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ISSN 2414-3146

3,5-Dichloro-*N*-(4-methylphenyl)benzenesulfonamide

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Received 5 March 2017
Accepted 8 March 2017

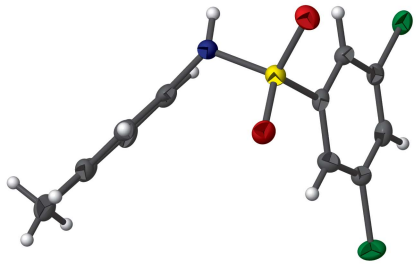
Edited by H. Stoeckli-Evans, University of Neuchâtel, Switzerland

Keywords: crystal structure; sulfonamides; N—H···O hydrogen bonding; C—H···O hydrogen bonding; C—H···π interactions.

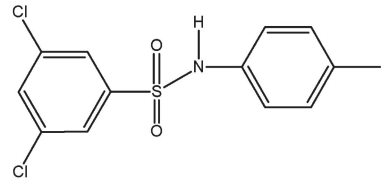
Structural data: full structural data are available from iucrdata.iucr.org

The molecule of the title compound, $C_{13}H_{11}Cl_2NO_2S$, is U-shaped with the central C—S—N—C segment having a torsion angle of 67.2 (4)°. The dihedral angle between the benzene rings is 57.0 (2)°. In the crystal, molecules are linked *via* N—H···O and C—H···O hydrogen bonds, forming chains propagating along the *a*-axis direction. The chains are linked by C—H···π interactions, forming a three-dimensional supramolecular structure.

3D view



Chemical scheme



Structure description

In recent years, extensive research has been carried out on the synthesis and evaluation of the pharmacological activities of molecules containing the sulfonamide moiety (Mohan *et al.*, 2013). As part of our ongoing studies on sulfonamides (Shakuntala *et al.*, 2017), we report herein on the synthesis and crystal structure of the title compound.

The molecule of the title compound, Fig. 1, is U-shaped with the central C1—S1—N1—C7 segment having a torsion angle of 67.2 (4)°. The dihedral angle between the benzene rings is 57.0 (2)°.

In the crystal, molecules are linked *via* N—H···O and C—H···O hydrogen bonds, forming chains propagating along [100]; see Table 1 and Fig. 2. The chains are linked by C—H···π interactions, forming a three-dimensional supramolecular structure (Fig. 3 and Table 1). The shortest Cl···Cl separation is 3.438 (1) Å (Fig. 3).

Synthesis and crystallization

The title compound was prepared according to a literature method (Rodrigues *et al.*, 2015). The purity of the compound was checked by determining its melting point.

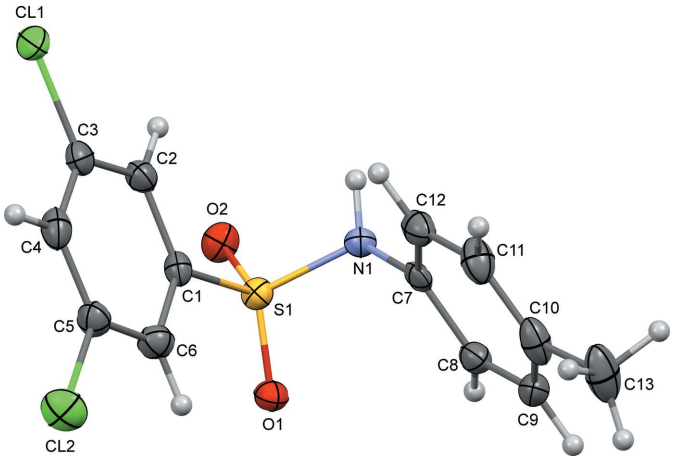


Figure 1
A view of the molecular structure of the title compound, with the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

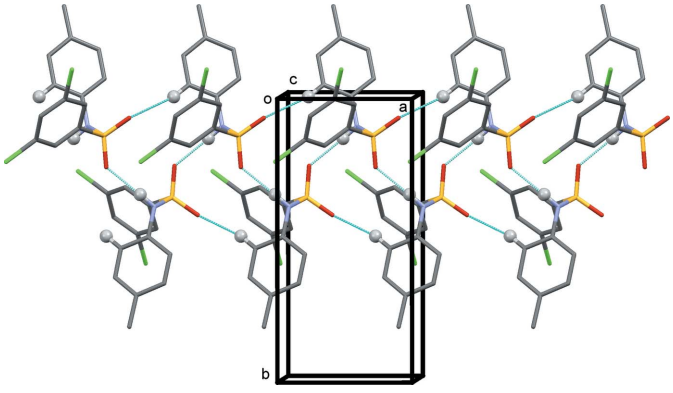


Figure 2
A partial view along the *c* axis of the crystal packing of the title compound. The hydrogen bonds are shown as dashed lines (see Table 1) and only the H atoms (grey balls) that are involved in hydrogen bonding are shown.

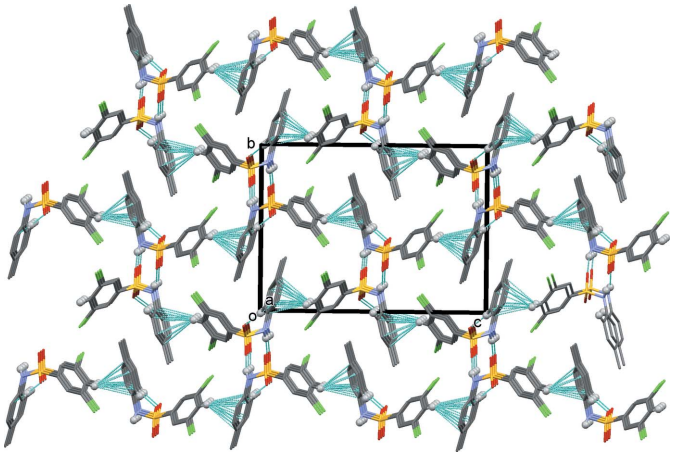


Figure 3
A view along the *a* axis of the crystal packing of the title compound. The hydrogen bonds and C—H$\cdots\pi$ interactions are shown as dashed lines (see Table 1) and only the H atoms (grey balls) that are involved in these interactions are shown.

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

Cg is the centroid of the C7–C12 aniline ring

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
N1—H1$\cdots$O2^{i}	0.87 (4)	2.00 (4)	2.866 (6)	171 (5)
C12—H12$\cdots$O1^{ii}	0.95	2.52	3.455 (6)	167
C4—H4$\cdots$*Cg*^{iii}	0.95	2.80	3.602 (6)	143

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (ii) $x-1, y, z$; (iii) $-x+\frac{3}{2}, -y+1, z+\frac{1}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{11}Cl_2NO_2S$
M_r	316.19
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	100
a, *b*, *c* (Å)	6.1673 (3), 13.0059 (7), 17.6433 (9)
V (Å^3)	1415.19 (13)
Z	4
Radiation type	Cu *K*α
μ (mm^{-1})	5.49
Crystal size (mm)	0.29 × 0.24 × 0.22
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.258, 0.299
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7822, 2292, 2210
R_{int}	0.060
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.584
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.052, 0.138, 0.99
No. of reflections	2292
No. of parameters	177
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.42, −0.59
Absolute structure	Flack *x* determined using 855 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.101 (13)

Computer programs: *APEX2*, *SAINT-Plus* and *XPREP* (Bruker, 2009), *SHELXT2016* (Sheldrick, 2015*a*), *Mercury* (Macrae *et al.*, 2008), *SHELXL2016* (Sheldrick, 2015*b*) and *PLATON* (Spek, 2009).

Colourless prismatic crystals were obtained by slow evaporation of a solution in ethanol, at room temperature, m.p. = 453 K.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors are thankful to the Institution of Excellence, Vijnana Bhavana, University of Mysore, Mysore, for providing the single-crystal X-ray diffraction data. KS is grateful to the

University Grants Commission (UGC), New Delhi, for the financial assistance under its MRP scheme.

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full crystallographic data

IUCrData (2017). **2**, x170375 [https://doi.org/10.1107/S2414314617003753]

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Crystal data

$C_{13}H_{11}Cl_2NO_2S$
M_r = 316.19
Orthorhombic, $P2_12_12_1$
a = 6.1673 (3) Å
b = 13.0059 (7) Å
c = 17.6433 (9) Å
V = 1415.19 (13) Å^3
Z = 4
$F(000)$ = 648
D_x = 1.484 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 143 reflections
θ = 6.1–64.2°
μ = 5.49 mm^{-1}
T = 100 K
Prism, colourless
0.29 × 0.24 × 0.22 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
ω and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
T_{min} = 0.258, T_{max} = 0.299
7822 measured reflections
2292 independent reflections
2210 reflections with $I > 2\sigma(I)$
R_{int} = 0.060
θ_{max} = 64.2°, θ_{min} = 6.1°
h = −7→7
k = −14→14
l = −18→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.138
S = 0.99
2292 reflections
177 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.115P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.59 e Å^{-3}
Absolute structure: Flack x determined using 855 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.101 (13)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
CL1	0.4625 (2)	0.27187 (11)	0.71332 (6)	0.0310 (4)
CL2	0.9584 (2)	0.59855 (10)	0.78818 (7)	0.0365 (4)
S1	1.17445 (18)	0.35953 (9)	0.54747 (6)	0.0191 (4)
O1	1.3636 (5)	0.4224 (3)	0.55489 (18)	0.0236 (8)
O2	1.1925 (6)	0.2491 (3)	0.54433 (18)	0.0256 (8)
C4	0.7242 (9)	0.4340 (4)	0.7437 (3)	0.0281 (12)
H4	0.628893	0.449882	0.784407	0.034*
C5	0.9032 (9)	0.4936 (4)	0.7298 (3)	0.0250 (11)
C6	1.0463 (9)	0.4729 (4)	0.6700 (3)	0.0260 (11)
H6	1.170170	0.514515	0.661025	0.031*
C1	0.9987 (8)	0.3891 (4)	0.6248 (2)	0.0193 (10)
N1	1.0491 (7)	0.3909 (3)	0.4694 (2)	0.0213 (9)
C7	1.0053 (8)	0.4973 (4)	0.4532 (2)	0.0200 (10)
C12	0.8004 (9)	0.5373 (4)	0.4662 (3)	0.0271 (11)
H12	0.689690	0.495458	0.487485	0.032*
C11	0.7588 (9)	0.6397 (5)	0.4478 (3)	0.0312 (12)
H11	0.618169	0.667346	0.456150	0.037*
C10	0.9196 (10)	0.7019 (4)	0.4174 (3)	0.0304 (13)
C13	0.8682 (13)	0.8123 (4)	0.3951 (3)	0.0437 (16)
H13A	0.788698	0.812679	0.346961	0.066*
H13B	1.003609	0.850936	0.389253	0.066*
H13C	0.779222	0.844344	0.434548	0.066*
C3	0.6852 (8)	0.3502 (4)	0.6972 (3)	0.0235 (11)
C2	0.8198 (8)	0.3268 (4)	0.6371 (3)	0.0231 (11)
H2	0.790606	0.269702	0.605079	0.028*
C9	1.1261 (9)	0.6600 (4)	0.4053 (3)	0.0259 (11)
H9	1.237690	0.701855	0.384760	0.031*
C8	1.1697 (8)	0.5578 (4)	0.4231 (2)	0.0236 (10)
H8	1.310023	0.529869	0.414837	0.028*
H1	0.944 (7)	0.348 (3)	0.460 (3)	0.022 (14)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
CL1	0.0262 (6)	0.0443 (8)	0.0225 (6)	−0.0092 (6)	0.0011 (5)	0.0037 (5)
CL2	0.0462 (8)	0.0292 (7)	0.0340 (7)	−0.0001 (6)	0.0017 (6)	−0.0137 (5)
S1	0.0187 (6)	0.0187 (6)	0.0199 (6)	0.0012 (5)	0.0017 (4)	−0.0006 (4)
O1	0.0188 (16)	0.0291 (19)	0.0231 (16)	−0.0022 (15)	−0.0015 (14)	−0.0001 (14)
O2	0.0279 (19)	0.0195 (18)	0.0293 (17)	0.0056 (15)	0.0035 (15)	−0.0003 (14)
C4	0.028 (3)	0.037 (3)	0.020 (2)	0.005 (2)	0.002 (2)	0.006 (2)
C5	0.031 (3)	0.022 (2)	0.022 (2)	−0.001 (2)	−0.002 (2)	−0.001 (2)
C6	0.028 (3)	0.022 (3)	0.029 (2)	−0.002 (2)	−0.003 (2)	0.000 (2)
C1	0.020 (2)	0.023 (2)	0.015 (2)	0.004 (2)	0.0004 (18)	0.0020 (17)
N1	0.0230 (19)	0.020 (2)	0.020 (2)	−0.0035 (19)	−0.0014 (17)	0.0001 (16)
C7	0.026 (2)	0.021 (2)	0.0126 (19)	−0.002 (2)	−0.006 (2)	−0.0027 (17)

C12	0.024 (2)	0.038 (3)	0.019 (2)	−0.002 (2)	0.001 (2)	−0.002 (2)
C11	0.030 (3)	0.040 (3)	0.024 (3)	0.012 (3)	0.000 (2)	0.003 (2)
C10	0.047 (3)	0.026 (3)	0.019 (2)	0.011 (3)	−0.007 (2)	−0.003 (2)
C13	0.076 (5)	0.026 (3)	0.029 (3)	0.015 (3)	−0.011 (3)	0.001 (2)
C3	0.024 (2)	0.026 (3)	0.021 (2)	0.000 (2)	−0.005 (2)	0.0057 (19)
C2	0.027 (3)	0.024 (3)	0.019 (2)	−0.002 (2)	−0.004 (2)	−0.0010 (18)
C9	0.029 (3)	0.027 (3)	0.021 (2)	−0.007 (2)	−0.003 (2)	0.005 (2)
C8	0.023 (2)	0.028 (3)	0.020 (2)	−0.003 (2)	−0.0027 (19)	−0.002 (2)

Geometric parameters (Å, º)

CL1—C3	1.734 (5)	C7—C8	1.389 (7)
CL2—C5	1.743 (5)	C12—C11	1.396 (8)
S1—O1	1.430 (4)	C12—H12	0.9500
S1—O2	1.442 (4)	C11—C10	1.387 (9)
S1—N1	1.631 (4)	C11—H11	0.9500
S1—C1	1.784 (5)	C10—C9	1.402 (8)
C4—C5	1.371 (8)	C10—C13	1.523 (7)
C4—C3	1.386 (8)	C13—H13A	0.9800
C4—H4	0.9500	C13—H13B	0.9800
C5—C6	1.402 (8)	C13—H13C	0.9800
C6—C1	1.383 (7)	C3—C2	1.381 (7)
C6—H6	0.9500	C2—H2	0.9500
C1—C2	1.386 (7)	C9—C8	1.391 (8)
N1—C7	1.439 (6)	C9—H9	0.9500
N1—H1	0.87 (3)	C8—H8	0.9500
C7—C12	1.386 (7)		
O1—S1—O2	120.7 (2)	C11—C12—H12	120.4
O1—S1—N1	108.7 (2)	C10—C11—C12	121.0 (5)
O2—S1—N1	104.7 (2)	C10—C11—H11	119.5
O1—S1—C1	107.6 (2)	C12—C11—H11	119.5
O2—S1—C1	106.9 (2)	C11—C10—C9	118.8 (5)
N1—S1—C1	107.7 (2)	C11—C10—C13	120.1 (6)
C5—C4—C3	118.6 (5)	C9—C10—C13	121.1 (6)
C5—C4—H4	120.7	C10—C13—H13A	109.5
C3—C4—H4	120.7	C10—C13—H13B	109.5
C4—C5—C6	122.2 (5)	H13A—C13—H13B	109.5
C4—C5—CL2	119.6 (4)	C10—C13—H13C	109.5
C6—C5—CL2	118.2 (4)	H13A—C13—H13C	109.5
C1—C6—C5	116.9 (5)	H13B—C13—H13C	109.5
C1—C6—H6	121.5	C2—C3—C4	121.6 (5)
C5—C6—H6	121.5	C2—C3—CL1	118.2 (4)
C6—C1—C2	122.6 (5)	C4—C3—CL1	120.2 (4)
C6—C1—S1	118.9 (4)	C3—C2—C1	118.0 (4)
C2—C1—S1	118.5 (4)	C3—C2—H2	121.0
C7—N1—S1	119.8 (3)	C1—C2—H2	121.0
C7—N1—H1	116 (4)	C8—C9—C10	120.9 (5)

S1—N1—H1	111 (4)	C8—C9—H9	119.6
C12—C7—C8	121.1 (5)	C10—C9—H9	119.6
C12—C7—N1	119.9 (5)	C9—C8—C7	119.1 (5)
C8—C7—N1	119.0 (4)	C9—C8—H8	120.4
C7—C12—C11	119.2 (5)	C7—C8—H8	120.4
C7—C12—H12	120.4		

C3—C4—C5—C6	0.5 (8)	C8—C7—C12—C11	0.9 (7)
C3—C4—C5—CL2	−179.2 (4)	N1—C7—C12—C11	−177.9 (4)
C4—C5—C6—C1	0.2 (7)	C7—C12—C11—C10	−0.6 (7)
CL2—C5—C6—C1	179.9 (4)	C12—C11—C10—C9	0.1 (8)
C5—C6—C1—C2	−0.4 (7)	C12—C11—C10—C13	177.8 (5)
C5—C6—C1—S1	179.7 (4)	C5—C4—C3—C2	−1.0 (7)
O1—S1—C1—C6	10.0 (4)	C5—C4—C3—CL1	179.3 (4)
O2—S1—C1—C6	141.0 (4)	C4—C3—C2—C1	0.8 (7)
N1—S1—C1—C6	−107.0 (4)	CL1—C3—C2—C1	−179.4 (3)
O1—S1—C1—C2	−169.9 (3)	C6—C1—C2—C3	−0.1 (7)
O2—S1—C1—C2	−39.0 (4)	S1—C1—C2—C3	179.8 (4)
N1—S1—C1—C2	73.0 (4)	C11—C10—C9—C8	0.2 (7)
O1—S1—N1—C7	−49.1 (4)	C13—C10—C9—C8	−177.4 (4)
O2—S1—N1—C7	−179.3 (4)	C10—C9—C8—C7	0.0 (7)
C1—S1—N1—C7	67.2 (4)	C12—C7—C8—C9	−0.6 (6)
S1—N1—C7—C12	−100.5 (5)	N1—C7—C8—C9	178.2 (4)
S1—N1—C7—C8	80.6 (5)		

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C7–C12 aniline ring

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.87 (4)	2.00 (4)	2.866 (6)	171 (5)
C12—H12···O1^{ii}	0.95	2.52	3.455 (6)	167
C4—H4···*Cg*^{iii}	0.95	2.80	3.602 (6)	143

Symmetry codes: (i) *x*−1/2, −*y*+1/2, −*z*+1; (ii) *x*−1, *y*, *z*; (iii) −*x*+3/2, −*y*+1, *z*+1/2.