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N,N'-[1,3-Phenylenebis(methylene)]-dibenzenesulfonamide

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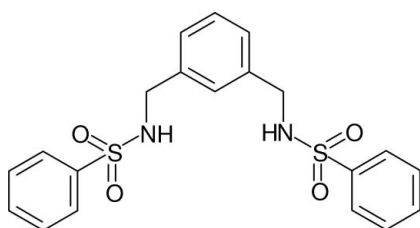
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 Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.037; wR factor = 0.103; data-to-parameter ratio = 17.9.

The complete molecule of the title compound, $\text{C}_{20}\text{H}_{20}\text{N}_2\text{O}_4\text{S}_2$, is generated by crystallographic twofold symmetry, with two C atoms lying on the rotation axis. The dihedral angle between the central benzene ring and the pendant ring is $68.42(6)^\circ$ and the dihedral angle between the pendant rings is $45.11(5)^\circ$. The torsion angles for the C–S–N–C and S–N–C–C fragments are $-73.22(15)$ and $-150.45(13)^\circ$, respectively. In the crystal, molecules are linked by N–H \cdots O hydrogen bonds, generating corrugated (001) sheets. Aromatic π – π stacking [centroid–centroid separation = $3.8925(12)$ and $3.9777(12)$ Å] and weak C–H \cdots O interactions also occur.

Related literature

 For a related structure, see: Khan *et al.* (2011).


Experimental

Crystal data

$\text{C}_{20}\text{H}_{20}\text{N}_2\text{O}_4\text{S}_2$
 $M_r = 416.50$
 Monoclinic, $C2/c$
 $a = 16.4319(6)$ Å
 $b = 7.8024(3)$ Å
 $c = 16.1217(6)$ Å
 $\beta = 111.543(2)^\circ$
 $V = 1922.54(12)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.31$ mm⁻¹
 $T = 296$ K
 $0.15 \times 0.11 \times 0.08$ mm

Data collection

Bruker APEXII CCD diffractometer
 8653 measured reflections
 2346 independent reflections
 1841 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.024$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.103$
 $S = 1.07$
 2346 reflections
 131 parameters
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.29$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.34$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{O2}^{\text{i}}$	0.81 (2)	2.16 (2)	2.960 (2)	166 (2)
$\text{C5}-\text{H5}\cdots\text{O1}^{\text{ii}}$	0.93	2.55	3.461 (2)	166
$\text{C10}-\text{H10}\cdots\text{O1}^{\text{iii}}$	0.93	2.57	3.379 (3)	146

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); 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: SU2323).

References

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 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supporting information

Acta Cryst. (2011). E67, o3037 [doi:10.1107/S1600536811040694]

N,N'*-[1,3-Phenylenebis(methylene)]dibenzenesulfonamide*Ejaz, Islam Ullah Khan, Hira Ahmad and William T. A. Harrison****S1. Comment**

As part of our ongoing structural studies of sulfonamides (Khan *et al.*, 2011), the synthesis and structure of the title compound are described herein.

The complete molecule of the title compound is generated by crystallographic twofold symmetry (Fig. 1), with atoms C9 and C11 lying on the rotation axis. The dihedral angle between the central benzene ring and the pendant ring is 68.42 (6)°. The dihedral angle between the two pendant rings is 45.11 (5)°. The conformation of the C4—S1—N1—C7 fragment is approximately *gauche* [-73.22 (15)°] whereas the torsion angle for S1—N1—C8—C9 of -150.45 (13)° indicates a conformation between *gauche* and *anti*. The bond-angle sum for N1 of 350.6° seems to indicate a valence state close to sp^2 hybridization.

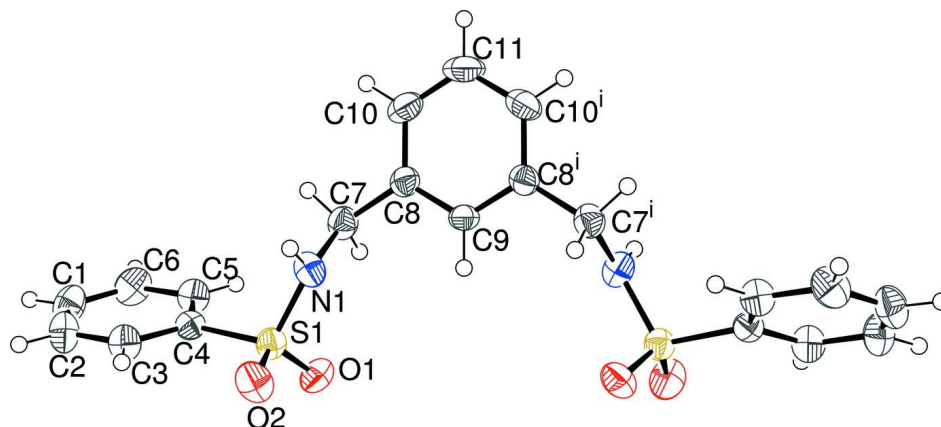
In the crystal, the molecules are linked by N—H···O hydrogen bonds (Table 1), to generate corrugated (010) sheets (Fig. 2). Weak aromatic π - π stacking [centroid-centroid separation = 3.8925 (12) and 3.9777 (12) Å] occurs between the layers and weak C—H···O interactions may help to consolidate the packing.

S2. Experimental

The *m*-xylylenediamine (0.132 ml, 1.0 mmol) was mixed with 20 ml distilled water in a 50 ml round bottom flask. Benzene sulfonyl chloride (0.255 ml, 2.0 mmol) was added while maintaining the pH of the reaction mixture at ca. 9.0 using sodium carbonate solution (3%) and the mixture was stirred for five hours. The white precipitated product was filtered, washed, dried and crystallized from methanol to generate colourless blocks of the title compound.

S3. Refinement

The N-bound H atom was located in a difference map and its position was freely refined with the constraint $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$. The C-bound hydrogen atoms were placed in calculated positions (C—H = 0.93–0.97 Å) and refined as riding atoms with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

**Figure 1**

The molecular structure of the title molecule, showing the numbering scheme and 50% displacement ellipsoids [Symmetry code: (i) $1-x, y, 1/2-z$].

N,N'-[1,3-Phenylenebis(methylene)]dibenzenesulfonamide

Crystal data

$C_{20}H_{20}N_2O_4S_2$

$M_r = 416.50$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 16.4319 (6) \text{ \AA}$

$b = 7.8024 (3) \text{ \AA}$

$c = 16.1217 (6) \text{ \AA}$

$\beta = 111.543 (2)^\circ$

$V = 1922.54 (12) \text{ \AA}^3$

$Z = 4$

$F(000) = 872$

$D_x = 1.439 \text{ Mg m}^{-3}$

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

Cell parameters from 2346 reflections

$\theta = 2.9\text{--}28.3^\circ$

$\mu = 0.31 \text{ mm}^{-1}$

$T = 296 \text{ K}$

Block, colourless

$0.15 \times 0.11 \times 0.08 \text{ mm}$

Data collection

Bruker APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

8653 measured reflections

2346 independent reflections

1841 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.024$

$\theta_{\text{max}} = 28.3^\circ$, $\theta_{\text{min}} = 2.9^\circ$

$h = -21 \rightarrow 21$

$k = -9 \rightarrow 10$

$l = -21 \rightarrow 21$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.103$

$S = 1.07$

2346 reflections

131 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

$w = 1/[\sigma^2(F_o^2) + (0.0466P)^2 + 1.1784P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 0.29 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.34 \text{ e \AA}^{-3}$

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.23478 (13)	0.4749 (3)	0.57485 (13)	0.0492 (5)
H1A	0.2133	0.4616	0.6204	0.059*
C2	0.18163 (12)	0.5397 (3)	0.49388 (14)	0.0493 (5)
H2	0.1239	0.5677	0.4846	0.059*
C3	0.21285 (11)	0.5637 (3)	0.42623 (12)	0.0405 (4)
H3	0.1766	0.6069	0.3713	0.049*
C4	0.29908 (10)	0.5225 (2)	0.44139 (10)	0.0313 (4)
C5	0.35276 (12)	0.4531 (2)	0.52240 (11)	0.0396 (4)
H5	0.4102	0.4230	0.5318	0.048*
C6	0.31953 (13)	0.4296 (3)	0.58870 (12)	0.0481 (5)
H6	0.3548	0.3828	0.6431	0.058*
C7	0.43547 (11)	0.2757 (2)	0.36899 (11)	0.0378 (4)
H7A	0.4162	0.1957	0.4041	0.045*
H7B	0.4827	0.3439	0.4096	0.045*
C8	0.46828 (10)	0.1780 (2)	0.30692 (11)	0.0332 (4)
C9	0.5000	0.2653 (3)	0.2500	0.0320 (5)
H9	0.5000	0.3845	0.2500	0.038*
C10	0.46821 (12)	0.0009 (3)	0.30605 (13)	0.0429 (4)
H10	0.4467	-0.0593	0.3434	0.052*
C11	0.5000	-0.0871 (4)	0.2500	0.0504 (7)
H11	0.5000	-0.2063	0.2500	0.061*
S1	0.34361 (3)	0.56500 (6)	0.35953 (3)	0.03403 (15)
N1	0.36255 (9)	0.3882 (2)	0.31843 (10)	0.0382 (4)
H1	0.3187 (13)	0.339 (3)	0.2865 (14)	0.046*
O1	0.42667 (8)	0.64544 (18)	0.40359 (9)	0.0459 (3)
O2	0.27803 (9)	0.65172 (19)	0.28749 (8)	0.0502 (4)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0510 (11)	0.0624 (14)	0.0390 (10)	-0.0120 (10)	0.0223 (9)	-0.0015 (9)
C2	0.0371 (10)	0.0647 (14)	0.0507 (11)	-0.0021 (9)	0.0213 (9)	-0.0029 (10)
C3	0.0355 (9)	0.0490 (12)	0.0342 (9)	0.0017 (8)	0.0097 (7)	0.0017 (8)
C4	0.0330 (8)	0.0318 (9)	0.0282 (7)	-0.0021 (6)	0.0101 (6)	-0.0021 (6)
C5	0.0362 (9)	0.0459 (11)	0.0343 (8)	0.0004 (7)	0.0101 (7)	0.0019 (7)
C6	0.0526 (11)	0.0575 (13)	0.0307 (9)	-0.0038 (9)	0.0110 (8)	0.0074 (8)

C7	0.0364 (9)	0.0416 (11)	0.0367 (9)	0.0013 (7)	0.0151 (7)	0.0044 (8)
C8	0.0264 (7)	0.0335 (10)	0.0379 (8)	-0.0002 (6)	0.0098 (7)	0.0026 (7)
C9	0.0316 (11)	0.0233 (12)	0.0409 (12)	0.000	0.0131 (10)	0.000
C10	0.0406 (9)	0.0337 (10)	0.0530 (11)	-0.0052 (8)	0.0155 (9)	0.0091 (9)
C11	0.0536 (16)	0.0234 (14)	0.0696 (19)	0.000	0.0171 (15)	0.000
S1	0.0348 (2)	0.0367 (3)	0.0307 (2)	0.00170 (17)	0.01223 (17)	0.00359 (17)
N1	0.0313 (7)	0.0452 (10)	0.0358 (8)	0.0011 (6)	0.0095 (6)	-0.0079 (7)
O1	0.0439 (7)	0.0438 (8)	0.0516 (7)	-0.0117 (6)	0.0195 (6)	0.0002 (6)
O2	0.0544 (8)	0.0605 (10)	0.0357 (7)	0.0195 (7)	0.0165 (6)	0.0149 (6)

Geometric parameters (Å, °)

C1—C2	1.373 (3)	C7—H7A	0.9700
C1—C6	1.373 (3)	C7—H7B	0.9700
C1—H1A	0.9300	C8—C10	1.382 (3)
C2—C3	1.378 (3)	C8—C9	1.389 (2)
C2—H2	0.9300	C9—C8 ⁱ	1.389 (2)
C3—C4	1.385 (2)	C9—H9	0.9300
C3—H3	0.9300	C10—C11	1.381 (2)
C4—C5	1.390 (2)	C10—H10	0.9300
C4—S1	1.7596 (17)	C11—C10 ⁱ	1.381 (2)
C5—C6	1.379 (3)	C11—H11	0.9300
C5—H5	0.9300	S1—O2	1.4314 (12)
C6—H6	0.9300	S1—O1	1.4315 (13)
C7—N1	1.467 (2)	S1—N1	1.6092 (16)
C7—C8	1.506 (2)	N1—H1	0.81 (2)
C2—C1—C6	120.16 (18)	H7A—C7—H7B	108.1
C2—C1—H1A	119.9	C10—C8—C9	118.86 (17)
C6—C1—H1A	119.9	C10—C8—C7	120.91 (16)
C1—C2—C3	120.73 (18)	C9—C8—C7	120.22 (17)
C1—C2—H2	119.6	C8 ⁱ —C9—C8	121.3 (2)
C3—C2—H2	119.6	C8 ⁱ —C9—H9	119.4
C2—C3—C4	118.88 (16)	C8—C9—H9	119.4
C2—C3—H3	120.6	C11—C10—C8	120.31 (19)
C4—C3—H3	120.6	C11—C10—H10	119.8
C3—C4—C5	120.78 (16)	C8—C10—H10	119.8
C3—C4—S1	120.29 (13)	C10—C11—C10 ⁱ	120.4 (3)
C5—C4—S1	118.89 (13)	C10—C11—H11	119.8
C6—C5—C4	119.00 (17)	C10 ⁱ —C11—H11	119.8
C6—C5—H5	120.5	O2—S1—O1	119.47 (9)
C4—C5—H5	120.5	O2—S1—N1	105.84 (8)
C1—C6—C5	120.41 (17)	O1—S1—N1	106.67 (8)
C1—C6—H6	119.8	O2—S1—C4	107.53 (8)
C5—C6—H6	119.8	O1—S1—C4	107.08 (8)
N1—C7—C8	110.62 (13)	N1—S1—C4	110.10 (8)
N1—C7—H7A	109.5	C7—N1—S1	121.73 (12)
C8—C7—H7A	109.5	C7—N1—H1	114.8 (15)

N1—C7—H7B	109.5	S1—N1—H1	114.1 (15)
C8—C7—H7B	109.5		
C6—C1—C2—C3	-1.4 (3)	C7—C8—C10—C11	-178.78 (13)
C1—C2—C3—C4	-0.4 (3)	C8—C10—C11—C10 ⁱ	-0.31 (11)
C2—C3—C4—C5	1.9 (3)	C3—C4—S1—O2	3.65 (17)
C2—C3—C4—S1	-175.61 (15)	C5—C4—S1—O2	-173.94 (14)
C3—C4—C5—C6	-1.6 (3)	C3—C4—S1—O1	133.19 (15)
S1—C4—C5—C6	175.99 (15)	C5—C4—S1—O1	-44.39 (17)
C2—C1—C6—C5	1.8 (3)	C3—C4—S1—N1	-111.21 (15)
C4—C5—C6—C1	-0.3 (3)	C5—C4—S1—N1	71.20 (16)
N1—C7—C8—C10	-121.05 (17)	C8—C7—N1—S1	-150.45 (13)
N1—C7—C8—C9	59.57 (18)	O2—S1—N1—C7	170.85 (14)
C10—C8—C9—C8 ⁱ	-0.30 (11)	O1—S1—N1—C7	42.63 (16)
C7—C8—C9—C8 ⁱ	179.09 (15)	C4—S1—N1—C7	-73.22 (15)
C9—C8—C10—C11	0.6 (2)		

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

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

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1 \cdots O2 ⁱⁱ	0.81 (2)	2.16 (2)	2.960 (2)	166 (2)
C5—H5 \cdots O1 ⁱⁱⁱ	0.93	2.55	3.461 (2)	166
C10—H10 \cdots O1 ^{iv}	0.93	2.57	3.379 (3)	146

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