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Crystal structure of 2-hydroxy-*N*-(2-hydroxyethyl)-*N*-{2-hydroxy-3-[(*E*)-*N*-hydroxyethanimidoyl]-5-methylbenzyl}-ethanaminium acetate monohydrate

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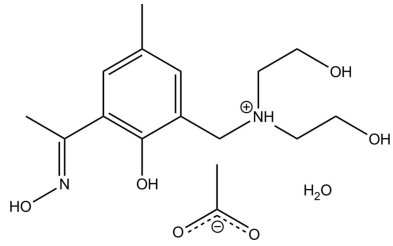
The structure of the title hydrated molecular salt, $C_{14}H_{23}N_2O_4^+\cdot C_2H_3O_2^-\cdot H_2O$, was determined as part of a wider study on the use of the molecule as a polydentate ligand in the synthesis of Mn^{III} clusters with magnetic properties. The cation features intramolecular O—H···N and N—H···O hydrogen-bond interactions. The crystal structure features a range of intermolecular hydrogen-bonding interactions, principally O—H···O interactions between all three species in the asymmetric unit. An $R_4^2(8)$ graph-set hydrogen-bonding motif between the anion and water molecules serves as a unit which links to the cation *via* the diethanolamine group. Each O atom of the acetate anion accepts two hydrogen bonds.

Keywords: crystal structure; hydrogen bonding; hydrate; organic salt; magnetism.

CCDC reference: 1047385

1. Related literature

For background literature on Mn-containing single molecule magnets, see: Inglis *et al.* (2012); Milios *et al.* (2007); Tasiopoulos & Perlepes (2008). For examples of the use of 3-{[bis(2-hydroxyethyl)amino]methyl}-2-hydroxy-5-methylbenzaldehyde in the synthesis of magnetic Mn cluster compounds, see: Sanz *et al.* (2014*a*,*b*) – molecular wheels; Frost *et al.* (2014) – tetrahedron cage. For examples of other magnentic oxime-containing clusters, see: Vlahopoulou *et al.* (2009); Stamatatos *et al.* (2007). For a review of pyridyl–oxime coordination chemistry, see: Milios *et al.* (2006). For the synthesis of 3-{[bis(2-hydroxyethyl)amino]methyl}-2-hydroxy-5-methylbenzaldehyde, see: Wang *et al.* (2006).



2. Experimental

2.1. Crystal data

$C_{14}H_{23}N_2O_4^+\cdot C_2H_3O_2^-\cdot H_2O$
M_r = 360.40
Monoclinic, $P2_1/c$
a = 14.4338 (5) Å
b = 10.4786 (3) Å
c = 12.4045 (4) Å
β = 101.593 (3)°
V = 1837.86 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 120 K
0.48 × 0.38 × 0.18 mm

2.2. Data collection

Agilent SuperNova diffractometer
Absorption correction: gaussian (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.942, T_{max} = 0.975
38067 measured reflections
5542 independent reflections
4362 reflections with $I > 2\sigma(I)$
R_{int} = 0.054

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.129
S = 1.09
5542 reflections
338 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.85 (2)	1.78 (2)	2.5368 (16)	148 (2)
O2—H2···O5	0.91 (2)	1.71 (2)	2.5985 (16)	165 (2)
O3—H3···O5^{i}	0.82 (3)	1.82 (3)	2.6335 (17)	171 (3)
O4—H4···O7	0.85 (2)	1.84 (2)	2.6875 (19)	176 (2)
N2—H2*A*···O1	0.903 (19)	2.168 (18)	2.8121 (16)	127.6 (15)
O7—H7*A*···O6	0.79 (3)	2.07 (3)	2.823 (2)	159 (3)
O7—H7*B*···O6^{ii}	0.89 (3)	1.86 (3)	2.738 (2)	169 (2)

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

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7350).

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Crystal structure of 2-hydroxy-*N*-(2-hydroxyethyl)-*N*-{2-hydroxy-3-[(*E*)-*N*-hydroxyethanimidoyl]-5-methylbenzyl}ethanaminium acetate monohydrate

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S1. Chemical context

The structure of the title hydrated salt was determined as part of a wider study on the synthesis of polymetallic compounds with potentially interesting magnetic properties. The phenolic oximes are a ligand family which have had enormous success in the construction of Mn cluster compounds that behave as single molecule magnets (Milios *et al.*, 2007; Inglis *et al.*, 2012). These ligand types tend to form systems based on the $[Mn_3O(L)_3]^+$ (L = salicylaldoxime) building block (Vlahopoulou *et al.*, 2009; Stamatatos *et al.*, 2007; Milios *et al.*, 2006). An additional functional group was introduced onto the aromatic framework of the ligand in an attempt to disrupt the formation of clusters based on this motif and to see if higher nuclearity compounds based on phenolic oximes could be isolated. A diethanolamine functional group was the obvious choice given that the this has an excellent track record of making magnetically interesting Mn clusters in its own right (Tasiopoulos & Perlepes, 2008). For examples of the use of the H_4L in the synthesis of magnetic materials, see Frost *et al.* (2014) and Sanz *et al.* (2014a , 2014b).

S2. Structural commentary

A check of the molecular geometry with Mogul showed all geometric parameters to be unexceptional. A mean plane fitted through atoms O1, O2, N1 and C1 to C10 (i.e. all ring atoms plus the oxime and hydroxyl groups) has an rms deviation of 0.029 Å.

S3. Supramolecular features

The crystal structure features extensive hydrogen bonding, principally O–H···O interactions involving all species in the asymmetric unit. Intramolecular interactions within the cation are, perhaps, less important but serve to support the overall structure by locking the cation conformation. The N2–H2A···O1 interaction in particular is probably quite weak. The $R^2_4(8)$ graph set motif between the anion and water molecules serves as an important unit which links to the cation via the hydroxyethane groups to propagate the three-dimensional structure. Hydrogen bonding information is summarised in Table 2.

S4. Synthesis and crystallization

Experimental Procedures

^{1}H and ^{13}CNMR spectra were recorded on a nav 500 MHz spectrometer. 3-((Bis(2-hydroxyethyl)amino)methyl)-2-hydroxy-5-methylbenzaldehyde was prepared according to a published procedure (Wang *et al.*, 2006). Solvents and reagents were used as received from commercial suppliers.

Synthesis of 3-{[Bis(2-hydroxyethyl)amino]methyl}-2-hydroxy-5-methylsalicylaldoxime (H_4L)

3-{[Bis(2-hydroxyethyl)amino]methyl}-2-hydroxy-5-methylbenzaldehyde (10.8 g, 40 mmol), hydroxylamine hydrochloride (3.5 g, 50 mmol) and sodium acetate (4.14 g, 50 mmol) were dissolved in 500 mL of ethanol. The mixture was refluxed under N_2 for 4 h. A white precipitate was filtered off from the warm ethanol solution. The solvent was evaporated to dryness, a minimum amount of CH_2Cl_2 added and the sample stored at -10°C for 24 hours. Clear block shaped crystals grew and were collected after filtration (10.16 g, 90%). ^{1}H NMR (500 MHz, DMSO): δ 7.12 (bs, 1H), 7.05 (bs, 1H), 3.60 (s, 2H), 3.54 (t, J=6.2 Hz, 4H), 2.53 (t, J= 6.2 Hz, 4H), 2.23 (s, 3H), 2.22 (s, 3H).^{13}C NMR (500 MHz, DMSO): δ 157.28 (1C, C_{ar}OH), 153.86 (1C, *C*NOH), 131.34 (1C, *C*H), 127.61 (1C, *C*H), 126.99 (1C, *C*), 124.34 (1C, *C*), 121.01 (1C, *C*), 59.14 (2C, *C*H_2), 56.51 (2C, *C*H_2), 54.78 (1C, *C*H_2), 21.69 (1C, *C*H_3), 12.73 (1C, *C*H_3).

S5. Refinement

Crystal data, data collection and structure refinement details are summarised in Table 1. All H atoms were located in a difference Fourier map and refined freely.

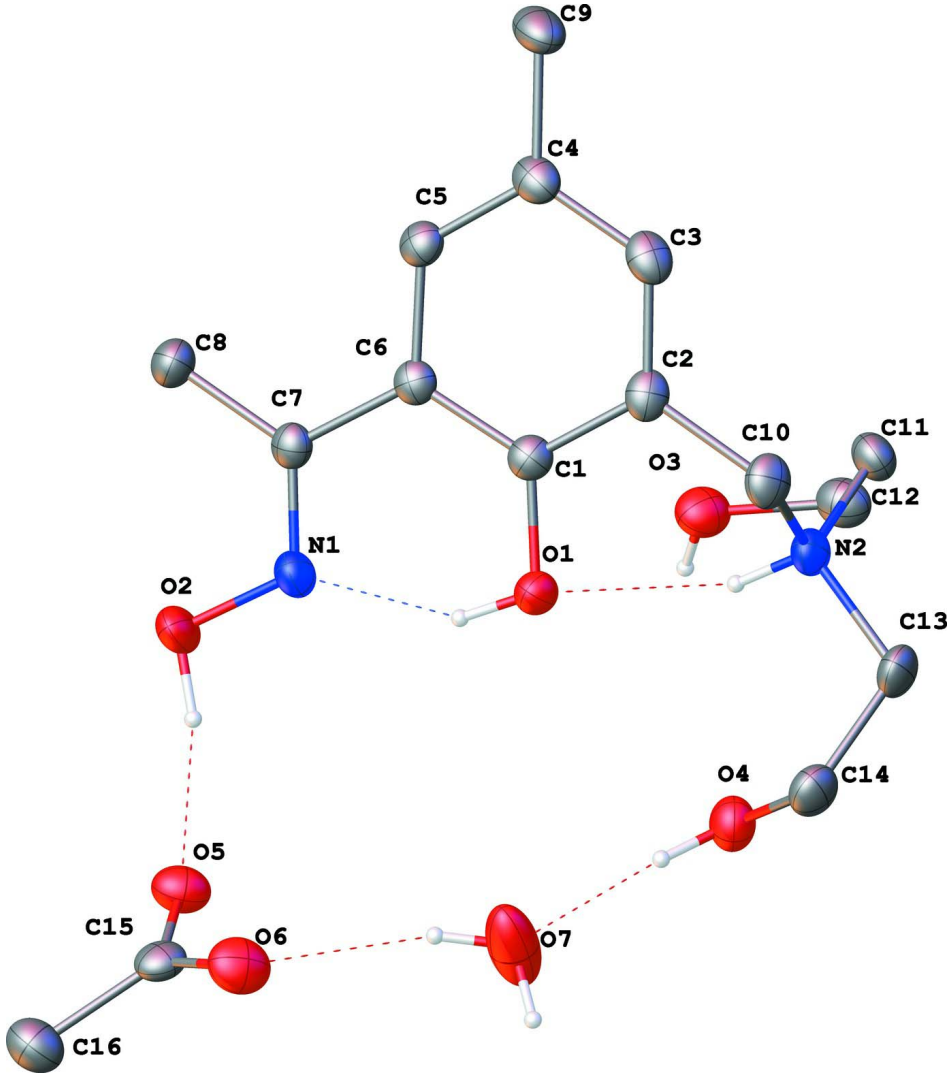

Figure 1

The asymmetric unit of H_4L. Displacement ellipsoids are at the 50% probability level and C-bound H atoms have been omitted.

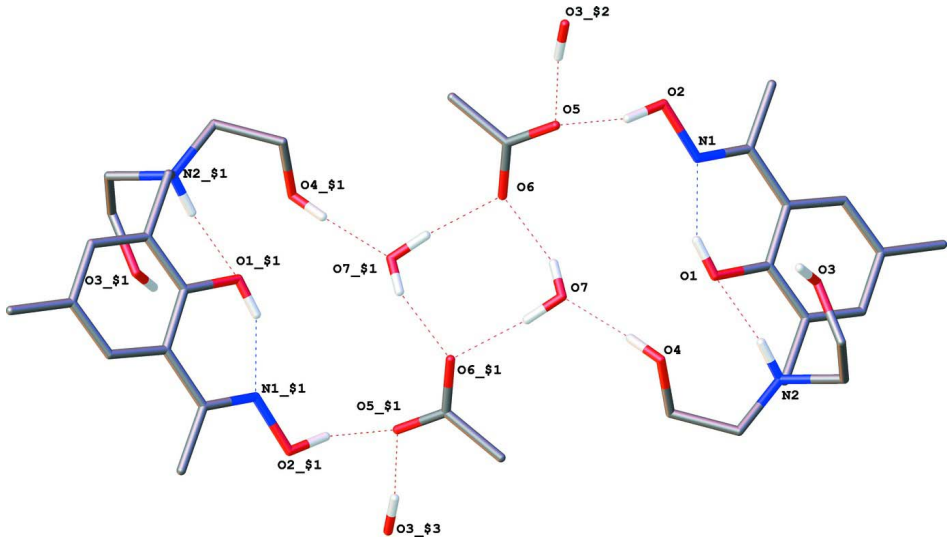

Figure 2

Hydrogen-bonding interactions, indicated by dashed lines, in the crystal structure of H_4L. Symmetry operations for equivalent atoms: \$1, 1 - *x*, -*y*, 2 - *z*; \$2, *x*, 1/2 - *y*, 1/2 + *z*; \$3, 1 - *x*, -1/2 + *y*, 3/2 - *z*.

2-Hydroxy-*N*-(2-hydroxyethyl)-*N*-{2-hydroxy-3-[(*E*)-*N*-hydroxyethanimidoyl]-5-methylbenzyl}ethanaminium acetate monohydrate

Crystal data

$C_{14}H_{23}N_2O_4^+ \cdot C_2H_3O_2^- \cdot H_2O$
$M_r = 360.40$
Monoclinic, $P2_1/c$
$a = 14.4338$ (5) Å
$b = 10.4786$ (3) Å
$c = 12.4045$ (4) Å
$\beta = 101.593$ (3)°
$V = 1837.86$ (10) Å^3
$Z = 4$
$F(000) = 776$
$D_x = 1.303$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 10187 reflections
$\theta = 3.5$–30.2°
$\mu = 0.10$ mm^{-1}
$T = 120$ K
Block, colourless
0.48 × 0.38 × 0.18 mm

Data collection

Agilent SuperNova diffractometer
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 5.1574 pixels mm^{-1}
ω scans
Absorption correction: gaussian (*CrysAlis PRO*; Agilent, 2014)
$T_{min} = 0.942$, $T_{max} = 0.975$
38067 measured reflections
5542 independent reflections
4362 reflections with $I > 2\sigma(I)$
$R_{int} = 0.054$
$\theta_{max} = 31.1°$, $\theta_{min} = 3.1°$
$h = -20 \rightarrow 20$
$k = -15 \rightarrow 13$
$l = -18 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.129$
$S = 1.09$
5542 reflections
338 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: difference Fourier map
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0431P)^2 + 0.9291P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ 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. All H atoms were located in a difference Fourier map and refined freely.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.25286 (7)	−0.10957 (10)	0.65849 (9)	0.0229 (2)
H1	0.2395 (15)	−0.050 (2)	0.6990 (18)	0.046 (6)*
O2	0.14106 (9)	0.14279 (11)	0.81376 (9)	0.0320 (3)
H2	0.2011 (17)	0.160 (2)	0.8484 (19)	0.054 (7)*
O3	0.24215 (8)	0.03539 (12)	0.40801 (10)	0.0304 (3)
H3	0.2659 (18)	0.107 (3)	0.418 (2)	0.060 (7)*
O4	0.45674 (8)	−0.04092 (11)	0.61916 (10)	0.0300 (3)
H4	0.4552 (16)	−0.026 (2)	0.686 (2)	0.055 (7)*
N1	0.15152 (9)	0.03976 (12)	0.74689 (10)	0.0232 (3)
N2	0.32127 (9)	−0.19342 (12)	0.47375 (10)	0.0220 (3)
H2A	0.3117 (13)	−0.1223 (18)	0.5112 (15)	0.028 (5)*
C1	0.16920 (10)	−0.16143 (13)	0.60648 (11)	0.0202 (3)
C2	0.17518 (10)	−0.26194 (13)	0.53438 (12)	0.0223 (3)
C3	0.09294 (11)	−0.32024 (14)	0.47871 (12)	0.0246 (3)
H3A	0.0993 (13)	−0.3890 (18)	0.4276 (15)	0.029 (5)*
C4	0.00394 (11)	−0.27997 (14)	0.49290 (12)	0.0243 (3)
C5	−0.00027 (10)	−0.17826 (14)	0.56389 (11)	0.0213 (3)
H5	−0.0609 (13)	−0.1492 (17)	0.5733 (14)	0.024 (4)*
C6	0.08079 (10)	−0.11679 (13)	0.62184 (11)	0.0192 (3)
C7	0.07274 (10)	−0.00789 (14)	0.69504 (11)	0.0203 (3)
C8	−0.02208 (11)	0.04239 (17)	0.70539 (14)	0.0264 (3)
H8A	−0.0567 (16)	0.073 (2)	0.638 (2)	0.053 (6)*
H8B	−0.0153 (17)	0.104 (2)	0.757 (2)	0.058 (7)*
H8C	−0.0601 (16)	−0.026 (2)	0.7244 (18)	0.052 (6)*
C9	−0.08552 (13)	−0.34031 (18)	0.43012 (14)	0.0323 (4)
H9A	−0.1036 (15)	−0.305 (2)	0.3569 (18)	0.042 (6)*
H9B	−0.1359 (18)	−0.334 (2)	0.474 (2)	0.063 (7)*
H9C	−0.0770 (15)	−0.429 (2)	0.4181 (18)	0.048 (6)*
C10	0.27174 (11)	−0.30209 (14)	0.51927 (14)	0.0261 (3)
H10A	0.2691 (13)	−0.3701 (19)	0.4675 (16)	0.032 (5)*
H10B	0.3144 (13)	−0.3244 (17)	0.5908 (15)	0.027 (5)*
C11	0.27852 (12)	−0.16896 (15)	0.35473 (12)	0.0259 (3)
H11A	0.2117 (13)	−0.1940 (17)	0.3425 (14)	0.024 (4)*
H11B	0.3108 (13)	−0.2206 (19)	0.3119 (15)	0.032 (5)*
C12	0.28636 (12)	−0.02870 (16)	0.33149 (13)	0.0294 (3)

H12A	0.3503 (14)	−0.0019 (18)	0.3386 (15)	0.032 (5)*
H12B	0.2514 (13)	−0.0128 (18)	0.2536 (15)	0.030 (5)*
C13	0.42648 (11)	−0.21255 (17)	0.49313 (14)	0.0288 (3)
H13A	0.4494 (12)	−0.1530 (17)	0.4413 (14)	0.025 (4)*
H13B	0.4385 (12)	−0.3021 (18)	0.4792 (14)	0.027 (4)*
C14	0.47242 (12)	−0.17376 (17)	0.60884 (14)	0.0298 (3)
H14A	0.5399 (14)	−0.1927 (18)	0.6200 (15)	0.034 (5)*
H14B	0.4445 (13)	−0.2225 (18)	0.6635 (15)	0.030 (5)*
O5	0.30047 (8)	0.22604 (11)	0.92796 (9)	0.0297 (3)
O6	0.38120 (9)	0.06607 (11)	1.01687 (10)	0.0349 (3)
C15	0.34399 (10)	0.17389 (14)	1.01535 (13)	0.0242 (3)
C16	0.35169 (15)	0.24691 (19)	1.12192 (15)	0.0347 (4)
H16A	0.409 (2)	0.234 (3)	1.167 (3)	0.098 (11)*
H16B	0.348 (2)	0.337 (3)	1.110 (3)	0.101 (11)*
H16C	0.307 (2)	0.221 (3)	1.162 (3)	0.108 (12)*
O7	0.46060 (14)	0.00383 (17)	0.83335 (12)	0.0569 (5)
H7A	0.4274 (18)	0.028 (3)	0.873 (2)	0.059 (8)*
H7B	0.5160 (19)	−0.016 (3)	0.875 (2)	0.064 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0198 (5)	0.0238 (5)	0.0247 (5)	0.0018 (4)	0.0037 (4)	−0.0027 (4)
O2	0.0276 (6)	0.0354 (6)	0.0320 (6)	0.0025 (5)	0.0035 (5)	−0.0167 (5)
O3	0.0300 (6)	0.0243 (6)	0.0361 (6)	−0.0018 (5)	0.0048 (5)	−0.0022 (5)
O4	0.0306 (6)	0.0328 (6)	0.0282 (6)	0.0025 (5)	0.0099 (5)	−0.0019 (5)
N1	0.0251 (6)	0.0229 (6)	0.0216 (6)	0.0016 (5)	0.0051 (5)	−0.0044 (5)
N2	0.0225 (6)	0.0208 (6)	0.0233 (6)	0.0035 (5)	0.0062 (5)	−0.0030 (5)
C1	0.0223 (7)	0.0177 (6)	0.0208 (6)	0.0002 (5)	0.0046 (5)	0.0031 (5)
C2	0.0268 (7)	0.0167 (6)	0.0251 (7)	0.0020 (5)	0.0096 (6)	0.0036 (5)
C3	0.0343 (8)	0.0166 (6)	0.0247 (7)	−0.0023 (6)	0.0098 (6)	0.0001 (5)
C4	0.0291 (8)	0.0232 (7)	0.0218 (6)	−0.0063 (6)	0.0077 (6)	0.0005 (5)
C5	0.0214 (7)	0.0226 (7)	0.0208 (6)	−0.0016 (5)	0.0070 (5)	0.0025 (5)
C6	0.0223 (7)	0.0180 (6)	0.0178 (6)	−0.0002 (5)	0.0053 (5)	0.0029 (5)
C7	0.0223 (7)	0.0218 (7)	0.0177 (6)	0.0008 (5)	0.0061 (5)	0.0017 (5)
C8	0.0227 (8)	0.0304 (8)	0.0271 (7)	0.0018 (6)	0.0075 (6)	−0.0024 (6)
C9	0.0333 (9)	0.0343 (9)	0.0303 (8)	−0.0124 (7)	0.0085 (7)	−0.0093 (7)
C10	0.0301 (8)	0.0185 (7)	0.0312 (8)	0.0048 (6)	0.0097 (6)	0.0005 (6)
C11	0.0294 (8)	0.0282 (8)	0.0204 (6)	−0.0018 (6)	0.0057 (6)	−0.0049 (6)
C12	0.0313 (9)	0.0310 (8)	0.0251 (7)	−0.0038 (6)	0.0039 (6)	0.0016 (6)
C13	0.0217 (8)	0.0337 (9)	0.0326 (8)	0.0081 (6)	0.0091 (6)	−0.0046 (7)
C14	0.0224 (8)	0.0337 (9)	0.0331 (8)	0.0074 (6)	0.0050 (6)	−0.0006 (7)
O5	0.0302 (6)	0.0252 (6)	0.0310 (6)	−0.0027 (4)	−0.0003 (5)	0.0003 (4)
O6	0.0394 (7)	0.0216 (6)	0.0417 (7)	0.0006 (5)	0.0032 (5)	−0.0002 (5)
C15	0.0192 (7)	0.0219 (7)	0.0311 (7)	−0.0064 (5)	0.0043 (6)	−0.0005 (6)
C16	0.0391 (10)	0.0332 (9)	0.0309 (8)	−0.0034 (7)	0.0044 (7)	−0.0042 (7)
O7	0.0679 (11)	0.0706 (11)	0.0285 (7)	0.0369 (9)	0.0011 (7)	−0.0062 (7)

Geometric parameters (Å, º)

O1—H1	0.85 (2)	C8—H8B	0.91 (3)
O1—C1	1.3625 (17)	C8—H8C	0.96 (2)
O2—H2	0.91 (2)	C9—H9A	0.97 (2)
O2—N1	1.3880 (16)	C9—H9B	0.99 (3)
O3—H3	0.82 (3)	C9—H9C	0.96 (2)
O3—C12	1.415 (2)	C10—H10A	0.95 (2)
O4—H4	0.85 (2)	C10—H10B	1.001 (18)
O4—C14	1.420 (2)	C11—H11A	0.982 (18)
N1—C7	1.2891 (19)	C11—H11B	0.94 (2)
N2—H2A	0.903 (19)	C11—C12	1.506 (2)
N2—C10	1.513 (2)	C12—H12A	0.952 (19)
N2—C11	1.5034 (19)	C12—H12B	1.009 (18)
N2—C13	1.503 (2)	C13—H13A	0.999 (18)
C1—C2	1.396 (2)	C13—H13B	0.976 (19)
C1—C6	1.408 (2)	C13—C14	1.511 (2)
C2—C3	1.389 (2)	C14—H14A	0.98 (2)
C2—C10	1.503 (2)	C14—H14B	0.996 (19)
C3—H3A	0.976 (19)	O5—C15	1.2628 (18)
C3—C4	1.396 (2)	O6—C15	1.2495 (19)
C4—C5	1.392 (2)	C15—C16	1.512 (2)
C4—C9	1.506 (2)	C16—H16A	0.91 (3)
C5—H5	0.955 (18)	C16—H16B	0.96 (4)
C5—C6	1.401 (2)	C16—H16C	0.92 (4)
C6—C7	1.4773 (19)	O7—H7A	0.79 (3)
C7—C8	1.496 (2)	O7—H7B	0.89 (3)
C8—H8A	0.95 (2)		

C1—O1—H1	106.8 (15)	H9A—C9—H9C	104.1 (18)
N1—O2—H2	103.4 (15)	H9B—C9—H9C	106 (2)
C12—O3—H3	107.3 (18)	N2—C10—H10A	105.6 (11)
C14—O4—H4	107.4 (17)	N2—C10—H10B	104.7 (10)
C7—N1—O2	114.11 (12)	C2—C10—N2	110.81 (12)
C10—N2—H2A	107.2 (12)	C2—C10—H10A	112.3 (11)
C11—N2—H2A	106.8 (11)	C2—C10—H10B	112.1 (10)
C11—N2—C10	111.31 (12)	H10A—C10—H10B	110.8 (15)
C13—N2—H2A	106.4 (11)	N2—C11—H11A	107.6 (10)
C13—N2—C10	112.21 (12)	N2—C11—H11B	107.6 (11)
C13—N2—C11	112.46 (12)	N2—C11—C12	108.81 (12)
O1—C1—C2	116.19 (13)	H11A—C11—H11B	109.9 (15)
O1—C1—C6	123.00 (13)	C12—C11—H11A	109.9 (10)
C2—C1—C6	120.80 (13)	C12—C11—H11B	112.7 (12)
C1—C2—C10	118.00 (13)	O3—C12—C11	105.86 (13)
C3—C2—C1	119.59 (14)	O3—C12—H12A	111.0 (11)
C3—C2—C10	122.41 (13)	O3—C12—H12B	111.0 (10)
C2—C3—H3A	117.7 (11)	C11—C12—H12A	112.2 (12)
C2—C3—C4	121.35 (14)	C11—C12—H12B	107.6 (11)

C4—C3—H3A	120.9 (11)	H12A—C12—H12B	109.2 (15)
C3—C4—C9	121.51 (14)	N2—C13—H13A	105.6 (10)
C5—C4—C3	118.00 (14)	N2—C13—H13B	108.1 (10)
C5—C4—C9	120.44 (15)	N2—C13—C14	110.65 (12)
C4—C5—H5	118.6 (11)	H13A—C13—H13B	113.1 (15)
C4—C5—C6	122.59 (14)	C14—C13—H13A	107.7 (10)
C6—C5—H5	118.8 (11)	C14—C13—H13B	111.5 (10)
C1—C6—C7	121.69 (13)	O4—C14—C13	107.57 (13)
C5—C6—C1	117.66 (13)	O4—C14—H14A	111.1 (12)
C5—C6—C7	120.65 (13)	O4—C14—H14B	110.4 (11)
N1—C7—C6	115.82 (13)	C13—C14—H14A	108.3 (11)
N1—C7—C8	123.45 (13)	C13—C14—H14B	110.4 (11)
C6—C7—C8	120.73 (13)	H14A—C14—H14B	109.1 (15)
C7—C8—H8A	112.0 (14)	O5—C15—C16	117.90 (14)
C7—C8—H8B	110.1 (15)	O6—C15—O5	122.85 (15)
C7—C8—H8C	109.7 (14)	O6—C15—C16	119.25 (15)
H8A—C8—H8B	110 (2)	C15—C16—H16A	111 (2)
H8A—C8—H8C	104.4 (19)	C15—C16—H16B	112 (2)
H8B—C8—H8C	110 (2)	C15—C16—H16C	113 (2)
C4—C9—H9A	111.6 (13)	H16A—C16—H16B	105 (3)
C4—C9—H9B	109.7 (14)	H16A—C16—H16C	106 (3)
C4—C9—H9C	111.5 (13)	H16B—C16—H16C	111 (3)
H9A—C9—H9B	113.5 (19)	H7A—O7—H7B	107 (2)

O1—C1—C2—C3	179.65 (12)	C3—C2—C10—N2	118.43 (15)
O1—C1—C2—C10	−0.96 (19)	C3—C4—C5—C6	−0.8 (2)
O1—C1—C6—C5	−179.83 (12)	C4—C5—C6—C1	−0.1 (2)
O1—C1—C6—C7	1.0 (2)	C4—C5—C6—C7	179.07 (13)
O2—N1—C7—C6	179.25 (11)	C5—C6—C7—N1	178.18 (13)
O2—N1—C7—C8	−0.1 (2)	C5—C6—C7—C8	−2.5 (2)
N2—C11—C12—O3	−55.59 (16)	C6—C1—C2—C3	−1.3 (2)
N2—C13—C14—O4	−63.57 (17)	C6—C1—C2—C10	178.13 (13)
C1—C2—C3—C4	0.3 (2)	C9—C4—C5—C6	−178.27 (14)
C1—C2—C10—N2	−60.94 (17)	C10—N2—C11—C12	148.01 (13)
C1—C6—C7—N1	−2.68 (19)	C10—N2—C13—C14	−81.01 (16)
C1—C6—C7—C8	176.67 (13)	C10—C2—C3—C4	−179.04 (14)
C2—C1—C6—C5	1.1 (2)	C11—N2—C10—C2	−72.47 (15)
C2—C1—C6—C7	−178.03 (12)	C11—N2—C13—C14	152.57 (14)
C2—C3—C4—C5	0.7 (2)	C13—N2—C10—C2	160.50 (12)
C2—C3—C4—C9	178.13 (14)	C13—N2—C11—C12	−85.09 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.85 (2)	1.78 (2)	2.5368 (16)	148 (2)
O2—H2···O5	0.91 (2)	1.71 (2)	2.5985 (16)	165 (2)
O3—H3···O5^{i}	0.82 (3)	1.82 (3)	2.6335 (17)	171 (3)
O4—H4···O7	0.85 (2)	1.84 (2)	2.6875 (19)	176 (2)

N2—H2*A*···O1	0.903 (19)	2.168 (18)	2.8121 (16)	127.6 (15)
O7—H7*A*···O6	0.79 (3)	2.07 (3)	2.823 (2)	159 (3)
O7—H7*B*···O6^{ii}	0.89 (3)	1.86 (3)	2.738 (2)	169 (2)

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