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4-Methylpyridinium bis(pyrocatecholato-κ^2O,O')-borate catechol solvate

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Key indicators

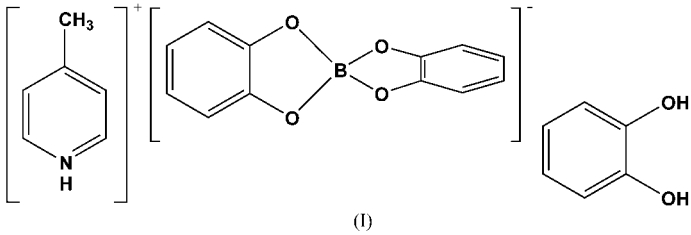
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.110
Data-to-parameter ratio = 16.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Unlike the previously reported salts of the 4-methylpyridinium cation and the bis(pyrocatecholato)borate anion [Clegg *et al.* (1998). *Acta Cryst.* C**54**, 1875–1880], the title compound, $C_6H_8N^+\cdot C_{12}H_8BO_4^-\cdot C_6H_6O_2$, is a solvate containing a molecule of catechol. The crystal packing is influenced by N—H···O and O—H···O hydrogen bonds.

Comment

In addition to the ammonium cations $[NH_4]^+$ (Goddard *et al.*, 1993) and $[NH_2Me_2]^+$ (Clegg, Elsegood *et al.*, 1998), the bis(pyrocatecholato)borate anion $[B(1,2\text{-}O_2C_6H_4)_2]^-$ has been found to crystallize with a number of pyridinium cations. These include $[2\text{-}MeC_5H_4NH]^+$ and two polymorphs containing $[4\text{-}MeC_5H_4NH]^+$ (Clegg, Scott *et al.*, 1998). $[NHEt_3]^+$ (Mohr *et al.*, 1990) and the unsubstituted pyridinium cation $[C_5H_5NH]^+$ (Griffin *et al.*, 1996) also form salts with $[B(1,2\text{-}O_2C_6H_4)_2]^-$, although in these cases a molecule of catechol is incorporated into the structure. The structures of [1,10-phenH][$B(1,2\text{-}O_2C_6H_4)_2$] (phen = phenanthroline), and its dichloromethane solvate (Clegg, Scott *et al.*, 1998) have also been determined as has the structure of the phosphonium salt [$PHMe_3$][$B(1,2\text{-}O_2C_6H_4)_2$] (Clegg, Scott *et al.*, 1998) and a range of salts containing cationic rhodium or iridium phosphine complexes (Clegg *et al.*, 1999). In this paper, we report the structure of a $[4\text{-}MeC_5H_4NH]^+$ salt of $[(C_6H_4O_2)_2B]^-$ that, unlike the crystal structures previously reported for salts of $[4\text{-}MeC_5H_4NH]^+$ and $[(C_6H_4O_2)_2B]^-$ (Clegg, Scott *et al.*, 1998), but in common with the pyridinium and triethylammonium salts, includes a molecule of catechol in the structure.



(I)

The molecular structure of (I) is shown in Fig. 1. The crystal structure contains hydrogen bonds between the catechol molecules and the catecholate ligands of the $[B(1,2\text{-}O_2C_6H_4)_2]^-$ anions. The 4-methylpyridinium cations also form hydrogen bonds to the catechol molecules, producing a ribbon structure (see Fig. 2). These ribbons crosslink through hydrogen bonds between the catecholate ligands and pyridinium cations to form a one-dimensional hydrogen-bonded polymer (see Fig. 3).

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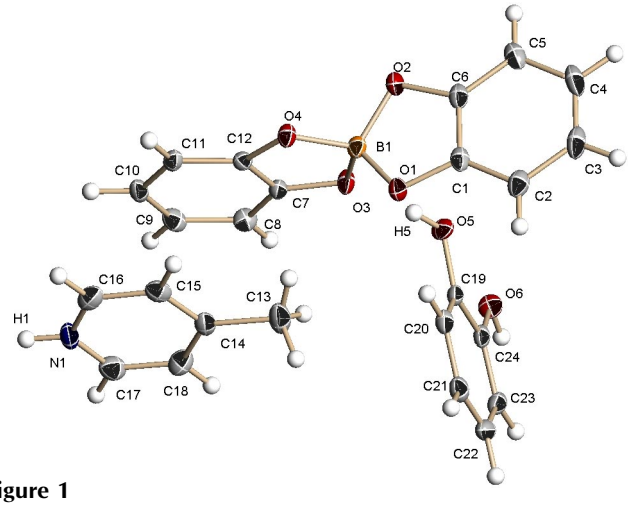

Figure 1
The molecular structure of (I), showing displacement ellipsoids drawn at the 50% probability level. The solvent catechol molecule has been transformed by the symmetry operation (x, y, $z-1$).

Experimental

$B_2(cat)_3$ (0.1 g, 0.029 mmol) was dissolved in CH_2Cl_2 (4 ml) in a small Schlenk tube to which 4-picoline (0.04 g, 0.058 mmol) was added and the mixture stirred for 1 h at room temperature. After this time, hexane (4 ml) was added as an overlayer and solvent diffusion over a period of days at 243 K afforded colourless crystals of [4-MeC_5H_4NH][$B(1,2\text{-}O_2C_6H_4)_2$]. $C_{18}H_{16}BNO_4$ requires: N 4.35, C 67.30, H 5.00%; found: N 4.40, C 67.65, H 5.75%. ^{11}B {^1H} NMR: δ 13.2 (*s*). Although the microanalytical data are consistent with the formula [4–MeC_5H_4NH][$B(1,2\text{-}O_2C_6H_4)_2$], confirmed by X-ray crystallography, one (colourless) crystal examined was found to have the composition [4-MeC_5H_4NH][$B(1,2\text{-}O_2C_6H_4)_2$]·1,2-$(HO)_2C_6H_4$. There are no obvious morphology differences between the two phases.

Crystal data

$C_6H_8N^+\cdot C_{12}H_8BO_4^-\cdot C_6H_6O_2$
M_r = 431.24
Monoclinic, $P2_1/n$
a = 10.0007 (14) Å
b = 12.9573 (17) Å
c = 16.396 (3) Å
β = 96.872 (11)°
V = 2109.3 (6) Å^3
Z = 4
D_x = 1.355 Mg m^{-3}
D_m = 1.340 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4189 reflections
θ = 2.5–25.8°
μ = 0.10 mm^{-1}
T = 100 (3) K
Block, colourless
0.05 × 0.05 × 0.05 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.929, T_{max} = 0.990
23718 measured reflections
4828 independent reflections
3973 reflections with $I > 2\sigma(I)$'
R_{int} = 0.037
θ_{max} = 27.5°
$h = -12 \rightarrow 12$
$k = -16 \rightarrow 16$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.110
S = 1.03
4828 reflections
299 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0504P)^2 + 0.7965P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

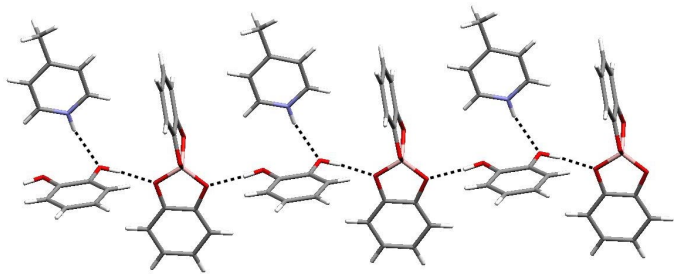

Figure 2
Stick representation (colour code: C grey, H white, O red, B pink, N blue) of the hydrogen-bonded (dashed lines) ribbon polymers formed in (I).

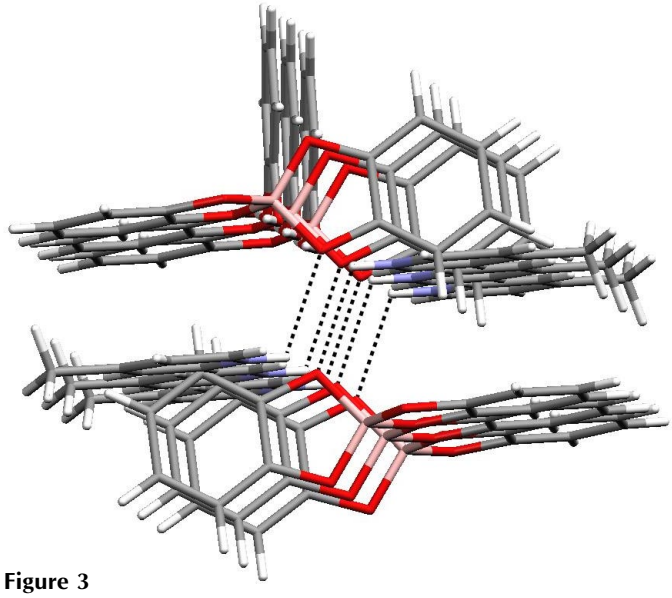

Figure 3
Cross-linking hydrogen bonds (dashed lines) between ribbons in (I). The colour code is as in Fig. 2.

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.3662 (17)	B1—O1	1.4746 (19)
C6—O2	1.3681 (17)	B1—O4	1.4820 (19)
C7—O3	1.3659 (17)	C19—O5	1.3731 (17)
C12—O4	1.3756 (16)	C24—O6	1.3664 (18)
B1—O2	1.4742 (19)		
O2—B1—O1	105.58 (11)	O4—B1—O3	103.32 (11)
O2—B1—O4	112.77 (12)	C1—O1—B1	105.89 (11)
O1—B1—O4	113.49 (12)	C6—O2—B1	105.98 (11)
O2—B1—O3	111.09 (12)	C7—O3—B1	108.12 (11)
O1—B1—O3	110.74 (12)	C12—O4—B1	108.15 (11)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2	0.872 (15)	2.425 (18)	2.8864 (17)	113.6 (14)
N1—H1···O5^{i}	0.872 (15)	2.096 (17)	2.8621 (18)	146.3 (16)
N1—H1···O6^{i}	0.872 (15)	2.587 (17)	3.1073 (18)	119.2 (14)
O5—H5···O3^{ii}	0.864 (15)	1.788 (16)	2.6469 (15)	172.5 (17)
O6—H6···O4^{iii}	0.871 (15)	1.793 (16)	2.6600 (15)	173.5 (18)

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

The NH H atom of the pyridinium cation and all hydroxy H atoms were located in difference maps. Distance restraints of 0.88 (3) and 0.84 (3) Å were applied to the N—H and O—H bond lengths, respectively. Methyl H atoms were located using a rotating group refinement, with C—H bond lengths constrained to 0.96 Å. All other H atoms were positioned in ideal geometries and refined by riding on their carrier atom. All H atoms were assigned displacement parameters equal to 1.5 times (methyl and hydroxyl H atoms) or 1.2 times (all other H atoms) U_{eq} of their parent atom.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT* and *SHELXTL* (Bruker, 2002); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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supporting information

Acta Cryst. (2004). E**60**, o1140–o1142 [https://doi.org/10.1107/S1600536804013108]

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(I)

Crystal data

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Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 10.0007\ (14)$ Å
$b = 12.9573\ (17)$ Å
$c = 16.396\ (3)$ Å
$\beta = 96.872\ (11)°$
$V = 2109.3\ (6)$ Å^3
$Z = 4$
$F(000) = 904$
$D_x = 1.355$ Mg m^{-3}
$D_m = 1.340$ Mg m^{-3}
D_m measured by not measured
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4189 reflections
$\theta = 2.5–25.8°$
$\mu = 0.10$ mm^{-1}
$T = 100$ K
Block, colourless
0.05 × 0.05 × 0.05 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.192 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
$T_{min} = 0.929$, $T_{max} = 0.990$
23718 measured reflections
4828 independent reflections
3973 reflections with $I > 2\sigma(I)'$
$R_{int} = 0.037$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.0°$
$h = -12\rightarrow12$
$k = -16\rightarrow16$
$l = -21\rightarrow21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.110$
$S = 1.03$
4828 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
$w = 1/[\sigma^2(F_o^2) + (0.0504P)^2 + 0.7965P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.81009 (14)	0.73736 (12)	−0.08826 (8)	0.0205 (3)
C2	0.83417 (16)	0.79292 (13)	−0.15693 (9)	0.0260 (3)
H2	0.8475	0.8655	−0.1547	0.031*
C3	0.83815 (16)	0.73753 (14)	−0.23020 (9)	0.0301 (4)
H3	0.8541	0.7734	−0.2786	0.036*
C4	0.81930 (16)	0.63191 (14)	−0.23323 (9)	0.0303 (4)
H4	0.8221	0.5966	−0.2838	0.036*
C5	0.79609 (15)	0.57553 (13)	−0.16294 (9)	0.0262 (3)
H5A	0.7840	0.5028	−0.1648	0.031*
C6	0.79161 (14)	0.63043 (12)	−0.09140 (8)	0.0211 (3)
C7	0.90025 (14)	0.66835 (10)	0.17549 (8)	0.0172 (3)
C8	0.98987 (15)	0.66026 (11)	0.24587 (9)	0.0219 (3)
H8	1.0839	0.6533	0.2435	0.026*
C9	0.93661 (16)	0.66266 (11)	0.32102 (9)	0.0243 (3)
H9	0.9957	0.6573	0.3707	0.029*
C10	0.79955 (16)	0.67271 (11)	0.32448 (9)	0.0225 (3)
H10	0.7661	0.6734	0.3763	0.027*
C11	0.70917 (15)	0.68191 (10)	0.25224 (9)	0.0195 (3)
H11	0.6150	0.6890	0.2541	0.023*
C12	0.76268 (14)	0.68016 (10)	0.17872 (8)	0.0161 (3)
B1	0.79639 (16)	0.68115 (13)	0.04148 (10)	0.0187 (3)
O1	0.80197 (10)	0.77338 (8)	−0.01069 (6)	0.0211 (2)
O2	0.76987 (10)	0.59384 (8)	−0.01582 (6)	0.0222 (2)
O3	0.92679 (10)	0.66676 (8)	0.09570 (6)	0.0202 (2)
O4	0.69591 (10)	0.68919 (8)	0.10068 (6)	0.0191 (2)
C13	0.6421 (2)	0.43363 (13)	0.34169 (10)	0.0342 (4)
H13A	0.7205	0.4080	0.3776	0.051*
H13B	0.5640	0.3899	0.3479	0.051*
H13C	0.6229	0.5048	0.3569	0.051*
C14	0.67129 (16)	0.43076 (11)	0.25411 (9)	0.0224 (3)
C15	0.80203 (16)	0.42629 (11)	0.23393 (9)	0.0236 (3)
H15	0.8758	0.4244	0.2762	0.028*
C16	0.82501 (16)	0.42467 (11)	0.15307 (10)	0.0264 (3)
H16	0.9145	0.4217	0.1393	0.032*
C17	0.59375 (17)	0.43127 (12)	0.11020 (10)	0.0290 (4)

H17	0.5221	0.4329	0.0666	0.035*
C18	0.56675 (16)	0.43292 (12)	0.19011 (10)	0.0271 (3)
H18	0.4761	0.4356	0.2019	0.033*
N1	0.72116 (15)	0.42732 (10)	0.09378 (8)	0.0266 (3)
H1	0.7377 (18)	0.4251 (14)	0.0428 (9)	0.032*
C19	0.77804 (14)	0.24734 (11)	0.95478 (8)	0.0177 (3)
C20	0.85497 (15)	0.15817 (11)	0.96525 (8)	0.0205 (3)
H20	0.9505	0.1625	0.9723	0.025*
C21	0.79204 (16)	0.06230 (12)	0.96543 (9)	0.0234 (3)
H21	0.8446	0.0011	0.9717	0.028*
C22	0.65281 (16)	0.05611 (12)	0.95649 (9)	0.0253 (3)
H22	0.6101	−0.0093	0.9568	0.030*
C23	0.57540 (15)	0.14533 (12)	0.94700 (8)	0.0227 (3)
H23	0.4799	0.1407	0.9415	0.027*
C24	0.63701 (14)	0.24125 (11)	0.94544 (8)	0.0188 (3)
O5	0.83169 (10)	0.34485 (8)	0.95439 (7)	0.0216 (2)
H5	0.9133 (15)	0.3402 (14)	0.9425 (11)	0.032*
O6	0.56912 (10)	0.33270 (8)	0.93487 (6)	0.0233 (2)
H6	0.4834 (15)	0.3205 (14)	0.9229 (12)	0.035*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0141 (7)	0.0329 (8)	0.0142 (7)	−0.0007 (6)	0.0004 (5)	−0.0012 (6)
C2	0.0220 (8)	0.0370 (9)	0.0189 (7)	−0.0014 (6)	0.0017 (6)	0.0054 (6)
C3	0.0214 (8)	0.0539 (11)	0.0153 (7)	−0.0004 (7)	0.0029 (6)	0.0049 (7)
C4	0.0215 (8)	0.0537 (11)	0.0158 (7)	0.0021 (7)	0.0024 (6)	−0.0084 (7)
C5	0.0198 (7)	0.0377 (9)	0.0211 (7)	0.0011 (6)	0.0023 (6)	−0.0076 (6)
C6	0.0149 (7)	0.0328 (8)	0.0156 (7)	−0.0002 (6)	0.0017 (5)	0.0000 (6)
C7	0.0198 (7)	0.0165 (6)	0.0162 (7)	−0.0013 (5)	0.0050 (5)	−0.0002 (5)
C8	0.0191 (7)	0.0227 (7)	0.0232 (7)	0.0017 (6)	0.0000 (6)	0.0017 (6)
C9	0.0334 (9)	0.0212 (7)	0.0168 (7)	0.0002 (6)	−0.0033 (6)	0.0015 (6)
C10	0.0356 (9)	0.0172 (7)	0.0157 (7)	−0.0009 (6)	0.0073 (6)	0.0000 (5)
C11	0.0219 (7)	0.0170 (7)	0.0207 (7)	−0.0002 (5)	0.0078 (6)	−0.0006 (5)
C12	0.0177 (7)	0.0148 (6)	0.0156 (7)	−0.0009 (5)	0.0018 (5)	0.0006 (5)
B1	0.0151 (7)	0.0239 (8)	0.0175 (8)	−0.0006 (6)	0.0038 (6)	−0.0002 (6)
O1	0.0243 (5)	0.0252 (5)	0.0140 (5)	−0.0018 (4)	0.0034 (4)	−0.0003 (4)
O2	0.0267 (6)	0.0249 (5)	0.0159 (5)	−0.0026 (4)	0.0058 (4)	−0.0014 (4)
O3	0.0152 (5)	0.0311 (6)	0.0150 (5)	0.0006 (4)	0.0045 (4)	0.0008 (4)
O4	0.0146 (5)	0.0276 (5)	0.0152 (5)	0.0003 (4)	0.0029 (4)	0.0005 (4)
C13	0.0561 (12)	0.0256 (8)	0.0235 (8)	−0.0040 (8)	0.0153 (8)	−0.0021 (6)
C14	0.0337 (9)	0.0136 (6)	0.0211 (7)	−0.0020 (6)	0.0079 (6)	−0.0010 (5)
C15	0.0282 (8)	0.0158 (7)	0.0260 (8)	−0.0013 (6)	−0.0004 (6)	−0.0003 (6)
C16	0.0262 (8)	0.0189 (7)	0.0362 (9)	−0.0023 (6)	0.0117 (7)	−0.0017 (6)
C17	0.0335 (9)	0.0276 (8)	0.0245 (8)	0.0032 (7)	−0.0025 (7)	−0.0013 (6)
C18	0.0238 (8)	0.0271 (8)	0.0315 (8)	−0.0009 (6)	0.0074 (6)	−0.0036 (6)
N1	0.0420 (8)	0.0218 (6)	0.0180 (6)	−0.0013 (6)	0.0114 (6)	0.0006 (5)
C19	0.0185 (7)	0.0228 (7)	0.0125 (6)	−0.0034 (5)	0.0047 (5)	−0.0013 (5)

C20	0.0199 (7)	0.0270 (8)	0.0151 (7)	0.0008 (6)	0.0044 (5)	0.0000 (6)
C21	0.0323 (9)	0.0228 (7)	0.0158 (7)	0.0022 (6)	0.0051 (6)	0.0013 (6)
C22	0.0343 (9)	0.0253 (8)	0.0167 (7)	−0.0100 (6)	0.0049 (6)	−0.0012 (6)
C23	0.0203 (7)	0.0327 (8)	0.0152 (7)	−0.0083 (6)	0.0022 (5)	0.0009 (6)
C24	0.0188 (7)	0.0264 (7)	0.0115 (6)	−0.0014 (6)	0.0033 (5)	−0.0002 (5)
O5	0.0155 (5)	0.0223 (5)	0.0285 (6)	−0.0021 (4)	0.0085 (4)	−0.0027 (4)
O6	0.0140 (5)	0.0294 (6)	0.0262 (6)	0.0002 (4)	0.0014 (4)	−0.0008 (4)

Geometric parameters (Å, º)

C1—O1	1.3662 (17)	C13—H13A	0.9800
C1—C2	1.382 (2)	C13—H13B	0.9800
C1—C6	1.398 (2)	C13—H13C	0.9800
C2—C3	1.404 (2)	C14—C15	1.388 (2)
C2—H2	0.9500	C14—C18	1.390 (2)
C3—C4	1.382 (3)	C15—C16	1.373 (2)
C3—H3	0.9500	C15—H15	0.9500
C4—C5	1.407 (2)	C16—N1	1.335 (2)
C4—H4	0.9500	C16—H16	0.9500
C5—C6	1.377 (2)	C17—N1	1.335 (2)
C5—H5A	0.9500	C17—C18	1.369 (2)
C6—O2	1.3681 (17)	C17—H17	0.9500
C7—O3	1.3659 (17)	C18—H18	0.9500
C7—C8	1.378 (2)	N1—H1	0.871 (14)
C7—C12	1.392 (2)	C19—O5	1.3731 (17)
C8—C9	1.400 (2)	C19—C20	1.387 (2)
C8—H8	0.9500	C19—C24	1.403 (2)
C9—C10	1.385 (2)	C20—C21	1.393 (2)
C9—H9	0.9500	C20—H20	0.9500
C10—C11	1.406 (2)	C21—C22	1.385 (2)
C10—H10	0.9500	C21—H21	0.9500
C11—C12	1.3764 (19)	C22—C23	1.390 (2)
C11—H11	0.9500	C22—H22	0.9500
C12—O4	1.3756 (16)	C23—C24	1.389 (2)
B1—O2	1.4742 (19)	C23—H23	0.9500
B1—O1	1.4746 (19)	C24—O6	1.3664 (18)
B1—O4	1.4820 (19)	O5—H5	0.864 (14)
B1—O3	1.4993 (18)	O6—H6	0.871 (15)
C13—C14	1.499 (2)		

O1—C1—C2	128.04 (14)	C12—O4—B1	108.15 (11)
O1—C1—C6	110.52 (12)	C14—C13—H13A	109.5
C2—C1—C6	121.44 (14)	C14—C13—H13B	109.5
C1—C2—C3	117.16 (15)	H13A—C13—H13B	109.5
C1—C2—H2	121.4	C14—C13—H13C	109.5
C3—C2—H2	121.4	H13A—C13—H13C	109.5
C4—C3—C2	121.29 (15)	H13B—C13—H13C	109.5
C4—C3—H3	119.4	C15—C14—C18	117.76 (14)

C2—C3—H3	119.4	C15—C14—C13	121.74 (15)
C3—C4—C5	121.34 (14)	C18—C14—C13	120.50 (15)
C3—C4—H4	119.3	C16—C15—C14	120.16 (14)
C5—C4—H4	119.3	C16—C15—H15	119.9
C6—C5—C4	117.06 (16)	C14—C15—H15	119.9
C6—C5—H5A	121.5	N1—C16—C15	119.82 (15)
C4—C5—H5A	121.5	N1—C16—H16	120.1
O2—C6—C5	128.12 (15)	C15—C16—H16	120.1
O2—C6—C1	110.17 (12)	N1—C17—C18	119.74 (15)
C5—C6—C1	121.71 (14)	N1—C17—H17	120.1
O3—C7—C8	128.28 (13)	C18—C17—H17	120.1
O3—C7—C12	110.14 (12)	C17—C18—C14	120.36 (15)
C8—C7—C12	121.57 (13)	C17—C18—H18	119.8
C7—C8—C9	117.23 (14)	C14—C18—H18	119.8
C7—C8—H8	121.4	C17—N1—C16	122.16 (14)
C9—C8—H8	121.4	C17—N1—H1	119.4 (12)
C10—C9—C8	121.39 (13)	C16—N1—H1	118.4 (12)
C10—C9—H9	119.3	O5—C19—C20	123.76 (13)
C8—C9—H9	119.3	O5—C19—C24	116.08 (12)
C9—C10—C11	120.87 (13)	C20—C19—C24	120.14 (13)
C9—C10—H10	119.6	C19—C20—C21	119.93 (14)
C11—C10—H10	119.6	C19—C20—H20	120.0
C12—C11—C10	117.27 (14)	C21—C20—H20	120.0
C12—C11—H11	121.4	C22—C21—C20	120.02 (14)
C10—C11—H11	121.4	C22—C21—H21	120.0
O4—C12—C11	128.09 (13)	C20—C21—H21	120.0
O4—C12—C7	110.25 (12)	C21—C22—C23	120.21 (14)
C11—C12—C7	121.66 (13)	C21—C22—H22	119.9
O2—B1—O1	105.58 (11)	C23—C22—H22	119.9
O2—B1—O4	112.77 (12)	C24—C23—C22	120.27 (14)
O1—B1—O4	113.49 (12)	C24—C23—H23	119.9
O2—B1—O3	111.09 (12)	C22—C23—H23	119.9
O1—B1—O3	110.74 (12)	O6—C24—C23	124.31 (13)
O4—B1—O3	103.32 (11)	O6—C24—C19	116.29 (12)
C1—O1—B1	105.89 (11)	C23—C24—C19	119.40 (13)
C6—O2—B1	105.98 (11)	C19—O5—H5	108.5 (12)
C7—O3—B1	108.12 (11)	C24—O6—H6	109.4 (12)

O1—C1—C2—C3	179.81 (14)	O3—B1—O2—C6	−106.83 (13)
C6—C1—C2—C3	0.5 (2)	C8—C7—O3—B1	−178.44 (14)
C1—C2—C3—C4	−0.3 (2)	C12—C7—O3—B1	1.39 (15)
C2—C3—C4—C5	−0.3 (2)	O2—B1—O3—C7	−121.96 (12)
C3—C4—C5—C6	0.7 (2)	O1—B1—O3—C7	121.05 (12)
C4—C5—C6—O2	179.55 (14)	O4—B1—O3—C7	−0.79 (14)
C4—C5—C6—C1	−0.4 (2)	C11—C12—O4—B1	−179.18 (14)
O1—C1—C6—O2	0.45 (16)	C7—C12—O4—B1	0.93 (15)
C2—C1—C6—O2	179.84 (13)	O2—B1—O4—C12	119.94 (12)
O1—C1—C6—C5	−179.60 (13)	O1—B1—O4—C12	−120.06 (12)

C2—C1—C6—C5	−0.2 (2)	O3—B1—O4—C12	−0.09 (14)
O3—C7—C8—C9	−179.13 (13)	C18—C14—C15—C16	0.3 (2)
C12—C7—C8—C9	1.0 (2)	C13—C14—C15—C16	−179.44 (13)
C7—C8—C9—C10	0.0 (2)	C14—C15—C16—N1	0.0 (2)
C8—C9—C10—C11	−0.7 (2)	N1—C17—C18—C14	0.2 (2)
C9—C10—C11—C12	0.2 (2)	C15—C14—C18—C17	−0.4 (2)
C10—C11—C12—O4	−179.01 (13)	C13—C14—C18—C17	179.38 (14)
C10—C11—C12—C7	0.9 (2)	C18—C17—N1—C16	0.1 (2)
O3—C7—C12—O4	−1.49 (15)	C15—C16—N1—C17	−0.2 (2)
C8—C7—C12—O4	178.36 (12)	O5—C19—C20—C21	179.52 (13)
O3—C7—C12—C11	178.61 (12)	C24—C19—C20—C21	0.8 (2)
C8—C7—C12—C11	−1.5 (2)	C19—C20—C21—C22	−1.0 (2)
C2—C1—O1—B1	−171.26 (15)	C20—C21—C22—C23	0.2 (2)
C6—C1—O1—B1	8.08 (15)	C21—C22—C23—C24	0.8 (2)
O2—B1—O1—C1	−12.99 (14)	C22—C23—C24—O6	178.73 (13)
O4—B1—O1—C1	−137.00 (12)	C22—C23—C24—C19	−1.0 (2)
O3—B1—O1—C1	107.34 (12)	O5—C19—C24—O6	1.66 (18)
C5—C6—O2—B1	171.28 (14)	C20—C19—C24—O6	−179.55 (12)
C1—C6—O2—B1	−8.77 (15)	O5—C19—C24—C23	−178.63 (12)
O1—B1—O2—C6	13.27 (14)	C20—C19—C24—C23	0.2 (2)
O4—B1—O2—C6	137.73 (12)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2	0.87 (2)	2.43 (2)	2.8864 (17)	114 (1)
N1—H1···O5^{i}	0.87 (2)	2.10 (2)	2.8621 (18)	146 (2)
N1—H1···O6^{i}	0.87 (2)	2.59 (2)	3.1073 (18)	119 (1)
O5—H5···O3^{ii}	0.86 (2)	1.79 (2)	2.6469 (15)	173 (2)
O6—H6···O4^{iii}	0.87 (2)	1.79 (2)	2.6600 (15)	174 (2)

Symmetry codes: (i) *x*, *y*, *z*−1; (ii) −*x*+2, −*y*+1, −*z*+1; (iii) −*x*+1, −*y*+1, −*z*+1.