N,N'-Dibenzenesulfonylputrescine

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N,*N*'-Dibenzenesulfonylputrescine

A. Linden and S. Bienz

Abstract

The title compound, *N*-{4-[(benzenesulfonyl)amino]butyl}benzenesulfonamide ($C_{16}H_{20}N_2O_4S_2$), is useful in the synthesis of natural or unnatural polyamine derivatives. The molecule is centrosymmetric with a straight-chain alkane core and N—H···O intermolecular hydrogen bonds link the molecules into an infinite two-dimensional network.

Comment

The title compound, (1), is of interest as a synthon for the synthesis of natural or unnatural polyamine derivatives. For example, it will be a useful reagent in our planned synthesis of polyamine spider toxins and analogues (Schäfer *et al.*, 1994).

The structure of (1) does not display any unusual geometrical features. The molecule is centrosymmetric and its alkane core has an essentially straight-chain conformation (Table 1). The N atoms are clearly pyramidal, as N1 deviates from the plane defined by S1, C2 and H1 by 0.31 (1) Å. This indicates that there is no delocalization of the lone pair of electrons on the N atom towards the sulfonyl group.

The N atom of each sulfonamide group of the molecule forms an intermolecular hydrogen bond with one of the sulfonyl O atoms of a neighbouring molecule (Table 2). When the donor and acceptor atoms immediately adjacent to each other are considered, the hydrogen bonds link adjacent molecules into infinite one-dimensional chains which run in the [010] direction and have a graph set motif of C(4) (Bernstein *et al.*, 1995). Bi-directional infinite one-dimensional chains are formed if one considers the pattern built by combining the donor at one end of the molecule with the acceptor at the opposite end of the molecule. These chains run parallel to the [101] direction and have a graph set motif of C(9). The complete pattern of interactions forms infinite two-dimensional networks which lie perpendicular to the [10$\bar{1}$] direction. This network also contains the ring sub-motif of $R^4_4(26)$ which involves four molecules.

Experimental

The title compound was synthesized according to the procedure of Wrede *et al.* (1927). Ccolourless plates were obtained by slowly cooling a warm solution of the compound in ethanol (m.p. 403.4–404.1 K).

Refinement

The positions of all H atoms were initially located in a difference electron density map. The position of the unique amide H atom was refined freely together with an isotropic displacement parameter. The positions of all other H atoms were geometrically idealized and refined with a riding model with U_{iso} constrained to be $1.2U_{eq}$ of the parent C atom.

CIF access

Computing details

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1991); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997b); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

N-{4-[(benzenesulfonyl)amino]butyl}benzenesulfonamide

Crystal data

$C_{16}H_{20}N_2O_4S_2$	V = 884.0 (3) Å^3
M_r = 368.46	Z = 2
Monoclinic, $P2_1/n$	Mo $K\alpha$
a = 10.649 (2) Å	μ = 0.32 mm^{-1}
b = 5.8777 (16) Å	T = 173 (2) K
c = 14.8257 (15) Å	0.46 × 0.35 × 0.12 mm
β = 107.705 (10)°	

Data collection

Rigaku AFC-5R diffractometer	1541 reflections with $I > 2\sigma(I)$
Absorption correction: ψ scan (North et al., 1968)	R_{int} = 0.024
T_{min} = 0.910, T_{max} = 1.000	3 standard reflections
2339 measured reflections	every 150 reflections
2025 independent reflections	intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036	113 parameters
$wR(F^2)$ = 0.105	H atoms treated by a mixture of independent and constrained refinement
S = 1.03	$\Delta\rho_{max}$ = 0.32 e Å^{-3}
2025 reflections	$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °)

S1—N1	1.6213 (16)	N1—C2	1.474 (2)
S1—C4	1.7654 (19)		
O1—S1—O2	119.27 (10)	C2—N1—S1	118.11 (12)
N1—S1—C4	107.01 (9)		
C4—S1—N1—C2	56.23 (16)	N1—C2—C3—C3^{i}	173.93 (19)

S1—N1—C2—C3	167.96 (13)	N1—S1—C4—C5	−107.12 (16)

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

Table 2
Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{ii}	0.87 (2)	2.07 (2)	2.917 (2)	164 (2)

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

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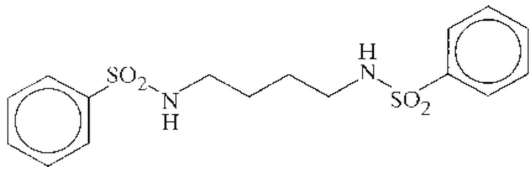
CIF access

Scheme 1



supplementary materials

N-{4-[(benzenesulfonyl)amino]butyl}benzenesulfonamide

Crystal data

$C_{16}H_{20}N_2O_4S_2$	$D_x = 1.384$ Mg m^{-3}
$M_r = 368.46$	Melting point: 403.4-404.1 K
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation $\lambda = 0.71069$ Å
$a = 10.649$ (2) Å	Cell parameters from 25 reflections
$b = 5.8777$ (16) Å	$\theta = 18.5$–20.0^o
$c = 14.8257$ (15) Å	$\mu = 0.32$ mm^{-1}
$\beta = 107.705$ (10)^o	$T = 173$ (2) K
$V = 884.0$ (3) Å^3	Plate, colourless
$Z = 2$	0.46 × 0.35 × 0.12 mm
$F_{000} = 388$	

Data collection

Rigaku AFC-5R diffractometer	$R_{int} = 0.024$
Radiation source: Rigaku rotating anode generator	$\theta_{max} = 27.5^o$
Monochromator: graphite	$\theta_{min} = 2.5^o$
$T = 173$(2) K	$h = 0 \rightarrow 13$
ω–2θ scans	$k = 0 \rightarrow 7$
Absorption correction: ψ scan (North et al., 1968)	$l = -19 \rightarrow 18$
$T_{min} = 0.910$, $T_{max} = 1.000$	3 standard reflections
2339 measured reflections	every 150 reflections
2025 independent reflections	intensity decay: none
1541 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Hydrogen site location: difference Fourier map
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.036$	$w = 1/[\sigma^2(F_o^2) + (0.0522P)^2 + 0.2504P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.105$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.03$	$\Delta\rho_{max} = 0.32$ e Å^{-3}
2025 reflections	$\Delta\rho_{min} = -0.27$ e Å^{-3}
113 parameters	Extinction correction: none
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.

Least-squares planes (*x*,*y*,*z* in crystal coordinates) and deviations from them (* indicates atom used to define plane)

5.8673 (0.1028) *x* + 4.0394 (0.0035) *y* + 4.2024 (0.2044) *z* = 5.3678 (0.0578)

* 0.0000 (0.0000) C2 * 0.0000 (0.0001) S1 * 0.0000 (0.0000) H1 − 0.3074 (0.0112) N1

Rms deviation of fitted atoms = 0.0000

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 $F^2 > 2\text{sigma}(F^2)$ 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}
S1	0.82476 (4)	−0.00661 (8)	0.13216 (3)	0.02752 (15)
O1	0.82203 (14)	−0.1007 (3)	0.04256 (10)	0.0399 (4)
O2	0.83649 (14)	−0.1564 (2)	0.21081 (10)	0.0401 (4)
N1	0.68807 (14)	0.1311 (3)	0.11752 (11)	0.0274 (3)
H1	0.677 (2)	0.168 (4)	0.1709 (16)	0.045 (7)*
C2	0.65789 (18)	0.3216 (3)	0.04969 (14)	0.0327 (4)
H21	0.6729	0.2741	−0.0103	0.039*
H22	0.7170	0.4514	0.0759	0.039*
C3	0.51554 (18)	0.3939 (3)	0.03070 (13)	0.0310 (4)
H31	0.4985	0.4243	0.0916	0.037*
H32	0.4567	0.2686	−0.0014	0.037*
C4	0.95358 (17)	0.1950 (3)	0.16515 (13)	0.0281 (4)
C5	1.01814 (19)	0.2573 (4)	0.10059 (14)	0.0359 (5)
H5	0.9969	0.1865	0.0402	0.043*
C6	1.1143 (2)	0.4248 (4)	0.12567 (16)	0.0463 (6)
H6	1.1590	0.4697	0.0820	0.056*
C7	1.1453 (2)	0.5263 (4)	0.21338 (18)	0.0476 (6)
H7	1.2114	0.6405	0.2300	0.057*
C8	1.0806 (2)	0.4629 (4)	0.27746 (16)	0.0436 (5)
H8	1.1027	0.5332	0.3380	0.052*
C9	0.9841 (2)	0.2978 (3)	0.25369 (14)	0.0356 (4)
H9	0.9390	0.2548	0.2973	0.043*

Atomic displacement parameters (Å^2)

U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}

S1	0.0315 (3)	0.0258 (2)	0.0290 (2)	0.00194 (18)	0.01481 (18)	0.00210 (18)
O1	0.0459 (8)	0.0398 (8)	0.0415 (8)	−0.0061 (7)	0.0243 (7)	−0.0120 (7)
O2	0.0428 (8)	0.0357 (8)	0.0471 (9)	0.0073 (6)	0.0214 (7)	0.0166 (7)
N1	0.0281 (8)	0.0306 (8)	0.0258 (8)	0.0029 (6)	0.0118 (6)	0.0035 (6)
C2	0.0307 (9)	0.0344 (10)	0.0321 (10)	−0.0001 (8)	0.0083 (8)	0.0091 (8)
C3	0.0278 (9)	0.0326 (10)	0.0293 (9)	−0.0021 (8)	0.0039 (7)	0.0041 (8)
C4	0.0264 (8)	0.0285 (9)	0.0299 (9)	0.0050 (7)	0.0090 (7)	0.0036 (8)
C5	0.0344 (10)	0.0427 (11)	0.0320 (10)	−0.0013 (9)	0.0121 (8)	0.0054 (9)
C6	0.0383 (12)	0.0518 (13)	0.0507 (13)	−0.0066 (10)	0.0164 (10)	0.0103 (11)
C7	0.0346 (11)	0.0358 (12)	0.0666 (16)	−0.0049 (9)	0.0067 (10)	0.0025 (11)
C8	0.0394 (11)	0.0405 (12)	0.0460 (12)	0.0048 (10)	0.0055 (10)	−0.0109 (10)
C9	0.0366 (10)	0.0373 (10)	0.0343 (10)	0.0070 (9)	0.0130 (8)	−0.0023 (9)

Geometric parameters (Å, °)

S1—O1	1.4310 (14)	C4—C5	1.387 (3)
S1—O2	1.4361 (14)	C4—C9	1.391 (3)
S1—N1	1.6213 (16)	C5—C6	1.387 (3)
S1—C4	1.7654 (19)	C5—H5	0.9500
N1—C2	1.474 (2)	C6—C7	1.376 (3)
N1—H1	0.87 (2)	C6—H6	0.9500
C2—C3	1.516 (3)	C7—C8	1.383 (3)
C2—H21	0.9900	C7—H7	0.9500
C2—H22	0.9900	C8—C9	1.379 (3)
C3—C3^{i}	1.520 (4)	C8—H8	0.9500
C3—H31	0.9900	C9—H9	0.9500
C3—H32	0.9900		
O1—S1—O2	119.27 (10)	H31—C3—H32	108.0
O1—S1—N1	107.74 (9)	C5—C4—C9	120.90 (19)
O2—S1—N1	105.52 (8)	C5—C4—S1	119.52 (15)
O1—S1—C4	108.14 (9)	C9—C4—S1	119.49 (14)
O2—S1—C4	108.55 (9)	C6—C5—C4	118.9 (2)
N1—S1—C4	107.01 (9)	C6—C5—H5	120.6
C2—N1—S1	118.11 (12)	C4—C5—H5	120.6
C2—N1—H1	111.7 (16)	C7—C6—C5	120.4 (2)
S1—N1—H1	112.0 (15)	C7—C6—H6	119.8
N1—C2—C3	109.76 (15)	C5—C6—H6	119.8
N1—C2—H21	109.7	C6—C7—C8	120.4 (2)
C3—C2—H21	109.7	C6—C7—H7	119.8
N1—C2—H22	109.7	C8—C7—H7	119.8
C3—C2—H22	109.7	C9—C8—C7	120.1 (2)
H21—C2—H22	108.2	C9—C8—H8	119.9
C2—C3—C3^{i}	111.4 (2)	C7—C8—H8	119.9
C2—C3—H31	109.3	C8—C9—C4	119.30 (19)
C3^{i}—C3—H31	109.3	C8—C9—H9	120.4
C2—C3—H32	109.3	C4—C9—H9	120.4
C3^{i}—C3—H32	109.3		
O1—S1—N1—C2	−59.85 (16)	N1—S1—C4—C9	69.48 (17)

O2—S1—N1—C2	171.72 (14)	C9—C4—C5—C6	0.0 (3)
C4—S1—N1—C2	56.23 (16)	S1—C4—C5—C6	176.56 (16)
S1—N1—C2—C3	167.96 (13)	C4—C5—C6—C7	0.3 (3)
N1—C2—C3—C3^{i}	173.93 (19)	C5—C6—C7—C8	−0.2 (3)
O1—S1—C4—C5	8.70 (18)	C6—C7—C8—C9	−0.2 (3)
O2—S1—C4—C5	139.44 (16)	C7—C8—C9—C4	0.5 (3)
N1—S1—C4—C5	−107.12 (16)	C5—C4—C9—C8	−0.4 (3)
O1—S1—C4—C9	−174.70 (15)	S1—C4—C9—C8	−176.98 (15)
O2—S1—C4—C9	−43.96 (18)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{ii}	0.87 (2)	2.07 (2)	2.917 (2)	164 (2)

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