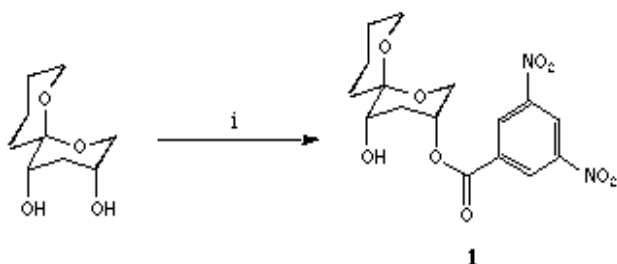


Molecules **1997**, *2*, M17**[3R*,5S*,6S*]-5-Hydroxy-1,7-dioxaspiro[5.5]undec-3-yl 3,5-Dinitrobenzoate****Margaret A. Brimble* and Andrew D. Johnston**

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Reagents and Conditions: (i) Et₃N (2.0 equiv), DMAP (cat), 3,5-dinitrobenzoyl chloride (1.0 equiv), CH₂Cl₂, room temp., 48 h., 45%.

To a solution of [3R*,5S*,6S*]-1,7-dioxaspiro[5.5]undecane-3,5-diol (50 mg, 0.27 mmol) in dichloromethane (5.0 ml), was added triethylamine (56 mg, 0.55 mmol), 4-dimethylaminopyridine (~1 mg), and 3,5-dinitrobenzoyl chloride (64 mg, 0.28 mmol) and the resultant solution allowed to stand at room temperature for 48 h. Removal of the solvent at reduced pressure gave a pale yellow oil, that was purified by flash chromatography using hexane-ethyl acetate (6:4) as eluent to afford the title compound (**1**) (46 mg, 45%) as pale yellow plates.

M.p. 54-56 deg.C.

IR (Nujol) cm⁻¹ 3680-3100 (br, s, OH), 1723 (s, C=O), 1110, 1083 (s, C-O).

High Res. MS calc. for C₁₆H₁₈N₂O₉ M⁺ 382.1012, found: M⁺, 382.0988.

¹H-NMR (400 MHz, CDCl₃) 1.35 (1H, ddd, *J*_{11ax,11eq} 13.4, *J*_{11ax,10ax} 13.4 and *J*_{11ax,10eq} 4.8 Hz, 11ax-H), 1.41-1.72 (4H, m, 9-CH₂ and 10-CH₂), 2.01 (1H, dt, *J*_{11eq,11ax} 13.4 and *J*_{11eq,10} 2.8 Hz, 11eq-H), 2.09 (1H, dddd, *J*_{4eq,4ax} 15.3, *J*_{4eq,5} 2.5, *J*_{4eq,3} 2.5 and *J*_{4eq,2eq} 2.5 Hz, 4eq-H), 2.12 (1H, m, OH), 2.35 (1H, ddd, *J*_{4ax,4eq} 15.3, *J*_{4ax,5} 3.6 and *J*_{4ax,3} 3.6 Hz, 4ax-H), 3.44 (1H, m, 5-H), 3.56-3.67 (2H, m, 8-CH₂), 3.85 (1H, ddd, *J*_{2ax,2eq} 13.3, *J*_{2ax,3} 2.5 and *J*_{2ax,4ax} 2.5 Hz, 2eq-H), 3.95 (1H, dd, *J*_{2eq,2ax} 13.3 and *J*_{2eq,3} 1.9 Hz, 2eq-H), 5.14 (1H, m, 3-H), 9.07-9.14 (3H, m, Ar-H).

¹³C-NMR (100 MHz, CDCl₃) 18.0, 24.8, 29.5, 30.8 (CH₂, C-4, C-9, C-10 and C-11), 61.4, 61.5 (CH₂, C-2 and C-8), 68.9 (CH, C-3), 70.6 (CH, C-5), 96.5 (quat, C-6), 122.4 (CH, C-4'), 129.4 (CH, C-2' and C-6'), 133.9 (quat, C-1'), 148.6 148.7 (quat, C-3' and C-5'), 162.0 (quat, C=O).

CI-MS 382 (M⁺, 2%), 295 (40), 213 (30), 195 (80), 179 (20), 149 (60), 114 (38), 101 (100), 83 (33), 75 (65), 69 (90), 59 (20), 55 (80), 41 (59).

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Sample Availability: No sample available.

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