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Crystal structure of 3-fluoro-3-methyl-1-((2-nitrophenyl)sulfonyl)-5,5-diphenylpiperidine, $C_{24}H_{23}FN_2O_4S$

Table 1: Data collection and handling.

Crystal:	Block
Size:	0.34 × 0.25 × 0.14 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.19 mm ⁻¹
Diffraction, scan mode:	Brker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.1°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	20955, 3916, 0.025
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3324
$N(\text{param})_{\text{refined}}$:	290
Programs:	SHELX [1], Bruker [2]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.64923(3)	0.23860(3)	0.53719(2)	0.04382(14)
F1	0.56183(7)	0.19174(7)	0.34211(6)	0.0596(3)
N1	0.84621(11)	0.22597(10)	0.61493(8)	0.0564(4)
N2	0.67534(10)	0.22113(8)	0.45801(7)	0.0436(3)
O1	0.88241(11)	0.18576(9)	0.56947(8)	0.0724(4)
O2	0.84328(15)	0.20273(11)	0.67345(8)	0.1040(7)
O3	0.67228(9)	0.16309(7)	0.57439(6)	0.0547(3)
O4	0.55706(8)	0.27155(8)	0.53861(7)	0.0596(3)
C1	0.80758(12)	0.31134(10)	0.59977(8)	0.0440(4)
C2	0.85969(13)	0.37999(13)	0.62181(9)	0.0574(5)
H2	0.9170	0.3710	0.6426	0.069*
C3	0.82649(14)	0.46174(12)	0.61289(10)	0.0610(5)
H3	0.8616	0.5085	0.6272	0.073*
C4	0.74157(13)	0.47428(11)	0.58296(10)	0.0560(5)
H4	0.7186	0.5297	0.5778	0.067*
C5	0.68984(12)	0.40578(10)	0.56049(8)	0.0461(4)
H5	0.6323	0.4154	0.5401	0.055*
C6	0.72223(11)	0.32248(9)	0.56777(8)	0.0390(3)
C7	0.65041(13)	0.28694(11)	0.40770(9)	0.0505(4)
H7A	0.6983	0.3307	0.4064	0.061*
H7B	0.5923	0.3140	0.4207	0.061*
C8	0.64032(12)	0.24653(11)	0.33812(9)	0.0483(4)
C9	0.72395(12)	0.19219(10)	0.31941(8)	0.0455(4)
H9A	0.7763	0.2300	0.3107	0.055*
H9B	0.7103	0.1620	0.2774	0.055*
C10	0.75306(11)	0.12599(10)	0.37353(7)	0.0390(3)
C11	0.76125(11)	0.17418(10)	0.44172(8)	0.0414(4)
H11A	0.7744	0.1334	0.4778	0.050*
H11B	0.8128	0.2142	0.4393	0.050*
C12	0.61871(16)	0.31316(14)	0.28417(11)	0.0692(6)

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Abstract

$C_{24}H_{23}FN_2O_4S$, orthorhombic, *Pbca* (no. 19), $a = 14.3646(18)$ Å, $b = 15.5927(19)$ Å, $c = 19.645(3)$ Å, $V = 4400.1(10)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0349$, $wR_{\text{ref}}(F^2) = 0.0982$, $T = 296(2)$ 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

The mixture solution of (diacetoxyiodo)benzene and fluoroboric acid in CH_2Cl_2 was stirred at room temperature

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Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H12A	0.6106	0.2854	0.2410	0.104*
H12B	0.6692	0.3533	0.2813	0.104*
H12C	0.5626	0.3430	0.2961	0.104*
C13	0.68651(11)	0.04841(10)	0.37521(8)	0.0396(3)
C14	0.63639(12)	0.02348(11)	0.43217(9)	0.0506(4)
H14	0.6388	0.0568	0.4714	0.061*
C15	0.58293(14)	−0.05027(12)	0.43152(11)	0.0616(5)
H15	0.5501	−0.0659	0.4704	0.074*
C16	0.57754(14)	−0.10070(12)	0.37467(11)	0.0607(5)
H16	0.5414	−0.1502	0.3746	0.073*
C17	0.62658(13)	−0.07693(12)	0.31754(10)	0.0571(5)
H17	0.6235	−0.1104	0.2784	0.068*
C18	0.68040(12)	−0.00358(11)	0.31804(8)	0.0487(4)
H18	0.7134	0.0114	0.2791	0.058*
C19	0.85073(10)	0.08953(10)	0.35946(8)	0.0399(3)
C20	0.88818(11)	0.03089(10)	0.40516(9)	0.0464(4)
H20	0.8544	0.0163	0.4439	0.056*
C21	0.97393(12)	−0.00622(11)	0.39462(9)	0.0496(4)
H21	0.9980	−0.0443	0.4265	0.060*
C22	1.02365(12)	0.01291(13)	0.33722(9)	0.0568(5)
H22	1.0807	−0.0133	0.3291	0.068*
C23	0.98860(14)	0.07077(15)	0.29204(10)	0.0712(6)
H23	1.0224	0.0844	0.2531	0.085*
C24	0.90320(13)	0.10971(14)	0.30321(9)	0.0610(5)
H24	0.8811	0.1500	0.2722	0.073*

for 10 min and *N*-(4-methyl-2,2-diphenylpent-4-en-1-yl)-2-nitrobenzenesulfonamide was added to continue the reaction for 10 min. The mixture was purified by flash column chromatography (petroleum ether/ethyl acetate = 20/1) to afford the title compound.

Experimental details

The U_{iso} values of the hydrogen atoms of methyl groups were set to 1.5 $U_{eq}(C)$. Besides, the U_{iso} values of phenyl, methylene hydrogen atoms were set to 1.2 $U_{eq}(C)$.

Comment

Fluorine-containing compounds are important building blocks for the synthesis of bioactive molecules [3–5]. The title structure can easily be deprotected of sulfonyl protection [6], and then it could be transformed into a range of compounds containing a piperidine ring and a fluorine atom [7–12]. These compounds have a wide range of biomedical activities.

The title structure contains a chair-type piperidine ring similar to the literature [13]. The angle between the fluorine atom and the methyl group attached to the C8 carbon atom on the piperidine ring is 106.79(15)°. The dihedral angles between the plane 1 of phenyl (C1, C2, C3, C4, C5 and C6)

and the plane 2 of sulfonyl group (O3, S1 and O4), the plane 3 of nitril (O1, N1 and O2) are 55.0(1)°, 75.2(1)°, respectively. Besides, the dihedral angle between the plane 2 and plane 3 is 52.8(1)°. The dihedral angle between two phenyl rings directly connected the piperidine ring is 83.534(55)°.

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