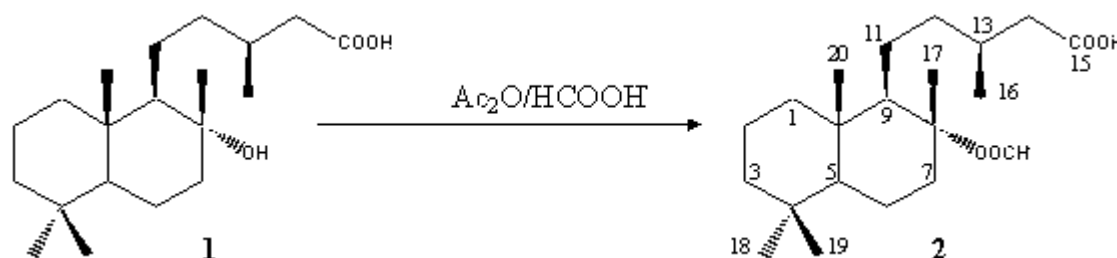


**8-Formyl-labdanolic Acid****(-)-(3S)-5-((1R,2R,4aS,8aS)-2-Formyloxy-2,5,5,8a-tetramethyldecahydro-1-naphthalenyl)-3-methylpentanoic Acid****Juan M. Castro, Sofia Salido, Joaquin Altarejos\*, Manuel Nogueras and Adolfo Sanchez**

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

A sample (0.85 mL, 6.5 mmol) of formic acid-acetic anhydride mixture (FAM), prepared from  $\text{Ac}_2\text{O}$  and formic acid as described in the literature [1], was added to the alcohol **1** [2] (255 mg, 0.79 mmol) at 10 °C. After stirring for 48 h at room temperature, water (10 mL) was added and the mixture extracted with  $\text{Et}_2\text{O}$  ( $3 \times 25$  mL). The combined organic layers were washed with 2N HCl (25 mL), saturated aq.  $\text{Na}_2\text{CO}_3$  (25 mL) and brine (25 mL). The organic phase was dried over anhydrous  $\text{Na}_2\text{SO}_4$  and the solvent evaporated under reduced pressure to yield a residue (258 mg) which was purified by flash chromatography on silica gel, using a 3:2 hexane/ $\text{EtOAc}$  mixture as eluent, to give the pure title compound **2** (180 mg, 0.51 mmol, 65%).

Mp: 102.4-105.5 °C (pale yellow crystals, from hexane).

[ $\alpha$ ]<sub>D</sub> = -33.7° (c 1.2  $\text{cg} \cdot \text{mL}^{-1}$ ,  $\text{CHCl}_3$ ).IR (neat,  $\text{cm}^{-1}$ ): 3600-2500, 1719 (COOH), 1719, 1200, 1180 (OOCH).

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ , d, ppm): 0.79 (3H, s,  $\text{Me}_b$ -4), 0.84 (3H, s, Me-10), 0.87 (3H, s,  $\text{Me}_a$ -4), 0.98 (3H, d,  $J=6.6$  Hz, Me-13), 1.49 (3H, s, Me-8), 0.99-2.00 (16H, m, H-1,2,3,5,6,7a,9,11,12,13), 2.15 (1H, dd,  $J=15.0$  Hz, 8.0 Hz, H-14), 2.36 (1H, dd,  $J=15.0$  Hz, 5.9 Hz, H'-14), 2.57 (1H, dt,  $J=12.3$  Hz, 3.2 Hz,  $\text{H}_b$ -7), 8.00 (1H, s, OOH).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ , d, ppm): 39.46 (C-1), 18.23 (C-2), 41.78 (C-3), 33.05 (C-4), 55.51 (C-5), 19.98 (C-6), 39.36 (C-7), 89.17 (C-8), 58.91 (C-9), 39.42 (C-10), 22.99 (C-11), 39.63 (C-12), 30.88 (C-13), 41.44 (C-14), 179.48 (C-15), 19.63 (C-16), 21.09 (C-17), 33.24 (C-18), 21.37 (C-19), 15.65 (C-20), 160.61 (OOCH).

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**References and Notes**

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*Sample availability:* Available from the authors and from MDPI

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