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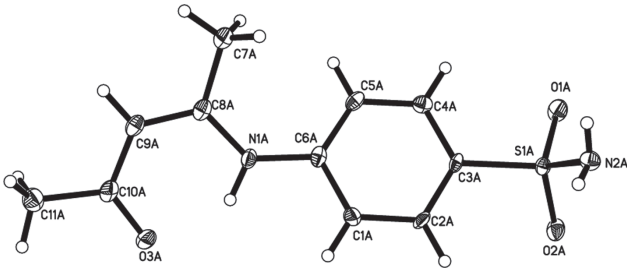
Crystal structure of (*Z*)-4-(4-oxopent-2-en-2-ylamino)benzenesulfonamide, $C_{11}H_{14}N_2O_3S$



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Abstract

$C_{11}H_{14}N_2O_3S$, orthorhombic, *Pbca* (no. 61), $a = 10.1973(3)$ Å, $b = 9.9487(4)$ Å, $c = 23.1332(8)$ Å, $V = 2346.86(14)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0527$, $wR_{ref}(F^2) = 0.1407$, $T = 100$ K.

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Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

A solution of sulfanilamide (1.72 g, 0.01 mol) and acetylacetone (1.00 g, 0.01 mol) in methanol (10 mL) containing glacial acetic acid (1 mL) was refluxed for 8 h. The reaction mixture was filtered then, the filtered solid was crystallized from ethanol to give the title compound [13]. Yield: 91%; m.p. 206.9°C, IR, cm^{-1}: 3361, 3283, 3193 (NH, NH_2), 3091 (CH arom.); 2986, 2831 (CH aliph.), 1696 (C = O), 1376, 1165 (SO2). ^{1}H-NMR (DMSO-d_6, ppm): 2.0 [s, 3H, $COCH_3$], 2.3 [s, 3H, CH_3], 5.6 [s, 1H, CH], 6.5, 8.3 [2d, 4H, Ar—H, AB system, $J = 7.3$ Hz], 8.8 [s, 2H, NH, SO_2NH_2, D_2O-exchangeable], 10.8 [s, 2H, NH, D_2O-exchangeable]. ^{13}C-NMR (DMSO-d_6, ppm): 18.6 (CH_3), 26.3 ($CH_3C = O$), 96.8 (CH), 113.7 (2), 129.8 (2), 131.6, 142.4, 160.7, 197.1 (C = O). Anal. calcd. for $C_{11}H_{14}N_2O_3S$ (254.31): C, 51.95; H, 5.55; N, 11.02%; found: C, 51.72; H, 5.87; N, 10.77%.

Table 1: Data collection and handling.

Crystal:	Colourless, plate, size 0.089×0.368×0.478 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.74 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω scans
$2\theta_{max}$:	49.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	21970, 2059
$N(param)_{refined}$:	156
Programs:	SHELX [14]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(17)	8*c*	−0.2775	0.4284	−0.0090	0.017
H(18)	8*c*	−0.6073	0.1597	−0.2580	0.017
H(19)	8*c*	−0.5368	0.2472	−0.2726	0.017
H(1)	8*c*	−0.2720	0.4430	−0.1086	0.017
H(2)	8*c*	−0.2971	0.3423	−0.1981	0.016
H(3)	8*c*	−0.5016	0.0430	−0.1234	0.017
H(4)	8*c*	−0.4749	0.1431	−0.0342	0.018
H(5)	8*c*	−0.5989	0.3129	0.0114	0.028
H(6)	8*c*	−0.5944	0.3312	0.0786	0.028
H(7)	8*c*	−0.5268	0.2065	0.0499	0.028
H(8)	8*c*	−0.4344	0.4615	0.1173	0.018
H(11A)	8*c*	−0.1736	0.6917	0.1274	0.032
H(11B)	8*c*	−0.2184	0.5746	0.1682	0.032
H(11C)	8*c*	−0.3216	0.6780	0.1448	0.032

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Table 3: Fractional coordinates and atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1A)	8*c*	−0.42093(6)	0.10338(6)	−0.23726(3)	0.0131(4)	0.0107(4)	0.0102(4)	0.0015(2)	0.0003(2)	−0.0015(2)
O(1A)	8*c*	−0.4601(2)	−0.0326(2)	−0.22662(8)	0.030(1)	0.012(1)	0.017(1)	0.0000(8)	0.0011(8)	−0.0011(8)
O(2A)	8*c*	−0.3032(2)	0.1289(2)	−0.26973(8)	0.014(1)	0.027(1)	0.015(1)	0.0015(9)	0.0023(7)	−0.0057(8)
O(3A)	8*c*	−0.1865(2)	0.5425(2)	0.04269(8)	0.022(1)	0.024(1)	0.012(1)	−0.0040(8)	0.0004(8)	0.0023(8)
N(1A)	8*c*	−0.3493(2)	0.3724(2)	−0.00988(9)	0.016(1)	0.018(1)	0.009(1)	0.0001(9)	−0.0006(9)	−0.0014(9)
N(2A)	8*c*	−0.5388(2)	0.1725(2)	−0.27270(9)	0.015(1)	0.012(1)	0.016(1)	0.0002(9)	0.0002(9)	−0.0001(9)
C(1A)	8*c*	−0.3184(3)	0.3628(3)	−0.1116(1)	0.015(1)	0.013(1)	0.015(1)	−0.002(1)	0.001(1)	−0.003(1)
C(2A)	8*c*	−0.3329(2)	0.3026(3)	−0.1653(1)	0.016(1)	0.017(1)	0.008(1)	−0.000(1)	0.005(1)	0.000(1)
C(3A)	8*c*	−0.4017(2)	0.1820(3)	−0.1695(1)	0.011(1)	0.015(1)	0.006(1)	0.004(1)	−0.0013(9)	0.001(1)
C(4A)	8*c*	−0.4553(3)	0.1232(3)	−0.1204(1)	0.015(1)	0.013(1)	0.016(1)	0.000(1)	−0.001(1)	0.003(1)
C(5A)	8*c*	−0.4399(3)	0.1836(3)	−0.0670(1)	0.017(1)	0.017(1)	0.010(1)	0.003(1)	0.001(1)	0.005(1)
C(6A)	8*c*	−0.3722(2)	0.3052(3)	−0.0621(1)	0.011(1)	0.017(1)	0.011(1)	0.005(1)	−0.002(1)	−0.001(1)
C(7A)	8*c*	−0.5467(3)	0.3002(3)	0.0455(1)	0.019(1)	0.022(2)	0.015(2)	−0.000(1)	0.003(1)	−0.003(1)
C(8A)	8*c*	−0.4219(3)	0.3783(3)	0.0399(1)	0.019(1)	0.013(1)	0.011(1)	0.005(1)	−0.000(1)	0.002(1)
C(9A)	8*c*	−0.3822(3)	0.4593(3)	0.0843(1)	0.018(1)	0.017(1)	0.010(1)	0.004(1)	0.000(1)	0.002(1)
C(10A)	8*c*	−0.2664(3)	0.5412(3)	0.0841(1)	0.023(1)	0.015(1)	0.009(1)	0.003(1)	−0.002(1)	0.004(1)
C(11A)	8*c*	−0.2429(3)	0.6293(3)	0.1358(1)	0.027(2)	0.019(2)	0.018(2)	−0.001(1)	−0.003(1)	0.001(1)

Experimental details

Cell refinement and data reduction were carried out by Bruker SAINT [15]. The hydrogen atoms were placed on calculated positions with the help of the SHELX program (AFIX 3, 43 or 137 option) [14].

Discussion

Heterocyclic sulfonamide derivatives have shown good anticancer activity with variety of mechanisms including cell cycle perturbation at G1 phase, disruption of microtubules assembly and the well-known carbonic anhydrase inhibition activity with selectivity to the tumor associated isoforms hCA IX and hCA XII [1–5]. Combination of a 4-(4-oxopent-2-en-2-ylamino) scaffold with the biologically active sulfonamide moiety has received great attention as PI3 K inhibitor, which is an important enzyme controlling signal transduction [4, 6–8]. Recently, diaryl sulfone derivatives, that were synthesized from dapson have shown good cytotoxic activity on breast cancer cell line (MCF-7) [9]. As a continuation of our work to design and synthesize novel anticancer agents [6–12], we have designed and synthesized the title compound.

The aim of this work was to design and synthesize of (*Z*)-4-(4-oxopent-2-en-2-ylamino)benzenesulfonamide having a biologically active sulfonamide moiety. Thus, interaction of sulfanilamide with acetylacetone in absolute methanol and glacial acetic acid furnished the title compound. The structure of this compound was established on the basis of elemental analysis, IR, ^{1}H-NMR, ^{13}C-NMR data and X-ray analysis.

The crystal structure of the title compound contains one molecule in the asymmetric unit (figure). In the title molecule C8A-C9A bond length is 1.367(4) Å, which is a typical C = C double bond. All geometric parameters are in the expected ranges. In the crystal structure the molecules are stabilized by intermolecular hydrogen bonds, of which O2A, O1A and N2A work as hydrogen bond acceptors and N2A and O1A work as hydrogen bond donors. These hydrogen bonding interactions lead to a layered structure parallel to the *ab* plane.

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