

endo-N-(5,5-Dimethyl-6-methylene-bicyclo[2.2.1]hept-2-yl)-4-methyl-benzenesulfonamide

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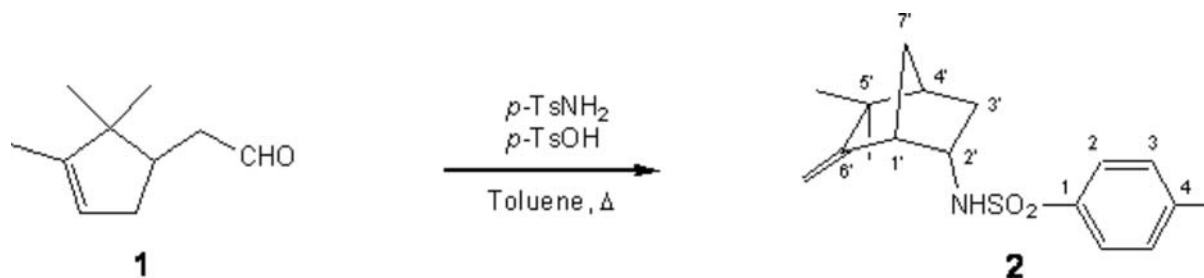
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p-Toluenesulfonic acid (35 mg, 0.20 mmol) was added to a stirred mixture of campholenic aldehyde (**1**) (725 mg, 3.81 mmol) and *p*-toluenesulfonamide (1.22 g, 6.96 mmol) in toluene (20 mL). Then a Dean-Stark trap device was fit and the reaction refluxed for 0.5 h. After that, the mixture was cooled to 0 °C, filtered through a silica gel pad and the solvent evaporated under reduced pressure to yield a residue (1.30g) which was purified by flash chromatography on silica gel, using a 2:1 Hexane/Et₂O mixture as eluent, to give the title compound **2** (591 mg, 1.93 mmol, 51%).

Mp: 101.2–104.3 °C (White crystals, from hexane)

IR (KBr, cm^{-1}): 3264 (N-H), 3062, 1656, 881 (C=C), 3062, 813 (Ar), 1337, 1164 (SO₂), 667 (C-N).

¹H NMR (300 MHz, CDCl₃, d, ppm): 1.00 (3H, s, Me_a-5'), 1.02 (3H, s, Me_b-5'), 1.17–1.30 (2H, *m*, H-3', H-7'), 1.70–1.82 (3H, *m*, H'-3', H'-7', H-4'), 2.34 (1H, *br d*, J=3.3 Hz, H-1'), 2.42 (3H, s, Me-4), 3.64–3.75 (1H, *m*, H-2'), 4.64 (1H, *d*, J=10.2 Hz, N-H), 4.67 (1H, s, CH₂-6'), 4.85 (1H, s, CH₂-6'), 7.30 (2H, *d*, J=8.3 Hz, H-3, H-5), 7.74 (2H, *d*, J=8.3 Hz, H-2, H-6). Some signals were assigned by means of 2D NMR experiments.

¹³C NMR (75 MHz, CDCl₃, d, ppm): 138.20 (C-1), 126.96 (C-2), 129.57 (C-3), 143.18 (C-4), 21.43 (Me-4), 50.49 (C-1'), 51.93 (C-2'), 31.99* (C-3'), 47.40 (C-4'), 42.30 (C-5'), 158.03 (C-6'), 36.31* (C-7'), 106.69 (CH₂-6'), 24.64 (Me_a-5'), 29.41 (Me_b-5'). Some signals were assigned by means of 2D NMR experiments.

*These signals may be interchanged.

MS (70 eV, m/z): 305 (M^+ , 4%), 240 ($M^+ - SO_2H$, 14), 185 (3), 171 (3), 162 (4), 155 (Ts^+ , 12), 150 ($M^+ - Ts$, 17), 134 ($M^+ - TsNH_2$, 23), 121 ($M^+ - TsNH - Me$, 39), 108 (72), 91 ($MePh^+$, 100), 79 (26), 65 (42), 53 (17), 41 (35), 39 (37).

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