

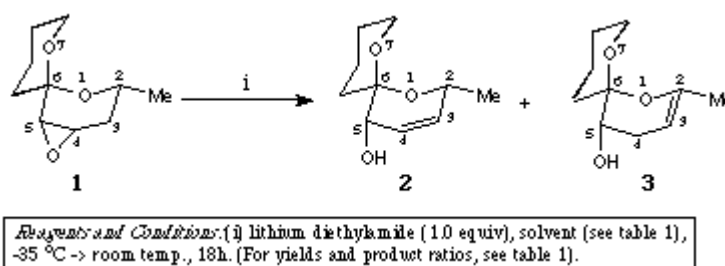
Molecules **1997**, *2*, M16

[2R*,5S*,6S*]-2-Methyl-1,7-dioxaspiro[5.5]undec-2-en-5-ol

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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 (6:4) as eluent to afford the title compound [2R*,5S*,6S*]-2-Methyl-1,7-dioxaspiro[5.5]undec-2-en-5-ol (**3** in the scheme) as a colourless oil (20 mg, 16%).

High Res. MS calc. for C₁₀H₁₆O₃ M⁺ 184.10994, found: M⁺ 184.10981.

IR (film) cm⁻¹ 3680-3100 (br, s, OH), 2942, 2850 (s, C-H), 1689 (m, C=C), 1091 (s, C-O).

¹H-NMR (400 MHz, CDCl₃) 1.48 (1H, ddd, *J*_{11ax,11eq} 13.4, *J*_{11ax,10ax} 13.4 and *J*_{11ax,10eq} 4.5 Hz, 11ax-H), 1.57-1.70 (4H, m, 9-CH₂ and 10-CH₂), 1.80 (3H, s, Me), 1.85-1.94 (1H, m, 11eq-H), 2.05-2.09 (1H, m, 4eq-H), 2.39-2.45 (1H, m, 4ax-H), 3.57 (1H, t, *J*_{5,4} 3.5 Hz, 5-H), 3.69 (1H, dt, *J*_{8eq,8ax} 11.4 and *J*_{8eq,9} 2.1 Hz, 8eq-H), 3.83 (1H, ddd, *J*_{8ax,8eq} 11.4, *J*_{8ax,9ax} 11.4 and *J*_{8ax,9eq} 3.1 Hz, 8ax-H), 4.47-4.49 (1H, m, 3-H).

¹³C-NMR (100 MHz, CDCl₃) 18.0, 25.1, 26.4, 29.6 (CH₂, C-4, C-9, C-10 and C-11), 19.6 (CH₃, Me), 61.8 (CH₂, C-8), 68.2 (CH, C-5), 93.3 (CH, C-3), 97.1 (quat, C-6), 147.1 (quat, C-2).

EI-MS 184 (M⁺, 5%), 167 (M⁺ -OH, 100), 125 (35), 111 (30), 101 (15), 98 (20), 55 (25), 43 (70).

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References

1. Brimble, M. A.; Johnston, A. D. *Molecules* **1997**, *2*, M15.

Sample Availability: No sample available.

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