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

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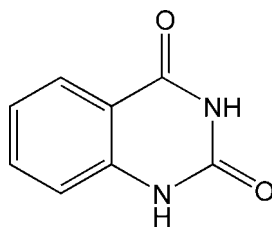
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.055; wR factor = 0.104; data-to-parameter ratio = 11.5.

In the title compound, $\text{C}_8\text{H}_6\text{N}_2\text{O}_2$, intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds involving the amine and carbonyl groups create centrosymmetric dimers between adjacent nearly coplanar molecules. These dimers are further connected by weak $\text{N}-\text{H}\cdots\text{O}$ 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 $\pi-\pi$ 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

$\text{C}_8\text{H}_6\text{N}_2\text{O}_2$	$V = 720.2$ (3) Å ³
$M_r = 162.15$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 10.891$ (2) Å	$\mu = 0.11$ mm ⁻¹
$b = 5.2810$ (11) Å	$T = 293$ (1) K
$c = 12.701$ (3) Å	$0.20 \times 0.18 \times 0.15$ mm
$\beta = 99.61$ (3)°	

Data collection

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

Refinement

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

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H2B}\cdots\text{O1}^{\text{i}}$	0.86	2.00	2.854	176
$\text{N1}-\text{H1A}\cdots\text{O1}^{\text{ii}}$	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/MS, 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

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supplementary materials

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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. Inter-molecular 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, CO₂ (1 bar) was charged at 293 K. The mixture was vigorously stirred under CO₂ (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 $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$.

Figures

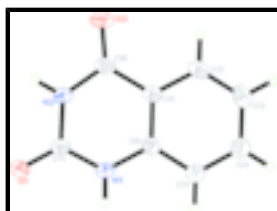


Fig. 1. The structure of the title compound with 30% displacement probability ellipsoids.

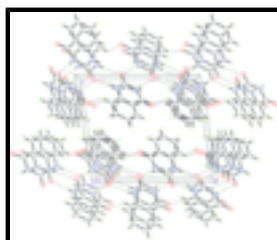


Fig. 2. Two dimensional sheet formed by hydrogen bonds (dashed lines) in the title compound.

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

Crystal data

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

Data collection

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

Refinement

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

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
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 (\AA^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 (\AA , $^\circ$)

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)

supplementary materials

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 (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2B \cdots O1 ⁱ	0.86	2.00	2.854	176
N1—H1A \cdots 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

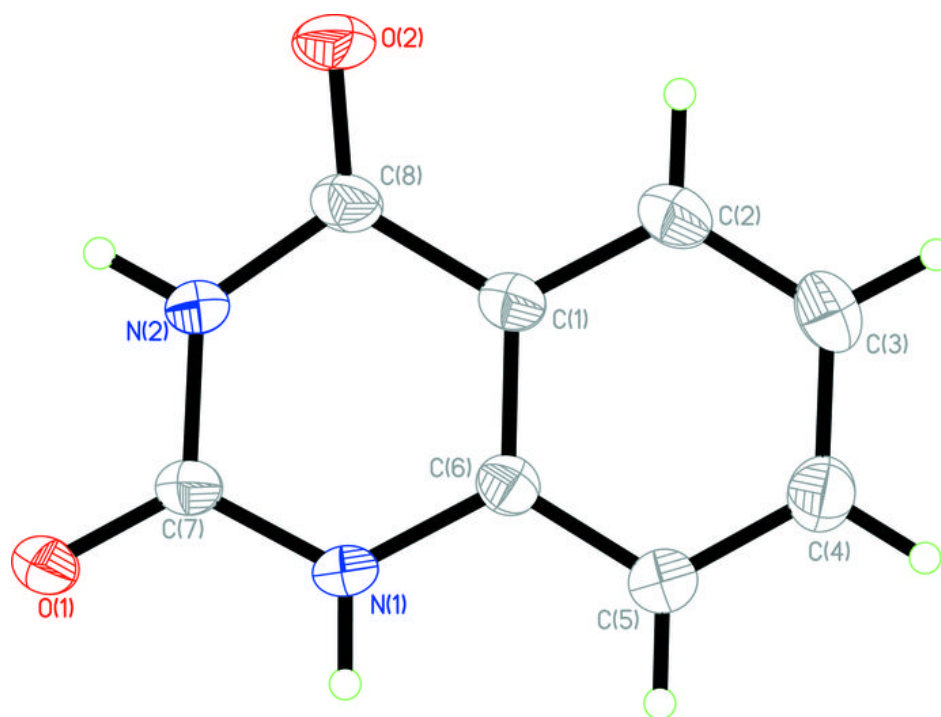


Fig. 2

