organic compounds

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

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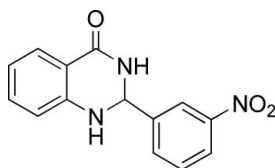Correspondence e-mail: jrli@bit.edu.cn

In the racemic title compound, $C_{14}H_{11}N_3O_3$, 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

$C_{14}H_{11}N_3O_3$	$V = 1199.0$ (2) Å^3
$M_r = 269.26$	$Z = 4$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 10.9766$ (13) Å	$\mu = 0.11$ mm^{-1}
$b = 9.8626$ (9) Å	$T = 113$ (2) K
$c = 11.7636$ (14) Å	$0.16 \times 0.12 \times 0.10$ mm
$\beta = 109.697$ (7)°	

Data collection

Rigaku Saturn diffractometer	12847 measured reflections
Absorption correction: multi-scan	2358 independent reflections
(*CrystalClear*; Rigaku, 2004)	2209 reflections with $I > 2\sigma(I)$
$T_{min} = 0.981$, $T_{max} = 0.988$	$R_{int} = 0.043$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.053$	H atoms treated by a mixture of
$wR(F^2) = 0.134$	independent and constrained
$S = 1.15$	refinement
2358 reflections	$\Delta\rho_{max} = 0.30$ e Å^{-3}
187 parameters	$\Delta\rho_{min} = -0.27$ e Å^{-3}
2 restraints	

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$\cdots$O1^i	0.857 (9)	2.075 (10)	2.9318 (19)	179.3 (19)
N2—H2$\cdots$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: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *SHELXL97*.

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

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII.* Report ORNL-6895. Oak Ridge National Laboratory, Oak Ridge, Tennessee, USA.
Chen, K., Al Aowad, A. F., Adelstein, S. J. & Kassis, A. I. (2007). *J. Med. Chem.* **50**, 663–673.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* B**46**, 256–262.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Rigaku (2004). *CrystalClear.* Version 1.36. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97.* University of Göttingen, Germany.
Watkin, D. M., Pearce, L. & Prout, C. K. (1993). *CAMERON.* Chemical Crystallography Laboratory, University of Oxford, England.

supplementary materials

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

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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 enveloppe conformation (Cremer & Pople, 1975) with the puckering Amplitude (Q) = 0.3338 (17) Å, Θ = 60.1 (3) ° and φ = 290.4 (3) °.

The two N—H groups form O—H···O hydrogen bonds with the ketone O atom of symmetry related molecules. Two N—H groups of symmetry related moleules 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).

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; ^{1}H NMR (DMSO-d_6) δ_H: 5.95 (1*H*, s, CH), 6.70 (1*H*, t, J=7.6 Hz, ArH), 6.79 (1*H*, d, J=8.0 Hz, ArH), 7.29 (1*H*, t, J=8.0 Hz, ArH), 7.35 (1*H*, s, NH), 7.62 (1*H*, dd, J=7.6 Hz, ArH) 7.70 (1*H*, t, J=7.6 Hz, ArH), 7.94 (1*H*, d, J=7.6 Hz, ArH), 8.21–8.22 (1*H*, m, J=1.4, 1.4 Hz, ArH), 8.36 (1*H*, t, J=1.8, 1.8 Hz, ArH), 8.53 (1*H*, 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.

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 $U_{iso}(H) = 1.2U_{eq}(N)$.
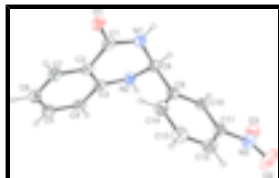
Figures

Fig. 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.
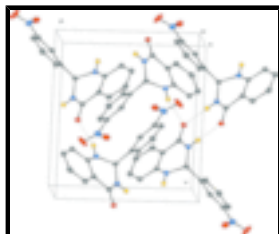

Fig. 2. Partial packing showing one sheet of molecules connected by N—H···O hydrogen bonds (dashed lines). H atoms not involved in hydrogen bondings have been omitted for clarity.

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

Crystal data

$C_{14}H_{11}N_3O_3$	$F_{000} = 560$
$M_r = 269.26$	$D_x = 1.492$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation $\lambda = 0.71070$ Å
Hall symbol: -P 2yn	Cell parameters from 4115 reflections
$a = 10.9766$ (13) Å	$\theta = 1.8–27.9°$
$b = 9.8626$ (9) Å	$\mu = 0.11$ mm^{-1}
$c = 11.7636$ (14) Å	$T = 113$ (2) K
$\beta = 109.697$ (7)°	Block, yellow
$V = 1199.0$ (2) Å^3	$0.16 \times 0.12 \times 0.10$ mm
$Z = 4$	

Data collection

Rigaku Saturn diffractometer	2358 independent reflections
Radiation source: rotating anode	2209 reflections with $I > 2\sigma(I)$
Monochromator: confocal	$R_{int} = 0.043$
Detector resolution: 14.63 pixels mm^{-1}	$\theta_{max} = 26.0°$
$T = 113(2)$ K	$\theta_{min} = 2.2°$
ω scans	$h = -13 \rightarrow 13$
Absorption correction: multi-scan (CrystalClear; Rigaku, 2004)	$k = -12 \rightarrow 12$
$T_{min} = 0.981$, $T_{max} = 0.988$	$l = -14 \rightarrow 14$
12847 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.053$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.134$	$w = 1/[\sigma^2(F_o^2) + (0.0628P)^2 + 0.2875P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.15$	$(\Delta/\sigma)_{max} = 0.001$
2358 reflections	$\Delta\rho_{max} = 0.30$ e Å^{-3}
187 parameters	$\Delta\rho_{min} = -0.27$ e Å^{-3}
2 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2$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 (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
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 (Å^2)

	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, °)

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1[i]	0.857 (9)	2.075 (10)	2.9318 (19)	179.3 (19)
N2—H2···O1[ii]	0.853 (9)	2.165 (11)	2.9837 (18)	160.9 (17)

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

Fig. 1

Fig. 2