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7-Nitroquinazolin-4(3*H*)-one

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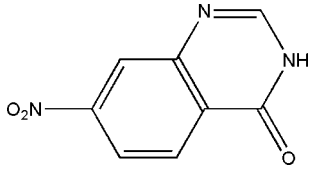
Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.002 Å; R factor = 0.034; wR factor = 0.096; data-to-parameter ratio = 10.6.

In the crystal structure of the title compound, $C_8H_5N_3O_3$, intermolecular N—H···O hydrogen bonds link molecules into centrosymmetric dimers. These dimers are, in turn, linked though weak intermolecular C—H···O and C—H···N hydrogen bonds and π–π stacking interactions, with centroid–centroid distances of 3.678 (3) Å, into a three-dimensional network.

Related literature

For related literature on biological activity, see: Masanori *et al.* (2003); Wolfe *et al.* (1990). For related structures, see: Chadwick & Easton (1983); Etter (1983).



Experimental

Crystal data

$C_8H_5N_3O_3$
M_r = 191.15
Monoclinic, $P2_1/n$
a = 5.1063 (10) Å
b = 11.206 (2) Å
c = 13.528 (3) Å
β = 99.19 (3)°
V = 764.1 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 153 (2) K
0.24 × 0.18 × 0.16 mm

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.969, T_{max} = 0.979
5749 measured reflections
1340 independent reflections
1215 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.096
S = 1.11
1340 reflections
127 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O3^{i}	0.88	1.98	2.8514 (14)	169
C1—H1B···O2^{ii}	0.95	2.54	3.2703 (17)	134
C1—H1B···O1^{iii}	0.95	2.55	3.0978 (17)	117
C5—H5A···O2^{iv}	0.95	2.49	3.2846 (16)	142
C7—H7A···N1^{ii}	0.95	2.55	3.4402 (18)	155

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

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2576).

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supporting information

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7-Nitroquinazolin-4(3*H*)-one

Jian-Ping Yong, Guan-Ping Yu, Jiu-Ming Li, Xue-Ling Hou and Haji Akber Aisa

S1. Comment

7-Nitro-4(3*H*)-Quinazolinone (I), is an important intermediate for drugs synthesis and its derivatives show many biological activities including anti-fungal, anti-convulsant (Masanori *et al.*, 2003), anti-bacterial, anti-cancer, anti-inflammatory, and anti-tumor (Wolfe *et al.*, 1990). We report here the crystal structure of (I) (Fig. 1).

In (I) (Fig. 1), all bond lengths and angles are normal and in a good agreement with those reported previously (Chadwick & Easton, 1983; Etter, 1983). Atoms N3 and O3 lie in the 1,2-dihydroquinazoline ring (C1—C8/N1/N2) plane, and the deviations from the least-squares plane through the ring atoms are all smaller than 0.026 (2) Å. The relatively short distances of 3.678 (3)Å between the centroids of 1,2-dihydropyrimidine (C1/C2/C3/C8/N1/N2) and benzene (C3—C8) rings related by (1 + *x*, *y*, *x*) indicates the presence of weak π-π interactions. In the crystal structure, intermolecular N—H···O hydrogen bonds link molecules into centrosymmetric dimers. These dimers, are in turn, linked though weak intermolecular C—H···O and C—H···N hydrogen bonds and $\pi\cdots\pi$ stacking interactions into a three-dimensional network.

S2. Experimental

The title compound was synthesized by the reaction of 4-nitro-2-amino-benezic acid 18.2 g (0.1 mol) and formamidine acetate 10.1 g (0.2 mol) in 100 mL andryous EtOH, refulxing for 6 h. The solid filtrated and washed with 20 ml H_2O, cool 30 ml EtOH and 30 ml e ther, respectively, dried under vacuum to obtain the title compound 15.8 g, yield: 82.8%. Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation the solution of 7-Nitro-4(3*H*)-Quinazolinone in EtOH/acetone/THF (1:1:1 V/V/V) at room temperature over a period of one week.

S3. Refinement

The H atoms were placed in calculated positions, with C—H = 0.95 Å, N—H = 0.88Å and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2 times U_{eq}(C, N).

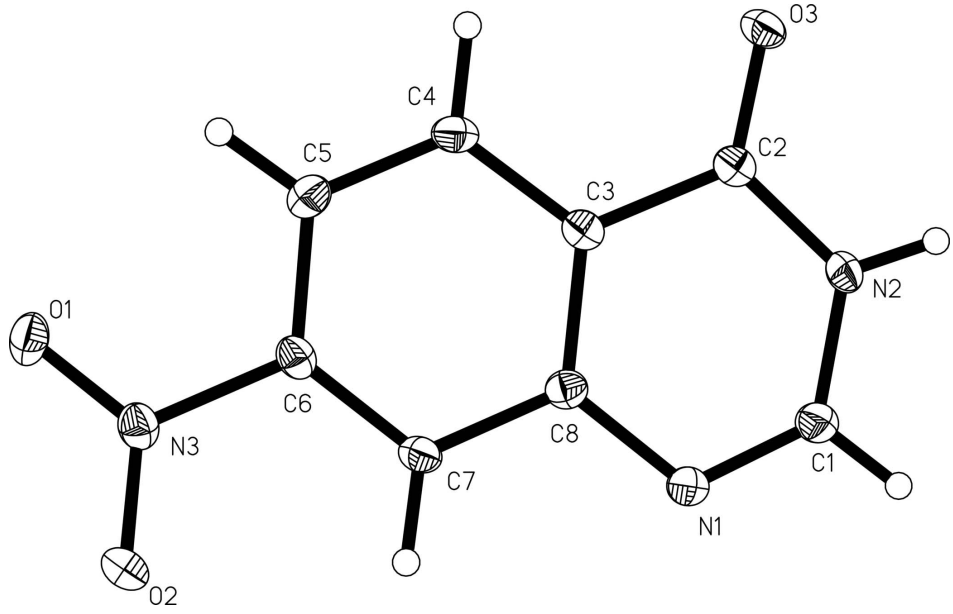

Figure 1

The molecular structure with displacement ellipsoids drawn at the 35% probability level.

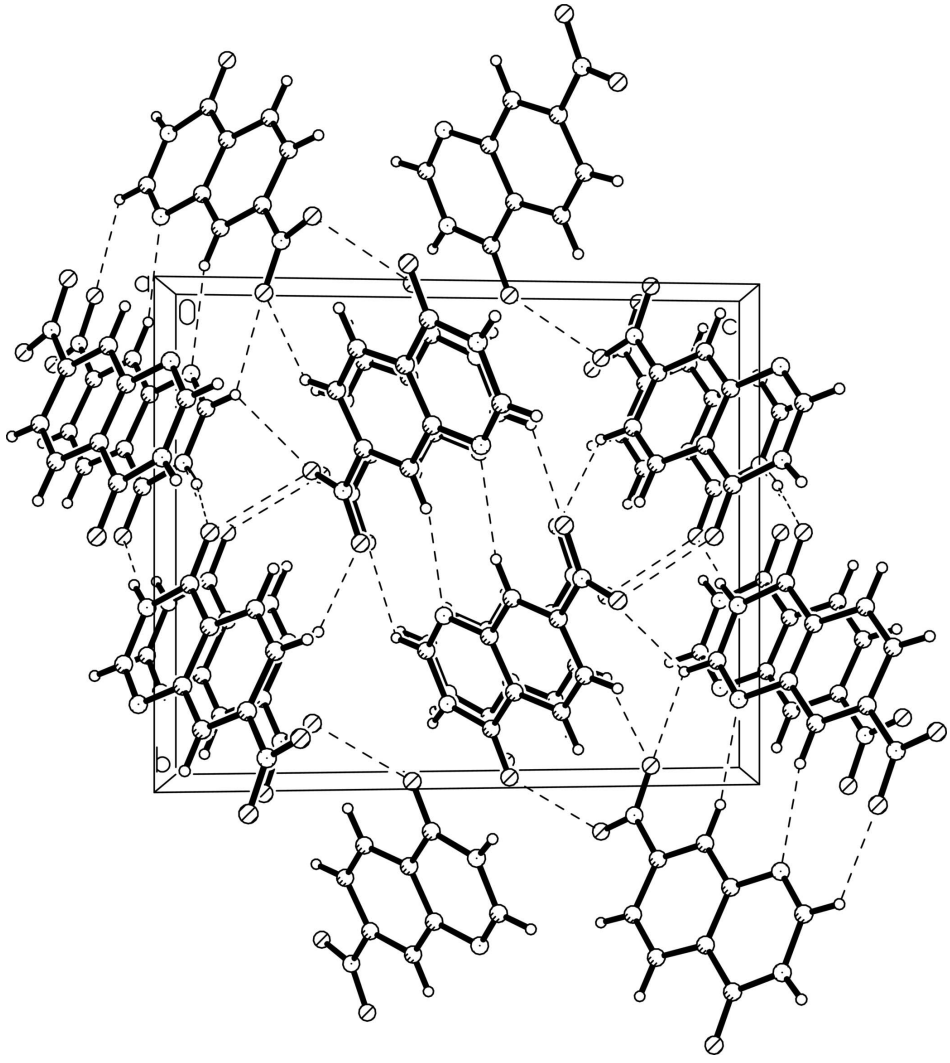

Figure 2

The packing of the title compound with hydrogen bonds shown as dashed lines.

7-NitroQuinazolin-4(3*H*)-one

Crystal data

$C_8H_5N_3O_3$
M_r = 191.15
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 5.1063 (10) Å
b = 11.206 (2) Å
c = 13.528 (3) Å
β = 99.19 (3)°
V = 764.1 (3) Å^3
Z = 4
$F(000)$ = 392
D_x = 1.662 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6207 reflections
θ = 6.0–55.0°
μ = 0.13 mm^{-1}
T = 153 K
Needle, colorless
0.24 × 0.18 × 0.16 mm

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer
Radiation source: Rotating Anode
Graphite monochromator
ω Oscillation scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.969, T_{max} = 0.979
5749 measured reflections
1340 independent reflections
1215 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 25.0°, θ_{min} = 3.1°
$h = -6 \rightarrow 6$
$k = -13 \rightarrow 13$
$l = -16 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.096
S = 1.11
1340 reflections
127 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.064P)^2 + 0.0923P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 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_{iso}*/U_{eq}
O1	−0.37255 (17)	0.37262 (8)	0.24708 (6)	0.0228 (3)
O2	−0.1637 (2)	0.51746 (9)	0.33145 (8)	0.0324 (3)
O3	0.69259 (17)	−0.00398 (7)	0.41669 (6)	0.0203 (3)
N1	0.6334 (2)	0.33230 (10)	0.53066 (8)	0.0214 (3)
N2	0.8443 (2)	0.14538 (9)	0.52489 (7)	0.0183 (3)
H2A	0.9807	0.1015	0.5512	0.022*
C1	0.8209 (2)	0.25752 (11)	0.56195 (9)	0.0206 (3)
H1B	0.9536	0.2827	0.6152	0.025*
C2	0.6655 (2)	0.09785 (11)	0.44863 (8)	0.0163 (3)
C3	0.4471 (2)	0.17821 (10)	0.40975 (8)	0.0160 (3)
C4	0.2491 (2)	0.14293 (11)	0.33110 (9)	0.0188 (3)
H4A	0.2557	0.0658	0.3025	0.023*
C5	0.0451 (2)	0.21960 (11)	0.29509 (9)	0.0194 (3)
H5A	−0.0904	0.1964	0.2421	0.023*
C6	0.0434 (2)	0.33253 (11)	0.33893 (9)	0.0171 (3)
C7	0.2345 (2)	0.37169 (11)	0.41533 (9)	0.0176 (3)

H7A	0.2274	0.4498	0.4421	0.021*
C8	0.4409 (2)	0.29261 (11)	0.45270 (8)	0.0166 (3)
N3	−0.17982 (19)	0.41378 (10)	0.30265 (7)	0.0194 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0179 (5)	0.0261 (6)	0.0224 (5)	−0.0023 (4)	−0.0028 (3)	0.0042 (3)
O2	0.0344 (6)	0.0166 (5)	0.0416 (6)	0.0077 (4)	−0.0086 (4)	−0.0043 (4)
O3	0.0219 (5)	0.0143 (5)	0.0248 (5)	0.0033 (4)	0.0043 (3)	−0.0018 (3)
N1	0.0228 (6)	0.0167 (6)	0.0227 (5)	0.0021 (4)	−0.0023 (4)	−0.0026 (4)
N2	0.0167 (5)	0.0155 (6)	0.0219 (5)	0.0034 (4)	0.0003 (4)	0.0017 (4)
C1	0.0225 (7)	0.0170 (7)	0.0208 (6)	0.0016 (5)	−0.0005 (5)	−0.0008 (4)
C2	0.0170 (6)	0.0146 (7)	0.0183 (6)	−0.0009 (5)	0.0062 (4)	0.0020 (4)
C3	0.0171 (6)	0.0146 (7)	0.0174 (6)	−0.0001 (5)	0.0056 (4)	0.0017 (4)
C4	0.0208 (7)	0.0145 (7)	0.0217 (6)	−0.0011 (5)	0.0049 (5)	−0.0034 (5)
C5	0.0192 (6)	0.0196 (7)	0.0185 (6)	−0.0032 (5)	0.0010 (4)	−0.0011 (5)
C6	0.0165 (6)	0.0156 (6)	0.0195 (6)	0.0014 (5)	0.0036 (4)	0.0033 (5)
C7	0.0201 (7)	0.0127 (6)	0.0199 (6)	0.0010 (5)	0.0033 (5)	−0.0006 (4)
C8	0.0182 (6)	0.0150 (6)	0.0168 (6)	−0.0016 (5)	0.0034 (4)	0.0009 (5)
N3	0.0192 (6)	0.0191 (6)	0.0197 (5)	0.0013 (4)	0.0020 (4)	0.0037 (4)

Geometric parameters (Å, º)

O1—N3	1.2289 (14)	C3—C4	1.4023 (17)
O2—N3	1.2240 (15)	C3—C8	1.4099 (17)
O3—C2	1.2358 (15)	C4—C5	1.3779 (18)
N1—C1	1.2916 (17)	C4—H4A	0.9500
N1—C8	1.3946 (16)	C5—C6	1.3982 (18)
N2—C1	1.3652 (16)	C5—H5A	0.9500
N2—C2	1.3713 (16)	C6—C7	1.3750 (17)
N2—H2A	0.8800	C6—N3	1.4799 (16)
C1—H1B	0.9500	C7—C8	1.4076 (18)
C2—C3	1.4644 (17)	C7—H7A	0.9500
C1—N1—C8	115.91 (11)	C4—C5—C6	118.03 (11)
C1—N2—C2	123.16 (10)	C4—C5—H5A	121.0
C1—N2—H2A	118.4	C6—C5—H5A	121.0
C2—N2—H2A	118.4	C7—C6—C5	123.78 (11)
N1—C1—N2	125.49 (11)	C7—C6—N3	118.00 (11)
N1—C1—H1B	117.3	C5—C6—N3	118.21 (11)
N2—C1—H1B	117.3	C6—C7—C8	118.02 (11)
O3—C2—N2	121.57 (11)	C6—C7—H7A	121.0
O3—C2—C3	124.30 (11)	C8—C7—H7A	121.0
N2—C2—C3	114.12 (11)	N1—C8—C7	117.89 (11)
C4—C3—C8	120.54 (11)	N1—C8—C3	122.83 (11)
C4—C3—C2	120.97 (11)	C7—C8—C3	119.28 (11)
C8—C3—C2	118.49 (11)	O2—N3—O1	123.89 (10)

C5—C4—C3	120.34 (11)	O2—N3—C6	117.99 (10)
C5—C4—H4A	119.8	O1—N3—C6	118.10 (10)
C3—C4—H4A	119.8		
C8—N1—C1—N2	0.28 (19)	N3—C6—C7—C8	177.39 (10)
C2—N2—C1—N1	−0.5 (2)	C1—N1—C8—C7	−179.22 (11)
C1—N2—C2—O3	179.92 (11)	C1—N1—C8—C3	0.03 (18)
C1—N2—C2—C3	0.33 (16)	C6—C7—C8—N1	−179.57 (10)
O3—C2—C3—C4	−0.08 (19)	C6—C7—C8—C3	1.15 (17)
N2—C2—C3—C4	179.50 (10)	C4—C3—C8—N1	−179.68 (11)
O3—C2—C3—C8	−179.62 (10)	C2—C3—C8—N1	−0.14 (17)
N2—C2—C3—C8	−0.04 (16)	C4—C3—C8—C7	−0.44 (18)
C8—C3—C4—C5	−0.31 (18)	C2—C3—C8—C7	179.10 (10)
C2—C3—C4—C5	−179.83 (11)	C7—C6—N3—O2	10.75 (16)
C3—C4—C5—C6	0.30 (18)	C5—C6—N3—O2	−170.57 (11)
C4—C5—C6—C7	0.47 (19)	C7—C6—N3—O1	−167.81 (10)
C4—C5—C6—N3	−178.12 (10)	C5—C6—N3—O1	10.87 (16)
C5—C6—C7—C8	−1.21 (18)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O3^{i}	0.88	1.98	2.8514 (14)	169
C1—H1*B*···O2^{ii}	0.95	2.54	3.2703 (17)	134
C1—H1*B*···O1^{iii}	0.95	2.55	3.0978 (17)	117
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C7—H7*A*···N1^{ii}	0.95	2.55	3.4402 (18)	155

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