

## 2,4-Dichloro-*N*-(3,4-dimethylphenyl)-benzenesulfonamide

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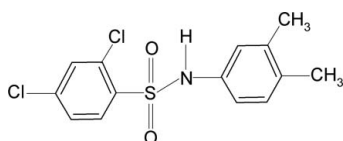
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Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.038;  $wR$  factor = 0.105; data-to-parameter ratio = 17.0.

In the title compound,  $\text{C}_{14}\text{H}_{13}\text{Cl}_2\text{NO}_2\text{S}$ , the  $\text{C}-\text{SO}_2-\text{NH}-\text{C}$  torsion angle is  $-60.84$  (18). The sulfonyl and the aniline benzene rings are tilted relative to each other by  $66.4$  (1)°. The crystal structure features inversion-related dimers linked by pairs of  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds.

### Related literature

For the preparation of the title compound, see: Savitha & Gowda (2006). For hydrogen-bonding modes of sulfonamides, see: Adsmund & Grant (2001). For our studies on the effects of substituents on the structures and other aspects of *N*-(aryl)-amides, see: Arjunan *et al.* (2004); Gowda *et al.* (2000), on *N*-(aryl)-methanesulfonamides, see: Gowda *et al.* (2007) and on *N*-(aryl)-arylsulfonamides, see: Gelbrich *et al.* (2007); Perlovich *et al.* (2006); Gowda *et al.* (2005); Rodrigues *et al.* (2011).



### Experimental

#### Crystal data

$\text{C}_{14}\text{H}_{13}\text{Cl}_2\text{NO}_2\text{S}$   
 $M_r = 330.21$   
Monoclinic,  $P2_1/c$   
 $a = 7.8381$  (6) Å  
 $b = 14.778$  (1) Å  
 $c = 13.660$  (1) Å  
 $\beta = 101.840$  (9)°

$V = 1548.59$  (19) Å<sup>3</sup>  
 $Z = 4$   
Mo  $K\alpha$  radiation  
 $\mu = 0.55$  mm<sup>-1</sup>  
 $T = 293$  K  
 $0.46 \times 0.44 \times 0.32$  mm

#### Data collection

Oxford Diffraction Xcalibur diffractometer with Sapphire CCD detector  
Absorption correction: multi-scan (*CrysAlis RED*; Oxford)

Diffraction, 2009)  
 $T_{\min} = 0.785$ ,  $T_{\max} = 0.843$   
6193 measured reflections  
3155 independent reflections  
2447 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.011$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$   
 $wR(F^2) = 0.105$   
 $S = 1.03$   
3155 reflections  
186 parameters  
1 restraint

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\max} = 0.36$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.40$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^1$	0.83 (2)	2.17 (2)	2.945 (2)	154 (2)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5644).

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**supplementary materials**

*Acta Cryst.* (2011). E67, o2712 [ doi:10.1107/S1600536811038256 ]

## 2,4-Dichloro-*N*-(3,4-dimethylphenyl)benzenesulfonamide

V. Z. Rodrigues, S. Foro and B. T. Gowda

### Comment

Several biologically important compounds contain the sulfonamide moiety. The hydrogen bonding preferences of sulfonamides have been investigated (Adsmond & Grant, 2001). As part of our studies on the substituent effects on the structures and other aspects of *N*-(aryl)-amides (Arjunan *et al.*, 2004; Gowda *et al.*, 2000), *N*-(aryl)-methanesulfonamides (Gowda *et al.*, 2007) and *N*-(aryl)-arylsulfonamides (Gowda *et al.*, 2005; Rodrigues *et al.*, 2011), in the present work, the crystal structure of 2,4-dichloro-*N*-(3,4-dimethylphenyl)-benzenesulfonamide (I) has been determined (Fig. 1).

In (I), the N—H bond in the C—SO<sub>2</sub>—NH—C segment is *syn* to the *meta*-methyl group in the anilino benzene ring and orients itself towards the *ortho*-chloro group in the sulfonyl benzene ring. Further, the conformations of the N—C bonds in the C—SO<sub>2</sub>—NH—C segment have *gauche* torsions with respect to the S=O bonds.

The molecule is bent at the S atom with C—SO<sub>2</sub>—NH—C torsion angle of -60.84 (18), compared to the value of -71.38 (39)° in 2,4-dichloro-*N*-(2,4-dimethylphenyl)-benzenesulfonamide (II) (Rodrigues *et al.*, 2011).

The sulfonyl and the anilino benzene rings are tilted relative to each other by 66.4 (1)°, compared to the value of 44.6 (1)° in (II)

The other bond parameters in (I) are similar to those observed in (II) and other aryl sulfonamides (Perlovich *et al.*, 2006; Gelbrich *et al.*, 2007).

In the crystal structure, the pairs of intermolecular N—H...O hydrogen bonds (Table 1) link the molecules into inversion-related dimers. Part of the crystal structure is shown in Fig. 2.

### Experimental

The solution of 1,3-dichlorobenzene (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 2,4-dichlorobenzenesulfonylchloride was treated with 3,4-dimethylaniline 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 solid 2,4-dichloro-*N*-(3,4-dimethylphenyl)-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 (Savitha & Gowda, 2006).

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

## Refinement

The H atom of the NH group was located in a difference map and later restrained to N—H = 0.86 (2) %Å. The other H atoms were positioned with idealized geometry using a riding model with the aromatic C—H = 0.93 Å and methyl C—H = 0.96 Å. The isotropic displacement parameters of the H atoms were set to  $1.2U_{\text{eq}}(\text{C-aromatic, N})$  and  $1.5U_{\text{eq}}(\text{C-methyl})$ .

## Figures

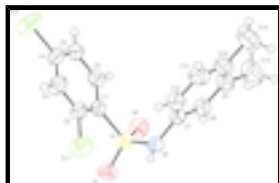


Fig. 1. Molecular structure of the title compound, showing the atom labelling scheme and displacement ellipsoids are drawn at the 50% probability level.

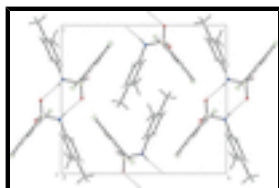


Fig. 2. Molecular packing of (I) with hydrogen bonding shown as dashed lines.

## 2,4-Dichloro-*N*-(3,4-dimethylphenyl)benzenesulfonamide

### Crystal data

$\text{C}_{14}\text{H}_{13}\text{Cl}_2\text{NO}_2\text{S}$

$M_r = 330.21$

Monoclinic,  $P2_1/c$

Hall symbol: -P 2ybc

$a = 7.8381$  (6) Å

$b = 14.778$  (1) Å

$c = 13.660$  (1) Å

$\beta = 101.840$  (9)°

$V = 1548.59$  (19) Å<sup>3</sup>

$Z = 4$

$F(000) = 680$

$D_x = 1.416$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 2657 reflections

$\theta = 2.7\text{--}27.8^\circ$

$\mu = 0.55$  mm<sup>-1</sup>

$T = 293$  K

Prism, light pink

$0.46 \times 0.44 \times 0.32$  mm

### Data collection

Oxford Diffraction Xcalibur diffractometer with Sapphire CCD detector

Radiation source: fine-focus sealed tube graphite

Rotation method data acquisition using  $\omega$  scans.

Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)

$T_{\text{min}} = 0.785$ ,  $T_{\text{max}} = 0.843$

6193 measured reflections

3155 independent reflections

2447 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.011$

$\theta_{\text{max}} = 26.4^\circ$ ,  $\theta_{\text{min}} = 2.7^\circ$

$h = -9 \rightarrow 9$

$k = -14 \rightarrow 18$

$l = -17 \rightarrow 16$

Refinement

Refinement on $F^2$	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.038$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.105$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.0533P)^2 + 0.5615P]$
3155 reflections	where $P = (F_o^2 + 2F_c^2)/3$
186 parameters	$(\Delta/\sigma)_{\max} < 0.001$
1 restraint	$\Delta\rho_{\max} = 0.36 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\min} = -0.40 \text{ e } \text{\AA}^{-3}$

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.

**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	0.34354 (8)	0.43015 (5)	0.08084 (5)	0.0707 (2)
Cl2	0.60591 (10)	0.20253 (6)	0.37736 (7)	0.0976 (3)
S1	-0.05081 (6)	0.38265 (3)	0.10982 (4)	0.04088 (15)
O1	-0.04339 (19)	0.37506 (10)	0.00605 (10)	0.0515 (4)
O2	-0.19273 (18)	0.34323 (11)	0.14429 (12)	0.0558 (4)
N1	-0.0465 (2)	0.48983 (12)	0.13356 (12)	0.0444 (4)
H1N	0.010 (3)	0.5191 (15)	0.0985 (16)	0.053*
C1	0.1397 (2)	0.33428 (13)	0.18378 (13)	0.0372 (4)
C2	0.3069 (3)	0.35329 (14)	0.16991 (15)	0.0451 (5)
C3	0.4500 (3)	0.31241 (17)	0.22924 (18)	0.0579 (6)
H3	0.5617	0.3246	0.2195	0.069*
C4	0.4251 (3)	0.25347 (16)	0.30303 (17)	0.0557 (6)
C5	0.2625 (3)	0.23435 (15)	0.31959 (16)	0.0502 (5)
H5	0.2483	0.1949	0.3704	0.060*
C6	0.1197 (3)	0.27492 (13)	0.25921 (15)	0.0433 (5)
H6	0.0084	0.2622	0.2694	0.052*
C7	-0.0452 (3)	0.52380 (13)	0.23208 (14)	0.0424 (4)

## supplementary materials

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C8	-0.1957 (3)	0.52055 (16)	0.26998 (17)	0.0530 (5)
H8	-0.2953	0.4936	0.2327	0.064*
C9	-0.1994 (4)	0.55708 (16)	0.36325 (19)	0.0631 (7)
C10	-0.0514 (4)	0.59808 (16)	0.41788 (18)	0.0655 (7)
C11	0.0974 (4)	0.60113 (17)	0.37911 (18)	0.0648 (7)
H11	0.1966	0.6289	0.4158	0.078*
C12	0.1028 (3)	0.56374 (15)	0.28668 (17)	0.0532 (5)
H12	0.2048	0.5656	0.2620	0.064*
C13	-0.3662 (5)	0.5535 (2)	0.4015 (3)	0.1035 (12)
H13A	-0.4509	0.5183	0.3566	0.124*
H13B	-0.4098	0.6138	0.4056	0.124*
H13C	-0.3442	0.5262	0.4666	0.124*
C14	-0.0508 (6)	0.6415 (2)	0.5189 (2)	0.1010 (12)
H14A	-0.0870	0.5978	0.5625	0.121*
H14B	-0.1297	0.6919	0.5102	0.121*
H14C	0.0647	0.6622	0.5477	0.121*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C11	0.0485 (3)	0.0890 (5)	0.0782 (4)	-0.0019 (3)	0.0215 (3)	0.0324 (4)
C12	0.0711 (5)	0.1047 (6)	0.1041 (6)	0.0292 (4)	-0.0126 (4)	0.0312 (5)
S1	0.0337 (3)	0.0458 (3)	0.0413 (3)	-0.0019 (2)	0.00329 (19)	-0.0010 (2)
O1	0.0539 (9)	0.0573 (9)	0.0392 (8)	-0.0029 (7)	0.0001 (6)	-0.0048 (7)
O2	0.0350 (8)	0.0619 (10)	0.0708 (10)	-0.0070 (7)	0.0113 (7)	0.0043 (8)
N1	0.0491 (10)	0.0431 (10)	0.0424 (9)	0.0035 (8)	0.0125 (7)	0.0046 (7)
C1	0.0359 (9)	0.0371 (10)	0.0373 (10)	-0.0003 (8)	0.0050 (7)	-0.0038 (8)
C2	0.0386 (10)	0.0484 (12)	0.0489 (11)	0.0012 (9)	0.0105 (9)	0.0046 (9)
C3	0.0366 (11)	0.0658 (15)	0.0698 (15)	0.0056 (10)	0.0075 (10)	0.0073 (12)
C4	0.0505 (13)	0.0530 (13)	0.0573 (13)	0.0127 (10)	-0.0037 (10)	0.0027 (11)
C5	0.0624 (14)	0.0438 (12)	0.0429 (11)	0.0035 (10)	0.0070 (10)	0.0024 (9)
C6	0.0468 (11)	0.0412 (11)	0.0428 (11)	-0.0043 (9)	0.0110 (9)	-0.0029 (8)
C7	0.0489 (11)	0.0374 (10)	0.0415 (10)	0.0080 (9)	0.0111 (9)	0.0056 (8)
C8	0.0531 (12)	0.0522 (13)	0.0566 (13)	0.0066 (10)	0.0175 (10)	0.0058 (10)
C9	0.0836 (18)	0.0539 (14)	0.0609 (14)	0.0125 (13)	0.0358 (14)	0.0099 (12)
C10	0.109 (2)	0.0430 (13)	0.0481 (13)	0.0076 (13)	0.0249 (14)	0.0051 (10)
C11	0.0867 (19)	0.0515 (14)	0.0520 (14)	-0.0050 (12)	0.0047 (13)	-0.0016 (11)
C12	0.0547 (13)	0.0509 (13)	0.0538 (13)	0.0017 (10)	0.0106 (10)	0.0001 (10)
C13	0.109 (3)	0.117 (3)	0.104 (2)	0.014 (2)	0.067 (2)	0.005 (2)
C14	0.187 (4)	0.0650 (18)	0.0592 (17)	0.007 (2)	0.045 (2)	-0.0059 (14)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

C11—C2	1.731 (2)	C7—C12	1.377 (3)
C12—C4	1.736 (2)	C7—C8	1.382 (3)
S1—O2	1.4190 (15)	C8—C9	1.390 (3)
S1—O1	1.4349 (15)	C8—H8	0.9300
S1—N1	1.6157 (18)	C9—C10	1.384 (4)
S1—C1	1.7724 (19)	C9—C13	1.505 (4)

N1—C7	1.434 (2)	C10—C11	1.377 (4)
N1—H1N	0.834 (16)	C10—C14	1.521 (3)
C1—C6	1.386 (3)	C11—C12	1.387 (3)
C1—C2	1.391 (3)	C11—H11	0.9300
C2—C3	1.381 (3)	C12—H12	0.9300
C3—C4	1.376 (3)	C13—H13A	0.9600
C3—H3	0.9300	C13—H13B	0.9600
C4—C5	1.369 (3)	C13—H13C	0.9600
C5—C6	1.383 (3)	C14—H14A	0.9600
C5—H5	0.9300	C14—H14B	0.9600
C6—H6	0.9300	C14—H14C	0.9600
O2—S1—O1	119.28 (9)	C8—C7—N1	119.79 (19)
O2—S1—N1	108.75 (9)	C9—C8—C7	120.7 (2)
O1—S1—N1	105.76 (9)	C9—C8—H8	119.7
O2—S1—C1	105.73 (9)	C7—C8—H8	119.7
O1—S1—C1	109.36 (9)	C10—C9—C8	119.4 (2)
N1—S1—C1	107.48 (9)	C10—C9—C13	121.4 (3)
C7—N1—S1	121.82 (14)	C8—C9—C13	119.2 (3)
C7—N1—H1N	117.0 (16)	C11—C10—C9	119.3 (2)
S1—N1—H1N	112.7 (16)	C11—C10—C14	119.4 (3)
C6—C1—C2	118.84 (18)	C9—C10—C14	121.3 (3)
C6—C1—S1	117.93 (15)	C10—C11—C12	121.6 (2)
C2—C1—S1	123.23 (15)	C10—C11—H11	119.2
C3—C2—C1	120.41 (19)	C12—C11—H11	119.2
C3—C2—C11	117.90 (16)	C7—C12—C11	119.0 (2)
C1—C2—C11	121.67 (15)	C7—C12—H12	120.5
C4—C3—C2	119.1 (2)	C11—C12—H12	120.5
C4—C3—H3	120.4	C9—C13—H13A	109.5
C2—C3—H3	120.4	C9—C13—H13B	109.5
C5—C4—C3	121.9 (2)	H13A—C13—H13B	109.5
C5—C4—C12	119.33 (18)	C9—C13—H13C	109.5
C3—C4—C12	118.78 (18)	H13A—C13—H13C	109.5
C4—C5—C6	118.6 (2)	H13B—C13—H13C	109.5
C4—C5—H5	120.7	C10—C14—H14A	109.5
C6—C5—H5	120.7	C10—C14—H14B	109.5
C1—C6—C5	121.09 (19)	H14A—C14—H14B	109.5
C1—C6—H6	119.5	C10—C14—H14C	109.5
C5—C6—H6	119.5	H14A—C14—H14C	109.5
C12—C7—C8	120.1 (2)	H14B—C14—H14C	109.5
C12—C7—N1	120.07 (19)		
O2—S1—N1—C7	53.19 (18)	C2—C1—C6—C5	-0.5 (3)
O1—S1—N1—C7	-177.58 (15)	S1—C1—C6—C5	179.74 (15)
C1—S1—N1—C7	-60.84 (18)	C4—C5—C6—C1	-0.5 (3)
O2—S1—C1—C6	-2.88 (18)	S1—N1—C7—C12	109.5 (2)
O1—S1—C1—C6	-132.49 (15)	S1—N1—C7—C8	-73.8 (2)
N1—S1—C1—C6	113.15 (16)	C12—C7—C8—C9	-0.2 (3)
O2—S1—C1—C2	177.36 (17)	N1—C7—C8—C9	-176.85 (19)
O1—S1—C1—C2	47.75 (19)	C7—C8—C9—C10	0.8 (3)

## supplementary materials

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N1—S1—C1—C2	-66.61 (19)	C7—C8—C9—C13	179.1 (2)
C6—C1—C2—C3	1.1 (3)	C8—C9—C10—C11	-0.6 (4)
S1—C1—C2—C3	-179.13 (17)	C13—C9—C10—C11	-178.9 (3)
C6—C1—C2—C11	-177.31 (15)	C8—C9—C10—C14	178.1 (2)
S1—C1—C2—C11	2.4 (3)	C13—C9—C10—C14	-0.1 (4)
C1—C2—C3—C4	-0.8 (4)	C9—C10—C11—C12	-0.3 (4)
C11—C2—C3—C4	177.71 (19)	C14—C10—C11—C12	-179.0 (2)
C2—C3—C4—C5	-0.2 (4)	C8—C7—C12—C11	-0.7 (3)
C2—C3—C4—C12	-179.91 (18)	N1—C7—C12—C11	175.98 (19)
C3—C4—C5—C6	0.8 (3)	C10—C11—C12—C7	0.9 (3)
C12—C4—C5—C6	-179.48 (16)		

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1N $\cdots$ O1 <sup>i</sup>	0.83 (2)	2.17 (2)	2.945 (2)	154 (2)

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



Fig. 1

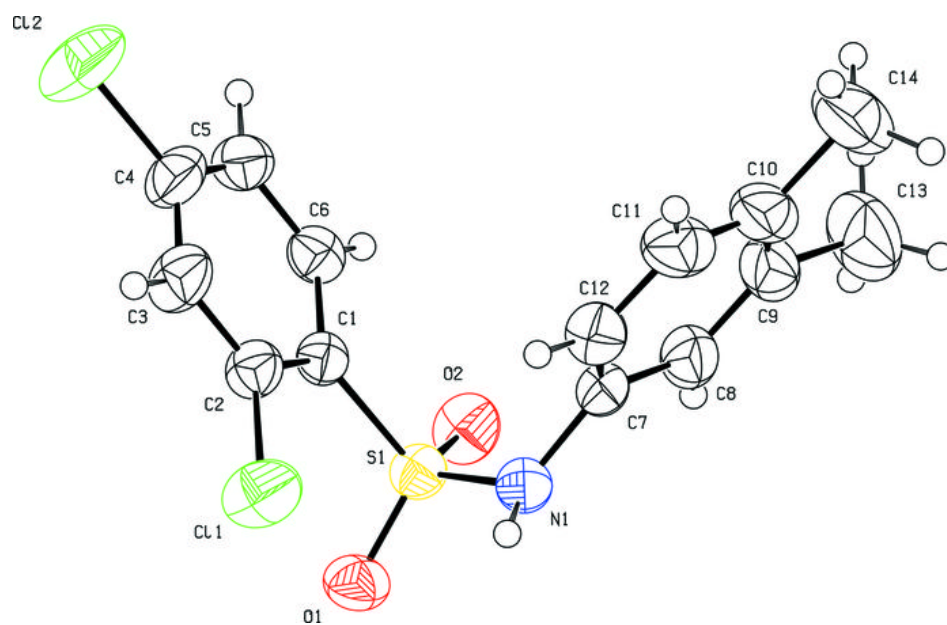


Fig. 2

