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Esterification and hydrolysis under microwave irradiation

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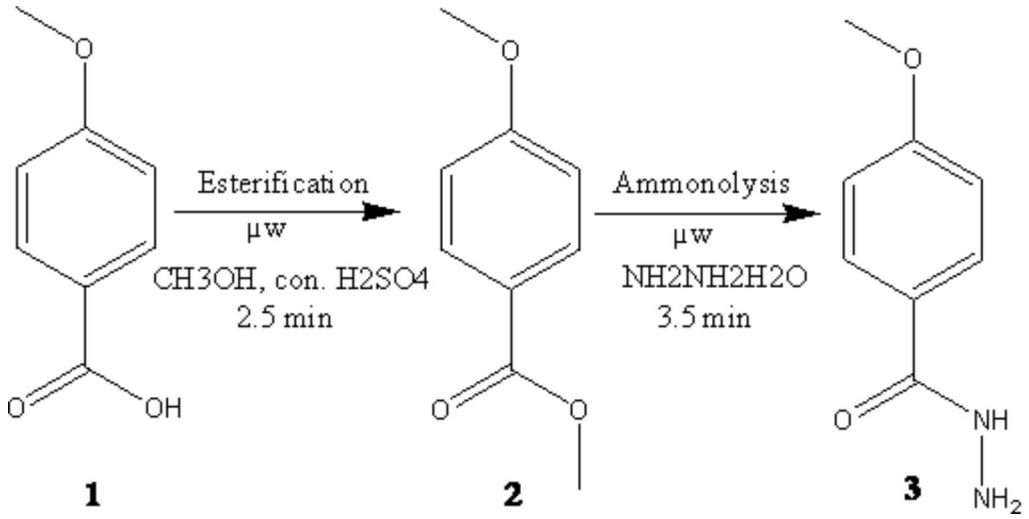

p-methoxy benzoicacid **1** (1.67 g, 0.01 mole) and catalytic amount of conc. H_2SO_4 (2-3 drops) in absolute methanol (30 mL) was taken in RBF placed in a microwave oven and irradiated (400w, 61-62°C) for 2.5 min [1]. Upon completion of reaction (monitored by TLC), using petroleumether-ethylacetate (8:2) as the eluent solvent system. The reaction mixtures was allowed to attain room temperature and treated with cold water. The solid separated was filtered, washed with water and recrystallised from ethanol to furnish compound **2**, yield 90%.
Methyl ester of p-methoxy benzoicacid **2** (1.66 g, 0.01 mole) and hydrazine hydrate (0.9 mL, 0.01 mole) in ethanol (20 mL) was taken in RBF placed in a microwave oven and irradiated (450w, 76-78°C) for 3.5 min [1]. After completion of reaction (monitored by TLC), using petroleumether-ethylacetate (8:2) as the eluent solvent system. The mixture was cooled and the resulting solid was filtered, dried and recrystallized from ethanol to get compound **3**, yield 83%.

Melting point: 88 ^{oC} **2** and 136 ^{oC} **3**.

IR (KBr) (cm^{-1}): **2** 1722 (>C=O of ester), 1225, 1044 (C-O-C), 3023 (C-H, aromatic ring), 1510 (C=C, aromatic ring), 2825 (-OCH_3). **3** 1661 (>C=O of amide), 3355, 3382 (-$NHNH_2$), 2837 (-OCH_3), 3028, 1522 (C-H, C=C, aromatic ring).

^{1}H-NMR ($CDCl_3$-DMSO-d_6) (400 MHz) δ (ppm): **2** 6.70-7.90 (4H, m, Ar-H), 4.0 (3H, s, -$COOCH_3$), 3.87 (3H, s, -OCH_3). **3** 6.85-7.96 (4H, m, Ar-H), 4.41 (2H, s, -NH_2), 7.85 (1H, s, -CONH-), 3.89 (3H, s, -OCH_3).

^{13}C-NMR ($CDCl_3$-DMSO-d_6) (62.90 MHz) δ (ppm): **2** 56.60 (-OCH_3), 115.29-134.1 (aromatic carbons), 170 (>C=O of ester), 20 (-$COOCH_3$). **3** 57.68 (-OCH_3), 117.31-130.0 (aromatic carbons), 162 (>C=O of amide).

MS (m/z): **2** 166 (M^+) ($C_9H_{10}O_3^+$), 135 ($C_8H_7O_2^+$), 107 ($C_7H_7O^+$), 59 ($C_2H_3O_2^+$), 31 (CH_3O^+). **3** 166 (M^+) ($C_8H_{10}O_2N_2^+$), 135 ($C_8H_7O_2^+$), 107 ($C_7H_7O^+$), 31 ($N_2H_3^+$), 16 (NH_2^+).

Elemental Analysis: Calculated for **2** $C_9H_{10}O_3$: C 65.06, H 6.02. Found: C 65.10, H 6.00.
Elemental Analysis: Calculated for **3** $C_8H_{10}N_2O_2$: C 72.18, H 6.02, N 16.86. Found: C 72.20, H 6.04, N, 16.89.

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References
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Sample Availability: Available from MDPI.