

N-(2,6-Dichlorophenyl)-4-methylbenzenesulfonamide

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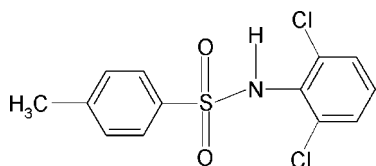
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.038; wR factor = 0.102; data-to-parameter ratio = 16.5.

In the title compound, $\text{C}_{13}\text{H}_{11}\text{Cl}_2\text{NO}_2\text{S}$, the molecule is bent at the S atom with a $\text{C}-\text{SO}_2-\text{NH}-\text{C}$ torsion angle of -90.4 (2)°. The sulfonyl benzene and the aniline benzene rings are tilted relative to each other by 51.7 (1)°. In the crystal, molecules are linked by $\text{N}-\text{H}\cdots\text{O}$ interactions into chains with graph-set notation $C(4)$ along $[100]$.

Related literature

For our study of the effect of substituents on the structures of N -(aryl)arylsulfonamides, see: Gowda *et al.* (2009); Nirmala *et al.* (2010); Shakuntala *et al.* (2010). For related structures, see: Gelbrich *et al.* (2007); Perlovich *et al.* (2006). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$\text{C}_{13}\text{H}_{11}\text{Cl}_2\text{NO}_2\text{S}$

$M_r = 316.19$

Monoclinic, $P2_1/n$

$a = 5.0456$ (6) Å

$b = 17.128$ (2) Å

$c = 16.540$ (2) Å

$\beta = 97.13$ (1)°

$V = 1418.4$ (3) Å³

$Z = 4$

Mo $K\alpha$ radiation

$\mu = 0.60$ mm⁻¹

$T = 293$ K

$0.55 \times 0.28 \times 0.25$ mm

Data collection

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

Diffraction, 2009)
 $T_{\min} = 0.734$, $T_{\max} = 0.864$
5631 measured reflections
2904 independent reflections
2493 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.011$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$

$wR(F^2) = 0.102$

$S = 1.05$

2904 reflections

176 parameters

1 restraint

H atoms treated by a mixture of independent and constrained refinement

$\Delta\rho_{\text{max}} = 0.30$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.48$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^i$	0.83 (2)	2.16 (2)	2.971 (2)	165 (2)

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

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: BX2335).

References

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supplementary materials

Acta Cryst. (2011). E67, o142 [doi:10.1107/S1600536810051792]

***N*-(2,6-Dichlorophenyl)-4-methylbenzenesulfonamide**

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Comment

In the present work, as part of a study of the substituent effects on the crystal structures of *N*-(aryl)arylsulfonamides (Gowda *et al.*, 2009; Nirmala *et al.*, 2010; Shakuntala *et al.*, 2010), the structure of *N*-(2,6-dichlorophenyl)-4-methylbenzenesulfonamide (I) has been determined (Fig. 1). The conformation of the N—H bond orients away from the two *ortho*-chloro groups in the adjacent benzene ring.

The molecule in (I) is bent at the S atom with the C—SO₂—NH—C torsion angle of -90.4 (2)°, compared to the values of 88.0 (2)° in *N*-(2,6-dimethylphenyl)-4-methylbenzenesulfonamide (II) (Nirmala *et al.*, 2010), 65.4 (2)° (molecule 1) and -61.7 (2)° (molecule 2) in *N*-(2,3-dichlorophenyl)-4-methylbenzenesulfonamide (III) (Shakuntala *et al.*, 2010) and 69.3 (4)° in *N*-(3,5-dichlorophenyl)-4-methylbenzenesulfonamide (IV) (Gowda *et al.*, 2009).

The two benzene rings in (I) are tilted relative to each other by 51.7 (1)°, compared to the values of 49.8 (1)° in (II), 76.0 (1)° (molecule 1) and 79.9 (1)° (molecule 2) in (III) and 79.6 (1)° in (IV).

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

In the crystal, molecules are linked by N—H···O interaction into chain with graph-set notation C(4) along [100] (Bernstein *et al.*, 1995), 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 4-methylbenzenesulfonylchloride was treated with 2,6-dichloroaniline 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 *N*-(2,6-dichlorophenyl)-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.

Rod like colourless 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 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. H-atoms were refined with isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent atom).

Figures

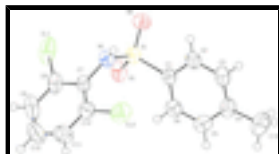


Fig. 1. Molecular structure of (I), showing the atom labelling scheme and displacement ellipsoids are drawn at the 50% probability level.

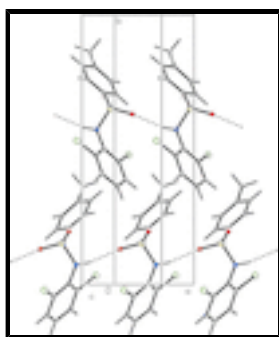


Fig. 2. Molecular packing of (I) with hydrogen bonding shown as dashed lines.

N-(2,6-Dichlorophenyl)-4-methylbenzenesulfonamide

Crystal data

$C_{13}H_{11}Cl_2NO_2S$

$M_r = 316.19$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1n$

$a = 5.0456$ (6) Å

$b = 17.128$ (2) Å

$c = 16.540$ (2) Å

$\beta = 97.13$ (1)°

$V = 1418.4$ (3) Å³

$Z = 4$

$F(000) = 648$

$D_x = 1.481$ Mg m⁻³

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

Cell parameters from 3098 reflections

$\theta = 3.4$ – 27.7 °

$\mu = 0.60$ mm⁻¹

$T = 293$ K

Rod, colourless

$0.55 \times 0.28 \times 0.25$ mm

Data collection

Oxford Diffraction Xcalibur
diffractometer with a Sapphire CCD detector

Radiation source: fine-focus sealed tube

graphite

Rotation method data acquisition using ω and ϕ
scans

2904 independent reflections

2493 reflections with $I > 2\sigma(I)$

$R_{int} = 0.011$

$\theta_{max} = 26.4$ °, $\theta_{min} = 3.4$ °

Absorption correction: multi-scan
(*CrysAlis RED*; Oxford Diffraction, 2009)
 $T_{\min} = 0.734$, $T_{\max} = 0.864$
5631 measured reflections

$h = -3 \rightarrow 6$
 $k = -21 \rightarrow 18$
 $l = -20 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.102$
 $S = 1.05$
2904 reflections
176 parameters
1 restraint

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
 $w = 1/[\sigma^2(F_o^2) + (0.0469P)^2 + 0.7136P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.028$
 $\Delta\rho_{\max} = 0.30 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.48 \text{ e } \text{Å}^{-3}$

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 (Å^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.0019 (4)	0.70942 (11)	0.33030 (12)	0.0361 (4)
C2	-0.2076 (4)	0.76146 (13)	0.32461 (14)	0.0471 (5)
H2	-0.3280	0.7645	0.2773	0.057*
C3	-0.2359 (5)	0.80913 (14)	0.39037 (16)	0.0559 (6)
H3	-0.3784	0.8439	0.3869	0.067*
C4	-0.0594 (5)	0.80678 (14)	0.46089 (15)	0.0537 (6)
C5	0.1467 (5)	0.75322 (15)	0.46540 (15)	0.0582 (6)
H5	0.2656	0.7498	0.5130	0.070*
C6	0.1799 (5)	0.70457 (14)	0.40068 (14)	0.0508 (5)
H6	0.3202	0.6690	0.4045	0.061*
C7	-0.0179 (4)	0.50337 (11)	0.28274 (13)	0.0387 (4)

supplementary materials

C8	0.1533 (5)	0.45552 (13)	0.24465 (16)	0.0521 (6)
C9	0.2543 (6)	0.38721 (15)	0.2808 (2)	0.0736 (8)
H9	0.3728	0.3570	0.2554	0.088*
C10	0.1807 (7)	0.36409 (16)	0.3536 (2)	0.0793 (9)
H10	0.2488	0.3180	0.3776	0.095*
C11	0.0071 (6)	0.40828 (15)	0.39182 (17)	0.0674 (7)
H11	-0.0466	0.3917	0.4408	0.081*
C12	-0.0877 (4)	0.47785 (13)	0.35667 (14)	0.0471 (5)
C13	-0.0895 (7)	0.8623 (2)	0.5296 (2)	0.0855 (9)
H13A	-0.2690	0.8819	0.5242	0.103*
H13B	0.0328	0.9050	0.5278	0.103*
H13C	-0.0513	0.8354	0.5807	0.103*
N1	-0.1228 (3)	0.57274 (10)	0.24474 (10)	0.0388 (4)
H1N	-0.286 (3)	0.5809 (14)	0.2422 (14)	0.047*
O1	0.3260 (3)	0.63094 (9)	0.25207 (10)	0.0495 (4)
O2	-0.0611 (3)	0.69646 (9)	0.17450 (9)	0.0538 (4)
C11	0.23560 (17)	0.47929 (4)	0.14965 (5)	0.0797 (3)
C12	-0.30503 (14)	0.53278 (4)	0.40686 (4)	0.0675 (2)
S1	0.05144 (9)	0.65342 (3)	0.24407 (3)	0.03684 (15)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0349 (9)	0.0306 (9)	0.0445 (10)	-0.0027 (7)	0.0116 (8)	-0.0009 (8)
C2	0.0412 (11)	0.0452 (12)	0.0547 (12)	0.0063 (9)	0.0046 (9)	-0.0015 (10)
C3	0.0526 (13)	0.0481 (13)	0.0695 (15)	0.0120 (10)	0.0170 (12)	-0.0079 (11)
C4	0.0646 (14)	0.0481 (12)	0.0520 (13)	-0.0031 (11)	0.0211 (11)	-0.0068 (10)
C5	0.0648 (15)	0.0631 (15)	0.0456 (12)	0.0016 (12)	0.0021 (10)	-0.0030 (11)
C6	0.0508 (12)	0.0485 (12)	0.0526 (12)	0.0097 (10)	0.0045 (10)	0.0011 (10)
C7	0.0349 (10)	0.0309 (9)	0.0502 (11)	-0.0052 (8)	0.0040 (8)	-0.0045 (8)
C8	0.0499 (12)	0.0367 (11)	0.0716 (15)	-0.0060 (9)	0.0150 (11)	-0.0127 (10)
C9	0.0655 (16)	0.0384 (13)	0.117 (3)	0.0088 (12)	0.0133 (16)	-0.0165 (15)
C10	0.085 (2)	0.0417 (14)	0.105 (3)	0.0090 (14)	-0.0109 (18)	0.0073 (15)
C11	0.0817 (18)	0.0513 (14)	0.0654 (16)	-0.0093 (13)	-0.0055 (13)	0.0132 (12)
C12	0.0463 (12)	0.0438 (11)	0.0502 (12)	-0.0056 (9)	0.0021 (9)	-0.0019 (9)
C13	0.104 (2)	0.086 (2)	0.0706 (18)	0.0060 (19)	0.0242 (17)	-0.0281 (17)
N1	0.0267 (7)	0.0374 (9)	0.0524 (10)	-0.0016 (7)	0.0047 (7)	-0.0004 (7)
O1	0.0310 (7)	0.0471 (8)	0.0728 (10)	-0.0049 (6)	0.0158 (7)	-0.0143 (8)
O2	0.0686 (10)	0.0477 (9)	0.0462 (8)	0.0004 (8)	0.0111 (7)	0.0086 (7)
C11	0.1007 (6)	0.0582 (4)	0.0912 (5)	-0.0164 (4)	0.0558 (4)	-0.0252 (3)
C12	0.0720 (4)	0.0775 (4)	0.0574 (4)	0.0006 (3)	0.0258 (3)	-0.0011 (3)
S1	0.0334 (2)	0.0338 (3)	0.0449 (3)	-0.00172 (19)	0.01091 (19)	-0.00076 (19)

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

C1—C2	1.377 (3)	C8—C11	1.723 (3)
C1—C6	1.382 (3)	C9—C10	1.363 (4)
C1—S1	1.7620 (19)	C9—H9	0.9300
C2—C3	1.381 (3)	C10—C11	1.370 (4)

C2—H2	0.9300	C10—H10	0.9300
C3—C4	1.377 (4)	C11—C12	1.385 (3)
C3—H3	0.9300	C11—H11	0.9300
C4—C5	1.382 (3)	C12—C12	1.733 (2)
C4—C13	1.504 (3)	C13—H13A	0.9600
C5—C6	1.383 (3)	C13—H13B	0.9600
C5—H5	0.9300	C13—H13C	0.9600
C6—H6	0.9300	N1—S1	1.6386 (17)
C7—C12	1.385 (3)	N1—H1N	0.830 (16)
C7—C8	1.396 (3)	O1—S1	1.4283 (14)
C7—N1	1.416 (3)	O2—S1	1.4241 (16)
C8—C9	1.382 (4)		
C2—C1—C6	120.64 (19)	C8—C9—H9	119.9
C2—C1—S1	118.80 (16)	C9—C10—C11	120.5 (3)
C6—C1—S1	120.44 (15)	C9—C10—H10	119.8
C1—C2—C3	118.8 (2)	C11—C10—H10	119.8
C1—C2—H2	120.6	C10—C11—C12	119.3 (3)
C3—C2—H2	120.6	C10—C11—H11	120.4
C4—C3—C2	122.0 (2)	C12—C11—H11	120.4
C4—C3—H3	119.0	C7—C12—C11	122.0 (2)
C2—C3—H3	119.0	C7—C12—C12	119.81 (17)
C3—C4—C5	117.9 (2)	C11—C12—C12	118.2 (2)
C3—C4—C13	120.4 (2)	C4—C13—H13A	109.5
C5—C4—C13	121.6 (3)	C4—C13—H13B	109.5
C4—C5—C6	121.4 (2)	H13A—C13—H13B	109.5
C4—C5—H5	119.3	C4—C13—H13C	109.5
C6—C5—H5	119.3	H13A—C13—H13C	109.5
C1—C6—C5	119.2 (2)	H13B—C13—H13C	109.5
C1—C6—H6	120.4	C7—N1—S1	122.66 (13)
C5—C6—H6	120.4	C7—N1—H1N	118.4 (17)
C12—C7—C8	116.9 (2)	S1—N1—H1N	112.8 (17)
C12—C7—N1	122.35 (18)	O2—S1—O1	119.96 (10)
C8—C7—N1	120.7 (2)	O2—S1—N1	106.35 (9)
C9—C8—C7	121.2 (3)	O1—S1—N1	106.68 (9)
C9—C8—C11	118.5 (2)	O2—S1—C1	106.85 (10)
C7—C8—C11	120.36 (19)	O1—S1—C1	107.77 (9)
C10—C9—C8	120.1 (3)	N1—S1—C1	108.88 (9)
C10—C9—H9	119.9		
C6—C1—C2—C3	0.4 (3)	C8—C7—C12—C11	0.1 (3)
S1—C1—C2—C3	-175.68 (18)	N1—C7—C12—C11	177.3 (2)
C1—C2—C3—C4	0.8 (4)	C8—C7—C12—C12	-178.62 (16)
C2—C3—C4—C5	-1.7 (4)	N1—C7—C12—C12	-1.4 (3)
C2—C3—C4—C13	177.1 (3)	C10—C11—C12—C7	1.7 (4)
C3—C4—C5—C6	1.5 (4)	C10—C11—C12—C12	-179.5 (2)
C13—C4—C5—C6	-177.2 (3)	C12—C7—N1—S1	104.6 (2)
C2—C1—C6—C5	-0.6 (3)	C8—C7—N1—S1	-78.4 (2)
S1—C1—C6—C5	175.45 (18)	C7—N1—S1—O2	154.79 (16)
C4—C5—C6—C1	-0.4 (4)	C7—N1—S1—O1	25.65 (18)

supplementary materials

C12—C7—C8—C9	-2.0 (3)	C7—N1—S1—C1	-90.40 (17)
N1—C7—C8—C9	-179.2 (2)	C2—C1—S1—O2	25.70 (19)
C12—C7—C8—C11	176.09 (16)	C6—C1—S1—O2	-150.40 (17)
N1—C7—C8—C11	-1.1 (3)	C2—C1—S1—O1	155.87 (16)
C7—C8—C9—C10	2.1 (4)	C6—C1—S1—O1	-20.2 (2)
C11—C8—C9—C10	-176.0 (2)	C2—C1—S1—N1	-88.79 (17)
C8—C9—C10—C11	-0.2 (5)	C6—C1—S1—N1	95.11 (18)
C9—C10—C11—C12	-1.7 (4)		

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	0.83 (2)	2.16 (2)	2.971 (2)	165 (2)

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

Fig. 1

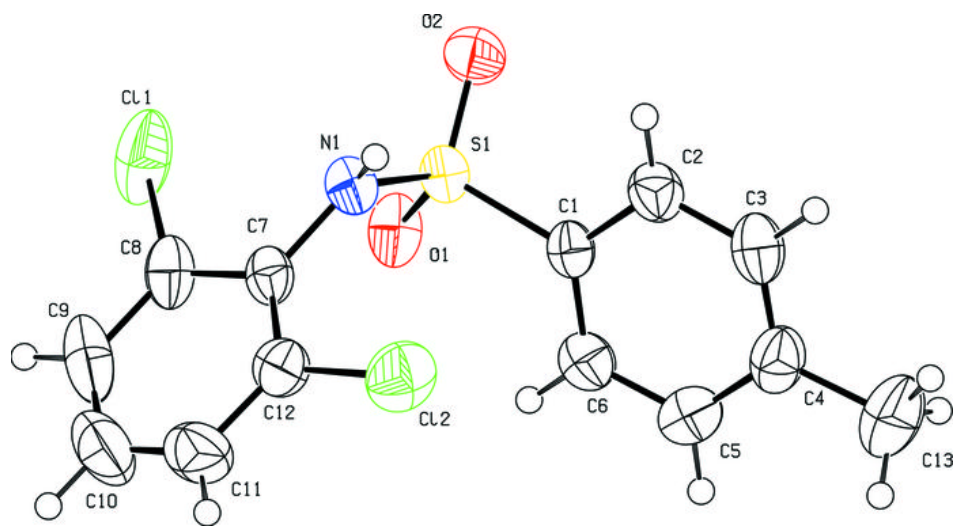


Fig. 2

