

Synthesis of N-(N'-benzoylhydrazinomethyl)benzamide

Emil Popovski

Institute of Chemistry, Faculty of Natural Sciences & Mathematics, Sts. Cyril and Methodius University, Arhimedova 5, PO Box 162, 1000 Skopje, Macedonia

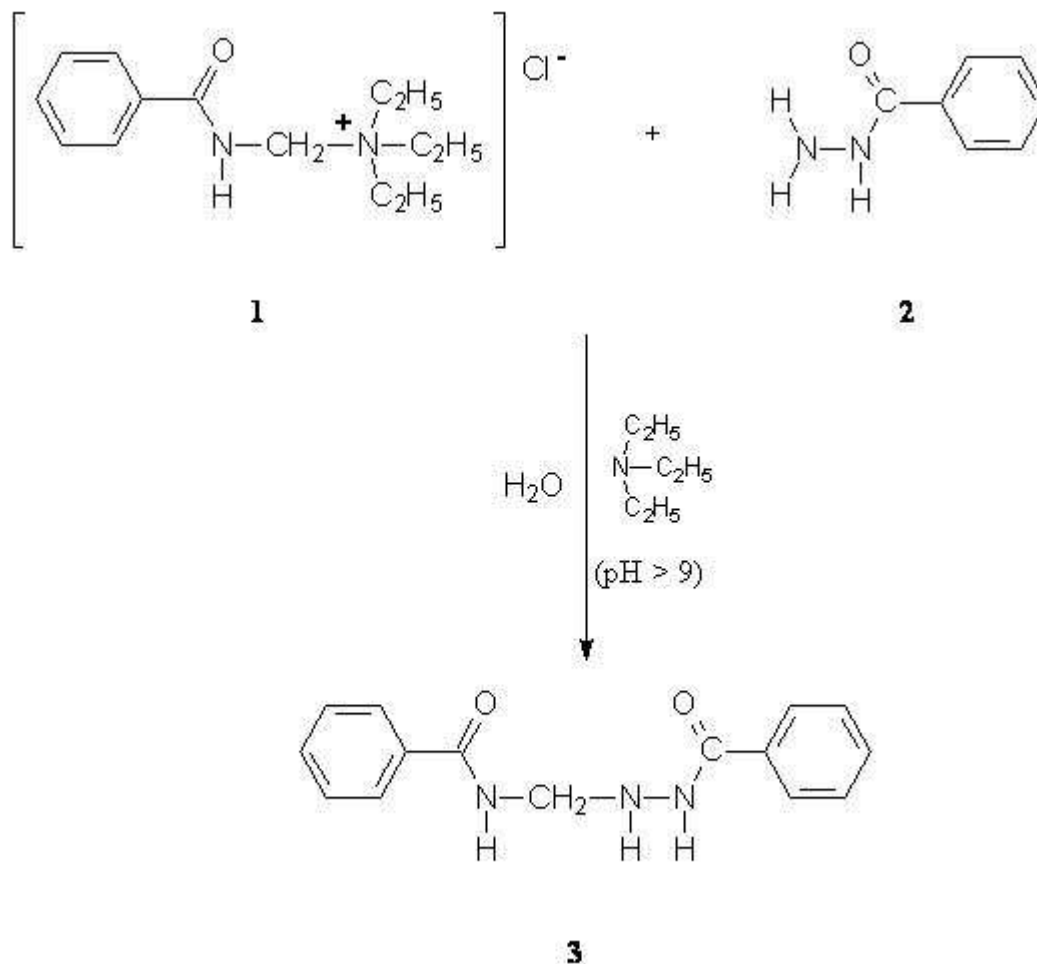
Tel: +389 (0)2 3249918; Fax: +389 (0)2 3226865; E-mail: emilp@iunona.pmf.ukim.edu.mk

Received: 9 January 2007 / Accepted: 21 January 2007 / Published: 31 May 2007

Keywords: Benzamidomethylation, aqueous media, hydrazide

Our previous research has shown that (benzamidomethyl)triethylammonium chloride (**1**) is an excellent benzamidomethylating agent for different types of compounds in aqueous media because of the mild reaction conditions, high yields and simple isolation of products [1-3].

In the course of this work, we have also carried out reaction of **1** with benzhydrazide (**2**) in aqueous media.



A solution of (benzamidomethyl)triethylammonium chloride (**1**) (0.273 g; 1.01 mmol) in water (10 mL) was added in small portions to a solution of benzhydrazide (**2**) (0.204 g; 1.50 mmol) in water (20 mL). In the same time, triethylamine (TEA) (0,2-0,4 mL; pH >9), drop by drop was added to the mixture. The mixture was stirred for 4-5 h at room temperature, afterward, 30 min. in ice bath. Colorless crystals were collected by filtration and purification was performed by recrystallization from dioxane.

Yield: 86 %

Melting Point: 144 °C (dioxane)

IR (KBr; cm^{-1}): $\nu(\text{N-H})$ 3329 and 3302; Amide I 1651 and 1638; Amide II 1543.

$^1\text{H-NMR}$ ($\text{DMSO-}d_6$; 250 MHz); δ/ppm = 10.03 (d, 1H, N-NHCO); 8.85 (t, 1H, CONH-C); 7.83-7.40 (m, 10H, Ar); 5.67 (q, 1H, C-NH-N) and 4.34 (t, 2H, CH_2).

$^{13}\text{C-NMR}$ ($\text{DMSO-}d_6$; 63 MHz); δ/ppm = 167.0 C=O; 165.6 C=O; 56.3 CH_2 , Ar: 134.6; 133.4; 131.3; 128.3; 127.3 and 127.2.

Elemental Analysis: Calculated (%) for $\text{C}_{15}\text{H}_{15}\text{N}_3\text{O}_2$: C 66.9, H 5.6, N 15.6. Found: C 66.7, H 5.7, N 15.3.

References and Notes:

1. Popovski, E.; Klisarova, L.; Vikić-Topić, D. Simple Method for Benzamidomethylation of Phenols in Water Solution. *Synth. Commun.* **1999**, *29*, 3451-3458.
2. Popovski, E.; Klisarova, L.; Vikić-Topić, D. Benzamidomethylation with (Benzamidomethyl)-triethylammonium Chloride. 2. A Simple Method for Benzamidomethylation of Thiols, Amines and Carboxylic acids. *Molecules* **2000**, *5*, 927-936.
3. Popovski, E.; Bogdanov, J.; Najdoski, M. and Hey-Hawkins, E. Reactions of (Benzamidomethyl)triethylammonium Chloride with Some Inorganic Nucleophiles in Aqueous Media. *Molecules* **2006**, *11*, 279-285.

© 2007 by MDPI (<http://www.mdpi.org/>). Reproduction is permitted for noncommercial purposes.