

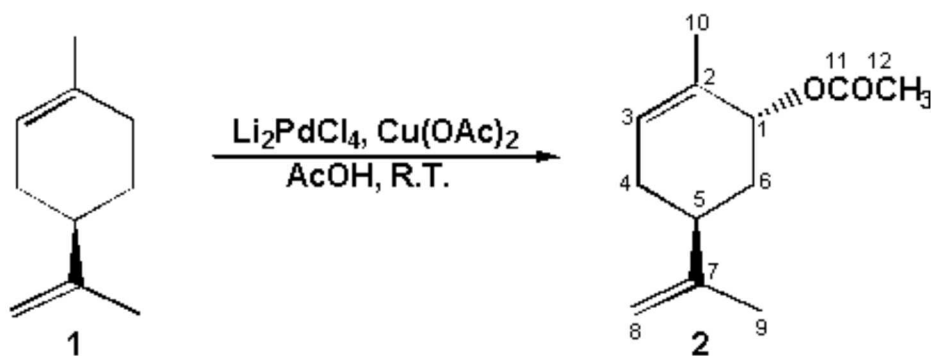
Molecules **2001**, *6*, M240

Trans Acetic Acid 5-isopropyl-2-methyl Cyclohex-2-enyl Ester (Trans Carvyl Acetate)

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Received: 10 January 2001 / Accepted: 15 May 2001 / Published: 25 May 2001



To a red solution of Li_2PdCl_4 , prepared in situ by mixing LiCl (30.8 mg, 0.72 mmol) and PdCl_2 (43 mg, 0.24 mmol) in 20 mL of AcOH stirred at 85°C , was added $\text{Cu}(\text{OAc})_2$ (4 g, 20 mmol) and stirred for 30 min. Then the reaction mixture was cooled to room temperature and the limonene **1** (1 g, 7.3 mmol) was added [1]. the evolution of the reaction was followed by GC. Once the reaction was finished, a mixture of hexane/ether (1:1, 30mL) was added and the solution was stirred for 30 min. the organic phase was separated and the remaining acetic acid phase was diluted with saturated NaCl (10mL) and extracted with hexane/ether (1:1, 3x30 mL). The combined extracts were neutralized with a saturated solution of NaHCO_3 , dried over MgSO_4 . Removal of the solvent under reduced pressure and purification of the residue by flash chromatography, using hexane/ether (9:1) as eluent, gave the compound **2** (86%) as a colorless oil [2, 3].

IR (cm^{-1}) 3080(C-H, sp^2) 1740(C=O) 1640(C=C) 1240(C-O).

^1H NMR (400 MHz, CDCl_3) 1.63(1H, ddd, $J_{6\text{ax}}, 5$ 13, $J_{6\text{ax}}, 6\text{eq}}$ 11.2, $J_{6\text{ax}}, 1$ 4.2Hz, 6ax-H) 1.68(3H, s, 9-CH₃) 1.72(3H, s, 10-CH₃) 1.83(1H, m, 4ax-H) 1.95(1H, ddd, $J_{6\text{eq}}, 6\text{ax}}$ 11.2, $J_{6\text{eq}}, 1$ 2.6 and $J_{6\text{eq}}, 5$ 4.2Hz, 6eq-H) 2.07(3H, s, 12-CH₃) 2.21(1H, m, 4eq-H) 2.32(1H, m, $J_5, 6\text{ax}$ 13, $J_5, 6\text{eq}$ 4.2, $J_5, 4\text{ax}$ 11.2 and $J_5, 4\text{eq}$ 4.2Hz, 5-CH) 4.72(2H, d, J 19 Hz, 8-CH₂) 5.26(1H, dd, $J_1, 6\text{ax}$ 4.2 and $J_1, 6\text{eq}$ 2.6Hz, 1-CH) 5.73(1H, m, 3-CH).

^{13}C NMR (100 MHz, CDCl_3) 20.6 and 20.8(CH₃, C-9 and C-10) 21.3(CH₃, C-12) 30.9 and 33.7(CH₂, C-4 and C-6) 70.6(CH, C-1), 109.2(CH₂, C-8) 127.8(CH, C-3) 130.9(C-7) 148.6(C-2) 170.7(C-11).

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Sample Availability: Available from the authors and from MDPI.

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