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N-(9-Fluorenylmethoxycarbonyl)-L-serine Amide

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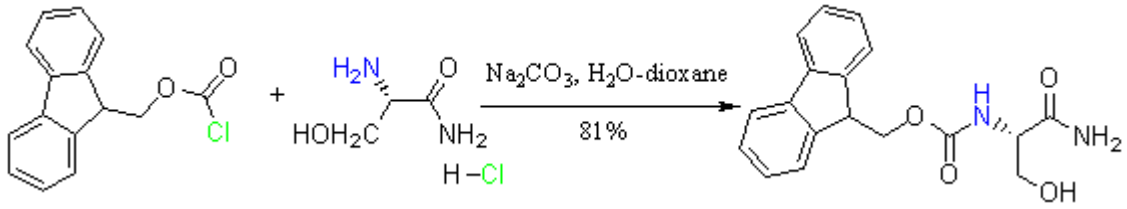

The synthesis of *N*-Fmoc-L-serine amide was performed by the well known Schotten-Baumann acylation method [1]. L-Serine amide hydrochloride (1.00 g, 7.1 mmol) and 1.50 g (14.2 mmol) of Na_2CO_3 were dissolved in dioxane-water 2:1 (45 ml). Then a solution of 2.00 g (7.7 mmol, 1.1 equivalent) of Fmoc chloride in dioxane (20 ml) was added dropwise over 1 h. The reaction mixture was stirred overnight at room temperature. According to TLC analysis, all the starting material was converted. The reaction mixture was evaporated, the resulting solid was triturated with 10% aq. $NaHSO_4$ (50 ml), filtered, washed with H_2O and ether and dried. As the crude product does not dissolve in apolar solvents nor ethyl acetate, crystallization was performed in tetrahydrofuran-ethyl acetate (1:3 v/v) to give 1.87 g (81%) of the title compound as a white solid.

Mp.: 155-156 °C.

$[a]_D^{20}$= +13.7 (c 2.5, tetrahydrofuran).

^{1}H NMR (DMSO-d_6, 500 MHz): 3.56 (m, 2H, C*H*$_2$OH); 3.96 (m, 1H, C*H*NH); 4.21 (t, *J* 6.6 Hz, 1H, C*H*CH$_2$O); 4.26 (d, *J* 6.2 Hz, 2H, CH$_2$O); 4.82 (t, *J* 5.7 Hz, 1H, OH); 7.04 (s, 1H, 1/2 NH$_2$); 7.26 (s, 1H, 1/2 NH$_2$); 7.13 (d, *J* 8.3 Hz, 1H, NH); 7.32 (dd, *J* 7.4 Hz, 2H, aromatic CH), 7.40 (dd, *J* 7.4 Hz, 2H, aromatic CH), 7.72 (d, *J* 5.2 Hz, 2H, aromatic CH), 7.87 (d, *J*7.5 Hz, 2H, aromatic CH).

^{13}C NMR (DMSO-d_6, 125 MHz, assignment based on *J*-modulated spin-echo, HMQC and COSY experiments): 46.6 (*C*HCH$_2$); 57.0 (CHNH); 61.7 (CH$_2$OH); 65.6 (CH$_2$O); 120.0; 125.2; 127.0; 127.6 (aromatic CHs); 140.6; 143.8 (aromatic C$_q$s); 155.8 (CONH); 172.0 (CONH$_2$).

ESI-MS (in methanol-H_2O, *m/z*, %): 326.9 (64, $[M+H]^+$), 343.9 (61, $[M+NH_4]^+$), 348.9 (100, $[M+Na]^+$), 653.3 (3, $[2M+H]^+$), 675.3 (13, $[2M+Na]^+$).

Anal calcd. for $C_{18}H_{18}N_2O_4$ (326.35): C, 66.25; H,5.56; N, 8.58; found C, 66.11; H, 5.45; N, 8.55 %.

Reference

1. Kocienski, P. *Protecting Groups*; Georg Thieme Verlag: Stuttgart, 1994: p 204.

Sample availability: available from the authors and MDPI (MDPI ID 18866).