

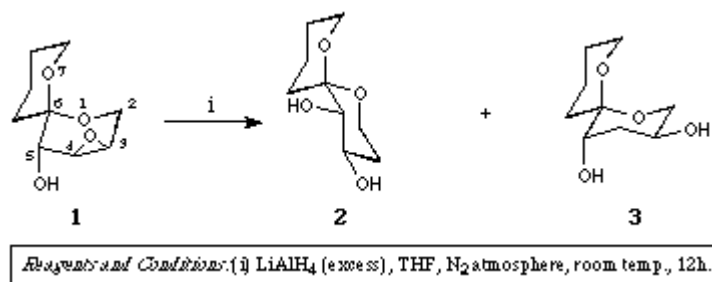
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## [3S\*,5S\*,6S\*]-1,7-Dioxaspiro[5.5]undecane-3,5-diol

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**Scheme**

The reaction and the product ratio shown in the scheme was reported [1]. The two products (**2** [1] and **3**) were separated by flash chromatography using hexane-ethyl acetate (1:1) as eluent to afford the title compound **3** in the scheme) as a colourless glass (33 mg, 6%).

M.p. 63-64 deg.C.

High Res. MS calc. for  $\text{C}_9\text{H}_{16}\text{O}_4 \text{M}^+\text{H}$  (CI,  $\text{NH}_3$ ) 189.1128, found:  $\text{M}^+$  189.1129.

IR (Nujol)  $\text{cm}^{-1}$  3610-3140 (br, s, OH), 1101 (s, C-O).

$^1\text{H}$ -NMR (200 MHz,  $\text{CDCl}_3$ ) 1.37 (1H, ddd,  $J_{11\text{ax},11\text{eq}}$  12.7,  $J_{11\text{ax},10\text{ax}}$  12.7 and  $J_{11\text{ax},10\text{eq}}$  4.7 Hz, 11ax-H), 1.49-1.70 (4H, m, 9- $\text{CH}_2$  and 10- $\text{CH}_2$ ), 1.85-1.98 (3H, m, 11eq-H and 4- $\text{CH}_2$ ), 2.51 (2H, m, 2 x OH), 3.39 (1H, dd,  $J_{2\text{ax},2\text{eq}}$  10.4 and  $J_{2\text{ax},3\text{ax}}$  10.4 Hz, 2ax-H), 3.61-3.69 (4H, m, 2eq-H, 8- $\text{CH}_2$  and 5-H), 3.94-4.09 (1H, m, 3ax-H).

$^{13}\text{C}$ -NMR (50 MHz,  $\text{CDCl}_3$ ) 18.3, 24.9, 29.9, 35.6 ( $\text{CH}_2$ , C-4, C-9, C-10 and C-11), 60.8 ( $\text{CH}_2$ , C-8), 61.9 (CH, C-3), 64.3 ( $\text{CH}_2$ , C-2), 71.5 (CH, C-5), 95.7 (quat, C-6).

CI-MS 189 ( $\text{M}^+\text{H}$ , 8%), 171 ( $\text{M}^+\text{H}-\text{H}_2\text{O}$ , 100), 153 ( $\text{M}^+\text{H}-2\text{H}_2\text{O}$ , 15), 111 (13), 101 (6).

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## References

1. Brimble, M. A.; Johnston, A. D. *Molecules* **1997**, M19.

*Sample Availability:* Available from Author, 0.1g and from MDPI, MDPI 11866.

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