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N,*N*′-Bis(2-chlorophenylsulfonyl)adipamide

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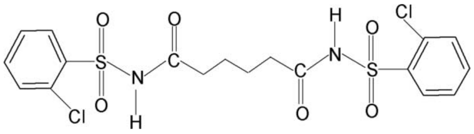
Key indicators: single-crystal X-ray study; *T* = 293 K; mean σ(C–C) = 0.011 Å; *R* factor = 0.089; *wR* factor = 0.143; data-to-parameter ratio = 14.2.

In the centrosymmetric title compound, $C_{18}H_{18}Cl_2N_2O_6S_2$, the conformation of the N—H and C═O bonds in the C—SO$_2$—NH—C(O)—C—C segment is *anti* to each other. The dihedral angle between the planes of the benzene ring and the central part of the molecule is 89.6 (2)°. In the crystal, intermolecular N—H···O(S) hydrogen bonds link the molecules into sheets along the *b* axis.

Related literature

For the effect of substituents on the structures of amides and sulfonamides, see: Gowda *et al.* (2000, 2005); Rodrigues *et al.* (2011).



Experimental

Crystal data

$C_{18}H_{18}Cl_2N_2O_6S_2$
M_r = 493.36
Monoclinic, $P2_1/n$
a = 11.899 (2) Å
b = 5.564 (1) Å
c = 16.333 (3) Å
β = 96.56 (2)°
V = 1074.3 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.54 mm^{-1}
T = 293 K
0.44 × 0.08 × 0.01 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.799, T_{max} = 0.995
3439 measured reflections
1971 independent reflections
1120 reflections with $I > 2\sigma(I)$
R_{int} = 0.049

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.089
$wR(F^2)$ = 0.143
S = 1.26
1971 reflections
139 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^{i}	0.84 (3)	2.08 (3)	2.901 (6)	168 (6)

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

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: BQ2284).

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supporting information

Acta Cryst. (2011). E**67**, o837 [doi:10.1107/S1600536811008464]

N,*N′*-Bis(2-chlorophenylsulfonyl)adipamide

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S1. Comment

The amide and sulfonamide moieties are important constituents of many biologically significant compounds. As a part of studying the effect of substituents on the structures of this class of compounds (Gowda *et al.*, 2000, 2005; Rodrigues *et al.*, 2011), in the present work, the structure of *N*,*N*-bis(2-chlorophenylsulfonyl)-adipamide (I) has been determined (Fig.1). The asymmetric unit comprises half of a molecule, the remaining portion is generated through an inversion centre, similar to that observed in *N*,*N*-bis(2-methylphenylsulfonyl)-adipamide (II) (Rodrigues *et al.*, 2011). The conformation of the N—H and C=O bonds in the C—SO_2—NH—C(O)—C—C segment is *anti* to each other and the amide O atom is also *anti* to the H atoms attached to the adjacent C atom. The molecule is bent at the S atom with the C—SO_2—NH—C(O) torsion angle of -65.1 (6)°, compared to the value of -63.7 (4)° in (II). Further, the S1—N1—C7—C8 and C7—N1—S1—O1 segments are nearly linear. The torsion angles C2—C1—S1—N1 and C6—C1—S1—N1 are -69.5 (6)° and 108.8 (5)°, respectively. The corresponding values in (II) are -71.3 (4)° and 106.9 (4)°.

The dihedral angle between the planes of the benzene ring and the SO_2—NH—C(O)—C—C segment in (I) is 89.6 (2)°, compared to the value of 89.9 (1)° in (II).

N—H···O1(S) H-bond formation results in an S=O1 bond longer than the S=O2 bond. A series of N—H···O(S) intermolecular hydrogen bonds (Table 1) link the molecules into sheets running in the direction of *b* axis (Fig. 2).

S2. Experimental

N,*N*-Bis(2-chlorophenylsulfonyl)-adipamide was prepared by refluxing a mixture of adipic acid (0.01 mol) with 2-chlorobenzenesulfonamide (0.02 mol) and $POCl_3$ 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 the solution of the compound in ethanol at room temperature.

S3. Refinement

The H atom of the NH group was located in a difference Fourier map and later restrained to the distance N—H = 0.86 (3) Å. The other H atoms were positioned with idealized geometry using a riding model with aromatic C—H distance = 0.93 Å and 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).

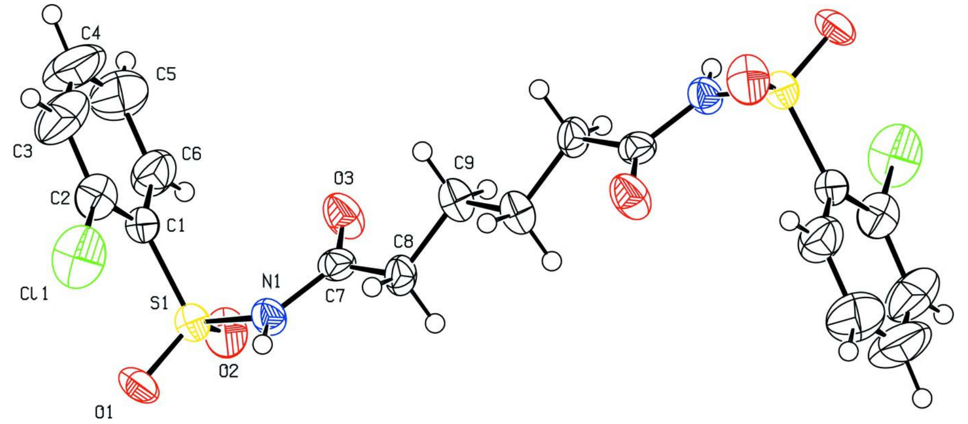

Figure 1

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

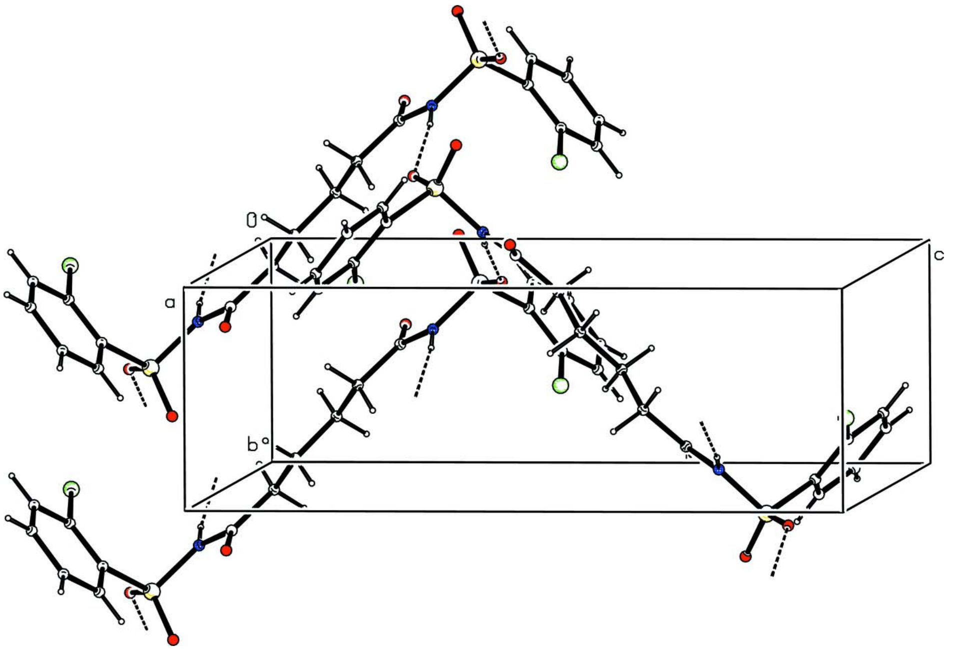

Figure 2

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

N,*N′*-Bis(2-chlorophenylsulfonyl)heptanediamide

Crystal data

$C_{18}H_{18}Cl_2N_2O_6S_2$
M_r = 493.36
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 11.899 (2) Å
b = 5.564 (1) Å
c = 16.333 (3) Å
β = 96.56 (2)°
V = 1074.3 (3) Å^3
Z = 2
F(000) = 508
D_x = 1.525 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 660 reflections
θ = 2.9–27.9°
μ = 0.54 mm^{-1}

T = 293 K	0.44 × 0.08 × 0.01 mm
Needle, colourless	

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector	3439 measured reflections
Radiation source: fine-focus sealed tube	1971 independent reflections
Graphite monochromator	1120 reflections with $I > 2\sigma(I)$
Rotation method data acquisition using ω scans.	R_{int} = 0.049
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)	θ_{max} = 25.7°, θ_{min} = 3.5°
T_{min} = 0.799, T_{max} = 0.995	$h = -11 \rightarrow 14$
	$k = -6 \rightarrow 6$
	$l = -19 \rightarrow 17$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.089	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.143	$w = 1/[\sigma^2(F_o^2) + (0.0112P)^2 + 2.7686P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.26	$(\Delta/\sigma)_{\text{max}}$ = 0.004
1971 reflections	$\Delta\rho_{\text{max}}$ = 0.34 e Å^{-3}
139 parameters	$\Delta\rho_{\text{min}}$ = −0.30 e Å^{-3}
2 restraints	
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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Cl1	−0.14610 (17)	0.2330 (4)	0.10849 (12)	0.0762 (7)
S1	−0.08046 (13)	−0.2093 (3)	0.23869 (9)	0.0378 (4)
O1	−0.1884 (3)	−0.2383 (8)	0.1916 (2)	0.0465 (12)
O2	−0.0261 (4)	−0.4127 (8)	0.2782 (3)	0.0538 (13)
O3	0.0751 (3)	0.0106 (9)	0.3729 (2)	0.0498 (13)
N1	−0.1028 (4)	−0.0070 (10)	0.3079 (3)	0.0365 (13)
H1N	−0.166 (3)	0.059 (10)	0.302 (3)	0.044*
C1	0.0147 (5)	−0.0762 (12)	0.1772 (3)	0.0361 (15)
C2	−0.0119 (6)	0.1105 (12)	0.1239 (4)	0.0470 (18)
C3	0.0707 (8)	0.2089 (17)	0.0807 (5)	0.081 (3)
H3	0.0530	0.3363	0.0447	0.098*

C4	0.1783 (9)	0.119 (2)	0.0912 (6)	0.098 (3)
H4	0.2336	0.1867	0.0627	0.118*
C5	0.2053 (7)	−0.069 (2)	0.1429 (6)	0.087 (3)
H5	0.2783	−0.1315	0.1486	0.105*
C6	0.1249 (6)	−0.1668 (15)	0.1864 (4)	0.060 (2)
H6	0.1437	−0.2941	0.2222	0.072*
C7	−0.0219 (5)	0.0781 (11)	0.3683 (4)	0.0349 (15)
C8	−0.0660 (4)	0.2510 (12)	0.4273 (3)	0.0375 (15)
H8A	−0.1021	0.1608	0.4679	0.045*
H8B	−0.1233	0.3515	0.3973	0.045*
C9	0.0245 (5)	0.4113 (11)	0.4718 (3)	0.0389 (16)
H9A	0.0803	0.3122	0.5039	0.047*
H9B	0.0625	0.4984	0.4315	0.047*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0866 (15)	0.0669 (15)	0.0744 (14)	0.0315 (13)	0.0064 (11)	0.0128 (12)
S1	0.0402 (9)	0.0383 (10)	0.0347 (9)	−0.0033 (8)	0.0034 (7)	−0.0051 (9)
O1	0.033 (2)	0.056 (3)	0.048 (3)	−0.014 (2)	−0.0038 (19)	−0.013 (2)
O2	0.076 (3)	0.035 (3)	0.049 (3)	0.002 (3)	0.001 (2)	0.003 (2)
O3	0.036 (2)	0.061 (3)	0.051 (3)	0.008 (2)	−0.005 (2)	−0.016 (2)
N1	0.028 (3)	0.047 (4)	0.035 (3)	0.002 (3)	0.002 (2)	−0.008 (3)
C1	0.038 (4)	0.039 (4)	0.031 (4)	−0.005 (3)	0.006 (3)	−0.005 (3)
C2	0.059 (4)	0.035 (4)	0.048 (4)	0.006 (3)	0.011 (4)	−0.003 (4)
C3	0.108 (8)	0.072 (6)	0.070 (6)	−0.004 (6)	0.036 (5)	0.022 (5)
C4	0.084 (7)	0.120 (10)	0.101 (8)	−0.021 (7)	0.054 (6)	0.008 (7)
C5	0.052 (5)	0.124 (9)	0.091 (7)	0.005 (6)	0.030 (5)	−0.006 (7)
C6	0.050 (4)	0.080 (6)	0.051 (5)	0.005 (4)	0.014 (4)	0.004 (4)
C7	0.030 (3)	0.039 (4)	0.036 (4)	−0.002 (3)	0.003 (3)	0.009 (3)
C8	0.039 (3)	0.045 (4)	0.028 (3)	0.002 (3)	0.005 (3)	−0.006 (3)
C9	0.041 (4)	0.038 (4)	0.036 (4)	0.006 (3)	−0.004 (3)	−0.006 (3)

Geometric parameters (Å, º)

Cl1—C2	1.728 (7)	C4—C5	1.359 (12)
S1—O2	1.421 (4)	C4—H4	0.9300
S1—O1	1.429 (4)	C5—C6	1.367 (10)
S1—N1	1.638 (5)	C5—H5	0.9300
S1—C1	1.760 (6)	C6—H6	0.9300
O3—C7	1.208 (6)	C7—C8	1.499 (8)
N1—C7	1.381 (7)	C8—C9	1.518 (7)
N1—H1N	0.84 (3)	C8—H8A	0.9700
C1—C2	1.370 (8)	C8—H8B	0.9700
C1—C6	1.397 (8)	C9—C9^{i}	1.511 (11)
C2—C3	1.386 (9)	C9—H9A	0.9700
C3—C4	1.367 (11)	C9—H9B	0.9700
C3—H3	0.9300		

O2—S1—O1	119.3 (3)	C4—C5—C6	119.9 (9)
O2—S1—N1	109.6 (3)	C4—C5—H5	120.1
O1—S1—N1	104.0 (2)	C6—C5—H5	120.1
O2—S1—C1	107.7 (3)	C5—C6—C1	120.4 (8)
O1—S1—C1	109.7 (3)	C5—C6—H6	119.8
N1—S1—C1	105.7 (3)	C1—C6—H6	119.8
C7—N1—S1	125.0 (4)	O3—C7—N1	121.4 (6)
C7—N1—H1N	119 (4)	O3—C7—C8	124.2 (5)
S1—N1—H1N	116 (4)	N1—C7—C8	114.4 (5)
C2—C1—C6	119.2 (6)	C7—C8—C9	113.8 (4)
C2—C1—S1	124.4 (5)	C7—C8—H8A	108.8
C6—C1—S1	116.4 (5)	C9—C8—H8A	108.8
C1—C2—C3	119.8 (7)	C7—C8—H8B	108.8
C1—C2—Cl1	122.3 (5)	C9—C8—H8B	108.8
C3—C2—Cl1	117.9 (6)	H8A—C8—H8B	107.7
C4—C3—C2	119.9 (8)	C9^{i}—C9—C8	112.0 (6)
C4—C3—H3	120.0	C9^{i}—C9—H9A	109.2
C2—C3—H3	120.0	C8—C9—H9A	109.2
C5—C4—C3	120.8 (8)	C9^{i}—C9—H9B	109.2
C5—C4—H4	119.6	C8—C9—H9B	109.2
C3—C4—H4	119.6	H9A—C9—H9B	107.9

O2—S1—N1—C7	50.8 (6)	C1—C2—C3—C4	0.3 (12)
O1—S1—N1—C7	179.4 (5)	Cl1—C2—C3—C4	179.7 (7)
C1—S1—N1—C7	−65.1 (6)	C2—C3—C4—C5	0.8 (15)
O2—S1—C1—C2	173.5 (5)	C3—C4—C5—C6	−1.4 (16)
O1—S1—C1—C2	42.1 (6)	C4—C5—C6—C1	0.8 (13)
N1—S1—C1—C2	−69.5 (6)	C2—C1—C6—C5	0.3 (11)
O2—S1—C1—C6	−8.2 (6)	S1—C1—C6—C5	−178.1 (6)
O1—S1—C1—C6	−139.6 (5)	S1—N1—C7—O3	2.1 (9)
N1—S1—C1—C6	108.8 (5)	S1—N1—C7—C8	−176.2 (5)
C6—C1—C2—C3	−0.9 (10)	O3—C7—C8—C9	23.4 (9)
S1—C1—C2—C3	177.3 (6)	N1—C7—C8—C9	−158.5 (5)
C6—C1—C2—Cl1	179.8 (5)	C7—C8—C9—C9^{i}	177.9 (6)
S1—C1—C2—Cl1	−1.9 (8)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^{ii}	0.84 (3)	2.08 (3)	2.901 (6)	168 (6)

Symmetry code: (ii) −*x*−1/2, *y*+1/2, −*z*+1/2.