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

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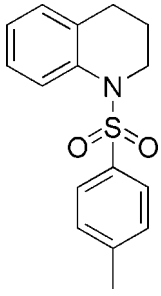
In the title compound, $C_{16}H_{17}NO_2S$, the heterocyclic ring adopts a half-chair conformation and the bond-angle sum at the N atom is 350.2°. The dihedral angle between the planes of the aromatic rings is 47.74 (10)°. In the crystal, molecules are linked by C—H···O hydrogen bonds to generate [010] 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

$C_{16}H_{17}NO_2S$
$M_r$ = 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) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.22 mm$^{-1}$
$T$ = 94 K
0.24 × 0.22 × 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 $I > 2\sigma(I)$
$R_{int}$ = 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
$\Delta\rho_{max}$ = 0.23 e Å$^{-3}$
$\Delta\rho_{min}$ = −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

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). BSPM thanks Rajegowda, Department of Studies and Research in Physics, UCS, Tumkur University, Karnataka 572 103, India, for his suport.

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

**S. Jeyaseelan, K.V. Asha, G. Venkateshappa, P. Raghavendrakumar and B.S. Palakshamurthy**

### 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 perticlar 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. Database survey

### S5. 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% $Na_2CO_3$ and brine. 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 in the mixture of ethyl acetate and hexane(1:1) to get colourless prisms.

### S6. 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).

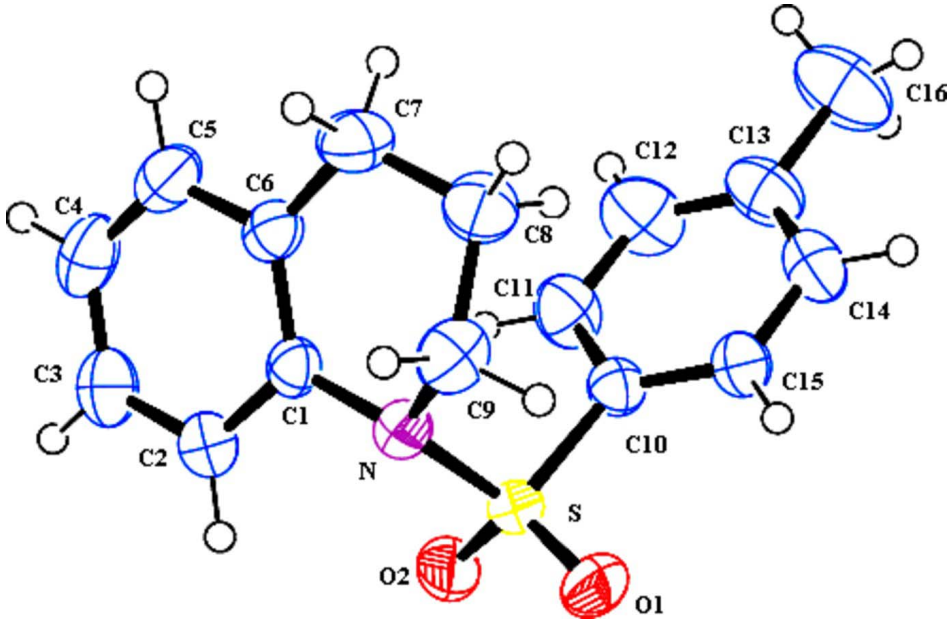


**Figure 1**

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

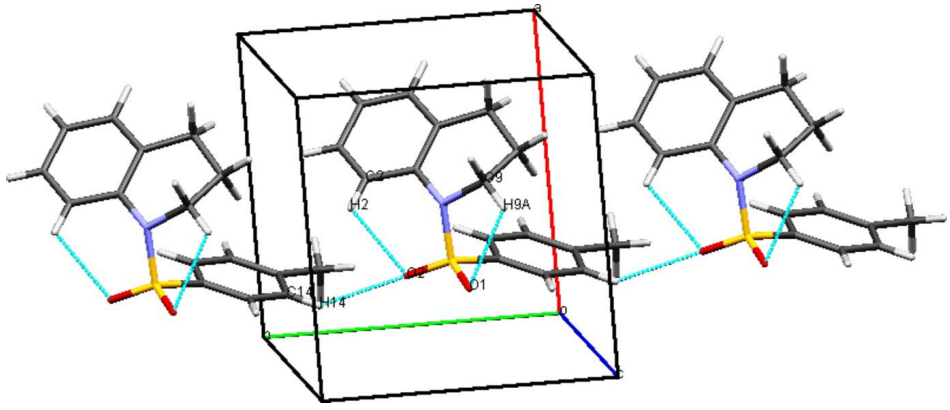


**Figure 2**

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 2yn
$a = 8.2176\ (7)$ Å
$b = 8.0468\ (6)$ Å
$c = 22.2439\ (18)$ Å
$\beta = 98.107\ (4)°$
$V = 1456.2\ (2)$ Å$^3$
$Z = 4$
$F(000) = 608$

Prism
$D_x = 1.311$ Mg m$^{-3}$
Melting point: 402 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2327 reflections
$\theta = 1.9–25.0°$
$\mu = 0.22$ mm$^{-1}$
$T = 94$ K
Prism, colourless
0.24 × 0.22 × 0.18 mm

*Data collection*

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 1.9 pixels mm$^{-1}$
phi and $\omega$ 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 $I > 2\sigma(I)$
$R_{int}$ = 0.046
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 1.9°
$h$ = −9→9
$k$ = −9→9
$l$ = −26→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 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.37 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$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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, º)*

| | | | |
|---|---|---|---|
| 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, º)*

| *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*.