

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

3-(4-Bromophenyl)quinazolin-4(3H)-one

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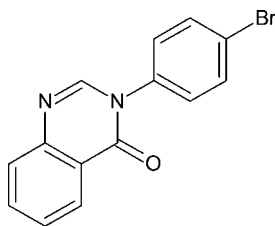
Received 15 September 2011; accepted 3 October 2011

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.036; wR factor = 0.094; data-to-parameter ratio = 17.4.

In the title compound, $\text{C}_{14}\text{H}_9\text{BrN}_2\text{O}$, the quinazoline unit is essentially planar, with a mean deviation of 0.058 (2) Å from the least-squares plane defined by the ten constituent ring atoms. The dihedral angle between the mean plane of the quinazoline ring system and the 4-bromophenyl ring is 47.6 (1)°. In the crystal, molecules are linked by intermolecular $\text{C}-\text{H}\cdots\text{N}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, forming infinite chains of alternating $R_2^2(6)$ dimers and $R_2^2(14)$ ring motifs.

Related literature

For the synthesis of the title compound, see: Priya, Zulykama *et al.* (2011). For a related structure, see: Priya, Srinivasan *et al.* (2011). For the biological activity of quinazoline derivatives, see: Wolfe *et al.* (1990); Tereshima *et al.* (1995); Pandeya *et al.* (1999). For graph-set notation, see: Bernstein *et al.* (1995).



Experimental

Crystal data

 $\text{C}_{14}\text{H}_9\text{BrN}_2\text{O}$
 $M_r = 301.14$ Monoclinic, $P2_1/n$
 $a = 16.961$ (3) Å $b = 3.9530$ (8) Å
 $c = 17.698$ (3) Å
 $\beta = 93.168$ (11)°
 $V = 1184.8$ (4) Å³
 $Z = 4$ Mo $K\alpha$ radiation
 $\mu = 3.46$ mm⁻¹
 $T = 293$ K
 $0.20 \times 0.20 \times 0.20$ mm

Data collection

Bruker SMART APEXII area-
detector diffractometer
10371 measured reflections2840 independent reflections
1772 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.050$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.094$
 $S = 1.01$
2840 reflections163 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.34$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.54$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C8}-\text{H8}\cdots\text{N1}^i$	0.93	2.48	3.286 (4)	146
$\text{C11}-\text{H11}\cdots\text{O1}^{\text{ii}}$	0.93	2.32	3.224 (4)	165

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

Data collection: APEX2 (Bruker, 2008); cell refinement: SAINT (Bruker, 2008); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97 and PLATON (Spek, 2009).

TS and DV thank the TBI X-ray facility, CAS in Crystallography and Biophysics, University of Madras, India, for the data collection and TS also thanks the DST for the Inspire fellowship

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2320).

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supporting information

Acta Cryst. (2011). E67, o2928 [doi:10.1107/S1600536811040736]

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S1. Comment

4(3H)-Quinazolinones are an important class of fused heterocycles with a wide range of biological activities such as anti-cancer (Wolfe *et al.*, 1990), anti-inflammatory (Tereshima *et al.*, 1995) and anti-HIV (Pandeya *et al.*, 1999). In addition to that, quinazolinones exhibit anti-bacterial and anti-fungal activities (Priya, Zulykama *et al.*, 2011).

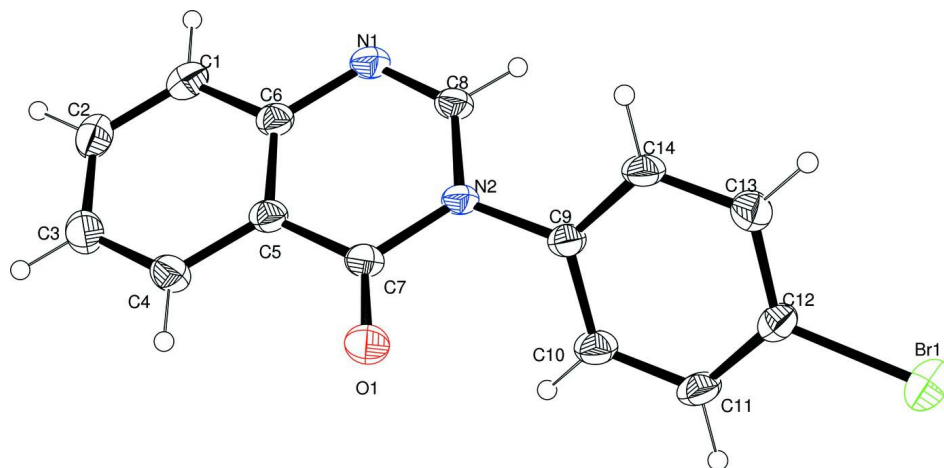
In title molecule (Fig. 1), the quinazoline unit is essentially planar, with a mean deviation of 0.058 (2) Å from the least square plane defined by the ten constituent atoms. The dihedral angle formed by the 4-bromophenyl ring and the mean plane of the quinazoline fragment is 47.6 (1)°. In the crystal packing, molecules are linked by intermolecular C–H⋯N and C–H⋯O hydrogen bonds (Table 1). These hydrogen bonds are forming infinite chains of alternating $R_2^2(6)$ dimer and $R_2^2(14)$ ring motifs (Bernstein *et al.*, 1995) as shown in Fig. 2.

S2. Experimental

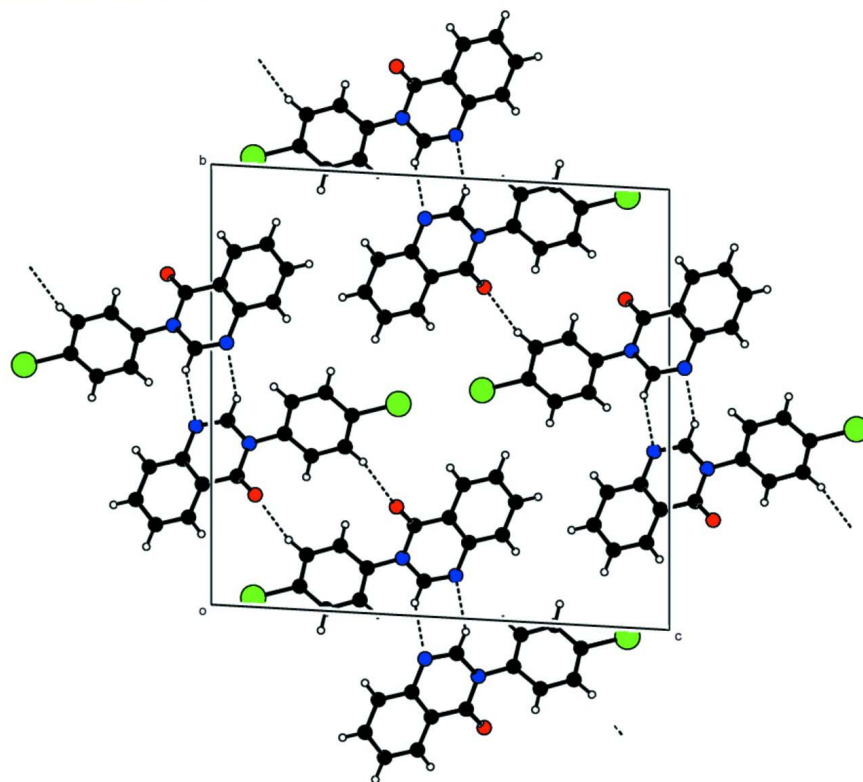
To an ice-cold solution of 2.8 ml POCl₃ in 5 ml DMF was added anthranilic acid (2 g, 0.0146 mole) and stirred for 5-10 min until TLC indicated the disappearance of anthranilic acid. The reaction mixture was then treated with an equimolar amount of *p*-bromo-aniline (2.511 g) and supported on anhydrous sodium sulfate (five times the weight of anthranilic acid) and exposed to microwave (BPL company) irradiation (600 W) for 2-4 min with 30 sec pulse. The reaction mixture was quenched with water (50 ml) and extracted with ethyl acetate (2 x 50 ml). The organic layer was dried over anhydrous sodium sulfate, concentrated and purified by silica gel column chromatography (60-20 mesh) using hexane/EtOAc (7.5 : 2.5) as eluent to yield the pure product (yield: 4,397 g, 84%). Single crystals suitable for X-ray diffraction were prepared by slow evaporation of a solution of the title compound in methanol at room temperature.

S3. Refinement

Hydrogen atoms were placed in calculated positions with C–H = 0.93 Å and refined using a riding model with fixed a isotropic displacement parameter of $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

**Figure 1**

Molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level. H atoms are presented as a small spheres of arbitrary radius.

**Figure 2**

View of the C-H...N and C-H...O hydrogen bonds (dotted lines) in the crystal structure of the title compound. [Symmetry codes: (i) $-x + 1, -y - 1, -z$; (ii) $-x + 1/2, y - 1/2, -z + 1/2$.]

3-(4-Bromophenyl)quinazolin-4(3H)-one

Crystal data

C₁₄H₉BrN₂O $M_r = 301.14$ Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

 $a = 16.961 (3) \text{ \AA}$ $b = 3.9530 (8) \text{ \AA}$ $c = 17.698 (3) \text{ \AA}$ $\beta = 93.168 (11)^\circ$ $V = 1184.8 (4) \text{ \AA}^3$ $Z = 4$ $F(000) = 600$ $D_x = 1.688 \text{ Mg m}^{-3}$ Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2840 reflections

 $\theta = 1.6\text{--}28.3^\circ$ $\mu = 3.46 \text{ mm}^{-1}$ $T = 293 \text{ K}$

Block, colourless

 $0.20 \times 0.20 \times 0.20 \text{ mm}$

Data collection

Bruker SMART APEXII area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 ω and φ scans

10371 measured reflections

2840 independent reflections

1772 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.050$ $\theta_{\text{max}} = 28.3^\circ$, $\theta_{\text{min}} = 1.6^\circ$ $h = -22 \rightarrow 14$ $k = -4 \rightarrow 5$ $l = -23 \rightarrow 23$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.036$ $wR(F^2) = 0.094$ $S = 1.01$

2840 reflections

163 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0381P)^2 + 0.2934P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} = 0.001$ $\Delta\rho_{\text{max}} = 0.34 \text{ e \AA}^{-3}$ $\Delta\rho_{\text{min}} = -0.54 \text{ e \AA}^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.30495 (19)	-0.4078 (7)	-0.13542 (16)	0.0457 (8)
H1	0.3413	-0.5172	-0.1644	0.055*
C2	0.22902 (19)	-0.3495 (8)	-0.16454 (17)	0.0494 (8)
H2	0.2143	-0.4201	-0.2134	0.059*
C3	0.17450 (19)	-0.1869 (8)	-0.12177 (18)	0.0500 (8)

H3	0.1233	-0.1520	-0.1418	0.060*
C4	0.19570 (17)	-0.0771 (8)	-0.04997 (17)	0.0445 (7)
H4	0.1591	0.0340	-0.0216	0.053*
C5	0.27242 (16)	-0.1327 (7)	-0.01962 (15)	0.0354 (7)
C6	0.32663 (17)	-0.3015 (7)	-0.06239 (15)	0.0360 (6)
N2	0.37065 (13)	-0.1237 (5)	0.08283 (12)	0.0338 (5)
C9	0.39734 (16)	-0.0577 (7)	0.15992 (14)	0.0340 (6)
C10	0.35023 (17)	-0.1470 (7)	0.21832 (16)	0.0424 (7)
H10	0.3018	-0.2522	0.2077	0.051*
C11	0.37520 (18)	-0.0797 (7)	0.29172 (16)	0.0430 (7)
H11	0.3434	-0.1357	0.3310	0.052*
C12	0.44731 (17)	0.0707 (7)	0.30707 (15)	0.0383 (7)
C13	0.49575 (17)	0.1529 (7)	0.24960 (16)	0.0440 (7)
H13	0.5449	0.2506	0.2607	0.053*
C14	0.47056 (16)	0.0889 (7)	0.17584 (16)	0.0397 (7)
H14	0.5026	0.1440	0.1367	0.048*
C7	0.29483 (16)	-0.0184 (8)	0.05649 (15)	0.0392 (7)
C8	0.41999 (17)	-0.2860 (7)	0.03558 (16)	0.0380 (7)
H8	0.4704	-0.3381	0.0556	0.046*
O1	0.25479 (13)	0.1572 (6)	0.09574 (12)	0.0599 (6)
N1	0.40348 (14)	-0.3727 (6)	-0.03334 (13)	0.0411 (6)
Br1	0.47919 (2)	0.17499 (9)	0.408526 (17)	0.05840 (16)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.056 (2)	0.0462 (19)	0.0358 (15)	0.0011 (15)	0.0107 (14)	-0.0001 (13)
C2	0.057 (2)	0.053 (2)	0.0376 (15)	-0.0083 (17)	-0.0049 (15)	0.0042 (14)
C3	0.0436 (18)	0.056 (2)	0.0494 (18)	-0.0026 (16)	-0.0041 (15)	0.0095 (16)
C4	0.0351 (17)	0.0494 (19)	0.0493 (17)	0.0077 (14)	0.0058 (14)	0.0051 (14)
C5	0.0356 (16)	0.0347 (17)	0.0368 (14)	0.0037 (13)	0.0094 (12)	0.0068 (12)
C6	0.0361 (16)	0.0359 (15)	0.0367 (14)	0.0006 (13)	0.0081 (12)	0.0063 (12)
N2	0.0301 (12)	0.0408 (14)	0.0314 (11)	0.0064 (11)	0.0090 (9)	0.0002 (10)
C9	0.0335 (16)	0.0367 (16)	0.0325 (13)	0.0026 (13)	0.0077 (12)	0.0035 (12)
C10	0.0390 (17)	0.0477 (19)	0.0416 (15)	-0.0047 (14)	0.0122 (13)	0.0043 (14)
C11	0.0467 (18)	0.0506 (19)	0.0331 (14)	-0.0002 (15)	0.0153 (13)	0.0068 (13)
C12	0.0453 (18)	0.0410 (16)	0.0291 (13)	0.0023 (14)	0.0050 (12)	0.0026 (12)
C13	0.0379 (16)	0.0505 (19)	0.0434 (16)	-0.0070 (15)	0.0018 (13)	0.0042 (14)
C14	0.0341 (16)	0.0474 (19)	0.0389 (15)	-0.0011 (14)	0.0131 (12)	0.0086 (13)
C7	0.0316 (16)	0.0478 (18)	0.0390 (15)	0.0039 (15)	0.0083 (12)	0.0052 (14)
C8	0.0304 (15)	0.0447 (17)	0.0397 (15)	0.0077 (13)	0.0096 (12)	0.0039 (13)
O1	0.0482 (13)	0.0870 (17)	0.0452 (12)	0.0302 (12)	0.0080 (10)	-0.0133 (12)
N1	0.0372 (14)	0.0494 (16)	0.0377 (13)	0.0062 (12)	0.0117 (11)	-0.0007 (11)
Br1	0.0734 (3)	0.0652 (3)	0.03636 (18)	-0.00188 (19)	0.00039 (15)	-0.00357 (15)

Geometric parameters (Å, °)

C1—C2	1.380 (4)	C9—C14	1.385 (4)
C1—C6	1.389 (4)	C9—C10	1.387 (4)
C1—H1	0.9300	C10—C11	1.370 (4)
C2—C3	1.385 (4)	C10—H10	0.9300
C2—H2	0.9300	C11—C12	1.373 (4)
C3—C4	1.372 (4)	C11—H11	0.9300
C3—H3	0.9300	C12—C13	1.381 (4)
C4—C5	1.398 (4)	C12—Br1	1.892 (3)
C4—H4	0.9300	C13—C14	1.374 (4)
C5—C6	1.393 (4)	C13—H13	0.9300
C5—C7	1.451 (4)	C14—H14	0.9300
C6—N1	1.403 (4)	C7—O1	1.216 (3)
N2—C8	1.374 (3)	C8—N1	1.283 (4)
N2—C7	1.406 (3)	C8—H8	0.9300
N2—C9	1.437 (3)		
C2—C1—C6	119.4 (3)	C10—C9—N2	119.8 (2)
C2—C1—H1	120.3	C11—C10—C9	119.8 (3)
C6—C1—H1	120.3	C11—C10—H10	120.1
C1—C2—C3	120.7 (3)	C9—C10—H10	120.1
C1—C2—H2	119.6	C10—C11—C12	119.8 (3)
C3—C2—H2	119.6	C10—C11—H11	120.1
C4—C3—C2	120.3 (3)	C12—C11—H11	120.1
C4—C3—H3	119.9	C11—C12—C13	121.0 (3)
C2—C3—H3	119.9	C11—C12—Br1	119.1 (2)
C3—C4—C5	119.8 (3)	C13—C12—Br1	119.8 (2)
C3—C4—H4	120.1	C14—C13—C12	119.3 (3)
C5—C4—H4	120.1	C14—C13—H13	120.3
C6—C5—C4	119.7 (3)	C12—C13—H13	120.3
C6—C5—C7	120.4 (2)	C13—C14—C9	119.9 (2)
C4—C5—C7	119.9 (2)	C13—C14—H14	120.1
C1—C6—C5	120.1 (3)	C9—C14—H14	120.1
C1—C6—N1	118.2 (3)	O1—C7—N2	120.6 (3)
C5—C6—N1	121.6 (2)	O1—C7—C5	125.6 (3)
C8—N2—C7	120.9 (2)	N2—C7—C5	113.8 (2)
C8—N2—C9	119.5 (2)	N1—C8—N2	126.5 (3)
C7—N2—C9	119.7 (2)	N1—C8—H8	116.7
C14—C9—C10	120.1 (3)	N2—C8—H8	116.7
C14—C9—N2	120.1 (2)	C8—N1—C6	116.4 (2)
C6—C1—C2—C3	0.0 (4)	C10—C11—C12—Br1	177.9 (2)
C1—C2—C3—C4	0.9 (5)	C11—C12—C13—C14	1.3 (5)
C2—C3—C4—C5	-0.6 (5)	Br1—C12—C13—C14	-177.3 (2)
C3—C4—C5—C6	-0.5 (4)	C12—C13—C14—C9	-0.2 (4)
C3—C4—C5—C7	-179.7 (3)	C10—C9—C14—C13	-1.5 (4)
C2—C1—C6—C5	-1.1 (4)	N2—C9—C14—C13	179.7 (3)

C2—C1—C6—N1	178.2 (2)	C8—N2—C7—O1	-171.8 (3)
C4—C5—C6—C1	1.3 (4)	C9—N2—C7—O1	7.5 (4)
C7—C5—C6—C1	-179.5 (3)	C8—N2—C7—C5	6.9 (4)
C4—C5—C6—N1	-177.9 (2)	C9—N2—C7—C5	-173.8 (2)
C7—C5—C6—N1	1.3 (4)	C6—C5—C7—O1	172.8 (3)
C8—N2—C9—C14	48.8 (4)	C4—C5—C7—O1	-8.0 (4)
C7—N2—C9—C14	-130.5 (3)	C6—C5—C7—N2	-5.9 (4)
C8—N2—C9—C10	-130.0 (3)	C4—C5—C7—N2	173.3 (2)
C7—N2—C9—C10	50.7 (4)	C7—N2—C8—N1	-3.4 (4)
C14—C9—C10—C11	2.1 (4)	C9—N2—C8—N1	177.3 (3)
N2—C9—C10—C11	-179.1 (3)	N2—C8—N1—C6	-1.7 (4)
C9—C10—C11—C12	-1.1 (5)	C1—C6—N1—C8	-176.6 (3)
C10—C11—C12—C13	-0.6 (5)	C5—C6—N1—C8	2.7 (4)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C8—H8 \cdots N1 ⁱ	0.93	2.48	3.286 (4)	146
C11—H11 \cdots O1 ⁱⁱ	0.93	2.32	3.224 (4)	165

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