CRYSTALLOGRAPHIC COMMUNICATIONS

OPEN ACCESS

ISSN 2056-9890

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

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Received 9 November 2014; accepted 19 November 2014

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

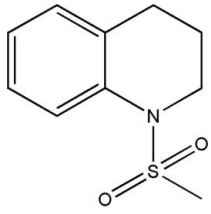
In the title compound, $C_{10}H_{13}NO_2S$, the heterocyclic ring adopts a half-chair conformation and the bond-angle sum at the N atom is 347.9°. In the crystal, inversion dimers linked by pairs of C—H···O hydrogen bonds generate $R_2^2(8)$ loops.

Keywords: crystal structure; 1,2,3,4-tetrahydroquinoline; physiological activities; photosensitizers.

CCDC reference: 1034951

1. Related literature

For background to tetrahydroquinolines, see: Chulakov *et al.* (2012); Kadutskii *et al.* (2012); Katritsky *et al.* (1996); Keith *et al.* (2001). For a related structure, see: Jeyaseelan *et al.* (2014).



2. Experimental

2.1. Crystal data

$C_{10}H_{13}NO_2S$
M_r = 211.27
Triclinic, $P\overline{1}$
a = 5.5865 (2) Å
b = 9.2195 (4) Å
c = 10.1924 (4) Å
α = 85.798 (2)°
β = 84.686 (2)°
γ = 77.166 (2)°
V = 508.89 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 294 K
0.24 × 0.20 × 0.16 mm

2.2. Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
T_{min} = 0.933, T_{max} = 0.955
7417 measured reflections
1973 independent reflections
1844 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.106
S = 1.07
1973 reflections
128 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 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
C10—H10C···O2^{i}	0.96	2.50	3.431 (2)	164

Symmetry code: (i) $-x, -y+1, -z+2$.

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, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL2014*.

Acknowledgements

SJ thanks Vision Group on Science and Technology, Government of Karnataka, for awarding a major project under CISE scheme (reference No. VGST/CISE/GRD-192/2013–14). BSP thanks Rajegowda, Department of Studies and Research in Physics, UCS, Tumkur University, Karnataka 572 103, India, for his support.

Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7314).

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supporting information

Acta Cryst. (2015). E**71**, o20 [doi:10.1107/S2056989014025353]

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

S. Jeyaseelan, S. L. Nagendra Babu, G. Venkateshappa, P. Raghavendra Kumar and B. S. Palakshamurthy

S1. Chemical context

Derivatives of tetrahydroquinolines display a wide range of physiological activities, they been found to be pesticides, antioxidants, photosensitizers, and dyes (Katritsky *et al.*, 1996). Heterocyclic compounds of 1,2,3,4-tetrahydroquinoline derivatives play important role in synthesize efficient kinetic resolution with predominant (S,S)-(R,R)-diastereoisomers (Chulakov *et al.*, 2012), optically active camphor moieties (Kadutskii *et al.*, 2012**)**, and biologically active compounds, synthetic intermediates (Keith *et al.*, 2001).

In due course of our study, we have synthised a series of 1,2,3,4-tetrahydroquinoline with derivatives of suloponyl 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 and the results are compared with crystal structure of 1-tosyl-1,2,3,4-tetrahydroquinoline(II) (Jeyaseelan *et al.*, 2014) .

S2. Structural commentary

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

S3. Supramolecular features

In the crystal, inversion dimers linked by pairs of C10—H10C···O2 hydrogen bonds generate $R_2^2(8)$ ring motifs.

S4. Synthesis and crystallization

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 methanesulfonyl chloride (12 mmol) in 10 ml dry dichloromethane 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 slovent mixture of ethyl acetate and hexane(1:2) to yield colourless prisms of (I).

S5. Refinement details

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 = 0.93-0.99 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).

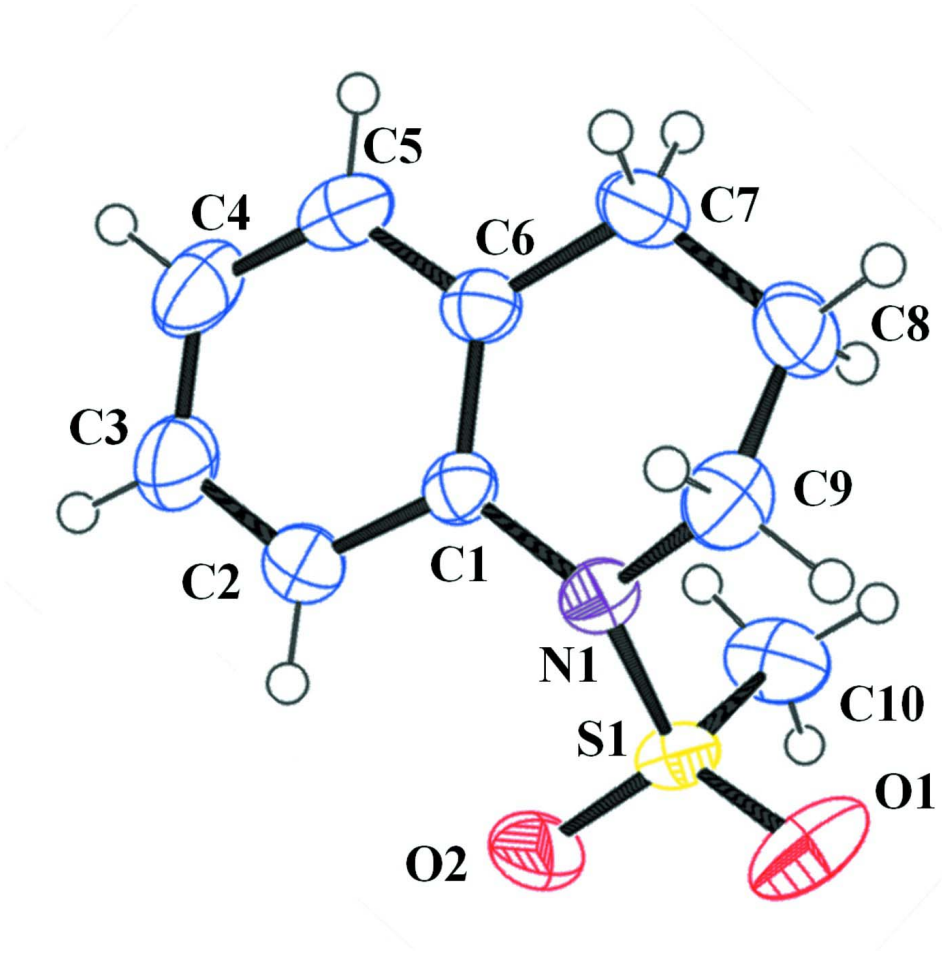

Figure 1

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

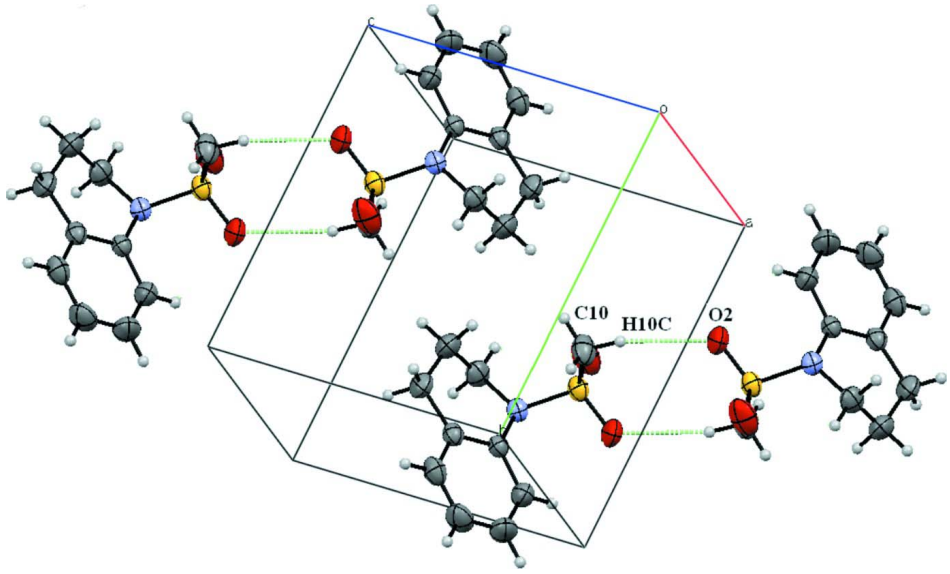

Figure 2

The molecular packing of the title compound, dashed lines indicates the inversion dimers linked by pairs of C—H···O hydrogen bonds with $R_2^2(8)$ ring motifs.

1-Methanesulfonyl-1,2,3,4-tetrahydroquinoline

Crystal data

$C_{10}H_{13}NO_2S$
$M_r = 211.27$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 5.5865$ (2) Å
$b = 9.2195$ (4) Å
$c = 10.1924$ (4) Å
$\alpha = 85.798$ (2)°
$\beta = 84.686$ (2)°
$\gamma = 77.166$ (2)°
$V = 508.89$ (4) Å^3
$Z = 2$
$F(000) = 224$
Prism
$D_x = 1.379$ Mg m^{-3}
Melting point: 414 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1844 reflections
$\theta = 2.0$–$26.0°$
$\mu = 0.29$ mm^{-1}
$T = 294$ K
Prism, colourless
0.24 × 0.20 × 0.16 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 1.09 pixels mm^{-1}
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
$T_{min} = 0.933$, $T_{max} = 0.955$
7417 measured reflections
1973 independent reflections
1844 reflections with $I > 2\sigma(I)$
$R_{int} = 0.042$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.0°$
$h = -6 \rightarrow 6$
$k = -11 \rightarrow 11$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.106$
$S = 1.07$
1973 reflections
128 parameters
0 restraints
0 constraints
Primary atom site location: difference Fourier map
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0543P)^2 + 0.1542P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.0188 (3)	0.52244 (15)	0.71208 (15)	0.0682 (4)
C1	0.3459 (3)	0.12140 (16)	0.73875 (15)	0.0323 (3)
C2	0.2249 (4)	0.0388 (2)	0.83231 (18)	0.0478 (4)
H2	0.0635	0.0778	0.8639	0.057*
C3	0.3448 (4)	−0.1007 (2)	0.8780 (2)	0.0620 (6)
H3	0.2647	−0.1549	0.9413	0.074*
C4	0.5827 (4)	−0.1601 (2)	0.8300 (2)	0.0593 (5)
H4	0.6643	−0.2534	0.8619	0.071*
C5	0.6980 (3)	−0.0810 (2)	0.73512 (18)	0.0480 (4)
H5	0.8574	−0.1227	0.7022	0.058*
C6	0.5840 (3)	0.06025 (17)	0.68628 (15)	0.0359 (4)
C7	0.7133 (3)	0.1364 (2)	0.57329 (19)	0.0489 (4)
H7A	0.8788	0.1355	0.5955	0.059*
H7B	0.7273	0.0797	0.4954	0.059*
C8	0.5850 (4)	0.2949 (2)	0.54063 (19)	0.0537 (5)
H8A	0.6378	0.3244	0.4512	0.064*
H8B	0.6293	0.3604	0.6001	0.064*
C9	0.3091 (4)	0.3101 (2)	0.55315 (16)	0.0461 (4)
H9A	0.2296	0.4127	0.5318	0.055*
H9B	0.2648	0.2483	0.4903	0.055*
N1	0.2186 (2)	0.26547 (14)	0.68785 (12)	0.0343 (3)
C10	0.3540 (4)	0.4495 (2)	0.8543 (2)	0.0548 (5)
H10A	0.4427	0.4978	0.7856	0.082*
H10B	0.4619	0.3619	0.8890	0.082*
H10C	0.2931	0.5165	0.9235	0.082*
O2	−0.0312 (3)	0.33906 (15)	0.89749 (14)	0.0592 (4)
S1	0.10556 (7)	0.39877 (4)	0.78957 (4)	0.03662 (17)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0777 (10)	0.0463 (8)	0.0683 (9)	0.0213 (7)	−0.0239 (8)	−0.0061 (7)
C1	0.0337 (7)	0.0307 (7)	0.0331 (7)	−0.0068 (6)	−0.0033 (6)	−0.0056 (6)
C2	0.0479 (10)	0.0421 (9)	0.0499 (10)	−0.0084 (8)	0.0107 (8)	−0.0023 (7)
C3	0.0783 (15)	0.0417 (10)	0.0591 (12)	−0.0096 (10)	0.0141 (10)	0.0071 (9)
C4	0.0768 (14)	0.0358 (9)	0.0578 (11)	0.0021 (9)	−0.0054 (10)	0.0022 (8)
C5	0.0419 (9)	0.0425 (9)	0.0553 (10)	0.0027 (7)	−0.0041 (8)	−0.0117 (8)
C6	0.0336 (8)	0.0364 (8)	0.0390 (8)	−0.0080 (6)	−0.0024 (6)	−0.0093 (6)
C7	0.0381 (9)	0.0537 (10)	0.0543 (10)	−0.0125 (8)	0.0100 (8)	−0.0084 (8)
C8	0.0626 (12)	0.0528 (11)	0.0455 (10)	−0.0200 (9)	0.0140 (9)	−0.0013 (8)
C9	0.0606 (11)	0.0445 (9)	0.0308 (8)	−0.0067 (8)	−0.0049 (7)	0.0003 (7)
N1	0.0350 (7)	0.0333 (7)	0.0336 (7)	−0.0043 (5)	−0.0030 (5)	−0.0042 (5)
C10	0.0488 (10)	0.0674 (12)	0.0529 (11)	−0.0158 (9)	−0.0023 (8)	−0.0261 (9)
O2	0.0526 (8)	0.0569 (8)	0.0645 (9)	−0.0122 (6)	0.0261 (7)	−0.0179 (7)
S1	0.0304 (2)	0.0346 (3)	0.0418 (3)	0.00108 (16)	−0.00305 (16)	−0.00688 (17)

Geometric parameters (Å, º)

O1—S1	1.4227 (13)	C7—H7B	0.9700
C1—C2	1.396 (2)	C8—C9	1.511 (3)
C1—C6	1.398 (2)	C8—H8A	0.9700
C1—N1	1.4446 (18)	C8—H8B	0.9700
C2—C3	1.381 (3)	C9—N1	1.480 (2)
C2—H2	0.9300	C9—H9A	0.9700
C3—C4	1.379 (3)	C9—H9B	0.9700
C3—H3	0.9300	N1—S1	1.6446 (13)
C4—C5	1.369 (3)	C10—S1	1.7555 (18)
C4—H4	0.9300	C10—H10A	0.9600
C5—C6	1.394 (2)	C10—H10B	0.9600
C5—H5	0.9300	C10—H10C	0.9600
C6—C7	1.515 (2)	O2—S1	1.4279 (13)
C7—C8	1.505 (3)	S1—O1	1.4227 (13)
C7—H7A	0.9700		
C2—C1—C6	120.12 (15)	C9—C8—H8A	109.6
C2—C1—N1	120.16 (14)	C7—C8—H8B	109.6
C6—C1—N1	119.53 (13)	C9—C8—H8B	109.6
C3—C2—C1	120.02 (17)	H8A—C8—H8B	108.1
C3—C2—H2	120.0	N1—C9—C8	111.80 (14)
C1—C2—H2	120.0	N1—C9—H9A	109.3
C4—C3—C2	120.28 (18)	C8—C9—H9A	109.3
C4—C3—H3	119.9	N1—C9—H9B	109.3
C2—C3—H3	119.9	C8—C9—H9B	109.3
C5—C4—C3	119.56 (18)	H9A—C9—H9B	107.9
C5—C4—H4	120.2	C1—N1—C9	114.89 (12)
C3—C4—H4	120.2	C1—N1—S1	119.76 (10)

C4—C5—C6	122.06 (16)	C9—N1—S1	117.41 (10)
C4—C5—H5	119.0	S1—C10—H10A	109.5
C6—C5—H5	119.0	S1—C10—H10B	109.5
C5—C6—C1	117.87 (15)	H10A—C10—H10B	109.5
C5—C6—C7	119.39 (15)	S1—C10—H10C	109.5
C1—C6—C7	122.61 (15)	H10A—C10—H10C	109.5
C8—C7—C6	114.00 (14)	H10B—C10—H10C	109.5
C8—C7—H7A	108.8	O1—S1—O2	118.38 (10)
C6—C7—H7A	108.8	O1—S1—N1	106.54 (8)
C8—C7—H7B	108.8	O2—S1—N1	108.22 (7)
C6—C7—H7B	108.8	O1—S1—C10	108.39 (10)
H7A—C7—H7B	107.6	O2—S1—C10	107.06 (9)
C7—C8—C9	110.45 (15)	N1—S1—C10	107.85 (8)
C7—C8—H8A	109.6		

C6—C1—C2—C3	3.1 (3)	C6—C1—N1—C9	22.44 (19)
N1—C1—C2—C3	178.13 (17)	C2—C1—N1—S1	59.22 (18)
C1—C2—C3—C4	−1.0 (3)	C6—C1—N1—S1	−125.77 (13)
C2—C3—C4—C5	−1.1 (3)	C8—C9—N1—C1	−51.15 (19)
C3—C4—C5—C6	1.2 (3)	C8—C9—N1—S1	97.83 (15)
C4—C5—C6—C1	0.9 (3)	C1—N1—S1—O1	−176.90 (12)
C4—C5—C6—C7	−174.89 (18)	C9—N1—S1—O1	35.68 (15)
C2—C1—C6—C5	−3.1 (2)	C1—N1—S1—O1	−176.90 (12)
N1—C1—C6—C5	−178.07 (13)	C9—N1—S1—O1	35.68 (15)
C2—C1—C6—C7	172.62 (15)	C1—N1—S1—O2	−48.59 (13)
N1—C1—C6—C7	−2.4 (2)	C9—N1—S1—O2	163.98 (12)
C5—C6—C7—C8	−173.18 (16)	C1—N1—S1—O2	−48.59 (13)
C1—C6—C7—C8	11.2 (2)	C9—N1—S1—O2	163.98 (12)
C6—C7—C8—C9	−38.3 (2)	C1—N1—S1—C10	66.91 (14)
C7—C8—C9—N1	58.9 (2)	C9—N1—S1—C10	−80.52 (14)
C2—C1—N1—C9	−152.58 (15)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10*C*···O2^{i}	0.96	2.50	3.431 (2)	164

Symmetry code: (i) −*x*, −*y*+1, −*z*+2.