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

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Key indicators

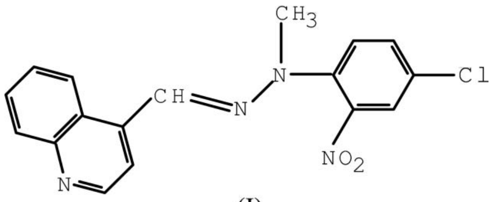
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.044
wR factor = 0.067
Data-to-parameter ratio = 13.9

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

In the title compound, $C_{17}H_{13}ClN_4O_2$, the dihedral angle between the benzene and quinoline ring systems is 20.95 (4)°. C—H⋯O and C—H⋯N hydrogen bonds, as well as π–π stacking interactions, are observed in the crystal structure.

Comment

Hydrazones, the most important derivatives of carboxaldehyde, are widely used both in organic syntheses and in industrial work because of their reaction abilities, such as ring closure, coupling, *etc*. Moreover, conjugated unsaturated ring systems and their derivatives containing nitrogen are biologically important compounds, which are being tested as potential anticancer agents and active materials against the causative virus of AIDS. As a result of their anti-HIV and anticancer activities, the chemical and pharmacological properties of hydrazones have been studied extensively (Kirk–Othmer Encyclopedia of Chemical Technology, 1980; Gürsoy *et al.*, 1997). The structures of the newly synthesized compounds have been evaluated by elemental analysis, UV, IR, ^{1}H NMR and mass spectroscopic techniques (Kaban & Aydemir, 1992). We report here the crystal structure of the title compound, (I).



(I)

The X-ray analysis shows that the bond distances and angles in (I) (Fig. 1) are within normal ranges (Table 1). The C1—Cl1 bond distance agrees with the value of 1.731 (7) Å reported by Karadayı *et al.* (2005). The N2—N3 and N3—C4 bond distances in (I) are comparable to those in *N*-(4-bromo-2-nitrophenyl)-*N*-methyl-*N*′-(quinolin-4-ylmethylene)hydrazine [1.359 (3) and 1.393 (4) Å; Öztürk *et al.*, 2003] and thiophene-2-carbaldehyde *N*-(2,4-dinitrophenyl)-*N*-methylhydrazone [1.371 (2) and 1.375 (2) Å; Aygün *et al.*, 1998]. The N3—C7 bond distance is nearly equal to the value of 1.455 (2) Å reported by Aygün *et al.* (1998). The bond distances and angles in the benzene and quinoline ring systems are in agreement with the values reported by Öztürk *et al.* (2003). The torsion angle about the N═N bond, C4—N3═N2—C8, is −175.6 (2)°.

The C8/N2/N3/C7 plane is rotated 13.57 (6)° from the plane of the C1–C6 ring and 15.39 (5)° from the plane of the

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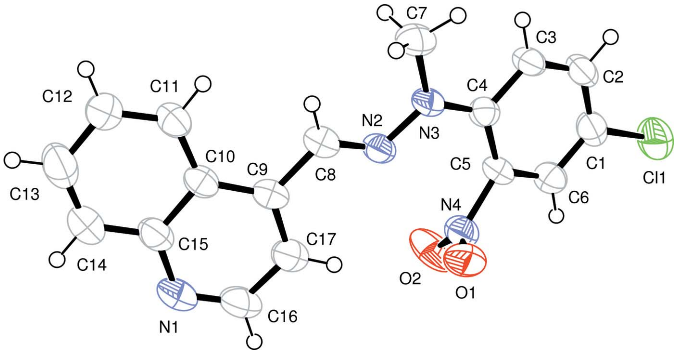

Figure 1
An *ORTEP-3* (Farrugia, 1997) drawing of (I), showing the atomic numbering scheme and 50% probability displacement ellipsoids.

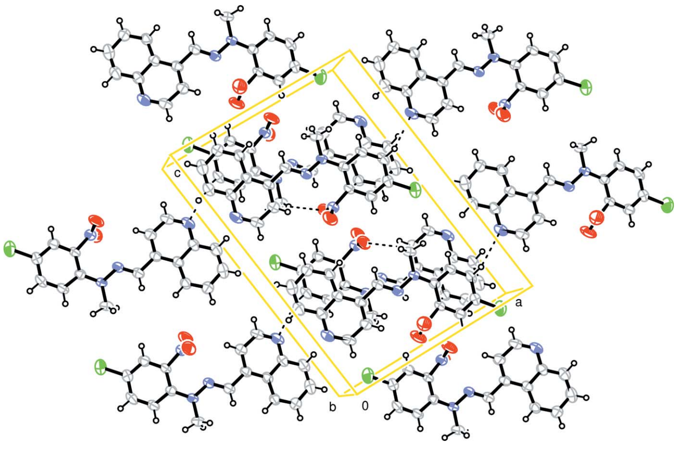

Figure 2
The crystal packing of (I), viewed approximately along the *b* axis. Hydrogen bonds are shown as dashed lines.

quinoline ring system. The dihedral angle between the two rings is 20.95 (4)°, and the C9—C8—N2—N3 and C4—N3—N2—C8 torsion angles are −177.9 (2) and −175.6 (2)°, respectively.

The crystal packing is stabilized by C—H···O and C—H···N hydrogen bonds (Table 2 and Fig.2) and by π–π stacking interactions between the C1–C6 ring and the C10–C15 benzene ring of the symmetry-related molecule at $(1 - x, y - \frac{1}{2}, \frac{1}{2} - z)$ [centroid–centroid distance 3.642 (2) Å].

Experimental

Quinoline-4-carboxaldehyde (1.0 mmol) was dissolved in hot absolute ethanol (7 ml) and an equimolar amount of *N*-(4-chloro-2-nitrophenyl)-*N*-methylhydrazine dissolved in hot absolute ethanol (10 ml) was added. The mixture was refluxed for 3 h. The crude product which precipitated on cooling was recrystallized from ethanol (yield 92%, m.p. 446–447 K). UV (CHCl$_3$): λ_{max} 249.5, 350.8 nm; IR (KBr): γ 3100–3000, 2990–2920, 1595, 1485 and 1365, 1110, 745 cm^{-1}; ^{1}H NMR (DMSO-d_6, 200 MHz): δ 3.68 (*s*, N—CH$_3$, 3H), 7.55–8.92 (*m*, ArH and CH, 10H); MS: *m*/*z* 340 (M^+), 294 (*M*-46), 155 (*M*-185), 129 (*M*-211), 101 (*M*-239). Analysis $C_{17}H_{13}ClN_4O_2$ requires: C 59.91, H 3.84, N 16.44%; found: C 59.86, H 3.86, N 16.12% (Kaban & Aydemir, 1992).

Crystal data

$C_{17}H_{13}ClN_4O_2$
M_r = 340.76
Monoclinic, $P2_1/c$
a = 12.5083 (15) Å
b = 6.9995 (5) Å
c = 17.885 (2) Å
β = 96.988 (10)°
V = 1554.2 (3) Å^3
Z = 4
D_x = 1.456 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6356 reflections
θ = 1.6–25.7°
μ = 0.26 mm^{-1}
T = 296 (2) K
Prism, yellow
0.50 × 0.42 × 0.32 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.918, T_{max} = 0.975
21401 measured reflections
3049 independent reflections
1276 reflections with $I > 2\sigma(I)$
R_{int} = 0.149
θ_{max} = 26.0°
$h = -15 \rightarrow 15$
$k = -8 \rightarrow 8$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.067
S = 0.94
3049 reflections
219 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0033P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.00128 (8)

Table 1
Selected geometric parameters (Å, °).

Cl1—C1	1.725 (3)	N2—N3	1.367 (2)
O1—N4	1.212 (3)	N3—C4	1.386 (3)
O2—N4	1.213 (3)	N3—C7	1.459 (3)
N1—C16	1.298 (3)	N4—C5	1.487 (3)
N1—C15	1.374 (3)	C8—C9	1.482 (3)
N2—C8	1.276 (3)		
C8—N2—N3	120.7 (2)	N3—C4—C3	121.3 (2)
N2—N3—C4	115.8 (2)	C6—C5—N4	113.0 (2)
N2—N3—C7	120.2 (2)	C4—C5—N4	123.3 (3)
C4—N3—C7	123.3 (2)	N2—C8—C9	119.1 (2)
O1—N4—O2	125.6 (2)	C11—C10—C9	125.3 (2)
O1—N4—C5	117.7 (2)	N1—C15—C14	118.1 (3)
O2—N4—C5	116.7 (3)	N1—C15—C10	122.4 (3)
N3—C4—C5	123.8 (2)	N1—C16—C17	125.3 (3)
N2—N3—C4—C3	162.8 (2)	O1—N4—C5—C4	−66.9 (4)
O1—N4—C5—C6	107.7 (3)	O2—N4—C5—C4	115.2 (3)
O2—N4—C5—C6	−70.1 (3)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···N1^i	0.93	2.57	3.420 (3)	153
C7—H7A···O1^{ii}	0.96	2.60	3.468 (3)	151

Symmetry codes: (i) $x + 1, y, z$; (ii) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$.

H atoms were placed in idealized positions and allowed to ride on their parent atoms, with C—H distances of 0.93 (aromatic) or 0.96 Å (methyl). The U$_{iso}$(H) values were set equal to 1.2U_{eq} of the carrier atom, or to 1.5U_{eq} of the carrier atom for methyl H atoms.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLUTON* (Spek, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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