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Synthesis of Glucopyranosyl Amides Using Polymer-Supported Reagents

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Abstract

2,3,4,6-Tetra-O-acetyl-β-D-glucopyranosyl azide reacts efficiently with polymer-supported triphenylphosphine and various acid chlorides to yield glucopyranosyl amides with retention of the β-gluco stereochemistry.

Keywords:

Glycosyl amides | Triphenylphosphine | Polymer-bound iminophosphorane | Polymer-supported Reagents

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Notes

[a]All new compounds were homogeneous by TLC and at least 95% pure as indicated by ^{1}H NMR spectra. All compounds gave satisfactory analytical data, including ^{1}H NMR (400 MHz), ^{13}C NMR (100 MHz), and mass spectra. Typical procedure for the formation of glucopyranosyl amides using polymer-supported triphenylphosphine: D-glucosyl azide 7 (100 mg, 0.27 mmol) and p-nitrobenzoyl chloride (0.54 mmol) were dissolved in CH_2Cl_2 (5.0 mL). Polymer-supported triphenylphosphine (~3 mmol/g loading, 116 mg, ~0.35 mmol) was added to the tube, and the mixture was agitated until the release of nitrogen gas had ceased. The mixture was then agitated and refluxed gently for 6 hr. The mixture was cooled, gravity filtered into another test tube to remove polymer-supported triphenyphosphine oxide, which was washed with CH_2Cl_2 (2 × 5 mL). Polystyrene-bound tris(2-aminoethyl) amine (4.0–5.0 mmol/g loading, 200 mg, ~0.88 mmol) was added to the solution, and the mixture was agitated for 2 hr at room temperature. The polymer was removed via gravity filtration, washed with CH_2Cl_2 (2 × 5 mL), and the filtrate was concentrated in vacuo to leave the product residue. Physical characteristics for amide 9a: 400 MHz ^{1}H NMR ($CDCl_3$) δ 2.03, 2.04, 2.05 (3s, 12H total, 4 × $COCH_3$), 3.91 (m, 1H, H-5), 4.09 (dd, 1H, H-6, J = 1.83, 12.45 Hz), 4.31 (dd, 1H, H-6′, J = 4.39, 12.08 Hz), 5.05 (m, 2H, H-3, H-4), 5.39 (m, 2H, H-1, H-2), 7.32 (d, 1H, NH, J = 9.15 Hz), 7.92 (d, 2H, Ar-H), 8.30 (d, 2H, Ar-H). 100 MHz ^{13}C NMR ($CDCl_3$): δ 21.97, 62.63, 69.18, 72.09, 73.41, 74.87, 80.06, 124.96, 129.40, 129.60, 139.08, 151.05, 166.04, 170.77, 171.52, 172.84. Mass calculated: 497.15. Found: 497.18. $[\alpha]_D^{20}$ -19.3 (c 5.1, CH_2Cl_2). TLC R_f-values for glycosyl amides (aluminum-backed silica gel plates using 1:1 EtOAc/hexane as eluent and visualization with 5% H_2SO_4 in ethanol followed by heating on a hot plate): 9a, 0.72; 9b, 0.66; 9c, 0.69; 9d, 0.70; 9e, 0.70; 9f, 0.70; 9h, 0.60; 9j, 0.69; 9k, 0.66; 9l, 0.36.