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(S)-2-(3-Nitrophenyl)-1,2-dihydroquinazolin-4(3H)-one

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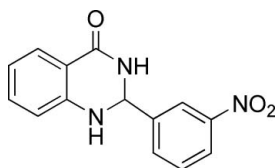
Received 20 November 2007; accepted 9 December 2007

Key indicators: single-crystal X-ray study; $T = 113$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.053; wR factor = 0.134; data-to-parameter ratio = 12.6.

In the racemic title compound, $\text{C}_{14}\text{H}_{11}\text{N}_3\text{O}_3$, the pyrimidine ring has an envelope conformation with the puckering parameters $Q = 0.3338$ (17) Å, $\Theta = 60.1$ (3) and $\varphi = 290.4$ (3)°. The two N—H groups form hydrogen bonds with symmetry-related molecules, building a two-dimensional network parallel to the (10 $\bar{1}$) plane.

Related literature

For related literature, see: Bernstein *et al.* (1995); Cremer & Pople (1975); Etter *et al.* (1990); Chen *et al.* (2007).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{11}\text{N}_3\text{O}_3$
 $M_r = 269.26$
 Monoclinic, $P2_1/n$
 $a = 10.9766$ (13) Å
 $b = 9.8626$ (9) Å
 $c = 11.7636$ (14) Å
 $\beta = 109.697$ (7)°

$V = 1199.0$ (2) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.11$ mm⁻¹
 $T = 113$ (2) K
 $0.16 \times 0.12 \times 0.10$ mm

Data collection

Rigaku Saturn diffractometer
 Absorption correction: multi-scan
 (*CrystalClear*; Rigaku, 2004)
 $T_{\min} = 0.981$, $T_{\max} = 0.988$

12847 measured reflections
 2358 independent reflections
 2209 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.043$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.053$
 $wR(F^2) = 0.134$
 $S = 1.15$
 2358 reflections
 187 parameters
 2 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.30$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.27$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{O1}^i$	0.857 (9)	2.075 (10)	2.9318 (19)	179.3 (19)
$\text{N2}-\text{H2}\cdots\text{O1}^{ii}$	0.853 (9)	2.165 (11)	2.9837 (18)	160.9 (17)

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

Data collection: *CrystalClear* (Rigaku, 2004); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-III* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *SHELXL97*.

We thank Beijing Institute of Technology for financial support and Naikai University for the X-ray diffraction analysis.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2300).

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supporting information

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(S)-2-(3-Nitrophenyl)-1,2-dihydroquinazolin-4(3H)-one

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S1. Comment

The title compound (I), $C_{14}H_{11}N_3O_3$, a derivative of the most useful 1,2-dihydroquinazolinones (Chen *et al.*, 2007), was synthesized directly from the reaction of 2-aminobenzonitrile and 3-nitrobenzaldehyde. In order to further confirm its structure and determine the correlation of structural features with biological activity, its single-crystal was undertaken.

The title compound (I), $C_{14}H_{11}N_3O_3$, is built up from dihydroquinazolin made by two six membered fused rings and a nitrophenyl ring linked through a C—C single bond (Fig. 1). The pyrimidine ring has an envelope conformation (Cremer & Pople, 1975) with the puckering Amplitude (Q) = 0.3338 (17) Å, $\Theta = 60.1$ (3) ° and $\varphi = 290.4$ (3) °.

The two N—H groups form O—H \cdots O hydrogen bonds with the ketone O atom of symmetry related molecules. Two N—H groups of symmetry related molecules form an $R_2^2(8)$ motif (Etter *et al.*, 1990; Bernstein *et al.*, 1995) whereas the other N—H group links these motifs to each other building a two dimensionnal network parallel to the (1 0 - 1) plane (Table 1, Fig. 2).

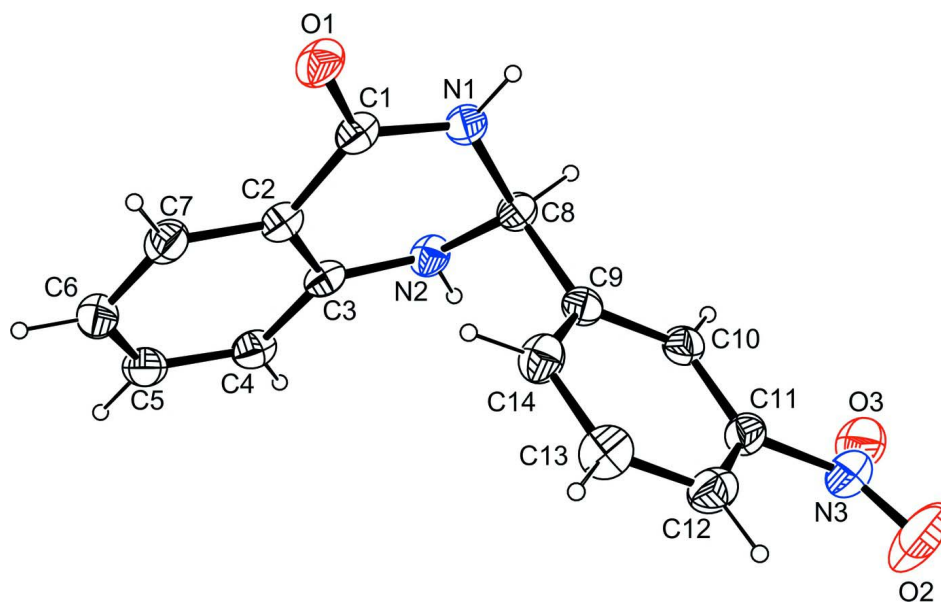
S2. Experimental

The title compound was obtained from the reaction of 2-aminobenzonitrile with 3-nitrobenzaldehyde in the present of zinc chloride, refluxing for 1.5 h in DMF and its single-crystal was cultured from a solution of ethanol by slow evaporation at room temperature.

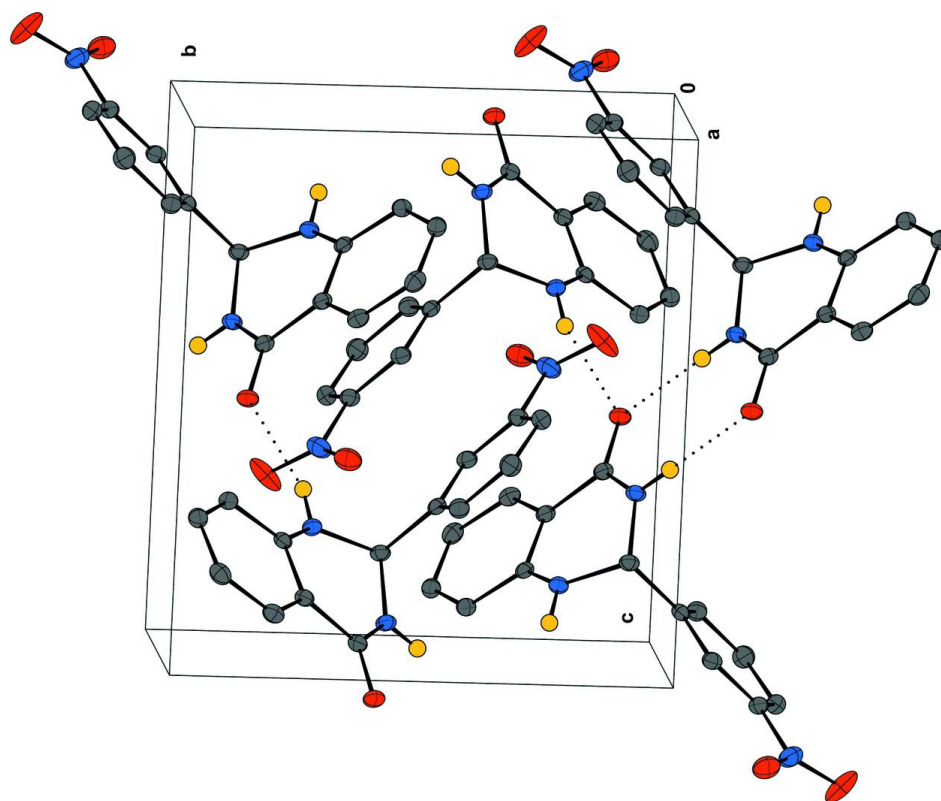
Mp. 210–212°C. Spectra data: IR (KBr, cm^{-1}): 3296, 3188, 1653, 1610, 1532, 1353; 1H NMR (DMSO- d_6) δ_H : 5.95 (1H, s, CH), 6.70 (1H, t, J=7.6 Hz, ArH), 6.79 (1H, d, J=8.0 Hz, ArH), 7.29 (1H, t, J=8.0 Hz, ArH), 7.35 (1H, s, NH), 7.62 (1H, dd, J=7.6 Hz, ArH), 7.70 (1H, t, J=7.6 Hz, ArH), 7.94 (1H, d, J=7.6 Hz, ArH), 8.21–8.22 (1H, m, J=1.4, 1.4 Hz, ArH), 8.36 (1H, t, J=1.8, 1.8 Hz, ArH), 8.53 (1H, s, NH); ^{13}C NMR (DMSO- d_6) δ_C : 65.20, 114.61, 114.97, 117.55, 121.59, 123.29, 127.43, 130.06, 133.39, 133.59, 144.32, 147.32, 147.73, 163.36; MS (ESI): m/z (%) =270.1 (100) [$M+H$] $^+$; $C_{14}H_{11}N_3O_3$: calcd. C 62.45, H 4.12, N 15.61; found C 62.16, H 4.20, N 15.24.

S3. Refinement

All H atoms attached to C atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic) or 0.98 Å (methine) with $U_{iso}(H) = 1.2U_{eq}(C)$. H atoms attached to N were located in difference Fourier maps and included in the subsequent refinement using restraints (O—N = 0.85 (1) Å) with $U_{iso}(H) = 1.2U_{eq}(N)$.

**Figure 1**

The molecular structure of (I) with atom the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

**Figure 2**

Partial packing showing one sheet of molecules connected by N—H...O hydrogen bonds (dashed lines). H atoms not involved in hydrogen bonding have been omitted for clarity.

(S)-2-(3-Nitrophenyl)-1,2-dihydroquinazolin-4(3H)-one*Crystal data*C₁₄H₁₁N₃O₃ $M_r = 269.26$ Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

 $a = 10.9766$ (13) Å $b = 9.8626$ (9) Å $c = 11.7636$ (14) Å $\beta = 109.697$ (7)° $V = 1199.0$ (2) Å³ $Z = 4$ $F(000) = 560$ $D_x = 1.492$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71070$ Å

Cell parameters from 4115 reflections

 $\theta = 1.8$ – 27.9 ° $\mu = 0.11$ mm⁻¹ $T = 113$ K

Block, yellow

 $0.16 \times 0.12 \times 0.10$ mm*Data collection*Rigaku Saturn
diffractometer

Radiation source: rotating anode

Confocal monochromator

Detector resolution: 14.63 pixels mm⁻¹ ω scans

Absorption correction: multi-scan

(CrystalClear; Rigaku, 2004)

 $T_{\min} = 0.981$, $T_{\max} = 0.988$

12847 measured reflections

2358 independent reflections

2209 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.043$ $\theta_{\text{max}} = 26.0$ °, $\theta_{\text{min}} = 2.2$ ° $h = -13$ → 13 $k = -12$ → 12 $l = -14$ → 14 *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.053$ $wR(F^2) = 0.134$ $S = 1.15$

2358 reflections

187 parameters

2 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sitesH atoms treated by a mixture of independent
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.0628P)^2 + 0.2875P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} = 0.001$ $\Delta\rho_{\text{max}} = 0.30$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.27$ e Å⁻³*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.

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.38313 (11)	0.37578 (12)	0.01748 (10)	0.0300 (3)
O2	0.88573 (16)	0.82212 (18)	0.63964 (16)	0.0661 (6)
O3	1.00163 (13)	0.66568 (15)	0.59857 (12)	0.0405 (4)

N1	0.59073 (14)	0.40329 (15)	0.13946 (12)	0.0251 (3)
H1	0.5980 (18)	0.4683 (15)	0.0939 (15)	0.030*
N2	0.69081 (13)	0.25348 (14)	0.30194 (13)	0.0241 (3)
H2	0.7543 (14)	0.2352 (19)	0.3656 (12)	0.029*
N3	0.89721 (16)	0.71853 (17)	0.58678 (14)	0.0369 (4)
C1	0.47429 (16)	0.34319 (17)	0.11042 (15)	0.0248 (4)
C2	0.46290 (16)	0.23302 (17)	0.19167 (14)	0.0237 (4)
C3	0.57453 (16)	0.19070 (17)	0.28587 (14)	0.0226 (4)
C4	0.56527 (17)	0.07855 (17)	0.35660 (15)	0.0265 (4)
H4	0.6379	0.0482	0.4185	0.032*
C5	0.44777 (17)	0.01378 (18)	0.33363 (16)	0.0287 (4)
H5	0.4425	-0.0606	0.3803	0.034*
C6	0.33707 (18)	0.05748 (18)	0.24215 (16)	0.0299 (4)
H6	0.2584	0.0138	0.2288	0.036*
C7	0.34564 (17)	0.16652 (18)	0.17147 (16)	0.0272 (4)
H7	0.2723	0.1958	0.1098	0.033*
C8	0.69237 (16)	0.39113 (17)	0.25765 (14)	0.0235 (4)
H8	0.7758	0.4049	0.2459	0.028*
C9	0.67990 (15)	0.49851 (16)	0.34659 (14)	0.0227 (4)
C10	0.79123 (16)	0.55921 (17)	0.42414 (15)	0.0248 (4)
H10	0.8723	0.5349	0.4220	0.030*
C11	0.77919 (17)	0.65619 (17)	0.50431 (15)	0.0269 (4)
C12	0.66122 (18)	0.69635 (18)	0.51111 (16)	0.0306 (4)
H12	0.6560	0.7633	0.5649	0.037*
C13	0.55171 (18)	0.63387 (19)	0.43545 (17)	0.0326 (4)
H13	0.4711	0.6573	0.4391	0.039*
C14	0.56071 (17)	0.53628 (18)	0.35387 (16)	0.0282 (4)
H14	0.4858	0.4954	0.3031	0.034*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0275 (7)	0.0325 (7)	0.0214 (6)	-0.0004 (5)	-0.0029 (5)	0.0017 (5)
O2	0.0589 (11)	0.0685 (12)	0.0687 (12)	-0.0165 (9)	0.0185 (9)	-0.0473 (10)
O3	0.0295 (8)	0.0512 (9)	0.0354 (8)	-0.0077 (6)	0.0040 (6)	-0.0056 (6)
N1	0.0249 (8)	0.0281 (8)	0.0182 (7)	-0.0030 (6)	0.0018 (6)	0.0025 (6)
N2	0.0206 (7)	0.0258 (8)	0.0206 (7)	0.0013 (6)	0.0001 (6)	0.0013 (6)
N3	0.0398 (10)	0.0399 (10)	0.0290 (9)	-0.0098 (8)	0.0089 (7)	-0.0087 (7)
C1	0.0261 (9)	0.0254 (9)	0.0202 (9)	0.0000 (7)	0.0042 (7)	-0.0027 (7)
C2	0.0248 (9)	0.0246 (9)	0.0194 (8)	0.0003 (7)	0.0047 (7)	-0.0027 (7)
C3	0.0260 (9)	0.0227 (8)	0.0183 (8)	0.0012 (7)	0.0062 (7)	-0.0044 (6)
C4	0.0316 (9)	0.0254 (9)	0.0210 (9)	0.0025 (7)	0.0070 (7)	-0.0004 (7)
C5	0.0393 (10)	0.0244 (9)	0.0246 (9)	-0.0023 (8)	0.0138 (8)	-0.0009 (7)
C6	0.0305 (10)	0.0310 (10)	0.0309 (10)	-0.0069 (8)	0.0138 (8)	-0.0067 (7)
C7	0.0239 (9)	0.0313 (9)	0.0246 (9)	-0.0011 (7)	0.0057 (7)	-0.0046 (7)
C8	0.0213 (8)	0.0277 (9)	0.0183 (8)	-0.0019 (7)	0.0026 (7)	0.0009 (6)
C9	0.0258 (9)	0.0219 (8)	0.0188 (8)	-0.0004 (7)	0.0056 (7)	0.0044 (6)
C10	0.0253 (9)	0.0280 (9)	0.0204 (8)	-0.0023 (7)	0.0066 (7)	0.0023 (7)

C11	0.0305 (10)	0.0267 (9)	0.0205 (9)	-0.0050 (7)	0.0045 (7)	0.0000 (7)
C12	0.0397 (11)	0.0254 (9)	0.0256 (9)	0.0033 (8)	0.0097 (8)	-0.0014 (7)
C13	0.0286 (10)	0.0329 (10)	0.0343 (10)	0.0066 (8)	0.0079 (8)	0.0000 (8)
C14	0.0253 (9)	0.0279 (9)	0.0271 (9)	0.0009 (7)	0.0034 (7)	0.0000 (7)

Geometric parameters (Å, °)

O1—C1	1.250 (2)	C5—H5	0.9300
O2—N3	1.224 (2)	C6—C7	1.382 (2)
O3—N3	1.224 (2)	C6—H6	0.9300
N1—C1	1.344 (2)	C7—H7	0.9300
N1—C8	1.465 (2)	C8—C9	1.527 (2)
N1—H1	0.857 (9)	C8—H8	0.9800
N2—C3	1.373 (2)	C9—C10	1.390 (2)
N2—C8	1.456 (2)	C9—C14	1.390 (2)
N2—H2	0.853 (9)	C10—C11	1.381 (2)
N3—C11	1.466 (2)	C10—H10	0.9300
C1—C2	1.480 (2)	C11—C12	1.382 (3)
C2—C7	1.392 (2)	C12—C13	1.377 (3)
C2—C3	1.410 (2)	C12—H12	0.9300
C3—C4	1.408 (2)	C13—C14	1.386 (3)
C4—C5	1.382 (2)	C13—H13	0.9300
C4—H4	0.9300	C14—H14	0.9300
C5—C6	1.392 (3)		
C1—N1—C8	124.04 (14)	C6—C7—C2	120.70 (16)
C1—N1—H1	116.9 (13)	C6—C7—H7	119.7
C8—N1—H1	116.9 (13)	C2—C7—H7	119.7
C3—N2—C8	119.58 (14)	N2—C8—N1	108.59 (13)
C3—N2—H2	118.2 (13)	N2—C8—C9	112.76 (13)
C8—N2—H2	114.0 (13)	N1—C8—C9	112.21 (13)
O3—N3—O2	123.41 (17)	N2—C8—H8	107.7
O3—N3—C11	118.77 (15)	N1—C8—H8	107.7
O2—N3—C11	117.82 (17)	C9—C8—H8	107.7
O1—C1—N1	121.41 (16)	C10—C9—C14	118.78 (16)
O1—C1—C2	122.46 (15)	C10—C9—C8	119.10 (15)
N1—C1—C2	116.08 (14)	C14—C9—C8	122.11 (15)
C7—C2—C3	120.22 (16)	C11—C10—C9	118.77 (16)
C7—C2—C1	120.68 (15)	C11—C10—H10	120.6
C3—C2—C1	118.99 (15)	C9—C10—H10	120.6
N2—C3—C4	121.45 (15)	C10—C11—C12	123.05 (16)
N2—C3—C2	119.73 (15)	C10—C11—N3	118.41 (16)
C4—C3—C2	118.70 (16)	C12—C11—N3	118.53 (16)
C5—C4—C3	119.74 (16)	C13—C12—C11	117.73 (17)
C5—C4—H4	120.1	C13—C12—H12	121.1
C3—C4—H4	120.1	C11—C12—H12	121.1
C4—C5—C6	121.46 (17)	C12—C13—C14	120.52 (17)
C4—C5—H5	119.3	C12—C13—H13	119.7

C6—C5—H5	119.3	C14—C13—H13	119.7
C7—C6—C5	119.16 (16)	C13—C14—C9	121.12 (16)
C7—C6—H6	120.4	C13—C14—H14	119.4
C5—C6—H6	120.4	C9—C14—H14	119.4

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N1—H1...O1 ⁱ	0.86 (1)	2.08 (1)	2.9318 (19)	179 (2)
N2—H2...O1 ⁱⁱ	0.85 (1)	2.17 (1)	2.9837 (18)	161 (2)

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