organic compounds

4-Chloro-*N*-(3,5-dichlorophenyl)-2-methylbenzenesulfonamide

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In the title compound, $C_{13}H_{10}Cl_3NO_2S$, torsion angle of the $C-SO_2-NH-C$ group in the molecule is $-58.57\,(26)°$. The sulfonyl and aniline benzene rings are tilted relative to each other by $84.2\,(1)°$. The crystal structure features inversion-related dimers linked by pairs of $N-H\cdots O$ hydrogen bonds.

Related literature

For the preparation of the title compound, see: Savitha & Gowda (2006). For the hydrogen-bonding preferences of sulfonamides, see: Adsmond & Grant (2001). For studies of the effects of substituents on the structure and other aspects of *N*-(aryl)amides, see: Gowda *et al.* (2000); of *N*-(aryl)-methanesulfonamides, see: Gowda *et al.* (2007); of *N*-(aryl)-arylsulfonamides, see: Gelbrich *et al.* (2007); Perlovich *et al.* (2006); Rodrigues *et al.* (2011); Shetty & Gowda (2005); and of *N*-(chloro)arylsulfonamides, see: Gowda *et al.* (2004).
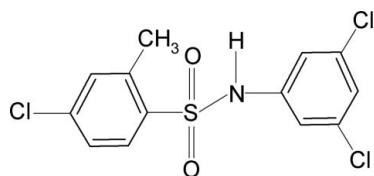
Experimental

Crystal data

$C_{13}H_{10}Cl_3NO_2S$	$b = 8.8494\,(9)$ Å
$M_r = 350.63$	$c = 11.649\,(1)$ Å
Triclinic, $P\bar{1}$	$\alpha = 97.002\,(8)°$
$a = 7.9638\,(8)$ Å	$\beta = 102.726\,(9)°$
$\gamma = 100.408\,(9)°$	$\mu = 0.72$ mm^{-1}
$V = 776.35\,(13)$ Å^3	$T = 293$ K
$Z = 2$	$0.44 \times 0.42 \times 0.36$ mm
Mo $K\alpha$ radiation	

Data collection

Oxford Xcalibur diffractometer with a Sapphire CCD detector	5369 measured reflections
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)	3137 independent reflections
	2649 reflections with $I > 2\sigma(I)$
$T_{min} = 0.742$, $T_{max} = 0.781$	$R_{int} = 0.009$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.140$	
$S = 1.06$	
3137 reflections	$\Delta\rho_{max} = 0.44$ e Å^{-3}
185 parameters	$\Delta\rho_{min} = -0.47$ e Å^{-3}
1 restraint	

Table 1
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1N$\cdots$O1^i	0.83 (2)	2.06 (2)	2.884 (3)	172 (3)

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

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 materials

4-Chloro-*N*-(3,5-dichlorophenyl)-2-methylbenzenesulfonamide

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Comment

The sulfonamide moiety is a constituent of many biologically significant compounds. The hydrogen bonding preferences of sulfonamides have been investigated (Adsmond & Grant, 2001). As part of our studies on the effects of substituents on the structures and other aspects of *N*-(aryl)-amides (Gowda *et al.*, 2000), *N*-(aryl)-methanesulfonamides (Gowda *et al.*, 2007), *N*-(aryl)-arylsulfonamides (Rodrigues *et al.*, 2011; Shetty & Gowda, 2005) and *N*-(chloro)-arylsulfonamides (Gowda *et al.*, 2004), in the present work, the crystal structure of 4-Chloro-2-methyl-*N*-(3,5-dichlorophenyl)benzenesulfonamide (I) has been determined (Fig. 1).

In (I), the conformation of the N—C bond in the C—SO$_2$—NH—C segment has *trans* and *gauche* torsion angles with the S=O bonds. Further, the N—H bond is *syn* to the *ortho*-methyl group in the sulfonyl benzene ring and to one of the *meta*-Cl atoms in the anilino ring of (I).

The molecule is bent at the S atom with the C—SO$_2$—NH—C torsion angle of -58.57 (26)°, compared to the value of -49.42 (23)° in 4-Chloro-2-methyl-*N*-(3,4-dichlorophenyl)benzenesulfonamide (II) (Rodrigues *et al.*, 2011).

The sulfonyl and the aniline benzene rings are tilted relative to each other by 84.2 (1)°, compared to the value of 54.6 (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, the intermolecular N–H···O hydrogen bonds (Table 1) link the molecules into dimeric chains. Part of the crystal structure is shown in Fig. 2.

Experimental

The solution of *m*-chlorotoluene (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-methyl-4-chlorobenzenesulfonylchloride was treated with 3,5-dichlorolaniline in the stoichiometric ratio and boiled for 10 min. The reaction mixture was then cooled to room temperature and added to ice cold water (100 ml). The resultant solid 4-chloro-*N*-(3,5-dichlorophenyl)-2-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 (Savitha & Gowda, 2006).

Prism like colourless single crystals used in X-ray diffraction studies were grown in ethanolic solution by slow evaporation at room temperature.

supplementary materials

Refinement

The H atom of the NH group was located in a difference map and its coordinates were refined with the N—H distance restrained to 0.86 (2) %A. The other H atoms were positioned with idealized geometry using a riding model with the aromatic C—H = 0.93Å and methyl C—H = 0.96 Å. All H atoms were refined with isotropic displacement parameters U_{iso}(H) set to $1.2U_{eq}$(C aromatic, N) and $1.5U_{eq}$(C methyl).

Figures
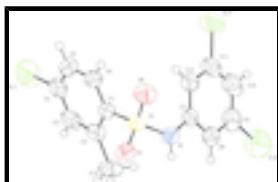
Fig. 1. Molecular structure of the title compound, showing the atom labelling scheme and displacement ellipsoids are drawn at the 50% probability level.
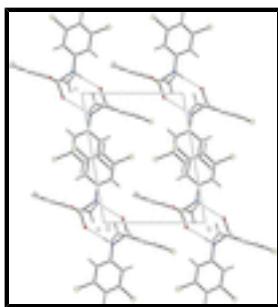
Fig. 2. Molecular packing of (I) with hydrogen bonding shown as dashed lines.

4-Chloro-*N*-(3,5-dichlorophenyl)-2-methylbenzenesulfonamide

Crystal data

$C_{13}H_{10}Cl_3NO_2S$	$Z = 2$
$M_r = 350.63$	$F(000) = 356$
Triclinic, $P\bar{1}$	$D_x = 1.500$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 7.9638$ (8) Å	Cell parameters from 3195 reflections
$b = 8.8494$ (9) Å	$\theta = 3.2–27.7°$
$c = 11.649$ (1) Å	$\mu = 0.72$ mm^{-1}
$\alpha = 97.002$ (8)°	$T = 293$ K
$\beta = 102.726$ (9)°	Prism, colourless
$\gamma = 100.408$ (9)°	$0.44 \times 0.42 \times 0.36$ mm
$V = 776.35$ (13) Å^3	

Data collection

Oxford Xcalibur diffractometer with a Sapphire CCD detector	3137 independent reflections
Radiation source: fine-focus sealed tube	2649 reflections with $I > 2\sigma(I)$

graphite	$R_{int} = 0.009$
rotation method data acquisition using ω scans	$\theta_{max} = 26.4°$, $\theta_{min} = 3.2°$
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)	$h = -9 \rightarrow 9$
$T_{min} = 0.742$, $T_{max} = 0.781$	$k = -11 \rightarrow 6$
5369 measured reflections	$l = -14 \rightarrow 13$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.048$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.140$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.06$	$w = 1/[\sigma^2(F_o^2) + (0.0686P)^2 + 0.5149P]$ where $P = (F_o^2 + 2F_c^2)/3$
3137 reflections	$(\Delta/\sigma)_{max} < 0.001$
185 parameters	$\Delta\rho_{max} = 0.44$ e Å^{-3}
1 restraint	$\Delta\rho_{min} = -0.47$ e Å^{-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 (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
C1	0.6313 (3)	0.3284 (3)	0.8732 (2)	0.0413 (5)
C2	0.7898 (3)	0.2789 (3)	0.8801 (2)	0.0520 (6)
C3	0.9242 (4)	0.3774 (4)	0.8502 (3)	0.0671 (8)
H3	1.0310	0.3479	0.8525	0.080*
C4	0.9016 (4)	0.5179 (4)	0.8174 (3)	0.0660 (8)
C5	0.7472 (4)	0.5659 (3)	0.8117 (3)	0.0643 (8)
H5	0.7342	0.6614	0.7895	0.077*
C6	0.6109 (4)	0.4700 (3)	0.8396 (2)	0.0523 (6)
H6	0.5043	0.5006	0.8359	0.063*
C7	0.3567 (3)	0.0027 (3)	0.7092 (2)	0.0473 (6)
C8	0.3722 (4)	−0.1450 (3)	0.6648 (2)	0.0549 (6)

H8	0.4155	−0.2093	0.7163	0.066*
C9	0.3226 (5)	−0.1955 (4)	0.5433 (3)	0.0707 (8)
C10	0.2603 (5)	−0.1027 (5)	0.4647 (3)	0.0769 (10)
H10	0.2280	−0.1379	0.3827	0.092*
C11	0.2473 (4)	0.0441 (4)	0.5115 (3)	0.0667 (8)
C12	0.2941 (4)	0.0998 (3)	0.6324 (2)	0.0570 (7)
H12	0.2841	0.1995	0.6617	0.068*
C13	0.8193 (4)	0.1183 (4)	0.9168 (3)	0.0657 (8)
H13A	0.7491	0.0347	0.8550	0.079*
H13B	0.7852	0.1076	0.9900	0.079*
H13C	0.9415	0.1148	0.9278	0.079*
N1	0.4035 (3)	0.0459 (3)	0.83447 (19)	0.0530 (5)
H1N	0.440 (4)	−0.018 (3)	0.874 (3)	0.064*
O1	0.5024 (3)	0.1945 (2)	1.03359 (15)	0.0547 (5)
O2	0.3065 (3)	0.2924 (3)	0.8798 (2)	0.0652 (5)
Cl1	1.07180 (15)	0.63564 (18)	0.77947 (12)	0.1240 (5)
Cl2	0.3407 (2)	−0.38086 (15)	0.49006 (10)	0.1233 (5)
Cl3	0.16476 (15)	0.16169 (14)	0.41414 (9)	0.1002 (4)
S1	0.45050 (8)	0.21891 (7)	0.91211 (5)	0.04505 (19)

Atomic displacement parameters ($\mathring{A}^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0421 (12)	0.0438 (12)	0.0388 (11)	0.0124 (9)	0.0091 (9)	0.0067 (9)
C2	0.0455 (13)	0.0640 (16)	0.0511 (14)	0.0199 (12)	0.0100 (11)	0.0186 (12)
C3	0.0386 (13)	0.099 (2)	0.0649 (18)	0.0144 (14)	0.0081 (12)	0.0280 (17)
C4	0.0526 (16)	0.081 (2)	0.0532 (15)	−0.0097 (14)	0.0028 (12)	0.0232 (14)
C5	0.0736 (19)	0.0516 (16)	0.0627 (17)	0.0044 (14)	0.0087 (14)	0.0187 (13)
C6	0.0579 (15)	0.0477 (14)	0.0540 (14)	0.0182 (12)	0.0128 (12)	0.0104 (11)
C7	0.0420 (12)	0.0527 (14)	0.0440 (12)	0.0003 (10)	0.0105 (10)	0.0107 (10)
C8	0.0544 (15)	0.0579 (15)	0.0492 (14)	0.0075 (12)	0.0113 (11)	0.0066 (12)
C9	0.077 (2)	0.072 (2)	0.0558 (17)	0.0080 (16)	0.0166 (15)	−0.0060 (15)
C10	0.079 (2)	0.100 (3)	0.0421 (15)	0.0053 (19)	0.0079 (14)	0.0072 (16)
C11	0.0576 (17)	0.086 (2)	0.0512 (15)	0.0018 (15)	0.0064 (13)	0.0256 (15)
C12	0.0555 (15)	0.0586 (16)	0.0530 (15)	0.0050 (12)	0.0076 (12)	0.0154 (12)
C13	0.0505 (15)	0.082 (2)	0.085 (2)	0.0451 (15)	0.0225 (14)	0.0335 (16)
N1	0.0683 (14)	0.0466 (12)	0.0415 (11)	0.0092 (10)	0.0088 (10)	0.0104 (9)
O1	0.0713 (12)	0.0540 (10)	0.0429 (9)	0.0162 (9)	0.0200 (8)	0.0084 (8)
O2	0.0515 (11)	0.0776 (14)	0.0795 (14)	0.0308 (10)	0.0258 (10)	0.0203 (11)
Cl1	0.0745 (6)	0.1562 (12)	0.1290 (10)	−0.0240 (7)	0.0142 (6)	0.0702 (9)
Cl2	0.1735 (13)	0.0994 (8)	0.0837 (7)	0.0466 (8)	0.0144 (7)	−0.0283 (6)
Cl3	0.1019 (7)	0.1234 (9)	0.0713 (6)	0.0149 (6)	0.0016 (5)	0.0515 (6)
S1	0.0460 (3)	0.0480 (3)	0.0460 (3)	0.0156 (2)	0.0163 (2)	0.0092 (2)

Geometric parameters ($\mathring{A}$, °)

C1—C6	1.383 (3)	C8—H8	0.9300
C1—C2	1.399 (3)	C9—C10	1.375 (5)
C1—S1	1.763 (2)	C9—Cl2	1.725 (4)

C2—C3	1.388 (4)	C10—C11	1.377 (5)
C2—C13	1.575 (4)	C10—H10	0.9300
C3—C4	1.376 (5)	C11—C12	1.377 (4)
C3—H3	0.9300	C11—Cl3	1.734 (3)
C4—C5	1.362 (5)	C12—H12	0.9300
C4—Cl1	1.727 (3)	C13—H13A	0.9600
C5—C6	1.376 (4)	C13—H13B	0.9600
C5—H5	0.9300	C13—H13C	0.9600
C6—H6	0.9300	N1—S1	1.615 (2)
C7—C8	1.383 (4)	N1—H1N	0.825 (17)
C7—C12	1.391 (4)	O1—S1	1.4384 (19)
C7—N1	1.410 (3)	O2—S1	1.4206 (19)
C8—C9	1.376 (4)		
C6—C1—C2	121.3 (2)	C8—C9—Cl2	118.2 (3)
C6—C1—S1	116.38 (19)	C9—C10—C11	117.7 (3)
C2—C1—S1	122.24 (19)	C9—C10—H10	121.2
C3—C2—C1	116.9 (2)	C11—C10—H10	121.2
C3—C2—C13	119.8 (2)	C10—C11—C12	122.6 (3)
C1—C2—C13	123.3 (2)	C10—C11—Cl3	118.5 (2)
C4—C3—C2	121.0 (3)	C12—C11—Cl3	118.9 (3)
C4—C3—H3	119.5	C11—C12—C7	118.0 (3)
C2—C3—H3	119.5	C11—C12—H12	121.0
C5—C4—C3	121.8 (3)	C7—C12—H12	121.0
C5—C4—Cl1	118.9 (3)	C2—C13—H13A	109.5
C3—C4—Cl1	119.3 (3)	C2—C13—H13B	109.5
C4—C5—C6	118.6 (3)	H13A—C13—H13B	109.5
C4—C5—H5	120.7	C2—C13—H13C	109.5
C6—C5—H5	120.7	H13A—C13—H13C	109.5
C5—C6—C1	120.5 (3)	H13B—C13—H13C	109.5
C5—C6—H6	119.8	C7—N1—S1	128.18 (19)
C1—C6—H6	119.8	C7—N1—H1N	117 (2)
C8—C7—C12	120.6 (2)	S1—N1—H1N	112 (2)
C8—C7—N1	116.5 (2)	O2—S1—O1	118.37 (12)
C12—C7—N1	122.8 (3)	O2—S1—N1	109.26 (13)
C9—C8—C7	119.0 (3)	O1—S1—N1	104.09 (11)
C9—C8—H8	120.5	O2—S1—C1	107.01 (12)
C7—C8—H8	120.5	O1—S1—C1	109.78 (11)
C10—C9—C8	122.0 (3)	N1—S1—C1	107.94 (12)
C10—C9—Cl2	119.8 (3)		
C6—C1—C2—C3	−0.8 (4)	Cl2—C9—C10—C11	179.7 (3)
S1—C1—C2—C3	−178.2 (2)	C9—C10—C11—C12	0.1 (5)
C6—C1—C2—C13	−179.5 (3)	C9—C10—C11—Cl3	−178.7 (3)
S1—C1—C2—C13	3.1 (4)	C10—C11—C12—C7	0.0 (5)
C1—C2—C3—C4	1.0 (4)	Cl3—C11—C12—C7	178.7 (2)
C13—C2—C3—C4	179.7 (3)	C8—C7—C12—C11	0.4 (4)
C2—C3—C4—C5	−0.6 (5)	N1—C7—C12—C11	−178.1 (2)
C2—C3—C4—Cl1	−179.3 (2)	C8—C7—N1—S1	161.9 (2)
C3—C4—C5—C6	−0.1 (5)	C12—C7—N1—S1	−19.5 (4)

Cl1—C4—C5—C6	178.6 (2)	C7—N1—S1—O2	57.5 (3)
C4—C5—C6—C1	0.3 (4)	C7—N1—S1—O1	−175.2 (2)
C2—C1—C6—C5	0.2 (4)	C7—N1—S1—C1	−58.6 (3)
S1—C1—C6—C5	177.7 (2)	C6—C1—S1—O2	9.9 (2)
C12—C7—C8—C9	−0.8 (4)	C2—C1—S1—O2	−172.5 (2)
N1—C7—C8—C9	177.8 (3)	C6—C1—S1—O1	−119.7 (2)
C7—C8—C9—C10	0.9 (5)	C2—C1—S1—O1	57.8 (2)
C7—C8—C9—Cl2	−179.3 (2)	C6—C1—S1—N1	127.4 (2)
C8—C9—C10—C11	−0.5 (5)	C2—C1—S1—N1	−55.0 (2)

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1N⋯O1[i]	0.83 (2)	2.06 (2)	2.884 (3)	172 (3)

Symmetry codes: (i) −x+1, −y, −z+2.

Fig. 1

Fig. 2