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3-(2-Aminoethyl)-2-anilinoquinazolin-4(3*H*)-one methanol hemisolvate

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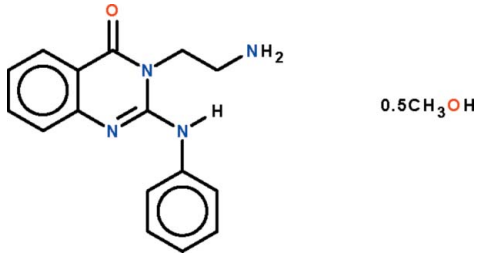
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; disorder in solvent or counterion; R factor = 0.062; wR factor = 0.186; data-to-parameter ratio = 16.2.

The title methanol hemisolvated quinazolin-(3*H*)-one, $C_{16}H_{16}N_4O{\cdot}0.5CH_3OH$, has an anilino substituent in the 2-position and an aminoethyl substituent in the 3-position of the planar fused-ring system (r.m.s. deviation = 0.019 Å). The anilino N atom donates an intramolecular hydrogen bond to the aminoethyl N atom. The molecule and the solvent methanol molecule are linked by N—H···N, N—H···O and O—H···O hydrogen bonds. The methanol molecule is disordered over two equally occupied positions about a twofold rotation axis.

Related literature

For the synthesis of this class of compounds, see: Yang *et al.* (2008). For the crystal structure of a chlorine-substituted derivative, see: Yang *et al.* (2009).



Experimental

Crystal data

$C_{16}H_{16}N_4O{\cdot}0.5CH_4O$
M_r = 296.35
Monoclinic, $C2/c$
a = 19.5972 (11) Å
b = 12.2035 (7) Å
c = 12.8681 (8) Å
β = 103.301 (1)°
V = 2994.9 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 295 K
0.30 × 0.20 × 0.10 mm

Data collection

Bruker APEXII diffractometer
Absorption correction: none
14007 measured reflections
3399 independent reflections
2377 reflections with $I > 2\sigma(I)$
R_{int} = 0.073

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.186
S = 1.09
3399 reflections
210 parameters
13 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H31···N4	0.88	2.04	2.811 (3)	146
N4—H41···O2^{i}	0.88	2.13	2.990 (6)	168
O2—H2*O*···O1	0.84	2.01	2.755 (6)	147

Symmetry code: (i) $x, -y+1, z-\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5121).

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Yang, X.-H., Wu, M.-H., Sun, S.-F., Ding, M.-W., Xie, J.-L. & Xia, Q.-H. (2008). *J. Heterocycl. Chem.* **45**, 1365–1369.

supporting information

Acta Cryst. (2009). E**65**, o2989 [doi:10.1107/S1600536809045516]

3-(2-Aminoethyl)-2-anilinoquinazolin-4(3*H*)-one methanol hemisolvate

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S1. Experimental

To a THF (10 ml) solution of 2-ethoxycarbonyliminophosphorane (1.27 g, 3.0 mmol) was added phenylisocyanate (0.36 g, 3.0 mmol). The solution was set aside undisturbed for 6 h at 273 K. To this solution was added ethanolamine (0.18 g, 3 mmol) in THF (5 ml). The mixture was stirred overnight. The solvent was removed and the solid recrystallized from a chloroform/methanol (1/1) mixture to give colorless crystals in 80% yield; m.p. 433–434 K.

S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 0.97 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2 to 1.5*U*(C). The amino and hydroxy H atoms were similarly generated.

The methanol molecule is disordered over two equally occupied positions about a two-fold rotation axis. The C–O distance was restrained to 1.500±0.002 Å. The anisotropic displacemnt parameters of the methanolic O and C atoms were restrained to be nearly isotropic.

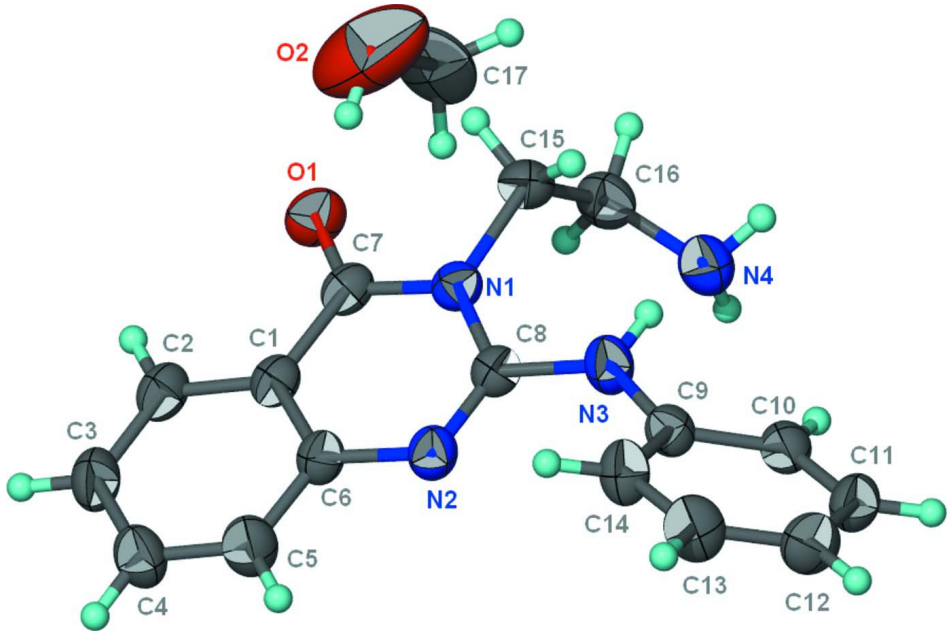

Figure 1

Anisotropic displacement ellipsoid plot (Barbour, 2001) of the title compound at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

3-(2-Aminoethyl)-2-anilinoquinazolin-4(3*H*)-one methanol hemisolvate

Crystal data

$C_{16}H_{16}N_4O\cdot 0.5CH_4O$
$M_r = 296.35$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 19.5972\ (11)$ Å
$b = 12.2035\ (7)$ Å
$c = 12.8681\ (8)$ Å
$\beta = 103.301\ (1)°$
$V = 2994.9\ (3)$ Å^3
$Z = 8$
$F(000) = 1256$
$D_x = 1.314$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3816 reflections
$\theta = 2.4–25.9°$
$\mu = 0.09$ mm^{-1}
$T = 295$ K
Block, colorless
0.30 × 0.20 × 0.10 mm

Data collection

Bruker APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
14007 measured reflections
3399 independent reflections
2377 reflections with $I > 2\sigma(I)$
$R_{int} = 0.073$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.0°$
$h = -17\rightarrow25$
$k = -15\rightarrow14$
$l = -16\rightarrow16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.186$
$S = 1.09$
3399 reflections
210 parameters
13 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
$w = 1/[\sigma^2(F_o^2) + (0.1044P)^2 + 0.1504P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.55$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.68958 (7)	0.41303 (12)	0.81309 (11)	0.0573 (4)	
N1	0.73674 (8)	0.41620 (12)	0.66714 (11)	0.0424 (4)	
N2	0.84958 (8)	0.34284 (13)	0.67038 (11)	0.0458 (4)	
N3	0.77752 (9)	0.40366 (13)	0.51241 (12)	0.0498 (4)	
H31	0.7345	0.4239	0.4817	0.060*	
N4	0.63136 (9)	0.37965 (16)	0.44490 (14)	0.0632 (5)	
H41	0.6131	0.4263	0.3939	0.076*	
H42	0.6131	0.3143	0.4284	0.076*	
C1	0.80430 (10)	0.34133 (14)	0.83148 (13)	0.0414 (4)	
C2	0.81360 (11)	0.31401 (16)	0.93932 (14)	0.0503 (5)	
H2	0.7782	0.3290	0.9746	0.060*	
C3	0.87449 (12)	0.26533 (18)	0.99341 (15)	0.0576 (6)	
H3	0.8804	0.2465	1.0650	0.069*	
C4	0.92707 (11)	0.24457 (19)	0.94030 (15)	0.0589 (6)	

H4	0.9684	0.2115	0.9767	0.071*	
C5	0.91908 (11)	0.27202 (19)	0.83510 (16)	0.0573 (6)	
H5	0.9552	0.2579	0.8011	0.069*	
C6	0.85720 (10)	0.32115 (15)	0.77791 (13)	0.0427 (5)	
C7	0.73916 (10)	0.39143 (15)	0.77321 (14)	0.0437 (5)	
C8	0.79099 (10)	0.38642 (15)	0.61962 (14)	0.0414 (4)	
C9	0.82431 (10)	0.39287 (14)	0.44502 (14)	0.0426 (5)	
C10	0.79487 (10)	0.36669 (15)	0.33875 (14)	0.0462 (5)	
H10	0.7472	0.3515	0.3175	0.055*	
C11	0.83579 (12)	0.36311 (17)	0.26507 (15)	0.0530 (5)	
H11	0.8154	0.3462	0.1942	0.064*	
C12	0.90627 (13)	0.38417 (19)	0.29508 (18)	0.0614 (6)	
H12	0.9338	0.3820	0.2451	0.074*	
C13	0.93592 (12)	0.40868 (19)	0.40098 (18)	0.0616 (6)	
H13	0.9838	0.4225	0.4220	0.074*	
C14	0.89551 (11)	0.41293 (17)	0.47595 (16)	0.0534 (5)	
H14	0.9161	0.4292	0.5468	0.064*	
C15	0.67613 (10)	0.48235 (17)	0.61029 (15)	0.0516 (5)	
H15A	0.6922	0.5335	0.5634	0.062*	
H15B	0.6587	0.5248	0.6624	0.062*	
C16	0.61626 (11)	0.41579 (18)	0.54480 (17)	0.0586 (6)	
H16A	0.6080	0.3524	0.5857	0.070*	
H16B	0.5739	0.4599	0.5298	0.070*	
O2	0.5495 (3)	0.4637 (6)	0.7850 (7)	0.176 (3)	0.50
H2O	0.5856	0.4244	0.8006	0.211*	0.50
C17	0.4883 (4)	0.3949 (6)	0.7344 (11)	0.101 (3)	0.50
H17A	0.4553	0.3937	0.7792	0.152*	0.50
H17B	0.5037	0.3216	0.7254	0.152*	0.50
H17C	0.4662	0.4250	0.6660	0.152*	0.50

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0468 (8)	0.0701 (10)	0.0607 (8)	0.0019 (7)	0.0243 (7)	−0.0043 (6)
N1	0.0402 (9)	0.0437 (9)	0.0438 (8)	0.0002 (7)	0.0110 (7)	−0.0028 (6)
N2	0.0417 (9)	0.0570 (10)	0.0402 (8)	0.0011 (7)	0.0130 (7)	−0.0002 (6)
N3	0.0430 (9)	0.0665 (11)	0.0405 (8)	0.0019 (8)	0.0107 (7)	0.0053 (7)
N4	0.0614 (12)	0.0661 (12)	0.0558 (10)	0.0053 (9)	0.0006 (9)	−0.0015 (8)
C1	0.0435 (10)	0.0412 (10)	0.0411 (9)	−0.0070 (8)	0.0128 (8)	−0.0050 (7)
C2	0.0578 (13)	0.0540 (12)	0.0437 (10)	−0.0063 (10)	0.0214 (9)	−0.0038 (8)
C3	0.0697 (14)	0.0649 (13)	0.0384 (9)	−0.0019 (11)	0.0128 (10)	0.0028 (9)
C4	0.0490 (12)	0.0744 (15)	0.0494 (11)	0.0035 (11)	0.0035 (10)	0.0062 (9)
C5	0.0446 (12)	0.0782 (15)	0.0510 (11)	0.0058 (10)	0.0152 (9)	0.0035 (10)
C6	0.0424 (10)	0.0490 (11)	0.0378 (9)	−0.0038 (8)	0.0112 (8)	−0.0014 (7)
C7	0.0443 (11)	0.0438 (10)	0.0459 (10)	−0.0063 (8)	0.0164 (8)	−0.0070 (7)
C8	0.0413 (10)	0.0424 (10)	0.0415 (9)	−0.0040 (8)	0.0112 (8)	−0.0027 (7)
C9	0.0445 (11)	0.0432 (10)	0.0411 (9)	−0.0007 (8)	0.0122 (8)	0.0067 (7)
C10	0.0482 (11)	0.0448 (11)	0.0440 (9)	−0.0001 (8)	0.0072 (8)	0.0011 (7)

C11	0.0636 (14)	0.0570 (12)	0.0404 (9)	0.0007 (10)	0.0159 (9)	−0.0006 (8)
C12	0.0654 (15)	0.0711 (15)	0.0554 (12)	0.0043 (11)	0.0293 (11)	0.0041 (10)
C13	0.0466 (12)	0.0792 (16)	0.0620 (13)	−0.0042 (10)	0.0187 (10)	0.0085 (10)
C14	0.0490 (12)	0.0683 (14)	0.0423 (10)	−0.0097 (10)	0.0091 (9)	0.0032 (9)
C15	0.0507 (12)	0.0492 (12)	0.0548 (11)	0.0090 (9)	0.0119 (9)	−0.0007 (8)
C16	0.0445 (12)	0.0671 (14)	0.0618 (13)	0.0074 (10)	0.0071 (10)	0.0025 (10)
O2	0.109 (4)	0.189 (6)	0.231 (6)	−0.017 (4)	0.043 (4)	−0.115 (5)
C17	0.044 (6)	0.143 (5)	0.117 (8)	0.021 (4)	0.017 (5)	0.021 (5)

Geometric parameters (Å, º)

O1—C7	1.227 (2)	C5—H5	0.9300
N1—C7	1.388 (2)	C9—C14	1.382 (3)
N1—C8	1.391 (2)	C9—C10	1.394 (2)
N1—C15	1.482 (2)	C10—C11	1.376 (3)
N2—C8	1.297 (2)	C10—H10	0.9300
N2—C6	1.383 (2)	C11—C12	1.370 (3)
N3—C8	1.360 (2)	C11—H11	0.9300
N3—C9	1.406 (2)	C12—C13	1.386 (3)
N3—H31	0.8800	C12—H12	0.9300
N4—C16	1.453 (3)	C13—C14	1.383 (3)
N4—H41	0.8800	C13—H13	0.9300
N4—H42	0.8800	C14—H14	0.9300
C1—C6	1.393 (3)	C15—C16	1.514 (3)
C1—C2	1.398 (2)	C15—H15A	0.9700
C1—C7	1.458 (3)	C15—H15B	0.9700
C2—C3	1.371 (3)	C16—H16A	0.9700
C2—H2	0.9300	C16—H16B	0.9700
C3—C4	1.385 (3)	O2—C17	1.486 (2)
C3—H3	0.9300	O2—H2O	0.8400
C4—C5	1.368 (3)	C17—H17A	0.9600
C4—H4	0.9300	C17—H17B	0.9600
C5—C6	1.400 (3)	C17—H17C	0.9600
C7—N1—C8	121.23 (15)	N2—C8—N1	124.28 (16)
C7—N1—C15	116.50 (15)	N3—C8—N1	114.57 (16)
C8—N1—C15	122.13 (15)	C14—C9—C10	119.07 (18)
C8—N2—C6	117.49 (16)	C14—C9—N3	124.37 (17)
C8—N3—C9	127.54 (17)	C10—C9—N3	116.44 (17)
C8—N3—H31	116.2	C11—C10—C9	120.53 (19)
C9—N3—H31	116.2	C11—C10—H10	119.7
C16—N4—H41	109.5	C9—C10—H10	119.7
C16—N4—H42	109.5	C12—C11—C10	120.61 (19)
H41—N4—H42	109.5	C12—C11—H11	119.7
C6—C1—C2	120.49 (17)	C10—C11—H11	119.7
C6—C1—C7	118.89 (16)	C11—C12—C13	119.0 (2)
C2—C1—C7	120.62 (18)	C11—C12—H12	120.5
C3—C2—C1	120.45 (19)	C13—C12—H12	120.5

C3—C2—H2	119.8	C14—C13—C12	121.1 (2)
C1—C2—H2	119.8	C14—C13—H13	119.5
C2—C3—C4	119.24 (17)	C12—C13—H13	119.5
C2—C3—H3	120.4	C9—C14—C13	119.66 (19)
C4—C3—H3	120.4	C9—C14—H14	120.2
C5—C4—C3	120.99 (19)	C13—C14—H14	120.2
C5—C4—H4	119.5	N1—C15—C16	114.39 (17)
C3—C4—H4	119.5	N1—C15—H15A	108.7
C4—C5—C6	120.9 (2)	C16—C15—H15A	108.7
C4—C5—H5	119.6	N1—C15—H15B	108.7
C6—C5—H5	119.6	C16—C15—H15B	108.7
N2—C6—C1	122.70 (17)	H15A—C15—H15B	107.6
N2—C6—C5	119.27 (17)	N4—C16—C15	111.46 (19)
C1—C6—C5	117.96 (16)	N4—C16—H16A	109.3
O1—C7—N1	120.87 (17)	C15—C16—H16A	109.3
O1—C7—C1	123.97 (17)	N4—C16—H16B	109.3
N1—C7—C1	115.15 (16)	C15—C16—H16B	109.3
N2—C8—N3	121.13 (18)	H16A—C16—H16B	108.0

C6—C1—C2—C3	−1.1 (3)	C6—N2—C8—N3	−175.64 (16)
C7—C1—C2—C3	178.97 (17)	C6—N2—C8—N1	2.5 (3)
C1—C2—C3—C4	0.7 (3)	C9—N3—C8—N2	−9.1 (3)
C2—C3—C4—C5	0.1 (3)	C9—N3—C8—N1	172.63 (16)
C3—C4—C5—C6	−0.5 (4)	C7—N1—C8—N2	−6.1 (3)
C8—N2—C6—C1	1.8 (3)	C15—N1—C8—N2	169.49 (18)
C8—N2—C6—C5	178.68 (18)	C7—N1—C8—N3	172.12 (15)
C2—C1—C6—N2	177.50 (16)	C15—N1—C8—N3	−12.3 (2)
C7—C1—C6—N2	−2.5 (3)	C8—N3—C9—C14	−31.8 (3)
C2—C1—C6—C5	0.6 (3)	C8—N3—C9—C10	152.29 (18)
C7—C1—C6—C5	−179.41 (17)	C14—C9—C10—C11	−1.4 (3)
C4—C5—C6—N2	−176.84 (19)	N3—C9—C10—C11	174.75 (17)
C4—C5—C6—C1	0.2 (3)	C9—C10—C11—C12	0.6 (3)
C8—N1—C7—O1	−176.29 (16)	C10—C11—C12—C13	0.3 (3)
C15—N1—C7—O1	7.9 (3)	C11—C12—C13—C14	−0.5 (3)
C8—N1—C7—C1	4.9 (2)	C10—C9—C14—C13	1.2 (3)
C15—N1—C7—C1	−170.91 (15)	N3—C9—C14—C13	−174.6 (2)
C6—C1—C7—O1	−179.64 (17)	C12—C13—C14—C9	−0.3 (3)
C2—C1—C7—O1	0.3 (3)	C7—N1—C15—C16	−96.6 (2)
C6—C1—C7—N1	−0.9 (2)	C8—N1—C15—C16	87.6 (2)
C2—C1—C7—N1	179.07 (16)	N1—C15—C16—N4	−77.5 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H31···N4	0.88	2.04	2.811 (3)	146

N4—H41···O2^{i}	0.88	2.13	2.990 (6)	168
O2—H2*O*···O1	0.84	2.01	2.755 (6)	147

Symmetry code: (i) x, $-y+1$, $z-1/2$.