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Crystal structure of 5-methyl-3-phenyl-1-tosyl-1,2,3,4-tetrahydropyridine, C₁₉H₂₁NO₂S

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.35 × 0.28 × 0.15 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.19 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.1°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	16417, 3115, 0.077
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1758
$N(\text{param})_{\text{refined}}$:	210
Programs:	SHELX [1], Bruker [2]

Source of material

The title compound was prepared by a reported method, which was slightly modified [3]. The mixture of (diacetoxyiodo)benzene and BF₃·Et₂O in anhydrous CH₂Cl₂ was stirred at room temperature for 1 min and 4-methyl-N-(4-methyl-2-phenylpent-4-en-1-yl)benzenesulfonamide was added to continue the reaction for 3 h. The mixture was concentrated and the residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 5/1) to afford the desired compound.

Experimental details

The U_{iso} values of the hydrogen atoms of methyl groups were set to 1.5 $U_{\text{eq}}(\text{C})$ while the U_{iso} values of phenyl, methylene hydrogen atoms were set to 1.2 $U_{\text{eq}}(\text{C})$. There is a small (not refined) disorder in the region of the C8, C9 moiety, which leads to an artificial flattening of this moiety and shortening of the bond lengths.

Comment

As useful building blocks in diverse biologically active nitrogen-containing heterocyclic structures, tetrahydropyridines are widely studied in clinical and preclinical trials [4–6]. The title compound could be used as main precursor for the synthesis of nitrogen-heterocycles, such as piperidine [7, 8], diludine [9] and pyridine [10, 11] compounds.

In this structure, the substituted phenyl group on C9 and the *p*-toluenesulfonyl group on N1 are located on the equatorial bond. These substituents are also located in the same side of the 1,2,3,4-tetrahydropyridine ring. The dihedral

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Abstract

C₁₉H₂₁NO₂S, orthorhombic, *Pbca* (no. 61), $a = 16.792(5)$ Å, $b = 9.153(3)$ Å, $c = 22.809(6)$ Å, $V = 3505.8(16)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0622$, $wR_{\text{ref}}(F^2) = 0.1494$, $T = 296(2)$ 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.

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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	0.07474(6)	0.85365(12)	0.38375(4)	0.0705(3)
N1	0.03435(16)	0.8290(3)	0.44825(12)	0.0658(8)
O1	0.01643(16)	0.9260(3)	0.34859(11)	0.0915(9)
O2	0.15099(15)	0.9197(3)	0.39390(11)	0.0844(8)
C1	0.1290(2)	0.2527(5)	0.2811(2)	0.0941(14)
H1A	0.0829	0.1934	0.2886	0.141*
H1B	0.1747	0.2089	0.2992	0.141*
H1C	0.1375	0.2597	0.2396	0.141*
C2	0.1161(2)	0.4032(5)	0.30609(15)	0.0646(10)
C3	0.0439(2)	0.4756(5)	0.29946(16)	0.0744(12)
H3	0.0032	0.4309	0.2784	0.089*
C4	0.0313(2)	0.6106(5)	0.32302(15)	0.0700(11)
H4	−0.0179	0.6560	0.3185	0.084*
C5	0.0914(2)	0.6802(4)	0.35364(13)	0.0581(9)
C6	0.1639(2)	0.6111(5)	0.36073(14)	0.0626(10)
H6	0.2047	0.6565	0.3814	0.075*
C7	0.1750(2)	0.4750(5)	0.33697(15)	0.0652(10)
H7	0.2240	0.4293	0.3418	0.078*
C8	0.0840(2)	0.7682(6)	0.49465(16)	0.0961(15)
H8A	0.1007	0.6712	0.4827	0.115*
H8B	0.1316	0.8278	0.4978	0.115*
C9	0.0504(2)	0.7578(7)	0.54960(19)	0.116(2)
H9	0.0477	0.8615	0.5600	0.139*
C10	−0.0353(2)	0.7187(5)	0.55439(15)	0.0715(11)
H10A	−0.0576	0.7660	0.5887	0.086*
H10B	−0.0399	0.6139	0.5599	0.086*
C11	−0.0825(2)	0.7619(4)	0.50197(16)	0.0657(10)
C12	−0.04899(19)	0.8092(4)	0.45380(16)	0.0655(10)
H12	−0.0813	0.8308	0.4218	0.079*
C13	−0.1719(2)	0.7560(5)	0.50770(18)	0.0975(15)
H13A	−0.1889	0.8248	0.5369	0.146*
H13B	−0.1878	0.6594	0.5191	0.146*
H13C	−0.1959	0.7802	0.4708	0.146*
C14	0.1028(2)	0.7023(6)	0.59875(18)	0.0762(12)
C15	0.1141(3)	0.7821(5)	0.6491(2)	0.0891(13)
H15	0.0903	0.8733	0.6529	0.107*
C16	0.1600(3)	0.7285(6)	0.6935(2)	0.0951(14)
H16	0.1673	0.7839	0.7272	0.114*
C17	0.1951(3)	0.5956(6)	0.6891(2)	0.0974(15)
H17	0.2265	0.5602	0.7195	0.117*
C18	0.1841(3)	0.5143(6)	0.6397(2)	0.1043(16)
H18	0.2082	0.4232	0.6363	0.125*
C19	0.1373(3)	0.5667(6)	0.59483(19)	0.0935(14)
H19	0.1290	0.5099	0.5616	0.112*

angles between the plane 1 of phenyl (C2, C3, C4, C5, C6 and C7) and the plane 2 of sulfonyl group (O1, S1 and O2), the plane 3 of phenyl (C14, C15, C16, C17, C18 and C19) are 51.56(14)°,

37.91(14)°, respectively. Moreover, the atoms of N1, C10 and C13 located on alkene are not strictly in the same plane. The dihedral angle between the plane 4 (N1, C12 and C11) and plane 5 (C13, C11 and C12), plane 6 (C10, C11 and C12) are 5.8(4)°, 2.7(4)°, respectively. The distance of carbon-carbon double bond from C11 to C12 is calculated to be 1.308(5) Å. However, the distance of carbon-carbon single bonds of C11–C10 and C11–C13 are calculated to be 1.488(5) Å, 1.507(5) Å, respectively. The angles of C10–C11–C12 and C13–C11–C12 are 122(3)°, 120.9(4)°, respectively.

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