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4-Hydroxy-1-methyl-3-phenylquinolin-2(1H)-one

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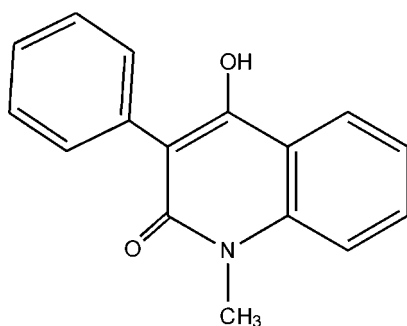
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.039; wR factor = 0.102; data-to-parameter ratio = 8.5.

In the title compound, $\text{C}_{16}\text{H}_{13}\text{NO}_2$, the quinoline system is approximately planar with a maximum deviation from the least-squares plane of 0.059 (1) Å for the N atom. The phenyl ring is rotated by 62.16 (4)° with respect to the plane of the quinoline system. In the crystal, $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds link molecules into infinite chains running along the b -axis direction.

Related literature

For the preparation of the title compound and other 4-hydroxyquinolin-2-ones, see: Baumgarten & Kärgel (1927); Lange *et al.*, (2001); Martensson & Nilsson (1960); Bezuglyi *et al.* (1992). For synthetic utilization of the title compound, see: Kafka *et al.* (2002); Klásek *et al.* (2002).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{13}\text{NO}_2$
 $M_r = 251.27$
 Monoclinic, $P2_1$
 $a = 6.1787$ (2) Å
 $b = 8.2696$ (2) Å
 $c = 12.3665$ (4) Å
 $\beta = 101.632$ (2)°

$V = 618.89$ (3) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 293$ K
 $0.50 \times 0.25 \times 0.10$ mm

Data collection

Nonius KappaCCD area-detector diffractometer
 Absorption correction: multi-scan (SCALEPACK; Otwinowski & Minor, 1997)
 $T_{\min} = 0.957$, $T_{\max} = 0.991$
 2580 measured reflections
 1479 independent reflections
 1235 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.102$
 $S = 1.02$
 1479 reflections
 174 parameters
 1 restraint
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.13$ e Å⁻³
 $\Delta\rho_{\min} = -0.16$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O}2-\text{H}2\text{O}\cdots\text{O}1^i$	0.82	1.89	2.655 (2)	156

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

Data collection: COLLECT (Nonius, 1998); cell refinement: DENZO and SCALEPACK (Otwinowski & Minor, 1997); data reduction: DENZO and SCALEPACK; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); 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, 2012).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FY2079).

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supporting information

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4-Hydroxy-1-methyl-3-phenylquinolin-2(1*H*)-one

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S1. Comment

The title compound, (I) (Fig. 1), was first prepared by the thermal condensation of diethyl phenylmalonate with *N*-methylaniline (Baumgarten & Kärgel 1927). This reaction, performed with various malonates and anilines, is still the most widely used general method for the preparation of 4-hydroxyquinolin-2-ones, including compound I. The performance of the reaction under microwave irradiation was described by Lange *et al.* (2001). Among other approaches to the preparation of compound I and other 4-hydroxyquinoline-2-diones, intramolecular condensations of 2-acylamino-benzoates could be particularly feasible (Martensson & Nilsson, 1960; Bezuglyi *et al.*, 1992). Recently, compound I was utilized for the preparation of the corresponding 3-bromo- and 3-chloro-1-methyl-3-phenylquinoline-2,4(1*H*,3*H*)-diones, from which other compounds were prepared by nucleophilic substitution of the halogen atom (Kafka *et al.*, 2002; Klásek *et al.*, 2002).

In the crystal structure of the title compound (I) (Fig. 2) 4-hydroxy-1-methyl-3-phenylquinolin-2(1*H*)-one molecules are connected by intermolecular O—H \cdots O hydrogen bonds between the hydroxyl and carbonyl groups (Table 1). These connections form linear chains along the *b*-axis in the crystal structure.

S2. Experimental

Title compound was prepared according to a modified procedure published by Baumgarten & Kärgel (1927). A mixture of *N*-methylaniline (10.7 g, 100 mmol) and diethyl phenylmalonate (24.8 g, 105 mmol) was gradually heated in a Wood's metal bath at 200–290 °C for 4.5 h (until the distillation of ethanol stopped; reached 8.57 g, *i.e.* 93% of theoretical mass of distillate). The hot reaction mixture was poured into a mortar, crushed after cooling and dissolved in the mixture of aqueous sodium hydroxide solution (0.5 *M*, 300 ml) and toluene (50 ml). The aqueous phase was separated, washed with toluene, shortly stirred with active carbon, filtered and acidified by addition of 10% hydrochloric acid to Congo red. The precipitated white solid was filtered off, washed with water and air dried affording 23.4 g (93% of theory) of crude product, m. p. 223–225 °C. Crystallization of the crude product from ethanol afforded 20.1 g (80% of theoretical yield) of the title compound (I), m. p. 222–226 °C. In the literature (Martensson & Nilsson, 1960), the same m. p. is given.

S3. Refinement

All 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 CH), 0.96 Å (methyl CH₃), and O—H = 0.82 Å, and with $U_{\text{iso}}(\text{H})$ values of $1.2U_{\text{eq}}(\text{C})$ [for aromatic CH] or $1.5U_{\text{eq}}(\text{C})$ [for OH and methyl groups]. In the absence of significant anomalous scattering, the Flack parameter could not be determined reliably. Therefore Friedel-pairs were merged prior to the final refinement cycle. 16 low angle reflections were dropped by the integration routines because of detector saturation.

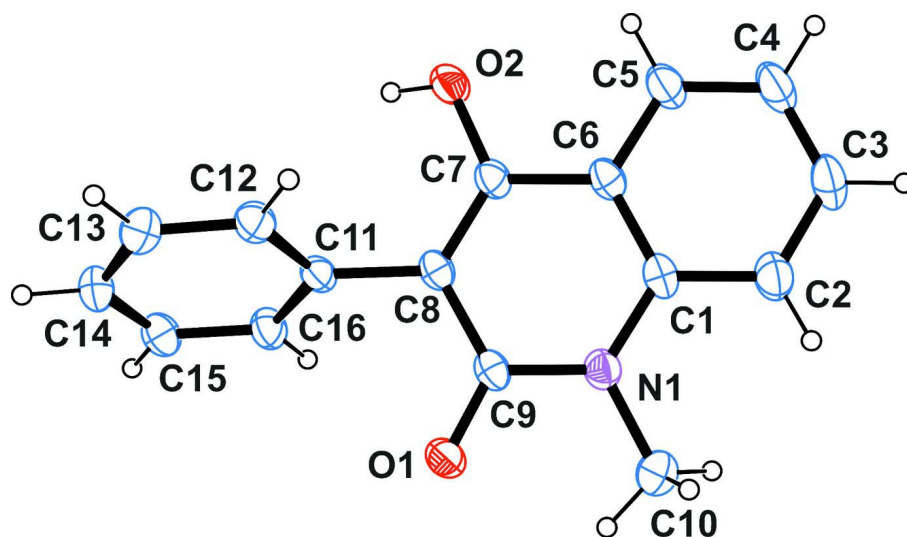
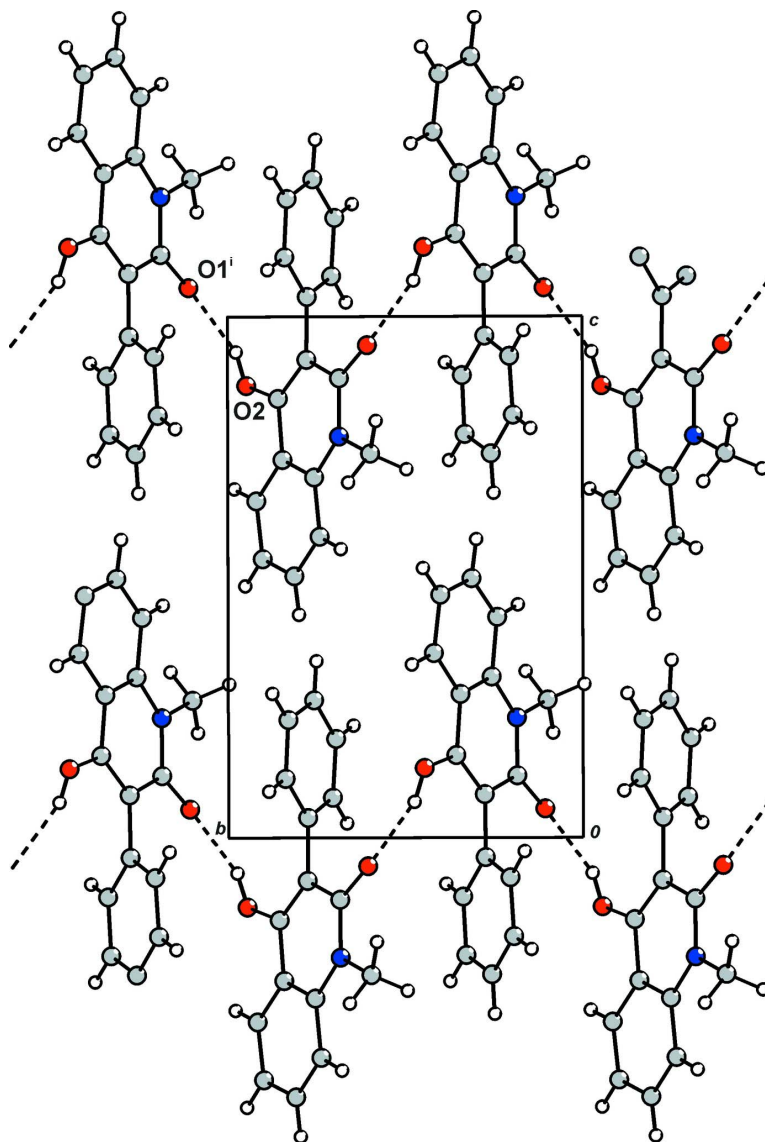


Figure 1

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 hydrogen bonds. [Symmetry code: (i) $-x, y + 1/2, -z + 2.$]

4-Hydroxy-1-methyl-3-phenylquinolin-2(1*H*)-one

Crystal data

$C_{16}H_{13}NO_2$

$M_r = 251.27$

Monoclinic, $P2_1$

Hall symbol: $P\ 2yb$

$a = 6.1787(2)\ \text{\AA}$

$b = 8.2696(2)\ \text{\AA}$

$c = 12.3665(4)\ \text{\AA}$

$\beta = 101.632(2)^\circ$

$V = 618.89(3)\ \text{\AA}^3$

$Z = 2$

$F(000) = 264$

$D_x = 1.348\ \text{Mg m}^{-3}$

Melting point = 495–499 K

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

Cell parameters from 1471 reflections

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

$\mu = 0.09\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Prism, colorless

$0.50 \times 0.25 \times 0.10\ \text{mm}$

Data collection

Nonius KappaCCD area-detector diffractometer	2580 measured reflections 1479 independent reflections
Radiation source: fine-focus sealed tube	1235 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{\text{int}} = 0.017$
φ scans + ω scans	$\theta_{\text{max}} = 27.4^\circ$, $\theta_{\text{min}} = 5.5^\circ$
Absorption correction: multi-scan (SCALEPACK; Otwinowski & Minor, 1997)	$h = -7 \rightarrow 7$
$T_{\text{min}} = 0.957$, $T_{\text{max}} = 0.991$	$k = -10 \rightarrow 8$
	$l = -16 \rightarrow 15$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.039$	H-atom parameters constrained
$wR(F^2) = 0.102$	$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2 + 0.0786P]$
$S = 1.02$	where $P = (F_o^2 + 2F_c^2)/3$
1479 reflections	$(\Delta/\sigma)_{\text{max}} = 0.0001$
174 parameters	$\Delta\rho_{\text{max}} = 0.13 \text{ e } \text{\AA}^{-3}$
1 restraint	$\Delta\rho_{\text{min}} = -0.16 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

Experimental. 216 frames in 4 sets of φ scans + ω scans. Rotation/frame = 2.0° . Crystal-detector distance = 31 mm. Measuring time = 200 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	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	-0.3506 (3)	0.6870 (2)	0.76995 (14)	0.0421 (5)
O1	-0.3484 (2)	0.6084 (2)	0.94498 (12)	0.0461 (4)
O2	0.2230 (3)	0.9476 (3)	0.86700 (12)	0.0527 (5)
H2O	0.2433	0.9751	0.9320	0.079*
C1	-0.2491 (4)	0.7574 (3)	0.69150 (17)	0.0408 (5)
C2	-0.3367 (5)	0.7420 (4)	0.57760 (19)	0.0563 (7)
H2	-0.4659	0.6833	0.5536	0.068*
C3	-0.2321 (5)	0.8131 (4)	0.50211 (19)	0.0640 (8)
H3	-0.2903	0.8004	0.4272	0.077*
C4	-0.0430 (6)	0.9027 (4)	0.5350 (2)	0.0631 (7)
H4	0.0251	0.9512	0.4827	0.076*
C5	0.0453 (5)	0.9202 (3)	0.64626 (19)	0.0531 (6)
H5	0.1720	0.9821	0.6688	0.064*
C6	-0.0539 (4)	0.8458 (3)	0.72531 (17)	0.0408 (5)

C7	0.0393 (4)	0.8579 (3)	0.84224 (17)	0.0392 (5)
C8	-0.0553 (4)	0.7782 (3)	0.91735 (16)	0.0374 (5)
C9	-0.2559 (3)	0.6871 (3)	0.88098 (16)	0.0374 (5)
C10	-0.5599 (4)	0.6003 (4)	0.7363 (2)	0.0558 (6)
H10A	-0.5330	0.4973	0.7057	0.084*
H10B	-0.6579	0.6625	0.6817	0.084*
H10C	-0.6261	0.5843	0.7994	0.084*
C11	0.0412 (3)	0.7767 (3)	1.03788 (15)	0.0374 (5)
C12	0.2473 (4)	0.7070 (3)	1.07702 (19)	0.0456 (5)
H12	0.3288	0.6669	1.0274	0.055*
C13	0.3319 (4)	0.6971 (4)	1.1895 (2)	0.0537 (6)
H13	0.4682	0.6483	1.2151	0.064*
C14	0.2142 (5)	0.7594 (4)	1.26318 (19)	0.0568 (7)
H14	0.2709	0.7523	1.3386	0.068*
C15	0.0133 (4)	0.8321 (4)	1.22571 (19)	0.0535 (6)
H15	-0.0640	0.8764	1.2758	0.064*
C16	-0.0749 (4)	0.8396 (3)	1.11342 (18)	0.0460 (6)
H16	-0.2124	0.8871	1.0886	0.055*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0427 (10)	0.0467 (10)	0.0362 (9)	0.0025 (9)	0.0062 (7)	0.0040 (9)
O1	0.0438 (8)	0.0554 (10)	0.0408 (8)	0.0007 (8)	0.0124 (6)	0.0101 (7)
O2	0.0653 (10)	0.0598 (10)	0.0366 (8)	-0.0198 (9)	0.0191 (8)	-0.0084 (8)
C1	0.0524 (12)	0.0392 (11)	0.0311 (10)	0.0092 (10)	0.0095 (9)	0.0020 (9)
C2	0.0630 (15)	0.0639 (17)	0.0397 (12)	0.0002 (13)	0.0047 (11)	0.0030 (12)
C3	0.087 (2)	0.0729 (19)	0.0299 (11)	0.0020 (17)	0.0058 (12)	0.0045 (12)
C4	0.0925 (19)	0.0656 (17)	0.0352 (11)	-0.0053 (16)	0.0220 (12)	0.0065 (12)
C5	0.0719 (15)	0.0535 (15)	0.0376 (11)	-0.0069 (13)	0.0201 (11)	-0.0013 (12)
C6	0.0531 (13)	0.0377 (11)	0.0339 (10)	0.0027 (10)	0.0143 (9)	-0.0009 (9)
C7	0.0472 (12)	0.0402 (12)	0.0325 (10)	-0.0009 (9)	0.0136 (9)	-0.0039 (9)
C8	0.0448 (11)	0.0383 (11)	0.0312 (10)	0.0041 (9)	0.0128 (8)	-0.0010 (9)
C9	0.0418 (11)	0.0392 (11)	0.0327 (10)	0.0083 (10)	0.0115 (8)	0.0030 (9)
C10	0.0476 (12)	0.0669 (16)	0.0497 (13)	-0.0029 (13)	0.0022 (10)	0.0035 (13)
C11	0.0432 (11)	0.0398 (11)	0.0315 (10)	-0.0029 (10)	0.0127 (9)	0.0005 (9)
C12	0.0439 (12)	0.0535 (14)	0.0410 (11)	0.0019 (11)	0.0126 (9)	-0.0017 (11)
C13	0.0470 (13)	0.0618 (15)	0.0492 (13)	0.0024 (12)	0.0019 (10)	0.0052 (13)
C14	0.0621 (15)	0.0732 (17)	0.0317 (11)	-0.0115 (14)	0.0017 (10)	0.0033 (11)
C15	0.0594 (15)	0.0693 (16)	0.0353 (11)	-0.0037 (13)	0.0177 (10)	-0.0062 (12)
C16	0.0483 (12)	0.0557 (14)	0.0354 (11)	0.0045 (11)	0.0118 (9)	-0.0011 (11)

Geometric parameters (Å, °)

N1—C9	1.380 (3)	C7—C8	1.363 (3)
N1—C1	1.385 (3)	C8—C9	1.443 (3)
N1—C10	1.464 (3)	C8—C11	1.490 (3)
O1—C9	1.248 (3)	C10—H10A	0.9600

O2—C7	1.339 (3)	C10—H10B	0.9600
O2—H2O	0.8200	C10—H10C	0.9600
C1—C6	1.400 (3)	C11—C16	1.389 (3)
C1—C2	1.409 (3)	C11—C12	1.393 (3)
C2—C3	1.370 (4)	C12—C13	1.386 (3)
C2—H2	0.9300	C12—H12	0.9300
C3—C4	1.374 (4)	C13—C14	1.376 (4)
C3—H3	0.9300	C13—H13	0.9300
C4—C5	1.382 (3)	C14—C15	1.373 (4)
C4—H4	0.9300	C14—H14	0.9300
C5—C6	1.397 (3)	C15—C16	1.387 (3)
C5—H5	0.9300	C15—H15	0.9300
C6—C7	1.448 (3)	C16—H16	0.9300
C9—N1—C1	122.40 (19)	O1—C9—N1	118.3 (2)
C9—N1—C10	117.14 (19)	O1—C9—C8	123.29 (18)
C1—N1—C10	120.37 (18)	N1—C9—C8	118.40 (18)
C7—O2—H2O	109.5	N1—C10—H10A	109.5
N1—C1—C6	119.64 (18)	N1—C10—H10B	109.5
N1—C1—C2	121.7 (2)	H10A—C10—H10B	109.5
C6—C1—C2	118.7 (2)	N1—C10—H10C	109.5
C3—C2—C1	120.2 (2)	H10A—C10—H10C	109.5
C3—C2—H2	119.9	H10B—C10—H10C	109.5
C1—C2—H2	119.9	C16—C11—C12	118.78 (19)
C2—C3—C4	121.3 (2)	C16—C11—C8	120.87 (19)
C2—C3—H3	119.3	C12—C11—C8	120.32 (18)
C4—C3—H3	119.3	C13—C12—C11	120.4 (2)
C3—C4—C5	119.5 (2)	C13—C12—H12	119.8
C3—C4—H4	120.3	C11—C12—H12	119.8
C5—C4—H4	120.3	C14—C13—C12	120.0 (2)
C4—C5—C6	120.7 (2)	C14—C13—H13	120.0
C4—C5—H5	119.7	C12—C13—H13	120.0
C6—C5—H5	119.7	C15—C14—C13	120.2 (2)
C5—C6—C1	119.60 (19)	C15—C14—H14	119.9
C5—C6—C7	121.7 (2)	C13—C14—H14	119.9
C1—C6—C7	118.66 (19)	C14—C15—C16	120.2 (2)
O2—C7—C8	124.90 (19)	C14—C15—H15	119.9
O2—C7—C6	114.51 (18)	C16—C15—H15	119.9
C8—C7—C6	120.6 (2)	C15—C16—C11	120.3 (2)
C7—C8—C9	119.96 (18)	C15—C16—H16	119.8
C7—C8—C11	123.1 (2)	C11—C16—H16	119.8
C9—C8—C11	116.93 (18)		
C9—N1—C1—C6	-6.3 (3)	C6—C7—C8—C11	175.7 (2)
C10—N1—C1—C6	177.3 (2)	C1—N1—C9—O1	-173.6 (2)
C9—N1—C1—C2	173.3 (2)	C10—N1—C9—O1	2.9 (3)
C10—N1—C1—C2	-3.0 (3)	C1—N1—C9—C8	6.6 (3)
N1—C1—C2—C3	-180.0 (2)	C10—N1—C9—C8	-177.0 (2)

C6—C1—C2—C3	-0.3 (4)	C7—C8—C9—O1	178.3 (2)
C1—C2—C3—C4	-1.1 (5)	C11—C8—C9—O1	-0.4 (3)
C2—C3—C4—C5	0.7 (5)	C7—C8—C9—N1	-1.8 (3)
C3—C4—C5—C6	1.0 (5)	C11—C8—C9—N1	179.48 (19)
C4—C5—C6—C1	-2.3 (4)	C7—C8—C11—C16	118.9 (3)
C4—C5—C6—C7	178.0 (3)	C9—C8—C11—C16	-62.4 (3)
N1—C1—C6—C5	-178.4 (2)	C7—C8—C11—C12	-63.2 (3)
C2—C1—C6—C5	1.9 (3)	C9—C8—C11—C12	115.5 (2)
N1—C1—C6—C7	1.4 (3)	C16—C11—C12—C13	1.7 (4)
C2—C1—C6—C7	-178.3 (2)	C8—C11—C12—C13	-176.3 (2)
C5—C6—C7—O2	1.2 (3)	C11—C12—C13—C14	-1.4 (4)
C1—C6—C7—O2	-178.5 (2)	C12—C13—C14—C15	-0.2 (5)
C5—C6—C7—C8	-177.1 (2)	C13—C14—C15—C16	1.6 (5)
C1—C6—C7—C8	3.2 (3)	C14—C15—C16—C11	-1.3 (4)
O2—C7—C8—C9	179.0 (2)	C12—C11—C16—C15	-0.3 (4)
C6—C7—C8—C9	-2.9 (3)	C8—C11—C16—C15	177.6 (2)
O2—C7—C8—C11	-2.4 (4)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O2—H2O...O1 ⁱ	0.82	1.89	2.655 (2)	156

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