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N,N'-(Ethane-1,2-diyl)bis(4-chlorobenzenesulfonamide)

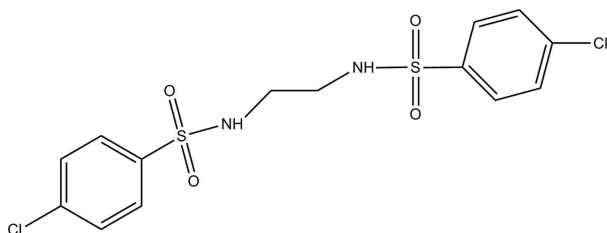
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 Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.054; wR factor = 0.175; data-to-parameter ratio = 32.4.

 The title molecule, $\text{C}_{14}\text{H}_{14}\text{Cl}_2\text{N}_2\text{O}_4\text{S}_2$, lies on an inversion center. The molecule is twisted in the region of the sulfonamide group with a $\text{C}-\text{S}-\text{N}-\text{C}$ torsion angle of -67.49 (16)°. In the crystal, molecules are connected *via* intermolecular $\text{N}-\text{H}\cdots\text{O}$ and weak $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, forming layers parallel to (100).

Related literature

 For details of the chemistry of sulfonamides, see: Gowda *et al.* (2003, 2007). For related structures, see: O'Connor & Maslen (1965); Kumar *et al.* (1992); Shakuntala *et al.* (2011).


Experimental

Crystal data

 $\text{C}_{14}\text{H}_{14}\text{Cl}_2\text{N}_2\text{O}_4\text{S}_2$
 $M_r = 409.29$
 Monoclinic, $P2_1/c$
 $a = 13.2640$ (5) Å
 $b = 5.3390$ (2) Å

 $c = 13.1792$ (5) Å
 $\beta = 110.270$ (1)°
 $V = 875.51$ (6) Å³
 $Z = 2$
 Mo $K\alpha$ radiation

 $\mu = 0.63$ mm⁻¹
 $T = 296$ K

 $0.86 \times 0.42 \times 0.13$ mm

Data collection

 Bruker APEXII DUO CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2009)
 $T_{\min} = 0.613$, $T_{\max} = 0.925$

 25142 measured reflections
 3689 independent reflections
 2869 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.054$
 $wR(F^2) = 0.175$
 $S = 1.03$
 3689 reflections
 114 parameters

 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.84$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.71$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N1}\cdots\text{O2}^{\text{i}}$	0.79 (3)	2.13 (3)	2.903 (2)	167 (3)
$\text{C4}-\text{H4A}\cdots\text{O1}^{\text{ii}}$	0.93	2.50	3.157 (2)	127

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

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5285).

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Acta Cryst. (2011). E67, o2241 [doi:10.1107/S1600536811028443]

***N,N'*-(Ethane-1,2-diyl)bis(4-chlorobenzenesulfonamide)**

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S1. Comment

The chemistry of sulfonamides is of interest as they show distinct physical, chemical and biological properties. Many arylsulfonamides and their *N*-halo compounds exhibit pharmacological, fungicidal and herbicidal activities due to their oxidizing action in aqueous, partial aqueous and non-aqueous media. 2-chlorobenzenesulfonamide has been used to explore the substituent effects on the solid state structures of sulfonamides and *N*-haloarylsulfonamides (Gowda *et al.*, 2003, 2007). The crystal structures of 4-aminobenzenesulfonamide (O'Connor & Maslen, 1965) and 4-methylbenzenesulfonamide (Kumar *et al.*, 1992) have been reported in the literature. In this paper, we present the X-ray single-crystal structure of *N,N'*-(ethane-1,2-diyl)bis(4-chlorobenzenesulfonamide) (I).

The asymmetric unit of the title compound, (I), consists of a half molecule of *N,N'*-(ethane-1,2-diyl)bis(4-chlorobenzenesulfonamide). The other half is generated by a crystallographic inversion center, as shown in Fig. 1. The molecule is twisted at the S atom with an C-S-N-C torsion angle of $-67.49(16)^\circ$. This value agrees with that previously reported for the crystal structure of 4-Chloro-*N*-(2,6-dimethylphenyl)benzene sulfonamide (Shakuntala *et al.*, 2011). In the crystal, molecules are connected *via* N1—H1N1 \cdots O2ⁱⁱ and C4—H4A \cdots O1ⁱⁱⁱ (Table 1) hydrogen bonds (Fig. 2) forming layers parallel to (100).

S2. Experimental

In a round bottom flask, 25ml of toluene was mixed with 4-chlorobenzenesulfonyl chloride (0.02 mol, 3.5 g) with stirring. Drops of ethylenediamine (0.01mol, 0.5 g) were added and the mixture was refluxed for 30 min. The yellow gum formed was dissolved in hot water and sodium bicarbonate was added. The yellow precipitate formed was dissolved in methanol at 333K, yielding colourless crystals.

S3. Refinement

Atom H1N1 was located from a difference Fourier maps and refined freely [N—H = 0.78 (3) Å]. The remaining H atoms were positioned geometrically [C—H = 0.93–0.97 Å] and were refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

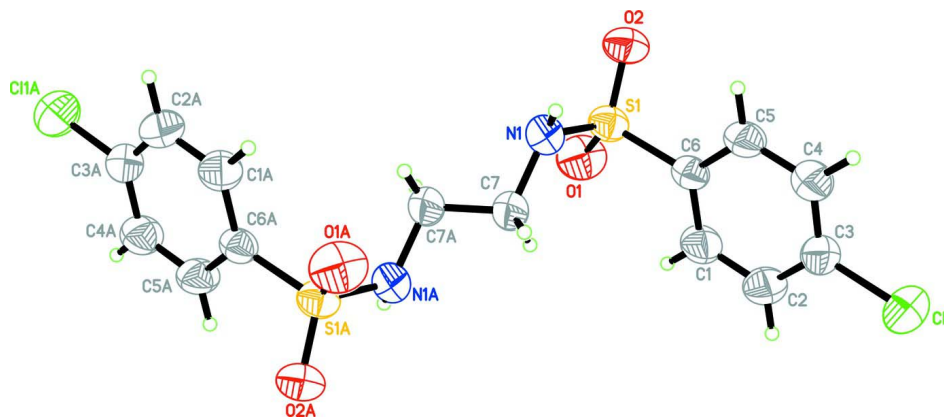


Figure 1

The molecular structure of the title compound showing 50% probability displacement ellipsoids (symmetry code: (A) - $x+1, -y+2, -z$).

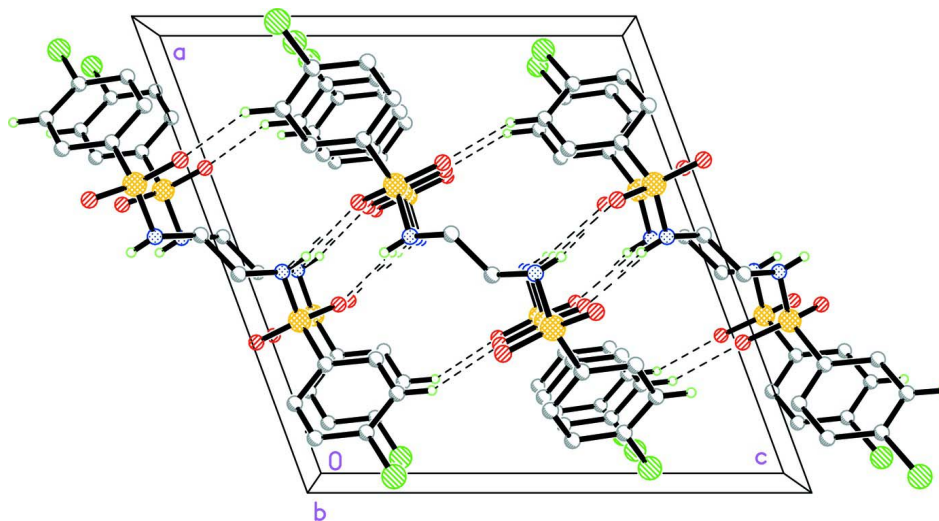


Figure 2

The crystal packing of (I) with dashed lines representing hydrogen bonds.

4-chloro-*N*-[2-(4-chlorobenzenesulfonamido)ethyl]benzenesulfonamide

Crystal data

$C_{14}H_{14}Cl_2N_2O_4S_2$

$M_r = 409.29$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 13.2640$ (5) Å

$b = 5.3390$ (2) Å

$c = 13.1792$ (5) Å

$\beta = 110.270$ (1)°

$V = 875.51$ (6) Å³

$Z = 2$

$F(000) = 420$

$D_x = 1.553$ Mg m⁻³

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

Cell parameters from 9471 reflections

$\theta = 3.3$ – 33.5 °

$\mu = 0.63$ mm⁻¹

$T = 296$ K

Plate, colourless

$0.86 \times 0.42 \times 0.13$ mm

Data collection

Bruker APEXII DUO CCD area-detector
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
 φ and ω scans
Absorption correction: multi-scan
(SADABS; Bruker, 2009)
 $T_{\min} = 0.613$, $T_{\max} = 0.925$

25142 measured reflections
3689 independent reflections
2869 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$
 $\theta_{\max} = 34.5^\circ$, $\theta_{\min} = 3.1^\circ$
 $h = -21 \rightarrow 21$
 $k = -8 \rightarrow 8$
 $l = -19 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.054$
 $wR(F^2) = 0.175$
 $S = 1.03$
3689 reflections
114 parameters
0 restraints
Primary atom site location: structure-invariant
direct methods
Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites
H atoms treated by a mixture of independent
and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0979P)^2 + 0.221P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.84 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.71 \text{ e } \text{\AA}^{-3}$
Extinction correction: SHELXTL (Sheldrick,
2008), $F_c^* = kFc[1 + 0.001x \text{Fc}^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.037 (6)

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.05348 (6)	1.46054 (14)	0.19657 (6)	0.0881 (2)
S1	0.35715 (3)	0.67863 (7)	0.09928 (3)	0.04701 (14)
O1	0.30882 (14)	0.5925 (3)	-0.00946 (11)	0.0722 (4)
O2	0.39055 (12)	0.5017 (2)	0.18611 (12)	0.0615 (3)
N1	0.46306 (13)	0.8326 (3)	0.10557 (14)	0.0537 (3)
C1	0.18737 (14)	1.0017 (4)	0.04025 (13)	0.0572 (4)
H1A	0.1784	0.9592	-0.0308	0.069*
C2	0.12110 (16)	1.1788 (4)	0.06200 (16)	0.0648 (5)
H2A	0.0680	1.2581	0.0056	0.078*
C3	0.13465 (14)	1.2362 (4)	0.16811 (17)	0.0564 (4)
C4	0.21201 (17)	1.1220 (4)	0.25315 (15)	0.0602 (4)
H4A	0.2191	1.1612	0.3240	0.072*
C5	0.27916 (15)	0.9482 (4)	0.23194 (12)	0.0543 (4)
H5A	0.3325	0.8707	0.2888	0.065*
C6	0.26714 (11)	0.8888 (3)	0.12547 (11)	0.0428 (3)

C7	0.45903 (17)	1.0240 (4)	0.02466 (18)	0.0635 (5)
H7A	0.4710	1.1877	0.0587	0.076*
H7B	0.3884	1.0241	-0.0311	0.076*
H1N1	0.496 (2)	0.868 (6)	0.166 (2)	0.076 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0889 (4)	0.0823 (4)	0.1081 (5)	0.0331 (3)	0.0532 (4)	0.0217 (3)
S1	0.0555 (2)	0.0437 (2)	0.03914 (19)	-0.00060 (14)	0.01292 (15)	-0.00090 (12)
O1	0.0873 (10)	0.0712 (9)	0.0468 (6)	0.0035 (8)	0.0090 (6)	-0.0168 (6)
O2	0.0717 (8)	0.0495 (7)	0.0620 (7)	0.0049 (6)	0.0214 (6)	0.0146 (5)
N1	0.0538 (7)	0.0590 (8)	0.0516 (7)	0.0028 (6)	0.0226 (6)	0.0065 (6)
C1	0.0508 (8)	0.0756 (12)	0.0390 (7)	0.0038 (7)	0.0079 (6)	0.0079 (7)
C2	0.0524 (9)	0.0799 (13)	0.0563 (9)	0.0152 (8)	0.0113 (7)	0.0193 (9)
C3	0.0522 (8)	0.0566 (9)	0.0653 (10)	0.0068 (7)	0.0265 (7)	0.0122 (8)
C4	0.0711 (11)	0.0637 (10)	0.0484 (8)	0.0112 (9)	0.0241 (8)	0.0060 (7)
C5	0.0620 (9)	0.0605 (9)	0.0374 (6)	0.0110 (7)	0.0135 (6)	0.0078 (6)
C6	0.0431 (6)	0.0460 (7)	0.0366 (5)	-0.0031 (5)	0.0104 (5)	0.0047 (5)
C7	0.0724 (11)	0.0577 (10)	0.0775 (12)	0.0207 (8)	0.0477 (10)	0.0202 (9)

Geometric parameters (Å, °)

Cl1—C3	1.7359 (19)	C2—C3	1.381 (3)
S1—O1	1.4282 (14)	C2—H2A	0.9300
S1—O2	1.4308 (13)	C3—C4	1.373 (3)
S1—N1	1.6048 (16)	C4—C5	1.380 (3)
S1—C6	1.7574 (16)	C4—H4A	0.9300
N1—C7	1.465 (2)	C5—C6	1.393 (2)
N1—H1N1	0.78 (3)	C5—H5A	0.9300
C1—C6	1.386 (2)	C7—C7 ⁱ	1.469 (3)
C1—C2	1.387 (3)	C7—H7A	0.9700
C1—H1A	0.9300	C7—H7B	0.9700
O1—S1—O2	119.79 (10)	C2—C3—Cl1	119.94 (14)
O1—S1—N1	107.30 (10)	C3—C4—C5	119.05 (17)
O2—S1—N1	105.90 (9)	C3—C4—H4A	120.5
O1—S1—C6	107.66 (8)	C5—C4—H4A	120.5
O2—S1—C6	107.92 (8)	C4—C5—C6	120.03 (15)
N1—S1—C6	107.76 (8)	C4—C5—H5A	120.0
C7—N1—S1	120.62 (14)	C6—C5—H5A	120.0
C7—N1—H1N1	116 (2)	C1—C6—C5	120.40 (16)
S1—N1—H1N1	110 (2)	C1—C6—S1	119.90 (12)
C6—C1—C2	119.32 (16)	C5—C6—S1	119.66 (11)
C6—C1—H1A	120.3	N1—C7—C7 ⁱ	110.75 (19)
C2—C1—H1A	120.3	N1—C7—H7A	109.5
C3—C2—C1	119.42 (16)	C7 ⁱ —C7—H7A	109.5
C3—C2—H2A	120.3	N1—C7—H7B	109.5

C1—C2—H2A	120.3	C7 ⁱ —C7—H7B	109.5
C4—C3—C2	121.75 (18)	H7A—C7—H7B	108.1
C4—C3—C11	118.31 (16)		
O1—S1—N1—C7	48.19 (17)	C2—C1—C6—S1	-176.04 (15)
O2—S1—N1—C7	177.22 (15)	C4—C5—C6—C1	-0.5 (3)
C6—S1—N1—C7	-67.49 (16)	C4—C5—C6—S1	176.93 (15)
C6—C1—C2—C3	-1.0 (3)	O1—S1—C6—C1	-20.18 (17)
C1—C2—C3—C4	-0.2 (3)	O2—S1—C6—C1	-150.79 (14)
C1—C2—C3—C11	179.13 (16)	N1—S1—C6—C1	95.26 (15)
C2—C3—C4—C5	1.1 (3)	O1—S1—C6—C5	162.35 (16)
C11—C3—C4—C5	-178.26 (16)	O2—S1—C6—C5	31.74 (16)
C3—C4—C5—C6	-0.7 (3)	N1—S1—C6—C5	-82.21 (15)
C2—C1—C6—C5	1.4 (3)	S1—N1—C7—C7 ⁱ	-126.2 (2)

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

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N1—H1N1...O2 ⁱⁱ	0.79 (3)	2.13 (3)	2.903 (2)	167 (3)
C4—H4A...O1 ⁱⁱⁱ	0.93	2.50	3.157 (2)	127

Symmetry codes: (ii) $-x+1, y+1/2, -z+1/2$; (iii) $x, -y+3/2, z+1/2$.