

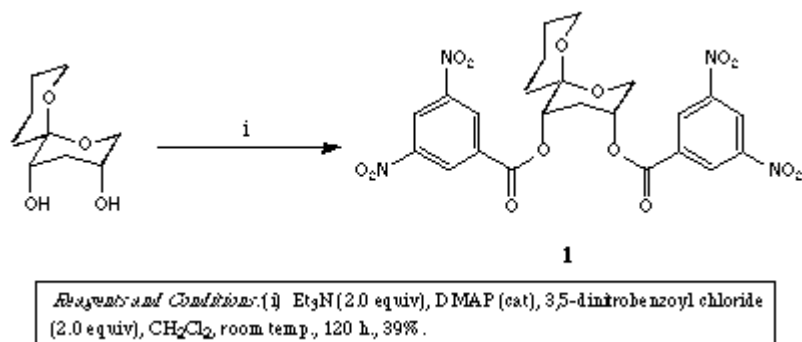
Molecules **1997**, *2*, M18

[3*R,5*S**,6*S**]-1,7-Dioxaspiro[5.5]undecane-3,5-diyl Bis(3,5-dinitrobenzoate)**

Margaret A. Brimble* and Andrew D. Johnston

Division of Organic Chemistry, School of Chemistry F11, The University of Sydney, N.S.W 2006, Australia. phone +61-2-9351-2750, fax +61-2-9351-6650, e-mail brimble_m@alf.chem.su.oz.au, <http://www.chem.usyd.edu.au/~brimble>

Received: 16 June 1997 / Published: 20 June 1997



To a solution of [3*R**,5*S**,6*S**]-1,7-dioxaspiro[5.5]undecane-3,5-diol (50 mg, 0.27 mmol) in dichloromethane (5.0 cm³), was added triethylamine (60 mg, 0.59 mmol), 4-dimethylaminopyridine (~1 mg), 3,5-dinitrobenzoyl chloride (136 mg, 0.59 mmol) and the resultant solution allowed to stand at room temperature for 120 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**) (61 mg, 39%) as pale yellow plates.

M.p. 64-66 deg.C.

IR (Nujol) cm⁻¹ 1727 (s, C=O), 1100, 1083, 1016 (s, C-O).

High Res. MS calc. for C₂₃H₂₀N₄O₁₄ M⁺ (DEI), 576.09760 requires: M⁺ 576.09705.

¹H-NMR (400 MHz, CDCl₃) 1.43 (1H, ddd, *J*_{11ax,11eq} 12.3, *J*_{11ax,10ax} 12.3 and *J*_{11ax,10eq} 4.6 Hz, 11ax-H), 1.62-1.96 (5H, m, 9-CH₂, 10-CH₂, and 11eq-H), 2.45 (1H, d, *J*_{4eq,4ax} 16.3 Hz, 4eq-H), 2.72 (1H, ddd, *J*_{4ax,4eq} 16.3, *J*_{4ax,5} 4.1 and *J*_{4ax,3} 4.1 Hz, 4ax-H), 3.72-3.78 (2H, m, 8-CH₂), 4.06-4.17 (2H, m, 2-CH₂), 5.11-5.20 (2H, m, 5-H and 3-H), 8.97-9.19 (6H, m, Ar-H).

¹³C-NMR (100 MHz, CDCl₃) 17.8, 24.5, 27.4, 30.9 (CH₂, C-4, C-9, C-10 and C-11), 61.0, 61.3 (CH₂, C-8 and C-2), 68.7 (CH, C-3), 71.6 (CH, C-5), 94.8 (quat, C-6), 122.4, 122.7 (CH, Ar-C), 129.1, 129.2 (quat, C-NO₂), 148.8 (quat, C-1', C-1''), 162.0 (quat, 2 x C=O).

DEI-MS 576 (M⁺, 100), 548 (50), 646 (48), 521 (60), 504 (100), 476 (100), 266 (75).

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

Sample Availability: Available from MDPI, MDPI 11864.

© 1997 MDPI. All rights reserved