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6′-Methyl-1′,2′,3′,4′-tetrahydrospiro-cyclohexane-2′-quinazolin-4′-one

Zhang Ling, Daxin Shi, Fan Yanqiu, Xia Wei and Jiarong Li*

School of Chemical Engineering and Environment, Beijing Institute of Technology, Beijing 100081, People's Republic of China
Correspondence e-mail: jrli@bit.edu.cn

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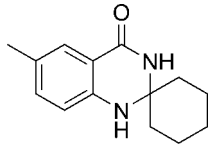
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.109; data-to-parameter ratio = 17.2.

The title compound, $C_{14}H_{18}N_2O$, was synthesized by the reaction of cyclohexanone and 2-amino-5-methylbenzonitrile. In the molecule, the cyclohexane ring displays a chair conformation, whereas the 1,3-diazacyclohexane moiety of the bicyclic system has a sofa conformation with the spiro C atom displaced by 0.603 (2) Å from the rest of the atoms of the 1,3-diazacyclohexane ring [planar within 0.052 (2) Å]. Molecules are linked into centrosymmetric dimers *via* N—H···O hydrogen bonds.

Related literature

For medicinal and biological properties of dihydroquinazolin-4(3*H*)-one derivatives, see: Jackson *et al.* (2007); Shi *et al.* (2003, 2004).



Experimental

Crystal data

$C_{14}H_{18}N_2O$
M_r = 230.30
Monoclinic, $P2_1/n$
a = 9.4077 (19) Å
b = 11.853 (2) Å
c = 11.067 (2) Å
β = 106.44 (3)°
V = 1183.6 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 113 K
0.28 × 0.24 × 0.20 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.977, T_{max} = 0.984
14356 measured reflections
2810 independent reflections
2346 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.109
S = 1.09
2810 reflections
163 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1^{i}	0.901 (16)	2.058 (16)	2.9563 (13)	174.5 (13)

Symmetry code: (i) $-x, -y+1, -z+1$.

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: YA2085).

References

Jackson, J. R., Patrick, D. R., Dar, M. M. & Huang, P. S. (2007). *Nat. Rev. Cancer*, **7**, 107–117.
Rigaku/MSC (2005). *CrystalClear*. Rigaku/MSC Inc., The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Shi, D. Q., Rong, L. C., Wang, J. X., Wang, X. S., Tu, S. J. & Hu, H. W. (2004). *Chem. J. Chin. Univ.* **25**, 2051–2053.
Shi, D. Q., Rong, L., Wang, J., Zhuang, Q., Wang, X. & Hu, H. (2003). *Tetrahedron Lett.* **44**, 3199–3201.

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6′-Methyl-1′,2′,3′,4′-tetrahydrospirocyclohexane-2′-quinazolin-4′-one

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S1. Comment

Derivatives of dihydroquinazolin-4(3*H*)-one are valuable synthetic intermediates featuring common structural motif found in a variety of compounds with interesting medicinal and biological properties (Shi *et al.*, 2004; Jackson *et al.*, 2007).

In the molecule of the title compound (Fig. 1) the cyclohexane ring displays a regular chair conformation, whereas, the 1,3-diazacyclohexane moiety of the bicyclic system has a sofa conformation with the C9 atom displaced by 0.603 (2) Å from the rest of the atoms of the 1,3-diazacyclohexane ring (planar within 0.052 (2) Å).

Molecules in crystal are linked into centrosymmetric dimers *via* N2—H2A···O1^{i} [symmetry code (i): -*x*, 1 - *y*, 1 - *z*] bond (Fig. 2).

The molecular geometry and overall crystal structure of the title compound are quite similar to those observed in the structure of its close analog which lacks the methyl substituent in position 6 of the tetrahydroquinazolinone system (Shi *et al.*, 2003).

S2. Experimental

A solution of 2-amino-5-methylbenzonitrile (10 mmol) and zinc chloride (10 mmol) in cyclohexanone (2 ml) was refluxed for 2 h. The reaction mixture was cooled to room temperature and poured into 20 ml of water (previously cooled to 20°); it was then filtered *in vacuo* to give the title compound. The product was recrystallizated from ethanol to give colorless crystalline powder. m.p. 527–528 K; IR (KBr): 3367 (N—H), 3028, 2936 (C—H), 1648 (C=O) cm^{-1}; ^{1H}-NMR(DMSO, p.p.m.): 1.25–1.78 (10*H*, m), 2.35(3*H*, s) 6.63 (1*H*, m), 6.87 (1*H*, d), 6.91 (1*H*, s), 7.55 (1*H*, d), 8.06(1*H*, s). 50 mg of the obtained product was dissolved in ethyl acetate (5 ml) and the solution was kept at room temperature for 4 days to give colorless single crystals.

S3. Refinement

The H atoms bonded to C were included in the riding model approximation with C—H distances 0.95–0.99 Å, and with U_{iso}=1.2U_{eq} or 1.5U_{eq} (for methyl H atoms). The H atoms bonded to N were located in the difference Fourier map and refined isotropically [N1—H1 0.89 (2); N2—H2A 0.90 (2)].

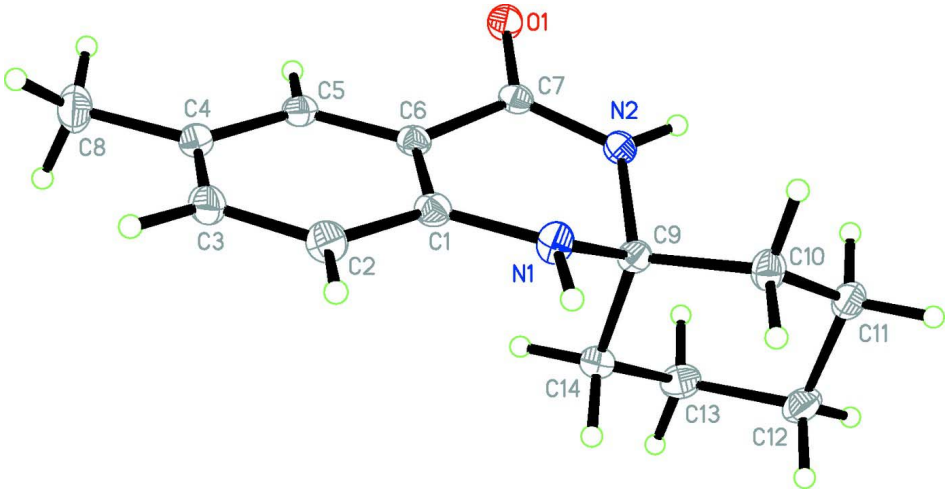

Figure 1

Molecular structure of the title compound with thermal displacement ellipsoids drawn at the 50% probability level; the H atoms are represented as small circles of arbitrary radius.

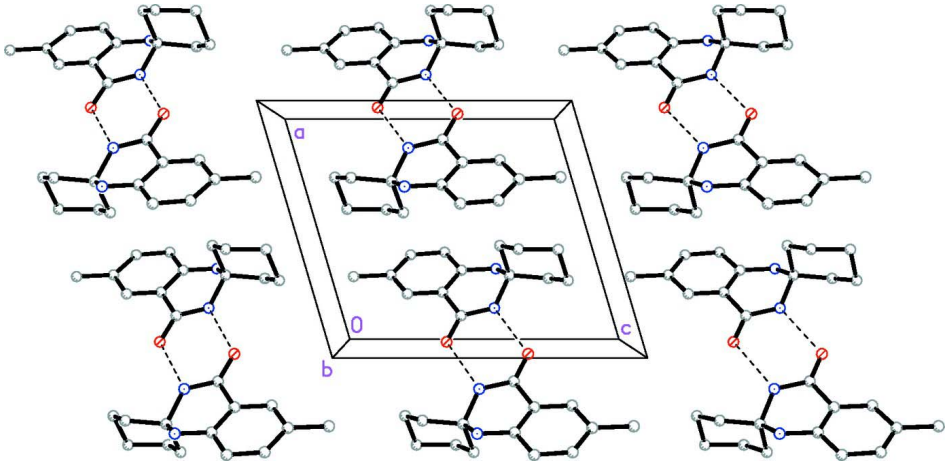

Figure 2

The crystal packing of the title compound, viewed down the *b* axis; H-bonds are shown as dashed lines.

6′-Methyl-1′,2′,3′,4′-tetrahydrospirocyclohexane-2′-quinazolin-4′-one

Crystal data

$C_{14}H_{18}N_2O$
$M_r = 230.30$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 9.4077\ (19)$ Å
$b = 11.853\ (2)$ Å
$c = 11.067\ (2)$ Å
$\beta = 106.44\ (3)°$
$V = 1183.6\ (4)$ Å^3
$Z = 4$

$F(000) = 496$
$D_x = 1.292$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3810 reflections
$\theta = 1.7–27.9°$
$\mu = 0.08$ mm^{-1}
$T = 113$ K
Cube, colorless
0.28 × 0.24 × 0.20 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Radiation source: rotating anode
Confocal monochromator
Detector resolution: 7.31 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.977, T_{max} = 0.984

14356 measured reflections
2810 independent reflections
2346 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 27.9°, θ_{min} = 2.5°
h = −12→12
k = −15→15
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.109
S = 1.09
2810 reflections
163 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0601P)^2 + 0.2329P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.01942 (8)	0.42294 (7)	0.36579 (7)	0.0182 (2)
N1	0.32706 (11)	0.24403 (8)	0.61566 (9)	0.0167 (2)
N2	0.16255 (10)	0.39739 (8)	0.56703 (8)	0.0144 (2)
C1	0.31997 (12)	0.22860 (9)	0.49040 (10)	0.0153 (2)
C2	0.39770 (12)	0.14331 (9)	0.44884 (11)	0.0191 (2)
H2	0.4588	0.0928	0.5078	0.023*
C3	0.38557 (12)	0.13253 (9)	0.32154 (11)	0.0197 (2)
H3	0.4379	0.0736	0.2946	0.024*
C4	0.29853 (12)	0.20585 (9)	0.23162 (10)	0.0180 (2)
C5	0.21867 (12)	0.28808 (9)	0.27333 (10)	0.0163 (2)
H5	0.1573	0.3381	0.2139	0.020*
C6	0.22636 (11)	0.29905 (9)	0.40055 (10)	0.0143 (2)
C7	0.12913 (11)	0.37917 (9)	0.44220 (10)	0.0141 (2)
C8	0.29082 (14)	0.19553 (11)	0.09415 (11)	0.0268 (3)
H8A	0.2017	0.2339	0.0431	0.040*

H8B	0.2869	0.1156	0.0707	0.040*
H8C	0.3789	0.2304	0.0793	0.040*
C9	0.30268 (11)	0.35816 (9)	0.65516 (9)	0.0142 (2)
C10	0.28781 (13)	0.35125 (9)	0.78910 (10)	0.0176 (2)
H10A	0.1961	0.3095	0.7872	0.021*
H10B	0.3726	0.3080	0.8423	0.021*
C11	0.28289 (12)	0.46671 (10)	0.84905 (10)	0.0192 (2)
H11A	0.2840	0.4566	0.9381	0.023*
H11B	0.1895	0.5055	0.8046	0.023*
C12	0.41467 (13)	0.53975 (10)	0.84304 (10)	0.0215 (3)
H12A	0.4071	0.6148	0.8799	0.026*
H12B	0.5081	0.5040	0.8927	0.026*
C13	0.41659 (13)	0.55324 (10)	0.70664 (10)	0.0204 (3)
H13A	0.5013	0.6013	0.7029	0.024*
H13B	0.3242	0.5906	0.6574	0.024*
C14	0.43013 (12)	0.43771 (9)	0.64986 (10)	0.0169 (2)
H14A	0.5257	0.4030	0.6963	0.020*
H14B	0.4303	0.4474	0.5610	0.020*
H2A	0.1067 (16)	0.4493 (13)	0.5919 (13)	0.027 (4)*
H1	0.3942 (18)	0.2055 (13)	0.6744 (14)	0.031 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0160 (4)	0.0208 (4)	0.0164 (4)	0.0053 (3)	0.0024 (3)	0.0005 (3)
N1	0.0198 (5)	0.0136 (5)	0.0159 (5)	0.0048 (4)	0.0040 (4)	0.0011 (3)
N2	0.0131 (4)	0.0143 (4)	0.0157 (4)	0.0030 (3)	0.0039 (3)	−0.0006 (3)
C1	0.0143 (5)	0.0135 (5)	0.0181 (5)	−0.0014 (4)	0.0048 (4)	−0.0013 (4)
C2	0.0187 (5)	0.0154 (5)	0.0226 (6)	0.0040 (4)	0.0045 (4)	−0.0011 (4)
C3	0.0175 (5)	0.0178 (5)	0.0247 (6)	0.0007 (4)	0.0073 (4)	−0.0065 (4)
C4	0.0153 (5)	0.0199 (6)	0.0192 (5)	−0.0034 (4)	0.0058 (4)	−0.0052 (4)
C5	0.0135 (5)	0.0171 (5)	0.0176 (5)	−0.0014 (4)	0.0034 (4)	−0.0018 (4)
C6	0.0124 (5)	0.0126 (5)	0.0178 (5)	−0.0014 (4)	0.0042 (4)	−0.0015 (4)
C7	0.0124 (5)	0.0128 (5)	0.0172 (5)	−0.0012 (4)	0.0045 (4)	0.0005 (4)
C8	0.0291 (6)	0.0327 (7)	0.0198 (6)	0.0046 (5)	0.0090 (5)	−0.0052 (5)
C9	0.0140 (5)	0.0135 (5)	0.0142 (5)	0.0020 (4)	0.0027 (4)	−0.0001 (4)
C10	0.0207 (5)	0.0178 (5)	0.0148 (5)	0.0019 (4)	0.0056 (4)	0.0020 (4)
C11	0.0196 (5)	0.0226 (6)	0.0144 (5)	0.0036 (4)	0.0031 (4)	−0.0017 (4)
C12	0.0196 (6)	0.0231 (6)	0.0186 (5)	0.0002 (5)	0.0004 (4)	−0.0059 (4)
C13	0.0196 (5)	0.0175 (6)	0.0224 (6)	−0.0032 (4)	0.0033 (4)	−0.0012 (4)
C14	0.0140 (5)	0.0183 (5)	0.0180 (5)	−0.0009 (4)	0.0042 (4)	−0.0003 (4)

Geometric parameters (Å, º)

O1—C7	1.2472 (13)	C8—H8B	0.9800
N1—C1	1.3810 (14)	C8—H8C	0.9800
N1—C9	1.4596 (14)	C9—C10	1.5299 (14)
N1—H1	0.893 (16)	C9—C14	1.5394 (15)

N2—C7	1.3446 (13)	C10—C11	1.5273 (16)
N2—C9	1.4757 (14)	C10—H10A	0.9900
N2—H2A	0.901 (16)	C10—H10B	0.9900
C1—C2	1.3995 (15)	C11—C12	1.5289 (16)
C1—C6	1.4022 (15)	C11—H11A	0.9900
C2—C3	1.3867 (15)	C11—H11B	0.9900
C2—H2	0.9500	C12—C13	1.5232 (16)
C3—C4	1.3982 (17)	C12—H12A	0.9900
C3—H3	0.9500	C12—H12B	0.9900
C4—C5	1.3870 (15)	C13—C14	1.5269 (15)
C4—C8	1.5074 (15)	C13—H13A	0.9900
C5—C6	1.3953 (14)	C13—H13B	0.9900
C5—H5	0.9500	C14—H14A	0.9900
C6—C7	1.4797 (14)	C14—H14B	0.9900
C8—H8A	0.9800		

C1—N1—C9	117.15 (9)	N2—C9—C10	110.35 (9)
C1—N1—H1	119.1 (10)	N1—C9—C14	111.48 (9)
C9—N1—H1	113.2 (10)	N2—C9—C14	109.97 (8)
C7—N2—C9	122.26 (9)	C10—C9—C14	110.82 (9)
C7—N2—H2A	115.9 (9)	C11—C10—C9	113.28 (9)
C9—N2—H2A	120.1 (9)	C11—C10—H10A	108.9
N1—C1—C2	122.97 (10)	C9—C10—H10A	108.9
N1—C1—C6	118.37 (10)	C11—C10—H10B	108.9
C2—C1—C6	118.61 (10)	C9—C10—H10B	108.9
C3—C2—C1	120.00 (10)	H10A—C10—H10B	107.7
C3—C2—H2	120.0	C10—C11—C12	111.34 (9)
C1—C2—H2	120.0	C10—C11—H11A	109.4
C2—C3—C4	121.94 (10)	C12—C11—H11A	109.4
C2—C3—H3	119.0	C10—C11—H11B	109.4
C4—C3—H3	119.0	C12—C11—H11B	109.4
C5—C4—C3	117.59 (10)	H11A—C11—H11B	108.0
C5—C4—C8	121.13 (10)	C13—C12—C11	109.86 (9)
C3—C4—C8	121.28 (10)	C13—C12—H12A	109.7
C4—C5—C6	121.54 (10)	C11—C12—H12A	109.7
C4—C5—H5	119.2	C13—C12—H12B	109.7
C6—C5—H5	119.2	C11—C12—H12B	109.7
C5—C6—C1	120.19 (10)	H12A—C12—H12B	108.2
C5—C6—C7	120.94 (9)	C12—C13—C14	109.84 (9)
C1—C6—C7	118.74 (9)	C12—C13—H13A	109.7
O1—C7—N2	122.54 (10)	C14—C13—H13A	109.7
O1—C7—C6	121.44 (9)	C12—C13—H13B	109.7
N2—C7—C6	115.95 (9)	C14—C13—H13B	109.7
C4—C8—H8A	109.5	H13A—C13—H13B	108.2
C4—C8—H8B	109.5	C13—C14—C9	112.16 (9)
H8A—C8—H8B	109.5	C13—C14—H14A	109.2
C4—C8—H8C	109.5	C9—C14—H14A	109.2
H8A—C8—H8C	109.5	C13—C14—H14B	109.2

H8B—C8—H8C	109.5	C9—C14—H14B	109.2
N1—C9—N2	106.31 (8)	H14A—C14—H14B	107.9
N1—C9—C10	107.80 (8)		

C9—N1—C1—C2	152.24 (10)	C5—C6—C7—N2	−168.95 (9)
C9—N1—C1—C6	−30.19 (14)	C1—C6—C7—N2	15.21 (14)
N1—C1—C2—C3	179.98 (10)	C1—N1—C9—N2	51.63 (12)
C6—C1—C2—C3	2.41 (16)	C1—N1—C9—C10	169.95 (9)
C1—C2—C3—C4	0.84 (17)	C1—N1—C9—C14	−68.21 (12)
C2—C3—C4—C5	−2.60 (16)	C7—N2—C9—N1	−42.79 (13)
C2—C3—C4—C8	177.80 (11)	C7—N2—C9—C10	−159.42 (9)
C3—C4—C5—C6	1.10 (16)	C7—N2—C9—C14	78.03 (12)
C8—C4—C5—C6	−179.30 (10)	N1—C9—C10—C11	172.15 (9)
C4—C5—C6—C1	2.13 (16)	N2—C9—C10—C11	−72.16 (11)
C4—C5—C6—C7	−173.64 (10)	C14—C9—C10—C11	49.90 (12)
N1—C1—C6—C5	178.45 (9)	C9—C10—C11—C12	−53.09 (12)
C2—C1—C6—C5	−3.87 (16)	C10—C11—C12—C13	57.52 (12)
N1—C1—C6—C7	−5.69 (15)	C11—C12—C13—C14	−60.01 (12)
C2—C1—C6—C7	171.99 (9)	C12—C13—C14—C9	58.30 (12)
C9—N2—C7—O1	−171.57 (10)	N1—C9—C14—C13	−172.67 (8)
C9—N2—C7—C6	11.38 (14)	N2—C9—C14—C13	69.67 (11)
C5—C6—C7—O1	13.96 (15)	C10—C9—C14—C13	−52.60 (11)
C1—C6—C7—O1	−161.87 (10)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O1^{i}	0.901 (16)	2.058 (16)	2.9563 (13)	174.5 (13)

Symmetry code: (i) −*x*, −*y*+1, −*z*+1.