

2-Hydroxy-7-methoxy-9H-carbazole-3-carbaldehyde

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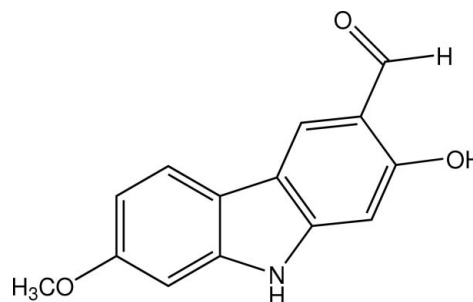
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.030; wR factor = 0.097; data-to-parameter ratio = 11.9.

The title compound, $\text{C}_{14}\text{H}_{11}\text{NO}_3$, was isolated from the roots of *Clausena wallichii*. The carbazole ring system is approximately planar (r.m.s. deviation = 0.039 Å) and the dihedral angle between the two benzene rings is 4.63 (7)°. An intramolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond generates an $S(6)$ ring motif. In the crystal, molecules are linked into a zigzag network extending parallel to the ac plane by $\text{O}-\text{H}\cdots\text{N}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For compounds isolated from plants of genera Rutaceae and their pharmacological activity, see: Ito *et al.* (1997); Kongkathip & Kongkathip (2009); Laphookhieo *et al.* (2009); Li *et al.* (1991); Maneerat & Laphookhieo (2010); Maneerat *et al.* (2010); Sripisut & Laphookhieo (2010); Tangyue-nyongwatthana *et al.* (1992); Yenjai *et al.* (2000). For a related structure, see: Fun *et al.* (2009). For bond-length data, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For the stability of the temperature controller used in the data collection, see Cosier & Glazer, (1986).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{11}\text{NO}_3$
 $M_r = 241.24$
 Orthorhombic, $Pna2_1$
 $a = 12.4352$ (4) Å
 $b = 17.6564$ (5) Å
 $c = 5.0839$ (1) Å
 $V = 1116.23$ (5) Å³
 $Z = 4$
 Cu $K\alpha$ radiation
 $\mu = 0.84$ mm⁻¹
 $T = 100$ K
 $0.23 \times 0.19 \times 0.10$ mm

Data collection

Bruker APEXII DUO CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2009)
 $T_{\min} = 0.831$, $T_{\max} = 0.918$
 25207 measured reflections
 2026 independent reflections
 2018 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$
 $wR(F^2) = 0.097$
 $S = 1.31$
 2026 reflections
 170 parameters
 1 restraint
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.64$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.63$ e Å⁻³
 Absolute structure: Flack (1983), 851 Friedel pairs
 Flack parameter: 0.17 (19)

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1O1}\cdots\text{O2}$	0.81	1.96	2.6453 (17)	143
$\text{O1}-\text{H1O1}\cdots\text{N1}^i$	0.81	2.53	3.0457 (15)	123
$\text{N1}-\text{H1N1}\cdots\text{O2}^{ii}$	0.85 (2)	2.10 (2)	2.941 (2)	172 (2)

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

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI5167).

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supporting information

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2-Hydroxy-7-methoxy-9H-carbazole-3-carbaldehyde

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S1. Comment

Rutaceae plants are known to be rich sources of coumarins and carbazole alkaloids. Many of them have been isolated from several genera of Rutaceae especially from *Clausena* genus (Laphookhieo *et al.*, 2009; Maneerat *et al.*, 2010; Sripisut & Laphookhieo 2010; Kongkathip & Kongkathip 2009; Ito *et al.*, 1997; Li *et al.*, 1991; Tangyuenyongwatthana *et al.*, 1992) and some of these compounds show interesting pharmacological activities (Maneerat & Laphookhieo 2010; Yenjai *et al.* 2000). Although *Clausena wallichii* is one of the Rutaceae plants, however phytochemical reports on the chemical constituents from this plant are rare. As part of our continuing study of chemical constituents and bioactive compounds from Thai medicinal plants, we report herein the crystal structure of the title compound, which was isolated from the roots of *C. wallichii* collected from Phrae province in the northern region of Thailand.

The non-hydrogen atoms of the title molecule (Fig. 1) are almost coplanar. The carbazole ring system (C1–C12/N1) is planar with an r.m.s. deviation of 0.039 Å [maximum deviation 0.072 (1) Å for atom C7]. The pyrrole ring makes dihedral angle of 1.66 (7) and 3.12 (8)°, respectively, with the C1–C4/C10–C11 and C5–C9/C12 benzene rings. The dihedral angle between the two benzene rings being 4.63 (7)°. The cabaldehyde and methoxy substituents at atoms C3 and C7, respectively, are coplanar with the benzene ring, as indicated by torsion angles C2–C3–C13–O2 = 0.0 (2)° and C14–O3–C7–C8 = 4.0 (2)°. An intramolecular O1—H1O1···O2 hydrogen bond (Table 1) generates an S(6) ring motif (Fig. 1 and Table 1) (Bernstein *et al.*, 1995). The bond distances are within normal ranges (Allen *et al.*, 1987) and comparable to a related structure (Fun *et al.*, 2009).

The crystal packing of the title compound is stabilized by intermolecular O—H···N and N—H···O hydrogen bonds (Table 1) which link the molecules into a zigzag network extending parallel to the *ac* plane.

S2. Experimental

The roots of *C. wallichii* (1.02 Kg) were successively extracted with CH₂Cl₂ over the period of 3 days each at room temperature to provide the crude CH₂Cl₂ extract which subjected to quick column chromatography (QCC) over silica gel eluted with a gradient of hexane–EtOAc (100% hexane to 100% EtOAc) to provide nine fractions (A–I). Fraction G (3.12 g) was further separated by QCC with a gradient of 10% EtOAc–hexane to 100% EtOAc to give seven subfractions (G1–G7). Subfraction G4 (118.9 mg) was subjected to repeated column chromatography using 30% EtOAc–hexane to yield the yellow solid of the title compound (12.8 mg). Yellow plate-shaped single crystals of the title compound suitable for X-ray structure determination were recrystallized from CH₂Cl₂/acetone (1:1 v/v) by the slow evaporation of the solvent at room temperature after several days; m.p. 496.7–498.8 K (decomposition).

S3. Refinement

Atom H1N1 was located in a difference map and refined isotropically. The remaining H atoms were placed in calculated positions with O–H = 0.81, C–H = 0.93 for aromatic and CH, and 0.96 Å for CH₃ atoms. The U_{iso} values were constrained

to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups. The highest residual electron density peak is located at 1.81 \AA from C9 and the deepest hole is located at 1.29 \AA from C3. 851 Friedel pairs were used to determine the absolute structure.

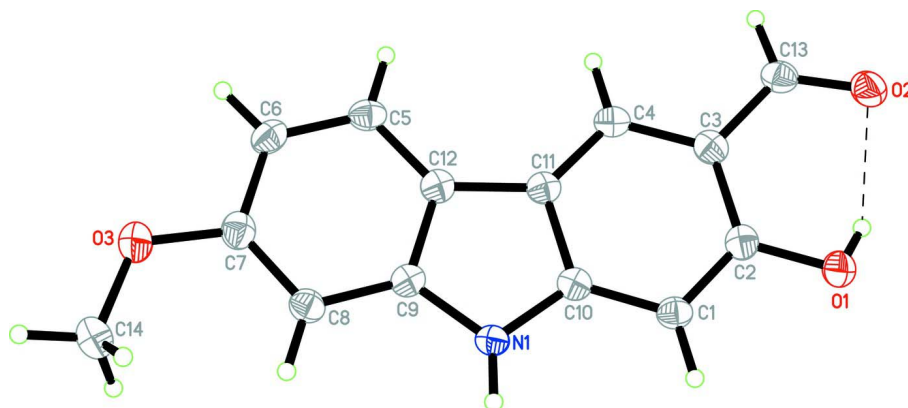


Figure 1

The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme. The O—H...O hydrogen bond is shown as a dashed line.

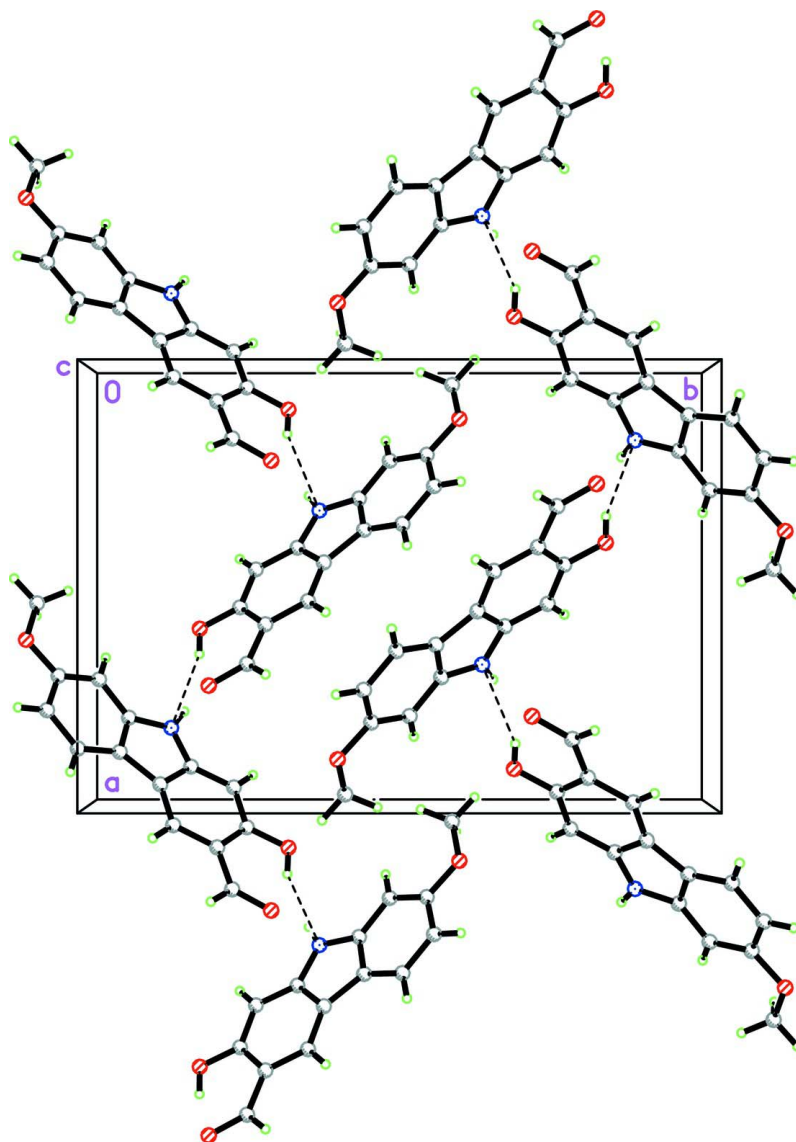


Figure 2

Part of the crystal packing of the title compound, viewed along the *c* axis. Hydrogen bonds are shown as dashed lines.

2-Hydroxy-7-methoxy-9*H*-carbazole-3-carbaldehyde

Crystal data

$C_{14}H_{11}NO_3$

$M_r = 241.24$

Orthorhombic, $Pna2_1$

Hall symbol: $P\ 2c\ -2n$

$a = 12.4352\ (4)\ \text{\AA}$

$b = 17.6564\ (5)\ \text{\AA}$

$c = 5.0839\ (1)\ \text{\AA}$

$V = 1116.23\ (5)\ \text{\AA}^3$

$Z = 4$

$F(000) = 504$

$D_x = 1.435\ \text{Mg m}^{-3}$

Melting point = 496.7–498.8 K

Cu $K\alpha$ radiation, $\lambda = 1.54178\ \text{\AA}$

Cell parameters from 2026 reflections

$\theta = 4.4\text{--}69.9^\circ$

$\mu = 0.84\ \text{mm}^{-1}$

$T = 100\ \text{K}$

Plate, yellow

$0.23 \times 0.19 \times 0.10\ \text{mm}$

Data collection

Bruker APEXII DUO CCD area-detector
diffractometer

Radiation source: sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2009)

$T_{\min} = 0.831$, $T_{\max} = 0.918$

25207 measured reflections

2026 independent reflections

2018 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 69.9^\circ$, $\theta_{\min} = 4.4^\circ$

$h = -14 \rightarrow 14$

$k = -21 \rightarrow 21$

$l = -5 \rightarrow 6$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.097$

$S = 1.31$

2026 reflections

170 parameters

1 restraint

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

$w = 1/[\sigma^2(F_o^2) + (0.0656P)^2 + 0.054P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.64 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.63 \text{ e } \text{\AA}^{-3}$

Extinction correction: *SHELXTL* (Sheldrick,
2008), $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.053 (3)

Absolute structure: Flack (1983), 851 Friedel
pairs

Absolute structure parameter: 0.17 (19)

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.59667 (8)	0.17861 (6)	0.4400 (2)	0.0264 (3)
H1O1	0.6537	0.1796	0.5149	0.056 (7)*
O2	0.72058 (8)	0.20104 (6)	0.8557 (3)	0.0294 (3)
O3	0.12071 (9)	0.59553 (6)	0.5822 (3)	0.0325 (3)
N1	0.32559 (9)	0.37021 (7)	0.3111 (3)	0.0203 (3)
H1N1	0.2898 (14)	0.3514 (10)	0.185 (5)	0.029 (5)*
C1	0.46098 (10)	0.26763 (8)	0.3568 (3)	0.0213 (3)
H1A	0.4366	0.2391	0.2150	0.026*
C2	0.54764 (10)	0.24453 (8)	0.5070 (3)	0.0212 (3)
C3	0.58509 (11)	0.28796 (8)	0.7249 (3)	0.0220 (3)
C4	0.53291 (10)	0.35607 (8)	0.7921 (3)	0.0208 (3)

H4A	0.5569	0.3847	0.9339	0.025*
C5	0.36091 (11)	0.50464 (7)	0.8333 (3)	0.0226 (3)
H5A	0.4092	0.5117	0.9708	0.027*
C6	0.27613 (11)	0.55440 (8)	0.7967 (3)	0.0241 (3)
H6A	0.2680	0.5955	0.9094	0.029*
C7	0.20217 (11)	0.54348 (8)	0.5906 (3)	0.0238 (3)
C8	0.21259 (10)	0.48413 (7)	0.4134 (3)	0.0222 (4)
H8A	0.1644	0.4774	0.2756	0.027*
C9	0.29943 (10)	0.43496 (7)	0.4528 (3)	0.0195 (3)
C10	0.41167 (10)	0.33567 (7)	0.4277 (3)	0.0196 (3)
C11	0.44591 (10)	0.37986 (8)	0.6456 (3)	0.0194 (3)
C12	0.37263 (11)	0.44372 (8)	0.6603 (3)	0.0203 (3)
C13	0.67188 (10)	0.26163 (8)	0.8877 (3)	0.0244 (3)
H13A	0.6930	0.2923	1.0273	0.029*
C14	0.03861 (12)	0.58496 (9)	0.3899 (4)	0.0354 (4)
H14A	-0.0179	0.6211	0.4184	0.053*
H14B	0.0101	0.5346	0.4043	0.053*
H14C	0.0684	0.5921	0.2174	0.053*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0261 (5)	0.0281 (5)	0.0251 (6)	0.0080 (4)	-0.0009 (4)	-0.0024 (5)
O2	0.0282 (5)	0.0358 (6)	0.0241 (7)	0.0098 (4)	-0.0016 (4)	0.0011 (5)
O3	0.0345 (6)	0.0291 (6)	0.0340 (7)	0.0104 (4)	-0.0052 (5)	-0.0053 (5)
N1	0.0207 (6)	0.0215 (6)	0.0187 (6)	0.0000 (4)	-0.0025 (5)	-0.0007 (5)
C1	0.0224 (6)	0.0225 (7)	0.0188 (8)	-0.0013 (5)	0.0010 (6)	-0.0003 (5)
C2	0.0202 (6)	0.0226 (6)	0.0209 (8)	0.0007 (5)	0.0047 (5)	0.0017 (6)
C3	0.0206 (7)	0.0242 (7)	0.0212 (8)	-0.0011 (5)	0.0019 (6)	0.0032 (6)
C4	0.0207 (6)	0.0234 (7)	0.0183 (8)	-0.0035 (5)	0.0000 (5)	0.0005 (6)
C5	0.0257 (7)	0.0221 (7)	0.0199 (7)	-0.0044 (5)	-0.0007 (6)	0.0000 (6)
C6	0.0303 (7)	0.0204 (6)	0.0217 (8)	-0.0017 (5)	0.0022 (6)	-0.0028 (6)
C7	0.0258 (7)	0.0210 (7)	0.0246 (8)	0.0016 (5)	0.0025 (6)	0.0029 (6)
C8	0.0226 (7)	0.0224 (7)	0.0216 (9)	-0.0004 (5)	-0.0014 (5)	0.0023 (6)
C9	0.0201 (6)	0.0195 (6)	0.0191 (8)	-0.0028 (5)	0.0020 (5)	0.0005 (6)
C10	0.0179 (6)	0.0217 (7)	0.0193 (8)	-0.0026 (4)	0.0014 (5)	0.0020 (6)
C11	0.0211 (6)	0.0191 (6)	0.0180 (8)	-0.0036 (5)	0.0023 (5)	0.0016 (5)
C12	0.0214 (7)	0.0188 (6)	0.0206 (8)	-0.0030 (5)	0.0015 (5)	0.0031 (6)
C13	0.0238 (6)	0.0297 (7)	0.0198 (9)	-0.0003 (5)	-0.0001 (6)	0.0025 (6)
C14	0.0353 (8)	0.0360 (9)	0.0348 (11)	0.0131 (6)	-0.0077 (7)	-0.0022 (8)

Geometric parameters (Å, °)

O1—C2	1.3574 (17)	C5—C6	1.3850 (19)
O1—H1O1	0.81	C5—C12	1.397 (2)
O2—C13	1.2401 (17)	C5—H5A	0.93
O3—C7	1.3685 (16)	C6—C7	1.407 (2)
O3—C14	1.426 (2)	C6—H6A	0.93

N1—C10	1.3671 (18)	C7—C8	1.388 (2)
N1—C9	1.3899 (18)	C8—C9	1.4000 (17)
N1—H1N1	0.85 (2)	C8—H8A	0.93
C1—C2	1.382 (2)	C9—C12	1.402 (2)
C1—C10	1.3960 (18)	C10—C11	1.420 (2)
C1—H1A	0.93	C11—C12	1.4517 (18)
C2—C3	1.425 (2)	C13—H13A	0.93
C3—C4	1.4086 (19)	C14—H14A	0.96
C3—C13	1.438 (2)	C14—H14B	0.96
C4—C11	1.379 (2)	C14—H14C	0.96
C4—H4A	0.93		
C2—O1—H1O1	104.9	C8—C7—C6	121.71 (13)
C7—O3—C14	117.62 (13)	C7—C8—C9	116.58 (13)
C10—N1—C9	109.00 (13)	C7—C8—H8A	121.7
C10—N1—H1N1	124.3 (12)	C9—C8—H8A	121.7
C9—N1—H1N1	126.2 (12)	N1—C9—C8	128.06 (14)
C2—C1—C10	116.99 (14)	N1—C9—C12	109.20 (12)
C2—C1—H1A	121.5	C8—C9—C12	122.69 (13)
C10—C1—H1A	121.5	N1—C10—C1	128.02 (14)
O1—C2—C1	117.68 (14)	N1—C10—C11	109.13 (12)
O1—C2—C3	120.64 (13)	C1—C10—C11	122.84 (13)
C1—C2—C3	121.67 (13)	C4—C11—C10	119.28 (13)
C4—C3—C2	119.83 (13)	C4—C11—C12	134.53 (14)
C4—C3—C13	118.83 (14)	C10—C11—C12	106.18 (12)
C2—C3—C13	121.25 (13)	C5—C12—C9	119.41 (12)
C11—C4—C3	119.36 (14)	C5—C12—C11	134.10 (14)
C11—C4—H4A	120.3	C9—C12—C11	106.45 (12)
C3—C4—H4A	120.3	O2—C13—C3	124.75 (14)
C6—C5—C12	118.88 (14)	O2—C13—H13A	117.6
C6—C5—H5A	120.6	C3—C13—H13A	117.6
C12—C5—H5A	120.6	O3—C14—H14A	109.5
C5—C6—C7	120.71 (14)	O3—C14—H14B	109.5
C5—C6—H6A	119.6	H14A—C14—H14B	109.5
C7—C6—H6A	119.6	O3—C14—H14C	109.5
O3—C7—C8	123.77 (14)	H14A—C14—H14C	109.5
O3—C7—C6	114.52 (14)	H14B—C14—H14C	109.5
C10—C1—C2—O1	-179.79 (13)	C2—C1—C10—N1	179.92 (13)
C10—C1—C2—C3	0.13 (19)	C2—C1—C10—C11	-1.0 (2)
O1—C2—C3—C4	-179.74 (12)	C3—C4—C11—C10	-0.9 (2)
C1—C2—C3—C4	0.3 (2)	C3—C4—C11—C12	177.97 (14)
O1—C2—C3—C13	-3.2 (2)	N1—C10—C11—C4	-179.38 (12)
C1—C2—C3—C13	176.88 (12)	C1—C10—C11—C4	1.4 (2)
C2—C3—C4—C11	0.0 (2)	N1—C10—C11—C12	1.49 (15)
C13—C3—C4—C11	-176.59 (13)	C1—C10—C11—C12	-177.74 (12)
C12—C5—C6—C7	0.7 (2)	C6—C5—C12—C9	0.8 (2)
C14—O3—C7—C8	4.0 (2)	C6—C5—C12—C11	-176.30 (14)

C14—O3—C7—C6	-175.73 (14)	N1—C9—C12—C5	-178.86 (12)
C5—C6—C7—O3	178.13 (13)	C8—C9—C12—C5	-1.4 (2)
C5—C6—C7—C8	-1.7 (2)	N1—C9—C12—C11	-1.06 (15)
O3—C7—C8—C9	-178.74 (13)	C8—C9—C12—C11	176.40 (12)
C6—C7—C8—C9	1.0 (2)	C4—C11—C12—C5	-1.9 (3)
C10—N1—C9—C8	-175.26 (13)	C10—C11—C12—C5	177.08 (15)
C10—N1—C9—C12	2.04 (15)	C4—C11—C12—C9	-179.18 (15)
C7—C8—C9—N1	177.45 (14)	C10—C11—C12—C9	-0.25 (15)
C7—C8—C9—C12	0.49 (19)	C4—C3—C13—O2	176.58 (13)
C9—N1—C10—C1	176.99 (13)	C2—C3—C13—O2	0.0 (2)
C9—N1—C10—C11	-2.19 (15)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H1O1 \cdots O2	0.81	1.96	2.6453 (17)	143
O1—H1O1 \cdots N1 ⁱ	0.81	2.53	3.0457 (15)	123
N1—H1N1 \cdots O2 ⁱⁱ	0.85 (2)	2.10 (2)	2.941 (2)	172 (2)

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