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Crystal structure of 5-methyl-3,3-diphenyl-1-tosyl-1,2,3,4-tetrahydropyridine, C₂₅H₂₅NO₂S

Table 1: Data collection and handling.

Crystal:	Block, colorless
Size:	0.39 × 0.30 × 0.17 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.18 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	25.1°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	5068, 3373, 0.025
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2874
$N(\text{param})_{\text{refined}}$:	264
Programs:	Bruker programs [1], SHELX [2]

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Abstract

C₂₅H₂₅NO₂S, monoclinic, $P2_1$ (no. 4), $a = 9.371(4)$ Å, $b = 11.397(4)$ Å, $c = 9.777(4)$ Å, $\beta = 95.117(6)^\circ$, $V = 1040.0(7)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0423$, $wR_{\text{ref}}(F^2) = 0.0921$, $T = 296(2)$ K.

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

Source of material

The mixture of 3-chlorobenzoperoxoic acid and 1-iodo-4-methylbenzene in CH₂Cl₂ was stirred at 0 °C for

30 min and BF₃·Et₂O was added to continue the reaction for 1 h. 4-Methyl-*N*-(4-methyl-2,2-diphenylpent-4-en-1-yl) benzenesulfonamide was added to the reaction solution for an additional 1 h at room temperature and then the reaction mixture was evaporated. The residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 46/1) to afford the desired product.

Experimental details

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

Discussion

Nitrogen-containing heterocyclic compounds are valuable in pharmaceutical chemistry [3–6]. The title structure can easily be converted into a series of compounds containing a piperidine ring due to the activity of the C=C bonds [7, 8]. They are widely used in antitumor [9], anti-inflammatory [10], anxiety [11], anticonvulsant [12] studies.

In the title structure, the 1,2,3,4-tetrahydropyridine ring adopts a chair conformation. The dihedral angles between the plane 3 of sulfonyl group (O1, S1 and O2) and the plane 1 of phenyl (C14, C15, C16, C17, C18 and C19), the plane 2 of phenyl (C20, C21, C22, C23, C24 and C25), and its connected plane 4 of phenyl (C2, C3, C4, C5, C6 and C7) are 19.64(1)°, 63.87(9)°, 50.82(1)°, respectively. Besides, the dihedral angle between two phenyl rings (plane 1 and plane 2) directly connected the 1,2,3,4-tetrahydropyridine ring is 79.78(9)°, and the dihedral angles between the plane 4 and plane 1, plane 2 are 58.93(8)°, 88.15(8)°, respectively. No hydrogen bonds were found.

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

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
S1	0.75726(9)	0.25438(6)	0.78180(7)	0.0423(2)
N1	0.7289(2)	0.2942(2)	0.6209(2)	0.0393(6)
O1	0.6424(2)	0.17697(18)	0.8069(2)	0.0542(6)
O2	0.9025(2)	0.2161(2)	0.8022(2)	0.0602(7)
C1	0.7110(4)	0.7004(3)	1.1058(4)	0.0701(11)
H1A	0.8040	0.7226	1.1468	0.105*
H1B	0.6470	0.6902	1.1763	0.105*
H1C	0.6747	0.7608	1.0436	0.105*
C2	0.7223(3)	0.5872(3)	1.0285(3)	0.0449(8)
C3	0.6010(3)	0.5315(3)	0.9699(3)	0.0477(8)
H3	0.5115	0.5638	0.9803	0.057*
C4	0.6100(3)	0.4296(3)	0.8967(3)	0.0436(7)
H4	0.5273	0.3930	0.8582	0.052*
C5	0.7422(3)	0.3820(2)	0.8808(2)	0.0342(6)
C6	0.8644(3)	0.4348(3)	0.9405(3)	0.0444(8)
H6	0.9537	0.4014	0.9315	0.053*
C7	0.8540(3)	0.5371(3)	1.0135(3)	0.0494(8)
H7	0.9367	0.5729	1.0532	0.059*
C8	0.8287(3)	0.3717(3)	0.5678(3)	0.0444(8)
H8	0.9178	0.3816	0.6165	0.053*
C9	0.8003(3)	0.4302(3)	0.4526(3)	0.0411(7)
C10	0.6593(3)	0.4179(3)	0.3693(3)	0.0430(8)
H10A	0.6748	0.4208	0.2725	0.052*
H10B	0.5986	0.4837	0.3887	0.052*
C11	0.5819(3)	0.3033(3)	0.3987(2)	0.0351(6)
C12	0.5811(3)	0.3008(3)	0.5577(3)	0.0403(7)
H12A	0.5353	0.3711	0.5887	0.048*
H12B	0.5272	0.2334	0.5849	0.048*
C13	0.9056(4)	0.5146(4)	0.4000(3)	0.0661(10)
H13A	0.9912	0.5160	0.4616	0.099*
H13B	0.8642	0.5917	0.3940	0.099*
H13C	0.9286	0.4901	0.3106	0.099*
C14	0.4247(3)	0.3014(3)	0.3381(3)	0.0370(6)
C15	0.3631(3)	0.3923(3)	0.2617(3)	0.0469(8)
H15	0.4182	0.4576	0.2441	0.056*
C16	0.2206(4)	0.3886(3)	0.2103(4)	0.0635(10)
H16	0.1813	0.4514	0.1592	0.076*
C17	0.1372(4)	0.2940(3)	0.2338(4)	0.0625(10)
H17	0.0417	0.2914	0.1988	0.075*
C18	0.1975(4)	0.2028(3)	0.3102(4)	0.0586(9)
H18	0.1420	0.1379	0.3283	0.070*
C19	0.3381(3)	0.2063(3)	0.3598(3)	0.0494(8)
H19	0.3769	0.1427	0.4098	0.059*
C20	0.6597(3)	0.1964(3)	0.3463(3)	0.0337(6)
C21	0.7077(3)	0.1977(3)	0.2154(3)	0.0503(8)
H21	0.6947	0.2649	0.1618	0.060*
C22	0.7735(4)	0.1021(4)	0.1641(3)	0.0612(10)
H22	0.8063	0.1058	0.0772	0.073*
C23	0.7913(4)	0.0020(3)	0.2387(4)	0.0615(10)
H23	0.8356	−0.0626	0.2027	0.074*
C24	0.7435(3)	−0.0037(3)	0.3687(4)	0.0554(9)
H24	0.7544	−0.0723	0.4202	0.066*
C25	0.6796(3)	0.0937(3)	0.4204(3)	0.0446(7)
H25	0.6489	0.0901	0.5082	0.053*

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