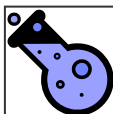
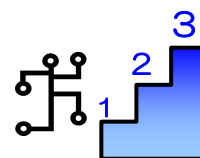




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逆合成検索 - 原料を合成する反応の検索 -

1 合成方法を知りたい物質 (原料 a) にカーソルを合わせ、>> ボタンをクリックし Synthesize this... を選択します。

2 原料 a を合成する反応が表示されました。

3 Experimental Procedure をクリックすると詳細な実験項が表示されます。

Experimental Procedure

Journal of Medicinal Chemistry

Naphthalen-1-yl 2-Bromo-4-methylbenzoate (57). Thionyl chloride (0.17 mL, 2.40 mmol) was added to 2-bromo-4-methylbenzoic acid (430 mg, 2 mmol) in DCM (3 mL) and DMF (0.1 mL), and the mixture was refluxed under nitrogen atmosphere for 1 h. After cooling to rt, it was concentrated in vacuo to give the title compound as a pale-yellow solid, which was used directly in the next step. Naphthalen-1-ol (268 mg, 2.00 mmol) was dissolved in THF (5 mL), then DMAP (5 mg) and ethyldiisopropylamine (0.36 mL, 2.05 mmol) were added, and the mixture was cooled to 0 °C for 10 min. Freshly prepared 2-bromo-4-methylbenzoyl chloride in dry THF (10 mL) was added to the mixture via cannula, and the resulting mixture was stirred at 25 °C for 2 h, diluted with diethyl ether (150 mL), and quenched by the addition of water (15 mL). The organic layer was washed with HCl and NaHCO₃ and then dried (Na₂SO₄) and concentrated in vacuo. The residue was purified with flash chromatography, eluting with hexane:EtOAc = 10:1, to give 57. Naphthalen-1-yl 2-Bromo-4-methylbenzoate (57). Yield 96%. ¹H NMR (300 MHz, CDCl₃, ppm): 8.2-8.1 (s, 3H, CH), 7.27 (dd, J=0.9, 8.1 Hz, 1H, aromatic), 7.40 (dd, J=1.2, 7.5 Hz, 1H, aromatic), 7.48-7.52 (m, 3H, aromatic), 7.87-7.90 (m, 1H, aromatic), 7.96-7.99 (m, 1H, aromatic), 8.13 (d, J=8.1 Hz, 1H, aromatic), 7.87-7.90 (m, 1H, aromatic), 7.96-7.99 (m, 1H, aromatic), 8.13 (d, J=8.1 Hz, 1H, aromatic).

* 実験項が表示されるのは 2000-2010 年の ACS 発行の雑誌と 2000-2010 年の米国特許, ヨーロッパ特許, PCT 出願で 英語記載の文献です. (2011 年 11 月現在)

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