(Benzoylamino)methyl 4-Acetyloxybenzoate

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Abstract: (Benzoylamino)methyl 4-acetyloxybenzoate (3) was obtained in a reaction of benzamidomethylation of 4-acetyloxybenzoic acid (2) with (benzamidomethyl)triethylammonium chloride (1).

Keywords: 4-acetyloxybenzoic acid; benzamidomethylation

In the course of our work on the synthesis of some new derivatives of 4-hydroxybenzoic acid [1–3], an additional compound, (benzoylamino)methyl 4-acetyloxybenzoate (3) was synthesized.

4-Acetyloxybenzoic acid (2) was benzamidomethylated with (benzamidomethyl)triethylammonium chloride (1) as a reagent for benzamidomethylation [4]. The reaction was performed in dioxane suspension of 1 in the presence of a small amount of triethylamine at 50 °C (Scheme 1). At the end of the reaction, water was added to the reaction mixture to precipitate the product. However, the crystals of 3 were formed slowly, which is unusual comparing to similar procedures for isolation of benzamidomethyl esters [4]. The maximal yield of almost pure crude product was 48%.
Scheme 1. Synthetic routes to the title compound 3.

Experimental

Compound 2 is not commercially available and it was synthesised as described previously [5].

**(Benzoylaminomethyl 4-acetyloxybenzoate (3))**

To a suspension of 1 (0.674 g, 2.49 mmol) in dioxane (30 mL), 2 (0.370 g, 2.05 mmol) and TEA (0.1 mL, 0.72 mmol) were added. The mixture was stirred and heated at 50 °C for 20 h. After cooling, cold water was added to the mixture until a white precipitate occurred. Colorless crystals were collected by simple filtration. Purification was performed by dissolving the crystals in dioxane and by precipitation with water. Significant loss of 3 during the purification was observed.

Melting point of pure crystals: 112–114 °C.

FT-IR (KBr): 3,343 cm⁻¹ (vNH); 1,759 and 1,726 cm⁻¹ (vOC=O); 1,657 cm⁻¹ Amide I; 1,534 cm⁻¹ Amide II.

¹H-NMR (250 MHz, DMSO-d₆): δ/ppm 9.69 (t, J = 6.7 Hz, 1H, NH); 8.01–7.27 (m, 9H, Ar); 5.60 (d, J = 6.7 Hz, 2H, N-CH₂-O); 2.29 (s, 3H, CH₃)

¹³C-NMR (63 MHz, DMSO-d₆): δ/ppm 168.9 (C=O); 167.2 (C=O); 164.8 (C=O); 65.9 (CH₂); 20.9 (CH₃); Ar: 154.5, 133.2, 132.1, 130.9, 128.6, 127.6, 127.1 and 122.4.

Anal. Calcd. (found) for C₁₇H₁₅NO₅: C, 65.17 (64.99); H, 4.82 (5.03); N, 4.47 (4.62).
References


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