(1S,4R,5R,8R,9S,12R,13S) 12-(1S-hydroxyethyl)-13-(3-hydroxypropyl...

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(1S,4R,5R,8R,9S,12R,13S) 12-(1S-Hydroxyethyl)-13-(3-hydroxypropyl)-5-(1,5-dimethylhexyl)-1,9-epoxy-4,8,13 Trimethyl Tricyclo [7,4,0,0^{4,8}] Tridecane

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A mixture of 1 (1 g, 2.25 mmol) and lithium aluminium hydride (0.34 g, 8.94 mmol) in 40 mL of THF was refluxed for 12 h. After being diluted with 20 mL of THF, the mixture was cooled to 0°C and carefully treated with 2 mL of distilled water. The resulting mixture was stirred for 40 min at room temperature and treated with anhydrous sodium sulfate. Filtration, and concentration under reduced pressure gave the crude product, which was purified by column chromatography on silica gel using hexane/EtOAc: 35/65) to give 2 (0.93 g, 92 %). The structure of the compound 2 was established by X-ray crystal structure determination.

Mp: 140-141 °C.

EI-MS (70eV): 448.72 (M⁺·).

¹H NMR (400 MHz, CDCl₃): 3.92 (t, C16-H₂); 3.60 (m, C17-H); 0.74 (s, C28- H₃); 1.02 (s, C29-H₃); 1.09 (d, J= 6Hz, C20-H₃); 0.86 (s, C19-H₃); 1.25 (d, J=6Hz, C18-H₃).

¹³C NMR (100 MHz, CDCl₃): 64.74 (C1); 39.91 (C2); 32.02 (C3); 36.06 (C4); 43.15 (C5); 29.98 (C6); 30.10 (C7); 43.51 (C8); 69.34 (C9); 39.55 (C10); 28.12 (C11); 48.05 (C12); 48.69 (C13); 32.83 (C14); 38.33 (C15); 63.02 (C16); 67.26 (C17); 27.72(C18); 22.97 (C19); 18.0 (C20); 35.99 (C21); 23.78 (C22); 23.47 (C23); 22.26 (C24); 35.06 (C25); 16.21 (C26); 16.22 (C27); 15.1 (C28); 18.77 (C29).

References

- 1. Daoubi, M.; Benharref, A.; Kossareva, E.; Pierrot, M. Molecules 2000, 5, M188.
- 2. Guan, H. P.; Ksebati, M. B.; Kern, E. R.; ZemLicka, J. J. Org. Chem. 2000, 65, 5177-5184.

Sample Availability: Available from the authors and from MDPI.

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