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

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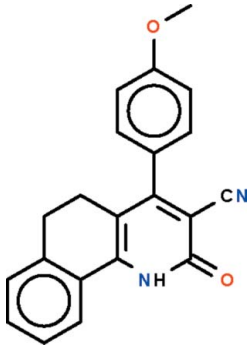
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.035; wR factor = 0.096; data-to-parameter ratio = 14.0.

In the molecule of the title compound, $C_{21}H_{16}N_2O_2$, 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 19.7 (1)°. The 4-substituted aromatic ring is bent away from the pyridine ring by 50.3 (1)° in order to avoid crowding the cyanide substituent. In the crystal, 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

$C_{21}H_{16}N_2O_2$
M_r = 328.36
Monoclinic, $P2_1/c$
a = 14.2016 (2) Å
b = 14.4725 (2) Å
c = 7.9935 (1) Å
β = 96.017 (1)°
V = 1633.87 (4) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.70 mm^{-1}
T = 100 K
0.30 × 0.25 × 0.20 mm

Data collection

Agilent SuperNova Dual diffractometer with Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.818, T_{max} = 0.873
6187 measured reflections
3211 independent reflections
3011 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.096
S = 1.03
3211 reflections
230 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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}	0.90 (2)	1.94 (2)	2.823 (1)	166 (1)

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: XU5292).

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supplementary materials

Acta Cryst. (2011). E**67**, o2470 [**doi:10.1107/S1600536811033897**]

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

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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 19.7 (1)°. The 4-subsituted aromatic ring is bent away from the pyridine ring by 50.3 (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).

Experimental

A mixture of *p*-anisaldehyde (1.36 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. 587–589 K.

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.

Figures

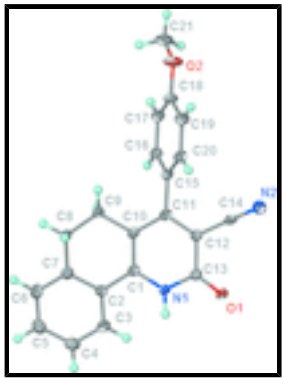


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $C_{21}H_{16}N_2O_2$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

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

Crystal data

$C_{21}H_{16}N_2O_2$	$F(000) = 688$
$M_r = 328.36$	$D_x = 1.335$ Mg m^{-3}
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Hall symbol: -P 2ybc	Cell parameters from 4172 reflections
$a = 14.2016$ (2) Å	$\theta = 3.1–74.1°$
$b = 14.4725$ (2) Å	$\mu = 0.70$ mm^{-1}
$c = 7.9935$ (1) Å	$T = 100$ K
$\beta = 96.017$ (1)°	Prism, yellow
$V = 1633.87$ (4) Å^3	0.30 × 0.25 × 0.20 mm
$Z = 4$	

Data collection

Agilent SuperNova Dual diffractometer with Atlas detector	3211 independent reflections
Radiation source: SuperNova (Cu) X-ray Source	3011 reflections with $I > 2\sigma(I)$
mirror	$R_{int} = 0.014$
Detector resolution: 10.4041 pixels mm^{-1}	$\theta_{max} = 74.2°$, $\theta_{min} = 4.4°$
ω scans	$h = -17\rightarrow14$
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)	$k = -9\rightarrow17$
$T_{min} = 0.818$, $T_{max} = 0.873$	$l = -9\rightarrow9$
6187 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.035$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.096$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.0566P)^2 + 0.481P]$ where $P = (F_o^2 + 2F_c^2)/3$
3211 reflections	$(\Delta/\sigma)_{max} = 0.001$
230 parameters	$\Delta\rho_{max} = 0.21$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.21$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}

O1	0.38937 (5)	0.48227 (6)	0.53692 (9)	0.01885 (19)
O2	−0.13689 (6)	0.71903 (6)	0.05350 (12)	0.0263 (2)
N1	0.42821 (6)	0.55604 (6)	0.30260 (11)	0.0154 (2)
H1	0.4886 (11)	0.5401 (11)	0.3377 (19)	0.026 (4)*
N2	0.15375 (7)	0.53084 (7)	0.58833 (12)	0.0215 (2)
C1	0.40584 (8)	0.60068 (7)	0.15327 (13)	0.0150 (2)
C2	0.48272 (8)	0.62138 (7)	0.04885 (13)	0.0159 (2)
C3	0.57808 (8)	0.62155 (8)	0.11363 (14)	0.0186 (2)
H3	0.5951	0.6082	0.2292	0.022*
C4	0.64792 (8)	0.64118 (8)	0.00983 (15)	0.0218 (2)
H4	0.7127	0.6404	0.0540	0.026*
C5	0.62308 (9)	0.66197 (8)	−0.15877 (15)	0.0228 (3)
H5	0.6708	0.6763	−0.2294	0.027*
C6	0.52862 (8)	0.66177 (8)	−0.22408 (14)	0.0208 (2)
H6	0.5122	0.6759	−0.3395	0.025*
C7	0.45770 (8)	0.64121 (7)	−0.12269 (13)	0.0168 (2)
C8	0.35509 (8)	0.63521 (8)	−0.19115 (13)	0.0184 (2)
H8A	0.3387	0.5700	−0.2181	0.022*
H8B	0.3447	0.6714	−0.2965	0.022*
C9	0.29074 (8)	0.67172 (8)	−0.06574 (13)	0.0178 (2)
H9A	0.2994	0.7393	−0.0531	0.021*
H9B	0.2239	0.6599	−0.1085	0.021*
C10	0.31283 (7)	0.62548 (7)	0.10368 (13)	0.0151 (2)
C11	0.24237 (7)	0.60637 (7)	0.21242 (13)	0.0150 (2)
C12	0.26806 (7)	0.55905 (7)	0.36226 (13)	0.0149 (2)
C13	0.36372 (7)	0.52863 (7)	0.40914 (13)	0.0151 (2)
C14	0.20310 (7)	0.54250 (7)	0.48475 (13)	0.0159 (2)
C15	0.14293 (7)	0.63712 (7)	0.16970 (13)	0.0153 (2)
C16	0.12282 (8)	0.72862 (7)	0.12434 (13)	0.0161 (2)
H16	0.1736	0.7709	0.1196	0.019*
C17	0.03069 (8)	0.75948 (8)	0.08598 (14)	0.0177 (2)
H17	0.0184	0.8223	0.0569	0.021*
C18	−0.04357 (7)	0.69700 (8)	0.09073 (14)	0.0181 (2)
C19	−0.02511 (8)	0.60501 (8)	0.13544 (14)	0.0193 (2)
H19	−0.0759	0.5626	0.1386	0.023*
C20	0.06719 (8)	0.57570 (8)	0.17509 (13)	0.0174 (2)
H20	0.0793	0.5132	0.2063	0.021*
C21	−0.15928 (8)	0.81389 (9)	0.01842 (17)	0.0261 (3)
H21A	−0.2279	0.8207	−0.0075	0.039*
H21B	−0.1275	0.8344	−0.0781	0.039*
H21C	−0.1378	0.8516	0.1169	0.039*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0173 (4)	0.0238 (4)	0.0149 (4)	0.0045 (3)	−0.0007 (3)	0.0043 (3)
O2	0.0117 (4)	0.0218 (4)	0.0445 (5)	0.0017 (3)	−0.0010 (3)	0.0067 (4)
N1	0.0124 (4)	0.0186 (5)	0.0147 (4)	0.0027 (3)	−0.0015 (3)	0.0015 (3)

N2	0.0212 (5)	0.0219 (5)	0.0217 (5)	0.0001 (4)	0.0034 (4)	0.0016 (4)
C1	0.0168 (5)	0.0133 (5)	0.0141 (5)	0.0009 (4)	−0.0014 (4)	−0.0011 (4)
C2	0.0160 (5)	0.0136 (5)	0.0178 (5)	0.0014 (4)	0.0004 (4)	0.0001 (4)
C3	0.0179 (5)	0.0175 (5)	0.0199 (5)	0.0004 (4)	−0.0005 (4)	0.0024 (4)
C4	0.0157 (5)	0.0212 (6)	0.0281 (6)	−0.0009 (4)	0.0009 (4)	0.0022 (5)
C5	0.0221 (6)	0.0220 (6)	0.0254 (6)	−0.0017 (4)	0.0079 (4)	0.0020 (4)
C6	0.0252 (6)	0.0194 (5)	0.0181 (5)	0.0008 (4)	0.0032 (4)	0.0019 (4)
C7	0.0191 (5)	0.0143 (5)	0.0167 (5)	0.0017 (4)	0.0009 (4)	−0.0002 (4)
C8	0.0189 (5)	0.0224 (5)	0.0134 (5)	0.0023 (4)	−0.0009 (4)	0.0013 (4)
C9	0.0173 (5)	0.0208 (5)	0.0148 (5)	0.0033 (4)	−0.0004 (4)	0.0019 (4)
C10	0.0155 (5)	0.0151 (5)	0.0142 (5)	0.0011 (4)	−0.0013 (4)	−0.0006 (4)
C11	0.0157 (5)	0.0128 (5)	0.0156 (5)	0.0001 (4)	−0.0023 (4)	−0.0025 (4)
C12	0.0144 (5)	0.0147 (5)	0.0152 (5)	0.0005 (4)	−0.0003 (4)	−0.0010 (4)
C13	0.0162 (5)	0.0151 (5)	0.0138 (5)	0.0015 (4)	−0.0002 (4)	−0.0010 (4)
C14	0.0153 (5)	0.0141 (5)	0.0172 (5)	0.0014 (4)	−0.0038 (4)	−0.0003 (4)
C15	0.0146 (5)	0.0180 (5)	0.0128 (5)	0.0012 (4)	−0.0012 (4)	−0.0007 (4)
C16	0.0139 (5)	0.0175 (5)	0.0165 (5)	−0.0018 (4)	−0.0006 (4)	−0.0004 (4)
C17	0.0172 (5)	0.0155 (5)	0.0199 (5)	0.0009 (4)	−0.0003 (4)	0.0015 (4)
C18	0.0119 (5)	0.0206 (6)	0.0212 (5)	0.0016 (4)	−0.0013 (4)	0.0011 (4)
C19	0.0147 (5)	0.0186 (5)	0.0240 (6)	−0.0030 (4)	−0.0003 (4)	0.0006 (4)
C20	0.0179 (5)	0.0153 (5)	0.0183 (5)	0.0004 (4)	−0.0011 (4)	0.0008 (4)
C21	0.0168 (5)	0.0229 (6)	0.0383 (7)	0.0060 (5)	0.0012 (5)	0.0064 (5)

Geometric parameters (Å, °)

O1—C13	1.2443 (13)	C8—H8B	0.9900
O2—C18	1.3653 (13)	C9—C10	1.5137 (14)
O2—C21	1.4304 (14)	C9—H9A	0.9900
N1—C1	1.3652 (13)	C9—H9B	0.9900
N1—C13	1.3729 (14)	C10—C11	1.4197 (15)
N1—H1	0.904 (16)	C11—C12	1.3947 (15)
N2—C14	1.1519 (15)	C11—C15	1.4859 (14)
C1—C10	1.3859 (15)	C12—C14	1.4341 (15)
C1—C2	1.4729 (15)	C12—C13	1.4399 (14)
C2—C3	1.3984 (15)	C15—C16	1.3947 (15)
C2—C7	1.4095 (15)	C15—C20	1.3994 (15)
C3—C4	1.3875 (16)	C16—C17	1.3860 (15)
C3—H3	0.9500	C16—H16	0.9500
C4—C5	1.3898 (17)	C17—C18	1.3928 (16)
C4—H4	0.9500	C17—H17	0.9500
C5—C6	1.3875 (17)	C18—C19	1.3961 (16)
C5—H5	0.9500	C19—C20	1.3826 (15)
C6—C7	1.3896 (16)	C19—H19	0.9500
C6—H6	0.9500	C20—H20	0.9500
C7—C8	1.5044 (15)	C21—H21A	0.9800
C8—C9	1.5204 (15)	C21—H21B	0.9800
C8—H8A	0.9900	C21—H21C	0.9800
C18—O2—C21	117.24 (9)	C1—C10—C11	119.10 (9)
C1—N1—C13	124.74 (9)	C1—C10—C9	118.18 (10)

C1—N1—H1	121.2 (10)	C11—C10—C9	122.72 (9)
C13—N1—H1	114.0 (9)	C12—C11—C10	118.93 (9)
N1—C1—C10	120.00 (10)	C12—C11—C15	120.07 (10)
N1—C1—C2	118.25 (9)	C10—C11—C15	120.99 (9)
C10—C1—C2	121.74 (9)	C11—C12—C14	122.63 (10)
C3—C2—C7	119.70 (10)	C11—C12—C13	121.90 (10)
C3—C2—C1	122.49 (10)	C14—C12—C13	115.35 (9)
C7—C2—C1	117.81 (10)	O1—C13—N1	120.54 (10)
C4—C3—C2	120.28 (10)	O1—C13—C12	124.44 (10)
C4—C3—H3	119.9	N1—C13—C12	115.01 (9)
C2—C3—H3	119.9	N2—C14—C12	177.04 (11)
C3—C4—C5	119.97 (11)	C16—C15—C20	118.30 (10)
C3—C4—H4	120.0	C16—C15—C11	120.52 (10)
C5—C4—H4	120.0	C20—C15—C11	121.18 (10)
C6—C5—C4	120.10 (11)	C17—C16—C15	121.76 (10)
C6—C5—H5	119.9	C17—C16—H16	119.1
C4—C5—H5	119.9	C15—C16—H16	119.1
C5—C6—C7	120.81 (10)	C16—C17—C18	118.99 (10)
C5—C6—H6	119.6	C16—C17—H17	120.5
C7—C6—H6	119.6	C18—C17—H17	120.5
C6—C7—C2	119.13 (10)	O2—C18—C17	124.26 (10)
C6—C7—C8	122.27 (10)	O2—C18—C19	115.50 (10)
C2—C7—C8	118.55 (10)	C17—C18—C19	120.24 (10)
C7—C8—C9	111.49 (9)	C20—C19—C18	119.96 (10)
C7—C8—H8A	109.3	C20—C19—H19	120.0
C9—C8—H8A	109.3	C18—C19—H19	120.0
C7—C8—H8B	109.3	C19—C20—C15	120.75 (10)
C9—C8—H8B	109.3	C19—C20—H20	119.6
H8A—C8—H8B	108.0	C15—C20—H20	119.6
C10—C9—C8	110.71 (9)	O2—C21—H21A	109.5
C10—C9—H9A	109.5	O2—C21—H21B	109.5
C8—C9—H9A	109.5	H21A—C21—H21B	109.5
C10—C9—H9B	109.5	O2—C21—H21C	109.5
C8—C9—H9B	109.5	H21A—C21—H21C	109.5
H9A—C9—H9B	108.1	H21B—C21—H21C	109.5
C13—N1—C1—C10	3.20 (16)	C1—C10—C11—C15	175.99 (9)
C13—N1—C1—C2	−176.95 (9)	C9—C10—C11—C15	−4.29 (16)
N1—C1—C2—C3	−18.44 (15)	C10—C11—C12—C14	175.55 (9)
C10—C1—C2—C3	161.41 (10)	C15—C11—C12—C14	−3.61 (16)
N1—C1—C2—C7	161.30 (10)	C10—C11—C12—C13	−0.36 (16)
C10—C1—C2—C7	−18.86 (15)	C15—C11—C12—C13	−179.53 (9)
C7—C2—C3—C4	0.04 (16)	C1—N1—C13—O1	174.67 (10)
C1—C2—C3—C4	179.77 (10)	C1—N1—C13—C12	−6.45 (15)
C2—C3—C4—C5	0.84 (17)	C11—C12—C13—O1	−176.24 (10)
C3—C4—C5—C6	−0.96 (18)	C14—C12—C13—O1	7.56 (16)
C4—C5—C6—C7	0.19 (18)	C11—C12—C13—N1	4.93 (15)
C5—C6—C7—C2	0.69 (17)	C14—C12—C13—N1	−171.27 (9)
C5—C6—C7—C8	−176.55 (11)	C12—C11—C15—C16	128.40 (11)
C3—C2—C7—C6	−0.80 (16)	C10—C11—C15—C16	−50.74 (14)

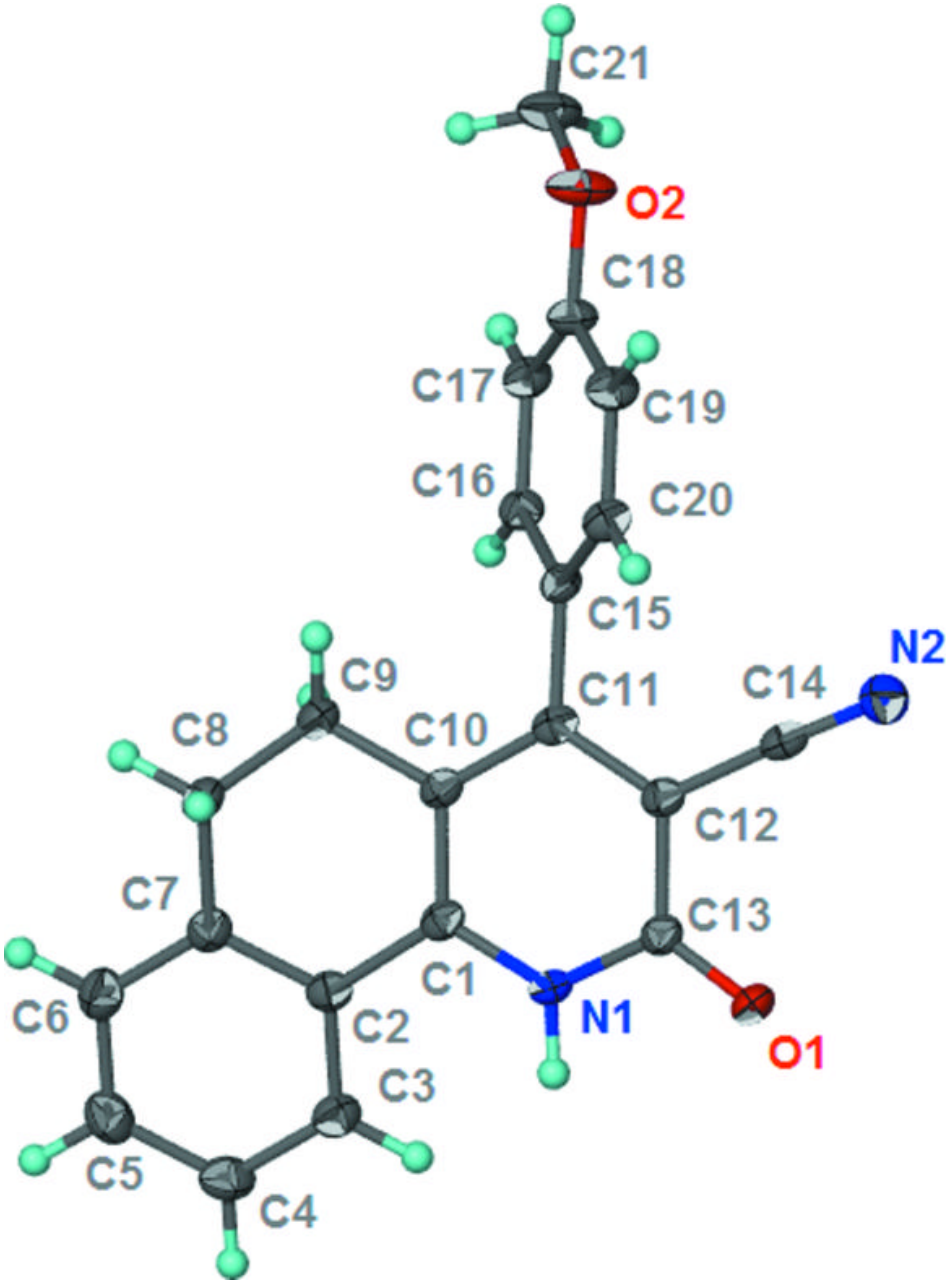
C1—C2—C7—C6	179.46 (10)	C12—C11—C15—C20	−51.08 (14)
C3—C2—C7—C8	176.55 (10)	C10—C11—C15—C20	129.78 (11)
C1—C2—C7—C8	−3.20 (15)	C20—C15—C16—C17	0.34 (16)
C6—C7—C8—C9	−144.07 (11)	C11—C15—C16—C17	−179.16 (10)
C2—C7—C8—C9	38.67 (14)	C15—C16—C17—C18	−0.89 (16)
C7—C8—C9—C10	−52.36 (12)	C21—O2—C18—C17	−4.32 (17)
N1—C1—C10—C11	1.94 (15)	C21—O2—C18—C19	175.79 (10)
C2—C1—C10—C11	−177.91 (9)	C16—C17—C18—O2	−179.15 (10)
N1—C1—C10—C9	−177.80 (9)	C16—C17—C18—C19	0.73 (16)
C2—C1—C10—C9	2.36 (15)	O2—C18—C19—C20	179.86 (10)
C8—C9—C10—C1	33.28 (13)	C17—C18—C19—C20	−0.04 (17)
C8—C9—C10—C11	−146.44 (10)	C18—C19—C20—C15	−0.53 (17)
C1—C10—C11—C12	−3.17 (15)	C16—C15—C20—C19	0.38 (16)
C9—C10—C11—C12	176.55 (10)	C11—C15—C20—C19	179.87 (10)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.90 (2)	1.94 (2)	2.823 (1)	166 (1)

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

Fig. 1


C21
O2
C18
C17
C19
C16
C20
C15
C9
C11
C14
N2
C8
C10
C12
C7
C13
C1
C6
C2
N1
O1
C3
C5
C4