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## Structure Reports

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## 4-Chloro-*N*-(2-chlorophenyl)-2-methylbenzenesulfonamide

 B. Thimme Gowda,<sup>a\*</sup> Sabine Foro,<sup>b</sup> P. G. Nirmala,<sup>a</sup>  
 K. S. Babitha<sup>a</sup> and Hartmut Fuess<sup>b</sup>
<sup>a</sup>Department of Chemistry, Mangalore University, Mangalagangothri 574 199, Mangalore, India, and <sup>b</sup>Institute of Materials Science, Darmstadt University of Technology, Petersenstrasse 23, D-64287 Darmstadt, Germany  
 Correspondence e-mail: gowdabt@yahoo.com

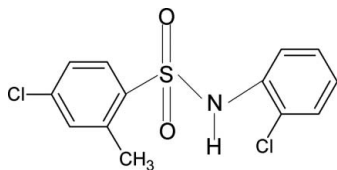
Received 28 February 2009; accepted 4 March 2009

 Key indicators: single-crystal X-ray study;  $T = 299$  K; mean  $\sigma(\text{C}-\text{C}) = 0.007$  Å;  $R$  factor = 0.083;  $wR$  factor = 0.236; data-to-parameter ratio = 13.6.

In the crystal structure of the title compound,  $\text{C}_{13}\text{H}_{11}\text{Cl}_2\text{NO}_2\text{S}$ , the conformations of the  $\text{N}-\text{C}$  bond in the  $\text{C}-\text{SO}_2-\text{NH}-\text{C}$  segment are *trans* and *gauche* with respect to the  $\text{S}=\text{O}$  bonds. The  $\text{C}-\text{S}(\text{O}_2)-\text{N}(\text{H})-\text{C}$  torsion angle is  $74.8(4)^\circ$ , indicating that the molecule is bent at the S atom. In the crystal structure, inversion dimers linked by pairs of  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds occur. An intramolecular  $\text{N}-\text{H}\cdots\text{Cl}$  interaction is also present.

### Related literature

For related structures of *N*-(aryl)-arylsulfonamides, see: Gelbrich *et al.* (2007); Gowda *et al.* (2009a,b); Perlovich *et al.* (2006).



### Experimental

#### Crystal data

 $\text{C}_{13}\text{H}_{11}\text{Cl}_2\text{NO}_2\text{S}$   
 $M_r = 316.19$   
 Triclinic,  $P\bar{1}$ 
 $a = 8.089(2)$  Å  
 $b = 8.096(2)$  Å  
 $c = 10.946(3)$  Å

 $\alpha = 96.00(1)^\circ$   
 $\beta = 97.11(2)^\circ$   
 $\gamma = 105.67(2)^\circ$   
 $V = 677.7(3)$  Å<sup>3</sup>  
 $Z = 2$ 

 Cu  $K\alpha$  radiation  
 $\mu = 5.73$  mm<sup>-1</sup>  
 $T = 299$  K  
 $0.45 \times 0.33 \times 0.08$  mm

#### Data collection

 Enraf–Nonius CAD-4  
 diffractometer  
 Absorption correction:  $\psi$  scan  
 (North *et al.*, 1968)  
 $T_{\min} = 0.150$ ,  $T_{\max} = 0.640$   
 2684 measured reflections

 2414 independent reflections  
 1932 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.037$   
 3 standard reflections  
 frequency: 120 min  
 intensity decay: 1.0%

#### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.083$   
 $wR(F^2) = 0.236$   
 $S = 1.05$   
 2414 reflections  
 177 parameters

 H atoms treated by a mixture of  
 independent and constrained  
 refinement  
 $\Delta\rho_{\max} = 0.60$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.67$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O2}^i$	0.88 (5)	2.17 (5)	2.994 (5)	157 (4)
$\text{N1}-\text{H1N}\cdots\text{Cl2}$	0.88 (5)	2.67 (5)	3.011 (4)	104 (4)

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

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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2384).

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## supporting information

*Acta Cryst.* (2009). E65, o717 [doi:10.1107/S1600536809007880]

## 4-Chloro-*N*-(2-chlorophenyl)-2-methylbenzenesulfonamide

B. Thimme Gowda, Sabine Foro, P. G. Nirmala, K. S. Babitha and Hartmut Fuess

### S1. Comment

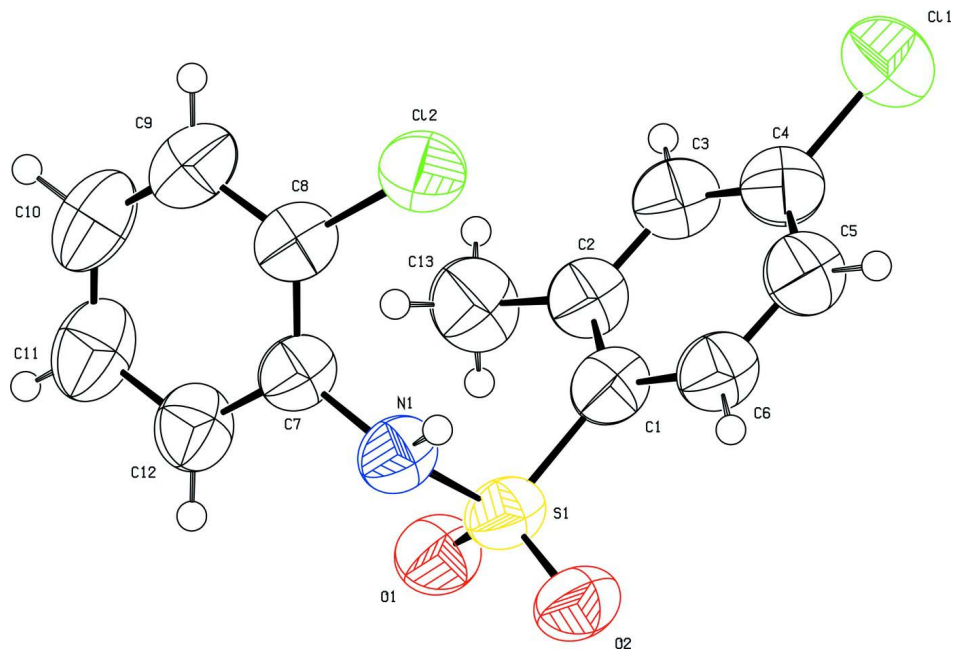
In the present work, as part of a study of substituent effects on the structures of *N*-(aryl)-arylsulfonamides (Gowda *et al.* 2008*a,b*, 2009), the structure of 2-methyl-4-chloro-*N*-(2-chlorophenyl)benzenesulfonamide (I) has been determined. The conformations of the N—C bond in the C—S(O<sub>2</sub>)—N(H)—C segment are *trans* and *gauche* with respect to the S=O bonds (Fig. 1). The torsion angle of C1—S1—N1—C1 is 74.8 (4)°, indicating the molecule is bent at the S1 atom. The two benzene rings are tilted relative to each other by 45.5 (2)°, compared with the values of 86.6 (2)° (molecule 1) and 83.0 (2)° (molecule 2), in the two independent molecules of 2-methyl-4-chloro-*N*-(phenyl)-benzenesulfonamide (II) (Gowda *et al.*, 2009). Bond distance parameters in (I) are similar to those observed in (II), 2,4-dimethyl-*N*-(phenyl)-benzenesulfonamide (Gowda *et al.*, 2008*b*) and other aryl sulfonamides (Perlovich *et al.*, 2006; Gelbrich *et al.*, 2007). The crystal structure comprises the packing of centrosymmetric molecules connected via N—H···O hydrogen bonds (Table 1 & Fig. 2).

### S2. Experimental

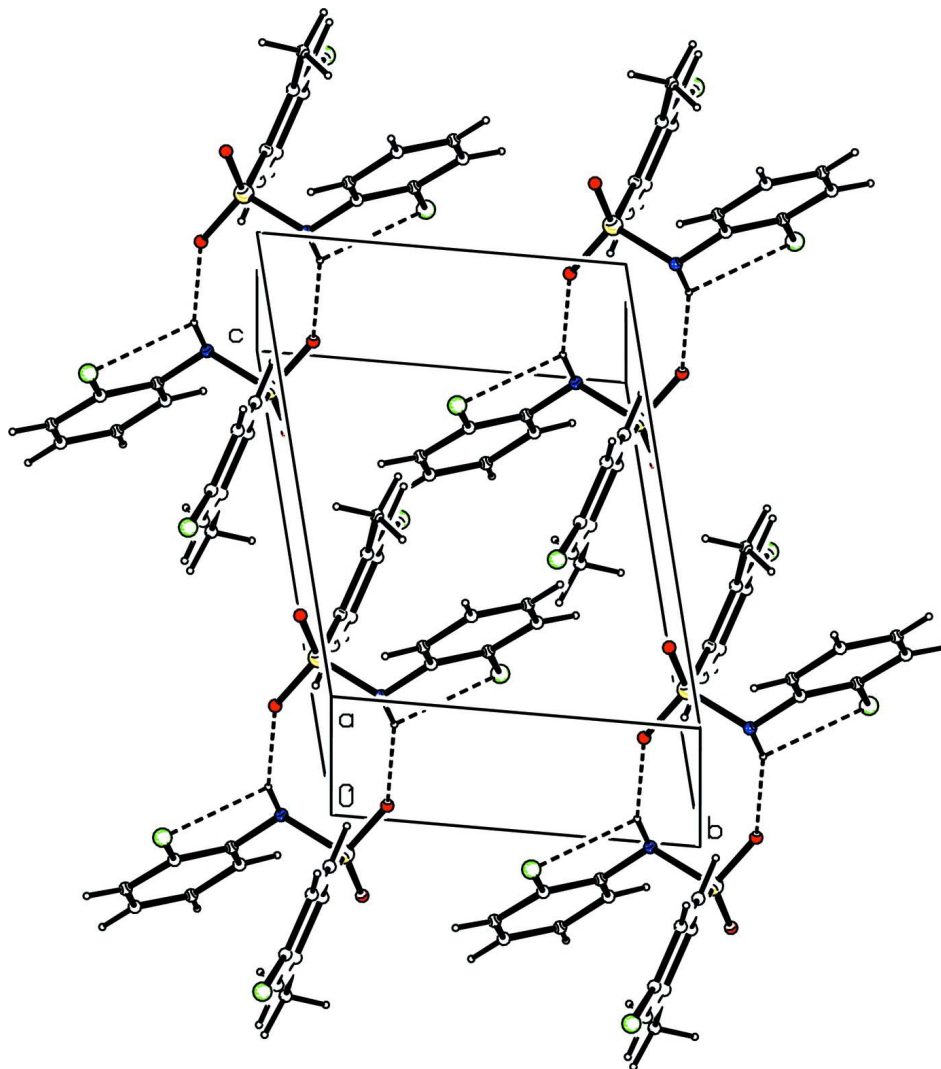
*m*-Chlorotoluene (10 ml) in chloroform (40 ml) was treated dropwise with chlorosulfonic acid (25 ml) at 0° C. After the initial evolution of HCl subsided, the reaction mixture was brought to room temperature and poured into crushed ice in a beaker. The chloroform layer was separated, washed with cold water and allowed to evaporate slowly. The residual 2-methyl-4-chlorobenzesulfonylchloride was treated with 2-chloroaniline in a 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 solid 2-methyl-4-chloro-*N*-(2-chlorophenyl)-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. The single crystals used in X-ray diffraction studies were grown from an ethanolic solution by slow evaporation at room temperature.

### S3. Refinement

The N-bound H atom was located in difference map and its positional parameters refined; N—H = 0.88 (5) Å. The remaining H atoms were positioned with idealized geometries using a riding model with C—H = 0.93–0.96 Å. All H atoms were refined with isotropic displacement parameters set to 1.2 ×  $U_{eq}$ (parent atom).

**Figure 1**

Molecular structure of (I), showing the atom labeling scheme. The displacement ellipsoids are drawn at the 50% probability level. The H atoms are represented as small spheres of arbitrary radii.



**Figure 2**

Molecular packing of (I) with hydrogen bonding shown as dashed lines.

#### 4-Chloro-*N*-(2-chlorophenyl)-2-methylbenzenesulfonamide

##### *Crystal data*

$C_{13}H_{11}Cl_2NO_2S$

$M_r = 316.19$

Triclinic,  $P\bar{1}$

Hall symbol: -P 1

$a = 8.089$  (2) Å

$b = 8.096$  (2) Å

$c = 10.946$  (3) Å

$\alpha = 96.00$  (1)°

$\beta = 97.11$  (2)°

$\gamma = 105.67$  (2)°

$V = 677.7$  (3) Å<sup>3</sup>

$Z = 2$

$F(000) = 324$

$D_x = 1.549$  Mg m<sup>-3</sup>

Cu  $K\alpha$  radiation,  $\lambda = 1.54180$  Å

Cell parameters from 25 reflections

$\theta = 5.7\text{--}19.7^\circ$

$\mu = 5.73$  mm<sup>-1</sup>

$T = 299$  K

Plate, colourless

$0.45 \times 0.33 \times 0.08$  mm

*Data collection*

Enraf–Nonius CAD-4  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega$  scans

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

$T_{\min} = 0.150$ ,  $T_{\max} = 0.640$

2684 measured reflections

2414 independent reflections

1932 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.037$

$\theta_{\max} = 66.9^\circ$ ,  $\theta_{\min} = 4.1^\circ$

$h = -9 \rightarrow 1$

$k = -9 \rightarrow 9$

$l = -12 \rightarrow 13$

3 standard reflections every 120 min

intensity decay: 1.0%

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.083$

$wR(F^2) = 0.236$

$S = 1.05$

2414 reflections

177 parameters

0 restraints

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

$w = 1/[\sigma^2(F_o^2) + (0.179P)^2 + 0.1079P]$

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

$(\Delta/\sigma)_{\max} = 0.008$

$\Delta\rho_{\max} = 0.60 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.67 \text{ e } \text{\AA}^{-3}$

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

Extinction coefficient: 0.039 (6)

*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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.35954 (18)	0.30027 (19)	0.56480 (12)	0.0932 (5)
Cl2	0.70514 (17)	0.48939 (15)	0.15780 (13)	0.0901 (5)
S1	0.71199 (12)	-0.00409 (12)	0.15693 (9)	0.0651 (4)
O1	0.8667 (4)	-0.0424 (4)	0.2054 (3)	0.0761 (8)
O2	0.5732 (4)	-0.1359 (4)	0.0795 (3)	0.0757 (8)
N1	0.7673 (4)	0.1494 (4)	0.0701 (3)	0.0659 (9)
H1N	0.678 (7)	0.178 (6)	0.034 (5)	0.079*
C1	0.6225 (5)	0.0807 (5)	0.2792 (4)	0.0637 (9)
C2	0.7215 (5)	0.1757 (5)	0.3907 (4)	0.0660 (10)
C3	0.6344 (6)	0.2423 (6)	0.4761 (4)	0.0719 (11)
H3	0.6974	0.3069	0.5507	0.086*
C4	0.4585 (6)	0.2158 (6)	0.4536 (4)	0.0708 (10)
C5	0.3607 (6)	0.1217 (6)	0.3426 (4)	0.0732 (11)
H5	0.2410	0.1036	0.3271	0.088*

C6	0.4441 (5)	0.0565 (5)	0.2569 (4)	0.0686 (10)
H6	0.3800	-0.0057	0.1818	0.082*
C7	0.9177 (5)	0.2931 (5)	0.1116 (4)	0.0625 (9)
C8	0.9064 (6)	0.4560 (5)	0.1521 (4)	0.0693 (10)
C9	1.0553 (7)	0.5950 (6)	0.1862 (4)	0.0812 (12)
H9	1.0466	0.7053	0.2114	0.097*
C10	1.2140 (6)	0.5692 (7)	0.1826 (5)	0.0900 (15)
H10	1.3139	0.6619	0.2069	0.108*
C11	1.2280 (6)	0.4087 (7)	0.1436 (5)	0.0900 (15)
H11	1.3372	0.3921	0.1427	0.108*
C12	1.0796 (6)	0.2698 (6)	0.1052 (5)	0.0760 (11)
H12	1.0891	0.1615	0.0753	0.091*
C13	0.9170 (6)	0.2092 (8)	0.4254 (4)	0.0875 (14)
H13A	0.9430	0.1011	0.4307	0.105*
H13B	0.9567	0.2823	0.5044	0.105*
H13C	0.9747	0.2655	0.3630	0.105*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C11	0.0896 (9)	0.1076 (10)	0.0887 (9)	0.0410 (7)	0.0150 (6)	0.0072 (7)
C12	0.0869 (8)	0.0730 (8)	0.1186 (10)	0.0325 (6)	0.0255 (7)	0.0141 (6)
S1	0.0608 (6)	0.0547 (6)	0.0758 (7)	0.0165 (4)	-0.0011 (4)	0.0066 (4)
O1	0.0695 (18)	0.0690 (17)	0.093 (2)	0.0285 (14)	0.0023 (15)	0.0136 (14)
O2	0.0690 (17)	0.0601 (16)	0.0872 (19)	0.0119 (13)	-0.0024 (14)	-0.0020 (13)
N1	0.0591 (18)	0.0635 (19)	0.0692 (19)	0.0144 (14)	-0.0032 (14)	0.0074 (14)
C1	0.058 (2)	0.060 (2)	0.073 (2)	0.0185 (16)	-0.0008 (17)	0.0152 (17)
C2	0.061 (2)	0.071 (2)	0.065 (2)	0.0205 (17)	-0.0021 (17)	0.0108 (17)
C3	0.072 (2)	0.073 (2)	0.065 (2)	0.0210 (19)	-0.0043 (18)	0.0072 (18)
C4	0.075 (2)	0.070 (2)	0.071 (2)	0.0278 (19)	0.0071 (19)	0.0153 (18)
C5	0.062 (2)	0.079 (3)	0.079 (3)	0.0260 (19)	0.0022 (19)	0.013 (2)
C6	0.061 (2)	0.067 (2)	0.072 (2)	0.0168 (18)	-0.0026 (18)	0.0084 (17)
C7	0.058 (2)	0.062 (2)	0.063 (2)	0.0149 (16)	-0.0013 (15)	0.0093 (15)
C8	0.072 (2)	0.065 (2)	0.068 (2)	0.0178 (18)	0.0016 (18)	0.0123 (17)
C9	0.087 (3)	0.067 (2)	0.077 (3)	0.006 (2)	0.000 (2)	0.0105 (19)
C10	0.070 (3)	0.086 (3)	0.095 (3)	-0.005 (2)	-0.011 (2)	0.025 (2)
C11	0.061 (2)	0.094 (3)	0.109 (4)	0.014 (2)	-0.005 (2)	0.032 (3)
C12	0.062 (2)	0.080 (3)	0.089 (3)	0.023 (2)	0.008 (2)	0.022 (2)
C13	0.062 (3)	0.114 (4)	0.075 (3)	0.022 (2)	-0.008 (2)	-0.003 (2)

*Geometric parameters (Å, °)*

C11—C4	1.719 (5)	C5—H5	0.9300
C12—C8	1.728 (5)	C6—H6	0.9300
S1—O1	1.423 (3)	C7—C8	1.378 (6)
S1—O2	1.430 (3)	C7—C12	1.381 (6)
S1—N1	1.643 (4)	C8—C9	1.388 (6)
S1—C1	1.761 (4)	C9—C10	1.360 (8)

N1—C7	1.424 (5)	C9—H9	0.9300
N1—H1N	0.88 (5)	C10—C11	1.364 (8)
C1—C6	1.390 (5)	C10—H10	0.9300
C1—C2	1.398 (5)	C11—C12	1.389 (6)
C2—C3	1.387 (7)	C11—H11	0.9300
C2—C13	1.522 (6)	C12—H12	0.9300
C3—C4	1.366 (6)	C13—H13A	0.9600
C3—H3	0.9300	C13—H13B	0.9600
C4—C5	1.388 (6)	C13—H13C	0.9600
C5—C6	1.364 (7)		
O1—S1—O2	120.08 (19)	C1—C6—H6	119.2
O1—S1—N1	107.27 (18)	C8—C7—C12	119.1 (4)
O2—S1—N1	104.75 (18)	C8—C7—N1	122.1 (4)
O1—S1—C1	109.99 (19)	C12—C7—N1	118.8 (4)
O2—S1—C1	106.96 (19)	C7—C8—C9	120.6 (4)
N1—S1—C1	107.03 (18)	C7—C8—C12	120.0 (3)
C7—N1—S1	120.4 (3)	C9—C8—C12	119.4 (4)
C7—N1—H1N	114 (3)	C10—C9—C8	119.6 (5)
S1—N1—H1N	113 (3)	C10—C9—H9	120.2
C6—C1—C2	120.1 (4)	C8—C9—H9	120.2
C6—C1—S1	116.1 (3)	C9—C10—C11	120.6 (4)
C2—C1—S1	123.7 (3)	C9—C10—H10	119.7
C3—C2—C1	117.4 (4)	C11—C10—H10	119.7
C3—C2—C13	118.0 (4)	C10—C11—C12	120.2 (5)
C1—C2—C13	124.6 (4)	C10—C11—H11	119.9
C4—C3—C2	121.9 (4)	C12—C11—H11	119.9
C4—C3—H3	119.0	C7—C12—C11	119.8 (5)
C2—C3—H3	119.0	C7—C12—H12	120.1
C3—C4—C5	120.6 (4)	C11—C12—H12	120.1
C3—C4—C11	119.2 (4)	C2—C13—H13A	109.5
C5—C4—C11	120.3 (4)	C2—C13—H13B	109.5
C6—C5—C4	118.5 (4)	H13A—C13—H13B	109.5
C6—C5—H5	120.8	C2—C13—H13C	109.5
C4—C5—H5	120.8	H13A—C13—H13C	109.5
C5—C6—C1	121.6 (4)	H13B—C13—H13C	109.5
C5—C6—H6	119.2		
O1—S1—N1—C7	-43.2 (4)	C11—C4—C5—C6	-179.9 (3)
O2—S1—N1—C7	-171.9 (3)	C4—C5—C6—C1	0.7 (6)
C1—S1—N1—C7	74.8 (4)	C2—C1—C6—C5	-0.9 (6)
O1—S1—C1—C6	-153.8 (3)	S1—C1—C6—C5	-177.6 (3)
O2—S1—C1—C6	-21.8 (3)	S1—N1—C7—C8	-106.8 (4)
N1—S1—C1—C6	90.0 (3)	S1—N1—C7—C12	76.4 (5)
O1—S1—C1—C2	29.7 (4)	C12—C7—C8—C9	-0.1 (6)
O2—S1—C1—C2	161.6 (3)	N1—C7—C8—C9	-176.9 (4)
N1—S1—C1—C2	-86.6 (4)	C12—C7—C8—C12	178.6 (3)
C6—C1—C2—C3	0.2 (6)	N1—C7—C8—C12	1.9 (6)

S1—C1—C2—C3	176.7 (3)	C7—C8—C9—C10	-1.7 (7)
C6—C1—C2—C13	179.5 (4)	C12—C8—C9—C10	179.6 (4)
S1—C1—C2—C13	-4.0 (6)	C8—C9—C10—C11	1.2 (8)
C1—C2—C3—C4	0.6 (6)	C9—C10—C11—C12	1.0 (8)
C13—C2—C3—C4	-178.8 (4)	C8—C7—C12—C11	2.3 (6)
C2—C3—C4—C5	-0.7 (7)	N1—C7—C12—C11	179.2 (4)
C2—C3—C4—C11	179.2 (3)	C10—C11—C12—C7	-2.7 (7)
C3—C4—C5—C6	0.1 (6)		

*Hydrogen-bond geometry (Å, °)*

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N1—H1N...O2 <sup>i</sup>	0.88 (5)	2.17 (5)	2.994 (5)	157 (4)
N1—H1N...Cl2	0.88 (5)	2.67 (5)	3.011 (4)	104 (4)

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