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

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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 Et2O (3×15 mL). The combined organic layers were washed with 2N HCl (2×15 mL), saturated Na2CO3 (2×15 mL) and brine (3×15 mL). The organic phase was dried over anhydrous Na2SO4 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).

¹H NMR (300 MHz, CDCl₃, 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).

¹³C NMR (75 MHz, CDCl₃, 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 (C₈H₁₂⁺, 53), 98 (56), 93 (55), 79 (33), 67 (29), 57 (C₃H₅O⁺, 100), 41 (49).

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