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(*E*)-6-(2,2,3-Trimethyl-cyclopent-3-enyl)-hex-4-en-3-one

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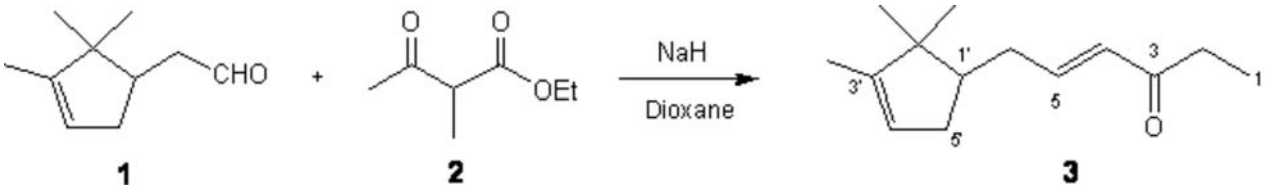

1 **2** **3**

Ethyl 2-methylacetoacetate (**2**) (727 mg, 4.53 mmol) was added to a stirred solution of NaH (184 mg, 4.60 mmol) in dioxane (25 mL). Then campholenic aldehyde (**1**) (707 mg, 3.72 mmol) was added and the mixture refluxed for 15 h. Then 2N HCl (15 mL) was added and the mixture extracted with Et_2O (3×15 mL). The combined organic layers were washed with 2N HCl (2×15 mL), saturated Na_2CO_3 (2×15 mL) and brine (3×15 mL). The organic phase was dried over anhydrous Na_2SO_4 and the solvent evaporated under reduced pressure to yield a residue (900 mg) which was purified by distillation under reduced pressure to give the title compound **3** (505 mg, 2.63 mmol, 58%).

IR (neat, n, cm^{-1}): 1700, 1676 (CO), 3036, 1631, 984 (C=C).

^{1}H NMR (300 MHz, $CDCl_3$, d, ppm): 0.81 (3H, *s*, Me-2'), 1.00 (3H, *s*, Me'-2'), 1.10 (3H, *t*, J=7.4 Hz, H-1), 1.61 (3H, *br s*, Me-3'), 1.77–2.42 (5H, *m*, H-5', H-1', H-6), 2.57 (2H, *q*, J=7.4 Hz, H-2), 5.22 (1H, *br s*, H-4'), 6.14 (1H, *dt*, J=15.8 Hz, 1.4 Hz, H-4), 6.85 (1H, *dt*, J=15.8 Hz, 7.3 Hz, H-5).

^{13}C NMR (75 MHz, $CDCl_3$, d, ppm): 8.13 (C-1), 33.46* (C-2), 201.04 (C-3), 130.50 (C-4), 146.89 (C-5), 33.14* (C-6), 49.27 (C-1'), 46.90 (C-2'), 148.30 (C-3'), 121.45 (C-4'), 35.42 (C-5'), 19.72 (Me-2'), 25.80 (Me'-2'), 12.54 (Me-3').
*These signals may be interchanged.

MS (70 eV, *m/z*): 206 (M^+, 3%), 191 (M^+–Me, 2), 177 (M^+–Et, 4), 173 (5), 163 (4), 149 (M^+–COEt, 5), 145 (7), 136 (7), 121 (11), 108 ($C_8H_{12}^+$, 53), 98 (56), 93 (55), 79 (33), 67 (29), 57 ($C_3H_5O^+$, 100), 41 (49).

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