

Short Note

[(3-Chlorobenzamido)methyl]triethylammonium Chloride

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Abstract: We report the synthesis of [(3-chlorobenzamido)methyl]triethylammonium chloride in a reaction of *N*-(chloromethyl)-3-chlorobenzamide and triethylamine in dry acetone. The structure of the newly synthesized compound was characterized with ¹H-NMR, ¹³C-NMR, FTIR and Mass spectroscopy.

Keywords: benzamidomethylation; *N*-(chloromethyl)-3-chlorobenzamide

1. Introduction

(Benzamidomethyl)triethylammonium chloride is a versatile agent used for benzamidomethylation of phenols [1], thiols, amines, dithiocarbamates [2], hydrazines [3,4], hydrazides [5], *etc.* The benzamidomethylating reactions proceed fast at mild conditions in aqueous media, and the products are easily isolated from reaction mixture by simple filtration, with high yields. Carboxylic acids and alcohols can also be benzamidomethylated at mild conditions but in organic solvents [1,2].

In the course of our research, synthesis of new bioactive compounds with substituted-benzamidomethyl group, as a reagent, the title compound (**2**) was synthesized in a reaction of *N*-(chloromethyl)-3-chlorobenzamide and triethylamine in dry acetone.

Here, we present the synthesis and some physicochemical characteristics of **2**.

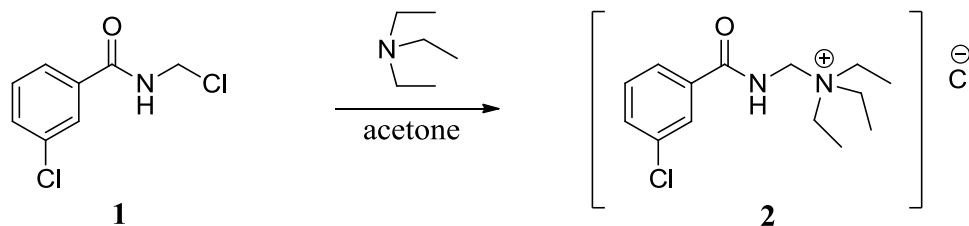


Figure 1. Reaction scheme of the title compound.

2. Experimental Section

2.1. Materials

Compound **1** was synthesized as *N*-(chloromethyl)benzamide in reference [1] and was used without further purification. All other reagents and solvents were obtained from commercial sources and used without further purification.

2.2. Instrumentation

The compound was structurally characterized by different spectroscopic techniques. Infrared spectra were measured on a Perkin-Elmer System 2000 FT-IR-ATR. The 1D (¹H, ¹³C {¹H}, DEPT) and 2D (COSY, HSQC, HMBC) NMR spectra were recorded on a Bruker Avance II+ 600 MHz BioSpin GmbH (Rheinstetten, Germany) in DMSO-*d*₆ as solvent and tetramethylsilane as an internal standard. The HR mass spectra were recorded on a MICROMASS Q-ToF Premier (ESI, pos, V-mode) (Waters, Milford, MA, USA). Melting points were determined on a Mel-Temp II[®] in open glass capillaries (Barnstead/ThermoLyne, Dubuque, IA, USA).

2.3. Synthesis of [(3-Chlorobenzamido)methyl]triethylammonium Chloride (**2**)

A solution of 2.85 g (0.014 mol) freshly synthesized **1** in 50 mL dry acetone was completely and at once added to a vigorously stirred solution of 5.85 mL (0.042 mol) triethylamine in 150 mL dry acetone. As a result of the reaction, a white precipitate occurred immediately. To this mixture, 50 mL of dry acetone was added, followed by stirring within 15 min. Colorless crystals were obtained, which were collected by filtration under pressure, washed with dry acetone and left to dry at a room temperature. The yield of the crude product was 78.70%. Purification was performed by recrystallization from acetone.

Recrystallization proceeded with difficulties and significant loss of **2**. For these reasons and to obtain better results, it is recommended **2** to be used without purification.

The melting point of the purified product was 195 °C and was followed by decomposition.

FT-IR-ATR: 3487.51 cm^{-1} and 3401.247 cm^{-1} $\nu(\text{N-H})$; 3136.06 cm^{-1} and 2982.7 cm^{-1} $\nu(\text{C-H})$; 1666.35 cm^{-1} (Amide I); 1548 cm^{-1} (Amide II).

$^1\text{H-NMR}$ (600.13 MHz, $\text{DMSO-}d_6$) δ (ppm): 1.26 (t, $J = 7.0$ Hz, CH_3); 3.23 (q, $J = 7.0$, CH_2CH_3) 4.74 (d, $J = 7.0$ Hz, 2H, CH_2), 7.56 (dd, $J = 8.0$, 8.0 Hz, 1H, ArH-5), 7.69 (d, $J = 8.0$ Hz, 1H, ArH-4), 8.01 (br d, $J = 8.0$ Hz, 1H, ArH-6), 8.12 (br s, 1H, ArH-2), 9.78 (t, $J = 7.0$ Hz, 1H, CO-NH).

$^{13}\text{C-NMR}$ (150.91 MHz, $\text{DMSO-}d_6$) δ (ppm): 7.5 CH_3 , 50.2 CH_2 , 59.7 NHCH_2N , 128.1 (ArC-2), 133.2 (ArC-3), 127.42 (ArC-6), 130.4 (ArC-5), 132.3 (ArC-4), 134.6 (ArC-1), 167.4 (C=O).

TOF MS (ESI, pos) m/z calculated for $\text{C}_{14}\text{H}_{22}\text{N}_2\text{OCl}^+$: 269.1421. Found: 269.1417.

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Author Contributions

P. B-S., Z. K., B. M. and G. D. performed the experimental work and product characterization. K. M. and E. P. prepared the manuscript and managed the overall project.

Conflicts of Interest

The authors declare no conflict of interest.

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