Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-(4-Bromophenyl)-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

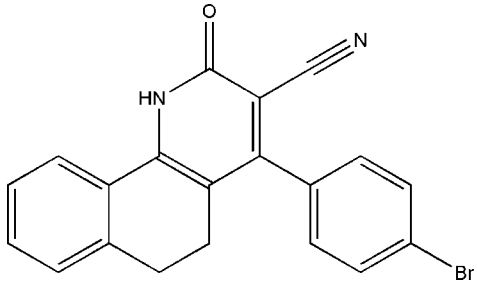
Received 7 August 2011; accepted 19 August 2011

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.032; wR factor = 0.094; data-to-parameter ratio = 14.7.

In the molecule of the title compound, $C_{20}H_{13}BrN_2O$, 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 17.7 (1)°. The 4-substituted aromatic ring is bent away from the pyridine ring by 82.3 (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

$C_{20}H_{13}BrN_2O$
M_r = 377.23
Monoclinic, $C2/c$
a = 22.6906 (5) Å
b = 8.5060 (2) Å
c = 17.6112 (5) Å
β = 106.498 (3)°
V = 3259.13 (14) Å^3
Z = 8
Cu $K\alpha$ radiation
μ = 3.50 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} = 0.420, T_{max} = 0.541
6063 measured reflections
3244 independent reflections
3132 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.094
S = 1.06
3244 reflections
221 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 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}	0.86 (3)	1.96 (3)	2.807 (2)	172 (3)

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: XU5291).

References

Agilent (2010). *CrysAlis PRO*. Agilent Technologies, Yarnton, Oxfordshire, England.
Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Rostom, S. A. F., Faidallah, H. M. & Al-Saadi, M. S. (2011). *Med. Chem. Res.* **20** (DOI: 10.1007/s00044-010-9469-0).
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Acta Cryst. (2011). E**67**, o2469 [doi:10.1107/S1600536811033885]

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

Abdullah M. Asiri, Hassan M. Faidallah, Abdulrahman O. Al-Youbi, Khalid A. Alamry and Seik Weng Ng

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 17.7 (1)°. The 4-subsituted aromatic ring is bent away from the pyridine ring by 83.2 (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 *p*-bromobenzaldehyde (1.85 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 orange precipitate that formed was filtered, washed with water, dried and recrystallized from ethanol; m.p. >630 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.

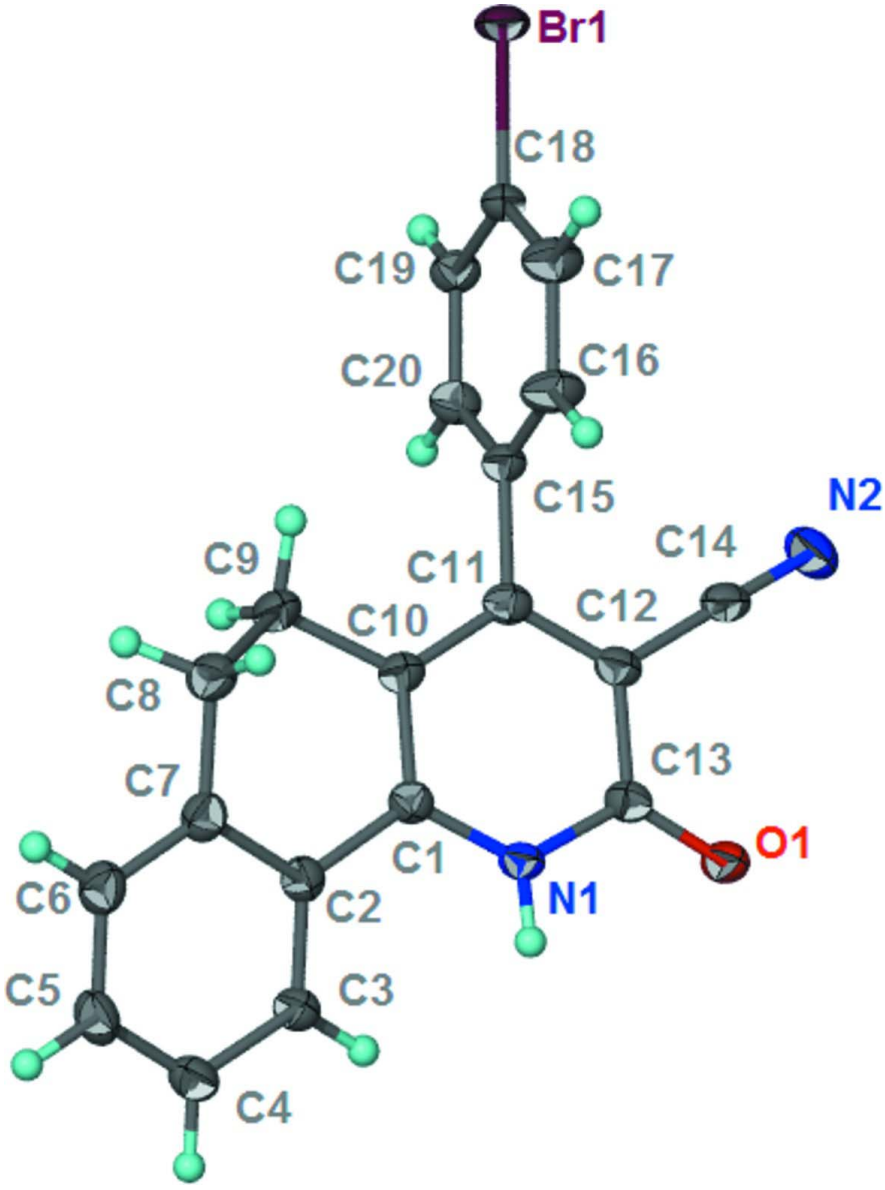


Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{20}H_{13}BrN_2O$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

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

Crystal data

$C_{20}H_{13}BrN_2O$
M_r = 377.23
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 22.6906 (5) Å
b = 8.5060 (2) Å
c = 17.6112 (5) Å
β = 106.498 (3)°
V = 3259.13 (14) Å^3
Z = 8
F(000) = 1520
D_x = 1.538 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 4349 reflections
θ = 4.2–74.3°
μ = 3.50 mm^{-1}

T = 100 K
Octahedron, yellow

0.30 × 0.25 × 0.20 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} = 0.420, T_{max} = 0.541
6063 measured reflections
3244 independent reflections
3132 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 74.4°, θ_{min} = 5.2°
h = −27→27
k = −7→10
l = −14→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.094
S = 1.06
3244 reflections
221 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
$w = 1/[\sigma^2(F_o^2) + (0.0634P)^2 + 3.4873P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
Br1	0.918537 (8)	−0.05660 (2)	0.742895 (12)	0.02179 (11)
O1	0.57369 (7)	0.53089 (18)	0.51145 (10)	0.0241 (3)
N1	0.53722 (7)	0.3095 (2)	0.55547 (10)	0.0172 (3)
N2	0.73458 (8)	0.5190 (3)	0.57289 (12)	0.0264 (4)
C1	0.54467 (9)	0.1682 (2)	0.59371 (11)	0.0167 (4)
C2	0.48982 (9)	0.0824 (2)	0.60012 (12)	0.0175 (4)
C3	0.43270 (9)	0.1549 (2)	0.58744 (12)	0.0195 (4)
H3	0.4284	0.2638	0.5751	0.023*
C4	0.38190 (10)	0.0686 (2)	0.59274 (14)	0.0223 (4)
H4	0.3432	0.1187	0.5848	0.027*
C5	0.38810 (10)	−0.0913 (3)	0.60971 (13)	0.0244 (4)
H5	0.3533	−0.1510	0.6123	0.029*
C6	0.44492 (10)	−0.1637 (3)	0.62285 (13)	0.0240 (4)
H6	0.4487	−0.2728	0.6349	0.029*
C7	0.49638 (10)	−0.0790 (2)	0.61870 (12)	0.0203 (4)
C8	0.55804 (9)	−0.1564 (3)	0.63177 (14)	0.0253 (4)
H8A	0.5629	−0.1912	0.5803	0.030*
H8B	0.5602	−0.2505	0.6655	0.030*
C9	0.61001 (10)	−0.0444 (2)	0.67123 (14)	0.0240 (5)
H9A	0.6096	−0.0241	0.7264	0.029*
H9B	0.6499	−0.0932	0.6728	0.029*
C10	0.60314 (9)	0.1088 (2)	0.62645 (11)	0.0181 (4)

C11	0.65406 (9)	0.1960 (2)	0.61906 (11)	0.0179 (4)
C12	0.64485 (9)	0.3399 (2)	0.58053 (11)	0.0177 (4)
C13	0.58428 (9)	0.4025 (2)	0.54653 (12)	0.0178 (4)
C14	0.69504 (10)	0.4366 (2)	0.57531 (13)	0.0193 (4)
C15	0.71808 (8)	0.1347 (2)	0.65091 (11)	0.0176 (4)
C16	0.74530 (10)	0.0579 (3)	0.59968 (13)	0.0252 (5)
H16	0.7228	0.0448	0.5456	0.030*
C17	0.80490 (10)	0.0000 (3)	0.62658 (12)	0.0251 (4)
H17	0.8232	−0.0532	0.5916	0.030*
C18	0.83693 (9)	0.0216 (2)	0.70531 (12)	0.0182 (4)
C19	0.81110 (9)	0.0985 (2)	0.75721 (12)	0.0194 (4)
H19	0.8340	0.1127	0.8110	0.023*
C20	0.75125 (9)	0.1550 (2)	0.72992 (12)	0.0200 (4)
H20	0.7330	0.2073	0.7652	0.024*
H1	0.5015 (13)	0.349 (3)	0.5343 (15)	0.026 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.01372 (15)	0.02727 (16)	0.02247 (15)	0.00536 (7)	0.00206 (10)	0.00254 (7)
O1	0.0149 (7)	0.0195 (7)	0.0366 (9)	0.0000 (6)	0.0050 (6)	0.0078 (6)
N1	0.0119 (7)	0.0173 (8)	0.0211 (8)	−0.0001 (6)	0.0026 (6)	0.0008 (6)
N2	0.0188 (9)	0.0326 (10)	0.0289 (10)	−0.0042 (8)	0.0084 (8)	0.0001 (8)
C1	0.0157 (9)	0.0182 (9)	0.0154 (8)	−0.0006 (7)	0.0032 (7)	−0.0010 (7)
C2	0.0176 (9)	0.0195 (9)	0.0147 (9)	−0.0015 (8)	0.0034 (7)	0.0000 (7)
C3	0.0160 (9)	0.0202 (9)	0.0217 (9)	−0.0007 (8)	0.0043 (7)	0.0011 (8)
C4	0.0161 (10)	0.0266 (11)	0.0242 (11)	−0.0010 (8)	0.0054 (8)	0.0009 (8)
C5	0.0209 (10)	0.0280 (11)	0.0241 (10)	−0.0076 (9)	0.0063 (8)	0.0018 (9)
C6	0.0237 (10)	0.0210 (10)	0.0257 (10)	−0.0040 (8)	0.0046 (8)	0.0032 (8)
C7	0.0217 (10)	0.0198 (10)	0.0184 (10)	−0.0014 (8)	0.0039 (8)	0.0011 (7)
C8	0.0214 (10)	0.0193 (10)	0.0340 (11)	0.0006 (8)	0.0062 (9)	0.0048 (9)
C9	0.0172 (10)	0.0245 (11)	0.0278 (11)	0.0024 (8)	0.0023 (9)	0.0085 (8)
C10	0.0153 (9)	0.0193 (10)	0.0186 (9)	0.0012 (8)	0.0028 (7)	0.0019 (8)
C11	0.0155 (9)	0.0210 (9)	0.0162 (8)	0.0011 (8)	0.0028 (7)	−0.0020 (7)
C12	0.0133 (8)	0.0214 (9)	0.0180 (9)	0.0001 (7)	0.0037 (7)	−0.0006 (7)
C13	0.0148 (9)	0.0187 (9)	0.0192 (9)	−0.0006 (8)	0.0037 (7)	−0.0014 (8)
C14	0.0147 (10)	0.0237 (11)	0.0187 (10)	0.0035 (7)	0.0036 (8)	0.0009 (7)
C15	0.0133 (8)	0.0186 (9)	0.0193 (9)	0.0003 (7)	0.0024 (7)	0.0027 (7)
C16	0.0190 (10)	0.0354 (13)	0.0176 (10)	0.0066 (8)	−0.0008 (8)	−0.0028 (8)
C17	0.0216 (10)	0.0333 (12)	0.0193 (10)	0.0065 (9)	0.0039 (8)	−0.0027 (9)
C18	0.0128 (8)	0.0210 (9)	0.0199 (9)	0.0019 (8)	0.0032 (7)	0.0038 (8)
C19	0.0157 (9)	0.0216 (9)	0.0181 (9)	−0.0007 (8)	0.0004 (7)	−0.0005 (8)
C20	0.0177 (9)	0.0225 (10)	0.0195 (9)	0.0018 (8)	0.0050 (8)	−0.0018 (8)

Geometric parameters (Å, º)

Br1—C18	1.9009 (19)	C8—H8A	0.9900
O1—C13	1.244 (3)	C8—H8B	0.9900

N1—C1	1.365 (3)	C9—C10	1.508 (3)
N1—C13	1.374 (3)	C9—H9A	0.9900
N1—H1	0.86 (3)	C9—H9B	0.9900
N2—C14	1.149 (3)	C10—C11	1.410 (3)
C1—C10	1.383 (3)	C11—C12	1.387 (3)
C1—C2	1.474 (3)	C11—C15	1.494 (3)
C2—C3	1.395 (3)	C12—C14	1.429 (3)
C2—C7	1.410 (3)	C12—C13	1.436 (3)
C3—C4	1.392 (3)	C15—C20	1.392 (3)
C3—H3	0.9500	C15—C16	1.393 (3)
C4—C5	1.391 (3)	C16—C17	1.390 (3)
C4—H4	0.9500	C16—H16	0.9500
C5—C6	1.388 (3)	C17—C18	1.383 (3)
C5—H5	0.9500	C17—H17	0.9500
C6—C7	1.392 (3)	C18—C19	1.382 (3)
C6—H6	0.9500	C19—C20	1.391 (3)
C7—C8	1.504 (3)	C19—H19	0.9500
C8—C9	1.522 (3)	C20—H20	0.9500

C1—N1—C13	124.93 (17)	C8—C9—H9B	109.5
C1—N1—H1	121.9 (19)	H9A—C9—H9B	108.1
C13—N1—H1	113.2 (18)	C1—C10—C11	118.91 (18)
N1—C1—C10	119.81 (18)	C1—C10—C9	118.56 (18)
N1—C1—C2	118.98 (17)	C11—C10—C9	122.49 (18)
C10—C1—C2	121.20 (18)	C12—C11—C10	119.75 (18)
C3—C2—C7	119.98 (19)	C12—C11—C15	119.14 (17)
C3—C2—C1	122.38 (18)	C10—C11—C15	121.11 (18)
C7—C2—C1	117.64 (18)	C11—C12—C14	121.83 (18)
C4—C3—C2	120.39 (19)	C11—C12—C13	121.65 (18)
C4—C3—H3	119.8	C14—C12—C13	116.48 (18)
C2—C3—H3	119.8	O1—C13—N1	121.05 (18)
C5—C4—C3	119.7 (2)	O1—C13—C12	124.01 (18)
C5—C4—H4	120.2	N1—C13—C12	114.94 (18)
C3—C4—H4	120.2	N2—C14—C12	177.1 (2)
C6—C5—C4	120.2 (2)	C20—C15—C16	119.48 (18)
C6—C5—H5	119.9	C20—C15—C11	121.61 (18)
C4—C5—H5	119.9	C16—C15—C11	118.89 (18)
C5—C6—C7	121.0 (2)	C17—C16—C15	120.9 (2)
C5—C6—H6	119.5	C17—C16—H16	119.6
C7—C6—H6	119.5	C15—C16—H16	119.6
C6—C7—C2	118.8 (2)	C18—C17—C16	118.6 (2)
C6—C7—C8	121.56 (19)	C18—C17—H17	120.7
C2—C7—C8	119.62 (19)	C16—C17—H17	120.7
C7—C8—C9	111.25 (18)	C17—C18—C19	121.67 (18)
C7—C8—H8A	109.4	C17—C18—Br1	119.04 (16)
C9—C8—H8A	109.4	C19—C18—Br1	119.29 (15)
C7—C8—H8B	109.4	C18—C19—C20	119.37 (19)
C9—C8—H8B	109.4	C18—C19—H19	120.3

H8A—C8—H8B	108.0	C20—C19—H19	120.3
C10—C9—C8	110.52 (18)	C15—C20—C19	120.03 (18)
C10—C9—H9A	109.5	C15—C20—H20	120.0
C8—C9—H9A	109.5	C19—C20—H20	120.0
C10—C9—H9B	109.5		

C13—N1—C1—C10	−0.5 (3)	C9—C10—C11—C12	−176.75 (19)
C13—N1—C1—C2	178.78 (18)	C1—C10—C11—C15	−177.91 (18)
N1—C1—C2—C3	−16.9 (3)	C9—C10—C11—C15	4.3 (3)
C10—C1—C2—C3	162.37 (19)	C10—C11—C12—C14	176.52 (19)
N1—C1—C2—C7	162.20 (18)	C15—C11—C12—C14	−4.5 (3)
C10—C1—C2—C7	−18.5 (3)	C10—C11—C12—C13	−0.9 (3)
C7—C2—C3—C4	−0.2 (3)	C15—C11—C12—C13	178.03 (18)
C1—C2—C3—C4	178.85 (19)	C1—N1—C13—O1	−179.27 (19)
C2—C3—C4—C5	−0.9 (3)	C1—N1—C13—C12	0.6 (3)
C3—C4—C5—C6	1.3 (3)	C11—C12—C13—O1	179.99 (19)
C4—C5—C6—C7	−0.6 (3)	C14—C12—C13—O1	2.4 (3)
C5—C6—C7—C2	−0.6 (3)	C11—C12—C13—N1	0.1 (3)
C5—C6—C7—C8	−179.2 (2)	C14—C12—C13—N1	−177.43 (17)
C3—C2—C7—C6	0.9 (3)	C12—C11—C15—C20	96.8 (2)
C1—C2—C7—C6	−178.17 (18)	C10—C11—C15—C20	−84.2 (3)
C3—C2—C7—C8	179.62 (19)	C12—C11—C15—C16	−82.1 (2)
C1—C2—C7—C8	0.5 (3)	C10—C11—C15—C16	96.8 (2)
C6—C7—C8—C9	−146.7 (2)	C20—C15—C16—C17	0.6 (3)
C2—C7—C8—C9	34.7 (3)	C11—C15—C16—C17	179.5 (2)
C7—C8—C9—C10	−51.9 (2)	C15—C16—C17—C18	−0.6 (4)
N1—C1—C10—C11	−0.4 (3)	C16—C17—C18—C19	0.1 (3)
C2—C1—C10—C11	−179.60 (18)	C16—C17—C18—Br1	−179.90 (17)
N1—C1—C10—C9	177.51 (18)	C17—C18—C19—C20	0.4 (3)
C2—C1—C10—C9	−1.7 (3)	Br1—C18—C19—C20	−179.60 (16)
C8—C9—C10—C1	37.1 (3)	C16—C15—C20—C19	0.0 (3)
C8—C9—C10—C11	−145.1 (2)	C11—C15—C20—C19	−178.99 (19)
C1—C10—C11—C12	1.0 (3)	C18—C19—C20—C15	−0.4 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86 (3)	1.96 (3)	2.807 (2)	172 (3)

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