3-Methyl-3-(6,6a-trimethyl-hexahydro-cyclopenta[b]furan-2-yl)-butan-2-one

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A 40% aqueous solution of sulfuric acid (0.5 mL) was added to a stirred solution of 3,3-dimethyl-4-hydroxy-5-(2,2,3-trimethyl-3-cyclopentenyl)-pentan-2-one (I) (630 mg, 2.65 mmol) in methanol (5 mL) and the mixture refluxed for 1.5 h. The resulting solution was partially evaporated under reduced pressure, resolved in Et₂O and washed with saturated Na₂CO₃ (3×25 mL) and brine (3×25 mL). The organic layer was dried over anhydrous Na₂SO₄ and evaporated under reduced pressure to give a crude, which was filtered through a silica gel pad to yield the cyclized and rearranged title compound 2 (504 mg, 2.11 mmol, 80%) as a yellow liquid.

IR (neat, cm⁻¹): 1705 (CO), 1077, 906 (C=O-C).

¹H NMR (300 MHz, CDCl₃): δ = 0.82 (3H, s, Me-6’); 0.96 (3H, s, Me’-6’); 1.03 (3H, s, Me-6a’); 1.05 (3H, s, Me-3’); 1.09 (3H, s, H-4); 1.16-1.28 (1H, m, H-4’); 1.35 (1H, dd, J=12.2 Hz, 8.1 Hz, H-5’); 1.50-1.63 (2H, m, H-3’, H’-5’); 1.78-1.88 (1H, m, H’-3’); 1.90-2.06 (1H, m, H-4’); 2.20 (3H, s, H-1); 2.31-2.42 (1H, m, H-3a’); 3.97 (1H, dd, J=11.5 Hz, 4.7 Hz, H-2’). Some signals were assigned by means of 2D NMR experiments.

¹³C NMR (75 MHz, CDCl₃): δ = 26.87 (C-1); 213.73 (C-2); 50.09 (C-3); 18.53 (C-4); 21.82 (Me-3); 84.68 (C-2’); 34.81 (C-3’); 46.84 (C-3a’); 29.33 (C-4’); 40.54 (C-5’); 46.43 (C-6’); 94.96 (C-6a’); 22.08 (Me-6’); 25.23 (Me’-6’); 19.60 (Me-6a’). Some signals were assigned by means of 2D NMR experiments.

EI-MS (70 eV, m/z): 238 (M⁺, 0.2%); 223 (M⁺-Me, 0.4); 195 (M⁺-COMe, 0.1); 153 (M⁺-C₅H₉O, 15); 109 (10); 96 (22); 86 (47); 71 (13); 55 (13); 43 (CH₃CO⁺, 100).

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