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# Synthesis of Glucopyranosyl Amides Using Polymer-Supported Reagents

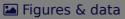
Yuriko Y. Root, Maximillian S. Bailor & Peter Norris

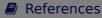
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## **Abstract**

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

Q Keywords: Glycosyl amides

Polymer-s

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The aut work as PACER award. M.S.B. was a summer student who participated in YSU's National Science Foundation-sponsored Research Experience for Undergraduates program.

# Notes

<sup>a</sup>All new compounds were homogeneous by TLC and at least 95% pure as indicated by <sup>1</sup>H NMR spectra. All compounds gave satisfactory analytical data, including <sup>1</sup>H NMR (400 MHz), <sup>13</sup>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 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 CH<sub>2</sub>Cl<sub>2</sub>  $(2 \times 5 \text{ mL})$ , and the filtrate was concentrated in vacuo to leave the product residue. Physical characteristics for amide 9a: 400 MHz <sup>1</sup>H NMR (CDCl<sub>3</sub>) 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  $^{13}$ C NMR (CDCl<sub>3</sub>):  $\delta$  21.97,  $\delta$ 2.63,  $\delta$ 9.18,  $\delta$ 72.09,  $\delta$ 73.41,  $\delta$ 74.87,  $\delta$ 80.06,  $\delta$ 74.96,  $\delta$ 72.40,  $\delta$ 74.87,  $\delta$ 80.06,  $\delta$ 80.06,  $\delta$ 9.18,  $\delta$ 9.18, 139.08, 151.05, 166.04, 170.77, 171.52, 172.84. Mass calculated: 497.15. Found: 497.18.  $[\alpha]_D$  <sup>20</sup> -19.3 (c 5.1, CH<sub>2</sub>Cl<sub>2</sub>). TLC R <sub>f</sub>-values for glycosyl amides (aluminum-

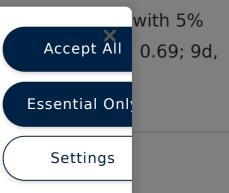
backed s H<sub>2</sub>SO<sub>4</sub> ir 0.70; 9e



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