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

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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, C₁₄H₂₃N₂O₄·C₂H₃O₂·H₂O, was determined as part of a wider study on the use of the molecule as a polydentate ligand in the synthesis of Mn¾ 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 R₁(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. (2014a,b) – molecular wheels; Frost et al. (2014) – tetrahedron cage. For examples of other magnetic oxime-containing clusters, see: Vlahopoulou et al. (2009); Stamatakos 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

C₁₄H₂₃N₂O₄·C₂H₃O₂·H₂O
Mᵣ = 360.40
Monoclinic, P₂₁/c
a = 14.4338 (5) A
b = 10.4786 (3) A
\( c = 12.4045 (4) \) A
\( \beta = 101.593 (3) ^\circ \)

V = 1837.86 (10) A³
Z = 4
Mo Kα radiation
\( \mu = 0.10 \) mm⁻¹
\( T = 120 \) K
0.48 × 0.38 × 0.18 mm

2.2. Data collection

Agilent SuperNova diffractometer
Absorption correction: gaussian

(1047385)

38067 measured reflections
5542 independent reflections
4362 reflections with \( I > 2\sigma(I) \)
Rint = 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
\( \Delta \rho_{\text{max}} = 0.33 e \text{ Å}^{-3} \)
\( \Delta \rho_{\text{min}} = -0.24 e \text{ Å}^{-3} \)

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

\( \begin{array}{cccc}
D–H···A & D–H & H···A & D···A \\
O1–H1··· N1 & 0.85 (2) & 1.78 (2) & 2.5368 (16) & 148 (2) \\
O2–H2··· O5 & 0.91 (2) & 1.71 (2) & 2.5983 (16) & 165 (2) \\
O3–H3··· O5\textsuperscript{a} & 0.82 (3) & 1.82 (3) & 2.6335 (17) & 171 (3) \\
O4–H4··· O7 & 0.85 (2) & 1.84 (2) & 2.6875 (19) & 176 (2) \\
N2–H2A··· O1 & 0.903 (19) & 2.168 (18) & 2.8121 (16) & 127.6 (15) \\
O7–H7A··· O6 & 0.79 (3) & 2.07 (3) & 2.823 (2) & 159 (3) \\
O7–H7B··· O6\textsuperscript{b} & 0.89 (3) & 1.86 (3) & 2.738 (2) & 169 (2) \\
\end{array} \)

Symmetry codes: (i) x, -y, 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

Gary S. Nichol, Jamie M. Frost, Sergio Sanz and Euan K. Brechin

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 [Mn3O(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 H4L 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 R24(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

1H and 13CNMR 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 (H4L)
3-\{[\text{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\textsubscript{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\textsubscript{2}Cl\textsubscript{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%). \textsuperscript{1}H NMR (500 MHz, DMSO): \(\delta\) 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). \textsuperscript{13}C NMR (500 MHz, DMSO): \(\delta\) 157.28 (1C, C\textsubscript{arOH}), 153.86 (1C, CNOH), 131.34 (1C, CH), 127.61 (1C, CH), 126.99 (1C, C), 124.34 (1C, C), 121.01 (1C, C), 59.14 (2C, CH\textsubscript{2}), 56.51 (2C, CH\textsubscript{2}), 54.78 (1C, CH\textsubscript{2}), 21.69 (1C, CH\textsubscript{3}), 12.73 (1C, CH\textsubscript{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\textsubscript{L}. 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 H4L. 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

$C_{14}H_{23}N_{2}O_{4}^+\cdot C_{2}H_{3}O_{2}^-\cdot H_{2}O$

$M_r = 360.40$

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) Å³

$Z = 4$

$F(000) = 776$

$D_x = 1.303$ Mg m⁻³

Mo Kα 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

$0.48 \times 0.38 \times 0.18$ 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

5542 independent reflections

$T_{min} = 0.942, T_{max} = 0.975$

38067 measured reflections

$\theta_{max} = 31.1°, \theta_{min} = 3.1°$

3574 reflections with $I > 2\sigma(I)$

$h = -20→20$

$k = -15→13$

$l = -18→17$

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
\[ w = \frac{1}{\sigma^2(F_o^2) + (0.0431P)^2 + 0.9291P} \]
\[ \text{where } P = (F_o^2 + 2F_c^2)/3 \]
\[ (\Delta/\sigma)_{\text{max}} < 0.001 \]
\[ \Delta \rho_{\text{max}} = 0.33 \text{ e Å}^{-3} \]
\[ \Delta \rho_{\text{min}} = -0.24 \text{ e Å}^{-3} \]

**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      | Uiso* Ueq |
|-----|--------|--------|--------|-----------|
| 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)* |
| O5  | 0.16920 (10) | -0.16143 (13) | 0.60648 (11) | 0.0202 (3) |
| C1  | 0.17518 (10) | -0.26194 (13) | 0.53438 (12) | 0.0223 (3) |
| O6  | 0.09294 (11) | -0.32024 (14) | 0.47871 (12) | 0.0246 (3) |
| H6A | 0.0993 (13) | -0.3890 (18) | 0.4276 (15) | 0.029 (5)* |
| C2  | 0.00394 (11) | -0.27997 (14) | 0.49290 (12) | 0.0243 (3) |
| O7  | -0.00027 (10) | -0.17826 (14) | 0.56389 (11) | 0.0213 (3) |
| H7B | -0.00601 (16) | -0.1492 (17) | 0.5733 (14) | 0.024 (4)* |
| C3  | 0.08079 (10) | -0.11679 (13) | 0.62184 (11) | 0.0192 (3) |
| O8  | 0.07274 (10) | -0.00789 (14) | 0.69504 (11) | 0.0203 (3) |
| H8A | -0.02208 (11) | 0.04239 (17) | 0.70539 (14) | 0.0264 (3) |
| O9  | -0.0567 (16) | 0.073 (2) | 0.638 (2) | 0.053 (6)* |
| C4  | -0.0153 (17) | 0.104 (2) | 0.757 (2) | 0.058 (7)* |
| H8A | -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)* |
| H9A | -0.1359 (18) | -0.334 (2) | 0.474 (2) | 0.063 (7)* |
| H9B | -0.0770 (15) | -0.429 (2) | 0.4181 (18) | 0.048 (6)* |
| C10 | 0.27174 (11) | -0.30299 (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) | 0.03 (5)* |
| C12 | 0.28636 (12) | -0.02870 (16) | 0.33149 (13) | 0.0294 (3) |
**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) |
| Bond                                | Length (Å) | Angle (°) |
|-------------------------------------|------------|-----------|
| O1—H1                               | 0.85 (2)   |           |
| O1—C1                               | 1.3625 (17)|           |
| O2—H2                               | 0.91 (2)   |           |
| O2—N1                               | 1.3880 (16)|           |
| O3—H3                               | 0.82 (3)   |           |
| O3—C12                              | 1.415 (2)  |           |
| O4—H4                               | 0.85 (2)   |           |
| O4—C14                              | 1.420 (2)  |           |
| N1—C7                               | 1.2891 (19)|           |
| N2—H2A                              | 0.903 (19) |           |
| N2—C10                              | 1.513 (2)  |           |
| N2—C11                              | 1.5034 (19)|           |
| N2—C13                              | 1.503 (2)  |           |
| C1—C2                               | 1.396 (2)  |           |
| C1—C6                               | 1.408 (2)  |           |
| C2—C3                               | 1.389 (2)  |           |
| C2—C10                              | 1.503 (2)  |           |
| C3—H3A                              | 0.976 (19) |           |
| C3—C4                               | 1.396 (2)  |           |
| C4—C5                               | 1.392 (2)  |           |
| C4—C9                               | 1.506 (2)  |           |
| C5—H5                               | 0.955 (18) |           |
| C5—C6                               | 1.401 (2)  |           |
| C6—C7                               | 1.4773 (19)|           |
| C7—C8                               | 1.496 (2)  |           |
| C1—O1—H1                            |           | 106.8 (15)|           |
| N1—O2—H2                            |           | 103.4 (15)|           |
| C12—O3—H3                           |           | 107.3 (18)|           |
| C14—O4—H4                           |           | 107.4 (17)|           |
| C7—N1—O2                            |           | 114.11 (12)|          |
| C10—N2—H2A                          |           | 107.2 (12)|           |
| C11—N2—H2A                          |           | 106.8 (11)|           |
| C11—N2—C10                          |           | 111.31 (12)|          |
| C13—N2—H2A                          |           | 106.4 (11)|           |
| C13—N2—C10                          |           | 112.21 (12)|          |
| C13—N2—C11                          |           | 112.46 (12)|          |
| O1—C1—C2                            |           | 116.19 (13)|          |
| O1—C1—C6                            |           | 123.00 (13)|          |
| C2—C1—C6                            |           | 120.80 (13)|          |
| C1—C2—C10                           |           | 118.00 (13)|          |
| C3—C2—C1                            |           | 119.59 (14)|          |
| C3—C2—C10                           |           | 122.41 (13)|          |
| C2—C3—H3A                           |           | 117.7 (11)|           |
| C2—C3—C4                            |           | 121.35 (14)|          |

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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—C6 122.59 (14) C14—C13—H13A 113.1 (15)
C6—C5—C4 118.8 (11) C14—C13—H13B 111.5 (10)
C1—C6—C7 121.69 (13) O4—C14—C13 110.4 (11)
C5—C6—C7 117.66 (13) O4—C14—H14A 109.1 (12)
C5—C6—C4 120.65 (13) O4—C14—H14B 109.1 (12)
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)
C1—C6—C7—N1 178.18 (13) C15—C16—H16A 107 (2)
C1—C6—C7—C8 10.8 (2) C15—C16—H16B 107 (2)
C2—C1—C6—C7 −1.3 (2) C15—C16—H16C 111 (2)
C1—C6—C7—N2 178.03 (12) H7A—O7—H7B 107 (2)
C6—C1—C2—C3 179.65 (12) C3—C2—C10—N2 118.43 (15)
C1—C2—C3—C4 0.3 (2) C3—C4—C5—C6 −0.8 (2)
C1—C2—C10—N2 178.13 (12) C4—C5—C6—C1 −0.1 (2)
C1—C6—C7—N1 −2.68 (19) C4—C5—C6—C7 179.07 (13)
C1—C6—C7—C8 176.67 (13) C5—C6—C7—N1 178.18 (13)
C2—C1—C6—C7 −1.3 (2) C5—C6—C7—C8 −2.5 (2)
C2—C1—C2—C3 −179.04 (14) C6—C1—C2—C3 −1.3 (2)
C2—C1—C10—N2 −60.94 (17) C6—C1—C2—C3 178.13 (13)
C1—C6—C7—N2 148.01 (13) C9—C4—C5—C6 −178.27 (14)
C1—C6—C7—N1 −81.01 (16) C10—N2—C11—C12 148.01 (13)
C1—C6—C7—C8 152.57 (14) C10—N2—C11—C12 −72.47 (15)
C2—C1—C6—C7 160.50 (12) C11—N2—C10—C2 −72.47 (15)
C2—C3—C4—C5 0.7 (2) C11—N2—C10—C2 −85.09 (16)
C2—C3—C4—C9 178.13 (14) C13—N2—C10—C2 −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···O5i | 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) |

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N2—H2A···O1  &  0.903 (19) & 2.168 (18) & 2.8121 (16) & 127.6 (15)  
O7—H7A···O6  &  0.79 (3) & 2.07 (3) & 2.823 (2) & 159 (3)  
O7—H7B···O6ii  &  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.