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

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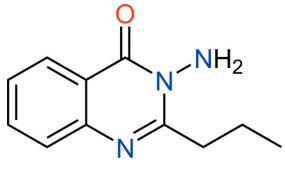
In the title molecule, $C_{11}H_{13}N_3O$, the propyl group is almost perpendicular to the quinazolin-4(3*H*)-one mean plane, making a dihedral angle of 88.98 (9)°. In the crystal, molecules related by an inversion centre are paired *via* π–π overlap, indicated by the short distances of 3.616 (5) and 3.619 (5) Å between the centroids of the aromatic rings of neighbouring molecules. Intermolecular N—H···N and N—H···O hydrogen bonds form $R_6^6(30)$ rings and C(5) chains, respectively, generating a three-dimensional network. Weak C—H···O interactions are also observed.

Keywords: crystal structure; quinazolin-4(3*H*)-one; hydrogen bonding; π–π overlap.

CCDC reference: 1411448

1. Related literature

For biological applications of related compounds, see: Sasmal *et al.* (2012); Rohini *et al.* (2010); Chandregowda *et al.* (2009); Gupta *et al.* (2008); Alagarsamy *et al.* (2007). For the synthesis of substituted quinazolin-4(3*H*)-ones, see: Ma *et al.* (2013); Adib *et al.* (2012); Xu *et al.* (2012); Kumar *et al.* (2011). For modification of the quinazolin-4(3*H*)-one ring system *via* lithiation, see: Smith *et al.* (2004, 1996, 1995). For the crystal structures for related compounds, see: El-Hiti *et al.* (2014); Yang *et al.* (2009); Coogan *et al.* (1999).



2. Experimental

2.1. Crystal data

$C_{11}H_{13}N_3O$
M_r = 203.24
Trigonal, $R\overline{3}$: *H*
a = 24.1525 (5) Å
c = 9.6500 (2) Å
V = 4875.1 (2) Å^3
Z = 18
Cu *K*α radiation
μ = 0.67 mm^{-1}
T = 296 K
0.34 × 0.25 × 0.19 mm

2.2. Data collection

Agilent SuperNova Dual Source diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.975, T_{max} = 0.984
3734 measured reflections
2136 independent reflections
1913 reflections with $I > 2\sigma(I)$
R_{int} = 0.013

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.120
S = 1.06
2136 reflections
146 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···N2^{i}	0.91 (2)	2.16 (2)	3.0677 (17)	176.1 (16)
N3—H3*B*···O1^{ii}	0.87 (2)	2.51 (2)	3.0599 (16)	122.0 (15)
C5—H5···O1^{iii}	0.93	2.44	3.3163 (16)	157

Symmetry codes: (i) $y, -x+y, -z$; (ii) $-y+\frac{1}{3}, x-y-\frac{1}{3}, z-\frac{1}{3}$; (iii) $-x+y+\frac{2}{3}, -x+\frac{1}{3}, z+\frac{1}{3}$.

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, 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

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Supporting information for this paper is available from the IUCr electronic archives (Reference: CV5493).

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supporting information

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

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

S1. Introduction

Quinazolines have a range of biological activities such as anti-cancer (Chandregowda *et al.*, 2009), anti-bacterial (Rohini *et al.*, 2010), anti-inflammatory (Alagarsamy *et al.*, 2007), anti-obesity (Sasmal *et al.*, 2012) and anti-spasm (Gupta *et al.*, 2008). Synthesis of quinazolin-4(3*H*)-ones involves use of various synthetic procedures. Recent examples involve reactions of 2-aminobenzonitrile with carbon dioxide in water (Ma *et al.*, 2013), 2-bromobenzamides with formamide catalysed by CuI and 4-hydroxy-l-proline (Xu *et al.*, 2012) and isatoic anhydride, benzyl halides and primary amines under mild Kornblum conditions (Adib *et al.*, 2012). 2-Alkyl-3-aminoquinazolin-4(3*H*)-ones can be obtained from reactions of 2-alkyl-4*H*-3,1-benzoxazin-4-ones with hydrazine hydrate (Kumar *et al.*, 2011). Lithiation of 2-unsubstituted and 2-n-alkyl-3-acylaminoquinazolin-4(3*H*)-ones followed by reactions of the lithium reagents produced *in-situ* with electrophiles gave the corresponding substituted derivatives in high 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

3-Amino-2-propylquinazolin-4(3*H*)-one was obtained in 82% yield by reaction of 2-propyl-4*H*-3,1-benzoxazin-4-one with excess hydrazine hydrate (three mole equivalents) in methanol under reflux conditions for 3 h (Kumar *et al.*, 2011). Crystallization from ethanol gave colourless crystals of the title compound. The NMR and mass spectral data for the title compound were identical with those reported (Kumar *et al.*, 2011).

S2.2. Refinement

The amino hydrogen atoms were located in the difference Fourier map and refined freely. The rest of the 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.

S3. Results and discussion

In the title compound (I) (Fig. 1), the propyl group is perpendicular to the quinazolin-4(3*H*)-one group with a dihedral angle of 88.98 (9)° between the least-squares planes of the two groups. In the crystal (Fig. 2), π–π overlap is observed for paired molecules with a centroid-centroid distance of *ca* 3.62 (1) Å between the benzene and pyrimidine rings of parallel 3-aminoquinazolin-4(3*H*)-one groups. N—H···N hydrogen bonds form $R_6^6(30)$ rings and N—H···O form C(5) chains to generate three dimensional packing. Weak C—H···O contacts (C(5)) are also observed.

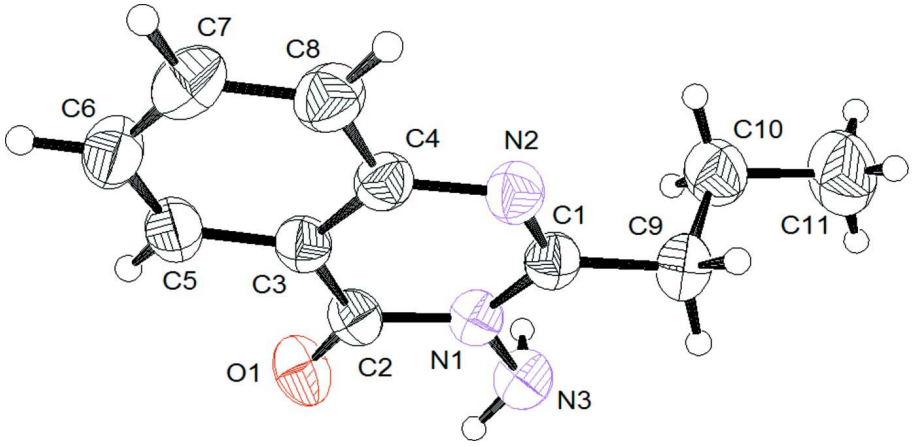

Figure 1

View of (I) showing the atom labels and 50% probability displacement ellipsoids.

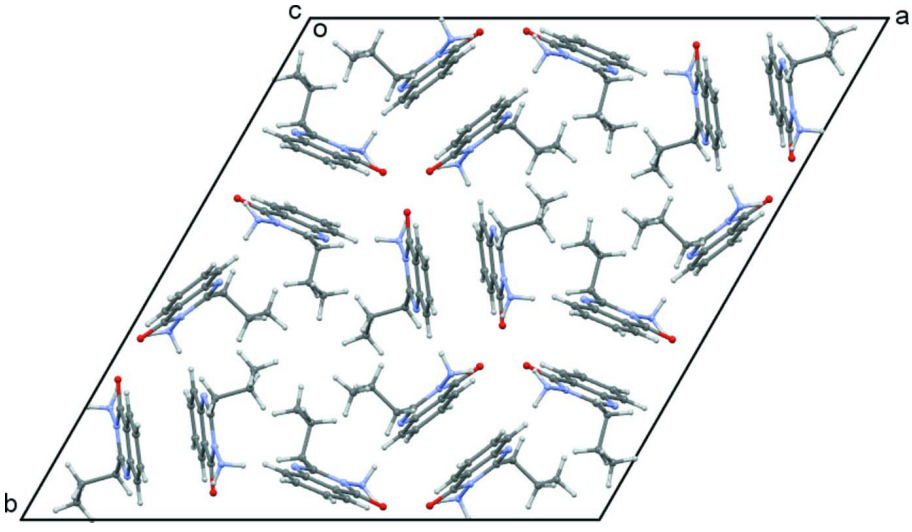

Figure 2

Crystal packing viewed along the *c* axis.

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

Crystal data

$C_{11}H_{13}N_3O$
$M_r = 203.24$
Trigonal, $R\bar{3}:H$
$a = 24.1525$ (5) Å
$c = 9.6500$ (2) Å
$V = 4875.1$ (2) Å^3
$Z = 18$
$F(000) = 1944$
$D_x = 1.246$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 2040 reflections
$\theta = 5.0$–$74.1°$
$\mu = 0.67$ mm^{-1}
$T = 296$ K
Block, colourless
0.34 × 0.25 × 0.19 mm

Data collection

Agilent SuperNova Dual Source diffractometer with an Atlas detector
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.975, T_{max} = 0.984
3734 measured reflections
2136 independent reflections
1913 reflections with $I > 2\sigma(I)$
R_{int} = 0.013
θ_{max} = 74.1°, θ_{min} = 3.7°
h = −21→30
k = −26→18
l = −11→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.120
S = 1.06
2136 reflections
146 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0637P)^2 + 1.4157P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Extinction correction: *SHELXL2013* (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00114 (10)

Special details

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	0.23343 (6)	0.11120 (6)	0.00970 (12)	0.0453 (3)
C2	0.28221 (6)	0.06090 (5)	0.14259 (13)	0.0457 (3)
C3	0.27352 (5)	0.09197 (5)	0.26270 (12)	0.0440 (3)
C4	0.24674 (6)	0.13113 (6)	0.24439 (12)	0.0450 (3)
C5	0.29168 (6)	0.08293 (7)	0.39466 (14)	0.0535 (3)
H5	0.3089	0.0563	0.4064	0.064*
C6	0.28403 (7)	0.11345 (8)	0.50654 (14)	0.0618 (4)
H6	0.2958	0.1074	0.5946	0.074*
C7	0.25865 (8)	0.15367 (8)	0.48822 (14)	0.0630 (4)
H7	0.2544	0.1749	0.5643	0.076*
C8	0.23997 (7)	0.16241 (7)	0.36013 (14)	0.0567 (3)
H8	0.2228	0.1891	0.3498	0.068*
C9	0.20961 (7)	0.11882 (6)	−0.12831 (14)	0.0546 (3)
H9A	0.2357	0.1157	−0.2009	0.066*
H9B	0.2141	0.1610	−0.1337	0.066*
C10	0.13977 (8)	0.06846 (8)	−0.15299 (18)	0.0697 (4)
H10A	0.1359	0.0265	−0.1554	0.084*
H10B	0.1141	0.0691	−0.0763	0.084*
C11	0.11417 (10)	0.07946 (12)	−0.2869 (2)	0.0996 (7)
H11A	0.1222	0.1226	−0.2894	0.149*
H11B	0.0690	0.0503	−0.2924	0.149*

H11C	0.1351	0.0724	−0.3639	0.149*
N1	0.25905 (5)	0.07168 (5)	0.01918 (10)	0.0447 (3)
N2	0.22710 (5)	0.14035 (5)	0.11608 (11)	0.0486 (3)
N3	0.26483 (7)	0.04247 (6)	−0.10370 (12)	0.0550 (3)
O1	0.30729 (5)	0.02780 (5)	0.14300 (11)	0.0625 (3)
H3A	0.2287 (9)	0.0033 (10)	−0.1045 (18)	0.069 (5)*
H3B	0.2972 (10)	0.0370 (9)	−0.087 (2)	0.069 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0447 (6)	0.0405 (6)	0.0464 (6)	0.0179 (5)	−0.0012 (5)	0.0016 (4)
C2	0.0425 (6)	0.0391 (5)	0.0518 (7)	0.0177 (5)	−0.0073 (5)	−0.0046 (5)
C3	0.0400 (6)	0.0401 (6)	0.0462 (6)	0.0158 (5)	−0.0028 (4)	−0.0002 (4)
C4	0.0441 (6)	0.0438 (6)	0.0439 (6)	0.0196 (5)	0.0021 (4)	0.0025 (4)
C5	0.0514 (7)	0.0554 (7)	0.0518 (7)	0.0253 (6)	−0.0067 (5)	0.0018 (5)
C6	0.0650 (8)	0.0724 (9)	0.0432 (7)	0.0307 (7)	−0.0043 (6)	0.0016 (6)
C7	0.0736 (9)	0.0695 (9)	0.0450 (7)	0.0350 (7)	0.0067 (6)	−0.0039 (6)
C8	0.0653 (8)	0.0603 (8)	0.0504 (7)	0.0358 (7)	0.0057 (6)	−0.0003 (6)
C9	0.0631 (8)	0.0512 (7)	0.0486 (7)	0.0279 (6)	−0.0060 (5)	0.0022 (5)
C10	0.0616 (9)	0.0732 (10)	0.0692 (9)	0.0300 (8)	−0.0086 (7)	0.0004 (7)
C11	0.0780 (12)	0.1036 (15)	0.0986 (15)	0.0315 (11)	−0.0333 (11)	0.0058 (12)
N1	0.0467 (5)	0.0401 (5)	0.0434 (5)	0.0189 (4)	−0.0036 (4)	−0.0052 (4)
N2	0.0546 (6)	0.0488 (6)	0.0462 (6)	0.0287 (5)	0.0001 (4)	0.0023 (4)
N3	0.0630 (7)	0.0499 (6)	0.0494 (6)	0.0263 (6)	−0.0042 (5)	−0.0120 (4)
O1	0.0729 (6)	0.0611 (6)	0.0683 (6)	0.0446 (5)	−0.0198 (5)	−0.0166 (4)

Geometric parameters (Å, º)

C1—N2	1.2963 (16)	C7—H7	0.9300
C1—N1	1.3760 (16)	C8—H8	0.9300
C1—C9	1.4981 (17)	C9—C10	1.526 (2)
C2—O1	1.2209 (15)	C9—H9A	0.9700
C2—N1	1.3945 (16)	C9—H9B	0.9700
C2—C3	1.4520 (17)	C10—C11	1.512 (2)
C3—C4	1.3984 (18)	C10—H10A	0.9700
C3—C5	1.3991 (18)	C10—H10B	0.9700
C4—N2	1.3832 (16)	C11—H11A	0.9600
C4—C8	1.4032 (18)	C11—H11B	0.9600
C5—C6	1.371 (2)	C11—H11C	0.9600
C5—H5	0.9300	N1—N3	1.4219 (14)
C6—C7	1.395 (2)	N3—H3A	0.91 (2)
C6—H6	0.9300	N3—H3B	0.87 (2)
C7—C8	1.368 (2)		
N2—C1—N1	122.69 (11)	C1—C9—H9A	109.1
N2—C1—C9	118.73 (11)	C10—C9—H9A	109.1
N1—C1—C9	118.53 (11)	C1—C9—H9B	109.1

O1—C2—N1	120.11 (11)	C10—C9—H9B	109.1
O1—C2—C3	125.67 (12)	H9A—C9—H9B	107.9
N1—C2—C3	114.21 (10)	C11—C10—C9	112.30 (15)
C4—C3—C5	120.51 (12)	C11—C10—H10A	109.1
C4—C3—C2	118.94 (11)	C9—C10—H10A	109.1
C5—C3—C2	120.55 (11)	C11—C10—H10B	109.1
N2—C4—C3	122.27 (11)	C9—C10—H10B	109.1
N2—C4—C8	118.95 (12)	H10A—C10—H10B	107.9
C3—C4—C8	118.78 (12)	C10—C11—H11A	109.5
C6—C5—C3	119.73 (13)	C10—C11—H11B	109.5
C6—C5—H5	120.1	H11A—C11—H11B	109.5
C3—C5—H5	120.1	C10—C11—H11C	109.5
C5—C6—C7	119.93 (13)	H11A—C11—H11C	109.5
C5—C6—H6	120.0	H11B—C11—H11C	109.5
C7—C6—H6	120.0	C1—N1—C2	123.22 (10)
C8—C7—C6	121.04 (13)	C1—N1—N3	118.60 (10)
C8—C7—H7	119.5	C2—N1—N3	118.13 (10)
C6—C7—H7	119.5	C1—N2—C4	118.58 (11)
C7—C8—C4	119.99 (13)	N1—N3—H3A	104.0 (11)
C7—C8—H8	120.0	N1—N3—H3B	103.8 (13)
C4—C8—H8	120.0	H3A—N3—H3B	108.1 (17)
C1—C9—C10	112.34 (12)		

O1—C2—C3—C4	176.78 (12)	N2—C1—C9—C10	−89.31 (15)
N1—C2—C3—C4	−3.10 (16)	N1—C1—C9—C10	88.39 (15)
O1—C2—C3—C5	−3.2 (2)	C1—C9—C10—C11	175.23 (16)
N1—C2—C3—C5	176.95 (11)	N2—C1—N1—C2	−1.99 (18)
C5—C3—C4—N2	−178.79 (11)	C9—C1—N1—C2	−179.59 (11)
C2—C3—C4—N2	1.26 (17)	N2—C1—N1—N3	−179.17 (11)
C5—C3—C4—C8	1.61 (18)	C9—C1—N1—N3	3.23 (16)
C2—C3—C4—C8	−178.34 (11)	O1—C2—N1—C1	−176.35 (11)
C4—C3—C5—C6	−0.98 (19)	C3—C2—N1—C1	3.54 (16)
C2—C3—C5—C6	178.97 (12)	O1—C2—N1—N3	0.84 (17)
C3—C5—C6—C7	−0.4 (2)	C3—C2—N1—N3	−179.27 (10)
C5—C6—C7—C8	1.2 (2)	N1—C1—N2—C4	−0.22 (18)
C6—C7—C8—C4	−0.6 (2)	C9—C1—N2—C4	177.38 (11)
N2—C4—C8—C7	179.55 (13)	C3—C4—N2—C1	0.51 (18)
C3—C4—C8—C7	−0.8 (2)	C8—C4—N2—C1	−179.89 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···N2^{i}	0.91 (2)	2.16 (2)	3.0677 (17)	176.1 (16)
N3—H3*B*···O1^{ii}	0.87 (2)	2.51 (2)	3.0599 (16)	122.0 (15)
C5—H5···O1^{iii}	0.93	2.44	3.3163 (16)	157

Symmetry codes: (i) *y*, −*x*+*y*, −*z*; (ii) −*y*+1/3, *x*−*y*−1/3, *z*−1/3; (iii) −*x*+*y*+2/3, −*x*+1/3, *z*+1/3.