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[4R*,5R*,6S*]-1,7-Dioxaspiro[5.5]undec-4,5-diyl Diacetate

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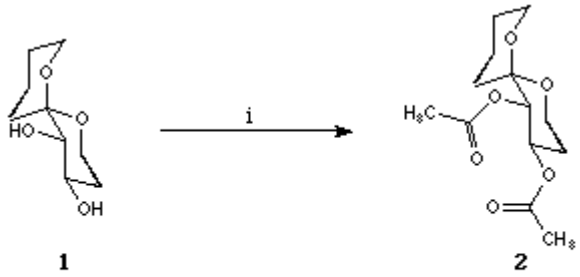

Reagents and Conditions: (i) Ac_2O (2.0 equiv), Et_3N (3.0 equiv), DMAP (cat), CH_2Cl_2, room temp., 1h,

To a solution of [4R*,5R*,6S*]-1,7-dioxaspiro[5.5]undecane-4,5-diol (**1**) (50 mg, 0.27 mmol) in dichloromethane (10 ml) was added triethylamine (100 mg, 0.99 mmol), acetic anhydride (67 mg, 0.66 mmol) and 4-dimethylaminopyridine (3 mg). The reaction mixture allowed to stand at room temperature for 1 h, then quenched with water (2.0 ml), extracted with dichloromethane (2x 50 ml) and dried over sodium sulphate. Removal of the solvent under reduced pressure gave a pale yellow oil, that was purified by flash chromatography using hexane-ethyl acetate (2:1) as eluent to afford [*4R*,5R*,6S**]-1,7-dioxaspiro[5.5]undec-4,5-diyl diacetate (**2**) (69 mg, 94%) as a colourless oil.

High Res. MS calc. for $C_{13}H_{20}O_6$ M^+H (CI, NH_3) 273.1338, found: M^+ 273.1337.

IR (Nujol) cm^{-1} 1731 [s, C=O (ester)], 1080 (s, C-O).

^{1}H-NMR (200 MHz, $CDCl_3$) 1.39-1.82 (8H, m, 4-CH_2, 9-CH_2, 10-CH_2, and 11-CH_2), 2.07 (3H, s, Ac), 2.09 (3H, s, Ac), 3.53-3.82 (4H, m, 2-CH_2, and 8-CH_2), 3.94 (1H, ddd, $J_{2ax,2eq}$ 11.3, $_{2ax,3ax}$ 11.3, and $_{2ax,3eq}$ 3.0 Hz, 2ax-H), 4.81-4.85 (2H, m, 4-H and 5-H).

^{13}C-NMR (50 MHz, $CDCl_3$) 17.6, 24.6 (CH_2, C-9 and C-10), 20.8, 21.1 (CH_3, 2x Ac), 26.3 (CH_2, C-3), 29.3 (CH_2, C-11), 55.9 (CH_2, C-2), 60.8 (CH_2, C-8), 67.9 (CH, C-4), 70.0 (CH, C-5), 96.1 (quat, C-6), 170.1, 171.0 (quat, 2x C=O).

CI-MS 273 (M^+H, 20%), 213 (100), 153 (30), 101 (5).

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Sample Availability: No sample available.