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2-Carbomethoxynor-31-lanosten-2-enol

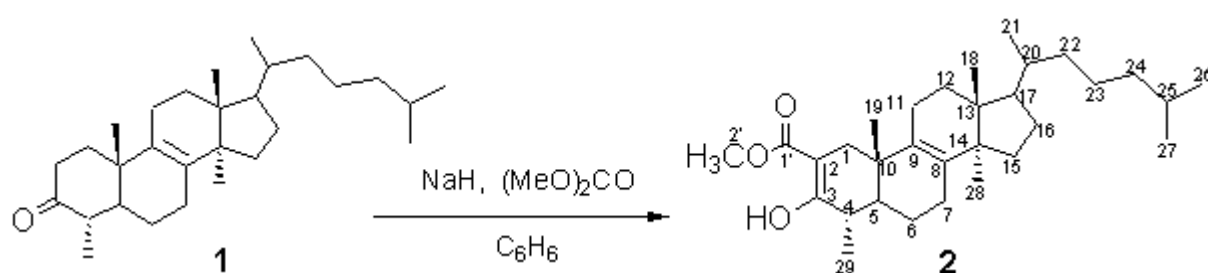
M. Daoubi^{1*}, A. Benharref¹ and M. Pierrot²

¹ Laboratoire de Chimie des Substances Naturelles et des Hétérocycles, Université Cadi Ayyad, Faculté des Sciences Semlalia, BP 2390. Bd Prince My Abdellah, Marrakech. Maroc. E-mail:

m_daoubi@hotmail.com

² LBS-UMR 6517, Centre Scientifique Saint-Jérôme, 13397 Marseille, CEDEX 20, France.

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To sodium hydride (0.3g, 12.7 mmole) carefully washed with anhydrous benzene under nitrogen (to eliminate mineral oil from the commercial product) [1], was added (0.8 ml, 8.43 mmole) of dimethylcarbonate (freshly distilled) in dry benzene (20 ml). Under nitrogen atmosphere, the mixture was vigorously stirred and heated at 70 °C. At this temperature was added drop wise and during 2 hours, (1g, 2.12 mmole) of **1** [2] in dry benzene (20 ml). The agitation was maintained at 100 °C during 12 hours. After cooling to 0 °C and acidification by 5.5 ml of acetic acid, the mixture was poured on 80 ml of ice added to (80 ml) of HCl (6N). The organic layer was washed with diluted solution of sodium bicarbonate, then dried. After evaporating the solvent *in vacuo*, the residue was purified by silica gel column chromatography using hexane as eluent to give **2** (0.85 g, 74 %).

Mp: 104-105°C.

IR: 1750 cm⁻¹.

MS (m/z) : 471.7 (M⁺).

¹H NMR (200 MHz, CDCl₃): 12.4 (s, OH); 3.75 (s, C2'-H₃); 0.75 (s, C18-H₃); 0.87 (s, C19-H₃); 0.95 (d, J= 6Hz, C21-H₃); 0.79 (s, C28-H₃); 1.22 (d, J=6Hz, C29-H₃).

¹³C NMR (50 MHz, CDCl₃): 35.2 (C1); 95.9 (C2); 174.81 (C3); 39.0 (C4); 46.9 (C5); 21.6 (C6); 28.0 (C7); 135.54 (C8); 131.96 (C9); 36.2 (C10); 21.6 (C11); 25.4 (C12); 44.4 (C13); 49.7 (C14); 30.9 (C15); 30.6 (C16); 50.4 (C17); 15.7 (C18); 20.1 (C19); 36.3 (C20); 18.6 (C21); 36.3 (C22); 23.9 (C23); 39.42 (C24); 27.8 (C25); 22.9 (C26); 22.5 (C27); 24.4 (C28); 27.9 (C29); 51.4 (C2'); 175.2 (C1').

References

1. Benharref, A.; Lavergne, J.-P. *Bull. Soc. Chim. Fr.* **1985**, 965.
2. Vander Roest, J. M.; Grieco, P. A. *J. Am. Chem. Soc.* **1993**, *115*, 5841.

Sample Availability: Available from the authors and MDPI.

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