

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

10-Hydroxy-2-azapentacyclo[10.8.0.-0²,10⁴,9¹⁵,20¹]icosa-1(12),4(9),5,7,13,-15(20),16,18-octaene-3,11-dione

Saeedeh Hashemian^{a*} and Behrouz Notash^b^aDepartment of Chemistry, Islamic Azad University, Yazd Branch, Yazd, Iran, and^bDepartment of Chemistry, Shahid Beheshti University, G. C., Evin, Tehran 1983963113, Iran

Correspondence e-mail: sa_hashemian@yahoo.com

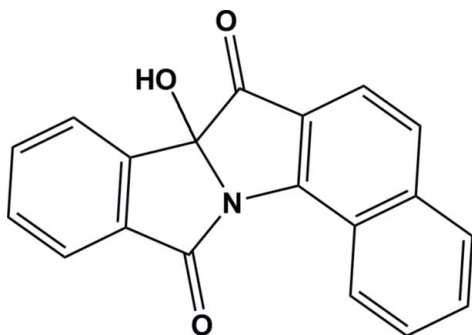
Received 8 February 2011; accepted 13 February 2011

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.044; wR factor = 0.136; data-to-parameter ratio = 17.1.

In the title compound, $\text{C}_{19}\text{H}_{11}\text{NO}_3$, the isoindolinone ring system is approximately planar with a maximum atomic deviation of 0.071 (1) Å and the five-membered ring of the dihydrobenzo[*g*]indol-3-one unit assumes an envelope conformation. The naphthalene ring system makes a dihedral angle of 39.47 (4)° with the mean plane of the isoindolinone system. Intermolecular $\text{O}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonding helps to stabilize the crystal structure.

Related literature

For applications of naphthylamines, see Valenti *et al.* (2006); Black *et al.* (1994).



Experimental

Crystal data

$\text{C}_{19}\text{H}_{11}\text{NO}_3$
 $M_r = 301.29$
 Triclinic, $P\bar{1}$
 $a = 7.3250$ (11) Å
 $b = 9.7916$ (16) Å
 $c = 10.4532$ (17) Å
 $\alpha = 70.401$ (13)°
 $\beta = 82.503$ (13)°
 $\gamma = 75.862$ (12)°
 $V = 683.94$ (19) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 298$ K
 $0.40 \times 0.30 \times 0.29$ mm

Data collection

Stoe IPDS II diffractometer
 7737 measured reflections
 3627 independent reflections
 3161 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.040$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.136$
 $S = 1.13$
 3627 reflections
 212 parameters
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.29$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.22$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O}2-\text{H}2\cdots\text{O}1^{\text{i}}$	0.96 (2)	1.85 (2)	2.7978 (15)	172.2 (17)
$\text{C}6-\text{H}6\cdots\text{O}3^{\text{ii}}$	0.93	2.52	3.1609 (18)	126

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

Data collection: *X-RED32* (Stoe & Cie, 2005); cell refinement: *X-AREA* (Stoe & Cie, 2005); data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

We are grateful to the Islamic Azad University, Yazd Branch, for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5159).

References

- Black, D. S. C., Bowyer, M. C., Condie, G. C., Craig, D. C. & Kumar, N. (1994). *Tetrahedron*, **50**, 10983–10994.
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Stoe & Cie (2005). *X-AREA* and *X-RED32*. Stoe & Cie, Darmstadt, Germany.
 Valenti, L. E., De Pauli, C. P. & Giacomelli, C. E. (2006). *J. Inorg. Biochem.* **100**, 192–200.

supporting information

Acta Cryst. (2011). E67, o680 [doi:10.1107/S1600536811005356]

10-Hydroxy-2-azapentacyclo- [10.8.0.0^{2,10}.0^{4,9}.0^{15,20}]icosa-1(12),4(9),5,7,13,15(20),16,18-octaene-3,11-dione

Saeedeh Hashemian and Behrouz Notash

S1. Comment

Naphthylamine is an aromatic amine which can be obtained from nitronaphthalene. Naphthylamines are used in the manufacture of dyes, condensation colors, and rubber and in the synthesis of a large number of chemical (Black *et al.* 1994; Valenti *et al.* 2006). Ninhydrin (2,2-dihydroxyindane-1,3-dione) is a chemical used to detect ammonia or primary and secondary amines. The carbon atom of a carbonyl bears a partial positive charge enhanced by neighboring electron withdrawing groups like carbonyl itself. So the central carbon of a 1,2,3-tricarbonyl compound is much more electrophilic than one in a simple ketone. Thus indane-1,2,3-trione reacts readily with nucleophiles, including water. Whereas for most carbonyl compounds, a carbonyl form is more stable than a product of water addition (hydrate), ninhydrin forms a stable hydrate of the central carbon because of the destabilizing effect of the adjacent carbonyl groups.

We report here the crystal structure of 7-hydroxy-7-*a*H-benzo[*g*] isoindolino[1,2-*α*]indole-7,10-dione. The title compound was prepared by the reaction of *α*-naphthylamine and ninhydrine in molar ratio of 1: 1 in acetone. The structure of title complex is shown in Fig. 1. There are intermolecular hydrogen bondings between O—H \cdots O and C—H \cdots O which play important role in the stabilization of crystalline network (Table 1 & Fig. 2).

S2. Experimental

The title compound was prepared by the reaction of *α*-naphthylamine and ninhydrine in molar ratio of 1: 1 in acetone. The mixture was stirred for about 2 h at room temperature. The resulting solution was kept in air. After slow evaporation, single crystals of the title compound suitable for an X-ray diffraction study formed at the bottom of the vessel after 2 weeks (m.p. 138 °C).

S3. Refinement

Hydroxyl H atom was found in a difference Fourier map and refined isotropically. Other H atoms were positioned geometrically and refined as riding atoms with C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

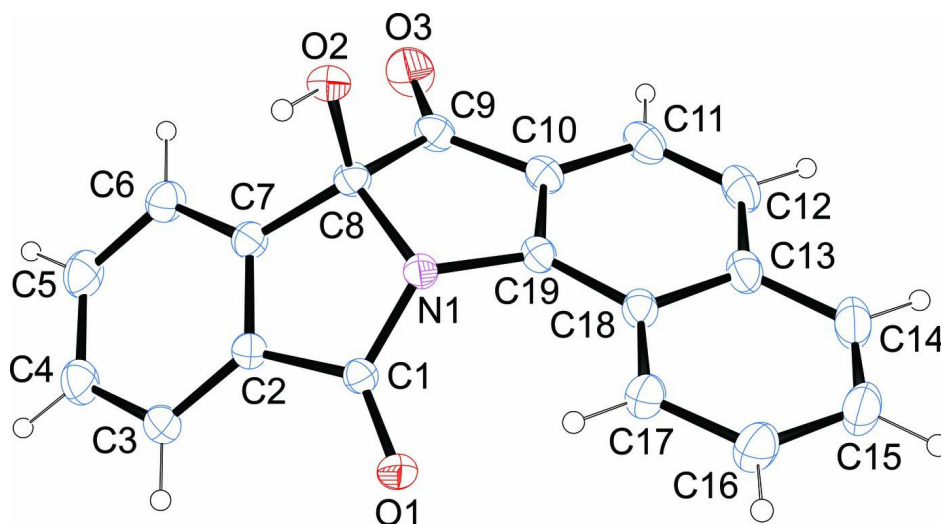


Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at 30% probability level.

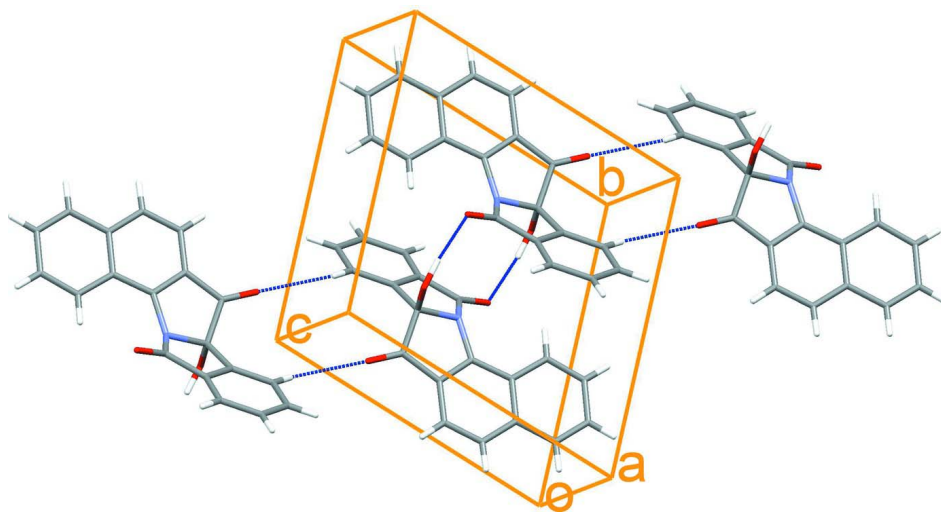


Figure 2

The packing diagram of the title compound. The intermolecular O—H...O and C—H...O hydrogen bonds are shown as blue dashed lines.

10-Hydroxy-2-azapentacyclo[10.8.0.0^{2,10}.0^{4,9}.0^{15,20}]icosa- 1(12),4(9),5,7,13,15 (20),16,18-octaene-3,11-dione

Crystal data

C₁₉H₁₁NO₃

M_r = 301.29

Triclinic, *P* $\bar{1}$

Hall symbol: -P 1

a = 7.3250 (11) Å

b = 9.7916 (16) Å

c = 10.4532 (17) Å

α = 70.401 (13)°

β = 82.503 (13)°

γ = 75.862 (12)°

V = 683.94 (19) Å³

Z = 2

F(000) = 312

D_x = 1.463 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 1011 reflections

θ = 2.3–25.0°

μ = 0.10 mm⁻¹

$T = 298$ K $0.40 \times 0.30 \times 0.29$ mm
 Block, colorless

Data collection

Stoe IPDS II diffractometer	3161 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.040$
Graphite monochromator	$\theta_{\text{max}} = 29.3^\circ$, $\theta_{\text{min}} = 2.3^\circ$
rotation method scans	$h = -10 \rightarrow 10$
7737 measured reflections	$k = -13 \rightarrow 13$
3627 independent reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.044$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.136$	$w = 1/[\sigma^2(F_o^2) + (0.072P)^2 + 0.1037P]$
$S = 1.13$	where $P = (F_o^2 + 2F_c^2)/3$
3627 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
212 parameters	$\Delta\rho_{\text{max}} = 0.29 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.22 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.60706 (15)	0.62937 (12)	0.49240 (11)	0.0298 (2)
C2	0.69780 (16)	0.61664 (13)	0.36032 (12)	0.0324 (2)
C3	0.87442 (18)	0.53663 (16)	0.33404 (14)	0.0410 (3)
H3	0.9488	0.4711	0.4042	0.049*
C4	0.9358 (2)	0.55825 (19)	0.19890 (16)	0.0494 (3)
H4	1.0540	0.5065	0.1779	0.059*
C5	0.8237 (2)	0.65604 (19)	0.09393 (15)	0.0502 (3)
H5	0.8694	0.6697	0.0042	0.060*
C6	0.6452 (2)	0.73333 (16)	0.12066 (13)	0.0425 (3)
H6	0.5690	0.7968	0.0507	0.051*
C7	0.58481 (16)	0.71222 (13)	0.25611 (12)	0.0332 (2)
C8	0.40104 (16)	0.77784 (12)	0.31604 (11)	0.0318 (2)
C9	0.33814 (17)	0.94765 (13)	0.28511 (13)	0.0374 (3)
C10	0.29102 (17)	0.97079 (13)	0.41761 (13)	0.0358 (3)

C11	0.19816 (19)	1.10289 (14)	0.44465 (16)	0.0441 (3)
H11	0.1640	1.1909	0.3742	0.053*
C12	0.15988 (19)	1.09807 (16)	0.57670 (17)	0.0469 (3)
H12	0.0981	1.1843	0.5963	0.056*
C13	0.21185 (17)	0.96457 (16)	0.68549 (15)	0.0413 (3)
C14	0.1701 (2)	0.9614 (2)	0.82289 (17)	0.0541 (4)
H14	0.1102	1.0485	0.8415	0.065*
C15	0.2156 (2)	0.8347 (2)	0.92698 (17)	0.0596 (4)
H15	0.1890	0.8359	1.0161	0.071*
C16	0.3028 (2)	0.7010 (2)	0.90168 (15)	0.0545 (4)
H16	0.3306	0.6140	0.9742	0.065*
C17	0.34726 (19)	0.69747 (16)	0.77121 (13)	0.0422 (3)
H17	0.4051	0.6082	0.7558	0.051*
C18	0.30558 (16)	0.82910 (14)	0.65996 (12)	0.0344 (2)
C19	0.34334 (15)	0.83897 (12)	0.52100 (12)	0.0310 (2)
O1	0.67384 (13)	0.57343 (10)	0.60386 (9)	0.0374 (2)
O2	0.24447 (12)	0.73626 (10)	0.28658 (9)	0.0376 (2)
H2	0.268 (3)	0.630 (2)	0.317 (2)	0.066 (6)*
O3	0.32112 (19)	1.03639 (12)	0.17284 (11)	0.0592 (3)
N1	0.43075 (13)	0.72309 (10)	0.46469 (9)	0.0303 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0310 (5)	0.0265 (5)	0.0314 (5)	-0.0044 (4)	-0.0037 (4)	-0.0091 (4)
C2	0.0318 (5)	0.0332 (5)	0.0334 (5)	-0.0057 (4)	-0.0021 (4)	-0.0128 (4)
C3	0.0329 (6)	0.0468 (7)	0.0462 (7)	-0.0022 (5)	-0.0040 (5)	-0.0221 (6)
C4	0.0366 (6)	0.0628 (9)	0.0545 (8)	-0.0073 (6)	0.0071 (6)	-0.0317 (7)
C5	0.0490 (8)	0.0656 (9)	0.0402 (7)	-0.0176 (7)	0.0120 (6)	-0.0237 (7)
C6	0.0460 (7)	0.0487 (7)	0.0309 (6)	-0.0114 (5)	0.0016 (5)	-0.0106 (5)
C7	0.0339 (5)	0.0342 (5)	0.0308 (5)	-0.0075 (4)	-0.0004 (4)	-0.0096 (4)
C8	0.0328 (5)	0.0321 (5)	0.0268 (5)	-0.0041 (4)	-0.0038 (4)	-0.0057 (4)
C9	0.0367 (6)	0.0319 (5)	0.0370 (6)	-0.0045 (4)	-0.0026 (4)	-0.0040 (4)
C10	0.0323 (5)	0.0302 (5)	0.0430 (6)	-0.0053 (4)	-0.0014 (4)	-0.0104 (5)
C11	0.0384 (6)	0.0293 (6)	0.0628 (8)	-0.0041 (5)	-0.0020 (6)	-0.0146 (5)
C12	0.0378 (6)	0.0398 (7)	0.0697 (9)	-0.0068 (5)	0.0044 (6)	-0.0297 (7)
C13	0.0310 (5)	0.0490 (7)	0.0540 (7)	-0.0105 (5)	0.0038 (5)	-0.0301 (6)
C14	0.0438 (7)	0.0725 (10)	0.0611 (9)	-0.0126 (7)	0.0084 (6)	-0.0447 (9)
C15	0.0510 (8)	0.0929 (13)	0.0467 (8)	-0.0148 (8)	0.0065 (6)	-0.0413 (9)
C16	0.0540 (8)	0.0735 (10)	0.0354 (7)	-0.0126 (7)	0.0017 (6)	-0.0188 (7)
C17	0.0428 (7)	0.0495 (7)	0.0346 (6)	-0.0087 (5)	0.0009 (5)	-0.0158 (5)
C18	0.0290 (5)	0.0408 (6)	0.0379 (6)	-0.0088 (4)	0.0016 (4)	-0.0184 (5)
C19	0.0278 (5)	0.0304 (5)	0.0353 (5)	-0.0060 (4)	0.0001 (4)	-0.0119 (4)
O1	0.0403 (5)	0.0356 (4)	0.0330 (4)	-0.0006 (3)	-0.0097 (3)	-0.0091 (3)
O2	0.0338 (4)	0.0389 (5)	0.0380 (4)	-0.0049 (3)	-0.0085 (3)	-0.0089 (4)
O3	0.0753 (8)	0.0408 (6)	0.0421 (5)	-0.0017 (5)	-0.0040 (5)	0.0047 (4)
N1	0.0325 (5)	0.0292 (4)	0.0265 (4)	-0.0019 (3)	-0.0028 (3)	-0.0080 (3)

Geometric parameters (Å, °)

C1—O1	1.2192 (14)	C10—C19	1.3826 (16)
C1—N1	1.3908 (14)	C10—C11	1.4075 (17)
C1—C2	1.4829 (16)	C11—C12	1.359 (2)
C2—C3	1.3855 (16)	C11—H11	0.9300
C2—C7	1.3858 (16)	C12—C13	1.421 (2)
C3—C4	1.387 (2)	C12—H12	0.9300
C3—H3	0.9300	C13—C14	1.420 (2)
C4—C5	1.393 (2)	C13—C18	1.4307 (17)
C4—H4	0.9300	C14—C15	1.349 (3)
C5—C6	1.387 (2)	C14—H14	0.9300
C5—H5	0.9300	C15—C16	1.404 (3)
C6—C7	1.3885 (17)	C15—H15	0.9300
C6—H6	0.9300	C16—C17	1.3704 (18)
C7—C8	1.4983 (16)	C16—H16	0.9300
C8—O2	1.4071 (14)	C17—C18	1.4148 (19)
C8—N1	1.4905 (14)	C17—H17	0.9300
C8—C9	1.5445 (16)	C18—C19	1.4177 (16)
C9—O3	1.2037 (16)	C19—N1	1.4278 (14)
C9—C10	1.4616 (18)	O2—H2	0.96 (2)
O1—C1—N1	125.83 (10)	C11—C10—C9	127.75 (12)
O1—C1—C2	127.52 (10)	C12—C11—C10	118.08 (13)
N1—C1—C2	106.62 (9)	C12—C11—H11	121.0
C3—C2—C7	121.55 (11)	C10—C11—H11	121.0
C3—C2—C1	128.81 (11)	C11—C12—C13	121.64 (12)
C7—C2—C1	109.36 (10)	C11—C12—H12	119.2
C2—C3—C4	117.33 (12)	C13—C12—H12	119.2
C2—C3—H3	121.3	C14—C13—C12	120.91 (13)
C4—C3—H3	121.3	C14—C13—C18	118.03 (14)
C3—C4—C5	121.23 (13)	C12—C13—C18	121.06 (12)
C3—C4—H4	119.4	C15—C14—C13	121.45 (14)
C5—C4—H4	119.4	C15—C14—H14	119.3
C6—C5—C4	121.25 (12)	C13—C14—H14	119.3
C6—C5—H5	119.4	C14—C15—C16	120.45 (14)
C4—C5—H5	119.4	C14—C15—H15	119.8
C5—C6—C7	117.34 (13)	C16—C15—H15	119.8
C5—C6—H6	121.3	C17—C16—C15	120.69 (16)
C7—C6—H6	121.3	C17—C16—H16	119.7
C2—C7—C6	121.28 (11)	C15—C16—H16	119.7
C2—C7—C8	109.15 (10)	C16—C17—C18	120.21 (14)
C6—C7—C8	129.57 (11)	C16—C17—H17	119.9
O2—C8—N1	111.82 (9)	C18—C17—H17	119.9
O2—C8—C7	113.83 (9)	C17—C18—C19	125.32 (11)
N1—C8—C7	103.29 (9)	C17—C18—C13	119.13 (12)
O2—C8—C9	104.10 (9)	C19—C18—C13	115.52 (12)
N1—C8—C9	103.13 (9)	C10—C19—C18	121.92 (11)

C7—C8—C9	120.25 (10)	C10—C19—N1	109.84 (10)
O3—C9—C10	129.58 (12)	C18—C19—N1	128.21 (10)
O3—C9—C8	124.77 (12)	C8—O2—H2	107.6 (12)
C10—C9—C8	105.45 (9)	C1—N1—C19	126.89 (9)
C19—C10—C11	121.77 (12)	C1—N1—C8	111.06 (9)
C19—C10—C9	110.43 (10)	C19—N1—C8	108.74 (9)
O1—C1—C2—C3	1.8 (2)	C11—C12—C13—C18	-0.5 (2)
N1—C1—C2—C3	179.64 (12)	C12—C13—C14—C15	178.73 (14)
O1—C1—C2—C7	-172.16 (12)	C18—C13—C14—C15	-0.5 (2)
N1—C1—C2—C7	5.70 (13)	C13—C14—C15—C16	-1.3 (2)
C7—C2—C3—C4	1.3 (2)	C14—C15—C16—C17	1.6 (3)
C1—C2—C3—C4	-171.95 (12)	C15—C16—C17—C18	0.0 (2)
C2—C3—C4—C5	-0.3 (2)	C16—C17—C18—C19	-179.45 (13)
C3—C4—C5—C6	-1.2 (2)	C16—C17—C18—C13	-1.7 (2)
C4—C5—C6—C7	1.6 (2)	C14—C13—C18—C17	2.00 (18)
C3—C2—C7—C6	-0.90 (19)	C12—C13—C18—C17	-177.24 (12)
C1—C2—C7—C6	173.56 (11)	C14—C13—C18—C19	179.92 (11)
C3—C2—C7—C8	177.95 (11)	C12—C13—C18—C19	0.68 (17)
C1—C2—C7—C8	-7.60 (13)	C11—C10—C19—C18	0.37 (18)
C5—C6—C7—C2	-0.6 (2)	C9—C10—C19—C18	-177.05 (10)
C5—C6—C7—C8	-179.17 (13)	C11—C10—C19—N1	178.75 (11)
C2—C7—C8—O2	-115.06 (11)	C9—C10—C19—N1	1.33 (14)
C6—C7—C8—O2	63.66 (17)	C17—C18—C19—C10	177.17 (12)
C2—C7—C8—N1	6.39 (12)	C13—C18—C19—C10	-0.61 (17)
C6—C7—C8—N1	-174.89 (13)	C17—C18—C19—N1	-0.9 (2)
C2—C7—C8—C9	120.48 (11)	C13—C18—C19—N1	-178.66 (10)
C6—C7—C8—C9	-60.81 (18)	O1—C1—N1—C19	40.41 (18)
O2—C8—C9—O3	-72.49 (16)	C2—C1—N1—C19	-137.50 (11)
N1—C8—C9—O3	170.63 (13)	O1—C1—N1—C8	176.42 (11)
C7—C8—C9—O3	56.47 (18)	C2—C1—N1—C8	-1.48 (12)
O2—C8—C9—C10	102.75 (10)	C10—C19—N1—C1	125.80 (12)
N1—C8—C9—C10	-14.13 (12)	C18—C19—N1—C1	-55.95 (17)
C7—C8—C9—C10	-128.29 (11)	C10—C19—N1—C8	-11.01 (13)
O3—C9—C10—C19	-176.69 (14)	C18—C19—N1—C8	167.23 (11)
C8—C9—C10—C19	8.38 (13)	O2—C8—N1—C1	119.96 (10)
O3—C9—C10—C11	6.1 (2)	C7—C8—N1—C1	-2.84 (12)
C8—C9—C10—C11	-168.84 (12)	C9—C8—N1—C1	-128.76 (10)
C19—C10—C11—C12	-0.18 (19)	O2—C8—N1—C19	-95.95 (11)
C9—C10—C11—C12	176.77 (12)	C7—C8—N1—C19	141.25 (9)
C10—C11—C12—C13	0.3 (2)	C9—C8—N1—C19	15.33 (11)
C11—C12—C13—C14	-179.76 (13)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O2—H2 \cdots O1 ⁱ	0.96 (2)	1.85 (2)	2.7978 (15)	172.2 (17)

C6—H6···O3 ⁱⁱ	0.93	2.52	3.1609 (18)	126
--------------------------	------	------	-------------	-----

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