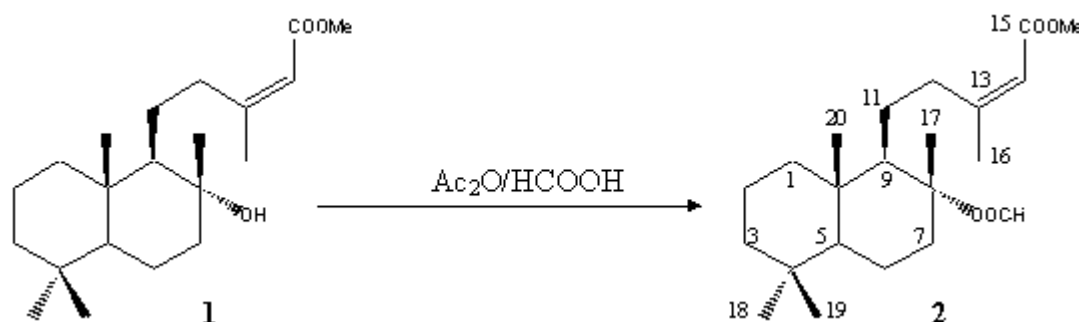


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www.molbank.org**Methyl 8a-Formyloxy-labd-13Z-en-15-oate****[(-)-(2Z)-5-((1R,2R,4aS,8aS)-2-Formyloxy-2,5,5,8a-tetramethyldecahydro-1-naphthalenyl)-3-methyl-2-pentenoic Acid Methyl Ester]****Juan M. Castro, Sofia Salido, Joaquin Altarejos*, Manuel Nogueras and Adolfo Sanchez**

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Tel.: 34-953-002743, fax: 34-953-012141, E-mail: jaltare@ujaen.es*Received: 12 December 2001 / Accepted: 19 September 2002 / Published: 8 March 2003***Keywords:** Diterpenes, labdanes, formylation, FAM, tertiary alcohol

A sample (0.03 mL, 0.45 mmol) of formic acid-acetic anhydride mixture (FAM), prepared from Ac_2O and formic acid as described in the literature [1], was added to the alcohol **1** [2] (29 mg, 0.09 mmol) at 10 °C. After stirring for 48 h at room temperature, water (10 mL) was added and the mixture extracted with Et_2O (3×25 mL). The combined organic layers were washed with 2N HCl (25 mL), saturated aq. Na_2CO_3 (25 mL) and brine (25 mL). The organic phase was dried over anhydrous Na_2SO_4 and the solvent evaporated under reduced pressure to yield a residue (29 mg) which was purified by flash chromatography on silica gel, using a 4:1 hexane/ Et_2O mixture as eluent, to give the title compound **2** (19 mg, 0.05 mmol, 60%).

Mp: 58.0-59.6 °C (white crystals, from hexane).

[α]_D = -12.7° (c 1.02 $\text{cg} \cdot \text{mL}^{-1}$, CHCl_3).IR (KBr, cm^{-1}): 1728, 1193 (OOCH), 1705, 1241, 1171 (COOMe), 1649, 875 (C=C).

^1H NMR (300 MHz, CDCl_3 , d, ppm): 0.77 (3H, s, Me_b -4), 0.82 (3H, s, Me -10), 0.86 (3H, s, Me_a -4), 1.49 (3H, s, Me -8), 1.88 (3H, d, $J=1.4$ Hz, Me -13), 0.99-1.88 (13H, m, H-1,2,3,5,6,7a,9,11), 2.53 (1H, dt, $J=12.5$ Hz, 3.3 Hz, Hb-7), 2.59-2.76 (2H, m, H-12), 3.65 (3H, s, OMe), 5.60 (1H, br s, H-14), 8.04 (1H, s, OOCH).

^{13}C NMR (75 MHz, CDCl_3 , d, ppm): 39.36* (C-1), 18.35 (C-2), 41.80 (C-3), 33.13 (C-4), 55.52 (C-5), 20.01 (C-6), 39.32* (C-7), 89.08 (C-8), 58.91 (C-9), 39.53 (C-10), 24.10 (C-11), 36.13 (C-12), 160.49 (C-13), 115.44 (C-14), 166.53 (C-15), 25.16 (C-16), 21.24 (C-17), 33.29 (C-18), 21.42 (C-19), 15.69 (C-20), 50.78 (OMe), 160.65 (OOCH) (* these signals may be interchanged).

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References and Notes

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Sample availability: Available from the authors and from MDPI

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