Benzylsulfamide

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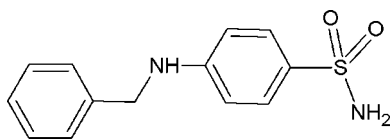Correspondence e-mail: thomas.gelbrich@uibk.ac.at

The crystal of the title compound [systematic name: 4-(benzylamino)benzenesulfonamide], $C_{13}H_{14}N_2O_2S$, displays a hydrogen-bonded framework structure. Molecules are doubly N—H···O hydrogen bonded to one another via their NH_2 groups and sulfonyl O atoms. These interactions generate a hydrogen-bonded ladder structure parallel to the a axis, which contains fused $R_2^2(8)$ rings. The NH group serves as the hydrogen-bond donor for a second set of intermolecular N—H···O=S interactions.

Related literature

For the pharmacology and synthesis of the title compound, see Goissedet et al. (1936); Goissedet & Despois (1938); Mellon et al. (1938); Long & Bliss (1939). For related structures, see: Hursthouse et al. (1998, 1999a,b); Gelbrich et al. (2008); Davis et al. (1996); Costanzo et al. (1999); Kubicki & Codding (2001); Yathirajan et al. (2005); Denehy et al. (2006); Toumieux et al. (2006). For graph-set analysis, see: Bernstein et al. (1995).

Experimental

Crystal data

$C_{13}H_{14}N_2O_2S$	$V = 1214.30$ (13) Å³
$M_r = 262.32$	$Z = 4$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 7.8426$ (1) Å	$\mu = 0.26$ mm^{-1}
$b = 10.5549$ (11) Å	$T = 120$ K
$c = 14.6694$ (3) Å	$0.20 \times 0.20 \times 0.15$ mm

Data collection

Bruker–Nonius Roper CCD camera on κ-goniostat diffractometer	11460 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2007)	2364 independent reflections
$T_{min} = 0.950$, $T_{max} = 0.962$	2312 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.023$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.061$	
$S = 1.06$	$\Delta\rho_{max} = 0.19$ e Å^{-3}
2364 reflections	$\Delta\rho_{min} = -0.30$ e Å^{-3}
176 parameters	Absolute structure: Flack (1983), 972 Friedel pairs
3 restraints	Flack parameter: -0.01 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H3N···O2^i	0.87 (2)	2.20 (2)	3.0264 (16)	160 (2)
N1—H2N···O1^{ii}	0.89 (2)	2.09 (2)	2.9613 (16)	168 (2)
N1—H1N···O2^{iii}	0.86 (1)	2.18 (1)	3.0281 (16)	172 (2)

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

Data collection: COLLECT (Hooft, 1998); cell refinement: DENZO (Otwinowski & Minor, 1997) and COLLECT; data reduction: DENZO and COLLECT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: XP in SHELXTL (Sheldrick, 2008) and Mercury (Bruno et al., 2002); software used to prepare material for publication: publCIF (Westrip, 2010).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2246).

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supplementary materials

Benzylsulfamide

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Comment

The title compound (synonyms: proseptazine, septazine, benzylsulfanilamide, chemodyn; CAS No. 104–22–3), first marketed in 1936, was one of the early antibacterial agents of the sulfonamide class (Goissedet *et al.*, 1936; Goissedet & Despois, 1938; Mellon *et al.*, 1938; Long & Bliss, 1939). The C–N–(C$_6$H$_4$)–S fragment of the molecule (see Fig. 1) is essentially planar, and the molecular geometry is characterized by the torsion angles N1–S1–C1–C2 and N2–C7–C8–C9 of 43.6 (1)° and -65.9 (2)°, respectively.

The crystal structure contains three independent intermolecular N—H···O=S bonds which lead to the formation of an H-bonded framework. Each molecule is doubly H-bonded, *via* its NH$_2$ and sulfonyl groups, to two neighbouring molecules. These interactions generate an N—H···O=S-bonded ladder structure parallel to [100], which consists of fused $R^2_2(8)$ rings (Bernstein *et al.*, 1995) and displays a 2$_1$ symmetry. This situation is illustrated in Fig. 2. The same one-dimensional structure has been found previously in only a few other compounds of the sulfonamide class, see Davis *et al.* (1996); Costanzo *et al.* (1999); Kubicki & Codding (2001);Yathirajan *et al.* (2005); Denehy *et al.* (2006); Toumieux *et al.* (2006).

The sulfonyl oxygen atom O2 accepts an additional H-bond from the NH group of a neighbouring molecule. This interaction links molecules which are related to one another by a 2$_1$ operation parallel to the *c*-axis (see Fig. 3).

Refinement

All H atoms were identified in a difference map. Secondary CH$_2$ (C—H = 0.99 Å) and aromatic carbon atoms (C—H = 0.95 Å) were positioned geometrically and refined with U_{iso} = 1.2 U_{eq}(C). Hydrogen atoms attached to N and O were refined with restrained distances [N—H = 0.88 (2) Å]; and their U_{iso} parameters were refined freely.
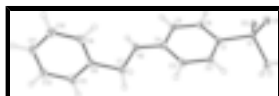
Figures

Fig. 1. The molecular structure with displacement ellipsoids drawn at the 50% probability level. Hydrogen atoms are shown as spheres of arbitrary size.
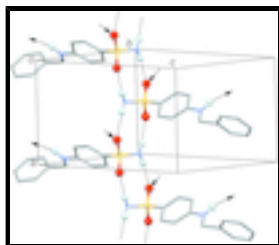

Fig. 2. Ladder structure parallel to [100] formed by H-bonds involving the NH$_2$ group. The interactions between the NH group and O2 are indicated by arrows. O and H atoms directly engaged in N–H···O bonds are drawn as balls.
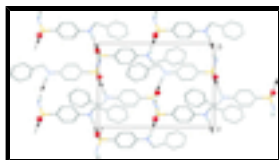
supplementary materials

Fig. 3. H-bonded framework structure viewed parallel to the *a*-axis, with H-bonds indicated by arrows.

4-(benzylamino)benzenesulfonamide

Crystal data

$C_{13}H_{14}N_2O_2S$	$F(000) = 552$
$M_r = 262.32$	$D_x = 1.435$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 6120 reflections
$a = 7.8426$ (1) Å	$\theta = 2.9–27.5°$
$b = 10.5549$ (11) Å	$\mu = 0.26$ mm^{-1}
$c = 14.6694$ (3) Å	$T = 120$ K
$V = 1214.30$ (13) Å^3	Block, colourless
$Z = 4$	$0.20 \times 0.20 \times 0.15$ mm

Data collection

Bruker–Nonius Roper CCD camera on κ-goniostat diffractometer	2364 independent reflections
Radiation source: Bruker-Nonius FR591 rotating anode	2312 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.027$
Detector resolution: 9.091 pixels mm^{-1}	$\theta_{max} = 26.0°$, $\theta_{min} = 3.2°$
φ and ω scans	$h = -9 \rightarrow 9$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2007)	$k = -12 \rightarrow 13$
$T_{min} = 0.950$, $T_{max} = 0.962$	$l = -18 \rightarrow 18$
11460 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.023$	$w = 1/[\sigma^2(F_o^2) + (0.032P)^2 + 0.3394P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.061$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.06$	$\Delta\rho_{max} = 0.19$ e Å^{-3}
2364 reflections	$\Delta\rho_{min} = -0.30$ e Å^{-3}
176 parameters	Extinction correction: *SHELXS97* (Sheldrick, 2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
3 restraints	Extinction coefficient: 0.021 (4)

Primary atom site location: structure-invariant direct methods

Absolute structure: Flack (1983), **972 Friedel pairs**

Secondary atom site location: difference Fourier map

Flack parameter: −0.01 (5)

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 ($\mathring{A}^2$)

	x	y	z	$U_{iso}*/U_{eq}$
S1	0.88925 (4)	0.14891 (3)	0.47921 (2)	0.00933 (11)
O1	1.04460 (12)	0.09326 (9)	0.51381 (7)	0.0132 (2)
O2	0.73029 (12)	0.08853 (9)	0.50432 (7)	0.0127 (2)
N1	0.88280 (16)	0.29082 (11)	0.51938 (8)	0.0124 (3)
H2N	0.789 (2)	0.3327 (18)	0.5037 (13)	0.027 (5)*
H1N	0.9760 (19)	0.3323 (16)	0.5125 (12)	0.016 (4)*
N2	0.94618 (16)	0.14533 (12)	0.07743 (8)	0.0141 (3)
H3N	0.879 (2)	0.0925 (18)	0.0498 (13)	0.032 (5)*
C1	0.89793 (18)	0.15385 (13)	0.36038 (9)	0.0103 (3)
C2	1.00520 (19)	0.23999 (13)	0.31668 (10)	0.0131 (3)
H2	1.0688	0.2990	0.3518	0.016*
C3	1.01965 (19)	0.24017 (13)	0.22311 (10)	0.0135 (3)
H3	1.0908	0.3008	0.1940	0.016*
C4	0.92945 (17)	0.15099 (14)	0.17015 (9)	0.0110 (3)
C5	0.82057 (18)	0.06548 (14)	0.21519 (10)	0.0115 (3)
H5	0.7574	0.0057	0.1805	0.014*
C6	0.80389 (18)	0.06679 (13)	0.30896 (9)	0.0107 (3)
H6	0.7291	0.0090	0.3383	0.013*
C9	1.15434 (18)	0.09176 (14)	−0.09378 (10)	0.0145 (3)
H9	1.2109	0.0486	−0.0456	0.017*
C7	1.03437 (19)	0.24066 (13)	0.02344 (10)	0.0138 (3)
H7A	1.1487	0.2564	0.0497	0.017*
H7B	0.9696	0.3211	0.0251	0.017*
C8	1.05223 (18)	0.19639 (14)	−0.07416 (10)	0.0119 (3)
C10	1.1744 (2)	0.04982 (15)	−0.18294 (11)	0.0182 (3)
H10	1.2445	−0.0214	−0.1957	0.022*
C11	1.0911 (2)	0.11273 (15)	−0.25348 (10)	0.0187 (3)
H11	1.1038	0.0840	−0.3145	0.022*
C12	0.9903 (2)	0.21671 (15)	−0.23489 (11)	0.0184 (3)
H12	0.9340	0.2596	−0.2832	0.022*

| C13 | 0.97056 (19) | 0.25909 (14) | −0.14530 (10) | 0.0150 (3) |
| H13 | 0.9013 | 0.3309 | −0.1329 | 0.018* |

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.00952 (18)	0.01096 (17)	0.00751 (17)	0.00004 (13)	0.00065 (13)	0.00008 (13)
O1	0.0124 (5)	0.0159 (5)	0.0115 (5)	0.0029 (4)	−0.0014 (4)	0.0013 (4)
O2	0.0118 (5)	0.0143 (5)	0.0120 (5)	−0.0022 (4)	0.0026 (4)	0.0015 (4)
N1	0.0110 (6)	0.0127 (6)	0.0136 (6)	−0.0003 (5)	0.0009 (6)	−0.0031 (5)
N2	0.0181 (6)	0.0152 (6)	0.0090 (6)	−0.0072 (5)	0.0009 (5)	0.0005 (5)
C1	0.0105 (6)	0.0132 (7)	0.0073 (6)	0.0018 (6)	0.0007 (5)	0.0008 (5)
C2	0.0137 (7)	0.0134 (7)	0.0122 (7)	−0.0033 (6)	−0.0011 (6)	−0.0012 (6)
C3	0.0145 (7)	0.0136 (7)	0.0125 (7)	−0.0049 (6)	0.0011 (6)	0.0017 (6)
C4	0.0117 (6)	0.0116 (6)	0.0099 (6)	0.0011 (6)	0.0000 (5)	0.0004 (6)
C5	0.0109 (7)	0.0114 (6)	0.0123 (6)	−0.0016 (5)	−0.0006 (5)	−0.0019 (6)
C6	0.0106 (7)	0.0099 (6)	0.0117 (6)	−0.0007 (5)	0.0008 (5)	0.0006 (6)
C9	0.0132 (7)	0.0150 (7)	0.0154 (7)	−0.0002 (6)	−0.0015 (5)	0.0027 (6)
C7	0.0167 (7)	0.0139 (7)	0.0109 (7)	−0.0039 (6)	0.0016 (6)	0.0016 (6)
C8	0.0117 (7)	0.0128 (6)	0.0112 (7)	−0.0047 (5)	0.0002 (5)	0.0006 (6)
C10	0.0176 (8)	0.0158 (7)	0.0212 (8)	−0.0008 (6)	0.0041 (6)	−0.0029 (6)
C11	0.0220 (8)	0.0236 (8)	0.0105 (7)	−0.0104 (6)	0.0039 (6)	−0.0030 (6)
C12	0.0187 (8)	0.0236 (8)	0.0129 (8)	−0.0059 (6)	−0.0035 (6)	0.0058 (6)
C13	0.0128 (7)	0.0175 (8)	0.0146 (7)	0.0006 (6)	0.0002 (6)	0.0029 (6)

Geometric parameters (Å, °)

S1—O1	1.4447 (10)	C5—H5	0.9500
S1—O2	1.4477 (10)	C6—H6	0.9500
S1—N1	1.6104 (12)	C9—C10	1.390 (2)
S1—C1	1.7452 (13)	C9—C8	1.394 (2)
N1—H2N	0.889 (15)	C9—H9	0.9500
N1—H1N	0.858 (14)	C7—C8	1.5126 (19)
N2—C4	1.3677 (17)	C7—H7A	0.9900
N2—C7	1.4553 (17)	C7—H7B	0.9900
N2—H3N	0.867 (15)	C8—C13	1.392 (2)
C1—C2	1.3948 (19)	C10—C11	1.392 (2)
C1—C6	1.399 (2)	C10—H10	0.9500
C2—C3	1.377 (2)	C11—C12	1.380 (2)
C2—H2	0.9500	C11—H11	0.9500
C3—C4	1.411 (2)	C12—C13	1.397 (2)
C3—H3	0.9500	C12—H12	0.9500
C4—C5	1.407 (2)	C13—H13	0.9500
C5—C6	1.3817 (19)		
O1—S1—O2	117.25 (6)	C5—C6—C1	119.57 (13)
O1—S1—N1	106.02 (6)	C5—C6—H6	120.2
O2—S1—N1	106.81 (6)	C1—C6—H6	120.2
O1—S1—C1	109.28 (6)	C10—C9—C8	120.77 (14)

O2—S1—C1	107.51 (6)	C10—C9—H9	119.6
N1—S1—C1	109.81 (6)	C8—C9—H9	119.6
S1—N1—H2N	113.2 (13)	N2—C7—C8	110.22 (11)
S1—N1—H1N	113.9 (12)	N2—C7—H7A	109.6
H2N—N1—H1N	114.9 (17)	C8—C7—H7A	109.6
C4—N2—C7	123.82 (12)	N2—C7—H7B	109.6
C4—N2—H3N	115.7 (14)	C8—C7—H7B	109.6
C7—N2—H3N	118.6 (14)	H7A—C7—H7B	108.1
C2—C1—C6	119.88 (13)	C13—C8—C9	119.09 (14)
C2—C1—S1	120.14 (11)	C13—C8—C7	121.35 (13)
C6—C1—S1	119.89 (10)	C9—C8—C7	119.56 (13)
C3—C2—C1	120.57 (13)	C9—C10—C11	119.64 (14)
C3—C2—H2	119.7	C9—C10—H10	120.2
C1—C2—H2	119.7	C11—C10—H10	120.2
C2—C3—C4	120.44 (13)	C12—C11—C10	120.08 (14)
C2—C3—H3	119.8	C12—C11—H11	120.0
C4—C3—H3	119.8	C10—C11—H11	120.0
N2—C4—C5	119.81 (13)	C11—C12—C13	120.27 (15)
N2—C4—C3	121.92 (13)	C11—C12—H12	119.9
C5—C4—C3	118.27 (13)	C13—C12—H12	119.9
C6—C5—C4	121.22 (13)	C8—C13—C12	120.15 (14)
C6—C5—H5	119.4	C8—C13—H13	119.9
C4—C5—H5	119.4	C12—C13—H13	119.9
N1—S1—C1—C2	43.59 (14)	N2—C7—C8—C9	−65.94 (17)

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
N2—H3N···O2^i	0.87 (2)	2.20 (2)	3.0264 (16)	160.(2)
N1—H2N···O1^{ii}	0.89 (2)	2.09 (2)	2.9613 (16)	168.(2)
N1—H1N···O2^{iii}	0.86 (1)	2.18 (1)	3.0281 (16)	172.(2)

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

Fig. 1

Fig. 2

Fig. 3