

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

B. Thimme Gowda,^{a*} Sabine Foro,^b P. G. Nirmala^a and Hartmut Fuess^b

^aDepartment of Chemistry, Mangalore University, Mangalagangothri 574 199, Mangalore, India, and ^bInstitute of Materials Science, Darmstadt University of Technology, Petersenstrasse 23, D-64287 Darmstadt, Germany
Correspondence e-mail: gowdabt@yahoo.com

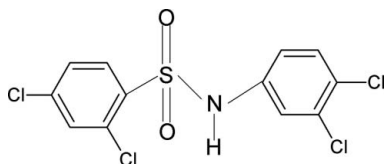
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Key indicators: single-crystal X-ray study; $T = 299$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.044; wR factor = 0.152; data-to-parameter ratio = 14.8.

In the crystal structure of the title compound, $\text{C}_{12}\text{H}_7\text{Cl}_4\text{NO}_2\text{S}$, the conformation of the N—H bond is *syn* to the *meta*-chloro residue in the aniline benzene ring. The two aromatic rings are tilted relative to each other by 68.9 (1)°. N—H...O hydrogen bonds connect the molecules into centrosymmetric dimers.

Related literature

For related structures, see: Gowda *et al.* (2008, 2009*a,b*). For comparative bond lengths in other aryl sulfonamides, see: Gelbrich *et al.* (2007); Perlovich *et al.* (2006).



Experimental

Crystal data

$\text{C}_{12}\text{H}_7\text{Cl}_4\text{NO}_2\text{S}$

$M_r = 371.05$

Triclinic, $P\bar{1}$

$a = 8.1498$ (9) Å

$b = 8.2633$ (9) Å

$c = 11.887$ (1) Å

$\alpha = 81.857$ (9)°

$\beta = 72.728$ (9)°

$\gamma = 78.213$ (9)°

$V = 745.49$ (13) Å³

$Z = 2$

Mo $K\alpha$ radiation

$\mu = 0.93$ mm⁻¹

$T = 299$ K

$0.48 \times 0.48 \times 0.28$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector

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

Diffraction, 2009)

$T_{\min} = 0.664$, $T_{\max} = 0.781$

7103 measured reflections

2722 independent reflections

2388 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.022$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$

$wR(F^2) = 0.152$

$S = 1.27$

2722 reflections

184 parameters

1 restraint

H atoms treated by a mixture of independent and constrained refinement

$\Delta\rho_{\text{max}} = 0.68$ e Å⁻³

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

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^{\text{i}}$	0.833 (17)	2.079 (18)	2.903 (3)	170 (3)

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

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: BT5008).

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supplementary materials

Acta Cryst. (2009). E65, o1940 [doi:10.1107/S1600536809027883]

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

B. T. Gowda, S. Foro, P. G. Nirmala and H. Fuess

Comment

In the present work, as part of a study of substituent effects on the structures of *N*-(aryl)-arylsulfonamides (Gowda *et al.*, 2008; Gowda *et al.*, 2009a; Gowda *et al.*, 2009b), the structure of 2,4-dichloro-*N*-(3,4-dichlorophenyl)benzenesulfonamide has been determined. The conformations of the N—C bonds in the C—SO₂—NH—C segment have *trans* and *gauche* torsion angles with the S=O bonds (Fig. 1). The molecule is bent at the S atom with the C—SO₂—NH—C torsion angle of -48.2 (2)°. The conformation of the N—H bond is *syn* to the *meta*-chloro group in the anilino benzene ring. The two aromatic rings are tilted relative to each other by 68.9 (1)°. The other bond parameters are similar to those observed in 2,4-dimethyl-*N*-(phenyl)benzenesulfonamide (Gowda *et al.*, 2009a); 4-methyl-*N*-(3,4-dimethylphenyl)-benzenesulfonamide (Gowda *et al.*, 2009b); *N*-(3-chlorophenyl)benzenesulfonamide (Gowda *et al.*, 2008) and other aryl sulfonamides (Perlovich *et al.*, 2006; Gelbrich *et al.*, 2007). The crystal packing *via* N—H···O(S) hydrogen bonds (Table 1) is shown in Fig. 2.

Experimental

The solution of 1,3-dichlorobenzene (10 cc) in chloroform (40 cc) was treated dropwise with chlorosulfonic acid (25 cc) 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 2,4-dichlorobenzenesulfonylchloride was treated with 3,4-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 cc). The resultant solid 2,4-dichloro-*N*-(3,4-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. The single crystals used in X-ray diffraction studies were grown in ethanolic solution by a slow evaporation at room temperature.

Refinement

The amino H atom was located in difference map and refined with restrained geometry to 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.

Figures

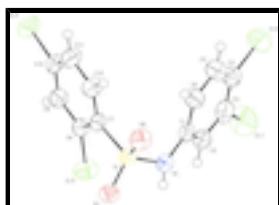


Fig. 1. Molecular structure of the title compound, showing the atom labelling scheme and displacement ellipsoids are drawn at the 50% probability level.

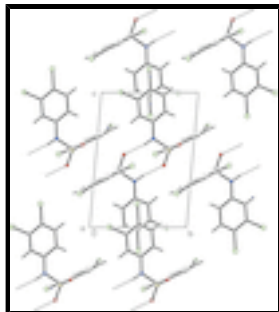


Fig. 2. Molecular packing of the title compound with hydrogen bonding shown as dashed lines.

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

Crystal data

$C_{12}H_7Cl_4NO_2S$	$Z = 2$
$M_r = 371.05$	$F_{000} = 372$
Triclinic, $P\bar{1}$	$D_x = 1.653 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 8.1498 (9) \text{ \AA}$	Cell parameters from 3818 reflections
$b = 8.2633 (9) \text{ \AA}$	$\theta = 2.5\text{--}27.5^\circ$
$c = 11.887 (1) \text{ \AA}$	$\mu = 0.93 \text{ mm}^{-1}$
$\alpha = 81.857 (9)^\circ$	$T = 299 \text{ K}$
$\beta = 72.728 (9)^\circ$	Plate, colourless
$\gamma = 78.213 (9)^\circ$	$0.48 \times 0.48 \times 0.28 \text{ mm}$
$V = 745.49 (13) \text{ \AA}^3$	

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector	2722 independent reflections
Radiation source: fine-focus sealed tube	2388 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.022$
$T = 100 \text{ K}$	$\theta_{\text{max}} = 25.4^\circ$
Rotation method data acquisition using ω and φ scans	$\theta_{\text{min}} = 2.5^\circ$
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2009)	$h = -9 \rightarrow 9$
$T_{\text{min}} = 0.664$, $T_{\text{max}} = 0.781$	$k = -9 \rightarrow 9$
7103 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.044$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.152$	$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$
	where $P = (F_o^2 + 2F_c^2)/3$

$S = 1.27$ $(\Delta/\sigma)_{\max} = 0.011$
 2722 reflections $\Delta\rho_{\max} = 0.68 \text{ e } \text{\AA}^{-3}$
 184 parameters $\Delta\rho_{\min} = -0.48 \text{ e } \text{\AA}^{-3}$
 1 restraint Extinction correction: none
 Primary atom site location: structure-invariant direct methods

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3911 (3)	0.5584 (3)	0.7834 (2)	0.0400 (6)
C2	0.3221 (3)	0.4144 (4)	0.8289 (2)	0.0464 (6)
H2	0.3248	0.3369	0.7785	0.056*
C3	0.2503 (3)	0.3869 (4)	0.9484 (3)	0.0551 (7)
C4	0.2429 (4)	0.5027 (4)	1.0245 (2)	0.0559 (7)
C5	0.3100 (4)	0.6460 (4)	0.9785 (3)	0.0590 (7)
H5	0.3034	0.7251	1.0288	0.071*
C6	0.3868 (4)	0.6734 (4)	0.8589 (2)	0.0509 (7)
H6	0.4355	0.7685	0.8291	0.061*
C7	0.2467 (3)	0.8593 (3)	0.61967 (19)	0.0337 (5)
C8	0.1074 (3)	0.7982 (3)	0.6056 (2)	0.0352 (5)
C9	-0.0613 (3)	0.8837 (3)	0.6440 (2)	0.0428 (6)
H9	-0.1543	0.8439	0.6338	0.051*
C10	-0.0895 (3)	1.0267 (3)	0.6968 (2)	0.0447 (6)
C11	0.0473 (3)	1.0924 (4)	0.7108 (3)	0.0513 (7)
H11	0.0262	1.1911	0.7458	0.062*
C12	0.2136 (3)	1.0071 (3)	0.6715 (2)	0.0448 (6)
H12	0.3063	1.0493	0.6798	0.054*
N1	0.4667 (3)	0.5772 (3)	0.65959 (18)	0.0410 (5)
H1N	0.478 (4)	0.500 (3)	0.618 (2)	0.049*
O1	0.5157 (2)	0.7111 (2)	0.46042 (14)	0.0419 (4)
O2	0.5641 (2)	0.8522 (2)	0.61286 (16)	0.0485 (5)
Cl1	0.16795 (16)	0.20525 (13)	1.00219 (9)	0.0922 (4)
Cl2	0.15005 (13)	0.47338 (13)	1.17468 (7)	0.0813 (3)

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C13	0.13671 (8)	0.61471 (9)	0.54309 (6)	0.0544 (3)
C14	-0.30088 (9)	1.12858 (12)	0.75011 (8)	0.0705 (3)
S1	0.46469 (7)	0.75391 (7)	0.57981 (5)	0.0351 (2)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0316 (12)	0.0451 (14)	0.0404 (12)	0.0022 (10)	-0.0130 (9)	-0.0006 (10)
C2	0.0439 (14)	0.0462 (15)	0.0468 (14)	-0.0028 (12)	-0.0137 (11)	-0.0012 (12)
C3	0.0428 (15)	0.0574 (18)	0.0573 (16)	-0.0035 (13)	-0.0135 (12)	0.0126 (14)
C4	0.0497 (16)	0.063 (2)	0.0445 (14)	0.0031 (14)	-0.0093 (12)	0.0021 (13)
C5	0.0676 (19)	0.0600 (19)	0.0475 (16)	-0.0058 (15)	-0.0149 (14)	-0.0083 (14)
C6	0.0569 (16)	0.0503 (17)	0.0459 (14)	-0.0101 (13)	-0.0143 (12)	-0.0040 (12)
C7	0.0279 (11)	0.0346 (12)	0.0387 (11)	-0.0069 (9)	-0.0102 (9)	0.0007 (9)
C8	0.0336 (12)	0.0363 (13)	0.0376 (11)	-0.0085 (10)	-0.0121 (9)	-0.0012 (9)
C9	0.0296 (12)	0.0553 (17)	0.0459 (13)	-0.0081 (11)	-0.0133 (10)	-0.0050 (12)
C10	0.0344 (12)	0.0496 (15)	0.0466 (13)	0.0040 (11)	-0.0136 (10)	-0.0041 (11)
C11	0.0471 (15)	0.0437 (15)	0.0649 (17)	0.0001 (12)	-0.0191 (13)	-0.0141 (13)
C12	0.0394 (14)	0.0414 (14)	0.0582 (15)	-0.0095 (11)	-0.0165 (11)	-0.0089 (12)
N1	0.0415 (11)	0.0400 (12)	0.0400 (11)	-0.0023 (9)	-0.0121 (9)	-0.0036 (9)
O1	0.0400 (9)	0.0414 (10)	0.0410 (9)	-0.0075 (7)	-0.0059 (7)	-0.0036 (7)
O2	0.0312 (9)	0.0583 (12)	0.0615 (11)	-0.0144 (8)	-0.0142 (8)	-0.0106 (9)
Cl1	0.1158 (8)	0.0755 (7)	0.0797 (7)	-0.0406 (6)	-0.0147 (6)	0.0204 (5)
Cl2	0.0879 (6)	0.0890 (7)	0.0450 (4)	-0.0018 (5)	0.0006 (4)	0.0053 (4)
Cl3	0.0432 (4)	0.0551 (5)	0.0720 (5)	-0.0139 (3)	-0.0140 (3)	-0.0250 (4)
Cl4	0.0396 (4)	0.0852 (6)	0.0818 (6)	0.0187 (4)	-0.0207 (4)	-0.0253 (5)
S1	0.0265 (3)	0.0384 (4)	0.0406 (4)	-0.0070 (3)	-0.0087 (2)	-0.0033 (3)

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

C1—C6	1.386 (4)	C7—S1	1.768 (2)
C1—C2	1.391 (4)	C8—C9	1.385 (3)
C1—N1	1.416 (3)	C8—Cl3	1.724 (2)
C2—C3	1.372 (4)	C9—C10	1.363 (4)
C2—H2	0.9300	C9—H9	0.9300
C3—C4	1.388 (4)	C10—C11	1.396 (4)
C3—Cl1	1.731 (3)	C10—Cl4	1.729 (3)
C4—C5	1.380 (4)	C11—C12	1.371 (4)
C4—Cl2	1.723 (3)	C11—H11	0.9300
C5—C6	1.380 (4)	C12—H12	0.9300
C5—H5	0.9300	N1—S1	1.625 (2)
C6—H6	0.9300	N1—H1N	0.833 (17)
C7—C12	1.387 (3)	O1—S1	1.4297 (17)
C7—C8	1.394 (3)	O2—S1	1.4208 (16)
C6—C1—C2	119.9 (2)	C7—C8—Cl3	121.98 (19)
C6—C1—N1	123.2 (2)	C10—C9—C8	119.3 (2)
C2—C1—N1	116.9 (2)	C10—C9—H9	120.3
C3—C2—C1	119.9 (3)	C8—C9—H9	120.3

C3—C2—H2	120.1	C9—C10—C11	121.9 (2)
C1—C2—H2	120.1	C9—C10—C14	119.22 (19)
C2—C3—C4	120.7 (3)	C11—C10—C14	118.8 (2)
C2—C3—C11	118.6 (2)	C12—C11—C10	118.0 (3)
C4—C3—C11	120.7 (2)	C12—C11—H11	121.0
C5—C4—C3	119.2 (3)	C10—C11—H11	121.0
C5—C4—C12	119.1 (2)	C11—C12—C7	121.5 (2)
C3—C4—C12	121.8 (2)	C11—C12—H12	119.2
C6—C5—C4	120.8 (3)	C7—C12—H12	119.2
C6—C5—H5	119.6	C1—N1—S1	124.52 (17)
C4—C5—H5	119.6	C1—N1—H1N	121 (2)
C5—C6—C1	119.6 (3)	S1—N1—H1N	110 (2)
C5—C6—H6	120.2	O2—S1—O1	118.63 (10)
C1—C6—H6	120.2	O2—S1—N1	109.79 (11)
C12—C7—C8	119.0 (2)	O1—S1—N1	104.74 (10)
C12—C7—S1	117.67 (17)	O2—S1—C7	105.89 (11)
C8—C7—S1	123.32 (18)	O1—S1—C7	111.08 (10)
C9—C8—C7	120.2 (2)	N1—S1—C7	106.12 (11)
C9—C8—C13	117.83 (17)		
C6—C1—C2—C3	0.0 (4)	C8—C9—C10—C11	1.7 (4)
N1—C1—C2—C3	178.6 (2)	C8—C9—C10—C14	-177.29 (18)
C1—C2—C3—C4	1.1 (4)	C9—C10—C11—C12	-1.1 (4)
C1—C2—C3—C11	-179.28 (19)	C14—C10—C11—C12	177.9 (2)
C2—C3—C4—C5	-0.4 (4)	C10—C11—C12—C7	-0.3 (4)
C11—C3—C4—C5	179.9 (2)	C8—C7—C12—C11	1.1 (4)
C2—C3—C4—C12	178.9 (2)	S1—C7—C12—C11	-176.5 (2)
C11—C3—C4—C12	-0.8 (4)	C6—C1—N1—S1	-34.0 (3)
C3—C4—C5—C6	-1.3 (5)	C2—C1—N1—S1	147.43 (19)
C12—C4—C5—C6	179.4 (2)	C1—N1—S1—O2	65.2 (2)
C4—C5—C6—C1	2.3 (5)	C1—N1—S1—O1	-166.37 (17)
C2—C1—C6—C5	-1.7 (4)	C1—N1—S1—C7	-48.8 (2)
N1—C1—C6—C5	179.8 (2)	C12—C7—S1—O2	0.2 (2)
C12—C7—C8—C9	-0.5 (4)	C8—C7—S1—O2	-177.34 (19)
S1—C7—C8—C9	176.96 (18)	C12—C7—S1—O1	-129.9 (2)
C12—C7—C8—C13	-179.53 (19)	C8—C7—S1—O1	52.6 (2)
S1—C7—C8—C13	-2.0 (3)	C12—C7—S1—N1	116.8 (2)
C7—C8—C9—C10	-0.8 (4)	C8—C7—S1—N1	-60.7 (2)
C13—C8—C9—C10	178.2 (2)		

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	0.833 (17)	2.079 (18)	2.903 (3)	170 (3)

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

Fig. 1

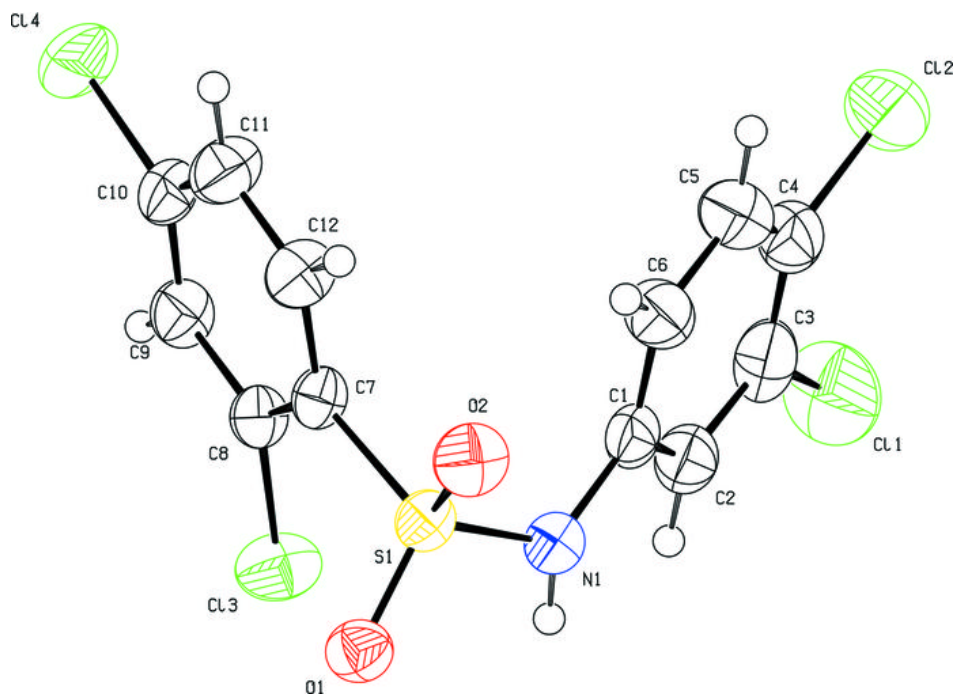


Fig. 2

