

2,4-Dichloro-N-(3,5-dichlorophenyl)-benzenesulfonamide

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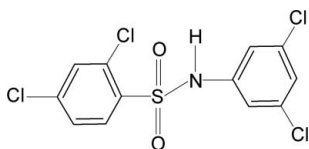
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.038; wR factor = 0.104; data-to-parameter ratio = 16.5.

In the title compound, $\text{C}_{12}\text{H}_7\text{Cl}_4\text{NO}_2\text{S}$, the N—H bond in the C—SO₂—NH—C segment is *syn* with respect to the *ortho*-Cl atom of the sulfonylbenzene ring and one of the *meta*-Cl atoms of the aniline ring. The C—SO₂—NH—C torsion angle is -93.9 (2)°. The benzene rings are tilted relative to each other by 61.9 (1)°. The crystal structure features inversion-related dimers linked by pairs of N—H···O hydrogen bonds.

Related literature

For the preparation of the title compound, see: Savitha & Gowda (2006). For hydrogen-bonding modes of sulfonamides, see: Adsmund & Grant (2001). For studies on the effects of substituents on the structures and other aspects of *N*-(aryl)-substituted amides, see: Gowda *et al.* (2000), on methane-sulfonamides, see: Gowda *et al.* (2007), on arylsulfonamides, see: Gelbrich *et al.* (2007); Perlovich *et al.* (2006); Gowda *et al.* (2009); Shetty & Gowda (2005) and on *N*-(chloro)-aryl-sulfonamides, see: Gowda *et al.* (2003).



Experimental

Crystal data

$\text{C}_{12}\text{H}_7\text{Cl}_4\text{NO}_2\text{S}$
 $M_r = 371.05$
Triclinic, $P\bar{1}$
 $a = 8.075$ (1) Å

$b = 9.479$ (1) Å
 $c = 10.103$ (2) Å
 $\alpha = 84.90$ (2)°
 $\beta = 78.49$ (1)°

$\gamma = 79.28$ (1)°
 $V = 743.46$ (19) Å³
 $Z = 2$
Mo $K\alpha$ radiation

$\mu = 0.93$ mm⁻¹
 $T = 293$ K
 $0.48 \times 0.44 \times 0.32$ mm

Data collection

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

Diffraction, 2009)
 $T_{\min} = 0.663$, $T_{\max} = 0.754$
4974 measured reflections
3027 independent reflections
2423 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.014$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.104$
 $S = 1.03$
3027 reflections
184 parameters
1 restraint

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.36$ e Å⁻³
 $\Delta\rho_{\min} = -0.29$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1N···O2 ⁱ	0.82 (2)	2.07 (2)	2.889 (3)	177 (3)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2009); 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: RZ22649).

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supplementary materials

Acta Cryst. (2011). E67, o2976 [doi:10.1107/S160053681104219X]

2,4-Dichloro-*N*-(3,5-dichlorophenyl)benzenesulfonamide

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Comment

The sulfonamide moiety is the 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 substituent effects on the structures and other aspects of *N*-(aryl)-amides (Gowda *et al.*, 2000), *N*-(aryl)-methanesulfonamides (Gowda *et al.*, 2007), *N*-(aryl)-arylsulfonamides (Gowda *et al.*, 2009; Shetty & Gowda, 2005) and *N*-(chloro)-arylsulfonamides (Gowda *et al.*, 2003), in the present work, the crystal structure of 2,4-dichloro-*N*-(3,5-dichlorophenyl)benzenesulfonamide (I) has been determined (Fig. 1).

In (I), the N—H bond in the C—SO₂—NH—C segment is *syn* with respect to the *ortho*-Cl atom of the sulfonylbenzene ring and one of the *meta*-Cl atoms of the anilino ring. The molecule is bent at the S atom with the C—SO₂—NH—C torsion angle of -93.9 (2)°, compared to the value of -48.2 (2)° in 2,4-dichloro-*N*-(3,4-dichlorophenyl)-benzenesulfonamide (II) (Gowda *et al.*, 2009). The sulfonyl and the anilino benzene rings are tilted relative to each other by 61.9 (1)°, compared to the value of 68.9 (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, intermolecular N—H···O hydrogen bonds (Table 1) link the molecules into dimeric units. Part of the crystal structure is shown in Fig. 2.

Experimental

A solution of 1,3-dichlorobenzene (10 ml) in chloroform (40 ml) was treated dropwise with chlorosulfonic acid (25 ml) at 273 K. 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 2,4-dichlorobenzenesulfonylchloride was treated with 3,5-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 solid 2,4-dichloro-*N*-(3,5-dichlorophenyl)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 (Savitha & Gowda, 2006). Prism like light pink single crystals used in X-ray diffraction studies were grown in ethanolic solution by slow evaporation of the solvent at room temperature.

Refinement

The H atoms of the NH group was located in a difference Fourier map and refined with the N—H distance restrained to 0.86 (2) Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$. All other H atoms were positioned with idealized geometry using a riding model with C—H = 0.93 Å, and refined with $U_{\text{iso}}(\text{H})$ values set at $1.2U_{\text{eq}}(\text{C})$.

Figures

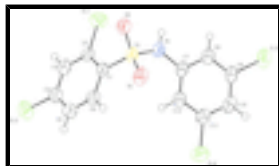


Fig. 1. Molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level.

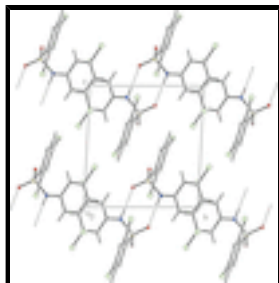


Fig. 2. Molecular packing of the title compound with hydrogen bonding shown as dashed lines.

2,4-Dichloro-*N*-(3,5-dichlorophenyl)benzenesulfonamide

Crystal data

$C_{12}H_7Cl_4NO_2S$

$M_r = 371.05$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 8.075$ (1) Å

$b = 9.479$ (1) Å

$c = 10.103$ (2) Å

$\alpha = 84.90$ (2)°

$\beta = 78.49$ (1)°

$\gamma = 79.28$ (1)°

$V = 743.46$ (19) Å³

$Z = 2$

$F(000) = 372$

$D_x = 1.657$ Mg m⁻³

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

Cell parameters from 2149 reflections

$\theta = 2.6$ – 27.7 °

$\mu = 0.93$ mm⁻¹

$T = 293$ K

Prism, light pink

$0.48 \times 0.44 \times 0.32$ 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 scans

Absorption correction: multi-scan (*Crys.Alis RED*; Oxford Diffraction, 2009)

$T_{\min} = 0.663$, $T_{\max} = 0.754$

4974 measured reflections

3027 independent reflections

2423 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.014$

$\theta_{\max} = 26.4$ °, $\theta_{\min} = 2.6$ °

$h = -10 \rightarrow 10$

$k = -11 \rightarrow 11$

$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.038$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.104$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.0494P)^2 + 0.2632P]$
3027 reflections	where $P = (F_o^2 + 2F_c^2)/3$
184 parameters	$(\Delta/\sigma)_{\max} = 0.001$
1 restraint	$\Delta\rho_{\max} = 0.36 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\min} = -0.29 \text{ e } \text{\AA}^{-3}$

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	0.2231 (3)	0.3850 (2)	0.6848 (2)	0.0383 (5)
C2	0.3719 (3)	0.3794 (3)	0.7381 (2)	0.0401 (5)
C3	0.5311 (3)	0.3381 (3)	0.6599 (3)	0.0454 (6)
H3	0.6298	0.3351	0.6951	0.055*
C4	0.5432 (3)	0.3010 (3)	0.5291 (3)	0.0481 (6)
C5	0.3988 (3)	0.3048 (3)	0.4738 (3)	0.0520 (6)
H5	0.4086	0.2792	0.3855	0.062*
C6	0.2400 (3)	0.3475 (3)	0.5529 (2)	0.0466 (6)
H6	0.1419	0.3512	0.5167	0.056*
C7	-0.0988 (3)	0.2062 (2)	0.9076 (2)	0.0397 (5)
C8	-0.1821 (3)	0.1626 (3)	1.0339 (3)	0.0431 (5)
H8	-0.1863	0.2132	1.1096	0.052*
C9	-0.2587 (3)	0.0423 (3)	1.0452 (3)	0.0442 (6)
C10	-0.2563 (3)	-0.0345 (3)	0.9350 (3)	0.0482 (6)
H10	-0.3092	-0.1149	0.9440	0.058*
C11	-0.1723 (3)	0.0125 (3)	0.8107 (3)	0.0455 (6)
C12	-0.0919 (3)	0.1312 (3)	0.7950 (3)	0.0436 (5)

supplementary materials

H12	-0.0345	0.1599	0.7105	0.052*
N1	-0.0178 (3)	0.3284 (2)	0.9021 (2)	0.0470 (5)
H1N	-0.017 (4)	0.358 (3)	0.975 (2)	0.056*
O1	-0.1015 (2)	0.4407 (2)	0.68767 (18)	0.0528 (5)
O2	0.0147 (2)	0.57618 (18)	0.83484 (17)	0.0488 (4)
Cl1	0.36340 (9)	0.42083 (9)	0.90298 (7)	0.0630 (2)
Cl2	0.74509 (9)	0.25049 (9)	0.43243 (8)	0.0707 (2)
Cl3	-0.36154 (9)	-0.01334 (8)	1.20398 (7)	0.0631 (2)
Cl4	-0.16598 (10)	-0.08094 (8)	0.66939 (8)	0.0692 (2)
S1	0.01475 (7)	0.44418 (6)	0.77605 (6)	0.04076 (17)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0362 (12)	0.0411 (12)	0.0409 (12)	-0.0147 (10)	-0.0093 (9)	0.0030 (10)
C2	0.0375 (12)	0.0447 (13)	0.0423 (13)	-0.0134 (10)	-0.0122 (10)	-0.0015 (10)
C3	0.0359 (12)	0.0481 (14)	0.0548 (15)	-0.0136 (11)	-0.0101 (11)	0.0010 (11)
C4	0.0428 (14)	0.0466 (14)	0.0531 (15)	-0.0151 (11)	0.0007 (11)	0.0003 (11)
C5	0.0567 (16)	0.0606 (16)	0.0418 (14)	-0.0193 (13)	-0.0069 (12)	-0.0047 (12)
C6	0.0455 (14)	0.0566 (15)	0.0436 (14)	-0.0189 (12)	-0.0148 (11)	0.0007 (11)
C7	0.0274 (11)	0.0447 (13)	0.0487 (13)	-0.0103 (10)	-0.0098 (9)	0.0027 (10)
C8	0.0339 (12)	0.0466 (13)	0.0491 (14)	-0.0096 (10)	-0.0080 (10)	0.0016 (11)
C9	0.0288 (11)	0.0477 (14)	0.0547 (14)	-0.0092 (10)	-0.0071 (10)	0.0089 (11)
C10	0.0354 (13)	0.0437 (13)	0.0681 (17)	-0.0129 (11)	-0.0125 (12)	0.0020 (12)
C11	0.0335 (12)	0.0459 (13)	0.0584 (15)	-0.0044 (10)	-0.0117 (11)	-0.0081 (12)
C12	0.0311 (12)	0.0501 (14)	0.0495 (14)	-0.0098 (10)	-0.0057 (10)	-0.0004 (11)
N1	0.0489 (12)	0.0557 (12)	0.0430 (11)	-0.0264 (10)	-0.0104 (10)	0.0028 (10)
O1	0.0390 (9)	0.0673 (12)	0.0570 (11)	-0.0153 (8)	-0.0199 (8)	0.0081 (9)
O2	0.0475 (10)	0.0473 (10)	0.0518 (10)	-0.0135 (8)	-0.0059 (8)	-0.0004 (8)
Cl1	0.0509 (4)	0.0957 (6)	0.0500 (4)	-0.0160 (4)	-0.0187 (3)	-0.0163 (4)
Cl2	0.0511 (4)	0.0808 (5)	0.0728 (5)	-0.0128 (4)	0.0116 (3)	-0.0131 (4)
Cl3	0.0566 (4)	0.0687 (5)	0.0631 (4)	-0.0273 (4)	-0.0004 (3)	0.0123 (3)
Cl4	0.0673 (5)	0.0699 (5)	0.0748 (5)	-0.0190 (4)	-0.0071 (4)	-0.0263 (4)
S1	0.0344 (3)	0.0473 (3)	0.0433 (3)	-0.0139 (3)	-0.0101 (2)	0.0040 (3)

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

C1—C6	1.383 (3)	C7—N1	1.424 (3)
C1—C2	1.402 (3)	C8—C9	1.381 (3)
C1—S1	1.765 (2)	C8—H8	0.9300
C2—C3	1.376 (3)	C9—C10	1.378 (4)
C2—Cl1	1.731 (2)	C9—Cl3	1.738 (2)
C3—C4	1.378 (4)	C10—C11	1.381 (4)
C3—H3	0.9300	C10—H10	0.9300
C4—C5	1.383 (4)	C11—C12	1.383 (3)
C4—Cl2	1.731 (3)	C11—Cl4	1.733 (3)
C5—C6	1.379 (4)	C12—H12	0.9300
C5—H5	0.9300	N1—S1	1.618 (2)
C6—H6	0.9300	N1—H1N	0.815 (16)

C7—C12	1.381 (3)	O1—S1	1.4254 (17)
C7—C8	1.386 (3)	O2—S1	1.4319 (18)
C6—C1—C2	118.7 (2)	C7—C8—H8	120.7
C6—C1—S1	118.24 (17)	C10—C9—C8	122.1 (2)
C2—C1—S1	123.06 (18)	C10—C9—C13	119.25 (18)
C3—C2—C1	120.3 (2)	C8—C9—C13	118.6 (2)
C3—C2—C11	117.68 (17)	C9—C10—C11	117.6 (2)
C1—C2—C11	121.97 (18)	C9—C10—H10	121.2
C2—C3—C4	119.5 (2)	C11—C10—H10	121.2
C2—C3—H3	120.3	C10—C11—C12	122.3 (2)
C4—C3—H3	120.3	C10—C11—C14	119.07 (19)
C3—C4—C5	121.6 (2)	C12—C11—C14	118.7 (2)
C3—C4—C12	118.57 (19)	C7—C12—C11	118.5 (2)
C5—C4—C12	119.9 (2)	C7—C12—H12	120.8
C6—C5—C4	118.4 (2)	C11—C12—H12	120.8
C6—C5—H5	120.8	C7—N1—S1	127.32 (17)
C4—C5—H5	120.8	C7—N1—H1N	115 (2)
C5—C6—C1	121.6 (2)	S1—N1—H1N	114 (2)
C5—C6—H6	119.2	O1—S1—O2	119.41 (11)
C1—C6—H6	119.2	O1—S1—N1	109.48 (10)
C12—C7—C8	120.9 (2)	O2—S1—N1	105.64 (11)
C12—C7—N1	122.7 (2)	O1—S1—C1	106.86 (11)
C8—C7—N1	116.4 (2)	O2—S1—C1	108.24 (10)
C9—C8—C7	118.7 (2)	N1—S1—C1	106.57 (11)
C9—C8—H8	120.7		
C6—C1—C2—C3	0.3 (3)	C13—C9—C10—C11	-179.56 (18)
S1—C1—C2—C3	-177.25 (18)	C9—C10—C11—C12	0.4 (4)
C6—C1—C2—C11	-178.67 (18)	C9—C10—C11—C14	179.96 (17)
S1—C1—C2—C11	3.7 (3)	C8—C7—C12—C11	0.9 (3)
C1—C2—C3—C4	-0.5 (4)	N1—C7—C12—C11	179.0 (2)
C11—C2—C3—C4	178.53 (19)	C10—C11—C12—C7	-1.1 (4)
C2—C3—C4—C5	0.2 (4)	C14—C11—C12—C7	179.34 (17)
C2—C3—C4—C12	179.58 (18)	C12—C7—N1—S1	30.7 (3)
C3—C4—C5—C6	0.3 (4)	C8—C7—N1—S1	-151.21 (19)
C12—C4—C5—C6	-179.1 (2)	C7—N1—S1—O1	21.3 (3)
C4—C5—C6—C1	-0.5 (4)	C7—N1—S1—O2	151.1 (2)
C2—C1—C6—C5	0.2 (4)	C7—N1—S1—C1	-93.9 (2)
S1—C1—C6—C5	177.9 (2)	C6—C1—S1—O1	0.0 (2)
C12—C7—C8—C9	-0.1 (3)	C2—C1—S1—O1	177.56 (19)
N1—C7—C8—C9	-178.3 (2)	C6—C1—S1—O2	-129.83 (19)
C7—C8—C9—C10	-0.6 (4)	C2—C1—S1—O2	47.8 (2)
C7—C8—C9—C13	179.45 (17)	C6—C1—S1—N1	116.95 (19)
C8—C9—C10—C11	0.5 (4)	C2—C1—S1—N1	-65.5 (2)

Hydrogen-bond geometry (\AA , $^\circ$)

<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 O2 ⁱ	0.82 (2)	2.07 (2)	2.889 (3)	177 (3)

supplementary materials

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

Fig. 1

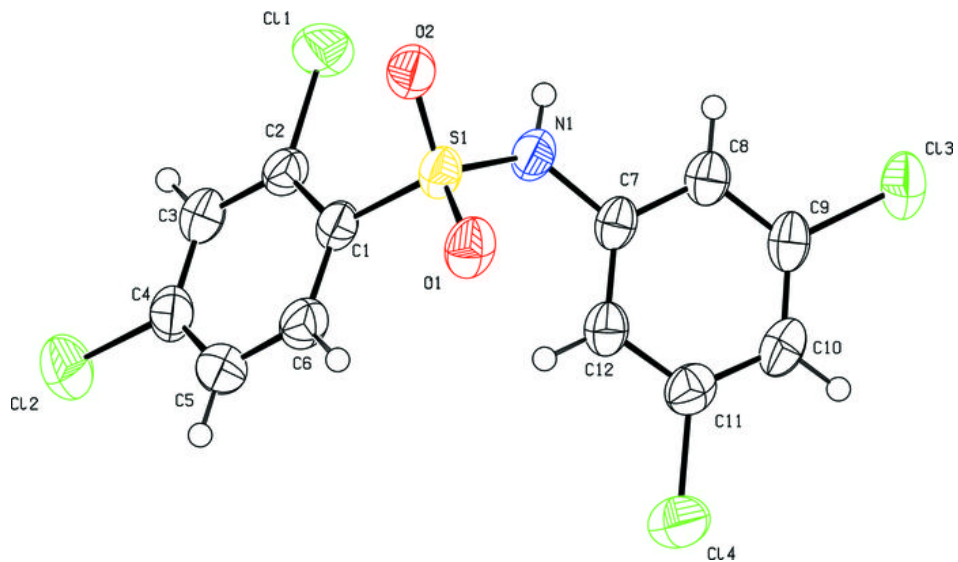


Fig. 2

