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2-Carbomethoxy-8a,9a-epoxy Nor-31-lanosten-3-enol

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To sodium hydride (0.34 g, 13.96 mmole) carefully washed with anhydrous benzene under nitrogen (to eliminate mineral oil from the commercial product) [1], was added 0.8 ml (9.27 mmol) 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 dropwise 1g (2.33 mmol) of **1** in dry benzene (20 ml) in 2 hours. 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 a mixture of 80 ml of ice and 80 ml of HCl (6N). The organic layer was washed with diluted solution of sodium bicarbonate, then dried. After evaporating the solvent *in vacuum*, the residue was purified by silica gel column chromatography using hexane as eluent to give **2** (0.93 g, 82 %).

IR : 1750 cm^{-1} .

MS (EI, 70eV) : 487.7 (M^{+.}).

¹H NMR (200 MHz, CDCl₃) : 12.27 (s, OH); 3.66 (s, C2'-H₃); 0.72 (s, C18-H₃); 0.89 (s, C19-H₃); 1.06 (d, J=6 Hz, C21-H₃); 0.84 (s, C28-H₃); 1.22 (d, J=6 Hz, C29-H₃).

¹³C NMR (50 MHz, CDCl₃): 32.46 (C1); 95.4 (C2); 173.84 (C3); 37.50 (C4); 43.99 (C5); 20.25 (C6);
28.31 (C7); 69.52 (C8); 68.42 (C9); 36.27 (C10); 19.36 (C11); 23.88 (C12); 39.83 (C13); 48.71 (C14);
31.98 (C15); 28.79 (C16); 49.26 (C17); 15.59 (C18); 16.54 (C19); 36.7 (C20); 15.97 (C21); 36.65 (C22);
22.89 (C23); 39.58 (C24); 24.42 (C25); 22.27 (C26); 21.10 (C27); 23.17 (C28); 27.17 (C29); 51.72 (C2');
174.06 (C1').

Reference

[1] 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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