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Crystal structure of (2*E*)-1-(4-hydroxy-1-methyl-2-oxo-1,2-dihydroquinolin-3-yl)-3-(4-hydroxy-3-methoxyphenyl)prop-2-en-1-one

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In the title compound, $C_{20}H_{17}NO_5$, the dihedral angle between the mean plane of the dihydroquinoline ring system (r.m.s. deviation = 0.003 Å) and the benzene ring is 1.83 (11)°. The almost planar conformation is a consequence of an intramolecular O—H···O hydrogen bond and the *E* configuration about the central C=C bond. In the crystal structure, O—H···O hydrogen bonds generate chains of molecules along the [10$\bar{1}$] direction. These chains are linked *via* π–π interactions [inter-centroid distances are in the range 3.6410 (16)–3.8663 (17) Å].

Keywords: crystal structure; 4-hydroxy-1,2-dihydroquinolin-2(1*H*)-one; α,β-unsaturated ketones; hydrogen bonding; π–π interactions

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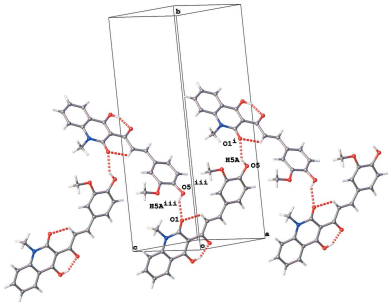
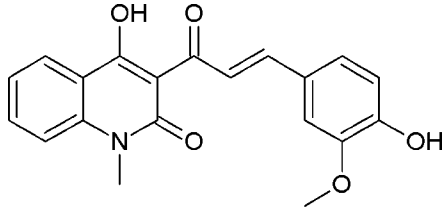
1. Chemical context

The quinoline ring is an important component of bioactive heterocycles because of its diversity (Larsen *et al.*, 1996; Chen *et al.*, 2001; Roma *et al.*, 2000; Dubé *et al.*, 1998; Billker *et al.*, 1998). Many derivatives containing 4-hydroxy-1,2-dihydroquinolin-2(1*H*)-one have wide applications in pharmaceuticals, such as anticancer (Hasegawa *et al.*, 1990), anti-inflammatory (Ukrainets *et al.*, 1996) and antiseizure (Rowley *et al.*, 1993). Some α,β-unsaturated ketones are known to have antimalarial, antibacterial and antifungal properties (Katritzky & Rees, 1984). The anticancer ability of some α,β-unsaturated ketones containing a quinoline ring has also been reported (Rezig *et al.*, 2000; Nguyen, 2007). A number of the α,β-unsaturated ketones containing quinoline synthesized by the Claisen–Schmidt reaction have been reported to inhibit antimalarial activity (Domínguez *et al.*, 2001). Moussaoui *et al.* (2002) also described the synthesis of α,β-unsaturated ketones containing a quinoline ring and claimed cytotoxicity with human leukemia cells. Here we present the synthesis and crystal structure of an α,β-unsaturated ketone derived from 3-acetyl-4-hydroxy-*N*-methylquinolin-2(1*H*)-one and 4-hydroxy-3-methoxybenzaldehyde.





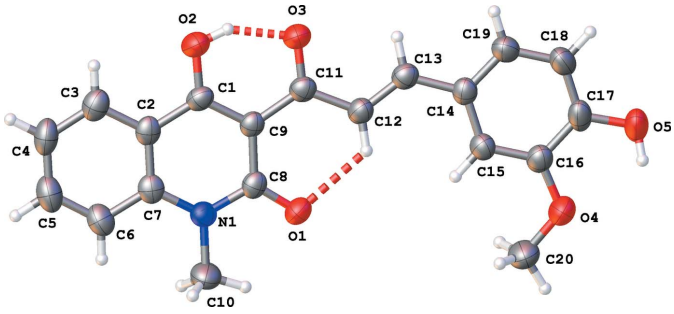


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines (see Table 1 for details).

2. Structural Commentary

The molecular structure of the title compound is illustrated in Fig. 1. The whole molecule is almost planar with a maximum deviation from the best plane through all atoms of 0.147 (3) Å for atom C20. The dihydroquinoline and benzene rings make a dihedral angle of 1.83 (11)° between the best planes. The configuration of the C12═C13 bond is *E*, with a C9—C11—C12—C13 torsion angle of 177.0 (2)°. In addition, intramolecular O2—H2···O3 and C12—H12···O1 hydrogen bonds assure the observed planarity of the structure (Table 1). Three short intramolecular contacts are observed: H10*B*···O1 (2.18 Å), H5*A*···O4 (2.25 Å) and H13···O3 (2.37 Å).

3. Supramolecular features

In the crystal, molecules are connected *via* O5—H5*A*···O1 hydrogen bonds, forming chains propagating along [10$\bar{1}$] (Fig. 2 and Table 1). These chains are linked by π–π interactions involving both ring systems (Fig. 3) and C—H···O interactions (Table 1). The inter-centroid distances are 3.6410 (16) and 3.8663 (17) Å for π–π interactions involving *Cg*1···*Cg*2^{iv} and *Cg*3···*Cg*2^{v}, respectively, where *Cg*1, *Cg*2 and *Cg*3 are the centroids of the N1/C1–C2/C7–C9, C2–C7 and C14–C19 rings, respectively [symmetry codes: (iv) $-x+1, -y, -z+2$; (v) $-x+2, -y, -z+2$].

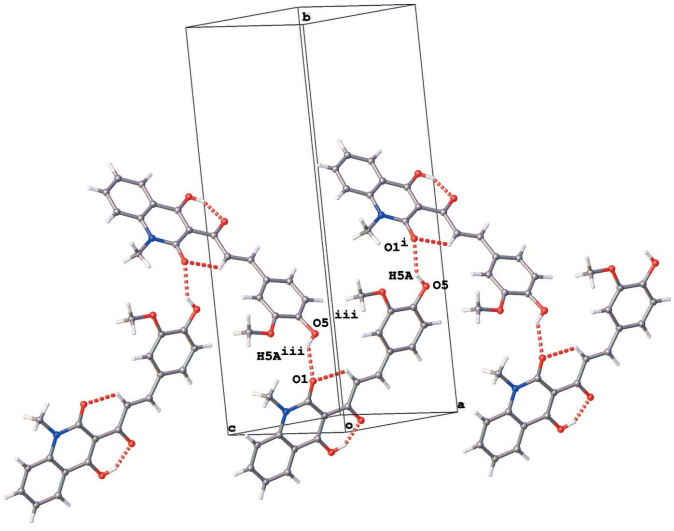


Figure 2
Infinite chains in the [10$\bar{1}$] direction formed by O5—H5*A*···O1 hydrogen bonds (shown as red dashed lines). [Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$; (iii) $x-\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$.]

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O3	0.84	1.65	2.407 (3)	148
O5—H5*A*···O1^{i}	0.84	2.05	2.730 (3)	137
C12—H12···O1	0.98	2.18	2.822 (3)	124
C10—H10*C*···O3^{ii}	0.98	2.56	3.523 (3)	167

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

4. Database survey

A search of the Cambridge Structural Database (Version 5.36; last update November 2014; Groom & Allen, 2014) for α,β-unsaturated ketones C—CH═CH—C(═O)—O gave 1281 hits of which the majority adopts an *E* configuration (C—C═C—C torsion angle around 180°) as in the title compound. For only 19 entries this torsion angle is centered around 0°. A search for 1,2-dihydroquinoline derivatives gave 706 hits of which none contains an α,β-unsaturated ketone at the 3-position. The angle between the best planes through the two six-membered rings in these 1,2-dihydroquinoline derivatives is in the range of 0–22.13°. In the title compound, this angle is 1.49 (12)°.

5. Synthesis and crystallization

The precursors 4-hydroxy-6-methyl-2*H*-pyrano[3,2-*c*]quinoline-2,5(6*H*)-dione and 3-acetyl-4-hydroxy-*N*-methylquinolin-2(1*H*)-one were prepared in high yield (87.0 and 92.5%, respectively) according to Kappe *et al.* (1994).

The title compound was synthesized by refluxing a solution of 2.17 g (0.01 mol) of 3-acetyl-4-hydroxy-*N*-methylquinolin-2(1*H*)-one, 1.52 g (0.01 mol) of 4-hydroxy-3-methoxybenzaldehyde, 22 ml of chloroform and 5 drops of piperidine

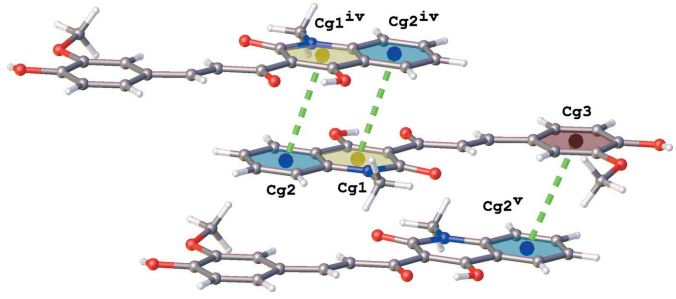


Figure 3
π–π interactions in the crystal of the title compound shown as green dashed lines. [Symmetry codes: (iv) $-x+1, -y, -z+2$; (v) $-x+2, -y, -z+2$.]

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{20}H_{17}NO_5$
M_r	351.35
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	100
a, b, c (Å)	8.3634 (8), 22.664 (2), 8.8079 (9)
β (°)	95.413 (3)
V (Å^3)	1662.1 (3)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.84
Crystal size (mm)	0.58 × 0.22 × 0.04
Data collection	
Diffractometer	Bruker SMART 6000
Absorption correction	Multi-scan (*SADABS*; Bruker, 2003)
T_{min}, T_{max}	0.641, 0.967
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15707, 2881, 1889
R_{int}	0.086
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.056, 0.156, 1.02
No. of reflections	2881
No. of parameters	239
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.19

Computer programs: *SMART* and *SAINT* (Bruker, 2003), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) and *OLEX2* (Dolomanov *et al.*, 2009).

(as a catalyst) in a 100 ml flask for 30 h. The precipitate was filtered off and recrystallized from ethanol to obtain the title product as yellow crystals. The yield was 2.03 g (58%); m.p. 505–506 K, R_f 0.7 ($CHCl_3$–C_2H_5OH = 7:1 *v*/*v*).

IR (KBr, cm^{-1}): 3357, 3115 (ν_{OH}); 1637 ($\nu_{C=O}$); 997 ($\nu_{CH=\ trans}$). ^{1}H NMR (δ p.p.m.; DMSO-d_6, Bruker Avance 500 MHz): 8.47 (1H, *d*, ^2J = 16.0 Hz, H_β), 7.92 (1H, *d*, ^2J = 16.0 Hz, H_α), 3.59 (3H, *s* CH_3-N), 7.33 (1H, *t*, ^3J = 8.0 Hz, C_6-H), 7.55 (1H, *d*, ^3J = 8.0 Hz, C_5-H), 7.81 (1H, *t*, ^3J = 8.0 Hz, C_7-H), 8.13 (1H, *d*, ^3J = 8.0 Hz, C_8-H), 3.85 (3H, *s*, OCH_3), 6.89 (2H, *d*, ^3J = 8.0 Hz, C_{13}-H), 7.27 (1H, *d*, ^3J = 8.0 Hz, C_{12}-H), 7.30 (1H, *s*, C_9-H), 9.89 (1H, *s*, C_4-OH). Calculation for $C_{20}H_{17}NO_5$: M = 351 au. Found (by ESI MS, m/z): 351 (M^+).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms were refined using a riding model with stretchable C—H and O—H distances and with U_{iso} = 1.2U_{eq}(C) (1.5 times for methyl and hydroxyl groups).

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Crystal structure of (2*E*)-1-(4-hydroxy-1-methyl-2-oxo-1,2-dihydroquinolin-3-yl)-3-(4-hydroxy-3-methoxyphenyl)prop-2-en-1-one

Peter Mangwala Kimpende, Ngoc Thanh Nguyen, Minh Thao Nguyen, Quoc Trung Vu and Luc Van Meervelt

Computing details

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT* (Bruker, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: OLEX2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: OLEX2 (Dolomanov *et al.*, 2009).

(2*E*)-1-(4-Hydroxy-1-methyl-2-oxo-1,2-dihydroquinolin-3-yl)-3-(4-hydroxy-3-methoxyphenyl)prop-2-en-1-one

Crystal data

$C_{20}H_{17}NO_5$
$M_r = 351.35$
Monoclinic, $P2_1/n$
$a = 8.3634$ (8) Å
$b = 22.664$ (2) Å
$c = 8.8079$ (9) Å
$\beta = 95.413$ (3)°
$V = 1662.1$ (3) Å^3
$Z = 4$
$F(000) = 736$
$D_x = 1.404$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
$\mu = 0.84$ mm^{-1}
$T = 100$ K
Block, yellow
0.58 × 0.22 × 0.04 mm

Data collection

Bruker SMART 6000 diffractometer
Radiation source: fine-focus sealed tube
Crossed Gobel mirrors monochromator
w\ and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 2003)
$T_{min} = 0.641$, $T_{max} = 0.967$
15707 measured reflections
2881 independent reflections
1889 reflections with $I > 2\sigma(I)$
$R_{int} = 0.086$
$\theta_{max} = 66.6°$, $\theta_{min} = 3.9°$
$h = -9 \rightarrow 9$
$k = -26 \rightarrow 26$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.156$
$S = 1.02$
2881 reflections
239 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0641P)^2 + 0.0033P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.7024 (3)	−0.04442 (11)	0.9256 (3)	0.0415 (6)
N1	0.7229 (2)	0.02301 (9)	1.1896 (2)	0.0420 (5)
O1	0.8821 (2)	0.08925 (8)	1.0865 (2)	0.0512 (5)
C2	0.6258 (3)	−0.06452 (11)	1.0557 (3)	0.0413 (6)
O2	0.6850 (2)	−0.07762 (9)	0.8038 (2)	0.0583 (6)
H2	0.7380	−0.0633	0.7362	0.088*
C3	0.5393 (3)	−0.11726 (12)	1.0516 (4)	0.0523 (7)
H3	0.5284	−0.1402	0.9609	0.063*
O3	0.8460 (2)	−0.00808 (8)	0.6787 (2)	0.0546 (5)
C4	0.4692 (3)	−0.13626 (13)	1.1794 (4)	0.0587 (8)
H4	0.4108	−0.1723	1.1772	0.070*
O4	1.2629 (3)	0.28691 (9)	0.7891 (2)	0.0675 (6)
C5	0.4853 (3)	−0.10230 (14)	1.3095 (4)	0.0620 (8)
H5	0.4376	−0.1152	1.3973	0.074*
O5	1.3629 (3)	0.29415 (9)	0.5092 (3)	0.0723 (7)
H5A	1.3606	0.3205	0.5759	0.109*
C6	0.5689 (3)	−0.05015 (13)	1.3150 (3)	0.0540 (7)
H6	0.5789	−0.0277	1.4064	0.065*
C7	0.6394 (3)	−0.02989 (11)	1.1873 (3)	0.0421 (6)
C8	0.8029 (3)	0.04333 (11)	1.0694 (3)	0.0394 (6)
C9	0.7893 (3)	0.00803 (10)	0.9302 (3)	0.0370 (5)
C10	0.7290 (4)	0.06001 (14)	1.3261 (3)	0.0635 (8)
H10A	0.6203	0.0733	1.3420	0.095*
H10B	0.7974	0.0944	1.3128	0.095*
H10C	0.7732	0.0372	1.4148	0.095*
C11	0.8604 (3)	0.02611 (11)	0.7932 (3)	0.0418 (6)
C12	0.9447 (3)	0.08179 (11)	0.7778 (3)	0.0447 (6)
H12	0.9507	0.1094	0.8594	0.054*
C13	1.0134 (3)	0.09468 (11)	0.6516 (3)	0.0429 (6)
H13	1.0054	0.0654	0.5741	0.051*
C14	1.0990 (3)	0.14816 (11)	0.6180 (3)	0.0420 (6)
C15	1.1320 (3)	0.19255 (11)	0.7263 (3)	0.0447 (6)

H15	1.0944	0.1890	0.8244	0.054*
C16	1.2192 (3)	0.24159 (12)	0.6913 (3)	0.0480 (7)
C17	1.2722 (3)	0.24754 (12)	0.5466 (4)	0.0536 (7)
C18	1.2366 (4)	0.20473 (12)	0.4387 (4)	0.0584 (8)
H18	1.2714	0.2090	0.3396	0.070*
C19	1.1502 (3)	0.15542 (12)	0.4737 (3)	0.0524 (7)
H19	1.1256	0.1261	0.3981	0.063*
C20	1.2328 (4)	0.28030 (14)	0.9429 (4)	0.0699 (9)
H20A	1.1166	0.2782	0.9499	0.105*
H20B	1.2773	0.3141	1.0019	0.105*
H20C	1.2834	0.2439	0.9839	0.105*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0403 (13)	0.0349 (13)	0.0493 (15)	0.0023 (10)	0.0045 (11)	−0.0069 (11)
N1	0.0485 (11)	0.0400 (12)	0.0372 (12)	0.0021 (9)	0.0034 (10)	0.0010 (8)
O1	0.0626 (11)	0.0393 (11)	0.0518 (11)	−0.0104 (9)	0.0065 (9)	−0.0053 (8)
C2	0.0355 (12)	0.0364 (14)	0.0522 (16)	0.0059 (10)	0.0046 (11)	0.0078 (11)
O2	0.0723 (13)	0.0472 (12)	0.0584 (13)	−0.0144 (10)	0.0210 (10)	−0.0155 (9)
C3	0.0465 (15)	0.0426 (16)	0.0679 (19)	−0.0003 (12)	0.0057 (14)	0.0037 (13)
O3	0.0706 (12)	0.0439 (11)	0.0515 (11)	−0.0091 (9)	0.0171 (10)	−0.0107 (8)
C4	0.0519 (16)	0.0476 (17)	0.077 (2)	−0.0073 (13)	0.0092 (15)	0.0175 (15)
O4	0.0914 (15)	0.0506 (13)	0.0635 (14)	−0.0232 (11)	0.0223 (12)	−0.0085 (10)
C5	0.0574 (18)	0.066 (2)	0.064 (2)	−0.0045 (15)	0.0134 (16)	0.0199 (15)
O5	0.0931 (16)	0.0444 (13)	0.0869 (18)	−0.0117 (11)	0.0471 (14)	0.0035 (10)
C6	0.0532 (15)	0.0613 (19)	0.0482 (16)	0.0000 (13)	0.0084 (13)	0.0105 (13)
C7	0.0345 (12)	0.0436 (15)	0.0476 (15)	0.0056 (10)	0.0016 (11)	0.0069 (11)
C8	0.0368 (12)	0.0336 (13)	0.0475 (15)	0.0028 (10)	0.0024 (11)	0.0029 (10)
C9	0.0355 (12)	0.0315 (13)	0.0443 (14)	0.0040 (10)	0.0051 (11)	0.0013 (10)
C10	0.086 (2)	0.064 (2)	0.0411 (16)	−0.0142 (17)	0.0089 (16)	−0.0072 (13)
C11	0.0407 (13)	0.0374 (14)	0.0479 (15)	0.0050 (11)	0.0068 (12)	−0.0028 (11)
C12	0.0455 (14)	0.0382 (14)	0.0519 (16)	−0.0020 (11)	0.0126 (12)	−0.0035 (11)
C13	0.0425 (13)	0.0378 (14)	0.0486 (15)	0.0030 (11)	0.0056 (12)	−0.0025 (11)
C14	0.0386 (12)	0.0382 (14)	0.0506 (16)	0.0026 (10)	0.0108 (12)	0.0028 (11)
C15	0.0451 (14)	0.0435 (15)	0.0470 (15)	−0.0019 (11)	0.0120 (12)	−0.0001 (11)
C16	0.0495 (15)	0.0400 (15)	0.0564 (17)	0.0009 (11)	0.0141 (13)	−0.0009 (12)
C17	0.0584 (17)	0.0380 (15)	0.068 (2)	0.0030 (12)	0.0270 (15)	0.0101 (13)
C18	0.077 (2)	0.0479 (17)	0.0552 (18)	0.0014 (14)	0.0308 (16)	0.0037 (13)
C19	0.0632 (17)	0.0456 (17)	0.0508 (17)	0.0012 (13)	0.0185 (14)	−0.0033 (12)
C20	0.089 (2)	0.063 (2)	0.059 (2)	−0.0198 (18)	0.0139 (18)	−0.0138 (15)

Geometric parameters (Å, º)

C1—C2	1.439 (4)	C8—C9	1.459 (3)
C1—O2	1.307 (3)	C9—C11	1.454 (3)
C1—C9	1.392 (3)	C10—H10A	0.9800
N1—C7	1.387 (3)	C10—H10B	0.9800

N1—C8	1.384 (3)	C10—H10C	0.9800
N1—C10	1.462 (3)	C11—C12	1.458 (3)
O1—C8	1.235 (3)	C12—H12	0.9500
C2—C3	1.396 (4)	C12—C13	1.331 (4)
C2—C7	1.395 (4)	C13—H13	0.9500
O2—H2	0.8400	C13—C14	1.452 (3)
C3—H3	0.9500	C14—C15	1.396 (4)
C3—C4	1.386 (4)	C14—C19	1.389 (4)
O3—C11	1.269 (3)	C15—H15	0.9500
C4—H4	0.9500	C15—C16	1.380 (4)
C4—C5	1.377 (4)	C16—C17	1.395 (4)
O4—C16	1.367 (3)	C17—C18	1.371 (4)
O4—C20	1.409 (4)	C18—H18	0.9500
C5—H5	0.9500	C18—C19	1.381 (4)
C5—C6	1.372 (4)	C19—H19	0.9500
O5—H5A	0.8400	C20—H20A	0.9800
O5—C17	1.359 (3)	C20—H20B	0.9800
C6—H6	0.9500	C20—H20C	0.9800
C6—C7	1.396 (4)		

O2—C1—C2	116.6 (2)	H10A—C10—H10B	109.5
O2—C1—C9	122.2 (2)	H10A—C10—H10C	109.5
C9—C1—C2	121.2 (2)	H10B—C10—H10C	109.5
C7—N1—C10	119.1 (2)	O3—C11—C9	118.2 (2)
C8—N1—C7	123.7 (2)	O3—C11—C12	117.7 (2)
C8—N1—C10	117.2 (2)	C9—C11—C12	124.1 (2)
C3—C2—C1	121.3 (3)	C11—C12—H12	119.4
C7—C2—C1	118.4 (2)	C13—C12—C11	121.2 (2)
C7—C2—C3	120.3 (2)	C13—C12—H12	119.4
C1—O2—H2	109.5	C12—C13—H13	116.1
C2—C3—H3	119.9	C12—C13—C14	127.8 (2)
C4—C3—C2	120.2 (3)	C14—C13—H13	116.1
C4—C3—H3	119.9	C15—C14—C13	122.1 (2)
C3—C4—H4	120.4	C19—C14—C13	119.1 (2)
C5—C4—C3	119.1 (3)	C19—C14—C15	118.8 (2)
C5—C4—H4	120.4	C14—C15—H15	119.9
C16—O4—C20	117.7 (2)	C16—C15—C14	120.2 (3)
C4—C5—H5	119.3	C16—C15—H15	119.9
C6—C5—C4	121.4 (3)	O4—C16—C15	125.4 (3)
C6—C5—H5	119.3	O4—C16—C17	114.5 (2)
C17—O5—H5A	109.5	C15—C16—C17	120.1 (3)
C5—C6—H6	119.7	O5—C17—C16	122.0 (3)
C5—C6—C7	120.5 (3)	O5—C17—C18	118.1 (3)
C7—C6—H6	119.7	C18—C17—C16	119.9 (3)
N1—C7—C2	120.0 (2)	C17—C18—H18	120.0
N1—C7—C6	121.5 (3)	C17—C18—C19	120.1 (3)
C2—C7—C6	118.5 (3)	C19—C18—H18	120.0
N1—C8—C9	117.1 (2)	C14—C19—H19	119.6

O1—C8—N1	118.6 (2)	C18—C19—C14	120.9 (3)
O1—C8—C9	124.3 (2)	C18—C19—H19	119.6
C1—C9—C8	119.5 (2)	O4—C20—H20A	109.5
C1—C9—C11	118.0 (2)	O4—C20—H20B	109.5
C11—C9—C8	122.5 (2)	O4—C20—H20C	109.5
N1—C10—H10A	109.5	H20A—C20—H20B	109.5
N1—C10—H10B	109.5	H20A—C20—H20C	109.5
N1—C10—H10C	109.5	H20B—C20—H20C	109.5

C1—C2—C3—C4	178.7 (2)	C7—C2—C3—C4	−1.4 (4)
C1—C2—C7—N1	1.1 (3)	C8—N1—C7—C2	−3.3 (4)
C1—C2—C7—C6	−178.3 (2)	C8—N1—C7—C6	176.0 (2)
C1—C9—C11—O3	−3.2 (3)	C8—C9—C11—O3	178.2 (2)
C1—C9—C11—C12	175.5 (2)	C8—C9—C11—C12	−3.1 (4)
N1—C8—C9—C1	−1.9 (3)	C9—C1—C2—C3	−179.7 (2)
N1—C8—C9—C11	176.7 (2)	C9—C1—C2—C7	0.5 (3)
O1—C8—C9—C1	177.2 (2)	C9—C11—C12—C13	177.0 (2)
O1—C8—C9—C11	−4.2 (4)	C10—N1—C7—C2	177.0 (2)
C2—C1—C9—C8	−0.1 (3)	C10—N1—C7—C6	−3.7 (4)
C2—C1—C9—C11	−178.7 (2)	C10—N1—C8—O1	4.2 (3)
C2—C3—C4—C5	0.4 (4)	C10—N1—C8—C9	−176.6 (2)
O2—C1—C2—C3	0.8 (4)	C11—C12—C13—C14	179.0 (2)
O2—C1—C2—C7	−179.0 (2)	C12—C13—C14—C15	5.8 (4)
O2—C1—C9—C8	179.5 (2)	C12—C13—C14—C19	−174.4 (3)
O2—C1—C9—C11	0.8 (4)	C13—C14—C15—C16	177.5 (2)
C3—C2—C7—N1	−178.8 (2)	C13—C14—C19—C18	−177.9 (3)
C3—C2—C7—C6	1.9 (4)	C14—C15—C16—O4	−178.0 (2)
C3—C4—C5—C6	0.1 (5)	C14—C15—C16—C17	1.2 (4)
O3—C11—C12—C13	−4.3 (4)	C15—C14—C19—C18	2.0 (4)
C4—C5—C6—C7	0.4 (4)	C15—C16—C17—O5	−177.8 (3)
O4—C16—C17—O5	1.5 (4)	C15—C16—C17—C18	0.4 (4)
O4—C16—C17—C18	179.7 (3)	C16—C17—C18—C19	−0.9 (5)
C5—C6—C7—N1	179.3 (2)	C17—C18—C19—C14	−0.4 (5)
C5—C6—C7—C2	−1.4 (4)	C19—C14—C15—C16	−2.4 (4)
O5—C17—C18—C19	177.4 (3)	C20—O4—C16—C15	7.6 (4)
C7—N1—C8—O1	−175.6 (2)	C20—O4—C16—C17	−171.6 (3)
C7—N1—C8—C9	3.6 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O3	0.84	1.65	2.407 (3)	148
O5—H5*A*···O1^{i}	0.84	2.05	2.730 (3)	137
C12—H12···O1	0.98	2.18	2.822 (3)	124
C10—H10*C*···O3^{ii}	0.98	2.56	3.523 (3)	167

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