(R,S)-Benzyl 5-(1-Hydroxyethyl)-1H-pyrrole-2-carboxylate

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A stirred solution of the pyrrole benzyl ester 1 [1] (1.20 g, 5.23 mmol, 1 equiv) in dry tetrahydrofuran (100 mL) under an inert atmosphere was cooled to -23°C (dry ice/carbon tetrachloride). Methyllithium (3.27 mL of a 1.6 M solution in ether, 5.23 mmol, 1 equiv) was added over 10 min, and the solution was stirred at -23°C for 1.5 h. 1H NMR analysis of an aliquot from the reaction indicated a 1.2:1 mixture of starting material to product. A further 3.93 mL of methyllithium (6.28 mmol, 1.2 equiv) was added over 10 min, and the solution was stirred at -23°C for an additional 1.5 h. The resultant solution was poured onto an ether/ice bath, and upon melting of the ice the layers were separated. The organic layer was washed with water (2 x 50 mL), aqueous saturated brine (50 mL), dried (MgSO4), and the solvent was removed by evaporation under reduced pressure. Flash chromatography on silica (ethyl acetate/petroleum ether, 3:1) afforded the title compound 2 (1.18 mg, 92%) as a light pink solid.

mp 82-84°C.

IR (CHCl3) 1676.0, 2359.7, 2971.1, 3165.5, 3389.2.

1H NMR (CDCl3, 500 MHz) d 1.54 (d, 3H, J = 6.8 Hz, CH(OH)CH3), 2.55 (s(br), 1H, CHOH), 4.94 (q, 1H, J = 6.2 Hz, CHOCH), 5.29 (s, 2H, CH2Ph), 6.05 (m, 1H, pyrrole H4), 6.89 (m, 1H, pyrrole H3), 7.31-7.41 (m, 5H, ArH), 9.67 (s(br), 1H pyrrole NH).

13C NMR (CDCl3, 75 MHz) d 23.1 (CH(OH)CH3), 63.9 (CHOH), 65.9 (CH2Ph), 106.4 (pyrrole C4), 116.4 (pyrrole C3), 121.3 (pyrrole C2), 127.9, 128.1, 128.5, 136.0 (ArC), 141.8 (pyrrole C5), 161.6 (CO2).


Reference


Sample availability: available from the authors.