Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Quinazoline-2,4(1*H*,3*H*)-dione

Ge Liu

Chifeng University, Chifeng 024000, People's Republic of China
Correspondence e-mail: liu_ge2008@163.com

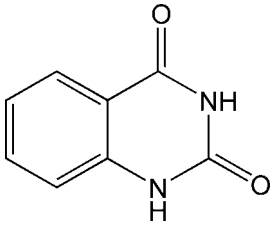
Received 5 July 2008; accepted 30 July 2008

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.055; wR factor = 0.104; data-to-parameter ratio = 11.5.

In the title compound, $C_8H_6N_2O_2$, intermolecular N—H···O hydrogen bonds involving the amine and carbonyl groups create centrosymmetric dimers between adjacent nearly coplanar molecules. These dimers are further connected by weak N—H···O hydrogen bonds, forming a two-dimensional network. Molecules are packed in the crystal structure with adjacent benzene and pyrimidine rings approximately coplanar; the centroid–centroid separation is 3.863 Å and the dihedral angle between the mean planes is 0.64°, indicating the presence of weak intermolecular face-to-face π–π stacking interactions.

Related literature

For background, see: Goto *et al.* (1993); Mohri (2001); For further synthetic details, see: Mizuno *et al.* (2007).



Experimental

Crystal data

$C_8H_6N_2O_2$
M_r = 162.15
Monoclinic, $P2_1/c$
a = 10.891 (2) Å
b = 5.2810 (11) Å
c = 12.701 (3) Å
β = 99.61 (3)°
V = 720.2 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 (1) K
0.20 × 0.18 × 0.15 mm

Data collection

Rigaku R-AXIS RAPID-S diffractometer
Absorption correction: none
5683 measured reflections
1262 independent reflections
869 reflections with $I > 2\sigma(I)$
R_{int} = 0.051

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.103
S = 1.06
1262 reflections
110 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2B···O1^{i}	0.86	2.00	2.854	176
N1—H1A···O1^{ii}	0.86	2.13	2.976	168

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The author thanks Chifeng University for supporting this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2183).

References

Goto, S., Tsuboi, H. & Kagara, K. (1993). *Chem. Express*, **8**, 761–764.
Mizuno, T., Mihara, M., Nakai, T., Iwai, T. & Ito, T. (2007). *Synthesis*, pp. 2524–2528.
Mohri, S. J. (2001). *Synth. Org. Chem. Jpn*, **59**, 514–515.
Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation,Tokyo, Japan.
Rigaku/MSC (2002). *CrystalStructure*. Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2008). E**64**, o1677 [**doi:10.1107/S1600536808024240**]

Quinazoline-2,4(1*H*,3*H*)-dione

G. Liu

Comment

2,4(1*H*,3*H*)-Quinazolinedione derivatives are of interest because of their biological activity, and they have been widely used as key structures in medicinal drugs (Goto *et al.*, 1993; Mohri, 2001; Mizuno *et al.*, 2007). We herein report the crystal structure of the title compound (I). In the molecule (Fig. 1), the bond lengths and angles are within normal ranges. Intermolecular N—H···O hydrogen bonds involving amine NH and carbonyl groups O atoms form a two dimensional network (Table 1 and Fig. 2). Weak π–π stacking interactions are also observed in the crystal structure.

Experimental

To a 100-ml, 3-necked flask equipped with condenser, were added 2-aminobenzonitrile (5.91 g, 50 mmol) and 1,8-diazabicyclo(5.4.0)undec-7-ene (DBU, 1.50 ml, 10 mmol) under argon, together with a large magnetic stirring bar. Then, CO_2 (1 bar) was charged at 293 K. The mixture was vigorously stirred under CO_2 (1 bar) at 423 K for 4 h. The resulting white solid was then poured into 1 *M* HCl (100 ml) and washed with *t*-BuOMe (200 ml) to give pure (I). Single crystals of (I) were obtained from a water solution, at room temperature, by slow evaporation.

Refinement

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with N—H = 0.86 Å, C—H = 0.93 Å, and with U_{iso}(H) = 1.2U_{eq}(parent atom).

Figures

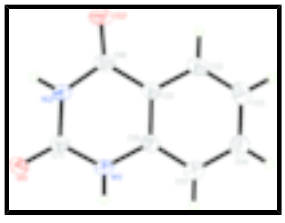


Fig. 1. The structure of the title compound with 30% displacement probability ellipsoids.

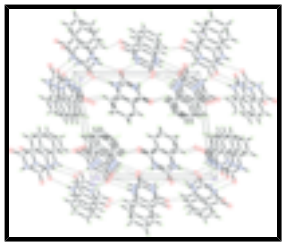


Fig. 2. Two dimensional sheet formed by hydrogen bonds (dashed lines) in the title compound.

Quinazoline-2,4(1*H*,3*H*)-dione

Crystal data

$C_8H_6N_2O_2$	F_{000} = 336
M_r = 162.15	D_x = 1.495 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 5473 reflections
a = 10.891 (2) Å	θ = 3.3–27.5º
b = 5.2810 (11) Å	μ = 0.11 mm^{-1}
c = 12.701 (3) Å	T = 293 (1) K
β = 99.61 (3)º	Block, colorless
V = 720.2 (3) Å^3	0.20 × 0.18 × 0.15 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID-S diffractometer	869 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.051
Monochromator: graphite	θ_{max} = 25.0º
T = 293(1) K	θ_{min} = 3.3º
ω scans	h = −12→12
Absorption correction: none	k = −6→6
5683 measured reflections	l = −15→15
1262 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.055	$w = 1/[\sigma^2(F_o^2) + (0.0327P)^2 + 0.2501P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.103	$(\Delta/\sigma)_{max}$ = 0.004
S = 1.07	$\Delta\rho_{max}$ = 0.12 e Å^{-3}
1262 reflections	$\Delta\rho_{min}$ = −0.12 e Å^{-3}
110 parameters	Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.009 (3)
Secondary atom site location: difference Fourier map	

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.2762 (2)	0.9421 (4)	0.57398 (17)	0.0395 (6)
N1	0.11953 (17)	1.0471 (4)	0.67998 (14)	0.0453 (6)
H1A	0.0866	1.0176	0.7357	0.054*
O1	−0.01889 (16)	1.3655 (3)	0.62957 (12)	0.0546 (5)
C2	0.3772 (2)	0.7919 (5)	0.5574 (2)	0.0494 (7)
H2A	0.4148	0.8190	0.4977	0.059*
N2	0.12516 (17)	1.2702 (4)	0.52514 (14)	0.0442 (6)
H2B	0.0924	1.3842	0.4809	0.053*
O2	0.26963 (17)	1.2019 (3)	0.41910 (13)	0.0633 (6)
C3	0.4210 (2)	0.6050 (5)	0.6289 (2)	0.0560 (8)
H3A	0.4884	0.5060	0.6179	0.067*
C4	0.3646 (2)	0.5637 (5)	0.7176 (2)	0.0540 (7)
H4A	0.3943	0.4359	0.7655	0.065*
C5	0.2655 (2)	0.7091 (5)	0.7357 (2)	0.0502 (7)
H5A	0.2287	0.6809	0.7956	0.060*
C6	0.2210 (2)	0.8984 (4)	0.66362 (17)	0.0388 (6)
C7	0.0702 (2)	1.2336 (5)	0.61374 (18)	0.0419 (6)
C8	0.2277 (2)	1.1435 (5)	0.49911 (18)	0.0444 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0414 (14)	0.0406 (14)	0.0384 (13)	−0.0030 (12)	0.0121 (11)	−0.0038 (12)
N1	0.0517 (13)	0.0533 (13)	0.0344 (11)	0.0086 (11)	0.0179 (10)	0.0109 (10)
O1	0.0556 (11)	0.0681 (13)	0.0450 (10)	0.0219 (10)	0.0221 (8)	0.0109 (9)
C2	0.0500 (16)	0.0520 (17)	0.0499 (16)	0.0001 (14)	0.0191 (13)	−0.0075 (14)
N2	0.0538 (13)	0.0474 (13)	0.0346 (11)	0.0079 (10)	0.0169 (10)	0.0077 (10)
O2	0.0853 (14)	0.0669 (13)	0.0469 (10)	0.0062 (11)	0.0380 (10)	0.0071 (10)
C3	0.0486 (16)	0.0527 (17)	0.0670 (19)	0.0098 (14)	0.0107 (15)	−0.0099 (16)
C4	0.0556 (17)	0.0519 (17)	0.0532 (16)	0.0063 (14)	0.0049 (14)	0.0017 (14)
C5	0.0523 (16)	0.0550 (17)	0.0445 (15)	0.0044 (14)	0.0117 (12)	0.0057 (13)
C6	0.0385 (14)	0.0404 (14)	0.0378 (13)	−0.0006 (12)	0.0077 (11)	−0.0037 (12)
C7	0.0456 (15)	0.0482 (15)	0.0336 (13)	0.0018 (13)	0.0118 (12)	0.0019 (12)
C8	0.0547 (17)	0.0442 (15)	0.0379 (14)	−0.0022 (13)	0.0181 (12)	−0.0070 (12)

Geometric parameters (Å, °)

C1—C6	1.393 (3)	N2—C8	1.389 (3)
C1—C2	1.401 (3)	N2—H2B	0.8600
C1—C8	1.465 (3)	O2—C8	1.221 (3)
N1—C7	1.348 (3)	C3—C4	1.388 (3)
N1—C6	1.399 (3)	C3—H3A	0.9300
N1—H1A	0.8600	C4—C5	1.375 (3)
O1—C7	1.238 (3)	C4—H4A	0.9300
C2—C3	1.372 (3)	C5—C6	1.387 (3)

C2—H2A	0.9300	C5—H5A	0.9300
N2—C7	1.373 (3)		
C6—C1—C2	119.1 (2)	C5—C4—C3	120.9 (3)
C6—C1—C8	119.6 (2)	C5—C4—H4A	119.5
C2—C1—C8	121.3 (2)	C3—C4—H4A	119.5
C7—N1—C6	124.0 (2)	C4—C5—C6	119.3 (2)
C7—N1—H1A	118.0	C4—C5—H5A	120.3
C6—N1—H1A	118.0	C6—C5—H5A	120.3
C3—C2—C1	120.2 (2)	C5—C6—C1	120.5 (2)
C3—C2—H2A	119.9	C5—C6—N1	120.3 (2)
C1—C2—H2A	119.9	C1—C6—N1	119.2 (2)
C7—N2—C8	127.2 (2)	O1—C7—N1	123.4 (2)
C7—N2—H2B	116.4	O1—C7—N2	121.0 (2)
C8—N2—H2B	116.4	N1—C7—N2	115.6 (2)
C2—C3—C4	119.9 (2)	O2—C8—N2	120.2 (2)
C2—C3—H3A	120.0	O2—C8—C1	125.4 (2)
C4—C3—H3A	120.0	N2—C8—C1	114.3 (2)
C6—C1—C2—C3	−0.1 (4)	C7—N1—C6—C1	0.0 (3)
C8—C1—C2—C3	179.8 (2)	C6—N1—C7—O1	−179.3 (2)
C1—C2—C3—C4	0.3 (4)	C6—N1—C7—N2	0.9 (3)
C2—C3—C4—C5	−0.5 (4)	C8—N2—C7—O1	177.6 (2)
C3—C4—C5—C6	0.4 (4)	C8—N2—C7—N1	−2.7 (3)
C4—C5—C6—C1	−0.3 (4)	C7—N2—C8—O2	−177.4 (2)
C4—C5—C6—N1	179.4 (2)	C7—N2—C8—C1	3.1 (3)
C2—C1—C6—C5	0.1 (3)	C6—C1—C8—O2	178.7 (2)
C8—C1—C6—C5	−179.9 (2)	C2—C1—C8—O2	−1.3 (4)
C2—C1—C6—N1	−179.6 (2)	C6—C1—C8—N2	−1.8 (3)
C8—C1—C6—N1	0.4 (3)	C2—C1—C8—N2	178.2 (2)
C7—N1—C6—C5	−179.7 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2B···O1^{i}	0.86	2.00	2.854	176
N1—H1A···O1^{ii}	0.86	2.13	2.976	168

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

Fig. 1

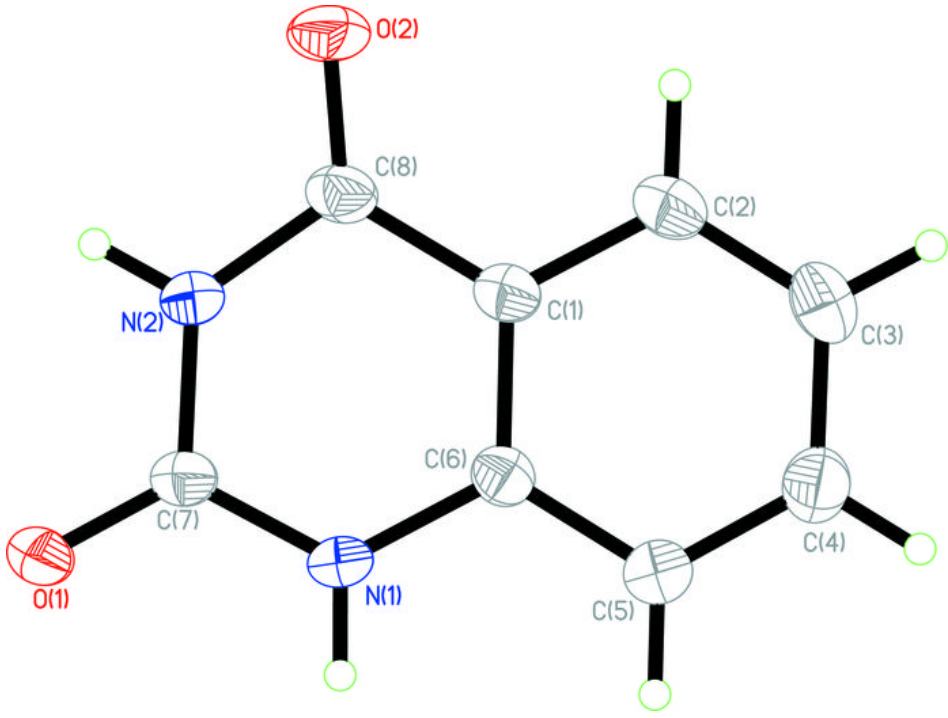


Fig. 2