

Supporting Information

Synthesis of Cyclic β -1,6-Oligosaccharides by Electrochemical Polyglycosylation of glucosamine monomers

Md Azadur Rahman¹, Hirofumi Endo¹, Takashi Yamamoto¹, Shoma Okushiba¹, Norihiko Sasaki^{1,2},
and Toshiki Nokami^{*1,2}.

¹ Department of Chemistry and Biotechnology, Tottori University, 4-101 Koyamacho-minami,
Tottori city, 680-8552 Tottori, Japan

²Center for Research on Green Sustainable Chemistry, Faculty of Engineering, Tottori University,
4-101 Koyamacho-minami, Tottori city, 680-8552 Tottori, Japan

*Corresponding author

Email: Toshiki Nokami — tnokami@tottori-u.ac.jp

Contents

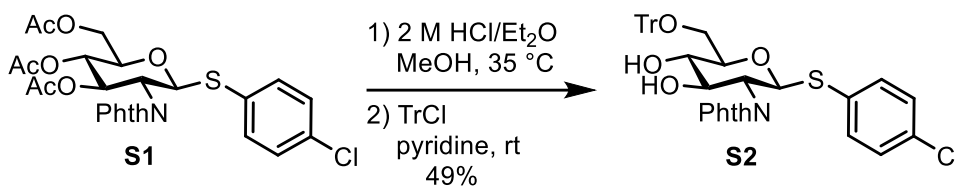
1. General	S2
2. Preparation of building blocks	S2
3. General procedure for cyclic oligoglucosamine synthesis with phthalimide group	S13
4. General procedure for cyclic oligoglucosamine synthesis with 2,3-oxazolidinone group	S17
5. General procedure for cyclic oligoglucosamine synthesis with azido group	S18
6. Molecular Orbital Calculations of anhydro sugars	S21
7. References	S23
8. ¹ H and ¹³ C NMR spectra of monosaccharides and oligosaccharides	S24

1. General

All reactions were conducted under argon atmosphere except for notice. ^1H NMR and ^{13}C NMR spectra were recorded on Bruker AVANCE II 600 (600 MHz for ^1H and 150 MHz for ^{13}C) and JEOL JNM-ECZ 600 (600 MHz for ^1H and 150 MHz for ^{13}C). ESI-MS spectra were recorded on Thermo Scientific Exactive spectrometer. MALDI-TOF MS spectra were recorded on Bruker Ultraflextreme spectrometer. Merck TLC (silica gel 60 F₂₅₄) was used for TLC analysis. Gel permeation chromatography (GPC) was used with JAI Labo Ace LC-5060 recycling preparative HPLC (eluent: CHCl_3). Kanto silica gel (spherical, neutral, 63–210 μm). Starting materials **S1**,¹ **S4**,² **S5**,² and **S11**³ were prepared according to the reported procedures. All reagents were purchased from commercial suppliers and used without extra purification. Products **7a**,⁴ **7b**,⁵ **7c**,⁶ and **8a**⁷ were known compounds and NMR spectra of these compounds were compared with reported values.

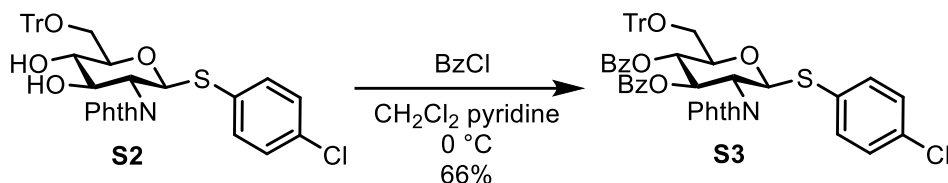
2. Preparation of building blocks

Preparation of 4-Chlorophenyl 2-deoxy-2-phthalimido-1-thio-6-*O*-trityl- β -D-glucopyranoside (**S2**)



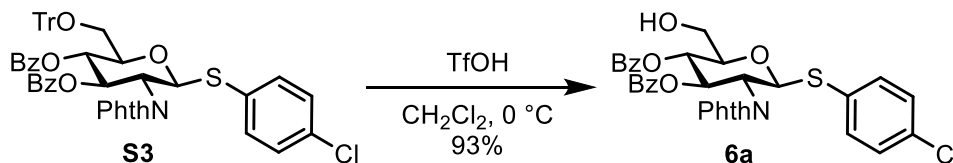
To a stirred solution of **S1**¹ (4.64 mmol, 2.61 g) in methanol (50 mL) at 35 °C was added 2 M HCl/Et₂O (10 mL). After 2 hours, the progress of the reaction was checked by TLC analysis, and the solution was concentrated under reduced pressure to afford deacetylated sugar. Then, the crude product and trityl chloride (TrCl) (6.96 mmol, 1.94 g, 1.5 eq.) were dissolved in pyridine (20 mL). The reaction mixture was kept stirred overnight at which point the TLC analysis indicated consumption of the starting material. The reaction was quenched with excessive methanol, and the solution was removed under the reduced pressure. The crude product was purified with silica gel chromatography (Hexane/EtOAc 3:2) to obtain **S2** (2.29 mmol, 1.55 g, 49%) as a white solid. **4-Chlorophenyl 2-deoxy-2-phthalimido-1-thio-6-*O*-trityl- β -D-glucopyranoside (**S2**)** TLC (eluent: Hexane/EtOAc 1:1) R_f = 0.22; ^1H NMR (CDCl_3 , 600 MHz) δ 7.92–7.82 (m, 2H), 7.78–7.74 (m, 2 H), 7.49–7.45 (m, 6 H), 7.42–7.39 (m, 2 H), 7.36–7.32 (m, 6 H), 7.30–7.27 (m, 3 H), 7.21–7.18 (m, 2 H), 5.56 (d, J = 10.3 Hz, 1 H), 4.30 (ddd, J = 12.7, 8.6, 4.1 Hz, 1 H), 4.20 (*pseudo-t*, J = 10.3 Hz, 1 H), 3.62 (td, J = 8.8, 3.2 Hz, 1 H), 3.59–3.54 (m, 1 H), 3.53 (dd, J = 10.0, 4.0 Hz, 1 H), 3.45 (dd, J = 10.0, 4.5 Hz, 1 H) 2.50 (d, J = 3.2 Hz, 1 H), 2.36 (d, J = 4.3 Hz, 1 H); ^{13}C NMR (CDCl_3 , 150 MHz) δ 143.6, 134.4, 134.3, 130.3, 129.1, 128.6, 128.0, 127.3, 87.1, 83.2, 78.3, 72.9, 72.7, 63.6, 56.2; HRMS (ESI) m/z calcd for $\text{C}_{39}\text{H}_{32}\text{ClKNO}_6\text{S}$; $[\text{M}+\text{K}]^+$, 716.1271, found 716.1211.

Preparation of 4-Chlorophenyl 3,4-di-*O*-benzoyl-2-deoxy-2-phthalimido-6-*O*-trityl-1-thio- β -D-glucopyranoside (**S3**)



To a stirred solution of **S2** (2.19 mmol, 1.48 g) in CH_2Cl_2 (25 mL) and pyridine (5.0 mL) was added benzyl chloride (BzCl) (11.0 mmol, 0.890 mL, 5.0 eq.) dropwise. The reaction was kept stirring over a period of 4 hours at 0 °C, and the progress of the reaction was checked by TLC analysis. The solution was concentrated under the reduced pressure. The mixture was diluted with in EtOAc (100 mL) and washed with 1 M HCl aqueous solution (50 mL \times 2), saturated aqueous NaHCO_3 solution (50 mL \times 2), and brine (50 mL). The organic layer was dried over anhydrous Na_2SO_4 , filtered and concentrated under reduced pressure to give the crude product. The crude product was purified with silica gel chromatography (Hexane/EtOAc 3:1) to afford **S3** (1.45 mmol, 1.28 g, 66%) as a white solid. **4-Chlorophenyl 3,4-di-*O*-benzoyl-2-deoxy-2-phthalimido-6-*O*-trityl-1-thio- β -D-glucopyranoside (**S3**)** TLC (eluent: Hexane/EtOAc 2:1) R_f = 0.55; ^1H NMR (CDCl_3 , 600 MHz) δ 7.92–7.88 (m, 2 H), 7.77–7.65 (m, 7 H), 7.55–7.52 (m, 2 H), 7.50–7.46 (m, 1 H), 7.44–7.40 (m, 6 H), 7.30–7.27 (m, 4 H), 7.24–7.22 (m, 2 H), 7.20–7.15 (m, 6 H), 7.14–7.12 (m, 3 H); 6.14 (dd, J = 10.3, 9.3 Hz, 1 H), 5.84 (d, J = 10.5 Hz, 1 H), 5.58 (*pseudo*-t, J = 9.8 Hz, 1 H), 4.61 (*pseudo*-t, J = 6.7 Hz, 1 H), 3.98 (ddd, J = 10.1, 5.3, 2.3 Hz, 1 H), 3.35 (dd, J = 11.0, 2.2 Hz, 1 H), 3.30 (dd, J = 10.6, 7.6 Hz, 1 H); ^{13}C NMR (CDCl_3 , 150 MHz) δ 168.1, 167.0, 165.8, 164.8, 143.6, 134.9, 134.8, 134.4, 134.3, 133.3, 133.1, 131.7, 129.8, 129.8, 129.7, 129.2, 129.0, 128.6, 128.6, 128.3, 128.2, 127.8, 127.0, 123.8, 123.7, 86.8, 83.2, 78.2, 72.4, 69.4, 62.4, 54.0; HRMS (ESI) m/z calcd for $\text{C}_{53}\text{H}_{40}\text{ClKNO}_8\text{S}$; $[\text{M}+\text{K}]^+$, 924.1795, found 924.1733.

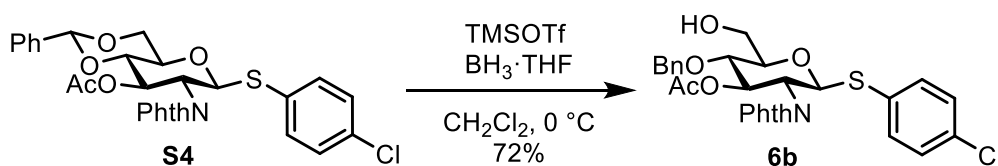
Preparation of 4-Chlorophenyl 3,4-di-*O*-benzoyl-2-deoxy-2-phthalimido-1-thio- β -D-glucopyranoside (**6a**)



To a stirred solution of **S3** (1.49 mmol, 1.32 g) in CH_2Cl_2 (20 mL) was added trifluoromethanesulfonic acid (TfOH) dropwise (1.63 mmol, 143 μL , 1.1 eq.) at 0 °C. The reaction was kept stirring until the reaction complete (*ca.* 3 hours). The reaction was quenched with saturated aqueous NaHCO_3 solution, and the solvent was removed under the reduced pressure. The reaction mixture was dissolved in EtOAc

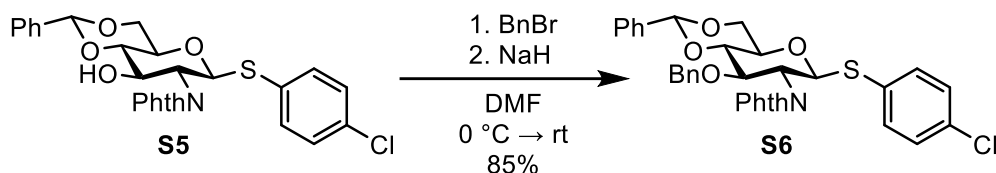
(50 mL) and washed with H₂O (50 mL ×3). The organic layer was dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure to obtain the crude product. The crude product was purified with silica gel chromatography (Hexane/EtOAc 3:1) to afford **6a** (1.38 mmol, 889 mg, 93%) as a white solid. **4-Chlorophenyl 3,4-di-O-benzoyl-2-deoxy-2-phthalimido-1-thio-β-D-glucopyranoside (6a)** TLC (eluent: Hexane/EtOAc 1:1) R_f = 0.54; ¹H NMR (CDCl₃, 600 MHz) δ 7.95–7.92 (m, 2 H), 7.92–7.87 (m, 1 H), 7.76–7.72 (m, 4 H), 7.72–7.67 (m, 1 H), 7.54–7.49 (m, 1 H), 7.45–7.35 (m, 5 H), 7.32–7.27 (m, 3 H), 7.26–7.24 (m, 1 H), 6.32 (*pseudo-t*, *J* = 9.8 Hz, 1 H), 5.85 (d, *J* = 10.7 Hz, 1 H), 5.49 (*pseudo-t*, *J* = 9.6 Hz, 1 H), 4.56 (*pseudo-t*, *J* = 10.2 Hz, 1 H) 3.95–3.85 (m, 2 H), 3.73 (dd, *J* = 8.0, 4.6 Hz, 1 H); 2.51 (*pseudo-t*, *J* = 7.9 Hz, 1 H); ¹³C NMR (CDCl₃, 150 MHz) δ 168.1, 166.9, 166.0, 165.7, 135.0, 134.8, 134.5, 134.3, 133.7, 133.4, 131.5, 131.1, 130.0, 129.7, 129.3, 129.3, 128.5, 128.5, 128.5, 128.4, 123.8, 83.1, 78.8, 71.7, 69.7, 61.5, 53.9; HRMS (ESI) *m/z* calcd for C₃₄H₂₆ClKNO₈S; [M+K]⁺, 682.0700, found 682.0688.

Preparation of 4-chlorophenyl 3-O-acetyl-4-O-benzyl-2-deoxy-2-phthalimido-1-thio-β-D-glucopyranoside (**6b**)



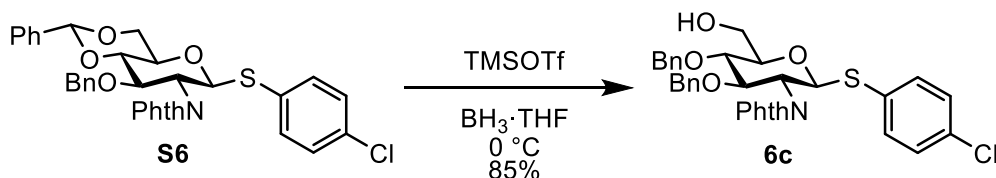
To a stirred solution of **S4**² (5.51 mmol, 3.12 g) in CH₂Cl₂ (24 mL) was added BH₃·THF (6.0 mL) at 0 °C. Then, trimethylsilyl trimethylsilyl trifluoromethanesulfonate (TMSOTf) (7.69 mmol, 1.39 mL, 1.4 eq.) was added dropwise. After 3 hours, the completion of the reaction was checked TLC analysis, and the reaction was quenched with saturated aqueous NaHCO₃ solution. Then, the solvent was removed under reduced pressure. The mixture was dissolved in EtOAc (100 mL) and washed with H₂O (100 mL ×3). The organic layer was dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure to give the crude product. The crude product was purified with silica gel chromatography (Hexane/EtOAc 1:1) to obtain **6b** (3.97 mmol, 2.26 g, 72%) as a white solid. **4-Chlorophenyl 3-O-acetyl-4-O-benzyl-2-deoxy-2-phthalimido-1-thio-β-D-glucopyranoside (6b)** TLC (eluent: Hexane/EtOAc 1:1) R_f = 0.48; ¹H NMR (CDCl₃, 600 MHz) δ 7.90–7.83 (m, 2 H), 7.77–7.73 (m, 2 H), 7.35–7.31 (m, 4 H), 7.29–7.27 (m, 3 H), 7.25–7.23 (m, 2 H), 5.79 (*pseudo-t*, *J* = 9.5 Hz, 1 H), 5.74 (d, *J* = 5.3 Hz, 1 H), 4.67 (d, *J* = 11.4 Hz, 1 H), 4.64 (d, *J* = 11.5 Hz, 1 H), 4.21 (*pseudo-t*, *J* = 10.2 Hz, 1 H), 3.95 (dd, *J* = 11.6, 3.5 Hz, 1 H), 3.80–3.76 (m, 1 H), 3.76 (*pseudo-t*, *J* = 8.0 Hz, 1 H) 3.65 (m, 1 H), 1.83 (dd, *J* = 8.5, 5.5 Hz, 1 H), 1.76 (s, 3 H); ¹³C NMR (CDCl₃, 150 MHz) δ 170.1, 167.8, 167.3, 137.6, 134.7, 134.5, 134.4, 134.2, 131.6, 131.1, 129.6, 129.2, 128.5, 123.0, 127.8, 123.7, 123.6, 82.8, 79.5, 75.9, 74.8, 73.9, 61.7, 54.1, 20.5; HRMS (ESI) *m/z* calcd for C₂₉H₂₆ClKNO₇S; [M+K]⁺, 606.0751, found 606.0741.

Preparation of 4-Chlorophenyl 3-*O*-benzyl-4,6-*O*-benzylidene-2-deoxy-2-phthalimido-1-thio-β-D-glucopyranoside (**S6**)



To a stirred solution of **S5**² (1.33 mmol, 0.696 g) in DMF (15 mL) was added benzyl bromide (BnBr) (4.00 mmol, 476 μL) at 0 °C. Then, 60% sodium hydride (NaH) (160 mg, 4.00 mmol) in DMF (4 mL) was added dropwise. The reaction was kept stirring overnight and the progress of the reaction was checked by TLC analysis. The reaction was quenched with saturated aqueous NaHCO₃ solution, and the solution was diluted with in EtOAc (50 mL) and washed with 1 M HCl aqueous solution (50 mL ×2), saturated aqueous NaHCO₃ solution (50 mL ×2), and brine (50 mL). The organic layer was dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure to give the crude product. The crude product was purified with silica gel chromatography (Hexane/EtOAc 5:1) to afford **S6** (1.13 mmol, 0.690 g, 85%) as a white solid. **4-Chlorophenyl 3-*O*-benzyl-4,6-*O*-benzylidene-2-deoxy-2-phthalimido-1-thio-β-D-glucopyranoside (**S6**)** TLC (eluent: Hexane/EtOAc 5:1) *R*_f = 0.39; ¹H NMR (600 MHz, CDCl₃) δ 7.89–7.84 (m, 1 H), 7.77–7.70 (m, 2 H), 7.66–7.60 (m, 1 H), 7.53–7.48 (m, 2 H), 7.44–7.36 (m, 3 H), 7.33–7.28 (m, 2 H), 7.24–7.20 (m, 2 H), 6.98–6.95 (m, 2 H), 6.93–6.88 (m, 1 H), 6.87–6.84 (m, 2 H), 5.63 (s, 1 H), 5.60 (d, *J* = 10.6 Hz, 1 H), 4.75 (d, *J* = 12.3 Hz, 1 H), 4.48 (d, *J* = 12.4 Hz, 1 H), 4.42 (dd, *J* = 10.5, 4.9 Hz, 1 H), 4.40 (*pseudo-t*, *J* = 9.3 Hz, 1 H), 4.24 (*pseudo-t*, *J* = 10.3 Hz, 1 H), 3.83 (*pseudo-t*, *J* = 10.3 Hz, 1 H), 3.77 (*pseudo-t*, *J* = 9.1 Hz, 1 H), 3.70 (td, *J* = 9.7, 4.9 Hz, 1 H); ¹³C NMR (150 MHz, CDCl₃) δ 167.8, 167.2, 137.7, 137.3, 134.7, 134.6, 134.1, 134.0, 131.5, 129.7, 129.1, 128.3, 128.2, 128.1, 127.5, 126.1, 123.6, 123.4, 101.4, 83.8, 82.8, 75.4, 74.3, 70.4, 68.6, 54.7; HRMS (ESI) *m/z* calcd for C₃₄H₂₈ClKNO₆S [M+Na]⁺, 652.0958; found, 652.0943.

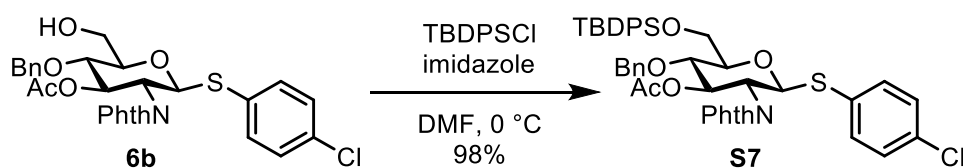
Preparation of 4-Chlorophenyl 3,4-di-*O*-benzyl-2-deoxy-2-phthalimido-1-thio-β-D-glucopyranoside (**6c**)



To a stirred solution of **S6** (1.38 mmol, .849 mg) and BH₃·THF (7.0 mL), TMSOTf (0.70 mL) was added dropwise at 0 °C. Then, the mixture was stirred for 4 hours at room temperature. The reaction was quenched with saturated aqueous NaHCO₃ solution, and the solution was diluted in EtOAc. The mixture was washed with saturated aqueous NaHCO₃ solution (3 times), H₂O (3 times) and brine respectively. The organic layer was dried over anhydrous Na₂SO₄, filtered, and concentrated under

reduced pressure. The crude product was purified with silica gel chromatography (Hexane/EtOAc 3:1) to give **6c** (1.17 mmol, 730 mg, 85%) as a white solid. **4-Chlorophenyl 3,4-di-O-benzyl-2-deoxy-2-phthalimido-1-thio-β-D-glucopyranoside (6c)** TLC (eluent: Hexane/EtOAc 2:1) R_f = 0.50; ^1H NMR (600 MHz, CDCl_3) δ 7.81–7.60 (m, 4 H), 7.37–7.32 (m, 4 H), 7.31–7.25 (m, 3 H), 7.20–7.18 (m, 2 H), 6.99–6.97 (m, 2 H), 6.89–6.82 (m, 3 H), 5.54 (d, J = 10.5 Hz, 1 H), 4.87 (d, J = 10.8 Hz, 1 H), 4.79 (d, J = 12.3 Hz, 1 H), 4.72 (d, J = 10.8 Hz, 1 H), 4.44 (d, J = 12.3 Hz, 1 H), 4.37 (dd, J = 10.2, 9.0 Hz, 1 H), 4.19 (*pseudo-t*, J = 10.5 Hz, 1 H), 3.93 (dd, J = 12.3, 2.4 Hz, 1 H), 3.76 (dd, J = 12.3, 4.5 Hz, 1 H), 3.72–3.68 (m, 1 H), 3.59 (dd, J = 9.9, 2.4 Hz, 1 H), 2.16–2.04 (m, 1 H); ^{13}C NMR (150 MHz, CDCl_3) δ 168.2, 167.5, 138.0, 137.9, 134.3, 134.2, 134.1, 133.9, 131.7, 131.5, 130.6, 129.2, 128.7, 128.3, 128.2, 128.1, 127.6, 123.6, 123.6, 83.4, 80.2, 80.0, 79.3, 77.7, 77.5, 77.3, 75.2, 75.1, 61.8, 55.1; HRMS (ESI) m/z calcd for $\text{C}_{34}\text{H}_{30}\text{ClNaO}_6\text{S}$ $[\text{M}+\text{Na}]^+$, 638.1375; found, 638.1357.

Preparation of 4-Chlorophenyl 3-*O*-acetyl-4-*O*-benzyl-6-*O*-*tert*-butyldiphenylsilyl-2-deoxy-1-thio-β-D-glucopyranoside (**S7**)

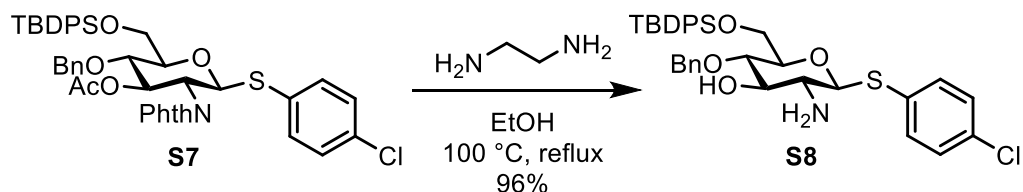


To a stirred solution of **6b** (3.61 mmol, 2.05 g) and imidazole (14.4 mmol, 1.06 g, 4.0 eq.) in DMF (15 mL) was added *tert*-butyldiphenylsilyl chloride (TBDPSCI) (10.8 mmol, 3.16 mL, 3.0 eq.) dropwise at 0 °C. The completion of the reaction was monitored by TLC analysis, and the reaction was quenched with saturated aqueous NaHCO_3 solution. The mixture was dissolved in EtOAc (100 mL) and washed with 1 M HCl aqueous solution (100 mL \times 2), saturated aqueous NaHCO_3 solution (50 mL \times 2), and brine (50 mL). The organic layer was dried over anhydrous Na_2SO_4 , filtered and concentrated under reduced pressure to give the crude product. The crude product was purified with silica gel chromatography (Hexane/EtOAc 5:1) to afford **S7** (3.52 mmol, 2.84 g, 98%) as a white solid.

4-Chlorophenyl 3-*O*-acetyl-4-*O*-benzyl-6-*O*-*tert*-butyldiphenylsilyl-2-deoxy-1-thio-β-D-glucopyranoside (S7**)** TLC (eluent: Hexane/EtOAc 3:1) R_f = 0.47; ^1H NMR (CDCl_3 , 600 MHz) δ 7.90–7.85 (m, 2 H), 7.80–7.77 (m, 2 H), 7.76–7.74 (m, 2 H), 7.72–7.69 (m, 2 H), 7.46–7.43 (m, 2 H), 7.40–7.35 (m, 6 H), 7.26–7.23 (m, 3 H), 7.15–7.10 (m, 4 H), 5.80 (*pseudo-t*, J = 9.5 Hz, 1 H), 5.71 (d, J = 10.4 Hz, 1 H), 4.68 (d, J = 11.6 Hz, 1 H), 4.63 (d, J = 11.6 Hz, 1 H), 4.26 (*pseudo-t*, J = 10.3 Hz, 1 H), 4.05–4.01 (m, 1 H), 3.97 (dd, J = 11.7, 2.8 Hz, 1 H), 3.93 (*pseudo-t*, J = 9.5 Hz, 1 H), 3.65 (dd, J = 10.1, 1.3 Hz, 1 H), 1.76 (s, 3 H), 1.12 (s, 9 H); ^{13}C NMR (CDCl_3 , 150 MHz) δ 170.2, 167.9, 167.4, 137.9, 135.9, 135.6, 134.5, 134.4, 134.3, 134.2, 133.3, 132.8, 131.8, 131.2, 130.0, 129.8, 129.1, 128.5,

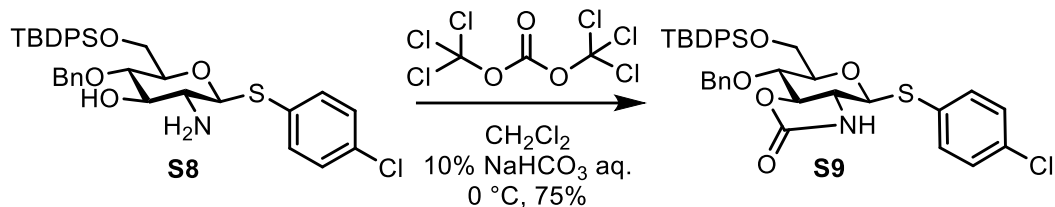
127.83, 127.79, 127.76, 127.5, 123.7, 123.6, 82.7, 80.1, 76.2, 74.9, 74.2, 62.5, 54.2, 26.9, 20.6, 19.4; HRMS (ESI) m/z calcd for $C_{45}H_{44}ClKNO_7SSi$; $[M+K]^+$, 844.1928, found 844.1914.

Preparation of 4-Chlorophenyl 2-amino-4-*O*-benzyl-6-*O*-*tert*-butyldiphenylsilyl-2-deoxy-1-thio- β -D-glucopyranoside (**S8**)



To a stirred solution of **S7** (3.82 mmol, 3.07 g) in ethanol (30 mL) was added ethylenediamine anhydrous (6.4 mL) at room temperature. The reaction temperature was gradually raised from room temperature to 100 °C and kept stirring. The progress of the reaction was checked by TLC analysis, and heating was halted in 3 hours. The solvent was removed under the reduced pressure, and the mixture was purified with silica gel chromatography (Hexane/EtOAc 1:3 + 1% Et_3N) to obtain **S8** (3.65 mmol, 2.31 g, 96%) as a white solid. **4-Chlorophenyl 2-amino-4-*O*-benzyl-6-*O*-*tert*-butyldiphenylsilyl-2-deoxy-1-thio- β -D-glucopyranoside (**S8**)** TLC (eluent: Hexane/EtOAc 1:3) R_f = 0.38; 1H NMR ($CDCl_3$, 600 MHz) δ 7.79–7.76 (m, 2 H), 7.73–7.70 (m, 2 H), 7.51–7.48 (m, 2 H), 7.45–7.40 (m, 2 H), 7.37–7.33 (m, 4 H), 7.32–7.27 (m, 3 H), 7.25–7.22 (m, 2 H), 7.17–7.13 (m, 2 H), 4.80 (d, J = 11.2 Hz, 1 H), 4.71 (d, J = 11.2 Hz, 1 H), 4.40 (d, J = 9.9 Hz, 1 H), 4.03–3.99 (m, 1 H), 3.95 (dd, J = 11.2, 3.9 Hz, 1 H), 3.64 (*pseudo-t*, J = 9.3 Hz, 1 H); 3.48 (*pseudo-t*, J = 9.2 Hz, 1 H), 3.41 (dd, J = 9.8, 2.5 Hz, 1 H), 2.69 (*pseudo-t*, J = 9.6 Hz, 1 H), 1.10 (s, 9 H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 138.3, 135.9, 135.6, 134.0, 133.6, 133.4, 133.0, 131.2, 129.8, 129.7, 129.1, 128.6, 127.9, 127.9, 127.8, 127.7, 89.3, 80.0, 78.3, 77.5, 74.8, 62.9, 56.1, 26.9, 19.4; HRMS (ESI) m/z calcd for $C_{35}H_{40}ClNNaO_4SSi$; $[M+Na]^+$, 656.2029, found 656.2006.

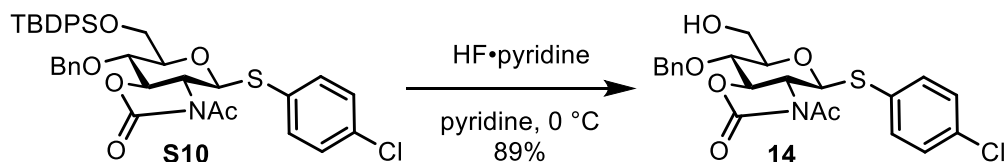
Preparation of 4-Chlorophenyl 4-*O*-benzyl-6-*O*-*tert*-butyldiphenylsilyl-2,3-*N,O*-carbonyl-2-deoxy-1-thio- β -D-glucopyranoside (**S9**)



To a stirred solution of **S8** (3.59 mmol, 2.28 g) in CH_2Cl_2 (50 mL) and 10% aqueous $NaHCO_3$ solution was added triphosgene (1.38 mmol, 408 mg, 0.383 eq.) at room temperature. The reaction was kept stirring for overnight. The completion of the reaction was checked by TLC analysis, then the solution

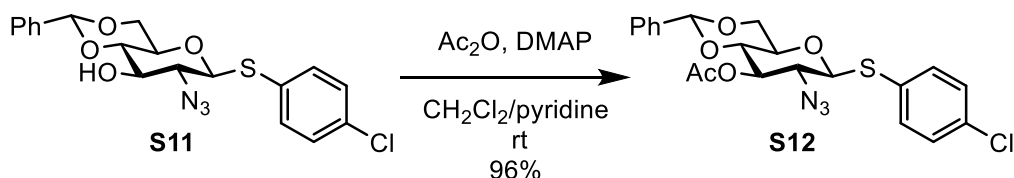
740.1654.

Preparation of 4-Chlorophenyl 2-acetamido-4-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy-1-thio- β -D-glucopyranoside (**14**)



To a stirred solution of **S10** (2.32 mmol, 1.63 g) in pyridine (20 mL) was added HF/pyridine (19.2 mmol, 2.53 mL, 8.3 eq.) dropwise at 0 °C. The reaction was monitored by TLC analysis and quenched with saturated aqueous NaHCO₃ solution when the reaction completed. The mixture was diluted with in EtOAc (50 mL) and washed with 1 M HCl aqueous solution (50 mL \times 3), saturated aqueous NaHCO₃ solution (50 mL \times 3), and brine (50 mL). The organic layer was dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure to obtain the crude product. The crude product was purified with silica gel chromatography to afford **14** (2.07 mmol, 963 mg, 89%) as a white solid. **4-Chlorophenyl 2-acetamido-4-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy-1-thio- β -D-glucopyranoside (**14**)** TLC (eluent: Hexane/EtOAc 3:1) R_f = 0.36; ¹H NMR (CDCl₃, 600 MHz) δ 7.43–7.40 (m, 2 H), 7.39–7.35 (m, 2 H), 7.35–7.30 (m, 3 H), 7.30–7.27 (m, 2 H), 4.894 (d, J = 11.0 Hz, 1 H), 4.892 (d, J = 8.6 Hz, 1 H), 4.63 (d, J = 11.4 Hz, 1 H), 4.38 (*pseudo-t*, J = 10.7 Hz, 1 H), 4.03 (dd, J = 11.3, 8.8 Hz, 1 H), 3.93 (*pseudo-t*, J = 9.3 Hz, 1 H), 3.82 (ddd, J = 11.5, 6.4, 2.2 Hz, 1 H), 3.72 (ddd, J = 13.7, 7.7, 4.5 Hz, 1 H), 3.52–3.46 (m, 1 H), 2.59 (s, 3 H), 1.84 (*pseudo-t*, J = 7.0 Hz, 1 H); ¹³C NMR (CDCl₃, 150 MHz) δ 173.1, 153.7, 136.9, 134.4, 133.5, 132.1, 129.2, 128.6, 128.3, 128.1, 86.7, 82.5, 80.7, 73.7, 73.5, 61.5, 59.8, 24.8; HRMS (ESI) m/z calcd for C₂₂H₂₂ClKNO₆S; [M+K]⁺, 502.0488, found 502.0474.

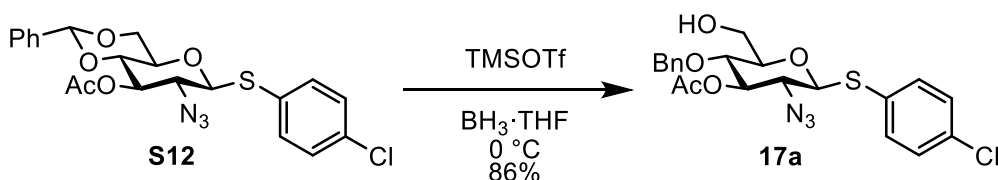
Preparation of 4-Chlorophenyl 3-*O*-acetyl-2-azido-4,6-*O*-benzylidene-2-deoxy-1-thio- β -D-glucopyranoside (**S12**)



To a stirred solution of **S11**³ (6.45 mmol, 2.70 g) and DMAP (84 mg) in CH₂Cl₂ (24 mL) and pyridine (7.0 mL), acetic anhydride (4.0 mL) was added dropwise. Then, the mixture was stirred overnight at room temperature. The reaction was quenched with 1 M HCl aqueous solution, and the mixture was diluted in EtOAc, and the organic solution was washed with 1 M HCl aqueous solution (3 times), saturated aqueous NaHCO₃ solution (3 times), H₂O (3 times), and brine respectively. The

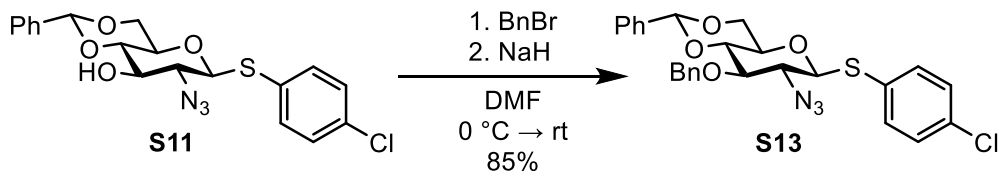
organic layer was dried over anhydrous Na_2SO_4 , filtered, and concentrated under reduced pressure. The crude product was purified with silica gel chromatography (Hexane/EtOAc 4:1) to afford **S12** (6.20 mmol, 2.89 g, 96%) as a white solid. **4-Chlorophenyl 3-O-acetyl-2-azido-4,6-O-benzylidene-2-deoxy-1-thio- β -D-glucopyranoside (S12)** TLC (eluent: Hexane/EtOAc 3:1) R_f = 0.50; ^1H NMR (600 MHz, CDCl_3) δ 7.50–7.48 (m, 2 H), 7.44–7.30 (m, 7 H), 5.45 (s, 1 H), 5.27 (*pseudo*-t, J = 9.6 Hz, 1 H), 4.55 (d, J = 10.2 Hz, 1 H), 4.32 (dd, J = 10.5, 4.8 Hz, 1 H), 3.72 (*pseudo*-t, J = 10.2 Hz, 1 H), 3.51 (*pseudo*-t, J = 9.6 Hz, 1 H), 3.46 (td, J = 9.6, 4.8, Hz, 1 H), 3.40 (*pseudo*-t, J = 9.9 Hz, 1 H), 2.10 (s, 3 H); ^{13}C NMR (150 MHz, CDCl_3) δ 169.6, 137.0, 135.2, 135.2, 129.5, 129.3, 128.4, 126.3, 101.5, 86.6, 78.3, 77.7, 77.5, 77.3, 73.0, 70.7, 68.3, 63.6, 20.9; HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{20}\text{ClKN}_3\text{O}_5\text{S}$ $[\text{M}+\text{K}]^+$, 500.0444; found, 500.0425.

Preparation of 4-Chlorophenyl 3-O-acetyl-2-azido-4-O-benzyl-2-deoxy-1-thio- β -D-glucopyranoside (**17a**)



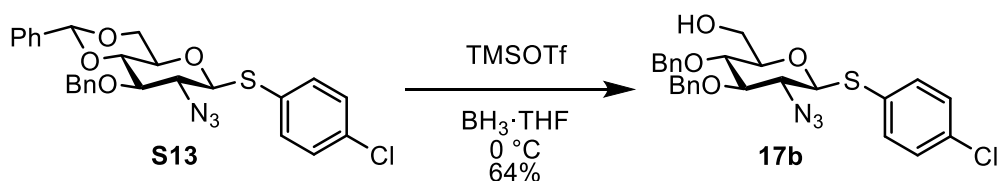
To a stirred solution of **S12** (1.37 mmol, 0.630 g), and $\text{BH}_3 \cdot \text{THF}$ (7.0 mL), TMSOTf (0.70 mL) was added dropwise at 0 °C. Then, the mixture was stirred for 4 hours at room temperature. The reaction was quenched with saturated aqueous NaHCO_3 solution. The mixture was diluted in EtOAc, and the organic solution was washed with saturated aqueous NaHCO_3 solution (3 times), H_2O (3 times) and brine respectively. The organic layer was dried over anhydrous Na_2SO_4 , filtered, and concentrated under reduced pressure. The crude product was purified with silica gel chromatography (Hexane/EtOAc 5:1) to afford **17a** (1.19 mmol, 0.550 g, 86%) as a white solid. **4-Chlorophenyl 3-O-acetyl-2-azido-4-O-benzyl-2-deoxy-1-thio- β -D-glucopyranoside (17a)** TLC (eluent: Hexane/EtOAc 2:1) R_f = 0.47; ^1H NMR (600 MHz, CDCl_3) δ 7.49–7.46 (m, 2 H), 7.34–7.28 (m, 5 H), 7.26–7.24 (m, 2 H), 5.15 (*pseudo*-t, J = 9.6 Hz, 1 H), 4.61 (d, J = 11.4 Hz, 1 H), 4.56 (d, J = 11.4 Hz, 1 H), 4.49 (d, J = 10.2 Hz, 1 H), 3.89 (ddd, J = 12.3, 5.4, 2.4 Hz, 1 H), 3.74–3.70 (m, 1 H), 3.57 (*pseudo*-t, J = 9.6 Hz, 1 H), 3.41 (ddd, J = 9.9, 3.9, 2.7 Hz, 1 H), 3.26 (*pseudo*-t, J = 9.9 Hz, 1 H), 2.00 (s, 3 H), 1.88 (dd, J = 8.0, 5.2 Hz, 1 H); ^{13}C NMR (150 MHz, CDCl_3) δ 169.9, 137.4, 135.2, 135.0, 129.5, 129.4, 128.7, 128.2, 128.1, 86.1, 79.7, 77.3, 77.1, 76.9, 75.9, 75.1, 74.8, 63.4, 61.6, 20.9; HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{22}\text{ClNaN}_3\text{O}_5\text{S}$ $[\text{M}+\text{Na}]^+$, 486.0861; found, 486.0842.

Preparation of 4-Chlorophenyl 2-azido-3-*O*-benzyl-4,6-*O*-benzylidene-2-deoxy-1-thio- β -D-glucopyranoside (**S13**)



To a stirred solution of **S11**³ (2.40 mmol, 1.01 g) in DMF (20 mL) was added 60% NaH (4.80 mmol, 192 mg) at 0 °C. Then, BnBr (3.60 mmol, 450 μ L) was added dropwise, and the reaction was kept stirring overnight. The progress of the reaction was monitored by TLC analysis, and the reaction was quenched with saturated aqueous NaHCO₃ solution. The solution was diluted with in EtOAc (100 mL) and washed with 1 M HCl aqueous solution (50 mL \times 2), saturated aqueous NaHCO₃ solution (50 mL \times 2), and brine (50 mL). The organic layer was dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure to give the crude product. The crude product was purified with silica gel chromatography (Hexane/EtOAc 8:1) to afford **S13** (2.06 mmol, 1.05 g, 86%) as a white solid. **4-Chlorophenyl 2-azido-3-*O*-benzyl-4,6-*O*-benzylidene-2-deoxy-1-thio- β -D-glucopyranoside (**S13**)** TLC (eluent: Hexane/EtOAc 8:1) R_f = 0.46; ¹H NMR (600 MHz, CDCl₃) δ 7.51–7.46 (m, 4 H), 7.40–7.37 (m, 3 H), 7.36–7.29 (m, 7 H), 5.57 (s, 1 H), 4.91 (d, J = 11.0 Hz, 1 H), 4.78 (d, J = 11.0 Hz, 1 H), 4.44 (d, J = 10.1 Hz, 1 H), 4.38 (dd, J = 10.6, 5.0 Hz, 1 H), 3.77 (*pseudo*-t, J = 10.3 Hz, 1 H), 3.66 (*pseudo*-t, J = 9.1 Hz, 1 H), 3.63 (*pseudo*-t, J = 9.2 Hz, 1 H), 3.48–3.42 (m, 1 H), 3.33 (*pseudo*-t, J = 9.3 Hz, 1 H); ¹³C NMR (150 MHz, CDCl₃) δ 137.5, 137.0, 135.4, 135.3, 129.4, 129.2, 129.0, 128.5, 128.4, 128.1, 126.0, 101.3, 86.5, 81.3, 80.9, 76.9, 75.2, 70.6, 68.5, 64.7; HRMS (ESI) m/z calcd for C₂₆H₂₄ClKN₃O₆S [M+Na]⁺, 548.0808; found, 548.0817.

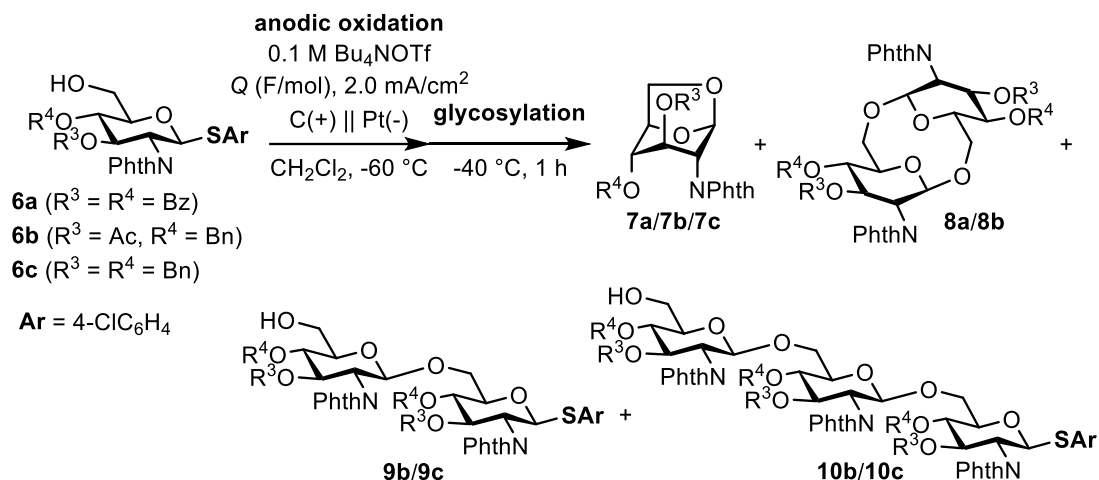
Preparation of 4-Chlorophenyl 2-azido-3,4-di-*O*-benzyl-2-deoxy-1-thio- β -D-glucopyranoside (**17b**)



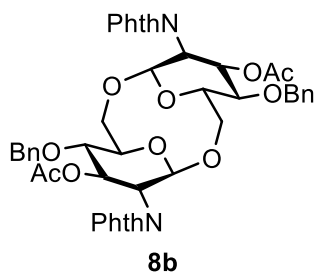
To a stirred solution of **S13** (2.31 mmol, 1.18 g), and BH₃·THF (12 mL), TMSOTf (1.2 mL) was added dropwise at 0 °C. Then, the mixture was stirred for 6 hours at room temperature. The reaction was quenched with saturated aqueous NaHCO₃ solution, and the reaction mixture diluted with in EtOAc. The solution was washed with saturated aqueous NaHCO₃ solution (3 times), H₂O (3 times) and brine respectively. The organic layer was dried over anhydrous Na₂SO₄, filtered, and concentrated under reduced pressure. The crude product was purified with silica gel chromatography (Hexane/EtOAc 5:1) to obtain **1d** (1.47 mmol, 0.750 g, 64%) as a white solid. **4-Chlorophenyl 2-**

azido-3,4-di-*O*-benzyl-2-deoxy-1-thio- β -D-glucopyranoside (17b) TLC (eluent: Hexane/EtOAc 3:1) R_f = 0.40; ^1H NMR (600 MHz, CDCl_3) δ 7.48–7.46 (m, 2 H), 7.36–7.27 (m, 12 H), 4.87 (d, J = 10.6 Hz, 1 H), 4.86 (d, J = 10.6 Hz, 1 H), 4.84–4.82 (m, 1 H), 4.64 (d, J = 11.1 Hz, 1 H), 4.41 (d, J = 10.2 Hz, 1 H), 3.87 (dd, J = 6.0, 2.7 Hz, 1 H), 3.71–3.67 (m, 1 H), 3.55–3.50 (m, 2 H), 3.36 (dd, J = 4.8, 2.7 Hz, 1 H), 3.30 (ddd, J = 10.2, 6.6, 2.7 Hz, 1 H), 1.82 (*pseudo*-t, J = 6.9 Hz, 1 H); ^{13}C NMR (150 MHz, CDCl_3) δ 137.6, 137.5, 135.1, 134f.9, 129.6, 129.4, 128.69, 128.65, 128.32, 128.21, 128.19, 128.12, 86.1, 84.9, 79.8, 76.0, 75.2, 65.3, 61.9; HRMS (ESI) m/z calcd for $\text{C}_{26}\text{H}_{26}\text{ClNaN}_3\text{O}_4\text{S}$ [$\text{M}+\text{Na}$] $^+$, 534.1225; found, 534.1211.

3. General procedure for cyclic oligoglucosamine synthesis with phthalimide group

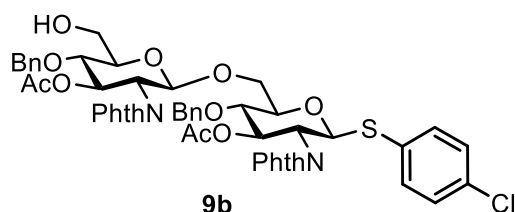


The electrochemical one-pot synthesis of cyclic oligosaccharides **8** and linear oligosaccharides **9-10**, was conducted with an H-type divided cell (4G glass filter). The cell had a carbon felt anode (Nippon Carbon JF-20-p7) and platinum square plate (20 mm×20 mm). Building block **6** (0.400 mmol), Bu₄NOTf (1.00 mmol), and CH₂Cl₂ (10 mL) were added to the anodic chamber. TfOH (0.400 mmol), Bu₄NOTf (1.00 mmol), and CH₂Cl₂ (10 mL) were added to the cathodic chamber. As the initiation phase of reaction, the constant current (8.0 mA (current density: 2.0 mA/cm²), (electrode distance: 4.5 cm)) was employed at -60 °C with magnetic stirring until electricity was consumed. After the electrolysis, the reaction temperature was raised from -60 °C to -40 °C as glycosylation phase. After glycosylation, Et₃N (0.5 mL) was added to both of the chambers. The mixture was collected, and the solvent was removed under reduced pressure. The mixture was dissolved in EtOAc and washed with H₂O to remove electrolyte. The solution was dried over anhydrous Na₂SO₄, filtered, and removed under reduced pressure. The crude product was purified with preparative-GPC to afford 1,6-anhydrosugar **7**,⁴⁻⁶ cyclic oligosaccharides **8**⁷ and linear oligosaccharides **9-10**.



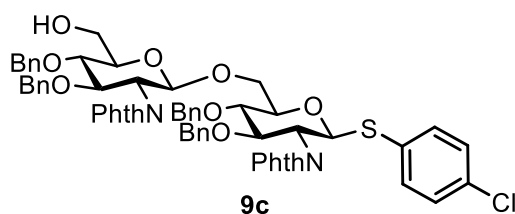
Building block **6b** (226 mg, 0.400 mmol) afforded **8b** as in 7% isolated yield (11 mg, 0.0130 mmol). **Cyclobis[(1→6)-3-O-acetyl-4-O-benzyl-2-deoxy-2-phthalimido-β-D-glucopyranosyl] (8b)** TLC (Hexane/EtOAc 1:1) R_f = 0.25; ¹H NMR (600 MHz, CDCl₃) δ 7.87–7.86 (m, 2 H), 7.74–7.72 (m, 2 H), 7.39–7.38 (m, 1 H), 7.32–7.26 (m, 4 H), 5.70 (dd, *J* = 10.8, 8.7 Hz, 1 H), 4.93 (d, *J* = 1.8 Hz, 1 H),

4.78 (d, $J = 11.7$ Hz, 1 H), 4.70 (d, $J = 11.7$ Hz, 1 H), 4.48–4.45 (m, 2 H), 4.16–4.14 (m, 1 H), 3.88 (dd, $J = 12.0, 2.7$ Hz, 1 H), 3.80 (d, $J = 11.7$ Hz, 1 H), 1.90 (s, 3 H); ^{13}C NMR (150 MHz, CDCl_3) δ 170.5, 167.7, 138.3, 134.4, 131.7, 129.4, 128.5, 127.9, 123.7, 100.1, 77.4, 75.0, 73.4, 71.8, 68.3, 57.7, 20.9; HRMS (ESI) m/z calcd for $\text{C}_{46}\text{H}_{42}\text{KN}_2\text{O}_{14}$ $[\text{M}+\text{K}]^+$, 885.2268; found, 885.2217.



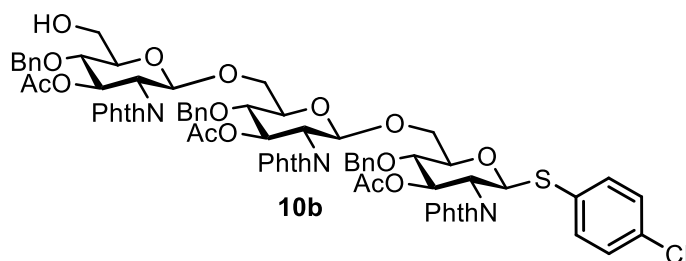
Building block **6b** (226 mg, 0.400 mmol) afforded **9b** as in 13% isolated yield (25 mg, 0.0253 mmol).

4-Chlorophenyl (3-*O*-acetyl-4-*O*-benzyl-2-deoxy-2-phthalimido- β -D-glucopyranosyl)-(1 \rightarrow 6)-3-*O*-acetyl-4-*O*-benzyl-2-deoxy-2-phthalimido-1-thio- β -D-glucopyranoside (9b**)** TLC (Hexane/EtOAc 1:1) $R_f = 0.33$; ^1H NMR (600 MHz, CDCl_3) δ 7.81–7.70 (m, 7 H), 7.61–7.58 (m, 2 H), 7.34–7.30 (m, 3 H), 7.29–7.27 (m, 4 H), 7.25–7.20 (m, 4 H), 7.02–7.01 (m, 2 H), 5.79 (dd, $J = 10.5, 9.0$ Hz, 1 H), 5.66 (dd, $J = 10.2, 9.0$ Hz, 1 H), 5.56 (d, $J = 10.5$ Hz, 1 H), 5.53 (d, $J = 8.4$ Hz, 1 H), 4.69 (d, $J = 11.4$ Hz, 1 H), 4.61 (d, $J = 11.5$ Hz, 1 H), 4.36 (d, $J = 11.5$ Hz, 1 H), 4.32 (d, $J = 11.3$ Hz, 1 H), 4.28 (dd, $J = 10.8, 8.4$ Hz, 1 H), 4.10 (*pseudo*-t, $J = 10.5$ Hz, 1 H), 4.05 (dd, $J = 11.1, 1.5$ Hz, 1 H), 3.95 (dd, $J = 12.2, 2.6$ Hz, 1 H), 3.84–3.78 (m, 2 H), 3.76 (dd, $J = 11.2, 4.9$ Hz, 1 H), 3.67–3.62 (m, 2 H), 3.52 (*pseudo*-t, $J = 9.6$ Hz, 1 H), 2.12–2.05 (m, 1 H), 1.79 (s, 3 H), 1.66 (s, 3 H); ^{13}C NMR (150 MHz, CDCl_3) δ 170.3, 170.1, 167.8, 167.3, 137.8, 137.5, 134.6, 134.5, 134.4, 134.3, 131.7, 131.2, 129.8, 129.2, 128.6, 128.5, 128.1, 127.9, 127.9, 127.5, 123.8, 123.7, 123.6, 123.6, 107.4, 98.3, 82.6, 78.5, 77.3, 77.1, 76.9, 76.6, 76.4, 75.4, 74.8, 74.7, 73.9, 73.2, 68.5, 61.8, 55.2, 53.9, 29.8, 20.7, 20.5; HRMS (ESI) m/z calcd for $\text{C}_{52}\text{H}_{47}\text{ClKN}_2\text{O}_{14}\text{S}$ $[\text{M}+\text{K}]^+$, 1029.2068; found, 1029.2040.

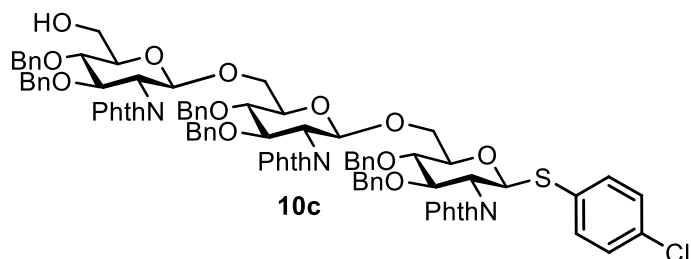


Building block **6c** (246 mg, 0.400 mmol) afforded **9c** in 4% isolated yield (8.0 mg, 6.84 μmol). **4-Chlorophenyl (3,4-di-*O*-benzyl-2-deoxy-2-phthalimido- β -D-glucopyranosyl)-(1 \rightarrow 6)-3,4-di-*O*-benzyl-2-deoxy-2-phthalimido-1-thio- β -D-glucopyranoside (**9c**)** TLC (Hexane/EtOAc 2:1) $R_f = 0.23$. ^1H NMR (600 MHz, CDCl_3) δ 7.76–7.62 (m, 4H), 7.56–7.48 (m, 3H), 7.36–7.33 (m, 4H), 7.32–

7.28 (m, 1H), 7.24–7.17 (m, 6H), 7.09–7.07 (m, 2H), 7.00–6.99 (m, 2 H), 6.89–6.84 (m, 5H), 6.84–6.78 (m, 5H), 5.31 (d, $J = 10.5$ Hz, 1 H), 5.28 (d, $J = 8.4$ Hz, 1 H), 4.89 (d, $J = 10.8$ Hz, 1 H), 4.81 (d, $J = 12.3$ Hz, 1 H), 4.74 (d, $J = 11.1$ Hz, 1 H), 4.64 (d, $J = 12.3$ Hz, 1 H), 4.52 (d, $J = 10.8$ Hz, 1 H), 4.45 (d, $J = 12.3$ Hz, 1 H), 4.37–4.33 (m, 2 H), 4.28 (d, $J = 12.3$ Hz, 1 H), 4.21 (dd, $J = 10.8, 8.7$ Hz, 2 H), 4.03 (*pseudo*-t, $J = 10.5$ Hz, 1 H), 3.99 (dd, $J = 10.8, 1.5$ Hz, 1 H), 3.91 (d, $J = 10.2$ Hz, 1 H), 3.78–3.72 (m, 2 H), 3.67 (dd, $J = 11.1, 5.1$ Hz, 1 H), 3.56–3.51 (m, 2 H), 3.44–3.40 (m, 1 H), 2.09–2.07 (m, 1 H); ^{13}C NMR (150 MHz, CDCl_3) δ 167.23, 167.21, 138.0, 137.9, 137.7, 137.5, 134.3, 134.0, 133.98, 133.97, 133.95, 133.9, 131.5, 131.49, 130.2, 129.1, 128.7, 128.6, 128.5, 128.4, 128.2, 128.15, 128.1, 128.07, 128.04, 128.01, 127.96, 127.5, 123.5, 123.48, 123.45, 123.4, 98.4, 82.9, 80.1, 79.6, 79.2, 79.1, 78.6, 75.5, 75.2, 75.0, 68.2, 62.0, 55.8, 54.6, 29.8; HRMS (ESI) m/z calcd for $\text{C}_{62}\text{H}_{55}\text{ClNaN}_2\text{O}_{12}\text{S}$ $[\text{M}+\text{Na}]^+$, 1109.3056; found, 1109.3041.



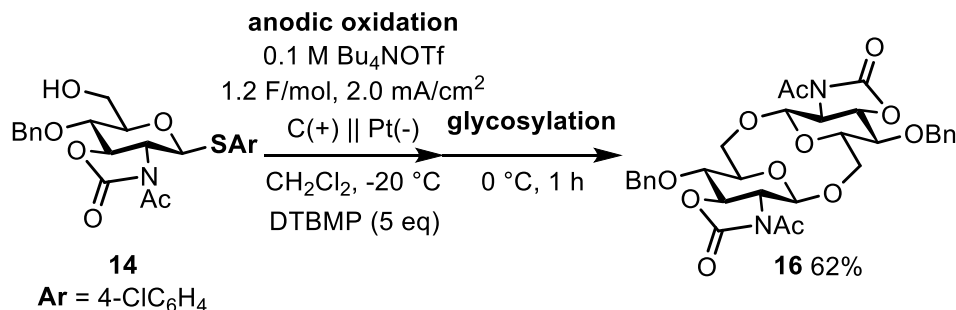
Building block **6b** (226 mg, 0.400 mmol) afforded **10b** in 6% isolated yield (11 mg, 7.80 μmol). **4-Chlorophenyl (3-*O*-acetyl-4-*O*-benzyl-2-deoxy-2-phthalimido- β -D-glucopyranosyl)-(1 \rightarrow 6)-(3-*O*-acetyl-4-*O*-benzyl-2-deoxy-2-phthalimido- β -D-glucopyranosyl)-(1 \rightarrow 6)-3-*O*-acetyl-4-*O*-benzyl-2-deoxy-2-phthalimido-1-thio- β -D-glucopyranoside (**10b**)** TLC (Hexane/EtOAc 1:1) $R_f = 0.23$; ^1H NMR (600 MHz, CDCl_3) δ 7.85–7.80 (m, 2 H), 7.76–7.68 (m, 6 H), 7.62–7.57 (m, 4 H), 7.31–7.26 (m, 7 H), 7.24–7.22 (m, 2 H), 7.21–7.18 (m, 6 H), 7.02–6.96 (m, 4 H), 5.78 (dd, $J = 10.8, 9.0$ Hz, 1 H), 5.66–5.62 (m, 2 H), 5.55 (d, $J = 8.4$ Hz, 1 H), 5.54 (d, $J = 10.5$ Hz, 1 H), 5.45 (d, $J = 8.4$ Hz, 1 H), 4.67 (d, $J = 11.7$ Hz, 1 H), 4.61 (d, $J = 11.7$ Hz, 1 H), 4.36–4.30 (m, 3 H), 4.28 (d, $J = 11.2$ Hz, 1 H), 4.24 (d, $J = 11.3$ Hz, 1 H), 4.21 (dd, $J = 10.7, 8.5$ Hz, 1 H), 4.13–4.09 (m, 1 H), 4.06–4.01 (m, 2 H), 3.96 (ddd, $J = 12.1, 4.9, 2.2$ Hz, 1 H), 3.87 (dd, $J = 11.4, 4.2$ Hz, 1 H), 3.80–3.75 (m, 2 H), 3.70 (dd, $J = 11.4, 4.8$ Hz, 1 H), 3.67–3.64 (m, 2 H), 3.63–3.57 (m, 2 H), 3.45 (*pseudo*-t, $J = 9.6$ Hz, 1 H), 2.32 (dd, $J = 8.5, 5.2$ Hz, 1 H), 1.76 (s, 3 H), 1.70 (s, 3 H), 1.62 (s, 3 H); ^{13}C NMR (150 MHz, CDCl_3) δ 170.3, 170.1, 167.9, 167.3, 137.9, 137.6, 134.9, 134.7, 134.5, 134.3, 134.2, 134.1, 131.8, 131.2, 129.3, 128.6, 128.5, 128.4, 128.4, 127.9, 127.8, 127.6, 127.5, 123.8, 123.7, 123.6, 98.2, 82.0, 78.3, 76.7, 76.5, 76.4, 75.5, 74.8, 74.7, 74.66, 74.65, 73.8, 73.3, 73.25, 73.2, 68.2, 68.16, 61.6, 60.5, 55.3, 55.0, 31.7, 22.7, 21.2, 20.7, 20.6, 20.5, 14.3, 14.2; HRMS (ESI) m/z calculated for $\text{C}_{75}\text{H}_{68}\text{ClNaN}_3\text{O}_{21}\text{S}$ $[\text{M}+\text{Na}]^+$, 1436.3647; found, 1436.3628.



Building block **6c** (246 mg, 0.40 mmol) afforded **10c** in 2% isolated yield (5.0 mg, 3.21 μ mol)). **4-Chlorophenyl (3,4-di-O-benzyl-2-deoxy-2-phthalimido- β -D-glucopyranosyl)-(1 \rightarrow 6)-(3,4-di-O-benzyl-2-deoxy-2-phthalimido- β -D-glucopyranosyl)-(1 \rightarrow 6)-3,4-di-O-benzyl-2-deoxy-2-phthalimido-1-thio- β -D-glucopyranoside (10c)** TLC (Hexane/EtOAc 1:1) R_f = 0.50; ^1H NMR (600 MHz, CDCl_3) δ 7.78–7.74 (m, 1 H), 7.72–7.62 (m, 5 H), 7.56–7.54 (m, 1 H), 7.53–7.48 (m, 2 H), 7.34–7.32 (m, 4 H), 7.30–7.26 (m, 4 H), 7.23–7.20 (m, 6 H), 7.20–7.17 (m, 4 H), 7.10–7.08 (m, 2 H), 7.07–7.04 (m, 2 H), 6.97–6.95 (m, 2 H), 6.87–6.85 (m, 2 H), 6.93–6.90 (m, 2 H), 6.97–6.77 (m, 9 H), 5.32 (d, J = 8.4 Hz, 1 H), 5.25 (d, J = 10.4 Hz, 1 H), 5.16 (d, J = 8.2 Hz, 1 H), 4.85 (d, J = 10.9 Hz, 1 H), 4.77 (d, J = 12.1 Hz, 1 H), 4.72 (d, J = 11.0 Hz, 1 H), 4.66 (d, J = 12.2 Hz, 1 H), 4.59 (d, J = 12.1 Hz, 1 H), 4.47 (d, J = 10.6 Hz, 1 H), 4.44–4.38 (m, 2 H), 4.37 (d, J = 12.0 Hz, 1 H), 4.35 (dd, J = 10.7, 8.6 Hz, 1 H), 4.33–4.30 (m, 2 H), 4.28–4.24 (m, 3 H), 4.21–4.12 (m, 4 H), 4.06 (dd, J = 10.9, 1.5 Hz, 1 H), 3.98–3.91 (m, 3 H), 3.78 (dd, J = 11.3, 4.5 Hz, 1 H), 3.72 (*pseudo-t*, J = 9.1 Hz, 1 H), 3.60–3.55 (m, 2 H), 3.50 (dd, J = 10.0, 3.8 Hz, 1 H), 3.42 (dd, J = 10.3, 3.1 Hz, 1 H), 3.37 (*pseudo-t*, J = 9.6 Hz, 1 H), 2.45–2.37 (m, 1 H); ^{13}C NMR (150 MHz, CDCl_3) δ 168.2, 168.0, 137.6, 137.4, 134.8, 134.6, 134.3, 134.2, 133.9, 131.8, 131.4, 129.2, 129.14, 129.1, 128.8, 128.7, 128.6, 128.59, 128.57, 128.5, 128.4, 128.3, 128.25, 128.2, 128.15, 128.13, 128.10, 128.09, 128.05, 128.04, 128.02, 127.99, 127.93, 127.91, 127.9, 127.6, 127.4, 127.36, 123.9, 123.6, 123.4, 102.2, 101.1, 85.4, 79.0, 76.5, 75.1, 74.8, 72.5, 71.8, 70.4, 70.3, 68.8, 68.2, 64.7, 57.8, 51.9, 29.8, 26.5; HRMS (ESI) m/z calcd for $\text{C}_{90}\text{H}_{80}\text{ClKN}_3\text{O}_{18}\text{S}$ $[\text{M}+\text{K}]^+$, 1596.4478; found, 1596.4441.

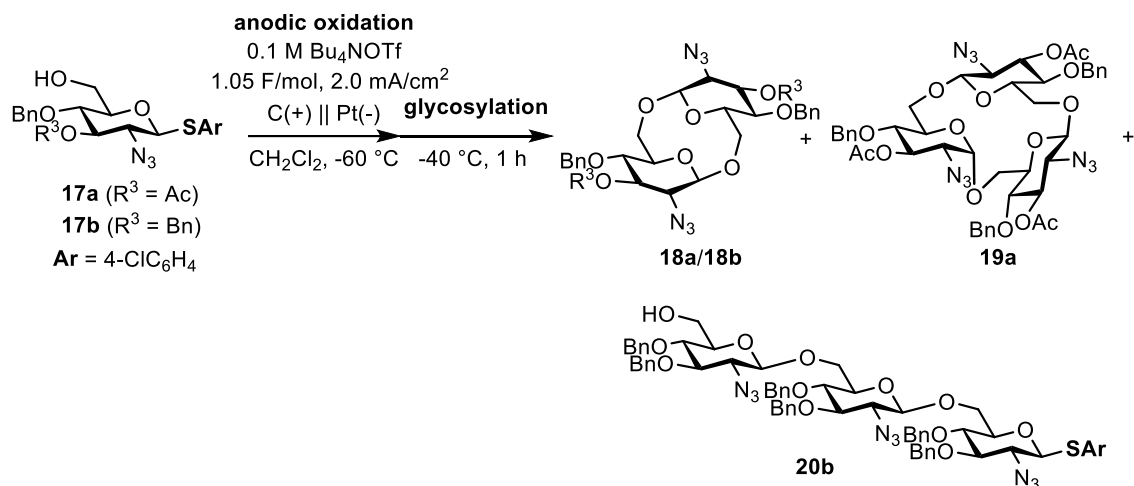
4. General procedure for cyclic oligoglucosamine synthesis with 2,3-oxazolidinone group

Synthesis of Cyclobis[(1→6)-2-acetamido-4-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy-β-D-glucopyranosyl] (**16**)

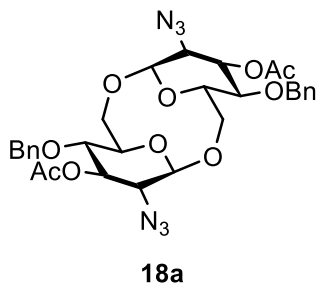


The electrochemical one-pot synthesis of cyclic disaccharide **16** was conducted with an H-type divided cell (4G glass filter). The cell had a carbon felt anode (Nippon Carbon JF-20-p7) and platinum square plate (20 mm×20 mm). Building block **14** (0.400 mmol, 186 mg), Bu₄NOTf (1.00 mmol, 392 mg), 2,6-di-*tert*-butyl-4-methylpyridine (DTBMP) (2.0 mmol, 411 mg), and CH₂Cl₂ (10 mL) were added to the anodic chamber. TfOH (0.400 mmol, 35.2 μL), Bu₄NOTf (1.00 mmol, 392 mg), and CH₂Cl₂ (10 mL) were added to the cathodic chamber. As the initiation phase of reaction, the constant current (8.0 mA (current density: 2.0 mA/cm²), 18~22 V (electrode distance: 4.5 cm)) was employed at -20 °C with magnetic stirring until 1.2 F/mol of electricity was consumed. After the electrolysis, the reaction temperature was raised from -20 °C to 0 °C as glycosylation phase. After glycosylation, Et₃N (0.5 mL) was added to both of the chambers. The solution was collected, and the solvent was removed under reduced pressure. The mixture was dissolved in EtOAc and washed with H₂O to remove electrolyte. It was further washed with 1N HCl (*aq.*) to remove excessive DTBMP. The solution was dried over anhydrous Na₂SO₄, and the solvent was removed under reduced pressure. The crude product was purified with preparative-GPC to afford cyclic oligosaccharide **16** (0.125 mmol, 79.7 mg, 62%) as a white solid. **Cyclobis[(1→6)-2-acetamido-4-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy-β-D-glucopyranosyl] (**16**)** TLC (eluent: Hexane/EtOAc 2:1) R_f = 0.31; ¹H NMR (CDCl₃, 600 MHz) δ 7.38–7.35 (m, 4 H), 7.33–7.30 (m, 1 H), 5.24 (d, *J* = 5.7 Hz, 1 H), 4.89 (d, *J* = 11.4 Hz, 1 H), 4.62 (d, *J* = 11.2 Hz, 1 H), 4.31 (dd, *J* = 12.5, 6.6 Hz, 1 H), 4.18 (dd, *J* = 9.7, 4.3 Hz, 1 H), 4.16–4.13 (m, 1 H), 4.01 (dd, *J* = 10.9, 2.5 Hz, 1 H), 3.94 (dd, *J* = 12.5, 5.8 Hz, 1 H), 3.59 (dd, *J* = 11.0, 1.5 Hz, 1 H), 2.53 (s, 3 H); ¹³C NMR (CDCl₃, 150 MHz) δ 170.6, 153.7, 137.3, 128.5, 128.0, 127.9, 97.0, 81.7, 73.1, 63.8, 62.0, 24.5; HRMS (ESI) *m/z* calcd for C₃₂H₃₄KN₂O₁₂; [M+K]⁺, 677.1744, found 677.1735.

5. General procedure for cyclic oligoglucosamine synthesis with azido group

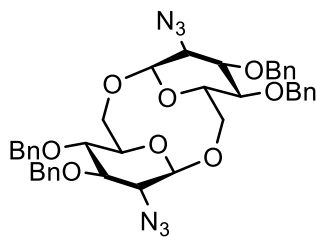


The electrochemical polymerization synthesis of cyclic oligosaccharides **18-19** and linear oligosaccharide **20** was carried out by an H-type divided cell (4G glass filter). The cell had a carbon felt anode (Nippon Carbon JF-20-P7) and platinum square plate (20 mm×20 mm). Building block **17** (0.40 mmol), Bu₄NOTf (1.00 mmol), and CH₂Cl₂ (10 mL) were added to the anodic chamber. TfOH (0.4 mmol), Bu₄NOTf (1.00 mmol), and CH₂Cl₂ (10 mL) were added to the cathodic chamber. The constant current (8 mA (current density: 2.0 mA/cm²), (electrode distance: 4.5 cm)) was employed at -60 °C with magnetic stirring until the electricity was consumed. After the electrolysis, the reaction was kept stirring at -40 °C for 1 h. After that, Et₃N (0.3 mL) was added to both chambers. The solution in both chambers was collected in eggplant flask, and the solvent was removed under reduced pressure. The mixture was dissolved in EtOAc and washed with H₂O to remove electrolyte. The solution was dried over anhydrous Na₂SO₄, filtered, and the solution was removed under reduced pressure. The crude product was purified with preparative-GPC to afford cyclic oligosaccharides **18-19** and linear trisaccharides **20b**.



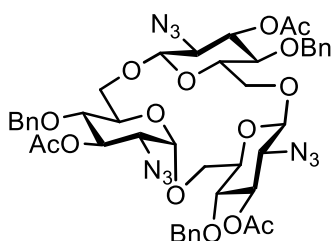
Building block **17a** (185 mg, 0.40 mmol) afforded **18a** in 49% isolated yield (63 mg, 0.987mmol).
Cyclobis[(1→6)-3-O-acetyl-2-azido-4-O-benzyl-2-deoxy-2-β-D-glucopyranosyl] (18a) TLC (Hexane/EtOAc 3:1) R_f = 0.28; ¹H NMR (600 MHz, CDCl₃) δ 7.35–7.29 (m, 5 H), 5.05 (dd, J = 9.0,

3.6 Hz, 1 H), 4.72 (d, $J = 1.8$ Hz, 1 H), 4.69 (d, $J = 11.6$ Hz, 1 H), 4.61 (d, $J = 11.5$ Hz, 1 H), 4.44 (dd, $J = 10.2, 9.6$ Hz, 1 H), 4.11 (dd, $J = 12.3, 1.2$ Hz, 1 H), 3.82–3.79 (m, 1 H), 3.71 (dd, $J = 12.6, 1.2$ Hz, 1 H), 3.65 (dd, $J = 3.6, 1.8$ Hz, 1 H), 1.96 (s, 3 H); ^{13}C NMR (150 MHz, CDCl_3) δ 170.2, 138.1, 128.6, 128.2, 128.0, 100.3, 75.7, 74.7, 74.5, 73.7, 70.9, 65.3, 21.0; HRMS (ESI) m/z calcd for $\text{C}_{30}\text{H}_{34}\text{KN}_6\text{O}_{10}$ $[\text{M}+\text{K}]^+$, 677.1968; found, 677.1933.



18b

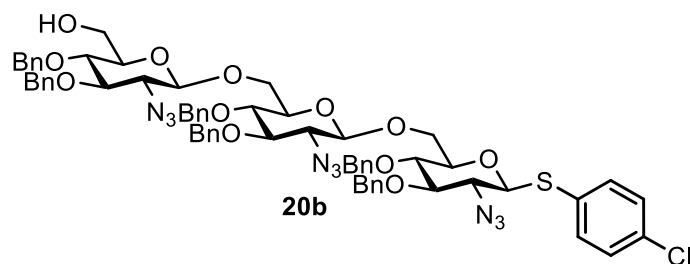
Building block **17b** (205 mg, 0.40 mmol) afforded **18b** in 14% isolated yield (21 mg, 0.0281 mmol). **Cyclobis[(1→6)-2-azido-3,4-di-O-benzyl-2-deoxy-2-β-D-glucopyranosyl] (18b)** TLC (Hexane/EtOAc 3:1) $R_f = 0.63$; ^1H NMR (600 MHz, CDCl_3) δ 7.36–7.28 (m, 10 H), 4.86 (d, $J = 11.3$ Hz, 1 H), 4.84 (d, $J = 11.0$ Hz, 1 H), 4.78 (d, $J = 11.1$ Hz, 1 H), 4.67 (d, $J = 11.4$ Hz, 1 H), 4.58–4.57 (m, 1 H), 4.20 (*pseudo-t*, $J = 9.6$ Hz, 1 H), 3.92 (dd, $J = 12.0, 2.4$ Hz, 1 H), 3.76 (dd, $J = 9.6, 1.8$ Hz, 1 H), 3.67–3.62 (m, 2 H), 3.50 (dd, $J = 6.6, 1.2$ Hz, 1 H), 1.43 (dd, $J = 15.0, 7.5$ Hz, 2 H); ^{13}C NMR (150 MHz, CDCl_3) δ 138.2, 137.8, 128.54, 128.49, 128.13, 128.09, 127.98, 127.97, 100.9, 81.7, 77.0, 75.0, 74.5, 74.2, 69.2, 67.7, 58.9, 24.0, 19.7, 13.7; HRMS (ESI) m/z calcd for $\text{C}_{40}\text{H}_{42}\text{KN}_6\text{O}_8$ $[\text{M}+\text{K}]^+$, 773.2696; found, 773.2650.



19a

Building block **17a** (185 mg, 0.40 mmol) afforded **19a** in 16% isolated yield (19 mg, 0.0208 mmol). **Cyclotris[(1→6)-3-O-acetyl-2-azido-4-O-benzyl-2-deoxy-2-α,β-D-glucopyranosyl] (19a)** TLC (Hexane/EtOAc 2:1) $R_f = 0.32$; ^1H NMR (600 MHz, CDCl_3) δ 7.37–7.28 (m, 10 H), 7.28–7.25 (m, 2 H), 7.22–7.21 (m, 3 H), 5.61 (dd, $J = 10.8, 9.0$ Hz, 1 H), 5.10 (dd, $J = 10.8, 9.6$ Hz, 1 H), 4.95 (*pseudo-t*, $J = 10.2$ Hz, 1 H), 4.80 (d, $J = 3.6$ Hz, 1 H), 4.72 (d, $J = 11.4$ Hz, 1 H), 4.63 (d, $J = 11.4$ Hz, 1 H), 4.61 (d, $J = 11.4$ Hz, 2 H), 4.53 (d, $J = 12.0$ Hz, 1 H), 4.52 (d, $J = 7.8$ Hz, 1 H), 4.51 (d, $J = 11.4$ Hz, 1 H), 4.33 (dd, $J = 12.6, 1.8$ Hz, 1 H), 4.22 (d, $J = 7.8$ Hz, 1 H), 4.03 (dd, $J = 12.6, 8.4$ Hz, 1 H), 3.91–

3.89 (m, 1 H), 3.81–3.77 (m, 3 H), 3.75 (*pseudo*-t, $J = 9.6$ Hz, 1 H), 3.70 (ddd, $J = 9.0, 6.6, 1.2$ Hz, 1 H), 3.45 (dd, $J = 10.2, 7.8$ Hz, 1 H), 3.44 (dd, $J = 10.3, 6.0$ Hz, 1H), 3.29 (*pseudo*-t, $J = 9.6$ Hz, 1H), 3.18–3.15 (m, 1 H), 3.15 (*pseudo*-t, $J = 9.6$ Hz, 1 H), 3.06 (dd, $J = 10.8, 3.6$ Hz, 1 H), 2.08 (s, 3 H), 2.06 (s, 3 H), 1.96 (s, 3 H); ^{13}C NMR (150 MHz, CDCl_3) δ 170.0, 169.7, 137.9, 137.2, 137.15, 128.8, 128.7, 128.5, 128.48, 128.3, 128.0, 127.9, 127.74, 127.7, 105.7, 101.3, 97.9, 77.7, 76.4, 75.3, 75.28, 74.9, 74.3, 74.2, 74.0, 73.9, 73.5, 71.8, 71.6, 69.6, 68.8, 64.9, 63.7, 61.2, 21.1, 21.0, 20.96, 14.3; HRMS (ESI) m/z calcd for $\text{C}_{45}\text{H}_{51}\text{KN}_9\text{O}_{15}$ $[\text{M}+\text{K}]^+$, 996.3136; found, 996.3195.



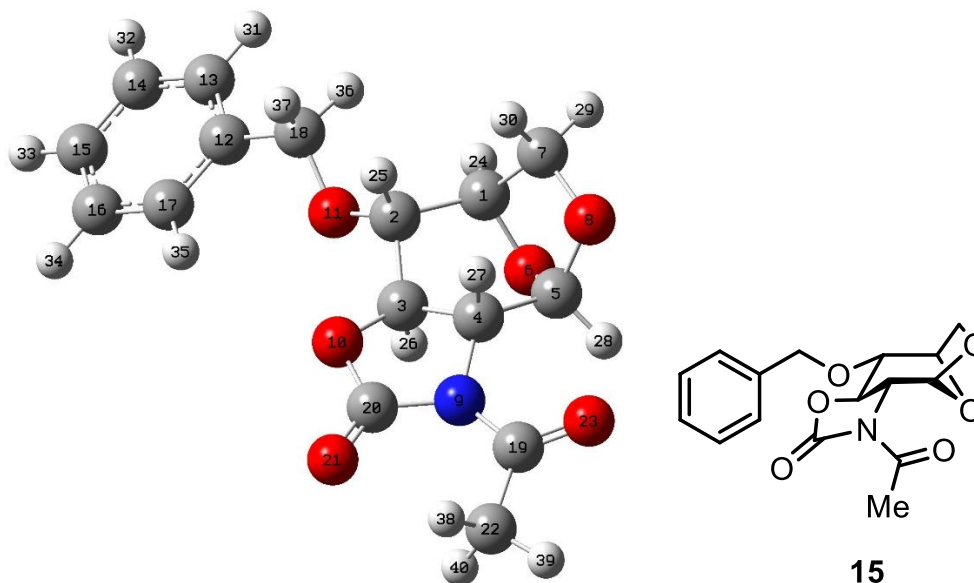
Building block **17b** (205 mg, 0.40 mmol) afforded **20b** in 13% isolated yield (21 mg, 0.0171 mmol).

4-Chlorophenyl (2-azido-2-deoxy-3,4-di-*O*-benzyl- β -D-glucopyranosyl)-(1 \rightarrow 6)-(2-azido-2-deoxy-3,4-di-*O*-benzyl- β -D-glucopyranosyl)-(1 \rightarrow 6)-2-azido-2-deoxy-3,4-di-*O*-benzyl-1-thio- β -D-glucopyranoside (20b) TLC (Hexane/EtOAc 2:1) $R_f = 0.40$; ^1H NMR (600 MHz, CDCl_3) δ 7.55–7.53 (m, 2 H), 7.37–7.34 (m, 5 H), 7.33–7.30 (m, 15 H), 7.29–7.26 (m, 12 H), 4.64–4.61 (m, 3 H), 4.42 (d, $J = 10.2$ Hz, 1 H), 4.29–4.26 (m, 2 H), 4.16 (dd, $J = 11.4, 1.8$ Hz, 1 H), 4.04 (dd, $J = 11.4, 1.5$ Hz, 1 H), 3.79 (dd, $J = 11.4, 5.1$ Hz, 1 H), 3.66–3.62 (m, 2 H), 3.54–3.45 (m, 4 H), 3.43–3.40 (m, 1 H), 3.38–3.36 (m, 1 H), 3.31 (dd, $J = 10.2, 9.3$ Hz, 1 H), 3.21 (*pseudo*-t, $J = 8.7$ Hz, 4 H), 1.90 (dd, $J = 7.7, 6.0$ Hz, 1H); ^{13}C NMR (150 MHz, CDCl_3) δ 137.9, 137.82, 137.79, 137.6, 135.0, 129.3, 128.82, 128.77, 128.66, 128.62, 128.61, 128.54, 128.49, 128.3, 128.21, 128.19, 128.18, 128.15, 128.13, 128.09, 128.08, 128.05, 128.02, 127.99, 127.98, 129.92, 127.9, 127.7, 102.6, 102.4, 86.0, 85.0, 83.2, 82.9, 78.9, 77.8, 77.6, 75.9, 75.8, 75.5, 75.4, 75.13, 75.10, 75.09, 74.8, 66.5, 66.4, 65.1, 61.7, 58.9, 24.0, 19.8, 13.7; HRMS (ESI) m/z calcd for $\text{C}_{66}\text{H}_{68}\text{ClNaN}_9\text{O}_{12}\text{S}$ $[\text{M}+\text{Na}]^+$, 1268.4289; found, 1268.4249.

6. Molecular Orbital Calculations of anhydro sugar

The molecular orbital calculations were carried out with 1,6-Anhydro-2-acetamido-4-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy- β -D-glucopyranoside (**15**) at B3LYP/6-31G(d) level using the Gaussian 16, Revision C.02.⁸ Geometries were fully optimized. All the optimized structures were local minima according to the vibration analysis. Cartesian coordinates and energies of computationally characterized species are as follows:

1,6-Anhydro-2-acetamido-4-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy- β -D-glucopyranoside (**15**)



(white H, black C, blue N, red O)

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms)		
			X	Y	Z
1	6	0	-0.16492	2.131337	0.2187
2	6	0	0.274667	0.754509	-0.42251
3	6	0	-0.7847	-0.2157	0.051899
4	6	0	-2.17365	0.346913	-0.18699
5	6	0	-2.30435	1.679611	0.613134
6	8	0	-1.08166	1.900996	1.306095
7	6	0	-1.02417	3.036293	-0.6985
8	8	0	-2.37958	2.739594	-0.32505
9	7	0	-2.93857	-0.87696	0.077304
10	8	0	-0.86217	-1.52716	-0.54015
11	8	0	1.526591	0.308336	0.050603
12	6	0	3.895483	0.079124	-0.22469
13	6	0	5.10813	0.755448	-0.05983

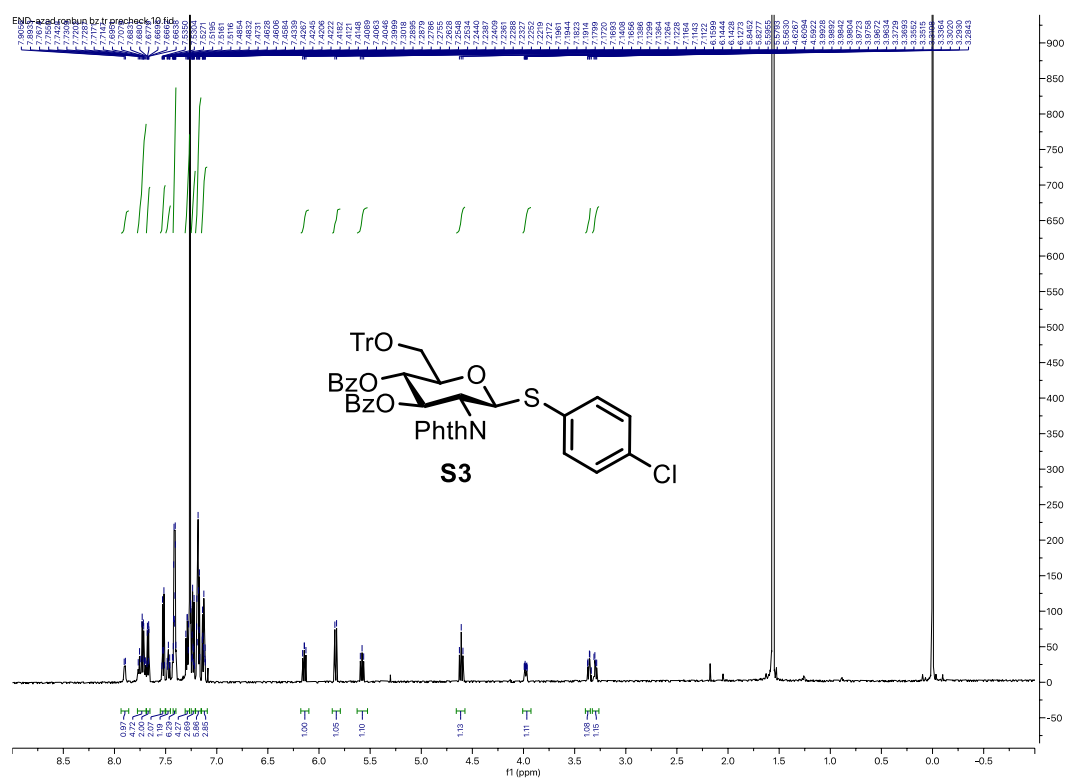
14	6	0	6.269702	0.058067	0.277473
15	6	0	6.224147	-1.32373	0.464531
16	6	0	5.013341	-2.00364	0.310461
17	6	0	3.855851	-1.30808	-0.03621
18	6	0	2.654427	0.828476	-0.64793
19	6	0	-4.33893	-0.85859	0.207606
20	6	0	-2.13822	-1.97578	-0.3258
21	8	0	-2.47812	-3.11809	-0.48533
22	6	0	-5.05385	-2.18061	0.34572
23	8	0	-4.90638	0.219384	0.233441
24	1	0	0.711775	2.634626	0.628949
25	1	0	0.285138	0.831605	-1.52156
26	1	0	-0.63025	-0.35481	1.125877
27	1	0	-2.31666	0.608144	-1.2439
28	1	0	-3.14116	1.728496	1.304974
29	1	0	-0.83681	4.09738	-0.5056
30	1	0	-0.86848	2.837402	-1.76574
31	1	0	5.146116	1.834702	-0.19313
32	1	0	7.205483	0.596154	0.403168
33	1	0	7.125442	-1.86806	0.733453
34	1	0	4.970403	-3.07924	0.460004
35	1	0	2.911867	-1.83238	-0.14819
36	1	0	2.778026	1.905583	-0.45278
37	1	0	2.487933	0.714053	-1.73309
38	1	0	-4.98025	-2.75917	-0.57938
39	1	0	-6.09876	-1.96667	0.57474
40	1	0	-4.60718	-2.79555	1.131953

7. References

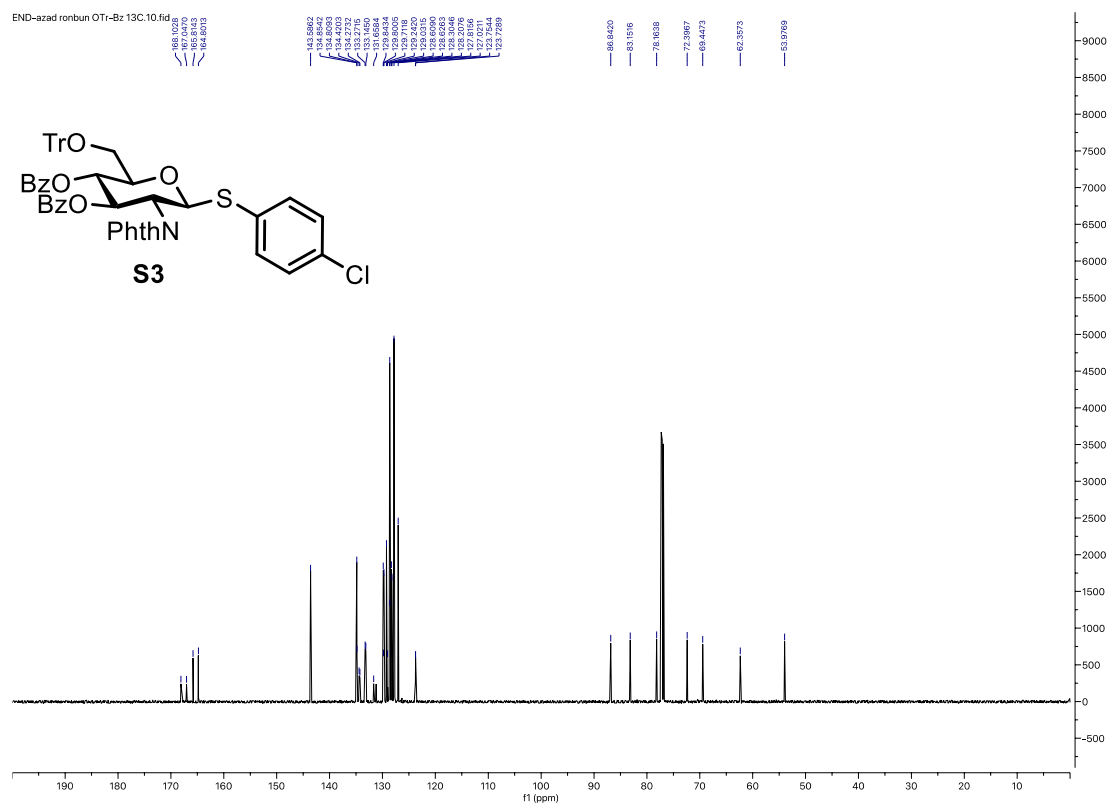
1. Sugiyama, S.; Diakur, J. M. *Org. Lett.* **2000**, *2*, 2713.
2. Yano, K.; Itoh, T.; Nokami, T. *Carbohydr. Res.* **2020**, *492*, 109018.
3. Rahman, A. M.; Takahashi, S.; Sasaki, N.; Itoh, T.; Ohnuma, T.; Nokami, T. *Electrochemistry* **2023**, *91*, 112013.
4. Chun, Y.; Yan, S.; Li, X.; Ding, N.; Zhang, W.; Wang, P.; Li, M.; Li, Y. *Tetrahedron Lett.* **2011**, *52*, 6196.
5. Nokami, T.; Hayashi, R.; Saigusa, Y.; Shimizu, A.; Liu, C.-Y.; Mong, T. K.; Yoshida, J. *Org. Lett.* **2013**, *15*, 4520.
6. Garcia, J.; Vilarrasa, J.; Bordas, X.; Banaszek, A. *Tetrahedron Lett.* **1986**, *27*, 639.
7. Gening, M. L.; Titov, D. V.; Grachev, A. A.; Gerbst, A. G.; Yudina, O. N.; Shashkov, A. S.; Chizhov, A. O. Tsvetkov, Y. E.; Nifantiev, N. E. *Eur. J. Org. Chem.* **2010**, *13*, 2465.
8. Gaussian 16, Revision C.02, Frisch, M. J.; Trucks, G. W.; Schlegel, H. B.; Scuseria, G. E.; Robb, M. A.; Cheeseman, J. R.; Scalmani, G.; Barone, V.; Petersson, G. A.; Nakatsuji, H.; Li, X.; Caricato, M.; Marenich, A. V.; Bloino, J.; Janesko, B. G.; Gomperts, R.; Mennucci, B.; Hratchian, H. P.; Ortiz, J. V.; Izmaylov, A. F.; Sonnenberg, J. L.; Williams-Young, D.; Ding, F.; Lipparini, F.; Egidi, F.; Goings, J.; Peng, B.; Petrone, A.; Henderson, T.; Ranasinghe, D.; Zakrzewski, V. G.; Gao, J.; Rega, N.; Zheng, G.; Liang, W.; Hada, M.; Ehara, M.; Toyota, K.; Fukuda, R.; Hasegawa, J.; Ishida, M.; Nakajima, T.; Honda, Y.; Kitao, O.; Nakai, H.; Vreven, T.; Throssell, K.; Montgomery, Jr., J. A.; Peralta, J. E.; Ogliaro, F.; Bearpark, M. J.; Heyd, J. J.; Brothers, E. N.; Kudin, K. N.; Staroverov, V. N.; Keith, T. A.; Kobayashi, R.; Normand, J.; Raghavachari, K.; Rendell, A. P.; Burant, J. C.; Iyengar, S. S.; Tomasi, J.; Cossi, M.; Millam, J. M.; Klene, M.; Adamo, C.; Cammi, R.; Ochterski, J. W.; Martin, R. L.; Morokuma, K.; Farkas, O.; Foresman, J. B. and Fox, D. J., Gaussian, Inc., Wallingford CT, **2016**.

¹H NMR

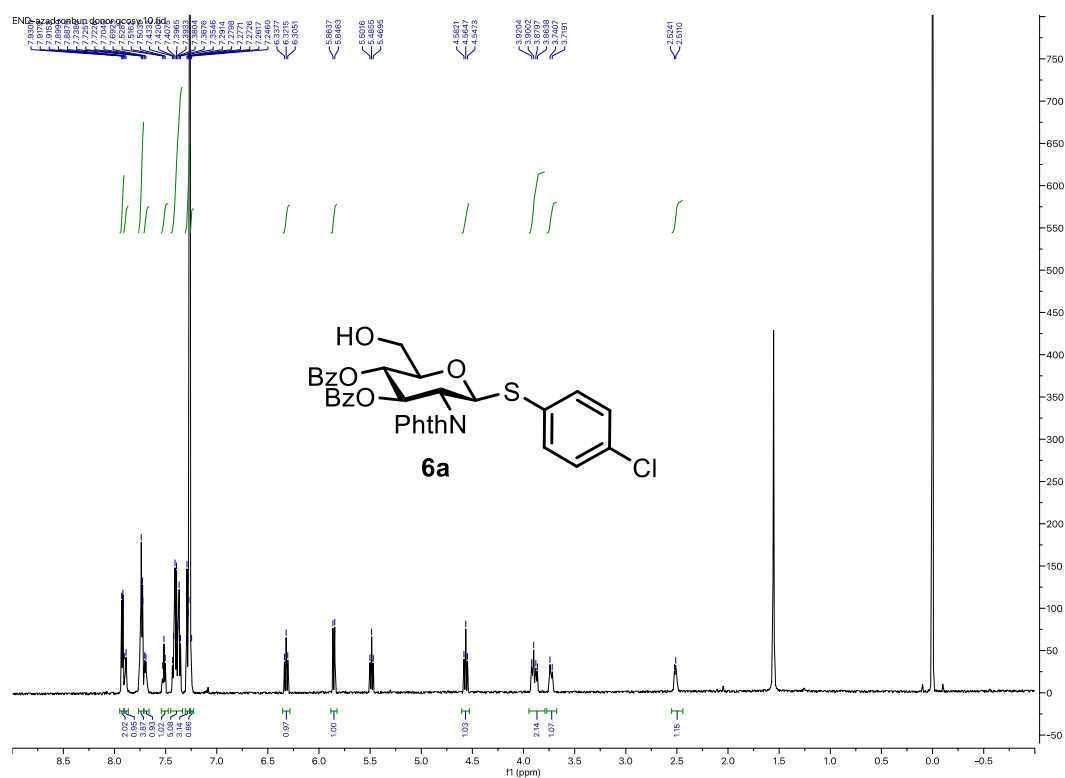
¹H NMR



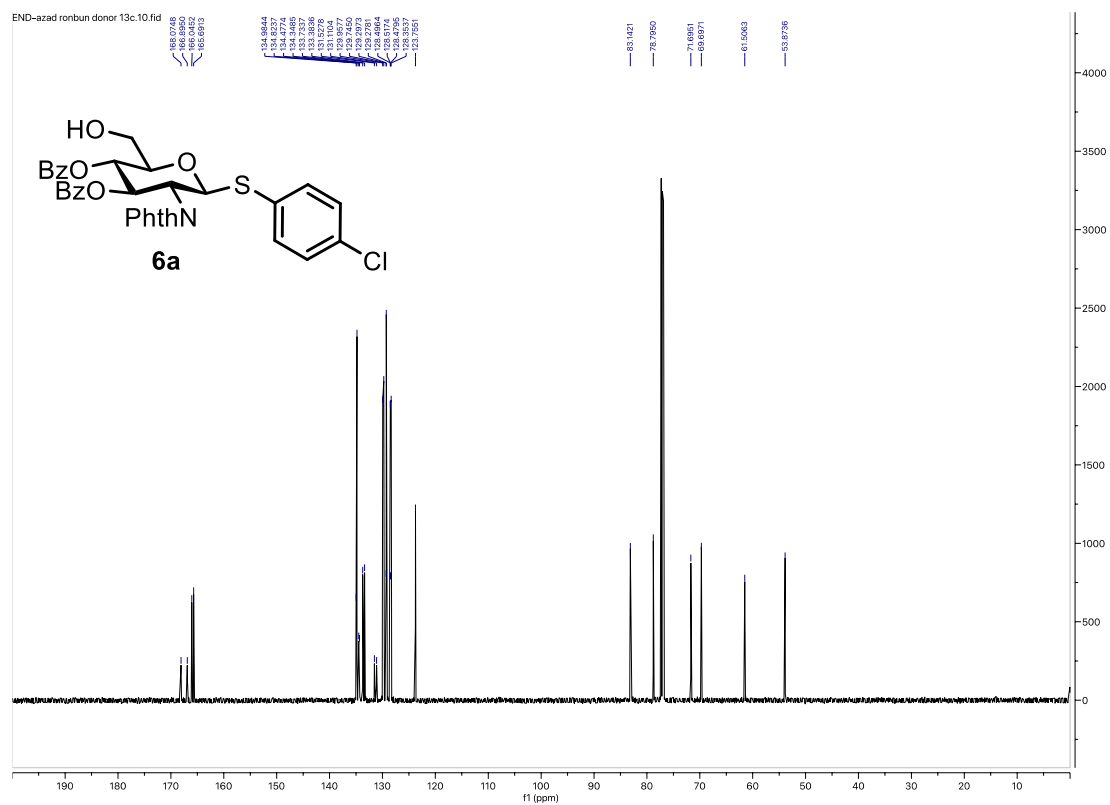
¹³C NMR



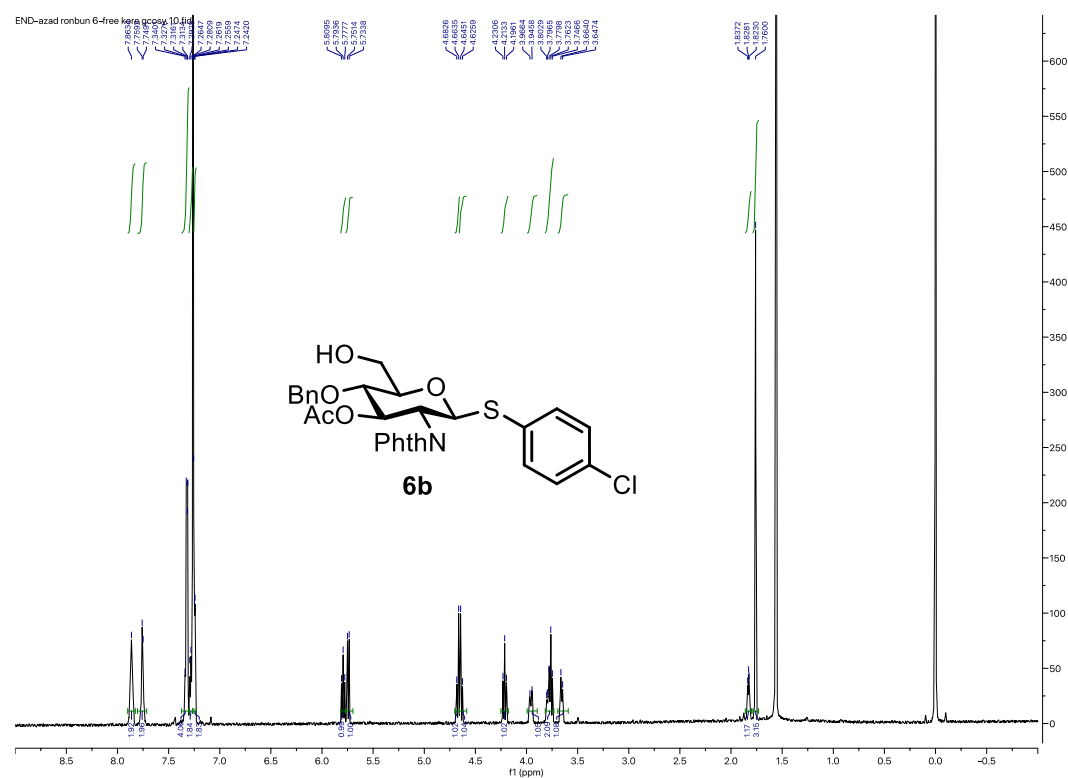
¹H NMR



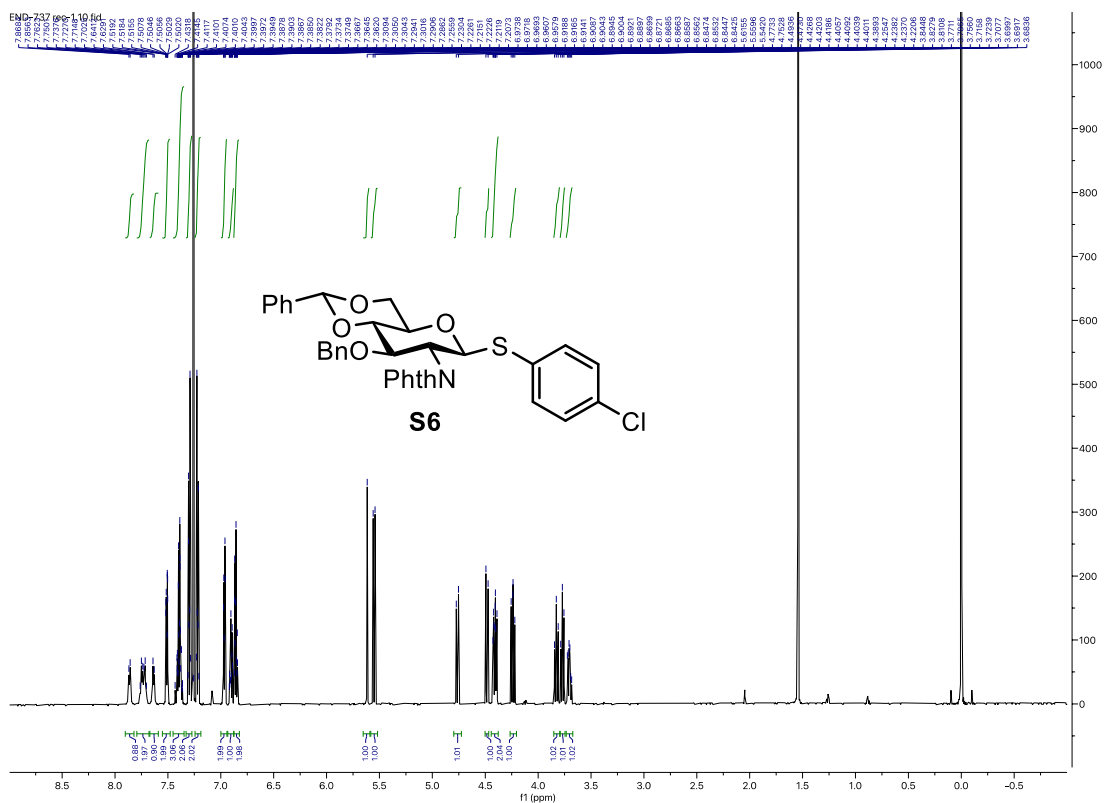
¹³C NMR



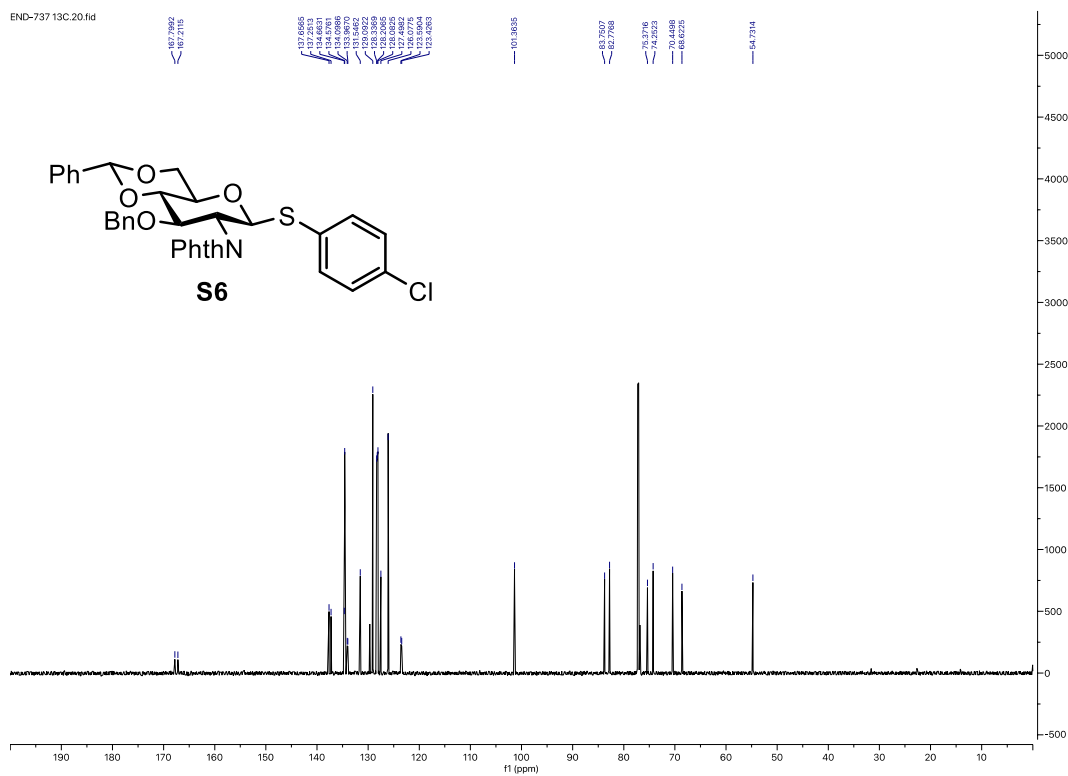
¹H NMR



¹H NMR



¹³C NMR

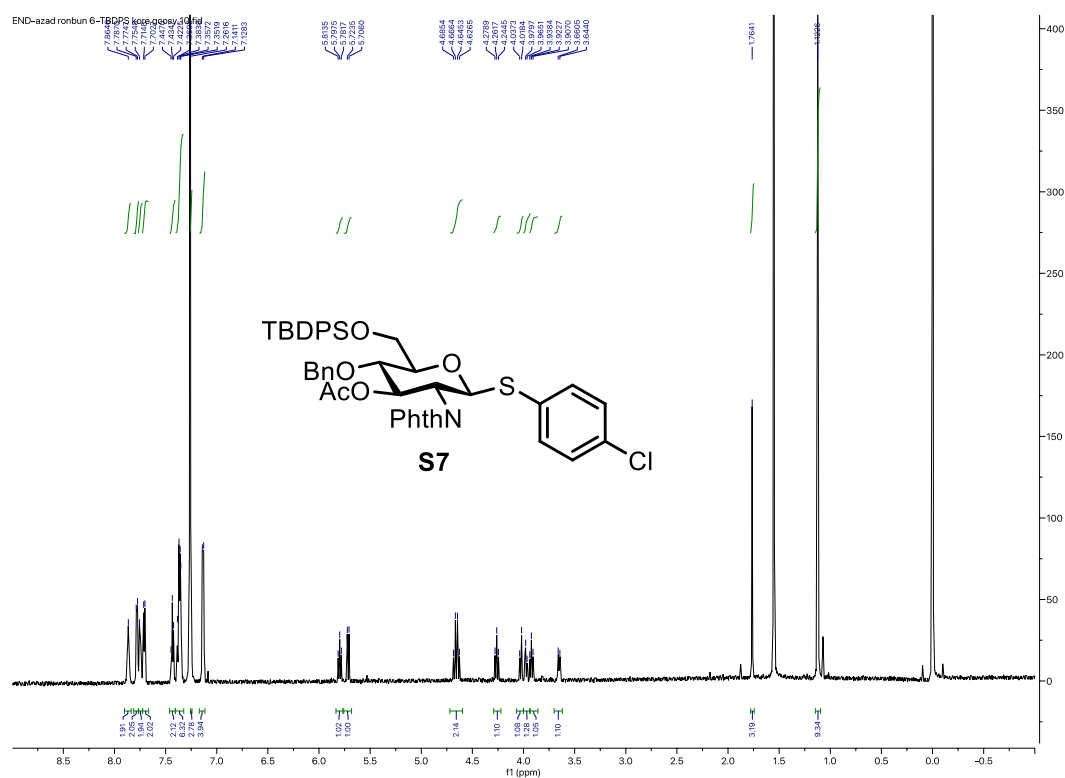
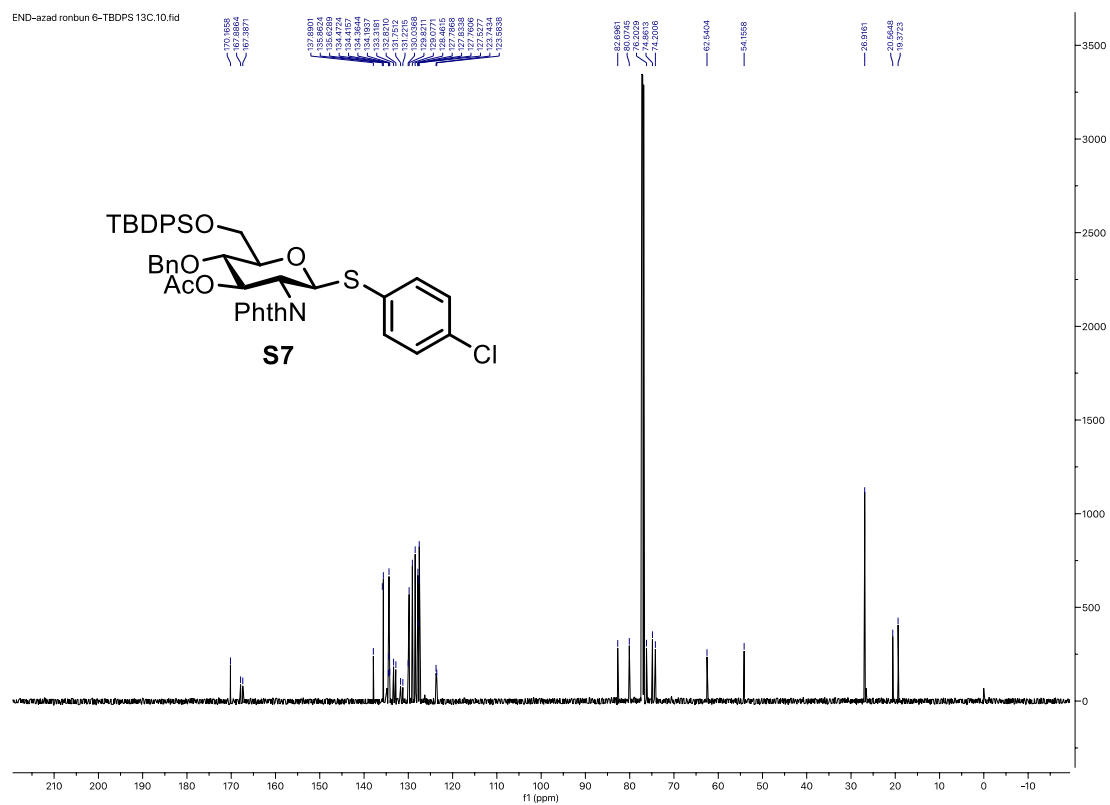


Chemical structure of **6c** is shown on the right. It is a substituted furanose derivative with a benzoyl group (BnO), a benzoyl group (BnO), a phenyl group (Ph), and a 4-chlorophenyl group (4-Cl-Ph).

1H NMR spectrum (CDCl₃) of compound 6c:

- Chemical Shifts (ppm):** 8.491, 8.483, 8.475, 8.467, 8.459, 8.451, 8.443, 8.435, 8.427, 8.419, 8.411, 8.403, 8.395, 8.387, 8.379, 8.371, 8.363, 8.355, 8.347, 8.339, 8.331, 8.323, 8.315, 8.307, 8.299, 8.291, 8.283, 8.275, 8.267, 8.259, 8.251, 8.243, 8.235, 8.227, 8.219, 8.211, 8.203, 8.195, 8.187, 8.179, 8.171, 8.163, 8.155, 8.147, 8.139, 8.131, 8.123, 8.115, 8.107, 8.099, 8.091, 8.083, 8.075, 8.067, 8.059, 8.051, 8.043, 8.035, 8.027, 8.019, 8.011, 8.003, 7.995, 7.987, 7.979, 7.971, 7.963, 7.955, 7.947, 7.939, 7.931, 7.923, 7.915, 7.907, 7.899, 7.891, 7.883, 7.875, 7.867, 7.859, 7.851, 7.843, 7.835, 7.827, 7.819, 7.811, 7.803, 7.795, 7.787, 7.779, 7.771, 7.763, 7.755, 7.747, 7.739, 7.731, 7.723, 7.715, 7.707, 7.699, 7.691, 7.683, 7.675, 7.667, 7.659, 7.651, 7.643, 7.635, 7.627, 7.619, 7.611, 7.603, 7.595, 7.587, 7.579, 7.571, 7.563, 7.555, 7.547, 7.539, 7.531, 7.523, 7.515, 7.507, 7.499, 7.491, 7.483, 7.475, 7.467, 7.459, 7.451, 7.443, 7.435, 7.427, 7.419, 7.411, 7.403, 7.395, 7.387, 7.379, 7.371, 7.363, 7.355, 7.347, 7.339, 7.331, 7.323, 7.315, 7.307, 7.299, 7.291, 7.283, 7.275, 7.267, 7.259, 7.251, 7.243, 7.235, 7.227, 7.219, 7.211, 7.203, 7.195, 7.187, 7.179, 7.171, 7.163, 7.155, 7.147, 7.139, 7.131, 7.123, 7.115, 7.107, 7.099, 7.091, 7.083, 7.075, 7.067, 7.059, 7.051, 7.043, 7.035, 7.027, 7.019, 7.011, 7.003, 6.995, 6.987, 6.979, 6.971, 6.963, 6.955, 6.947, 6.939, 6.931, 6.923, 6.915, 6.907, 6.899, 6.891, 6.883, 6.875, 6.867, 6.859, 6.851, 6.843, 6.835, 6.827, 6.819, 6.811, 6.803, 6.795, 6.787, 6.779, 6.771, 6.763, 6.755, 6.747, 6.739, 6.731, 6.723, 6.715, 6.707, 6.699, 6.691, 6.683, 6.675, 6.667, 6.659, 6.651, 6.643, 6.635, 6.627, 6.619, 6.611, 6.603, 6.595, 6.587, 6.579, 6.571, 6.563, 6.555, 6.547, 6.539, 6.531, 6.523, 6.515, 6.507, 6.499, 6.491, 6.483, 6.475, 6.467, 6.459, 6.451, 6.443, 6.435, 6.427, 6.419, 6.411, 6.403, 6.395, 6.387, 6.379, 6.371, 6.363, 6.355, 6.347, 6.339, 6.331, 6.323, 6.315, 6.307, 6.299, 6.291, 6.283, 6.275, 6.267, 6.259, 6.251, 6.243, 6.235, 6.227, 6.219, 6.211, 6.203, 6.195, 6.187, 6.179, 6.171, 6.163, 6.155, 6.147, 6.139, 6.131, 6.123, 6.115, 6.107, 6.099, 6.091, 6.083, 6.075, 6.067, 6.059, 6.051, 6.043, 6.035, 6.027, 6.019, 6.011, 6.003, 5.995, 5.987, 5.979, 5.971, 5.963, 5.955, 5.947, 5.939, 5.931, 5.923, 5.915, 5.907, 5.899, 5.891, 5.883, 5.875, 5.867, 5.859, 5.851, 5.843, 5.835, 5.827, 5.819, 5.811, 5.803, 5.795, 5.787, 5.779, 5.771, 5.763, 5.755, 5.747, 5.739, 5.731, 5.723, 5.715, 5.707, 5.699, 5.691, 5.683, 5.675, 5.667, 5.659, 5.651, 5.643, 5.635, 5.627, 5.619, 5.611, 5.603, 5.595, 5.587, 5.579, 5.571, 5.563, 5.555, 5.547, 5.539, 5.531, 5.523, 5.515, 5.507, 5.499, 5.491, 5.483, 5.475, 5.467, 5.459, 5.451, 5.443, 5.435, 5.427, 5.419, 5.411, 5.403, 5.395, 5.387, 5.379, 5.371, 5.363, 5.355, 5.347, 5.339, 5.331, 5.323, 5.315, 5.307, 5.299, 5.291, 5.283, 5.275, 5.267, 5.259, 5.251, 5.243, 5.235, 5.227, 5.219, 5.211, 5.203, 5.195, 5.187, 5.179, 5.171, 5.163, 5.155, 5.147, 5.139, 5.131, 5.123, 5.115, 5.107, 5.099, 5.091, 5.083, 5.075, 5.067, 5.059, 5.051, 5.043, 5.035, 5.027, 5.019, 5.011, 5.003, 4.995, 4.987, 4.979, 4.971, 4.963, 4.955, 4.947, 4.939, 4.931, 4.923, 4.915, 4.907, 4.899, 4.891, 4.883, 4.875, 4.867, 4.859, 4.851, 4.843, 4.835, 4.827, 4.819, 4.811, 4.803, 4.795, 4.787, 4.779, 4.771, 4.763, 4.755, 4.747, 4.739, 4.731, 4.723, 4.715, 4.707, 4.699, 4.691, 4.683, 4.675, 4.667, 4.659, 4.651, 4.643, 4.635, 4.627, 4.619, 4.611, 4.603, 4.595, 4.587, 4.579, 4.571, 4.563, 4.555, 4.547, 4.539, 4.531, 4.523, 4.515, 4.507, 4.499, 4.491, 4.483, 4.475, 4.467, 4.459, 4.451, 4.443, 4.435, 4.427, 4.419, 4.411, 4.403, 4.395, 4.387, 4.379, 4.371, 4.363, 4.355, 4.347, 4.339, 4.331, 4.323, 4.315, 4.307, 4.299, 4.291, 4.283, 4.275, 4.267, 4.259, 4.251, 4.243, 4.235, 4.227, 4.219, 4.211, 4.203, 4.195, 4.187, 4.179, 4.171, 4.163, 4.155, 4.147, 4.139, 4.131, 4.123, 4.115, 4.107, 4.099, 4.091,

13C NMR spectrum of compound **6c** in CDCl₃. The chemical structure of **6c** is shown as an inset: a pyranose ring with a 4-chlorophenylthio group at C1, a benzoyloxy group at C2, a hydroxyl group at C3, and a benzoyloxy group at C4. The NMR peaks are labeled with their corresponding chemical shifts in ppm: 138.0145, 137.9904, 137.9668, 137.9429, 134.9144, 133.9827, 133.9582, 131.6849, 130.9561, 130.9300, 128.7169, 128.2205, 128.1925, 128.1473, 128.1230, 123.6330, 123.5821, 83.3569, 80.1742, 79.9906, 79.9668, 75.2279, 75.1997, 61.8457, and 55.9513.

¹H NMR¹³C NMR

[illegible]

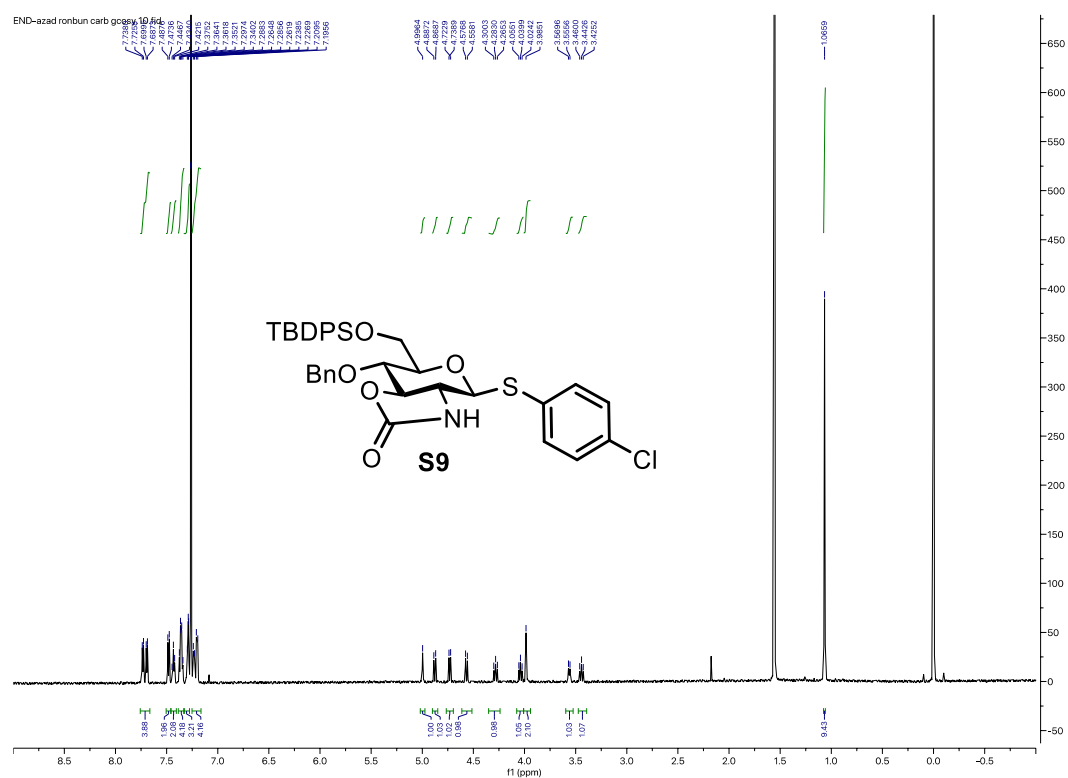
END-azad ronicun NH2 dep 13C, 10.fid

Chemical structure of S8 is shown above the spectrum. The structure is a cyclohexane ring with a TBDPSO group, a BnO group, a hydroxyl group, an amino group, and a 4-chlorophenylthio group.

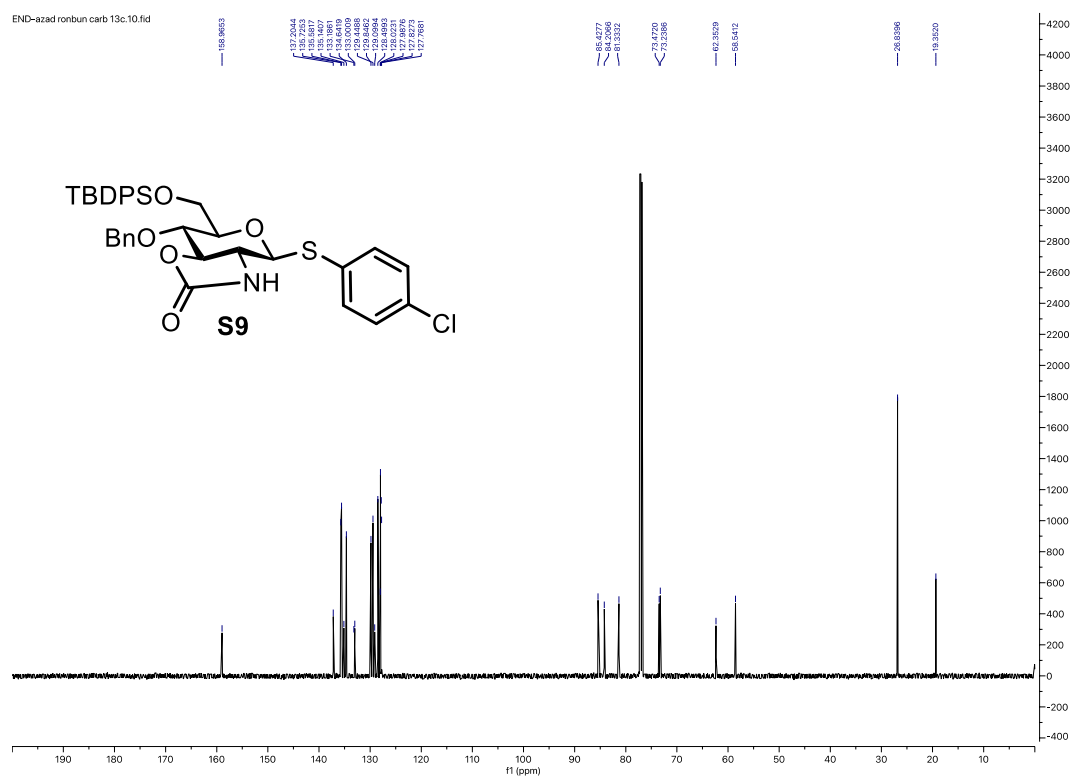
Peak list (ppm):

- 138.2014
- 136.6515
- 135.6509
- 133.9649
- 133.9640
- 133.9603
- 133.9592
- 131.2715
- 129.7845
- 129.7844
- 129.6988
- 129.6987
- 127.8909
- 127.8908
- 127.7183
- 127.7188
- 89.2023
- 79.9833
- 79.9834
- 74.7863
- 62.9154
- 56.0023
- 28.8863
- 19.9582

¹H NMR



¹³C NMR



1H NMR spectrum of compound S10 in CDCl₃. The chemical structure of S10 is shown above the spectrum. The structure is a substituted cyclohexane with a TBDPSO group, a BnO group, a NAc group, and a 4-chlorophenyl group. The NMR spectrum shows peaks for the TBDPSO group (7.1-7.8 ppm), the BnO group (4.5-4.9 ppm), the NAc group (2.0-2.1 ppm), the 4-chlorophenyl group (7.2-7.8 ppm), and the cyclohexane protons (1.1-1.9 ppm). The x-axis is labeled 'f1 (ppm)' and ranges from 8.5 to -0.5. The y-axis is labeled 'Intensity' and ranges from 0 to 500. The chemical structure is labeled 'S10'.

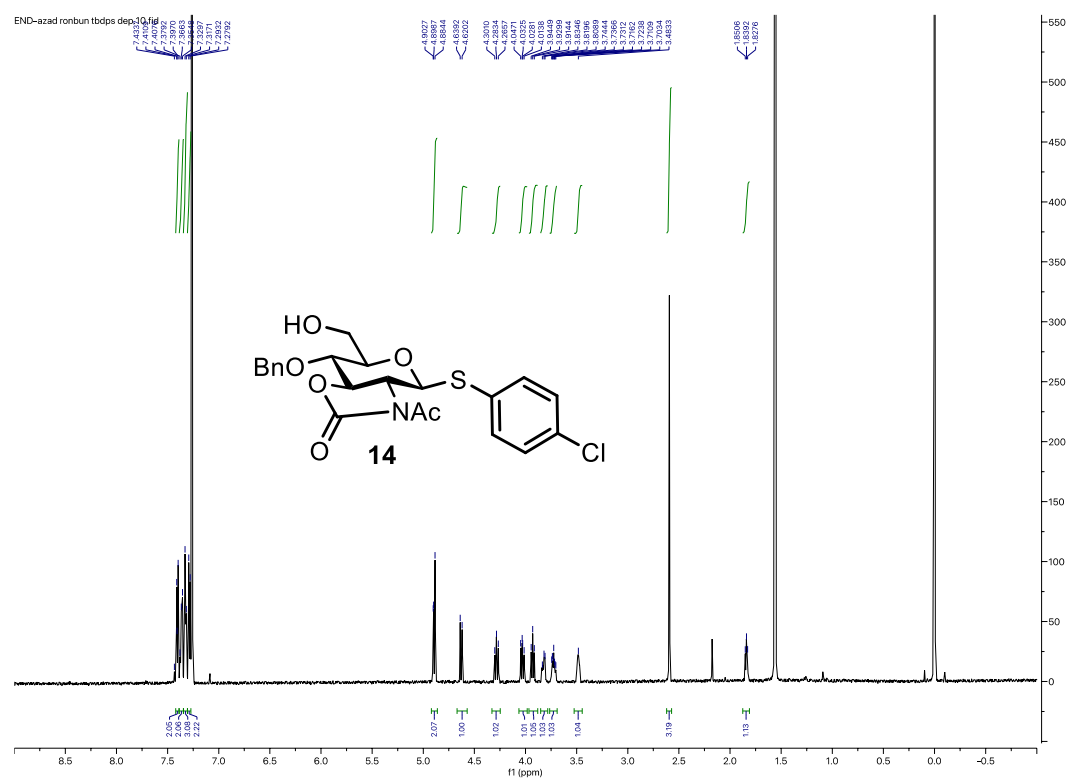
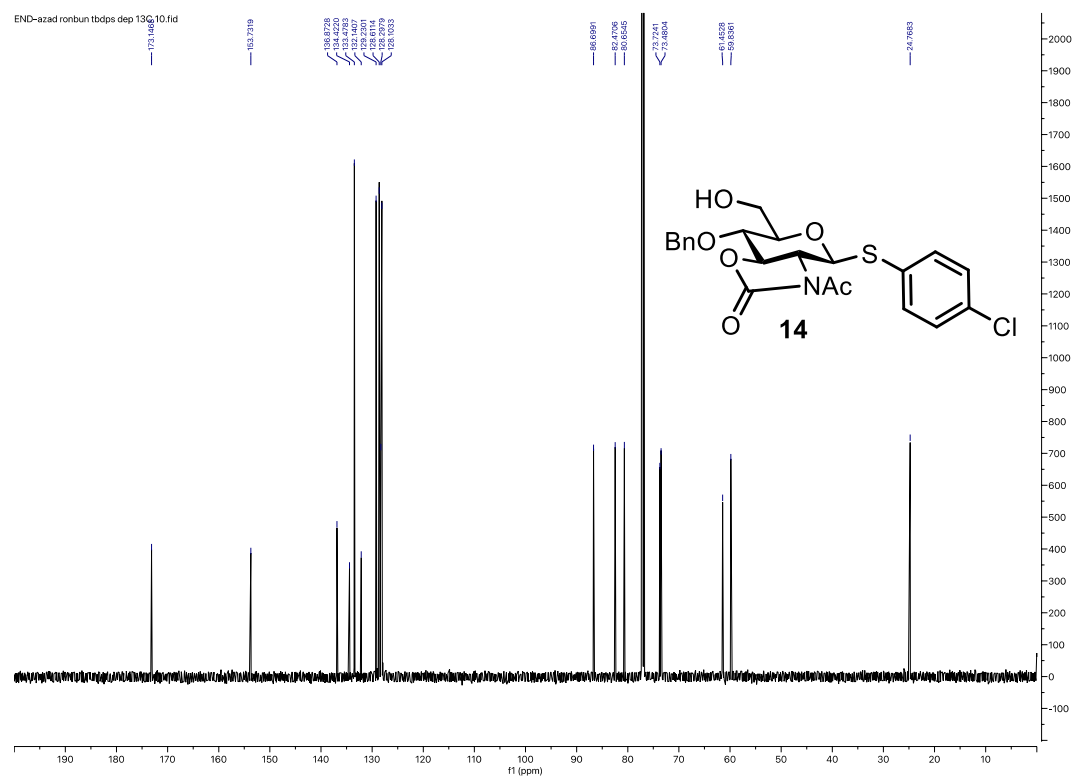
END-azad ronbun NAc 13C-10.fid

173.20167
153.96657
137.62223
135.81856
134.25175
133.95922
133.77255
132.77255
129.85444
129.85555
128.64188
128.58444
127.78009
86.39924
82.78904
81.22728
73.86965
73.64333
62.47799
59.84644
26.88910
24.89002
19.30222

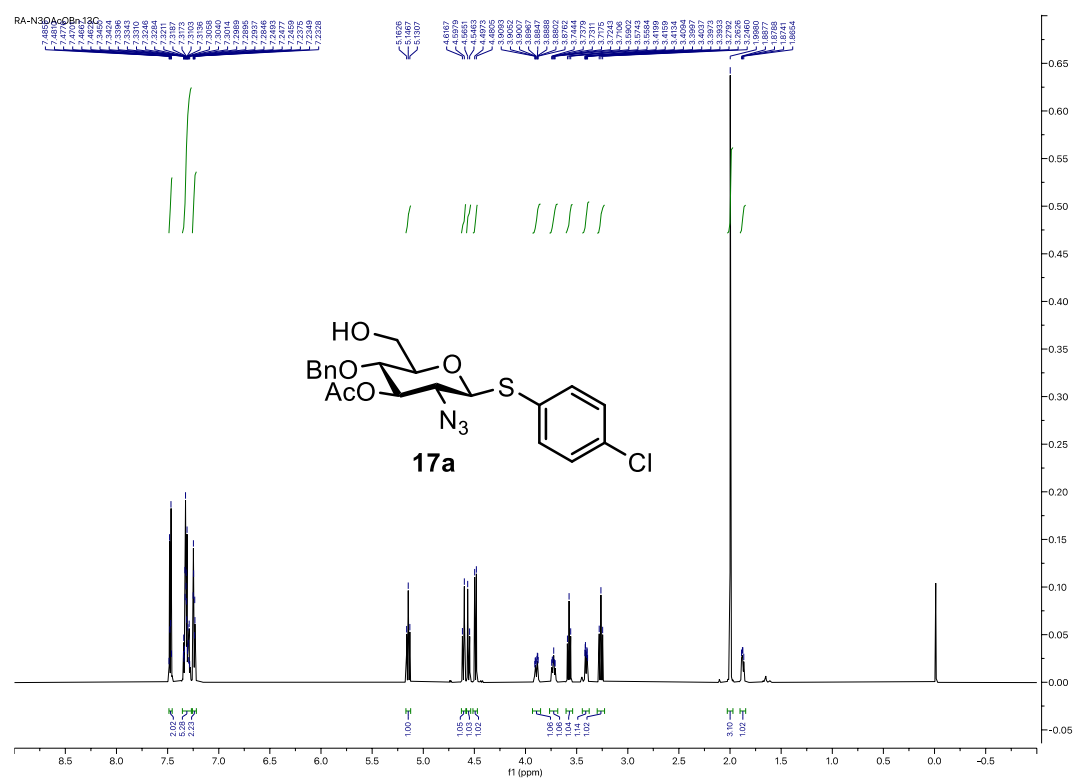
TBDPSO
BnO
NAC
S
Cl

S10

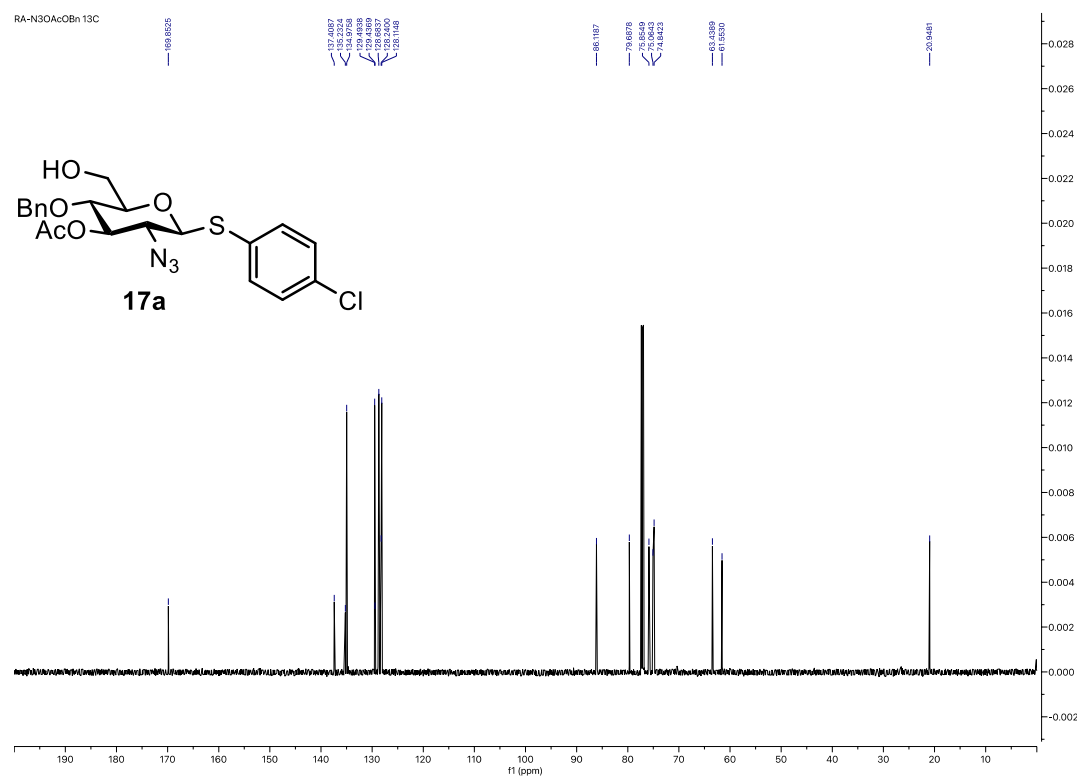
f1 (ppm)

¹H NMR¹³C NMR

¹H NMR



¹³C NMR



END-738 rec-1 13C.10.tif

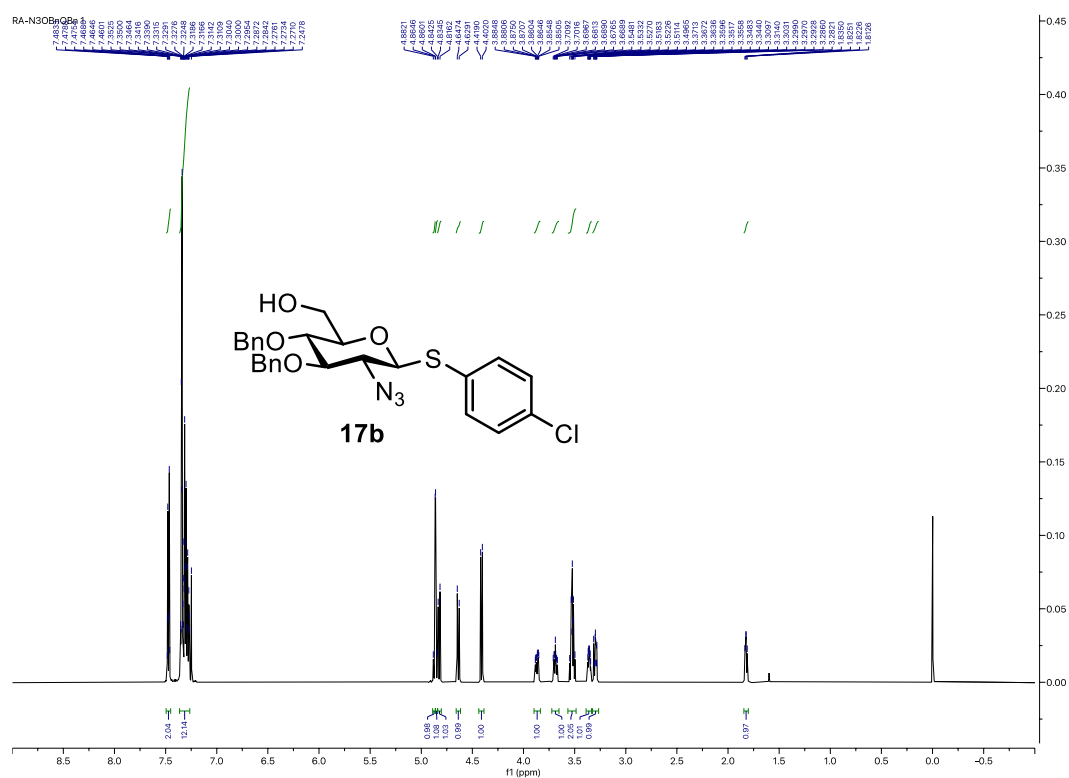
Chemical structure of compound **S13** is shown, which is a substituted tetrahydropyran derivative. The structure features a benzylidene acetal protecting group, a benzyl ether, and a 4-chlorophenylthio group.

The ¹³C NMR spectrum (CDCl₃) is displayed below the structure, showing peaks corresponding to the carbon atoms in the molecule. The x-axis represents the chemical shift in ppm (f1), ranging from 190 to 60. The y-axis represents the intensity, ranging from -1000 to 15000.

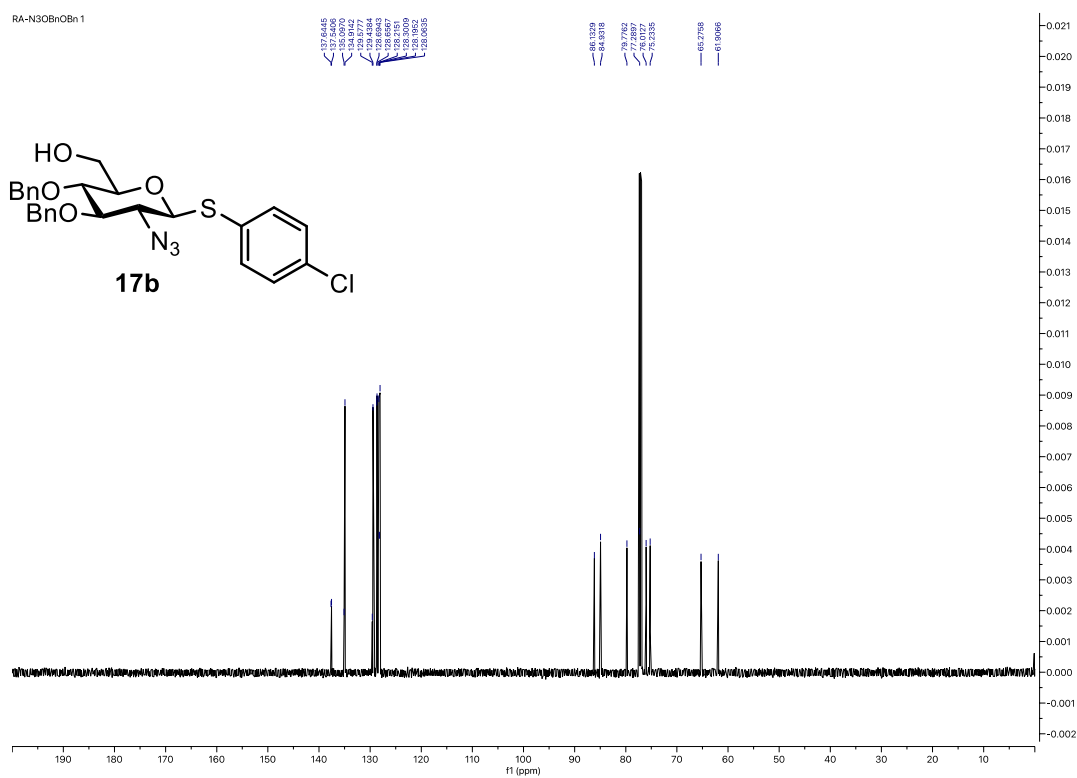
Key peaks in the spectrum are labeled with their chemical shifts (ppm):

- 137.5508
- 136.7614
- 135.7942
- 135.2689
- 134.7612
- 129.2041
- 128.9312
- 128.8207
- 128.5507
- 128.3996
- 126.2517
- 107.3246
- 88.4700
- 81.8272
- 80.3869
- 78.2414
- 76.5953
- 68.4729
- 64.6967

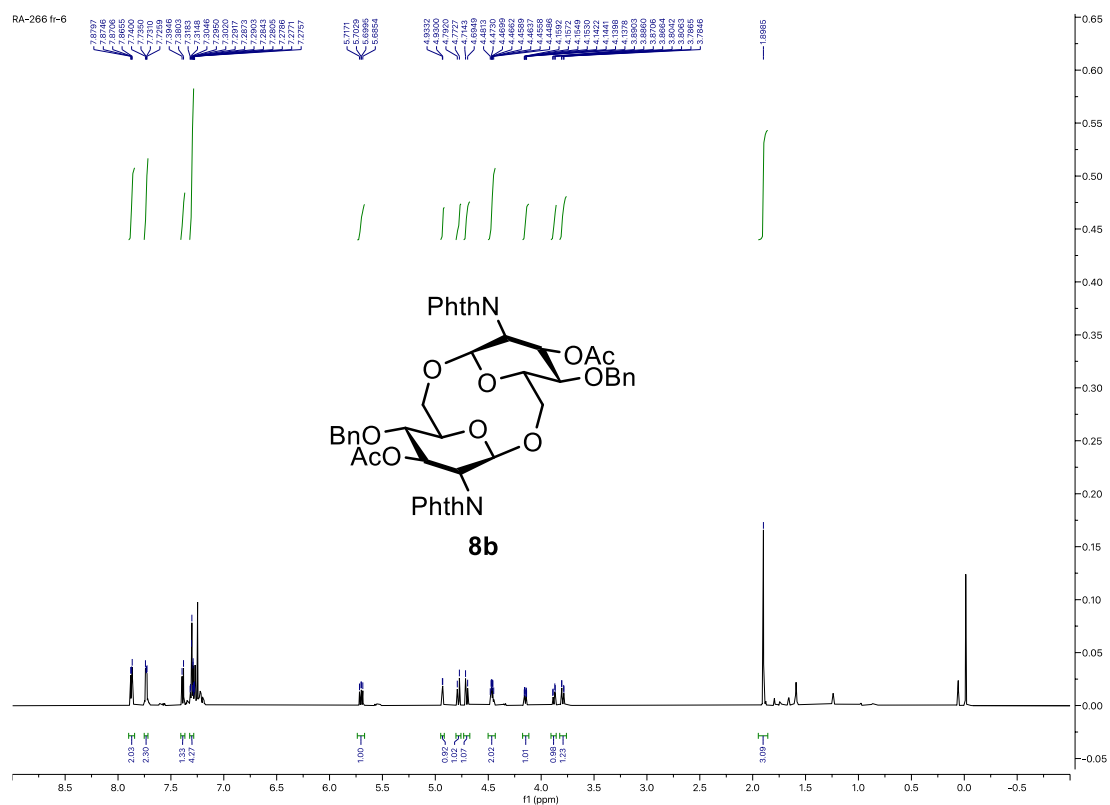
¹H NMR



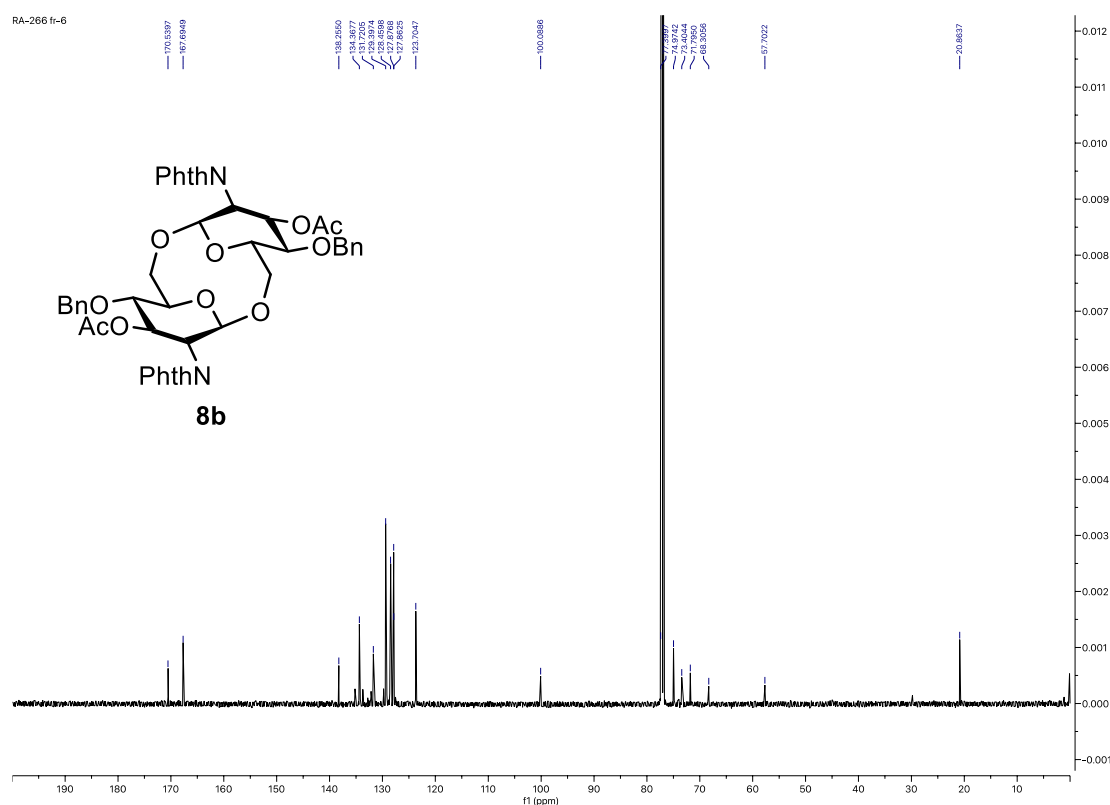
¹³C NMR



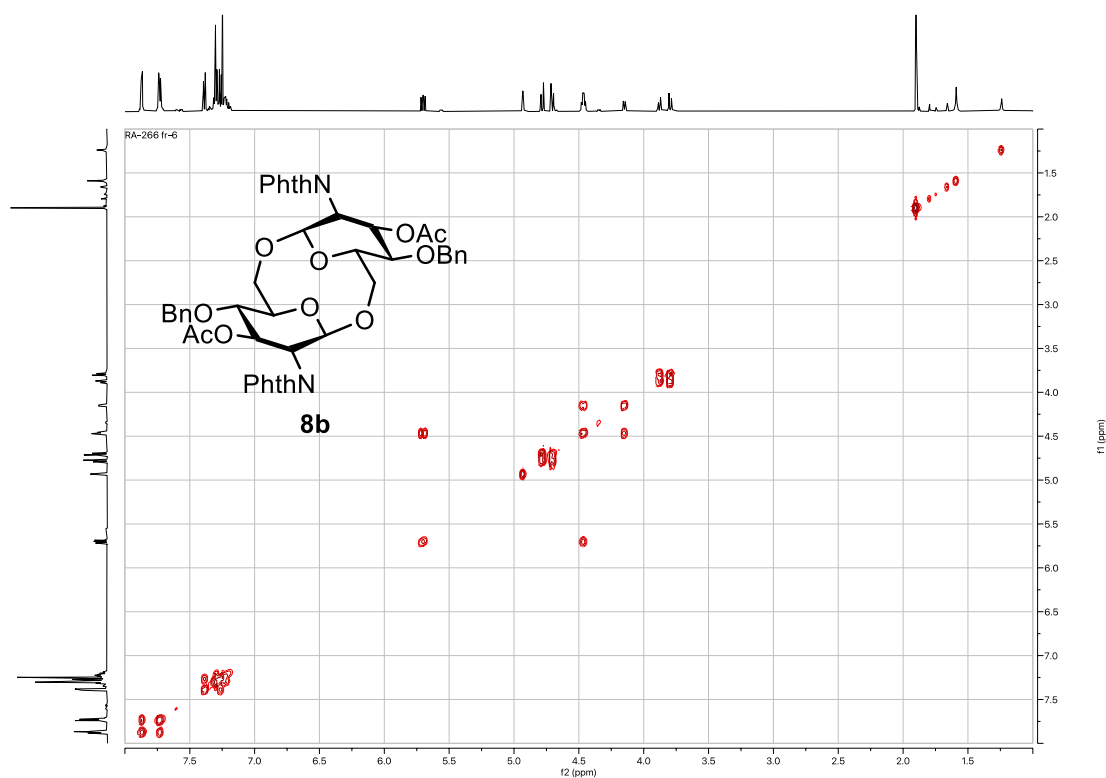
¹H NMR



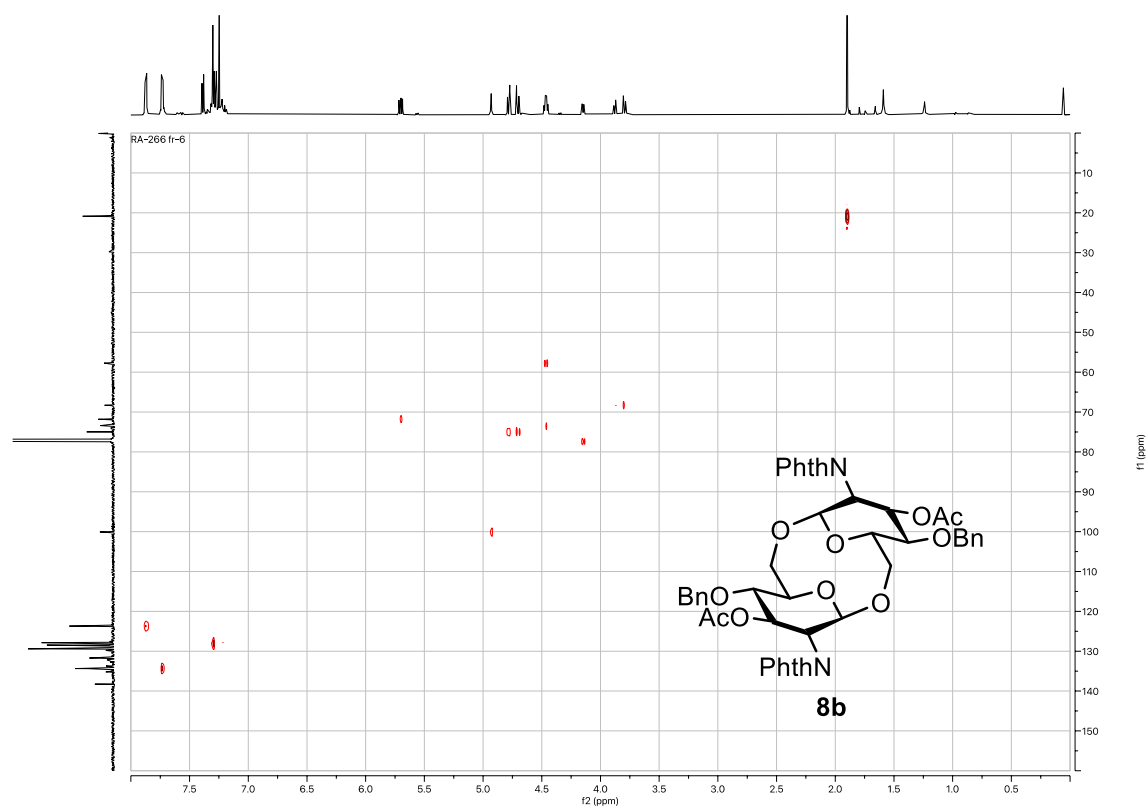
¹³C NMR



H-H cosy

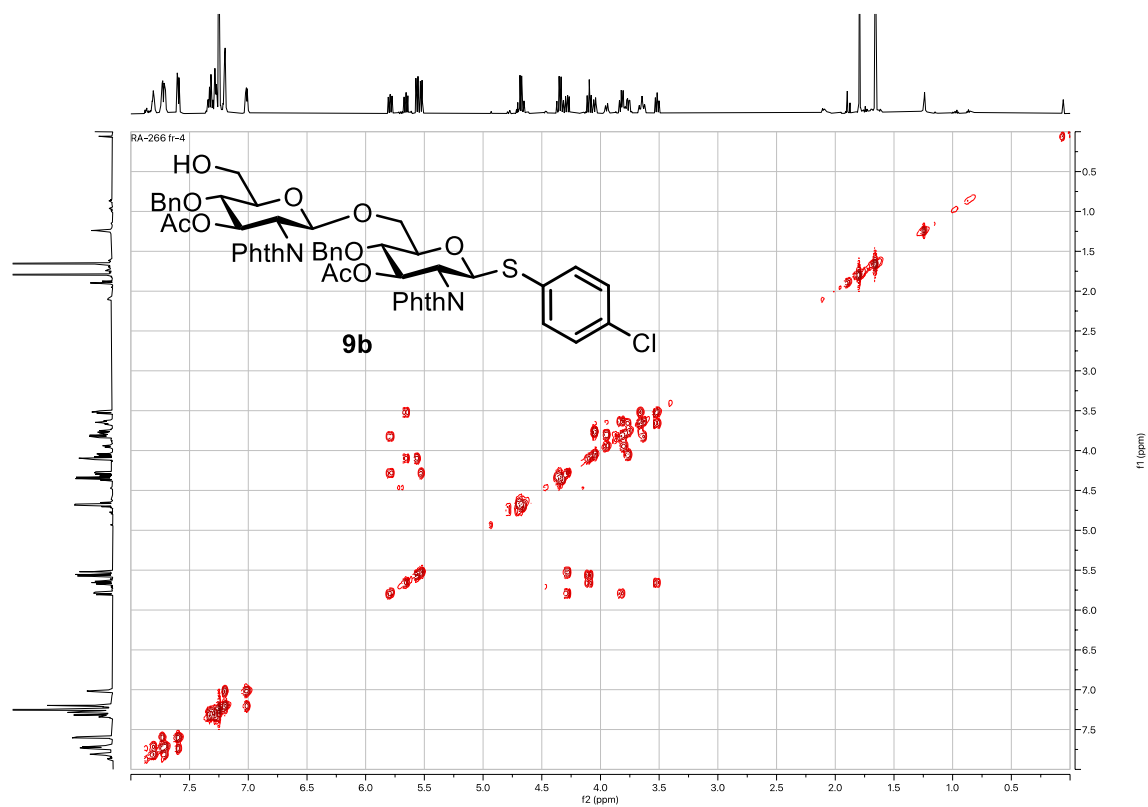


HMQC

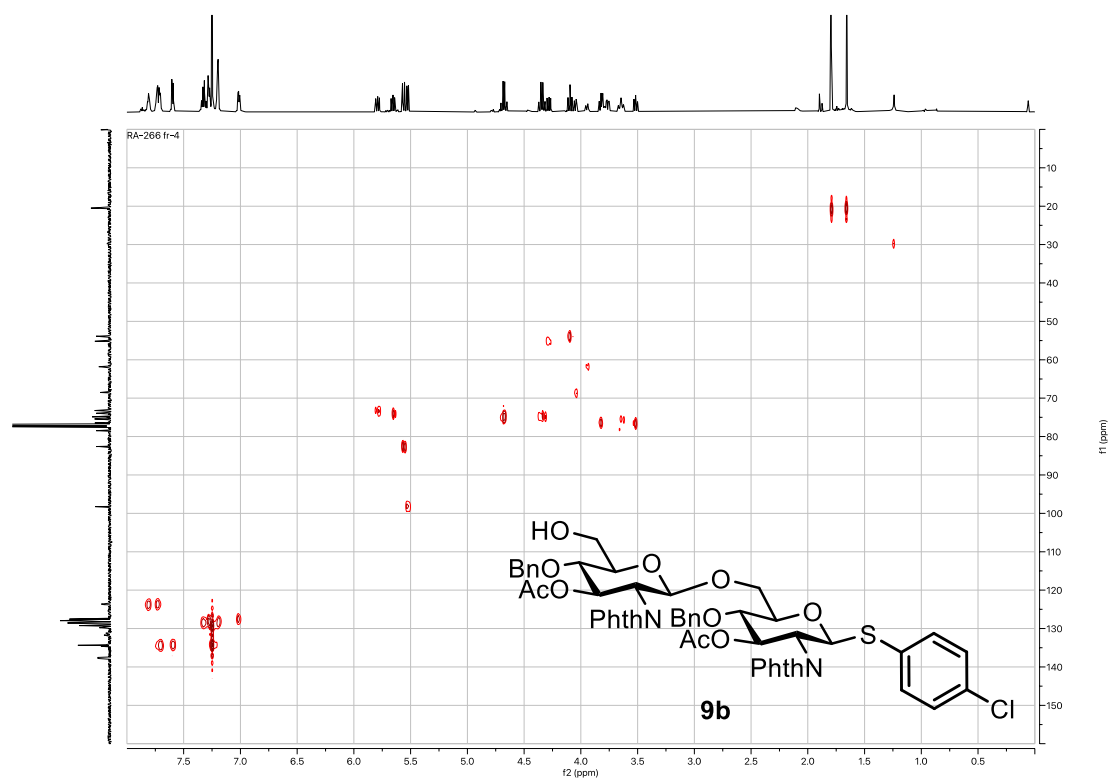


[illegible]

H-H cosy



HMQC

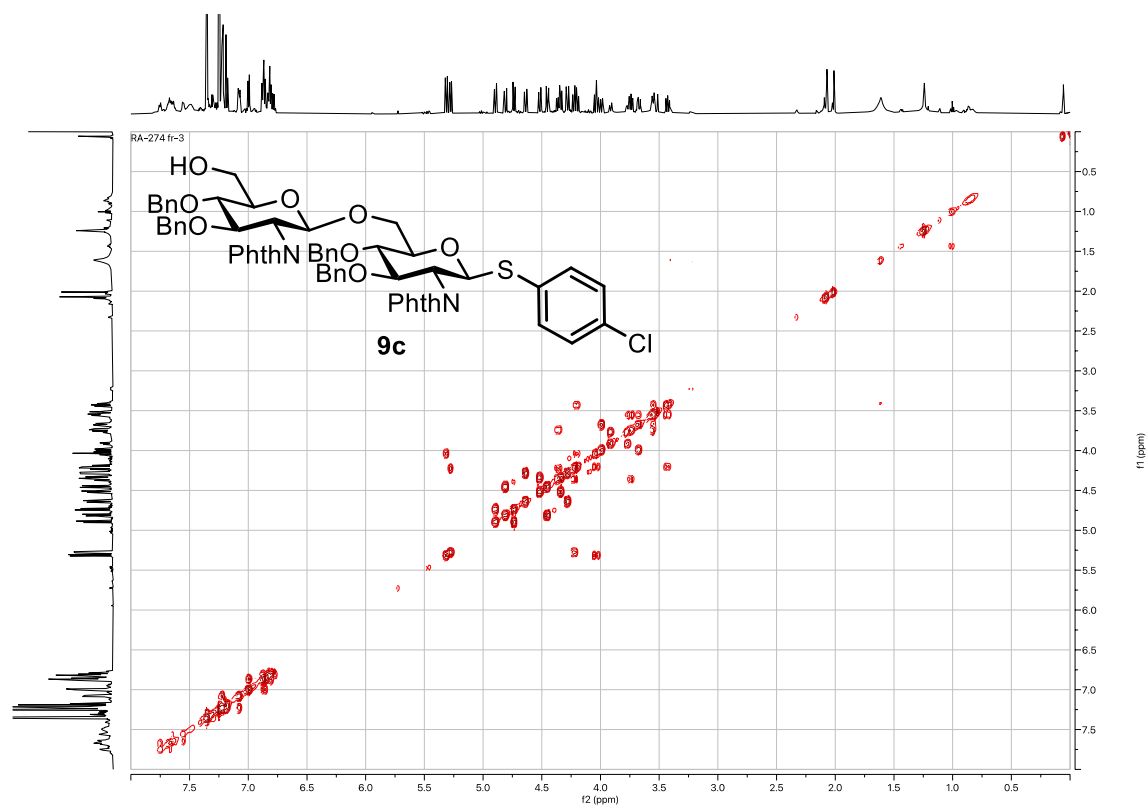


Chemical structure of compound **9c** is shown above the spectrum. The structure is a dimer of a substituted sugar derivative. It consists of two pyranose rings linked by an acetal bridge. Each ring has a PhthN group, a BnO group, and a Cl group. The Cl atom is on a para-substituted phenyl ring.

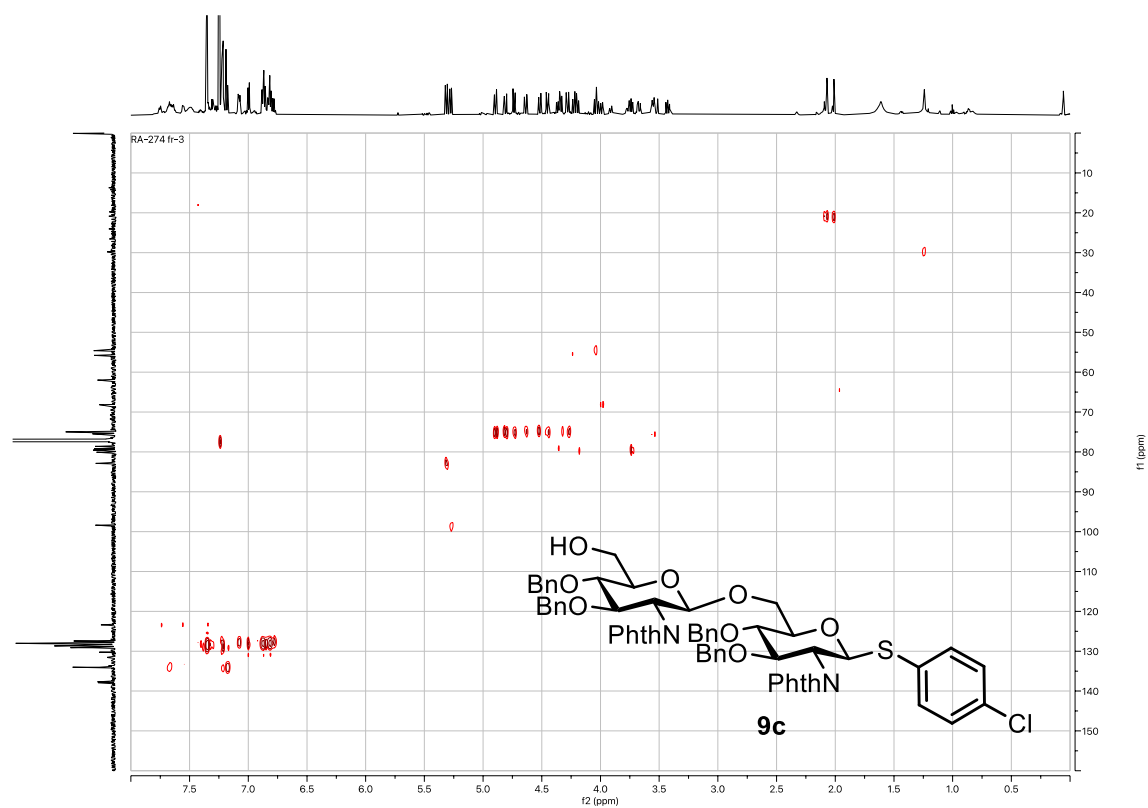
1H NMR spectrum (CDCl₃) data:

Chemical Shift (ppm)	Integration
7.500	0.0005
7.400	0.0005
7.300	0.0005
7.200	0.0005
7.100	0.0005
7.000	0.0005
6.900	0.0005
6.800	0.0005
6.700	0.0005
6.600	0.0005
6.500	0.0005
6.400	0.0005
6.300	0.0005
6.200	0.0005
6.100	0.0005
6.000	0.0005
5.900	0.0005
5.800	0.0005
5.700	0.0005
5.600	0.0005
5.500	0.0005
5.400	0.0005
5.300	0.0005
5.200	0.0005
5.100	0.0005
5.000	0.0005
4.900	0.0005
4.800	0.0005
4.700	0.0005
4.600	0.0005
4.500	0.0005
4.400	0.0005
4.300	0.0005
4.200	0.0005
4.100	0.0005
4.000	0.0005
3.900	0.0005
3.800	0.0005
3.700	0.0005
3.600	0.0005
3.500	0.0005
3.400	0.0005
3.300	0.0005
3.200	0.0005
3.100	0.0005
3.000	0.0005
2.900	0.0005
2.800	0.0005
2.700	0.0005
2.600	0.0005
2.500	0.0005
2.400	0.0005
2.300	0.0005
2.200	0.0005
2.100	0.0005
2.000	0.0005
1.900	0.0005
1.800	0.0005
1.700	0.0005
1.600	0.0005
1.500	0.0005
1.400	0.0005
1.300	0.0005
1.200	0.0005
1.100	0.0005
1.000	0.0005
0.900	0.0005
0.800	0.0005
0.700	0.0005
0.600	0.0005
0.500	0.0005
0.400	0.0005
0.300	0.0005
0.200	0.0005
0.100	0.0005
0.000	0.0005

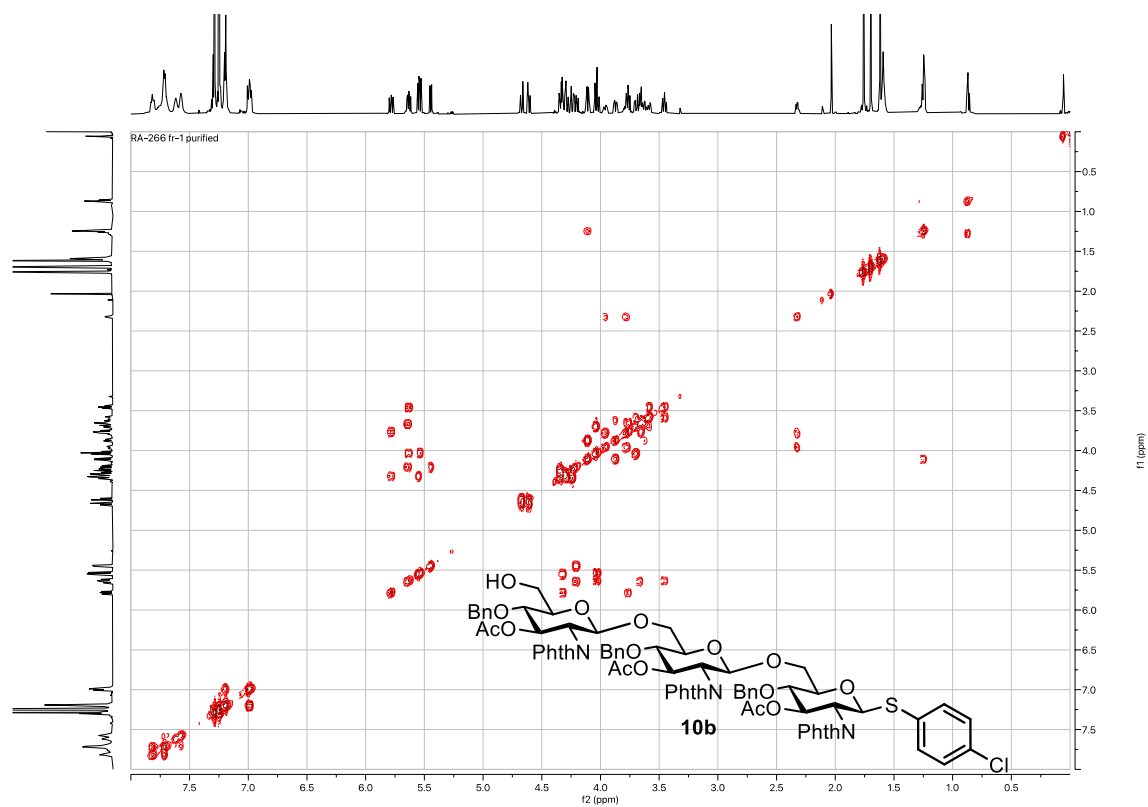
^1H NMR



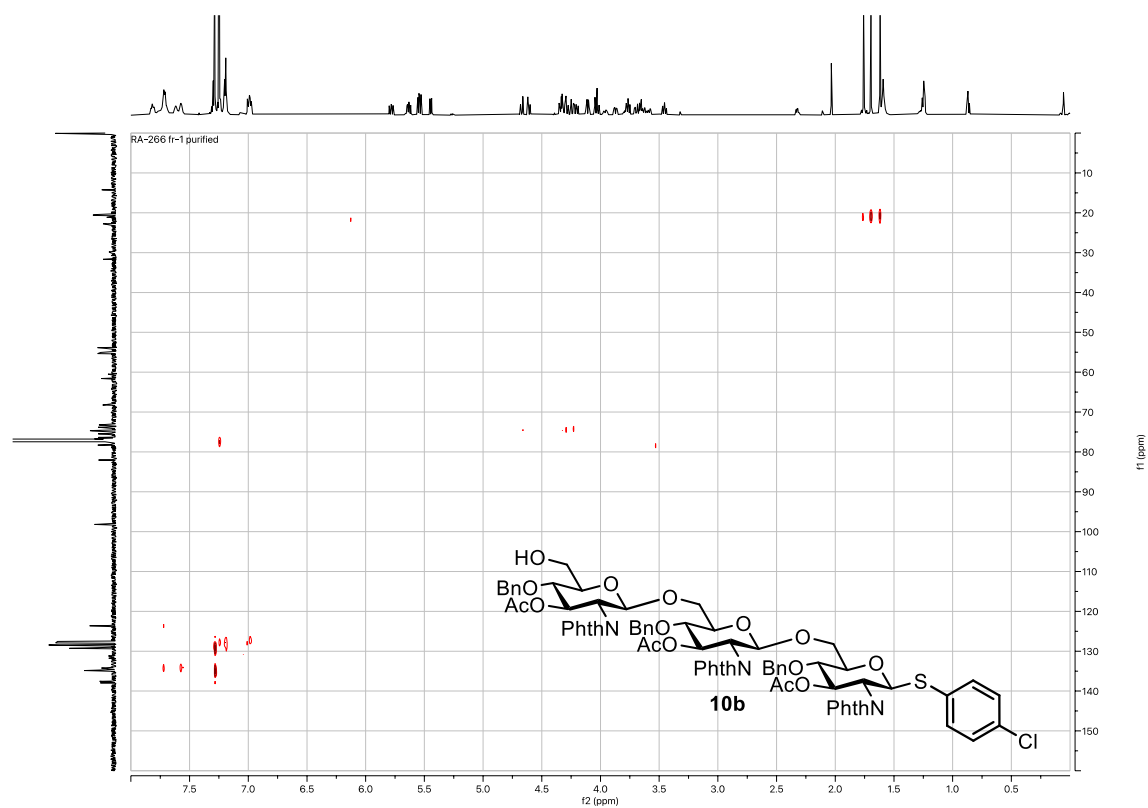
^{13}C NMR



H-H cosy



HMQC



Chemical structure of compound **10c** is shown above the spectrum. The structure is a trisaccharide with three glucose units linked by (1->3) glycosidic bonds. The top and middle units are protected with benzyl (BnO) and phthalimide (PhthN) groups. The bottom unit is linked to a 4-chlorophenyl group via a thioether bond.

¹H NMR spectrum (CDCl₃) of compound **10c**. The spectrum shows peaks from 0 to 8 ppm. The chemical structure of **10c** is shown above the spectrum.

Peak lists (ppm):

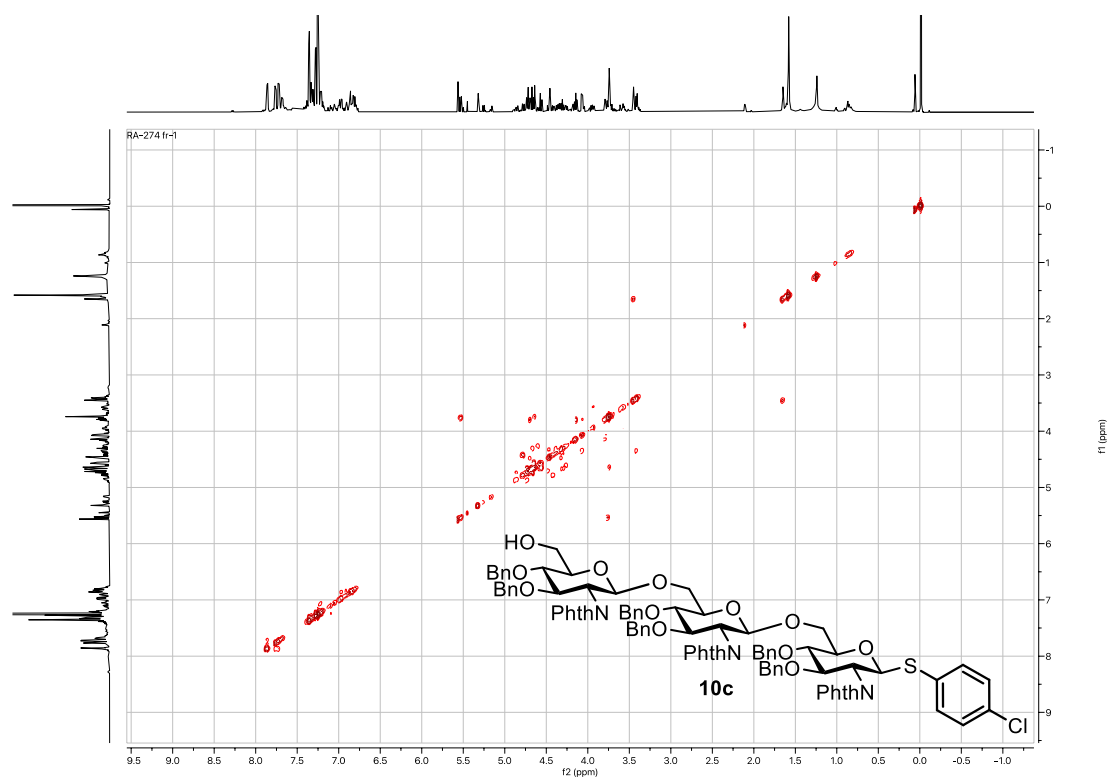
- Top list (7.070 to 1.9600): 7.070, 7.069, 7.068, 7.067, 7.066, 7.065, 7.064, 7.063, 7.062, 7.061, 7.060, 7.059, 7.058, 7.057, 7.056, 7.055, 7.054, 7.053, 7.052, 7.051, 7.050, 7.049, 7.048, 7.047, 7.046, 7.045, 7.044, 7.043, 7.042, 7.041, 7.040, 7.039, 7.038, 7.037, 7.036, 7.035, 7.034, 7.033, 7.032, 7.031, 7.030, 7.029, 7.028, 7.027, 7.026, 7.025, 7.024, 7.023, 7.022, 7.021, 7.020, 7.019, 7.018, 7.017, 7.016, 7.015, 7.014, 7.013, 7.012, 7.011, 7.010, 7.009, 7.008, 7.007, 7.006, 7.005, 7.004, 7.003, 7.002, 7.001, 7.000, 6.999, 6.998, 6.997, 6.996, 6.995, 6.994, 6.993, 6.992, 6.991, 6.990, 6.989, 6.988, 6.987, 6.986, 6.985, 6.984, 6.983, 6.982, 6.981, 6.980, 6.979, 6.978, 6.977, 6.976, 6.975, 6.974, 6.973, 6.972, 6.971, 6.970, 6.969, 6.968, 6.967, 6.966, 6.965, 6.964, 6.963, 6.962, 6.961, 6.960, 6.959, 6.958, 6.957, 6.956, 6.955, 6.954, 6.953, 6.952, 6.951, 6.950, 6.949, 6.948, 6.947, 6.946, 6.945, 6.944, 6.943, 6.942, 6.941, 6.940, 6.939, 6.938, 6.937, 6.936, 6.935, 6.934, 6.933, 6.932, 6.931, 6.930, 6.929, 6.928, 6.927, 6.926, 6.925, 6.924, 6.923, 6.922, 6.921, 6.920, 6.919, 6.918, 6.917, 6.916, 6.915, 6.914, 6.913, 6.912, 6.911, 6.910, 6.909, 6.908, 6.907, 6.906, 6.905, 6.904, 6.903, 6.902, 6.901, 6.900, 6.899, 6.898, 6.897, 6.896, 6.895, 6.894, 6.893, 6.892, 6.891, 6.890, 6.889, 6.888, 6.887, 6.886, 6.885, 6.884, 6.883, 6.882, 6.881, 6.880, 6.879, 6.878, 6.877, 6.876, 6.875, 6.874, 6.873, 6.872, 6.871, 6.870, 6.869, 6.868, 6.867, 6.866, 6.865, 6.864, 6.863, 6.862, 6.861, 6.860, 6.859, 6.858, 6.857, 6.856, 6.855, 6.854, 6.853, 6.852, 6.851, 6.850, 6.849, 6.848, 6.847, 6.846, 6.845, 6.844, 6.843, 6.842, 6.841, 6.840, 6.839, 6.838, 6.837, 6.836, 6.835, 6.834, 6.833, 6.832, 6.831, 6.830, 6.829, 6.828, 6.827, 6.826, 6.825, 6.824, 6.823, 6.822, 6.821, 6.820, 6.819, 6.818, 6.817, 6.816, 6.815, 6.814, 6.813, 6.812, 6.811, 6.810, 6.809, 6.808, 6.807, 6.806, 6.805, 6.804, 6.803, 6.802, 6.801, 6.800, 6.799, 6.798, 6.797, 6.796, 6.795, 6.794, 6.793, 6.792, 6.791, 6.790, 6.789, 6.788, 6.787, 6.786, 6.785, 6.784, 6.783, 6.782, 6.781, 6.780, 6.779, 6.778, 6.777, 6.776, 6.775, 6.774, 6.773, 6.772, 6.771, 6.770, 6.769, 6.768, 6.767, 6.766, 6.765, 6.764, 6.763, 6.762, 6.761, 6.760, 6.759, 6.758, 6.757, 6.756, 6.755, 6.754, 6.753, 6.752, 6.751, 6.750, 6.749, 6.748, 6.747, 6.746, 6.745, 6.744, 6.743, 6.742, 6.741, 6.740, 6.739, 6.738, 6.737, 6.736, 6.735, 6.734, 6.733, 6.732, 6.731, 6.730, 6.729, 6.728, 6.727, 6.726, 6.725, 6.724, 6.723, 6.722, 6.721, 6.720, 6.719, 6.718, 6.717, 6.716, 6.715, 6.714, 6.713, 6.712, 6.711, 6.710, 6.709, 6.708, 6.707, 6.706, 6.705, 6.704, 6.703, 6.702, 6.701, 6.700, 6.699, 6.698, 6.697, 6.696, 6.695, 6.694, 6.693, 6.692, 6.691, 6.690, 6.689, 6.688, 6.687, 6.686, 6.685, 6.684, 6.683, 6.682, 6.681, 6.680, 6.679, 6.678, 6.677, 6.676, 6.675, 6.674, 6.673, 6.672, 6.671, 6.670, 6.669, 6.668, 6.667, 6.666, 6.665, 6.664, 6.663, 6.662, 6.661, 6.660, 6.659, 6.658, 6.657, 6.656, 6.655, 6.654, 6.653, 6.652, 6.651, 6.650, 6.649, 6.648, 6.647, 6.646, 6.645, 6.644, 6.643, 6.642, 6.641, 6.640, 6.639, 6.638, 6.637, 6.636, 6.635, 6.634, 6.633, 6.632, 6.631, 6.630, 6.629, 6.628, 6.627, 6.626, 6.625, 6.624, 6.623, 6.622, 6.621, 6.620, 6.619, 6.618, 6.617, 6.616, 6.615, 6.614, 6.613, 6.612, 6.611, 6.610, 6.609, 6.608, 6.607, 6.606, 6.605, 6.604, 6.603, 6.602, 6.601, 6.600, 6.599, 6.598, 6.597, 6.596, 6.595, 6.594, 6.593, 6.592, 6.591, 6.590, 6.589, 6.588, 6.587, 6.586, 6.585, 6.584, 6.583, 6.582, 6.581, 6.580, 6.579, 6.578, 6.577, 6.576, 6.575, 6.574, 6.573, 6.572, 6.571, 6.570, 6.569, 6.568, 6.567, 6.566, 6.565, 6.564, 6.563, 6.562, 6.561, 6.560, 6.559, 6.558, 6.557, 6.556, 6.555, 6.554, 6.553, 6.552, 6.551, 6.550, 6.549, 6.548, 6.547, 6.546, 6.545, 6.544, 6.543, 6.542, 6.541, 6.540, 6.539, 6.538

Chemical structure of **10c** is shown above the spectrum. The structure is a trisaccharide derivative with a 4-chlorophenyl group attached to the rightmost sugar unit. The sugar units are linked by glycosidic bonds. The structure includes a benzylidene protecting group (BnO) and a phthalimide group (PhthN).

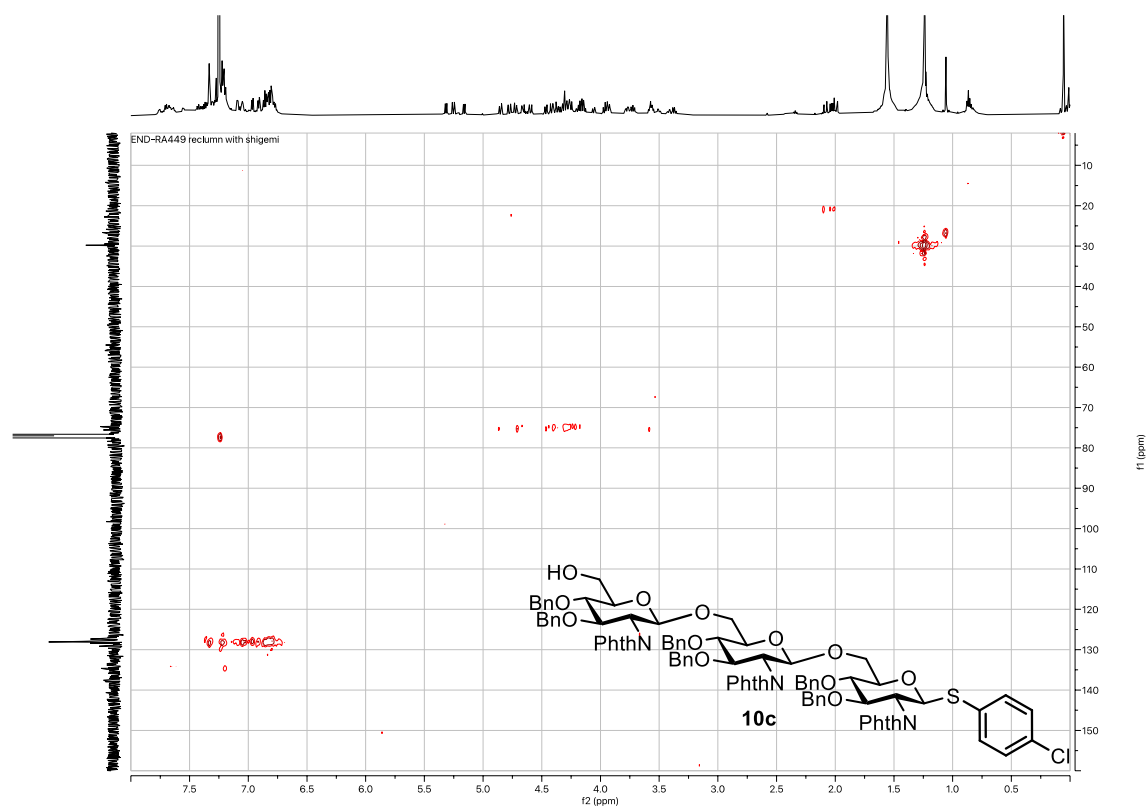
1H NMR spectrum (CDCl₃) data:

Chemical Shift (ppm)	Integration
7.26	1.00
7.24	1.00
7.22	1.00
7.20	1.00
7.18	1.00
7.16	1.00
7.14	1.00
7.12	1.00
7.10	1.00
7.08	1.00
7.06	1.00
7.04	1.00
7.02	1.00
7.00	1.00
6.98	1.00
6.96	1.00
6.94	1.00
6.92	1.00
6.90	1.00
6.88	1.00
6.86	1.00
6.84	1.00
6.82	1.00
6.80	1.00
6.78	1.00
6.76	1.00
6.74	1.00
6.72	1.00
6.70	1.00
6.68	1.00
6.66	1.00
6.64	1.00
6.62	1.00
6.60	1.00
6.58	1.00
6.56	1.00
6.54	1.00
6.52	1.00
6.50	1.00
6.48	1.00
6.46	1.00
6.44	1.00
6.42	1.00
6.40	1.00
6.38	1.00
6.36	1.00
6.34	1.00
6.32	1.00
6.30	1.00
6.28	1.00
6.26	1.00
6.24	1.00
6.22	1.00
6.20	1.00
6.18	1.00
6.16	1.00
6.14	1.00
6.12	1.00
6.10	1.00
6.08	1.00
6.06	1.00
6.04	1.00
6.02	1.00
6.00	1.00
5.98	1.00
5.96	1.00
5.94	1.00
5.92	1.00
5.90	1.00
5.88	1.00
5.86	1.00
5.84	1.00
5.82	1.00
5.80	1.00
5.78	1.00
5.76	1.00
5.74	1.00
5.72	1.00
5.70	1.00
5.68	1.00
5.66	1.00
5.64	1.00
5.62	1.00
5.60	1.00
5.58	1.00
5.56	1.00
5.54	1.00
5.52	1.00
5.50	1.00
5.48	1.00
5.46	1.00
5.44	1.00
5.42	1.00
5.40	1.00
5.38	1.00
5.36	1.00
5.34	1.00
5.32	1.00
5.30	1.00
5.28	1.00
5.26	1.00
5.24	1.00
5.22	1.00
5.20	1.00
5.18	1.00
5.16	1.00
5.14	1.00
5.12	1.00
5.10	1.00
5.08	1.00
5.06	1.00
5.04	1.00
5.02	1.00
5.00	1.00
4.98	1.00
4.96	1.00
4.94	1.00
4.92	1.00
4.90	1.00
4.88	1.00
4.86	1.00
4.84	1.00
4.82	1.00
4.80	1.00
4.78	1.00
4.76	1.00
4.74	1.00
4.72	1.00
4.70	1.00
4.68	1.00
4.66	1.00
4.64	1.00
4.62	1.00
4.60	1.00
4.58	1.00
4.56	1.00
4.54	1.00
4.52	1.00
4.50	1.00
4.48	1.00
4.46	1.00
4.44	1.00
4.42	1.00
4.40	1.00
4.38	1.00
4.36	1.00
4.34	1.00
4.32	1.00
4.30	1.00
4.28	1.00
4.26	1.00
4.24	1.00
4.22	1.00
4.20	1.00
4.18	1.00
4.16	1.00
4.14	1.00
4.12	1.00
4.10	1.00
4.08	1.00
4.06	1.00
4.04	1.00
4.02	1.00
4.00	1.00
3.98	1.00
3.96	1.00
3.94	1.00
3.92	1.00
3.90	1.00
3.88	1.00
3.86	1.00
3.84	1.00
3.82	1.00

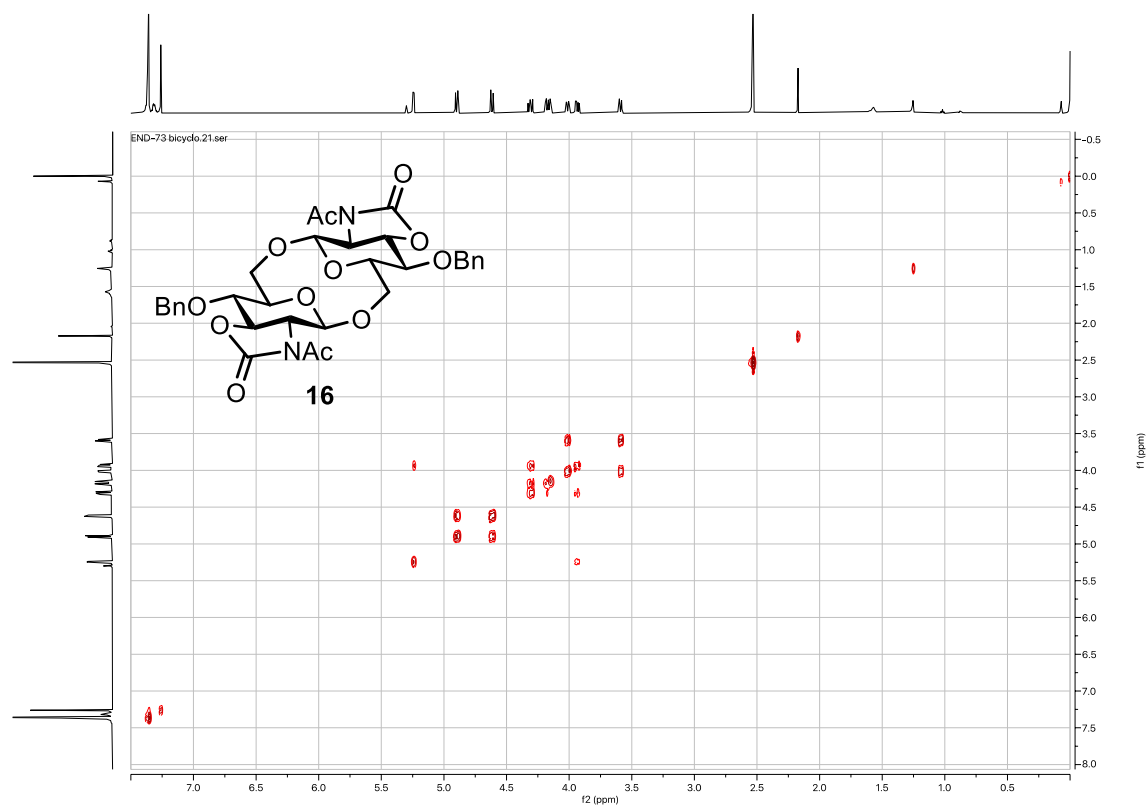
H-H cosy



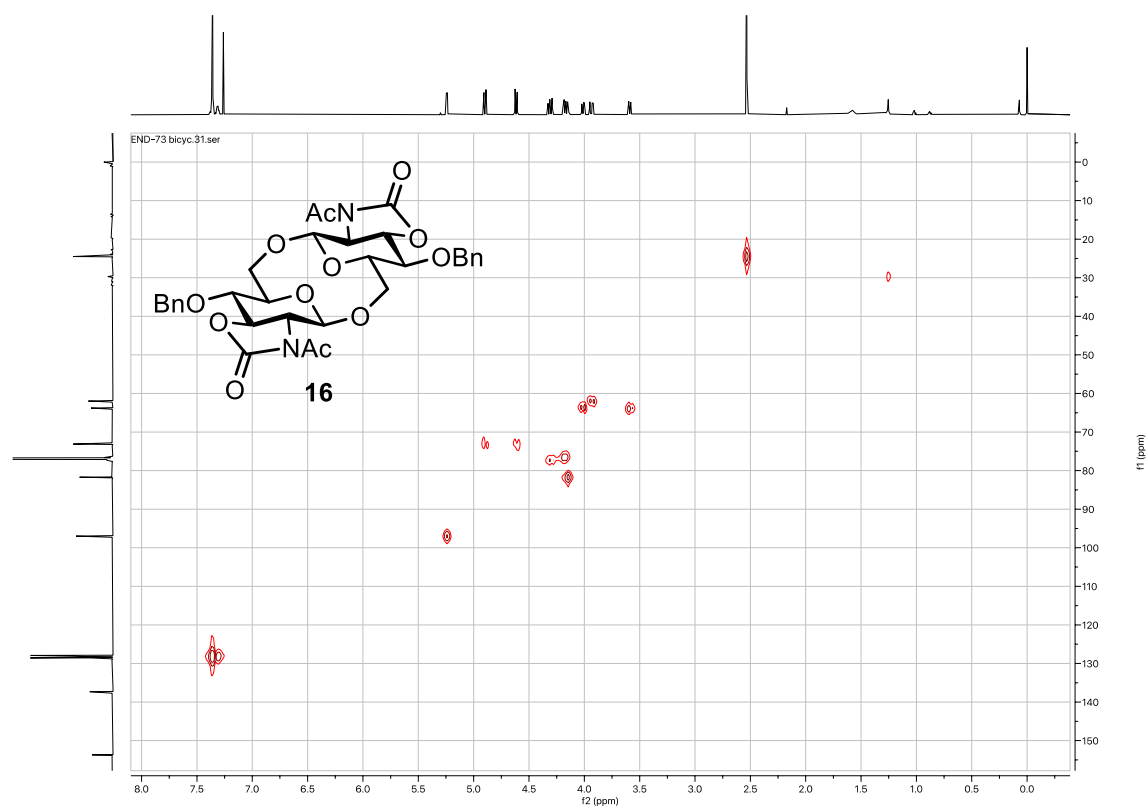
HMQC



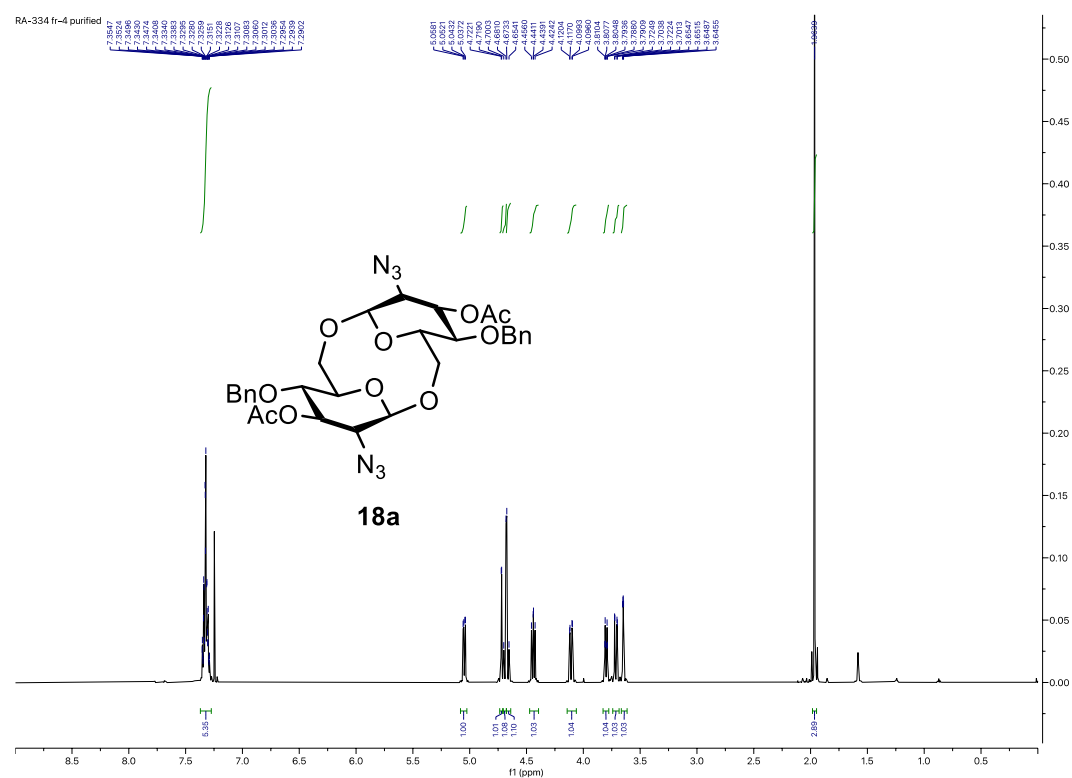
H-H cosy



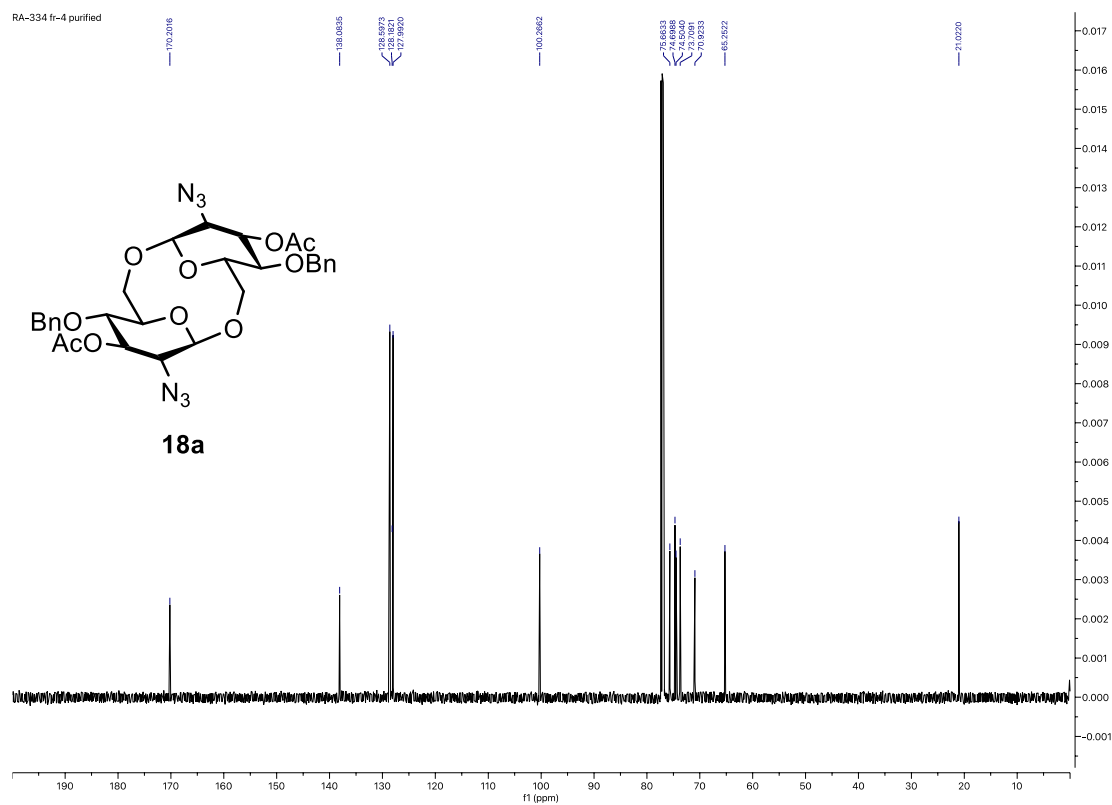
^{13}C NMR



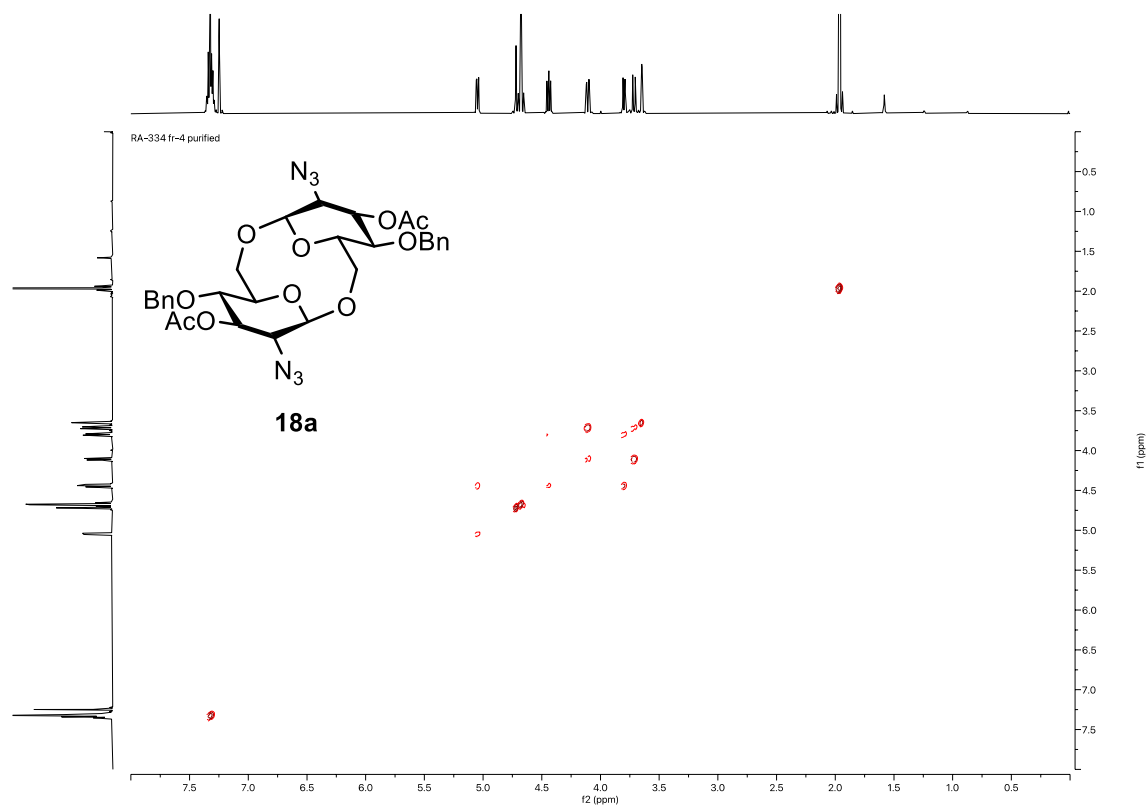
¹H NMR



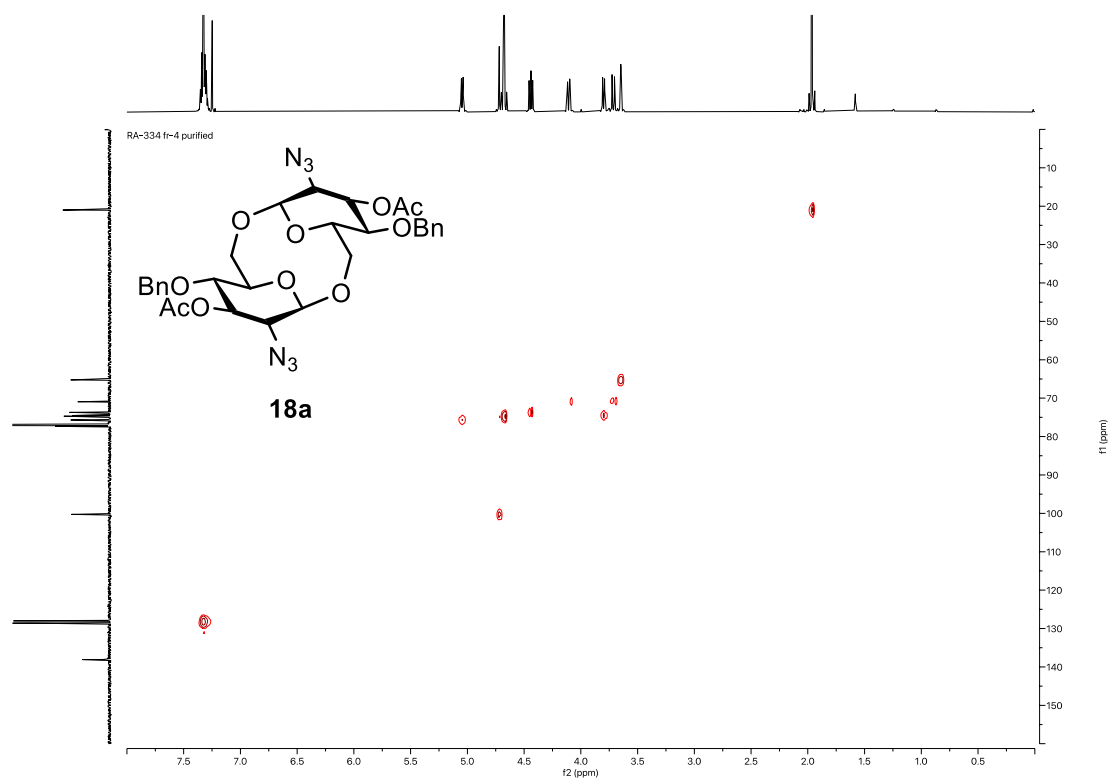
¹³C NMR



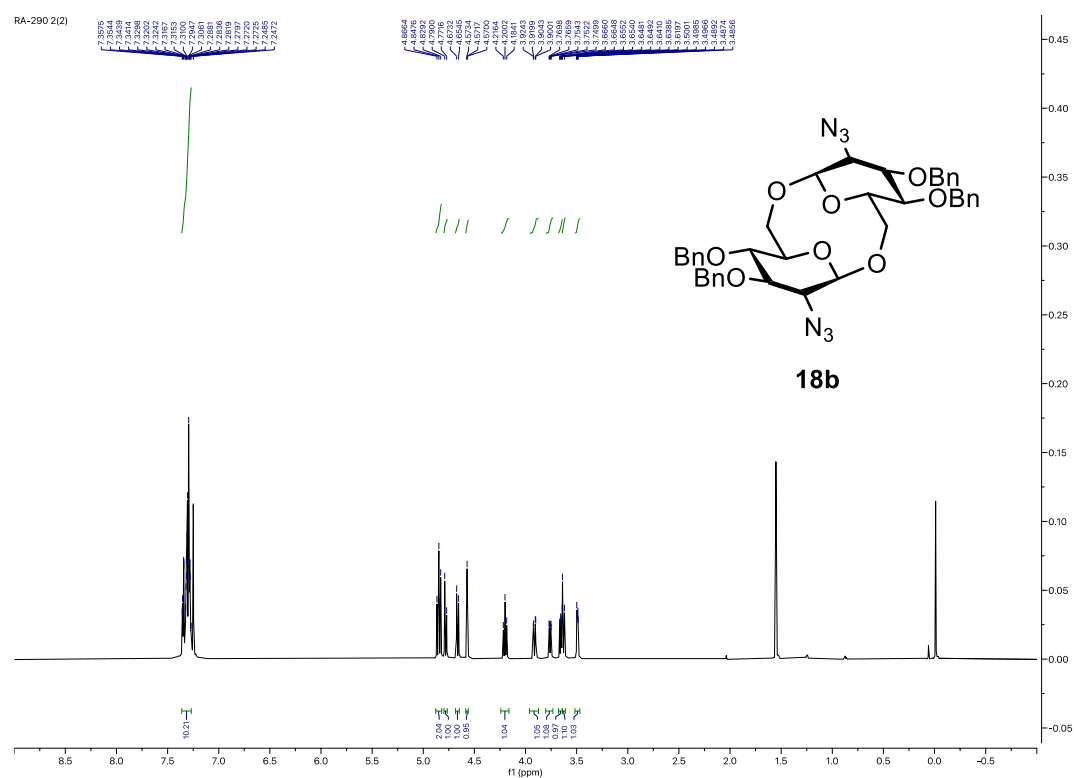
H-H cosy



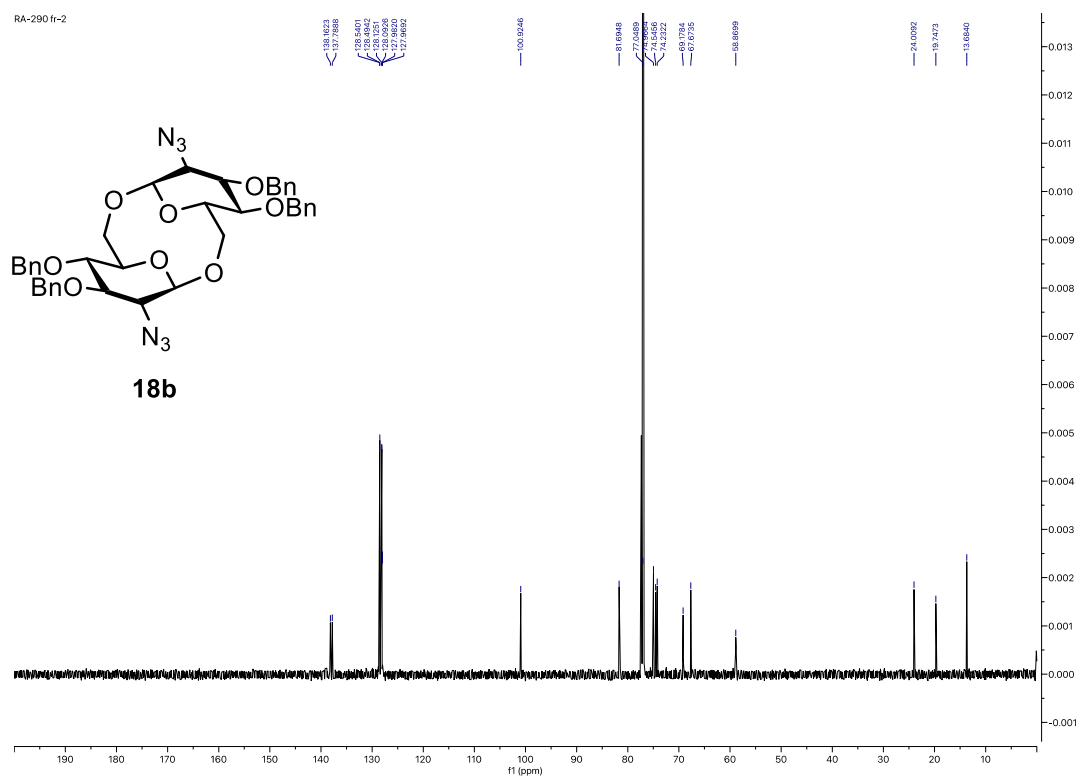
HMQC



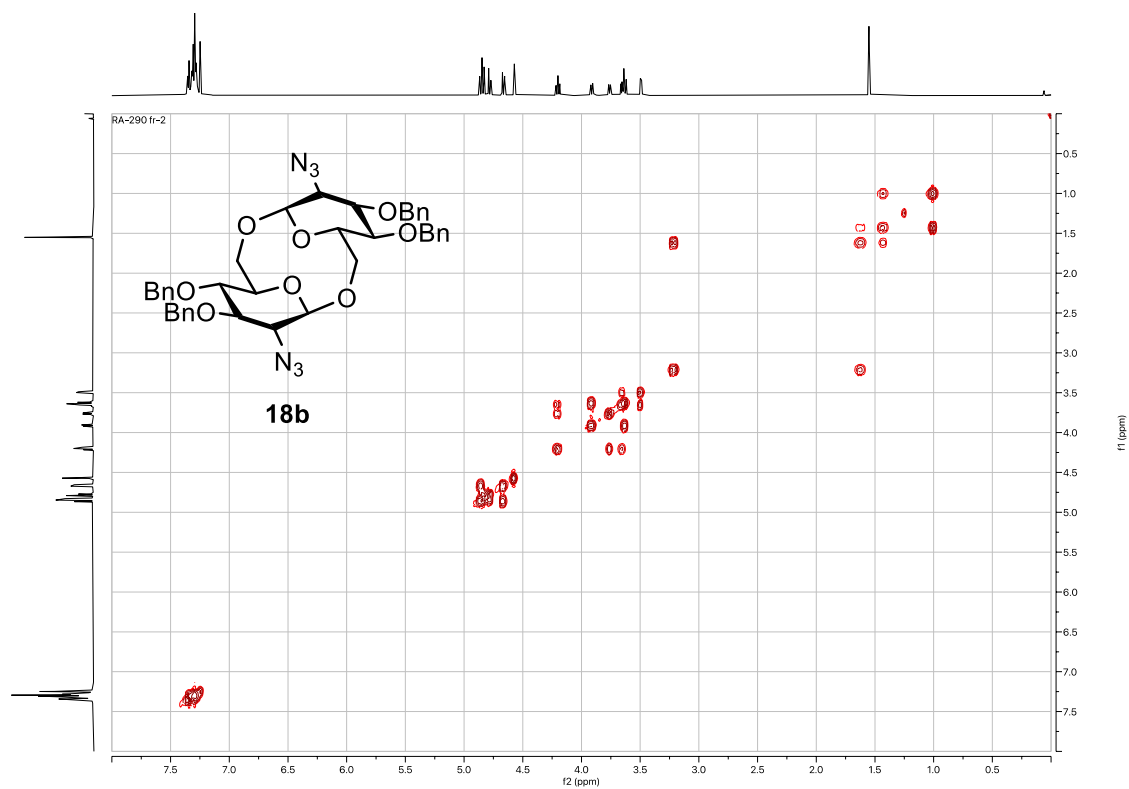
¹H NMR



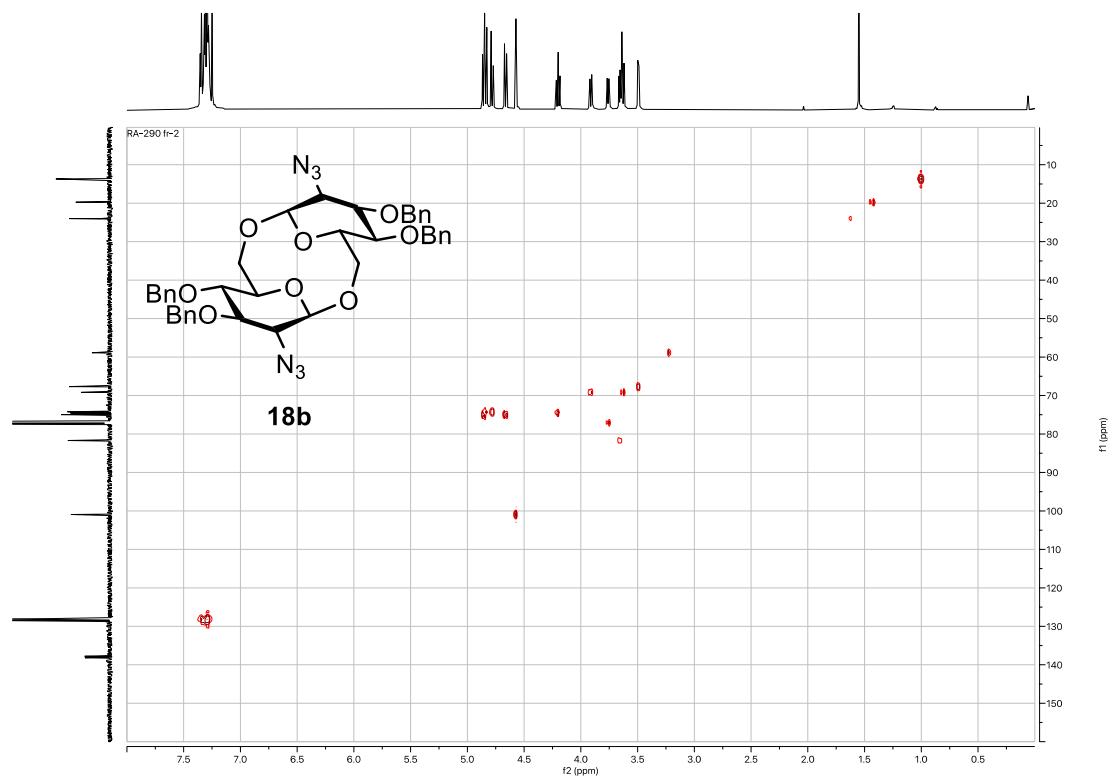
¹³C NMR

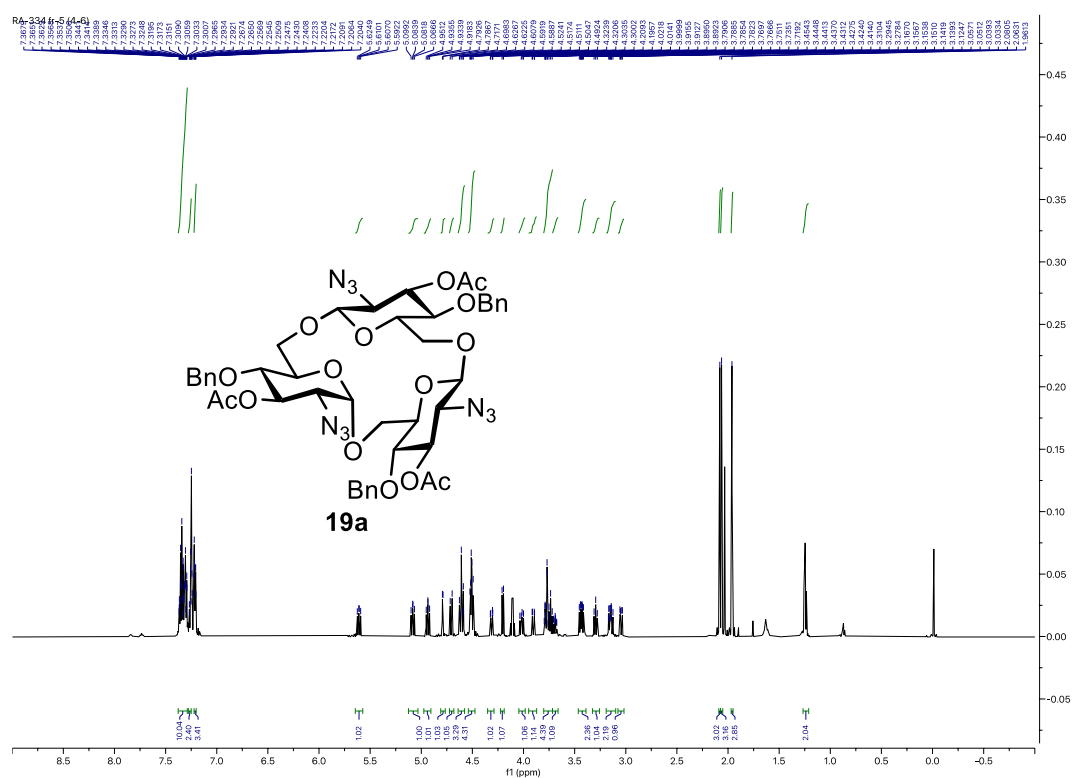
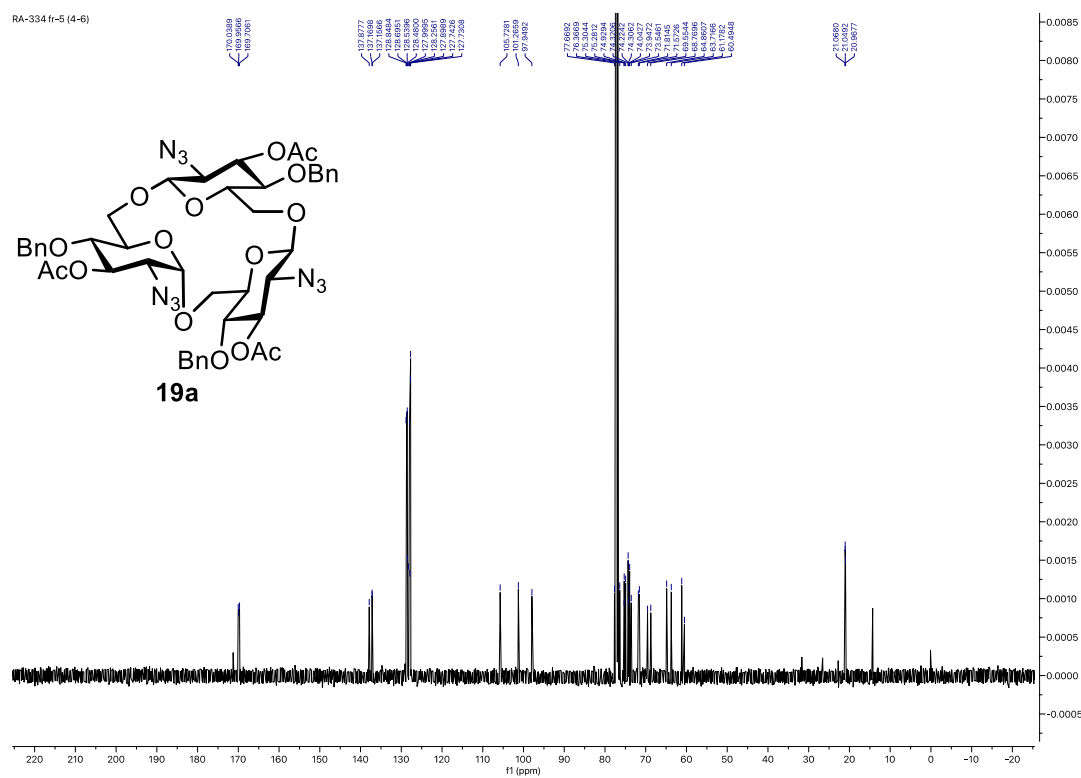


H-H cosy

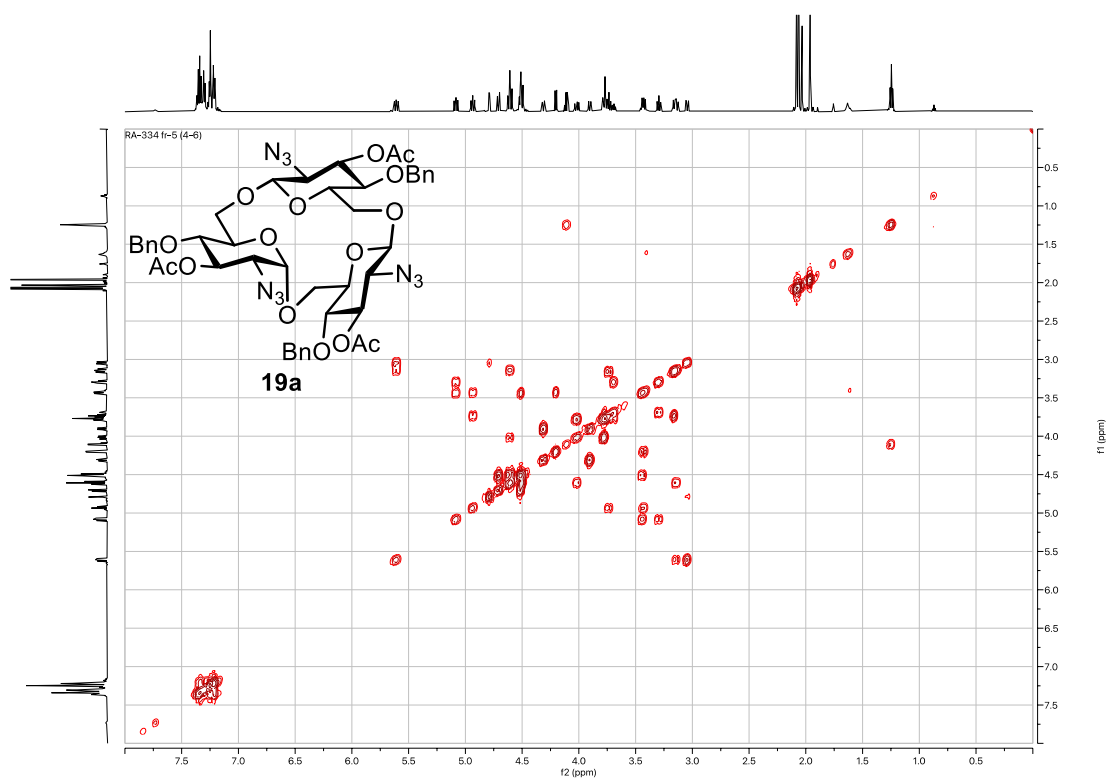


HMQC

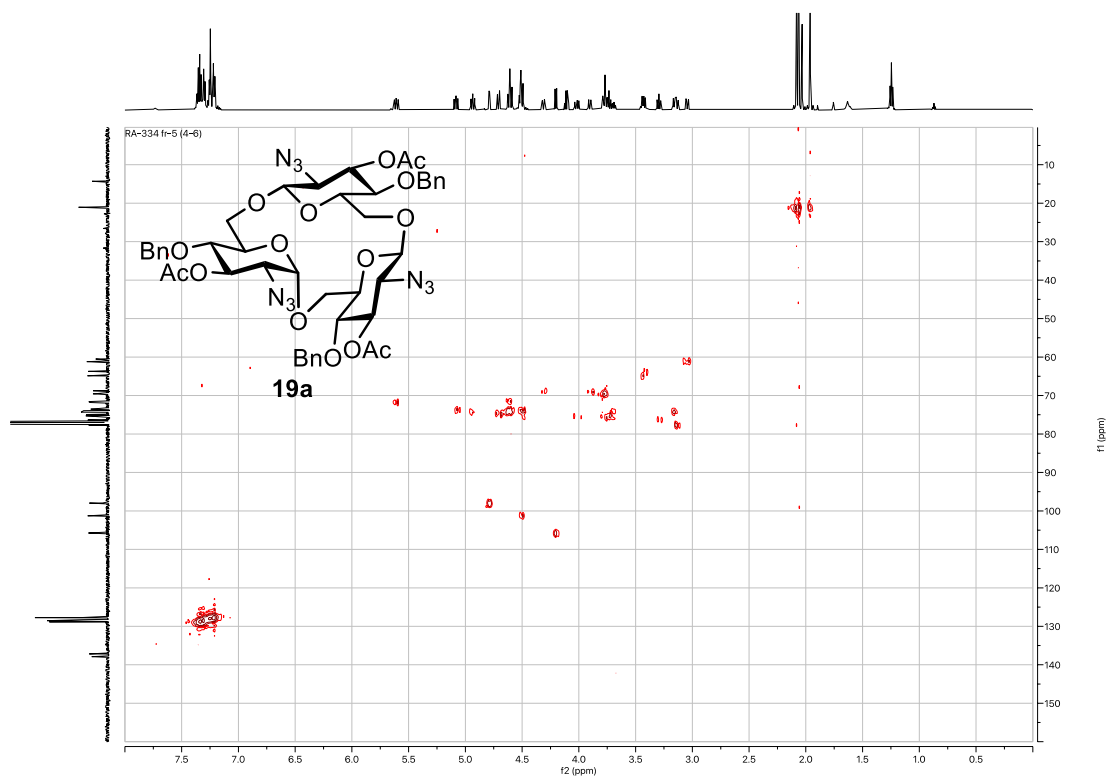


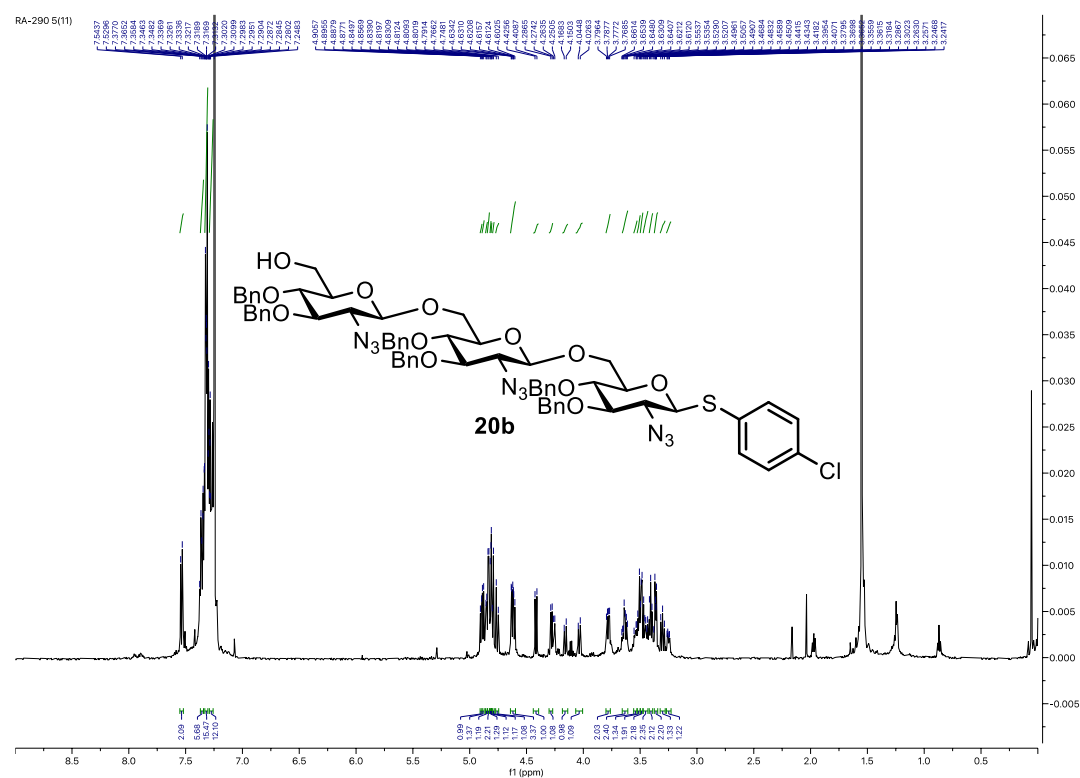
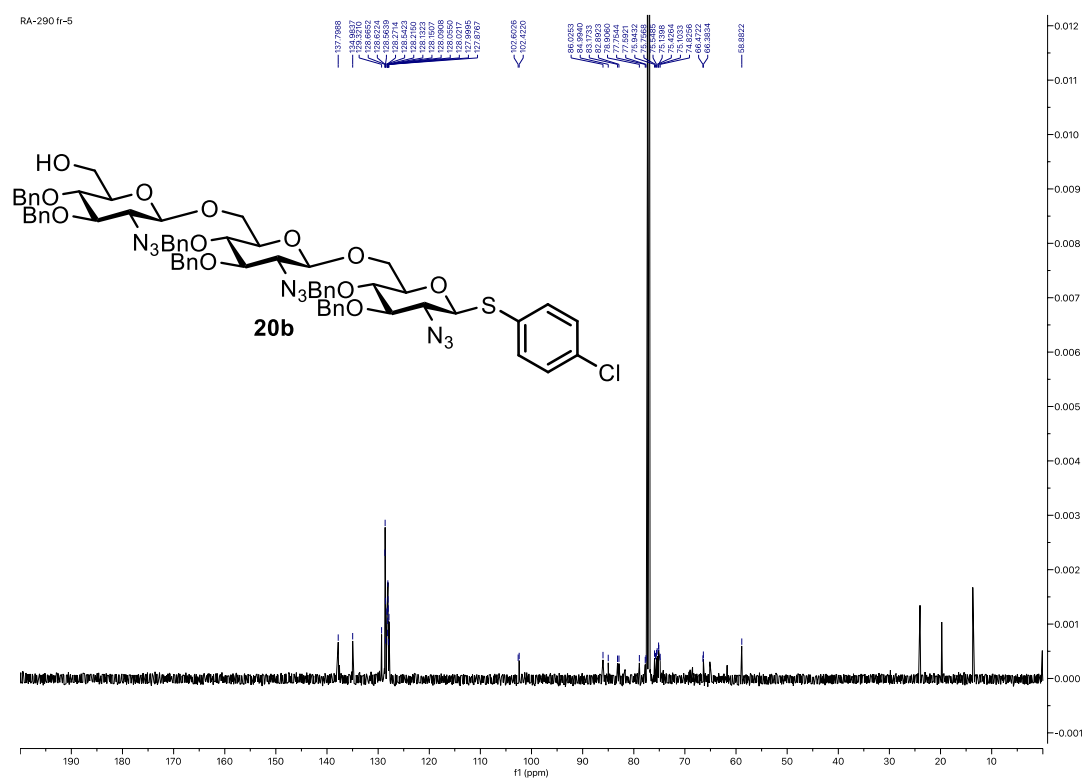
¹H NMR¹³C NMR

H-H cosy

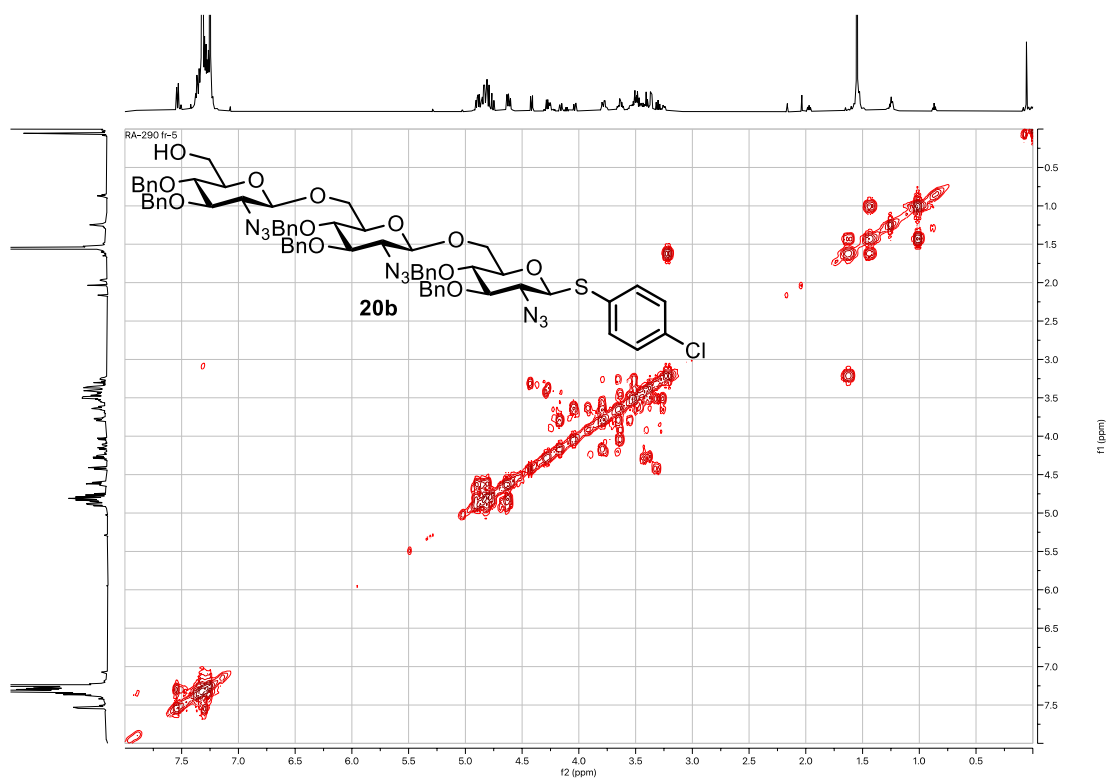


HMQC



¹H NMR¹³C NMR

H-H cosy



HMQC

