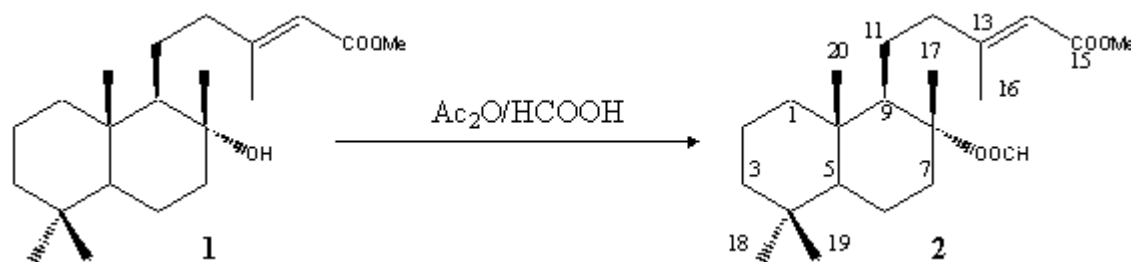


Molbank 2003, M301

www.molbank.org**Methyl 8a-Formyloxy-labd-13E-en-15-oate****[(-)-(2E)-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**

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*Received: 12 December 2001 / Accepted: 19 September 2002 / Published: 8 March 2003***Keywords:** Diterpenes, labdanes, formylation, FAM, tertiary alcohol

A sample (0.10 mL, 1.27 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] (83 mg, 0.25 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 (83 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** (54 mg, 0.15 mmol, 60%).

Mp: 89.1-91.0 °C (white crystals, from hexane).

[α]_D = -18.9° (c 1.06 $\text{cg} \cdot \text{mL}^{-1}$, CHCl_3).IR (KBr, cm^{-1}): 1716, 1184 (OOCH), 1716, 1218, 1184 (COOMe), 1651, 854 (C=C).

^1H NMR (300 MHz, CDCl_3 , d, ppm): 0.79 (3H, s, Me_b -4), 0.84 (3H, s, Me -10), 0.88 (3H, s, Me_a -4), 1.50 (3H, s, Me -8), 2.17 (3H, s, Me -13), 0.90-2.33 (15H, m, H-1,2,3,5,6,7a,9,11,12), 2.60 (1H, dt, $J=12.1$ Hz, 3.1 Hz, H_b -7), 3.69 (3H, s, OMe), 5.67 (1H, br s, H-14), 8.01 (1H, s, OOH).

^{13}C NMR (75 MHz, CDCl_3 , d, ppm): 39.50 (C-1), 18.21 (C-2), 41.72 (C-3), 33.07 (C-4), 55.52 (C-5), 19.95 (C-6), 39.37 (C-7), 89.00 (C-8), 58.46 (C-9), 39.50 (C-10), 23.87 (C-11), 43.62 (C-12), 160.77 (C-13), 114.83 (C-14), 167.23 (C-15), 19.02 (C-16), 21.03 (C-17), 33.22 (C-18), 21.36 (C-19), 15.60 (C-20), 50.74 (OMe), 160.28 (OOCH).

Acknowledgements: We wish to thank the Junta de Andalucía for financial support and the Ministerio de Educación, Cultura y Deporte for a Fellowship to J. M. Castro.

References and Notes

1. Strazzolini, P.; Giumanini, A. G.; Cauci, S. Acetic Formic Anhydride. A Review. *Tetrahedron* **1990**, *46*, 1081-1118.
2. Urones, J. G.; Basabe, P.; Marcos, I. S.; González, J. L.; Jiménez, V.; Sexmero, M. J.; Lithgow, A. M. Ambergris Compounds from Labdanolic Acid. *Tetrahedron* **1992**, *48*, 9991-9998.

Sample availability: Available from the authors and from MDPI

© 2003 [MDPI](#). All rights reserved.