

Short Note

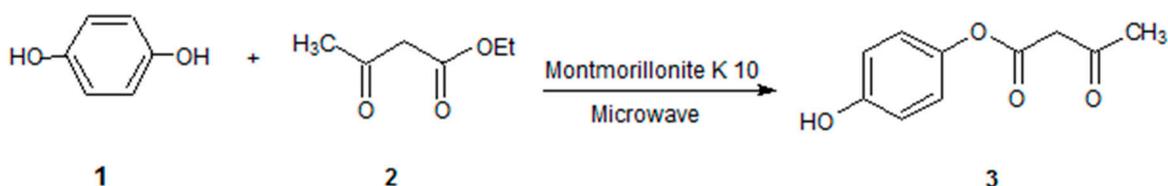
Microwave Synthesis of (4-hydroxy Phenyl) 3-oxobutanoate

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The product **3** was previously prepared [1] using C₆H₆ in the presence of pyridine. A mixture of hydroquinone **1** (0.66 g, 6 mmol), ethyl acetoacetate **2** (0.78 g, 6 mmol) and monmorillonite K10 (0.43 g, 30 % by weight of the total reactants) [2,3] was placed in a pyrex tube which was then introduced into a Maxidigest MX 350 Prolabo microwave monomode reactor fitted with a rotational system [4]. An approximate final temperature (120 °C) was measured by introducing a digital thermometer at the end of the irradiation time (20 min on 180 W as irradiation power). The mixture was cooled to ambient temperature. After elution with ethyl acetate (30 mL) and subsequent filtration through florisil, the organic product was purified by chromatography on silicagel (dichloromethane : ethyl acetate, 90 :10), yield : 65 % of **3** white solid.

Melting point: 96-98 °C.

¹H NMR δ (CDCl₃, 200 MHz): 7-6.7 (2d, 4H, Ph); 5,65 (s, OH) ; 3.7 (s, CH₂); 2.38 (s, CH₃).

¹³C NMR δ (CDCl₃, 100 MHz): 30 (CH₃); 50 (CH₂); 116 and 123 (C=C arom.); 155 (CO₂); 202 (C=O acyl).

MS (IC-NH₃, m / z): 212(M⁺ + 18) / 100 %.

References

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