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Synthesis of Glucopyranosyl Amides Using 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 mixture was cooled, gravity filtered into another test tube to remove the polymer-bound amine, which was washed with CH₂Cl₂ (2 × 5 mL). The combined filtrate and washes were concentrated under reduced pressure to give the residue. Physical data: mp 105–106 °C; IR (KBr): 3400 (broad, OH), 2900 (C–H), 1700 (C=O), 1500 (C=C), 1200 (C–O), 1000 (C–O), 700 (C=C); ¹H NMR (CDCl₃): 12.05 (s, 1H, H-1), 7.32 (d, 1H, H-2), 7.31 (d, 1H, H-3), 7.30 (d, 1H, H-4), 7.29 (d, 1H, H-5), 7.28 (d, 1H, H-6), 7.27 (d, 1H, H-7), 7.26 (d, 1H, H-8), 7.25 (d, 1H, H-9), 7.24 (d, 1H, H-10), 7.23 (d, 1H, H-11), 7.22 (d, 1H, H-12), 7.21 (d, 1H, H-13), 7.20 (d, 1H, H-14), 7.19 (d, 1H, H-15), 7.18 (d, 1H, H-16), 7.17 (d, 1H, H-17), 7.16 (d, 1H, H-18), 7.15 (d, 1H, H-19), 7.14 (d, 1H, H-20), 7.13 (d, 1H, H-21), 7.12 (d, 1H, H-22), 7.11 (d, 1H, H-23), 7.10 (d, 1H, H-24), 7.09 (d, 1H, H-25), 7.08 (d, 1H, H-26), 7.07 (d, 1H, H-27), 7.06 (d, 1H, H-28), 7.05 (d, 1H, H-29), 7.04 (d, 1H, H-30), 7.03 (d, 1H, H-31), 7.02 (d, 1H, H-32), 7.01 (d, 1H, H-33), 7.00 (d, 1H, H-34), 6.99 (d, 1H, H-35), 6.98 (d, 1H, H-36), 6.97 (d, 1H, H-37), 6.96 (d, 1H, H-38), 6.95 (d, 1H, H-39), 6.94 (d, 1H, H-40), 6.93 (d, 1H, H-41), 6.92 (d, 1H, H-42), 6.91 (d, 1H, H-43), 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6.40 (d, 1H, H-94), 6.39 (d, 1H, H-95), 6.38 (d, 1H, H-96), 6.37 (d, 1H, H-97), 6.36 (d, 1H, H-98), 6.35 (d, 1H, H-99), 6.34 (d, 1H, H-100), 6.33 (d, 1H, H-101), 6.32 (d, 1H, H-102), 6.31 (d, 1H, H-103), 6.30 (d, 1H, H-104), 6.29 (d, 1H, H-105), 6.28 (d, 1H, H-106), 6.27 (d, 1H, H-107), 6.26 (d, 1H, H-108), 6.25 (d, 1H, H-109), 6.24 (d, 1H, H-110), 6.23 (d, 1H, H-111), 6.22 (d, 1H, H-112), 6.21 (d, 1H, H-113), 6.20 (d, 1H, H-114), 6.19 (d, 1H, H-115), 6.18 (d, 1H, H-116), 6.17 (d, 1H, H-117), 6.16 (d, 1H, H-118), 6.15 (d, 1H, H-119), 6.14 (d, 1H, H-120), 6.13 (d, 1H, H-121), 6.12 (d, 1H, H-122), 6.11 (d, 1H, H-123), 6.10 (d, 1H, H-124), 6.09 (d, 1H, H-125), 6.08 (d, 1H, H-126), 6.07 (d, 1H, H-127), 6.06 (d, 1H, H-128), 6.05 (d, 1H, H-129), 6.04 (d, 1H, H-130), 6.03 (d, 1H, H-131), 6.02 (d, 1H, H-132), 6.01 (d, 1H, H-133), 6.00 (d, 1H, H-134), 5.99 (d, 1H, H-135), 5.98 (d, 1H, H-136), 5.97 (d, 1H, H-137), 5.96 (d, 1H, H-138), 5.95 (d, 1H, H-139), 5.94 (d, 1H, H-140), 5.93 (d, 1H, H-141), 5.92 (d, 1H, H-142), 5.91 (d, 1H, H-143), 5.90 (d, 1H, H-144), 5.89 (d, 1H, H-145), 5.88 (d, 1H, H-146), 5.87 (d, 1H, H-147), 5.86 (d, 1H, H-148), 5.85 (d, 1H, H-149), 5.84 (d, 1H, H-150), 5.83 (d, 1H, H-151), 5.82 (d, 1H, H-152), 5.81 (d, 1H, H-153), 5.80 (d, 1H, H-154), 5.79 (d, 1H, H-155), 5.78 (d, 1H, H-156), 5.77 (d, 1H, H-157), 5.76 (d, 1H, H-158), 5.75 (d, 1H, H-159), 5.74 (d, 1H, H-160), 5.73 (d, 1H, H-161), 5.72 (d, 1H, H-162), 5.71 (d, 1H, H-163), 5.70 (d, 1H, H-164), 5.69 (d, 1H, H-165), 5.68 (d, 1H, H-166), 5.67 (d, 1H, H-167), 5.66 (d, 1H, H-168), 5.65 (d, 1H, H-169), 5.64 (d, 1H, H-170), 5.63 (d, 1H, H-171), 5.62 (d, 1H, H-172), 5.61 (d, 1H, H-173), 5.60 (d, 1H, H-174), 5.59 (d, 1H, H-175), 5.58 (d, 1H, H-176), 5.57 (d, 1H, H-177), 5.56 (d, 1H, H-178), 5.55 (d, 1H, H-179), 5.54 (d, 1H, H-180), 5.53 (d, 1H, H-181), 5.52 (d, 1H, H-182), 5.51 (d, 1H, H-183), 5.50 (d, 1H, H-184), 5.49 (d, 1H, H-185), 5.48 (d, 1H, H-186), 5.47 (d, 1H, H-187), 5.46 (d, 1H, H-188), 5.45 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