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3-Ethyl-3-hydroxy-8-methoxyquinoline-2,4(1H,3H)-dione monohydrate

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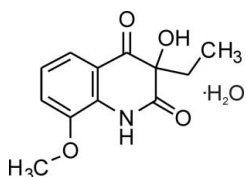
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.044; wR factor = 0.127; data-to-parameter ratio = 15.9.

In the title hydrate, $\text{C}_{12}\text{H}_{13}\text{NO}_4 \cdot \text{H}_2\text{O}$, the piperidine ring that is fused to the benzene ring is in a sofa conformation with the chiral C atom lying 0.4084 (18) Å out of the plane of the nine fused-ring atoms. In the crystal, $\text{O}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds link the organic molecules and water molecules into chains running along the b -axis direction. The chains are further connected into layers parallel to the bc plane by $\pi-\pi$ interactions between inversion-related benzene rings [centroid-centroid distance = 3.8846 (9) Å].

Related literature

For methods of preparation of 3-alkyl- or 3-aryl-3-hydroxyquinoline-2,4-diones by oxidation of the corresponding 3-alkyl- or 3-arylquinolin-2-ones, see: Stadlbauer & Kappe (1982); Stadlbauer *et al.* (1992). For naturally occurring 3-hydroxyquinoline-2,4-diones, see: Neuenhaus & Budzikiewicz (1979); Luo *et al.* (2009). For the biological activity of 3-hydroxyquinoline-2,4-diones, see: Prisyazhnyuk *et al.* (1984); Luo *et al.* (2009). For a related structure, see: Kafka *et al.* (2012).



Experimental

Crystal data

 $\text{C}_{12}\text{H}_{13}\text{NO}_4 \cdot \text{H}_2\text{O}$
 $M_r = 253.25$

 Orthorhombic, $Pbca$
 $a = 16.5055$ (4) Å

 $b = 8.8068$ (2) Å

 $c = 16.6690$ (4) Å

 $V = 2423.02$ (10) Å³
 $Z = 8$

 Mo $K\alpha$ radiation

 $\mu = 0.11$ mm⁻¹
 $T = 293$ K

 $0.50 \times 0.25 \times 0.20$ mm

Data collection

Nonius KappaCCD area-detector diffractometer

 Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)

 $T_{\min} = 0.948$, $T_{\max} = 0.979$

5200 measured reflections

2779 independent reflections

 1963 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.019$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.127$
 $S = 1.03$

2779 reflections

175 parameters

3 restraints

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.26$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.23$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O3}-\text{H3}\cdots\text{O1}^{\text{i}}$	0.82	2.35	2.9430 (17)	130
$\text{N1}-\text{H1N}\cdots\text{O1W}$	0.86 (1)	2.01 (2)	2.8438 (19)	163 (2)
$\text{O1W}-\text{H1W}\cdots\text{O2}^{\text{ii}}$	0.92 (2)	2.07 (2)	2.9425 (19)	158 (2)
$\text{O1W}-\text{H2W}\cdots\text{O3}^{\text{iii}}$	0.92 (2)	2.15 (2)	2.9401 (19)	144 (2)

 Symmetry codes: (i) $-x, y - \frac{1}{2}, -z + \frac{3}{2}$; (ii) $x, y + 1, z$; (iii) $-x, y + \frac{1}{2}, -z + \frac{3}{2}$.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK5162).

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supporting information

Acta Cryst. (2012). E68, o3199–o3200 [doi:10.1107/S1600536812043280]

3-Ethyl-3-hydroxy-8-methoxyquinoline-2,4(1*H*,3*H*)-dione monohydrate

Stanislav Kafka, Andrej Pevec, Karel Proisl, Roman Kimmel and Janez Košmrlj

S1. Comment

3-Alkyl- and 3-aryl-3-hydroxyquinoline-2,4-diones have been prepared by oxidation of the corresponding 3-alkyl- or 3-arylquinolin-2-ones by means of oxygen under UV irradiation (Stadlbauer & Kappe, 1982), *m*-chloroperoxybenzoic acid (Stadlbauer & Kappe, 1982), hydrogen peroxide (Stadlbauer & Kappe, 1982), nitric acid (Stadlbauer *et al.*, 1992), or peracetic acid (Stadlbauer *et al.*, 1992). Several 3-hydroxyquinoline-2,4-diones were isolated from bacteria *Pseudomonas aeruginosa* (Neuenhaus & Budzikiewicz, 1979) and from stem bark of *Micromelum falcatum* (Luo *et al.*, 2009). Biological activity of several 3-hydroxyquinoline-2,4-diones has been investigated (Prisyazhnyuk *et al.*, 1984; Luo *et al.*, 2009). 3-Alkyl- and 3-aryl-3-hydroxyquinoline-2,4-diones are important synthetic intermediates for the preparation of new types of heterocyclic compounds.

The asymmetric unit of the title compound (I) consists of a single 3-ethyl-3-hydroxy-8-methoxyquinoline-2,4(1*H*,3*H*)-dione molecule and solvated water molecule (Fig. 1). The piperidine ring that is fused to the benzene ring has a sofa shape with chiral carbon center 0.4084 (18) Å out of the plane for 9 ring atoms. In the crystal packing organic molecules and solvated water molecules are connected by two intermolecular O—H \cdots O and one N—H \cdots O hydrogen bonds. These connections altogether with additional O—H \cdots O hydrogen bonding between hydroxyl and carbonyl groups (Table 1) form linear chain running along the *b* axis. Non-covalent π – π interactions occur between inversion-related benzene rings [centroid–centroid distance = 3.8846 (9) Å], which stabilize the crystal packing and connect the chains into a two-dimensional supramolecular layer parallel to the *bc* plane (Fig. 2).

S2. Experimental

To a solution of 3-ethyl-4-hydroxy-8-methoxyquinolin-2(1*H*)-one (4.38 g, 20.0 mmol) in 0.5 *M* sodium hydroxide (120 ml), 30% peroxyacetic acid in acetic acid (6.7 ml, 30 mmol) was added drop-wise within 30 min under stirring at room temperature. After several minutes a precipitate started to form. The reaction mixture was stirred for additional 30 min and then left at 10 °C overnight. The solid phase was filtered off under suction, dispersed in 5% aqueous sodium bicarbonate solution (25 ml), filtered off and washed with water (3 x 20 ml). Crystallization of the above air-dried solid from toluene afforded 3.92 g (83%) of the final product (I), *M.pt* 371–372 K.

S3. Refinement

The N-bonded hydrogen atom was located in a difference map and refined with the using distance restraint with N—H = 0.86±0.02 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$. Water H atoms were located in a difference map and refined with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$. The O—H distance of water were restrained to be 0.96±0.02 Å. All other H atoms were included in the model at geometrically calculated positions and refined using a riding model, with C—H bond lengths constrained to 0.93 Å (aromatic H), 0.96 Å (methyl H), 0.97 Å (methylene H) and O—H = 0.82 Å, and with $U_{\text{iso}}(\text{H})$ values of 1.2 $U_{\text{eq}}(\text{C})$ [for aromatic and methylene H] or 1.5 $U_{\text{eq}}(\text{C})$ [for oxygen and methyl H].

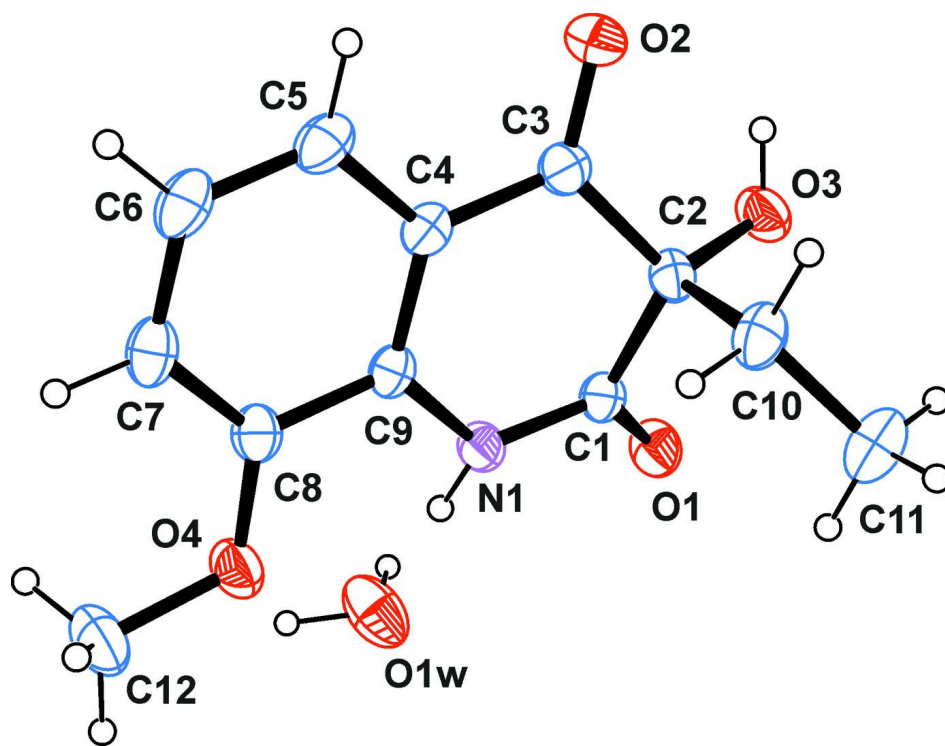
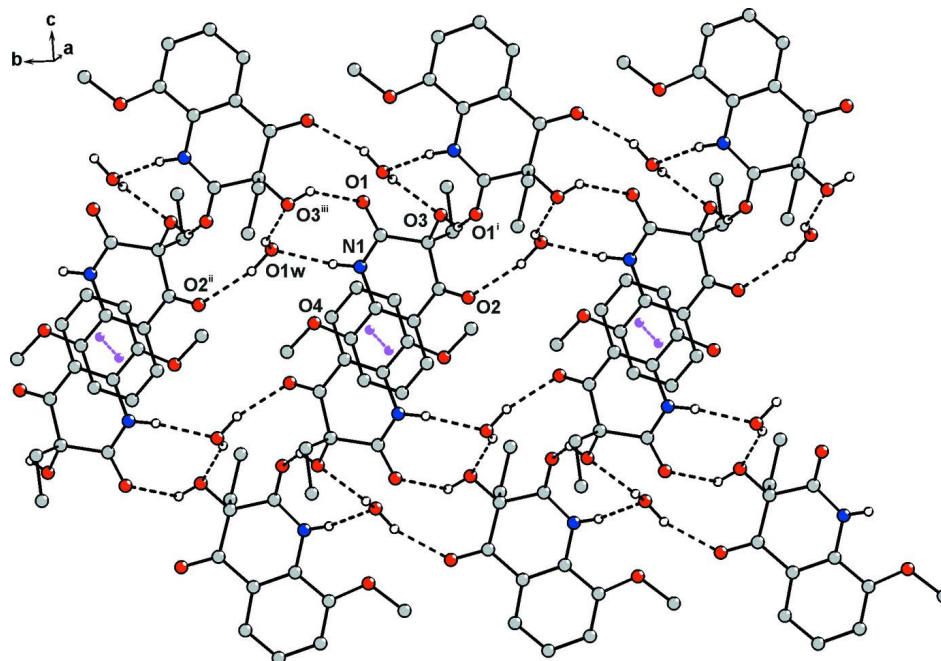


Figure 1

A view of the title compound showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

**Figure 2**

The packing of (I), with the O—H...O and N—H...O hydrogen bonds, and π — π interactions denoted by dashed lines. Hydrogen atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry codes: (i) $-x, y - 1/2, -z + 3/2$; (ii) $x, y + 1, z$; (iii) $-x, y + 1/2, -z + 3/2$]

3-Ethyl-3-hydroxy-8-methoxyquinoline-2,4(1*H*,3*H*)-dione monohydrate

Crystal data

$C_{12}H_{13}NO_4 \cdot H_2O$

$M_r = 253.25$

Orthorhombic, *Pbca*

Hall symbol: $-P\ 2ac\ 2ab$

$a = 16.5055\ (4)\ \text{\AA}$

$b = 8.8068\ (2)\ \text{\AA}$

$c = 16.6690\ (4)\ \text{\AA}$

$V = 2423.02\ (10)\ \text{\AA}^3$

$Z = 8$

$F(000) = 1072$

$D_x = 1.388\ \text{Mg m}^{-3}$

Melting point = 371–372 K

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 3161 reflections

$\theta = 1.0\text{--}27.5^\circ$

$\mu = 0.11\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Prism, yellow

$0.50 \times 0.25 \times 0.20\ \text{mm}$

Data collection

Nonius KappaCCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ scans + ω scans

Absorption correction: multi-scan

(*SCALEPACK*; Otwinowski & Minor, 1997)

$T_{\min} = 0.948, T_{\max} = 0.979$

5200 measured reflections

2779 independent reflections

1963 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.019$

$\theta_{\max} = 27.5^\circ, \theta_{\min} = 3.4^\circ$

$h = -21 \rightarrow 21$

$k = -11 \rightarrow 11$

$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.127$
 $S = 1.03$
 2779 reflections
 175 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
 $w = 1/[\sigma^2(F_o^2) + (0.0662P)^2 + 0.4669P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.0001$
 $\Delta\rho_{\max} = 0.26 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.23 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. 211 frames in 4 sets of φ scans + ω scans. Rotation/frame = 2°. Crystal-detector distance = 31 mm. Measuring time = 20 s/°.

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.08014 (8)	0.56475 (12)	0.75104 (6)	0.0505 (3)
O2	0.04557 (8)	0.18242 (13)	0.56925 (8)	0.0576 (4)
O3	0.05871 (8)	0.26982 (13)	0.72594 (7)	0.0527 (3)
H3	0.0359	0.1965	0.7058	0.079*
O4	0.14902 (8)	0.81485 (13)	0.51080 (7)	0.0542 (3)
N1	0.11348 (8)	0.61105 (15)	0.62211 (7)	0.0395 (3)
H1N	0.1065 (10)	0.7068 (16)	0.6299 (11)	0.047*
C1	0.09586 (9)	0.51728 (17)	0.68405 (8)	0.0374 (3)
C2	0.10269 (9)	0.34757 (17)	0.66669 (9)	0.0392 (4)
C3	0.07607 (9)	0.30617 (16)	0.58203 (10)	0.0408 (4)
C4	0.09477 (9)	0.41692 (17)	0.51912 (9)	0.0381 (3)
C5	0.09051 (10)	0.3782 (2)	0.43780 (10)	0.0481 (4)
H5	0.0777	0.2795	0.4227	0.058*
C6	0.10534 (11)	0.4863 (2)	0.38047 (10)	0.0527 (4)
H6	0.1023	0.4608	0.3264	0.063*
C7	0.12485 (10)	0.6336 (2)	0.40285 (9)	0.0492 (4)
H7	0.1344	0.7062	0.3634	0.059*
C8	0.13022 (9)	0.67423 (18)	0.48253 (9)	0.0410 (4)
C9	0.11398 (9)	0.56549 (17)	0.54186 (8)	0.0358 (3)
C10	0.19372 (10)	0.3023 (2)	0.67026 (10)	0.0523 (4)
H10A	0.1986	0.1950	0.6581	0.063*
H10B	0.2228	0.3579	0.6291	0.063*

C11	0.23315 (12)	0.3326 (3)	0.74990 (11)	0.0683 (6)
H11A	0.2035	0.2817	0.7915	0.102*
H11B	0.2333	0.4399	0.7602	0.102*
H11C	0.2879	0.2957	0.7490	0.102*
C12	0.16490 (14)	0.9320 (2)	0.45424 (12)	0.0670 (6)
H12A	0.1179	0.9477	0.4214	0.101*
H12B	0.2099	0.9033	0.4210	0.101*
H12C	0.1777	1.0243	0.4822	0.101*
O1W	0.06321 (11)	0.90745 (16)	0.66799 (10)	0.0752 (5)
H1W	0.0478 (14)	0.978 (3)	0.6301 (13)	0.090*
H2W	0.0132 (11)	0.900 (3)	0.6914 (14)	0.090*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0709 (8)	0.0477 (7)	0.0329 (6)	0.0008 (5)	0.0073 (5)	-0.0009 (5)
O2	0.0692 (8)	0.0379 (6)	0.0657 (8)	-0.0052 (5)	-0.0121 (6)	-0.0034 (5)
O3	0.0674 (8)	0.0436 (6)	0.0472 (7)	-0.0100 (6)	0.0055 (6)	0.0084 (5)
O4	0.0786 (9)	0.0454 (6)	0.0386 (6)	-0.0116 (6)	0.0060 (6)	0.0066 (5)
N1	0.0543 (8)	0.0341 (6)	0.0301 (6)	-0.0024 (5)	0.0009 (5)	-0.0008 (5)
C1	0.0403 (8)	0.0402 (8)	0.0318 (7)	0.0000 (6)	0.0002 (6)	0.0013 (6)
C2	0.0440 (8)	0.0380 (8)	0.0357 (8)	0.0022 (6)	0.0002 (6)	0.0046 (6)
C3	0.0398 (8)	0.0368 (8)	0.0457 (9)	0.0065 (6)	-0.0034 (7)	-0.0034 (6)
C4	0.0369 (7)	0.0427 (8)	0.0346 (7)	0.0054 (6)	-0.0030 (6)	-0.0040 (6)
C5	0.0512 (9)	0.0533 (10)	0.0399 (9)	0.0082 (8)	-0.0058 (7)	-0.0118 (7)
C6	0.0588 (10)	0.0682 (11)	0.0312 (8)	0.0108 (9)	-0.0013 (7)	-0.0079 (8)
C7	0.0531 (9)	0.0626 (11)	0.0319 (8)	0.0079 (8)	0.0049 (7)	0.0053 (7)
C8	0.0429 (8)	0.0451 (9)	0.0349 (8)	0.0033 (6)	0.0040 (6)	0.0024 (7)
C9	0.0360 (7)	0.0418 (8)	0.0295 (7)	0.0037 (6)	-0.0001 (6)	0.0000 (6)
C10	0.0496 (9)	0.0584 (10)	0.0490 (10)	0.0130 (8)	-0.0069 (8)	0.0009 (8)
C11	0.0583 (11)	0.0913 (16)	0.0553 (11)	0.0106 (11)	-0.0155 (9)	-0.0012 (10)
C12	0.0872 (15)	0.0552 (11)	0.0587 (12)	-0.0077 (10)	0.0127 (10)	0.0164 (9)
O1W	0.0992 (11)	0.0548 (8)	0.0715 (10)	0.0047 (8)	0.0365 (9)	0.0061 (7)

Geometric parameters (Å, °)

O1—C1	1.2202 (17)	C6—C7	1.388 (3)
O2—C3	1.2193 (19)	C6—H6	0.9300
O3—C2	1.4040 (18)	C7—C8	1.378 (2)
O3—H3	0.8200	C7—H7	0.9300
O4—C8	1.361 (2)	C8—C9	1.402 (2)
O4—C12	1.422 (2)	C10—C11	1.502 (2)
N1—C1	1.3537 (19)	C10—H10A	0.9700
N1—C9	1.3966 (18)	C10—H10B	0.9700
N1—H1N	0.861 (14)	C11—H11A	0.9600
C1—C2	1.527 (2)	C11—H11B	0.9600
C2—C3	1.522 (2)	C11—H11C	0.9600
C2—C10	1.555 (2)	C12—H12A	0.9600

C3—C4	1.465 (2)	C12—H12B	0.9600
C4—C9	1.399 (2)	C12—H12C	0.9600
C4—C5	1.399 (2)	O1W—H1W	0.923 (16)
C5—C6	1.371 (3)	O1W—H2W	0.916 (16)
C5—H5	0.9300		
C2—O3—H3	109.5	C8—C7—H7	119.5
C8—O4—C12	118.23 (14)	C6—C7—H7	119.5
C1—N1—C9	123.82 (13)	O4—C8—C7	125.75 (15)
C1—N1—H1N	117.0 (12)	O4—C8—C9	114.89 (13)
C9—N1—H1N	115.2 (12)	C7—C8—C9	119.34 (15)
O1—C1—N1	122.32 (14)	N1—C9—C4	121.80 (13)
O1—C1—C2	121.64 (13)	N1—C9—C8	118.71 (13)
N1—C1—C2	115.90 (12)	C4—C9—C8	119.41 (13)
O3—C2—C3	112.71 (13)	C11—C10—C2	114.01 (15)
O3—C2—C1	107.81 (12)	C11—C10—H10A	108.8
C3—C2—C1	112.89 (12)	C2—C10—H10A	108.8
O3—C2—C10	110.35 (13)	C11—C10—H10B	108.8
C3—C2—C10	104.65 (12)	C2—C10—H10B	108.8
C1—C2—C10	108.35 (13)	H10A—C10—H10B	107.6
O2—C3—C4	123.84 (15)	C10—C11—H11A	109.5
O2—C3—C2	119.69 (14)	C10—C11—H11B	109.5
C4—C3—C2	116.32 (13)	H11A—C11—H11B	109.5
C9—C4—C5	120.10 (14)	C10—C11—H11C	109.5
C9—C4—C3	118.46 (13)	H11A—C11—H11C	109.5
C5—C4—C3	121.38 (14)	H11B—C11—H11C	109.5
C6—C5—C4	119.83 (16)	O4—C12—H12A	109.5
C6—C5—H5	120.1	O4—C12—H12B	109.5
C4—C5—H5	120.1	H12A—C12—H12B	109.5
C5—C6—C7	120.20 (15)	O4—C12—H12C	109.5
C5—C6—H6	119.9	H12A—C12—H12C	109.5
C7—C6—H6	119.9	H12B—C12—H12C	109.5
C8—C7—C6	121.09 (16)	H1W—O1W—H2W	95 (2)
C9—N1—C1—O1	-165.15 (15)	C4—C5—C6—C7	0.2 (2)
C9—N1—C1—C2	19.1 (2)	C5—C6—C7—C8	0.4 (3)
O1—C1—C2—O3	22.8 (2)	C12—O4—C8—C7	-0.1 (3)
N1—C1—C2—O3	-161.35 (13)	C12—O4—C8—C9	-178.93 (16)
O1—C1—C2—C3	147.98 (15)	C6—C7—C8—O4	179.85 (16)
N1—C1—C2—C3	-36.19 (18)	C6—C7—C8—C9	-1.4 (2)
O1—C1—C2—C10	-96.60 (17)	C1—N1—C9—C4	0.9 (2)
N1—C1—C2—C10	79.22 (16)	C1—N1—C9—C8	177.65 (14)
O3—C2—C3—O2	-26.4 (2)	C5—C4—C9—N1	175.61 (14)
C1—C2—C3—O2	-148.87 (14)	C3—C4—C9—N1	-1.8 (2)
C10—C2—C3—O2	93.51 (17)	C5—C4—C9—C8	-1.1 (2)
O3—C2—C3—C4	157.87 (13)	C3—C4—C9—C8	-178.52 (14)
C1—C2—C3—C4	35.42 (18)	O4—C8—C9—N1	3.8 (2)
C10—C2—C3—C4	-82.20 (16)	C7—C8—C9—N1	-175.08 (14)

O2—C3—C4—C9	167.10 (15)	O4—C8—C9—C4	-179.40 (14)
C2—C3—C4—C9	-17.4 (2)	C7—C8—C9—C4	1.7 (2)
O2—C3—C4—C5	-10.3 (2)	O3—C2—C10—C11	-58.1 (2)
C2—C3—C4—C5	165.20 (14)	C3—C2—C10—C11	-179.62 (16)
C9—C4—C5—C6	0.1 (2)	C1—C2—C10—C11	59.70 (19)
C3—C4—C5—C6	177.47 (15)		

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
O3—H3...O1 ⁱ	0.82	2.35	2.9430 (17)	130
N1—H1N...O1W	0.86 (1)	2.01 (2)	2.8438 (19)	163 (2)
O1W—H1W...O2 ⁱⁱ	0.92 (2)	2.07 (2)	2.9425 (19)	158 (2)
O1W—H2W...O3 ⁱⁱⁱ	0.92 (2)	2.15 (2)	2.9401 (19)	144 (2)

Symmetry codes: (i) $-x, y-1/2, -z+3/2$; (ii) $x, y+1, z$; (iii) $-x, y+1/2, -z+3/2$.