During the synthesis of a series of carboximides using acyl isocyanates [1], the title compound 3 was synthesised as a model compound from benzoyl isocyanate (1) and lithium phenylacetylide (2).

To benzoyl isocyanate (342 ml, 2.72 mmol, Aldrich) in tetrahydrofuran (8 ml), a solution of lithium phenylacetylide in tetrahydrofuran (2.72 ml, 1.0 M, 2.72 mmol, Aldrich) was added under argon at -15°C, and the mixture stirred for 90 min at -15°C. Dilute hydrochloric acid (20 ml, 2 M) was added and the mixture was extracted three times with dichloromethane (20 ml each). The combined organic layers were dried (Na₂SO₄), filtered, and the solvent removed in vacuo. The residue was chromatographed on SiO₂ (60 g, dichloromethane) to give 414 mg (61%) of 3 as yellowish needles.

Yellowish needles, m.p. 110-112°C

IR (KBr): 3266; 3079; 2235; 2192; 1713; 1648; 1459; 1325; 1238; 761; 694.

¹H-NMR (300 MHz, CDCl₃): 10.02 (s br, 1H, NH); 7.97 (d, J = 7.1, 2H, H-C(2''), H-C(6'')); 7.63 - 7.30 (m, 8H, H-C(3''), H-C(4''), H-C(5''), H-C(2'), H-C(3'), H-C(4'), H-C(5'), H-C(6')).

¹³C-NMR (75 MHz, CDCl₃): 165.1 (s, C₆H₅-C=O); 153.3 (s, C(1)); 133.2 (d, C(4'')); 132.9 (d, C(2'), C(6')); 131.9 (s, C(1'')); 130.7 (d, C(4')); 128.6, 128.3, 127.9 (3d, C(2'), C(3''), C(5''), C(6'), C(3'), C(5')); 119.5 (s, C(1'')); 93.0 (s, C(3)); 82.9 (s, C(2)).

EI-MS (70 eV): 249 (5, M⁺); 221 (8); 178 (18); 129 (16, [C₆H₅C=CC=O]⁺); 118 (100); 105 (40, [C₆H₅CO]⁺); 90 (39); 77 (21, [C₆H₅⁺]); 63 (6); 51 (11).

CI-MS (NH₃): 250 (100, [M+H]⁺); 118 (10).

Anal. Calcd for C₁₆H₁₁NO₂ (249.27): C 77.10, H 4.45, N 5.62, O 12.84; Found: C 76.92, H 4.60, N 5.59, O 12.74.

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References


Sample availability: available from the authors and from MDPI. MDPI ID 17925.