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 Bis[(*m*-phenylenedimethylene)-diammonium] tetradecaborate

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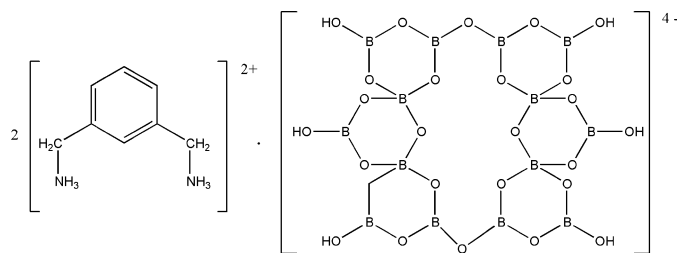
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 Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.035; wR factor = 0.128; data-to-parameter ratio = 11.1.

 The title compound $2\text{C}_8\text{H}_{14}\text{N}_2^{2+} \cdot [\text{B}_{14}\text{O}_{20}(\text{OH})_6]^{4-}$, contains diprotonated $\text{C}_8\text{H}_{14}\text{N}_2^{2+}$ cations and centrosymmetric tetradecaborate anions. The crystal structure is stabilized by $\text{O}-\text{H} \cdots \text{O}$ and $\text{N}-\text{H} \cdots \text{O}$ hydrogen bonds.

Related literature

 For background on the importance of borate compounds, see: Chen *et al.* (1995); Grice *et al.* (1999). For previous work on boron oxoanions, see: Liu *et al.* (2006); Pan *et al.* (2007); Grice *et al.* (1999); Schubert *et al.* (2000); Touboul *et al.* (2003); Burns (1995).


Experimental

Crystal data

 $2\text{C}_8\text{H}_{14}\text{N}_2^{2+} \cdot \text{B}_{14}\text{H}_6\text{O}_{26}^{4-}$
 $M_r = 849.81$
 Triclinic, $P\bar{1}$
 $a = 9.1025$ (18) Å
 $b = 10.293$ (2) Å
 $c = 10.942$ (2) Å
 $\alpha = 109.68$ (3)°
 $\beta = 108.24$ (3)°

 $\gamma = 102.19$ (3)°
 $V = 857.4$ (5) Å³
 $Z = 1$
 Mo $K\alpha$ radiation
 $\mu = 0.14$ mm⁻¹
 $T = 295$ (2) K
 $0.34 \times 0.26 \times 0.18$ mm

Data collection

 Rigaku R-Axis RAPID
 diffractometer
 Absorption correction: multi-scan
 (ABSCOR; Higashi, 1995)
 $T_{\min} = 0.964$, $T_{\max} = 0.973$

 6803 measured reflections
 3009 independent reflections
 2002 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.023$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.128$
 $S = 1.24$
 3009 reflections

 272 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.42$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.46$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H} \cdots A$ | $D-\text{H}$ | $\text{H} \cdots A$ | $D \cdots A$ | $D-\text{H} \cdots A$ |
|--|--------------|---------------------|--------------|-----------------------|
| $\text{O}10-\text{H}10\text{A} \cdots \text{O}2^{\text{i}}$ | 0.82 | 1.98 | 2.784 (2) | 168 |
| $\text{O}11-\text{H}11\text{A} \cdots \text{O}3^{\text{ii}}$ | 0.82 | 1.84 | 2.659 (2) | 179 |
| $\text{O}13-\text{H}13\text{A} \cdots \text{O}11^{\text{iii}}$ | 0.82 | 2.00 | 2.815 (2) | 173 |
| $\text{N}1-\text{H}1\text{A} \cdots \text{O}7$ | 0.89 | 2.08 | 2.863 (2) | 146 |
| $\text{N}1-\text{H}1\text{B} \cdots \text{O}13^{\text{iv}}$ | 0.89 | 1.98 | 2.850 (2) | 166 |
| $\text{N}1-\text{H}1\text{C} \cdots \text{O}6^{\text{v}}$ | 0.89 | 2.20 | 2.916 (2) | 137 |
| $\text{N}1-\text{H}1\text{C} \cdots \text{O}1^{\text{v}}$ | 0.89 | 2.54 | 3.394 (2) | 161 |
| $\text{N}2-\text{H}2\text{A} \cdots \text{O}4^{\text{vi}}$ | 0.89 | 1.97 | 2.822 (2) | 159 |
| $\text{N}2-\text{H}2\text{B} \cdots \text{O}1^{\text{v}}$ | 0.89 | 1.99 | 2.877 (2) | 173 |
| $\text{N}2-\text{H}2\text{B} \cdots \text{O}9^{\text{v}}$ | 0.89 | 2.55 | 3.052 (2) | 116 |
| $\text{N}2-\text{H}2\text{C} \cdots \text{O}12^{\text{vii}}$ | 0.89 | 2.19 | 3.067 (2) | 168 |

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

 Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2801).

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supporting information

Acta Cryst. (2008). E64, o2129 [doi:10.1107/S1600536808033333]

Bis[*m*-phenylenedimethylene]diammonium] tetradecaborate

Xiao Jiang, Shu-Li Wu, Zhi-Dong Shao and Yun-Xiao Liang

S1. Comment

Borate compounds have considerable mineralogical and industrial importance (Chen *et al.*, 1995; Grice *et al.*, 1999). Boron atoms form strong bonds with oxygen atoms not only in trigonal planar BO₃, but also in tetrahedral BO₄ groups. These BO₃ and BO₄ groups may be linked together by sharing common oxygen to form isolated rings and cages or extended chains, sheets, and networks. So far, a number of isolated boron oxoanions have been found in mineral and synthetic borates, such as [B(OH)₄]⁻, [B₂O(OH)₆]²⁻, [B₃O₃(OH)₄]⁻, [B₄O₅(OH)₄]²⁻, [B₅O₆(OH)₄]⁻, [B₆O₇(OH)₆]²⁻ (Grice *et al.*, 1999; Touboul *et al.*, 2003), [B₇O₉(OH)₅]²⁻ (Liu *et al.*, 2006; Pan *et al.*, 2007), [B₉O₁₂(OH)₆]³⁻ (Schubert *et al.*, 2000), and [B₁₄O₂₀(OH)₆]⁴⁻ (Liu *et al.*, 2006; Pan *et al.*, 2007). Compared with metal borates, the synthesis of organically modified nonmetal borates was less well explored in the past decades. Herein, we describe the synthesis and crystal structure of a new nonmetal borate with [B₁₄O₂₀(OH)₆]⁴⁻ as polyanions.

As shown in Fig. 1, the title compound consists of isolated [B₁₄O₂₀(OH)₆]⁴⁻ polyborate anions and [C₈H₁₄N₂]²⁺ cations. The [B₁₄O₂₀(OH)₆]⁴⁻ borate anion is composed of four BO₄, four BO₃, and six BO₂(OH) groups (Burns, 1995). It can also be seen as two [B₇O₉(OH)₅]²⁻ clusters combined with each other through the dehydration of four hydroxyl groups. Each [B₇O₉(OH)₅]²⁻ group contains three B₃O₃ cycles held together *via* two common BO₄ tetrahedra. The B—O bond lengths and O—B—O bond angles are in the range of 1.332 (4)–1.509 (4) Å and 105.9 (3)–124.5 (3)° (Table 1), which are in good agreement with other borates reported previously (Liu *et al.*, 2006; Pan *et al.*, 2007).

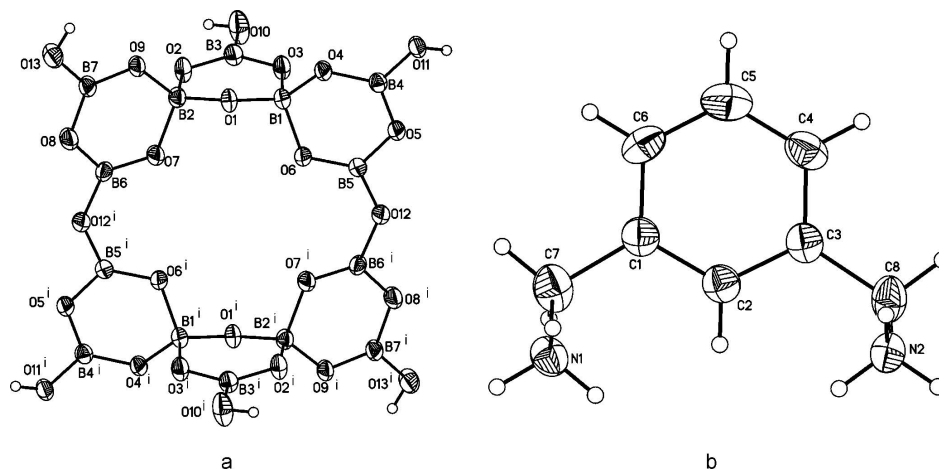
In the present instance, the isolated tetradecaborates anions are linked together through hydrogen bonds: O10—H10A···O2, O11—H11A···O3 (Fig. 2), forming a two-dimensional sheetlike structure. The adjacent borate sheets are further linked together by strong H-bonding interactions [O13—H13A···O11] to form a three-dimensional network (Fig. 3). The hydrogen bonds are listed in Table 2.

S2. Experimental

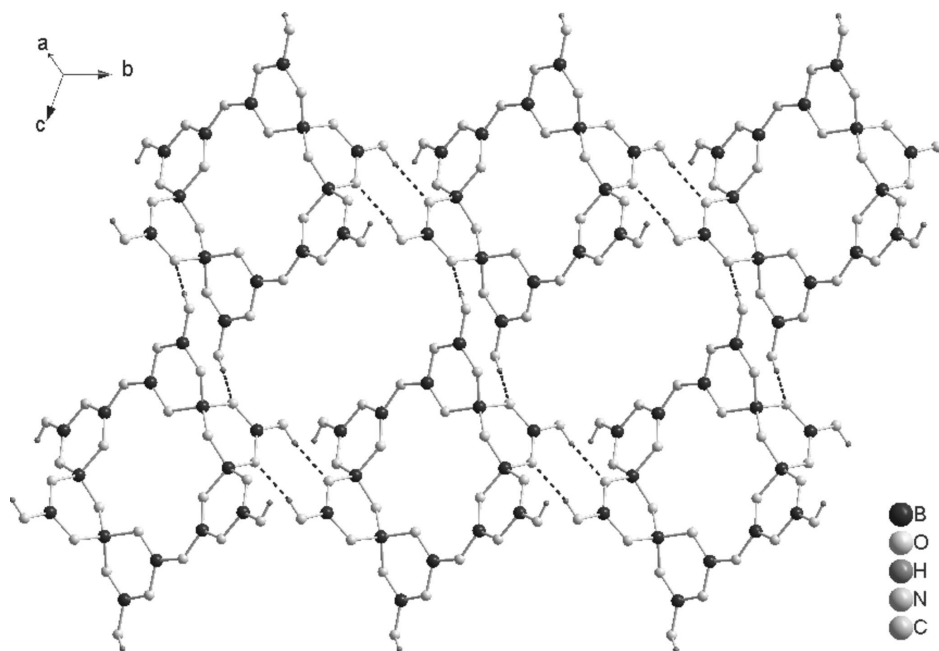
The title compound was obtained by the reaction of H₃BO₃ and 1,3-Bis(aminomethyl)benzene under mild solvothermal conditions. Typically, a mixture of H₃BO₃ (0.9882 g), 1,3-Bis(aminomethyl)benzene (3 ml) was stirred at room temperature. The final mixture was sealed in a Teflon-lined autoclave, heated to 443 K at a rate of 10 K/h, kept at 443 K for 4 days and then cooled to room temperature at a rate of 5 K/h. Colorless transparent block-like crystals were collected and dried in air.

S3. Refinement

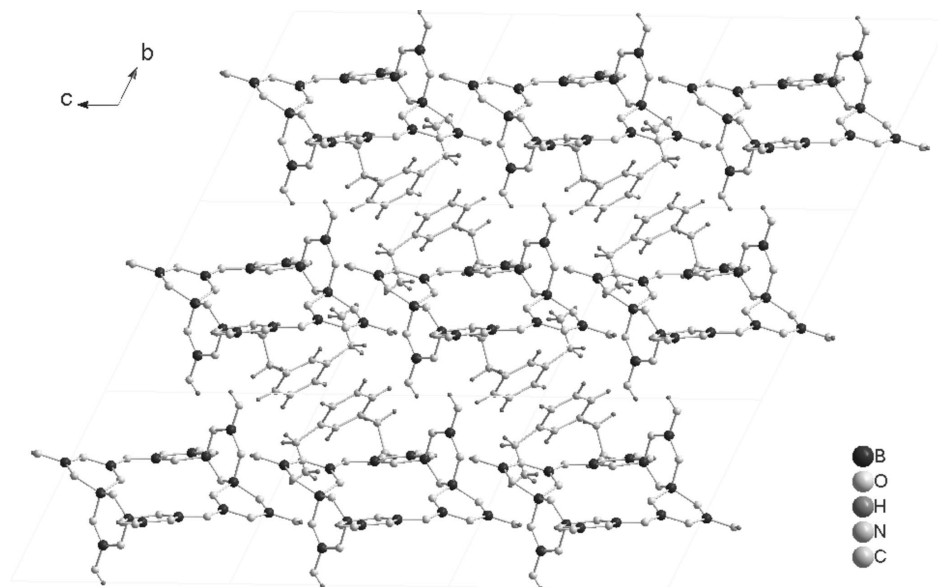
All H atoms were positioned geometrically and refined as riding model [O—H = 0.82 Å, N—H = 0.89 Å, C—H_{aromatic} = 0.93 Å, C—H₂ = 0.97 Å, and with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$, $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{N})$, $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$].

**Figure 1**

The crystal structure of $[(C_8H_{14}N_2)]_2[B_{14}O_{20}(OH)_6]$: (a) $[B_{14}O_{20}(OH)_6]^{4-}$; (b) $[(C_8H_{14}N_2)]^{2+}$, drawn at the 50% probability level. [Symmetry codes: (i) $-x+1, -y+1, -z+1$.]

**Figure 2**

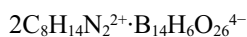
Formation of the two-dimensional sheet from the $[B_{14}O_{20}(OH)_6]^{4-}$ polyborate anions. Hydrogen bonds are indicated by dashed lines.

**Figure 3**

View of the diprotonated of organic amines in the inorganic borate network along *a* axis.

Bis[(*m*-phenylenedimethylene)diammonium] tetradecaborate

Crystal data



$M_r = 849.81$

Triclinic, $P\bar{1}$

Hall symbol: $-P1$

$a = 9.1025$ (18) Å

$b = 10.293$ (2) Å

$c = 10.942$ (2) Å

$\alpha = 109.68$ (3)°

$\beta = 108.24$ (3)°

$\gamma = 102.19$ (3)°

$V = 857.4$ (5) Å³

$Z = 1$

$F(000) = 436$

$D_x = 1.646$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4940 reflections

$\theta = 6.7\text{--}54.9^\circ$

$\mu = 0.14$ mm⁻¹

$T = 295$ K

Block, colourless

$0.34 \times 0.26 \times 0.18$ mm

Data collection

Rigaku R-AXIS RAPID
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

Absorption correction: multi-scan
(*ABSCOR*; Higashi, 1995)

$T_{\min} = 0.964$, $T_{\max} = 0.973$

6803 measured reflections

3009 independent reflections

2002 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.023$

$\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 3.4^\circ$

$h = -10 \rightarrow 10$

$k = -12 \rightarrow 12$

$l = -12 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.035$

$wR(F^2) = 0.128$

$S = 1.24$

3009 reflections

272 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
 $w = 1/[\sigma^2(F_o^2) + (0.0299P)^2 + 1.2547P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.42 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.46 \text{ e } \text{\AA}^{-3}$
Extinction correction: *SHELXL97* (Sheldrick,
2008), $F_c^* = kFc[1 + 0.001x Fc^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.012 (2)

Special details

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.

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> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|-------------|------------|------------|----------------------------------|
| B1 | 0.6460 (5) | 0.4668 (4) | 0.7888 (4) | 0.0240 (8) |
| B2 | 0.7413 (4) | 0.3345 (4) | 0.6060 (4) | 0.0222 (8) |
| B3 | 0.5338 (4) | 0.1936 (4) | 0.6602 (4) | 0.0247 (8) |
| B4 | 0.6535 (4) | 0.6248 (4) | 1.0187 (4) | 0.0209 (7) |
| B5 | 0.4617 (5) | 0.6104 (4) | 0.8040 (4) | 0.0233 (8) |
| B6 | 0.7437 (4) | 0.3478 (4) | 0.3829 (4) | 0.0225 (8) |
| B7 | 0.9709 (5) | 0.3173 (4) | 0.5343 (4) | 0.0252 (8) |
| O1 | 0.7649 (3) | 0.4597 (2) | 0.7304 (2) | 0.0238 (5) |
| O2 | 0.6220 (3) | 0.1961 (2) | 0.5811 (2) | 0.0279 (5) |
| O3 | 0.5410 (3) | 0.3195 (2) | 0.7601 (2) | 0.0266 (5) |
| O4 | 0.7324 (3) | 0.5555 (2) | 0.9474 (2) | 0.0231 (5) |
| O5 | 0.5160 (3) | 0.6519 (2) | 0.9489 (2) | 0.0259 (5) |
| O6 | 0.5330 (3) | 0.5377 (2) | 0.7283 (2) | 0.0248 (5) |
| O7 | 0.6718 (3) | 0.3576 (2) | 0.4736 (2) | 0.0263 (5) |
| O8 | 0.8946 (3) | 0.3301 (3) | 0.4095 (2) | 0.0297 (6) |
| O9 | 0.9009 (3) | 0.3166 (2) | 0.6244 (2) | 0.0260 (5) |
| O10 | 0.4275 (3) | 0.0652 (3) | 0.6413 (3) | 0.0447 (7) |
| H10A | 0.4225 | -0.0042 | 0.5733 | 0.067* |
| O11 | 0.7074 (3) | 0.6690 (2) | 1.1630 (2) | 0.0269 (5) |
| H11A | 0.6297 | 0.6722 | 1.1852 | 0.040* |
| O12 | 0.3292 (3) | 0.6490 (3) | 0.7475 (2) | 0.0284 (5) |
| O13 | 1.1239 (3) | 0.3064 (3) | 0.5617 (2) | 0.0374 (6) |
| H13A | 1.1659 | 0.3147 | 0.6433 | 0.056* |
| N1 | 0.3291 (3) | 0.3057 (3) | 0.4112 (3) | 0.0329 (7) |
| H1A | 0.4353 | 0.3287 | 0.4654 | 0.049* |
| H1B | 0.2747 | 0.3228 | 0.4662 | 0.049* |
| H1C | 0.3203 | 0.3605 | 0.3633 | 0.049* |
| N2 | -0.1153 (3) | 0.4082 (3) | 0.0950 (3) | 0.0320 (7) |
| H2A | -0.1372 | 0.4667 | 0.0531 | 0.048* |

| | | | | |
|-----|-------------|-------------|-------------|-------------|
| H2B | -0.0078 | 0.4427 | 0.1513 | 0.048* |
| H2C | -0.1730 | 0.4060 | 0.1471 | 0.048* |
| C1 | 0.0788 (4) | 0.1079 (4) | 0.2176 (4) | 0.0316 (8) |
| C2 | 0.0378 (4) | 0.1765 (4) | 0.1291 (4) | 0.0313 (8) |
| H2D | 0.1214 | 0.2350 | 0.1185 | 0.038* |
| C3 | -0.1245 (4) | 0.1600 (4) | 0.0564 (4) | 0.0310 (8) |
| C4 | -0.2492 (4) | 0.0650 (4) | 0.0667 (4) | 0.0380 (9) |
| H4A | -0.3593 | 0.0505 | 0.0165 | 0.046* |
| C5 | -0.2107 (5) | -0.0074 (4) | 0.1508 (4) | 0.0452 (10) |
| H5A | -0.2949 | -0.0713 | 0.1563 | 0.054* |
| C6 | -0.0475 (5) | 0.0143 (4) | 0.2272 (4) | 0.0387 (9) |
| H6A | -0.0220 | -0.0337 | 0.2850 | 0.046* |
| C7 | 0.2573 (5) | 0.1467 (4) | 0.3083 (4) | 0.0413 (9) |
| H7A | 0.2681 | 0.0865 | 0.3600 | 0.050* |
| H7B | 0.3166 | 0.1274 | 0.2483 | 0.050* |
| C8 | -0.1624 (5) | 0.2555 (4) | -0.0168 (4) | 0.0373 (9) |
| H8A | -0.2794 | 0.2172 | -0.0789 | 0.045* |
| H8B | -0.1006 | 0.2570 | -0.0744 | 0.045* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|-------------|-------------|-------------|
| B1 | 0.0293 (19) | 0.027 (2) | 0.0191 (19) | 0.0131 (16) | 0.0134 (16) | 0.0092 (16) |
| B2 | 0.0212 (18) | 0.025 (2) | 0.0231 (19) | 0.0095 (15) | 0.0124 (16) | 0.0104 (16) |
| B3 | 0.0267 (19) | 0.024 (2) | 0.024 (2) | 0.0084 (16) | 0.0132 (16) | 0.0098 (17) |
| B4 | 0.0219 (18) | 0.0198 (18) | 0.0207 (19) | 0.0057 (14) | 0.0104 (15) | 0.0081 (15) |
| B5 | 0.0287 (19) | 0.0248 (19) | 0.0197 (19) | 0.0128 (16) | 0.0115 (16) | 0.0101 (16) |
| B6 | 0.0245 (19) | 0.0242 (19) | 0.0179 (18) | 0.0128 (15) | 0.0081 (15) | 0.0060 (15) |
| B7 | 0.0265 (19) | 0.032 (2) | 0.0162 (18) | 0.0142 (17) | 0.0085 (16) | 0.0072 (16) |
| O1 | 0.0236 (11) | 0.0247 (12) | 0.0230 (12) | 0.0071 (9) | 0.0145 (10) | 0.0067 (10) |
| O2 | 0.0325 (12) | 0.0254 (12) | 0.0273 (13) | 0.0089 (10) | 0.0197 (11) | 0.0073 (10) |
| O3 | 0.0327 (13) | 0.0219 (12) | 0.0295 (13) | 0.0083 (10) | 0.0211 (11) | 0.0095 (10) |
| O4 | 0.0253 (11) | 0.0272 (12) | 0.0160 (11) | 0.0119 (9) | 0.0086 (9) | 0.0071 (10) |
| O5 | 0.0291 (12) | 0.0354 (13) | 0.0188 (12) | 0.0186 (10) | 0.0117 (10) | 0.0119 (10) |
| O6 | 0.0292 (12) | 0.0320 (13) | 0.0190 (11) | 0.0182 (10) | 0.0119 (10) | 0.0113 (10) |
| O7 | 0.0251 (12) | 0.0394 (14) | 0.0243 (12) | 0.0182 (10) | 0.0145 (10) | 0.0168 (11) |
| O8 | 0.0280 (12) | 0.0458 (15) | 0.0253 (13) | 0.0196 (11) | 0.0161 (10) | 0.0183 (11) |
| O9 | 0.0248 (12) | 0.0392 (14) | 0.0237 (12) | 0.0175 (10) | 0.0153 (10) | 0.0157 (11) |
| O10 | 0.0624 (17) | 0.0251 (14) | 0.0473 (16) | 0.0051 (12) | 0.0399 (15) | 0.0069 (12) |
| O11 | 0.0280 (12) | 0.0377 (14) | 0.0196 (12) | 0.0158 (10) | 0.0130 (10) | 0.0123 (10) |
| O12 | 0.0315 (13) | 0.0418 (14) | 0.0240 (12) | 0.0234 (11) | 0.0155 (10) | 0.0178 (11) |
| O13 | 0.0271 (13) | 0.0688 (18) | 0.0272 (13) | 0.0275 (13) | 0.0146 (11) | 0.0238 (13) |
| N1 | 0.0276 (15) | 0.0430 (18) | 0.0291 (16) | 0.0123 (13) | 0.0137 (13) | 0.0152 (14) |
| N2 | 0.0338 (16) | 0.0351 (17) | 0.0316 (16) | 0.0160 (13) | 0.0139 (13) | 0.0170 (14) |
| C1 | 0.0338 (19) | 0.0238 (18) | 0.0276 (19) | 0.0091 (15) | 0.0082 (15) | 0.0054 (15) |
| C2 | 0.0323 (19) | 0.0286 (19) | 0.0274 (19) | 0.0091 (15) | 0.0107 (16) | 0.0085 (15) |
| C3 | 0.0325 (19) | 0.0279 (19) | 0.0270 (19) | 0.0117 (15) | 0.0102 (16) | 0.0072 (15) |
| C4 | 0.0290 (19) | 0.029 (2) | 0.045 (2) | 0.0075 (16) | 0.0120 (17) | 0.0087 (18) |

| | | | | | | |
|----|-----------|-----------|-------------|-------------|-------------|-------------|
| C5 | 0.044 (2) | 0.033 (2) | 0.054 (3) | 0.0065 (18) | 0.024 (2) | 0.014 (2) |
| C6 | 0.050 (2) | 0.028 (2) | 0.043 (2) | 0.0132 (17) | 0.0213 (19) | 0.0194 (18) |
| C7 | 0.041 (2) | 0.037 (2) | 0.038 (2) | 0.0188 (18) | 0.0109 (18) | 0.0096 (18) |
| C8 | 0.041 (2) | 0.038 (2) | 0.0250 (19) | 0.0164 (17) | 0.0063 (16) | 0.0113 (17) |

Geometric parameters (Å, °)

| | | | |
|---------------------|-----------|------------|-----------|
| B1—O1 | 1.421 (4) | O13—H13A | 0.8200 |
| B1—O3 | 1.480 (4) | N1—C7 | 1.489 (5) |
| B1—O4 | 1.496 (4) | N1—H1A | 0.8900 |
| B1—O6 | 1.499 (4) | N1—H1B | 0.8900 |
| B2—O1 | 1.442 (4) | N1—H1C | 0.8900 |
| B2—O9 | 1.463 (4) | N2—C8 | 1.497 (4) |
| B2—O2 | 1.477 (4) | N2—H2A | 0.8900 |
| B2—O7 | 1.509 (4) | N2—H2B | 0.8900 |
| B3—O2 | 1.354 (4) | N2—H2C | 0.8900 |
| B3—O3 | 1.358 (4) | C1—C2 | 1.385 (5) |
| B3—O10 | 1.370 (4) | C1—C6 | 1.391 (5) |
| B4—O4 | 1.351 (4) | C1—C7 | 1.495 (5) |
| B4—O11 | 1.370 (4) | C2—C3 | 1.383 (5) |
| B4—O5 | 1.386 (4) | C2—H2D | 0.9300 |
| B5—O6 | 1.338 (4) | C3—C4 | 1.394 (5) |
| B5—O12 | 1.377 (4) | C3—C8 | 1.492 (5) |
| B5—O5 | 1.380 (4) | C4—C5 | 1.377 (6) |
| B6—O7 | 1.338 (4) | C4—H4A | 0.9300 |
| B6—O8 | 1.377 (4) | C5—C6 | 1.383 (5) |
| B6—O12 ⁱ | 1.388 (4) | C5—H5A | 0.9300 |
| B7—O9 | 1.332 (4) | C6—H6A | 0.9300 |
| B7—O13 | 1.368 (4) | C7—H7A | 0.9700 |
| B7—O8 | 1.388 (4) | C7—H7B | 0.9700 |
| O10—H10A | 0.8200 | C8—H8A | 0.9700 |
| O11—H11A | 0.8200 | C8—H8B | 0.9700 |
| O12—B6 ⁱ | 1.388 (4) | | |
| O1—B1—O3 | 112.6 (3) | C7—N1—H1B | 109.5 |
| O1—B1—O4 | 109.6 (3) | H1A—N1—H1B | 109.5 |
| O3—B1—O4 | 107.5 (2) | C7—N1—H1C | 109.5 |
| O1—B1—O6 | 111.1 (3) | H1A—N1—H1C | 109.5 |
| O3—B1—O6 | 107.2 (3) | H1B—N1—H1C | 109.5 |
| O4—B1—O6 | 108.6 (2) | C8—N2—H2A | 109.5 |
| O1—B2—O9 | 108.6 (3) | C8—N2—H2B | 109.5 |
| O1—B2—O2 | 112.9 (2) | H2A—N2—H2B | 109.5 |
| O9—B2—O2 | 109.0 (3) | C8—N2—H2C | 109.5 |
| O1—B2—O7 | 110.2 (3) | H2A—N2—H2C | 109.5 |
| O9—B2—O7 | 110.1 (2) | H2B—N2—H2C | 109.5 |
| O2—B2—O7 | 105.9 (3) | C2—C1—C6 | 118.9 (3) |
| O2—B3—O3 | 121.8 (3) | C2—C1—C7 | 118.6 (3) |
| O2—B3—O10 | 122.5 (3) | C6—C1—C7 | 122.2 (3) |

| | | | |
|------------------------|-----------|------------|-----------|
| O3—B3—O10 | 115.7 (3) | C3—C2—C1 | 121.5 (3) |
| O4—B4—O11 | 120.4 (3) | C3—C2—H2D | 119.2 |
| O4—B4—O5 | 121.5 (3) | C1—C2—H2D | 119.2 |
| O11—B4—O5 | 118.1 (3) | C2—C3—C4 | 118.6 (3) |
| O6—B5—O12 | 124.5 (3) | C2—C3—C8 | 120.2 (3) |
| O6—B5—O5 | 121.6 (3) | C4—C3—C8 | 120.9 (3) |
| O12—B5—O5 | 113.8 (3) | C5—C4—C3 | 120.5 (3) |
| O7—B6—O8 | 122.9 (3) | C5—C4—H4A | 119.8 |
| O7—B6—O12 ⁱ | 122.9 (3) | C3—C4—H4A | 119.8 |
| O8—B6—O12 ⁱ | 114.2 (3) | C4—C5—C6 | 120.3 (4) |
| O9—B7—O13 | 120.9 (3) | C4—C5—H5A | 119.8 |
| O9—B7—O8 | 122.5 (3) | C6—C5—H5A | 119.8 |
| O13—B7—O8 | 116.6 (3) | C5—C6—C1 | 120.1 (3) |
| B1—O1—B2 | 123.5 (3) | C5—C6—H6A | 120.0 |
| B3—O2—B2 | 122.1 (3) | C1—C6—H6A | 120.0 |
| B3—O3—B1 | 121.8 (2) | N1—C7—C1 | 109.5 (3) |
| B4—O4—B1 | 119.7 (3) | N1—C7—H7A | 109.8 |
| B5—O5—B4 | 118.9 (2) | C1—C7—H7A | 109.8 |
| B5—O6—B1 | 120.7 (2) | N1—C7—H7B | 109.8 |
| B6—O7—B2 | 122.4 (2) | C1—C7—H7B | 109.8 |
| B6—O8—B7 | 117.6 (3) | H7A—C7—H7B | 108.2 |
| B7—O9—B2 | 124.2 (3) | C3—C8—N2 | 108.3 (3) |
| B3—O10—H10A | 109.5 | C3—C8—H8A | 110.0 |
| B4—O11—H11A | 109.5 | N2—C8—H8A | 110.0 |
| B5—O12—B6 ⁱ | 132.5 (3) | C3—C8—H8B | 110.0 |
| B7—O13—H13A | 109.5 | N2—C8—H8B | 110.0 |
| C7—N1—H1A | 109.5 | H8A—C8—H8B | 108.4 |

Symmetry code: (i) $-x+1, -y+1, -z+1$.

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-------------------------------------|-------|-------------|-------------|---------------|
| O10—H10A \cdots O2 ⁱⁱ | 0.82 | 1.98 | 2.784 (2) | 168 |
| O11—H11A \cdots O3 ⁱⁱⁱ | 0.82 | 1.84 | 2.659 (2) | 179 |
| O13—H13A \cdots O11 ^{iv} | 0.82 | 2.00 | 2.815 (2) | 173 |
| N1—H1A \cdots O7 | 0.89 | 2.08 | 2.863 (2) | 146 |
| N1—H1B \cdots O13 ^v | 0.89 | 1.98 | 2.850 (2) | 166 |
| N1—H1C \cdots O6 ⁱ | 0.89 | 2.20 | 2.916 (2) | 137 |
| N1—H1C \cdots O1 ⁱ | 0.89 | 2.54 | 3.394 (2) | 161 |
| N2—H2A \cdots O4 ^{vi} | 0.89 | 1.97 | 2.822 (2) | 159 |
| N2—H2B \cdots O1 ⁱ | 0.89 | 1.99 | 2.877 (2) | 173 |
| N2—H2B \cdots O9 ⁱ | 0.89 | 2.55 | 3.052 (2) | 116 |
| N2—H2C \cdots O12 ^{vii} | 0.89 | 2.19 | 3.067 (2) | 168 |

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