organic compounds

$\mu = 0.82$ mm^{-1}

$T = 293$ K

$0.32 \times 0.20 \times 0.10$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with Sapphire CCD detector

Absorption correction: multi-scan (*CrysAlis RED*; Oxford

Diffraction, 2009)

$T_{min} = 0.780$, $T_{max} = 0.923$

4440 measured reflections

2785 independent reflections

2236 reflections with $I > 2\sigma(I)$

$R_{int} = 0.013$

4-Chloro-*N*-(3,5-dichlorophenyl)-benzenesulfonamide

K. Shakuntala,^{a} Sabine Foro^b and B. Thimme Gowda^{a*}

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Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$

$wR(F^2) = 0.086$

$S = 1.03$

2785 reflections

175 parameters

1 restraint

H atoms treated by a mixture of independent and constrained refinement

$\Delta\rho_{max} = 0.36$ e Å^{-3}

$\Delta\rho_{min} = -0.33$ e Å^{-3}

In the title compound, $C_{12}H_8Cl_3NO_2S$, the dihedral angle between the aromatic rings is 87.9 (1)° and the C—S—N—C torsion angle is 77.8 (2)°. In the crystal, inversion dimers linked by pairs of N—H···O hydrogen bonds occur.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O2^i	0.84 (2)	2.08 (2)	2.917 (2)	170 (2)

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

Related literature

For hydrogen-bonding preferences of sulfonamides, see; Adsmond & Grant (2001). For our study of the effect of substituents on the structures of *N*-(aryl)-amides, see: Gowda *et al.* (2004); on the structures of *N*-(aryl)arylsulfonamides, see: Shakuntala *et al.* (2011*a*,*b*); and on the oxidative strengths of *N*-chloro-*N*-arylsulfonamides, see: Gowda & Kumar (2003).

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*.
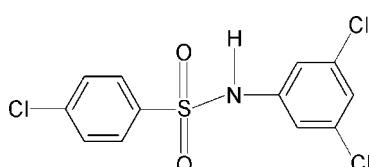
Experimental

Crystal data

$C_{12}H_8Cl_3NO_2S$

$M_r = 336.60$

Triclinic, $P\bar{1}$

$a = 4.935$ (1) Å

$b = 11.630$ (2) Å

$c = 13.115$ (2) Å

$\alpha = 113.52$ (2)°

$\beta = 90.49$ (1)°

$\gamma = 96.50$ (1)°

$V = 684.6$ (2) Å^3

$Z = 2$

Mo $K\alpha$ radiation

References

Adsmond, D. A. & Grant, D. J. W. (2001). *J. Pharm. Sci.* **90**, 2058–2077.
Gowda, B. T. & Kumar, B. H. A. (2003). *Oxid. Commun.* **26**, 403–425.
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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
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supporting information

4-Chloro-*N*-(3,5-dichlorophenyl)benzenesulfonamide

K. Shakuntala, Sabine Foro and B. Thimme Gowda

S1. Comment

The amide and sulfonamide moieties are important constituents of many biologically important compounds. As a part of studying the substituent effects on the structures and other aspects of this class of compounds (Gowda & Kumar, 2003; Gowda *et al.*, 2004; Shakuntala *et al.*, 2011*a*,*b*), in the present work, the crystal structure of 4-chloro-*N*-(3,5-dichloro-phenyl)benzenesulfonamide (I) has been determined (Fig. 1). The molecule is twisted at the S atom with the C—SO$_2$—NH—C torsion angle of 77.8 (2)°, compared to the values of -58.4 (3)° in 4-chloro-*N*-(3-chlorophenyl)benzene-sulfonamide (II) (Shakuntala *et al.*, 2011*b*) and -56.7 (2)° in 4-chloro-*N*-(2,3-dichlorophenyl)-benzenesulfonamide (III) (Shakuntala *et al.*, 2011*a*). The conformation of the N—H bond is *anti* to one of the *meta*-chloro group in the anilino benzene ring and *syn* to the other.

The sulfonyl and the anilino benzene rings in (I) are tilted relative to each other by 87.9 (1)°, compared to the values of 77.1 (1)° in (II) and 56.5 (1)° in (III).

Intermolecular N—H···O(S) hydrogen bonding interactions generate inversion related dimers which are further packed *via* van der Waals interactions in the crystal structure (Fig. 2).

S2. Experimental

The solution of chlorobenzene (10 ml) in chloroform (40 ml) was added 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-chlorobenzenesulfonylchloride was treated with 3,5-dichloroaniline in the stoichiometric ratio and boiled for 15 min. The reaction mixture was then cooled to room temperature and added to ice cold water (100 ml). The resultant 4-chloro-*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 compound was characterized by FT–IR and NMR spectra.

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

S3. 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 Å. All H atoms were refined with isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent atom).
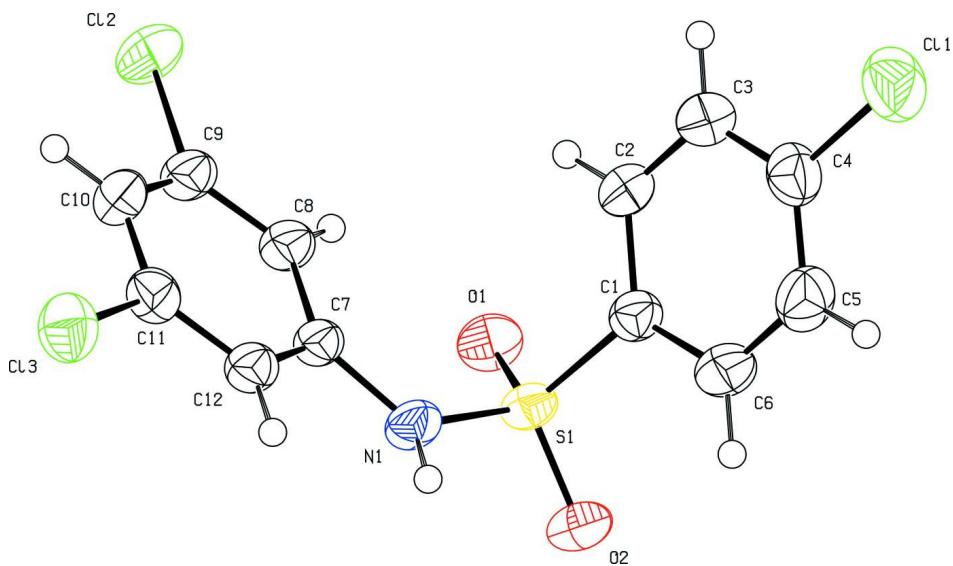
Figure 1

Molecular structure of (I), showing the atom labeling scheme and displacement ellipsoids are drawn at the 50% probability level.
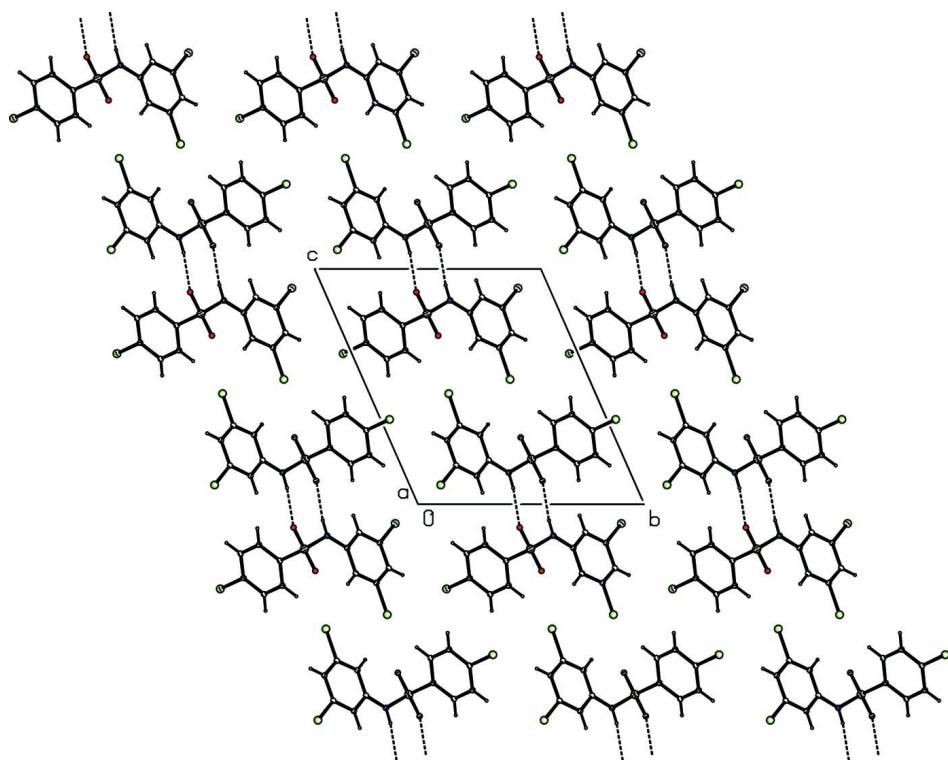
Figure 2

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

4-Chloro-*N*-(3,5-dichlorophenyl)benzenesulfonamide

Crystal data

$C_{12}H_8Cl_3NO_2S$	$Z = 2$
$M_r = 336.60$	$F(000) = 340$
Triclinic, $P\overline{1}$	$D_x = 1.633$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 4.935$ (1) Å	Cell parameters from 1578 reflections
$b = 11.630$ (2) Å	$\theta = 3.0–27.8°$
$c = 13.115$ (2) Å	$\mu = 0.82$ mm^{-1}
$\alpha = 113.52$ (2)°	$T = 293$ K
$\beta = 90.49$ (1)°	Prism, colourless
$\gamma = 96.50$ (1)°	$0.32 \times 0.20 \times 0.10$ mm
$V = 684.6$ (2) Å^3	

Data collection

Oxford Diffraction Xcalibur diffractometer with Sapphire CCD detector	4440 measured reflections
	2785 independent reflections
Radiation source: fine-focus sealed tube	2236 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.013$
Rotation method data acquisition using ω and φ scans	$\theta_{max} = 26.4°$, $\theta_{min} = 3.0°$
	$h = -6\rightarrow4$
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)	$k = -14\rightarrow14$
	$l = -15\rightarrow16$
$T_{min} = 0.780$, $T_{max} = 0.923$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.034$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.086$	
$S = 1.03$	H atoms treated by a mixture of independent and constrained refinement
2785 reflections	
175 parameters	$w = 1/[\sigma^2(F_o^2) + (0.0393P)^2 + 0.3103P]$ where $P = (F_o^2 + 2F_c^2)/3$
1 restraint	
Primary atom site location: structure-invariant direct methods	$(\Delta/\sigma)_{max} = 0.005$
	$\Delta\rho_{max} = 0.36$ e Å^{-3}
	$\Delta\rho_{min} = -0.33$ e 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.7071 (4)	0.7199 (2)	0.24024 (17)	0.0357 (5)
C2	0.8325 (5)	0.7573 (2)	0.34566 (19)	0.0443 (5)
H2	0.7746	0.7164	0.3913	0.053*
C3	1.0431 (5)	0.8552 (2)	0.3823 (2)	0.0506 (6)
H3	1.1295	0.8807	0.4525	0.061*
C4	1.1242 (5)	0.9149 (2)	0.3139 (2)	0.0466 (6)
C5	0.9996 (5)	0.8800 (2)	0.2095 (2)	0.0507 (6)
H5	1.0565	0.9221	0.1648	0.061*
C6	0.7896 (5)	0.7817 (2)	0.17258 (19)	0.0454 (5)
H6	0.7035	0.7569	0.1024	0.055*
C7	0.7357 (4)	0.39831 (19)	0.18639 (16)	0.0330 (4)
C8	0.6937 (4)	0.4095 (2)	0.29448 (17)	0.0388 (5)
H8	0.5776	0.4644	0.3387	0.047*
C9	0.8290 (5)	0.3369 (2)	0.33442 (18)	0.0416 (5)
C10	1.0005 (5)	0.2536 (2)	0.27211 (19)	0.0463 (6)
H10	1.0892	0.2057	0.3010	0.056*
C11	1.0357 (4)	0.2439 (2)	0.16442 (19)	0.0400 (5)
C12	0.9083 (4)	0.3151 (2)	0.12094 (18)	0.0365 (5)
H12	0.9373	0.3077	0.0487	0.044*
N1	0.5991 (4)	0.46264 (17)	0.13400 (14)	0.0382 (4)
H1N	0.645 (5)	0.452 (2)	0.0694 (15)	0.046*
O1	0.3111 (3)	0.58358 (15)	0.28347 (12)	0.0442 (4)
O2	0.2996 (3)	0.59558 (15)	0.09918 (12)	0.0442 (4)
Cl1	1.39180 (14)	1.03860 (7)	0.36017 (7)	0.0681 (2)
Cl2	0.77531 (16)	0.35067 (7)	0.46970 (5)	0.0603 (2)
Cl3	1.24838 (13)	0.13818 (6)	0.08193 (6)	0.05370 (18)
S1	0.45009 (11)	0.58943 (5)	0.19092 (4)	0.03502 (14)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0393 (11)	0.0375 (11)	0.0312 (10)	0.0088 (9)	0.0033 (9)	0.0137 (9)
C2	0.0567 (14)	0.0441 (13)	0.0365 (12)	0.0058 (11)	−0.0029 (10)	0.0210 (10)
C3	0.0594 (15)	0.0460 (13)	0.0460 (14)	0.0057 (11)	−0.0123 (11)	0.0187 (11)
C4	0.0438 (13)	0.0368 (12)	0.0563 (15)	0.0068 (10)	−0.0006 (11)	0.0154 (11)
C5	0.0588 (15)	0.0504 (14)	0.0501 (14)	0.0054 (12)	0.0082 (12)	0.0279 (12)
C6	0.0553 (14)	0.0514 (14)	0.0321 (11)	0.0054 (11)	0.0030 (10)	0.0197 (10)
C7	0.0362 (11)	0.0341 (11)	0.0301 (10)	−0.0015 (8)	−0.0025 (8)	0.0159 (8)
C8	0.0454 (12)	0.0407 (12)	0.0311 (11)	0.0013 (9)	0.0002 (9)	0.0164 (9)
C9	0.0505 (13)	0.0444 (12)	0.0329 (11)	−0.0039 (10)	−0.0028 (10)	0.0212 (10)
C10	0.0528 (14)	0.0444 (13)	0.0480 (14)	0.0032 (11)	−0.0076 (11)	0.0263 (11)
C11	0.0390 (12)	0.0362 (11)	0.0423 (12)	0.0001 (9)	−0.0002 (9)	0.0145 (10)
C12	0.0388 (11)	0.0382 (11)	0.0327 (11)	−0.0005 (9)	0.0000 (9)	0.0160 (9)
N1	0.0495 (11)	0.0440 (10)	0.0249 (9)	0.0105 (8)	0.0057 (8)	0.0166 (8)
O1	0.0453 (9)	0.0560 (10)	0.0342 (8)	0.0078 (7)	0.0101 (7)	0.0206 (7)

O2	0.0448 (9)	0.0574 (10)	0.0330 (8)	0.0125 (7)	−0.0027 (7)	0.0194 (7)
Cl1	0.0579 (4)	0.0520 (4)	0.0911 (6)	−0.0058 (3)	−0.0129 (4)	0.0292 (4)
Cl2	0.0874 (5)	0.0668 (4)	0.0378 (3)	0.0071 (3)	0.0023 (3)	0.0333 (3)
Cl3	0.0543 (4)	0.0469 (3)	0.0610 (4)	0.0135 (3)	0.0063 (3)	0.0209 (3)
S1	0.0375 (3)	0.0434 (3)	0.0262 (3)	0.0081 (2)	0.0021 (2)	0.0154 (2)

Geometric parameters (Å, º)

C1—C6	1.385 (3)	C7—N1	1.415 (3)
C1—C2	1.389 (3)	C8—C9	1.381 (3)
C1—S1	1.760 (2)	C8—H8	0.9300
C2—C3	1.376 (3)	C9—C10	1.375 (3)
C2—H2	0.9300	C9—Cl2	1.742 (2)
C3—C4	1.373 (4)	C10—C11	1.385 (3)
C3—H3	0.9300	C10—H10	0.9300
C4—C5	1.379 (3)	C11—C12	1.377 (3)
C4—Cl1	1.744 (2)	C11—Cl3	1.742 (2)
C5—C6	1.376 (3)	C12—H12	0.9300
C5—H5	0.9300	N1—S1	1.6274 (19)
C6—H6	0.9300	N1—H1N	0.844 (16)
C7—C12	1.391 (3)	O1—S1	1.4207 (16)
C7—C8	1.390 (3)	O2—S1	1.4387 (15)
C6—C1—C2	120.5 (2)	C7—C8—H8	120.9
C6—C1—S1	120.01 (17)	C10—C9—C8	123.1 (2)
C2—C1—S1	119.48 (17)	C10—C9—Cl2	118.69 (17)
C3—C2—C1	119.6 (2)	C8—C9—Cl2	118.17 (19)
C3—C2—H2	120.2	C9—C10—C11	117.1 (2)
C1—C2—H2	120.2	C9—C10—H10	121.4
C2—C3—C4	119.2 (2)	C11—C10—H10	121.4
C2—C3—H3	120.4	C12—C11—C10	122.1 (2)
C4—C3—H3	120.4	C12—C11—Cl3	119.20 (18)
C3—C4—C5	121.8 (2)	C10—C11—Cl3	118.70 (18)
C3—C4—Cl1	119.2 (2)	C11—C12—C7	119.2 (2)
C5—C4—Cl1	119.0 (2)	C11—C12—H12	120.4
C6—C5—C4	119.0 (2)	C7—C12—H12	120.4
C6—C5—H5	120.5	C7—N1—S1	128.77 (14)
C4—C5—H5	120.5	C7—N1—H1N	116.4 (17)
C5—C6—C1	119.8 (2)	S1—N1—H1N	111.1 (17)
C5—C6—H6	120.1	O1—S1—O2	120.01 (10)
C1—C6—H6	120.1	O1—S1—N1	108.60 (10)
C12—C7—C8	120.25 (19)	O2—S1—N1	104.03 (9)
C12—C7—N1	116.01 (18)	O1—S1—C1	108.36 (10)
C8—C7—N1	123.65 (19)	O2—S1—C1	107.64 (10)
C9—C8—C7	118.2 (2)	N1—S1—C1	107.59 (10)
C9—C8—H8	120.9		
C6—C1—C2—C3	−1.0 (3)	C9—C10—C11—Cl3	−179.32 (18)

S1—C1—C2—C3	176.71 (18)	C10—C11—C12—C7	−0.9 (3)
C1—C2—C3—C4	0.5 (4)	Cl3—C11—C12—C7	179.18 (16)
C2—C3—C4—C5	0.4 (4)	C8—C7—C12—C11	0.3 (3)
C2—C3—C4—Cl1	−179.78 (18)	N1—C7—C12—C11	−176.30 (19)
C3—C4—C5—C6	−0.6 (4)	C12—C7—N1—S1	−162.46 (16)
Cl1—C4—C5—C6	179.53 (18)	C8—C7—N1—S1	21.1 (3)
C4—C5—C6—C1	0.0 (4)	C7—N1—S1—O1	−39.3 (2)
C2—C1—C6—C5	0.8 (3)	C7—N1—S1—O2	−168.24 (18)
S1—C1—C6—C5	−176.95 (18)	C7—N1—S1—C1	77.8 (2)
C12—C7—C8—C9	0.5 (3)	C6—C1—S1—O1	−151.25 (18)
N1—C7—C8—C9	176.8 (2)	C2—C1—S1—O1	31.0 (2)
C7—C8—C9—C10	−0.6 (3)	C6—C1—S1—O2	−20.1 (2)
C7—C8—C9—Cl2	−179.79 (16)	C2—C1—S1—O2	162.20 (17)
C8—C9—C10—C11	0.0 (4)	C6—C1—S1—N1	91.51 (19)
Cl2—C9—C10—C11	179.18 (17)	C2—C1—S1—N1	−86.23 (19)
C9—C10—C11—C12	0.8 (3)		

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O2[i]	0.84 (2)	2.08 (2)	2.917 (2)	170 (2)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*.