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 C\(_{16}\)H\(_{18}\)N\(_2\)O\(_9\) M\(^+\) 382.1012, found: M\(^+\), 382.0988.

\(^1\)H-NMR (400 MHz, CDCl\(_3\)) 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-CH\(_2\) and 10-CH\(_2\)), 2.01 (1H, dt, J\(_{11eq,11ax}\) 13.4 and J\(_{11eq,10}\) 2.8 Hz, 11eq-H), 2.09 (1H, ddd, J\(_{4ax,4eq}\) 15.3, J\(_{4ax,5}\) 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-CH\(_2\)), 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).

\(^{13}\)C-NMR (100 MHz, CDCl\(_3\)) 18.0, 24.8, 29.5, 30.8 (CH\(_2\), C-4, C-9, C-10 and C-11), 61.4, 61.5 (CH\(_2\), 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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