

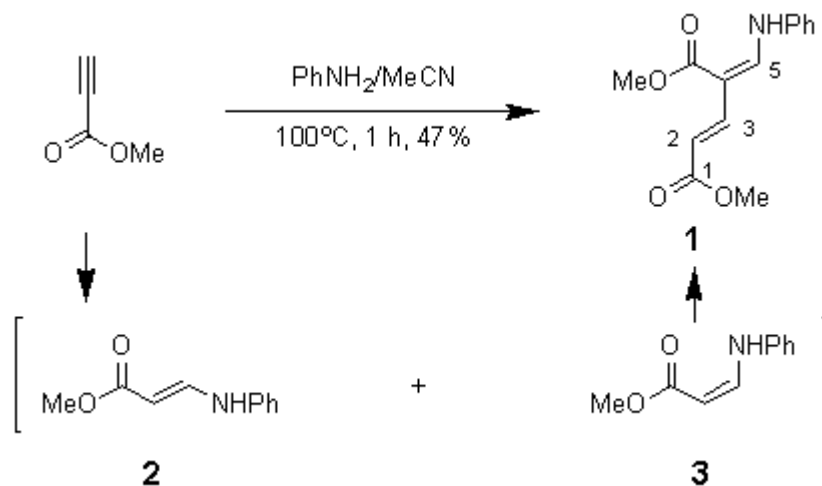
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Methyl (2*E*,4*E*)-5-Anilino-4-(methoxycarbonyl)penta-2,4-dienoate

Jin-Cong Zhuo

College of Pharmacy, The Ohio State University, 500 West 12th Avenue, Columbus, OH 43210, USA.
Fax: (614) 292 2435; E-mail: zhuo.1@osu.edu

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Method A: A mixture of methyl propiolate (0.42 g, 5 mmol) and aniline (0.47 g, 5 mmol) in MeCN (3 ml) was heated at 100°C in a sealed-tube for 1 h. After cooling, the volatiles were evaporated under reduced pressure. The residue was treated with Et₂O (5 ml) and filtered. The solid is compound **2** (0.4 g). The filtrate was concentrated to give compound **1** (0.31 g, 47%).

Method B: A solution of methyl (Z)-3-anilinoacrylate (**3**, 0.5 g) [1] in MeCN (2 ml) was heated under reflux for 5 h (or at r.t. for 15 d). The volatiles were evaporated under reduced pressure to give the compound **1** as a solid (0.36 g, 97.8%).

Data for compound **1**:

M.p. 116.4–117.7 °C (lit. [2]:118.9°C).

¹H-NMR(CDCl₃): 10.75 (d br, J = 13.2, 1H, NH), 7.76 (d, J = 13.2, 1H, H-5), 7.48 (d, J = 16.0, 1H, H-3), 7.34–7.40 (m, 2H, Ph), 7.11–7.16 (m, 1H, Ph), 7.06–7.10 (m, 2H, Ph), 6.19 (d, J = 16.0, 1H, H-2), 3.86 (s, 3H, OMe), 3.76 (s, 3H, OMe).

¹³C-NMR(CDCl₃): 169.4 (C, COOMe), 168.8 (C, COOMe), 148.0 (CH, C-5), 142.4 (CH, C-3), 139.3 (C, Ph), 129.9 (2CH, Ph), 124.5 (CH, Ph), 116.7 (2CH, Ph), 110.7 (CH, C-2), 98.4 (C-4), 51.4 (OMe), 51.3 (OMe).

IR (cm⁻¹, KBr): 2300–3200 br, 1694_{vs}, 1655_{vs}, 1629_{vs}, 1615_{vs}, 1597_{vs}, 1590_{vs}, 1439_{vs}, 1371_{vs}, 1309_{vs}, 1261_{vs}, 1223_{vs}, 994_s, 793_s, 749_{vs}.

CI-MS (NH₃): 262 (M+H⁺, 6), 261 (M⁺, 1), 115 (3), 106 (2), 98 (6), 94 (100), 93 (34), 77 (49), 73 (38).

References

1. Huisgen, R.; Herbig, K.; Siegl, A.; Huber, H. *Chem. Ber.* **1966**, *99*, 2526.

2. Bottomley, W. *Tetrahedron Lett.* **1967**, 8, 1997.

Sample availability: available from MDPI. MDPI ID 13337.

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