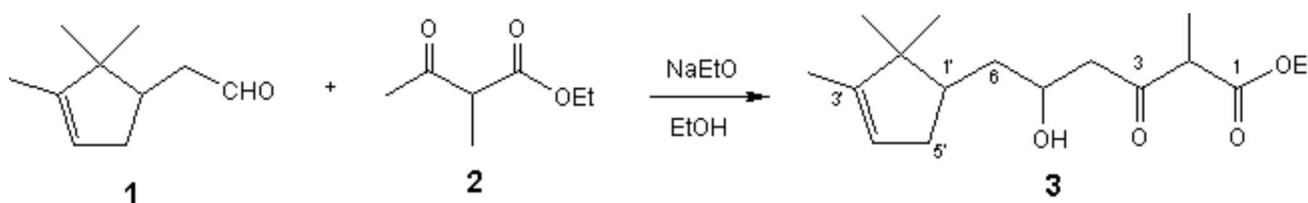


**5-Hydroxy-2-methyl-3-oxo-6-(2,2,3-trimethyl-cyclopent-3-enyl)-hexanoic acid ethyl ester****Mercedes Pérez-Bonilla, Juan M. Castro, Pablo J. Linares-Palomino, Sofía Salido, Joaquín Altarejos,\* Manuel Nogueras and Adolfo Sánchez**

Departamento de Química Inorgánica y Orgánica, Facultad de Ciencias Experimentales, Universidad de Jaén, 23071 Jaén, Spain.

Tel.: 34-953-002743, Fax: 34-953-012141, e-mail: [jaltare@ujaen.es](mailto:jaltare@ujaen.es)*Received: 16 February 2004 / Accepted: 6 July 2004 / Published: 1 July 2005***Keywords:** *a*-Campholenic aldehyde, ethyl 2-methylacetoacetate, aldol reaction.

Sodium (241 mg, 10.48 mmol) was added to absolute ethanol (40 mL) and the mixture refluxed until sodium reacted completely. Then ethyl 2-methylacetoacetate (**2**) (1.2 mL, 8.25 mmol) was added to the solution. The mixture was refluxed for 1 h and, then, after reaching room temperature, aldehyde **1** (1.26 g, 8.27 mmol) was added and the mixture refluxed again for 2 h. Finally, ethanol was partially evaporated under reduced pressure and the residue neutralized with aqueous 2N HCl and extracted with EtOAc (3×15 mL). The combined organic layers were washed with 2N HCl (10 mL), saturated NaHCO<sub>3</sub> (2×15 mL) and brine (3×10 mL). The organic phase was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and the solvent evaporated under reduced pressure to yield a residue (1.65 g) which was purified by flash chromatography on silica gel, using a 6.5:4.5 hexane/EtOAc mixture as eluent, to give the title compound **3** (356 mg, 1.20 mmol, 15%).

Melting point: 131.0–132.8 °C (white dust, from hexane/ethyl acetate).

IR (neat, cm<sup>-1</sup>): 3450–2600, 1101 (OH); 3036 (C=C); 1610 (CO); 1610, 1246, 1153 (COOEt).

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ= 0.79 (3H, *s*, Me-2'); 1.01 (3H, *s*, Me'-2'); 1.21 (3H, *t*, J=7.0 Hz, CH<sub>3</sub>CH<sub>2</sub>O), 1.36 (3H, *d*, J=6.6 Hz, Me-2); 1.62 (3H, *br s*, Me-3'); 1.70–2.87 (7H, *m*, H-4, H-6, H-1', H-5'); 3.48 (2H, *q*, J=7.0 Hz, CH<sub>3</sub>CH<sub>2</sub>O); 3.61 (1H, *q*, J=6.6 Hz, H-2); 4.69–4.81 (1H, *m*, H-5); 5.24 (1H, *br s*, H-4').

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ= 169.92 (C-1); 51.68 (C-2); 201.52 (C-3); 43.98 (C-4); 73.19 (C-5); 35.25\* (C-6); 7.71 (Me-2); 15.22 (CH<sub>3</sub>CH<sub>2</sub>O); 65.81 (CH<sub>3</sub>CH<sub>2</sub>O); 44.91 (C-1'); 46.79 (C-2'); 148.67 (C-3'); 121.00 (C-4'); 35.17\* (C-5'); 19.73 (Me-2'); 25.46 (Me'-2'); 12.54 (Me-3').

\*These signals may be interchanged.

EI-MS (70 eV, *m/z*): 279 (M<sup>+</sup>–OH, 0.1%); 267 (M<sup>+</sup>–Et, 0.1); 251 (M<sup>+</sup>–OEt, 0.8); 235 (0.7); 217 (0.9); 205 (0.4); 193 (1); 171 (2); 161 (9); 141 (9); 133 (11); 119 (20); 108 (C<sub>8</sub>H<sub>12</sub><sup>+</sup>, 100); 93 (29); 79 (12); 43 (34).

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