# organic compounds

# 4-Chloro-*N*-cyclohexylbenzene-sulfonamide

**Shahzad Sharif,[a] Shumaila Younas Mughal,[a] Islam Ullah Khan,[a] Mehmet Akkurt[b]\* and Muneeb Hayat Khan[c]**

[a]Materials Chemistry Laboratory, Department of Chemistry, Government College University, Lahore 54000, Pakistan, [b]Department of Physics, Faculty of Sciences, Erciyes University, 38039 Kayseri, Turkey, and [c]Materials Chemistry Laboratory, Department of Chemistry, Government College University, Lahore 54000, Pakistan, and, Pakistan and Punjab Forensic Science Agency, Thokar Niaz Baig, Lahore, Pakistan

Correspondence e-mail: akkurt@erciyes.edu.tr, iukhan@gcu.edu.pk

The title compound, $C_{12}H_{16}ClNO_2S$, adopts an L-shaped conformation, with the central C—S—N—C torsion angle being $-78.0$ (2)°. The cyclohexyl ring adopts a chair conformation. In the crystal, adjacent molecules are connected by pairs of N—H···O hydrogen bonds around an inversion centre, forming cyclic dimers [graph set $R_2^2(8)$].

## Related literature

For background to the pharmacological uses of sulfonamides, see: Korolkovas (1988); Mandell & Sande (1992). For related structures, see: Sharif *et al.* (2011); Khan *et al.* (2010); John *et al.* (2010). For ring conformational analysis, see: Cremer & Pople (1975).
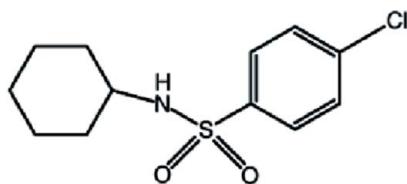


## Experimental

### Crystal data

| | |
|---|---|
| $C_{12}H_{16}ClNO_2S$ | $V = 1371.73$ (10) Å$^3$ |
| $M_r = 273.78$ | $Z = 4$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation |
| $a = 11.1226$ (5) Å | $\mu = 0.42$ mm$^{-1}$ |
| $b = 6.2490$ (2) Å | $T = 296$ K |
| $c = 19.8635$ (9) Å | $0.29 \times 0.15 \times 0.11$ mm |
| $\beta = 96.505$ (2)° | |

### Data collection

| | |
|---|---|
| Bruker APEXII CCD diffractometer | 3365 independent reflections |
| 12714 measured reflections | 2075 reflections with $I > 2\sigma(I)$ |
| | $R_{int} = 0.030$ |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)] = 0.053$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.150$ | |
| $S = 1.03$ | $\Delta\rho_{max} = 0.39$ e Å$^{-3}$ |
| 3365 reflections | $\Delta\rho_{min} = -0.25$ e Å$^{-3}$ |
| 157 parameters | |
| 1 restraint | |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1$N$···O1$^i$ | 0.85 (4) | 2.05 (4) | 2.891 (4) | 170 (3) |

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* for Windows (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

## References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
John, P., Anwar, F., Khan, I. U., Sharif, S. & Tiekink, E. R. T. (2010). *Acta Cryst.* E**66**, o1989.
Khan, I. U., Mariam, I., Zia-ur-Rehman, M., Arif Sajjad, M. & Sharif, S. (2010). *Acta Cryst.* E**66**, o1088.
Korolkovas, A. (1988). *Essentials of Medicinal Chemistry*, 2nd ed., pp. 699–716. New York: Wiley.
Mandell, G. L. & Sande, M. A. (1992). In *Goodman and Gilman, The Pharmacological Basis of Therapeutics 2*, edited by A. Gilman, T. W. Rall, A. S. Nies & P. Taylor, 8th ed., pp. 1047–1057. Singapore: McGraw–Hill; New York: Wiley.
Sharif, S., Khan, I. U., Mahmood, T. & Kang, S. K. (2011). *Acta Cryst.* E**67**, o1570.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

# supporting information

## 4-Chloro-*N*-cyclohexylbenzenesulfonamide

## Shahzad Sharif, Shumaila Younas Mughal, Islam Ullah Khan, Mehmet Akkurt and Muneeb Hayat Khan

### S1. Comment

Sulfonamide group containing drugs are extensively used for the treatment of certain infections caused by Gram-positive and Gram-negative microorganisms (Korolkovas, 1988; Mandell & Sande, 1992). In continuation of our on going structural studies of cyclohexylamine and sulfonamides synthesis (John *et al.*, 2010; Khan *et al.*, 2010; Sharif *et al.*, 2011), herein the crystal structure of title compound (I) is described.

In (I), (Fig. 1), the S atom has a distorted tetrahedral geometry within a $CNO_2$ donor set [maximum deviation: O—S—O = 119.45 (12)°]. The central C6–S1–N1–C7 torsion angle is -78.0 (2)°. The C7–C12 cyclohexyl ring adopts a chair conformation, with puckering parameters (Cremer & Pople, 1975) $Q$ = 0.536 (4) Å, $\theta$ = 180.0 (4) °, $\varphi$ = 196 (16) °.

In the crystal, two adjacent molecules are linked by a pair of N—H⋯O hydrogen bonds, forming an inversion dimer with an $R_2^2(8)$ ring motif (Table 1, Fig. 2).

### S2. Experimental

To 115 $\mu$l (1 mmol) of cyclohexylamine in 10 ml distilled water, was added 211 mg (1 mmol) of 4-chlorobenzenesulfonyl chloride while maintaining the pH of reaction mixture at 8 by using 3% sodium carbonate solution. Consumption of the reactants was confirmed by TLC. The pH of reaction mixture was adjusted by 3 N HCl at 3. Precipitates formed, washed with water and crystallized from methanol.

### S3. Refinement

The NH H–atom was located in a difference Fourier map and isotropically refined with a distance restraint: N—H = 0.86 (2) Å. C-bound H atoms were placed in calculated positions with C—H distances in the range 0.93–0.98 Å and and were refined using a riding model with $U_{iso}(H) = 1.2U_{eq}(C)$. In the final refinement two low angle reflections evidently effected by the beam stop were omitted, *i.e.* 0 0 2 and 1 0 0.
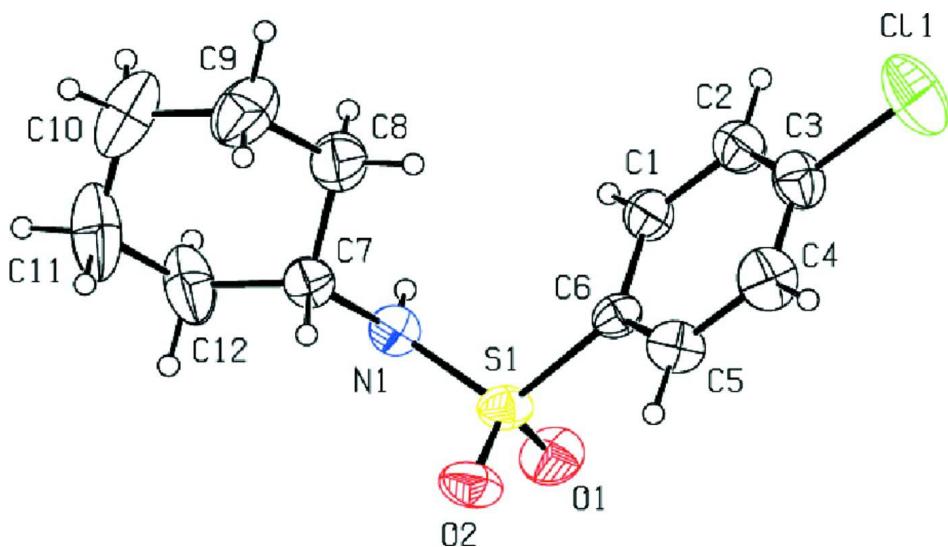
**Figure 1**

The title molecule, showing the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.
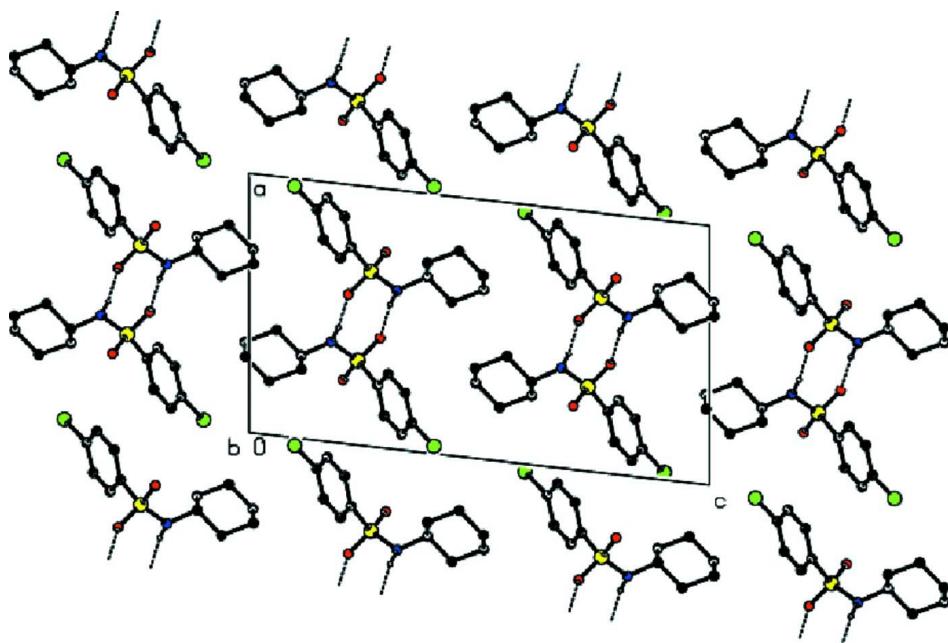


**Figure 2**

The crystal packing of (I) down *b* axis, showing the molecules are linked into dimers by pairs of N—H ⋯ O hydrogen bonds, forming $R_2^2(8)$ graph-set motif. Hydrogen atoms not involved in hydrogen bonding have been omitted for clarity.

**4-Chloro-*N*-cyclohexylbenzenesulfonamide**

*Crystal data*

| | |
|---|---|
| $C_{12}H_{16}ClNO_2S$ | Hall symbol: -P 2ybc |
| $M_r = 273.78$ | $a = 11.1226 (5)$ Å |
| Monoclinic, $P2_1/c$ | $b = 6.2490 (2)$ Å |

| | |
|---|---|
| $c = 19.8635$ (9) Å | Cell parameters from 3062 reflections |
| $\beta = 96.505$ (2)° | $\theta = 2.6–21.6°$ |
| $V = 1371.73$ (10) Å$^3$ | $\mu = 0.42$ mm$^{-1}$ |
| $Z = 4$ | $T = 296$ K |
| $F(000) = 576$ | Needle, light brown |
| $D_x = 1.326$ Mg m$^{-3}$ | $0.29 \times 0.15 \times 0.11$ mm |
| Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å | |

*Data collection*

| | |
|---|---|
| Bruker APEXII CCD diffractometer | 2075 reflections with $I > 2\sigma(I)$ |
| Radiation source: sealed tube | $R_{int} = 0.030$ |
| Graphite monochromator | $\theta_{max} = 28.3°$, $\theta_{min} = 3.4°$ |
| $\varphi$ and $\omega$ scans | $h = -14 \rightarrow 14$ |
| 12714 measured reflections | $k = -6 \rightarrow 8$ |
| 3365 independent reflections | $l = -26 \rightarrow 26$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.053$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.150$ | |
| $S = 1.03$ | H atoms treated by a mixture of independent and constrained refinement |
| 3365 reflections | |
| 157 parameters | $w = 1/[\sigma^2(F_o^2) + (0.0613P)^2 + 0.526P]$ |
| 1 restraint | where $P = (F_o^2 + 2F_c^2)/3$ |
| Primary atom site location: structure-invariant direct methods | $(\Delta/\sigma)_{max} < 0.001$ |
| | $\Delta\rho_{max} = 0.39$ e Å$^{-3}$ |
| | $\Delta\rho_{min} = -0.25$ e Å$^{-3}$ |

*Special details*

**Geometry**. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

**Refinement**. Refinement on $F^2$ for ALL reflections except those flagged by the user for potential systematic errors. Weighted $R$-factors $wR$ and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -$R$-factor-obs *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* | $U_{iso}*/U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.97047 (9) | 0.63719 (19) | 0.09885 (6) | 0.1159 (5) |
| S1 | 0.67445 (5) | 0.02579 (10) | 0.26455 (3) | 0.0538 (2) |
| O1 | 0.57744 (18) | −0.0412 (3) | 0.21563 (11) | 0.0788 (8) |
| O2 | 0.75531 (17) | −0.1301 (3) | 0.29647 (11) | 0.0710 (7) |
| N1 | 0.61305 (18) | 0.1518 (3) | 0.32146 (11) | 0.0527 (7) |
| C1 | 0.7127 (2) | 0.3981 (4) | 0.19816 (13) | 0.0581 (9) |
| C2 | 0.7777 (3) | 0.5312 (4) | 0.16141 (14) | 0.0650 (9) |
| C3 | 0.8901 (3) | 0.4698 (5) | 0.14686 (14) | 0.0654 (10) |
| C4 | 0.9396 (2) | 0.2788 (5) | 0.16961 (16) | 0.0733 (11) |
| C5 | 0.8750 (2) | 0.1454 (4) | 0.20712 (14) | 0.0606 (9) |

| | | | |
|---|---|---|---|
| C6 | 0.7611 (2) | 0.2037 (4) | 0.22098 (12) | 0.0465 (7) |
| C7 | 0.6789 (2) | 0.2145 (4) | 0.38711 (12) | 0.0541 (8) |
| C8 | 0.7329 (3) | 0.4325 (5) | 0.38666 (16) | 0.0829 (11) |
| C9 | 0.7924 (4) | 0.4979 (7) | 0.45609 (18) | 0.1069 (17) |
| C10 | 0.7084 (4) | 0.4802 (9) | 0.5084 (2) | 0.117 (2) |
| C11 | 0.6528 (5) | 0.2674 (10) | 0.50951 (18) | 0.135 (2) |
| C12 | 0.5920 (4) | 0.1970 (7) | 0.44002 (17) | 0.1033 (16) |
| H1 | 0.63600 | 0.43810 | 0.20780 | 0.0700* |
| H1N | 0.555 (3) | 0.234 (6) | 0.3060 (19) | 0.1390* |
| H2 | 0.74580 | 0.66260 | 0.14640 | 0.0780* |
| H4 | 1.01630 | 0.23970 | 0.15970 | 0.0880* |
| H5 | 0.90830 | 0.01600 | 0.22310 | 0.0730* |
| H7 | 0.74450 | 0.11150 | 0.39850 | 0.0650* |
| H8A | 0.67010 | 0.53500 | 0.37150 | 0.0990* |
| H8B | 0.79270 | 0.43550 | 0.35470 | 0.0990* |
| H9A | 0.86230 | 0.40740 | 0.46850 | 0.1280* |
| H9B | 0.82060 | 0.64450 | 0.45420 | 0.1280* |
| H10A | 0.75200 | 0.51000 | 0.55240 | 0.1410* |
| H10B | 0.64520 | 0.58700 | 0.49970 | 0.1410* |
| H11A | 0.59300 | 0.26840 | 0.54140 | 0.1620* |
| H11B | 0.71460 | 0.16380 | 0.52540 | 0.1620* |
| H12A | 0.56470 | 0.05020 | 0.44260 | 0.1240* |
| H12B | 0.52180 | 0.28630 | 0.42710 | 0.1240* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0867 (7) | 0.1367 (9) | 0.1293 (9) | −0.0197 (6) | 0.0339 (6) | 0.0505 (7) |
| S1 | 0.0456 (4) | 0.0438 (3) | 0.0725 (4) | −0.0069 (3) | 0.0087 (3) | −0.0065 (3) |
| O1 | 0.0634 (12) | 0.0815 (13) | 0.0904 (14) | −0.0310 (10) | 0.0034 (11) | −0.0211 (11) |
| O2 | 0.0691 (12) | 0.0436 (9) | 0.1030 (15) | 0.0083 (9) | 0.0210 (11) | 0.0082 (9) |
| N1 | 0.0393 (11) | 0.0562 (12) | 0.0629 (13) | 0.0041 (9) | 0.0071 (10) | 0.0035 (10) |
| C1 | 0.0540 (15) | 0.0589 (15) | 0.0637 (16) | 0.0058 (13) | 0.0169 (13) | 0.0006 (12) |
| C2 | 0.0704 (18) | 0.0594 (15) | 0.0664 (16) | 0.0034 (14) | 0.0132 (15) | 0.0056 (13) |
| C3 | 0.0553 (16) | 0.0765 (18) | 0.0646 (16) | −0.0132 (15) | 0.0076 (13) | 0.0050 (14) |
| C4 | 0.0398 (14) | 0.091 (2) | 0.090 (2) | −0.0036 (15) | 0.0118 (15) | 0.0011 (18) |
| C5 | 0.0418 (14) | 0.0602 (15) | 0.0793 (18) | 0.0021 (12) | 0.0048 (13) | 0.0025 (13) |
| C6 | 0.0410 (12) | 0.0461 (12) | 0.0514 (13) | −0.0033 (10) | 0.0015 (10) | −0.0093 (10) |
| C7 | 0.0463 (14) | 0.0565 (14) | 0.0597 (15) | 0.0112 (12) | 0.0063 (12) | 0.0059 (12) |
| C8 | 0.095 (2) | 0.082 (2) | 0.0727 (19) | −0.0204 (19) | 0.0134 (18) | −0.0077 (16) |
| C9 | 0.106 (3) | 0.119 (3) | 0.095 (3) | −0.014 (2) | 0.009 (2) | −0.038 (2) |
| C10 | 0.092 (3) | 0.178 (5) | 0.082 (2) | 0.019 (3) | 0.009 (2) | −0.049 (3) |
| C11 | 0.132 (4) | 0.216 (6) | 0.059 (2) | −0.014 (4) | 0.018 (2) | 0.022 (3) |
| C12 | 0.100 (3) | 0.141 (3) | 0.072 (2) | −0.027 (2) | 0.023 (2) | 0.021 (2) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1—C3 | 1.731 (4) | C10—C11 | 1.468 (8) |
| S1—O1 | 1.431 (2) | C11—C12 | 1.531 (6) |
| S1—O2 | 1.425 (2) | C1—H1 | 0.9300 |
| S1—N1 | 1.594 (2) | C2—H2 | 0.9300 |
| S1—C6 | 1.762 (3) | C4—H4 | 0.9300 |
| N1—C7 | 1.475 (3) | C5—H5 | 0.9300 |
| N1—H1N | 0.85 (4) | C7—H7 | 0.9800 |
| C1—C2 | 1.366 (4) | C8—H8A | 0.9700 |
| C1—C6 | 1.384 (4) | C8—H8B | 0.9700 |
| C2—C3 | 1.370 (5) | C9—H9A | 0.9700 |
| C3—C4 | 1.370 (4) | C9—H9B | 0.9700 |
| C4—C5 | 1.374 (4) | C10—H10A | 0.9700 |
| C5—C6 | 1.376 (3) | C10—H10B | 0.9700 |
| C7—C12 | 1.510 (5) | C11—H11A | 0.9700 |
| C7—C8 | 1.489 (4) | C11—H11B | 0.9700 |
| C8—C9 | 1.517 (5) | C12—H12A | 0.9700 |
| C9—C10 | 1.478 (6) | C12—H12B | 0.9700 |
| | | | |
| O1—S1—O2 | 119.45 (12) | C5—C4—H4 | 120.00 |
| O1—S1—N1 | 106.00 (12) | C4—C5—H5 | 120.00 |
| O1—S1—C6 | 105.28 (12) | C6—C5—H5 | 120.00 |
| O2—S1—N1 | 108.77 (12) | N1—C7—H7 | 108.00 |
| O2—S1—C6 | 107.29 (11) | C8—C7—H7 | 108.00 |
| N1—S1—C6 | 109.82 (11) | C12—C7—H7 | 108.00 |
| S1—N1—C7 | 123.29 (16) | C7—C8—H8A | 109.00 |
| S1—N1—H1N | 114 (3) | C7—C8—H8B | 109.00 |
| C7—N1—H1N | 116 (3) | C9—C8—H8A | 109.00 |
| C2—C1—C6 | 120.0 (2) | C9—C8—H8B | 109.00 |
| C1—C2—C3 | 119.6 (3) | H8A—C8—H8B | 108.00 |
| Cl1—C3—C2 | 119.2 (2) | C8—C9—H9A | 109.00 |
| C2—C3—C4 | 121.1 (3) | C8—C9—H9B | 109.00 |
| Cl1—C3—C4 | 119.7 (2) | C10—C9—H9A | 109.00 |
| C3—C4—C5 | 119.5 (2) | C10—C9—H9B | 109.00 |
| C4—C5—C6 | 119.9 (2) | H9A—C9—H9B | 108.00 |
| S1—C6—C1 | 120.05 (17) | C9—C10—H10A | 109.00 |
| S1—C6—C5 | 119.92 (19) | C9—C10—H10B | 109.00 |
| C1—C6—C5 | 120.0 (2) | C11—C10—H10A | 109.00 |
| N1—C7—C8 | 113.5 (2) | C11—C10—H10B | 109.00 |
| C8—C7—C12 | 111.2 (3) | H10A—C10—H10B | 108.00 |
| N1—C7—C12 | 107.7 (2) | C10—C11—H11A | 109.00 |
| C7—C8—C9 | 112.1 (3) | C10—C11—H11B | 109.00 |
| C8—C9—C10 | 111.9 (4) | C12—C11—H11A | 109.00 |
| C9—C10—C11 | 112.3 (4) | C12—C11—H11B | 109.00 |
| C10—C11—C12 | 113.0 (4) | H11A—C11—H11B | 108.00 |
| C7—C12—C11 | 110.8 (4) | C7—C12—H12A | 110.00 |
| C2—C1—H1 | 120.00 | C7—C12—H12B | 109.00 |

| | | | |
|---|---|---|---|
| C6—C1—H1 | 120.00 | C11—C12—H12A | 109.00 |
| C1—C2—H2 | 120.00 | C11—C12—H12B | 109.00 |
| C3—C2—H2 | 120.00 | H12A—C12—H12B | 108.00 |
| C3—C4—H4 | 120.00 | | |
| | | | |
| O1—S1—N1—C7 | 168.78 (18) | C1—C2—C3—Cl1 | 178.7 (2) |
| O2—S1—N1—C7 | 39.2 (2) | Cl1—C3—C4—C5 | −179.3 (2) |
| C6—S1—N1—C7 | −78.0 (2) | C2—C3—C4—C5 | 0.6 (5) |
| N1—S1—C6—C5 | 135.6 (2) | C3—C4—C5—C6 | 0.6 (4) |
| O2—S1—C6—C1 | −165.3 (2) | C4—C5—C6—S1 | 176.1 (2) |
| N1—S1—C6—C1 | −47.2 (2) | C4—C5—C6—C1 | −1.1 (4) |
| O1—S1—C6—C1 | 66.5 (2) | N1—C7—C8—C9 | 176.1 (3) |
| O2—S1—C6—C5 | 17.5 (2) | C12—C7—C8—C9 | 54.6 (4) |
| O1—S1—C6—C5 | −110.7 (2) | N1—C7—C12—C11 | −178.0 (3) |
| S1—N1—C7—C12 | −144.8 (2) | C8—C7—C12—C11 | −53.2 (4) |
| S1—N1—C7—C8 | 91.8 (2) | C7—C8—C9—C10 | −54.4 (4) |
| C6—C1—C2—C3 | 0.7 (4) | C8—C9—C10—C11 | 53.3 (5) |
| C2—C1—C6—S1 | −176.7 (2) | C9—C10—C11—C12 | −53.2 (6) |
| C2—C1—C6—C5 | 0.5 (4) | C10—C11—C12—C7 | 53.0 (5) |
| C1—C2—C3—C4 | −1.3 (4) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*N*···O1[i] | 0.85 (4) | 2.05 (4) | 2.891 (4) | 170 (3) |

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