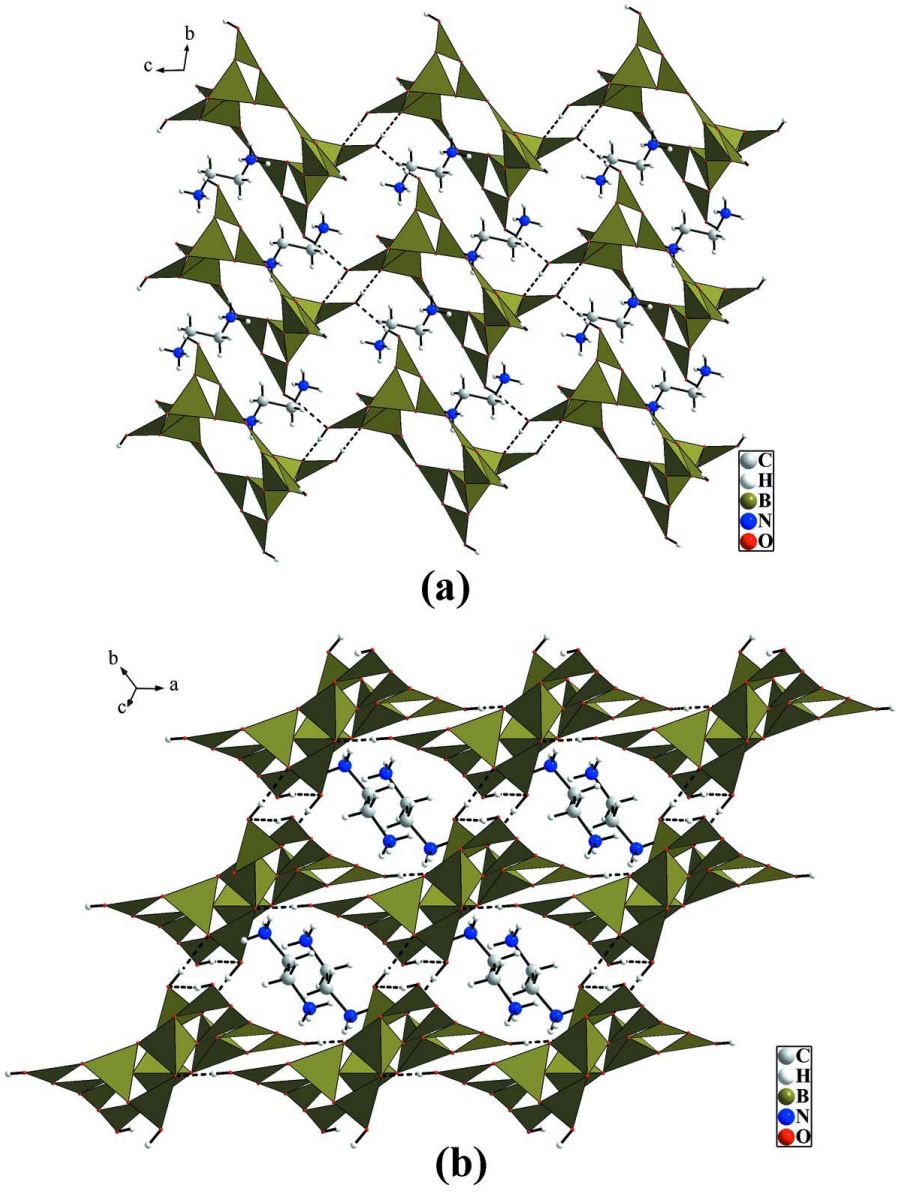

Figure 2

Different views (a) along [100] and (b) along [111] of the three-dimensional framework constructed from $[B_{14}O_{20}(OH)_6]^{4-}$ anions, with $[C_2H_{10}N_2]^{2+}$ cations occupying channels. Hydrogen bonds are shown as dashed lines.

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Crystal data

$2C_2H_{10}N_2^{2+}\cdot B_{14}H_6O_{26}^{4-}$
M_r = 697.63
Triclinic, $P\overline{1}$
Hall symbol: -P 1
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b = 8.8387 (3) Å
c = 10.0406 (2) Å
α = 95.085 (2)°
β = 96.942 (3)°
γ = 116.856 (4)°
V = 658.08 (3) Å^3
Z = 1
$F(000)$ = 356
D_x = 1.760 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5101 reflections
θ = 2.1–26.0°
μ = 0.16 mm^{-1}

T = 293 K
Prism, colorless

0.28 × 0.13 × 0.04 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.956, T_{max} = 0.994

5101 measured reflections
2541 independent reflections
2033 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 26.0°, θ_{min} = 2.1°
h = −10→10
k = −10→10
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.107
S = 1.03
2541 reflections
217 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
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0518P)^2 + 0.1264P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
B1	−0.5849 (3)	−0.5170 (3)	0.8129 (2)	0.0190 (5)
B2	−0.4605 (3)	−0.2456 (3)	0.9547 (2)	0.0180 (5)
B3	−0.2914 (3)	−0.2948 (3)	0.7854 (2)	0.0166 (5)
B4	−0.2007 (3)	−0.1473 (3)	0.5881 (2)	0.0166 (5)
B5	0.0303 (3)	−0.1771 (3)	0.7443 (2)	0.0175 (5)
B6	0.3485 (3)	0.0104 (3)	0.8520 (2)	0.0174 (5)
B7	0.2775 (3)	−0.2278 (3)	0.6835 (2)	0.0189 (5)
O1	−0.73216 (19)	−0.67207 (18)	0.76829 (15)	0.0286 (4)
H1F	−0.7172	−0.7203	0.7011	0.043*
O2	−0.42739 (18)	−0.47558 (17)	0.77218 (13)	0.0203 (3)
O3	−0.60553 (17)	−0.40525 (18)	0.90605 (14)	0.0232 (3)
O4	−0.48111 (18)	−0.15100 (18)	1.06075 (14)	0.0211 (3)
O5	−0.31106 (17)	−0.18999 (17)	0.90041 (13)	0.0188 (3)
O6	−0.11436 (17)	−0.27927 (17)	0.80925 (13)	0.0175 (3)
O7	−0.32891 (17)	−0.23425 (18)	0.65880 (13)	0.0218 (3)
O8	−0.25158 (18)	−0.10364 (18)	0.46895 (14)	0.0245 (4)
H8A	−0.1620	−0.0404	0.4401	0.037*
O9	−0.02594 (18)	−0.10073 (19)	0.63517 (14)	0.0246 (4)
O10	0.10144 (17)	−0.28587 (18)	0.68168 (14)	0.0230 (3)
O11	0.17413 (17)	−0.03462 (17)	0.84417 (13)	0.0196 (3)
O12	0.40563 (17)	−0.08058 (18)	0.76988 (14)	0.0221 (3)
O13	0.33259 (18)	−0.31501 (19)	0.59850 (14)	0.0248 (4)
H13A	0.4399	−0.2832	0.6234	0.037*

C1	0.8308 (3)	0.2448 (3)	0.7230 (2)	0.0251 (5)
H1A	0.9116	0.2282	0.6699	0.030*
H1B	0.7200	0.1376	0.7087	0.030*
C2	0.9149 (3)	0.2927 (3)	0.8712 (2)	0.0248 (5)
H2A	1.0184	0.4058	0.8868	0.030*
H2B	0.8291	0.2977	0.9248	0.030*
N1	0.7928 (3)	0.3808 (2)	0.67745 (18)	0.0284 (4)
H1C	0.7433	0.3510	0.5897	0.043*
H1D	0.8947	0.4789	0.6897	0.043*
H1E	0.7173	0.3948	0.7254	0.043*
N2	0.9717 (2)	0.1666 (2)	0.91544 (17)	0.0236 (4)
H2C	1.0208	0.1977	1.0033	0.035*
H2D	1.0518	0.1633	0.8673	0.035*
H2E	0.8765	0.0631	0.9024	0.035*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
B1	0.0211 (12)	0.0190 (12)	0.0172 (12)	0.0090 (10)	0.0043 (10)	0.0053 (10)
B2	0.0159 (11)	0.0207 (12)	0.0179 (11)	0.0094 (9)	0.0017 (9)	0.0020 (9)
B3	0.0139 (11)	0.0185 (11)	0.0171 (11)	0.0069 (9)	0.0044 (9)	0.0032 (9)
B4	0.0171 (11)	0.0167 (11)	0.0167 (11)	0.0083 (9)	0.0042 (9)	0.0019 (9)
B5	0.0131 (11)	0.0209 (12)	0.0183 (12)	0.0076 (9)	0.0030 (9)	0.0035 (9)
B6	0.0175 (11)	0.0228 (12)	0.0138 (11)	0.0103 (10)	0.0045 (9)	0.0054 (9)
B7	0.0158 (11)	0.0266 (12)	0.0158 (11)	0.0112 (10)	0.0024 (9)	0.0036 (10)
O1	0.0228 (8)	0.0213 (8)	0.0322 (9)	0.0024 (6)	0.0106 (7)	−0.0046 (7)
O2	0.0184 (7)	0.0178 (7)	0.0222 (8)	0.0061 (6)	0.0065 (6)	0.0002 (6)
O3	0.0155 (7)	0.0220 (8)	0.0262 (8)	0.0042 (6)	0.0069 (6)	−0.0024 (6)
O4	0.0150 (7)	0.0232 (8)	0.0213 (8)	0.0072 (6)	0.0033 (6)	−0.0041 (6)
O5	0.0158 (7)	0.0186 (7)	0.0197 (7)	0.0063 (6)	0.0051 (6)	−0.0010 (6)
O6	0.0141 (7)	0.0220 (7)	0.0173 (7)	0.0085 (6)	0.0040 (6)	0.0051 (6)
O7	0.0133 (7)	0.0334 (8)	0.0201 (7)	0.0107 (6)	0.0048 (6)	0.0096 (6)
O8	0.0175 (7)	0.0308 (8)	0.0231 (8)	0.0081 (6)	0.0045 (6)	0.0115 (6)
O9	0.0146 (7)	0.0334 (8)	0.0254 (8)	0.0085 (6)	0.0063 (6)	0.0147 (7)
O10	0.0150 (7)	0.0255 (8)	0.0251 (8)	0.0086 (6)	0.0020 (6)	−0.0056 (6)
O11	0.0141 (7)	0.0216 (7)	0.0221 (8)	0.0085 (6)	0.0036 (6)	−0.0025 (6)
O12	0.0140 (7)	0.0267 (8)	0.0233 (8)	0.0091 (6)	0.0027 (6)	−0.0045 (6)
O13	0.0149 (7)	0.0349 (9)	0.0233 (8)	0.0133 (7)	−0.0001 (6)	−0.0064 (7)
C1	0.0281 (12)	0.0204 (11)	0.0263 (12)	0.0123 (9)	0.0008 (9)	0.0005 (9)
C2	0.0277 (12)	0.0251 (12)	0.0238 (12)	0.0141 (10)	0.0047 (9)	0.0039 (9)
N1	0.0355 (11)	0.0266 (10)	0.0234 (10)	0.0175 (9)	−0.0022 (8)	−0.0021 (8)
N2	0.0239 (10)	0.0296 (10)	0.0188 (9)	0.0133 (8)	0.0046 (7)	0.0054 (8)

Geometric parameters (Å, º)

B1—O2	1.343 (3)	B7—O10	1.340 (3)
B1—O1	1.360 (3)	B7—O13	1.359 (3)
B1—O3	1.383 (3)	B7—O12	1.386 (3)

B2—O5	1.341 (3)	O1—H1F	0.8200
B2—O4	1.372 (3)	O4—B6^{i}	1.372 (3)
B2—O3	1.381 (3)	O8—H8A	0.8200
B3—O6	1.433 (3)	O13—H13A	0.8200
B3—O2	1.471 (3)	C1—N1	1.474 (3)
B3—O7	1.476 (3)	C1—C2	1.503 (3)
B3—O5	1.489 (3)	C1—H1A	0.9700
B4—O7	1.344 (3)	C1—H1B	0.9700
B4—O9	1.355 (3)	C2—N2	1.481 (3)
B4—O8	1.369 (3)	C2—H2A	0.9700
B5—O6	1.430 (3)	C2—H2B	0.9700
B5—O11	1.474 (3)	N1—H1C	0.8900
B5—O9	1.477 (3)	N1—H1D	0.8900
B5—O10	1.480 (3)	N1—H1E	0.8900
B6—O11	1.337 (3)	N2—H2C	0.8900
B6—O4^{i}	1.372 (3)	N2—H2D	0.8900
B6—O12	1.376 (3)	N2—H2E	0.8900

O2—B1—O1	122.44 (19)	B5—O6—B3	125.67 (16)
O2—B1—O3	121.71 (19)	B4—O7—B3	122.80 (16)
O1—B1—O3	115.84 (18)	B4—O8—H8A	109.5
O5—B2—O4	123.39 (19)	B4—O9—B5	122.54 (16)
O5—B2—O3	121.17 (18)	B7—O10—B5	121.89 (17)
O4—B2—O3	115.44 (18)	B6—O11—B5	123.54 (16)
O6—B3—O2	110.41 (17)	B6—O12—B7	118.51 (17)
O6—B3—O7	112.48 (16)	B7—O13—H13A	109.5
O2—B3—O7	106.77 (16)	N1—C1—C2	110.38 (17)
O6—B3—O5	109.26 (16)	N1—C1—H1A	109.6
O2—B3—O5	109.91 (16)	C2—C1—H1A	109.6
O7—B3—O5	107.96 (16)	N1—C1—H1B	109.6
O7—B4—O9	120.90 (18)	C2—C1—H1B	109.6
O7—B4—O8	117.95 (18)	H1A—C1—H1B	108.1
O9—B4—O8	121.13 (18)	N2—C2—C1	111.20 (17)
O6—B5—O11	110.14 (16)	N2—C2—H2A	109.4
O6—B5—O9	112.55 (16)	C1—C2—H2A	109.4
O11—B5—O9	107.35 (16)	N2—C2—H2B	109.4
O6—B5—O10	109.49 (17)	C1—C2—H2B	109.4
O11—B5—O10	109.92 (16)	H2A—C2—H2B	108.0
O9—B5—O10	107.33 (16)	C1—N1—H1C	109.5
O11—B6—O4^{i}	122.59 (19)	C1—N1—H1D	109.5
O11—B6—O12	121.46 (19)	H1C—N1—H1D	109.5
O4^{i}—B6—O12	115.93 (18)	C1—N1—H1E	109.5
O10—B7—O13	119.34 (19)	H1C—N1—H1E	109.5
O10—B7—O12	121.74 (19)	H1D—N1—H1E	109.5
O13—B7—O12	118.91 (18)	C2—N2—H2C	109.5
B1—O1—H1F	109.5	C2—N2—H2D	109.5
B1—O2—B3	120.65 (17)	H2C—N2—H2D	109.5
B2—O3—B1	118.42 (16)	C2—N2—H2E	109.5

B2—O4—B6^{i}	127.25 (17)	H2C—N2—H2E	109.5
B2—O5—B3	122.53 (16)	H2D—N2—H2E	109.5

Symmetry code: (i) −*x*, −*y*, −*z*+2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*F*···O8^{ii}	0.82	2.11	2.909 (2)	165
O8—H8*A*···O9^{iii}	0.82	1.83	2.6433 (19)	176
O13—H13*A*···O7^{iv}	0.82	1.79	2.6030 (18)	172
N1—H1*C*···O13^{v}	0.89	1.87	2.755 (2)	172
N1—H1*D*···O10^{vi}	0.89	2.04	2.919 (2)	168
N1—H1*E*···O2^{vi}	0.89	2.09	2.892 (2)	150
N2—H2*C*···O6^{vii}	0.89	1.89	2.777 (2)	174
N2—H2*D*···O1^{viii}	0.89	2.18	2.926 (2)	141
N2—H2*E*···O5^{iv}	0.89	2.08	2.951 (2)	168

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