data reports

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

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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.

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).
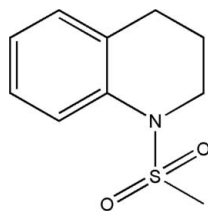
2. Experimental

2.1. Crystal data

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

2.2. Data collection

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

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$	128 parameters
$wR(F^2) = 0.106$	H-atom parameters constrained
$S = 1.07$	$\Delta\rho_{max} = 0.24$ e Å^{-3}
1973 reflections	$\Delta\rho_{min} = -0.31$ 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

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supporting information

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

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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).
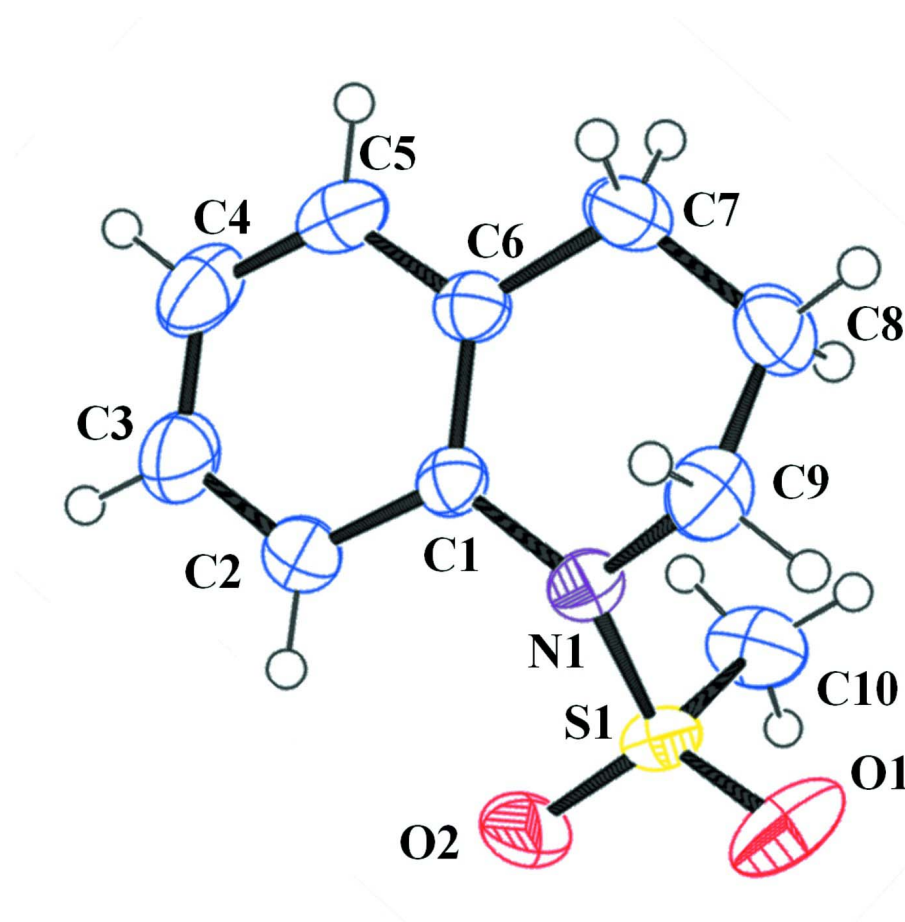
Figure 1

The molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level.
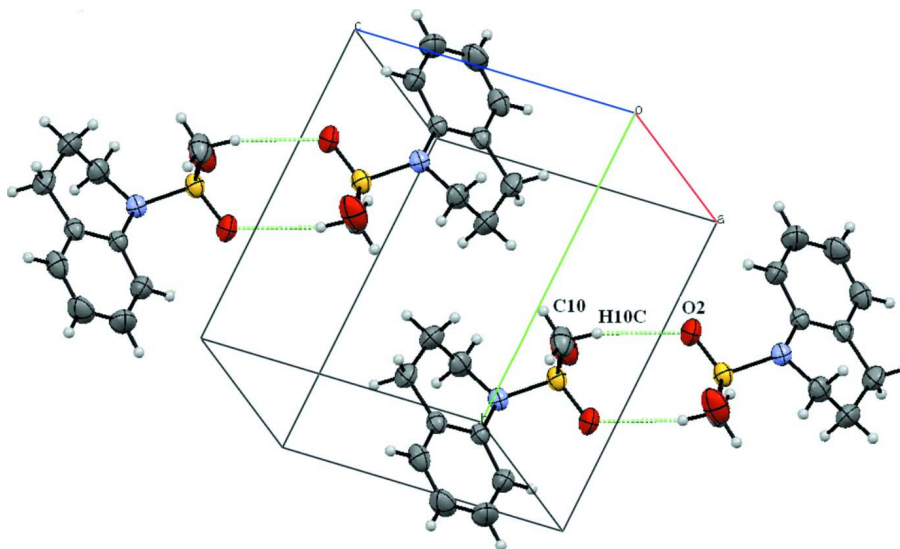
Figure 2

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

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

Data collection

Bruker APEXII CCD diffractometer	7417 measured reflections
Radiation source: fine-focus sealed tube	1973 independent reflections
Graphite monochromator	1844 reflections with $I > 2\sigma(I)$
Detector resolution: 1.09 pixels mm^{-1}	$R_{int} = 0.042$
phi and ω scans	$\theta_{max} = 26.0°$, $\theta_{min} = 2.0°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)	$h = -6\rightarrow6$
	$k = -11\rightarrow11$
$T_{min} = 0.933$, $T_{max} = 0.955$	$l = -12\rightarrow12$

Refinement

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

	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 (Å^2)

	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*⋯O2ⁱ	0.96	2.50	3.431 (2)	164

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