Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

OPEN ACCESS

ISSN 2056-9890

Crystal structure of 3-amino-2-ethylquinazolin-4(3*H*)-one

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Received 20 July 2015; accepted 31 July 2015

Edited by P. C. Healy, Griffith University, Australia

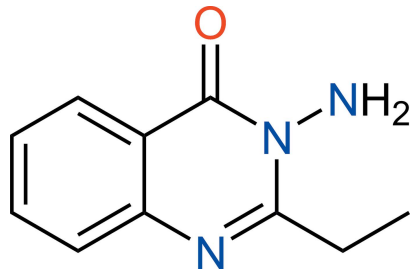
The molecule of the title compound, $C_{10}H_{11}N_3O$, is planar, including the ethyl group, as indicated by the N—C—C—C torsion angle of 1.5 (2)°. In the crystal, inversion-related molecules are stacked along the *a* axis. Molecules are oriented head-to-tail and display π–π interactions with a centroid-to-centroid distance of 3.6664 (8) Å. N—H···O hydrogen bonds between molecules generate a 'step' structure through formation of an $R_2^2(10)$ ring.

Keywords: crystal structure; 3-amino-2-ethylquinazolin-4(3*H*)-one; π–π interactions.

CCDC reference: 1416070

1. Related literature

For related compounds, see: Ma *et al.* (2013); Adib *et al.* (2012); Xu *et al.* (2012); Sasmal *et al.* (2012); Kumar *et al.* (2011); Rohini *et al.* (2010); Davies *et al.* (2010). For quinazolin-4(3*H*)-one ring-system modification through lithiation, see: Smith *et al.* (2004, 1996, 1995). For the crystal structures of related compounds, see: El-Hiti *et al.* (2014); Yang *et al.* (2009); Coogan *et al.* (1999).



2. Experimental

2.1. Crystal data

$C_{10}H_{11}N_3O$
M_r = 189.22
Triclinic, $P\overline{1}$
a = 7.0230 (5) Å
b = 7.6198 (7) Å
c = 9.7868 (6) Å
α = 69.709 (7)°
β = 89.242 (5)°
γ = 75.191 (7)°
V = 473.27 (7) Å^3
Z = 2
Cu $K\alpha$ radiation
μ = 0.73 mm^{-1}
T = 293 K
0.38 × 0.20 × 0.08 mm

2.2. Data collection

Agilent SuperNova Dual Source diffractometer with an Atlas detector
Absorption correction: Gaussian (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.741, T_{max} = 0.924
3303 measured reflections
1858 independent reflections
1657 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
Standard reflections: 0

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.196
S = 1.07
1858 reflections
136 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···O1^{i}	0.91 (2)	2.12 (2)	2.974 (2)	157.1 (19)

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

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, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *CHEMDRAW Ultra* (Cambridge Soft, 2001).

Acknowledgements

The authors extend their appreciation to the British Council, Riyadh, Saudi Arabia, for funding this research and to Cardiff University for continued support.

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HG5454).

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supporting information

Acta Cryst. (2015). E**71**, o650–o651 [https://doi.org/10.1107/S2056989015014450]

Crystal structure of 3-amino-2-ethylquinazolin-4(3*H*)-one

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

S1. Introduction

Quinazolines have various interesting biological applications (Sasmal *et al*., 2012; Rohini *et al.*, 2010). Quinazolin-4(3*H*)-ones synthesis involves use of various synthetic procedures. The most common starting materials are 2-aminobenzonitrile (Ma *et al.*, 2013), 2-bromobenzamides (Xu *et al.*, 2012), isatoic anhydride (Adib *et al.*, 2012), anthranilic acid (Kumar *et al.*, 2011), methyl 2-aminobenzoate (Davies *et al.*, 2010). Lithiation of 2-*n*-alkyl- and 2-unsubstituted 3-acylaminoquinazolin-4(3*H*)-ones with a lithium reagent in tetrahydrofuran at a low temperature followed by reactions of various electrophiles with the lithium reagents produced in-situ gave the corresponding 2-substituted derivatives in good to excellent yields (Smith *et al.*, 2004, 1996, 1995). For the X-ray structures for related compounds, see: El-Hiti *et al.* (2014); Yang *et al.* (2009); Coogan *et al.* (1999).

S2. Experimental

S2.1. Synthesis and crystallization

A mixture of methyl 2-aminobenzoate and propionic anhydride (1.4 mole equivalents) was heated for 30 minutes at 105 °C. The mixture was cooled to 75 °C and diluted with ethanol (50 mL). Hydrazine monohydrate (10 mole equivalents) was added in a dropwise manner over 10 minutes and the mixture was refluxed for 1 h. The mixture was cooled to room temperature and the solvent was removed under reduced pressure. The residue obtained was purified by column chromatography (silica gel hexane/diethyl ether in 4:1 by volume) to give 3-amino-2-ethylquinazolin-4(3*H*)-one in 82% yield (Davies *et al.*, 2010). Crystallization from a mixture of ethyl acetate and diethyl ether (1:2 by volume) gave colourless crystals of the title compound. The spectroscopic data for the title compound were identical with those reported (Davies *et al.*, 2010).

S2.2. Refinement

H atoms were positioned geometrically and refined using a riding model with U_{iso}(H) constrained to be 1.2 times U_{eq} for the atom it is bonded to except for methyl groups where it was 1.5 times with free rotation about the C—C bond. The amide hydrogen atoms were located in the difference Fourier map and refined freely.

S3. Results and discussion

The asymmetric unit comprises a molecule of $C_{10}H_{11}N_3O$ (Fig. 1). The molecule is planar, including the ethyl group as indicated by the N2—C1—C9—C10 torsion angle of 1.5 (2)°. Inversion related molecules are stacked along the *a* axis (Fig. 2). Molecules (x,y,z) and (1-x, -y,1-z) are oriented head-to-tail and display π - π interaction with a centroid to centroid distance of 3.66 (2)Å. N—H···O hydrogen bonds between molecules (x,y,z) and (-x,-y+1, -z+1) generate a 'step′ structure through formation of a $R_2^2(10)$ ring.

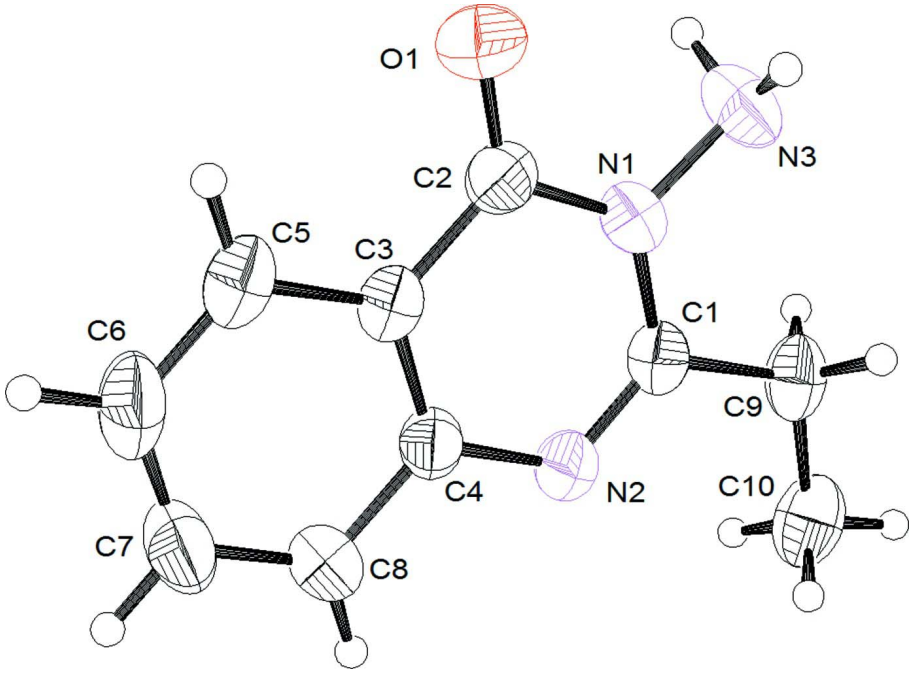

Figure 1

The asymmetric unit of $C_{10}H_{11}N_3O$, with atom labels and 50% probability displacement ellipsoids for non-hydrogen atoms.

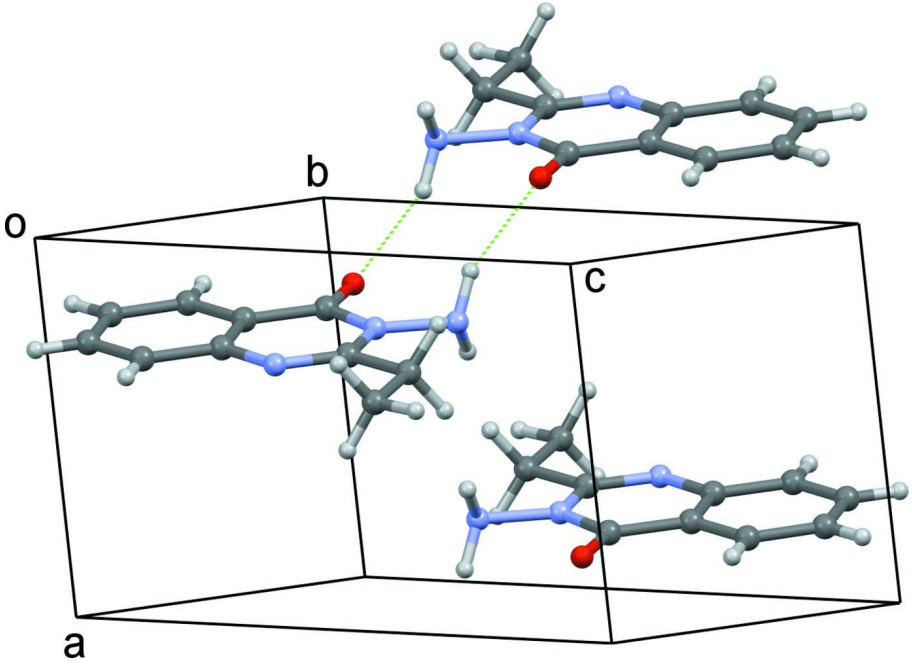

Figure 2

Crystal packing with hydrogen-bonding contacts shown as dotted lines.

3-Amino-2-ethylquinazolin-4(3*H*)-one

Crystal data

$C_{10}H_{11}N_3O$
$M_r = 189.22$
Triclinic, $P\overline{1}$
$a = 7.0230$ (5) Å
$b = 7.6198$ (7) Å
$c = 9.7868$ (6) Å
$\alpha = 69.709$ (7)°
$\beta = 89.242$ (5)°
$\gamma = 75.191$ (7)°
$V = 473.27$ (7) Å^3
$Z = 2$
$F(000) = 200$
$D_x = 1.328$ Mg m^{-3}
Melting point: 398 K
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 1907 reflections
$\theta = 6.5$–$73.7°$
$\mu = 0.73$ mm^{-1}
$T = 293$ K
Block, colourless
0.38 × 0.20 × 0.08 mm

Data collection

Agilent SuperNova Dual Source diffractometer with an Atlas detector
ω scans
Absorption correction: gaussian (*CrysAlis PRO*; Agilent, 2014)
$T_{min} = 0.741$, $T_{max} = 0.924$
3303 measured reflections
1858 independent reflections
1657 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 73.9°$, $\theta_{min} = 6.5°$
$h = -8 \rightarrow 8$
$k = -9 \rightarrow 8$
$l = -10 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.196$
$S = 1.07$
1858 reflections
136 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1458P)^2 + 0.021P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.26318 (18)	−0.0469 (2)	0.59059 (14)	0.0441 (4)
C2	0.18669 (19)	0.2641 (2)	0.38779 (16)	0.0485 (4)
C3	0.21085 (18)	0.1504 (2)	0.29325 (14)	0.0455 (4)
C4	0.25874 (19)	−0.0513 (2)	0.35723 (14)	0.0453 (4)
C5	0.1873 (2)	0.2421 (3)	0.14073 (16)	0.0585 (4)
H5	0.1547	0.3768	0.0991	0.070*

C6	0.2123 (3)	0.1324 (3)	0.05335 (16)	0.0711 (5)
H6	0.1976	0.1923	−0.0478	0.085*
C7	0.2597 (3)	−0.0691 (3)	0.11692 (19)	0.0738 (5)
H7	0.2761	−0.1428	0.0571	0.089*
C8	0.2829 (3)	−0.1613 (2)	0.26567 (18)	0.0616 (5)
H8	0.3144	−0.2961	0.3059	0.074*
C9	0.2868 (2)	−0.1443 (2)	0.75358 (14)	0.0541 (4)
H9A	0.1644	−0.0989	0.7931	0.065*
H9B	0.3894	−0.1063	0.7928	0.065*
C10	0.3393 (3)	−0.3628 (3)	0.80379 (17)	0.0703 (5)
H10A	0.2385	−0.4018	0.7655	0.105*
H10B	0.3492	−0.4162	0.9087	0.105*
H10C	0.4636	−0.4096	0.7691	0.105*
N1	0.21763 (16)	0.15389 (17)	0.53566 (13)	0.0475 (4)
N2	0.28333 (17)	−0.14907 (17)	0.50721 (12)	0.0479 (4)
N3	0.2043 (3)	0.2510 (2)	0.63756 (16)	0.0683 (5)
O1	0.14455 (19)	0.44171 (16)	0.34527 (14)	0.0704 (4)
H3A	0.080 (3)	0.329 (3)	0.630 (2)	0.079 (6)*
H3B	0.292 (4)	0.328 (5)	0.609 (3)	0.111 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0404 (6)	0.0519 (7)	0.0392 (7)	−0.0129 (5)	0.0037 (5)	−0.0148 (5)
C2	0.0494 (7)	0.0445 (7)	0.0510 (8)	−0.0144 (5)	0.0072 (5)	−0.0151 (6)
C3	0.0457 (7)	0.0494 (8)	0.0400 (7)	−0.0154 (5)	0.0048 (5)	−0.0122 (6)
C4	0.0494 (7)	0.0498 (7)	0.0403 (7)	−0.0174 (5)	0.0067 (5)	−0.0173 (6)
C5	0.0588 (8)	0.0659 (9)	0.0424 (8)	−0.0201 (7)	0.0043 (6)	−0.0066 (6)
C6	0.0789 (11)	0.0986 (14)	0.0375 (7)	−0.0316 (10)	0.0073 (7)	−0.0206 (8)
C7	0.0926 (12)	0.0963 (13)	0.0520 (9)	−0.0368 (10)	0.0153 (8)	−0.0421 (9)
C8	0.0781 (10)	0.0627 (9)	0.0559 (9)	−0.0253 (8)	0.0121 (7)	−0.0312 (7)
C9	0.0482 (7)	0.0729 (9)	0.0367 (7)	−0.0158 (6)	0.0031 (5)	−0.0142 (6)
C10	0.0729 (10)	0.0711 (10)	0.0464 (8)	−0.0147 (8)	−0.0002 (7)	0.0010 (7)
N1	0.0511 (6)	0.0505 (7)	0.0451 (7)	−0.0128 (5)	0.0045 (4)	−0.0227 (5)
N2	0.0556 (7)	0.0458 (6)	0.0408 (7)	−0.0147 (5)	0.0055 (5)	−0.0129 (5)
N3	0.0801 (10)	0.0733 (9)	0.0628 (9)	−0.0136 (8)	0.0049 (7)	−0.0428 (7)
O1	0.0890 (8)	0.0436 (6)	0.0744 (8)	−0.0149 (5)	0.0118 (6)	−0.0182 (5)

Geometric parameters (Å, º)

C1—N2	1.2937 (19)	C6—H6	0.9300
C1—N1	1.3840 (19)	C7—C8	1.370 (2)
C1—C9	1.4986 (18)	C7—H7	0.9300
C2—O1	1.2245 (18)	C8—H8	0.9300
C2—N1	1.3853 (19)	C9—C10	1.508 (2)
C2—C3	1.453 (2)	C9—H9A	0.9700
C3—C4	1.394 (2)	C9—H9B	0.9700
C3—C5	1.4027 (19)	C10—H10A	0.9600

C4—N2	1.3862 (18)	C10—H10B	0.9600
C4—C8	1.406 (2)	C10—H10C	0.9600
C5—C6	1.370 (3)	N1—N3	1.4227 (16)
C5—H5	0.9300	N3—H3A	0.91 (2)
C6—C7	1.392 (3)	N3—H3B	0.93 (3)

N2—C1—N1	122.58 (12)	C7—C8—H8	120.1
N2—C1—C9	120.35 (13)	C4—C8—H8	120.1
N1—C1—C9	117.07 (12)	C1—C9—C10	113.52 (13)
O1—C2—N1	120.90 (14)	C1—C9—H9A	108.9
O1—C2—C3	124.96 (14)	C10—C9—H9A	108.9
N1—C2—C3	114.14 (12)	C1—C9—H9B	108.9
C4—C3—C5	120.75 (14)	C10—C9—H9B	108.9
C4—C3—C2	118.64 (13)	H9A—C9—H9B	107.7
C5—C3—C2	120.61 (14)	C9—C10—H10A	109.5
N2—C4—C3	123.07 (12)	C9—C10—H10B	109.5
N2—C4—C8	118.33 (13)	H10A—C10—H10B	109.5
C3—C4—C8	118.60 (14)	C9—C10—H10C	109.5
C6—C5—C3	119.79 (16)	H10A—C10—H10C	109.5
C6—C5—H5	120.1	H10B—C10—H10C	109.5
C3—C5—H5	120.1	C1—N1—C2	123.65 (12)
C5—C6—C7	119.60 (14)	C1—N1—N3	117.72 (12)
C5—C6—H6	120.2	C2—N1—N3	118.63 (13)
C7—C6—H6	120.2	C1—N2—C4	117.90 (12)
C8—C7—C6	121.48 (16)	N1—N3—H3A	110.0 (14)
C8—C7—H7	119.3	N1—N3—H3B	104.8 (18)
C6—C7—H7	119.3	H3A—N3—H3B	109 (2)
C7—C8—C4	119.78 (16)		

O1—C2—C3—C4	−179.89 (12)	N2—C1—C9—C10	1.5 (2)
N1—C2—C3—C4	0.7 (2)	N1—C1—C9—C10	−179.13 (11)
O1—C2—C3—C5	0.4 (2)	N2—C1—N1—C2	0.9 (2)
N1—C2—C3—C5	−178.98 (10)	C9—C1—N1—C2	−178.40 (10)
C5—C3—C4—N2	−179.87 (11)	N2—C1—N1—N3	−178.35 (11)
C2—C3—C4—N2	0.4 (2)	C9—C1—N1—N3	2.30 (19)
C5—C3—C4—C8	0.0 (2)	O1—C2—N1—C1	179.18 (12)
C2—C3—C4—C8	−179.75 (11)	C3—C2—N1—C1	−1.4 (2)
C4—C3—C5—C6	−0.3 (2)	O1—C2—N1—N3	−1.5 (2)
C2—C3—C5—C6	179.44 (12)	C3—C2—N1—N3	177.87 (11)
C3—C5—C6—C7	0.4 (3)	N1—C1—N2—C4	0.3 (2)
C5—C6—C7—C8	−0.2 (3)	C9—C1—N2—C4	179.64 (10)
C6—C7—C8—C4	−0.1 (3)	C3—C4—N2—C1	−1.0 (2)
N2—C4—C8—C7	−179.95 (14)	C8—C4—N2—C1	179.19 (11)
C3—C4—C8—C7	0.2 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···O1^{i}	0.91 (2)	2.12 (2)	2.974 (2)	157.1 (19)

Symmetry code: (i) −*x*, −*y*+1, −*z*+1.