

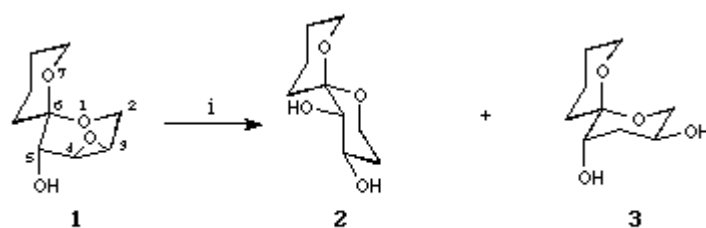
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[4R*,5R*,6S*]-1,7-Dioxaspiro[5.5]undecane-4,5-diol

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Reagents and Conditions: (i) LiAlH₄ (excess), THF, N₂ atmosphere, room temp., 12h.

A solution of [3R*,4R*,5S*,6S*]-3,4-epoxy-1,7-dioxaspiro[5.5]undecan-5-ol (**1**) (561 mg, 3.01 mmol) in dry tetrahydrofuran (30 ml) under a nitrogen atmosphere was cooled to 0 deg.C in an ice/water bath.

Lithium aluminium hydride (4.52 ml, of a 1 mol L⁻¹ solution in THF, 4.52 mmol) was added in 3 portions over 1 minute, and the reaction mixture allowed to stir at 0 deg.C for 1 h then room temperature for 12 h. After quenching with sodium dihydrogen phosphate solution (10 ml, 10% w/v), the reaction mixture was extracted with ethyl acetate (3x 50 ml), the combined extracts washed with water (10 ml) and dried over sodium sulphate. Removal of the solvent under reduced pressure gave a colourless oil, which was purified by flash chromatography using hexane-ethyl acetate (2:1) as eluent to afford [4R*,5R*,6S*]-1,7-Dioxaspiro[5.5]undecane-4,5-diol (**2**) (310 mg, 55%) as colourless needles [1].

M.p. 135-137 deg.C.

IR (Nujol) cm⁻¹ 3600-3150 (br, s, OH), 1082 (s, C-O).

¹H-NMR (400 MHz, CDCl₃) 1.47 (1H, ddd, *J*_{11ax,11eq} 13.3, *J*_{11ax,10ax} 13.3 and *J*_{11ax,10eq} 4.4 Hz, 11ax-H), 1.54-1.81 (5H, m, 9-CH₂, 10-CH₂ and 3ax-H), 1.90 (1H, dt, *J*_{11eq,11ax} 13.3 and *J*_{11eq,10} 2.6 Hz, 11eq-H), 2.05-2.14 (1H, m, 3eq-H), 3.41 (1H, dd, *J*_{5,4} 7.4 and *J*_{5,OH} 3.7 Hz, 5-H), 3.62 (1H, ddd, *J*_{2eq,2ax} 11.8, *J*_{2eq,3ax} 5.5 and *J*_{2eq,3eq} 2.3 Hz, 2eq-H), 3.71-3.99 (6H, m, 8-CH₂, 4-H, 2ax-H, 4-OH and 5-OH).

¹³C-NMR (100 MHz, CDCl₃) 17.6, 24.8, 27.6, 30.6 (CH₂, C-3, C-9, C-10 and C-11), 55.6 (CH₂, C-2), 61.3 (CH₂, C-8), 68.6 (CH, C-4), 71.1 (CH, C-5), 98.4 (quat, C-6).

CI-MS 189 (M⁺H, 85%), 171 (M⁺H-H₂O, 100), 153 (10), 118 (13), 115 (8), 101 (80), 83 (10).

Anal. calc. for C₉H₁₆O₄ C, 57.44; H, 8.57%; M⁺H, 189.1127, found C, 57.50; H, 8.42%; M⁺H (CI, NH₃), 189.1134.

Acknowledgment: The authors gratefully acknowledge financial support from the Australian Research Council and The University of Sydney.

References and Notes

1. Another product (**3** in the Scheme, a colourless glass, 33mg, 6%) will be reported in the following short

note. Brimble, M. A.; Johnston, A. D. *Molecules* **1997**, *2*, M20.

Sample Availability: No sample available.

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