

Synthesis of N-[N'-(2-hydroxy-2,2-diphenylacetyl)hydrazinomethyl]benzamide

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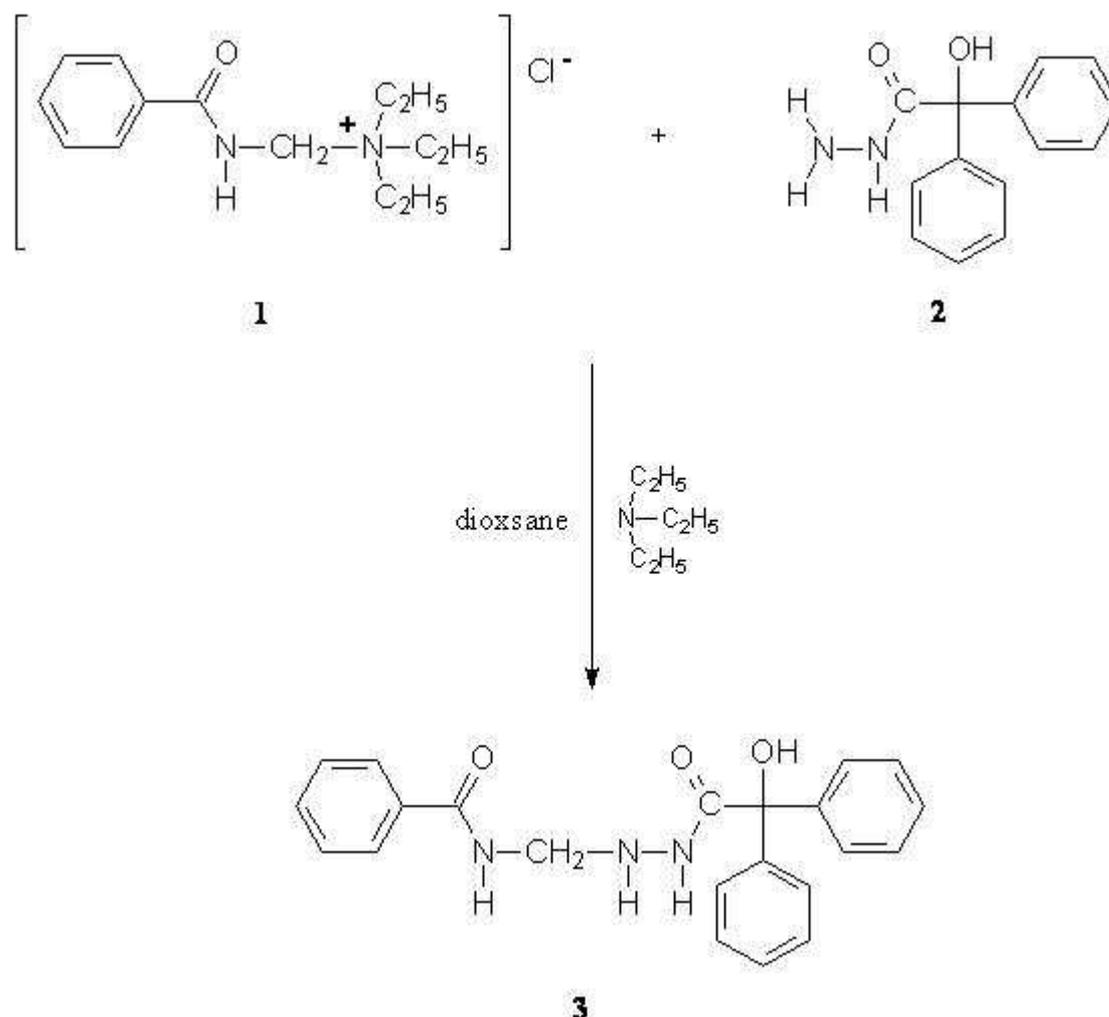
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Our previous research has shown that (benzamidomethyl)triethylammonium chloride (**1**) is a good benzamidomethylating agent for different types of compounds in non-aqueous media [1-2].

In the course of this work, we have also carried out reaction of **1** (in dioxane) with in water insoluble hydrazide of benzoic acid (**2**).



To a suspension of **1** (0.271 g, 1.00 mmol) in dioxane (15 mL) was added **2** (0.368 g, 1.52 mmol) and triethylamine (TEA) (0.2-0.4 mL). The mixture was stirred and heated at 50 °C for 1 h. After cooling water was added to the mixture until white precipitate appears. Colorless crystals were filtered and first purification was performed by dissolving the product in dioxane and precipitated with water.

Yield: 85 %

Melting Point: 188 °C (toluene)

IR (KBr; cm^{-1}): $\nu(\text{N-H})$ and $\nu(\text{O-H})$ 3421, 3313 and 3237; Amide I 1656 and 1636; Amide II 1514.

$^1\text{H-NMR}$ ($\text{DMSO-}d_6$; 400 MHz); δ/ppm = 9.68 (d, 1H, N-NHCO); 8.78 (t, 1H, CONH-C); 7.82-7.18 (m, 15H, Ar); 6.60 (s, 1H, OH); 5.65 (d(q), 1H, C-NH-N) and 4.31 (s(t), 2H, CH_2).

$^{13}\text{C-NMR}$ ($\text{DMSO-}d_6$; 100 MHz); δ/ppm = 170.61 C=O; 166.69 C=O; 80.01 C-OH; 56.3 CH_2 , Ar: 143.88; 134.15; 131.14; 128.13; 127.40; 127.30; 127.17 and 127.01.

Elemental Analysis: Calculated (%) for $\text{C}_{22}\text{H}_{21}\text{N}_3\text{O}_3$: C 70.4, H 5.6, N 11.2. Found: C 70.5, H 5.7, N 10.9.

References and Notes:

1. Popovski, E.; Klisarova, L.; Vikic-Topic, D. Simple Method for Benzamidomethylation of Phenols in Water Solution. *Synth. Commun.* **1999**, *29*, 3451-3458.
2. Popovski, E.; Klisarova, L.; Vikic-Topic, D. Benzamidomethylation with (Benzamidomethyl)-triethylammonium Chloride. 2. A Simple Method for Benzamidomethylation of Thiols, Amines and Carboxylic acids. *Molecules* **2000**, *5*, 927-936.

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