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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

^aAll new compounds were homogeneous by TLC and at least 95% pure as indicated by ¹H NMR spectra. All compounds gave satisfactory analytical data, including ¹H NMR (400 MHz), ¹³C 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 CH₂Cl₂ (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 triphenylphosphine oxide, which was washed with CH₂Cl₂ (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 CH₂Cl₂ (2 × 5 mL), and the filtrate was concentrated in vacuo to leave the product residue. Physical characteristics for amide **9a**: 400 MHz ¹H NMR (CDCl₃) δ 2.03, 2.04, 2.05 (3s, 12H total, 4 × COCH₃), 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 ¹³C NMR (CDCl₃): 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. [α]_D²⁰ -19.3 (c 5.1, CH₂Cl₂). TLC R_f-values for glycosyl amides (aluminum-backed silica gel plates using 1:1 EtOAc/hexane as eluent and visualization with 5% H₂SO₄ 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.

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