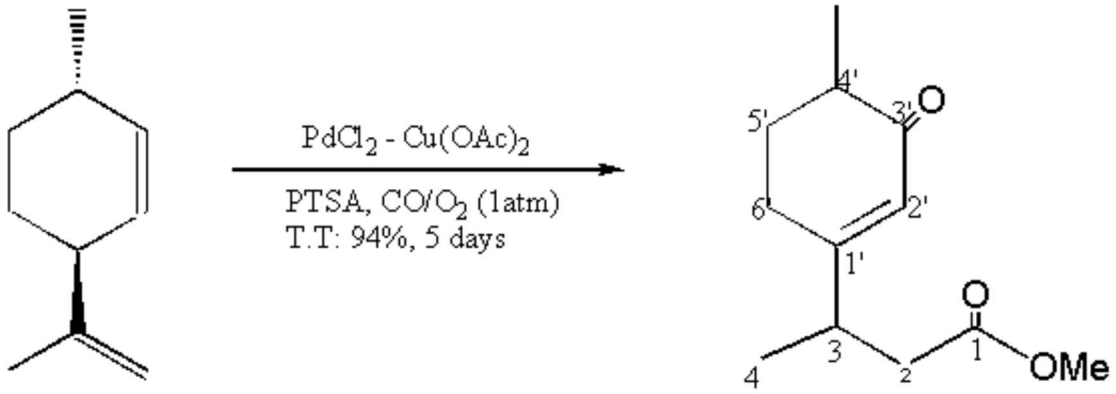
Molecules **2001**, *6*, M267

3-(4'-Methyl-3'-oxocyclohex-1'-enyl)butyric Acid Methyl Ester

Soufiane El Houssame*, Larbi El Firdoussi and Abdellah Karim

Laboratoire de Chimie de Coordination, Faculté des Sciences Semlalia, BP 2390 Marrakech Morocco.
E-mail: soufiane@ucam.ac.ma

Received: 14 June 2001 / Accepted: 15 December 2001 / Published: 20 December 2001



1 **2** 46%

The alkoxycarbonylation of terpenic olefins which affords the corresponding esters is an elegant synthesis [1-2]. Morover, the metal-complex-catalyzed hydrocarboxylation is among the most extensively investigated processes in homogeneous catalysis[3].

In a flask fitted with a reflux condenser was placed $PdCl_2$ (34 mg, 0.19 mmol), $Cu(OAc)_2$ (1.1 g, 5.5 mmol) and p-Toluenesulfonic acid (PTSA) (69.6 mg, 0.36 mmol), and the atmosphere was replaced with CO/O_2 (1:1). 15 ml of methanol were added and the mixture was stirred for 15 min at room temperature under atmospheric pressure of CO/O_2. 3-Isopropenyl-6-methyl-cyclohexene, **1**, (3.6 mmol) prepared by pyrolysis of 2-carene at high temperature (>220 °C) was added. The resulting mixture was further stirred at room temperature and at CO/O_2 was bubbled in for 5 days. The reaction was monitored by GC (conversion = 94%).

At the end of the reaction, the solvent was removed by rotary evaporation. The residue was purified by using silica gel chromatography column with Hexane/AcOEt (96:4) to afford **2** as colorless oil (46%).

^{1}H-NMR (300 MHz, $CDCl_3$): 5.72 (1H, dd, J_1=3.26 Hz/J_2=1.31Hz); 3.6 (3H, S, O-CH_3); 1.07 (3H, d, J=7.18 Hz, CH_3); 1.01(3H, d, J=6.53 Hz, CH_3).

^{13}C-NMR (100 MHz, $CDCl_3$): 202.2 (C=O); 172.1 (C=O); 167.4 (=C); 124 (=CH); 51.6 (OCH_3); 40.9 (-CH); 39.3 (-CH_2); 37.5 (-CH); 30.7 (-CH_2); 27.1 (CH_2); 19 (CH_3); 14.9 (CH_3).

M.S: m/z 210 (M^+); 151 (59); 109 (42).

References

1. Naigre, R.; Chenal, T.; Ciprès, I.; Kalck, P.; Daran, J.C.; Vaissermann, J. *J. Org. Chem.* **1994**, *480*, 91-102.
2. Chenal, T.; Ciprès, I.; Jenck, J.; Kalck, P.; Peres, Y. *J. Mol .Catal.* **1993**, *78*, 351-366.
3. Tkatchenko, I. *Comprehensive Organometallic Chemistry*, Vol. 8, Pergamon, Oxford, (1982),

101-223.

Sample Availability: Available from the authors and from MDPI.