Molecules 2001, 6, M268

(4-Isopropenyl-cyclohex-1-enyl)acetic Acid Ethylester

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

The carbonylation of allylic compounds catalyzed by transition metal complexes under atmospheric pressure of CO is one of the most attractive tools to synthesize the b,g - unsaturated carbonyl compounds, which are versatile building blocks [1-2].

Pd(OAc)$_2$ (7.5 mg, 0.033 mmol), anhydrous K$_2$CO$_3$ (135 mg, 9.7 mmol) and a stirring bar were placed in a three necked flask. The atmosphere was replaced with carbon monoxide and 1-Chloromethyl-4-isopropenyl-cyclohexene, 1, [3] (3.3 mmol) in 5 ml of ethanol was added under CO. The reaction mixture was stirred at 25 °C for 1.5 hours. The reaction was followed by GC. At the end of the reaction, the mixture was filtered and the solvent was removed under vacuum. The residue was chromatographed on silica gel with Hexane / Ethyl acetate as eluent to provide the carbonylated product 2 [4] (Conversion: 100 %, Yield: 96 %).

$[\alpha]_D^{20} = -43.1^\circ$ (c = 2.1; MeOH).

$^1$H-NMR (300 MHz, CDCl$_3$): 5.52 (1H, m, =CH); 4.66 (2H, m, =CH$_2$); 4.1 (2H, q (J=7.1 Hz), OCH$_2$CH$_3$); 2.9 (2H, S, -CH$_2$CO); 1.7 (3H, S, CH$_3$); 1.2 (3H, t (J=7.1 Hz), OCH$_2$CH$_3$).

$^{13}$C-NMR (100 MHz, CDCl$_3$): 172 (C=O); 149.8 (=C); 130.9 (=C); 125.1 (=CH); 108.7 (=CH$_2$); 60.5 (OCH$_2$CH$_3$); 43.2 (-CH$_2$); 40.7 (-CH); 30.8 (-CH$_2$); 28.9 (-CH$_2$); 27.7 (-CH$_2$); 20.8 (OCH$_2$CH$_3$); 14.3 (CH$_3$).

References


Sample Availability: Available from the authors and from MDPI.

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