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

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Edited by W. T. A. Harrison, University of Aberdeen, Scotland

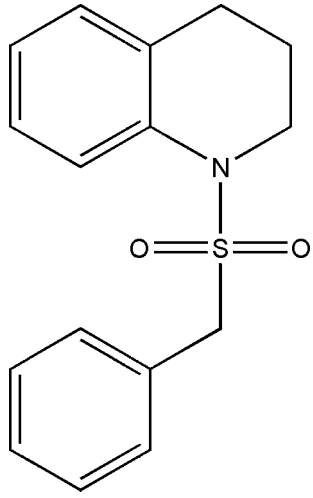
In the title compound, $C_{16}H_{17}NO_2S$, the heterocyclic ring adopts a half-chair conformation and the bond-angle sum at the N atom is 354.6°. The dihedral angle between the planes of the aromatic rings is 74.15 (10)°. In the crystal, molecules are linked by weak C—H···O hydrogen bonds, generating $C(8)$ and $C(4)$ chains propagating along [100] and [010], respectively, which together generate (001) sheets.

Keywords: crystal structure; 1,2,3,4-tetrahydroquinoline; weak C—H···O interactions.

CCDC reference: 1052632

1. Related literature

For the biological properties of 1,2,3,4-tetrahydroquinoline derivatives, see: Bendale *et al.* (2007); Singer *et al.* (2005). For related structures, see: Jeyaseelan *et al.* (2014, 2015).



2. Experimental

2.1. Crystal data

$C_{16}H_{17}NO_2S$
M_r = 287.36
Monoclinic, $P2_1/n$
a = 13.5690 (5) Å
b = 6.7128 (2) Å
c = 16.8317 (6) Å
β = 110.243 (1)°
V = 1438.44 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 295 K
0.24 × 0.20 × 0.18 mm

2.2. Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
T_{min} = 0.947, T_{max} = 0.960
19601 measured reflections
2529 independent reflections
2264 reflections with $I > 2\sigma(I)$
R_{int} = 0.051

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.101
S = 1.08
2529 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C14—H14···O1^{i}	0.93	2.69	3.573 (2)	158
C10—H10A···O2^{ii}	0.97	2.68	3.575 (2)	153

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

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: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL2014*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7377).

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supporting information

Acta Cryst. (2015). E**71**, o249–o250 [doi:10.1107/S2056989015004727]

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

S. Jeyaseelan, B. R. Sowmya, G. Venkateshappa, P. Raghavendra Kumar and B. S. Palakshamurthy

S1. Introduction

Heterocyclic compounds of 1,2,3,4-tetrahydroquinoline derivatives play important role in synthesize antimalarial (Bendale *et al.*, 2007), antipsychotic (Singer *et al.*, 2005) drugs. Keeping this in mind we have synthised a series of 1,2,3,4-tetrahydroquinoline with derivatives of Sulfonyl chlorides they exhibit a few pharmacological activities (our unpublished data). As a part of our study we have undertaken crystal structure determination of the title compound(I) and the results are compared with crystal structure of 1- tosyl-1,2,3,4-tetrahydroquinoline(II) and 1-methanesulfonyl-1,2,3,4-tetrahydroquinoline(III) (Jeyaseelan *et al.*, 2014a & 2014b).

S2. Structural commentary

The molecular structure of the title compound (I) is shown in Fig. 1. In all the compounds (I),(II) and (III), the C1/C6–C9/N1 rings are in a half-chair conformation, but the bond-angle sum at the N atom in the compound (I), (II) and (III) are 354.61°, 347.9° and 350.2°, respectively.

The crystal structure of (I) features C14–H14···O1 weak hydrogen bonds generating C(8) chain along [100] and C10–H10A···O2 weak hydrogen bonds generating C(4) along [010]: together these generate (001) sheets.

S3. Experimental

To a stirred solution of 1,2,3,4-tetrahydroquinoline (10 mmol) in 30 ml dry methylene dichloride, triethylamine (15 mmol) was added at 0–5°C. To this reaction mixture phenylmethanesulfonyl chloride (12 mmol) in 10 ml dry dichloro-methane was added drop wise. After 2h of stirring at 15–20°C, the reaction mixture was washed with 5% Na_2CO_3 and brine. The organic phase was dried over Na_2SO_4 and then it was concentrated on vacuum to yield titled compound as colourless solid. The crude product was recrystallized from a solvent mixture of ethyl acetate and hexane (1:2) to yield colourless prisms of (I).

S3.1. 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 = 0.93-0.97 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).

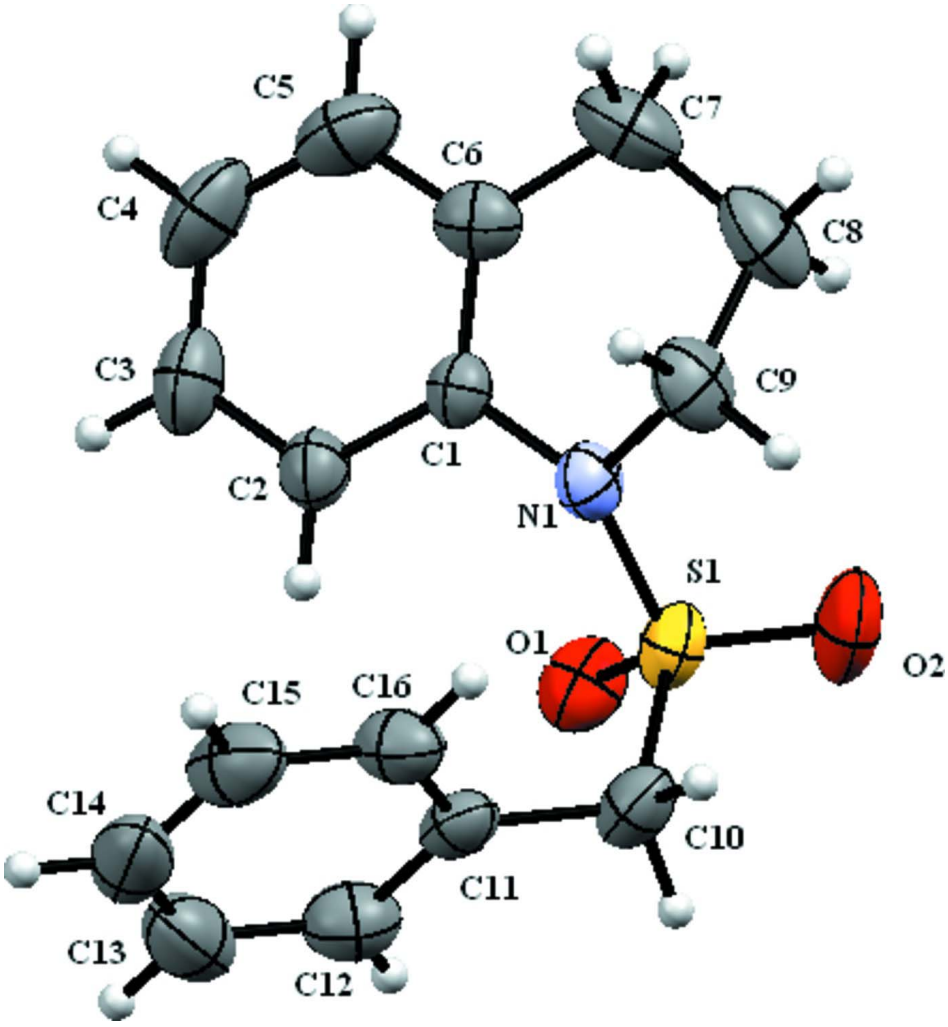

Figure 1

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

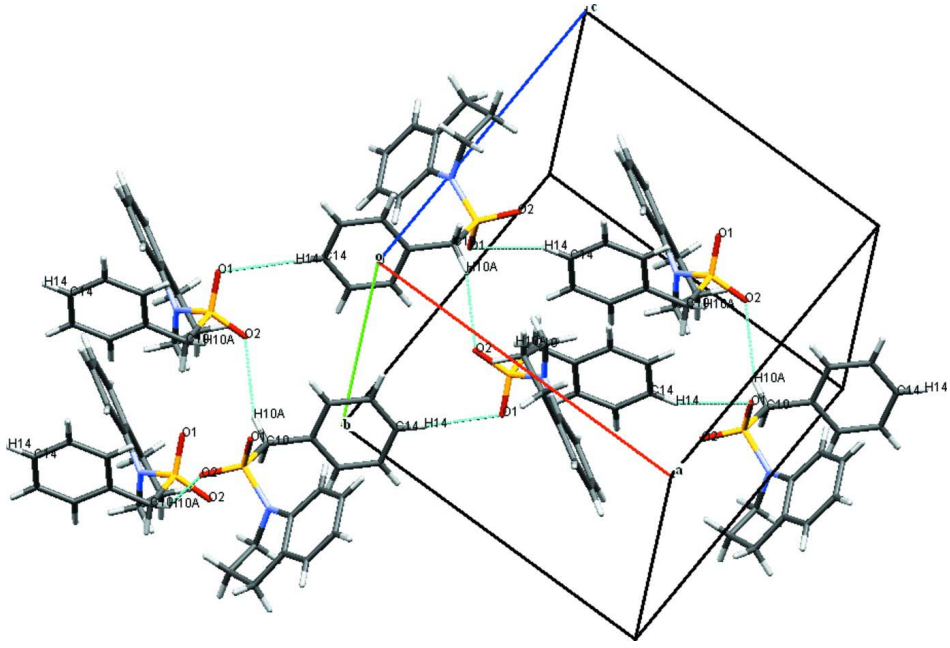

Figure 2

The molecular packing of the title compound, dashed lines indicates the C—H···O weak hydrogen bonds in the *ab* plane.

1-Benzylsulfonyl-1,2,3,4-tetrahydroquinoline

Crystal data

$C_{16}H_{17}NO_2S$
$M_r = 287.36$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 13.5690\ (5)$ Å
$b = 6.7128\ (2)$ Å
$c = 16.8317\ (6)$ Å
$\beta = 110.243\ (1)°$
$V = 1438.44\ (9)$ Å^3
$Z = 4$
$F(000) = 608$

Prism
$D_x = 1.327$ Mg m^{-3}
Melting point: 514 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2529 reflections
$\theta = 1.7–25°$
$\mu = 0.23$ mm^{-1}
$T = 295$ K
Prism, colourless
0.24 × 0.20 × 0.18 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 1.90 pixels mm^{-1}
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
$T_{min} = 0.947$, $T_{max} = 0.960$

19601 measured reflections
2529 independent reflections
2264 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.7°$
$h = -16\rightarrow16$
$k = -7\rightarrow7$
$l = -19\rightarrow20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.101$
$S = 1.08$
2529 reflections
181 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
$w = 1/[\sigma^2(F_o^2) + (0.0563P)^2 + 0.3073P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.36706 (3)	0.52581 (6)	0.20696 (2)	0.04077 (16)
N1	0.47088 (9)	0.40403 (18)	0.20485 (8)	0.0377 (3)
O1	0.37882 (9)	0.73063 (17)	0.18920 (8)	0.0515 (3)
C11	0.47391 (11)	0.5739 (2)	0.37732 (10)	0.0395 (4)
C1	0.60202 (12)	0.6674 (2)	0.22242 (10)	0.0419 (4)
H1	0.5827	0.7225	0.2656	0.050*
O2	0.27373 (9)	0.4236 (2)	0.15618 (8)	0.0634 (4)
C6	0.55139 (11)	0.4980 (2)	0.18039 (9)	0.0334 (3)
C12	0.55291 (13)	0.4389 (3)	0.41491 (11)	0.0488 (4)
H12	0.5430	0.3051	0.3999	0.059*
C5	0.58183 (13)	0.4084 (2)	0.11812 (10)	0.0448 (4)
C10	0.37241 (13)	0.5045 (3)	0.31416 (11)	0.0477 (4)
H10A	0.3155	0.5818	0.3211	0.057*
H10B	0.3615	0.3662	0.3257	0.057*
C9	0.46039 (15)	0.1865 (2)	0.19022 (11)	0.0516 (4)
H9A	0.4017	0.1368	0.2047	0.062*
H9B	0.5236	0.1200	0.2260	0.062*
C16	0.49091 (15)	0.7733 (3)	0.40037 (12)	0.0547 (4)
H16	0.4387	0.8669	0.3758	0.066*
C2	0.68073 (13)	0.7538 (3)	0.20020 (13)	0.0555 (5)
H2	0.7137	0.8689	0.2275	0.067*
C3	0.71051 (15)	0.6695 (3)	0.13749 (14)	0.0679 (6)
H3	0.7633	0.7280	0.1221	0.081*
C13	0.64626 (15)	0.4993 (3)	0.47439 (12)	0.0632 (5)
H13	0.6984	0.4061	0.4996	0.076*
C14	0.66280 (15)	0.6951 (4)	0.49666 (12)	0.0646 (6)
H14	0.7262	0.7354	0.5366	0.078*
C7	0.53441 (17)	0.2149 (3)	0.07532 (12)	0.0625 (5)

H7A	0.5113	0.2333	0.0145	0.075*
H7B	0.5885	0.1131	0.0903	0.075*
C4	0.66236 (16)	0.4995 (3)	0.09785 (14)	0.0634 (5)
H4	0.6840	0.4431	0.0562	0.076*
C8	0.44285 (18)	0.1420 (3)	0.09850 (13)	0.0667 (6)
H8A	0.4346	−0.0005	0.0888	0.080*
H8B	0.3789	0.2067	0.0629	0.080*
C15	0.58539 (18)	0.8325 (3)	0.45973 (13)	0.0654 (5)
H15	0.5966	0.9662	0.4747	0.079*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0284 (2)	0.0475 (3)	0.0419 (3)	−0.00260 (14)	0.00645 (17)	0.00823 (16)
N1	0.0394 (7)	0.0304 (6)	0.0437 (7)	−0.0068 (5)	0.0147 (5)	−0.0008 (5)
O1	0.0455 (7)	0.0438 (7)	0.0627 (7)	0.0085 (5)	0.0155 (6)	0.0154 (5)
C11	0.0358 (8)	0.0506 (9)	0.0373 (8)	0.0036 (7)	0.0193 (6)	0.0022 (7)
C1	0.0376 (8)	0.0386 (8)	0.0478 (9)	−0.0041 (6)	0.0127 (7)	−0.0021 (7)
O2	0.0352 (6)	0.0850 (9)	0.0578 (8)	−0.0170 (6)	0.0004 (5)	0.0074 (7)
C6	0.0308 (7)	0.0326 (7)	0.0349 (8)	−0.0002 (5)	0.0088 (6)	0.0035 (6)
C12	0.0503 (10)	0.0531 (10)	0.0431 (9)	0.0111 (8)	0.0165 (8)	−0.0011 (7)
C5	0.0502 (9)	0.0451 (9)	0.0392 (8)	0.0065 (7)	0.0157 (7)	0.0047 (7)
C10	0.0347 (8)	0.0621 (10)	0.0496 (10)	0.0006 (7)	0.0187 (7)	0.0069 (8)
C9	0.0659 (11)	0.0311 (8)	0.0573 (10)	−0.0103 (7)	0.0205 (9)	0.0010 (7)
C16	0.0605 (11)	0.0515 (10)	0.0575 (11)	0.0093 (8)	0.0272 (9)	0.0028 (8)
C2	0.0405 (9)	0.0500 (10)	0.0697 (12)	−0.0115 (7)	0.0108 (8)	0.0085 (9)
C3	0.0477 (10)	0.0809 (14)	0.0836 (14)	−0.0053 (10)	0.0336 (10)	0.0238 (12)
C13	0.0463 (10)	0.0953 (16)	0.0442 (10)	0.0197 (10)	0.0107 (8)	0.0000 (10)
C14	0.0509 (10)	0.1025 (17)	0.0424 (10)	−0.0162 (11)	0.0185 (8)	−0.0123 (10)
C7	0.0889 (14)	0.0505 (10)	0.0481 (10)	0.0043 (10)	0.0236 (10)	−0.0111 (8)
C4	0.0630 (12)	0.0794 (14)	0.0605 (12)	0.0072 (10)	0.0375 (10)	0.0079 (10)
C8	0.0895 (15)	0.0432 (10)	0.0611 (12)	−0.0192 (10)	0.0180 (10)	−0.0149 (9)
C15	0.0842 (14)	0.0607 (12)	0.0607 (12)	−0.0209 (11)	0.0368 (11)	−0.0156 (10)

Geometric parameters (Å, º)

S1—O1	1.4277 (12)	C9—H9A	0.9700
S1—O2	1.4341 (12)	C9—H9B	0.9700
S1—N1	1.6397 (13)	C16—C15	1.383 (3)
S1—C10	1.7863 (18)	C16—H16	0.9300
N1—C6	1.4397 (18)	C2—C3	1.376 (3)
N1—C9	1.4794 (19)	C2—H2	0.9300
C11—C12	1.379 (2)	C3—C4	1.368 (3)
C11—C16	1.390 (2)	C3—H3	0.9300
C11—C10	1.494 (2)	C13—C14	1.364 (3)
C1—C2	1.376 (2)	C13—H13	0.9300
C1—C6	1.389 (2)	C14—C15	1.374 (3)
C1—H1	0.9300	C14—H14	0.9300

C6—C5	1.389 (2)	C7—C8	1.507 (3)
C12—C13	1.376 (3)	C7—H7A	0.9700
C12—H12	0.9300	C7—H7B	0.9700
C5—C4	1.394 (3)	C4—H4	0.9300
C5—C7	1.517 (2)	C8—H8A	0.9700
C10—H10A	0.9700	C8—H8B	0.9700
C10—H10B	0.9700	C15—H15	0.9300
C9—C8	1.508 (3)		

O1—S1—O2	118.41 (8)	H9A—C9—H9B	108.2
O1—S1—N1	108.44 (7)	C15—C16—C11	120.08 (18)
O2—S1—N1	109.67 (8)	C15—C16—H16	120.0
O1—S1—C10	108.68 (8)	C11—C16—H16	120.0
O2—S1—C10	106.42 (8)	C1—C2—C3	119.78 (17)
N1—S1—C10	104.30 (7)	C1—C2—H2	120.1
C6—N1—C9	115.08 (12)	C3—C2—H2	120.1
C6—N1—S1	122.03 (10)	C4—C3—C2	119.99 (17)
C9—N1—S1	117.50 (10)	C4—C3—H3	120.0
C12—C11—C16	118.48 (16)	C2—C3—H3	120.0
C12—C11—C10	120.05 (15)	C14—C13—C12	120.44 (18)
C16—C11—C10	121.46 (15)	C14—C13—H13	119.8
C2—C1—C6	120.02 (16)	C12—C13—H13	119.8
C2—C1—H1	120.0	C13—C14—C15	119.68 (18)
C6—C1—H1	120.0	C13—C14—H14	120.2
C1—C6—C5	120.99 (14)	C15—C14—H14	120.2
C1—C6—N1	120.25 (13)	C8—C7—C5	114.05 (15)
C5—C6—N1	118.62 (13)	C8—C7—H7A	108.7
C13—C12—C11	120.92 (17)	C5—C7—H7A	108.7
C13—C12—H12	119.5	C8—C7—H7B	108.7
C11—C12—H12	119.5	C5—C7—H7B	108.7
C6—C5—C4	117.24 (16)	H7A—C7—H7B	107.6
C6—C5—C7	122.76 (15)	C3—C4—C5	121.93 (18)
C4—C5—C7	119.95 (16)	C3—C4—H4	119.0
C11—C10—S1	113.53 (11)	C5—C4—H4	119.0
C11—C10—H10A	108.9	C7—C8—C9	110.39 (15)
S1—C10—H10A	108.9	C7—C8—H8A	109.6
C11—C10—H10B	108.9	C9—C8—H8A	109.6
S1—C10—H10B	108.9	C7—C8—H8B	109.6
H10A—C10—H10B	107.7	C9—C8—H8B	109.6
N1—C9—C8	109.76 (14)	H8A—C8—H8B	108.1
N1—C9—H9A	109.7	C14—C15—C16	120.39 (19)
C8—C9—H9A	109.7	C14—C15—H15	119.8
N1—C9—H9B	109.7	C16—C15—H15	119.8
C8—C9—H9B	109.7		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14···O1^{i}	0.93	2.69	3.573 (2)	158
C10—H10*A*···O2^{ii}	0.97	2.68	3.575 (2)	153

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