Molbank **2008**, M561 http://www.mdpi.org/molbank/

Microwave Synthesis of (4-hydroxy Phenyl) 3-oxo butanoate

Mohammed Ramdani *, Nour-Eddine Benchat, Abderrahmane Anaflous, Fouad El Kalai

Applied Chemistry and Environement Laboratory, Faculty of Sciences, Univerity Mohamed 1st, Oujda, Morooco

* Author to whom correspondence should be addressed. Fax : +212 36 50 06 03;
E-Mail: moharamdani2000@yahoo.fr

Received: 2 May 2007; in revised form: 20 November 2007 / Accepted: 15 January 2008 / Published: 25 March 2008

Scheme 1

The product **3** was previously prepared [1] using C_6H_6 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.

^{1}H NMR d ($CDCl_3$, 200 MHz): 7-6.7 (2d, 4H, Ph); 5,65 (s, OH) ; 3.7 (s, CH_2); 2.38 (s, CH_3).

^{13}C NMR d ($CDCl_3$, 100 MHz): 30 (CH_3); 50 (CH_2); 116 and 123 (C=C arom.); 155 (CO_2); 202 (C=O acyl).

MS (IC-NH_3, m / z): 212(M^+ + 18) / 100 %.

References

1. Rall, K.B.; Perekalin, V.V. *Zhurnal Obshchei Khimii* **1955**, *25*, 259.
2. Li, T.S.; Zhang, Z.H.; Yang, F.; Fu, C.G. *J. Chem. Research (S)* **1998**, 38-39.
3. Singh, J.; Kaur, J.; Nayyar, S.; Kad, G.L. *J. Chem. Research (S)* **1998**, 280-281.
4. (a) Kappe, C.O.; Dallinger, D. *Nature Reviews Drug Discovery* **2006**, *5*, 51; (b) De la Hoz, A.; Diaz-Ortiz, A.; Moreno, A. *Chem. Soc. Rev.*, **2005**, *34*, 164.