IUCrData

ISSN 2414-3146

5,6,7,8-Tetrahydro-[1,2,4]triazolo[5,1-*b*]quinazol-in-9(4*H*)-one

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Received 3 May 2023
Accepted 9 May 2023

Edited by E. R. T. Tiekink, Sunway University, Malaysia

This article is part of a collection of articles to commemorate the founding of the African Crystallographic Association and the 75th anniversary of the IUCr.

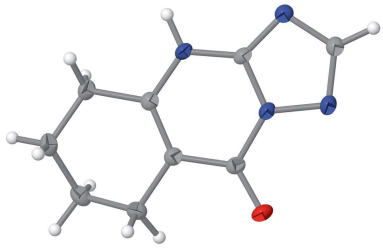
Keywords: crystal structure; π-stacking; hydrogen bonding; triazole; tetrahydroquinazoline.

CCDC reference: 2259950

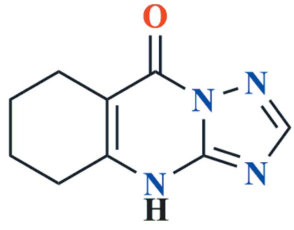
Structural data: full structural data are available from iucrdata.iucr.org

The triazole ring in the title molecule, $C_9H_{10}N_4O$, is not quite coplanar with the six-membered ring to which it is fused, the dihedral angle between the two least-squares planes being 2.52 (6)°. In the crystal, a layered structure is formed by N—H···N and C—H···O hydrogen bonds plus slipped π-stacking interactions, with the fused cyclohexene rings projecting to either side.

3D view



Chemical scheme



Structure description

Compounds containing nitrogen heterocycles make up a significant portion (approximately 60%) of small drug molecules that have been approved by the FDA (Ramli & Essassi, 2015; Martins *et al.*, 2015). Quinazoline is a frequently occurring structural feature in natural products and pharmaceutically active molecules., which possess a range of useful biological properties, including anti-SARS-CoV-2 (*e.g.* Karan *et al.*, 2021), anticancer (*e.g.* Zhao *et al.*, 2021), antiviral (*e.g.* El-Shershaby *et al.*, 2021), antimicrobial, anti-inflammatory (*e.g.* Zhang *et al.*, 2020), and antifungal activities (*e.g.* Ibrahim *et al.*, 2021).

A puckering analysis of the C2–C7 ring of the title compound (Fig. 1) gave the parameters Q = 0.4922 (12) Å, θ = 129.71 (14)° and φ = 326.36 (18)°. This conformation is quite similar to a half-chair form. The C8/N2/C9/N3/N4 ring is closer to planarity than is the C1/C2/C7/N1/C8/N4 ring (r.m.s. deviations of the fitted atoms are 0.0128 and 0.0042 Å, respectively) and the dihedral angle between their mean planes is 2.52 (6)°. In

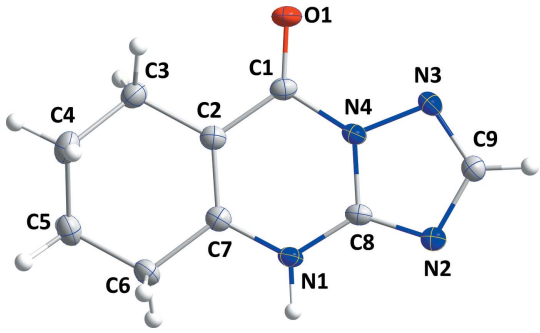


Figure 1
The title molecule with labeling scheme and 50% probability ellipsoids.

the crystal, N1—H1···N3 hydrogen bonds (Table 1) form chains of molecules extending along the *c*-axis direction, which are linked into layers parallel to the *bc* plane by weak C—H···O hydrogen bonds (Table 1 and Fig. 2). The layer

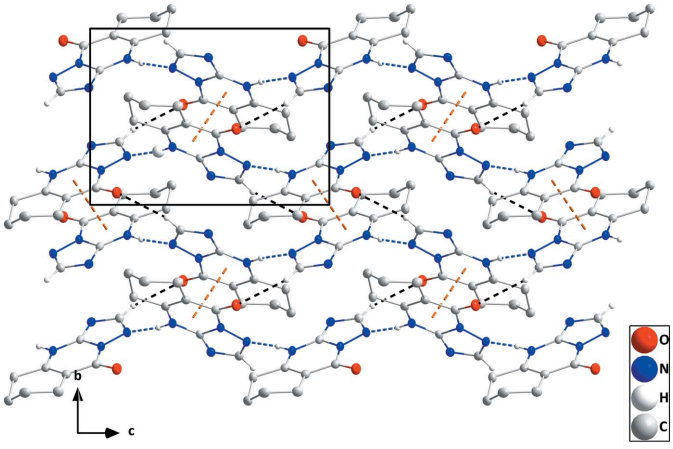


Figure 2
A portion of one layer viewed along the *a*-axis direction with N—H···N and C—H···O hydrogen bonds depicted by blue and black dashed lines, respectively. The slipped π-stacking interactions are depicted by orange dashed lines and non-interacting hydrogen atoms are omitted for clarity.

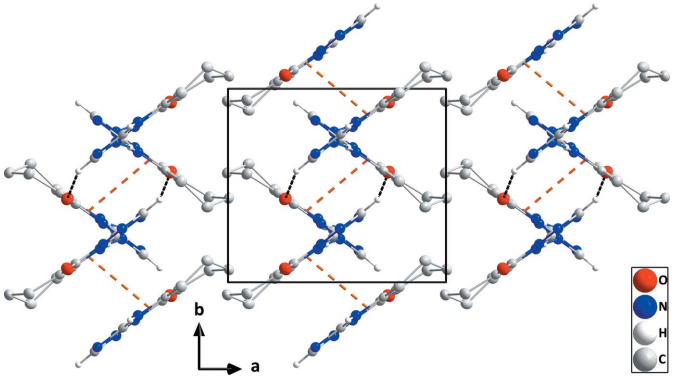


Figure 3
Packing viewed along the *c*-axis direction giving edge views of portions of three layers. Intermolecular interactions are depicted as in Fig. 2 and non-interacting hydrogen atoms are omitted for clarity.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···N3^{i}	0.919 (15)	1.907 (15)	2.8208 (12)	173.1 (13)
C9—H9···O1^{ii}	0.95	2.57	3.3282 (12)	137

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_9H_{10}N_4O$
M_r	190.21
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	150
a, *b*, *c* (Å)	9.7925 (3), 7.9648 (3), 11.8039 (4)
β (°)	113.553 (1)
V (Å^3)	843.95 (5)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.86
Crystal size (mm)	0.36 × 0.15 × 0.12
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 3 CPAD
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.84, 0.91
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	18079, 1657, 1626
R_{int}	0.021
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.618
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.030, 0.080, 1.07
No. of reflections	1657
No. of parameters	131
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.24, −0.19

Computer programs: *APEX4* and *SAINT* (Bruker, 2021), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2018/1* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

formation is assisted by slipped π-stacking interactions between inversion-related C1/C2/C7/N1/C8/N4 rings [centroid–centroid distance = 3.4033 (6) Å, slippage = 0.96 Å]. The layers pack along the *a*-axis direction with van der Waals contacts between them (Fig. 3).

Synthesis and crystallization

1*H*-1,2,4-Triazol-5-amine (0.5 g, 5.95 mmol) and ethyl 2-oxocyclohexanecarboxylate (0.951 ml, 5.95 mmol) were combined and heated under reflux in 10 ml of acetic acid for 1 h. The solid product obtained was recrystallized from ethanol solution to afford colorless crystals.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The support of NSF-MRI Grant #1228232 for the purchase of the diffractometer and Tulane University for support of the Tulane Crystallography Laboratory are gratefully acknowledged. Author contributions are as follows. Conceptualization, MT and AB; methodology, WE and AB; investigation, WE, AD; writing (original draft), JTM and YR; writing (review and editing of the manuscript), YR; formal analysis, YR; supervision, YR and MT; crystal-structure determination and validation, JTM.

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full crystallographic data

IUCrData (2023). **8**, x230409 [https://doi.org/10.1107/S2414314623004091]

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Crystal data

$C_9H_{10}N_4O$
M_r = 190.21
Monoclinic, $P2_1/c$
a = 9.7925 (3) Å
b = 7.9648 (3) Å
c = 11.8039 (4) Å
β = 113.553 (1)°
V = 843.95 (5) Å^3
Z = 4
$F(000)$ = 400
D_x = 1.497 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 9958 reflections
θ = 4.1–72.3°
μ = 0.86 mm^{-1}
T = 150 K
Column, colourless
0.36 × 0.15 × 0.12 mm

Data collection

Bruker D8 VENTURE PHOTON 3 CPAD diffractometer
Radiation source: INCOATEC IμS micro—-focus source
Mirror monochromator
Detector resolution: 7.3910 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min} = 0.84, T_{max} = 0.91
18079 measured reflections
1657 independent reflections
1626 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 72.4°, θ_{min} = 7.4°
h = −12→12
k = −9→9
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.080
S = 1.07
1657 reflections
131 parameters
0 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0413P)^2 + 0.2836P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Special details

Experimental. The diffraction data were obtained from 18 sets of frames, each of width 0.5° in ω or φ, collected with scan parameters determined by the "strategy" routine in *APEX4*. The scan time was θ-dependent and ranged from 4 to 15 sec/frame.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 - 0.98 Å) and included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms. That attached to nitrogen was placed in a location derived from a difference map and was refined independently.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.26314 (8)	0.43095 (10)	0.61185 (6)	0.0244 (2)
N1	0.41264 (9)	0.32811 (10)	0.34454 (7)	0.0175 (2)
H1	0.4489 (16)	0.3032 (18)	0.2857 (14)	0.034 (4)*
N2	0.59918 (9)	0.16425 (10)	0.50516 (7)	0.0183 (2)
N3	0.51360 (9)	0.22338 (11)	0.65438 (7)	0.0189 (2)
N4	0.42719 (9)	0.30291 (10)	0.54577 (7)	0.0168 (2)
C1	0.30105 (11)	0.40365 (12)	0.52669 (9)	0.0180 (2)
C2	0.22902 (11)	0.46080 (12)	0.40036 (9)	0.0180 (2)
C3	0.08681 (11)	0.55964 (13)	0.36710 (10)	0.0227 (2)
H3A	0.111393	0.675596	0.398974	0.027*
H3B	0.025843	0.507898	0.407433	0.027*
C4	−0.00393 (12)	0.56563 (14)	0.22750 (10)	0.0245 (3)
H4A	−0.050592	0.454782	0.198573	0.029*
H4B	−0.084433	0.649834	0.208425	0.029*
C5	0.09473 (12)	0.61123 (13)	0.15965 (9)	0.0236 (2)
H5A	0.142182	0.721576	0.189077	0.028*
H5B	0.032941	0.620358	0.069973	0.028*
C6	0.21483 (11)	0.47830 (13)	0.18183 (9)	0.0215 (2)
H6A	0.169975	0.379239	0.129506	0.026*
H6B	0.292459	0.523786	0.156502	0.026*
C7	0.28651 (11)	0.42377 (12)	0.31498 (9)	0.0172 (2)
C8	0.48158 (10)	0.26443 (12)	0.45921 (8)	0.0160 (2)
C9	0.61124 (11)	0.14311 (12)	0.62317 (9)	0.0185 (2)
H9	0.686309	0.073934	0.680293	0.022*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0287 (4)	0.0296 (4)	0.0208 (4)	0.0032 (3)	0.0161 (3)	−0.0006 (3)
N1	0.0201 (4)	0.0205 (4)	0.0139 (4)	0.0001 (3)	0.0089 (3)	−0.0007 (3)
N2	0.0187 (4)	0.0190 (4)	0.0178 (4)	−0.0010 (3)	0.0079 (3)	−0.0009 (3)
N3	0.0206 (4)	0.0212 (4)	0.0143 (4)	−0.0004 (3)	0.0063 (3)	0.0019 (3)
N4	0.0188 (4)	0.0193 (4)	0.0133 (4)	−0.0007 (3)	0.0076 (3)	0.0002 (3)
C1	0.0195 (5)	0.0171 (5)	0.0196 (5)	−0.0020 (4)	0.0102 (4)	−0.0016 (4)

C2	0.0190 (5)	0.0170 (5)	0.0186 (5)	−0.0019 (4)	0.0083 (4)	−0.0008 (4)
C3	0.0218 (5)	0.0230 (5)	0.0254 (5)	0.0030 (4)	0.0115 (4)	0.0009 (4)
C4	0.0194 (5)	0.0222 (5)	0.0283 (6)	0.0025 (4)	0.0057 (4)	−0.0001 (4)
C5	0.0258 (5)	0.0211 (5)	0.0189 (5)	0.0010 (4)	0.0037 (4)	0.0013 (4)
C6	0.0239 (5)	0.0240 (5)	0.0157 (5)	0.0000 (4)	0.0069 (4)	0.0004 (4)
C7	0.0178 (5)	0.0161 (5)	0.0175 (5)	−0.0030 (3)	0.0067 (4)	−0.0011 (4)
C8	0.0182 (4)	0.0164 (5)	0.0152 (4)	−0.0036 (3)	0.0087 (4)	−0.0025 (3)
C9	0.0182 (5)	0.0188 (5)	0.0176 (5)	−0.0015 (4)	0.0063 (4)	0.0006 (4)

Geometric parameters (Å, º)

O1—C1	1.2222 (12)	C3—C4	1.5287 (14)
N1—C8	1.3471 (12)	C3—H3A	0.9900
N1—C7	1.3724 (13)	C3—H3B	0.9900
N1—H1	0.919 (15)	C4—C5	1.5247 (15)
N2—C8	1.3258 (13)	C4—H4A	0.9900
N2—C9	1.3605 (12)	C4—H4B	0.9900
N3—C9	1.3192 (13)	C5—C6	1.5253 (14)
N3—N4	1.3762 (11)	C5—H5A	0.9900
N4—C8	1.3625 (12)	C5—H5B	0.9900
N4—C1	1.4135 (13)	C6—C7	1.5066 (13)
C1—C2	1.4449 (14)	C6—H6A	0.9900
C2—C7	1.3687 (14)	C6—H6B	0.9900
C2—C3	1.5087 (13)	C9—H9	0.9500
C8—N1—C7	120.21 (8)	C3—C4—H4B	109.5
C8—N1—H1	119.2 (9)	H4A—C4—H4B	108.1
C7—N1—H1	120.5 (9)	C4—C5—C6	110.58 (8)
C8—N2—C9	101.49 (8)	C4—C5—H5A	109.5
C9—N3—N4	101.96 (7)	C6—C5—H5A	109.5
C8—N4—N3	108.42 (8)	C4—C5—H5B	109.5
C8—N4—C1	125.93 (8)	C6—C5—H5B	109.5
N3—N4—C1	125.63 (8)	H5A—C5—H5B	108.1
O1—C1—N4	120.21 (9)	C7—C6—C5	112.64 (8)
O1—C1—C2	127.54 (9)	C7—C6—H6A	109.1
N4—C1—C2	112.23 (8)	C5—C6—H6A	109.1
C7—C2—C1	121.15 (9)	C7—C6—H6B	109.1
C7—C2—C3	121.96 (9)	C5—C6—H6B	109.1
C1—C2—C3	116.88 (9)	H6A—C6—H6B	107.8
C2—C3—C4	112.04 (8)	C2—C7—N1	121.71 (9)
C2—C3—H3A	109.2	C2—C7—C6	123.29 (9)
C4—C3—H3A	109.2	N1—C7—C6	114.96 (8)
C2—C3—H3B	109.2	N2—C8—N1	129.96 (9)
C4—C3—H3B	109.2	N2—C8—N4	111.37 (8)
H3A—C3—H3B	107.9	N1—C8—N4	118.66 (9)
C5—C4—C3	110.87 (8)	N3—C9—N2	116.74 (9)
C5—C4—H4A	109.5	N3—C9—H9	121.6
C3—C4—H4A	109.5	N2—C9—H9	121.6

C5—C4—H4B	109.5		
C9—N3—N4—C8	−0.54 (10)	C1—C2—C7—C6	179.19 (9)
C9—N3—N4—C1	177.90 (9)	C3—C2—C7—C6	0.39 (15)
C8—N4—C1—O1	−179.38 (9)	C8—N1—C7—C2	1.57 (14)
N3—N4—C1—O1	2.46 (15)	C8—N1—C7—C6	−176.34 (8)
C8—N4—C1—C2	1.78 (14)	C5—C6—C7—C2	13.45 (14)
N3—N4—C1—C2	−176.39 (8)	C5—C6—C7—N1	−168.67 (8)
O1—C1—C2—C7	178.33 (10)	C9—N2—C8—N1	−178.78 (10)
N4—C1—C2—C7	−2.93 (13)	C9—N2—C8—N4	0.72 (10)
O1—C1—C2—C3	−2.81 (15)	C7—N1—C8—N2	176.72 (9)
N4—C1—C2—C3	175.93 (8)	C7—N1—C8—N4	−2.74 (14)
C7—C2—C3—C4	16.83 (14)	N3—N4—C8—N2	−0.13 (11)
C1—C2—C3—C4	−162.02 (9)	C1—N4—C8—N2	−178.56 (8)
C2—C3—C4—C5	−47.48 (12)	N3—N4—C8—N1	179.43 (8)
C3—C4—C5—C6	62.20 (11)	C1—N4—C8—N1	1.00 (14)
C4—C5—C6—C7	−43.86 (11)	N4—N3—C9—N2	1.08 (11)
C1—C2—C7—N1	1.46 (15)	C8—N2—C9—N3	−1.16 (11)
C3—C2—C7—N1	−177.34 (9)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···N3^{i}	0.919 (15)	1.907 (15)	2.8208 (12)	173.1 (13)
C9—H9···O1^{ii}	0.95	2.57	3.3282 (12)	137

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