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4-(1,3-Benzodioxol-5-yl)-2-oxo-1,2,5,6-tetrahydrobenzo[*h*]quinoline-3-carbonitrile

Abdullah M. Asiri,[a] Hassan M. Faidallah,[a] Abdulrahman O. Al-Youbi,[a] Khalid A. Alamry[a] and Seik Weng Ng[b,a]*

[a]Chemistry Department, Faculty of Science, King Abdulaziz University, PO Box 80203 Jeddah, Saudi Arabia, and [b]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

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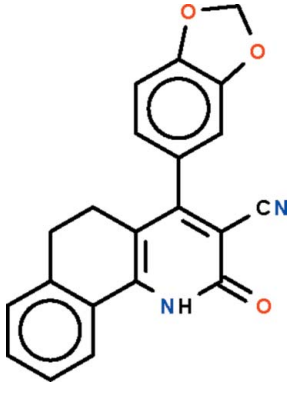
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.100; data-to-parameter ratio = 13.6.

In the molecule of the title compound, $C_{21}H_{14}N_2O_3$, the tetrahydrobenzo[*h*]quinoline fused-ring system is buckled owing to the ethylene $-CH_2CH_2-$ fragment, the benzene ring and the pyridine ring being twisted by 24.3 (1)°. The ring of the benzodioxol system is bent away from the pyridine ring by 61.4 (1)° in order to avoid crowding the cyanide substituent. Two molecules are linked by a pair of N—H···O hydrogen bonds to form a centrosymmetric dimer.

Related literature

For background to the anticancer properties of this class of compounds, see: Rostom *et al.* (2011).



Experimental

Crystal data

$C_{21}H_{14}N_2O_3$
M_r = 342.34
Monoclinic, $P2_1/n$
a = 7.6586 (3) Å
b = 16.5858 (5) Å
c = 13.3220 (6) Å
β = 104.164 (4)°
V = 1640.77 (11) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.77 mm^{-1}
T = 100 K
0.30 × 0.20 × 0.05 mm

Data collection

Agilent SuperNova Dual diffractometer with Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.802, T_{max} = 0.963
6078 measured reflections
3241 independent reflections
2962 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.100
S = 1.02
3241 reflections
239 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.93 (2)	1.85 (2)	2.778 (1)	175 (2)

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

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

The authors thank King Abdulaziz University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5293).

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supporting information

Acta Cryst. (2011). E**67**, o2471 [doi:10.1107/S1600536811033903]

4-(1,3-Benzodioxol-5-yl)-2-oxo-1,2,5,6-tetrahydrobenzo[*h*]quinoline-3-carbonitrile

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S1. Comment

The compound (Scheme I) belongs to a series of cyano-pyridinones that have been evaluated for their anticancer properties (Rostom *et al.*, 2011). The tetrahydrobenzo[*h*]quinoline fused-ring system is buckled owing to the ethylene –CH_2CH_2– fragment, the benzene ring and the pyridine ring being twisted by 24.3 (1)°. The 4-subsituted aromatic ring is bent away from the pyridine ring by 61.4 (1) ° in order to avoid crowding the cyanide substituent (Fig. 1). Two molecules are linked by an N—H···O hydrogen bonds to form a centrosymmetric dimer (Table 1).

S2. Experimental

A mixture of piperonaldehyde (1.50 g, 10 mmol), 1-tetralone (1.46 g, 10 mmol), ethyl cyanoacetate (1.1 g, 10 mmol) and ammonium acetate (6.2 g, 80 mmol) in absolute ethanol (50 ml) was refluxed for 6 h. The reaction mixture was allowed to cool, and the yellow precipitate that formed was filtered, washed with water, dried and recrystallized from ethanol; m.p. 593–595 K.

S3. Refinement

Carbon- and nitrogen-bound H atoms were placed in calculated positions [C—H 0.95 to 0.99 Å, U_{iso}(H) = 1.2U_{eq}(C)] and were included in the refinement in the riding model approximation.

The amino H atom was located in a difference Fourier map and was freely refined.

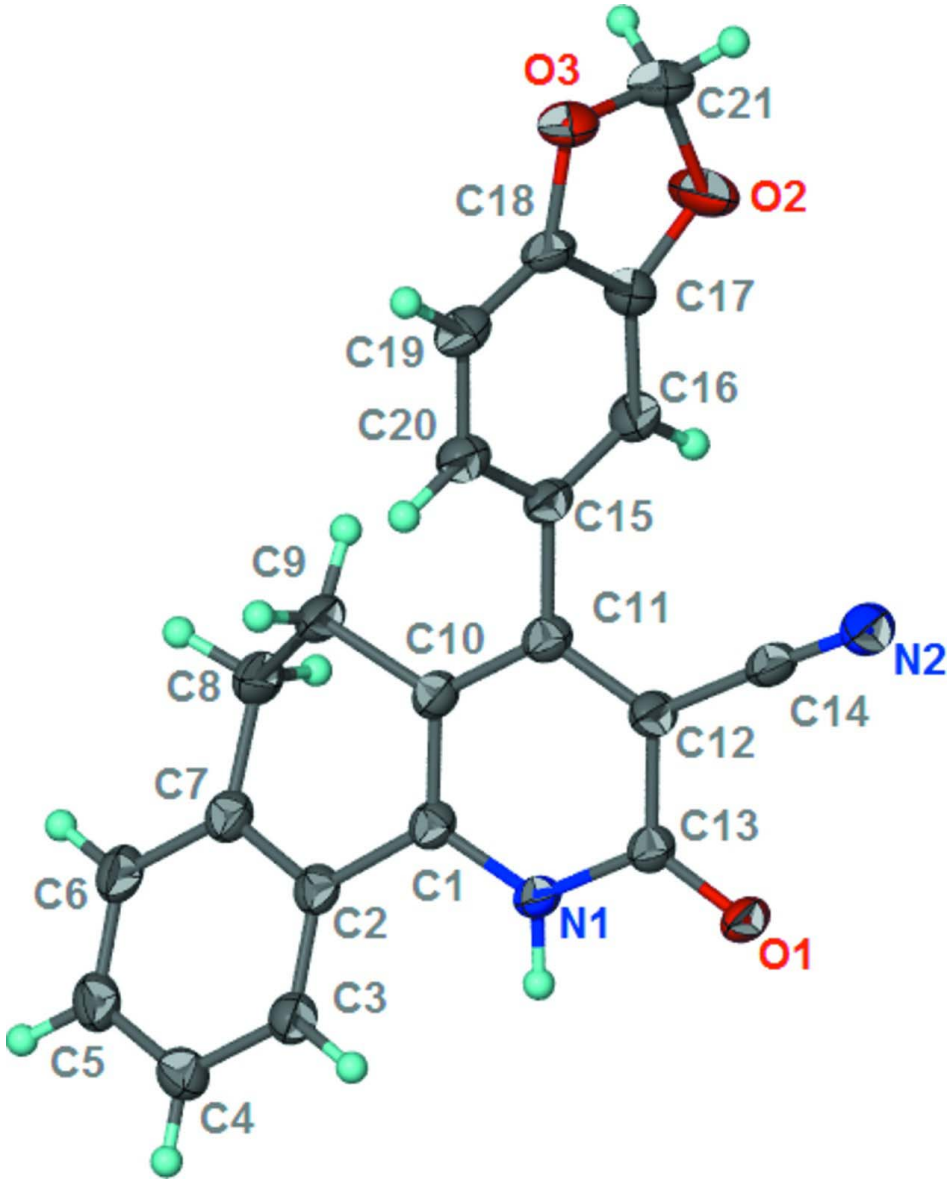

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{21}H_{14}N_2O_3$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

4-(1,3-Benzodioxol-5-yl)-2-oxo-1,2,5,6-tetrahydrobenzo[*h*]quinoline- 3-carbonitrile

Crystal data

$C_{21}H_{14}N_2O_3$
M_r = 342.34
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 7.6586 (3) Å
b = 16.5858 (5) Å
c = 13.3220 (6) Å
β = 104.164 (4)°
V = 1640.77 (11) Å^3
Z = 4
F(000) = 712
D_x = 1.386 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 3826 reflections
θ = 3.4–74.1°
μ = 0.77 mm^{-1}

T = 100 K
Prism, yellow

0.30 × 0.20 × 0.05 mm

Data collection

Agilent SuperNova Dual diffractometer with Atlas detector
Radiation source: SuperNova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)

T_{min} = 0.802, T_{max} = 0.963
6078 measured reflections
3241 independent reflections
2962 reflections with $I > 2\sigma(I)$
R_{int} = 0.014
θ_{max} = 74.3°, θ_{min} = 4.3°
h = −9→9
k = −20→18
l = −12→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.100
S = 1.02
3241 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0584P)^2 + 0.3916P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	0.70432 (10)	0.53335 (5)	0.56250 (6)	0.0240 (2)
O2	1.17132 (13)	0.94538 (6)	0.60416 (10)	0.0444 (3)
O3	1.07114 (12)	1.02058 (5)	0.72509 (8)	0.0325 (2)
N1	0.46189 (12)	0.61383 (6)	0.49726 (7)	0.0200 (2)
H1	0.400 (2)	0.5663 (10)	0.4755 (12)	0.036 (4)*
N2	1.07002 (14)	0.65042 (6)	0.67925 (9)	0.0305 (3)
C1	0.38331 (15)	0.68751 (7)	0.47365 (8)	0.0197 (2)
C2	0.19123 (15)	0.69051 (7)	0.41849 (9)	0.0210 (2)
C3	0.07235 (15)	0.62711 (7)	0.42051 (9)	0.0233 (2)
H3	0.1138	0.5799	0.4594	0.028*
C4	−0.10680 (16)	0.63304 (7)	0.36557 (10)	0.0270 (3)
H4	−0.1877	0.5899	0.3671	0.032*
C5	−0.16769 (16)	0.70217 (8)	0.30833 (10)	0.0288 (3)
H5	−0.2899	0.7060	0.2705	0.035*
C6	−0.05002 (16)	0.76546 (7)	0.30652 (9)	0.0268 (3)
H6	−0.0922	0.8124	0.2672	0.032*
C7	0.12963 (16)	0.76085 (7)	0.36181 (9)	0.0232 (2)
C8	0.26177 (16)	0.82766 (7)	0.36083 (10)	0.0282 (3)
H8A	0.1958	0.8789	0.3413	0.034*
H8B	0.3302	0.8158	0.3084	0.034*
C9	0.39269 (16)	0.83692 (7)	0.46727 (10)	0.0271 (3)
H9A	0.4852	0.8778	0.4636	0.032*

H9B	0.3266	0.8555	0.5182	0.032*
C10	0.48223 (15)	0.75711 (7)	0.50141 (9)	0.0210 (2)
C11	0.66347 (15)	0.75062 (7)	0.55957 (9)	0.0212 (2)
C12	0.74037 (15)	0.67503 (7)	0.57985 (9)	0.0212 (2)
C13	0.63948 (15)	0.60256 (7)	0.54712 (8)	0.0201 (2)
C14	0.92402 (15)	0.66289 (7)	0.63508 (9)	0.0228 (3)
C15	0.76691 (15)	0.82441 (6)	0.60147 (9)	0.0213 (2)
C16	0.92690 (16)	0.84474 (7)	0.57344 (10)	0.0252 (3)
H16	0.9722	0.8130	0.5261	0.030*
C17	1.01415 (15)	0.91297 (7)	0.61812 (10)	0.0262 (3)
C18	0.95379 (16)	0.95848 (7)	0.68946 (9)	0.0238 (2)
C19	0.79788 (16)	0.94009 (7)	0.71780 (9)	0.0250 (3)
H19	0.7560	0.9718	0.7665	0.030*
C20	0.70363 (16)	0.87202 (7)	0.67114 (9)	0.0239 (2)
H20	0.5935	0.8579	0.6875	0.029*
C21	1.20431 (18)	1.01643 (8)	0.66633 (11)	0.0346 (3)
H21A	1.1967	1.0647	0.6218	0.041*
H21B	1.3263	1.0143	0.7134	0.041*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0226 (4)	0.0149 (4)	0.0296 (4)	0.0014 (3)	−0.0028 (3)	0.0014 (3)
O2	0.0336 (5)	0.0346 (5)	0.0716 (8)	−0.0158 (4)	0.0256 (5)	−0.0236 (5)
O3	0.0289 (4)	0.0224 (4)	0.0442 (5)	−0.0065 (3)	0.0051 (4)	−0.0121 (4)
N1	0.0196 (5)	0.0147 (4)	0.0226 (5)	−0.0005 (4)	−0.0007 (4)	−0.0004 (4)
N2	0.0250 (5)	0.0230 (5)	0.0375 (6)	0.0003 (4)	−0.0040 (4)	−0.0018 (4)
C1	0.0213 (5)	0.0186 (5)	0.0181 (5)	0.0018 (4)	0.0027 (4)	−0.0002 (4)
C2	0.0204 (5)	0.0203 (5)	0.0205 (5)	0.0026 (4)	0.0018 (4)	−0.0029 (4)
C3	0.0224 (5)	0.0219 (6)	0.0240 (6)	0.0024 (4)	0.0027 (4)	−0.0020 (4)
C4	0.0216 (5)	0.0269 (6)	0.0306 (6)	−0.0011 (5)	0.0028 (5)	−0.0049 (5)
C5	0.0209 (5)	0.0296 (6)	0.0316 (6)	0.0046 (5)	−0.0019 (5)	−0.0058 (5)
C6	0.0250 (6)	0.0229 (6)	0.0281 (6)	0.0067 (5)	−0.0022 (5)	−0.0018 (5)
C7	0.0234 (5)	0.0209 (5)	0.0235 (5)	0.0030 (4)	0.0021 (4)	−0.0032 (4)
C8	0.0254 (6)	0.0209 (6)	0.0338 (7)	0.0033 (4)	−0.0013 (5)	0.0056 (5)
C9	0.0245 (6)	0.0164 (5)	0.0362 (7)	0.0022 (4)	−0.0005 (5)	−0.0007 (5)
C10	0.0218 (5)	0.0174 (5)	0.0226 (5)	0.0020 (4)	0.0031 (4)	−0.0010 (4)
C11	0.0233 (5)	0.0181 (5)	0.0211 (5)	0.0002 (4)	0.0031 (4)	−0.0015 (4)
C12	0.0200 (5)	0.0189 (5)	0.0219 (5)	−0.0004 (4)	−0.0003 (4)	0.0000 (4)
C13	0.0210 (5)	0.0181 (5)	0.0190 (5)	0.0012 (4)	0.0007 (4)	0.0010 (4)
C14	0.0250 (6)	0.0150 (5)	0.0260 (6)	−0.0006 (4)	0.0016 (5)	−0.0020 (4)
C15	0.0226 (5)	0.0157 (5)	0.0222 (5)	0.0007 (4)	−0.0010 (4)	−0.0002 (4)
C16	0.0243 (5)	0.0214 (6)	0.0291 (6)	0.0003 (4)	0.0050 (5)	−0.0060 (5)
C17	0.0219 (5)	0.0217 (6)	0.0343 (6)	−0.0022 (4)	0.0054 (5)	−0.0026 (5)
C18	0.0267 (6)	0.0147 (5)	0.0258 (6)	−0.0017 (4)	−0.0015 (4)	−0.0020 (4)
C19	0.0315 (6)	0.0195 (5)	0.0232 (6)	0.0009 (4)	0.0051 (5)	−0.0026 (4)
C20	0.0266 (6)	0.0189 (5)	0.0255 (6)	−0.0012 (4)	0.0050 (5)	0.0000 (4)
C21	0.0342 (7)	0.0237 (6)	0.0449 (8)	−0.0085 (5)	0.0081 (6)	−0.0066 (6)

Geometric parameters (Å, º)

O1—C13	1.2477 (14)	C8—C9	1.5309 (17)
O2—C17	1.3718 (15)	C8—H8A	0.9900
O2—C21	1.4269 (16)	C8—H8B	0.9900
O3—C18	1.3730 (14)	C9—C10	1.5088 (15)
O3—C21	1.4312 (17)	C9—H9A	0.9900
N1—C1	1.3645 (14)	C9—H9B	0.9900
N1—C13	1.3725 (14)	C10—C11	1.4185 (15)
N1—H1	0.931 (17)	C11—C12	1.3834 (15)
N2—C14	1.1477 (15)	C11—C15	1.4902 (15)
C1—C10	1.3807 (15)	C12—C14	1.4331 (15)
C1—C2	1.4756 (15)	C12—C13	1.4377 (15)
C2—C3	1.3955 (16)	C15—C20	1.3928 (16)
C2—C7	1.4068 (16)	C15—C16	1.4065 (17)
C3—C4	1.3916 (16)	C16—C17	1.3736 (16)
C3—H3	0.9500	C16—H16	0.9500
C4—C5	1.3928 (18)	C17—C18	1.3790 (17)
C4—H4	0.9500	C18—C19	1.3718 (17)
C5—C6	1.3875 (18)	C19—C20	1.4010 (16)
C5—H5	0.9500	C19—H19	0.9500
C6—C7	1.3948 (15)	C20—H20	0.9500
C6—H6	0.9500	C21—H21A	0.9900
C7—C8	1.5029 (17)	C21—H21B	0.9900
C17—O2—C21	106.35 (10)	C1—C10—C11	118.83 (10)
C18—O3—C21	105.58 (9)	C1—C10—C9	118.27 (10)
C1—N1—C13	124.23 (9)	C11—C10—C9	122.89 (10)
C1—N1—H1	121.6 (10)	C12—C11—C10	119.24 (10)
C13—N1—H1	114.0 (10)	C12—C11—C15	120.61 (10)
N1—C1—C10	120.33 (10)	C10—C11—C15	120.11 (10)
N1—C1—C2	118.34 (10)	C11—C12—C14	123.03 (10)
C10—C1—C2	121.33 (10)	C11—C12—C13	121.79 (10)
C3—C2—C7	120.03 (10)	C14—C12—C13	115.17 (10)
C3—C2—C1	122.72 (10)	O1—C13—N1	120.82 (10)
C7—C2—C1	117.25 (10)	O1—C13—C12	123.82 (10)
C4—C3—C2	120.04 (11)	N1—C13—C12	115.36 (9)
C4—C3—H3	120.0	N2—C14—C12	177.69 (12)
C2—C3—H3	120.0	C20—C15—C16	120.58 (10)
C5—C4—C3	120.07 (11)	C20—C15—C11	118.39 (10)
C5—C4—H4	120.0	C16—C15—C11	121.02 (10)
C3—C4—H4	120.0	C17—C16—C15	116.46 (11)
C6—C5—C4	120.03 (11)	C17—C16—H16	121.8
C6—C5—H5	120.0	C15—C16—H16	121.8
C4—C5—H5	120.0	O2—C17—C16	127.88 (11)
C5—C6—C7	120.68 (11)	O2—C17—C18	109.40 (10)
C5—C6—H6	119.7	C16—C17—C18	122.68 (11)
C7—C6—H6	119.7	O3—C18—C19	127.72 (11)

C6—C7—C2	119.13 (11)	O3—C18—C17	110.33 (11)
C6—C7—C8	122.30 (10)	C19—C18—C17	121.95 (11)
C2—C7—C8	118.55 (10)	C18—C19—C20	116.47 (11)
C7—C8—C9	110.84 (10)	C18—C19—H19	121.8
C7—C8—H8A	109.5	C20—C19—H19	121.8
C9—C8—H8A	109.5	C15—C20—C19	121.81 (11)
C7—C8—H8B	109.5	C15—C20—H20	119.1
C9—C8—H8B	109.5	C19—C20—H20	119.1
H8A—C8—H8B	108.1	O2—C21—O3	107.97 (10)
C10—C9—C8	109.72 (10)	O2—C21—H21A	110.1
C10—C9—H9A	109.7	O3—C21—H21A	110.1
C8—C9—H9A	109.7	O2—C21—H21B	110.1
C10—C9—H9B	109.7	O3—C21—H21B	110.1
C8—C9—H9B	109.7	H21A—C21—H21B	108.4
H9A—C9—H9B	108.2		

C13—N1—C1—C10	−1.05 (17)	C10—C11—C12—C13	−2.52 (17)
C13—N1—C1—C2	179.18 (10)	C15—C11—C12—C13	175.21 (10)
N1—C1—C2—C3	22.74 (16)	C1—N1—C13—O1	−177.03 (10)
C10—C1—C2—C3	−157.03 (11)	C1—N1—C13—C12	3.52 (16)
N1—C1—C2—C7	−156.96 (10)	C11—C12—C13—O1	178.92 (11)
C10—C1—C2—C7	23.27 (16)	C14—C12—C13—O1	−1.53 (17)
C7—C2—C3—C4	0.69 (17)	C11—C12—C13—N1	−1.66 (16)
C1—C2—C3—C4	−179.00 (10)	C14—C12—C13—N1	177.90 (10)
C2—C3—C4—C5	0.11 (18)	C12—C11—C15—C20	−116.89 (13)
C3—C4—C5—C6	−0.38 (19)	C10—C11—C15—C20	60.82 (15)
C4—C5—C6—C7	−0.15 (19)	C12—C11—C15—C16	61.65 (16)
C5—C6—C7—C2	0.94 (17)	C10—C11—C15—C16	−120.64 (12)
C5—C6—C7—C8	179.27 (12)	C20—C15—C16—C17	−0.08 (17)
C3—C2—C7—C6	−1.21 (17)	C11—C15—C16—C17	−178.59 (11)
C1—C2—C7—C6	178.50 (10)	C21—O2—C17—C16	179.29 (13)
C3—C2—C7—C8	−179.60 (11)	C21—O2—C17—C18	−2.82 (15)
C1—C2—C7—C8	0.11 (16)	C15—C16—C17—O2	179.76 (12)
C6—C7—C8—C9	143.06 (11)	C15—C16—C17—C18	2.13 (18)
C2—C7—C8—C9	−38.60 (15)	C21—O3—C18—C19	−176.17 (12)
C7—C8—C9—C10	54.40 (14)	C21—O3—C18—C17	4.47 (13)
N1—C1—C10—C11	−3.38 (17)	O2—C17—C18—O3	−1.08 (14)
C2—C1—C10—C11	176.38 (10)	C16—C17—C18—O3	176.94 (11)
N1—C1—C10—C9	175.95 (10)	O2—C17—C18—C19	179.52 (11)
C2—C1—C10—C9	−4.29 (16)	C16—C17—C18—C19	−2.46 (19)
C8—C9—C10—C1	−34.42 (15)	O3—C18—C19—C20	−178.70 (11)
C8—C9—C10—C11	144.89 (11)	C17—C18—C19—C20	0.59 (17)
C1—C10—C11—C12	5.05 (17)	C16—C15—C20—C19	−1.71 (17)
C9—C10—C11—C12	−174.25 (11)	C11—C15—C20—C19	176.83 (10)
C1—C10—C11—C15	−172.70 (10)	C18—C19—C20—C15	1.44 (17)
C9—C10—C11—C15	8.01 (17)	C17—O2—C21—O3	5.54 (15)
C10—C11—C12—C14	177.96 (10)	C18—O3—C21—O2	−6.13 (14)
C15—C11—C12—C14	−4.31 (18)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.93 (2)	1.85 (2)	2.778 (1)	175 (2)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.