



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

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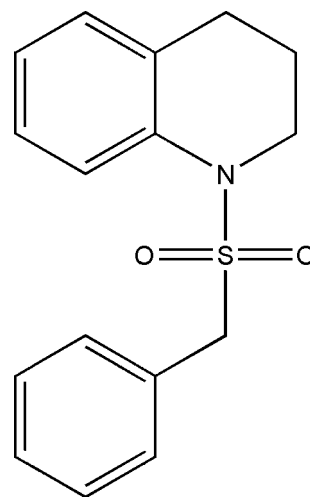
In the title compound, C₁₆H₁₇NO₂S, the heterocyclic ring adopts a half-chair conformation and the bond-angle sum at the N atom is 354.6°. The dihedral angle between the planes of the aromatic rings is 74.15 (10)°. In the crystal, molecules are linked by weak C—H...O hydrogen bonds, generating C(8) and 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 ₂ S | <i>V</i> = 1438.44 (9) Å ³ |
| <i>M_r</i> = 287.36 | <i>Z</i> = 4 |
| Monoclinic, <i>P</i> 2 ₁ / <i>n</i> | Mo <i>K</i> α radiation |
| <i>a</i> = 13.5690 (5) Å | <i>μ</i> = 0.23 mm ⁻¹ |
| <i>b</i> = 6.7128 (2) Å | <i>T</i> = 295 K |
| <i>c</i> = 16.8317 (6) Å | 0.24 × 0.20 × 0.18 mm |
| <i>β</i> = 110.243 (1)° | |

2.2. Data collection

| | |
|--|---|
| Bruker APEXII CCD diffractometer | 19601 measured reflections |
| Absorption correction: multi-scan (SADABS; Bruker, 2013) | 2529 independent reflections |
| <i>T</i> _{min} = 0.947, <i>T</i> _{max} = 0.960 | 2264 reflections with <i>I</i> > 2σ(<i>I</i>) |
| | <i>R</i> _{int} = 0.051 |

2.3. Refinement

| | |
|---|---|
| <i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)] = 0.036 | 181 parameters |
| <i>wR</i> (<i>F</i> ²) = 0.101 | H-atom parameters constrained |
| <i>S</i> = 1.08 | Δ <i>ρ</i> _{max} = 0.15 e Å ⁻³ |
| 2529 reflections | Δ <i>ρ</i> _{min} = -0.38 e Å ⁻³ |

Table 1

Hydrogen-bond geometry (Å, °).

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|-----------------------------|-------------|---------------|-----------------------|-------------------------|
| 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.

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

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supporting information

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

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S1. Introduction

Heterocyclic compounds of 1,2,3,4-tetrahydroquinoline derivatives play important role in synthesise antimalarial (Bendale *et al.*, 2007), antipsychotic (Singer *et al.*, 2005) drugs. Keeping this in mind we have synthesised 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 dichloromethane was added drop wise. After 2h of stirring at 15–20°C, the reaction mixture was washed with 5% Na₂CO₃ and brine. The 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 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).

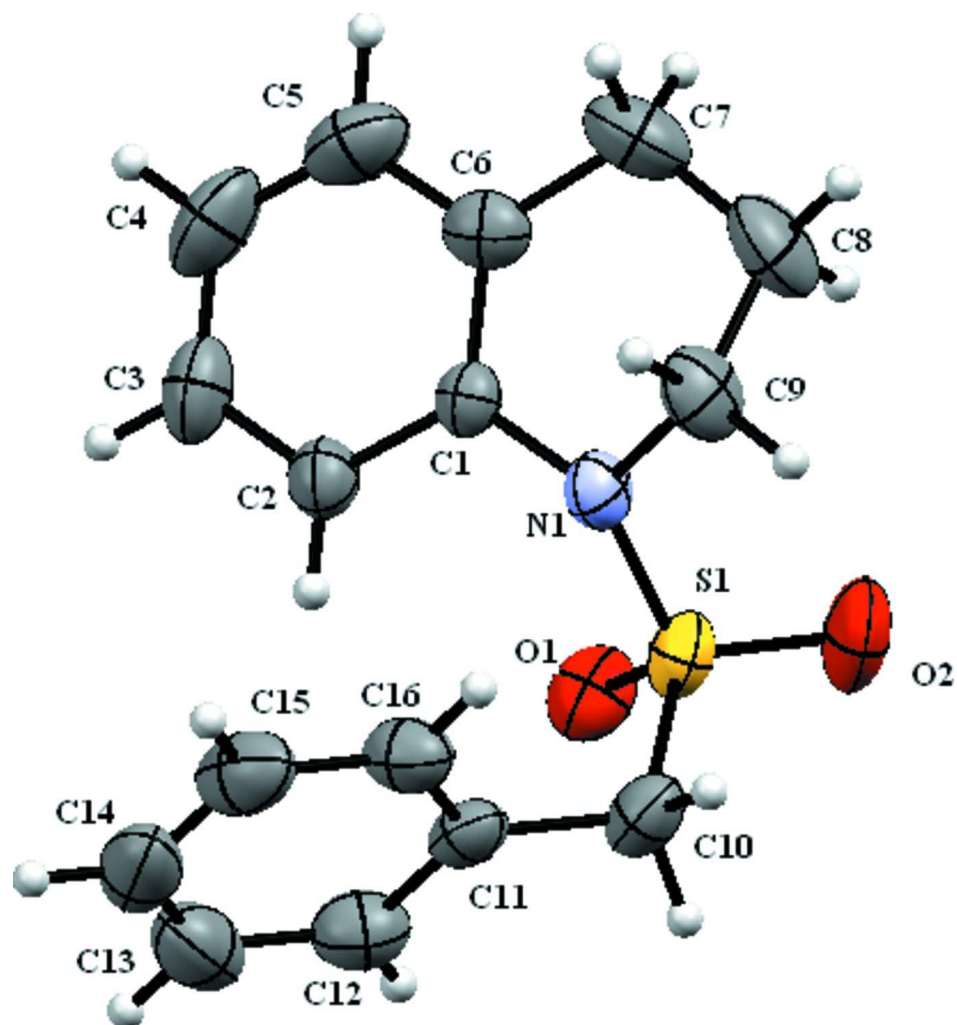
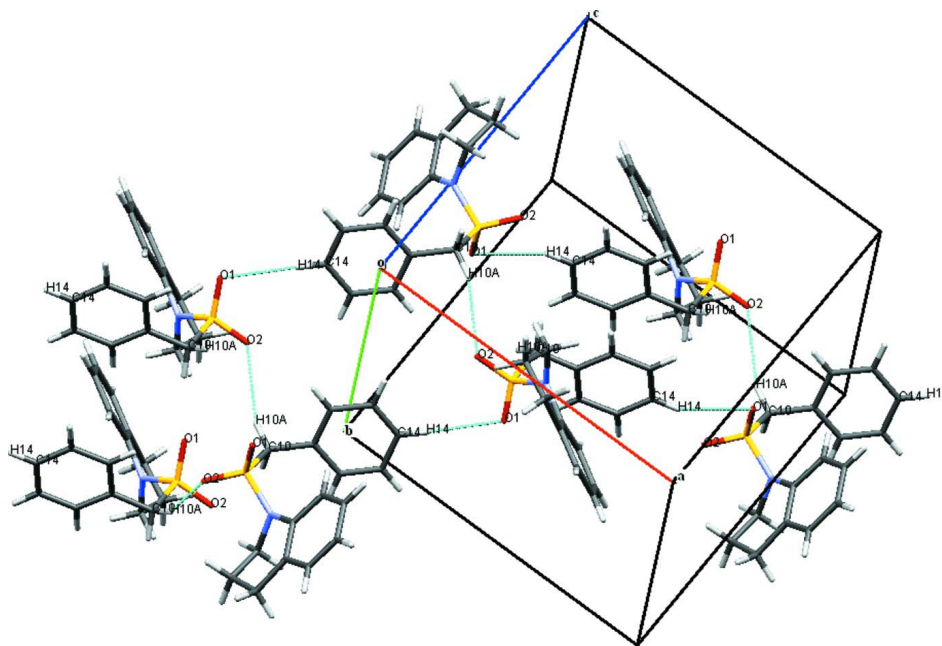


Figure 1

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: $-P\ 2_1n$

$a = 13.5690$ (5) Å

$b = 6.7128$ (2) Å

$c = 16.8317$ (6) Å

$\beta = 110.243$ (1)°

$V = 1438.44$ (9) Å³

$Z = 4$

$F(000) = 608$

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

$0.24 \times 0.20 \times 0.18$ mm

Data collection

Bruker APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 1.90 pixels mm⁻¹

ϕ 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 $I > 2\sigma(I)$

$R_{\text{int}} = 0.051$

$\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 1.7^\circ$

$h = -16 \rightarrow 16$

$k = -7 \rightarrow 7$

$l = -19 \rightarrow 20$

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 $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.15 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.38 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|--------------|--------------|----------------------------------|
| 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, °)

| | | | |
|---------|-------------|---------|-----------|
| 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, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|--|-------------|---------------------|----------------------------|-------------------------------|
| C14—H14 \cdots O1 ⁱ | 0.93 | 2.69 | 3.573 (2) | 158 |
| C10—H10 <i>A</i> \cdots O2 ⁱⁱ | 0.97 | 2.68 | 3.575 (2) | 153 |

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