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Crystal structure of 1-tosyl-1,2,3,4-tetrahydroquinoline

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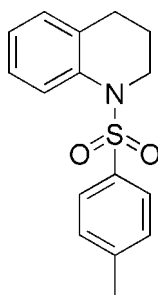
In the title compound, C₁₆H₁₇NO₂S, the heterocyclic ring adopts a half-chair conformation and the bond-angle sum at the N atom is 350.2°. The dihedral angle between the planes of the aromatic rings is 47.74 (10)°. In the crystal, molecules are linked by C—H...O hydrogen bonds to generate [010] chains.

Keywords: crystal structure; quinolines; C—H...O interactions; biotransformations; pharmacological activity.

CCDC reference: 1028050

1. Related literature

For reactions related to biotransformations, see: Leresche *et al.* (2006); Astudillo *et al.* (2009). For pharmacological activities, see: Bendale *et al.* (2007); Chen *et al.* (2007); Singer *et al.* (2005).



2. Experimental

2.1. Crystal data

C₁₆H₁₇NO₂S
 $M_r = 287.37$
 Monoclinic, $P2_1/n$
 $a = 8.2176$ (7) Å
 $b = 8.0468$ (6) Å
 $c = 22.2439$ (18) Å
 $\beta = 98.107$ (4)°
 $V = 1456.2$ (2) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.22$ mm⁻¹

$T = 94$ K
 $0.24 \times 0.22 \times 0.18$ mm

2.2. Data collection

Bruker APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2013)
 $T_{\min} = 0.949$, $T_{\max} = 0.961$
 20017 measured reflections
 2568 independent reflections
 2327 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.046$

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.101$
 $S = 1.09$
 2568 reflections
 182 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.23$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.37$ e Å⁻³

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

D—H...A	D—H	H...A	D...A	D—H...A
C14—H14...O2 ⁱ	0.95	2.53	3.340 (2)	143

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

Data collection: APEX2 (Bruker, 2013); cell refinement: SAINT (Bruker, 2013); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 2012) and Mercury (Macrae *et al.*, 2008); software used to prepare material for publication: SHELXL97.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7292).

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supporting information

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Crystal structure of 1-tosyl-1,2,3,4-tetrahydroquinoline

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S1. Chemical context

Chemical reactions by biotransformations have a number of advantages because they play an important role in the production of chiral products from racemic mixtures (Leresche *et al.*, 2006) in particular the tetrahydroquinoline derivatives can be transformed to other by *Mortierella isabelina* (Astudillo *et al.*, 2009). The tetrahydroquinoline compounds are core structures in pharmacological activities such as antimalarial activities (Bendale *et al.*, 2007), anti-psychotic (Singer *et al.*, 2005), estrogenic receptors (Chen *et al.*, 2007). In the course of our study, we noticed that 1,2,3,4-tetrahydroquinoline derivatives exhibit a few pharmacological activities (our unpublished data). As a part of our study we have undertaken crystal structure determination of the title compound and the results are presented here.

S2. Structural commentary

The molecular structure of the title compound is shown in Fig. 1. In the title molecule, the planes of the C1–C6 and C10–C15 benzene rings form a dihedral angle of 47.74 (9)°. The C1/C6–C9/N1 ring is in a half-chair conformation, with the methylene C9 atom as the flap. The molecular structure is stabilized by intramolecular C9—H9A···O1 and C2—H2···O2 hydrogen bonds (Fig. 2).

S3. Supramolecular features

In the crystal structure, intermolecular C14—H14···O2 hydrogen bonds link molecules into *C*(6) chains along [010] (Fig. 2 and Table 1)

S4. Synthesis and crystallization

To a stirred solution of 1,2,3,4-tetrahydroquinoline (10 mmol) in 30 mL dry dichloroethane, triethylamine (15 mmol) was added at 0 – 5°C. To this reaction mixture 4-methylbenzene-1-sulfonylchloride (12 mmol) was added drop wise. After 2h of stirring at room temperature, the reaction mixture was washed with 5% Na₂CO₃ and brine. Organic phase was dried over Na₂SO₄ and then it was concentrated on vacuum to yield titled compound as colourless solid. The crude product was recrystallized in the mixture of ethyl acetate and hexane(1:1) to get colourless prisms.

S5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. The H atoms were positioned with idealized geometry using a riding model with C—H = 0.95–0.99 Å. All H-atoms were refined with isotropic displacement parameters (set to 1.2–1.5 times of the U eq of the parent atom).

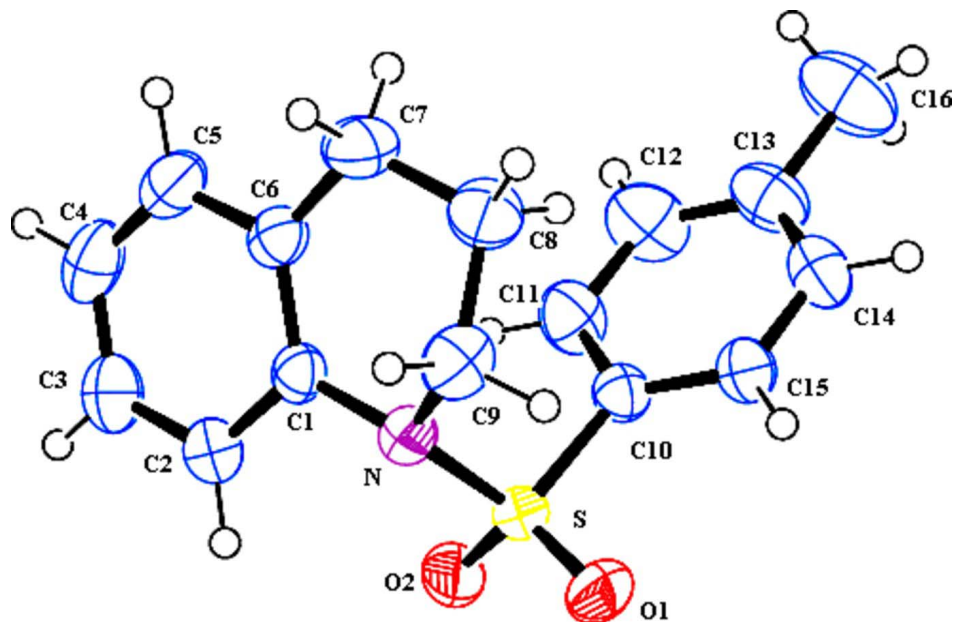


Figure 1

The molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level.

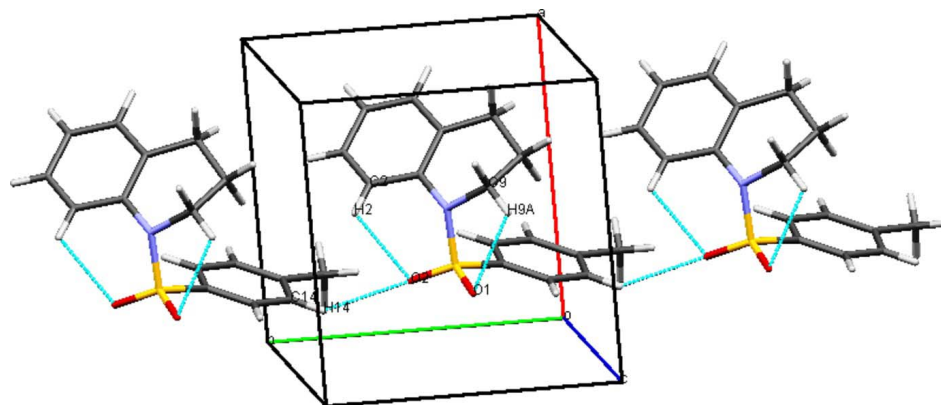


Figure 2

The molecular packing of the title compound, dashed lines indicate intramolecular C—H...O and intermolecular C—H...O hydrogen bonds forming C(6) chains viewed along [010].

1-Tosyl-1,2,3,4-tetrahydroquinoline

Crystal data

$C_{16}H_{17}NO_2S$

$M_r = 287.37$

Monoclinic, $P2_1/n$

Hall symbol: $-P 2_1n$

$a = 8.2176 (7) \text{ \AA}$

$b = 8.0468 (6) \text{ \AA}$

$c = 22.2439 (18) \text{ \AA}$

$\beta = 98.107 (4)^\circ$

$V = 1456.2 (2) \text{ \AA}^3$

$Z = 4$

$F(000) = 608$

Prism

$D_x = 1.311 \text{ Mg m}^{-3}$

Melting point: 402 K

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2327 reflections

$\theta = 1.9\text{--}25.0^\circ$

$\mu = 0.22 \text{ mm}^{-1}$

$T = 94 \text{ K}$

Prism, colourless

$0.24 \times 0.22 \times 0.18 \text{ mm}$

Data collection

Bruker APEXII CCD
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 1.9 pixels mm⁻¹
phi and ω scans
Absorption correction: multi-scan
(SADABS; Bruker, 2013)
 $T_{\min} = 0.949$, $T_{\max} = 0.961$

20017 measured reflections
2568 independent reflections
2327 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.046$
 $\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 1.9^\circ$
 $h = -9 \rightarrow 9$
 $k = -9 \rightarrow 9$
 $l = -26 \rightarrow 26$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.101$
 $S = 1.09$
2568 reflections
182 parameters
0 restraints
0 constraints
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
 $w = 1/[\sigma^2(F_o^2) + (0.0362P)^2 + 0.8395P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.23 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.36 \text{ e } \text{Å}^{-3}$

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	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.5482 (2)	0.4736 (2)	0.17137 (8)	0.0359 (4)
C2	0.5261 (3)	0.6396 (2)	0.15480 (9)	0.0461 (5)
H2	0.4386	0.7015	0.1675	0.055*
C3	0.6312 (3)	0.7140 (3)	0.12003 (10)	0.0565 (6)
H3	0.6146	0.8265	0.1078	0.068*
C4	0.7605 (3)	0.6253 (3)	0.10294 (10)	0.0593 (6)
H4	0.8319	0.6758	0.0782	0.071*
C5	0.7859 (2)	0.4634 (3)	0.12175 (9)	0.0509 (5)
H5	0.8772	0.4043	0.1106	0.061*
C6	0.6818 (2)	0.3838 (2)	0.15666 (8)	0.0400 (4)
C7	0.7232 (3)	0.2110 (3)	0.18038 (11)	0.0553 (6)
H7A	0.7451	0.1398	0.1461	0.066*
H7B	0.8255	0.2161	0.2098	0.066*
C8	0.5908 (3)	0.1313 (3)	0.21082 (11)	0.0553 (6)
H8A	0.6394	0.0426	0.2386	0.066*

H8B	0.5079	0.0799	0.1798	0.066*
C9	0.5085 (3)	0.2589 (3)	0.24637 (9)	0.0505 (5)
H9A	0.4200	0.2043	0.2651	0.061*
H9B	0.5900	0.3033	0.2795	0.061*
C10	0.2228 (2)	0.2132 (2)	0.12881 (8)	0.0368 (4)
C11	0.2794 (3)	0.2300 (3)	0.07339 (9)	0.0536 (5)
H11	0.3210	0.3333	0.0616	0.064*
C12	0.2741 (4)	0.0936 (3)	0.03583 (10)	0.0661 (7)
H12	0.3131	0.1040	-0.0022	0.079*
C13	0.2137 (3)	-0.0582 (3)	0.05179 (10)	0.0577 (6)
C14	0.1579 (3)	-0.0714 (3)	0.10703 (10)	0.0524 (5)
H14	0.1156	-0.1745	0.1187	0.063*
C15	0.1626 (2)	0.0630 (2)	0.14573 (9)	0.0427 (4)
H15	0.1244	0.0520	0.1839	0.051*
C16	0.2090 (4)	-0.2057 (4)	0.00972 (13)	0.0920 (10)
H16A	0.2151	-0.3085	0.0336	0.138*
H16B	0.3025	-0.2004	-0.0131	0.138*
H16C	0.1062	-0.2042	-0.0186	0.138*
O1	0.15775 (18)	0.33842 (19)	0.22946 (7)	0.0548 (4)
O2	0.19664 (17)	0.52807 (17)	0.14578 (7)	0.0532 (4)
N	0.43800 (18)	0.39748 (19)	0.20781 (7)	0.0381 (4)
S	0.24138 (5)	0.38103 (6)	0.17987 (2)	0.03916 (16)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0337 (9)	0.0389 (10)	0.0349 (9)	-0.0051 (8)	0.0038 (7)	-0.0037 (8)
C2	0.0459 (11)	0.0401 (11)	0.0527 (11)	-0.0041 (9)	0.0082 (9)	-0.0020 (9)
C3	0.0605 (14)	0.0486 (12)	0.0607 (13)	-0.0124 (11)	0.0102 (11)	0.0080 (10)
C4	0.0523 (13)	0.0752 (16)	0.0527 (12)	-0.0209 (12)	0.0152 (10)	0.0045 (12)
C5	0.0367 (11)	0.0699 (15)	0.0470 (11)	-0.0057 (10)	0.0092 (9)	-0.0098 (11)
C6	0.0341 (10)	0.0479 (11)	0.0373 (9)	-0.0024 (8)	0.0025 (8)	-0.0061 (8)
C7	0.0451 (12)	0.0545 (13)	0.0660 (14)	0.0119 (10)	0.0070 (10)	-0.0018 (11)
C8	0.0496 (12)	0.0458 (12)	0.0689 (14)	0.0112 (10)	0.0029 (10)	0.0139 (10)
C9	0.0495 (12)	0.0565 (13)	0.0457 (11)	0.0021 (10)	0.0074 (9)	0.0137 (10)
C10	0.0365 (10)	0.0350 (10)	0.0381 (9)	0.0010 (8)	0.0028 (7)	0.0014 (8)
C11	0.0704 (15)	0.0471 (12)	0.0444 (11)	-0.0039 (11)	0.0118 (10)	0.0067 (9)
C12	0.0952 (19)	0.0675 (16)	0.0370 (11)	0.0029 (14)	0.0138 (12)	-0.0015 (11)
C13	0.0697 (15)	0.0497 (13)	0.0498 (12)	0.0089 (11)	-0.0056 (11)	-0.0092 (10)
C14	0.0595 (13)	0.0374 (11)	0.0578 (13)	-0.0024 (10)	-0.0002 (10)	0.0005 (9)
C15	0.0454 (11)	0.0390 (10)	0.0439 (10)	-0.0020 (8)	0.0066 (9)	0.0028 (8)
C16	0.127 (3)	0.0716 (19)	0.0739 (18)	0.0083 (18)	0.0008 (18)	-0.0298 (15)
O1	0.0478 (8)	0.0610 (9)	0.0612 (9)	-0.0083 (7)	0.0268 (7)	-0.0128 (7)
O2	0.0399 (8)	0.0352 (7)	0.0831 (11)	0.0066 (6)	0.0040 (7)	0.0050 (7)
N	0.0361 (8)	0.0379 (8)	0.0410 (8)	-0.0006 (7)	0.0084 (7)	0.0007 (7)
S	0.0332 (3)	0.0345 (3)	0.0516 (3)	0.00026 (18)	0.0119 (2)	-0.0035 (2)

Geometric parameters (Å, °)

C1—C2	1.390 (3)	C10—C15	1.378 (3)
C1—C6	1.392 (3)	C10—C11	1.384 (3)
C1—N	1.435 (2)	C10—S	1.7577 (19)
C2—C3	1.375 (3)	C11—C12	1.377 (3)
C2—H2	0.9500	C11—H11	0.9500
C3—C4	1.377 (3)	C12—C13	1.384 (3)
C3—H3	0.9500	C12—H12	0.9500
C4—C5	1.375 (3)	C13—C14	1.374 (3)
C4—H4	0.9500	C13—C16	1.508 (3)
C5—C6	1.390 (3)	C14—C15	1.379 (3)
C5—H5	0.9500	C14—H14	0.9500
C6—C7	1.509 (3)	C15—H15	0.9500
C7—C8	1.504 (3)	C16—H16A	0.9800
C7—H7A	0.9900	C16—H16B	0.9800
C7—H7B	0.9900	C16—H16C	0.9800
C8—C9	1.512 (3)	O1—S	1.4213 (14)
C8—H8A	0.9900	O2—S	1.4254 (14)
C8—H8B	0.9900	N—S	1.6526 (16)
C9—N	1.475 (2)	S—O1	1.4213 (14)
C9—H9A	0.9900	S—O2	1.4254 (14)
C9—H9B	0.9900		
C2—C1—C6	120.95 (17)	H9A—C9—H9B	107.9
C2—C1—N	119.39 (17)	C15—C10—C11	120.52 (18)
C6—C1—N	119.52 (17)	C15—C10—S	119.94 (14)
C3—C2—C1	119.9 (2)	C11—C10—S	119.40 (15)
C3—C2—H2	120.1	C12—C11—C10	118.5 (2)
C1—C2—H2	120.1	C12—C11—H11	120.8
C2—C3—C4	120.0 (2)	C10—C11—H11	120.8
C2—C3—H3	120.0	C11—C12—C13	122.0 (2)
C4—C3—H3	120.0	C11—C12—H12	119.0
C5—C4—C3	119.8 (2)	C13—C12—H12	119.0
C5—C4—H4	120.1	C14—C13—C12	118.3 (2)
C3—C4—H4	120.1	C14—C13—C16	120.8 (2)
C4—C5—C6	121.8 (2)	C12—C13—C16	120.9 (2)
C4—C5—H5	119.1	C13—C14—C15	121.0 (2)
C6—C5—H5	119.1	C13—C14—H14	119.5
C5—C6—C1	117.40 (19)	C15—C14—H14	119.5
C5—C6—C7	119.59 (18)	C10—C15—C14	119.75 (19)
C1—C6—C7	122.88 (17)	C10—C15—H15	120.1
C8—C7—C6	114.12 (17)	C14—C15—H15	120.1
C8—C7—H7A	108.7	C13—C16—H16A	109.5
C6—C7—H7A	108.7	C13—C16—H16B	109.5
C8—C7—H7B	108.7	H16A—C16—H16B	109.5
C6—C7—H7B	108.7	C13—C16—H16C	109.5
H7A—C7—H7B	107.6	H16A—C16—H16C	109.5

C7—C8—C9	110.59 (19)	H16B—C16—H16C	109.5
C7—C8—H8A	109.5	C1—N—C9	115.09 (15)
C9—C8—H8A	109.5	C1—N—S	118.90 (12)
C7—C8—H8B	109.5	C9—N—S	116.17 (13)
C9—C8—H8B	109.5	O1—S—O2	119.76 (9)
H8A—C8—H8B	108.1	O1—S—N	106.36 (9)
N—C9—C8	112.16 (16)	O2—S—N	107.38 (8)
N—C9—H9A	109.2	O1—S—C10	107.98 (9)
C8—C9—H9A	109.2	O2—S—C10	107.57 (9)
N—C9—H9B	109.2	N—S—C10	107.19 (8)
C8—C9—H9B	109.2		

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
C14—H14...O2 ⁱ	0.95	2.53	3.340 (2)	143

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