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Synthesis of Glucopyranosyl Amides Using Polymer-Supported Reagents

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Acknow

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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 tempera CH_2Cl_2 (2 × 5 m residue. Physical , 2.05 (3s, 12H tota Hz), 4.31 (dd, 1H, H-2), 7.32 (d, 1H, N NMR ($CDCl_3$): 129.60, 139. Found: 497.1 minum-backed s with 5% H_2SO_4 in 0.69; 9d, 0.70; 9e