Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

OPEN ACCESS

ISSN 2056-9890

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

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Received 16 October 2015; accepted 26 October 2015

Edited by M. Gdaniec, Adam Mickiewicz University, Poland

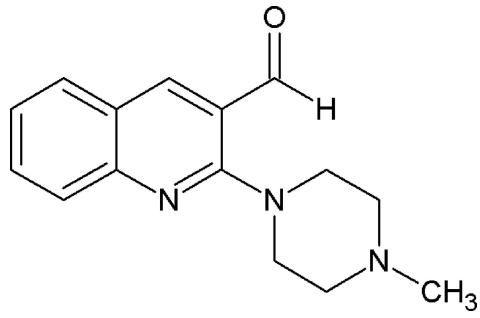
In the title compound, $C_{15}H_{17}N_3O$, the aldehyde group is twisted relative to the quinoline group by17.6 (2)° due to the presence of a bulky piperazinyl group in the *ortho* position. The piperazine N atom attached to the aromatic ring is sp^3-hybridized and the dihedral angle between the mean 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.

CCDC reference: 1433198

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); Strekowski *et al.* (1991). For photonic and electronic properties of poly-substituted quinolines, see: Gyoten *et al.* (2003).



2. Experimental

2.1. Crystal data

$C_{15}H_{17}N_3O$
M_r = 255.32
Monoclinic, $P2_1/n$
a = 12.3282 (4) Å
b = 5.8935 (2) Å
c = 18.9202 (7) Å
β = 103.591 (2)°
V = 1336.18 (8) Å^3
Z = 4
Cu $K\alpha$ 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 $I > 2\sigma(I)$
R_{int} = 0.048

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.162
S = 1.06
2181 reflections
173 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −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*.

Acknowledgements

The authors are thankful to the Institution of Excellence, Vijnana Bhavana, University of Mysore, Mysuru, for providing the single-crystal X-ray diffraction facility. RND, SS, PAS and DBAK are thankful to Tumkur University for providing laboratory facilities to carry out this work.

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

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supporting information

Acta Cryst. (2015). E**71**, o900–o901 [https://doi.org/10.1107/S2056989015020186]

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

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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), tyrokinase PDGF-RTK inhibition (Wilson *et al.*, 1992) and anti-HIV properties (Strekowski *et al.*, 1991). In addition, polysubstituted quinoline can achieve hierchical 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 K_2CO_3 (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^{o}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 Ueq 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.

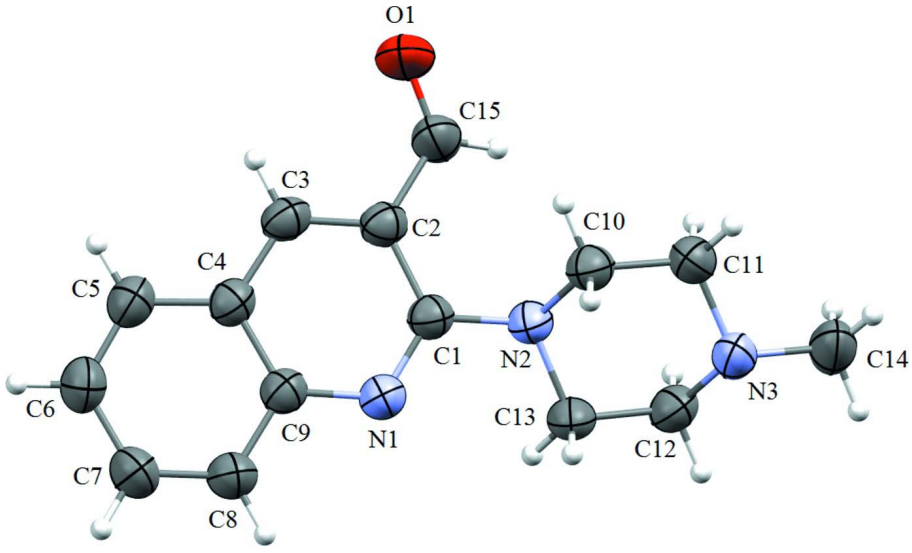

Figure 1

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 2yn
$a = 12.3282$ (4) Å
$b = 5.8935$ (2) Å
$c = 18.9202$ (7) Å
$\beta = 103.591$ (2)°
$V = 1336.18$ (8) Å^3
$Z = 4$
$F(000) = 544$

Prism
$D_x = 1.269$ Mg m^{-3}
Melting point: 384 K
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 143 reflections
$\theta = 3.9$–$64.5°$
$\mu = 0.65$ mm^{-1}
$T = 296$ K
Prism, colourless
0.28 × 0.26 × 0.24 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min} = 0.838$, $T_{max} = 0.859$

9762 measured reflections
2181 independent reflections
1859 reflections with $I > 2\sigma(I)$
$R_{int} = 0.048$
$\theta_{max} = 64.5°$, $\theta_{min} = 3.9°$
$h = -14 \rightarrow 14$
$k = -6 \rightarrow 6$
$l = -21 \rightarrow 21$

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

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

$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{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, º)

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)		