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6-Bromo-1,2,3,4-tetrahydroquinoline-8-carbonitrile

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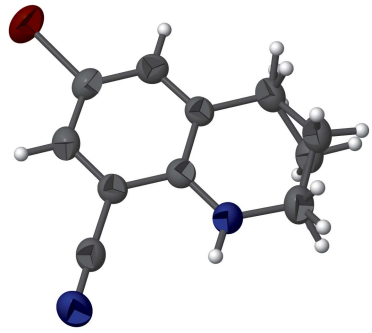
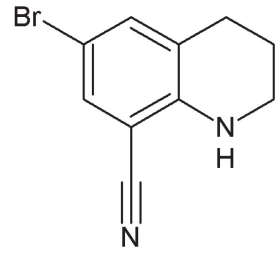
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{10}H_9BrN_2$, one of the methylene groups of the piperidine ring is disordered over two sets of sites in a 0.692 (8):0.308 (8) ratio, which leads to two envelope conformations. In the crystal, inversion dimers linked by pairs of N—H···N hydrogen bonds generate $R_2^2(12)$ loops.

3D view



Chemical scheme



Structure description

Cyano quinoline compounds can deactivate the action of growth factor receptor protein tyrocine kinases (Berger *et al.*, 2008) and bind with biological systems (Fleming *et al.*, 2010). As part of our ongoing synthetic and structural studies in this area (Ökten & Çakmak, 2015), we now describe the title compound (Fig. 1).

The piperidine ring is disordered over two conformations in a 0.692 (8):0.308 (8) ratio. Both disorder components lead to an envelope conformation for the ring, with methylene atom C8 as the flap with it and its attached H atoms either above or below the plane of the other atoms.

In the crystal, inversion dimers linked by pairs of N—H···N hydrogen bonds (Table 1 and Fig. 2) generate $R_2^2(12)$ loops. Br···Br contacts [3.6394 (14) Å] just shorter than the van der Waals contact distance (3.70 Å) are also observed.

Synthesis and crystallization

6-Bromo-8-cyano-1,2,3,4-tetrahydroquinoline was prepared by the literature method (Ökten & Çakmak, 2015). Recrystallization from a CH_2Cl_2/hexane (2:1, 5 ml) solvent mixture gave colourless prisms.

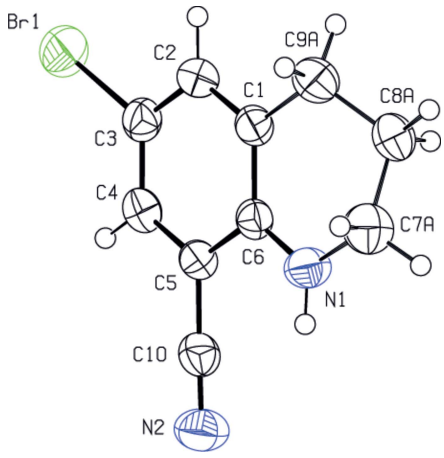


Figure 1
A view of the title compound, with displacement ellipsoids drawn at the 50% probability level. The minor component of the disorder is not shown.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The central carbon atom (C8) and its attached H atoms of the three methylene groups in the piperidine ring are positionally disordered over two sets of sites (C8*A* and C8*B*) in a 0.692 (8): 0.308 (8) ratio. The other two methylene C atoms (C6 and C7) in the piperidine ring are also refined as disordered and constrained with the EXYZ and EADP instructions to aid in the location of their attached H atoms.

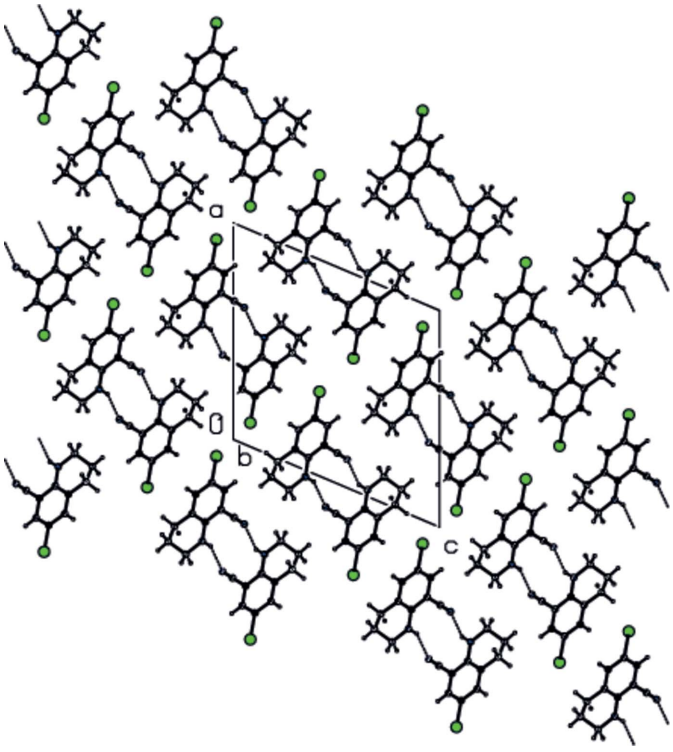


Figure 2
A view down the *b* axis of the dimers linked by pairs of N—H···N hydrogen bonds (shown as dashed lines).

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···N2^{i}	0.85 (2)	2.29 (3)	3.075 (4)	153 (2)

Symmetry code: (i) $-x+2, -y+2, -z+1$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{10}H_9BrN_2$
M_r	237.09
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	296
a, *b*, *c* (Å)	14.189 (5), 5.0064 (14), 14.683 (5)
β (°)	113.008 (13)
V (Å^3)	960.1 (5)
Z	4
Radiation type	Mo *K*α
μ (mm^{-1})	4.23
Crystal size (mm)	0.19 × 0.15 × 0.14
Data collection	
Diffractometer	Bruker APEXII CCD
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	35875, 2412, 1837
R_{int}	0.043
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.672
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.031, 0.080, 1.10
No. of reflections	2412
No. of parameters	133
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.36, −0.47

Computer programs: *APEX2* and *SAINT* (Bruker, 2007), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2009).

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x161854 [https://doi.org/10.1107/S241431461601854X]

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Crystal data

$C_{10}H_9BrN_2$
M_r = 237.09
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 14.189 (5) Å
b = 5.0064 (14) Å
c = 14.683 (5) Å
β = 113.008 (13)°
V = 960.1 (5) Å^3
Z = 4
$F(000)$ = 472
D_x = 1.640 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9423 reflections
θ = 3.0–27.8°
μ = 4.23 mm^{-1}
T = 296 K
Prism, colourless
0.19 × 0.15 × 0.14 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
35875 measured reflections
2412 independent reflections
1837 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 28.5°, θ_{min} = 3.0°
h = −19→18
k = −6→6
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.080
S = 1.10
2412 reflections
133 parameters
1 restraint
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0285P)^2 + 0.7134P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0135 (11)

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses 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 observed criterion of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating -R-factor-obs 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Br1	0.61037 (2)	0.18750 (7)	0.58211 (2)	0.0649 (1)	
N1	1.03058 (15)	0.6179 (5)	0.65068 (16)	0.0490 (7)	
N2	0.86937 (18)	0.9940 (5)	0.44726 (18)	0.0611 (8)	
C1	0.92333 (18)	0.3281 (4)	0.70001 (16)	0.0394 (7)	
C2	0.82674 (19)	0.2339 (5)	0.68257 (17)	0.0446 (8)	
C3	0.74161 (17)	0.3303 (5)	0.60479 (17)	0.0431 (7)	
C4	0.75099 (17)	0.5260 (5)	0.54363 (16)	0.0426 (7)	
C5	0.84797 (17)	0.6238 (5)	0.55964 (16)	0.0388 (7)	
C6	0.93628 (16)	0.5253 (4)	0.63672 (15)	0.0373 (7)	
C7A	1.12305 (19)	0.5279 (6)	0.7306 (2)	0.0585 (9)	0.692 (8)
C7B	1.12305 (19)	0.5279 (6)	0.7306 (2)	0.0585 (9)	0.308 (8)
C8A	1.0989 (3)	0.4295 (9)	0.8157 (3)	0.0525 (14)	0.692 (8)
C8B	1.1163 (6)	0.2658 (19)	0.7700 (7)	0.051 (3)	0.308 (8)
C9A	1.0160 (2)	0.2262 (6)	0.7852 (2)	0.0527 (8)	0.692 (8)
C9B	1.0160 (2)	0.2262 (6)	0.7852 (2)	0.0527 (8)	0.308 (8)
C10	0.85870 (18)	0.8300 (5)	0.49630 (18)	0.0447 (7)	
H7A1	1.17160	0.67400	0.75250	0.0700*	0.692 (8)
H1N	1.037 (2)	0.731 (4)	0.6103 (17)	0.054 (8)*	
H2	0.81840	0.10300	0.72380	0.0540*	
H7A2	1.15410	0.38510	0.70720	0.0700*	0.692 (8)
H8A1	1.16030	0.35260	0.86540	0.0630*	0.692 (8)
H4	0.69340	0.59240	0.49220	0.0510*	
H8A2	1.07860	0.57980	0.84560	0.0630*	0.692 (8)
H9A1	0.99750	0.18500	0.84060	0.0630*	0.692 (8)
H9A2	1.04010	0.06330	0.76580	0.0630*	0.692 (8)
H7B1	1.14110	0.65690	0.78400	0.0700*	0.308 (8)
H7B2	1.17820	0.52530	0.70710	0.0700*	0.308 (8)
H8B1	1.17370	0.24230	0.83280	0.0610*	0.308 (8)
H8B2	1.12130	0.13010	0.72490	0.0610*	0.308 (8)
H9B1	1.00690	0.03740	0.79430	0.0630*	0.308 (8)
H9B2	1.02110	0.31810	0.84510	0.0630*	0.308 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0467 (2)	0.0867 (3)	0.0617 (2)	−0.0181 (1)	0.0216 (1)	−0.0032 (2)
N1	0.0394 (10)	0.0536 (13)	0.0496 (12)	−0.0030 (9)	0.0125 (9)	0.0126 (10)
N2	0.0559 (13)	0.0667 (16)	0.0585 (14)	0.0000 (11)	0.0200 (11)	0.0202 (12)
C1	0.0435 (12)	0.0372 (12)	0.0339 (11)	0.0006 (9)	0.0114 (9)	−0.0004 (9)

C2	0.0499 (13)	0.0471 (14)	0.0375 (12)	−0.0058 (10)	0.0178 (10)	0.0019 (10)
C3	0.0385 (11)	0.0510 (14)	0.0406 (12)	−0.0074 (10)	0.0163 (9)	−0.0061 (11)
C4	0.0392 (11)	0.0488 (14)	0.0352 (11)	0.0017 (10)	0.0097 (9)	−0.0016 (10)
C5	0.0431 (12)	0.0387 (12)	0.0328 (10)	−0.0010 (9)	0.0128 (9)	−0.0001 (9)
C6	0.0406 (11)	0.0364 (12)	0.0331 (11)	−0.0009 (9)	0.0124 (9)	−0.0031 (9)
C7A	0.0393 (13)	0.0695 (19)	0.0601 (16)	0.0044 (12)	0.0122 (12)	0.0094 (14)
C7B	0.0393 (13)	0.0695 (19)	0.0601 (16)	0.0044 (12)	0.0122 (12)	0.0094 (14)
C8A	0.044 (2)	0.060 (3)	0.042 (2)	0.0004 (18)	0.0044 (16)	0.0050 (19)
C8B	0.042 (4)	0.059 (6)	0.046 (5)	0.006 (4)	0.010 (4)	0.006 (4)
C9A	0.0500 (14)	0.0538 (15)	0.0462 (13)	0.0050 (12)	0.0102 (11)	0.0093 (12)
C9B	0.0500 (14)	0.0538 (15)	0.0462 (13)	0.0050 (12)	0.0102 (11)	0.0093 (12)
C10	0.0400 (12)	0.0499 (14)	0.0395 (12)	0.0001 (10)	0.0105 (10)	0.0018 (11)

Geometric parameters (Å, º)

Br1—C3	1.899 (3)	C8A—C9A	1.486 (5)
N1—C6	1.354 (3)	C8B—C9B	1.537 (10)
N1—C7A	1.448 (4)	C2—H2	0.9300
N1—C7B	1.448 (4)	C4—H4	0.9300
N2—C10	1.141 (4)	C7A—H7A1	0.9700
C1—C2	1.376 (4)	C7A—H7A2	0.9700
C1—C6	1.416 (3)	C7B—H7B1	0.9700
C1—C9A	1.506 (4)	C7B—H7B2	0.9700
C1—C9B	1.506 (4)	C8A—H8A1	0.9700
N1—H1N	0.85 (2)	C8A—H8A2	0.9700
C2—C3	1.385 (4)	C8B—H8B1	0.9700
C3—C4	1.370 (3)	C8B—H8B2	0.9700
C4—C5	1.391 (4)	C9A—H9A1	0.9700
C5—C10	1.437 (4)	C9A—H9A2	0.9700
C5—C6	1.409 (3)	C9B—H9B1	0.9700
C7A—C8A	1.502 (5)	C9B—H9B2	0.9700
C7B—C8B	1.452 (10)		
C6—N1—C7A	123.0 (2)	C5—C4—H4	121.00
C6—N1—C7B	123.0 (2)	N1—C7A—H7A1	110.00
C2—C1—C6	119.5 (2)	N1—C7A—H7A2	110.00
C2—C1—C9A	121.4 (2)	C8A—C7A—H7A1	110.00
C2—C1—C9B	121.4 (2)	C8A—C7A—H7A2	110.00
C6—C1—C9A	119.1 (2)	H7A1—C7A—H7A2	108.00
C6—C1—C9B	119.1 (2)	H7B1—C7B—H7B2	108.00
C6—N1—H1N	119.4 (19)	N1—C7B—H7B2	108.00
C7A—N1—H1N	117.5 (19)	C8B—C7B—H7B1	109.00
C7B—N1—H1N	117.5 (19)	N1—C7B—H7B1	108.00
C1—C2—C3	121.2 (2)	C8B—C7B—H7B2	108.00
Br1—C3—C2	119.54 (19)	C7A—C8A—H8A2	109.00
Br1—C3—C4	119.61 (18)	C9A—C8A—H8A1	109.00
C2—C3—C4	120.9 (2)	C7A—C8A—H8A1	109.00
C3—C4—C5	118.9 (2)	H8A1—C8A—H8A2	108.00

C6—C5—C10	119.0 (2)	C9A—C8A—H8A2	109.00
C4—C5—C10	119.4 (2)	C7B—C8B—H8B2	109.00
C4—C5—C6	121.5 (2)	C7B—C8B—H8B1	109.00
N1—C6—C1	120.9 (2)	H8B1—C8B—H8B2	108.00
N1—C6—C5	121.2 (2)	C9B—C8B—H8B1	109.00
C1—C6—C5	117.9 (2)	C9B—C8B—H8B2	109.00
N1—C7A—C8A	110.3 (3)	C1—C9A—H9A2	110.00
N1—C7B—C8B	115.1 (4)	H9A1—C9A—H9A2	108.00
C7A—C8A—C9A	112.6 (3)	C8A—C9A—H9A1	110.00
C7B—C8B—C9B	112.5 (6)	C8A—C9A—H9A2	110.00
C1—C9A—C8A	110.5 (3)	C1—C9A—H9A1	110.00
C1—C9B—C8B	113.1 (4)	C1—C9B—H9B1	109.00
N2—C10—C5	178.6 (3)	C1—C9B—H9B2	109.00
C1—C2—H2	119.00	C8B—C9B—H9B1	109.00
C3—C2—H2	119.00	C8B—C9B—H9B2	109.00
C3—C4—H4	121.00	H9B1—C9B—H9B2	108.00

C7A—N1—C6—C1	−1.1 (4)	C1—C2—C3—C4	−1.0 (4)
C7A—N1—C6—C5	178.2 (2)	Br1—C3—C4—C5	−178.36 (18)
C6—N1—C7A—C8A	−25.7 (4)	C2—C3—C4—C5	1.2 (4)
C6—C1—C2—C3	−0.7 (4)	C3—C4—C5—C10	−179.7 (2)
C9A—C1—C2—C3	179.3 (2)	C3—C4—C5—C6	0.3 (4)
C2—C1—C6—N1	−178.6 (2)	C4—C5—C6—N1	178.7 (2)
C2—C1—C9A—C8A	−154.6 (3)	C4—C5—C6—C1	−2.0 (3)
C6—C1—C9A—C8A	25.3 (3)	C10—C5—C6—N1	−1.3 (3)
C2—C1—C6—C5	2.1 (3)	C10—C5—C6—C1	178.1 (2)
C9A—C1—C6—N1	1.5 (3)	N1—C7A—C8A—C9A	52.7 (4)
C9A—C1—C6—C5	−177.9 (2)	C7A—C8A—C9A—C1	−52.4 (4)
C1—C2—C3—Br1	178.54 (18)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···N2^{i}	0.85 (2)	2.29 (3)	3.075 (4)	153 (2)

Symmetry code: (i) −*x*+2, −*y*+2, −*z*+1.