

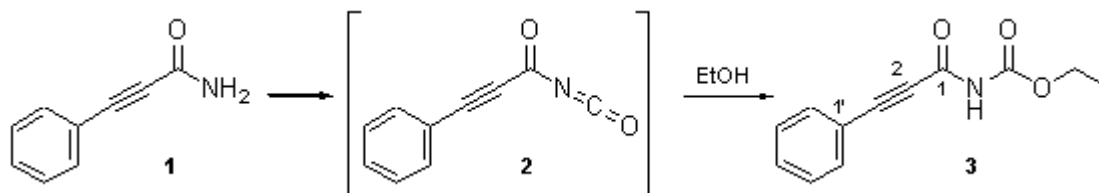
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## Ethyl *N*-(3-Phenyl-2-propynoyl)carbamate

Dieter Hubmann and Urs Séquin\*

Institut für Organische Chemie der Universität Basel, St. Johannis-Ring 19. CH-4056 Basel, Switzerland.  
Tel. +41 61 2671110, Fax +41 61 2671103, E-mail: [sequin@ubaclu.unibas.ch](mailto:sequin@ubaclu.unibas.ch)

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During the synthesis of a series of carboximides using acyl isocyanates [1], the title compound **3** was synthesised in a model reaction from 3-phenyl-2-propynoyl isocyanate (**2**) and ethanol. Isocyanate **2** was generated *in situ* from the corresponding carboxamide (**1**) with oxalyl chloride [2]. Compound **3** had been reported previously [3], but we give here a more detailed characterization.

To 3-phenyl-2-propynamide (**1** [3], 492 mg, 3.39 mmol) in dichloromethane (8 ml), oxalyl chloride (407 ml, 4.75 mmol) was added under argon at room temperature. The mixture was heated to reflux for 5 h and then allowed to cool to room temperature. Ethanol (1 ml, 177 mmol) was added and the mixture stirred for 10 min and the solvent removed *in vacuo*. The residue was chromatographed on SiO<sub>2</sub> (40 g, dichloromethane) to give 645 mg (89%) of **3** as a colorless solid.

Colorless prisms (ethanol), m.p. 98.5–100°C ([3]: 100°C).

IR (KBr): 3211; 3100; 2987; 2204; 1768; 1648; 1479; 1358; 1206; 1192; 1066; 769; 696.

<sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>): 7.96 (*s br*, 1H, NH); 7.63 (*d*, *J* = 6.8, 2H, H-C(2'), H-C(6')); 7.47 (*t*, *J* = 7.4, 1H, H-C(4')); 7.39 (*t*, *J* = 7.3, 2H, H-C(3'), H-C(5')); 4.29 (*q*, *J* = 7.1, 2H, CH<sub>2</sub>); 1.34 (*t*, *J* = 7.1, 3H, CH<sub>3</sub>).

<sup>13</sup>C-NMR (75 MHz, CDCl<sub>3</sub>): 151.9, 150.5 (2 C=O); 133.1 (C(2'), C(6')); 130.9 (C(4')); 128.6 (C(3'), C(5')); 119.6 (C(1')); 92.3 (C(3)); 81.8 (C(2)); 62.6 (CH<sub>2</sub>); 14.2 (CH<sub>3</sub>).

EI-MS (70 eV): 217 (6, *M*<sup>+</sup>); 189 (8); 171 (6); 145 (18); 129 (100); 118 (30); 101 (7); 89 (11); 75 (20); 51 (11).

CI-MS (NH<sub>3</sub>): 235 (10, [*M*+NH<sub>4</sub>]<sup>+</sup>); 218 (100, [*M*+H]<sup>+</sup>); 146 (8); 129 (16).

Anal. Calcd for C<sub>12</sub>H<sub>11</sub>NO<sub>3</sub> (217.22): C 66.35, H 5.10, N 6.45; Found: C 66.19, H 5.03, N 6.36.

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*Sample availability:* available from the authors and from MDPI. MDPI ID 17924.

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