

N-(3-Chlorophenyl)-4-methylbenzenesulfonamide

B. Thimme Gowda,^{a*} Sabine Foro,^b P. G. Nirmala^a and Hartmut Fuess^b

^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

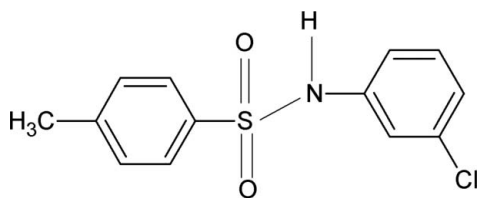
Received 13 December 2009; accepted 23 December 2009

Key indicators: single-crystal X-ray study; $T = 299$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.036; wR factor = 0.108; data-to-parameter ratio = 14.7.

In the title compound, $\text{C}_{13}\text{H}_{12}\text{ClNO}_2\text{S}$, the conformation of the $\text{N}-\text{H}$ bond is *anti* to the 3-chloro group in the aniline benzene ring. The dihedral angle between the two benzene rings is $73.7(1)^\circ$. 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: Gowda *et al.* (2005). For our study of the effects of substituents on the structures of *N*-(aryl)-arylsulfonamides, see: Gowda *et al.* (2008, 2009); Nirmala *et al.* (2009). For related structures, see: Gelbrich *et al.* (2007); Perlovich *et al.* (2006)



Experimental

Crystal data

$\text{C}_{13}\text{H}_{12}\text{ClNO}_2\text{S}$
 $M_r = 281.75$
Monoclinic, $P2_1/c$

$a = 9.774(1)$ Å
 $b = 13.589(1)$ Å
 $c = 10.066(1)$ Å

$\beta = 91.952(8)^\circ$
 $V = 1336.2(2)$ Å³
 $Z = 4$
Mo $K\alpha$ radiation

$\mu = 0.44$ mm⁻¹
 $T = 299$ K
 $0.46 \times 0.34 \times 0.20$ mm

Data collection

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

Diffraction, 2009)
 $T_{\min} = 0.825$, $T_{\max} = 0.918$
5167 measured reflections
2445 independent reflections
1957 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.011$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.108$
 $S = 1.08$
2445 reflections
166 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.22$ e Å⁻³
 $\Delta\rho_{\min} = -0.29$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^i$	0.84 (2)	2.09 (2)	2.932 (2)	175 (2)

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

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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2186).

References

- Gelbrich, T., Hursthouse, M. B. & Threlfall, T. L. (2007). *Acta Cryst.* **B63**, 621–632.
Gowda, B. T., Foro, S., Babitha, K. S. & Fuess, H. (2008). *Acta Cryst.* **E64**, o1825.
Gowda, B. T., Foro, S., Nirmala, P. G., Terao, H. & Fuess, H. (2009). *Acta Cryst.* **E65**, o1219.
Gowda, B. T., Shetty, M. & Jayalakshmi, K. L. (2005). *Z. Naturforsch. Teil A*, **60**, 106–112.
Nirmala, P. G., Gowda, B. T., Foro, S. & Fuess, H. (2009). *Acta Cryst.* **E65**, o3208.
Oxford Diffraction (2009). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Yarnton, England.
Perlovich, G. L., Tkachev, V. V., Schaper, K.-J. & Raevsky, O. A. (2006). *Acta Cryst.* **E62**, o780–o782.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

supplementary materials

Acta Cryst. (2010). E66, o278 [doi:10.1107/S1600536809055238]

***N*-(3-Chlorophenyl)-4-methylbenzenesulfonamide**

B. T. Gowda, S. Foro, P. G. Nirmala and H. Fuess

Comment

In the present work, as part of a study of the effect of substituents on the crystal structures of *N*-(aryl)-arylsulfonamides (Gowda *et al.*, 2008, 2009; Nirmala *et al.*, 2009), the structure of *N*-(3-chlorophenyl)4-methylbenzenesulfonamide (I) has been determined. The conformation of the N—C bond in the C1—SO₂—NH—C7 segment of the structure has *gauche* torsions with respect to the S=O bonds (Fig. 1). Further, the conformation of the N—H bond is *anti* to the 3-chloro group in the aniline benzene ring, similar to that observed in *N*-(3-chlorophenyl)-benzenesulfonamide (II) (Gowda *et al.*, 2008) and that between the N—H bond and the 3-methyl group in the aniline benzene ring of *N*-(3-methylphenyl)4-methylbenzenesulfonamide (III) (Nirmala *et al.*, 2009). The molecule is bent at the S atom with the C1—SO₂—NH—C7 torsion angle of 54.1 (2)°, compared to the values of -60.1 (2)° in (II), 56.7 (3)° in (III) and -51.6 (3)° in 4-methyl-*N*-(phenyl)-benzenesulfonamide (IV)(Gowda *et al.*, 2009),

The two benzene rings in (I) are tilted relative to each other by 73.7 (1)°, compared to the values of 65.4 (1)° in (II), 83.9 (1)° in (III) and 68.4 (1)° in (IV). The other bond parameters are similar to those observed in (II), (III), (IV) and other aryl sulfonamides (Perlovich *et al.*, 2006; Gelbrich *et al.*, 2007). The crystal packing stabilized by pairs of intermolecular N—H···O hydrogen bonds (Table 1) is shown in Fig.2.

Experimental

The solution of toluene (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 benzenesulfonylchloride was treated with 3-chloroaniline 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 *N*-(3-chlorophenyl)4-methylbenzenesulfonamide 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 (Gowda *et al.*, 2005). The rod like single crystals used in X-ray diffraction studies were grown in ethanolic solution by a slow evaporation at room temperature.

Refinement

The H atom of the NH group was located in a difference map and later restrained to the distance N—H = 0.86 (2) Å. The other H atoms were positioned with idealized geometry using a riding model with C—H = 0.93–0.96 Å A 11 H atoms were refined with isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent atom).

Figures

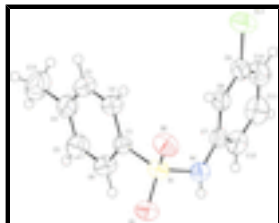


Fig. 1. Molecular structure of (I), showing the atom labelling scheme and displacement ellipsoids drawn at the 50% probability level.

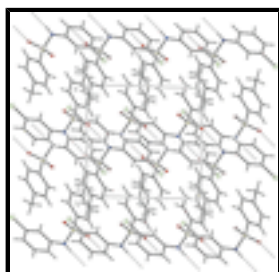


Fig. 2. Molecular packing of (I) with hydrogen bonding shown as dashed lines.

***N*-(3-Chlorophenyl)-4-methylbenzenesulfonamide**

Crystal data

$C_{13}H_{12}ClNO_2S$

$M_r = 281.75$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 9.774$ (1) Å

$b = 13.589$ (1) Å

$c = 10.066$ (1) Å

$\beta = 91.952$ (8)°

$V = 1336.2$ (2) Å³

$Z = 4$

$F(000) = 584$

$D_x = 1.401$ Mg m⁻³

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

Cell parameters from 2689 reflections

$\theta = 2.5$ – 27.8 °

$\mu = 0.44$ mm⁻¹

$T = 299$ K

Rod, colourless

$0.46 \times 0.34 \times 0.20$ mm

Data collection

Oxford Diffraction Xcalibur (TM) diffractometer with a Sapphire CCD detector

Radiation source: fine-focus sealed tube

graphite

Rotation method data acquisition using ω and phi scans

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

$T_{\min} = 0.825$, $T_{\max} = 0.918$

5167 measured reflections

2445 independent reflections

1957 reflections with $I > 2\sigma(I)$

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

$\theta_{\max} = 25.4$ °, $\theta_{\min} = 2.5$ °

$h = -11 \rightarrow 11$

$k = -16 \rightarrow 12$

$l = -12 \rightarrow 7$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.036$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.108$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.08$	$w = 1/[\sigma^2(F_o^2) + (0.058P)^2 + 0.340P]$
2445 reflections	where $P = (F_o^2 + 2F_c^2)/3$
166 parameters	$(\Delta/\sigma)_{\max} = 0.001$
0 restraints	$\Delta\rho_{\max} = 0.22 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\min} = -0.29 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. CrysAlis RED (Oxford Diffraction, 2009) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.18744 (19)	1.05082 (14)	0.08569 (19)	0.0411 (4)
C2	0.0703 (2)	1.05677 (18)	0.1578 (2)	0.0573 (6)
H2	0.0517	1.0093	0.2212	0.069*
C3	-0.0188 (2)	1.1340 (2)	0.1347 (2)	0.0686 (7)
H3	-0.0975	1.1382	0.1838	0.082*
C4	0.0059 (2)	1.20537 (18)	0.0404 (2)	0.0589 (6)
C5	0.1235 (2)	1.19686 (17)	-0.0317 (2)	0.0605 (6)
H5	0.1415	1.2436	-0.0963	0.073*
C6	0.2143 (2)	1.12092 (16)	-0.0097 (2)	0.0513 (5)
H6	0.2932	1.1167	-0.0585	0.062*
C7	0.42477 (19)	1.05956 (14)	0.31752 (19)	0.0409 (4)
C8	0.3351 (2)	1.03090 (15)	0.41417 (19)	0.0440 (5)
H8	0.2785	0.9765	0.4011	0.053*
C9	0.3317 (2)	1.08485 (16)	0.5303 (2)	0.0488 (5)
C10	0.4151 (3)	1.16417 (17)	0.5545 (2)	0.0608 (6)

supplementary materials

H10	0.4118	1.1987	0.6341	0.073*
C11	0.5039 (3)	1.19146 (16)	0.4578 (3)	0.0648 (6)
H11	0.5613	1.2452	0.4725	0.078*
C12	0.5092 (2)	1.14039 (15)	0.3391 (2)	0.0527 (5)
H12	0.5690	1.1602	0.2743	0.063*
C13	-0.0920 (3)	1.2896 (2)	0.0170 (3)	0.0881 (9)
H13A	-0.1003	1.3263	0.0978	0.106*
H13B	-0.1801	1.2643	-0.0110	0.106*
H13C	-0.0581	1.3318	-0.0509	0.106*
N1	0.43675 (18)	1.00545 (14)	0.19789 (17)	0.0474 (4)
H1N	0.496 (2)	1.0277 (16)	0.147 (2)	0.057*
O1	0.36308 (15)	0.92586 (11)	-0.00834 (14)	0.0531 (4)
O2	0.24646 (16)	0.88340 (11)	0.19738 (15)	0.0590 (4)
Cl1	0.21817 (7)	1.04894 (6)	0.65092 (6)	0.0776 (2)
S1	0.30698 (5)	0.95603 (3)	0.11563 (5)	0.04334 (18)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0400 (10)	0.0469 (11)	0.0364 (10)	-0.0042 (9)	0.0006 (8)	-0.0082 (8)
C2	0.0446 (12)	0.0796 (16)	0.0482 (13)	0.0006 (11)	0.0096 (10)	0.0020 (11)
C3	0.0439 (12)	0.103 (2)	0.0597 (14)	0.0120 (14)	0.0071 (10)	-0.0142 (15)
C4	0.0523 (13)	0.0638 (14)	0.0598 (14)	0.0104 (11)	-0.0099 (10)	-0.0213 (11)
C5	0.0644 (14)	0.0539 (13)	0.0634 (14)	0.0043 (11)	0.0028 (11)	0.0018 (11)
C6	0.0476 (12)	0.0504 (12)	0.0565 (13)	0.0019 (10)	0.0111 (10)	0.0015 (10)
C7	0.0384 (10)	0.0426 (10)	0.0413 (10)	0.0033 (8)	-0.0031 (8)	0.0064 (8)
C8	0.0414 (10)	0.0481 (11)	0.0422 (11)	-0.0033 (9)	-0.0019 (8)	0.0020 (9)
C9	0.0505 (12)	0.0542 (12)	0.0416 (11)	0.0068 (10)	-0.0005 (9)	0.0003 (9)
C10	0.0689 (15)	0.0522 (13)	0.0605 (14)	0.0055 (12)	-0.0088 (12)	-0.0128 (11)
C11	0.0680 (15)	0.0446 (12)	0.0808 (17)	-0.0101 (11)	-0.0119 (13)	-0.0024 (12)
C12	0.0505 (12)	0.0470 (11)	0.0604 (13)	-0.0047 (10)	-0.0010 (10)	0.0115 (10)
C13	0.0770 (18)	0.082 (2)	0.104 (2)	0.0302 (16)	-0.0137 (16)	-0.0218 (17)
N1	0.0413 (9)	0.0591 (11)	0.0421 (10)	-0.0023 (8)	0.0049 (7)	0.0019 (8)
O1	0.0603 (9)	0.0530 (8)	0.0468 (8)	0.0036 (7)	0.0123 (7)	-0.0074 (7)
O2	0.0740 (10)	0.0513 (8)	0.0522 (9)	-0.0142 (8)	0.0092 (7)	0.0052 (7)
Cl1	0.0747 (4)	0.1074 (6)	0.0518 (4)	-0.0022 (4)	0.0189 (3)	-0.0061 (3)
S1	0.0472 (3)	0.0436 (3)	0.0395 (3)	-0.0021 (2)	0.0063 (2)	-0.0014 (2)

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

C1—C2	1.379 (3)	C8—H8	0.9300
C1—C6	1.384 (3)	C9—C10	1.368 (3)
C1—S1	1.758 (2)	C9—Cl1	1.742 (2)
C2—C3	1.378 (3)	C10—C11	1.377 (3)
C2—H2	0.9300	C10—H10	0.9300
C3—C4	1.384 (4)	C11—C12	1.384 (3)
C3—H3	0.9300	C11—H11	0.9300
C4—C5	1.386 (3)	C12—H12	0.9300
C4—C13	1.505 (3)	C13—H13A	0.9600

C5—C6	1.374 (3)	C13—H13B	0.9600
C5—H5	0.9300	C13—H13C	0.9600
C6—H6	0.9300	N1—S1	1.6352 (18)
C7—C12	1.387 (3)	N1—H1N	0.84 (2)
C7—C8	1.387 (3)	O1—S1	1.4396 (14)
C7—N1	1.419 (3)	O2—S1	1.4263 (15)
C8—C9	1.381 (3)		
C2—C1—C6	120.4 (2)	C8—C9—C11	118.38 (17)
C2—C1—S1	120.76 (17)	C9—C10—C11	118.2 (2)
C6—C1—S1	118.84 (15)	C9—C10—H10	120.9
C3—C2—C1	119.1 (2)	C11—C10—H10	120.9
C3—C2—H2	120.4	C10—C11—C12	121.2 (2)
C1—C2—H2	120.4	C10—C11—H11	119.4
C2—C3—C4	121.7 (2)	C12—C11—H11	119.4
C2—C3—H3	119.1	C11—C12—C7	119.5 (2)
C4—C3—H3	119.1	C11—C12—H12	120.2
C3—C4—C5	117.9 (2)	C7—C12—H12	120.2
C3—C4—C13	121.1 (2)	C4—C13—H13A	109.5
C5—C4—C13	121.1 (3)	C4—C13—H13B	109.5
C6—C5—C4	121.4 (2)	H13A—C13—H13B	109.5
C6—C5—H5	119.3	C4—C13—H13C	109.5
C4—C5—H5	119.3	H13A—C13—H13C	109.5
C5—C6—C1	119.4 (2)	H13B—C13—H13C	109.5
C5—C6—H6	120.3	C7—N1—S1	123.88 (14)
C1—C6—H6	120.3	C7—N1—H1N	114.0 (16)
C12—C7—C8	119.96 (19)	S1—N1—H1N	112.3 (17)
C12—C7—N1	118.52 (18)	O2—S1—O1	118.73 (9)
C8—C7—N1	121.47 (18)	O2—S1—N1	108.77 (10)
C9—C8—C7	118.59 (19)	O1—S1—N1	104.13 (9)
C9—C8—H8	120.7	O2—S1—C1	108.64 (10)
C7—C8—H8	120.7	O1—S1—C1	109.30 (9)
C10—C9—C8	122.5 (2)	N1—S1—C1	106.58 (9)
C10—C9—C11	119.08 (17)		
C6—C1—C2—C3	-0.7 (3)	C9—C10—C11—C12	0.0 (4)
S1—C1—C2—C3	177.82 (18)	C10—C11—C12—C7	-0.7 (3)
C1—C2—C3—C4	0.4 (4)	C8—C7—C12—C11	0.5 (3)
C2—C3—C4—C5	0.4 (4)	N1—C7—C12—C11	-176.95 (19)
C2—C3—C4—C13	-179.6 (2)	C12—C7—N1—S1	-143.42 (17)
C3—C4—C5—C6	-0.8 (4)	C8—C7—N1—S1	39.2 (3)
C13—C4—C5—C6	179.2 (2)	C7—N1—S1—O2	-62.89 (19)
C4—C5—C6—C1	0.5 (3)	C7—N1—S1—O1	169.58 (16)
C2—C1—C6—C5	0.3 (3)	C7—N1—S1—C1	54.08 (19)
S1—C1—C6—C5	-178.30 (17)	C2—C1—S1—O2	12.5 (2)
C12—C7—C8—C9	0.5 (3)	C6—C1—S1—O2	-168.94 (16)
N1—C7—C8—C9	177.84 (18)	C2—C1—S1—O1	143.48 (17)
C7—C8—C9—C10	-1.3 (3)	C6—C1—S1—O1	-37.97 (19)
C7—C8—C9—C11	179.61 (15)	C2—C1—S1—N1	-104.55 (18)
C8—C9—C10—C11	1.0 (3)	C6—C1—S1—N1	74.00 (18)

supplementary materials

C11—C9—C10—C11 -179.88 (18)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1N \cdots O1 ⁱ	0.84 (2)	2.09 (2)	2.932 (2)	175 (2)

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

Fig. 1

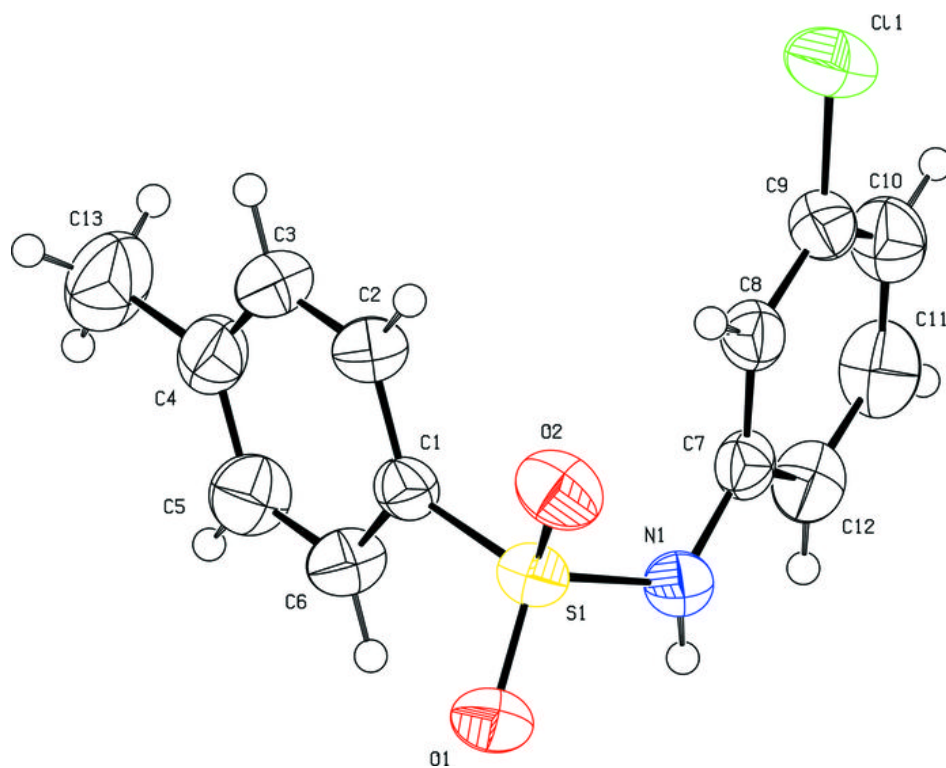


Fig. 2

