

4-Chloro-*N*-(2-chlorophenyl)-2-methylbenzenesulfonamide

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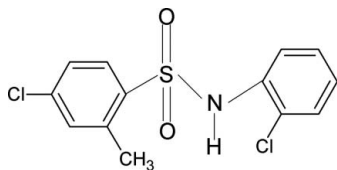
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Key indicators: single-crystal X-ray study; $T = 299$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.083; wR factor = 0.236; data-to-parameter ratio = 13.6.

In the crystal structure of the title compound, $\text{C}_{13}\text{H}_{11}\text{Cl}_2\text{NO}_2\text{S}$, the conformations of the $\text{N}-\text{C}$ bond in the $\text{C}-\text{SO}_2-\text{NH}-\text{C}$ segment are *trans* and *gauche* with respect to the $\text{S}=\text{O}$ bonds. The $\text{C}-\text{S}(\text{O}_2)-\text{N}(\text{H})-\text{C}$ torsion angle is 74.8 (4)°, indicating that the molecule is bent at the S atom. In the crystal structure, inversion dimers linked by pairs of $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds occur. An intramolecular $\text{N}-\text{H}\cdots\text{Cl}$ interaction is also present.

Related literature

For related structures of *N*-(aryl)-arylsulfonamides, see: Gelbrich *et al.* (2007); Gowda *et al.* (2009a,b); Perlovich *et al.* (2006).



Experimental

Crystal data

$\text{C}_{13}\text{H}_{11}\text{Cl}_2\text{NO}_2\text{S}$
 $M_r = 316.19$
Triclinic, $P\bar{1}$

$a = 8.089$ (2) Å
 $b = 8.096$ (2) Å
 $c = 10.946$ (3) Å

$\alpha = 96.00$ (1)°
 $\beta = 97.11$ (2)°
 $\gamma = 105.67$ (2)°
 $V = 677.7$ (3) Å³
 $Z = 2$

Cu $K\alpha$ radiation
 $\mu = 5.73$ mm⁻¹
 $T = 299$ K
 $0.45 \times 0.33 \times 0.08$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.150$, $T_{\max} = 0.640$
2684 measured reflections

2414 independent reflections
1932 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.037$
3 standard reflections
frequency: 120 min
intensity decay: 1.0%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.083$
 $wR(F^2) = 0.236$
 $S = 1.05$
2414 reflections
177 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.60$ e Å⁻³
 $\Delta\rho_{\min} = -0.67$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O2}^i$	0.88 (5)	2.17 (5)	2.994 (5)	157 (4)
$\text{N1}-\text{H1N}\cdots\text{Cl2}$	0.88 (5)	2.67 (5)	3.011 (4)	104 (4)

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

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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2384).

References

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supplementary materials

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Comment

In the present work, as part of a study of substituent effects on the structures of *N*-(aryl)-arylsulfonamides (Gowda *et al.* 2008*a,b*, 2009), the structure of 2-methyl-4-chloro-*N*-(2-chlorophenyl)benzenesulfonamide (I) has been determined. The conformations of the N—C bond in the C—S(O₂)—N(H)—C segment are *trans* and *gauche* with respect to the S=O bonds (Fig. 1). The torsion angle of C1—S1—N1—C1 is 74.8 (4)°, indicating the molecule is bent at the S1 atom. The two benzene rings are tilted relative to each other by 45.5 (2)°, compared with the values of 86.6 (2)° (molecule 1) and 83.0 (2)° (molecule 2), in the two independent molecules of 2-methyl-4-chloro-*N*-(phenyl)-benzenesulfonamide (II) (Gowda *et al.*, 2009). Bond distance parameters in (I) are similar to those observed in (II), 2,4-dimethyl-*N*-(phenyl)benzenesulfonamide (Gowda *et al.*, 2008*b*) and other aryl sulfonamides (Perlovich *et al.*, 2006; Gelbrich *et al.*, 2007). The crystal structure comprises the packing of centrosymmetric molecules connected via N—H⋯O hydrogen bonds (Table 1 & Fig. 2).

Experimental

m-Chlorotoluene (10 ml) in chloroform (40 ml) was treated dropwise with chlorosulfonic acid (25 ml) at 0° C. After the initial evolution of HCl 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-methyl-4-chlorobenzenesulfonylchloride was treated with 2-chloroaniline in a 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-methyl-4-chloro-*N*-(2-chlorophenyl)-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. The single crystals used in X-ray diffraction studies were grown from an ethanolic solution by slow evaporation at room temperature.

Refinement

The N-bound H atom was located in difference map and its positional parameters refined; N—H = 0.88 (5) Å. The remaining H atoms were positioned with idealized geometries 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 × U_{eq} (parent atom).

Figures

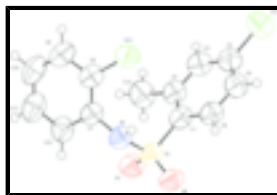


Fig. 1. Molecular structure of (I), showing the atom labeling scheme. The displacement ellipsoids are drawn at the 50% probability level. The H atoms are represented as small spheres of arbitrary radii.

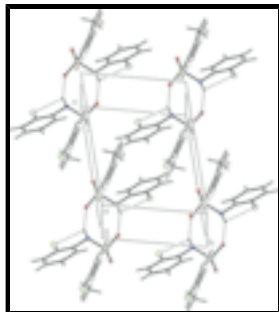


Fig. 2. Molecular packing of (I) with hydrogen bonding shown as dashed lines.

4-Chloro-*N*-(2-chlorophenyl)-2-methylbenzenesulfonamide

Crystal data

$C_{13}H_{11}Cl_2NO_2S$	$Z = 2$
$M_r = 316.19$	$F_{000} = 324$
Triclinic, $P\bar{1}$	$D_x = 1.549 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Cu $K\alpha$ radiation
$a = 8.089 (2) \text{ \AA}$	$\lambda = 1.54180 \text{ \AA}$
$b = 8.096 (2) \text{ \AA}$	Cell parameters from 25 reflections
$c = 10.946 (3) \text{ \AA}$	$\theta = 5.7\text{--}19.7^\circ$
$\alpha = 96.00 (1)^\circ$	$\mu = 5.73 \text{ mm}^{-1}$
$\beta = 97.11 (2)^\circ$	$T = 299 \text{ K}$
$\gamma = 105.67 (2)^\circ$	Plate, colourless
$V = 677.7 (3) \text{ \AA}^3$	$0.45 \times 0.33 \times 0.08 \text{ mm}$

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.037$
Radiation source: fine-focus sealed tube	$\theta_{\text{max}} = 66.9^\circ$
Monochromator: graphite	$\theta_{\text{min}} = 4.1^\circ$
$T = 299 \text{ K}$	$h = -9 \rightarrow 1$
ω scans	$k = -9 \rightarrow 9$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$l = -12 \rightarrow 13$
$T_{\text{min}} = 0.150$, $T_{\text{max}} = 0.640$	3 standard reflections
2684 measured reflections	every 120 min
2414 independent reflections	intensity decay: 1.0%
1932 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement

$R[F^2 > 2\sigma(F^2)] = 0.083$	$w = 1/[\sigma^2(F_o^2) + (0.179P)^2 + 0.1079P]$
$wR(F^2) = 0.236$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{\max} = 0.008$
2414 reflections	$\Delta\rho_{\max} = 0.60 \text{ e } \text{\AA}^{-3}$
177 parameters	$\Delta\rho_{\min} = -0.67 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.039 (6)

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	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.35954 (18)	0.30027 (19)	0.56480 (12)	0.0932 (5)
C12	0.70514 (17)	0.48939 (15)	0.15780 (13)	0.0901 (5)
S1	0.71199 (12)	-0.00409 (12)	0.15693 (9)	0.0651 (4)
O1	0.8667 (4)	-0.0424 (4)	0.2054 (3)	0.0761 (8)
O2	0.5732 (4)	-0.1359 (4)	0.0795 (3)	0.0757 (8)
N1	0.7673 (4)	0.1494 (4)	0.0701 (3)	0.0659 (9)
H1N	0.678 (7)	0.178 (6)	0.034 (5)	0.079*
C1	0.6225 (5)	0.0807 (5)	0.2792 (4)	0.0637 (9)
C2	0.7215 (5)	0.1757 (5)	0.3907 (4)	0.0660 (10)
C3	0.6344 (6)	0.2423 (6)	0.4761 (4)	0.0719 (11)
H3	0.6974	0.3069	0.5507	0.086*
C4	0.4585 (6)	0.2158 (6)	0.4536 (4)	0.0708 (10)
C5	0.3607 (6)	0.1217 (6)	0.3426 (4)	0.0732 (11)
H5	0.2410	0.1036	0.3271	0.088*
C6	0.4441 (5)	0.0565 (5)	0.2569 (4)	0.0686 (10)
H6	0.3800	-0.0057	0.1818	0.082*
C7	0.9177 (5)	0.2931 (5)	0.1116 (4)	0.0625 (9)
C8	0.9064 (6)	0.4560 (5)	0.1521 (4)	0.0693 (10)
C9	1.0553 (7)	0.5950 (6)	0.1862 (4)	0.0812 (12)
H9	1.0466	0.7053	0.2114	0.097*
C10	1.2140 (6)	0.5692 (7)	0.1826 (5)	0.0900 (15)
H10	1.3139	0.6619	0.2069	0.108*

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C11	1.2280 (6)	0.4087 (7)	0.1436 (5)	0.0900 (15)
H11	1.3372	0.3921	0.1427	0.108*
C12	1.0796 (6)	0.2698 (6)	0.1052 (5)	0.0760 (11)
H12	1.0891	0.1615	0.0753	0.091*
C13	0.9170 (6)	0.2092 (8)	0.4254 (4)	0.0875 (14)
H13A	0.9430	0.1011	0.4307	0.105*
H13B	0.9567	0.2823	0.5044	0.105*
H13C	0.9747	0.2655	0.3630	0.105*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0896 (9)	0.1076 (10)	0.0887 (9)	0.0410 (7)	0.0150 (6)	0.0072 (7)
C12	0.0869 (8)	0.0730 (8)	0.1186 (10)	0.0325 (6)	0.0255 (7)	0.0141 (6)
S1	0.0608 (6)	0.0547 (6)	0.0758 (7)	0.0165 (4)	-0.0011 (4)	0.0066 (4)
O1	0.0695 (18)	0.0690 (17)	0.093 (2)	0.0285 (14)	0.0023 (15)	0.0136 (14)
O2	0.0690 (17)	0.0601 (16)	0.0872 (19)	0.0119 (13)	-0.0024 (14)	-0.0020 (13)
N1	0.0591 (18)	0.0635 (19)	0.0692 (19)	0.0144 (14)	-0.0032 (14)	0.0074 (14)
C1	0.058 (2)	0.060 (2)	0.073 (2)	0.0185 (16)	-0.0008 (17)	0.0152 (17)
C2	0.061 (2)	0.071 (2)	0.065 (2)	0.0205 (17)	-0.0021 (17)	0.0108 (17)
C3	0.072 (2)	0.073 (2)	0.065 (2)	0.0210 (19)	-0.0043 (18)	0.0072 (18)
C4	0.075 (2)	0.070 (2)	0.071 (2)	0.0278 (19)	0.0071 (19)	0.0153 (18)
C5	0.062 (2)	0.079 (3)	0.079 (3)	0.0260 (19)	0.0022 (19)	0.013 (2)
C6	0.061 (2)	0.067 (2)	0.072 (2)	0.0168 (18)	-0.0026 (18)	0.0084 (17)
C7	0.058 (2)	0.062 (2)	0.063 (2)	0.0149 (16)	-0.0013 (15)	0.0093 (15)
C8	0.072 (2)	0.065 (2)	0.068 (2)	0.0178 (18)	0.0016 (18)	0.0123 (17)
C9	0.087 (3)	0.067 (2)	0.077 (3)	0.006 (2)	0.000 (2)	0.0105 (19)
C10	0.070 (3)	0.086 (3)	0.095 (3)	-0.005 (2)	-0.011 (2)	0.025 (2)
C11	0.061 (2)	0.094 (3)	0.109 (4)	0.014 (2)	-0.005 (2)	0.032 (3)
C12	0.062 (2)	0.080 (3)	0.089 (3)	0.023 (2)	0.008 (2)	0.022 (2)
C13	0.062 (3)	0.114 (4)	0.075 (3)	0.022 (2)	-0.008 (2)	-0.003 (2)

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

C11—C4	1.719 (5)	C5—H5	0.9300
C12—C8	1.728 (5)	C6—H6	0.9300
S1—O1	1.423 (3)	C7—C8	1.378 (6)
S1—O2	1.430 (3)	C7—C12	1.381 (6)
S1—N1	1.643 (4)	C8—C9	1.388 (6)
S1—C1	1.761 (4)	C9—C10	1.360 (8)
N1—C7	1.424 (5)	C9—H9	0.9300
N1—H1N	0.88 (5)	C10—C11	1.364 (8)
C1—C6	1.390 (5)	C10—H10	0.9300
C1—C2	1.398 (5)	C11—C12	1.389 (6)
C2—C3	1.387 (7)	C11—H11	0.9300
C2—C13	1.522 (6)	C12—H12	0.9300
C3—C4	1.366 (6)	C13—H13A	0.9600
C3—H3	0.9300	C13—H13B	0.9600
C4—C5	1.388 (6)	C13—H13C	0.9600

C5—C6	1.364 (7)		
O1—S1—O2	120.08 (19)	C1—C6—H6	119.2
O1—S1—N1	107.27 (18)	C8—C7—C12	119.1 (4)
O2—S1—N1	104.75 (18)	C8—C7—N1	122.1 (4)
O1—S1—C1	109.99 (19)	C12—C7—N1	118.8 (4)
O2—S1—C1	106.96 (19)	C7—C8—C9	120.6 (4)
N1—S1—C1	107.03 (18)	C7—C8—C12	120.0 (3)
C7—N1—S1	120.4 (3)	C9—C8—C12	119.4 (4)
C7—N1—H1N	114 (3)	C10—C9—C8	119.6 (5)
S1—N1—H1N	113 (3)	C10—C9—H9	120.2
C6—C1—C2	120.1 (4)	C8—C9—H9	120.2
C6—C1—S1	116.1 (3)	C9—C10—C11	120.6 (4)
C2—C1—S1	123.7 (3)	C9—C10—H10	119.7
C3—C2—C1	117.4 (4)	C11—C10—H10	119.7
C3—C2—C13	118.0 (4)	C10—C11—C12	120.2 (5)
C1—C2—C13	124.6 (4)	C10—C11—H11	119.9
C4—C3—C2	121.9 (4)	C12—C11—H11	119.9
C4—C3—H3	119.0	C7—C12—C11	119.8 (5)
C2—C3—H3	119.0	C7—C12—H12	120.1
C3—C4—C5	120.6 (4)	C11—C12—H12	120.1
C3—C4—C11	119.2 (4)	C2—C13—H13A	109.5
C5—C4—C11	120.3 (4)	C2—C13—H13B	109.5
C6—C5—C4	118.5 (4)	H13A—C13—H13B	109.5
C6—C5—H5	120.8	C2—C13—H13C	109.5
C4—C5—H5	120.8	H13A—C13—H13C	109.5
C5—C6—C1	121.6 (4)	H13B—C13—H13C	109.5
C5—C6—H6	119.2		
O1—S1—N1—C7	-43.2 (4)	C11—C4—C5—C6	-179.9 (3)
O2—S1—N1—C7	-171.9 (3)	C4—C5—C6—C1	0.7 (6)
C1—S1—N1—C7	74.8 (4)	C2—C1—C6—C5	-0.9 (6)
O1—S1—C1—C6	-153.8 (3)	S1—C1—C6—C5	-177.6 (3)
O2—S1—C1—C6	-21.8 (3)	S1—N1—C7—C8	-106.8 (4)
N1—S1—C1—C6	90.0 (3)	S1—N1—C7—C12	76.4 (5)
O1—S1—C1—C2	29.7 (4)	C12—C7—C8—C9	-0.1 (6)
O2—S1—C1—C2	161.6 (3)	N1—C7—C8—C9	-176.9 (4)
N1—S1—C1—C2	-86.6 (4)	C12—C7—C8—C12	178.6 (3)
C6—C1—C2—C3	0.2 (6)	N1—C7—C8—C12	1.9 (6)
S1—C1—C2—C3	176.7 (3)	C7—C8—C9—C10	-1.7 (7)
C6—C1—C2—C13	179.5 (4)	C12—C8—C9—C10	179.6 (4)
S1—C1—C2—C13	-4.0 (6)	C8—C9—C10—C11	1.2 (8)
C1—C2—C3—C4	0.6 (6)	C9—C10—C11—C12	1.0 (8)
C13—C2—C3—C4	-178.8 (4)	C8—C7—C12—C11	2.3 (6)
C2—C3—C4—C5	-0.7 (7)	N1—C7—C12—C11	179.2 (4)
C2—C3—C4—C11	179.2 (3)	C10—C11—C12—C7	-2.7 (7)
C3—C4—C5—C6	0.1 (6)		

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
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N1—H1N···O2 ⁱ	0.88 (5)	2.17 (5)	2.994 (5)	157 (4)
N1—H1N···Cl2	0.88 (5)	2.67 (5)	3.011 (4)	104 (4)

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

Fig. 1

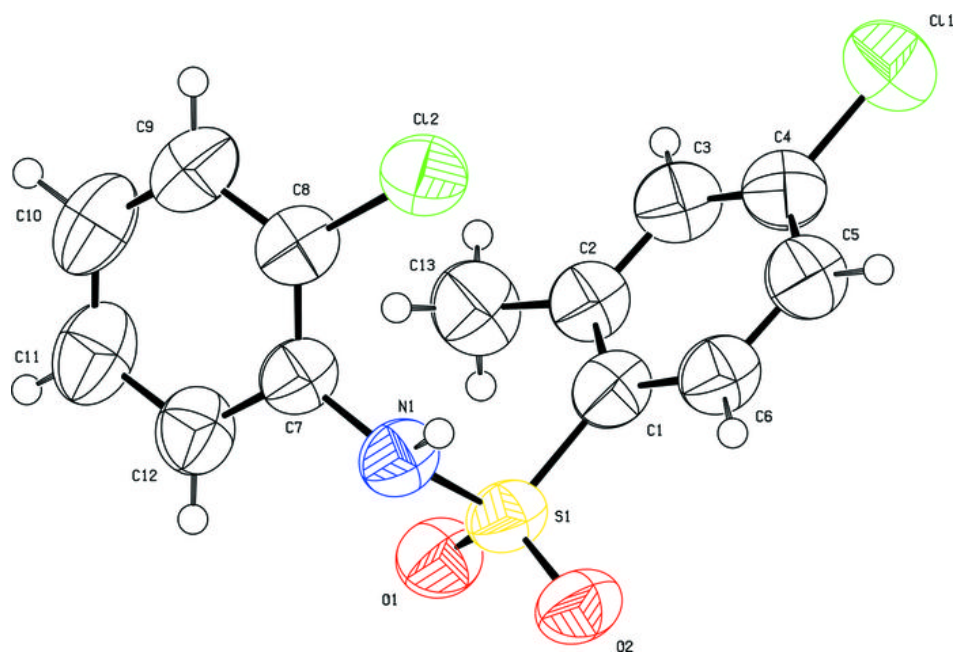


Fig. 2

