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[*3S*,5S*,6S**]-1,7-Dioxaspiro[5.5]undecane-3,5-diyl Diacetate

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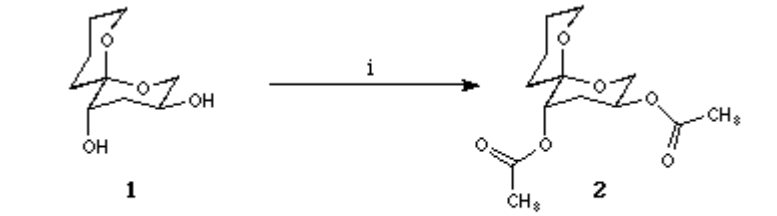

Reagents and Conditions: (i) Ac_2O (2.0 equiv), Et_3N (3.0 equiv), DMAP (cat), CH_2Cl_2, room temp., 1h.

To a solution of [*3S*,5S*,6S**]-1,7-dioxaspiro[5.5]undecane-3,5-diol (**1**) [1] (33 mg, 0.17 mmol) in dichloromethane (10 ml) was added triethylamine (54 mg, 0.53 mmol), acetic anhydride (34 mg, 0.34 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 [*3S*,5S*,6S**]-1,7-Dioxaspiro[5.5]undec-3,5-diyl diacetate (**2**) (43 mg, 90%) as a colourless oil.

High Res. MS calc. for $C_{13}H_{20}O_6$ M^+H (CI, NH_3) 273.1329, found: M^+ 273.1338.

IR (Nujol) cm^{-1} 1720 [s, C=O (ester)], 1010 (s, C-O).

^{1}H-NMR (200 MHz, $CDCl_3$) 1.34 (1H, ddd, $J_{11ax,11ex}$ 14.0, $J_{11ax,10ax}$ 14.0 and $J_{11ax,10eq}$ 5.2 Hz, 11ax-H), 1.49-1.77 (6H, m, 4-CH_2, 9-CH_2 and 10-CH_2), 2.01 (3H, s, Ac), 2.09 (3H, s, Ac), 1.99-2.11 (1H, m, 11eq-H), 3.48 (1H, dd, $J_{2ax,2eq}$10.5 and $J_{2ax,\ 3ax}$ 10.5 Hz, 2ax-H), 3.62-3.80 (3H, m, 8-CH_2 and 2eq-H), 4.87 (1H, t, $J_{5,4}$ 3.1 Hz, 5-H), 4.95-5.11 (1H, m, 3-H).

^{13}C-NMR (50 MHz, $CDCl_3$) 18.1, 24.7, 29.9, 30.2 (CH_2, C-4, C-9, C-10 and C-11), 21.1 (CH_3, Ac), 60.7, (CH_2, C-2), 60.8 (CH, C-3), 64.5 (CH_2, C-2), 72.1 (CH, C-5), 96.1 (quat, C-6), 170.0, 171.2 (quat, 2 x C=O).

CI-MS 273 (M^+H, 12%), 213 (100), 153 (72), 135 (8).

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References

1.Brimble, M. A.; Johnston, A. D. *Molecules* **1997**, *2*, M20.

Sample Availability: No sample available.