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2-Formyl nor-31-lanosten-3-enol

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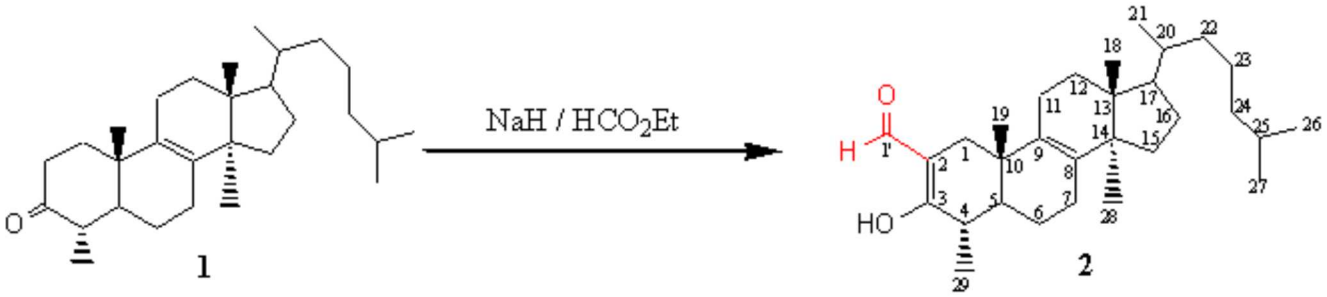

To a solution of **1** [1] (1 g, 2.25 mmol) in 50 mL of dry benzene held under nitrogen, was added 0.5 mL of ethyl formate [2] (distilled from phosphorus pentoxide) followed by a solution of (0.45 g, 8.33mmol) of sodium in 35 mL of absolute methyl alcohol. The resulting solution was then kept at room temperature under nitrogen for several hours (usually overnight). Reaction was indicated by the appearance of a deep colour. The mixture was poured into a cold solution of 5 mL of glacial acetic acid in 20 mL of water, and the resulting precipitate was extracted into benzene or methylene dichloride. The organic layer was washed with water, dried with Na_2SO_4, and concentrated. The residue was then chromatographed on a silica gel column using hexane/EtOAc (99/1) as eluant to give **2** (0.85 g, 80 %).

Mp: 161-163 °C.

MS (EI, 70eV) : 440.72 ($M^{+\cdot}$).

^{1}H NMR (400 MHz, $CDCl_3$) : 14.75 (s, OH); 8.64 (s, C1'-H); 0.73 (s, C18-H_3); 0.91 (s, C19-H_3); 1.13 (D, C21-H_3) ; 0.86 (s, C28-H_3); 1.23 (d, C29-H_3).

^{13}C NMR (100 MHz, $CDCl_3$):31.27 (C1); 105.93 (C2); 187,49 (C3); 38.49 (C4); 44.45 (C5); 21.41 (C6); 28.13 (C7); 136.00 (C8); 131.38 (C9); 36.43 (C10); 18.74 (C11); 24.11 (C12); 39.50 (C13); 49.99 (C14); 30.91 (C15); 28.01 (C16); 50.47 (C17); 14.92 (C18); 16.87 (C19); 36.48 (C20); 15.86 (C21); 23.73 (C22); 22.84 (C23); 21.50 (C24); 35.68 (C25); 21.86 (C26); 21.99 (C27); 24.41 (C28); 25.32 (C29); 188.46 (C1').

References

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Sample Availability: Available from the authors and from MDPI.