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1-(4-Hydroxy-3-methoxyphenyl)-4,4-dimethyl-3-pentanone

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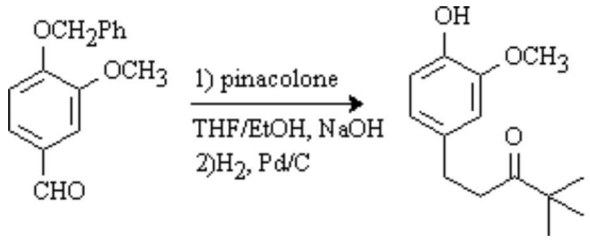

The discussion and purpose for the synthesis of this compound has been reported elsewhere [1]. To a solution of 4-benzyloxy-3-methoxybenzaldehyde (1.45 g, 6.0 mmol) in 50% THF/EtOH (100 mL) was added sodium hydroxide (0.5 g, 12.5 mmol, 2.1 eq) and pinacolone (2.0 g, 20 mmol, 3.3 eq) The resulting mixture was refluxed for 6 h (complete reaction by tlc). The solution was cooled to room temperature and 10% HCl (50 mL) was added. The mixture was concentrated in vacuo and the aqueous residue was extracted with dichloromethane (3 x 30 mL). The organic fractions were combined, dried ($MgSO_4$) and the solvent was evaporated in vacuo to give a brown oil. The crude product was partially purified by chromatography on silica gel (20% EtOAc/hexanes) to give a yellow oil. This oil was dissolved in EtOAc (100 mL), Pd/C (135 mg) was added and the solution was stirred under a positve atmosphere of H_2 for 20 h. The suspension was filtered through celite and the solvent was evaporated in vacuo. Chromatography on silica gel (20% EtOAc/hexanes) afforded a white solid (1.09 g, 77%).

mp: 65-66°C.

IR (KBr) cm^{-1}: 3441 (OH), 1708 (CO).

^{1}H-NMR ($CDCl_3$) d: 1.10 (s, 9H, CH_3), 2.78 (m, 4H, H-1 and H-2), 3.87 (s, 3H, OCH_3), 5.39 (s, 1H, exchangeable with D_2O, OH), 6.62 (d, 1H, J=7.8 Hz, ArH-6), 6.69 (s, 1H, ArH-2), 6.83 (d, 1H, J=7.8 Hz, ArH-5).

^{13}C-NMR ($CDCl_3$) d: 26.3 (CH_3), 30.0 (C-2), 39.0 (C-1), 44.3 (C-4), 56.1 (OCH_3), 111.2 (ArC-2), 114.5 (ArC-5), 121.0 (ArC-6), 133.8 (ArC-1), 144.0 (ArC-4), 146.5 (ArC-3), 215.4 (CO).

MS m/e (rel %): 236 [M+] (48), 179 (25), 151 (9), 137 (100).

Anal. calc. for $C_{14}H_{20}O_3$ C 71.14, H 8.54, found C 71.32, H 8.49.

Acknowlegment

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Reference

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

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