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Crystal structure of 4-methoxyquinazoline

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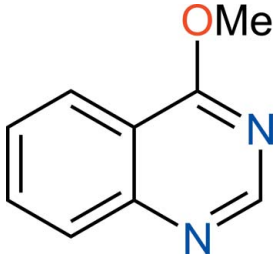
The title compound, $C_9H_8N_2O$, is almost planar, with the C atom of the methoxy group deviating from the mean plane of the quinazoline ring system (r.m.s. deviation = 0.011 Å) by 0.068 (4) Å. In the crystal, molecules form π–π stacks parallel to the *b*-axis direction [centroid–centroid separation = 3.5140 (18) Å], leading to a herringbone packing arrangement.

Keywords: crystal structure; 4-methoxyquinazoline; quinazoline derivatives; π–π stacks; herringbone packing.

CCDC reference: 1034363

1. Related literature

For the synthesis of quinazoline derivatives, see: Bogert & May (1909); Smith *et al.* (2005); Wang *et al.* (2010); Yang *et al.* (2010); Han *et al.* (2012). For the crystal structures of related compounds, see Alshammari *et al.* (2014); Derabli *et al.* (2013); Gao *et al.* (2012); Huang & Tan (2012); Jia *et al.* (2011).



2. Experimental

2.1. Crystal data

$C_9H_8N_2O$
M_r = 160.17
Monoclinic, $P2_1$
a = 6.9590 (6) Å
b = 4.0517 (3) Å
c = 13.5858 (12) Å
β = 91.754 (8)°
V = 382.88 (6) Å^3
Z = 2
Cu *K*α radiation
μ = 0.77 mm^{-1}
T = 150 K
0.57 × 0.12 × 0.08 mm

2.2. Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas) diffractometer
Absorption correction: gaussian (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.641, T_{max} = 0.895
2166 measured reflections
1435 independent reflections
1311 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.159
S = 1.08
1435 reflections
110 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *CHEMDRAW Ultra* (Cambridge Soft, 2001).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7317).

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supporting information

Acta Cryst. (2014). E**70**, o1279 [doi:10.1107/S1600536814025082]

Crystal structure of 4-methoxyquinazoline

Gamal A. El-Hiti, Keith Smith, Amany S. Hegazy, Mohammed B. Alshammari and Benson M. Kariuki

S1. Chemical context

S2. Structural commentary

Copper-catalyzed processes offer convenient approaches to various quinazoline derivatives starting from (2-bromophenyl)methylamines, 2-aminobenzylamines or 2-bromobenzonitriles (Wang *et al.*, 2010; Yang *et al.*, 2010; Han *et al.*, 2012). For ring substitution and modification of 4-methoxyquinazolines, see: Smith *et al.* (2005). 4-Methoxyquinazoline was synthesized in 81% yield from reaction of quinazoline-4(3H)-thione with iodomethane in aqueous methanol containing potassium hydroxide at room temperature for 24 h (Bogert & May, 1909). For the X-ray structures for related compounds, see Alshammari *et al.* (2014); Derabli *et al.* (2013); Gao *et al.* (2012); Huang & Tan (2012); Jia *et al.* (2011).

The asymmetric unit consists of one molecule of $C_9H_8N_2O$ (Fig. 1). The molecule is almost planar apart from the methyl hydrogen atoms, with C9 deviating from the least squares plane of the quinazoline group by 0.068 (4)Å. The molecules form π-π stacks parallel to the *b*-axis leading to a herring-bone pattern in the crystal structure (Fig. 2).

S3. Supramolecular features

S4. Database survey

S5. Synthesis and crystallization

To a solution of quinazoline-4(3*H*)-thione (4.9 g, 30.2 mmol) in a 1:1 mixture of methanol and water (50 ml) containing potassium hydroxide (3.0 g), was added iodomethane (5.7 g, 40.1 mmol) at room temperature. The reaction mixture was stirred for 24 h, then methanol was removed under reduced pressure and the remaining aqueous layer was extracted with diethyl ether (2 × 20 ml). The organic layer was separated, washed with water (2 × 10 ml), dried ($MgSO_4$), and evaporated under reduced pressure. The residue obtained was purified by column chromatography (silica gel, diethyl ether–hexane, 1:1) to give 4-methoxyquinazoline (3.9 g, 24.4 mmol, 81%) as a white solid. Crystallization from a mixture of ethyl acetate and diethyl ether (1:3 by volume) gave the title compound as colorless needles. mp 34–35°C [lit. 35.4°C; Bogert & May, (1909)]. ^{1}H NMR (400 MHz, $CDCl_3$, δ p.p.m.): 8.77 (s, 1 H, H-2), 8.08 (dd, *J* = 1, 8 Hz, 1 H, H-8), 7.88 (dd, *J* = 1,8 Hz, 1 H, H-5), 7.76 (app. dt, *J* = 1, 8 Hz, 1 H, H-7), 7.50 (app. t, *J* = 8 Hz, 1 H, H-6), 4.13 (s, 3 H, OCH_3). ^{13}C NMR (100 MHz, $CDCl_3$, δ, p.p.m.): 167.4 (*s*, C-4), 154.6 (*d*, C-2), 151.1 (*s*, C-8a), 133.8 (*d*, C-7), 128.0 (*d*, C-8), 127.3 (*d*, C-5), 123.8 (*d*, C-6), 116.9 (*s*, C-4a), 54.6 (*q*, OCH_3). EI–MS (*m/z*, %): 160 (M^+, 68), 131 (32), 130 (30), 103 (100), 90 (22), 76 (28), 63 (21). CI–MS (*m/z*, %): 161 (MH^+, 100). HRMS (CI): calculated for $C_9H_9N_2O$ [*MH*] 161.0709; found, 161.0708.

S6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

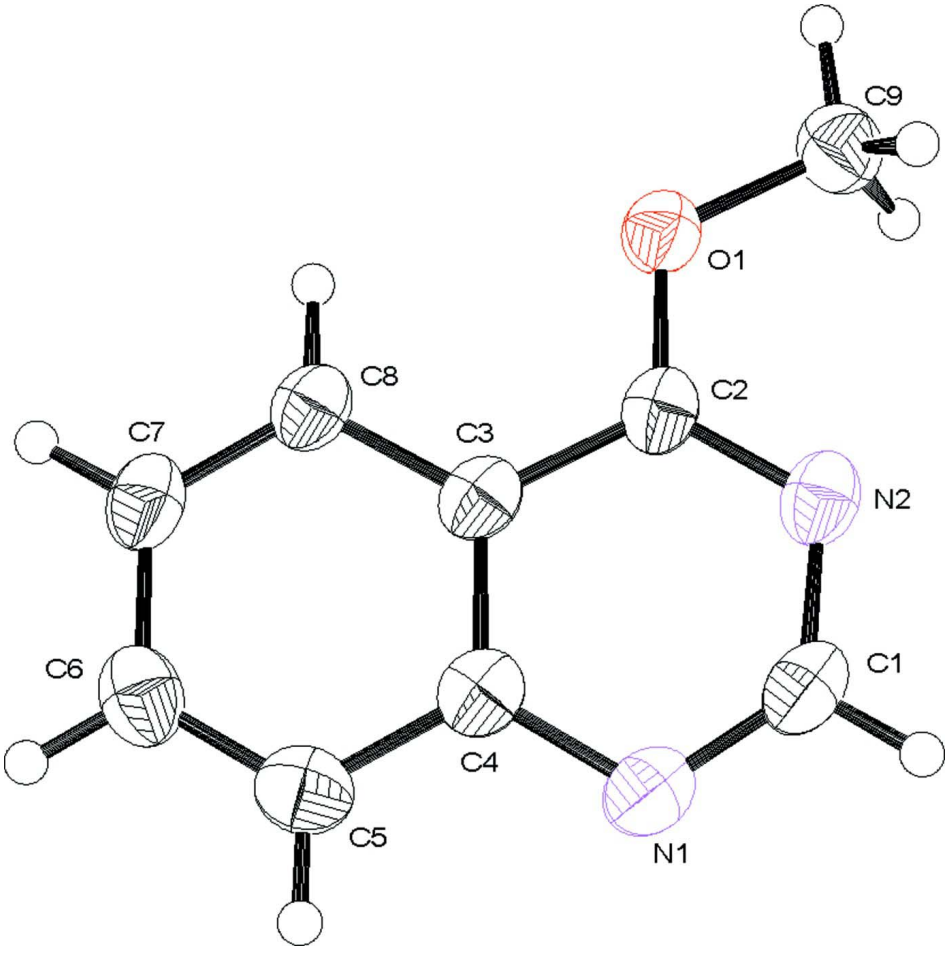

Figure 1

The title molecule showing 50% probability displacement ellipsoids.

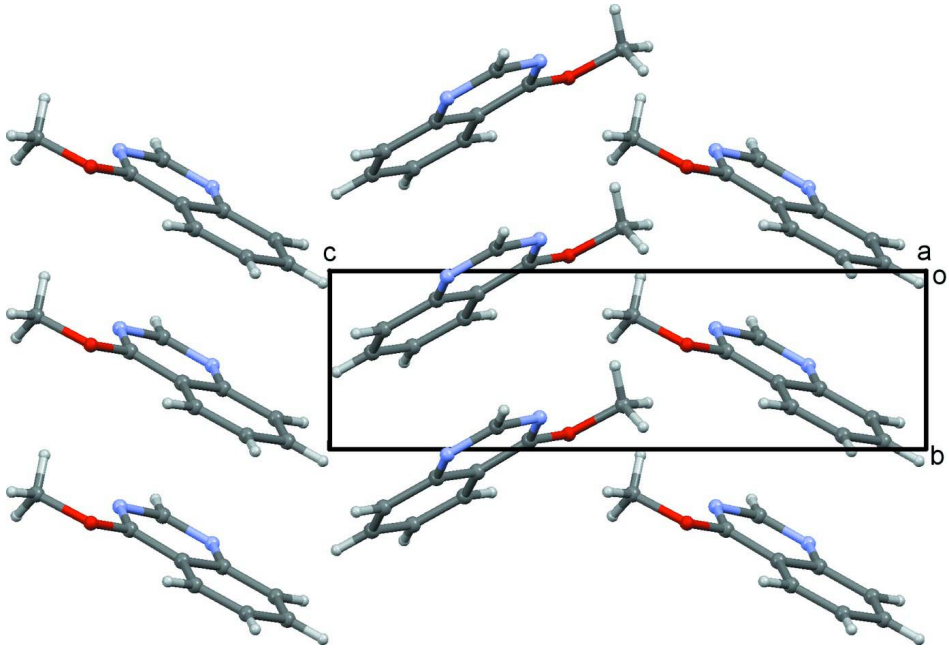

Figure 2

Crystal packing viewed down the *a* axis.

4-Methoxyquinazoline

Crystal data

$C_9H_8N_2O$
$M_r = 160.17$
Monoclinic, $P2_1$
$a = 6.9590\ (6)$ Å
$b = 4.0517\ (3)$ Å
$c = 13.5858\ (12)$ Å
$\beta = 91.754\ (8)°$
$V = 382.88\ (6)$ Å^3
$Z = 2$
$F(000) = 168$
$D_x = 1.389$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 1311 reflections
$\theta = 3.3–67.7°$
$\mu = 0.77$ mm^{-1}
$T = 150$ K
Needle, colourless
0.57 × 0.12 × 0.08 mm

Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas) diffractometer
ω scans
Absorption correction: gaussian (*CrysAlis PRO*; Agilent, 2014)
$T_{min} = 0.641$, $T_{max} = 0.895$
2166 measured reflections
1435 independent reflections
1311 reflections with $I > 2\sigma(I)$
$R_{int} = 0.040$
$\theta_{max} = 74.6°$, $\theta_{min} = 3.3°$
$h = -8\rightarrow8$
$k = -4\rightarrow5$
$l = -11\rightarrow16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.159$
$S = 1.08$
1435 reflections
110 parameters
1 restraint
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.110P)^2 + 0.0483P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

Special details

Experimental. Absorption correction: CrysAlisPro, Agilent Technologies, Version 1.171.37.33 (release 27-03-2014 CrysAlis171 .NET) (compiled Mar 27 2014,17:12:48) Numerical absorption correction based on gaussian integration over a multifaceted crystal model Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.1179 (5)	0.8736 (9)	0.7198 (2)	0.0403 (8)
H1	−0.2391	0.7831	0.7078	0.048*
C2	0.1829 (4)	0.9515 (7)	0.6650 (2)	0.0330 (7)
C3	0.2338 (4)	1.1283 (7)	0.7527 (2)	0.0330 (7)
C4	0.0855 (4)	1.1608 (7)	0.8208 (2)	0.0349 (7)
C5	0.1265 (5)	1.3290 (8)	0.9096 (3)	0.0426 (8)
H5	0.0308	1.3526	0.9554	0.051*
C6	0.3034 (5)	1.4570 (8)	0.9292 (2)	0.0431 (8)
H6	0.3276	1.5675	0.9883	0.052*
C7	0.4520 (5)	1.4246 (10)	0.8608 (3)	0.0411 (7)
H7	0.5731	1.5127	0.8751	0.049*
C8	0.4169 (5)	1.2640 (7)	0.7739 (2)	0.0364 (7)
H8	0.5138	1.2440	0.7287	0.044*
C9	0.2674 (5)	0.7530 (9)	0.5079 (3)	0.0433 (8)
H9A	0.1669	0.8757	0.4745	0.065*
H9B	0.2226	0.5340	0.5215	0.065*
H9C	0.3771	0.7410	0.4669	0.065*
N1	−0.0945 (4)	1.0312 (7)	0.8028 (2)	0.0412 (7)
N2	0.0121 (4)	0.8237 (6)	0.6479 (2)	0.0378 (7)
O1	0.3208 (3)	0.9159 (6)	0.59930 (16)	0.0381 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0293 (13)	0.0351 (16)	0.0562 (19)	−0.0019 (13)	−0.0033 (12)	0.0037 (15)
C2	0.0334 (14)	0.0278 (15)	0.0374 (15)	0.0007 (13)	−0.0059 (11)	0.0054 (12)
C3	0.0310 (14)	0.0268 (14)	0.0408 (15)	0.0051 (11)	−0.0036 (12)	0.0073 (12)
C4	0.0356 (16)	0.0283 (15)	0.0405 (16)	0.0033 (13)	−0.0020 (12)	0.0071 (13)
C5	0.0473 (18)	0.0386 (17)	0.0423 (18)	0.0053 (15)	0.0052 (14)	0.0023 (15)
C6	0.0528 (19)	0.0364 (18)	0.0396 (17)	0.0030 (15)	−0.0075 (14)	−0.0008 (14)
C7	0.0367 (14)	0.0373 (16)	0.0486 (17)	0.0006 (14)	−0.0104 (12)	0.0020 (14)
C8	0.0327 (14)	0.0346 (16)	0.0416 (18)	0.0033 (13)	−0.0051 (12)	0.0057 (14)

C9	0.0445 (17)	0.0437 (18)	0.0414 (17)	0.0051 (16)	−0.0052 (13)	−0.0020 (14)
N1	0.0336 (13)	0.0372 (14)	0.0528 (17)	−0.0001 (11)	0.0023 (11)	0.0013 (13)
N2	0.0346 (13)	0.0336 (14)	0.0446 (14)	−0.0001 (10)	−0.0084 (11)	0.0044 (11)
O1	0.0346 (10)	0.0430 (13)	0.0367 (11)	−0.0004 (11)	−0.0016 (8)	−0.0004 (10)

Geometric parameters (Å, º)

C1—N1	1.302 (4)	C5—H5	0.9300
C1—N2	1.366 (4)	C6—C7	1.417 (5)
C1—H1	0.9300	C6—H6	0.9300
C2—N2	1.311 (4)	C7—C8	1.363 (5)
C2—O1	1.338 (4)	C7—H7	0.9300
C2—C3	1.425 (4)	C8—H8	0.9300
C3—C8	1.409 (4)	C9—O1	1.445 (4)
C3—C4	1.413 (4)	C9—H9A	0.9600
C4—N1	1.373 (4)	C9—H9B	0.9600
C4—C5	1.407 (5)	C9—H9C	0.9600
C5—C6	1.355 (5)		
N1—C1—N2	128.6 (3)	C7—C6—H6	119.6
N1—C1—H1	115.7	C8—C7—C6	119.8 (3)
N2—C1—H1	115.7	C8—C7—H7	120.1
N2—C2—O1	120.3 (3)	C6—C7—H7	120.1
N2—C2—C3	123.2 (3)	C7—C8—C3	120.0 (3)
O1—C2—C3	116.5 (2)	C7—C8—H8	120.0
C8—C3—C4	120.2 (3)	C3—C8—H8	120.0
C8—C3—C2	124.6 (3)	O1—C9—H9A	109.5
C4—C3—C2	115.2 (3)	O1—C9—H9B	109.5
N1—C4—C5	119.8 (3)	H9A—C9—H9B	109.5
N1—C4—C3	121.9 (3)	O1—C9—H9C	109.5
C5—C4—C3	118.2 (3)	H9A—C9—H9C	109.5
C6—C5—C4	120.9 (3)	H9B—C9—H9C	109.5
C6—C5—H5	119.6	C1—N1—C4	115.5 (3)
C4—C5—H5	119.6	C2—N2—C1	115.6 (3)
C5—C6—C7	120.8 (3)	C2—O1—C9	116.9 (2)
C5—C6—H6	119.6		