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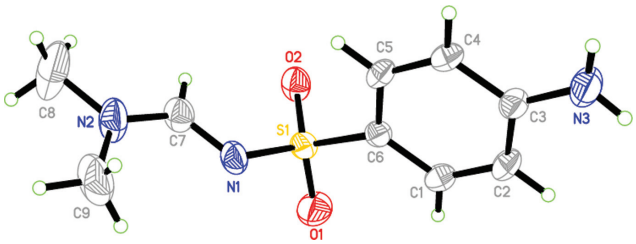
Crystal structure of (*E*)-N′-((4-aminophenyl) sulfonyl)-*N*,*N*-dimethylformimidamide, $C_9H_{13}N_3O_2S$



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Abstract

$C_9H_{13}N_3O_2S$, monoclinic, $P2_1/c$ (no. 14), $a = 8.0984(4)$ Å, $b = 17.3203(10)$ Å, $c = 9.6802(4)$ Å, $\beta = 124.031(3)°$, $V = 1125.26(10)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0504$, $wR_{ref}(F^2) = 0.1275$, $T = 293(2)$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

A mixture of benzenesulfonamide (344 mg, 2 mmol) and *N*,*N*-dimethylformamide dimethyl acetal (476 mg, 4 mmol) in ethanol (10 mL) was heated under reflux for 10 h. The reaction mixture was cooled and the separated solid was filtered, dried and recrystallized from ethanol. Yield 96%; **^{1}H NMR** (DMSO-d$_6$; 500 MHz): δ 8.10 (s, 1H, N—C(H)=N), 7.39–7.37

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Table 1: Data collection and handling.

Crystal:	Colourless needle
Size:	0.59 × 0.13 × 0.09 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.27 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	27.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	16215, 2581, 0.066
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1884
$N(param)_{refined}$:	138
Programs:	SHELX [1], Bruker [2]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
S1	0.31663(8)	−0.59517(3)	−0.17899(7)	0.03732(19)
O1	0.4058(3)	−0.64624(11)	−0.0384(2)	0.0635(5)
O2	0.2455(2)	−0.52250(9)	−0.1591(2)	0.0490(5)
N1	0.4747(3)	−0.58306(11)	−0.2299(3)	0.0449(5)
N2	0.6689(4)	−0.49539(14)	−0.2558(3)	0.0610(6)
N3	−0.3860(3)	−0.74972(13)	−0.7473(3)	0.0613(7)
H2	−0.4202	−0.7928	−0.7311	0.074*
H3	−0.4502	−0.7290	−0.8401	0.074*
C1	0.0513(4)	−0.71410(13)	−0.3266(3)	0.0438(6)
H1A	0.1191	−0.7371	−0.2219	0.053*
C2	−0.1152(4)	−0.74905(13)	−0.4584(3)	0.0472(6)
H2A	−0.1592	−0.7955	−0.4417	0.057*
C3	−0.2195(3)	−0.71587(12)	−0.6171(3)	0.0388(5)
C4	−0.1487(3)	−0.64599(12)	−0.6387(3)	0.0363(5)
H4A	−0.2143	−0.6232	−0.7436	0.044*
C5	0.0161(3)	−0.61130(12)	−0.5066(3)	0.0344(5)
H5A	0.0602	−0.5646	−0.5224	0.041*
C6	0.1189(3)	−0.64450(12)	−0.3492(3)	0.0329(5)
C7	0.5336(4)	−0.51297(15)	−0.2254(3)	0.0471(6)
H1	0.4779	−0.4732	−0.1997	0.057*
C8	0.7321(7)	−0.4158(2)	−0.2474(7)	0.1030(14)
H8A	0.6694	−0.3832	−0.2094	0.154*
H8B	0.6943	−0.3993	−0.3561	0.154*
H8C	0.8742	−0.4124	−0.1713	0.154*
C9	0.7652(5)	−0.5543(2)	−0.2925(5)	0.0894(12)
H9A	0.7915	−0.5985	−0.2234	0.134*
H9B	0.8883	−0.5345	−0.2708	0.134*
H9C	0.6797	−0.5688	−0.4076	0.134*

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(d, 2H, $J = 8.5$ Hz, Ar—H), 6.58–6.56 (d, 2H, $J = 9.0$ Hz, Ar—H), 5.82 (s, 2H, NH_2), 3.10 (s, 3H, CH_3), 2.87 (s, 3H, CH_3) ppm; **^{13}C NMR** (DMSO-d_6; 125 MHz): δ 35.30 (CH_3), 41.14 (CH_3), 113.01, 128.20, 129.03, 152.41, 159.43 (N—CH=N) ppm; **MS**: m/z = 227.1.

Experimental details

Cell refinement and data reduction were carried out by Bruker SAINT, SHELXT [1, 2].

Comment

Benzenesulfonamides ($ArSO_2NH_2$) incorporating aromatic and heterocyclic ring systems are an important group of compounds, which have a wide range of biological activity such as anti-inflammatory, COX-1/2 inhibiting activity, inhibitors of the metalloenzyme carbonic anhydrase, and antitumor activities [3–8]. Recently, the synthesis of *N*-sulfonylformamidines has been reported through condensation of formamides and versatile sulfonamides using thionylchloride in the presence of chloroform as solvent [9]. The title compound was synthesized and reported according to method described by Silva, et al. [10]. Detailed synthetic studies indicate that this procedure gives the title compound.

The asymmetric unit of the title compound contains one independent molecule. The bond length between N1—C7 is 1.296(3) Å which indicates that it is a double bond, compared to 1.320(3) Å between N2—C7. Bond lengths and angles of the title molecule are in the expected ranges [11]. The molecules pack in the crystal structure *via* two strong classical intermolecular hydrogen bonds, N3—H2···N1^{i} and N3—H3···O1^{ii} in addition to one non-classical hydrogen bond C9—H9B···O2^{iii}. The H···A distances are 2.31, 2.14 and 2.47 Å, respectively and the angles are 170, 164 and 161°, respectively. Symmetry codes: (i) $x - 1, -y - 3/2, z - 1/2$; (ii) $x - 1, y, z - 1$; (iii) $x + 1, y, z$.

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