

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

4-Chloro-*N*-(2,3-dichlorophenyl)-benzenesulfonamideK. Shakuntala,<sup>a</sup> Sabine Foro<sup>b</sup> and B. Thimme Gowda<sup>a\*</sup>

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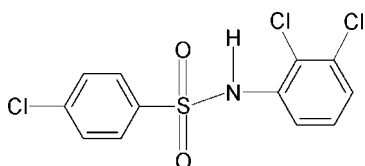
Received 21 December 2010; accepted 21 December 2010

Key indicators: single-crystal X-ray study;  $T = 299$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  $R$  factor = 0.043;  $wR$  factor = 0.137; data-to-parameter ratio = 14.3.

In the title compound,  $\text{C}_{12}\text{H}_8\text{Cl}_3\text{NO}_2\text{S}$ , the two aromatic rings are tilted relative to each other by  $56.5(1)^\circ$ . The crystal structure features centrosymmetric dimers in which molecules are linked by  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds.

## Related literature

For our study of the effect of substituents on the structures of *N*-(aryl)arylsulfonamides, see: Gowda *et al.* (2010); Nirmala *et al.* (2010); Shakuntala *et al.* (2010). For related structures, see: Gelbrich *et al.* (2007); Perlovich *et al.* (2006).



## Experimental

## Crystal data

 $\text{C}_{12}\text{H}_8\text{Cl}_3\text{NO}_2\text{S}$  $M_r = 336.60$ Monoclinic,  $P2_1/c$  $a = 7.224(1)$  Å $b = 14.975(2)$  Å $c = 13.170(2)$  Å $\beta = 97.16(1)^\circ$  $V = 1413.6(3)$  Å<sup>3</sup> $Z = 4$ Cu  $K\alpha$  radiation $\mu = 7.23$  mm<sup>-1</sup> $T = 299$  K $0.38 \times 0.30 \times 0.20$  mm

## Data collection

Enraf–Nonius CAD-4 diffractometer  
Absorption correction:  $\psi$  scan (North *et al.*, 1968)  
 $T_{\min} = 0.170$ ,  $T_{\max} = 0.326$   
2767 measured reflections

2516 independent reflections  
2277 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.062$   
3 standard reflections every 120 min  
intensity decay: 0.5%

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.043$  $wR(F^2) = 0.137$  $S = 1.15$ 

2516 reflections

176 parameters

1 restraint

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\max} = 0.46$  e Å<sup>-3</sup> $\Delta\rho_{\min} = -0.51$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^i$	0.86 (2)	2.11 (2)	2.944 (3)	163 (3)

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

Data collection: *CAD-4-PC* (Enraf–Nonius, 1996); cell refinement: *CAD-4-PC*; data reduction: *REDU4* (Stoe & Cie, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

KS thanks the University Grants Commission, Government of India, New Delhi, for the award of a research fellowship under its faculty improvement program.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5445).

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## supporting information

*Acta Cryst.* (2011). E67, o232 [https://doi.org/10.1107/S1600536810053638]

**4-Chloro-*N*-(2,3-dichlorophenyl)benzenesulfonamide**

**K. Shakuntala, Sabine Foro and B. Thimme Gowda**

**S1. Comment**

As part of a study of the substituent effects on the crystal structures of *N*-(aryl)-arylsulfonamides (Gowda *et al.*, 2010; Nirmala *et al.*, 2010; Shakuntala *et al.*, 2010), in the present work, the structure of 4-chloro-*N*-(2,3-dichlorophenyl)-benzenesulfonamide (I) has been determined. The conformation of the N—C bond in the C—SO<sub>2</sub>—NH—C segment of the structure has *gauche* torsions with respect to the S=O bonds (Fig. 1). The molecule is bent at the S atom with the C—SO<sub>2</sub>—NH—C torsion angle of -56.7 (2)°, compared to the values of 65.4 (2) and -61.7 (2) in the two molecules of *N*-(2,3-dichlorophenyl)-4-methylbenzenesulfonamide (II) (Shakuntala *et al.*, 2010). The conformations of the N—H bond and the *ortho*-chloro group in the anilino benzene ring are *syn* to each other.

The sulfonyl and the anilino benzene rings in (I) are tilted relative to each other by 56.5 (1)°, compared to the values of 76.0 (1)° (molecule 1) and 79.9 (1)° (molecule 2) in (II).

The other bond parameters in (I) are similar to those observed in (II), *N*-(2,3-dichlorophenyl)-2,4-dimethylbenzenesulfonamide (Nirmala *et al.*, 2010), *N*-(3,4-dimethylphenyl)-4-chlorobenzene-sulfonamide (Gowda *et al.*, 2010) and other aryl sulfonamides, (Perlovich *et al.*, 2006; Gelbrich *et al.*, 2007).

The structure shows N—H···O intermolecular H-bonding (Table 1). The crystal packing is shown in Fig. 2.

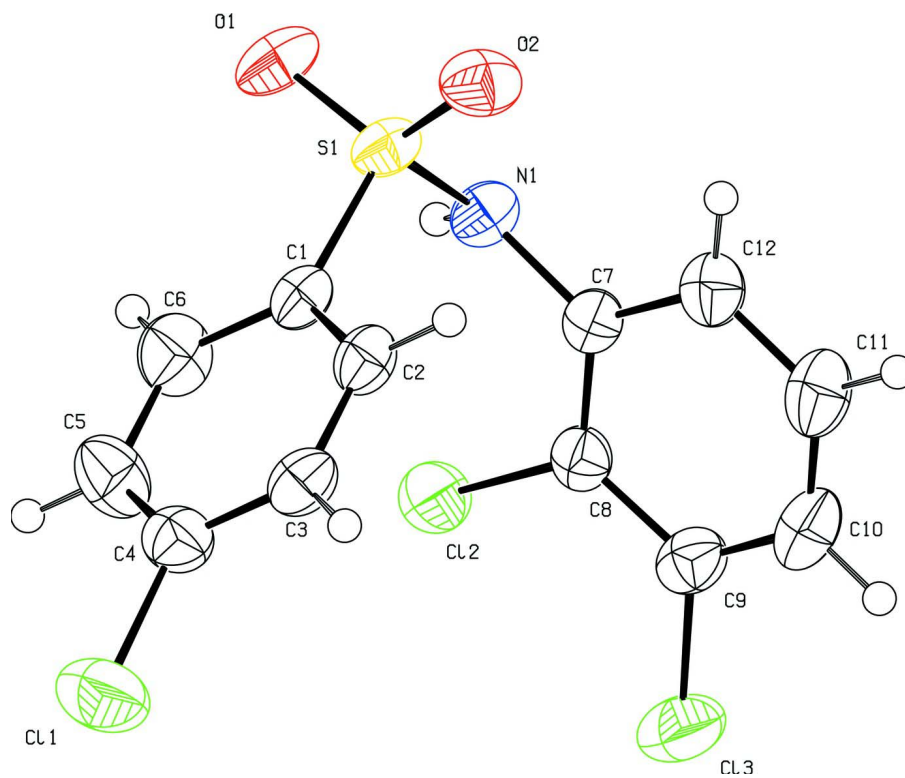
**S2. Experimental**

The solution of chlorobenzene (10 ml) in chloroform (40 ml) was treated dropwise with chlorosulfonic acid (25 ml) at 0 °C. After the initial evolution of hydrogen chloride subsided, the reaction mixture was brought to room temperature and poured into crushed ice in a beaker. The chloroform layer was separated, washed with cold water and allowed to evaporate slowly. The residual 4-chlorobenzenesulfonylchloride was treated with 2,3-dichloroaniline in the stoichiometric ratio and boiled for ten minutes. The reaction mixture was then cooled to room temperature and added to ice cold water (100 ml). The resultant 4-chloro-*N*-(2,3-dichlorophenyl)-benzenesulfonamide was filtered under suction and washed thoroughly with cold water. It was then recrystallized to constant melting point from dilute ethanol. The purity of the compound was checked and characterized by recording its infrared and NMR spectra.

Prism like colorless single crystals used in X-ray diffraction studies were grown in ethanolic solution by slow evaporation at room temperature.

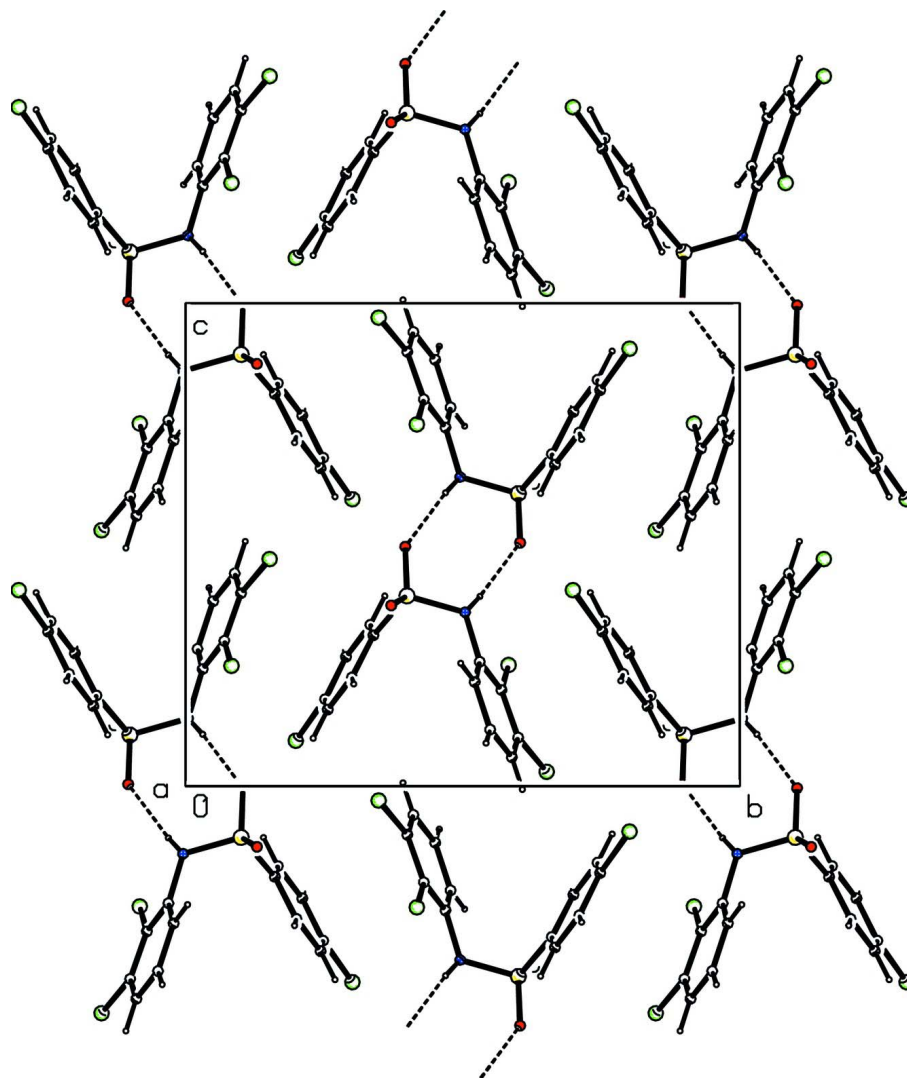
**S3. Refinement**

The H atom of the NH group was located in a difference and its coordinates were refined with the N—H distance restrained to 0.86 (2) Å. The other H atoms were positioned with idealized geometry using a riding model [C—H = 0.93 Å]. All H atoms were refined with isotropic displacement parameters set to 1.2 times of the  $U_{eq}$  of the parent atom.



**Figure 1**

Molecular structure of the title compound, showing the atom labelling scheme and displacement ellipsoids are drawn at the 50% probability level.



**Figure 2**

Molecular packing of the title compound with hydrogen bonding shown as dashed lines.

#### 4-Chloro-*N*-(2,3-dichlorophenyl)benzenesulfonamide

##### Crystal data

$C_{12}H_8Cl_3NO_2S$

$M_r = 336.60$

Monoclinic,  $P2_1/c$

Hall symbol:  $-P\ 2_1/c$

$a = 7.224\ (1)\ \text{\AA}$

$b = 14.975\ (2)\ \text{\AA}$

$c = 13.170\ (2)\ \text{\AA}$

$\beta = 97.16\ (1)^\circ$

$V = 1413.6\ (3)\ \text{\AA}^3$

$Z = 4$

$F(000) = 680$

$D_x = 1.582\ \text{Mg m}^{-3}$

Cu  $K\alpha$  radiation,  $\lambda = 1.54180\ \text{\AA}$

Cell parameters from 25 reflections

$\theta = 6.8\text{--}22.5^\circ$

$\mu = 7.23\ \text{mm}^{-1}$

$T = 299\ \text{K}$

Prism, colourless

$0.38 \times 0.30 \times 0.20\ \text{mm}$

*Data collection*

Enraf–Nonius CAD-4  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega/2\theta$  scans

Absorption correction:  $\psi$  scan  
(North *et al.*, 1968)

$T_{\min} = 0.170$ ,  $T_{\max} = 0.326$

2767 measured reflections

2516 independent reflections

2277 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.062$

$\theta_{\max} = 66.9^\circ$ ,  $\theta_{\min} = 4.5^\circ$

$h = -8 \rightarrow 8$

$k = -17 \rightarrow 0$

$l = -15 \rightarrow 1$

3 standard reflections every 120 min

intensity decay: 0.5%

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.137$

$S = 1.15$

2516 reflections

176 parameters

1 restraint

Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier  
map

Hydrogen site location: inferred from  
neighbouring sites

H atoms treated by a mixture of independent  
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0763P)^2 + 1.0223P]$

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

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

$\Delta\rho_{\max} = 0.46 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.51 \text{ e } \text{\AA}^{-3}$

Extinction correction: *SHELXL97* (Sheldrick,  
2008),  $F_c^* = kFc[1 + 0.001x \text{Fc}^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0074 (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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.5255 (4)	0.83900 (18)	0.1877 (2)	0.0332 (6)
C2	0.4733 (4)	0.80016 (19)	0.2749 (2)	0.0372 (6)
H2	0.3502	0.8034	0.2886	0.045*
C3	0.6057 (4)	0.7565 (2)	0.3415 (2)	0.0401 (7)
H3	0.5726	0.7303	0.4008	0.048*
C4	0.7869 (4)	0.7519 (2)	0.3199 (2)	0.0429 (7)
C5	0.8394 (5)	0.7896 (3)	0.2323 (3)	0.0551 (9)
H5	0.9622	0.7853	0.2183	0.066*
C6	0.7079 (5)	0.8336 (2)	0.1657 (3)	0.0504 (8)
H6	0.7412	0.8594	0.1064	0.060*
C7	0.3738 (4)	1.03085 (17)	0.2425 (2)	0.0326 (6)
C8	0.5279 (4)	1.06989 (18)	0.3008 (2)	0.0345 (6)
C9	0.5110 (5)	1.10001 (19)	0.3994 (2)	0.0416 (7)
C10	0.3474 (5)	1.0888 (2)	0.4403 (3)	0.0516 (8)

H10	0.3383	1.1076	0.5068	0.062*
C11	0.1954 (5)	1.0497 (2)	0.3829 (3)	0.0536 (8)
H11	0.0844	1.0422	0.4109	0.064*
C12	0.2082 (4)	1.0217 (2)	0.2843 (2)	0.0431 (7)
H12	0.1050	0.9964	0.2455	0.052*
N1	0.3873 (3)	1.00439 (16)	0.14018 (17)	0.0365 (6)
H1N	0.467 (4)	1.031 (2)	0.107 (2)	0.044*
O1	0.4094 (4)	0.89646 (15)	0.00411 (15)	0.0486 (6)
O2	0.1801 (3)	0.87121 (16)	0.12563 (17)	0.0469 (5)
C11	0.95260 (14)	0.69893 (7)	0.40587 (8)	0.0675 (3)
C12	0.73258 (11)	1.08276 (6)	0.24909 (6)	0.0517 (3)
C13	0.69792 (14)	1.15158 (6)	0.47020 (6)	0.0596 (3)
S1	0.35957 (10)	0.89965 (5)	0.10606 (5)	0.0353 (2)

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0408 (15)	0.0311 (13)	0.0289 (13)	−0.0055 (11)	0.0092 (11)	−0.0026 (10)
C2	0.0421 (15)	0.0385 (15)	0.0334 (14)	−0.0047 (12)	0.0137 (12)	−0.0011 (11)
C3	0.0514 (17)	0.0388 (15)	0.0309 (14)	−0.0032 (13)	0.0085 (12)	0.0011 (11)
C4	0.0419 (16)	0.0372 (15)	0.0479 (18)	−0.0019 (12)	−0.0004 (13)	−0.0040 (12)
C5	0.0352 (16)	0.064 (2)	0.067 (2)	−0.0019 (15)	0.0126 (15)	0.0076 (17)
C6	0.0483 (18)	0.059 (2)	0.0478 (18)	−0.0058 (15)	0.0201 (15)	0.0102 (15)
C7	0.0400 (14)	0.0281 (12)	0.0303 (13)	0.0033 (11)	0.0068 (11)	0.0040 (10)
C8	0.0387 (15)	0.0308 (13)	0.0343 (14)	0.0054 (11)	0.0055 (11)	0.0045 (11)
C9	0.0533 (18)	0.0326 (14)	0.0377 (16)	0.0021 (13)	0.0014 (13)	−0.0018 (11)
C10	0.066 (2)	0.0518 (19)	0.0397 (17)	0.0027 (16)	0.0189 (15)	−0.0070 (13)
C11	0.056 (2)	0.056 (2)	0.0528 (19)	−0.0010 (16)	0.0256 (16)	−0.0040 (15)
C12	0.0419 (16)	0.0428 (16)	0.0460 (17)	0.0003 (13)	0.0111 (13)	−0.0012 (13)
N1	0.0441 (13)	0.0362 (13)	0.0302 (12)	−0.0057 (10)	0.0082 (10)	0.0030 (9)
O1	0.0695 (15)	0.0514 (13)	0.0254 (10)	−0.0129 (11)	0.0077 (10)	−0.0030 (8)
O2	0.0413 (12)	0.0525 (13)	0.0460 (12)	−0.0151 (10)	0.0019 (9)	0.0011 (10)
C11	0.0594 (6)	0.0690 (6)	0.0686 (6)	0.0082 (4)	−0.0138 (4)	0.0057 (4)
C12	0.0380 (4)	0.0687 (6)	0.0490 (5)	−0.0056 (3)	0.0081 (3)	−0.0070 (3)
C13	0.0697 (6)	0.0598 (5)	0.0456 (5)	−0.0055 (4)	−0.0068 (4)	−0.0122 (3)
S1	0.0418 (4)	0.0381 (4)	0.0260 (4)	−0.0093 (3)	0.0045 (3)	−0.0011 (2)

*Geometric parameters (Å, °)*

C1—C2	1.381 (4)	C7—N1	1.420 (3)
C1—C6	1.386 (4)	C8—C9	1.394 (4)
C1—S1	1.760 (3)	C8—C12	1.714 (3)
C2—C3	1.379 (4)	C9—C10	1.369 (5)
C2—H2	0.9300	C9—C13	1.724 (3)
C3—C4	1.376 (4)	C10—C11	1.384 (5)
C3—H3	0.9300	C10—H10	0.9300
C4—C5	1.380 (5)	C11—C12	1.379 (4)
C4—C11	1.734 (3)	C11—H11	0.9300

C5—C6	1.378 (5)	C12—H12	0.9300
C5—H5	0.9300	N1—S1	1.637 (2)
C6—H6	0.9300	N1—H1N	0.858 (18)
C7—C12	1.384 (4)	O1—S1	1.434 (2)
C7—C8	1.399 (4)	O2—S1	1.418 (2)
C2—C1—C6	121.0 (3)	C7—C8—C12	119.7 (2)
C2—C1—S1	119.3 (2)	C10—C9—C8	120.4 (3)
C6—C1—S1	119.7 (2)	C10—C9—C13	119.9 (2)
C3—C2—C1	119.3 (3)	C8—C9—C13	119.7 (2)
C3—C2—H2	120.4	C9—C10—C11	120.1 (3)
C1—C2—H2	120.4	C9—C10—H10	119.9
C4—C3—C2	119.6 (3)	C11—C10—H10	119.9
C4—C3—H3	120.2	C12—C11—C10	120.1 (3)
C2—C3—H3	120.2	C12—C11—H11	120.0
C3—C4—C5	121.4 (3)	C10—C11—H11	120.0
C3—C4—C11	119.0 (2)	C11—C12—C7	120.5 (3)
C5—C4—C11	119.6 (2)	C11—C12—H12	119.8
C6—C5—C4	119.2 (3)	C7—C12—H12	119.8
C6—C5—H5	120.4	C7—N1—S1	120.50 (18)
C4—C5—H5	120.4	C7—N1—H1N	118 (2)
C5—C6—C1	119.5 (3)	S1—N1—H1N	112 (2)
C5—C6—H6	120.2	O2—S1—O1	120.13 (14)
C1—C6—H6	120.2	O2—S1—N1	108.73 (14)
C12—C7—C8	119.4 (3)	O1—S1—N1	104.63 (12)
C12—C7—N1	120.9 (3)	O2—S1—C1	107.60 (13)
C8—C7—N1	119.6 (2)	O1—S1—C1	108.90 (14)
C9—C8—C7	119.4 (3)	N1—S1—C1	106.03 (13)
C9—C8—C12	120.9 (2)		
C6—C1—C2—C3	0.9 (4)	C8—C9—C10—C11	-1.8 (5)
S1—C1—C2—C3	-176.7 (2)	C13—C9—C10—C11	178.3 (3)
C1—C2—C3—C4	-0.4 (4)	C9—C10—C11—C12	0.0 (5)
C2—C3—C4—C5	-0.5 (5)	C10—C11—C12—C7	1.3 (5)
C2—C3—C4—C11	178.6 (2)	C8—C7—C12—C11	-0.8 (4)
C3—C4—C5—C6	0.7 (5)	N1—C7—C12—C11	-178.4 (3)
C11—C4—C5—C6	-178.3 (3)	C12—C7—N1—S1	-63.5 (3)
C4—C5—C6—C1	-0.1 (6)	C8—C7—N1—S1	118.9 (2)
C2—C1—C6—C5	-0.7 (5)	C7—N1—S1—O2	58.7 (2)
S1—C1—C6—C5	177.0 (3)	C7—N1—S1—O1	-171.8 (2)
C12—C7—C8—C9	-0.9 (4)	C7—N1—S1—C1	-56.7 (2)
N1—C7—C8—C9	176.7 (2)	C2—C1—S1—O2	-20.9 (3)
C12—C7—C8—C12	-179.2 (2)	C6—C1—S1—O2	161.3 (2)
N1—C7—C8—C12	-1.6 (3)	C2—C1—S1—O1	-152.6 (2)
C7—C8—C9—C10	2.2 (4)	C6—C1—S1—O1	29.6 (3)
C12—C8—C9—C10	-179.5 (2)	C2—C1—S1—N1	95.3 (2)
C7—C8—C9—C13	-177.8 (2)	C6—C1—S1—N1	-82.5 (3)
C12—C8—C9—C13	0.4 (3)		

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*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>
N1—H1N···O1 <sup>i</sup>	0.86 (2)	2.11 (2)	2.944 (3)	163 (3)

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