

3 β -Tosyloxy-elemo-lanost-8-en-24-one

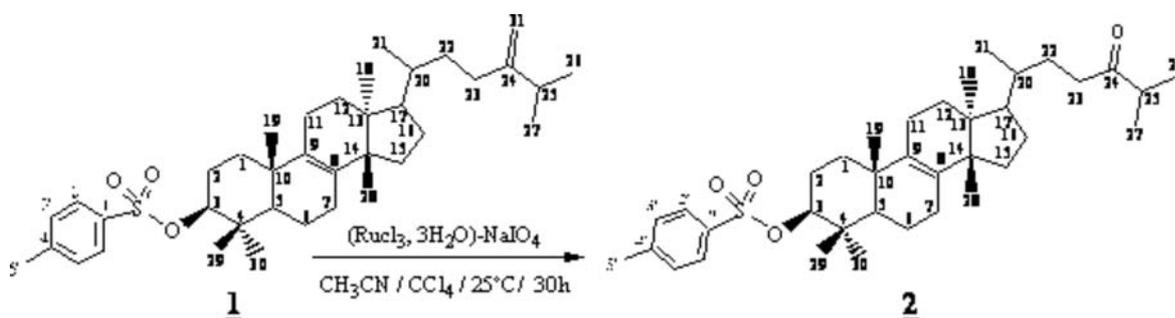
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The sodium periodate is prepared in situ with equimolecular quantity of soda NaOH (0.5g; 12.50 mmol) and periodic acid H₅IO₆ (2.85g; 12.50 mmol), the mixture is stirred at 0°C. After 15 min, 5 ml of CCl₄, 5 ml of H₃CCN and 93.03mg (0.12mmol) of ruthenium trichloride^{1,2} were added. The mixture was stirred during 15min, and then 1.85g (3.12 mmol) of **1**³ was added. The reaction was left under stirring at 25°C for 30h, then 20 ml of distilled water was added and the reactional mixture was extracted with the dichloromethane. After filtration on silica gel column to eliminate RuO₄, the organic layer was recovered, dried by Na₂SO₄ and evaporated under reduce pressure. The residue was purified on silica gel column using hexane: ethyl acetate (96: 4) as eluent to give 1.30g (2.18 mmol) of **2** in 70 % yield.

Melting point: 115-116 °C (Hexane)

MS (EI, 70eV): 596 (M⁺).

¹H NMR (300 MHz, CDCl₃) d(ppm): 4.22 (1H-3, dd, J₁ = 12 Hz, J₂ = 4 Hz); 7.96 (2H-2', d, J = 8.8Hz); 7.35 (2H-3', d, J = 8.8 Hz); 2.48 (3H-5'); 0.76 (3H-18, s); 0.85 (3H-19, s); 0.93 (3H-21, d, J = 6 Hz); 2.60 (2H-23, m); 1.10 (3H-26, d, J = 2 Hz); 1.11 (3H-27, d, J = 2 Hz); 0.80 (3H-28, s); 0.95 (3H-29, s); 1.05 (3H-30, s).

¹³C NMR (75 MHz, CDCl₃)d (ppm): 35.15 (C-1); 27.52 (C-2); 91.00 (C-3); 36.25 (C-4); 49.98 (C-5); 38.78 (C-6); 28.03 (C-7); 133.66 (C-8); 134.93 (C-9); 36.25 (C-10); 20.20 (C-11); 25.47 (C-12); 44.16 (C-13); 50.16 (C-14); 30.76 (C-15); 30.21 (C-16); 51.33 (C-17); 18.64 (C-18); 18.99 (C-19); 37.01 (C-20); 21.54 (C-21); 37.59 (C-22); 29.84 (C-23); 215.50 (C-24); 35.64 (C-25); 21.72 (C-26); 22.40 (C-27); 31.80 (C-25); 21.70 (C-26); 18.80 (C-27); 14.5 (C-28); 16.41 (C-29); 144.35 (C-1'); 129.76 (C-2'); 127.77 (C-3'); 134.82 (C-4'); 21.25 (C-5').

MS (m/z): 596 (35%), 425 (48%), 297 (75%).

Acknowledgments

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