

Electronic Supplementary Information

Rapid Glycosylation of 2'-Benzoylphenyl Glycosides Promoted by TfOH

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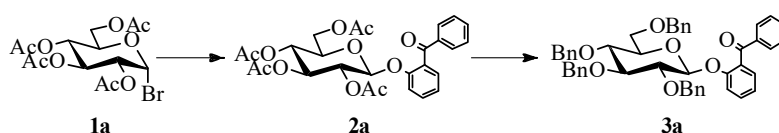
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1. Experimental Section

All solvents were dried before use according to standard methods. Commercial reagents were used without further purification unless otherwise noted. Reactions were monitored by analytical thin-layer chromatography (TLC) on silica gel-coated aluminium plates (60 F₂₅₄). Spots were detected under UV light (254 nm) and/or charring with a solution of (NH₄)₆Mo₇O₂₄•4H₂O (24.00 g, 19.4 mmol) and Ce(NH₄)₂(NO₃)₆ (0.50 g, 0.90 mmol) in sulfuric acid (5%, 500 mL). Column chromatography was performed on silica gel (200-300 mesh). ¹H NMR spectra were recorded at room temperature in CDCl₃ with TMS (δ = 0 ppm) as internal standard. ¹³C NMR spectra were obtained by using the same NMR spectrometer and were calibrated with CDCl₃ (δ = 77.00 ppm). The following standard abbreviations are used to indicate multiplicity: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublets, dt = doublet of triplets, and br = broad. High resolution mass spectra were performed on a Waters Xevo G2 Q-TOF mass spectrometer.

1) Synthesis of the donors 3a-3f

2'-Benzoylphenyl 2,3,4,6-tetra-O-benzyl-β-D-glucopyranoside (3a)

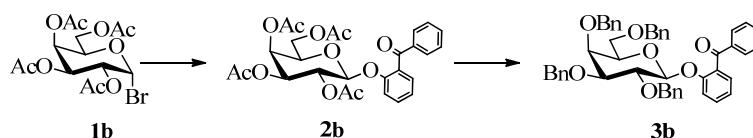


To a solution of **1a** (14.84 mmol, 6.10 g) in dichloromethane (DCM) (60 mL) and water (52.5 mL), 2-hydroxybenzophenone (29.68 mmol, 5.8 g), 2M NaOH solution (7.5 mL) and tetrabutylammonium bromide (14.84 mmol, 4.70 g) were added. The reaction mixture was stirred at room temperature for 6 h and monitored by TLC. The reaction mixture was extracted with DCM for three times. The combined organic phase was washed with water and brine, dried over Na₂SO₄, filtered and the filtrate was concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (petroleum ether: ethyl acetate = 3:1, v/v) to afford **2a** as a white solid (4.00 g, 51%). ¹H NMR (400 MHz, CDCl₃) δ 7.78 (dd, *J* = 8.4, 0.9 Hz, 2H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.47 – 7.41 (m, 3H), 7.33 (dd, *J* = 7.5, 1.6 Hz, 1H), 7.24 – 7.16 (m, 2H), 5.18 (t, *J* = 9.3 Hz, 1H), 5.10 – 5.00 (m, 2H), 4.93 (dd, *J* = 9.3, 7.9 Hz, 1H), 4.26 (dd, *J* = 12.3, 5.3 Hz, 1H), 4.15 (dd, *J* = 12.2, 2.4 Hz, 1H), 3.85 – 3.78 (m, 1H), 2.09 (s, 3H), 2.02 (s, 3H), 1.95 (s, 3H), 1.90 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 195.32, 170.50, 170.10, 169.32, 168.96, 153.95, 137.01, 133.38, 131.40, 131.07, 129.98, 128.99, 128.34, 123.53, 117.20, 99.74, 72.61, 72.00, 70.60, 68.18, 61.92, 20.70, 20.56, 20.52, 20.36. HRMS (ESI): Calcd for C₂₇H₃₂NO₁₁ [M+NH₄]⁺ 546.1970, found 546.1971.

To a solution of **2a** (7.56 mmol, 4.00 g) in dry MeOH (60 mL), NaOMe was added until the pH was 10. The reaction mixture was stirred at room temperature for 1 h, neutralized with acidic resin, filtered and the filtrate was concentrated. The residue was dissolved in DMF (40 mL), stirred in an ice bath, 60% sodium hydride (1.36 g, 45.36 mmol) was added at 0 °C, benzyl bromide (5.4 mL, 45.36 mmol) was then added dropwise. The reaction mixture was stirred for 12 h at room temperature, monitored by TLC, quenched by dropwise addition of methanol at 0 °C. The solvent was evaporated and concentrated. The residue was extracted with DCM for three times and the organic

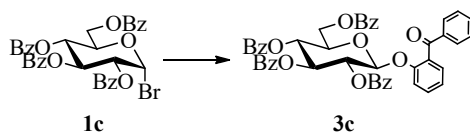
phases were combined. The combined organic phase was washed with water and brine, dried over Na₂SO₄, filtered and the filtrate was concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (petroleum ether: ethyl acetate = 8:1, v/v) to afford **3a** as colorless oil (5.18 g, 95%). ¹H NMR (400 MHz, CDCl₃) δ 7.83 (d, *J* = 7.2 Hz, 2H), 7.49 (t, *J* = 7.4 Hz, 1H), 7.45 – 7.39 (m, 2H), 7.35 (t, *J* = 7.8 Hz, 4H), 7.31 – 7.10 (m, 20H), 5.02 (d, *J* = 7.7 Hz, 1H), 4.84 (d, *J* = 10.9 Hz, 1H), 4.79 (d, *J* = 10.9 Hz, 1H), 4.73 (d, *J* = 10.9 Hz, 1H), 4.57 (d, *J* = 12.0 Hz, 1H), 4.54 (d, *J* = 11.6 Hz, 2H), 4.48 (d, *J* = 11.3 Hz, 1H), 4.31 (d, *J* = 10.9 Hz, 1H), 3.75 (d, *J* = 10.90 Hz, 1H), 3.70 – 3.62 (m, 2H), 3.59 – 3.52 (m, 2H), 3.31 (dd, *J* = 8.7, 8.0 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 196.02, 154.46, 138.51, 138.31, 138.18, 138.10, 137.78, 133.18, 132.03, 130.23, 129.99, 129.75, 128.53, 128.46, 128.36, 128.24, 128.05, 127.93, 127.89, 127.75, 127.72, 127.70, 127.08, 122.41, 115.60, 100.70, 84.59, 81.85, 77.73, 75.79, 75.38, 75.12, 74.91, 73.54, 68.95. HRMS (ESI): Calcd for C₄₇H₄₈NO₇ [M+NH₄]⁺ 738.3430, found 738.3431.

2'-Benzoylphenyl 2,3,4,6-tetra-*O*-benzyl-β-D-galactopyranoside (**3b**)



The same procedure for the synthesis of **3a** was applied, yielding **3b** (0.64 g, 71%) as colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.85 – 7.74 (m, 2H), 7.52 – 7.37 (m, 3H), 7.34 – 7.24 (m, 17H), 7.24 – 7.19 (m, 4H), 7.19 – 7.13 (m, 2H), 7.10 (t, *J* = 7.4 Hz, 1H), 4.96 (d, *J* = 7.6 Hz, 1H), 4.91 (d, *J* = 11.6 Hz, 1H), 4.73 – 4.63 (m, 2H), 4.55 (d, *J* = 11.6 Hz, 1H), 4.50 – 4.39 (m, 3H), 4.36 (d, *J* = 10.7 Hz, 1H), 3.86 (d, *J* = 2.8 Hz, 1H), 3.69 – 3.64 (m, 1H), 3.63 (d, *J* = 2.9 Hz, 1H), 3.60 (d, *J* = 5.4 Hz, 1H), 3.60 – 3.55 (m, 1H), 3.52 (dd, *J* = 9.7, 2.9 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 196.04, 154.65, 138.74, 138.53, 138.44, 138.05, 137.78, 133.07, 131.88, 130.26, 129.76, 128.58, 128.50, 128.38, 128.35, 128.27, 128.17, 127.97, 127.95, 127.75, 127.69, 127.57, 122.22, 115.79, 101.20, 82.03, 78.89, 77.48, 76.84, 75.23, 74.62, 74.10, 73.73, 73.69, 73.27, 69.05. HRMS (ESI): Calcd for C₄₇H₄₈NO₇ [M+NH₄]⁺ 738.3431, found 738.3425.

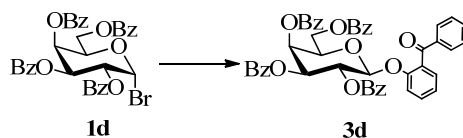
2'-Benzoylphenyl 2,3,4,6-tetra-*O*-benzyl-β-D-glucopyranoside (**3c**)



To a solution of **1c** (6.44 mmol, 4.25 g) in DCM (40 mL) and water (32.5 mL), 2-hydroxybenzophenone (12.88 mmol, 2.55 g), 2M NaOH solution (7.5 mL) and tetrabutylammonium bromide (12.88 mmol, 4.08 g) were added under an atmosphere of argon. The reaction mixture was stirred at room temperature for 6 h and monitored by TLC, extracted with DCM for three times. The combined organic phase was washed with water and brine, dried over Na₂SO₄, filtered and the filtrate was concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (petroleum ether: ethyl acetate = 6:1, v/v) to afford **3c** as a white solid (4.00 g, 48%).

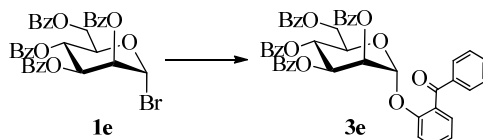
^1H NMR (400 MHz, CDCl_3) δ 8.04 (d, $J = 7.5$ Hz, 2H), 7.90 (d, $J = 7.6$ Hz, 2H), 7.81 – 7.56 (m, 4H), 7.56 (d, $J = 7.7$ Hz, 3H), 7.52 – 7.15 (m, 17H), 7.11 (t, $J = 7.4$ Hz, 1H), 5.89 (t, $J = 9.3$ Hz, 1H), 5.62 (t, $J = 9.6$ Hz, 1H), 5.52 – 5.35 (m, 2H), 4.64 (dd, $J = 12.1, 3.0$ Hz, 1H), 4.51 (dd, $J = 12.1, 6.4$ Hz, 1H), 4.32 – 4.25 (m, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 195.19, 166.04, 165.70, 165.26, 164.54, 153.83, 136.90, 133.59, 133.30, 133.13, 131.41, 131.36, 129.91, 129.84, 129.80, 129.79, 129.64, 129.25, 129.07, 128.73, 128.50, 128.31, 128.17, 123.67, 117.73, 99.91, 72.86, 72.63, 71.35, 69.57, 63.17. HRMS (ESI): Calcd for $\text{C}_{47}\text{H}_{40}\text{NO}_{11}$ $[\text{M}+\text{NH}_4]^+$ 794.2601, found 794.2592.

2'-Benzoylphenyl 2,3,4,6-tetra-*O*-benzoyl- β -D-galactopyranoside (3d)



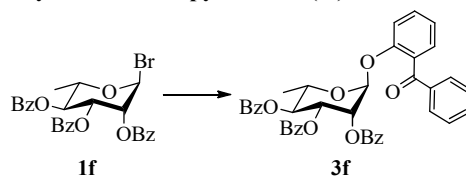
The same procedure for the synthesis of **3c** was applied, yielding **3d** (0.64 g, 71%) as a white solid. ^1H NMR (400 MHz, CDCl_3) δ 8.08 – 8.00 (m, 4H), 7.81 – 7.70 (m, 2H), 7.64 – 7.56 (m, 2H), 7.64 – 7.56 (m, 4H), 7.51 – 7.43 (m, 6H), 7.42 – 7.17 (m, 10H), 7.14 (t, $J = 7.4$ Hz, 1H), 5.98 (d, $J = 3.0$ Hz, 1H), 5.74 (dd, $J = 10.3, 7.9$ Hz, 1H), 5.57 (dd, $J = 10.4, 3.4$ Hz, 1H), 5.36 (d, $J = 7.9$ Hz, 1H), 4.64 (dd, $J = 11.3, 7.5$ Hz, 1H), 4.53 – 4.39 (m, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 195.28, 166.07, 165.60, 165.56, 164.67, 153.86, 136.93, 133.77, 133.50, 133.41, 133.18, 133.11, 131.44, 131.38, 130.14, 129.91, 129.85, 129.52, 129.32, 129.14, 128.91, 128.73, 128.67, 128.62, 128.36, 128.24, 128.13, 123.70, 117.82, 100.27, 71.92, 71.81, 69.06, 67.98, 62.36. HRMS (ESI): Calcd for $\text{C}_{47}\text{H}_{40}\text{NO}_{11}$ $[\text{M}+\text{NH}_4]^+$ 794.2601, found 794.2599.

2'-Benzoylphenyl 2,3,4,6-tetra-*O*-benzoyl- α -D-mannopyranoside (3e)



To a solution of **1e** (6.44 mmol, 4.25 g) in DCM (40 mL), 2-hydroxybenzophenone (12.88 mmol, 2.55 g) and activated 4 Å molecular sieves (4.00 g) were added under an argon atmosphere. After cooling to 0 °C, AgOTf (1.99 g, 7.73 mmol) was added. The reaction mixture was stirred for 4 h at room temperature, monitored by TLC, quenched by dropwise addition of Et_3N at 0 °C, filtered through Celite and concentrated in vacuum. The crude product was purified by column chromatography on silica gel (petroleum ether: ethyl acetate = 6:1, v/v) to afford **3e** as a white solid (5.00 g, 60%). ^1H NMR (400 MHz, CDCl_3) δ 8.08 – 7.99 (m, 4H), 7.97 – 7.89 (m, 4H), 7.79 (d, $J = 7.2$ Hz, 2H), 7.62 – 7.50 (m, 7H), 7.44 – 7.36 (m, 8H), 7.32 (d, $J = 8.3$ Hz, 1H), 7.28 – 7.22 (m, 2H), 7.20 (t, $J = 7.4$ Hz, 1H), 6.00 (t, $J = 9.8$ Hz, 1H), 5.76 (s, 1H), 5.31 – 5.24 (m, 2H), 4.56 (dd, $J = 12.2, 2.5$ Hz, 1H), 4.40 (dd, $J = 12.2, 5.0$ Hz, 1H), 4.31 – 4.24 (m, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.29, 166.09, 165.50, 165.31, 164.75, 153.63, 138.74, 133.70, 133.63, 133.19, 133.18, 133.08, 132.51, 130.27, 130.00, 129.95, 129.93, 129.85, 129.83, 129.74, 129.49, 129.17, 128.97, 128.80, 128.73, 128.55, 128.50, 128.38, 123.18, 115.01, 96.00, 70.01, 69.93, 69.34, 66.58, 62.72. HRMS (ESI): Calcd for $\text{C}_{47}\text{H}_{40}\text{NO}_{11}$ $[\text{M}+\text{NH}_4]^+$ 794.2596, found 794.2593.

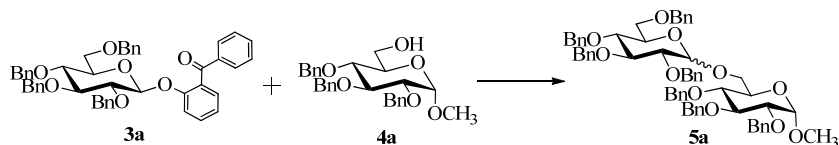
2'-Benzoylphenyl 2,3,4-tri-*O*-benzoyl- α -L-rhamnopyranoside (**3f**)



The same procedure for the synthesis of **3e** was applied, yielding **3f** (3.09 g, 60%) as a white solid. ^1H NMR (400 MHz, CDCl_3) δ 7.91 (dd, $J = 8.3, 1.2$ Hz, 2H), 7.82 (dd, $J = 8.3, 1.2$ Hz, 2H), 7.73 (dd, $J = 8.4, 1.3$ Hz, 2H), 7.63 – 7.56 (m, 3H), 7.54 – 7.47 (m, 2H), 7.44 – 7.34 (m, 6H), 7.27 – 7.10 (m, 7H), 5.68 – 5.66 (m, 1H), 5.55 – 5.48 (m, 2H), 5.40 (d, $J = 1.1$ Hz, 1H), 3.96 – 3.86 (m, 1H), 1.47 (d, $J = 6.2$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 195.86, 165.65, 165.58, 164.95, 154.14, 137.33, 133.55, 133.30, 133.15, 133.04, 131.97, 131.19, 130.19, 129.92, 129.88, 129.84, 129.56, 129.40, 129.22, 128.98, 128.73, 128.59, 128.36, 128.34, 128.11, 123.72, 116.61, 97.62, 71.62, 71.33, 71.23, 69.43, 17.93. HRMS (ESI): Calcd for $\text{C}_{40}\text{H}_{32}\text{NO}_9$ $[\text{M}+\text{NH}_4]^+$ 674.2390, found 674.2388.

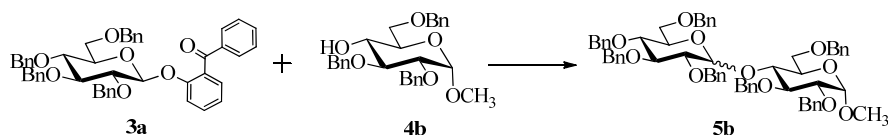
2) Substrate scope of the glycosylation reaction

Methyl 2,3,4-tri-*O*-benzyl-6-*O*-(2,3,4,6-tetra-*O*-benzyl- α/β -D-glucopyranosyl)- α -D-glucopyranoside (**5a**)



A dried flask fitted with a magnetic stir-bar was charged with donor **3a** (30.0 mg, 0.042 mmol), acceptor **4a** (16.3 mg, 0.035 mmol), activated 4Å molecular sieves (200.0 mg) and CH_2Cl_2 (2.0 mL) under an argon atmosphere. Then TfOH (1.2 equiv., 0.042 mmol) was added, the reaction mixture was stirred at room temperature for 10 min, monitored by TLC, quenched by dropwise addition of Et_3N (0.1 mL) at 0 °C, then filtered through Celite and concentrated in vacuum. The crude product was purified by column chromatography on silica gel (petroleum ether: ethyl acetate = 9:1, v/v) to afford **5a** as a white solid (31.1 mg, 90%, $\alpha/\beta = 1.8:1.0$). The ^1H NMR data for **5a** are in accordance with those reported previously. 1 ^1H NMR (400 MHz, CDCl_3) δ 7.47 – 6.99 (m, 52.01H), 4.98 – 4.95 (m, 3.18H), 4.92 – 4.89 (m, 1.86H), 4.83 – 4.38 (m, 18.37H), 4.34 (d, $J = 7.8$ Hz, 1H), 4.17 (d, $J = 9.1$ Hz, 1H), 4.03 – 3.91 (m, 2H), 3.83 – 3.41 (m, 15.52H), 3.35 (s, 1.49H), 3.32 (s, 2.61H).

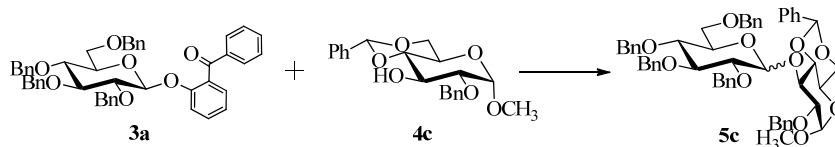
Methyl 2,3,6-tri-*O*-benzyl-4-*O*-(2,3,4,6-tetra-*O*-benzyl- α/β -D-glucopyranosyl)- α -D-glucopyranoside (**5b**)



The reaction of donor **3a** (30.0 mg, 0.042 mmol) and acceptor **4b** (16.3 mg, 0.035 mmol) was performed as described in the synthesis of **5a**, affording **5b** (eluent: petroleum ether/ethyl acetate = 6: 1 (v/v); 20.7 mg, 69%, $\alpha/\beta = 2.5:1.0$). The ^1H NMR data for **5b** are in accordance with those reported previously. 1 ^1H NMR (400 MHz, CDCl_3) δ 7.43 – 7.08 (m, 49.35H), 5.69 (d, $J = 3.5$ Hz, 1H), 5.09 (d, $J = 10.9$ Hz, 0.4H), 5.03 (d, $J = 11.6$ Hz, 1H), 4.91 –

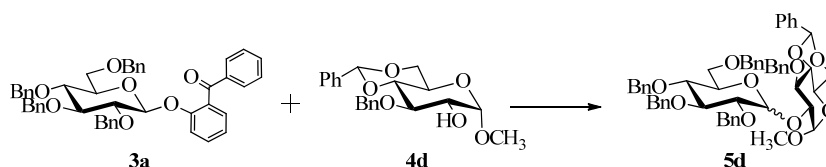
4.72 (m, 6.95H), 4.69 (d, $J = 12.5$ Hz, 1.48H), 4.62 – 4.34 (m, 11.84H), 4.29 (d, $J = 12.2$ Hz, 1H), 4.18 – 3.79 (m, 7.07H), 3.76 – 3.40 (m, 10.76H), 3.37 (s, 3.09H), 3.36 (s, 1.23H), 3.32 – 3.25 (m, 0.54H).

Methyl 2-*O*-benzyl-4,6-*O*-benzylidene-3-*O*-(2,3,4,6-tetra-*O*-benzoyl- α / β -D-glucopyranosyl)- α -D-glucopyranoside (5c**)**



The reaction of donor **3a** (30.0 mg, 0.042 mmol) and acceptor **4c** (13.0 mg, 0.035 mmol) was performed as described in the synthesis of **5a**, affording **5c** (eluent: petroleum ether/ethyl acetate = 6: 1 (v/v); 23.5 mg, 75%, $\alpha/\beta = 2.0:1.0$, α -isomer: 15.7 mg, 50%; β -isomer: 7.8 mg, 25%). The ^1H NMR data for **5c** are in accordance with those reported previously.¹ **α -isomer:** ^1H NMR (400 MHz, CDCl_3) δ 7.43 – 7.11 (m, 28H), 6.92 (d, $J = 7.1$ Hz, 2H), 5.59 (d, $J = 3.6$ Hz, 1H), 5.46 (s, 1H), 4.99 (d, $J = 10.8$ Hz, 1H), 4.81 (d, $J = 3.7$ Hz, 1H), 4.78 (d, $J = 3.9$ Hz, 1H), 4.71 (d, $J = 3.7$ Hz, 1H), 4.65 (d, $J = 11.4$ Hz, 1H), 4.61 – 4.53 (m, 3H), 4.42 – 4.34 (m, 2H), 4.30 (t, $J = 12.3$ Hz, 2H), 4.26 – 4.17 (m, 2H), 3.96 (t, $J = 9.4$ Hz, 1H), 3.92 – 3.83 (m, 1H), 3.78 (t, $J = 9.3$ Hz, 1H), 3.73 – 3.63 (m, 3H), 3.53 – 3.43 (m, 3H), 3.41 (s, 3H). **β -isomer:** ^1H NMR (400 MHz, CDCl_3) δ 7.43 – 7.39 (m, 2H), 7.38 – 7.20 (m, 26H), 7.17 – 7.12 (m, 2H), 5.46 (s, 1H), 5.06 (d, $J = 11.3$ Hz, 1H), 4.95 – 4.86 (m, 2H), 4.81 – 4.68 (m, 4H), 4.53 (d, $J = 10.7$ Hz, 1H), 4.50 – 4.43 (m, 4H), 4.36 (t, $J = 9.1$ Hz, 1H), 4.21 (dd, $J = 10.1, 4.6$ Hz, 1H), 3.86 – 3.77 (m, 1H), 3.71 – 3.54 (m, 7H), 3.49 (t, $J = 8.1$ Hz, 1H), 3.35 (s, 3H), 3.28 – 3.22 (m, 1H).

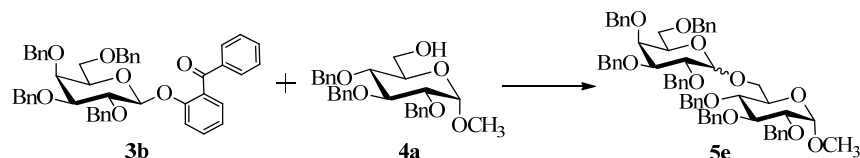
Methyl 3-*O*-benzyl-4,6-*O*-benzylidene-2-*O*-(2,3,4,6-tetra-*O*-benzyl- α / β -D-glucopyranosyl)- α -D-glucopyranoside (5d**)**



The reaction of donor **3a** (30.0 mg, 0.042 mmol) and acceptor **4d** (13.0 mg, 0.035 mmol) was performed as described in the synthesis of **5a**, affording **5d** (eluent: petroleum ether/ethyl acetate = 6: 1 (v/v); 25.7 mg, 82%, $\alpha/\beta = 2.6:1.0$, α -isomer: 18.6 mg, 59%; β -isomer: 7.1 mg, 23%). The ^1H NMR data for **5d** are in accordance with those reported previously.¹ **α -isomer:** ^1H NMR (400 MHz, CDCl_3) δ 7.52 – 7.50 (m, 2H), 7.42 – 7.23 (m, 20H), 7.25 – 7.23 (m, 3H), 7.15 – 7.10 (m, 3H), 7.05 – 7.01 (m, 2H), 5.57 (s, 1H), 5.02 (d, $J = 10.8$ Hz, 1H), 4.92 (d, $J = 3.6$ Hz, 1H), 4.89 – 4.69 (m, 7H), 4.53 (d, $J = 12.1$ Hz, 1H), 4.45 (d, $J = 11.2$ Hz, 1H), 4.33 – 4.27 (m, 2H), 4.17 – 4.08 (m, 3H), 3.90 – 3.83 (m, 2H), 3.78 – 3.57 (m, 4H), 3.50 – 3.45 (m, 4H), 3.41 – 3.37 (m, 1H). **β -isomer:** ^1H NMR (400 MHz, CDCl_3) δ 7.46 (dd, $J = 6.6$ Hz, 3.1 Hz, 2H), 7.38 – 7.13 (m, 28H), 5.56 (s, 1H), 5.04 (d, $J = 11.5$ Hz, 1H), 4.96 (d, $J = 3.6$ Hz, 1H), 4.92 (d, $J = 10.9$ Hz, 1H), 4.82 – 4.74 (m, 5H), 4.65 (d, $J = 10.8$ Hz, 1H), 4.57 (d, $J = 12.0$ Hz,

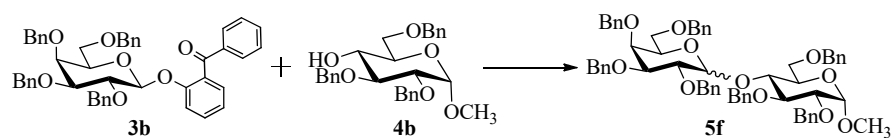
1H), 4.52 (d, $J = 5.6$ Hz, 1H), 4.49 (d, $J = 6.9$ Hz, 1H), 4.31 (dd, $J = 10.1, 4.8$ Hz, 1H), 4.10 (t, $J = 9.0$ Hz, 1H), 3.94 – 3.83 (m, 2H), 3.76 (t, $J = 10.6$ Hz, 1H), 3.68 – 3.50 (m, 6H), 3.43 – 3.38 (m, 4H).

Methyl 2,3,4-tri-*O*-benzyl-6-*O*-(2,3,4,6-tetra-*O*-benzyl- α / β -D-galactopyranosyl)- α -D-glucopyranoside (5e)



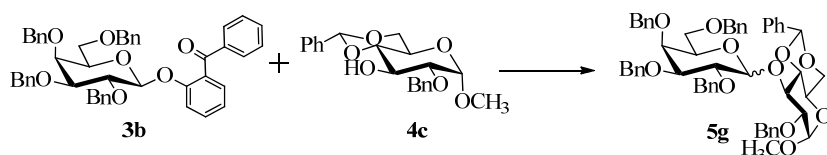
The reaction of donor **3b** (30.0 mg, 0.042 mmol) and acceptor **4a** (16.3 mg, 0.035 mmol) was performed as described in the synthesis of **5a**, affording **5e** (eluent: petroleum ether/ethyl acetate = 9: 1 (v/v); 31.4 mg, 91%, $\alpha/\beta = 1.9:1.0$). The ^1H NMR data for **5e** are in accordance with those reported previously.² ^1H NMR (400 MHz, CDCl_3) δ 7.40 – 7.13 (m, 53.2H), 4.99 (d, $J = 3.5$ Hz, 1H), 4.98 – 4.90 (m, 3.77H), 4.87 – 4.73 (m, 5.35H), 4.73 – 4.62 (m, 6.37H), 4.60 – 4.51 (m, 5.8H), 4.50 – 4.27 (m, 4.5H), 4.14 (dd, $J = 10.7, 1.7$ Hz, 0.54H), 4.06 – 3.69 (m, 11.15H), 3.64 – 3.38 (m, 8.62H), 3.29 (d, $J = 2.2$ Hz, 4.56H).

Methyl 2,3,6-tri-*O*-benzyl-4-*O*-(2,3,4,6-tetra-*O*-benzyl- α / β -D-galactopyranosyl)- α -D-glucopyranoside (5f)



The reaction of donor **3b** (30.0 mg, 0.042 mmol) and acceptor **4b** (13.0 mg, 0.035 mmol) was performed as described in the synthesis of **5a**, affording **5f** (eluent: petroleum ether/ethyl acetate = 6: 1 (v/v); 24.5 mg, 71%, $\alpha/\beta = 3.1:1.0$). The ^1H NMR data for **5f** are in accordance with those reported previously.² ^1H NMR (400 MHz, CDCl_3) δ 7.38 – 7.11 (m, 46.2H), 5.75 (d, $J = 3.8$ Hz, 1H), 5.03 (d, $J = 10.7$ Hz, 0.32H), 4.96 (d, $J = 11.5$ Hz, 1H), 4.86 (d, $J = 11.4$ Hz, 1H), 4.84 – 4.76 (m, 2H), 4.77 – 4.50 (m, 11.15H), 4.42 (d, $J = 12.3$ Hz, 1.12H), 4.40 – 4.26 (m, 2.05H), 4.23 (d, $J = 11.6$ Hz, 1.13H), 4.09 – 4.03 (t, $J = 10.7$ Hz, 1H), 4.01 – 3.79 (m, 7.41H), 3.78 – 3.40 (m, 7.62H), 3.37 (d, $J = 2.3$ Hz, 3.65H), 3.35 – 3.28 (m, 0.88H).

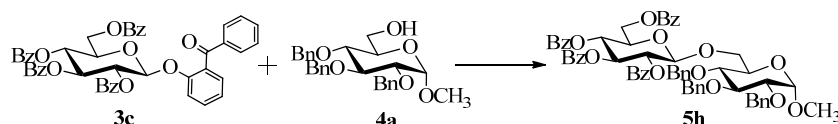
Methyl 2-*O*-benzyl-4,6-*O*-benzylidene-3-*O*-(2,3,4,6-tetra-*O*-benzyl- α / β -D-galactopyranosyl)- α -D-glucopyranoside (5g)



The reaction of donor **3b** (30.0 mg, 0.042 mmol) and acceptor **4c** (13.0 mg, 0.035 mmol) was performed as described in the synthesis of **5a**, affording **5g** (eluent: petroleum ether/ethyl acetate = 6: 1 (v/v); 23.8 mg, 76%, $\alpha/\beta = 3.5:1.0$, α -isomer: 18.5 mg, 59%; β -isomer: 5.3 mg, 17%). The ^1H NMR data for **5g** are in accordance with those

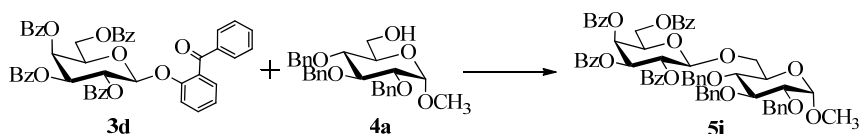
reported previously.² **α -isomer:** ¹H NMR (400 MHz, CDCl₃) δ 7.42 – 7.20 (m, 25H), 7.16 (t, J = 7.2 Hz, 1H), 7.09 (t, J = 7.4 Hz, 2H), 7.02 (d, J = 7.3 Hz, 2H), 5.61 (d, J = 3.4 Hz, 1H), 5.43 (s, 1H), 4.88 (d, J = 11.1 Hz, 1H), 4.85 (d, J = 11.1 Hz, 1H), 4.74 (d, J = 11.6 Hz, 1H), 4.71 (d, J = 12.0 Hz, 1H), 4.61 – 4.49 (m, 4H), 4.44 (d, J = 12.3 Hz, 1H), 4.40 – 4.30 (m, 4H), 4.21 (dd, J = 10.1, 4.6 Hz, 1H), 4.02 – 3.90 (m, 3H), 3.87 – 3.78 (m, 1H), 3.74 (t, J = 9.4 Hz, 1H), 3.68 (t, J = 10.2 Hz, 1H), 3.64 – 3.57 (m, 2H), 3.54 (t, J = 8.5 Hz, 1H), 3.33 (s, 3H). **β -isomer:** ¹H NMR (400 MHz, CDCl₃) δ 7.47 – 7.43 (m, 2H), 7.37 – 7.22 (m, 26H), 7.20 – 7.17 (m, 2H), 5.50 (s, 1H), 5.03 (d, J = 11.0 Hz, 1H), 4.93 (d, J = 11.6 Hz, 1H), 4.81 – 4.76 (m, 3H), 4.68 (s, 2H), 4.60 (d, J = 11.7 Hz, 1H), 4.51 (d, J = 12.1 Hz, 1H), 4.44 (d, J = 3.8 Hz, 1H), 4.33 (t, J = 8.6 Hz, 1H), 4.26 (d, J = 13.3 Hz, 1H), 4.23 – 4.16 (m, 1H), 3.89 – 3.80 (m, 3H), 3.71 – 3.56 (m, 4H), 3.48 (dd, J = 9.7, 2.9 Hz, 1H), 3.42 – 3.31 (m, 2H), 3.34 (s, 3H).

Methyl 2,3,4-tri-*O*-benzyl-6-*O*-(2,3,4,6-tetra-*O*-benzoyl- β -D-glucopyranosyl)- α -D-glucopyranoside (5h**)**



The reaction of donor **3c** (32.6 mg, 0.042 mmol) and acceptor **4a** (16.3 mg, 0.035 mmol) was performed as described in the synthesis of **5a**, affording **5h** (eluent: petroleum ether/ethyl acetate = 5: 1 (v/v); 28.47 mg, 78%). The ¹H NMR data for **5h** are in accordance with those reported previously.³ ¹H NMR (400 MHz, CDCl₃) δ 8.01 – 7.97 (m, 2H), 7.92 – 7.86 (m, 4H), 7.84 – 7.80 (m, 2H), 7.56 – 7.46 (m, 2H), 7.45 – 7.18 (m, 23H), 7.07 – 7.03 (m, 2H), 5.89 (t, J = 9.6 Hz, 1H), 5.67 (t, J = 9.7 Hz, 1H), 5.59 (dd, J = 9.7, 7.8 Hz, 1H), 4.89 (d, J = 10.9 Hz, 1H), 4.82 (d, J = 7.8 Hz, 1H), 4.73 (d, J = 12.1 Hz, 1H), 4.68 (d, J = 11.0 Hz, 1H), 4.63 – 4.57 (m, 2H), 4.54 – 4.47 (m, 3H), 4.28 (d, J = 11.2 Hz, 1H), 4.18 – 4.07 (m, 2H), 3.88 (t, J = 9.2 Hz, 1H), 3.77 – 3.69 (m, 2H), 3.43 (dd, J = 9.7, 3.5 Hz, 1H), 3.37 (t, J = 9.4 Hz, 1H), 3.21 (s, 3H).

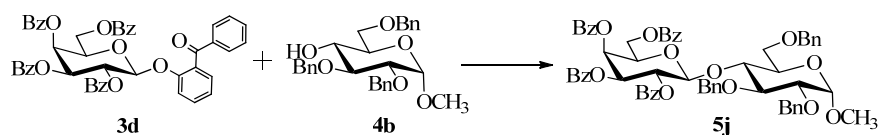
Methyl 2,3,4-tri-*O*-benzyl-6-*O*-(2,3,4,6-tetra-*O*-benzoyl- β -D-galactopyranosyl)- α -D-glucopyranoside (5i**)**



The reaction of donor **3d** (32.6 mg, 0.042 mmol) and acceptor **4a** (16.3 mg, 0.035 mmol) was performed as described in the synthesis of **5a**, affording **5i** (eluent: petroleum ether/ethyl acetate = 5: 1 (v/v); 29.2 mg, 80%). The ¹H NMR data for **5i** are in accordance with those reported previously.³ ¹H NMR (400 MHz, CDCl₃) δ 8.08 (d, J = 7.6 Hz, 2H), 8.01 (d, J = 7.7 Hz, 2H), 7.88 (d, J = 7.8 Hz, 2H), 7.76 (d, J = 7.7 Hz, 2H), 7.65 – 7.18 (m, 25H), 7.16 – 7.08 (m, 2H), 5.97 (d, J = 3.3 Hz, 1H), 5.84 (dd, J = 10.1, 8.2 Hz, 1H), 5.59 (dd, J = 10.4, 3.4 Hz, 1H), 4.89 (d, J = 10.9 Hz, 1H), 4.77 – 4.72 (m, 1H), 4.72 – 4.63 (m, 3H), 4.58 (d, J = 9.0 Hz, 1H), 4.55 (d, J = 8.3 Hz, 1H), 4.49 (d, J = 3.4 Hz, 1H), 4.43 – 4.34 (m, 2H), 4.27 – 4.17 (m, 2H), 3.89 (t, J = 9.2 Hz, 1H), 3.75 (d, J = 8.8 Hz, 2H), 3.42 –

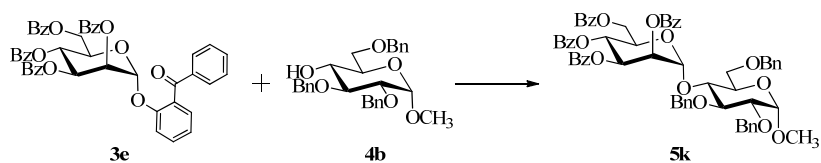
3.32 (m, 2H), 3.20 (s, 3H).

Methyl 2,3,6-tri-*O*-benzyl-4-*O*-(2,3,4,6-tetra-*O*-benzoyl- β -D-galactopyranosyl)- α -D-glucopyranoside (5j**)**



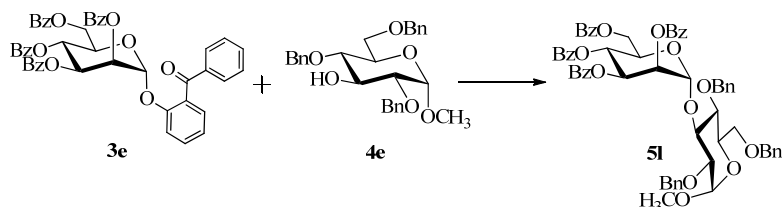
The reaction of donor **3d** (32.6 mg, 0.042 mmol) and acceptor **4b** (16.3 mg, 0.035 mmol) was performed as described in the synthesis of **5a**, affording **5j** (eluent: petroleum ether/ethyl acetate = 4: 1 (v/v); 24.8 mg, 68%). The ^1H NMR data for **5j** are in accordance with those reported previously.³ ^1H NMR (400 MHz, CDCl_3) δ 8.02 (d, J = 7.3 Hz, 2H), 7.94 (d, J = 7.4 Hz, 2H), 7.85 (d, J = 7.4 Hz, 2H), 7.76 (d, J = 7.4 Hz, 2H), 7.59 – 7.13 (m, 27H), 5.85 (d, J = 3.2 Hz, 1H), 5.70 (dd, J = 10.2, 8.2 Hz, 1H), 5.30 (dd, J = 10.4, 3.4 Hz, 1H), 5.18 (d, J = 11.1 Hz, 1H), 4.91 (d, J = 11.1 Hz, 1H), 4.82 – 4.73 (m, 3H), 4.64 (d, J = 12.3 Hz, 1H), 4.58 (d, J = 3.6 Hz, 1H), 4.40 (dd, J = 11.2, 6.2 Hz, 1H), 4.32 (d, J = 12.2 Hz, 1H), 4.18 (dd, J = 11.1, 7.8 Hz, 1H), 4.03 (t, J = 9.4 Hz, 1H), 3.91 (dd, J = 11.9, 6.4 Hz, 2H), 3.70 (dd, J = 10.7, 2.4 Hz, 1H), 3.56 – 3.48 (m, 2H), 3.43 (d, J = 9.7 Hz, 1H), 3.30 (s, 3H).

Methyl 2,3,6-tri-*O*-benzyl-4-*O*-(2,3,4,6-tetra-*O*-benzoyl- α -D-mannopyranosyl)- α -D-glucopyranoside (5k**)**



The reaction of donor **3e** (32.6 mg, 0.042 mmol) and acceptor **4b** (16.3 mg, 0.035 mmol) was performed as described in the synthesis of **5a**, affording **5k** (eluent: petroleum ether/ethyl acetate = 5: 1 (v/v); 25.6 mg, 63%). The ^1H NMR data for **5k** are in accordance with those reported previously.³ ^1H NMR (400 MHz, CDCl_3) δ 8.07 (d, J = 7.6 Hz, 2H), 7.92 (t, J = 6.7 Hz, 4H), 7.79 (d, J = 7.7 Hz, 2H), 7.63 – 7.48 (m, 3H), 7.46 – 7.18 (m, 20H), 7.12 (t, J = 7.3 Hz, 1H), 7.02 (t, J = 7.2 Hz, 2H), 6.99 – 6.92 (m, 1H), 6.03 (t, J = 10.1 Hz, 1H), 5.85 (d, J = 9.9 Hz, 1H), 5.72 (s, 1H), 5.60 (s, 1H), 5.06 (d, J = 10.9 Hz, 1H), 4.85 (d, J = 11.0 Hz, 1H), 4.74 (d, J = 12.2 Hz, 1H), 4.65 – 4.52 (m, 4H), 4.46 (d, J = 12.1 Hz, 1H), 4.34 (d, J = 9.8 Hz, 1H), 4.23 (d, J = 12.3 Hz, 1H), 4.09 (t, J = 8.4 Hz, 1H), 3.97 – 3.82 (m, 3H), 3.77 (d, J = 10.4 Hz, 1H), 3.56 (d, J = 9.4 Hz, 1H), 3.44 (s, 3H).

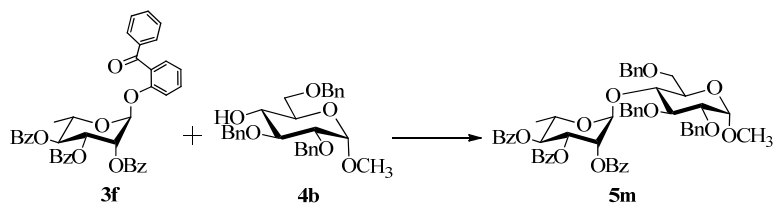
Methyl 2,4,6-tri-*O*-benzyl-3-*O*-(2,3,4,6-tetra-*O*-benzoyl- α -D-mannopyranosyl)- α -D-glucopyranoside (5l**)**



The reaction of donor **3e** (32.6 mg, 0.042 mmol) and acceptor **4e** (16.3 mg, 0.035 mmol) was performed as described in the synthesis of **5a**, affording **5l** (eluent: petroleum ether/ethyl acetate = 4: 1 (v/v); 28.4 mg, 70%). The ^1H NMR data for **5l** are in accordance with those reported previously.³ ^1H NMR (400 MHz, CDCl_3) δ 8.13 (d, J =

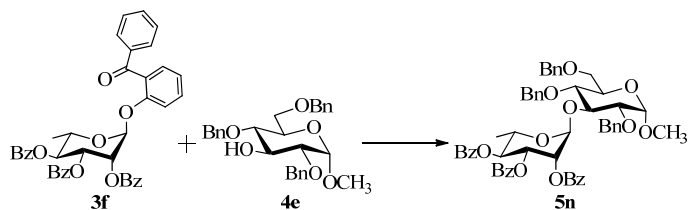
7.5 Hz, 2H), 7.94 (d, $J = 7.5$ Hz, 2H), 7.83 (t, $J = 7.6$ Hz, 4H), 7.56 (t, $J = 7.2$ Hz, 2H), 7.53 – 7.15 (m, 20H), 7.09 – 7.02 (m, 5H), 6.04 (t, $J = 10.1$ Hz, 1H), 5.94 (d, $J = 10.3$ Hz, 1H), 5.82 (s, 1H), 5.57 (s, 1H), 4.91 – 4.61 (m, 6H), 4.52 (dd, $J = 11.4, 6.6$ Hz, 2H), 4.44 (d, $J = 12.2$ Hz, 1H), 4.31 (t, $J = 9.1$ Hz, 1H), 3.98 (d, $J = 12.2$ Hz, 1H), 3.82 (t, $J = 9.1$ Hz, 1H), 3.75 (d, $J = 9.2$ Hz, 2H), 3.66 (d, $J = 9.7$ Hz, 2H), 3.39 (s, 3H).

Methyl 2,3,6-tri-*O*-benzyl-4-*O*-(2,3,4-tri-*O*-benzoyl- α -L-rhamnopyranosyl)- α -D-glucopyranoside (5m)



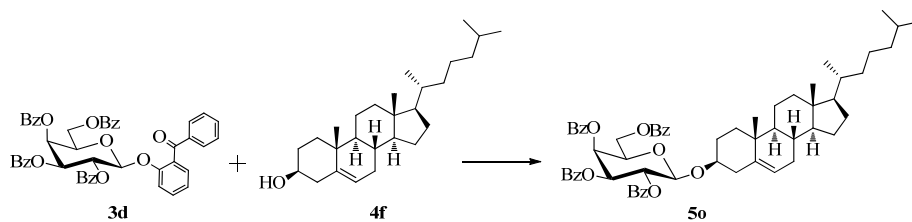
The reaction of donor **3f** (27.6 mg, 0.042 mmol) and acceptor **4b** (16.3 mg, 0.035 mmol) was performed as described in the synthesis of **5a**, affording **5m** (eluent: petroleum ether/ethyl acetate = 5: 1 (v/v); 22.6 mg, 70%). The ^1H NMR data for **5m** are in accordance with those reported previously.³ ^1H NMR (400 MHz, CDCl_3) δ 8.08 – 8.02 (m, 2H), 7.93 – 7.82 (m, 4H), 7.63 – 7.36 (m, 9H), 7.35 – 7.24 (m, 9H), 7.21 – 7.09 (m, 6H), 5.78 (dd, $J = 10.2$ Hz, 3.4 Hz, 1H), 5.64 – 5.54 (m, 2H), 5.21 (d, $J = 10.6$ Hz, 2H), 4.85 (d, $J = 11.1$ Hz, 1H), 4.76 (d, $J = 12.1$ Hz, 1H), 4.68 – 4.51 (m, 4H), 4.41 – 4.32 (m, 1H), 4.05 – 3.94 (m, 2H), 3.89 (dd, $J = 11.1$ Hz, 2.5 Hz, 1H), 3.84 (d, $J = 9.2$ Hz, 1H), 3.73 (d, $J = 9.8$ Hz, 1H), 3.65 (dd, $J = 9.0$ Hz, 3.5 Hz, 1H), 3.41 (s, 3H), 0.88 (d, $J = 6.2$ Hz, 3H).

Methyl 2,4,6-tri-*O*-benzyl-3-*O*-(2,3,4-tri-*O*-benzoyl- α -L-rhamnopyranosyl)- α -D-glucopyranoside (5n)



The reaction of donor **3f** (27.6 mg, 0.042 mmol) and acceptor **4e** (16.3 mg, 0.035 mmol) was performed as described in the synthesis of **5a**, affording **5n** (eluent: petroleum ether/ethyl acetate = 5: 1 (v/v); 26.7 mg, 81%). The ^1H NMR data for **5n** are in accordance with those reported previously.³ ^1H NMR (400 MHz, CDCl_3) δ 8.05 (d, $J = 8.2$ Hz, 2H), 7.83 (d, $J = 8.2$ Hz, 2H), 7.77 (d, $J = 8.2$ Hz, 2H), 7.59 (t, $J = 7.4$ Hz, 1H), 7.53 – 7.09 (m, 23H), 5.89 – 5.80 (m, 2H), 5.70 (s, 1H), 5.60 (t, $J = 10.0$ Hz, 1H), 4.98 (d, $J = 11.3$ Hz, 1H), 4.81 (d, $J = 12.1$ Hz, 1H), 4.69 – 4.59 (m, 4H), 4.52 (d, $J = 12.0$ Hz, 1H), 4.41 – 4.34 (m, 1H), 4.28 (t, $J = 9.3$ Hz, 1H), 3.81 (d, $J = 9.1$ Hz, 2H), 3.76 – 3.61 (m, 3H), 3.33 (s, 3H), 1.06 (d, $J = 6.1$ Hz, 3H).

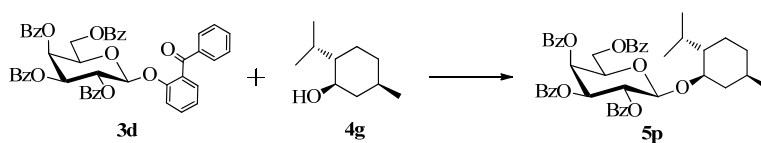
Cholesteryl 2,3,4,6-tetra-*O*-benzoyl- β -D-galactopyranoside (5o)



The reaction of donor **3d** (32.6 mg, 0.042 mmol) and acceptor **4f** (13.5 mg, 0.035 mmol) was performed as

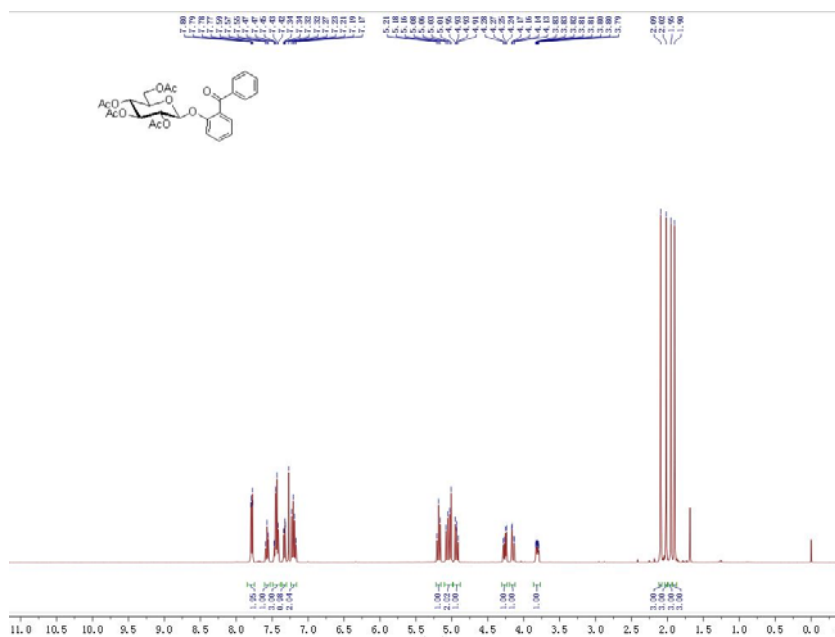
described in the synthesis of **5a**, affording **5o** (eluent: petroleum ether/ethyl acetate = 5: 1 (v/v); 27.0 mg, 80%). The ¹H NMR data for **5o** are in accordance with those reported previously.³ ¹H NMR (400 MHz, CDCl₃) δ 8.10 (d, *J* = 7.4 Hz, 2H), 8.02 (d, *J* = 7.5 Hz, 2H), 7.96 (d, *J* = 7.5 Hz, 2H), 7.79 (d, *J* = 7.6 Hz, 2H), 7.65 – 7.35 (m, 11H), 7.23 (d, *J* = 7.8 Hz, 1H), 5.98 (d, *J* = 3.4 Hz, 1H), 5.77 (dd, *J* = 10.2, 8.0 Hz, 1H), 5.59 (dd, *J* = 10.5, 3.1 Hz, 1H), 5.27 – 5.18 (m, 1H), 4.91 (d, *J* = 8.0 Hz, 1H), 4.67 (dd, *J* = 11.0, 6.9 Hz, 1H), 4.42 (dd, *J* = 11.1, 6.4 Hz, 1H), 4.32 (t, *J* = 6.7 Hz, 1H), 3.62 – 3.49 (m, 1H), 2.18 (d, *J* = 7.7 Hz, 2H), 2.05 – 0.80 (m, 41H), 0.66 (s, 3H).

(1R, 2S, 5R)-(-)-1-Menthyl 2,3,4,6-tetra-*O*-benzoyl-β-D-galactopyranoside (5p)

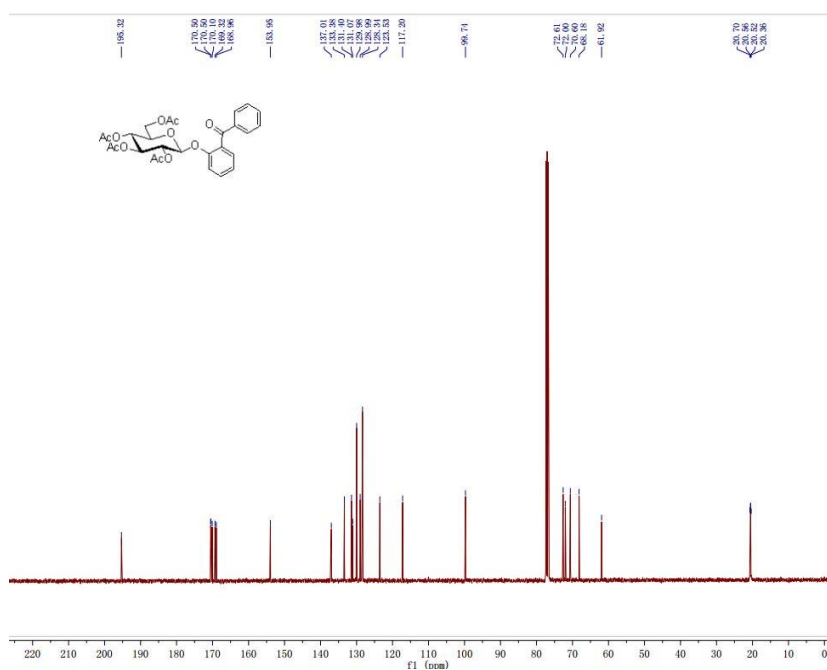


The reaction of donor **3d** (27.6 mg, 0.042 mmol) and acceptor **4g** (5.4 mg, 0.035 mmol) was performed as described in the synthesis of **5a**, affording **5p** (eluent: petroleum ether/ethyl acetate = 5: 1 (v/v); 20.8 mg, 81%). The ¹H NMR data for **5p** are in accordance with those reported previously.³ ¹H NMR (400 MHz, CDCl₃) δ 8.12 – 8.07 (m, 2H), 8.04 – 8.00 (m, 2H), 7.99 – 7.95 (m, 2H), 7.82 – 7.77 (m, 2H), 7.65 – 7.35 (m, 10H), 7.27 – 7.22 (m, 2H), 5.98 (d, *J* = 3.4 Hz, 1H), 5.74 (dd, *J* = 10.3, 8.0 Hz, 1H), 5.58 (dd, *J* = 10.4, 3.4 Hz, 1H), 4.87 (d, *J* = 7.9 Hz, 1H), 4.62 (dd, *J* = 11.3, 6.7 Hz, 1H), 4.42 (dd, *J* = 11.3, 6.3 Hz, 1H), 4.29 (t, *J* = 6.5 Hz, 1H), 3.48 (td, *J* = 10.7, 4.3 Hz, 1H), 2.42 – 2.27 (m, 1H), 1.94 (d, *J* = 12.3 Hz, 1H), 1.65 – 1.50 (m, 2H), 1.35 – 1.15 (m, 2H), 1.00 – 0.61 (m, 12H).

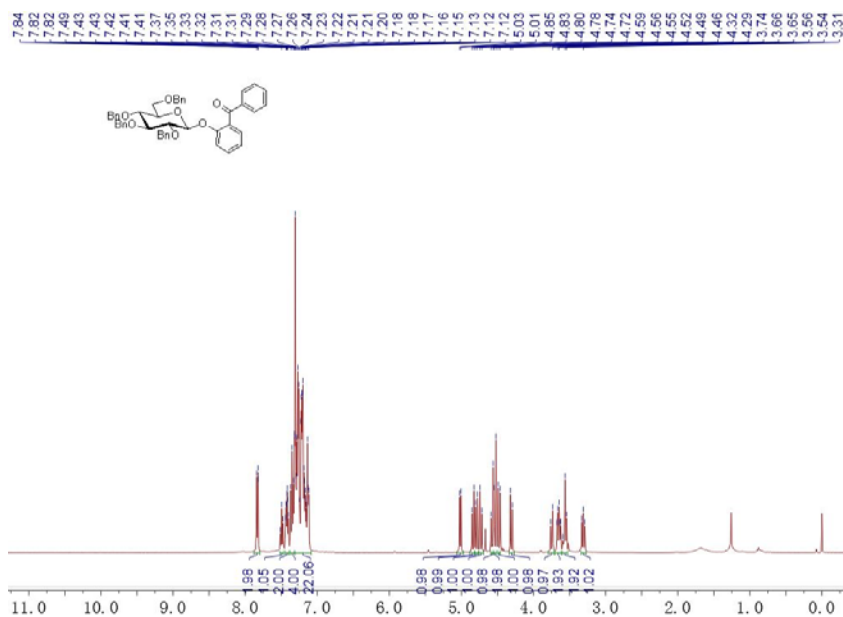
2. ¹H and ¹³C NMR Spectra



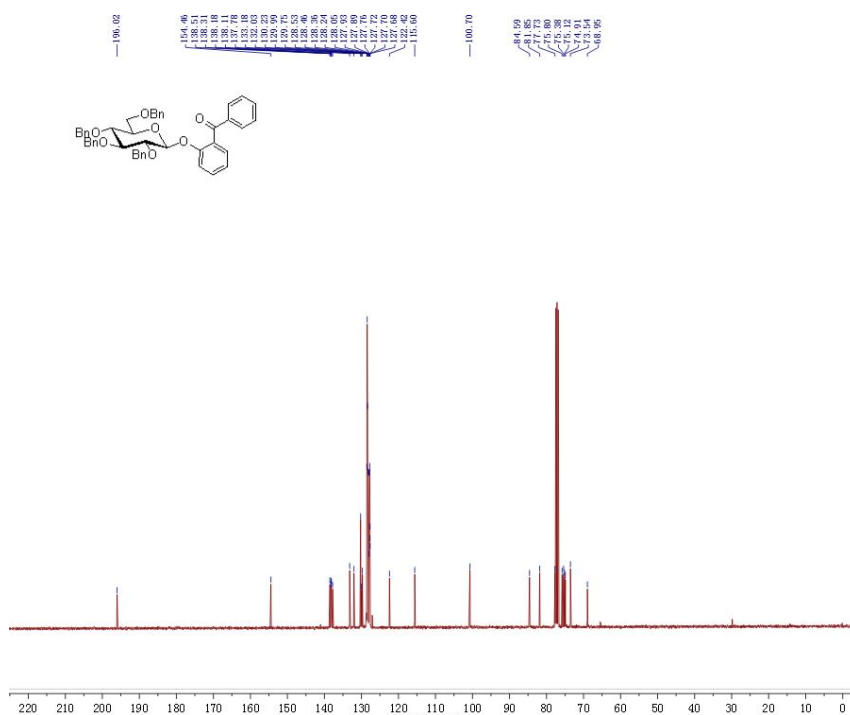
¹H NMR (400 MHz, CDCl₃) spectrum of compound 2a



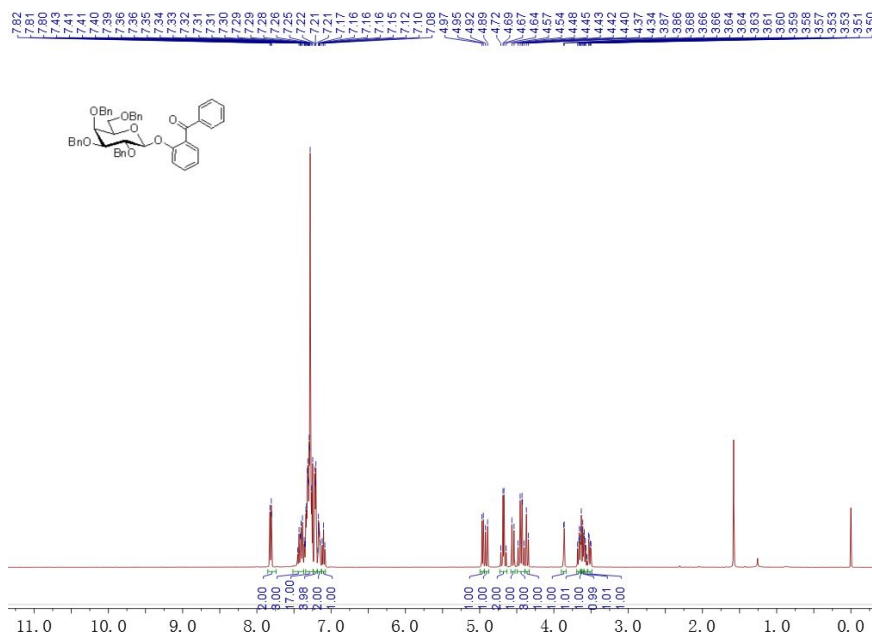
¹³C NMR (101 MHz, CDCl₃) spectrum of compound 2a



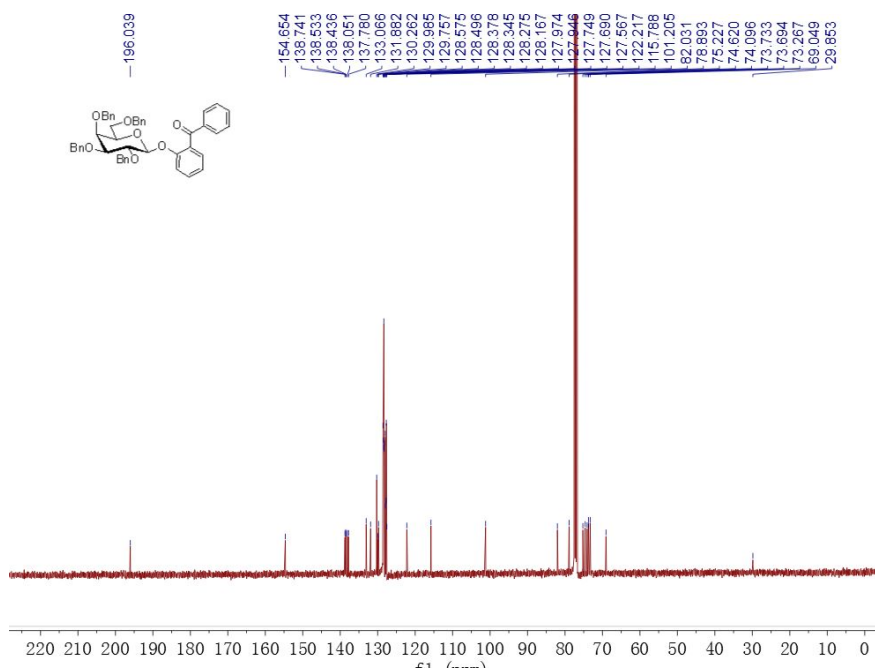
¹H NMR (400 MHz, CDCl₃) spectrum of compound 3a



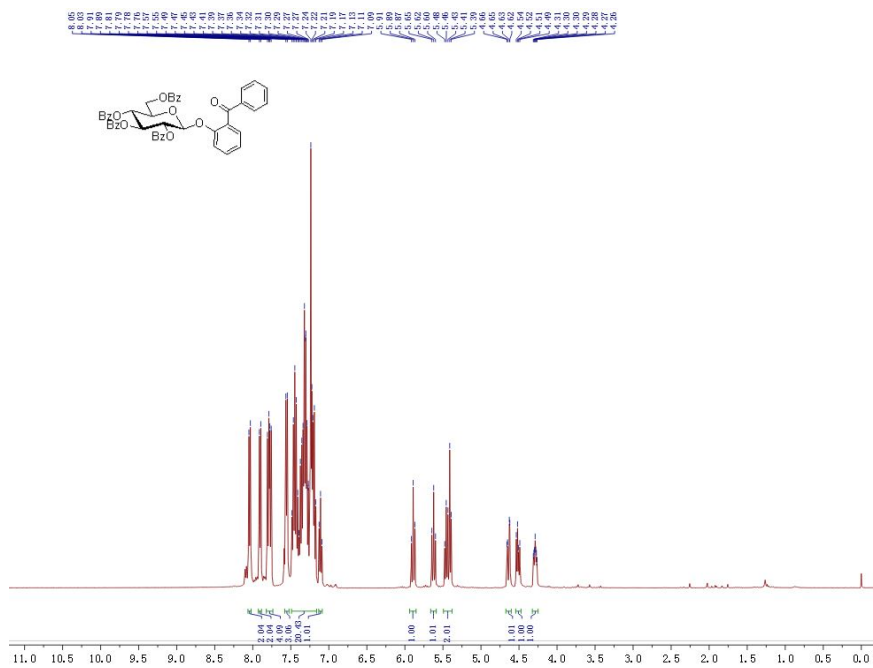
¹³C NMR (101 MHz, CDCl₃) spectrum of compound 3a



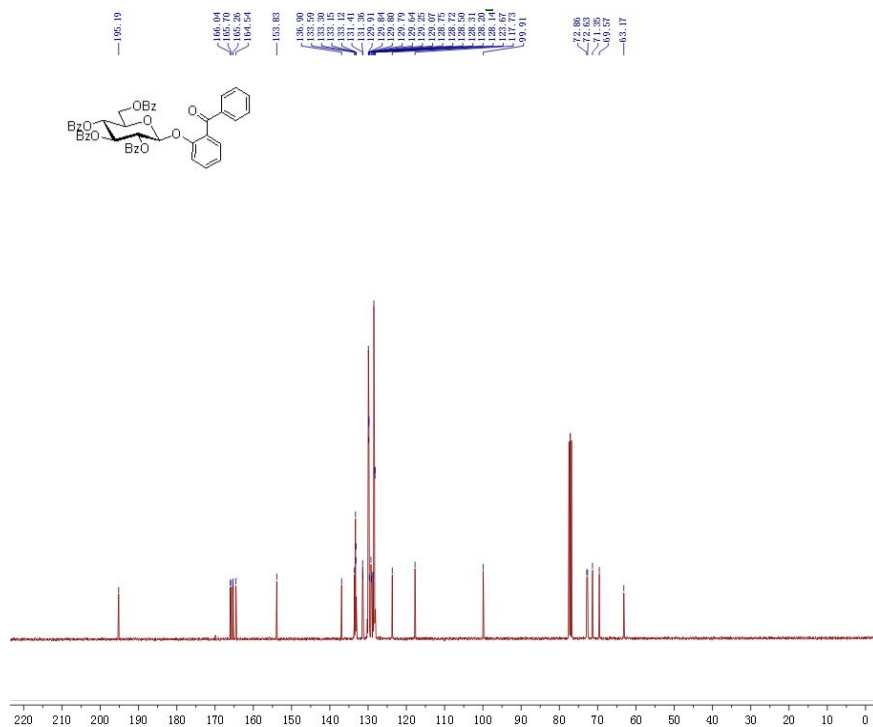
¹H NMR (400 MHz, CDCl₃) spectrum of compound 3b



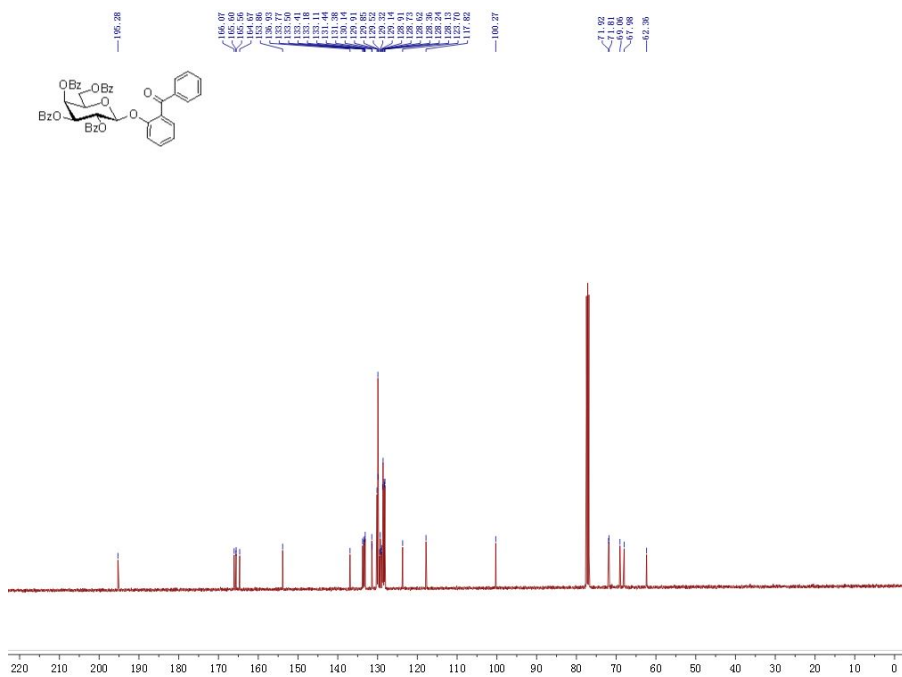
¹³C NMR (101 MHz, CDCl₃) spectrum of compound 3b



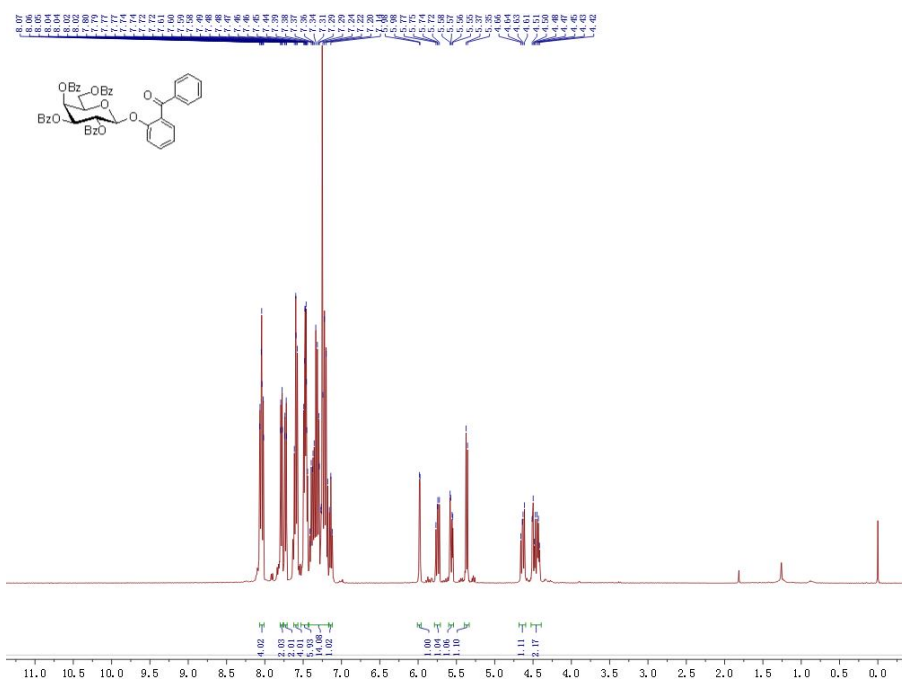
¹H NMR (400 MHz, CDCl₃) spectrum of compound 3c



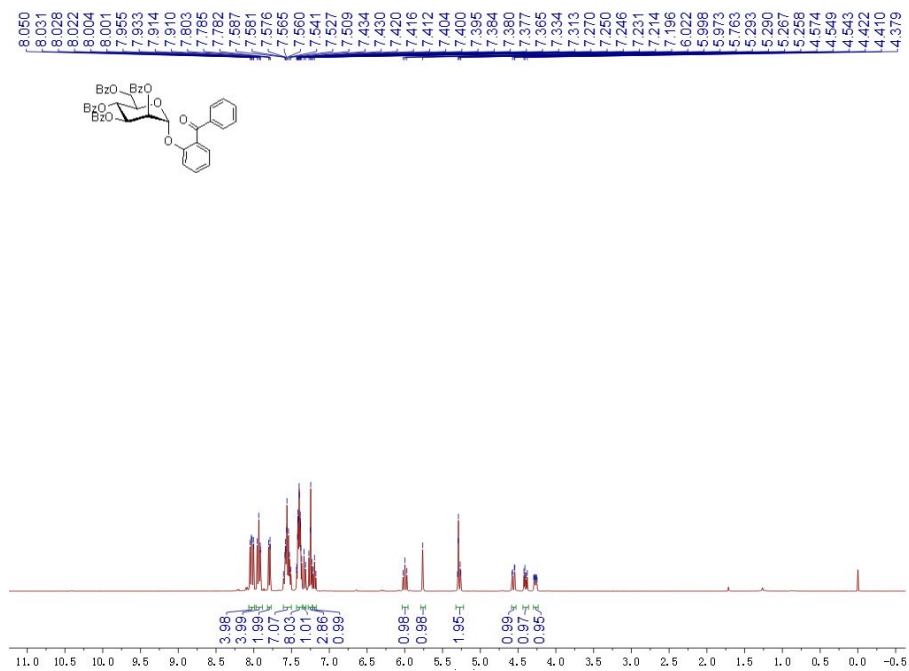
¹³C NMR (101 MHz, CDCl₃) spectrum of compound 3c



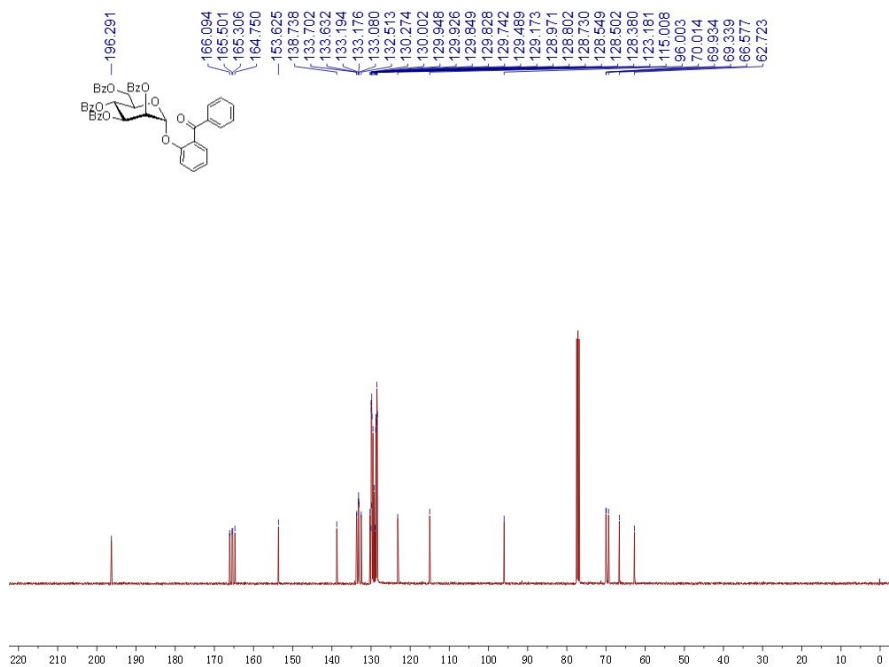
¹³C NMR (101 MHz, CDCl₃) spectrum of compound 3d



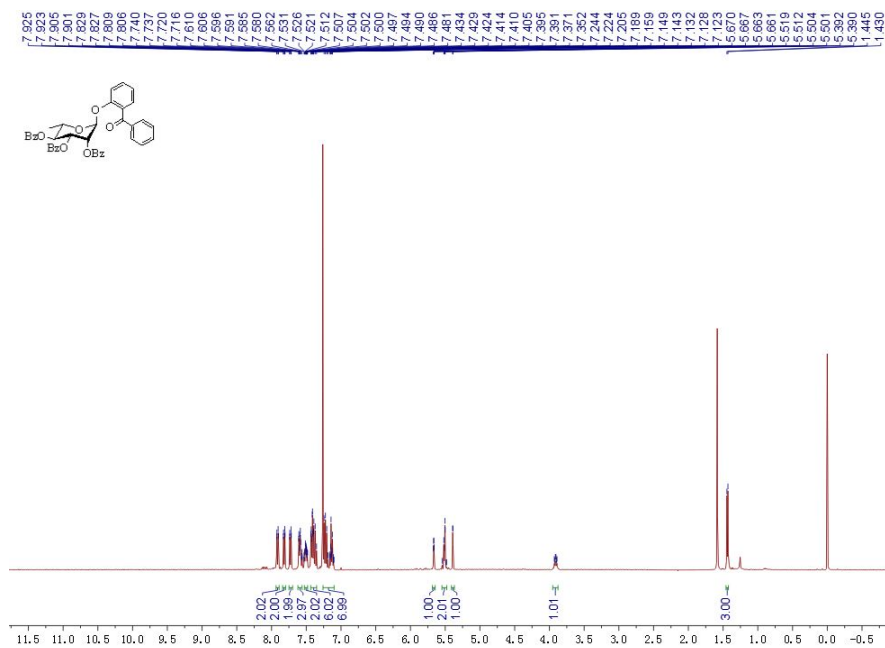
¹H NMR (400 MHz, CDCl₃) spectrum of compound 3d



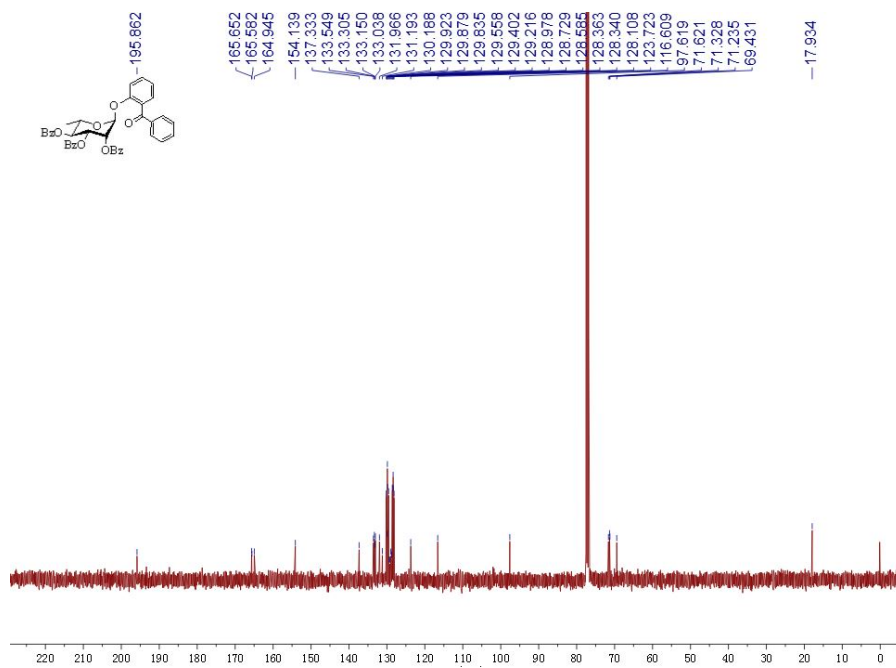
¹H NMR (400 MHz, CDCl₃) spectrum of compound 3e



¹³C NMR (101 MHz, CDCl₃) spectrum of compound 3e

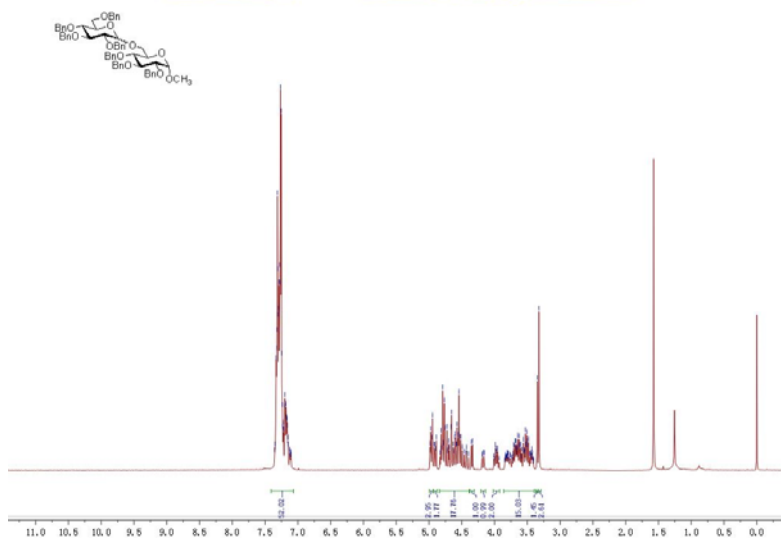


¹H NMR (400 MHz, CDCl₃) spectrum of compound 3f



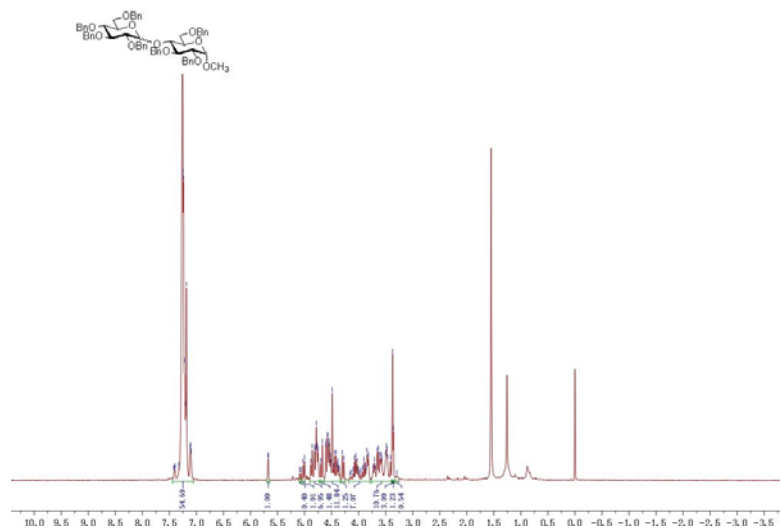
¹³C NMR (101 MHz, CDCl₃) spectrum of compound 3f

将下列数据与所测的谱图对照，以验证其正确性。如有不符，请重新测定。如有必要，请重新测定。如有必要，请重新测定。

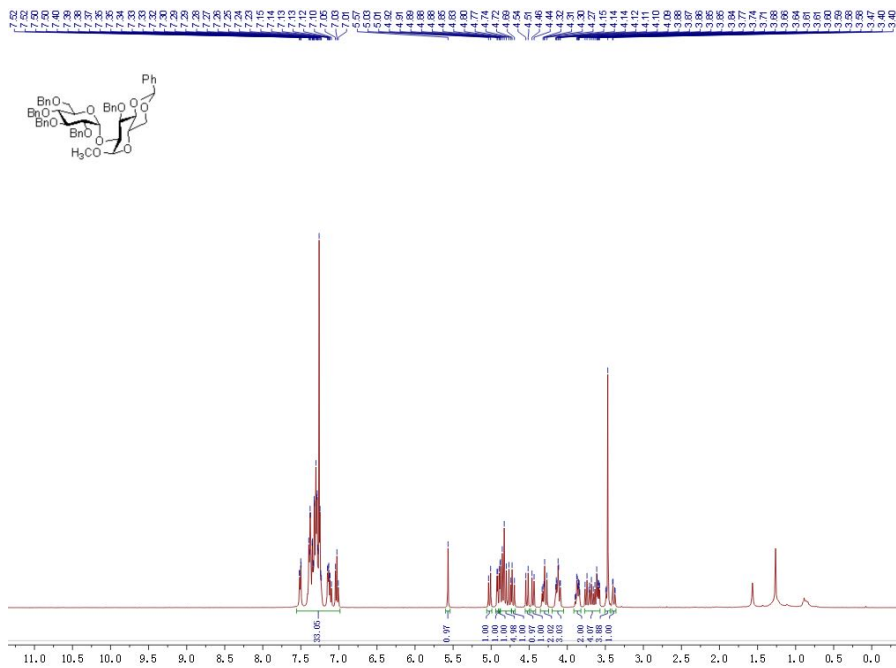


¹H NMR (400 MHz, CDCl₃) spectrum of compound 5a ($\alpha/\beta = 1.8:1.0$)

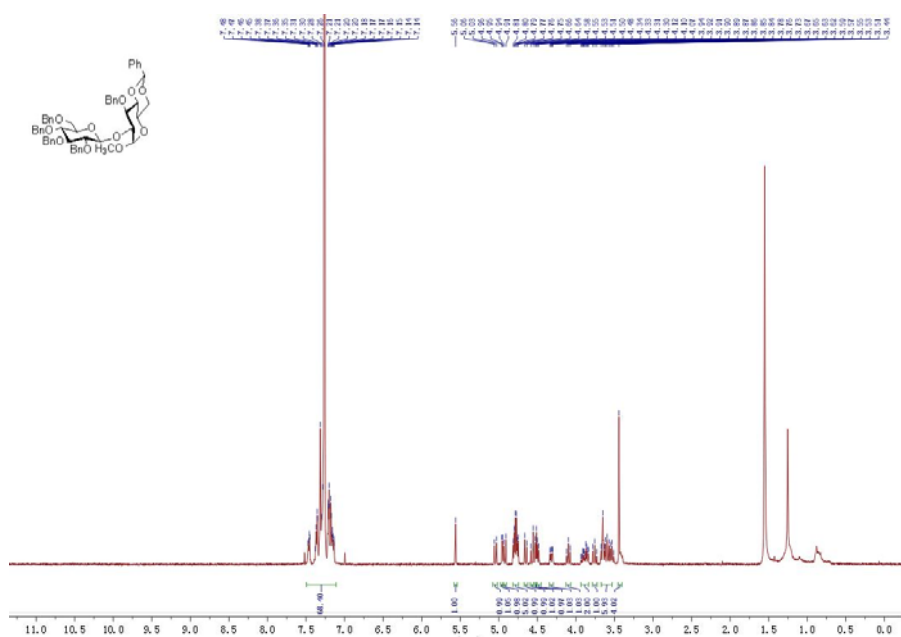
将下列数据与所测的谱图对照，以验证其正确性。如有不符，请重新测定。如有必要，请重新测定。如有必要，请重新测定。



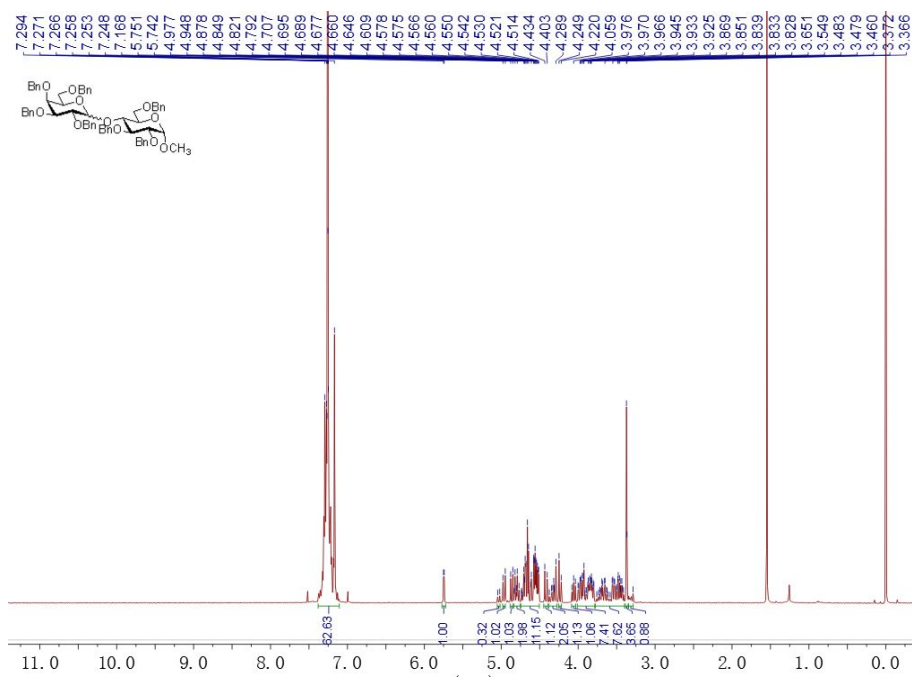
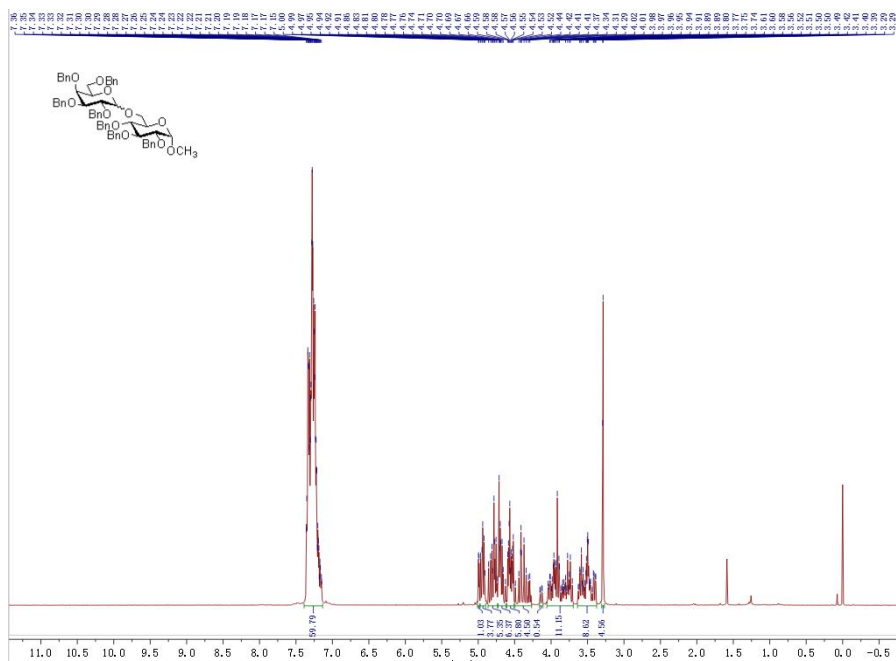
¹H NMR (400 MHz, CDCl₃) spectrum of compound 5b ($\alpha/\beta = 2.5:1.0$)

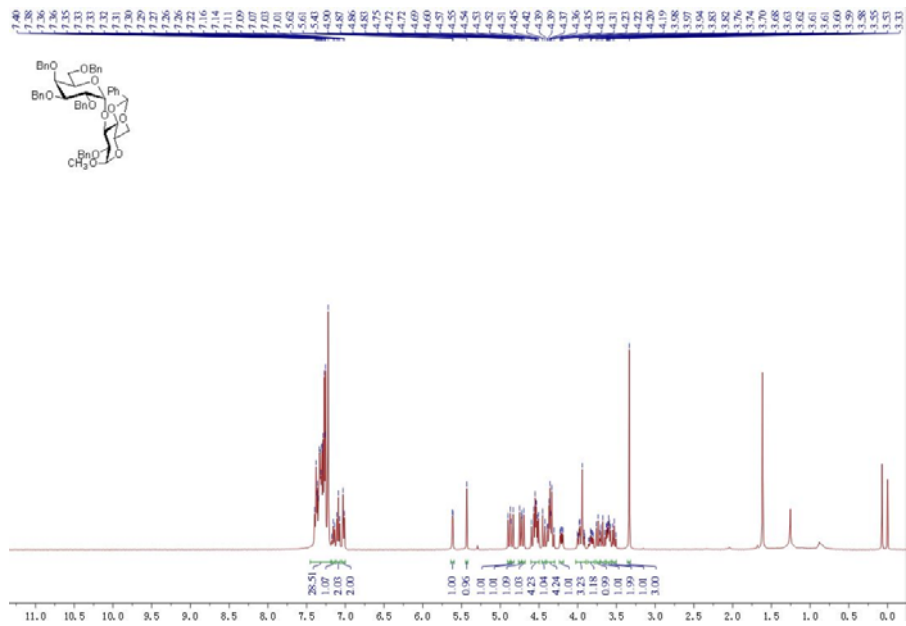


^1H NMR (400 MHz, CDCl_3) spectrum of compound 5d α

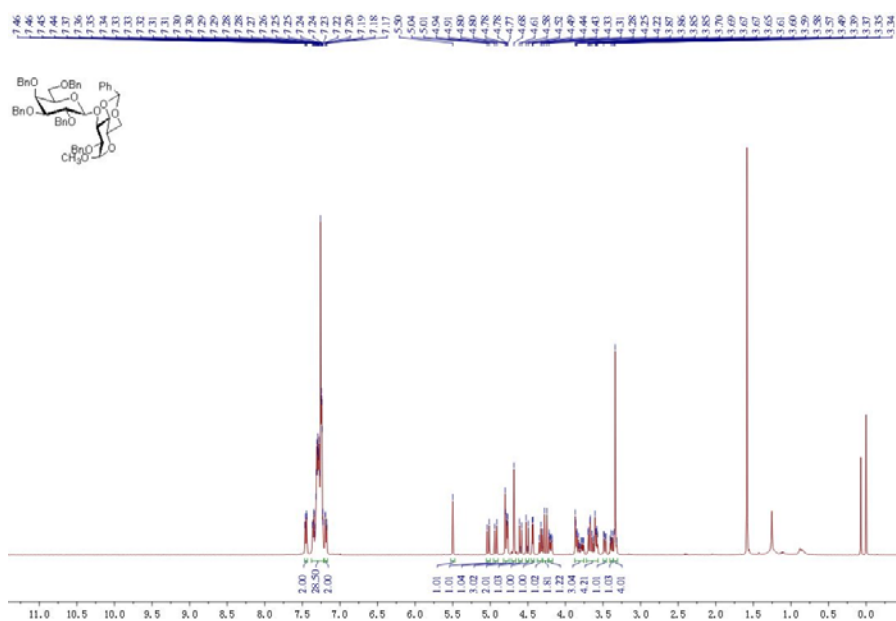


^1H NMR (400 MHz, CDCl_3) spectrum of compound 5d β

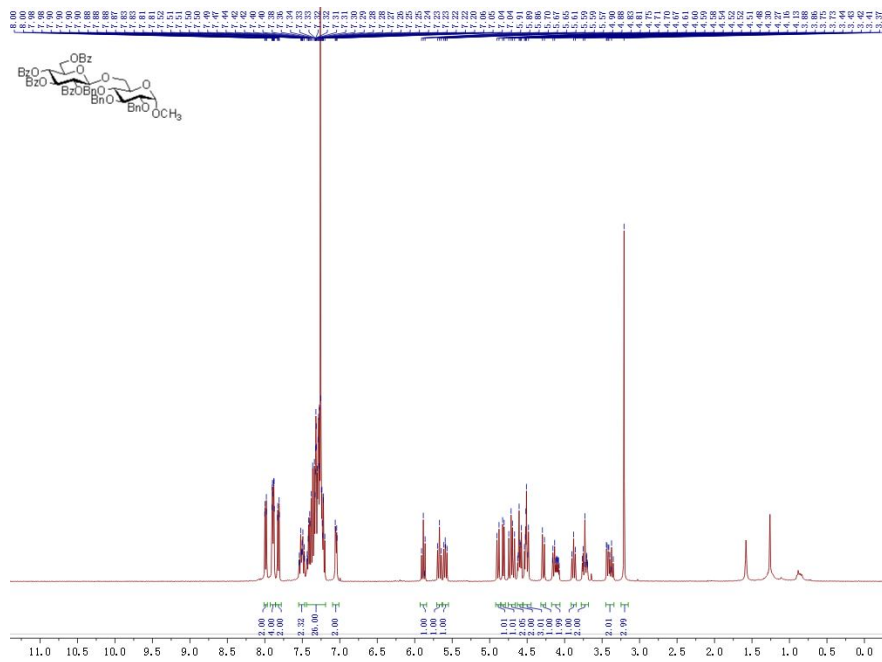




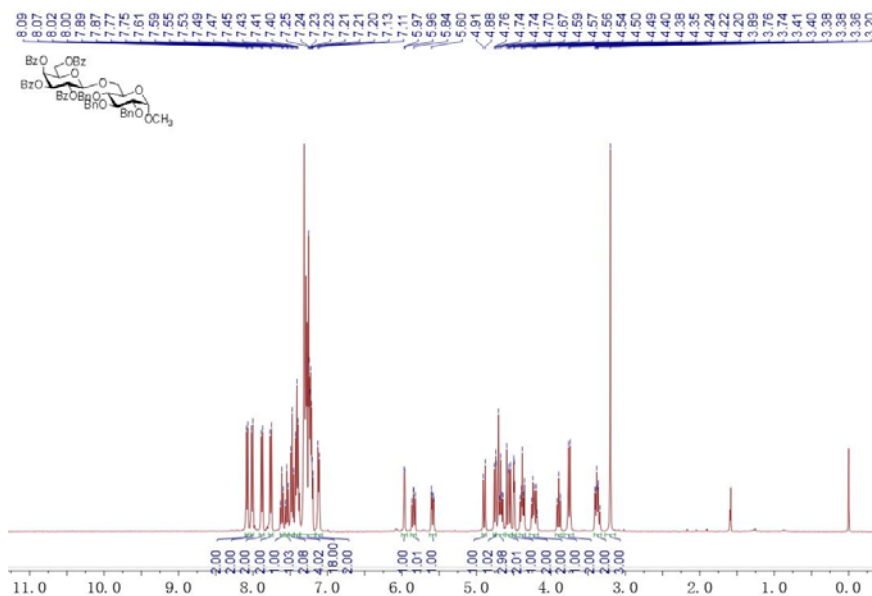
^1H NMR (400 MHz, CDCl_3) spectrum of compound 5g α



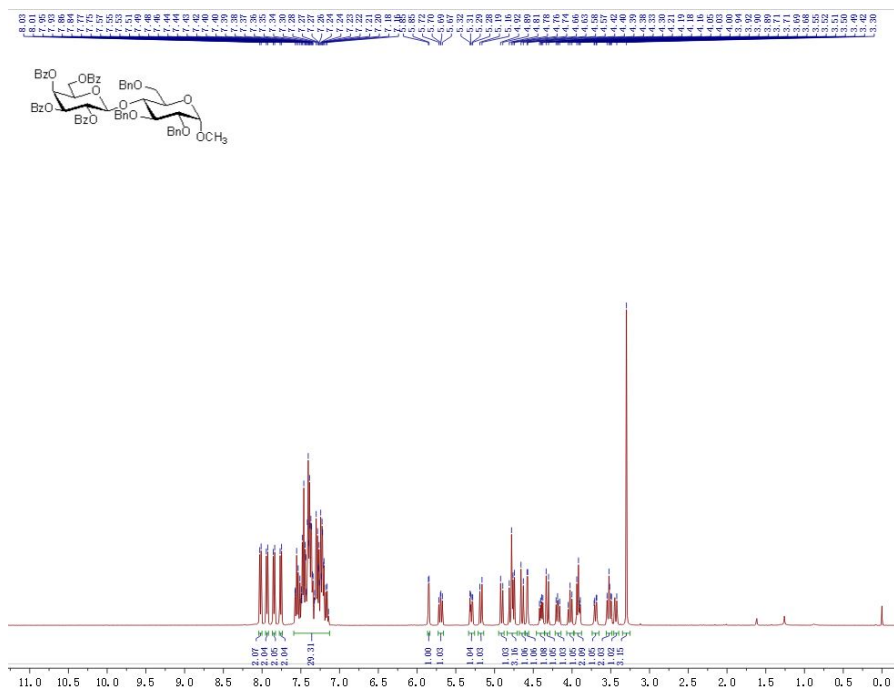
^1H NMR (400 MHz, CDCl_3) spectrum of compound 5g β



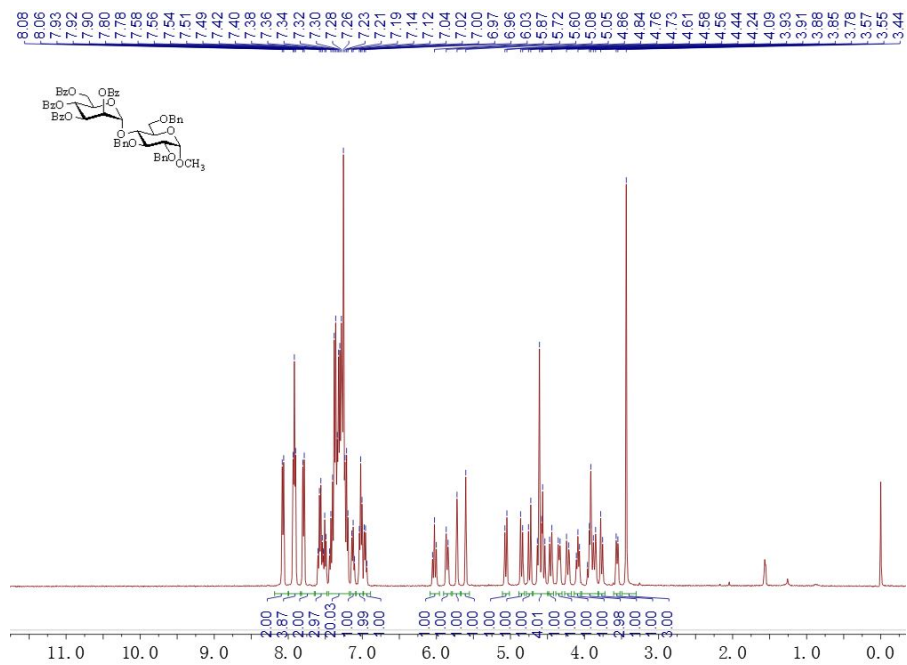
¹H NMR (400 MHz, CDCl₃) spectrum of compound 5h



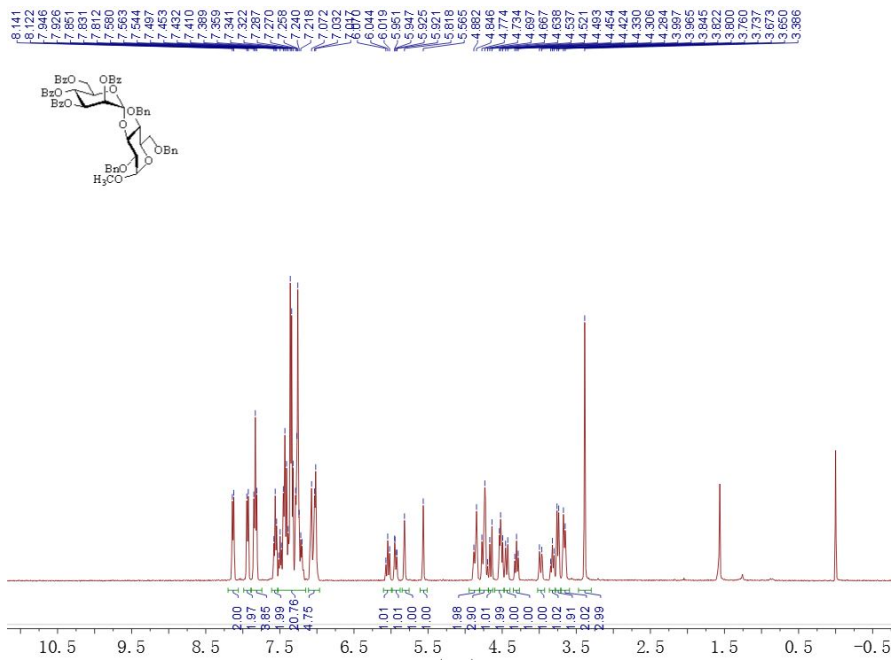
¹H NMR (400 MHz, CDCl₃) spectrum of compound 5i



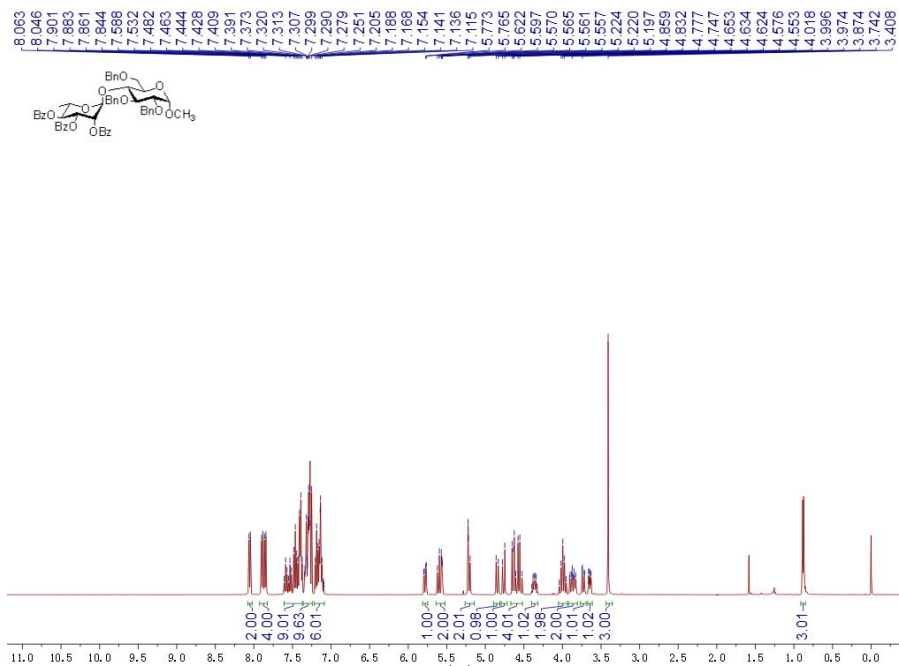
¹H NMR (400 MHz, CDCl₃) spectrum of compound 5j



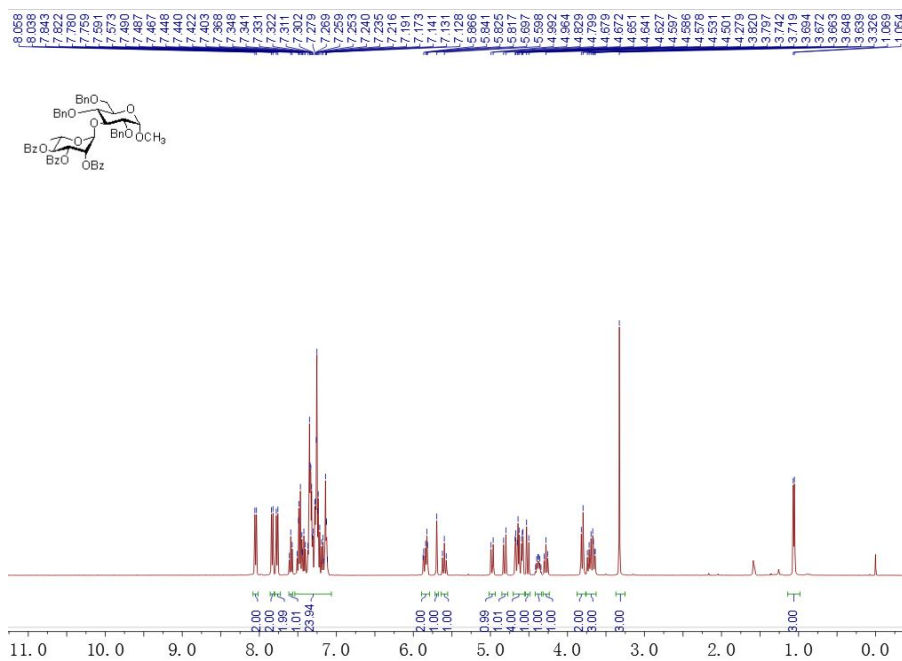
¹H NMR (400 MHz, CDCl₃) spectrum of compound 5k



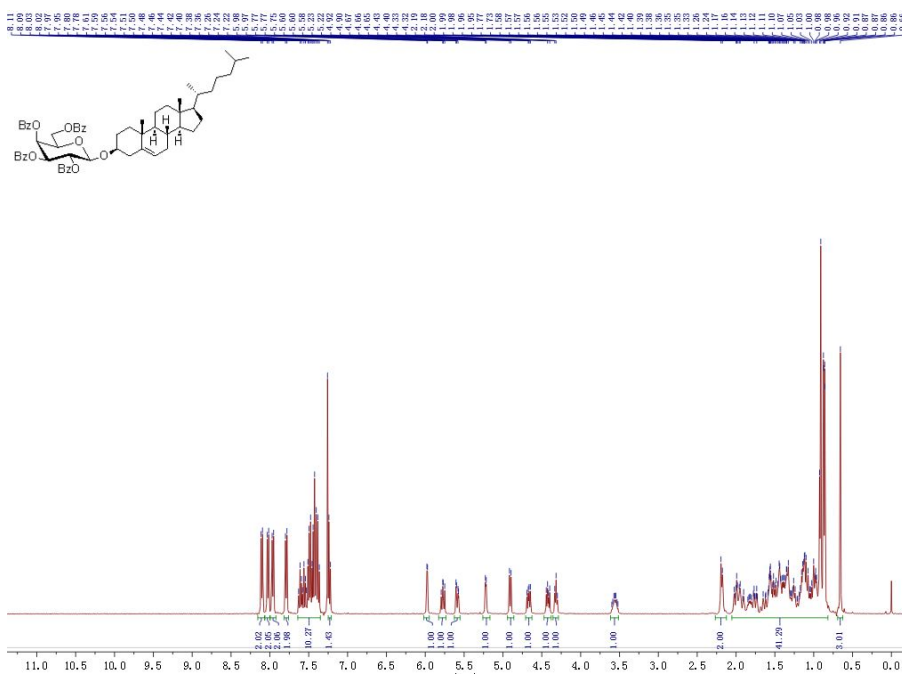
¹H NMR (400 MHz, CDCl₃) spectrum of compound 5l

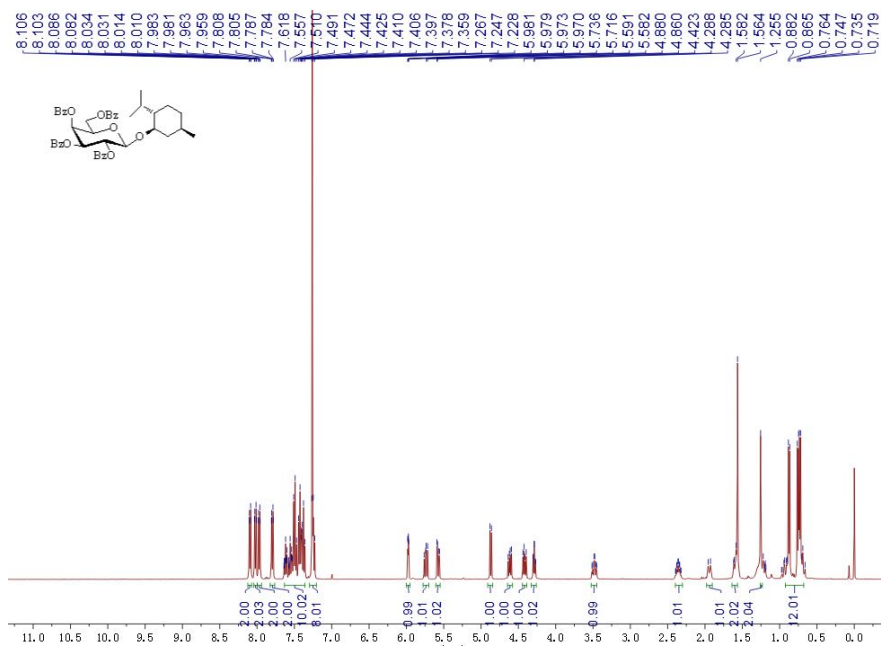


¹H NMR (400 MHz, CDCl₃) spectrum of compound 5m



¹H NMR (400 MHz, CDCl₃) spectrum of compound 5n





¹H NMR (400 MHz, CDCl₃) spectrum of compound 5p

References:

1. L. F. Sun, X. W. Wu, D-C. Xiong, X-S. Ye. *Angew. Chem., Int. Ed.* **2016**, *55*, 8041-8044.
2. D-C Xiong, L-H. Zhang, X-S. Ye. *Adv. Synth. Catal.* **2008**, *350*, 1696-1700.
3. M. Liu, B-H. Li, D-C. Xiong, X-S. Ye. *J. Org. Chem.* **2018**, *83*, 8292-8303.