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(±)-1-(4-Hydroxy-3-methoxyphenyl)-4,4-dimethyl-3-pentanol

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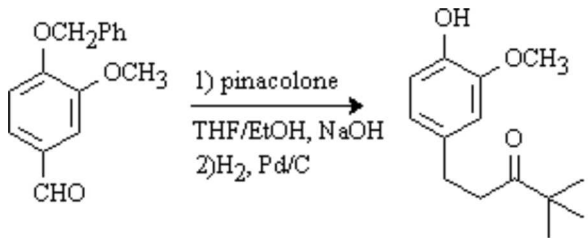

The discussion and purpose for the synthesis of this compound has been reported elsewhere [1]. To a cold (0°C) solution of 1-(4-hydroxy-3-methoxyphenyl)-4,4-dimethyl-3-pentanone (235 mg, 1.0 mmol) in EtOH (15 mL) was added sodium borohydride (40 mg, 1.1 mmol, 1.1 eq). The solution was stirred at 0°C for 30 min., then at room temperature for 1 h. 10% HCl (10 mL) was added and the solution was stirred at room temperature for 2 h. The solution was concentrated in vacuo and the aqueous residue was extracted with dichloromethane (3 x 15 mL). The organic fractions were combined, dried ($MgSO_4$) and the solvent was evaporated in vacuo. Chromatography on silica gel (40% EtOAc/hexanes) afforded a colorless oil (227 mg, 96%).

IR (neat) cm^{-1}: 3435 (OH).

^{1}H-NMR ($CDCl_3$) d: 0.89 (s, 9H, CH_3), 1.43 (broad s, 1H, exchangeable with D_2O, OH), 1.55 (m, 1H, H-2a), 1.80 (m, 1H, H-2b), 2.56 (m, 1H, H-1a), 2.85 (m, 1H, H-1b), 3.22 (broad d, 1H, J=10.5 Hz, H-3), 3.88 (s, 3H, OCH3), 5.49 (s, 1H, exchangeable with D_2O, OH), 6.72 (m, 2H, Ar-H2 and Ar-H6), 6.84 (d, 1H, J=8.7 Hz, Ar-H5).

^{13}C-NMR ($CDCl_3$) d: 25.9 (CH_3), 33.3 (C-1), 33.9 (C-2), 35.2 (C-4), 56.1 (OCH_3), 79.6 (C-3), 111.3 (ArC-2), 114.4 (ArC-5), 121.1 (ArC-6), 134.6 (ArC-1), 143.8 (ArC-4), 146.6 (ArC-3).

MS m/e (rel %): 238 [M+] (58), 164 (27), 150 (31), 138 (37), 137 (100), 131 (16).

Anal. calc. for $C_{14}H_{22}O_3$: C 70.54, H 9.31; found: C 70.63, H 9.22.

Acknowlegment

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Reference

1. Plourde G.L. *Tetrahedron Letters* **2002**, *43*, 3597-3599.