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Crystal structure of 2-(4-fluorophenyl)-*N*-phenyl-2-(phenylamino)ethanesulfonamide – toluene (1/0.5), $C_{23.5}H_{23}FN_2O_2S$

Table 1: Data collection and handling.

Crystal:	Needle, colorless
Size:	0.31 × 0.24 × 0.13 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.20 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	28.4°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	24448, 5003, 0.024
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4259
$N(\text{param})_{\text{refined}}$:	303
Programs:	Bruker programs [1], SHELX [2, 3], OLEX2 [4]

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Abstract

$C_{23.5}H_{23}FN_2O_2S$, Mr = 416.50, monoclinic, $C2/c$ (no. 15), $a = 27.2594(19)$ Å, $b = 5.7351(4)$ Å, $c = 26.1139(18)$ Å, $\beta = 102.009(2)^\circ$, $V = 3993.2(5)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0358$, $wR_{\text{ref}}(F^2) = 0.0958$ $T = 100(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The β -amino sulfonamide title compound was obtained through the following synthetic procedure: A 10 mL single-neck round-bottom flask was charged with (*E*)-2-(4-fluorophenyl)ethanesulfonyl fluoride (prepared according to [11]) (0.245 mmol, 0.05 g, 1 equiv), aniline (24.5 mmol,

223 μ L, 10 equiv) and DBU (50 mol%). The reaction mixture was stirred at room temperature until completion as judged by TLC analysis. The crude mixture was washed with water and extracted with Et₂O (3x). The combined organic layers were washed with brine and dried over MgSO₄ anhydrous. The solvent was evaporated under vacuum and the crude product was purified by column chromatography (SiO₂, EtOAc/Hexane, 3:7). ¹H-NMR (CDCl₃, 400 MHz): δ 7.34 (2H, t, $J = 7.83$ Hz), 7.27 (3H, m), 7.19 (1H, t, $J = 7.45$ Hz), 7.12 (3H, m), 7.00 (2H, t, $J = 8.56$ Hz), 6.75 (1H, t, $J = 7.40$ Hz), 6.54 (2H, d, $J = 7.84$ Hz), 6.37 (1H, s), 4.94 (1H, dd, $J = 3.55$), 4.65 (1H, b), 3.47 (2H, m). p.p.m.

Fifteen milligram of the title compound was dissolved in 1 mL of ethanol and kept in a 5 mL vial covered with aluminium foil for the slow evaporation of the solvent at ambient condition. Single crystals were obtained after three days upon complete evaporation of the solvent which was used for the data collection at single-crystal X-ray diffractometer. The source of the residual toluene contaminant was confirmed to have come from the aniline that was used in the reaction.

Experimental details

Single colourless needle-shaped crystals of were recrystallised from ethanol by slow evaporation. A crystal was selected and mounted on a MITIGEN holder in paratone oil on a Bruker APEX-II diffractometer. Using OLEX2 the structure was solved with the SHELXS-2013 program, using the

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
S1	0.24347(2)	0.27161(5)	0.31603(2)	0.01345(9)
F1	0.48592(3)	0.36466(17)	0.34266(4)	0.0289(2)
O1	0.23132(4)	0.11047(17)	0.35354(4)	0.0192(2)
O2	0.28256(4)	0.21137(19)	0.28887(4)	0.0221(2)
N1	0.19352(4)	0.3142(2)	0.27017(4)	0.0141(2)
H1	0.199724	0.418803	0.247685	0.017*
N2	0.29754(4)	0.7604(2)	0.42291(4)	0.0161(2)
C4	0.05088(5)	0.4392(3)	0.30282(6)	0.0220(3)
H4	0.018825	0.468200	0.310403	0.026*
C5	0.07275(5)	0.6032(3)	0.27569(5)	0.0206(3)
H5	0.055185	0.742515	0.263755	0.025*
C6	0.12041(5)	0.5649(2)	0.26581(5)	0.0164(3)
H6	0.135716	0.678716	0.247823	0.020*
C1	0.14539(5)	0.3577(2)	0.28262(5)	0.0135(2)
C7	0.25712(5)	0.5463(2)	0.34694(5)	0.0136(2)
H7A	0.266781	0.656436	0.321563	0.016*
H7B	0.226335	0.607348	0.356656	0.016*
C8	0.29934(5)	0.5385(2)	0.39625(5)	0.0139(2)
H8	0.291900	0.410566	0.419535	0.017*
C9	0.35038(5)	0.4921(2)	0.38311(5)	0.0142(2)
C10	0.37608(5)	0.2852(2)	0.39816(5)	0.0175(3)
H10	0.361956	0.172699	0.417571	0.021*
C11	0.42224(5)	0.2408(2)	0.38511(6)	0.0203(3)
H11	0.440171	0.101121	0.395857	0.024*
C12	0.44100(5)	0.4064(3)	0.35611(5)	0.0201(3)
C3	0.07580(5)	0.2324(3)	0.31893(5)	0.0210(3)
H3	0.060517	0.119511	0.337179	0.025*
C2	0.12293(5)	0.1892(2)	0.30855(5)	0.0172(3)
H2A	0.139665	0.046364	0.319036	0.021*
C13	0.41688(5)	0.6143(3)	0.34047(5)	0.0201(3)
H13	0.431007	0.724968	0.320632	0.024*
C14	0.37137(5)	0.6565(2)	0.35466(5)	0.0171(3)
H14	0.354294	0.799170	0.344864	0.021*
C15	0.31376(5)	0.7815(2)	0.47681(5)	0.0147(2)
C16	0.34210(5)	0.6093(2)	0.50747(5)	0.0185(3)
H16	0.350417	0.469452	0.491720	0.022*
C17	0.35812(5)	0.6434(3)	0.56124(5)	0.0216(3)
H17	0.376779	0.524366	0.581950	0.026*
C18	0.34734(5)	0.8471(3)	0.58494(5)	0.0221(3)
H18	0.359150	0.869953	0.621462	0.027*
C19	0.31900(5)	1.0182(3)	0.55462(5)	0.0210(3)
H19	0.311241	1.158409	0.570602	0.025*
C20	0.30196(5)	0.9860(2)	0.50136(5)	0.0175(3)
H20	0.282116	1.103120	0.481212	0.021*
C21 ^a	0.47069(16)	0.2774(7)	0.56027(14)	0.0358(9)
H21A ^a	0.434217	0.269302	0.556603	0.054*
H21B ^a	0.480278	0.435673	0.552156	0.054*
H21C ^a	0.487079	0.237755	0.596271	0.054*
C22 ^a	0.48572(13)	0.1192(7)	0.52562(14)	0.0260(7)
C23 ^a	0.45782(15)	−0.0810(8)	0.50905(16)	0.0254(8)
H23 ^a	0.426993	−0.105386	0.519796	0.031*
C24 ^a	0.47410(16)	−0.2460(8)	0.47710(17)	0.0303(8)
H24 ^a	0.454389	−0.381622	0.467226	0.036*
C25 ^a	0.51911(15)	−0.2172(8)	0.4590(2)	0.0327(8)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H25 ^a	0.530164	−0.327790	0.436765	0.039*
C26 ^a	0.54637(17)	−0.0139(8)	0.47612(19)	0.0341(9)
H26 ^a	0.577145	0.011562	0.465348	0.041*
C27 ^a	0.53057(15)	0.1526(9)	0.50812(18)	0.0287(9)
H27 ^a	0.550148	0.288551	0.518086	0.034*
H2	0.2738(7)	0.850(3)	0.4086(7)	0.026(5)*

^aOccupancy: 0.5.

direct method. The model was refined with version 2016/6 of SHELXL.

Discussion

Novel synthetic organic compounds of potential therapeutic value and of sustainable manufacturing process are of paramount importance to pharmaceutical industries. In this context, over 100 molecules bearing the sulfonamide functional group have featured on the market as approved drugs and they are found to have a broad spectrum of biological and pharmacological activities such as antibacterials, diuretics, anticonvulsants, hypoglycemics, and as HIV protease inhibitors [5, 6]. Due to the wide range of applications of sulfonamides in medicinal chemistry, several synthetic methods for the design of new derivatives and for installing the sulfonamide functionality have been reported [7, 8]. In addition, the synthesis of transition-metal *N*-substituted sulfonamide complexes of iridium and osmium, for instance, has demonstrated the growing importance for asymmetric transfer hydrogenation reactions as well as in the area of cancer chemotherapy [9–11]. Hence, given the comprehensive applicability of sulfonamides, we report herein the structure characterization of the β-amino sulfonamide compound 2-(4-fluorophenyl)-*N*-phenyl-2-(phenylamino)ethanesulfonamide by spectroscopic and X-ray crystallography techniques.

The asymmetric unit of the crystal structure of title compound contains 2-(4-fluorophenyl)-*N* phenyl-2-(phenylamino)ethanesulfonamide and disordered toluene molecules. In the title compound, fluorophenyl [C9–C8–C7–S1, 69.69(12)°] and phenylamino [C15–N2–C8–C7, 152.16(11)°] moieties are attached almost perpendicular to each other. The phenyl moiety is attached to sulphonamide group from the opposite direction of fluorophenyl group with the torsion angle (C1–N1–S1–C7) of −70.12(11)°. In the crystal structure, the toluene molecules which are in disorder in a 1:1 ratio are located around a centre of inversion. The crystal structure analysis reveals that four adjacent title molecules are interconnected through C–H⋯F (C⋯F, 3.3863(18) Å and ∠C–H⋯F, 150°) and C–H⋯π (C⋯π, 3.542(12) Å and ∠C–H⋯π, 156°) interactions, forming ternary units

with molecular cavities in which the disordered toluene molecules are located. Furthermore, the molecular cavities are interconnected through N—H...O hydrogen bonds formed between sulphonamide and phenyl amine groups [(i) N1—H1...O2, 2.8998(16) Å and ∠N1—H1...O2, 166° and (ii) N2—H2...O1, 3.0348(15) Å and ∠N2—H2...O1, 159.6(17)°]. The overall molecular packing displays one-dimensional channels running along [100] in which the toluene guest molecules are encapsulated.

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