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# *N*-(4-Bromo-2-nitrophenyl)-*N*-methyl-*N*′-(quinolin-4-ylmethylene)hydrazine

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**Key indicators**

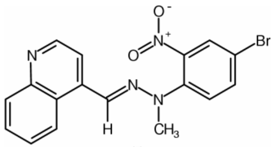
Single-crystal X-ray study
*T* = 293 K
Mean σ(C–C) = 0.005 Å
*R* factor = 0.044
*wR* factor = 0.107
Data-to-parameter ratio = 15.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{17}H_{13}BrN_4O_2$, is non-planar, with a dihedral angle between the quinoline and substituted phenyl rings of 20.8 (1)°. The N—N bond distance [1.359 (3) Å] is close to the value of 1.350 (10) Å quoted for the N—N bond in pyridazinium [Allen *et al.* (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19].

## Comment

Hydrazones are widely used in both organic syntheses and industrial applications because of their reaction abilities, such as ring closure, cycloaddiation, coupling, *etc*. In addition, because of their biological activities, heteroaromatic hydrazones and derivatives are medicinally important compounds, and they are under investigation as potential anticancer and anti-HIV reagents (Kirk-Othmer Encyclopedia of Chemical Technology, 1980; Gürsoy *et al.*, 1997). In view of this, the crystal structure determination of the title compound, (I), has been carried out in order to elucidate the molecular conformation.



(I)

The molecular structure of (I) is shown in Fig. 1. The C10═N2 bond length of 1.278 (4) Å is longer than typical double bonds observed in similar structures, *viz*. 1.265 (4) (Öztürk, Aygün *et al.*, 2001), 1.267 (3) (Akkurt *et al.*, 2001) and 1.265 (4) Å (Öztürk, Akkurt *et al.*, 2001). The C16—N4 bond length [1.471 (4) Å] of the nitro group in (I) is close to the standard value for this type of bond (Allen *et al.*, 1987). In the quinoline ring system, the C7—C8—C9 angle is larger than 120° [124.4 (3)°] and the N1—C3—C4 angle is smaller than 120° [118.3 (3)°].

The C9—C10═N2—N3(C17)—C11 central chain is planar [maximum displacement of −0.037 (3) Å for atom N3] and forms dihedral angles of 18.4 (1) and 12.8 (1)° with the quinoline and substituted phenyl planes, respectively.

There is a weak hydrogen bond, calculated by *PARST*97 (Nardelli, 1995), given in Table 2, and the H···*A* distance, 2.53 Å, is less than the sum of the van der Waals radii.

## Experimental

Quinoline-4-carboxaldehyde (1.0 mmol) was dissolved in hot absolute ethanol (10 ml) and an equimolar amount of *N*-(4-bromo-2-nitrophenyl)-*N*-methylhydrazine dissolved in hot absolute ethanol (10 ml) was added. The mixture was refluxed on a steam bath for 3 h. The crude product which precipitated on cooling was recrytallized from ethanol. Yield 97%, m.p.: 464–465 K; IR (KBr): $\nu$ 3090–3010, 3000–2930, 1585, 1495 and 1375, 1100, 750 $\text{cm}^{-1}$; $^1$H NMR (DMSO-$d_6$, $\delta$, 200 MHz): 3.66 (*s*, N—$CH_3$, 3H), 7.53–8.91 (*m*, ArH and CH, 10H); MS: *m*/*z* 385 ($M^+$), 155 ($M - 230$), 128 ($M - 257$), 101 ($M - 284$); UV ($CHCl_3$): $\lambda_{max}$ 248.1, 351.3 nm; elemental analysis, $C_{17}H_{13}BrN_4O_2$ requires/found: C 52.99/52.63, H 3.40/3.43, N 14.54/14.41%.

*Crystal data*

$C_{17}H_{13}BrN_4O_2$
$M_r = 385.22$
Monoclinic, $P2_1/c$
$a = 12.7415\ (11)$ Å
$b = 6.9924\ (6)$ Å
$c = 17.9671\ (15)$ Å
$\beta = 98.641\ (2)^\circ$
$V = 1582.6\ (2)$ Å$^3$
$Z = 4$
$D_x = 1.617$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 9640 reflections
$\theta = 2.6$–$28.3^\circ$
$\mu = 2.62$ mm$^{-1}$
$T = 293\ (2)$ K
Needle, yellow
$0.44 \times 0.22 \times 0.10$ mm

*Data collection*

Siemens SMART CCD area-detector diffractometer
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.506$, $T_{max} = 0.770$
9585 measured reflections
3858 independent reflections
1982 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 28.3^\circ$
$h = -16 \rightarrow 16$
$k = -9 \rightarrow 9$
$l = -23 \rightarrow 16$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.107$
$S = 1.04$
3858 reflections
245 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0291P)^2 + 1.2305P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.42$ e Å$^{-3}$
$\Delta\rho_{min} = -0.58$ e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Br1—C14 | 1.895 (3) | N2—C10 | 1.278 (4) |
| O1—N4 | 1.209 (4) | N2—N3 | 1.359 (3) |
| O2—N4 | 1.224 (4) | N3—C11 | 1.393 (4) |
| N1—C2 | 1.308 (5) | N3—C17 | 1.460 (4) |
| N1—C3 | 1.375 (4) | N4—C16 | 1.471 (4) |
| | | | |
| C2—N1—C3 | 116.9 (3) | N1—C3—C8 | 122.2 (3) |
| C10—N2—N3 | 120.3 (3) | C7—C8—C9 | 124.4 (3) |
| N2—N3—C11 | 116.2 (2) | N2—C10—C9 | 119.1 (3) |
| N2—N3—C17 | 120.9 (3) | N3—C11—C12 | 121.5 (3) |
| C11—N3—C17 | 122.5 (3) | N3—C11—C16 | 123.7 (3) |
| O1—N4—O2 | 124.5 (3) | C15—C14—Br1 | 119.8 (3) |
| O1—N4—C16 | 117.9 (3) | C13—C14—Br1 | 119.9 (2) |
| O2—N4—C16 | 117.6 (3) | C15—C16—N4 | 113.5 (3) |
| N1—C2—C1 | 125.1 (4) | C11—C16—N4 | 123.1 (3) |
| N1—C3—C4 | 118.3 (3) | | |
| | | | |
| C10—N2—N3—C11 | 176.4 (3) | C12—C13—C14—Br1 | 179.5 (3) |
| C10—N2—N3—C17 | 4.3 (5) | Br1—C14—C15—C16 | −177.1 (3) |
| N3—N2—C10—C9 | 178.2 (3) | C14—C15—C16—N4 | 172.8 (3) |
| C1—C9—C10—N2 | −17.7 (5) | N3—C11—C16—C15 | −177.6 (3) |
| C8—C9—C10—N2 | 162.4 (3) | N3—C11—C16—N4 | 7.4 (5) |
| N2—N3—C11—C12 | −163.7 (3) | C12—C11—C16—N4 | −173.8 (3) |
| C17—N3—C11—C12 | 8.3 (5) | O1—N4—C16—C15 | 69.7 (4) |
| N2—N3—C11—C16 | 15.1 (5) | O2—N4—C16—C15 | −107.1 (4) |
| C17—N3—C11—C16 | −172.9 (4) | O1—N4—C16—C11 | −114.9 (4) |
| N3—C11—C12—C13 | −179.8 (3) | O2—N4—C16—C11 | 68.3 (4) |

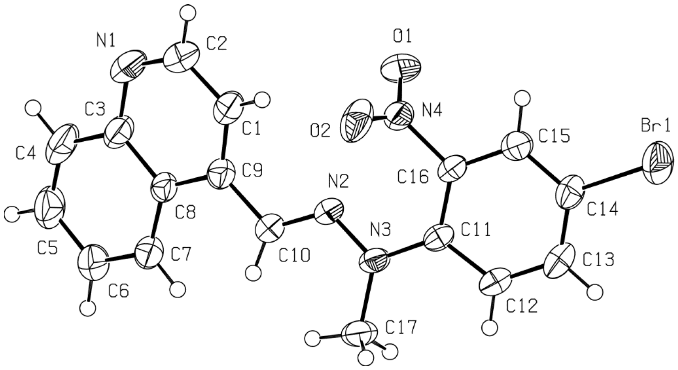


**Figure 1**
An *ORTEP*-3 (Farrugia, 1997) drawing of the title compound, showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 50% probability level.

**Table 2**
Hydrogen-bonding geometry (Å, °).

| *D*—H⋯*A* | *D*—H | H⋯*A* | *D*⋯*A* | *D*—H⋯*A* |
|---|---|---|---|---|
| C13—H13*A*⋯N1$^i$ | 0.93 | 2.53 | 3.424 (5) | 162 |

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

Some H atoms were located in a difference map and were refined freely with isotropic displacement parameters; C---H bond lengths are in the range 0.88 (4)–1.00 (4) Å. Others were positioned geometrically and refined riding on their parent atoms (C—H = 0.93 Å).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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