Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-(1*H*-Benzimidazol-2-ylmethoxy)-3-methoxybenzaldehyde tetrahydrate

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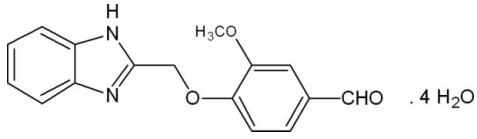
Received 30 June 2011; accepted 6 July 2011

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; disorder in main residue; R factor = 0.049; wR factor = 0.152; data-to-parameter ratio = 16.6.

In the title compound, $C_{16}H_{14}N_2O_3\cdot4H_2O$, the dihedral angle between the mean planes of the benzimidazole ring system and benzene ring is 2.9 (1)°. The aldehyde group is disordered over two sets of sites with refined occupancies of 0.559 (4) and 0.441 (4). In the crystal, extensive intermolecular O—H···O, O—H···N and N—H···O hydrogen bonds in concert with weak π–π stacking interactions [centroid–centroid distances = 3.6104 (9), 3.6288 (9) and 3.9167 (10) Å] create a three-dimensional network.

Related literature

For the pharmaceutical and biological activity of benzimidazole compounds, see: Pujar *et al.* (1988); Bouwman *et al.* (1990). For plant-protective agents in the field of pest control, see: Madkour *et al.* (2006). For related structures, see: Akkurt *et al.* (2011); Jian *et al.* (2003); Jasinski *et al.* (2010, 2011); Odabaşoğlu *et al.* (2007). For standard bond lengths, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{16}H_{14}N_2O_3\cdot4H_2O$
M_r = 354.36
Triclinic, $P\overline{1}$
a = 6.8953 (6) Å
b = 11.4266 (13) Å
c = 11.7287 (14) Å
α = 107.965 (10)°
β = 90.906 (8)°
γ = 91.769 (8)°
V = 878.32 (16) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 173 K
0.35 × 0.33 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur Eos Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2010)'
T_{min} = 0.964, T_{max} = 0.979
8245 measured reflections
4539 independent reflections
3499 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.152
S = 1.02
4539 reflections
273 parameters
17 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4*WB*···O5	0.84 (2)	1.98 (2)	2.809 (2)	170 (2)
O4—H4*WA*···O5^{i}	0.87 (2)	2.04 (2)	2.880 (2)	162 (2)
O5—H5*WB*···N2	0.85 (1)	1.96 (2)	2.8003 (16)	171 (2)
O5—H5*WA*···O7^{ii}	0.85 (2)	1.94 (2)	2.7882 (19)	171 (2)
O6—H6*WA*···O4^{iii}	0.85 (2)	2.16 (2)	2.996 (2)	171 (3)
O6—H6*WB*···O2	0.85 (2)	2.40 (2)	3.187 (3)	154 (3)
O7—H7*WB*···O4^{iv}	0.84 (2)	2.01 (2)	2.844 (2)	174 (2)
O7—H7*WA*···O3*A*	0.81 (2)	1.98 (2)	2.721 (3)	151 (2)
N1—H1*N*···O6	0.83 (1)	2.02 (1)	2.8152 (19)	160 (2)

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2010); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

BN thanks Mangalore University for the research facilities and the UGC SAP for financial assistance for the purchase of chemicals. HSY thanks the UOM for the facilities. JPJ acknowledges the NSF–MRI program (grant No. CHE1039027) for funds to purchase the X-ray diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5279).

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supporting information

Acta Cryst. (2011). E**67**, o2021–o2022 [doi:10.1107/S1600536811027164]

4-(1*H*-Benzimidazol-2-ylmethoxy)-3-methoxybenzaldehyde tetrahydrate

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S1. Comment

The benzimidazole ring system and its related compounds play an important role in pharmaceutical and agricultural fields due to their broad spectrum of biological activities (Pujar *et al.*, 1988, Bouwman *et al.*, 1990). The synthesis of novel benzimidazole derivatives remains a main focus of medicinal research. Benzimidazoles are also useful as insecticides, acaricides, nematocides, herbicides and other plant-protective agents in the field of pest control (Madkour *et al.*, 2006). In addition, benzimidazole derivatives have played a crucial role in the theoretical development of heterocyclic chemistry and are also used extensively in organic synthesis. The crystal structures of some benzimidazole derivatives viz., 2-chloromethyl-1H-benzimidazole nitrate (Jian *et al.*, 2003) and 5-methoxy-1H-benzo[d]imidazole-2(3H)-thione (Odabaşoğlu *et al.*, 2007) have been reported. In continuation of our work on the synthesis of benzimidazole containing aldehydes and their chalcones (Jasinski *et al.*, 2010, 2011; Akkurt *et al.*, 2011) and in view of the importance of benzimidazoles, the title compound, (I), was synthesized and its crystal structure is reported herein.

The molecular structure of the title compound is shown in Fig. 1. In (I) the dihedral angle between the mean planes of the benzimidazole ring system and benzene ring is 2.9 (1)°. The aldehyde group is disordered over two sets of sites corresponding to a rotation of approximately 180° about the C12-C16 bond with refined occupancies of 0.441 (4) and 0.559 (4). Bond distances are in normal ranges (Allen *et al.*, 1987). Extensive O—H···O, O—H···N and N—H···O hydrogen bonds (Table 1) in concert with weak π–π stacking interactions (Table 2) create a 3-D network (Fig. 2).

S2. Experimental

Vanillin (1.52g, 0.01 mole) was dissolved in 30 mL of ethanolic KOH (0.56g, 0.01 mole) and the solution was stirred for 1 h. 2-chloromethyl-1H-benzimidazole (1.66g, 0.01 mole) was added with continuous stirring and refluxed for 5 h (Fig. 3). The reaction mixture was cooled to room temperature and poured into crushed ice. The solid products that separated out were filtered off and recrystallized in ethanol. Single crystals were grown from ethanol by the slow evaporation method which yielded the tetrahydrate of the product (m.p.: 381-382 K) with an yield of 46%.

S3. Refinement

The N–H atom was located in a difference Fourier map and refined isotropically with DFIX = 0.86Å. The O–H atoms were also located in differnce Fourier maps and refined isotropically with DFIX = 0.84Å and DANG = 1.35Å. DFIX and DANG commands are in the SHELXL (Sheldrick, 2008) software. The C and O atoms on the aldehyde group were refined as disordered over two sets of sites for C16/C16A and O3/O3A [occupancy ratio 0.441 (4):0.559 (4)]. All of the remaining H atoms were placed in calculated positions and refined using a riding-model approximation with C—H lengths of 0.95Å (CH), 0.99Å (CH_2) or 0.98Å (CH_3). The isotropic displacement parameters for these atoms were set to 1.19-1.21 (CH, CH_2) or 1.50 (CH_3) times U_{eq} of the parent atom.

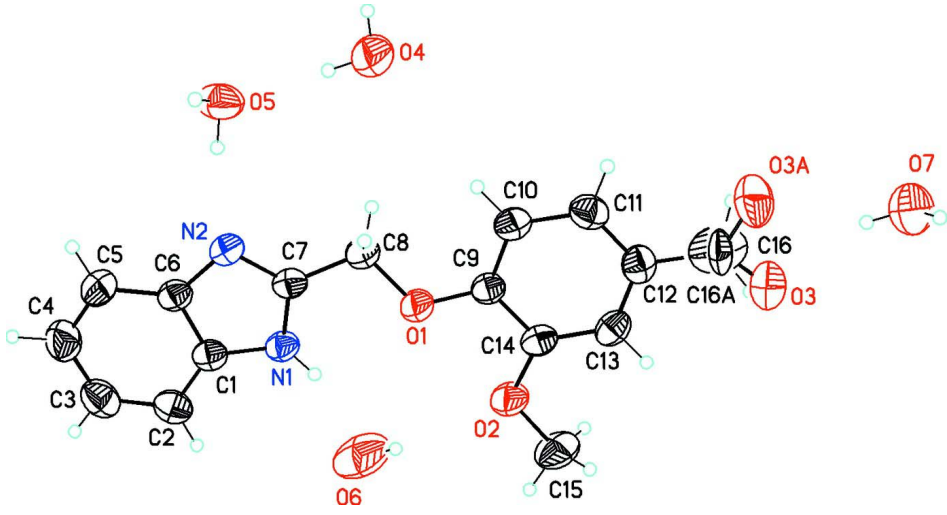

Figure 1

The molecular structure of the title compound showing 50% probability displacement ellipsoids.

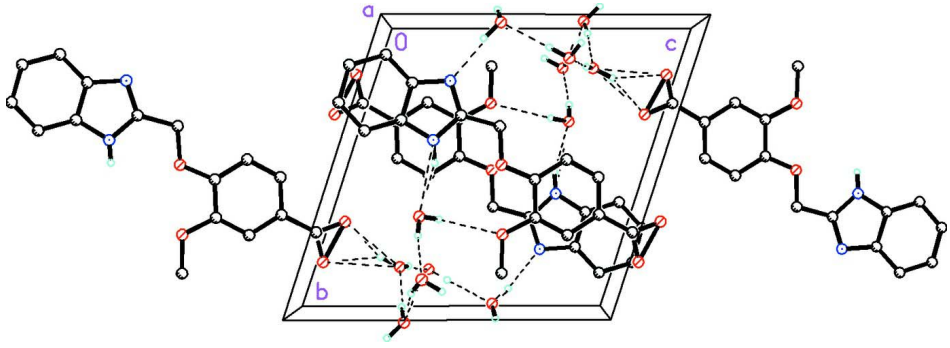

Figure 2

Packing diagram of the title compound viewed along the *a* axis. Dashed lines represent O—H···O, O—H···N and N—H···O hydrogen bonds.

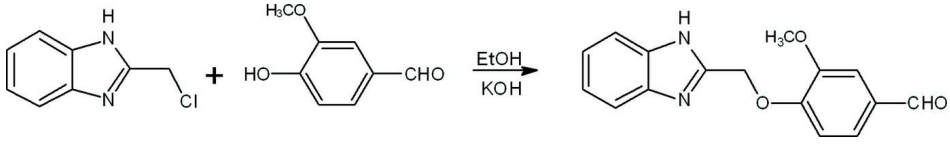

Figure 3

Reaction scheme of the title compound, (I).

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Crystal data

$C_{16}H_{14}N_2O_3·4H_2O$
$M_r = 354.36$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.8953$ (6) Å
$b = 11.4266$ (13) Å
$c = 11.7287$ (14) Å
$\alpha = 107.965$ (10)°
$\beta = 90.906$ (8)°
$\gamma = 91.769$ (8)°
$V = 878.32$ (16) Å^3
$Z = 2$
$F(000) = 376$
$D_x = 1.340$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4026 reflections

θ = 3.4–32.3°
μ = 0.11 mm^{-1}
T = 173 K

Block, pale yellow
0.35 × 0.33 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur Eos Gemini diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.1500 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2010)′
T_{min} = 0.964, T_{max} = 0.979

8245 measured reflections
4539 independent reflections
3499 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 28.7°, θ_{min} = 3.4°
h = −9→6
k = −15→12
l = −14→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.152
S = 1.02
4539 reflections
273 parameters
17 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
$w = 1/[\sigma^2(F_o^2) + (0.0835P)^2 + 0.139P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.25247 (16)	0.50917 (9)	0.48675 (9)	0.0433 (3)	
O2	0.27855 (17)	0.27445 (9)	0.41320 (10)	0.0504 (3)	
O3	0.3970 (4)	0.1784 (3)	−0.0482 (2)	0.0613 (10)	0.441 (4)
O3A	0.3989 (4)	0.3155 (2)	−0.0763 (2)	0.0688 (9)	0.559 (4)
O4	0.1293 (2)	0.86262 (11)	0.38472 (13)	0.0662 (4)	
H4WB	0.161 (3)	0.8992 (19)	0.4568 (14)	0.079*	
H4WA	0.045 (3)	0.9090 (19)	0.3647 (19)	0.079*	
O5	0.18563 (19)	0.98294 (11)	0.63137 (12)	0.0599 (3)	
H5WB	0.192 (3)	0.9297 (16)	0.6684 (18)	0.072*	
H5WA	0.286 (3)	1.0304 (17)	0.6559 (18)	0.072*	
O6	0.1819 (3)	0.33687 (14)	0.6899 (2)	0.0961 (6)	
H6WA	0.101 (3)	0.276 (2)	0.674 (2)	0.115*	

H6WB	0.246 (4)	0.326 (3)	0.626 (2)	0.115*	
O7	0.4891 (2)	0.15864 (14)	−0.29388 (13)	0.0701 (4)	
H7WB	0.603 (3)	0.158 (2)	−0.3182 (19)	0.084*	
H7WA	0.493 (3)	0.192 (2)	−0.2216 (14)	0.084*	
N1	0.18875 (16)	0.59173 (10)	0.72323 (11)	0.0364 (3)	
H1N	0.192 (2)	0.5154 (12)	0.6960 (14)	0.044*	
N2	0.19911 (17)	0.78893 (10)	0.72974 (10)	0.0386 (3)	
C1	0.16508 (18)	0.65805 (12)	0.84166 (12)	0.0363 (3)	
C2	0.1383 (2)	0.62248 (15)	0.94340 (14)	0.0462 (3)	
H2A	0.1349	0.5383	0.9401	0.055*	
C3	0.1168 (2)	0.71524 (17)	1.04909 (15)	0.0549 (4)	
H3A	0.0961	0.6948	1.1207	0.066*	
C4	0.1248 (3)	0.83881 (17)	1.05371 (15)	0.0588 (4)	
H4A	0.1095	0.9004	1.1285	0.071*	
C5	0.1541 (2)	0.87414 (14)	0.95290 (14)	0.0508 (4)	
H5A	0.1614	0.9586	0.9573	0.061*	
C6	0.17285 (18)	0.78171 (12)	0.84452 (12)	0.0375 (3)	
C7	0.20690 (18)	0.67405 (11)	0.66209 (12)	0.0348 (3)	
C8	0.2316 (2)	0.63843 (12)	0.53048 (12)	0.0407 (3)	
H8A	0.1171	0.6617	0.4915	0.049*	
H8B	0.3480	0.6816	0.5122	0.049*	
C9	0.28517 (18)	0.46020 (12)	0.36761 (12)	0.0352 (3)	
C10	0.3059 (2)	0.52749 (13)	0.28790 (13)	0.0413 (3)	
H10A	0.2975	0.6145	0.3152	0.050*	
C11	0.3390 (2)	0.46721 (14)	0.16864 (13)	0.0443 (3)	
H11A	0.3551	0.5132	0.1141	0.053*	
C12	0.34859 (19)	0.34084 (14)	0.12823 (13)	0.0418 (3)	
C13	0.32778 (19)	0.27242 (13)	0.20786 (13)	0.0409 (3)	
H13A	0.3346	0.1853	0.1796	0.049*	
C14	0.29751 (18)	0.33106 (12)	0.32700 (13)	0.0375 (3)	
C15	0.2914 (3)	0.14435 (14)	0.37694 (19)	0.0631 (5)	
H15A	0.2866	0.1158	0.4476	0.095*	
H15B	0.4139	0.1212	0.3363	0.095*	
H15C	0.1825	0.1063	0.3219	0.095*	
C16	0.3765 (19)	0.2805 (10)	0.0036 (9)	0.078 (5)	0.441 (4)
H16A	0.3785	0.3337	−0.0447	0.093*	0.441 (4)
C16A	0.3838 (12)	0.2765 (6)	0.0018 (4)	0.046 (2)	0.559 (4)
H16B	0.3959	0.1900	−0.0179	0.055*	0.559 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0595 (6)	0.0317 (5)	0.0400 (5)	0.0043 (4)	0.0048 (4)	0.0129 (4)
O2	0.0665 (7)	0.0350 (5)	0.0546 (6)	0.0035 (5)	0.0063 (5)	0.0206 (4)
O3	0.0618 (18)	0.064 (2)	0.0498 (16)	0.0027 (14)	0.0093 (12)	0.0047 (14)
O3A	0.0735 (16)	0.0759 (18)	0.0524 (14)	−0.0041 (12)	0.0180 (11)	0.0130 (12)
O4	0.0898 (10)	0.0485 (7)	0.0610 (8)	0.0035 (6)	0.0078 (7)	0.0177 (6)
O5	0.0660 (8)	0.0529 (7)	0.0692 (8)	−0.0063 (6)	−0.0137 (6)	0.0329 (6)

O6	0.0934 (13)	0.0469 (8)	0.1487 (18)	−0.0067 (8)	−0.0135 (11)	0.0330 (10)
O7	0.0674 (8)	0.0725 (9)	0.0644 (8)	−0.0059 (7)	0.0001 (7)	0.0131 (7)
N1	0.0372 (6)	0.0298 (5)	0.0432 (6)	−0.0010 (4)	−0.0011 (4)	0.0131 (5)
N2	0.0406 (6)	0.0322 (5)	0.0437 (6)	0.0043 (4)	0.0011 (5)	0.0126 (5)
C1	0.0276 (6)	0.0387 (6)	0.0441 (7)	0.0016 (5)	−0.0005 (5)	0.0149 (5)
C2	0.0415 (7)	0.0535 (8)	0.0491 (8)	−0.0006 (6)	−0.0005 (6)	0.0243 (7)
C3	0.0500 (9)	0.0739 (11)	0.0443 (8)	0.0067 (8)	0.0037 (7)	0.0229 (8)
C4	0.0606 (10)	0.0666 (11)	0.0439 (8)	0.0161 (8)	0.0049 (7)	0.0078 (7)
C5	0.0540 (9)	0.0436 (8)	0.0512 (9)	0.0139 (7)	0.0024 (7)	0.0083 (7)
C6	0.0310 (6)	0.0382 (7)	0.0439 (7)	0.0058 (5)	0.0007 (5)	0.0133 (5)
C7	0.0308 (6)	0.0327 (6)	0.0430 (7)	0.0003 (5)	−0.0015 (5)	0.0149 (5)
C8	0.0506 (8)	0.0315 (6)	0.0411 (7)	0.0011 (5)	0.0005 (6)	0.0128 (5)
C9	0.0326 (6)	0.0345 (6)	0.0394 (7)	0.0016 (5)	0.0006 (5)	0.0127 (5)
C10	0.0445 (7)	0.0350 (6)	0.0470 (7)	0.0024 (5)	0.0018 (6)	0.0162 (6)
C11	0.0419 (7)	0.0502 (8)	0.0461 (8)	0.0026 (6)	0.0031 (6)	0.0225 (6)
C12	0.0310 (6)	0.0509 (8)	0.0411 (7)	0.0033 (5)	0.0037 (5)	0.0104 (6)
C13	0.0324 (6)	0.0355 (6)	0.0516 (8)	0.0024 (5)	0.0024 (6)	0.0087 (6)
C14	0.0316 (6)	0.0342 (6)	0.0490 (8)	0.0015 (5)	0.0009 (5)	0.0162 (6)
C15	0.0776 (12)	0.0351 (8)	0.0826 (13)	0.0022 (8)	0.0069 (10)	0.0266 (8)
C16	0.039 (6)	0.085 (9)	0.116 (9)	−0.005 (5)	−0.002 (5)	0.042 (7)
C16A	0.042 (4)	0.060 (4)	0.0252 (19)	0.007 (3)	0.011 (2)	−0.004 (2)

Geometric parameters (Å, º)

O1—C9	1.3609 (17)	C3—H3A	0.9500
O1—C8	1.4190 (16)	C4—C5	1.378 (2)
O2—C14	1.3638 (17)	C4—H4A	0.9500
O2—C15	1.4206 (18)	C5—C6	1.390 (2)
O3—C16	1.151 (8)	C5—H5A	0.9500
O3A—C16A	1.140 (7)	C7—C8	1.4840 (19)
O4—H4WB	0.841 (15)	C8—H8A	0.9900
O4—H4WA	0.873 (15)	C8—H8B	0.9900
O5—H5WB	0.851 (14)	C9—C10	1.3880 (19)
O5—H5WA	0.853 (15)	C9—C14	1.4097 (18)
O6—H6WA	0.845 (16)	C10—C11	1.381 (2)
O6—H6WB	0.850 (16)	C10—H10A	0.9500
O7—H7WB	0.838 (15)	C11—C12	1.378 (2)
O7—H7WA	0.814 (15)	C11—H11A	0.9500
N1—C7	1.3514 (16)	C12—C13	1.397 (2)
N1—C1	1.3768 (18)	C12—C16	1.430 (9)
N1—H1N	0.832 (13)	C12—C16A	1.466 (4)
N2—C7	1.3110 (17)	C13—C14	1.373 (2)
N2—C6	1.3881 (18)	C13—H13A	0.9500
C1—C2	1.387 (2)	C15—H15A	0.9800
C1—C6	1.4023 (18)	C15—H15B	0.9800
C2—C3	1.374 (2)	C15—H15C	0.9800
C2—H2A	0.9500	C16—H16A	0.9500
C3—C4	1.396 (3)	C16A—H16B	0.9500

C9—O1—C8	116.72 (10)	C7—C8—H8B	110.0
C14—O2—C15	117.42 (13)	H8A—C8—H8B	108.4
H4WB—O4—H4WA	105.6 (17)	O1—C9—C10	124.95 (12)
H5WB—O5—H5WA	104.9 (17)	O1—C9—C14	114.89 (11)
H6WA—O6—H6WB	105 (2)	C10—C9—C14	120.15 (13)
H7WB—O7—H7WA	107.6 (19)	C11—C10—C9	119.63 (13)
C7—N1—C1	106.93 (11)	C11—C10—H10A	120.2
C7—N1—H1N	127.5 (11)	C9—C10—H10A	120.2
C1—N1—H1N	125.5 (11)	C12—C11—C10	120.42 (13)
C7—N2—C6	104.49 (11)	C12—C11—H11A	119.8
N1—C1—C2	132.25 (13)	C10—C11—H11A	119.8
N1—C1—C6	105.01 (11)	C11—C12—C13	120.31 (13)
C2—C1—C6	122.74 (13)	C11—C12—C16	119.2 (5)
C3—C2—C1	116.54 (14)	C13—C12—C16	120.4 (5)
C3—C2—H2A	121.7	C11—C12—C16A	120.6 (3)
C1—C2—H2A	121.7	C13—C12—C16A	119.1 (3)
C2—C3—C4	121.51 (15)	C14—C13—C12	120.00 (13)
C2—C3—H3A	119.2	C14—C13—H13A	120.0
C4—C3—H3A	119.2	C12—C13—H13A	120.0
C5—C4—C3	121.91 (16)	O2—C14—C13	125.26 (12)
C5—C4—H4A	119.0	O2—C14—C9	115.26 (12)
C3—C4—H4A	119.0	C13—C14—C9	119.48 (12)
C4—C5—C6	117.59 (15)	O2—C15—H15A	109.5
C4—C5—H5A	121.2	O2—C15—H15B	109.5
C6—C5—H5A	121.2	H15A—C15—H15B	109.5
N2—C6—C5	130.48 (13)	O2—C15—H15C	109.5
N2—C6—C1	109.82 (12)	H15A—C15—H15C	109.5
C5—C6—C1	119.70 (13)	H15B—C15—H15C	109.5
N2—C7—N1	113.74 (12)	O3—C16—C12	131.0 (10)
N2—C7—C8	122.82 (11)	O3—C16—H16A	114.5
N1—C7—C8	123.43 (11)	C12—C16—H16A	114.5
O1—C8—C7	108.39 (10)	O3A—C16A—C12	129.3 (5)
O1—C8—H8A	110.0	O3A—C16A—H16B	115.4
C7—C8—H8A	110.0	C12—C16A—H16B	115.4
O1—C8—H8B	110.0		

C7—N1—C1—C2	179.33 (14)	O1—C9—C10—C11	179.95 (13)
C7—N1—C1—C6	−0.62 (14)	C14—C9—C10—C11	−0.1 (2)
N1—C1—C2—C3	−179.16 (14)	C9—C10—C11—C12	0.9 (2)
C6—C1—C2—C3	0.8 (2)	C10—C11—C12—C13	−0.9 (2)
C1—C2—C3—C4	−1.0 (2)	C10—C11—C12—C16	177.9 (6)
C2—C3—C4—C5	0.1 (3)	C10—C11—C12—C16A	−179.9 (4)
C3—C4—C5—C6	1.0 (3)	C11—C12—C13—C14	0.0 (2)
C7—N2—C6—C5	−179.88 (14)	C16—C12—C13—C14	−178.8 (6)
C7—N2—C6—C1	−0.13 (14)	C16A—C12—C13—C14	179.0 (4)
C4—C5—C6—N2	178.55 (14)	C15—O2—C14—C13	0.1 (2)
C4—C5—C6—C1	−1.2 (2)	C15—O2—C14—C9	−179.54 (13)

N1—C1—C6—N2	0.47 (14)	C12—C13—C14—O2	−178.79 (12)
C2—C1—C6—N2	−179.48 (12)	C12—C13—C14—C9	0.8 (2)
N1—C1—C6—C5	−179.75 (12)	O1—C9—C14—O2	−1.17 (17)
C2—C1—C6—C5	0.3 (2)	C10—C9—C14—O2	178.85 (12)
C6—N2—C7—N1	−0.28 (15)	O1—C9—C14—C13	179.18 (11)
C6—N2—C7—C8	179.19 (12)	C10—C9—C14—C13	−0.81 (19)
C1—N1—C7—N2	0.59 (15)	C11—C12—C16—O3	176.2 (10)
C1—N1—C7—C8	−178.88 (12)	C13—C12—C16—O3	−4.9 (16)
C9—O1—C8—C7	−177.26 (11)	C16A—C12—C16—O3	50 (19)
N2—C7—C8—O1	174.58 (12)	C11—C12—C16A—O3A	−3.5 (10)
N1—C7—C8—O1	−6.00 (18)	C13—C12—C16A—O3A	177.5 (6)
C8—O1—C9—C10	2.33 (19)	C16—C12—C16A—O3A	51 (20)
C8—O1—C9—C14	−177.65 (11)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4*WB*···O5	0.84 (2)	1.98 (2)	2.809 (2)	170 (2)
O4—H4*WA*···O5^{i}	0.87 (2)	2.04 (2)	2.880 (2)	162 (2)
O5—H5*WB*···N2	0.85 (1)	1.96 (2)	2.8003 (16)	171 (2)
O5—H5*WA*···O7^{ii}	0.85 (2)	1.94 (2)	2.7882 (19)	171 (2)
O6—H6*WA*···O4^{iii}	0.85 (2)	2.16 (2)	2.996 (2)	171 (3)
O6—H6*WB*···O2	0.85 (2)	2.40 (2)	3.187 (3)	154 (3)
O7—H7*WB*···O4^{iv}	0.84 (2)	2.01 (2)	2.844 (2)	174 (2)
O7—H7*WA*···O3*A*	0.81 (2)	1.98 (2)	2.721 (3)	151 (2)
N1—H1*N*···O6	0.83 (1)	2.02 (1)	2.8152 (19)	160 (2)

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