



Crystal structure of 2-(4-methylpiperazin-1-yl)quinoline-3-carbaldehyde

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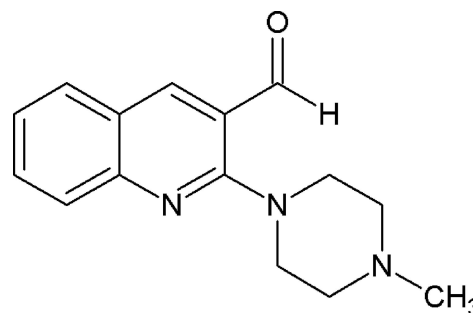
In the title compound, C₁₅H₁₇N₃O, the aldehyde group is twisted relative to the quinoline group by 17.6 (2)° due to the presence of a bulky piperazinyl group in the *ortho* position. The piperazine N atom attached to the aromatic ring is sp³-hybridized and the dihedral angle between the mean planes through the the six piperazine ring atoms and through the quinoline ring system is 40.59 (7)°. Both piperazine substituents are in equatorial positions.

Keywords: crystal structure; quinolines; piperazines.

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1. Related literature

For biological activity of quinoline derivatives, see: Nasveld *et al.* (2005); Eswaran *et al.* (2009); Leatham *et al.* (1983); Muruganantham *et al.* (2004); Maguire *et al.* (1994); Wilson *et al.* (1992); Streckowski *et al.* (1991). For photonic and electronic properties of poly-substituted quinolines, see: Gyoten *et al.* (2003).



2. Experimental

2.1. Crystal data

C₁₅H₁₇N₃O
M_r = 255.32
Monoclinic, P2₁/n
a = 12.3282 (4) Å
b = 5.8935 (2) Å
c = 18.9202 (7) Å
β = 103.591 (2)°

V = 1336.18 (8) Å³
Z = 4
Cu Kα radiation
μ = 0.65 mm⁻¹
T = 296 K
0.28 × 0.26 × 0.24 mm

2.2. Data collection

Bruker APEXII CCD
diffractometer
Absorption correction: multi-scan
(SADABS; Bruker, 2009)
T_{min} = 0.838, T_{max} = 0.859

9762 measured reflections
2181 independent reflections
1859 reflections with I > 2σ(I)
R_{int} = 0.048

2.3. Refinement

R[F² > 2σ(F²)] = 0.054
wR(F²) = 0.162
S = 1.06
2181 reflections

173 parameters
H-atom parameters constrained
Δρ_{max} = 0.22 e Å⁻³
Δρ_{min} = -0.24 e Å⁻³

Data collection: APEX2 (Bruker, 2009); cell refinement: APEX2 and SAINT-Plus (Bruker, 2009); data reduction: SAINT-Plus and XPREP (Bruker, 2009); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: Mercury (Macrae *et al.*, 2008); software used to prepare material for publication: SHELXL97.

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Supporting information for this paper is available from the IUCr electronic archives (Reference: GK2647).

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S1. Introduction

Quinoline and its derivatives have been well known in pharmaceutical chemistry because of their wide spectrum of biological activities and their presence in naturally occurring compounds. They have been shown to possess antimalarial (Nasveld *et al.*, 2005), antibiotic (Eswaran *et al.*, 2009), anticancer (Denny *et al.*, 1983), anti-inflammatory (Muruganantham *et al.*, 2004), antihypertensive (Maguire *et al.*, 1994), tyrosinase PDGF-RTK inhibition (Wilson *et al.*, 1992) and anti-HIV properties (Strekowski *et al.*, 1991). In addition, polysubstituted quinoline can achieve hierarchical self-assembly into variety of meso and nano structures with enhanced photonic and electronic properties (Gyoten *et al.*, 2003). In this view the title compound was synthesized to study its crystal structure.

S2. Experimental

S2.1. Synthesis and crystallization

2-Chloroquinoline-3-carbaldehyde (0.42 g, 0.00351 mmol), *N*-methyl piperazine (0.14 g, 0.00351 mmol) and anhydrous K₂CO₃ (1.0 g, 0.002920 mmol) were refluxed for 24 hrs in DMF. The progress of the reaction was monitored by thin layer chromatography. After the completion of the reaction, the reaction mixture was poured into water and extracted to ethyl acetate. The organic layer was washed with water, dried and concentrated under vacuum using rotary evaporator. Single crystals of the title compound were obtained by slow evaporation of the ethyl acetate solution at room temperature (27°C).

S2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. All H atoms were positioned with idealized geometry using a riding model with C—H = 0.93–0.97 Å. All H atoms were refined with isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent C atom).

S3. Results and discussion

The crystal packing of the compound does not feature any specific strong or weak intermolecular interactions.

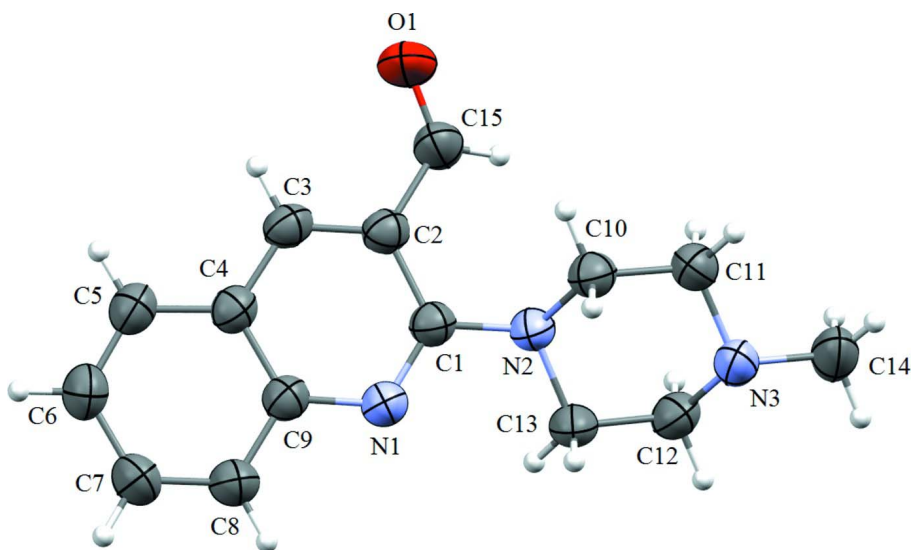


Figure 1

Molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

2-(4-Methylpiperazin-1-yl)quinoline-3-carbaldehyde

Crystal data

$C_{15}H_{17}N_3O$

$M_r = 255.32$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1n$

$a = 12.3282\ (4)\ \text{\AA}$

$b = 5.8935\ (2)\ \text{\AA}$

$c = 18.9202\ (7)\ \text{\AA}$

$\beta = 103.591\ (2)^\circ$

$V = 1336.18\ (8)\ \text{\AA}^3$

$Z = 4$

$F(000) = 544$

Data collection

Bruker APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ϕ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2009)

$T_{\min} = 0.838$, $T_{\max} = 0.859$

Prism

$D_x = 1.269\ \text{Mg m}^{-3}$

Melting point: 384 K

Cu $K\alpha$ radiation, $\lambda = 1.54178\ \text{\AA}$

Cell parameters from 143 reflections

$\theta = 3.9\text{--}64.5^\circ$

$\mu = 0.65\ \text{mm}^{-1}$

$T = 296\ \text{K}$

Prism, colourless

$0.28 \times 0.26 \times 0.24\ \text{mm}$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.054$

$wR(F^2) = 0.162$

$S = 1.06$

2181 reflections

173 parameters

0 restraints

9762 measured reflections

2181 independent reflections

1859 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.048$

$\theta_{\max} = 64.5^\circ$, $\theta_{\min} = 3.9^\circ$

$h = -14 \rightarrow 14$

$k = -6 \rightarrow 6$

$l = -21 \rightarrow 21$

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.1151P)^2 + 0.0654P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 0.22 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.24 \text{ e } \text{\AA}^{-3}$$

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness 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 threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.44347 (10)	0.7887 (2)	0.60405 (6)	0.0446 (4)
N2	0.31637 (10)	0.4894 (2)	0.58318 (6)	0.0443 (4)
N3	0.15374 (11)	0.2821 (2)	0.46867 (7)	0.0492 (4)
O1	0.33529 (13)	0.4038 (3)	0.79890 (7)	0.0800 (5)
C1	0.38623 (12)	0.6362 (3)	0.63074 (7)	0.0415 (4)
C2	0.39605 (12)	0.6082 (3)	0.70773 (8)	0.0435 (4)
C3	0.46317 (13)	0.7537 (3)	0.75426 (8)	0.0456 (4)
H3	0.4690	0.7410	0.8040	0.055*
C4	0.52382 (12)	0.9229 (3)	0.72789 (8)	0.0429 (4)
C5	0.59503 (14)	1.0786 (3)	0.77313 (9)	0.0517 (5)
H5	0.6035	1.0718	0.8232	0.062*
C6	0.65119 (15)	1.2380 (3)	0.74416 (10)	0.0571 (5)
H6	0.6980	1.3397	0.7744	0.068*
C7	0.63874 (16)	1.2497 (3)	0.66853 (10)	0.0580 (5)
H7	0.6768	1.3606	0.6490	0.070*
C8	0.57153 (14)	1.1003 (3)	0.62334 (9)	0.0516 (5)
H8	0.5648	1.1094	0.5734	0.062*
C9	0.51232 (12)	0.9326 (3)	0.65156 (8)	0.0430 (4)
C10	0.19665 (13)	0.5037 (3)	0.58101 (8)	0.0477 (4)
H10A	0.1659	0.6413	0.5560	0.057*
H10B	0.1861	0.5100	0.6302	0.057*
C11	0.13648 (13)	0.3007 (3)	0.54226 (9)	0.0517 (5)
H11A	0.1635	0.1642	0.5694	0.062*
H11B	0.0573	0.3142	0.5399	0.062*
C12	0.27299 (14)	0.2697 (3)	0.47221 (9)	0.0522 (5)
H12A	0.2846	0.2582	0.4234	0.063*
H12B	0.3038	0.1347	0.4988	0.063*
C13	0.33266 (14)	0.4768 (3)	0.50926 (8)	0.0516 (5)
H13A	0.4117	0.4670	0.5106	0.062*
H13B	0.3031	0.6123	0.4824	0.062*
C14	0.09682 (18)	0.0831 (4)	0.43236 (9)	0.0660 (6)
H14A	0.1106	0.0708	0.3846	0.099*

H14B	0.0181	0.0973	0.4285	0.099*
H14C	0.1242	-0.0501	0.4600	0.099*
C15	0.34545 (14)	0.4132 (3)	0.73732 (9)	0.0561 (5)
H15	0.3201	0.2913	0.7067	0.067*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0470 (7)	0.0482 (8)	0.0386 (7)	-0.0039 (6)	0.0102 (6)	0.0002 (5)
N2	0.0423 (7)	0.0538 (9)	0.0381 (7)	-0.0054 (6)	0.0119 (5)	-0.0038 (6)
N3	0.0541 (8)	0.0505 (9)	0.0400 (7)	-0.0114 (6)	0.0047 (6)	0.0021 (6)
O1	0.0885 (10)	0.1024 (13)	0.0487 (8)	-0.0299 (8)	0.0151 (7)	0.0178 (7)
C1	0.0400 (8)	0.0462 (9)	0.0389 (8)	0.0020 (6)	0.0102 (6)	0.0015 (6)
C2	0.0408 (8)	0.0503 (10)	0.0392 (8)	0.0025 (6)	0.0088 (6)	0.0038 (7)
C3	0.0456 (9)	0.0556 (10)	0.0348 (8)	0.0055 (7)	0.0076 (6)	0.0042 (7)
C4	0.0402 (8)	0.0467 (9)	0.0407 (8)	0.0049 (6)	0.0077 (6)	0.0001 (6)
C5	0.0529 (10)	0.0568 (11)	0.0431 (8)	0.0002 (8)	0.0064 (7)	-0.0061 (7)
C6	0.0583 (10)	0.0557 (11)	0.0549 (10)	-0.0102 (8)	0.0086 (8)	-0.0100 (8)
C7	0.0640 (11)	0.0530 (11)	0.0576 (10)	-0.0139 (8)	0.0155 (8)	-0.0007 (8)
C8	0.0574 (10)	0.0544 (11)	0.0439 (8)	-0.0055 (8)	0.0142 (7)	0.0014 (7)
C9	0.0427 (8)	0.0458 (10)	0.0402 (8)	0.0020 (6)	0.0090 (6)	-0.0002 (6)
C10	0.0450 (9)	0.0568 (11)	0.0420 (8)	-0.0011 (7)	0.0117 (6)	0.0002 (7)
C11	0.0481 (9)	0.0603 (11)	0.0465 (9)	-0.0097 (7)	0.0108 (7)	0.0025 (7)
C12	0.0614 (10)	0.0561 (11)	0.0402 (8)	-0.0041 (7)	0.0143 (7)	-0.0042 (7)
C13	0.0513 (9)	0.0644 (11)	0.0424 (8)	-0.0112 (8)	0.0180 (7)	-0.0056 (7)
C14	0.0820 (13)	0.0605 (12)	0.0491 (10)	-0.0239 (9)	0.0026 (9)	0.0008 (8)
C15	0.0567 (10)	0.0634 (12)	0.0449 (9)	-0.0096 (8)	0.0050 (7)	0.0107 (8)

Geometric parameters (Å, °)

N1—C1	1.315 (2)	C6—H6	0.9300
N1—C9	1.375 (2)	C7—C8	1.364 (3)
N2—C1	1.391 (2)	C7—H7	0.9300
N2—C13	1.4607 (18)	C8—C9	1.406 (2)
N2—C10	1.4693 (19)	C8—H8	0.9300
N3—C14	1.454 (2)	C10—C11	1.505 (2)
N3—C12	1.458 (2)	C10—H10A	0.9700
N3—C11	1.461 (2)	C10—H10B	0.9700
O1—C15	1.202 (2)	C11—H11A	0.9700
C1—C2	1.442 (2)	C11—H11B	0.9700
C2—C3	1.361 (2)	C12—C13	1.510 (2)
C2—C15	1.479 (2)	C12—H12A	0.9700
C3—C4	1.406 (2)	C12—H12B	0.9700
C3—H3	0.9300	C13—H13A	0.9700
C4—C5	1.411 (2)	C13—H13B	0.9700
C4—C9	1.419 (2)	C14—H14A	0.9600
C5—C6	1.357 (3)	C14—H14B	0.9600
C5—H5	0.9300	C14—H14C	0.9600

C6—C7	1.404 (3)	C15—H15	0.9300
C1—N1—C9	118.40 (12)	N2—C10—C11	110.18 (13)
C1—N2—C13	116.58 (12)	N2—C10—H10A	109.6
C1—N2—C10	116.60 (12)	C11—C10—H10A	109.6
C13—N2—C10	109.84 (11)	N2—C10—H10B	109.6
C14—N3—C12	110.53 (15)	C11—C10—H10B	109.6
C14—N3—C11	110.40 (13)	H10A—C10—H10B	108.1
C12—N3—C11	109.34 (12)	N3—C11—C10	111.00 (13)
N1—C1—N2	118.89 (12)	N3—C11—H11A	109.4
N1—C1—C2	122.74 (14)	C10—C11—H11A	109.4
N2—C1—C2	118.32 (13)	N3—C11—H11B	109.4
C3—C2—C1	118.36 (14)	C10—C11—H11B	109.4
C3—C2—C15	119.41 (14)	H11A—C11—H11B	108.0
C1—C2—C15	121.90 (15)	N3—C12—C13	110.89 (14)
C2—C3—C4	120.69 (14)	N3—C12—H12A	109.5
C2—C3—H3	119.7	C13—C12—H12A	109.5
C4—C3—H3	119.7	N3—C12—H12B	109.5
C3—C4—C5	123.55 (14)	C13—C12—H12B	109.5
C3—C4—C9	117.08 (14)	H12A—C12—H12B	108.0
C5—C4—C9	119.36 (15)	N2—C13—C12	108.90 (13)
C6—C5—C4	120.59 (15)	N2—C13—H13A	109.9
C6—C5—H5	119.7	C12—C13—H13A	109.9
C4—C5—H5	119.7	N2—C13—H13B	109.9
C5—C6—C7	120.09 (16)	C12—C13—H13B	109.9
C5—C6—H6	120.0	H13A—C13—H13B	108.3
C7—C6—H6	120.0	N3—C14—H14A	109.5
C8—C7—C6	120.80 (16)	N3—C14—H14B	109.5
C8—C7—H7	119.6	H14A—C14—H14B	109.5
C6—C7—H7	119.6	N3—C14—H14C	109.5
C7—C8—C9	120.59 (15)	H14A—C14—H14C	109.5
C7—C8—H8	119.7	H14B—C14—H14C	109.5
C9—C8—H8	119.7	O1—C15—C2	123.51 (18)
N1—C9—C8	118.78 (13)	O1—C15—H15	118.2
N1—C9—C4	122.64 (14)	C2—C15—H15	118.2
C8—C9—C4	118.56 (15)		
