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The crystal structure of *N*-((1*E*,2*E*)-1,3-bis(4-fluorophenyl)but-2-en-1-ylidene)-4-methylbenzenesulfonamide, C₂₃H₁₉F₂NO₂S

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.30 × 0.26 × 0.22 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.20 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	30.6°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	25700, 5918, 0.040
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4948
$N(\text{param})_{\text{refined}}$:	264
Programs:	Bruker [1], SHELX [2, 3]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.67666(5)	0.76333(10)	0.60380(5)	0.04259(17)
N1	0.74937(16)	0.8581(3)	0.54463(13)	0.0395(5)
F1	0.19538(14)	0.9388(4)	0.33216(15)	0.0808(8)
O1	0.59345(16)	0.6835(4)	0.57307(15)	0.0645(7)
C1	0.72464(18)	0.9038(3)	0.48069(15)	0.0337(5)
O2	0.6633(2)	0.8862(3)	0.66112(14)	0.0623(7)
F2	1.01725(18)	1.1620(3)	0.30411(15)	0.0789(7)
C2	0.62594(19)	0.8967(4)	0.44907(15)	0.0368(5)
H2	0.613745	0.812226	0.414414	0.044*
C3	0.55440(19)	1.0015(3)	0.46662(14)	0.0345(5)
C5	0.45930(18)	0.9899(3)	0.43025(15)	0.0332(5)
C4	0.5653(3)	1.1355(4)	0.5253(2)	0.0537(8)
H4A	0.632186	1.164913	0.530554	0.081*
H4B	0.541468	1.089827	0.570202	0.081*
H4C	0.529345	1.236956	0.512520	0.081*
C6	0.3752(2)	1.0301(4)	0.46695(16)	0.0424(6)
H6	0.378605	1.069358	0.514494	0.051*
C9	0.3635(2)	0.9185(5)	0.32505(18)	0.0491(7)
H9	0.359053	0.882029	0.277155	0.059*
C8	0.2824(2)	0.9569(5)	0.36452(19)	0.0501(8)
C7	0.2858(2)	1.0129(5)	0.43399(19)	0.0514(8)
H7	0.229538	1.039247	0.459014	0.062*
C10	0.4518(2)	0.9356(4)	0.35870(16)	0.0415(6)
H10	0.507580	0.910276	0.332891	0.050*
C11	0.8022(2)	0.9698(3)	0.43325(15)	0.0356(5)
C12	0.8986(2)	0.9525(4)	0.45340(18)	0.0411(6)
H12	0.914161	0.898263	0.496801	0.049*
C13	0.9707(2)	1.0151(4)	0.4095(2)	0.0520(8)
H13	1.035123	1.002025	0.422418	0.062*
C14	0.9458(3)	1.0973(4)	0.34618(19)	0.0522(8)

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Abstract

C₂₃H₁₉F₂NO₂S, orthorhombic, *Pna*2₁ (no. 33), $a = 13.8754(8)$ Å, $b = 7.7354(5)$ Å, $c = 18.4616(11)$ Å, $V = 1981.5(2)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0449$, $wR_{\text{ref}}(F^2) = 0.1066$, $T = 300(2)$ 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

In a representative experiment the ethanol (10 mL) solution of 4-fluoroacetophenone (14 mmol) was cooled under -15 °C in a brine bath, and thionyl chloride (20 mmol) was added in

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C15	0.8529(3)	1.1160(4)	0.32361(19)	0.0551(8)
H15	0.838536	1.169988	0.279904	0.066*
C16	0.7803(3)	1.0521(4)	0.36780(17)	0.0470(7)
H16	0.716311	1.064030	0.353736	0.056*
C17	0.75476(19)	0.5981(4)	0.63357(15)	0.0364(5)
C18	0.8467(2)	0.6366(4)	0.65922(17)	0.0448(7)
H18	0.867425	0.750683	0.662301	0.054*
C19	0.9065(2)	0.5035(4)	0.67994(17)	0.0476(7)
H19	0.968032	0.528994	0.696889	0.057*
C20	0.8772(2)	0.3322(4)	0.67612(16)	0.0451(7)
C21	0.7843(2)	0.2976(4)	0.65250(19)	0.0508(8)
H21	0.762717	0.183824	0.650977	0.061*
C22	0.7233(2)	0.4288(4)	0.63124(17)	0.0449(7)
H22	0.661245	0.403393	0.615404	0.054*
C23	0.9448(3)	0.1857(5)	0.6948(2)	0.0670(10)
H23A	0.941704	0.099054	0.657583	0.100*
H23B	0.926120	0.135808	0.740265	0.100*
H23C	1.009426	0.229029	0.698255	0.100*

30 min. After addition, the reaction solution was stirred for additional 2 h, then quenched by saturated K₂CO₃ solution. The resulting solution was extracted with dichloromethane for 3 times. The combined organic phase was concentrated under reduced pressure to obtain the reddish brown residue, which was subjected to silica gel column chromatography eluted with hexane/EtOAc (50:1) to afford the chalcone intermediate – 1,3-bis(4-fluorophenyl)but-2-en-1-one.

The chalcone intermediate (5 mmol) was mixed with TsNH₂ (6 mmol) and Et₃N (12.5 mmol) in 20 mL dichloromethane at 0 °C. To the solution was slowly added 1.0 M TiCl₄ (7.5 mL), which was initially stirred at 0 °C for 0.5 h, and then refluxed overnight. The organic layer was collected after addition of water which was then separated over a silica gel column eluted with hexane/dichloromethane/EtOAc (3:1.5:0.1) to obtain the viscous product, which was recrystallized from hexane/EtOAc solvent to get the title compound as colorless block crystal.

Experimental details

The structure was solved and refined using the Bruker SHELXTL software [2, 3].

Comment

Imines, on the one hand, belong to bioactive compounds with various biological activities including antidiabetic, anti-cancer and antimicrobial effects [4–6]. On the other hand, they are the important intermediates to achieve functional

molecules, such as quinolones and 1,3-oxazines [7, 8]. Thus, a lot of methods have been developed to approach them [9].

There is one crystallographically independent molecule in the asymmetric unit. The geometric parameters in the crystal structure of the title compound are all in normal ranges [10]. The length of C=C double bond was longer than that of C=N double bond (1.322(4) Å vs 1.279(4) Å). Both the configuration of double bonds are *E* which was usually ambiguous in the literature [11, 12]. The steric interactions between *β*-methyl and *p*-toluene sulfonyl destroy the conjugation effect of C=N and C=C double bonds, which was inferred from the dihedral angle of 74.09° between plane N1–C1–C2 and plane C2–C3–C4.

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