Electronic Supplementary Information for:
Direct Formation of β-Glycosides of N-Acetyl Glycosamines
Mediated by Rare Earth Metal Triflates

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2-Bromoethyl 2-acetamido-3,4,6-tri-O-acetyl-2-deoxy-β-D-glucopyranoside (7).
Donor 3 (197 mg, 0.51 mmol) and 2-bromoethanol (6) (0.11 mL, 1.54 mmol) were coupled according to the general procedure with Sc(OTf)₃ (37 mg, 0.075 mmol) as catalyst in dry CH₂Cl₂ (3 mL). After 27 h TLC analysis indicated no further reaction. The reaction mixture was worked up as described in the general procedure and purified by column chromatography (1:2 pentane/EtOAc) to give the desired glycoside 7 (153 mg, 67 %) as white crystals. Rₕ (EtOAc): 0.50; Mp(uncorr.): 166-167 °C (EtOAc/pentane). ¹H-NMR (CDCl₃, 400 MHz): δ 5.45 (d, 1H, JₙH₂ 8.4 Hz, NH), 5.31 (t, 1H, J₂₃, J₃₄ 9.6 Hz, H₃), 5.06 (t, 1H, H₄), 4.77 (d, 1H, J₁₂ 8.4 Hz, H-1), 4.25 (dd, 1H, J₅₆ 4.8 Hz, J₆₅, J₆₆ 12.2 Hz, H₆), 4.19-4.12 (m, 2H, OCH₂CH₂Br), 3.90-3.80 (m, 2H, H₂, OCH₂CH₂Br), 3.71 (ddd, 1H, J₆₅, J₆₆ 2.2 Hz, H₅), 3.49-3.46 (m, 2H, OCH₂CH₂Br), 2.09, 2.03, 2.02,, 1.97 (s, 12H, COC₃). ¹³C-NMR (CDCl₃, 100 MHz): δ 171.1, 170.9, 170.7, 169.6 (CO), 101.2 (C1), 72.4, 72.2 (C3, C5), 69.7, 68.8 (C2, C4), 62.3 (C6), 54.9 (OCH₂CH₂Br), 30.8 (OCH₂CH₂Br), 23.6, 21.0, 20.9, 20.8 (C(O)CH₃); HRMS: calcd for C₁₆H₂₄NO₉⁻BrNa: 476.0532, found: 476.0533. [α]D²¹ -11.8 (c 1.0, CHCl₃).

Cyclohexyl 2-acetamido-3,4,6-tri-O-acetyl-2-deoxy-β-D-glucopyranoside (9).
Donor 3 (200 mg, 0.51 mmol) and cyclohexanol (8) (0.16 mL, 1.52 mmol), freshly distilled from sodium, were coupled according to the general procedure with Sc(OTf)₃ (37 mg, 0.076 mmol) as catalyst in dry CH₂Cl₂ (2.5 mL). After 48 h TLC analysis indicated no further reaction. The reaction
mixture was worked up as described in the general procedure and purified by column chromatography (1:3 pentane/EtOAc → EtOAc) to give the desired cyclohexyl glycoside 9 (184 mg, 84 %) as colourless crystals. \(R_f\) (EtOAc): 0.53. Mp(uncorr.): 180-181 °C (EtOAc/pentane); LRMS(ES+): calcld. for C\(_{20}\)H\(_{24}\)NO\(_9\)Na: 452.2, found: 452.0. Spectral data was in accordance with previously reported results.i

\[N-(9-Fluorenylmethoxycarbonyl)-(2-acetamido-2-deoxy-3,4,6-tri-O-acetyl-\beta-D-glucopyranosyl)-L-serine methyl ester (11).\]

Donor 3 (129 mg, 0.33 mmol) and \(N-(9\text{-fluorenylmethyl-oxycarbonyl})\)-L-serine methyl ester (10) (169 mg, 0.50 mmol) were coupled according to the general procedure with Sc(OTf)\(_3\) (25 mg, 0.050 mmol) as catalyst in dry CH\(_2\)Cl\(_2\) (3 mL). After 72 h TLC analysis indicated no further reaction. The reaction mixture was worked up as described in the general procedure and purified by column chromatography (1:4 pentane/EtOAc → EtOAc) which gave glycoside 11 (111 mg, 50 %) as pale yellow crystals. \(R_f\) (EtOAc): 0.56; Mp(uncorr.): 162-165 °C (EtOAc/pentane); \(^1\)H-NMR (CDCl\(_3\), 400 MHz): \(\delta\) 7.72 (dd, 2H, \(J\) 3.4 Hz, \(J\) 7.4 Hz, Fmoc Ph), 7.58 (d, 2H, \(J\) 5.0 Hz, Fmoc Ph), 7.34 (t, 2H, \(J\) 7.4 Hz, Fmoc Ph), 7.27 (t, 2H, \(J\) 7.4 Hz, Fmoc Ph), 5.67 (d, 1H, \(J_{\text{NH,CH-Ser}}\) 7.6 Hz, NH-Ser), 5.41 (d, 1H, \(J_{\text{NH,2}}\) 8.0 Hz, NHAc), 5.17 (t, 1H, \(J_{2,3} = J_{3,4}\) 10.0 Hz, H3), 4.97 (t, 1H, H4), 4.56 (d, 1H, \(J_{1,2}\) 8.4 Hz, H1), 4.43-4.38 (m, 3H, CH-Ser, CH\(_2\)Fmoc), 4.20-4.12 (m, 3H, H6a, CH Fmoc, CHH-Ser), 4.05 (dd, 1H, \(J_{5,6b}\) 2.4 Hz, \(J_{5a,6b}\) 12.4 Hz, H6b), 3.79-3.71 (m, 2H, H2, CHH-Ser), 3.70 (s, 3H, CO\(_2\)CH\(_3\)), 3.60 (ddd, 1H, \(J_{5,6a}\) 4.8 Hz, H5), 2.01, 1.97, 1.96, 1.78 (s, 12H, COCH\(_3\)). \(^{13}\)C-NMR (CDCl\(_3\), 100 MHz): \(\delta\) 171.1, 170.9, 170.4, 169.6 (COCH\(_3\), CO\(_2\)CH\(_3\)), 156.3 (CO Fmoc), 144.1, 143.9, 141.6 (C-ipso Fmoc), 128.0, 127.4, 125.4, 120.3 (Ar Fmoc), 101.0 (C1), 72.4, 72.2 (C3, C5), 69.1, 68.7 (C4, CH\(_2\)-Ser), 67.1 (CH\(_2\) Fmoc), 62.3 (C6), 54.6, 54.4 (C2, CH-Ser), 53.0 (CO\(_2\)CH\(_3\)), 47.4 (CH Fmoc), 23.4, 20.9, 20.9, 20.8 (COCH\(_3\)); HRMS(ES+): calcd. for C\(_{33}\)H\(_{38}\)N\(_2\)O\(_{13}\)Na: 693.2272; found: 693.2243. \([\alpha]_D^{21}\) 3.5 (c 1.0, CHCl\(_3\)).
O-(2-Acetamido-3,4,6-tri-O-acetyl-2-deoxy-D-glucopyranosyl)-β-(1-6)-1,2;3,4-di-O-isopropylidene-α-D-galactopyranose (13).

Donor 3 (734 mg, 1.89 mmol) and 1,2;3,4-di-O-isopropylidene-α-D-galactopyranose (12) (220 mg, 0.85 mmol) were coupled according to the general procedure with Sc(OTf)₃ (63 mg, 0.13 mmol) as catalyst in dry CH₂Cl₂ (3 mL). After 20 h TLC analysis indicated no further reaction. The reaction mixture was worked up as described in the general procedure and purified by column chromatography (acetone/pentane 2:5) to give the desired disaccharide 13 (0.387 g, 78%) as a white powder. Rᵉ(acetone/pentane 4:5) 0.47; Mp(uncorr.): 72-74 °C (acetone/pentane). ¹H-NMR (CDCl₃, 400 MHz): δ 5.59 (d, 1H, J₉H₂ 8.8 Hz, NHAc), 5.50 (d, 1H, J₁₂H₂ 5.0 Hz, H1), 5.14 (t, 1H, J₂₂,₃₃ = J₁₂,₁₃ 5.0 Hz, H1'), 4.68 (d, 1 H, J₂₂,₃₃ 8.4 Hz, H3'), 4.04-3.92 (m, 3H, H2', H6a, H5'), 3.72 (dd, 1H, J₅₆b 12.4 Hz, J₆₆a 12.4 Hz, H6'a), 4.14-4.09 (m, 2H, H4, H6'b), 4.04-3.92 (m, 3H, H2', H6a, H5'), 3.72 (dd, 1H, J₆₆b,₆₆a 13.2 Hz, J₅₆b 9.2 Hz, H6b), 3.66 (dd, 1H, J₅₆b,₆₆a 12.4 Hz, H5'), 2.06, 2.00, 1.99, 1.94 (s, 12H, O₂CMeCH₃), 1.42 (s, 3H, O₂CCH₃Me), 1.30 (s, 6H, O₂CMeCH₃); ¹³C-NMR (CDCl₃, 100 MHz) δ 171.1, 170.9, 170.6, 169.5 (CO), 109.6, 108.8 ((CH₃)₂CO₂), 102.1 (C1'), 96.4 (C1), 73.4 (C3'), 72.0 (C5'), 71.2 (C4), 70.8 (C3), 70.5 (C2), 69.2 (C6), 68.7 (C4'), 68.5 (C2'/C5), 62.3 (C6'), 54.3 (C2'/C5), 26.3, 26.1, 25.2, 24.5, 23.5, 20.9, 20.9, 20.8 (C(O)CH₃); [α]D²¹ -51.3 (c 1.0, CHCl₃). Spectral data was in accordance with previously reported results.\(^{ii}\)

Methyl O-(2-acetamido-3,4,6-tri-O-acetyl-2-deoxy-β-D-glucopyranosyl-(1→6)-2,3,4-tri-O-benzyl-α-D-galactopyranoside (15):

Donor 3 (210 mg, 0.54 mmol, microwave heating; 410 mg, 0.9 mmol, conventional heating), methyl glucopyranoside 14 (100 mg, 0.22 mmol), and Sc(OTf)₃ (16 mg, 0.023 mmol) was dissolved in dry CH₂Cl₂ (2 mL) and heated to 80°C for 3 h. by microwave irradiation or to reflux for 72 h by conventional heating. The mixture was diluted with CH₂Cl₂ and washed with water followed by extraction with CH₂Cl₂ as described in the general procedure. The combined organic extracts were dried over MgSO₄ and purified by column chromatography (EtOAc/pentane 1:1 → 1:0) to give the desired disaccharide.
16 (128 mg, 75 %, microwave heating; 145 mg, 85% conventional heating) as a white solid. $R_f$(EtOAc) 0.6. $^1$H-NMR (400 MHz, CDCl$_3$): $\delta$ 7.29-7.19 (m, 15H, ArH), 5.33 (d, 1H, $J_{NH,2}$ 8.4 Hz, NH), 5.20 (t, 1H, $J_{2,3} = J_{3,4}$ 10.0 Hz, H3'), 4.97 (t, 1H, H4'), 4.90 (d, 1H, $J$ gem 10.8 Hz, PhCHH), 4.76 (d, 1H, $J$ gem 10.8 Hz, PhCHH), 4.72 (d, 1H, $J$ gem 10.8 Hz, PhCHH), 4.61 (d, 1H, $J_{1',2'}$ 8.0 Hz, H1'), 4.58 (d, 1H, $J$ gem 12.0 Hz, PhCHH), 4.52 (d, 1H, $J_{1,2}$ 3.2 Hz, H1), 4.50 (d, 1H, $J$ gem 10.8 Hz, PhCHH), 4.14 (dd, 1H, $J_{5',6'}$ 4.8 Hz, H6'a), 4.02 (dd, 1H, $J_{5',6'b}$ 2.4 Hz, H6'b), 3.98 (d, 1H, $J_{8a,6b}$ 10.2 Hz, H6a), 3.90 (t, 1H, H3), 3.78 (q, 1H, H2'), 3.70-3.57 (m, 3H, H5', H5, H6b), 3.44 (dd, 1H, $J_{2,3}$ 9.6 Hz, H2), 3.40 (t, 1H, $J_{3,4}$ 9.6 Hz, H4), 3.28 (s, 3H, OC$_3$H$_3$), 1.94, 1.74 (s, 12H, C(O)CH$_3$). $^{13}$C-NMR (100 MHz, CDCl$_3$) $\delta$ 171.0, 169.6 (CO), 139.0, 138.3, 128.7, 128.1, 127.8 (Ar), 100.8 (C1'), 98.3 (C1), 82.2 (C3), 80.0 (C2), 77.6 (C4), 75.9 (PhCH$_2$), 74.8 (PhCH$_2$), 73.6 (PhCH$_2$), 72.5 (C3'), 72.1, 69.7 (C5, C5'), 68.9 (C4'), 67.9 (C6), 62.4 (C6'), 55.4 (OCH$_3$), 54.9 (C2'), 23.5, 20.9, 20.9 (C(O)CH$_3$). Spectral data is in accordance with previously reported values.$^{iii}$

Methyl O-(2-acetamido-3,4,6-tri-O-acetyl-2-deoxy-β-D-glucopyranosyl)-(1→4)-2,3,6-tri-O-benzyl-α-D-glucopyranoside (17).

Acceptor 16 (103 mg, 0.22 mmol) and donor 3 (345 mg, 0.89 mmol) were coupled according to the general procedure with Sc(OTf)$_3$ (66 mg, 0.13 mmol) as catalyst in dry CH$_2$Cl$_2$. After 72 h TLC analysis indicated no further reaction. The reaction mixture was worked up as described in the general procedure and purified by column chromatography (EtOAc/pentane 1:5 → EtOAc) to give the desired disaccharide 17 (37 mg, 21%) and unreacted acceptor (16) (68 mg). $R_f$(EtOAc) 0.55. $^1$H-NMR (CDCl$_3$, 400 MHz) $\delta$ 7.44-7.15 (m, 15H, Ar-H), 4.92 (d, 1H, $J$ 11.3 Hz, PhCHHa), 4.87 (m, 1H, H4'), 4.82 (m, 1H, H3'), 4.77 (d, 1H, $J$ 12.2 Hz, PhCHHb), 4.70 (d, 1H, PhCHHb), 4.64 (d, 1H, $J$ 12.0, PhCHHc), 4.57 (d, 1H, PhCHHc), 4.56 (d, 1H, $J_{1,2}$ 3.6 Hz, H1), 4.53 (d, 1H, $J_{NH,2}$ 9.2 Hz, NH), 4.36 (d, 1H, $J_{1',2'}$ 8.4 Hz, H1'), 4.31 (d, 1H, PhCHHb), 4.02 (dd, 1H, $J_{5,6'a}$ 4.4 Hz, $J_{6a,6b}$ 12.4 Hz, H6a'), 3.80-3.72 (m, 4H, H2', H3, H4, H6b'), 3.59-3.53 (m, 2H, H5, H6a), 3.43-3.34 (m, 3H, H2, H5', H6b), 3.29 (s, 3H, OCH$_3$), 1.93, 1.91, 1.86, 1.64 (s, 12 H, C(O)CH$_3$). $^{13}$C-NMR
(CDCl₃, 100 MHz) δ 170.9, 170.8, 170.0, 169.6 (CO), 139.8, 138.5, 138.0, 129.2, 129.1, 129.0, 128.6, 128.4, 128.3, 128.0, 127.3, 127.2 (Ar), 100.7 (C1'), 98.6 (C1), 80.3 (C3), 79.1 (C2), 77.5 (C4), 75.2, 74.0, 73.7 (PhCH₂), 73.1 (C3'), 71.6 (C5') 69.6 (C5), 68.7 (C4'), 67.8 (C6), 62.1 (C6'), 55.6 (OCH₃), 54.8 (C2'), 23.4 (NHC(O)CH₃), 20.8 (OC(O)CH₃). [α]D²² 14 (c 1.0, CHCl₃); HRMS(ES+): calcd for C₄₂H₅₁NO₁₄Na: 816.3208; found, 816.3203.

Methyl O-(2-acetamido-3,4,6-tri-O-acetyl-2-deoxy-β-D-glucopyranosyl)-(1→4)-2,3,6-tri-O-benzyl-α-D-glucopyranoside (17) by microwave irradiation.

Donor 3 (168 mg, 0.43 mmol) and methyl glucoside 16 (100 mg, 0.23 mmol) were dissolved in CH₂Cl₂ (1.2 mL) before Sc(OTf)₃ (16 mg, 0.032 mmol) was added. The reaction mixture was heated by microwave irradiation to 80 °C for 8 h. After this period of time more donor (3) (167 mg, 0.43 mmol) and Sc(OTf)₃ (16 mg, 0.032 mmol) were added and the mixture heated as before for another 8 h. The reaction mixture was loaded directly onto a column of silicagel and purified as mentioned above. This gave desired disaccharide (17) (73 mg, 43%) and unreacted acceptor (16) (37 mg, recovered yield of 17: 68%).

Allyl 2,3,6-tri-O-benzyl-α-D-galactopyranoside (19):

To a stirred solution of benzylidene 24 (1.33 g, 2.7 mmol) in CH₂Cl₂ (10 mL) was added triethylsilane (3.55 mL, 22.2 mmol) and trifluoroacetic acid (1.83 mL, 24.6 mmol). The mixture was stirred for 5 min at 0 °C and then at ambient temperature for 24 h before neutralised with NaHCO₃ (sat. aq) and extracted with CH₂Cl₂. The resulting product was purified by column chromatography (silica, EtOAc/pentane 1:4). This gave the desired tribenzylated galactoside 19 as a colourless oil (813 mg, 61 %). Rf(EtOAc/pentane 1:4) 0.20. ¹H-NMR (CDCl₃, 400 MHz) δ 7.39-7.26 (m, 15H, ArH), 5.99-5.90 (m, 1H, CH₂CH=CH₂), 5.32 (dq, J 1.6 Hz, Jvic 17.2 Hz, CH=CHH), 5.21 (b d, Jvic 10.0 Hz, CH=CHH), 4.88 (d, 1H, J1,2 4.8 Hz, H1), 4.81 (d, 1H, Jgem 11.6 Hz, PhCHH), 4.80 (d, 1H, Jgem 12.0 Hz, PhCHH), 4.71 (d, 1H, Jgem 11.6 Hz, PhCHH), 4.66 (d, 1H, Jgem 12.0 Hz, PhCHH), 4.60 (d,
1H, $J_{\text{gem}}$ 12.0 Hz, PhCH(OH)O), 4.56 (d, 1H, PhCH(OH)O), 4.18 (ddt, $J_{\text{vic}}$ 5.2 Hz, $J_{\text{gem}}$ 13.2 Hz, OCHHCHCH$_2$), 4.09 (b s, 1H, H4), 4.04 (ddt, 1H, $J_{\text{vic}}$ 6.8 Hz, OCHHCHCH$_2$), 3.97 (b t, 1H, $J_{5,6a} = J_{5,6b}$ 5.6 Hz, H5), 3.93-3.86 (m, 2H, H2, H3), 3.74 (dd, 1H, $J_{6a,6b}$ 10.0 Hz, H6a), 3.68 (dd, 1H, H6b). $^{13}$C-NMR (100 MHz, CDCl$_3$) $\delta$ 138.8, 138.5, 138.4, 128.7, 128.6, 128.6, 128.2, 128.1, 127.9 (Ar), 134.2 (CH$_2$), 118.3 (CH$_2$), 96.5 (C1), 78.0, 76.1 (C2, C3), 73.8, 73.6, 73.1 (PhCH$_2$), 69.9 (C6), 68.9 (C5), 68.6 (CH$_2$CH=CH$_2$), 68.4 (C4). HRMS(ES+): calcd. for C$_{30}$H$_{34}$O$_6$Na: 513.2253; found: 513.2258. Spectral data was largely in accordance with previously published values.$^v$

**O-(2-acetamido-2-deoxy-β-D-glucopyranosyl)-(1→3)-2-acetamido-2-deoxy-α/β-D-glucopyranose (26):**

A balloon of H$_2$-gas was applied to a vigorously stirred solution of protected disaccharide 23 (120 mg, 0.16 mmol) and Pd(OH)$_2$/C (10%, 25 mg) in acetic acid (2 mL). The reaction mixture was stirred overnight before the catalyst was removed by filtration through a bed of Celite®. The remaining acetic acid was carefully removed by repeated co-evaporation with toluene before the residue was dissolved in methanol (3 mL) to which had been added a catalytic amount of Na-metal. The reaction mixture was stirred overnight at 40 °C before diluted with H$_2$O (5 mL) and neutralised with Amberlite (IR-120 H+). The solvents were removed under reduced pressure and the crude product underwent $^{13}$C-NMR analysis (D$_2$O, 100 MHz) to confirm the linkage being 1→3.$^\text{Error! Bookmark not defined.}$

**Benzyl 2-acetamido-3,4,6-tri-O-acetyl-2-deoxy-α/β-D-glucopyranoside from anomeric mixture of donors (5αβ):**

An anomeric mixture of D-αβ-N-acetyl glucosamine tetraacetate (3αβ) (α/β 4.6:1, 200 mg, 0.51 mmol), BnOH (4) (0.16 mL, 1.54 mmol), and Sc(OTf)$_3$ were dissolved in CH$_2$Cl$_2$ (1.45 mL). The mixture was heated to 110 °C under microwave conditions for 5 hours. The reaction mixture was diluted with CH$_2$Cl$_2$, washed with NaHCO$_3$ (sat. aq.), and dried (MgSO$_4$) before purified by
column chromatography (EtOAc/pentane 1:1 → 2:1) to give an anomeric mixture of benzyl glycosides 5αβ (α/β 21/79, 180 mg, 80%).

Data for α-anomer (less polar than β-anomer): $R_f$(EtOAc) 0.61. $^1$H-NMR (400 MHz, CDCl$_3$) $\delta$ 7.40-7.31 (m, 5H, ArH), 5.63 (d, 1H, $J_{2,NH}$ 9.2 Hz, NH), 5.23 (t, 1H, $J_{2,3;3,4}$ 10.2 Hz, H3), 5.13 (t, 1H, H4), 4.94 (d, 1H, $J_{1,2}$ 3.6 Hz, H1), 4.71 (d, 1H, $J_{gem}$ 11.6 Hz, PhCH$_2$O), 4.52 (d, 1H, PhCH2O), 4.35 (ddd, 1H, H6a), 4.23 (dd, 1H, $J_{5,6a}$ 4.4 Hz, $J_{6a,6b}$ 12.4 Hz, H6a), 4.03 (dd, 1H, $J_{5,6b}$ 2.4 Hz, H6b), 3.99-3.95 (m, 1H, H5), 2.10,  2.01, 2.00, 1.89 (s, 12H, C(O)C$_3$).

$^{13}$C-NMR (100 MHz, CDCl$_3$) $\delta$ 171.6, 170.9, 170.1, 169.5 (CO), 136.7, 128.9, 128.8, 128.5 (Ar), 96.9 (C1), 71.5 (C3), 70.5 (PhCH$_2$O), 68.3, 68.2 (C4, C5), 62.1 (C6), 52.0 (C2), 23.4, 21.0, 20.9, 20.8 (C(O)CH$_3$). $[^{\alpha}]$D$_{21}$ 113 (c 1.0, CHCl$_3$), HRMS(ES+): calcd. for C$_{21}$H$_{27}$NO$_9$Na: 460.1584; found: 460.1594.

**Treatment of benzyl 2-acetamido-3,4,6-tri-O-acetyl-2-deoxy-β-D-glucopyranose (5) with either Sc(OTf)$_3$ or acetic acid:**

To benzyl glucoside (5) (100 mg, 0.23 mmol) in CH$_2$Cl$_2$ (0.75 mL) was added either Sc(OTf)$_3$ (17 mg, 0.034 mmol) or CH$_3$CO$_2$H (13 μL, 0.23 mmol) before the reaction mixture was heated to 110 °C under microwave irradiation for 5 h. The reaction mixture was then diluted with CH$_2$Cl$_2$ (20 mL) and washed with NaHCO$_3$ (aq. sat. 20 mL), dried (MgSO$_4$), concentrated under reduced pressure (full recovery of material) and analysed by NMR spectroscopy to be starting material 5.

**Treatment of benzyl 2-acetamido-3,4,6-tri-O-acetyl-2-deoxy-β-D-glucopyranose (5) with Sc(OTf)$_3$ and acetic acid:**

To benzyl glucoside (5) (100 mg, 0.23 mmol) in CH$_2$Cl$_2$ (0.75 mL) was added Sc(OTf)$_3$ (17 mg, 0.034 mmol) and CH$_3$CO$_2$H (13 μL, 0.23 mmol) before the reaction mixture was heated to 110 °C under microwave irradiation for 5 h. The reaction mixture was then diluted with CH$_2$Cl$_2$ (20 mL) and washed with NaHCO$_3$ (aq. sat. 20 mL), dried (MgSO$_4$), concentrated under reduced pressure (near full recovery of material) and analysed by NMR spectroscopy to be an anomeric mixture of benzyl glycosides (5αβ).