

Short Note

## (Benzoylamino)methyl 4-Acetyloxybenzoate

Emil Popovski <sup>1,\*</sup> and Kristina Mladenovska <sup>2</sup>

<sup>1</sup> Institute of Chemistry, Faculty of Natural Sciences & Mathematics, Ss. Cyril and Methodius University, Arhimedova 5, PO Box 162, 1000 Skopje, Macedonia

<sup>2</sup> Department of Drug Design and Metabolism, Faculty of Pharmacy, Ss. Cyril and Methodius University, Vodnjanska 17, 1000 Skopje, Macedonia

\* Author to whom correspondence should be addressed; E-Mail: popovski.emil@gmail.com.

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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**).

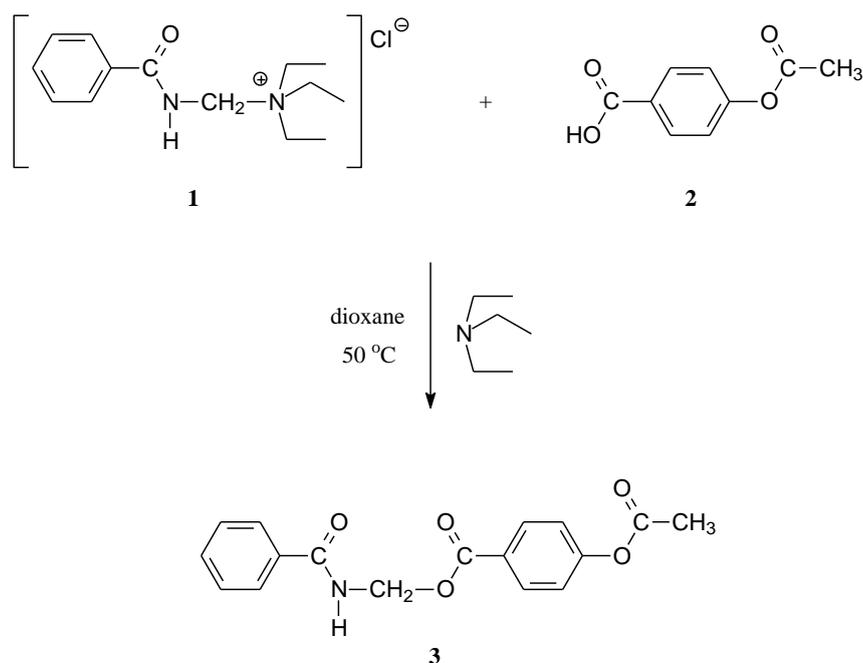
**Keywords:** 4-acetyloxybenzoic acid; benzamidomethylation

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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].

*(Benzoylamino)methyl 4-acetoxybenzoate (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<sup>-1</sup> (νNH); 1,759 and 1,726 cm<sup>-1</sup> (νOC=O); 1,657 cm<sup>-1</sup> Amide I; 1,534 cm<sup>-1</sup> Amide II.

<sup>1</sup>H-NMR (250 MHz, DMSO-*d*<sub>6</sub>): 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-CH<sub>2</sub>-O); 2.29 (s, 3H, CH<sub>3</sub>)

<sup>13</sup>C-NMR (63 MHz, DMSO-*d*<sub>6</sub>): δ/ppm 168.9 (C=O); 167.2 (C=O); 164.8 (C=O); 65.9 (CH<sub>2</sub>); 20.9 (CH<sub>3</sub>); Ar: 154.5, 133.2, 132.1, 130.9, 128.6, 127.6, 127.1 and 122.4.

Anal. Calcd. (found) for C<sub>17</sub>H<sub>15</sub>NO<sub>5</sub>: C, 65.17 (64.99); H, 4.82 (5.03); N, 4.47 (4.62).

## References

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