





Home ► All Journals ► Physical Sciences ► Synthetic Communications ► List of Issues ► Volume 34, Issue 13 ► Synthesis of Glucopyranosyl Amides Using

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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 1 H NMR spectra. All compounds gave satisfactory analytical data, including 1 H 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 polymersupported 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 CH₂Cl₂ $(2 \times 5 \text{ mL})$, 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, $4 \times COCH_3$), 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 ¹³C 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, CH₂Cl₂). TLC R _f-values for glycosyl amides (aluminumbacked 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.

6

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