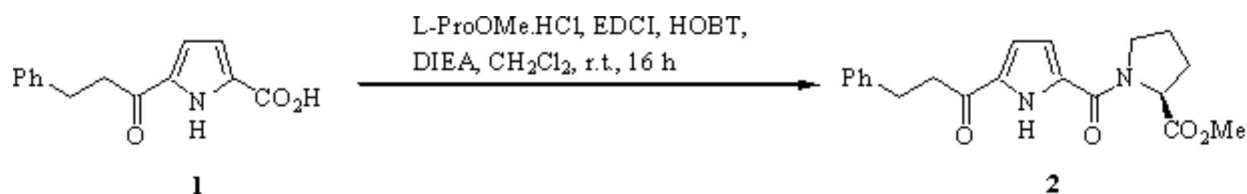


N-[5-(3-Phenylpropionyl)-1H-pyrrole-2-carbonyl]-L-proline Methyl Ester

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Received: 22 February 2003 / Accepted 22 May 2003 / Published 1 June 2003



To a stirring solution of the pyrrole carboxylic acid **1** [1] (100 mg, 0.41 mmol, 1 equiv) and L-proline methyl ester hydrochloride (75 mg, 0.45 mmol, 1.1 equiv) in dry dichloromethane (6 mL) at r.t. under an inert atmosphere were added 1-(3-dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride (102 mg, 0.53 mmol, 1.3 equiv) and 1-hydroxybenzotriazole hydrate (83 mg, 0.62 mmol, 1.5 equiv) [2]. *N,N*-Diisopropylethylamine (Hünig's base, 58 mg, 0.45 mmol, 1.1 equiv) was added, and the reaction mixture was stirred at r.t. for 16 h. The solution was then diluted with dichloromethane (10 mL), washed with 3 M hydrochloric acid (2 x 10 mL), water (2 x 10 mL), and the combined aqueous washings were back-extracted with dichloromethane (2 x 10 mL). The combined organic fractions were dried (MgSO₄) and the solvent was removed by evaporation under reduced pressure. Flash chromatography on silica (ethyl acetate/petroleum ether, 2:1) afforded the title compound **2** (100 mg, 69%) as a tan solid.

mp 84-86°C.

IR (KBr, diffuse refraction method) 1539.1, 1598.9, 1660.6, 1737.7, 2954.7, 3028.0, 3435.0.

¹H NMR (CDCl₃, 500 MHz) δ 2.03-2.27 (m, 4H, NCH₂CH₂CH₂CH), 3.03 (m, 2H, PhCH₂CH₂), 3.11 (m, 2H, PhCH₂CH₂), 3.75 (s, 3H, CO₂CH₃), 3.84 (m, 1H, NCH_{2a}CH₂CH₂CH), 3.96 (m, 1H, NCH_{2b}CH₂CH₂CH), 4.68 (q, 1H, J = 4.1 Hz, NCH₂CH₂CH₂CH), 6.61 (m, 1H, pyrrole H3), 6.82 (m, 1H, pyrrole H4), 7.17-7.29 (m, 5H, ArH), 10.10 (s (br), 1H, pyrrole NH).

¹³C NMR (CDCl₃, 75 MHz) δ 25.3, 28.6 (NCH₂CH₂CH₂CH), 30.3 (PhCH₂CH₂), 40.1 (PhCH₂CH₂), 48.4 (NCH₂CH₂CH₂CH), 52.4 (CO₂CH₃), 60.2 (NCH₂CH₂CH₂CH), 112.9 (pyrrole C3), 115.4 (pyrrole C4), 126.2, 128.3, 128.5, 140.9 (ArC), 129.5 (pyrrole C2), 132.5 (pyrrole C5), 159.6 (CON), 172.5 (CO₂), 189.9 (CH₂CO).

HRMS (M⁺) Calcd for C₂₀H₂₂N₂O₄: 354.1580. Found: 354.1583.

Anal. Calcd for C₂₀H₂₂N₂O₄: C, 67.78; H, 6.26; N, 7.90. Found: C, 67.75; H, 6.51; N, 7.97.

References

- Martyn, D. C.; Abell, A. D. *J. Chem. Soc., Org. Biomol. Chem.*, in press.
- Tian, Z-Q.; Brown, B. B.; Mack, D. P.; Hutton, C. A.; Bartlett, P. A. *J. Org. Chem.* **1997**, *62*, 514.

Sample availability: available from the authors.

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