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N,N'-Bis(4-chlorophenylsulfonyl)-adipamide

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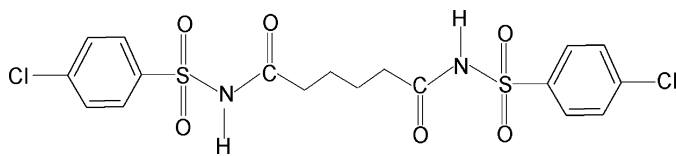
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.009$ Å; R factor = 0.081; wR factor = 0.104; data-to-parameter ratio = 12.6.

In the title compound, $\text{C}_{18}\text{H}_{18}\text{Cl}_2\text{N}_2\text{O}_6\text{S}_2$, the asymmetric unit contains half a molecule with a center of symmetry at the midpoint of the central C—C bond. The dihedral angle between the benzene ring and the $\text{SO}_2\text{—NH—C(O)}$ segment in the two halves of the molecule is $83.5(2)^\circ$. In the crystal, $\text{N—H}\cdots\text{O}(\text{S})$ intermolecular hydrogen bonds link the molecules into infinite chains running along the c axis. The O atom involved in the hydrogen bond has a longer S—O bond than the other O atom bonded to S [1.403 (4) versus 1.361 (4) Å].

Related literature

For hydrogen-bonding preferences of sulfonamides, see: Adsmoind & Grant (2001). For our studies on the effects of substituents on the structures of *N*-(aryl)-amides, see: Bhat & Gowda (2000); Gowda *et al.* (2000, 2007). For those on *N*-(arylsulfonyl)-amides, see: Rodrigues *et al.* (2011*a,b*). For those on *N*-(aryl)-arylsulfonamides, see: Gowda *et al.* (2005).



Experimental

Crystal data

 $\text{C}_{18}\text{H}_{18}\text{Cl}_2\text{N}_2\text{O}_6\text{S}_2$
 $M_r = 493.36$

 Triclinic, $P\bar{1}$
 $a = 5.593(1)$ Å

 $b = 8.827(2)$ Å
 $c = 9.908(2)$ Å
 $\alpha = 89.28(2)^\circ$
 $\beta = 87.75(2)^\circ$
 $\gamma = 81.16(1)^\circ$
 $V = 482.96(17)$ Å³
 $Z = 1$
 Mo $K\alpha$ radiation
 $\mu = 0.60$ mm⁻¹
 $T = 293$ K
 $0.12 \times 0.08 \times 0.04$ mm

Data collection

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

 Diffraction, 2009)
 $T_{\min} = 0.932$, $T_{\max} = 0.977$
 2942 measured reflections
 1757 independent reflections
 775 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.063$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.081$
 $wR(F^2) = 0.104$
 $S = 0.99$
 1757 reflections
 139 parameters
 2 restraints

 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.41$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.36$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D\text{—H}\cdots A$	$D\text{—H}$	$\text{H}\cdots A$	$D\cdots A$	$D\text{—H}\cdots A$
$\text{N1—HN}\cdots\text{O2}^i$	0.85 (2)	2.03 (3)	2.839 (7)	160 (6)

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

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: ZJ2018).

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supporting information

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N,N'*-Bis(4-chlorophenylsulfonyl)adipamide*Vinola Z. Rodrigues, Sabine Foro and B. Thimme Gowda****S1. Comment**

The amide moiety is an important constituent of many biologically significant compounds. As part of our studies on the effects of ring and side chain substitutions on the structures of *N*-(aryl)-amides (Bhat & Gowda, 2000; Gowda *et al.*, 2000, 2007), *N*-(arylsulfonyl)-amides (Rodrigues *et al.*, 2011*a,b*) and *N*-(aryl)-arylsulfonamides (Gowda *et al.*, 2005), the crystal structure of *N,N*-bis(4-chlorophenylsulfonyl)-adipamide has been determined (I) (Fig. 1).

In the two C—SO₂—NH—CO—CH₂—CH₂ central segments of the structure, the N—H, C=O and C—H bonds are *anti* to the adjacent bonds, similar to that observed in *N,N*-bis(2-chlorophenylsulfonyl)-adipamide (II) (Rodrigues *et al.*, 2011*a*) and *N,N*-bis(4-chlorophenylsulfonyl)-suberamide (III) (Rodrigues *et al.*, 2011*b*). The orientations of sulfonamide groups with respect to the attached phenyl rings are given by the torsion angles of C2—C1—S1—N1 = -117.1 (6)° and C6—C1—S1—N1 = 60.5 (6)°. The molecule is bent at the S atom with the C1—S1—N1—C7 torsion angle of 55.0 (6)°, compared to the value of -65.1 (6)° in (II).

The dihedral angle between the benzene ring and the SO₂—NH—C(O) segment in the two halves of the molecule is 83.5 (2)°, compared to the values of 89.6 (2)° in (II) and 79.5 (2)° in (III).

N—H...O2(S) H-bond formation results in an S=O2 bond longer than the S=O1 bond [1.403 (4)Å *versus* 1.361 (4) Å]. A series of N—H...O(S) intermolecular hydrogen bonds (Table 1) link the molecules into infinite chains running along *c*-axis (Fig. 2). The hydrogen bonding preferences of sulfonamides is described elsewhere (Adsmund & Grant, 2001)

S2. Experimental

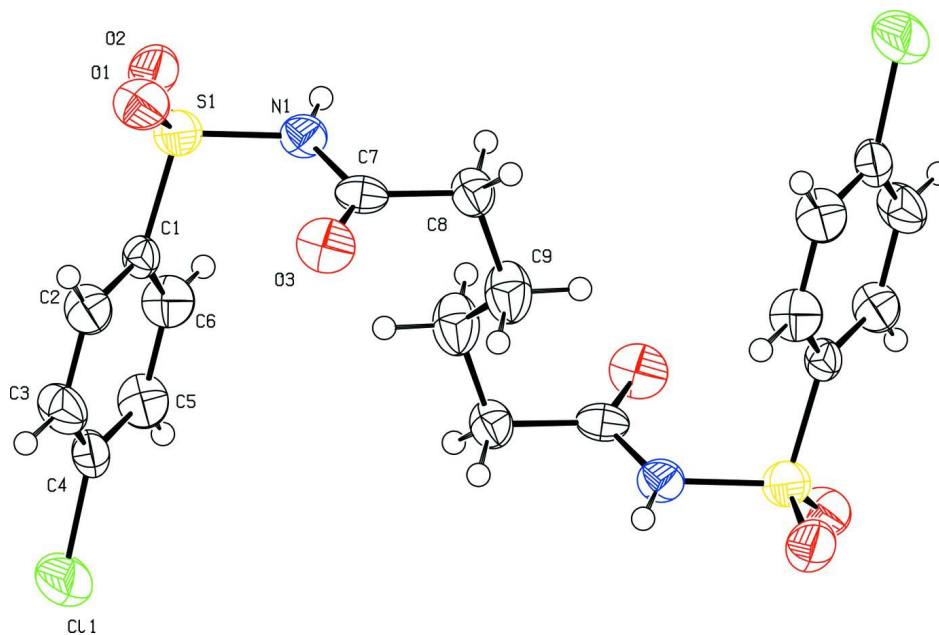
N,N-Bis(4-chlorophenylsulfonyl)-adipamide was prepared by refluxing a mixture of adipic acid (0.01 mol) with 4-chlorobenzenesulfonamide (0.02 mol) and POCl₃ for 1 hr on a water bath. The reaction mixture was allowed to cool and added ether to it. The solid product obtained was filtered, washed thoroughly with ether and hot ethanol. The compound was recrystallized to the constant melting point and was characterized by its infrared and NMR spectra.

Needle like colorless single crystals used in the X-ray diffraction studies were grown by a slow evaporation of a solution of the compound in ethanol at room temperature.

S3. Refinement

The H atom of the NH group was located in a difference map and later restrained to N—H = 0.86 (2) %A. The other H atoms were positioned with idealized geometry using a riding model with the aromatic C—H = 0.93 Å and the methylene C—H = 0.97 Å. All H atoms were refined with isotropic displacement parameters (set to 1.2 times of the *U*_{eq} of the parent atom).

The distance C1—C6 in the benzene ring was restrained to 1.39 (1) Å.

**Figure 1**

Molecular structure of (I) showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented as small spheres of arbitrary radii.

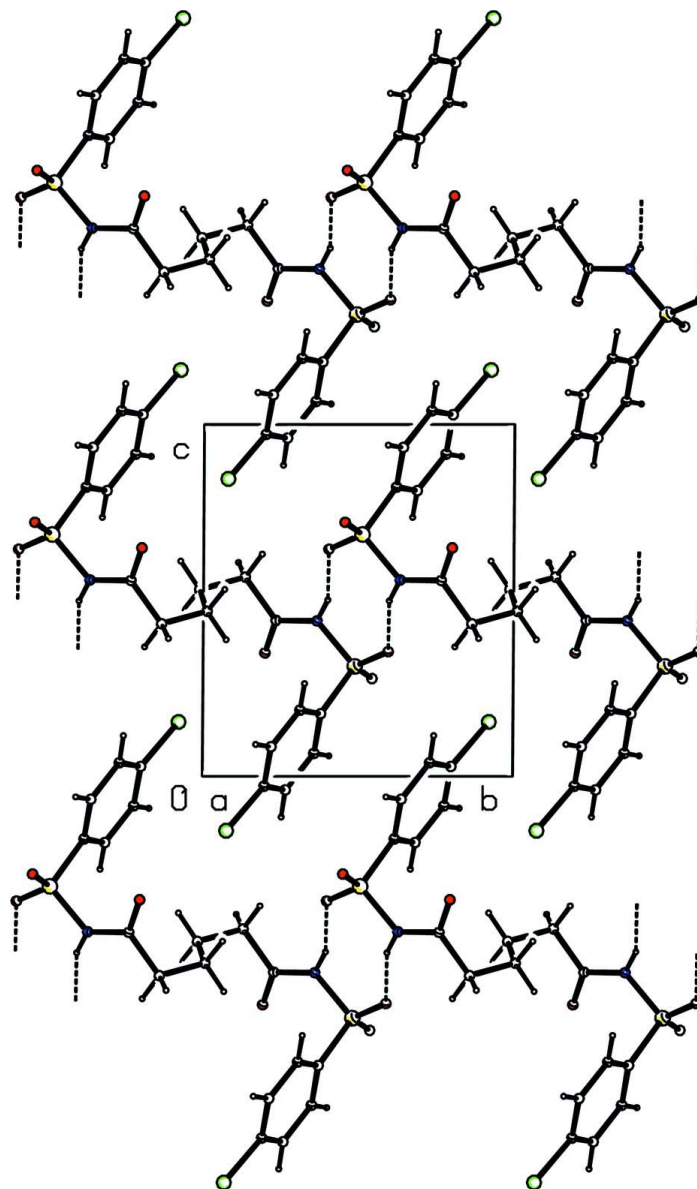


Figure 2

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

***N,N'*-Bis(4-chlorophenylsulfonyl)adipamide**

Crystal data

$C_{18}H_{18}Cl_2N_2O_6S_2$

$M_r = 493.36$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 5.593\ (1)\ \text{\AA}$

$b = 8.827\ (2)\ \text{\AA}$

$c = 9.908\ (2)\ \text{\AA}$

$\alpha = 89.28\ (2)^\circ$

$\beta = 87.75\ (2)^\circ$

$\gamma = 81.16\ (1)^\circ$

$V = 482.96\ (17)\ \text{\AA}^3$

$Z = 1$

$F(000) = 254$

$D_x = 1.696\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 528 reflections

$\theta = 3.1\text{--}28.0^\circ$

$\mu = 0.60\ \text{mm}^{-1}$

$T = 293$ K $0.12 \times 0.08 \times 0.04$ mm
 Needle, colourless

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector	2942 measured reflections 1757 independent reflections
Radiation source: fine-focus sealed tube	775 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{\text{int}} = 0.063$
Rotation method data acquisition using ω scans	$\theta_{\text{max}} = 25.4^\circ$, $\theta_{\text{min}} = 3.1^\circ$
Absorption correction: multi-scan (<i>CrysAlis RED</i> ; Oxford Diffraction, 2009)	$h = -6 \rightarrow 6$ $k = -10 \rightarrow 10$ $l = -11 \rightarrow 11$
$T_{\text{min}} = 0.932$, $T_{\text{max}} = 0.977$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.081$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.104$	$w = 1/[\sigma^2(F_o^2) + (0.P)^2]$
$S = 0.99$	where $P = (F_o^2 + 2F_c^2)/3$
1757 reflections	$(\Delta/\sigma)_{\text{max}} = 0.007$
139 parameters	$\Delta\rho_{\text{max}} = 0.41 \text{ e } \text{\AA}^{-3}$
2 restraints	$\Delta\rho_{\text{min}} = -0.36 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

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	0.1041 (11)	0.6193 (7)	0.8224 (6)	0.0293 (17)
C2	0.2221 (12)	0.6416 (7)	0.9348 (6)	0.042 (2)
H2	0.3810	0.5932	0.9421	0.051*
C3	0.1175 (13)	0.7325 (8)	1.0388 (7)	0.047 (2)
H3	0.2030	0.7448	1.1155	0.057*
C4	-0.1031 (13)	0.8010 (8)	1.0280 (7)	0.042 (2)
C5	-0.2239 (12)	0.7794 (8)	0.9163 (7)	0.045 (2)
H5	-0.3822	0.8291	0.9100	0.054*
C6	-0.1242 (11)	0.6877 (7)	0.8116 (7)	0.0433 (19)
H6	-0.2120	0.6738	0.7361	0.052*
C7	0.3253 (12)	0.7659 (8)	0.5567 (7)	0.0357 (18)
C8	0.2725 (11)	0.8657 (7)	0.4335 (6)	0.0394 (18)
H8A	0.4234	0.8879	0.3921	0.047*

H8B	0.1936	0.8111	0.3683	0.047*
C9	0.1140 (10)	1.0122 (8)	0.4692 (6)	0.054 (2)
H9A	0.1970	1.0689	0.5309	0.065*
H9B	0.0846	1.0738	0.3881	0.065*
N1	0.2328 (10)	0.6336 (6)	0.5582 (5)	0.0371 (15)
H1N	0.149 (9)	0.600 (6)	0.499 (4)	0.044*
O1	0.4677 (8)	0.4551 (5)	0.7221 (4)	0.0501 (14)
O2	0.0961 (8)	0.4035 (5)	0.6456 (4)	0.0508 (14)
O3	0.4340 (7)	0.8007 (5)	0.6492 (5)	0.0491 (14)
Cl1	-0.2314 (4)	0.9221 (2)	1.15456 (19)	0.0695 (7)
S1	0.2378 (4)	0.5111 (2)	0.68742 (19)	0.0419 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.032 (4)	0.030 (4)	0.026 (4)	-0.004 (4)	-0.006 (3)	0.003 (3)
C2	0.035 (5)	0.049 (5)	0.039 (5)	0.004 (4)	-0.001 (4)	0.002 (4)
C3	0.047 (5)	0.060 (6)	0.031 (5)	0.005 (5)	-0.011 (4)	-0.001 (4)
C4	0.049 (5)	0.042 (5)	0.031 (5)	0.002 (4)	0.004 (4)	0.009 (4)
C5	0.026 (4)	0.056 (5)	0.049 (5)	0.005 (4)	0.000 (4)	0.005 (4)
C6	0.032 (4)	0.050 (5)	0.048 (5)	-0.004 (4)	-0.012 (4)	0.001 (4)
C7	0.024 (4)	0.040 (5)	0.041 (5)	-0.001 (4)	0.004 (4)	-0.013 (4)
C8	0.035 (4)	0.045 (5)	0.036 (5)	0.000 (4)	0.000 (3)	0.005 (4)
C9	0.042 (5)	0.064 (6)	0.053 (5)	0.001 (5)	-0.001 (4)	0.017 (4)
N1	0.041 (4)	0.037 (4)	0.035 (4)	-0.008 (3)	-0.010 (3)	-0.005 (3)
O1	0.036 (3)	0.058 (3)	0.050 (3)	0.013 (3)	-0.006 (2)	-0.003 (3)
O2	0.067 (4)	0.042 (3)	0.047 (3)	-0.017 (3)	-0.018 (3)	0.000 (3)
O3	0.031 (3)	0.062 (4)	0.055 (4)	-0.008 (3)	-0.012 (3)	-0.005 (3)
Cl1	0.0784 (16)	0.0701 (16)	0.0514 (14)	0.0107 (12)	0.0185 (12)	-0.0023 (12)
S1	0.0447 (13)	0.0403 (13)	0.0394 (12)	-0.0009 (11)	-0.0076 (10)	-0.0022 (11)

Geometric parameters (Å, °)

C1—C6	1.333 (6)	C7—N1	1.348 (7)
C1—C2	1.348 (8)	C7—C8	1.508 (8)
C1—S1	1.731 (6)	C8—C9	1.489 (7)
C2—C3	1.370 (7)	C8—H8A	0.9700
C2—H2	0.9300	C8—H8B	0.9700
C3—C4	1.295 (8)	C9—C9 ⁱ	1.437 (10)
C3—H3	0.9300	C9—H9A	0.9700
C4—C5	1.350 (8)	C9—H9B	0.9700
C4—Cl1	1.719 (7)	N1—S1	1.664 (6)
C5—C6	1.371 (7)	N1—H1N	0.85 (2)
C5—H5	0.9300	O1—S1	1.361 (4)
C6—H6	0.9300	O2—S1	1.403 (4)
C7—O3	1.188 (7)		
C6—C1—C2	119.0 (6)	C9—C8—C7	111.2 (5)

C6—C1—S1	117.8 (5)	C9—C8—H8A	109.4
C2—C1—S1	123.2 (5)	C7—C8—H8A	109.4
C1—C2—C3	122.7 (7)	C9—C8—H8B	109.4
C1—C2—H2	118.7	C7—C8—H8B	109.4
C3—C2—H2	118.7	H8A—C8—H8B	108.0
C4—C3—C2	118.6 (7)	C9 ⁱ —C9—C8	112.4 (7)
C4—C3—H3	120.7	C9 ⁱ —C9—H9A	109.1
C2—C3—H3	120.7	C8—C9—H9A	109.1
C3—C4—C5	119.4 (7)	C9 ⁱ —C9—H9B	109.1
C3—C4—C11	119.0 (6)	C8—C9—H9B	109.1
C5—C4—C11	121.6 (6)	H9A—C9—H9B	107.9
C4—C5—C6	123.1 (7)	C7—N1—S1	125.5 (5)
C4—C5—H5	118.4	C7—N1—H1N	128 (4)
C6—C5—H5	118.4	S1—N1—H1N	106 (4)
C1—C6—C5	117.2 (6)	O1—S1—O2	116.6 (3)
C1—C6—H6	121.4	O1—S1—N1	112.0 (3)
C5—C6—H6	121.4	O2—S1—N1	103.7 (3)
O3—C7—N1	121.0 (7)	O1—S1—C1	106.7 (3)
O3—C7—C8	123.7 (7)	O2—S1—C1	112.4 (3)
N1—C7—C8	115.3 (6)	N1—S1—C1	105.0 (3)
C6—C1—C2—C3	-0.3 (10)	C7—C8—C9—C9 ⁱ	-59.2 (9)
S1—C1—C2—C3	177.3 (5)	O3—C7—N1—S1	4.7 (9)
C1—C2—C3—C4	-0.6 (11)	C8—C7—N1—S1	-173.2 (4)
C2—C3—C4—C5	0.8 (11)	C7—N1—S1—O1	-60.3 (6)
C2—C3—C4—C11	-177.1 (5)	C7—N1—S1—O2	173.2 (5)
C3—C4—C5—C6	-0.2 (11)	C7—N1—S1—C1	55.0 (6)
C11—C4—C5—C6	177.8 (5)	C6—C1—S1—O1	179.5 (5)
C2—C1—C6—C5	1.0 (10)	C2—C1—S1—O1	1.8 (6)
S1—C1—C6—C5	-176.8 (4)	C6—C1—S1—O2	-51.6 (6)
C4—C5—C6—C1	-0.8 (10)	C2—C1—S1—O2	130.8 (5)
O3—C7—C8—C9	-63.7 (9)	C6—C1—S1—N1	60.5 (6)
N1—C7—C8—C9	114.2 (6)	C2—C1—S1—N1	-117.1 (6)

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1N \cdots O2 ⁱⁱ	0.85 (2)	2.03 (3)	2.839 (7)	160 (6)

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