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Dimethylammonium bis(3-oxido-naphthalene-2-carboxylato)borate hemihydrate

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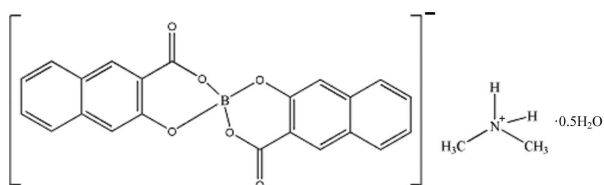
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Key indicators: single-crystal X-ray study; $T = 303$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; some non-H atoms missing; R factor = 0.093; wR factor = 0.152; data-to-parameter ratio = 14.5.

The title compound, $\text{C}_2\text{H}_8\text{N}^+\cdot\text{C}_{22}\text{H}_{12}\text{BO}_6^- \cdot 0.5\text{H}_2\text{O}$, was synthesized under atmospheric conditions in the presence of dimethylformamide acting as a template. The structure is composed of $[\text{NH}_2(\text{CH}_3)_2]^+$ cations, bis(3-oxidonaphthalene-2-carboxylato)borate anions and water molecules. The water molecule lies on a twofold rotation axis. The stabilization of the crystal structure comes from electrostatic interactions and is assisted by intermolecular $\text{O}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds between the layers.

Related literature

For related literature, see: Carr *et al.* (2005); Downard *et al.* (2002); Errington *et al.* (1999); Green *et al.* (2000); Grice *et al.* (1999); Li & Liu (2006); Schubert *et al.* (2000); Tombul *et al.* (2003); Tombul, Guven, Büyükgüngör *et al.* (2007); Touboul *et al.* (2003); Zhang & Liu (2006); Zhang *et al.* (2005).



Experimental

Crystal data

$\text{C}_2\text{H}_8\text{N}^+\cdot\text{C}_{22}\text{H}_{12}\text{BO}_6^- \cdot 0.5\text{H}_2\text{O}$
 $M_r = 438.24$
Monoclinic, $C2/c$
 $a = 32.011$ (3) Å
 $b = 9.774$ (1) Å

$c = 14.742$ (1) Å
 $\beta = 112.628$ (7)°
 $V = 4257.2$ (7) Å³
 $Z = 8$
Mo $K\alpha$ radiation

$\mu = 0.10$ mm⁻¹
 $T = 303$ (2) K

0.48 × 0.08 × 0.08 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: numerical [using a multifaceted crystal model based on expressions

derived (Clark & Reid, 1995)]
 $T_{\min} = 0.981$, $T_{\max} = 0.994$
16173 measured reflections
4321 independent reflections
1708 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.079$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.093$
 $wR(F^2) = 0.152$
 $S = 1.12$
4321 reflections
299 parameters
3 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.21$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.23$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

| | | | |
|----------|-----------|----------|-----------|
| O1—B1 | 1.427 (5) | O4—B1 | 1.426 (5) |
| O2—B1 | 1.496 (5) | O5—B1 | 1.502 (5) |
| O4—B1—O1 | 110.7 (4) | O4—B1—O5 | 112.0 (3) |
| O4—B1—O2 | 107.7 (3) | O1—B1—O5 | 107.9 (3) |
| O1—B1—O2 | 112.4 (3) | O2—B1—O5 | 106.1 (4) |

Table 2

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|------------------------------------|--------------|--------------------|-------------|----------------------|
| N5—H25A \cdots O3 ⁱ | 1.00 | 1.88 | 2.849 (4) | 162 |
| N5—H25B \cdots O6 ⁱⁱ | 0.95 | 2.12 | 2.839 (4) | 131 |
| N5—H25B \cdots O3 ⁱⁱⁱ | 0.95 | 2.33 | 2.871 (4) | 116 |
| O7—H27 \cdots O5 | 0.85 (3) | 2.18 (4) | 3.010 (3) | 163.3 (12) |

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2517).

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supplementary materials

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Dimethylammonium bis(3-oxidonaphthalene-2-carboxylato)borate hemihydrate

M. Tombul, K. Güven and I. Svoboda

Comment

Owing to their rich structural chemistry and potential applications in mineralogy (Grice *et al.*, 1999) and nonlinear optical materials (Touboul *et al.*, 2003), borates have provided an abounding area of research for over half a century. Boron can form a large variety of compounds due to the complexity of the structures involved. Boron atom coordinate with oxygen not only in fourfold coordination (tetrahedral, BO_4) but also in threefold coordination (triangular, BO_3). These BO_3 and BO_4 groups favour polymerization *via* common corners into large polynuclear anion units including isolated or finite clusters, chains, sheets, and networks (Grice *et al.*, 1999). Most borates synthesized and studied to date have been prepared under the templating effect of inorganic cations, such as alkali-metal, alkaline-earth, rare-earth or transition cations. Accordingly, many borate systems taking into account alkali metal, alkaline earth metal, rare-earth, and transition metals have been explored in recent years (Schubert *et al.*, 2000). Currently, studies conducted in this area are primarily focused on improving existing materials utilized and search for new materials. In comparison to inorganic borates, synthesis, crystal structure and application of organic borates seem to be insufficient (Li & Liu, 2006; Zhang & Liu, 2006; Carr *et al.*, 2005; Zhang *et al.*, 2005; Downard *et al.*, 2002; Green *et al.*, 2000). As part of our ongoing study of the synthesis and structure of organoborate complexes (Errington *et al.*, 1999; Tombul *et al.*, 2003; Tombul, Guven, Büyükgüngör *et al.*, 2007), we have prepared a new organically templated borate, (I), using dimethylformamide as the structure-directing agent.

The asymmetric unit of (I) (Fig.1) consists of $[\text{BO}_4(\text{C}_{11}\text{H}_6\text{O})_2]^-$ anions, $[\text{NH}_2(\text{CH}_3)_2]^+$ cations and water molecule. The $[\text{BO}_4(\text{C}_{11}\text{H}_6\text{O})_2]^-$ anion consists of one set of distorted $[\text{BO}_4]$ tetrahedra and two sets of slightly deformed $[\text{C}_{11}\text{H}_6\text{O}]$ planes with oxygen atoms as sharing vertexes. The boron atom is bonded to four oxygen atoms to form a highly tetrahedral environment (mean OBO bond angle of $109.46(3)^\circ$). The B—O bond lengths are typical for such tetrahedral borates, with B— $\text{O}_{\text{carboxyl}}$ bond lengths (mean $1.499(5) \text{ \AA}$) being slightly longer than B— $\text{O}_{\text{hydroxyl}}$ bond lengths (mean $1.427(5) \text{ \AA}$). Each $[\text{C}_{11}\text{H}_6\text{O}]$ unit is almost planar, and both $[\text{C}_{11}\text{H}_6\text{O}]$ units in $[\text{BO}_4(\text{C}_{11}\text{H}_6\text{O})_2]^-$ are nearly perpendicular to each other. The borate rings highly coplanar with the corresponding aryl rings [with 0.0779 \AA r.m.s. deviation for Plane A (C1—C11/O1—O3) and 0.0648 \AA r.m.s. deviation for Plane B (C12—C22/O4—O6)]. The coordination planes (Planes C (O2, B1, O1) and D (O5, B1, O4) intersects at an angle of $89.73(26)^\circ$. Dimethylammonium cation is distorted tetrahedral environment with CNC bond angle of $113.5(4)^\circ$ and located with a large distance from the anion (B—N distance $6.108(15) \text{ \AA}$).

In the crystal structure $[\text{BO}_4(\text{C}_{11}\text{H}_6\text{O})_2]^-$ anions and $[\text{NH}_2(\text{CH}_3)_2]^+$ cations are discrete units and they interact both electrostatically and *via* N—H \cdots O hydrogen bonds with N \cdots O distances in the range $2.839(4) \text{ \AA} - 2.872(4) \text{ \AA}$ (Fig.2). The water molecule is also involved in normal, slightly bent, hydrogen bond with the borate anion at a distance of $3.012(3) \text{ \AA}$. The acceptors are all carboxylate O atoms of the aromatic ring (Table 2). For the synthesis of (I), dimethylformamide acts not only as the solvent, but also as the reactant and it decomposes under experimental conditions forming $[\text{NH}_2(\text{CH}_3)_2]^+$ cations.

Experimental

For the preparation of title compound, (I), $B(OH)_3$ (133 mg, 2.16 mmol,) was carefully added to a stirred DMF (10.0 ml) solution of 3-hydroxynaphthalene-2-carboxylic acid (875 mg, 4.65 mmol) at ambient temperature. The reaction mixture initially gave a brown solution which was stirred at 398 K for 2.5 h until all became a gel-like material. This product was then redissolved in the mixture of MeOH/ CH_2Cl_2 (10 ml; 1:1) and allowed to stand at room temperature for a couple of hours, whereupon transparent and fine crystals were harvested. Yield, 79% (based on $B(OH)_3$); Elemental analysis: (Found): C 65.48, H 5.18, N 3.19%. Calculated for $C_{24}H_{22}O_7NB$: C 64.45, H 4.96, N 3.13%. 1H NMR (d^6 -DMSO- $CDCl_3$, 298 K, TMS): δ (p.p.m.): All aromatic signals are observed between the region at 7.12 and 8.43. ^{13}C NMR (d^6 -DMSO- $CDCl_3$, 298 K, TMS): δ (p.p.m.): δ 119.7, 122.5, 128.1, 132.2, 142.4, 160.0, 161.6, 169.7, 177.0.

Refinement

The H27 atom was located in a difference map and refined freely. Other H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93–0.96 Å and N—H = 0.9530–1.0035 Å with $U_{iso}(H) = 1.2$ or 1.5 times $U_{eq}(C,N)$ of the parent atom.

Figures

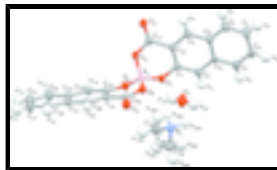


Fig. 1. Showing the atom-labelling scheme of (I). Displacement ellipsoids are drawn at the 50% probability level.

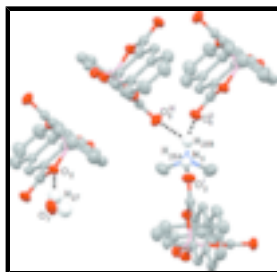


Fig. 2. Hydrogen bond labelling scheme for (I). Dashed lines indicate hydrogen bonds. Some hydrogen atoms were omitted for clarity). [Symmetry codes: (i): $x, -y + 1, z - 1/2$, (ii): $x, y + 1, z$, (iii): $-x, -y + 1, -z + 1$].

Dimethylammonium bis(3-oxidonaphthalene-2-carboxylato)borate hemihydrate

Crystal data

$C_2H_8N^+ \cdot C_{22}H_{12}BO_6^- \cdot 0.5H_2O$

$M_r = 438.24$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 32.011$ (3) Å

$F_{000} = 1832$

$D_x = 1.367$ Mg m $^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 2075 reflections

$\theta = 2.5$ – 21.1°

$b = 9.774 (1) \text{ \AA}$
 $c = 14.742 (1) \text{ \AA}$
 $\beta = 112.628 (7)^\circ$
 $V = 4257.2 (7) \text{ \AA}^3$
 $Z = 8$

$\mu = 0.10 \text{ mm}^{-1}$
 $T = 303 (2) \text{ K}$
 Rod shape, light yellow
 $0.48 \times 0.08 \times 0.08 \text{ mm}$

Data collection

Oxford Diffraction Xcalibur
 diffractometer with a Sapphire CCD detector
 Radiation source: fine-focus sealed tube
 Monochromator: graphite
 Detector resolution: $8.4012 \text{ pixels mm}^{-1}$
 $T = 303(2) \text{ K}$
 Rotation method data acquisition using ω and φ scans $h = -40 \rightarrow 40$
 Absorption correction: numerical
 [absorption correction using a multifaceted crystal
 model based on expressions derived (Clark & Reid,
 1995)]
 $T_{\min} = 0.981, T_{\max} = 0.994$
 16173 measured reflections

4321 independent reflections
 1708 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.079$
 $\theta_{\max} = 26.4^\circ$
 $\theta_{\min} = 2.5^\circ$
 $k = -9 \rightarrow 12$
 $l = -18 \rightarrow 18$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.093$
 $wR(F^2) = 0.152$
 $S = 1.12$
 4321 reflections
 299 parameters
 3 restraints
 Primary atom site location: structure-invariant direct
 methods

Secondary atom site location: difference Fourier map
 Hydrogen site location: inferred from neighbouring
 sites
 H atoms treated by a mixture of
 independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0247P)^2 + 7.7951P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.21 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.23 \text{ e \AA}^{-3}$
 Extinction correction: none

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 $F^2 > 2\sigma(F^2)$ is used only for calculat-

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ing 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)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|------------|--------------|----------------------------------|
| O1 | -0.01987 (8) | 0.4067 (3) | 0.37753 (18) | 0.0487 (8) |
| O2 | -0.03911 (9) | 0.2873 (3) | 0.5007 (2) | 0.0520 (8) |
| O3 | 0.01354 (9) | 0.2159 (3) | 0.6392 (2) | 0.0643 (9) |
| O4 | -0.09425 (8) | 0.4084 (3) | 0.37123 (19) | 0.0474 (7) |
| O5 | -0.06167 (9) | 0.2000 (3) | 0.33692 (19) | 0.0498 (8) |
| O6 | -0.10420 (9) | 0.0190 (3) | 0.2734 (2) | 0.0608 (9) |
| C1 | 0.02357 (13) | 0.3624 (4) | 0.4222 (3) | 0.0421 (11) |
| C2 | 0.05411 (13) | 0.3882 (4) | 0.3807 (3) | 0.0443 (11) |
| H2 | 0.0451 | 0.4358 | 0.3216 | 0.053* |
| C3 | 0.09922 (13) | 0.3435 (4) | 0.4264 (3) | 0.0423 (11) |
| C4 | 0.13136 (14) | 0.3649 (5) | 0.3838 (3) | 0.0564 (13) |
| H4 | 0.1230 | 0.4126 | 0.3249 | 0.068* |
| C5 | 0.17420 (16) | 0.3169 (5) | 0.4274 (4) | 0.0693 (15) |
| H5 | 0.1947 | 0.3314 | 0.3976 | 0.083* |
| C6 | 0.18799 (15) | 0.2461 (5) | 0.5163 (4) | 0.0712 (15) |
| H6 | 0.2175 | 0.2135 | 0.5452 | 0.085* |
| C7 | 0.15843 (14) | 0.2246 (5) | 0.5608 (3) | 0.0615 (13) |
| H7 | 0.1679 | 0.1784 | 0.6205 | 0.074* |
| C8 | 0.11340 (13) | 0.2721 (4) | 0.5170 (3) | 0.0443 (11) |
| C9 | 0.08061 (13) | 0.2483 (4) | 0.5579 (3) | 0.0457 (11) |
| H9 | 0.0890 | 0.2026 | 0.6176 | 0.055* |
| C10 | 0.03693 (13) | 0.2910 (4) | 0.5115 (3) | 0.0398 (10) |
| C11 | 0.00351 (14) | 0.2617 (4) | 0.5560 (4) | 0.0482 (12) |
| C12 | -0.13259 (13) | 0.3378 (4) | 0.3605 (3) | 0.0406 (10) |
| C13 | -0.16686 (12) | 0.4009 (4) | 0.3771 (3) | 0.0462 (11) |
| H13 | -0.1638 | 0.4923 | 0.3962 | 0.055* |
| C14 | -0.20682 (13) | 0.3303 (5) | 0.3661 (3) | 0.0470 (12) |
| C15 | -0.24235 (14) | 0.3923 (5) | 0.3855 (3) | 0.0686 (15) |
| H15 | -0.2397 | 0.4828 | 0.4065 | 0.082* |
| C16 | -0.28029 (16) | 0.3202 (6) | 0.3738 (4) | 0.0833 (17) |
| H16 | -0.3035 | 0.3628 | 0.3863 | 0.100* |
| C17 | -0.28545 (16) | 0.1834 (6) | 0.3434 (4) | 0.0858 (17) |
| H17 | -0.3117 | 0.1356 | 0.3362 | 0.103* |
| C18 | -0.25178 (14) | 0.1214 (5) | 0.3245 (3) | 0.0694 (14) |
| H18 | -0.2551 | 0.0304 | 0.3046 | 0.083* |
| C19 | -0.21161 (13) | 0.1922 (5) | 0.3345 (3) | 0.0457 (11) |
| C20 | -0.17613 (13) | 0.1312 (4) | 0.3159 (3) | 0.0463 (11) |
| H20 | -0.1792 | 0.0412 | 0.2937 | 0.056* |
| C21 | -0.13709 (12) | 0.2008 (4) | 0.3295 (3) | 0.0379 (10) |
| C22 | -0.10027 (14) | 0.1318 (5) | 0.3107 (3) | 0.0466 (11) |
| B1 | -0.05381 (15) | 0.3300 (5) | 0.3956 (4) | 0.0444 (13) |
| N5 | -0.06832 (10) | 0.8557 (4) | 0.1613 (2) | 0.0518 (9) |

| | | | | |
|------|---------------|-------------|------------|-------------|
| H25A | -0.0368 | 0.8299 | 0.1688 | 0.062* |
| H25B | -0.0619 | 0.9008 | 0.2225 | 0.062* |
| C23 | -0.09329 (15) | 0.7284 (5) | 0.1570 (4) | 0.0791 (16) |
| H23A | -0.0763 | 0.6713 | 0.2117 | 0.095* |
| H23B | -0.0979 | 0.6813 | 0.0967 | 0.095* |
| H23C | -0.1221 | 0.7494 | 0.1596 | 0.095* |
| C24 | -0.09181 (16) | 0.9503 (5) | 0.0806 (3) | 0.0898 (18) |
| H24A | -0.0997 | 0.9036 | 0.0190 | 0.108* |
| H24B | -0.0723 | 1.0261 | 0.0831 | 0.108* |
| H24C | -0.1188 | 0.9833 | 0.0869 | 0.108* |
| O7 | 0.0000 | 0.0715 (4) | 0.2500 | 0.0849 (15) |
| H27 | -0.0156 (12) | 0.1236 (14) | 0.271 (3) | 0.102* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| O1 | 0.0322 (16) | 0.061 (2) | 0.0533 (19) | -0.0011 (15) | 0.0168 (15) | 0.0074 (15) |
| O2 | 0.0364 (16) | 0.071 (2) | 0.053 (2) | -0.0023 (16) | 0.0217 (15) | 0.0056 (17) |
| O3 | 0.0504 (18) | 0.088 (3) | 0.058 (2) | -0.0010 (17) | 0.0247 (17) | 0.0188 (19) |
| O4 | 0.0370 (16) | 0.0427 (19) | 0.0651 (19) | -0.0031 (14) | 0.0226 (15) | -0.0049 (15) |
| O5 | 0.0442 (17) | 0.047 (2) | 0.063 (2) | 0.0006 (15) | 0.0262 (16) | -0.0089 (16) |
| O6 | 0.0632 (19) | 0.051 (2) | 0.078 (2) | 0.0015 (17) | 0.0375 (17) | -0.0150 (18) |
| C1 | 0.037 (3) | 0.042 (3) | 0.048 (3) | -0.006 (2) | 0.017 (2) | 0.000 (2) |
| C2 | 0.040 (3) | 0.050 (3) | 0.045 (3) | -0.008 (2) | 0.020 (2) | 0.000 (2) |
| C3 | 0.038 (3) | 0.043 (3) | 0.049 (3) | -0.010 (2) | 0.019 (2) | -0.012 (2) |
| C4 | 0.046 (3) | 0.067 (3) | 0.067 (3) | -0.013 (3) | 0.033 (3) | -0.011 (3) |
| C5 | 0.052 (3) | 0.084 (4) | 0.088 (4) | -0.009 (3) | 0.045 (3) | -0.012 (3) |
| C6 | 0.040 (3) | 0.076 (4) | 0.103 (4) | 0.001 (3) | 0.033 (3) | 0.001 (3) |
| C7 | 0.043 (3) | 0.060 (3) | 0.079 (3) | 0.003 (2) | 0.020 (3) | 0.007 (3) |
| C8 | 0.038 (3) | 0.042 (3) | 0.049 (3) | -0.004 (2) | 0.012 (2) | -0.003 (2) |
| C9 | 0.046 (3) | 0.045 (3) | 0.049 (3) | -0.003 (2) | 0.021 (2) | 0.000 (2) |
| C10 | 0.035 (2) | 0.040 (3) | 0.048 (3) | -0.005 (2) | 0.021 (2) | 0.001 (2) |
| C11 | 0.046 (3) | 0.047 (3) | 0.056 (3) | -0.004 (2) | 0.024 (3) | -0.002 (3) |
| C12 | 0.038 (2) | 0.038 (3) | 0.047 (3) | -0.001 (2) | 0.017 (2) | -0.002 (2) |
| C13 | 0.040 (2) | 0.041 (3) | 0.060 (3) | 0.001 (2) | 0.021 (2) | -0.008 (2) |
| C14 | 0.034 (2) | 0.054 (3) | 0.056 (3) | 0.005 (2) | 0.020 (2) | -0.003 (2) |
| C15 | 0.047 (3) | 0.069 (4) | 0.100 (4) | 0.002 (3) | 0.039 (3) | -0.007 (3) |
| C16 | 0.054 (3) | 0.085 (5) | 0.123 (5) | 0.004 (3) | 0.048 (3) | -0.006 (4) |
| C17 | 0.049 (3) | 0.086 (5) | 0.133 (5) | -0.015 (3) | 0.046 (3) | -0.006 (4) |
| C18 | 0.053 (3) | 0.070 (4) | 0.089 (4) | -0.011 (3) | 0.031 (3) | -0.004 (3) |
| C19 | 0.035 (2) | 0.053 (3) | 0.050 (3) | -0.003 (2) | 0.018 (2) | 0.001 (2) |
| C20 | 0.048 (3) | 0.045 (3) | 0.047 (3) | -0.002 (2) | 0.019 (2) | -0.005 (2) |
| C21 | 0.033 (2) | 0.041 (3) | 0.040 (3) | 0.004 (2) | 0.014 (2) | 0.001 (2) |
| C22 | 0.049 (3) | 0.045 (3) | 0.048 (3) | 0.001 (3) | 0.021 (2) | -0.002 (2) |
| B1 | 0.027 (3) | 0.057 (4) | 0.050 (3) | -0.002 (3) | 0.017 (3) | 0.001 (3) |
| N5 | 0.048 (2) | 0.056 (2) | 0.052 (2) | 0.006 (2) | 0.0200 (19) | -0.003 (2) |
| C23 | 0.069 (3) | 0.074 (4) | 0.097 (4) | -0.020 (3) | 0.035 (3) | -0.026 (3) |
| C24 | 0.106 (4) | 0.096 (5) | 0.065 (4) | 0.040 (4) | 0.029 (3) | 0.013 (3) |

supplementary materials

O7 0.086 (4) 0.060 (3) 0.135 (4) 0.000 0.071 (3) 0.000

Geometric parameters (Å, °)

| | | | |
|-----------|-----------|-------------|-----------|
| O1—C1 | 1.360 (4) | C12—C21 | 1.405 (5) |
| O1—B1 | 1.427 (5) | C13—C14 | 1.406 (5) |
| O2—C11 | 1.316 (4) | C13—H13 | 0.9300 |
| O2—B1 | 1.496 (5) | C14—C15 | 1.412 (5) |
| O3—C11 | 1.227 (4) | C14—C19 | 1.417 (5) |
| O4—C12 | 1.363 (4) | C15—C16 | 1.356 (6) |
| O4—B1 | 1.426 (5) | C15—H15 | 0.9300 |
| O5—C22 | 1.324 (4) | C16—C17 | 1.399 (6) |
| O5—B1 | 1.502 (5) | C16—H16 | 0.9300 |
| O6—C22 | 1.216 (4) | C17—C18 | 1.355 (6) |
| C1—C2 | 1.362 (5) | C17—H17 | 0.9300 |
| C1—C10 | 1.404 (5) | C18—C19 | 1.417 (5) |
| C2—C3 | 1.408 (5) | C18—H18 | 0.9300 |
| C2—H2 | 0.9300 | C19—C20 | 1.400 (5) |
| C3—C4 | 1.412 (5) | C20—C21 | 1.368 (5) |
| C3—C8 | 1.417 (5) | C20—H20 | 0.9300 |
| C4—C5 | 1.355 (6) | C21—C22 | 1.473 (5) |
| C4—H4 | 0.9300 | N5—C24 | 1.466 (5) |
| C5—C6 | 1.395 (6) | N5—C23 | 1.467 (5) |
| C5—H5 | 0.9300 | N5—H25A | 1.0035 |
| C6—C7 | 1.360 (5) | N5—H25B | 0.9530 |
| C6—H6 | 0.9300 | C23—H23A | 0.9600 |
| C7—C8 | 1.412 (5) | C23—H23B | 0.9600 |
| C7—H7 | 0.9300 | C23—H23C | 0.9600 |
| C8—C9 | 1.416 (5) | C24—H24A | 0.9600 |
| C9—C10 | 1.364 (5) | C24—H24B | 0.9600 |
| C9—H9 | 0.9300 | C24—H24C | 0.9600 |
| C10—C11 | 1.482 (5) | O7—H27 | 0.85 (3) |
| C12—C13 | 1.361 (5) | | |
| C1—O1—B1 | 116.9 (3) | C16—C15—H15 | 119.9 |
| C11—O2—B1 | 122.2 (3) | C14—C15—H15 | 119.9 |
| C12—O4—B1 | 116.5 (3) | C15—C16—C17 | 121.8 (5) |
| C22—O5—B1 | 121.6 (3) | C15—C16—H16 | 119.1 |
| O1—C1—C2 | 120.0 (4) | C17—C16—H16 | 119.1 |
| O1—C1—C10 | 119.9 (4) | C18—C17—C16 | 119.2 (5) |
| C2—C1—C10 | 120.1 (4) | C18—C17—H17 | 120.4 |
| C1—C2—C3 | 120.5 (4) | C16—C17—H17 | 120.4 |
| C1—C2—H2 | 119.7 | C17—C18—C19 | 121.4 (5) |
| C3—C2—H2 | 119.7 | C17—C18—H18 | 119.3 |
| C2—C3—C4 | 121.9 (4) | C19—C18—H18 | 119.3 |
| C2—C3—C8 | 120.2 (4) | C20—C19—C18 | 122.9 (4) |
| C4—C3—C8 | 117.9 (4) | C20—C19—C14 | 118.5 (4) |
| C5—C4—C3 | 121.0 (5) | C18—C19—C14 | 118.6 (4) |
| C5—C4—H4 | 119.5 | C21—C20—C19 | 121.6 (4) |
| C3—C4—H4 | 119.5 | C21—C20—H20 | 119.2 |

| | | | |
|--------------|------------|-----------------|------------|
| C4—C5—C6 | 120.9 (4) | C19—C20—H20 | 119.2 |
| C4—C5—H5 | 119.5 | C20—C21—C12 | 119.7 (4) |
| C6—C5—H5 | 119.5 | C20—C21—C22 | 119.8 (4) |
| C7—C6—C5 | 120.2 (4) | C12—C21—C22 | 120.5 (4) |
| C7—C6—H6 | 119.9 | O6—C22—O5 | 120.7 (4) |
| C5—C6—H6 | 119.9 | O6—C22—C21 | 123.3 (4) |
| C6—C7—C8 | 120.4 (5) | O5—C22—C21 | 116.0 (4) |
| C6—C7—H7 | 119.8 | O4—B1—O1 | 110.7 (4) |
| C8—C7—H7 | 119.8 | O4—B1—O2 | 107.7 (3) |
| C7—C8—C9 | 123.0 (4) | O1—B1—O2 | 112.4 (3) |
| C7—C8—C3 | 119.6 (4) | O4—B1—O5 | 112.0 (3) |
| C9—C8—C3 | 117.4 (4) | O1—B1—O5 | 107.9 (3) |
| C10—C9—C8 | 121.4 (4) | O2—B1—O5 | 106.1 (4) |
| C10—C9—H9 | 119.3 | C24—N5—C23 | 113.5 (4) |
| C8—C9—H9 | 119.3 | C24—N5—H25A | 115.4 |
| C9—C10—C1 | 120.4 (4) | C23—N5—H25A | 107.4 |
| C9—C10—C11 | 119.6 (4) | C24—N5—H25B | 109.4 |
| C1—C10—C11 | 120.0 (4) | C23—N5—H25B | 110.4 |
| O3—C11—O2 | 119.9 (4) | H25A—N5—H25B | 99.9 |
| O3—C11—C10 | 123.9 (4) | N5—C23—H23A | 109.5 |
| O2—C11—C10 | 116.2 (4) | N5—C23—H23B | 109.5 |
| C13—C12—O4 | 120.1 (4) | H23A—C23—H23B | 109.5 |
| C13—C12—C21 | 120.1 (4) | N5—C23—H23C | 109.5 |
| O4—C12—C21 | 119.7 (4) | H23A—C23—H23C | 109.5 |
| C12—C13—C14 | 121.2 (4) | H23B—C23—H23C | 109.5 |
| C12—C13—H13 | 119.4 | N5—C24—H24A | 109.5 |
| C14—C13—H13 | 119.4 | N5—C24—H24B | 109.5 |
| C13—C14—C15 | 122.3 (4) | H24A—C24—H24B | 109.5 |
| C13—C14—C19 | 118.9 (4) | N5—C24—H24C | 109.5 |
| C15—C14—C19 | 118.8 (4) | H24A—C24—H24C | 109.5 |
| C16—C15—C14 | 120.2 (5) | H24B—C24—H24C | 109.5 |
| B1—O1—C1—C2 | 152.4 (4) | C13—C14—C15—C16 | 180.0 (4) |
| B1—O1—C1—C10 | -28.0 (5) | C19—C14—C15—C16 | -0.2 (7) |
| O1—C1—C2—C3 | 179.7 (4) | C14—C15—C16—C17 | 0.6 (8) |
| C10—C1—C2—C3 | 0.1 (6) | C15—C16—C17—C18 | -0.4 (9) |
| C1—C2—C3—C4 | 178.0 (4) | C16—C17—C18—C19 | -0.3 (8) |
| C1—C2—C3—C8 | -0.6 (6) | C17—C18—C19—C20 | 179.8 (5) |
| C2—C3—C4—C5 | -177.7 (4) | C17—C18—C19—C14 | 0.8 (7) |
| C8—C3—C4—C5 | 1.0 (6) | C13—C14—C19—C20 | 0.2 (6) |
| C3—C4—C5—C6 | -0.7 (7) | C15—C14—C19—C20 | -179.6 (4) |
| C4—C5—C6—C7 | -0.2 (8) | C13—C14—C19—C18 | 179.3 (4) |
| C5—C6—C7—C8 | 0.8 (7) | C15—C14—C19—C18 | -0.5 (6) |
| C6—C7—C8—C9 | 177.5 (4) | C18—C19—C20—C21 | -177.6 (4) |
| C6—C7—C8—C3 | -0.5 (6) | C14—C19—C20—C21 | 1.4 (6) |
| C2—C3—C8—C7 | 178.3 (4) | C19—C20—C21—C12 | -1.7 (6) |
| C4—C3—C8—C7 | -0.4 (6) | C19—C20—C21—C22 | 178.9 (4) |
| C2—C3—C8—C9 | 0.2 (6) | C13—C12—C21—C20 | 0.2 (6) |
| C4—C3—C8—C9 | -178.5 (4) | O4—C12—C21—C20 | -178.1 (3) |
| C7—C8—C9—C10 | -177.3 (4) | C13—C12—C21—C22 | 179.6 (4) |

supplementary materials

| | | | |
|-----------------|------------|----------------|------------|
| C3—C8—C9—C10 | 0.7 (6) | O4—C12—C21—C22 | 1.4 (5) |
| C8—C9—C10—C1 | -1.2 (6) | B1—O5—C22—O6 | -171.3 (4) |
| C8—C9—C10—C11 | 178.9 (4) | B1—O5—C22—C21 | 8.7 (5) |
| O1—C1—C10—C9 | -178.8 (4) | C20—C21—C22—O6 | 8.0 (6) |
| C2—C1—C10—C9 | 0.8 (6) | C12—C21—C22—O6 | -171.4 (4) |
| O1—C1—C10—C11 | 1.1 (6) | C20—C21—C22—O5 | -172.0 (4) |
| C2—C1—C10—C11 | -179.3 (4) | C12—C21—C22—O5 | 8.5 (5) |
| B1—O2—C11—O3 | -173.9 (4) | C12—O4—B1—O1 | 163.5 (3) |
| B1—O2—C11—C10 | 6.0 (6) | C12—O4—B1—O2 | -73.2 (4) |
| C9—C10—C11—O3 | 9.7 (6) | C12—O4—B1—O5 | 43.1 (5) |
| C1—C10—C11—O3 | -170.2 (4) | C1—O1—B1—O4 | 161.8 (3) |
| C9—C10—C11—O2 | -170.2 (4) | C1—O1—B1—O2 | 41.4 (5) |
| C1—C10—C11—O2 | 9.9 (6) | C1—O1—B1—O5 | -75.3 (4) |
| B1—O4—C12—C13 | 153.2 (4) | C11—O2—B1—O4 | -153.3 (4) |
| B1—O4—C12—C21 | -28.6 (5) | C11—O2—B1—O1 | -31.1 (6) |
| O4—C12—C13—C14 | 179.7 (4) | C11—O2—B1—O5 | 86.6 (4) |
| C21—C12—C13—C14 | 1.5 (6) | C22—O5—B1—O4 | -34.1 (5) |
| C12—C13—C14—C15 | 178.2 (4) | C22—O5—B1—O1 | -156.1 (3) |
| C12—C13—C14—C19 | -1.7 (6) | C22—O5—B1—O2 | 83.2 (4) |

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|------------------------------------|----------|-------------|-------------|---------------|
| N5—H25A \cdots O3 ⁱ | 1.00 | 1.88 | 2.849 (4) | 162 |
| N5—H25B \cdots O6 ⁱⁱ | 0.95 | 2.12 | 2.839 (4) | 131 |
| N5—H25B \cdots O3 ⁱⁱⁱ | 0.95 | 2.33 | 2.871 (4) | 116 |
| O7—H27 \cdots O5 | 0.85 (3) | 2.18 (4) | 3.010 (3) | 163.3 (12) |

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

Fig. 1

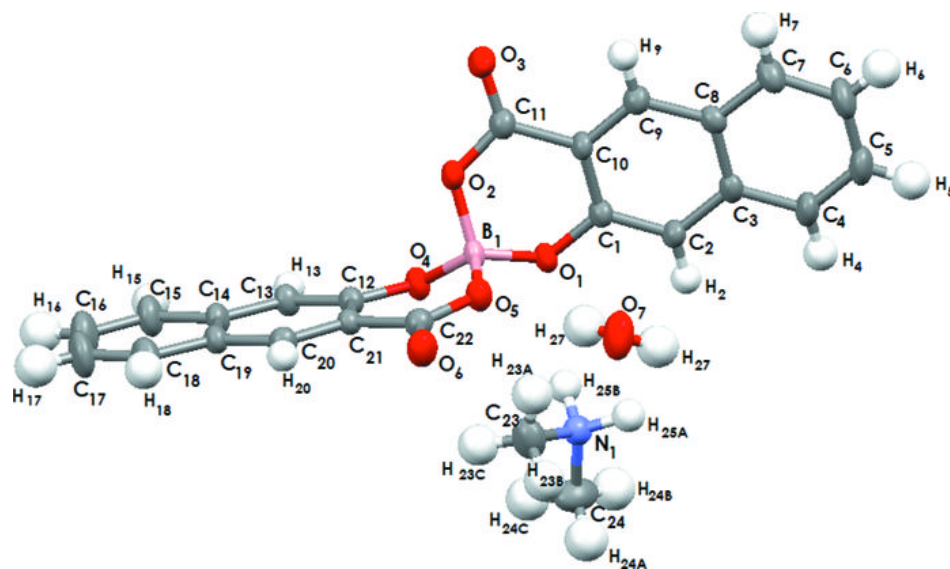


Fig. 2

