

4-(4-Bromophenyl)-2-oxo-1,2,5,6-tetrahydrobenzo[*h*]quinoline-3-carbonitrileAbdullah M. Asiri,^a Hassan M. Faidallah,^a Abdulrahman O. Al-Youbi,^a Khalid A. Alamry^a and Seik Weng Ng^{b,a*}^aChemistry Department, Faculty of Science, King Abdulaziz University, PO Box 80203 Jeddah, Saudi Arabia, and ^bDepartment of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

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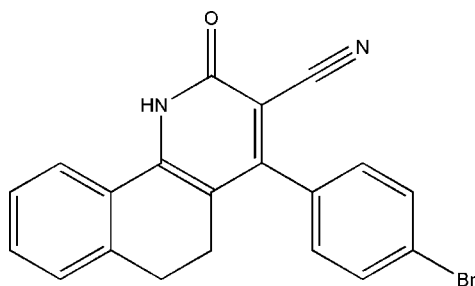
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.032; wR factor = 0.094; data-to-parameter ratio = 14.7.

In the molecule of the title compound, $\text{C}_{20}\text{H}_{13}\text{BrN}_2\text{O}$, the tetrahydrobenzo[*h*]quinoline fused-ring system is buckled owing to the ethylene $-\text{CH}_2\text{CH}_2-$ 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 $\text{N}-\text{H}\cdots\text{O}$ 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

$\text{C}_{20}\text{H}_{13}\text{BrN}_2\text{O}$
 $M_r = 377.23$
 Monoclinic, $C2/c$
 $a = 22.6906$ (5) Å
 $b = 8.5060$ (2) Å
 $c = 17.6112$ (5) Å
 $\beta = 106.498$ (3)°

$V = 3259.13$ (14) Å³
 $Z = 8$
 Cu $K\alpha$ radiation
 $\mu = 3.50$ mm⁻¹
 $T = 100$ K
 $0.30 \times 0.25 \times 0.20$ 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 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$

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
 $\Delta\rho_{\text{max}} = 0.56$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.57$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{O1}^i$	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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5291).

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supplementary materials

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

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Comment

The compound (Scheme 1) 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 –CH₂CH₂– 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 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).

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.

Refinement

Carbon- and nitrogen-bound H atoms were placed in calculated positions [C—H 0.95 to 0.99 Å, $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$] 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

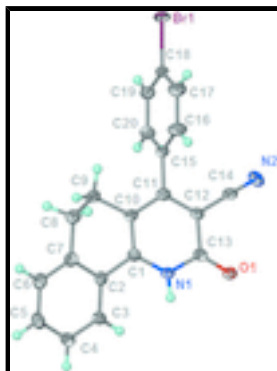


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of C₂₀H₁₃BrN₂O at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

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

Crystal data

$C_{20}H_{13}BrN_2O$	$F(000) = 1520$
$M_r = 377.23$	$D_x = 1.538 \text{ Mg m}^{-3}$
Monoclinic, $C2/c$	Cu $K\alpha$ radiation, $\lambda = 1.54184 \text{ \AA}$
Hall symbol: $-C 2yc$	Cell parameters from 4349 reflections
$a = 22.6906 (5) \text{ \AA}$	$\theta = 4.2\text{--}74.3^\circ$
$b = 8.5060 (2) \text{ \AA}$	$\mu = 3.50 \text{ mm}^{-1}$
$c = 17.6112 (5) \text{ \AA}$	$T = 100 \text{ K}$
$\beta = 106.498 (3)^\circ$	Octahedron, yellow
$V = 3259.13 (14) \text{ \AA}^3$	$0.30 \times 0.25 \times 0.20 \text{ mm}$
$Z = 8$	

Data collection

Agilent SuperNova Dual diffractometer with Atlas detector	3244 independent reflections
Radiation source: SuperNova (Cu) X-ray Source mirror	3132 reflections with $I > 2\sigma(I)$
Detector resolution: $10.4041 \text{ pixels mm}^{-1}$	$R_{\text{int}} = 0.022$
ω scans	$\theta_{\text{max}} = 74.4^\circ$, $\theta_{\text{min}} = 5.2^\circ$
Absorption correction: multi-scan (<i>CrysAlis PRO</i> ; Agilent, 2010)	$h = -27 \rightarrow 27$
$T_{\text{min}} = 0.420$, $T_{\text{max}} = 0.541$	$k = -7 \rightarrow 10$
6063 measured reflections	$l = -14 \rightarrow 21$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.032$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.094$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.06$	$w = 1/[\sigma^2(F_o^2) + (0.0634P)^2 + 3.4873P]$
3244 reflections	where $P = (F_o^2 + 2F_c^2)/3$
221 parameters	$(\Delta/\sigma)_{\text{max}} = 0.001$
0 restraints	$\Delta\rho_{\text{max}} = 0.56 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.57 \text{ e \AA}^{-3}$

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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 (\AA^2)

	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)

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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, °)

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)

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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, °)

<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>
N1—H1 \cdots O1 ⁱ	0.86 (3)	1.96 (3)	2.807 (2)	172 (3)

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

Fig. 1

