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# 1,4-Ditosyl-1,4-diazepane

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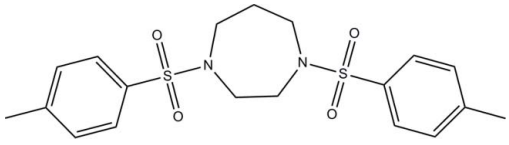
Key indicators: single-crystal X-ray study; $T$ = 173 K; mean $\sigma$(C–C) = 0.004 Å; disorder in main residue; $R$ factor = 0.036; $wR$ factor = 0.083; data-to-parameter ratio = 13.3.

In the title compound, $C_{19}H_{24}N_2O_4S_2$, the dihedral angle formed by the benzene rings is 82.88 (7)°, and the molecular conformation is enforced by weak intramolecular C—H···O contacts. Two C atoms of the 1,4-diazepane ring are disordered over two sets of sites with a refined occupancy ratio of 0.534 (13):0.466 (13). In the crystal, molecules are linked by weak intermolecular C—H···O interactions into chains parallel to the *a* axis.

## Related literature

For related structures, see: Romba *et al.* (2002). For the biological activity of heterocyclic compounds, see: Xu *et al.* (2006); Yu *et al.* (2009).



## Experimental

### Crystal data

$C_{19}H_{24}N_2O_4S_2$
$M_r$ = 408.52
Orthorhombic, $P2_12_12_1$
$a$ = 6.3407 (13) Å
$b$ = 10.367 (2) Å
$c$ = 30.516 (6) Å
$V$ = 2005.9 (7) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.29 mm$^{-1}$
$T$ = 173 K
0.20 × 0.20 × 0.10 mm

### Data collection

Rigaku Mercury CCD/AFC diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2007)
$T_{min}$ = 0.944, $T_{max}$ = 0.971
11747 measured reflections
3531 independent reflections
3430 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.035

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.083
$S$ = 1.07
3531 reflections
265 parameters
6 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.13 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.20 e Å$^{-3}$
Absolute structure: Flack (1983), 1442 Friedel pairs
Flack parameter: −0.03 (7)

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C10—H10$B$···O3 | 0.97 | 2.40 | 2.886 (3) | 110 |
| C12—H12$A$···O1 | 0.97 | 2.39 | 2.878 (3) | 111 |
| C10—H10$B$···O4$^{i}$ | 0.97 | 2.52 | 3.142 (3) | 122 |
| C12—H12$A$···O2$^{i}$ | 0.97 | 2.50 | 3.035 (3) | 115 |

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

Data collection: *CrystalClear* (Rigaku, 2007); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2746).

## References

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Xu, L. Z., Huang, Y. W., Yu, G. P., Si, G. D. & Zhu, Q. (2006). *Struct. Chem.* **17**, 235–239.
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# supplementary materials

*Acta Cryst.* (2012). E**68**, o1716 [doi:10.1107/S1600536812020958]

## 1,4-Ditosyl-1,4-diazepane

**Shuang-Hua Yang and Zhi-Wei Zhai**

### Comment

Many heterocyclic compounds have been widely used as potent and broad-spectrum fungicides (Xu *et al.*, 2006; Yu *et al.*, 2009). In order to search for new heterocylic compounds with higher biological activities, we synthesized the title compound and describe its structure herein.

In title compound (Fig. 1), all bond lengths are normal and in a good agreement with those reported for related compounds (Romba *et al.*, 2002). Two atoms (C8 and C9) of the 1,4-diazepane ring are disordered over two orientations with refined occupancy ratio of 0.466 (13)/0.534 (13). The dihedral angle formed by the phenyl rings is 82.88 (7)°. The molecular conformation is stabilized by intramolecular C—H···O hydrogen bonds (Table 1). In the crystal packing, molecules are linked by intermolecular C—H···O hydrogen bonds to form chains parallel to the *a* axis.

### Experimental

NaH (1.9 g, 0.08 mol) was dissolved in 30 ml DMF and cooled with an ice bath, then *N*,*N*′-di(*p*-toluenesulfonyl)-ethane-1,2-diamine (7.4 g, 0.02 mol) in 10 ml DMF was added dropwise to the solution. After stirring 30 min, 1,3-di-bromopropane in 5 ml DMF was added dropwise and the resulting suspension stirred overnight at room temperature. The residue was quenched by saturated $NH_4Cl$ and extracted with ethyl acetate (3 × 200 ml). The combined organic layer was washed with saturated NaCl and dried over sodium sulfate. The solvent was removed and the residue was recrystallized from EtOH/DMF (5:1 *v*/*v*) to give the title compound (6.7 g, 82%). Single crystals suitable for X-ray measurements were obtained by recrystallization from acetonitrile at room temperature.

### Refinement

H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95 or 0.99 Å and with $U_{iso}$(H) = 1.2 times $U_{eq}$(C) for methylene H atoms and 1.5$U_{eq}$(C) for the methyl H atoms. Distance constraints (N—C = 1.47 (1) Å; C—C = 1.52 (1) Å) were applied to the disordered atoms C8 and C9.

### Computing details

Data collection: *CrystalClear* (Rigaku, 2007); cell refinement: *CrystalClear* (Rigaku, 2007); data reduction: *CrystalClear* (Rigaku, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

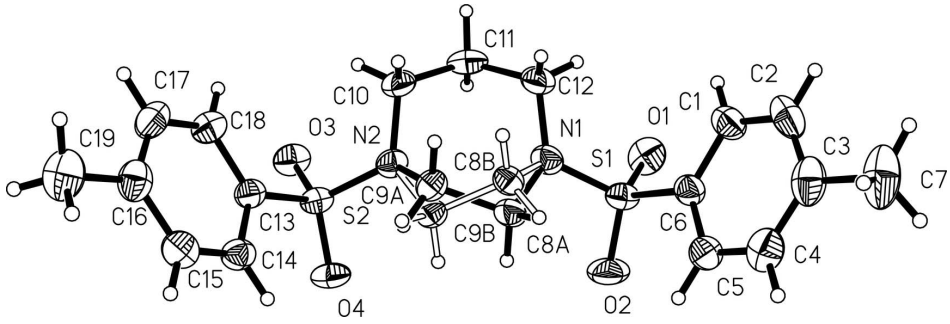


**Figure 1**

The molecular structure of the title compound, with 40% probability displacement ellipsoids.

### 1,4-Ditosyl-1,4-diazepane

*Crystal data*

$C_{19}H_{24}N_2O_4S_2$
$M_r$ = 408.52
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a$ = 6.3407 (13) Å
$b$ = 10.367 (2) Å
$c$ = 30.516 (6) Å
$V$ = 2005.9 (7) Å$^3$
$Z$ = 4
$F(000)$ = 864
$D_x$ = 1.353 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 7269 reflections
$\theta$ = 1.7–27.5°
$\mu$ = 0.29 mm$^{-1}$
$T$ = 173 K
Block, colourless
0.20 × 0.20 × 0.10 mm

*Data collection*

Rigaku Mercury CCD/AFC diffractometer
Radiation source: Sealed Tube
Graphite Monochromator monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2007)
$T_{min}$ = 0.944, $T_{max}$ = 0.971
11747 measured reflections
3531 independent reflections
3430 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.035
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 2.8°
$h$ = −7→7
$k$ = −8→12
$l$ = −36→35

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.083
$S$ = 1.07
3531 reflections
265 parameters
6 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.035P)^2 + 0.4874P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.13 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.20 e Å$^{-3}$
Absolute structure: Flack (1983), 1442 Friedel pairs
Flack parameter: −0.03 (7)

*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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| S1 | 0.84854 (9) | −0.00702 (6) | 0.40081 (2) | 0.04437 (16) | |
| S2 | 0.83732 (9) | 0.12928 (5) | 0.22154 (2) | 0.03921 (15) | |
| O1 | 0.7421 (3) | 0.08375 (18) | 0.42783 (6) | 0.0594 (5) | |
| O2 | 1.0685 (3) | 0.0089 (2) | 0.39251 (7) | 0.0716 (6) | |
| O3 | 0.7251 (3) | 0.24688 (15) | 0.21378 (6) | 0.0501 (4) | |
| O4 | 1.0600 (2) | 0.13432 (18) | 0.22902 (6) | 0.0532 (5) | |
| N1 | 0.7323 (3) | −0.0080 (2) | 0.35377 (6) | 0.0439 (5) | |
| N2 | 0.7313 (3) | 0.06018 (17) | 0.26359 (6) | 0.0388 (5) | |
| C1 | 0.6335 (4) | −0.1951 (3) | 0.44536 (8) | 0.0511 (6) | |
| H1 | 0.5214 | −0.1374 | 0.4464 | 0.061* | |
| C2 | 0.6164 (5) | −0.3155 (3) | 0.46458 (8) | 0.0603 (8) | |
| H2 | 0.4911 | −0.3386 | 0.4783 | 0.072* | |
| C3 | 0.7831 (6) | −0.4029 (3) | 0.46375 (8) | 0.0618 (8) | |
| C4 | 0.9649 (5) | −0.3682 (3) | 0.44218 (9) | 0.0589 (7) | |
| H4 | 1.0765 | −0.4263 | 0.4408 | 0.071* | |
| C5 | 0.9853 (4) | −0.2489 (3) | 0.42246 (8) | 0.0495 (6) | |
| H5 | 1.1091 | −0.2274 | 0.4079 | 0.059* | |
| C6 | 0.8205 (4) | −0.1615 (2) | 0.42453 (7) | 0.0419 (5) | |
| C8A | 0.8715 (15) | −0.0801 (9) | 0.3232 (2) | 0.044 (2) | 0.466 (13) |
| H8A1 | 0.8801 | −0.1695 | 0.3323 | 0.053* | 0.466 (13) |
| H8A2 | 1.0125 | −0.0438 | 0.3241 | 0.053* | 0.466 (13) |
| C9A | 0.7867 (18) | −0.0729 (7) | 0.2768 (3) | 0.040 (2) | 0.466 (13) |
| H9A1 | 0.8920 | −0.1065 | 0.2568 | 0.048* | 0.466 (13) |
| H9A2 | 0.6624 | −0.1270 | 0.2745 | 0.048* | 0.466 (13) |
| C8B | 0.7774 (14) | −0.1092 (6) | 0.31983 (19) | 0.0377 (15) | 0.534 (13) |
| H8B1 | 0.6480 | −0.1544 | 0.3126 | 0.045* | 0.534 (13) |
| H8B2 | 0.8765 | −0.1714 | 0.3316 | 0.045* | 0.534 (13) |
| C9B | 0.8687 (11) | −0.0489 (7) | 0.2785 (3) | 0.0353 (17) | 0.534 (13) |
| H9B1 | 1.0098 | −0.0173 | 0.2844 | 0.042* | 0.534 (13) |
| H9B2 | 0.8778 | −0.1135 | 0.2556 | 0.042* | 0.534 (13) |
| C10 | 0.5022 (4) | 0.0686 (3) | 0.26975 (8) | 0.0517 (7) | |
| H10A | 0.4390 | −0.0144 | 0.2633 | 0.062* | |
| H10B | 0.4447 | 0.1312 | 0.2494 | 0.062* | |
| C11 | 0.4459 (4) | 0.1078 (3) | 0.31613 (9) | 0.0518 (7) | |
| H11A | 0.5150 | 0.1891 | 0.3226 | 0.062* | |
| H11B | 0.2950 | 0.1226 | 0.3175 | 0.062* | |
| C12 | 0.5040 (4) | 0.0126 (3) | 0.35127 (8) | 0.0536 (7) | |

| | | | | |
|---|---|---|---|---|
| H12A | 0.4535 | 0.0438 | 0.3793 | 0.064* |
| H12B | 0.4350 | −0.0691 | 0.3453 | 0.064* |
| C13 | 0.7984 (4) | 0.0278 (2) | 0.17594 (7) | 0.0394 (5) |
| C14 | 0.9546 (4) | −0.0588 (2) | 0.16374 (8) | 0.0449 (6) |
| H14 | 1.0803 | −0.0630 | 0.1794 | 0.054* |
| C15 | 0.9215 (4) | −0.1392 (3) | 0.12792 (8) | 0.0515 (6) |
| H15 | 1.0266 | −0.1968 | 0.1196 | 0.062* |
| C16 | 0.7342 (5) | −0.1350 (3) | 0.10436 (9) | 0.0521 (6) |
| C17 | 0.5795 (4) | −0.0506 (3) | 0.11791 (9) | 0.0543 (7) |
| H17 | 0.4521 | −0.0486 | 0.1028 | 0.065* |
| C18 | 0.6080 (4) | 0.0313 (2) | 0.15328 (8) | 0.0469 (6) |
| H18 | 0.5015 | 0.0877 | 0.1618 | 0.056* |
| C19 | 0.7052 (6) | −0.2180 (3) | 0.06426 (9) | 0.0691 (9) |
| H19A | 0.5637 | −0.2509 | 0.0636 | 0.104* |
| H19B | 0.8030 | −0.2887 | 0.0652 | 0.104* |
| H19C | 0.7305 | −0.1673 | 0.0385 | 0.104* |
| C7 | 0.7658 (6) | −0.5318 (3) | 0.48659 (10) | 0.0877 (12) |
| H7A | 0.7705 | −0.5997 | 0.4652 | 0.131* |
| H7B | 0.6349 | −0.5359 | 0.5023 | 0.131* |
| H7C | 0.8810 | −0.5415 | 0.5067 | 0.131* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0326 (3) | 0.0467 (3) | 0.0537 (3) | −0.0031 (3) | 0.0021 (3) | 0.0001 (3) |
| S2 | 0.0305 (3) | 0.0333 (3) | 0.0538 (3) | −0.0003 (2) | −0.0042 (3) | 0.0032 (3) |
| O1 | 0.0676 (13) | 0.0522 (11) | 0.0584 (11) | 0.0016 (9) | −0.0065 (9) | −0.0171 (9) |
| O2 | 0.0299 (9) | 0.0798 (14) | 0.1052 (15) | −0.0070 (10) | 0.0024 (10) | 0.0327 (13) |
| O3 | 0.0498 (10) | 0.0296 (8) | 0.0710 (12) | 0.0033 (7) | −0.0041 (9) | 0.0093 (8) |
| O4 | 0.0271 (8) | 0.0608 (11) | 0.0717 (12) | −0.0036 (8) | −0.0061 (8) | −0.0080 (10) |
| N1 | 0.0411 (11) | 0.0450 (11) | 0.0457 (10) | 0.0089 (9) | 0.0087 (9) | −0.0023 (10) |
| N2 | 0.0373 (11) | 0.0326 (10) | 0.0464 (11) | 0.0048 (8) | −0.0031 (9) | 0.0025 (9) |
| C1 | 0.0479 (15) | 0.0597 (16) | 0.0458 (13) | −0.0042 (13) | 0.0092 (12) | −0.0037 (12) |
| C2 | 0.073 (2) | 0.0680 (18) | 0.0401 (14) | −0.0238 (16) | 0.0104 (14) | −0.0042 (13) |
| C3 | 0.094 (2) | 0.0522 (16) | 0.0395 (14) | −0.0121 (16) | −0.0093 (15) | −0.0031 (12) |
| C4 | 0.077 (2) | 0.0505 (15) | 0.0497 (15) | 0.0081 (16) | −0.0049 (14) | −0.0054 (13) |
| C5 | 0.0482 (15) | 0.0590 (16) | 0.0413 (14) | 0.0046 (13) | 0.0025 (12) | −0.0031 (12) |
| C6 | 0.0394 (13) | 0.0492 (14) | 0.0370 (12) | −0.0026 (11) | 0.0018 (11) | −0.0036 (10) |
| C8A | 0.035 (4) | 0.050 (4) | 0.048 (4) | 0.011 (4) | 0.005 (3) | 0.006 (3) |
| C9A | 0.046 (5) | 0.031 (3) | 0.043 (4) | −0.001 (3) | −0.004 (4) | 0.001 (3) |
| C8B | 0.036 (4) | 0.041 (3) | 0.036 (3) | 0.002 (3) | 0.003 (3) | −0.005 (2) |
| C9B | 0.035 (4) | 0.030 (3) | 0.041 (3) | 0.006 (2) | −0.001 (3) | 0.001 (3) |
| C10 | 0.0334 (13) | 0.0642 (17) | 0.0576 (16) | −0.0106 (12) | −0.0099 (11) | 0.0107 (14) |
| C11 | 0.0268 (11) | 0.0530 (16) | 0.0758 (18) | 0.0059 (11) | −0.0004 (12) | −0.0106 (13) |
| C12 | 0.0353 (13) | 0.0765 (19) | 0.0491 (14) | −0.0133 (13) | 0.0033 (11) | −0.0035 (15) |
| C13 | 0.0356 (12) | 0.0383 (12) | 0.0442 (12) | −0.0016 (10) | −0.0024 (10) | 0.0101 (10) |
| C14 | 0.0402 (13) | 0.0438 (13) | 0.0506 (14) | 0.0029 (11) | −0.0007 (12) | 0.0061 (12) |
| C15 | 0.0561 (16) | 0.0442 (14) | 0.0543 (15) | 0.0045 (13) | 0.0055 (13) | 0.0047 (13) |
| C16 | 0.0640 (17) | 0.0446 (13) | 0.0477 (14) | −0.0054 (13) | −0.0038 (13) | 0.0051 (13) |
| C17 | 0.0542 (16) | 0.0577 (16) | 0.0511 (14) | −0.0062 (13) | −0.0158 (13) | 0.0090 (13) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C18 | 0.0413 (14) | 0.0464 (14) | 0.0529 (14) | 0.0032 (11) | −0.0066 (11) | 0.0019 (11) |
| C19 | 0.092 (2) | 0.0543 (17) | 0.0613 (18) | −0.0067 (17) | −0.0096 (17) | −0.0056 (14) |
| C7 | 0.140 (4) | 0.063 (2) | 0.0591 (18) | −0.028 (2) | −0.021 (2) | 0.0131 (15) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—O1 | 1.4216 (19) | C8B—C9B | 1.522 (7) |
| S1—O2 | 1.4273 (18) | C8B—H8B1 | 0.9700 |
| S1—N1 | 1.614 (2) | C8B—H8B2 | 0.9700 |
| S1—C6 | 1.766 (2) | C9B—H9B1 | 0.9700 |
| S2—O4 | 1.4313 (17) | C9B—H9B2 | 0.9700 |
| S2—O3 | 1.4313 (16) | C10—C11 | 1.515 (4) |
| S2—N2 | 1.616 (2) | C10—H10A | 0.9700 |
| S2—C13 | 1.762 (2) | C10—H10B | 0.9700 |
| N1—C12 | 1.465 (3) | C11—C12 | 1.504 (4) |
| N1—C8A | 1.486 (6) | C11—H11A | 0.9700 |
| N1—C8B | 1.502 (5) | C11—H11B | 0.9700 |
| N2—C10 | 1.467 (3) | C12—H12A | 0.9700 |
| N2—C9A | 1.480 (7) | C12—H12B | 0.9700 |
| N2—C9B | 1.499 (6) | C13—C14 | 1.388 (3) |
| C1—C2 | 1.384 (4) | C13—C18 | 1.392 (3) |
| C1—C6 | 1.389 (4) | C14—C15 | 1.391 (4) |
| C1—H1 | 0.9300 | C14—H14 | 0.9300 |
| C2—C3 | 1.392 (4) | C15—C16 | 1.389 (4) |
| C2—H2 | 0.9300 | C15—H15 | 0.9300 |
| C3—C4 | 1.375 (4) | C16—C17 | 1.378 (4) |
| C3—C7 | 1.511 (4) | C16—C19 | 1.507 (4) |
| C4—C5 | 1.381 (4) | C17—C18 | 1.385 (4) |
| C4—H4 | 0.9300 | C17—H17 | 0.9300 |
| C5—C6 | 1.385 (3) | C18—H18 | 0.9300 |
| C5—H5 | 0.9300 | C19—H19A | 0.9600 |
| C8A—C9A | 1.516 (8) | C19—H19B | 0.9600 |
| C8A—H8A1 | 0.9700 | C19—H19C | 0.9600 |
| C8A—H8A2 | 0.9700 | C7—H7A | 0.9600 |
| C9A—H9A1 | 0.9700 | C7—H7B | 0.9600 |
| C9A—H9A2 | 0.9700 | C7—H7C | 0.9600 |
| | | | |
| O1—S1—O2 | 119.36 (13) | C9B—C8B—H8B2 | 109.5 |
| O1—S1—N1 | 107.66 (11) | H8B1—C8B—H8B2 | 108.0 |
| O2—S1—N1 | 106.80 (12) | N2—C9B—C8B | 109.9 (5) |
| O1—S1—C6 | 108.33 (12) | N2—C9B—H9B1 | 109.7 |
| O2—S1—C6 | 106.02 (12) | C8B—C9B—H9B1 | 109.7 |
| N1—S1—C6 | 108.25 (11) | N2—C9B—H9B2 | 109.7 |
| O4—S2—O3 | 119.05 (11) | C8B—C9B—H9B2 | 109.7 |
| O4—S2—N2 | 107.45 (11) | H9B1—C9B—H9B2 | 108.2 |
| O3—S2—N2 | 107.59 (10) | N2—C10—C11 | 111.7 (2) |
| O4—S2—C13 | 106.62 (11) | N2—C10—H10A | 109.3 |
| O3—S2—C13 | 107.95 (11) | C11—C10—H10A | 109.3 |
| N2—S2—C13 | 107.71 (10) | N2—C10—H10B | 109.3 |
| C12—N1—C8A | 128.8 (4) | C11—C10—H10B | 109.3 |

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| C12—N1—C8B | 104.7 (4) | H10A—C10—H10B | 107.9 |
| C12—N1—S1 | 119.77 (16) | C12—C11—C10 | 115.6 (2) |
| C8A—N1—S1 | 106.9 (3) | C12—C11—H11A | 108.4 |
| C8B—N1—S1 | 122.1 (3) | C10—C11—H11A | 108.4 |
| C10—N2—C9A | 104.8 (5) | C12—C11—H11B | 108.4 |
| C10—N2—C9B | 125.6 (3) | C10—C11—H11B | 108.4 |
| C10—N2—S2 | 119.14 (16) | H11A—C11—H11B | 107.4 |
| C9A—N2—S2 | 122.1 (5) | N1—C12—C11 | 112.0 (2) |
| C9B—N2—S2 | 109.5 (3) | N1—C12—H12A | 109.2 |
| C2—C1—C6 | 119.2 (3) | C11—C12—H12A | 109.2 |
| C2—C1—H1 | 120.4 | N1—C12—H12B | 109.2 |
| C6—C1—H1 | 120.4 | C11—C12—H12B | 109.2 |
| C1—C2—C3 | 121.3 (3) | H12A—C12—H12B | 107.9 |
| C1—C2—H2 | 119.3 | C14—C13—C18 | 120.2 (2) |
| C3—C2—H2 | 119.3 | C14—C13—S2 | 119.87 (18) |
| C4—C3—C2 | 118.4 (3) | C18—C13—S2 | 119.90 (19) |
| C4—C3—C7 | 120.9 (3) | C13—C14—C15 | 119.4 (2) |
| C2—C3—C7 | 120.8 (3) | C13—C14—H14 | 120.3 |
| C3—C4—C5 | 121.4 (3) | C15—C14—H14 | 120.3 |
| C3—C4—H4 | 119.3 | C16—C15—C14 | 121.1 (3) |
| C5—C4—H4 | 119.3 | C16—C15—H15 | 119.4 |
| C4—C5—C6 | 119.7 (3) | C14—C15—H15 | 119.4 |
| C4—C5—H5 | 120.1 | C17—C16—C15 | 118.3 (3) |
| C6—C5—H5 | 120.1 | C17—C16—C19 | 121.3 (3) |
| C5—C6—C1 | 120.0 (2) | C15—C16—C19 | 120.5 (3) |
| C5—C6—S1 | 119.9 (2) | C16—C17—C18 | 122.0 (3) |
| C1—C6—S1 | 120.1 (2) | C16—C17—H17 | 119.0 |
| N1—C8A—C9A | 110.6 (7) | C18—C17—H17 | 119.0 |
| N1—C8A—H8A1 | 109.5 | C17—C18—C13 | 119.0 (2) |
| C9A—C8A—H8A1 | 109.5 | C17—C18—H18 | 120.5 |
| N1—C8A—H8A2 | 109.5 | C13—C18—H18 | 120.5 |
| C9A—C8A—H8A2 | 109.5 | C16—C19—H19A | 109.5 |
| H8A1—C8A—H8A2 | 108.1 | C16—C19—H19B | 109.5 |
| N2—C9A—C8A | 112.6 (6) | H19A—C19—H19B | 109.5 |
| N2—C9A—H9A1 | 109.1 | C16—C19—H19C | 109.5 |
| C8A—C9A—H9A1 | 109.1 | H19A—C19—H19C | 109.5 |
| N2—C9A—H9A2 | 109.1 | H19B—C19—H19C | 109.5 |
| C8A—C9A—H9A2 | 109.1 | C3—C7—H7A | 109.5 |
| H9A1—C9A—H9A2 | 107.8 | C3—C7—H7B | 109.5 |
| N1—C8B—C9B | 110.9 (6) | H7A—C7—H7B | 109.5 |
| N1—C8B—H8B1 | 109.5 | C3—C7—H7C | 109.5 |
| C9B—C8B—H8B1 | 109.5 | H7A—C7—H7C | 109.5 |
| N1—C8B—H8B2 | 109.5 | H7B—C7—H7C | 109.5 |

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| O1—S1—N1—C12 | −34.0 (3) | S1—N1—C8A—C9A | −173.6 (8) |
| O2—S1—N1—C12 | −163.3 (2) | C10—N2—C9A—C8A | −98.8 (10) |
| C6—S1—N1—C12 | 82.9 (2) | C9B—N2—C9A—C8A | 60.8 (13) |
| O1—S1—N1—C8A | 167.7 (5) | S2—N2—C9A—C8A | 121.8 (9) |
| O2—S1—N1—C8A | 38.4 (5) | N1—C8A—C9A—N2 | 48.4 (15) |

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| C6—S1—N1—C8A | −75.4 (5) | C12—N1—C8B—C9B | 101.7 (7) |
| O1—S1—N1—C8B | −168.7 (4) | C8A—N1—C8B—C9B | −57.8 (9) |
| O2—S1—N1—C8B | 62.0 (4) | S1—N1—C8B—C9B | −118.0 (6) |
| C6—S1—N1—C8B | −51.8 (4) | C10—N2—C9B—C8B | −30.9 (10) |
| O4—S2—N2—C10 | 165.87 (18) | C9A—N2—C9B—C8B | −55.4 (14) |
| O3—S2—N2—C10 | 36.5 (2) | S2—N2—C9B—C8B | 176.4 (6) |
| C13—S2—N2—C10 | −79.6 (2) | N1—C8B—C9B—N2 | −51.2 (11) |
| O4—S2—N2—C9A | −60.1 (4) | C9A—N2—C10—C11 | 87.3 (4) |
| O3—S2—N2—C9A | 170.6 (4) | C9B—N2—C10—C11 | 77.9 (6) |
| C13—S2—N2—C9A | 54.4 (4) | S2—N2—C10—C11 | −131.8 (2) |
| O4—S2—N2—C9B | −39.4 (4) | N2—C10—C11—C12 | −65.3 (3) |
| O3—S2—N2—C9B | −168.8 (4) | C8A—N1—C12—C11 | −73.4 (6) |
| C13—S2—N2—C9B | 75.1 (4) | C8B—N1—C12—C11 | −84.8 (3) |
| C6—C1—C2—C3 | 0.5 (4) | S1—N1—C12—C11 | 133.74 (19) |
| C1—C2—C3—C4 | −1.8 (4) | C10—C11—C12—N1 | 62.6 (3) |
| C1—C2—C3—C7 | 177.3 (3) | O4—S2—C13—C14 | 20.1 (2) |
| C2—C3—C4—C5 | 1.3 (4) | O3—S2—C13—C14 | 149.10 (19) |
| C7—C3—C4—C5 | −177.7 (3) | N2—S2—C13—C14 | −95.0 (2) |
| C3—C4—C5—C6 | 0.4 (4) | O4—S2—C13—C18 | −162.33 (19) |
| C4—C5—C6—C1 | −1.7 (4) | O3—S2—C13—C18 | −33.3 (2) |
| C4—C5—C6—S1 | 178.10 (19) | N2—S2—C13—C18 | 82.6 (2) |
| C2—C1—C6—C5 | 1.2 (4) | C18—C13—C14—C15 | 1.9 (4) |
| C2—C1—C6—S1 | −178.58 (19) | S2—C13—C14—C15 | 179.45 (18) |
| O1—S1—C6—C5 | −143.5 (2) | C13—C14—C15—C16 | −0.5 (4) |
| O2—S1—C6—C5 | −14.2 (2) | C14—C15—C16—C17 | −1.3 (4) |
| N1—S1—C6—C5 | 100.1 (2) | C14—C15—C16—C19 | 176.9 (2) |
| O1—S1—C6—C1 | 36.3 (2) | C15—C16—C17—C18 | 1.6 (4) |
| O2—S1—C6—C1 | 165.6 (2) | C19—C16—C17—C18 | −176.5 (3) |
| N1—S1—C6—C1 | −80.1 (2) | C16—C17—C18—C13 | −0.3 (4) |
| C12—N1—C8A—C9A | 30.8 (12) | C14—C13—C18—C17 | −1.5 (4) |
| C8B—N1—C8A—C9A | 56.6 (10) | S2—C13—C18—C17 | −179.09 (19) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C10—H10*B*···O3 | 0.97 | 2.40 | 2.886 (3) | 110 |
| C12—H12*A*···O1 | 0.97 | 2.39 | 2.878 (3) | 111 |
| C10—H10*B*···O4$^{i}$ | 0.97 | 2.52 | 3.142 (3) | 122 |
| C12—H12*A*···O2$^{i}$ | 0.97 | 2.50 | 3.035 (3) | 115 |

Symmetry code: (i) *x*−1, *y*, *z*.