

4-Chloro-*N*-(2-methylbenzoyl)benzenesulfonamide

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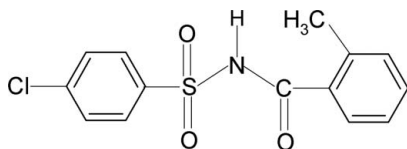
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Key indicators: single-crystal X-ray study; $T = 299$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.052; wR factor = 0.147; data-to-parameter ratio = 13.5.

In the title compound, $\text{C}_{14}\text{H}_{12}\text{ClNO}_3\text{S}$, the conformation of the $\text{N}-\text{H}$ bond in the $\text{C}-\text{SO}_2-\text{NH}-\text{C}(\text{O})$ segment is *anti* to the $\text{C}=\text{O}$ bond. The two aromatic rings are tilted relative to each other by 57.7 (1)°. In the crystal, molecules are linked by pairs of $\text{N}-\text{H}\cdots\text{O}(\text{S})$ hydrogen bonds, forming centrosymmetric dimers.

Related literature

For background to our study of the effect of ring and side-chain substituents on the crystal structures of *N*-aromatic sulfonamides and for similar structures, see: Gowda *et al.* (2010*a,b*); Suchetan *et al.* (2010).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{12}\text{ClNO}_3\text{S}$

$M_r = 309.76$

Triclinic, $P\bar{1}$

$a = 6.4328$ (9) Å

$b = 10.343$ (1) Å

$c = 11.207$ (1) Å

$\alpha = 78.78$ (1)°

$\beta = 73.84$ (1)°

$\gamma = 84.62$ (1)°

$V = 701.88$ (13) Å³

$Z = 2$
Cu $K\alpha$ radiation
 $\mu = 3.86$ mm⁻¹

$T = 299$ K
 $0.40 \times 0.15 \times 0.15$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
4317 measured reflections
2502 independent reflections

2283 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.039$
3 standard reflections every 120 min
intensity decay: 0.5%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.147$
 $S = 1.04$
2502 reflections
186 parameters
1 restraint

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.76$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.56$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{N1}-\text{H1N}\cdots\text{O1}^i$ | 0.82 (2) | 2.15 (2) | 2.925 (3) | 157 (3) |

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

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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5427).

References

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supplementary materials

Acta Cryst. (2011). E67, o22 [doi:10.1107/S1600536810050087]

4-Chloro-*N*-(2-methylbenzoyl)benzenesulfonamide

P. A. Suchetan, S. Foro and B. T. Gowda

Comment

As a part of studying the effect of ring and the side chain substituents on the crystal structures of *N*-aromatic sulfonamides (Gowda *et al.*, 2010*a,b*; Suchetan *et al.*, 2010), the structure of 4-Chloro-*N*-(2-methylbenzoyl)benzenesulfonamide (I) has been determined. The conformation of the N—H bond in the C—SO₂—NH—C(O) segment is *anti* to the C=O bond (Fig. 1), similar to those observed in *N*-(2-chlorobenzoyl)-4-chlorobenzenesulfonamide (II)(Gowda *et al.*, 2010*a*), *N*-(2-methylbenzoyl)-4-methylbenzenesulfonamide (III) (Gowda *et al.*, 2010*b*) and *N*-(2-methylbenzoyl)-2-chlorobenzenesulfonamide (IV) (Suchetan *et al.*, 2010).

The molecules are twisted at the *S* atom with the torsional angle of -69.2 (2)°, compared to those of 65.7 (2)° in (II), 67.7 (2)° in (III) and -64.0 (2)° in (IV).

The dihedral angles between the sulfonyl benzene ring and the —SO₂—NH—C—O segment is 87.2 (1)°, compared to the values of 88.5 (1)° in (II), 87.1 (1)° in (III) and 88.4 (1)° in (IV).

Furthermore, the dihedral angle between the sulfonyl and the benzoyl benzene rings is 57.7 (1)°, compared to the values of 58.0 (1)° in (II), 58.2 (1)° in (III) and 78.7 (1)° in (IV)

The packing of molecules linked by of N—H···O(S) hydrogen bonds (Table 1) is shown in Fig. 2.

Experimental

The title compound was prepared by refluxing a mixture of 2-methylbenzoic acid, 4-chlorobenzenesulfonamide and phosphorous oxy chloride for 3 h on a water bath. The resultant mixture was cooled and poured into ice cold water. The solid, *N*-(2-methylbenzoyl)-4-chlorobenzenesulfonamide obtained was filtered, washed thoroughly with water and then dissolved in sodium bicarbonate solution. The compound was later reprecipitated by acidifying the filtered solution with dilute HCl. It was filtered, dried and recrystallized.

Rod like colourless single crystals of the title compound used in X-ray diffraction studies were obtained by a slow evaporation of its toluene solution at room temperature.

Refinement

The H atom of the NH group was located in a difference map and 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 with C—H = 0.93–0.96 Å. All H atoms were refined with isotropic displacement parameters set to 1.2 times of the U_{eq} of the parent atom.

Figures

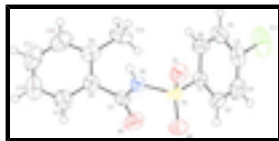


Fig. 1. Molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

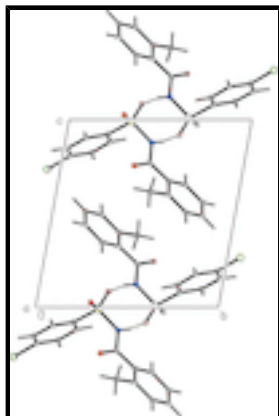


Fig. 2. Molecular packing in the title compound. Hydrogen bonds are shown as dashed lines.

4-Chloro-*N*-(2-methylbenzoyl)benzenesulfonamide

Crystal data

$C_{14}H_{12}ClNO_3S$

$M_r = 309.76$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 6.4328$ (9) Å

$b = 10.343$ (1) Å

$c = 11.207$ (1) Å

$\alpha = 78.78$ (1)°

$\beta = 73.84$ (1)°

$\gamma = 84.62$ (1)°

$V = 701.88$ (13) Å³

$Z = 2$

$F(000) = 320$

$D_x = 1.466$ Mg m⁻³

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

Cell parameters from 25 reflections

$\theta = 4.2$ – 22.5 °

$\mu = 3.86$ mm⁻¹

$T = 299$ K

Rod, colourless

$0.40 \times 0.15 \times 0.15$ mm

Data collection

Enraf–Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube
graphite

$\omega/2\theta$ scans

4317 measured reflections

2502 independent reflections

2283 reflections with $I > 2\sigma(I)$

$R_{int} = 0.039$

$\theta_{max} = 66.9$ °, $\theta_{min} = 4.2$ °

$h = -7 \rightarrow 4$

$k = -12 \rightarrow 12$

$l = -13 \rightarrow 13$

3 standard reflections every 120 min

intensity decay: 0.5%

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.052$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.147$ | $w = 1/[\sigma^2(F_o^2) + (0.1004P)^2 + 0.2209P]$ |
| $S = 1.04$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 2502 reflections | $(\Delta/\sigma)_{\max} < 0.001$ |
| 186 parameters | $\Delta\rho_{\max} = 0.76 \text{ e } \text{\AA}^{-3}$ |
| 1 restraint | $\Delta\rho_{\min} = -0.56 \text{ e } \text{\AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: <i>SHELXL97</i> (Sheldrick, 2008), $F_c^* = kF_c[1+0.001xF_c^2\lambda^3/\sin(2\theta)]^{-1/4}$ |
| | Extinction coefficient: 0.038 (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 | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|------------|--------------|----------------------------------|
| C1 | 0.2014 (3) | 0.2235 (2) | 0.92052 (19) | 0.0403 (5) |
| C2 | 0.3302 (4) | 0.1107 (2) | 0.9031 (2) | 0.0505 (5) |
| H2 | 0.4576 | 0.0973 | 0.9285 | 0.061* |
| C3 | 0.2687 (5) | 0.0180 (2) | 0.8478 (3) | 0.0605 (7) |
| H3 | 0.3518 | -0.0596 | 0.8373 | 0.073* |
| C4 | 0.0816 (5) | 0.0425 (2) | 0.8083 (2) | 0.0573 (6) |
| C5 | -0.0465 (4) | 0.1555 (3) | 0.8239 (3) | 0.0585 (6) |
| H5 | -0.1709 | 0.1703 | 0.7955 | 0.070* |
| C6 | 0.0124 (4) | 0.2463 (2) | 0.8821 (2) | 0.0512 (6) |
| H6 | -0.0739 | 0.3222 | 0.8955 | 0.061* |
| C7 | 0.4960 (3) | 0.4841 (2) | 0.7696 (2) | 0.0429 (5) |
| C8 | 0.4849 (4) | 0.6049 (2) | 0.6737 (2) | 0.0439 (5) |
| C9 | 0.3391 (4) | 0.6163 (2) | 0.6007 (2) | 0.0509 (6) |
| C10 | 0.3493 (5) | 0.7283 (3) | 0.5067 (2) | 0.0686 (8) |
| H10 | 0.2534 | 0.7394 | 0.4565 | 0.082* |

supplementary materials

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|------|--------------|--------------|--------------|------------|
| C11 | 0.4987 (6) | 0.8224 (3) | 0.4870 (3) | 0.0790 (9) |
| H11 | 0.5026 | 0.8959 | 0.4237 | 0.095* |
| C12 | 0.6422 (6) | 0.8090 (3) | 0.5598 (3) | 0.0768 (9) |
| H12 | 0.7427 | 0.8730 | 0.5460 | 0.092* |
| C13 | 0.6361 (5) | 0.7005 (3) | 0.6529 (3) | 0.0604 (6) |
| H13 | 0.7331 | 0.6907 | 0.7023 | 0.073* |
| C14 | 0.1835 (4) | 0.5118 (3) | 0.6165 (3) | 0.0661 (7) |
| H14A | 0.0593 | 0.5222 | 0.6857 | 0.079* |
| H14B | 0.2528 | 0.4265 | 0.6336 | 0.079* |
| H14C | 0.1385 | 0.5197 | 0.5405 | 0.079* |
| N1 | 0.3253 (3) | 0.47376 (17) | 0.87777 (17) | 0.0434 (4) |
| H1N | 0.229 (4) | 0.532 (2) | 0.883 (2) | 0.052* |
| O1 | 0.0959 (3) | 0.38337 (16) | 1.08307 (14) | 0.0554 (5) |
| O2 | 0.4730 (3) | 0.29640 (17) | 1.02583 (16) | 0.0581 (5) |
| O3 | 0.6391 (3) | 0.4000 (2) | 0.75586 (18) | 0.0674 (5) |
| Cl1 | 0.00535 (19) | -0.07131 (8) | 0.73539 (8) | 0.0981 (4) |
| S1 | 0.27927 (8) | 0.34281 (5) | 0.99049 (5) | 0.0427 (3) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C1 | 0.0376 (10) | 0.0380 (10) | 0.0435 (10) | -0.0018 (8) | -0.0103 (8) | -0.0029 (8) |
| C2 | 0.0508 (12) | 0.0421 (11) | 0.0553 (13) | 0.0059 (9) | -0.0157 (10) | -0.0023 (9) |
| C3 | 0.0726 (17) | 0.0405 (12) | 0.0620 (15) | 0.0046 (11) | -0.0101 (13) | -0.0089 (10) |
| C4 | 0.0726 (16) | 0.0482 (13) | 0.0489 (13) | -0.0211 (11) | -0.0081 (11) | -0.0063 (10) |
| C5 | 0.0505 (13) | 0.0616 (15) | 0.0664 (15) | -0.0146 (11) | -0.0199 (12) | -0.0062 (12) |
| C6 | 0.0405 (11) | 0.0467 (12) | 0.0681 (14) | 0.0009 (9) | -0.0176 (10) | -0.0111 (10) |
| C7 | 0.0394 (10) | 0.0468 (11) | 0.0468 (11) | -0.0035 (9) | -0.0170 (9) | -0.0094 (9) |
| C8 | 0.0441 (11) | 0.0461 (11) | 0.0410 (10) | -0.0014 (9) | -0.0094 (9) | -0.0094 (8) |
| C9 | 0.0502 (12) | 0.0609 (13) | 0.0424 (11) | 0.0084 (10) | -0.0136 (9) | -0.0144 (10) |
| C10 | 0.0818 (19) | 0.0769 (18) | 0.0469 (13) | 0.0194 (15) | -0.0237 (13) | -0.0114 (12) |
| C11 | 0.112 (3) | 0.0580 (16) | 0.0533 (15) | 0.0025 (16) | -0.0111 (16) | 0.0036 (12) |
| C12 | 0.101 (2) | 0.0567 (15) | 0.0668 (17) | -0.0248 (15) | -0.0129 (17) | 0.0003 (13) |
| C13 | 0.0670 (16) | 0.0575 (14) | 0.0588 (14) | -0.0147 (12) | -0.0176 (12) | -0.0082 (11) |
| C14 | 0.0479 (13) | 0.095 (2) | 0.0652 (16) | -0.0053 (13) | -0.0244 (12) | -0.0223 (14) |
| N1 | 0.0451 (10) | 0.0387 (9) | 0.0464 (10) | 0.0006 (7) | -0.0129 (8) | -0.0073 (7) |
| O1 | 0.0640 (10) | 0.0536 (9) | 0.0427 (8) | 0.0038 (8) | -0.0078 (7) | -0.0068 (7) |
| O2 | 0.0553 (10) | 0.0641 (10) | 0.0617 (10) | -0.0003 (8) | -0.0330 (8) | -0.0027 (8) |
| O3 | 0.0527 (10) | 0.0709 (11) | 0.0668 (11) | 0.0169 (9) | -0.0106 (9) | -0.0015 (9) |
| Cl1 | 0.1417 (9) | 0.0778 (6) | 0.0845 (6) | -0.0445 (5) | -0.0243 (6) | -0.0269 (4) |
| S1 | 0.0446 (4) | 0.0425 (4) | 0.0418 (3) | 0.0002 (2) | -0.0153 (2) | -0.0046 (2) |

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

| | | | |
|-------|-----------|---------|-----------|
| C1—C2 | 1.379 (3) | C9—C10 | 1.399 (4) |
| C1—C6 | 1.385 (3) | C9—C14 | 1.493 (4) |
| C1—S1 | 1.760 (2) | C10—C11 | 1.376 (5) |
| C2—C3 | 1.378 (4) | C10—H10 | 0.9300 |
| C2—H2 | 0.9300 | C11—C12 | 1.373 (5) |

| | | | |
|--------------|--------------|-----------------|-------------|
| C3—C4 | 1.381 (4) | C11—H11 | 0.9300 |
| C3—H3 | 0.9300 | C12—C13 | 1.371 (4) |
| C4—C5 | 1.377 (4) | C12—H12 | 0.9300 |
| C4—C11 | 1.732 (2) | C13—H13 | 0.9300 |
| C5—C6 | 1.376 (4) | C14—H14A | 0.9600 |
| C5—H5 | 0.9300 | C14—H14B | 0.9600 |
| C6—H6 | 0.9300 | C14—H14C | 0.9600 |
| C7—O3 | 1.205 (3) | N1—S1 | 1.6489 (18) |
| C7—N1 | 1.385 (3) | N1—H1N | 0.819 (17) |
| C7—C8 | 1.491 (3) | O1—S1 | 1.4280 (17) |
| C8—C9 | 1.389 (3) | O2—S1 | 1.4243 (17) |
| C8—C13 | 1.394 (3) | | |
| C2—C1—C6 | 121.3 (2) | C11—C10—C9 | 121.2 (3) |
| C2—C1—S1 | 119.81 (17) | C11—C10—H10 | 119.4 |
| C6—C1—S1 | 118.90 (17) | C9—C10—H10 | 119.4 |
| C3—C2—C1 | 119.5 (2) | C12—C11—C10 | 120.7 (3) |
| C3—C2—H2 | 120.3 | C12—C11—H11 | 119.7 |
| C1—C2—H2 | 120.3 | C10—C11—H11 | 119.7 |
| C2—C3—C4 | 118.8 (2) | C13—C12—C11 | 119.5 (3) |
| C2—C3—H3 | 120.6 | C13—C12—H12 | 120.2 |
| C4—C3—H3 | 120.6 | C11—C12—H12 | 120.2 |
| C5—C4—C3 | 122.1 (2) | C12—C13—C8 | 120.2 (3) |
| C5—C4—C11 | 118.6 (2) | C12—C13—H13 | 119.9 |
| C3—C4—C11 | 119.3 (2) | C8—C13—H13 | 119.9 |
| C6—C5—C4 | 119.0 (2) | C9—C14—H14A | 109.5 |
| C6—C5—H5 | 120.5 | C9—C14—H14B | 109.5 |
| C4—C5—H5 | 120.5 | H14A—C14—H14B | 109.5 |
| C5—C6—C1 | 119.3 (2) | C9—C14—H14C | 109.5 |
| C5—C6—H6 | 120.3 | H14A—C14—H14C | 109.5 |
| C1—C6—H6 | 120.3 | H14B—C14—H14C | 109.5 |
| O3—C7—N1 | 121.3 (2) | C7—N1—S1 | 125.08 (16) |
| O3—C7—C8 | 123.9 (2) | C7—N1—H1N | 118.8 (19) |
| N1—C7—C8 | 114.76 (18) | S1—N1—H1N | 115.6 (19) |
| C9—C8—C13 | 121.2 (2) | O2—S1—O1 | 119.23 (11) |
| C9—C8—C7 | 120.7 (2) | O2—S1—N1 | 110.43 (10) |
| C13—C8—C7 | 118.0 (2) | O1—S1—N1 | 103.74 (10) |
| C8—C9—C10 | 117.2 (2) | O2—S1—C1 | 108.92 (10) |
| C8—C9—C14 | 122.2 (2) | O1—S1—C1 | 109.13 (11) |
| C10—C9—C14 | 120.5 (2) | N1—S1—C1 | 104.33 (9) |
| C6—C1—C2—C3 | -0.8 (4) | C8—C9—C10—C11 | 0.5 (4) |
| S1—C1—C2—C3 | -179.73 (18) | C14—C9—C10—C11 | -176.9 (3) |
| C1—C2—C3—C4 | 1.6 (4) | C9—C10—C11—C12 | -0.2 (5) |
| C2—C3—C4—C5 | -0.8 (4) | C10—C11—C12—C13 | 0.0 (5) |
| C2—C3—C4—C11 | 178.54 (18) | C11—C12—C13—C8 | -0.2 (5) |
| C3—C4—C5—C6 | -0.8 (4) | C9—C8—C13—C12 | 0.5 (4) |
| C11—C4—C5—C6 | 179.78 (19) | C7—C8—C13—C12 | 175.5 (2) |
| C4—C5—C6—C1 | 1.7 (4) | O3—C7—N1—S1 | -7.6 (3) |
| C2—C1—C6—C5 | -0.9 (4) | C8—C7—N1—S1 | 171.71 (15) |

supplementary materials

| | | | |
|---------------|-------------|-------------|--------------|
| S1—C1—C6—C5 | 178.06 (18) | C7—N1—S1—O2 | 47.7 (2) |
| O3—C7—C8—C9 | 105.2 (3) | C7—N1—S1—O1 | 176.59 (17) |
| N1—C7—C8—C9 | -74.1 (3) | C7—N1—S1—C1 | -69.16 (19) |
| O3—C7—C8—C13 | -69.9 (3) | C2—C1—S1—O2 | -3.0 (2) |
| N1—C7—C8—C13 | 110.9 (2) | C6—C1—S1—O2 | 178.07 (18) |
| C13—C8—C9—C10 | -0.7 (3) | C2—C1—S1—O1 | -134.65 (18) |
| C7—C8—C9—C10 | -175.5 (2) | C6—C1—S1—O1 | 46.4 (2) |
| C13—C8—C9—C14 | 176.7 (2) | C2—C1—S1—N1 | 114.98 (19) |
| C7—C8—C9—C14 | 1.8 (3) | C6—C1—S1—N1 | -64.0 (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 | 0.82 (2) | 2.15 (2) | 2.925 (3) | 157 (3) |

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

Fig. 1

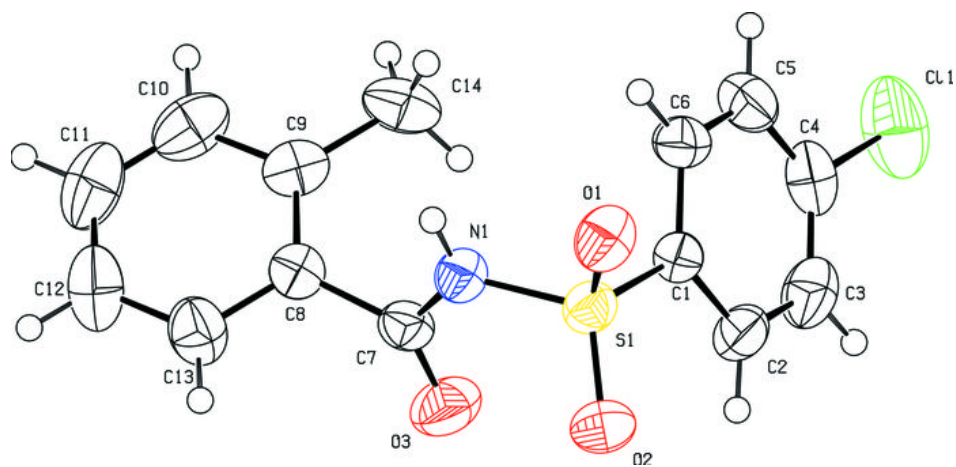


Fig. 2

