8a,13-Epoxy-14,15-dinorlabd-12-ene (Sclareol Oxide)

To a stirred solution of a mixture of E/Z isomers 1 (174 mg, 0.52 mmol, 45:55 ratio) in acetone (8 mL) was added a mixture of KMnO₄ (260 mg, 1.64 mmol) and anhydrous MgSO₄ (230 mg) at 10 °C [1]. After stirring for 10 min the reaction was allowed to warm to room temperature. After 20 min more the crude was filtered over Celite and the clean solution evaporated under reduced pressure to yield a residue which was solved in Et₂O (25 mL). This solution was washed with brine (3×10 mL), dried over anhydrous Na₂SO₄ and the solvent evaporated under reduced pressure to yield a residue (128 mg) which was solved in toluene (10 mL) and distilled in a Dean-Stark trap device for 1 h. The resulting solution was evaporated under reduced pressure to yield the title compound 2 (120 mg, 0.46 mmol, 88%).

Mp: 34.0-36.0 °C (white crystals, from methanol).

[a]D = +1.22º (c 0.95 cg·mL⁻¹, CHCl₃).

IR (neat, n, cm⁻¹): 3054, 1683 (C=C), 1127 (CTOTC).

¹H NMR (300 MHz, CDCl₃, d, ppm): 0.82 (6H, s, Me₄-4, Me-10), 0.88 (3H, s, Me₉-4), 1.16 (3H, s, Me-8), 1.68 (3H, br s, Me-13), 0.89-1.85 (13H, m, H₁,2,3,5,6,7a,9,11), 1.94 (1H, dt, J=12.3 Hz, 3.2 Hz, Hb-7), 4.43 (1H, br s, H-12).

¹³C NMR (75 MHz, CDCl₃, d, ppm): 39.29 (C-1), 18.56 (C-2), 41.91 (C-3), 33.14 (C-4), 56.17 (C-5), 19.74 (C-6), 41.12 (C-7), 76.17 (C-8), 52.43 (C-9), 36.66 (C-10), 18.26 (C-11), 94.54 (C-12), 147.83 (C-13), 20.43 (C-16), 20.07 (C-17), 33.43 (C-18), 21.55 (C-19), 15.00 (C-20).

Acknowledgements: We wish to thank the Junta de Andalucía for financial support and the Ministerio de Educación, Cultura y Deporte for a Fellowship to J. M. Castro.

References and Notes

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

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