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2,4-Dichloro-N-(3,5-dimethylphenyl)-benzenesulfonamide

Vinola Z. Rodrigues,^a Sabine Foro^b and B. Thimme Gowda^{a*}

^aDepartment of Chemistry, Mangalore University, Mangalagangothri 574 199, Mangalore, India, and ^bInstitute of Materials Science, Darmstadt University of Technology, Petersenstrasse 23, D-64287 Darmstadt, Germany
Correspondence e-mail: gowdabt@yahoo.com

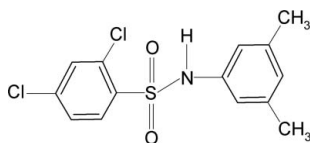
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.046; wR factor = 0.124; data-to-parameter ratio = 16.5.

In the crystal of the title compound, $\text{C}_{14}\text{H}_{13}\text{Cl}_2\text{NO}_2\text{S}$, the N—H bond in the C—SO₂—NH—C segment is *syn* to one of the *meta*-methyl groups in the aniline benzene ring and *anti* to the other. Further, the conformation of the N—C bond in the C—SO₂—NH—C segment is *gauche* with respect to the S=O bonds. The C—SO₂—NH—C torsion angle is 54.9 (2)°. The sulfonyl and aniline benzene rings are tilted relative to each other by 82.8 (1)°. The crystal structure features inversion-related dimers linked by pairs of N—H···O hydrogen bonds. There are also weak C—H···O hydrogen bonds between these dimers.

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: Gowda *et al.* (2003), on *N*-(aryl)-methane-sulfonamides, see: Gowda *et al.* (2007) and on *N*-(aryl)-aryl-sulfonamides, see: Gelbrich *et al.* (2007); Perlovich *et al.* (2006); Gowda & Kumar (2003); 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, $C2/c$
 $a = 23.067$ (2) Å
 $b = 8.0794$ (5) Å
 $c = 16.470$ (1) Å
 $\beta = 101.575$ (7)°
 $V = 3007.0$ (4) Å³

$Z = 8$
Mo $K\alpha$ radiation
 $\mu = 0.57$ mm⁻¹
 $T = 293$ K
 $0.50 \times 0.46 \times 0.42$ mm

Data collection

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

Diffraction, 2009)
 $T_{\min} = 0.764$, $T_{\max} = 0.796$
10045 measured reflections
3067 independent reflections
2555 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.018$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.124$
 $S = 1.09$
3067 reflections
186 parameters
1 restraint

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.42$ e Å⁻³
 $\Delta\rho_{\min} = -0.73$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^i$	0.85 (2)	2.11 (2)	2.948 (2)	176 (2)
$\text{C3}-\text{H3}\cdots\text{O2}^{ii}$	0.93	2.40	3.264 (2)	154 (1)

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

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: BQ2307).

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supplementary materials

Acta Cryst. (2011). E67, o2842 [doi:10.1107/S1600536811040189]

2,4-Dichloro-*N*-(3,5-dimethylphenyl)benzenesulfonamide

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

Comment

The sulfonamide moiety is present in several biologically important compounds. The hydrogen bonding preferences of sulfonamides have also been investigated (Adsmund & Grant, 2001). As part of our studies on the substituent effects on the structures and other aspects of *N*-(aryl)-amides (Gowda *et al.*, 2003), *N*-(aryl)-methanesulfonamides (Gowda *et al.*, 2007) and *N*-(aryl)-arylsulfonamides (Gowda & Kumar, 2003; Rodrigues *et al.*, 2011), in the present work, the crystal structure of 2,4-dichloro-*N*-(3,5-dimethylphenyl)-benzenesulfonamide (I) has been determined (Fig. 1).

In (I), the N—H bond in the C—SO₂—NH—C segment is *syn* to one of the *meta*-methyl groups in the aniline benzene ring and *anti* to the other. Further, the conformations of the N—C bonds in the C—SO₂—NH—C segment have *gauche* torsions with respect to the S=O bonds.

The molecule is bent at the S atom with C—SO₂—NH—C torsion angle of 54.94 (20)°, compared to the value of -60.84 (18)° in 2,4-dichloro-*N*-(3,4-dimethylphenyl)-benzenesulfonamide (II) (Rodrigues *et al.*, 2011).

The sulfonyl and the aniline benzene rings are tilted relative to each other by 66.4 (1)°, compared to the value of 82.8 (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. There is also weak C—H⋯O hydrogen bonds between these 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 3,5-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*-(3,5-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 atoms of the NH groups were 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. All H atoms were refined with isotropic displacement parameters. The $U_{\text{iso}}(\text{H})$ values were set at $1.2U_{\text{eq}}(\text{C-aromatic, N})$ and $1.5U_{\text{eq}}(\text{C-methyl})$.

Figures

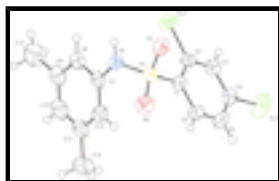


Fig. 1. Molecular structure of the title compound, showing the atom labeling scheme and displacement ellipsoids are drawn at the 50% probability level.

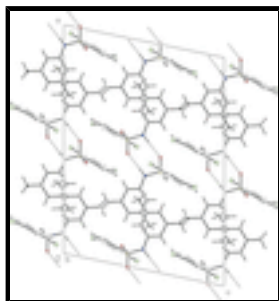


Fig. 2. Molecular packing of (I) with hydrogen bonding shown as dashed lines.

2,4-Dichloro-*N*-(3,5-dimethylphenyl)benzenesulfonamide

Crystal data

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

$M_r = 330.21$

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 23.067\ (2)\ \text{\AA}$

$b = 8.0794\ (5)\ \text{\AA}$

$c = 16.470\ (1)\ \text{\AA}$

$\beta = 101.575\ (7)^\circ$

$V = 3007.0\ (4)\ \text{\AA}^3$

$Z = 8$

$F(000) = 1360$

$D_x = 1.459\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 3755 reflections

$\theta = 2.7\text{--}28.0^\circ$

$\mu = 0.57\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Prism, light pink

$0.50 \times 0.46 \times 0.42\ \text{mm}$

Data collection

Oxford Diffraction Xcalibur
diffractometer with a Sapphire CCD Detector

Radiation source: fine-focus sealed tube
graphite

3067 independent reflections

2555 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.018$

Rotation method data acquisition using ω scans $\theta_{\max} = 26.4^\circ$, $\theta_{\min} = 2.7^\circ$
 Absorption correction: multi-scan $h = -26 \rightarrow 28$
 (*CrysAlis RED*; Oxford Diffraction, 2009)
 $T_{\min} = 0.764$, $T_{\max} = 0.796$ $k = -10 \rightarrow 10$
 10045 measured reflections $l = -20 \rightarrow 10$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.046$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.124$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.09$	$w = 1/[\sigma^2(F_o^2) + (0.080P)^2 + 1.2587P]$
3067 reflections	where $P = (F_o^2 + 2F_c^2)/3$
186 parameters	$(\Delta/\sigma)_{\max} = 0.005$
1 restraint	$\Delta\rho_{\max} = 0.42 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\min} = -0.73 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.46825 (3)	0.17165 (7)	0.04412 (4)	0.0546 (2)
Cl2	0.40676 (4)	-0.06394 (9)	0.31762 (4)	0.0740 (3)
S1	0.44325 (2)	0.54969 (6)	0.10219 (3)	0.03642 (17)
O1	0.50536 (6)	0.55754 (19)	0.10210 (9)	0.0465 (4)
O2	0.41625 (8)	0.68220 (18)	0.13769 (11)	0.0528 (4)
N1	0.41157 (8)	0.5283 (2)	0.00604 (11)	0.0404 (4)
H1N	0.4355 (10)	0.498 (3)	-0.0239 (14)	0.048*
C1	0.42987 (8)	0.3693 (2)	0.15700 (11)	0.0324 (4)
C2	0.44187 (8)	0.2097 (2)	0.13350 (12)	0.0343 (4)
C3	0.43374 (9)	0.0751 (2)	0.18203 (13)	0.0402 (5)
H3	0.4412	-0.0318	0.1659	0.048*
C4	0.41432 (9)	0.1028 (3)	0.25492 (12)	0.0423 (5)

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C5	0.40168 (10)	0.2587 (3)	0.27941 (13)	0.0453 (5)
H5	0.3883	0.2746	0.3285	0.054*
C6	0.40921 (9)	0.3917 (3)	0.22994 (12)	0.0406 (4)
H6	0.4003	0.4978	0.2456	0.049*
C7	0.35076 (9)	0.4915 (2)	−0.02514 (12)	0.0383 (4)
C8	0.33728 (10)	0.4228 (3)	−0.10365 (14)	0.0437 (5)
H8	0.3675	0.3984	−0.1315	0.052*
C9	0.27908 (11)	0.3901 (3)	−0.14115 (16)	0.0556 (6)
C10	0.23500 (11)	0.4246 (3)	−0.09667 (18)	0.0612 (7)
H10	0.1958	0.4018	−0.1208	0.073*
C11	0.24769 (10)	0.4914 (3)	−0.01807 (16)	0.0539 (6)
C12	0.30625 (10)	0.5275 (3)	0.01805 (14)	0.0471 (5)
H12	0.3155	0.5750	0.0705	0.057*
C13	0.26428 (15)	0.3176 (5)	−0.2271 (2)	0.0863 (10)
H13A	0.2571	0.2010	−0.2235	0.104*
H13B	0.2968	0.3349	−0.2544	0.104*
H13C	0.2295	0.3706	−0.2580	0.104*
C14	0.19926 (13)	0.5276 (4)	0.0292 (2)	0.0767 (9)
H14A	0.1899	0.4286	0.0560	0.092*
H14B	0.1646	0.5660	−0.0086	0.092*
H14C	0.2125	0.6114	0.0700	0.092*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0766 (4)	0.0469 (3)	0.0478 (3)	0.0090 (3)	0.0306 (3)	−0.0059 (2)
C12	0.1009 (6)	0.0607 (4)	0.0620 (4)	−0.0197 (4)	0.0203 (4)	0.0207 (3)
S1	0.0393 (3)	0.0314 (3)	0.0382 (3)	−0.00622 (18)	0.0070 (2)	−0.00107 (18)
O1	0.0392 (8)	0.0546 (9)	0.0446 (8)	−0.0151 (6)	0.0059 (6)	0.0006 (7)
O2	0.0694 (11)	0.0308 (7)	0.0597 (10)	−0.0001 (7)	0.0167 (8)	−0.0068 (6)
N1	0.0357 (9)	0.0483 (10)	0.0374 (9)	−0.0037 (7)	0.0081 (7)	0.0039 (7)
C1	0.0316 (9)	0.0322 (9)	0.0332 (9)	−0.0036 (7)	0.0063 (7)	−0.0024 (7)
C2	0.0337 (9)	0.0357 (10)	0.0341 (9)	−0.0014 (8)	0.0082 (7)	−0.0040 (7)
C3	0.0422 (11)	0.0324 (10)	0.0451 (11)	−0.0033 (8)	0.0062 (9)	−0.0012 (8)
C4	0.0411 (11)	0.0447 (11)	0.0393 (10)	−0.0114 (9)	0.0035 (8)	0.0077 (9)
C5	0.0492 (12)	0.0544 (13)	0.0344 (10)	−0.0061 (10)	0.0134 (9)	−0.0029 (9)
C6	0.0444 (11)	0.0406 (11)	0.0383 (10)	−0.0020 (9)	0.0123 (8)	−0.0064 (8)
C7	0.0369 (10)	0.0346 (10)	0.0422 (10)	−0.0004 (8)	0.0049 (8)	0.0104 (8)
C8	0.0419 (11)	0.0406 (11)	0.0467 (12)	0.0004 (9)	0.0044 (9)	0.0049 (9)
C9	0.0465 (13)	0.0568 (14)	0.0572 (14)	−0.0022 (11)	−0.0044 (10)	0.0029 (11)
C10	0.0358 (12)	0.0689 (17)	0.0728 (17)	−0.0036 (11)	−0.0036 (11)	0.0097 (13)
C11	0.0374 (11)	0.0594 (14)	0.0655 (16)	0.0035 (10)	0.0117 (10)	0.0199 (12)
C12	0.0429 (12)	0.0516 (13)	0.0471 (12)	0.0007 (9)	0.0097 (9)	0.0080 (10)
C13	0.0644 (18)	0.106 (3)	0.077 (2)	−0.0030 (17)	−0.0139 (15)	−0.0246 (18)
C14	0.0469 (15)	0.100 (2)	0.087 (2)	0.0041 (14)	0.0234 (14)	0.0196 (18)

Geometric parameters (\AA , $^\circ$)

C11—C2	1.7296 (19)	C7—C8	1.384 (3)
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C12—C4	1.727 (2)	C7—C12	1.392 (3)
S1—O2	1.4220 (16)	C8—C9	1.386 (3)
S1—O1	1.4343 (16)	C8—H8	0.9300
S1—N1	1.6148 (18)	C9—C10	1.395 (4)
S1—C1	1.7739 (19)	C9—C13	1.506 (4)
N1—C7	1.425 (3)	C10—C11	1.379 (4)
N1—H1N	0.845 (16)	C10—H10	0.9300
C1—C2	1.390 (3)	C11—C12	1.393 (3)
C1—C6	1.390 (3)	C11—C14	1.512 (4)
C2—C3	1.385 (3)	C12—H12	0.9300
C3—C4	1.381 (3)	C13—H13A	0.9600
C3—H3	0.9300	C13—H13B	0.9600
C4—C5	1.372 (3)	C13—H13C	0.9600
C5—C6	1.380 (3)	C14—H14A	0.9600
C5—H5	0.9300	C14—H14B	0.9600
C6—H6	0.9300	C14—H14C	0.9600
O2—S1—O1	119.16 (10)	C12—C7—N1	123.1 (2)
O2—S1—N1	109.55 (10)	C7—C8—C9	120.6 (2)
O1—S1—N1	105.02 (9)	C7—C8—H8	119.7
O2—S1—C1	105.91 (9)	C9—C8—H8	119.7
O1—S1—C1	108.22 (9)	C8—C9—C10	118.1 (2)
N1—S1—C1	108.67 (9)	C8—C9—C13	120.6 (2)
C7—N1—S1	126.50 (14)	C10—C9—C13	121.3 (2)
C7—N1—H1N	116.2 (17)	C11—C10—C9	122.1 (2)
S1—N1—H1N	112.5 (17)	C11—C10—H10	118.9
C2—C1—C6	118.90 (17)	C9—C10—H10	118.9
C2—C1—S1	123.77 (14)	C10—C11—C12	119.2 (2)
C6—C1—S1	117.23 (15)	C10—C11—C14	121.3 (2)
C3—C2—C1	120.70 (17)	C12—C11—C14	119.5 (3)
C3—C2—C11	117.57 (15)	C7—C12—C11	119.3 (2)
C1—C2—C11	121.72 (15)	C7—C12—H12	120.3
C4—C3—C2	118.62 (19)	C11—C12—H12	120.3
C4—C3—H3	120.7	C9—C13—H13A	109.5
C2—C3—H3	120.7	C9—C13—H13B	109.5
C5—C4—C3	121.99 (18)	H13A—C13—H13B	109.5
C5—C4—C12	119.18 (16)	C9—C13—H13C	109.5
C3—C4—C12	118.83 (17)	H13A—C13—H13C	109.5
C4—C5—C6	118.80 (19)	H13B—C13—H13C	109.5
C4—C5—H5	120.6	C11—C14—H14A	109.5
C6—C5—H5	120.6	C11—C14—H14B	109.5
C5—C6—C1	120.96 (19)	H14A—C14—H14B	109.5
C5—C6—H6	119.5	C11—C14—H14C	109.5
C1—C6—H6	119.5	H14A—C14—H14C	109.5
C8—C7—C12	120.7 (2)	H14B—C14—H14C	109.5
C8—C7—N1	116.14 (18)		
O2—S1—N1—C7	-60.3 (2)	C12—C4—C5—C6	178.44 (17)
O1—S1—N1—C7	170.56 (17)	C4—C5—C6—C1	-0.7 (3)
C1—S1—N1—C7	54.9 (2)	C2—C1—C6—C5	1.2 (3)

supplementary materials

O2—S1—C1—C2	170.58 (16)	S1—C1—C6—C5	-175.34 (16)
O1—S1—C1—C2	-60.58 (18)	S1—N1—C7—C8	-158.16 (16)
N1—S1—C1—C2	52.96 (18)	S1—N1—C7—C12	24.4 (3)
O2—S1—C1—C6	-13.03 (18)	C12—C7—C8—C9	0.7 (3)
O1—S1—C1—C6	115.81 (16)	N1—C7—C8—C9	-176.8 (2)
N1—S1—C1—C6	-130.65 (16)	C7—C8—C9—C10	-1.5 (3)
C6—C1—C2—C3	-0.4 (3)	C7—C8—C9—C13	179.1 (3)
S1—C1—C2—C3	175.98 (15)	C8—C9—C10—C11	0.9 (4)
C6—C1—C2—C11	-179.47 (15)	C13—C9—C10—C11	-179.8 (3)
S1—C1—C2—C11	-3.1 (2)	C9—C10—C11—C12	0.7 (4)
C1—C2—C3—C4	-1.0 (3)	C9—C10—C11—C14	-179.9 (3)
C11—C2—C3—C4	178.18 (16)	C8—C7—C12—C11	0.9 (3)
C2—C3—C4—C5	1.5 (3)	N1—C7—C12—C11	178.2 (2)
C2—C3—C4—C12	-177.60 (15)	C10—C11—C12—C7	-1.5 (3)
C3—C4—C5—C6	-0.6 (3)	C14—C11—C12—C7	179.0 (2)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1N \cdots O1 ⁱ	0.85 (2)	2.11 (2)	2.948 (2)	176 (2)
C3—H3 \cdots O2 ⁱⁱ	0.93	2.40	3.264 (2)	154 (1)

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

Fig. 1

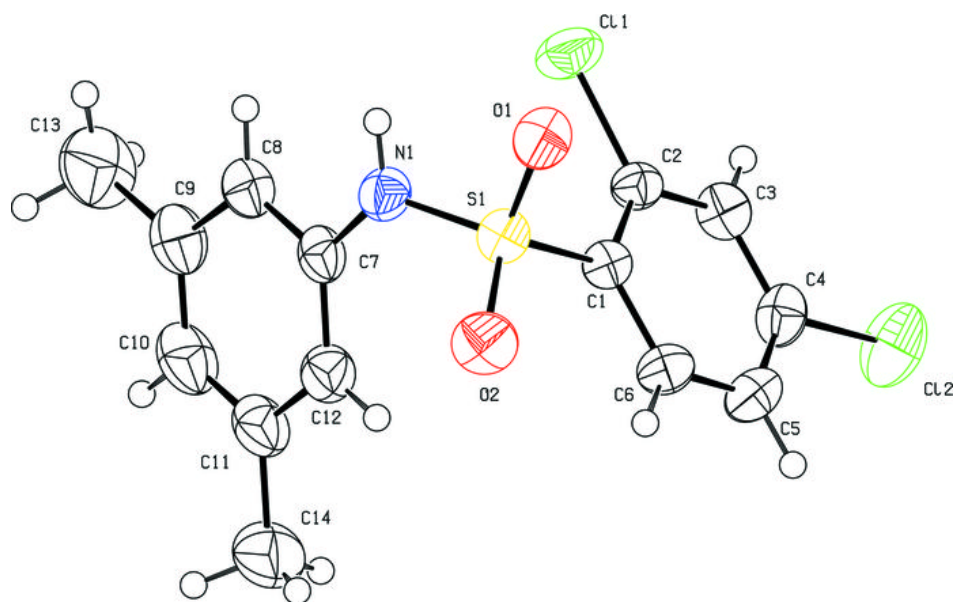


Fig. 2

