Electronic Supplementary Information

Synthesis of Cyclic α-1,4-Oligo-*N*-acetylglucosamine "Cyclokasaodorin" via One-pot Electrochemical Polyglycosylation-Isomerization-Cyclization Process

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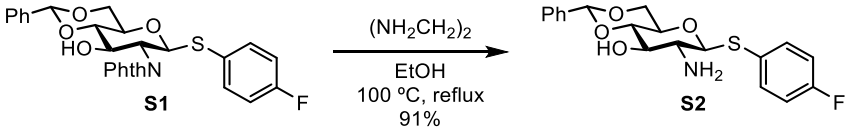
Contents

1. General

All reactions were carried out under argon atmosphere except notice. ^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker AVANCE II 600 (600 MHz for ^{1}H 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. Optical rotation data was recorded on JASCO DIP-370 digital polarimeter. Merck TLC (silica gel 60 F_{254}) was employed 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) and Sephadex LH-20 were used for Silica gel chromatography and gel filtration chromatography, respectively. Rotating-disk electrode voltammetry was carried out using BAS 700c analyzer and RRDE-3 rotating ring disk electrode. Measurements of oxidation potential of substrates (conc. 4.0 mM) were carried out in 0.1 M Bu_4NOTf/CH_2Cl_2 using a glassy carbon disk working electrode, a platinum wire counter electrode, and a saturated calomel electrode (SCE) as a reference electrode with sweep rate of 10 mV/s at 2000 r.p.m.. Compounds **S1** was synthesized according to the reported procedure.[1] Unless otherwise mentioned, all reagents were obtained from commercial suppliers and used without extra purification.

2. Preparation of building blocks

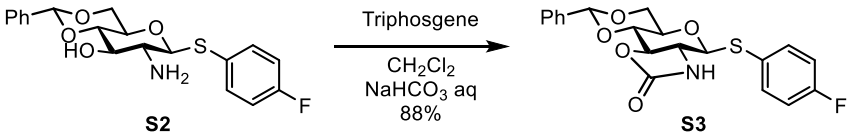
Preparation of 4-Fluorophenyl 2-amino-4,6-*O*-benzylidene-2-deoxy-1-thio-β-D-glucopyranoside (**S2**)[2]



Compound **S1**[1] (17.6 mmol, 8.94 g) was dissolved in EtOH (92.2 mL), and then ethylene diamine anhydrous (19.0 mL) was added to the solution. The temperature was raised slowly from room temperature (around 20 ℃) to 100 ℃. Then, the reflux was started and kept overnight. After completion of the reaction, the solvent was removed under reduced pressure. The crude product was purified with silica gel chromatography (CH_2Cl_2/MeOH 8:1) to afford **S2** (16.0 mmol, 6.03 g) as white solid in 91% yield. **4-Fluorophenyl 2-amino-4,6-*O*-benzylidene-2-deoxy-1-thio-β-D-glucopyranoside** (**S2**); TLC (CH_2Cl_2/MeOH 8:1): R_f 0.54. $[\alpha]_D$ = –50.2 (*c* = 1.1, $CHCl_3$, 22 ℃); ^{1}H NMR ($CDCl_3$, 600 MHz) δ 7.55–7.52 (m, 2 H, SPhF), 7.48–7.47 (m, 2 H, benzylidene-Ph), 7.39–7.35 (m, 3 H, benzylidene-Ph), 7.06–7.02 (m, 2 H, SPhF), 5.45 (s, 1 H, acetal-PhC*H*), 4.48 (d, *J* = 9.8 Hz, 1 H, H-1), 4.36 (dd, *J* = 10.6, 4.6 Hz, 1 H, H-6), 3.79 (*pseudo*-t, *J* = 9.6 Hz, 1 H, H-6), 3.65 (*pseudo*-t, *J* = 8.8 Hz, 1 H, H-3), 3.54-3.48 (m, 2 H, H-4 and H-5), 2.85 (s, 1 H, 4-O*H*), 2.76 (dd, *J* = 9.7, 9.2 Hz, 1 H, H-2) , 1.66 (s, 2 H, 2-NH_2); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 163.1 (d, *J* = 247.8 Hz), 137.1, 135.6 (d, *J* = 8.0 Hz), 129.4, 128.4, 126.6 (d, *J* = 3.2 Hz), 126.3, 116.2 (d, *J* =

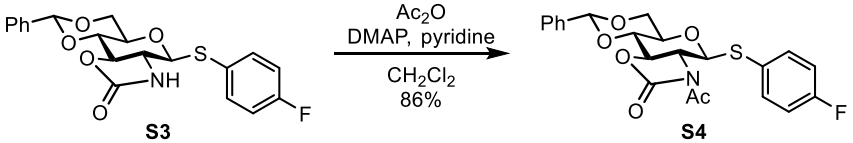
21.6 Hz), 101.9, 89.9, 80.1, 74.4, 70.5, 68.6, 56.3; HRMS (ESI) *m/z* calculated for $C_{19}H_{20}FKNO_4S$ $[M+K]^+$ 416.0729; found, 416.0730.

Preparation of 4-Fluorophenyl 4,6-*O*-benzylidene-2,3-*N,O*-carbonyl-2-deoxy-1-thio-β-D-glucopyranoside (**S3**)



Compound **S2** (16.0 mmol, 6.03 g) and triphosgene (5.33 mmol, 1.59 g) were dissolved in CH_2Cl_2 (262 mL), and then the mixture of saturated aqueous solution of $NaHCO_3$ (188 mL) was added to the solution. The reaction was kept stirring at room temperature for overnight. Then, the mixture was diluted by CH_2Cl_2 (200 mL), and the organic layer was washed with water (3 times) and brine, respectively. The organic layer was dried over Na_2SO_4, and the solvent was removed under reduced pressure. The crude product was purified by recrystallization to afford **S3** (14.1 mmol, 5.67 g) as a white solid in 88% yield. **4-Fluorophenyl 4,6-*O*-benzylidene-2,3-*N,O*-carbonyl-2-deoxy-1-thio-β-D-glucopyranoside** (**S3**); TLC (Hexane/EtOAc 1:1): R_f 0.60. $[\alpha]_D$ = –92.2 (*c* = 1.2, $CHCl_3$, 22 °C); ^{1}H NMR ($CDCl_3$, 600 MHz) δ 7.53–7.51 (m, 2 H, SPhF), 7.47–7.46 (m, 2 H, benzylidene-Ph), 7.38–7.36 (m, 3 H, benzylidene-Ph), 7.10–7.01 (m, 2 H, SPhF), 5.60 (s, 1 H, acetal-PhC*H*), 5.05 (s, 1 H, 1-N*H*), 4.79 (d, *J* = 9.8 Hz, 1 H, H-1), 4.39 (dd, *J* = 13.8, 10.5 Hz, 1 H, H-6), 4.38 (*pseudo*-t, *J* = 11.0 Hz, 1 H, H-3), 4.01 (dd, *J* = 10.0, 8.6 Hz, 1 H, H-4), 3.92 (*pseudo*-t, *J* = 10.4 Hz, 1 H, H-6), 3.63 (ddd, *J* = 10.1, 8.5, 4.7 Hz, 1 H, H-5), 3.46–3.43 (m, 1 H, H-2); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 163.5 (d, *J* = 249.2 Hz), 158.9, 136.6 (d, *J* = 8.5 Hz), 136.4, 129.4, 128.4, 126.2, 124.6 (d, *J* = 3.2 Hz), 116.5 (d, *J* = 21.8 Hz), 101.4, 85.2, 80.6, 78.6, 73.2, 68.3, 59.5; HRMS (ESI) *m/z* calculated for $C_{20}H_{18}FKNO_5S$ $[M+K]^+$ 442.0522; found, 442.0523.

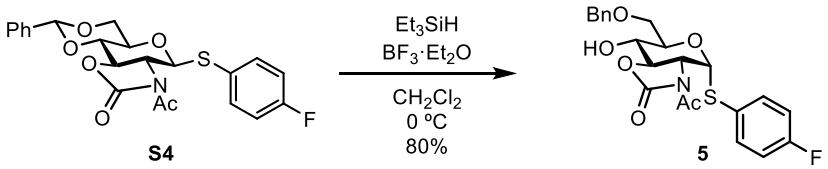
Preparation of 4-Fluorophenyl 2-acetamido-4,6-*O*-benzylidene-2,3-*N,O*-carbonyl-2-deoxy-1-thio-β-D-glucopyranoside (**S4**)



Compound **S3** (9.92 mmol, 4.00 g) and *N,N*-dimethylaminopyridine (1.90 mmol, 0.240 g) were dissolved in CH_2Cl_2 (29.5 mL). Then, pyridine (9.55 mL) and acetic anhydrous (95.3 mmol, 8.85 mL) were added to the mixture, and the reaction was kept stirring at room temperature for 3 h. The reaction was quenched by MeOH at 0 °C, and the solvent was removed under reduced pressure. The crude product was purified by recrystallization to afford the compounds **S4** (8.56 mmol, 3.81 g) as a white

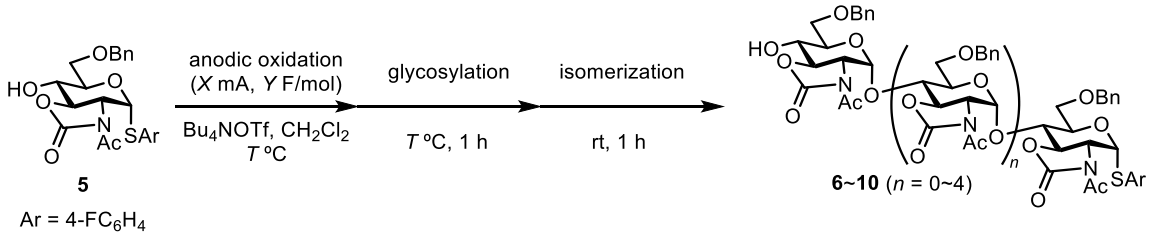
solid in 86% yield. **4-Fluorophenyl 2-acetamido-4,6-*O*-benzylidene-2,3-*N,O*-carbonyl-2-deoxy-1-thio-β-D-glucopyranoside** (**S4**); TLC (Hexane/EtOAc 1:1): R_f 0.76. $[\alpha]_D$ = –39.4 (*c* = 1.0, DMSO, 23 °C); ^{1}H NMR ($CDCl_3$, 600 MHz) δ 7.51–7.49 (m, 2 H, SPhF), 7.46–7.45 (m, 2 H, benzylidene-Ph), 7.37–7.36 (m, 3 H, benzylidene-Ph), 7.04–7.01 (m, 2 H, SPhF), 5.62 (s, 1 H, acetal-PhC*H*), 4.89 (d, *J* = 8.8 Hz, 1 H, H-1), 4.37 (*pseudo*-t, *J* = 10.8 Hz, 1 H, H-3), 4.27 (dd, *J* = 10.5, 4.7 Hz, 1 H, H-6), 4.13 (dd, *J* = 11.0, 8.8 Hz, 1 H, H-2), 4.09 (dd, *J* = 10.0, 8.6 Hz, 1 H, H-4), 3.94 (*pseudo*-t, *J* = 10.4 Hz, 1 H, H-6), 3.53 (ddd, *J* = 10.1, 8.7, 4.7 Hz, 1 H, H-5), 2.60 (s, 3 H, Ac); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 173.3, 163.1 (d, *J* = 247.3 Hz), 153.5, 136.2, 135.6 (d, *J* = 7.9 Hz), 129.4, 128.4, 128.2, 126.1, 116.1 (d, *J* = 21.5 Hz), 101.6, 88.6, 78.7, 78.3, 73.1, 68.2, 60.9, 24.7; HRMS (ESI) *m/z* calculated for $C_{22}H_{20}FKNO_6S$ $[M+K]^+$ 484.0627; found, 484.0619.

Preparation of 4-Fluorophenyl 2-acetamido-6-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy-1-thio-α-D-glucopyranoside (**5**)[3]



Compound **S4** (8.56 mmol, 3.81 g) was dissolved in CH_2Cl_2 (130 mL) at 0 °C. Then, triethylsilane (98.4 mmol, 15.7 mL) and $BF_3 \cdot Et_2O$ (12.3 mmol, 1.54 mL) were added to the mixture, and the reaction was kept stirring at 0 °C for 4 h. The reaction was quenched with saturated aqueous solution of $NaHCO_3$. The organic layer was washed with water (3 times). Then, the organic layer was dried over Na_2SO_4, and the solvent was removed under reduced pressure. The crude product was purified with silica gel chromatography (Hexane/EtOAc 2:1) to afford the compound **5** (6.86 mmol, 3.07 g) as a white solid in 80% yield. **4-Fluorophenyl 2-acetamido-6-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy-1-thio-α-D-glucopyranoside** (**5**); TLC (Hexane/EtOAc 2:1): R_f 0.25. $[\alpha]_D$ = 10.7 (*c* = 1.0, $CHCl_3$, 26 °C). E_{ox} = 1.74 V vs. SCE; ^{1}H NMR ($CDCl_3$, 600 MHz) δ 7.46–7.43 (m, 2 H, SPhF), 7.39–7.36 (m, 2 H, benzyl-Ph), 7.34–7.32 (m, 3 H, benzyl-Ph), 7.00–6.97 (m, 2 H, SPhF), 6.05 (d, *J* = 4.5 Hz, 1 H, H-1), 4.63 (d, *J* = 11.9 Hz, 1 H, PhCH_2), 4.55 (d, *J* = 11.9 Hz, 1 H, PhCH_2), 4.36 (dd, *J* = 12.1, 9.6 Hz, 1 H, H-3), 4.20–4.17 (m, 1 H, H-5), 4.11 (td, *J* = 8.9, 2.9 Hz, 1 H, H-4), 4.05 (dd, *J* = 12.1, 4.5 Hz, 1 H, H-2), 3.86 (dd, *J* = 10.3, 4.4 Hz, 1 H, H-6), 3.75 (dd, *J* = 10.3, 4.8 Hz, 1 H, H-6), 2.95 (d, *J* = 2.9 Hz, 1 H, 4-O*H*), 2.54 (s, 3 H, Ac); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 171.3, 163.0 (d, *J* = 247.2 Hz), 153.0, 137.3, 135.3 (d, *J* = 8.1 Hz), 128.6, 128.1, 127.8, 127.4 (d, *J* = 3.3 Hz), 116.4 (d, *J* = 21.9 Hz), 86.9, 78.2, 73.8, 72.6, 70.2, 69.3, 59.6, 23.8; HRMS (ESI) *m/z* calculated for $C_{22}H_{22}FKNO_6S$ $[M+K]^+$ 486.07834; found, 486.0781.

3. Synthesis of linear oligosaccharides[4,5]



The electrochemical polymerization synthesis of linear oligosaccharides (**6**~**10**) was carried out 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 **5** (0.600 mmol, 268 mg), Bu_4NOTf (1.00 mmol, 393 mg), and CH_2Cl_2 (10 mL) were added to the anodic chamber. Trifluoromethanesulfonic acid (0.600 mmol, 53 μL), Bu_4NOTf (1.00 mmol, 393 mg), and CH_2Cl_2 (10 mL) were added to the cathodic chamber. The constant current (8 mA (current density: 2.0 mA/cm^2), 35 V (electrode distance: 4.5 cm)) was employed at –40 °C with magnetic stirring until 0.6 F/mol of the electricity was consumed. After the electrolysis, the reaction was kept stirring at the same temperature for 1 h. After that, the temperature was raised to room temperature for 1 h as isomerization, and triethylamine (0.5 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 water (3 times) and brine, respectively. The solution was dried over Na_2SO_4, and the solvent was removed under reduced pressure. The crude product was purified with preparative-GPC to afford linear oligosaccharides **6** (*n* = 0, 0.0626 mmol, 48.0 mg, 21%), **7** (*n* = 1, 0.0383 mmol, 41.6 mg, 19%), **8** (*n* = 2, 0.0184 mmol, 25.9 mg, 12%), **9** (*n* = 3, 0.0109 mmol, 18.8 mg, 9.1%), and **10** (*n* = 4, 0.00690 mmol, 14.1 mg, 6.9%) as white solids.

4-Fluorophenyl (2-acetamido-6-*O*-benzyl-2,3-*N*,*O*-carbonyl-2-deoxy-α-D-glucopyranosyl)-(1→4)-2-acetamido-6-*O*-benzyl-2,3-*N*,*O*-carbonyl-2-deoxy-1-thio-α-D-gluco-pyranoside (**6**); TLC (Hexane:EtOAc 1:1): R_f 0.66. $[\alpha]_D$ = 164.4 (*c* = 1.1, $CHCl_3$, 27 °C). E_{ox} = 1.68 V vs. SCE; ^{1}H NMR ($CDCl_3$, 600 MHz) δ 7.46–7.42 (m, 2 H), 7.36–7.31 (m, 5 H), 7.29–7.27 (m, 5 H), 7.01–6.97 (m, 2 H), 6.08 (d, *J* = 4.4 Hz, 1 H), 6.03 (d, *J* = 2.7 Hz, 1 H), 4.55 (d, *J* = 11.9 Hz, 1 H), 4.53 (d, *J* = 11.9 Hz, 1 H), 4.52 (d, *J* = 11.9 Hz, 1 H), 4.48 (dd, *J* = 12.1, 10.0 Hz, 1 H), 4.46 (d, *J* = 11.9 Hz, 1 H), 4.37–4.31 (m, 2 H), 4.20 (d, *J* = 8.6 Hz, 1 H), 4.10 (td, *J* = 10.0, 8.9, 2.8 Hz, 1 H), 4.05 (dd, *J* = 11.5, 4.5 Hz, 1 H), 3.88 (dd, *J* = 11.2, 3.7 Hz, 1 H), 3.76–3.73 (m, 2 H), 3.71 (dd, *J* = 11.5, 1.4 Hz, 1 H), 3.64 (dd, *J* = 10.1, 4.3 Hz, 1 H), 3.56 (dd, *J* = 10.1, 5.2 Hz, 1 H), 2.89 (d, *J* = 2.7 Hz, 1 H), 2.532 (s, 3 H), 2.529 (s, 3 H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 171.7, 171.2, 163.1 (d, *J* = 247.6 Hz), 153.2, 152.6, 137.8, 137.4, 135.3 (d, *J* = 8.0 Hz), 128.6, 128.4, 128.0, 127.8, 127.7, 127.6, 127.3 (d, *J* = 3.3 Hz), 116.4 (d, *J* = 21.9 Hz), 95.8, 86.7, 77.5, 76.4, 73.8, 73.54, 73.48, 72.6, 69.7, 69.2, 68.0, 59.8, 59.6, 23.8, 23.7; HRMS (ESI) *m/z* calculated for $C_{38}H_{39}FKN_2O_{12}S$ $[M+K]^+$ 805.1840; found, 805.1835.

4-Fluorophenyl (2-acetamido-6-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy-α-D-gluco-pyranosyl)-(1→4)-(2-acetamido-6-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy-α-D-glucopyranosyl)-(1→4)-2-acetamido-6-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy-1-thio-α-D-glucopyranoside (**7**); TLC (Hexane:EtOAc 1:1): R_f 0.57. $[\alpha]_D$ = 141.4 (*c* = 1.3, $CHCl_3$, 27 °C); ^1H NMR ($CDCl_3$, 600 MHz) δ 7.45–7.42 (m, 2 H), 7.36–7.33 (m, 2 H), 7.32–7.27 (m, 11 H), 7.26–7.24 (m, 2 H), 7.00–6.97 (m, 2 H), 6.07 (d, *J* = 4.4 Hz, 1 H), 6.023 (d, *J* = 3.1 Hz, 1 H), 6.018 (d, *J* = 3.1 Hz, 1 H), 4.525 (d, *J* = 11.8 Hz, 1 H), 4.521 (d, *J* = 11.9 Hz, 1 H), 4.50 (dd, *J* = 11.9, 10.1 Hz, 1 H), 4.49–4.48 (m, 2 H), 4.47 (d, *J* = 11.8 Hz, 1 H), 4.46 (d, *J* = 11.9 Hz, 1 H), 4.42 (dd, *J* = 12.1, 10.0 Hz, 1 H), 4.37 (dd, *J* = 8.5, 6.5 Hz, 1 H), 4.34 (*pseudo*-t, *J* = 6.0 Hz, 1 H), 4.30 (*pseudo*-t, *J* = 9.1 Hz, 1 H), 4.19 (d, *J* = 9.0 Hz, 1 H), 4.10 (*pseudo*-t, *J* = 9.2 Hz, 1 H), 4.04 (dd, *J* = 11.9, 4.5 Hz, 1 H), 3.85 (dd, *J* = 11.2, 3.6 Hz, 1 H), 3.78 (d, *J* = 7.3 Hz, 1 H), 3.76 (dd, *J* = 10.3, 2.3 Hz, 1 H), 3.74 (dd, *J* = 10.3, 2.8 Hz, 1 H), 3.71 (dd. *J* = 11.0, 1.3 Hz, 1 H), 3.68–3.65 (m, 3 H), 3.54–3.51 (m, 2 H), 3.00–2.95 (m, 1 H), 2.53 (s, 3 H), 2.52 (s, 3 H), 2.51 (s, 3 H).; ^{13}C NMR ($CDCl_3$, 150 MHz) δ 171.7, 171.4, 171.1, 163.1 (d, *J* = 247.3 Hz), 153.0, 152.54, 152.46, 137.7, 137.6, 137.2, 135.2 (d, *J* = 8.0 Hz), 128.6, 128.45, 128.43, 128.1, 127.5, 127.82, 127.79, 127.4, 127.2 (d, *J* = 3.2 Hz), 116.4 (d, *J* = 21.9 Hz), 95.7, 95.5, 86.8, 77.2, 76.2, 75.5, 74.4, 73.8, 73.5, 73.2, 73.1, 73.0, 72.6, 70.2, 69.4, 68.0, 67.6, 59.9, 59.8, 59.6, 23.8, 23.70, 23.67; HRMS (ESI) *m/z* calculated for $C_{54}H_{56}FN_3NaO_{18}S$ $[M+Na]^+$, 1108.3156; found, 1108.3204.

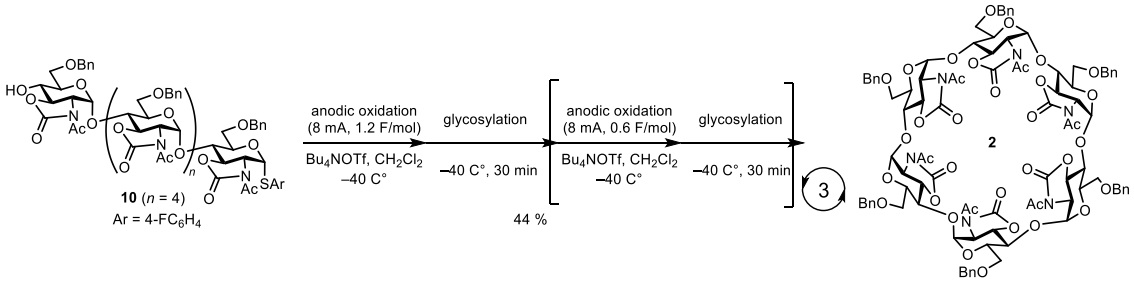
4-Fluorophenyl (2-acetamido-6-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy-α-D-glucopyranosyl)-(1→4)-(2-acetamido-6-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy-α-D-glucopyranosyl)-(1→4)-(2-acetamido-6-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy-α-D-glucopyranosyl)-(1→4)-2-acetamido-6-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy-1-thio-α-D-glucopyranoside (**8**); TLC (Hexane:EtOAc 1:1): R_f 0.53. $[\alpha]_D$ = 138.2 (*c* = 1.0, $CHCl_3$, 27 °C); ^1H NMR ($CDCl_3$, 600 MHz) δ 7.45–7.41 (m, 2 H), 7.35–7.27 (m, 15 H), 7.25–7.23 (m, 5 H), 7.00–6.96 (m, 2 H), 6.07 (d, *J* = 4.4 Hz, 1 H), 6.02 (d, *J* = 3.4 Hz, 1 H), 6.01 (d, *J* = 3.1 Hz, 1 H), 6.00 (d, *J* = 2.7 Hz, 1 H), 4.55–4.43 (m, 10 H), 4.42 (dd, *J* = 12.1, 10.0 Hz, 1 H), 4.37 (dd, *J* = 10.7, 8.8 Hz, 1 H), 4.34 (*pseudo*-t, *J* = 9.1 Hz, 1 H), 4.31 (*pseudo*-t, *J* = 9.5 Hz, 1 H), 4.29 (*pseudo*-t, *J* = 9.1 Hz, 1 H), 4.19 (d, *J* = 8.9 Hz, 1 H), 4.09 (*pseudo*-t, *J* = 8.8 Hz, 1 H), 4.03 (dd, *J* = 12.0, 4.5 Hz, 1 H), 3.84 (d, *J* = 11.2, 3.6 Hz, 1 H), 3.78 (d, *J* = 9.1 Hz, 1 H), 3.75 (dd, *J* = 12.0, 2.7 Hz, 2 H), 3.74 (dd, *J* = 12.0, 2.8 Hz, 1 H), 3.72–3.67 (m, 2 H), 3.67–3.58 (m, 2 H), 3.62 (*pseudo*-t, *J* = 10.1 Hz, 1 H), 3.59 (dd, *J* = 11.9, 3.1 Hz, 1 H), 3.54 (d, *J* = 11.6 Hz, 1 H), 3.53 (dd, *J* = 9.8, 4.8 Hz, 1 H), 3.48 (d, *J* = 10.9 Hz, 1 H), 3.07 (d, *J* = 1.4 Hz, 1 H), 2.52 (s, 3 H), 2.51 (s, 6 H), 2.50 (s, 3 H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 171.7, 171.5, 171.4, 171.1, 163.1 (d, *J* = 247.9 Hz), 153.1, 152.6, 152.50, 152.46, 137.66, 137.65, 137.60, 135.2 (d, *J* = 8.3 Hz), 128.6, 128.5, 128.44, 128.43, 128.1, 127.9, 127.82, 127.77, 127.5, 127.4, 127.3, 127.2 (d, *J* = 3.2 Hz), 116.4 (d, *J* = 21.9 Hz), 95.9, 95.57, 95.55, 86.7, 77.1, 76.2, 75.5, 75.2, 74.6, 73.9, 73.8, 73.6, 73.52, 73.49, 73.3, 73.1, 73.0, 72.6, 70.1, 69.3, 67.9, 67.7, 67.5, 59.9, 59.8, 59.6, 23.8, 23.69, 23.67, 23.6; HRMS (ESI) *m/z* calculated for $C_{70}H_{73}FKN_4NaO_{24}S$ $[M+Na]^+$, 1427.4212; found, 1427.4205.

4-Fluorophenyl (2-acetamido-6-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy-α-D-glucopyranosyl)-(1→4)-(2-acetamido-6-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy-α-D-glucopyranosyl)-(1→4)-(2-acetamido-6-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy-α-D-glucopyranosyl)-(1→4)-(2-acetamido-6-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy-α-D-glucopyranosyl)-(1→4)-2-acetamido-6-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy-1-thio-α-D-glucopyranoside (**9**); TLC (Hexane:EtOAc 1:1): R_f 0.43. $[\alpha]_D$ = 149.2 (*c* = 1.0, $CHCl_3$, 28 °C); ^{1}H NMR ($CDCl_3$, 600 MHz) δ 7.45–7.41 (m, 2 H), 7.36–7.31 (m, 5 H), 7.30–7.26 (m, 12 H), 7.25–7.22 (m, 8 H), 6.99–6.96 (m, 2 H), 6.07 (d, *J* = 4.4 Hz, 1 H), 6.03 (d, *J* = 2.8 Hz, 1 H), 6.02 (d, *J* = 2.7 Hz, 2 H), 6.00 (d, *J* = 2.6 Hz, 1 H), 4.54 (d, *J* = 11.6 Hz, 1 H), 4.52-4.42 (m, 12 H), 4.41 (dd, *J* = 12.1, 10.1 Hz, 1 H), 4.37 (dd, *J* = 11.9, 10.0 Hz, 1 H), 4.34 (*pseudo*-t, *J* = 9.9 Hz, 1 H), 4.32 (*pseudo*-t, *J* = 9.6 Hz, 1 H), 4.31 (*pseudo*-t, *J* = 4.31 Hz, 1 H), 4.28 (*pseudo*-t, *J* = 9.2 Hz, 1 H), 4.18 (d, *J* = 8.9 Hz, 1 H), 4.09 (*pseudo*-t, *J* = 9.3 Hz, 1 H), 4.03 (dd, *J* = 12.0, 4.4 Hz, 1 H), 3.84 (dd, *J* = 11.1, 3.5 Hz, 1 H), 3.79 (d, *J* = 9.0 Hz, 1 H), 3.75 (dd, *J* = 12.0, 3.1 Hz, 2 H), 3.74 (dd, *J* = 12.0, 3.2 Hz, 2 H), 3.70 (d, *J* = 11.0 Hz, 2 H), 3.68 (d, *J* = 11.2 Hz, 1 H), 3.66–3.60 (m, 3 H), 3.58 (dd, *J* = 11.2, 3.1 Hz, 1 H), 3.57 (dd, *J* = 11.0, 2.9 Hz, 1 H), 3.55–3.45 (m, 4 H), 3.03 (d, *J* = 1.9 Hz, 1 H), 2.52 (s, 3 H), 2.505 (s, 3 H), 2.496 (s, 3 H), 2.49 (s, 3 H), 2.48 (s, 3 H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 171.7, 171.54, 171.45, 171.4, 171.1, 163.1 (d, *J* = 248.0 Hz), 153.0, 152.6, 152.49, 152.46, 137.67, 137.65, 137.60, 137.58, 137.2, 135.2 (d, *J* = 8.4 Hz), 128.6, 128.5, 128.4, 128.1, 127.92, 127.90, 127.82, 127.78, 127.5, 127.4, 127.35, 127.29, 127.2 (d, *J* = 3.2 Hz), 116.4 (d, *J* = 21.9 Hz), 96.0, 95.5, 95.4, 86.7, 76.2, 75.6, 75.3, 75.1, 74.8, 74.0, 73.8, 73.61, 73.55, 73.52, 73.49, 73.2, 73.01, 72.97, 72.6, 70.2, 69.3, 67.9, 67.7, 67.6, 67.5, 60.0, 59.9, 59.8, 59.6, 23.8, 23.68, 23.66, 23.6; HRMS (ESI) *m/z* calculated for $C_{86}H_{90}FKN_5O_{30}S$ $[M+K]^+$, 1762.5007; found, 1762.5010.

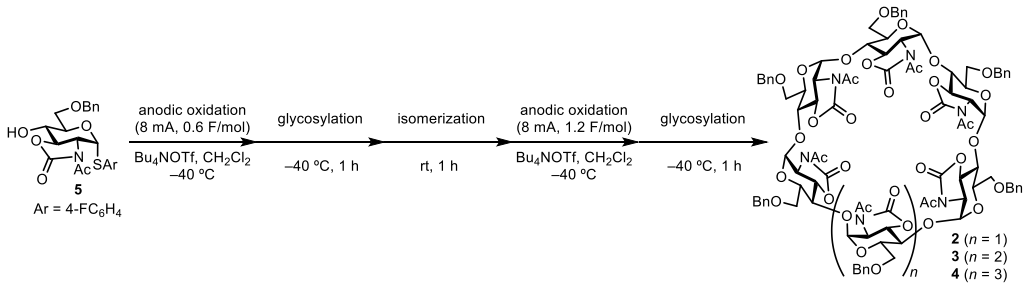
4-Fluorophenyl (2-acetamido-6-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy-α-D-glucopyranosyl)-(1→4)-(2-acetamido-6-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy-α-D-glucopyranosyl)-(1→4)-(2-acetamido-6-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy-α-D-glucopyranosyl)-(1→4)-(2-acetamido-6-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy-α-D-glucopyranosyl)-(1→4)-2-acetamido-6-*O*-benzyl-2,3-*N,O*-carbonyl-2-deoxy-1-thio-α-D-glucopyranoside (**10**); TLC (Hexane:EtOAc 1:1): R_f 0.40. $[\alpha]_D$ = 100.6 (*c* = 0.64, $CHCl_3$, 24 °C); ^{1}H NMR ($CDCl_3$, 600 MHz) δ 7.44–7.42 (m, 2 H), 7.35–7.32 (m, 5 H), 7.31–7.28 (m, 16 H), 7.25–7.22 (m, 9 H), 6.99–6.96 (m, 2 H), 6.07 (d, *J* = 4.4 Hz, 1 H), 6.032 (d, *J* = 3.1 Hz, 1 H), 6.027 (d, *J* = 3.2 Hz, 1 H), 6.02–6.01 (m, 2 H), 6.00 (d, *J* = 2.6 Hz, 1 H), 4.52–4.27 (m, 23 H), 4.18 (d, *J* = 8.7 Hz, 1 H), 4.09 (*pseudo*-t, *J* = 9.0 Hz, 1 H), 4.04 (dd, *J* = 12.1, 4.5 Hz, 1 H), 3.83 (dd, *J* = 11.1, 3.5 Hz, 1 H), 3.80-3.78 (m, 1 H), 3.75–3.45 (m, 20 H), 2.98-2.93 (m, 1 H), 2.52 (s, 3 H), 2.50 (s, 3 H), 2.495-2.490 (m, 6 H), 2.48 (s, 3 H), 2.47 (s, 3 H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 171.7, 171.54, 171.47, 171.4, 171.1, 163.0 (d, *J* = 247 Hz), 153.0, 152.54, 152.48, 137.7, 137.62, 137.58, 137.55, 137.0, 135.2 (d, *J* = 8.5 Hz), 128.6, 128.5, 128.4, 128.1, 127.9, 127.8, 127.5, 127.42, 127.37, 127.32, 127.27, 116.4 (d, *J* = 21.9 Hz), 95.9, 95.6, 95.34, 95.30, 86.7, 76.2, 75.6, 75.3, 75.14, 75.06, 74.7, 74.1,

73.8, 73.6, 73.51, 73.48, 73.4, 73.2, 73.00, 72.96, 72.91, 72.86, 72.6, 70.2, 69.3, 67.9, 67.7, 67.6, 67.5, 60.0, 59.9, 59.84, 59.81, 59.6, 23.8, 23.7; HRMS (ESI) *m/z* calculated for $C_{102}H_{107}FKN_6O_{36}S$ $[M+K]^+$, 2082.6097; found, 2082.6018.

4. Synthesis of cyclic oligosaccharides[6]



The electrochemical synthesis of Cyclohexakis-(1→4)-(2-acetamido-3-*O*-acetyl-6-*O*-benzyl-2,3-*N*,*O*-carbonyl-2-deoxy-α-D-glucopyranosyl) **2** was carried out 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). Linear hexasaccharide **10** (0.0116 mmol, 23.8 mg) in CH_2Cl_2 (1.0 mL), Bu_4NOTf (1.00 mmol, 393 mg), and CH_2Cl_2 (9.0 mL) were added to the anodic chamber. Trifluoromethanesulfonic acid (1.00 mmol, 88 μL), Bu_4NOTf (1.00 mmol, 393 mg), and CH_2Cl_2 (10 mL) were added to the cathodic chamber. The constant current (8.0 mA (current density: 2.0 mA/cm^2), 35 V (electrode distance: 4.5 cm)) was employed at –40 ℃ with magnetic stirring until the starting material consumed (3.0 F/mol of electricity). After the electrolysis, the reaction was kept stirring at the same temperature for 30 minutes. After that, triethylamine (0.50 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 water (3 times) and brine, respectively. The solution was dried over Na_2SO_4, and the solvent was removed under reduced pressure. The crude product was purified with preparative-GPC to afford **2** (0.00517 mmol, 9.90 mg) as a white solid in 44% yield. **Clclohexakis-(1→4)-(2-acetamido-3-*O*-acetyl-6-*O*-benzyl-2,3-*N*,*O*-carbonyl-2-deoxy-α-D-gluco-pyranosyl)** (**2**); TLC (Hexane/EtOAc 1:1): R_f 0.31. $[\alpha]_D$ = 60.4 (*c* = 1.0, $CHCl_3$, 25 ℃); ^{1}H NMR ($CDCl_3$, 600 MHz) δ 7.33–7.29 (m, 4 H), 7.27 (d, *J* = 1.6 Hz, 1 H), 5.80 (d, *J* = 2.3 Hz, 1 H), 4.54 (d, *J* = 11.9 Hz, 1 H), 4.51 (dd, *J* = 12.1, 10.3 Hz, 1 H), 4.43 (d, *J* = 11.9 Hz, 1 H), 4.17 (*pseudo*-t, *J* = 9.1 Hz, 1 H), 3.80 (dd, *J* = 10.6, 4.4 Hz, 1 H), 3.76 (dd, *J* = 8.8, 4.4 Hz, 1 H), 3.70 (dd, *J* = 12.2, 2.3 Hz, 1 H), 3.63 (d, *J* = 10.0 Hz, 1 H), 2.53 (s, 3 H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 172.1, 152.3, 137.4, 128.5, 128.0, 127.8, 98.0, 77.7, 74.8, 74.4, 73.7, 68.0, 59.8, 23.5; HRMS (ESI) *m/z* calculated for $C_{96}H_{102}N_6NaO_{36}$ $[M+Na]^+$, 1938.6261; found, 1938.6130.

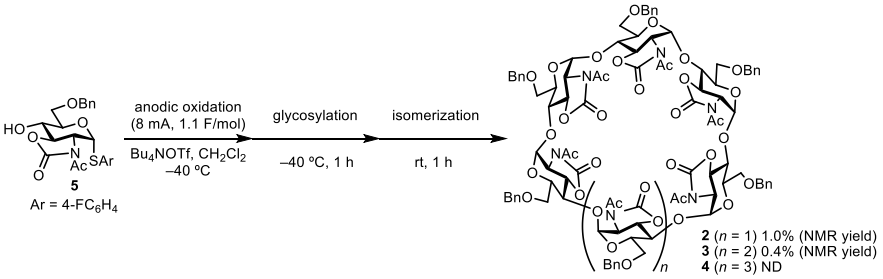


The electrochemical one-pot synthesis of cyclic oligosaccharides (**2**, **3**, **4**), was carried out an H-type divided cell (4G glass filter). The cell had a carbon felt anode (Nippon Carbon JF-20-P7) and platinum square plate (20 mm×20 mm). Building block **5** (0.600 mmol, 268 mg), Bu_4NOTf (1.00 mmol, 393 mg), and CH_2Cl_2 (10 mL) were added to the anodic chamber. Trifluoromethanesulfonic acid (1.60 mmol, 141 μL), Bu_4NOTf (1.00 mmol, 393 mg), and CH_2Cl_2 (10 mL) were added to the cathodic chamber. As the first electrochemical polygycosylation, the constant current (8.0 mA (current density: 2.0 mA/cm^2), 36 V (electrode distance: 4.5 cm)) was employed at –40 ℃ with magnetic stirring until 0.6 F/mol of electricity was consumed. After the electrolysis, the reaction was kept stirring at the same temperature for 1 h. After that, the temperature was raised to room temperature for 1 h as isomerization. Then, as the final electrochemical cyclization, electrolysis with the constant current ((8.0 mA (current density: 2.0 mA/cm^2), 36 V (electrode distance: 4.5 cm)) was repeated at –40 ℃ with magnetic stirring until 1.2 F/mol of electricity was consumed. After that, triethylamine (1.0 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 water (3 times) and brine, respectively. The solution was dried over Na_2SO_4, and the solvent was removed under reduced pressure. The crude product was purified with silica gel chromatography (Hexane/EtOAc 1:1) and preparative-GPC to afford cyclic oligosaccharides **2** (*n* = 1, 0.00621 mmol, 11.9 mg, 6.2%), **3** (*n* = 2, 0.00474 mmol, 10.6 mg, 5.5%), and **4** (*n* = 3, trace) as white solids.

Clcloheptakis-(1→4)-(2-acetamido-3-*O*-acetyl-6-*O*-benzyl-2,3-*N*,*O*-carbonyl-2-deoxy-α-D-glucopyranosyl) (**3**); TLC (Hexane/EtOAc 1:1): R_f 0.21. $[\alpha]_D$ = 40.0 (*c* = 0.36, $CHCl_3$, 22 ℃); ^{1}H NMR ($CDCl_3$, 600 Mhz) δ 7.30–7.26 (m, 2 H), 7.25–7.21 (m, 3 H), 5.79 (d, *J* = 2.5 Hz, 1 H), 4.56 (d, *J* = 12.1 Hz, 1 H), 4.37 (d, *J* =12.2 Hz, 1 H), 4.34 (dd, *J* = 11.9, 10.5 Hz, 1 H), 4.14 (*pseudo*-t, *J* = 9.5 Hz, 1 H), 3.73 (dd, *J* = 10.9, 4.1 Hz, 1 H), 3.69 (dd, *J* = 12.1 Hz, 2.5 Hz, 1 H), 3.58 (dd, *J* = 8.7, 3.4 Hz, 1 H), 3.54 (d, *J* = 10.3 Hz, 1 H), 2.54 (s, 3 H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 172.3, 152.4, 137.6, 128.4, 127.8, 127.5, 98.9, 78.5, 74.5, 74.3, 73.3, 67.6, 60.0, 23.6; HRMS (ESI) *m/z* calculated for $C_{112}H_{119}N_7NaO_{42}$ $[M+Na]^+$, 2257.7317; found, 2257.7339.

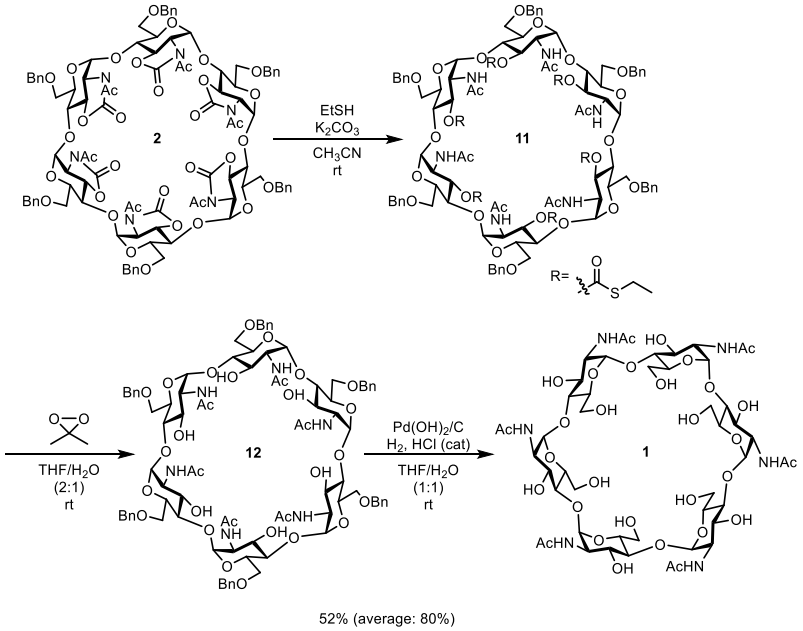
Clclooctakis-(1→4)-(2-acetamido-3-*O*-acetyl-6-*O*-benzyl-2,3-*N*,*O*-carbonyl-2-deoxy-α-D-glucopyranosyl) (**4**); TLC (Hexane/EtOAc 1:1): R_f 0.14. $[\alpha]_D$ = 40.6 (*c* = 0.33, $CHCl_3$, 25 ℃); ^{1}H NMR ($CDCl_3$, 600 MHz) δ 7.26-7.22 (m, 5 H), 5.83-5.82 (m, 1 H), 4.55 (d, *J* = 12.0 Hz, 1 H), 4.42 (*pseudo*-t, *J* = 11.4 Hz, 1 H), 4.38 (d, *J* = 12.0 Hz, 1 H), 4.15 (*pseudo*-t, *J* = 9.6 Hz, 1 H), 3.72-3.62 (m, 3 H),

3.55 (d, J = 9.6 Hz, 1 H), 2.52 (s, 3 H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 171.3, 151.4, 136.6, 127.4, 126.8, 126.5, 97.6, 77.2, 73.4, 73.1, 72.3, 66.7, 59.2, 22.6; HRMS (ESI) *m/z* calculated for $C_{128}H_{136}FKN_8NaO_{48}$ $[M+K]^+$, 2576.8373; found, 2576.8440.



The electrochemical one-pot synthesis of cyclic oligosaccharides (**2**, **3**, **4**) without the second anodic oxidation and glycosylation, was carried out 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 **5** (0.600 mmol, 268 mg), Bu_4NOTf (1.00 mmol, 393 mg), and CH_2Cl_2 (10 mL) were added to the anodic chamber. Trifluoromethanesulfonic acid (1.20 mmol, 106 μL), Bu_4NOTf (1.00 mmol, 393 mg), and CH_2Cl_2 (10 mL) were added to the cathodic chamber. The constant current (8.0 mA (current density: 2.0 mA/cm^2), 26 V (electrode distance: 4.5 cm)) was employed at –40 °C with magnetic stirring until 1.1 F/mol of electricity was consumed. After the electrolysis, the reaction was kept stirring at the same temperature for 1 h. After that, the temperature was raised to room temperature for 1 h as isomerization. After that, triethylamine (0.5 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 water (3 times) and brine, respectively. The solution was dried over Na_2SO_4, and the solvent was removed under reduced pressure. The crude product was purified with preparative-GPC. The yields of cyclic oligosaccharide **2** (n = 1, 0.00605 mmol, 1.0%) and **3** (n = 2, 0.00260 mmol, 0.4%) were decided by ^{1}H NMR with 1,1,2,2-tetrachloroethane as an internal standard.

5. Deprotection of cyclic hexasaccharide[7,8]



Cyclic hexasaccharide **2** (0.0157 mmol, 30.0 mg) and K_2CO_3 (0.141 mmol, 19.5 mg) were dissolved in CH_3CN (3.5 mL). Then, ethanethiol (1.77 mmol, 129 μL) was added dropwise to the solution. The reaction was kept stirring until **2** was completely consumed. The solution was diluted with EtOAc and washed with saturated aqueous solution of NH_4Cl, water, and brine by sequence. The organic solvent was removed under reduce pressure. Then, tetrahydrofuran (5.2 mL) and H_2O (2.6 mL) were added to the mixture. After dissolving, dimethyldioxirane in acetone (15 mL (87 mM: detected by *trans*-stilbene)) was put in the solution. After the reaction, the solvent was removed by freeze-drying. The dried mixture was dissolved in tetrahydrofuran (1.6 mL) and H_2O (1.6 mL). To the solution, $Pd(OH)_2/C$ (111 mg) and 35% aqueous HCl (1 drop as catalyst) were put in. The reaction was carried out under hydrogen atmosphere for 2 days. The residue was purified by size exclusion column chromatography on Sephadex LH-20 eluted with water to afford **1** (0.00821 mmol, 10.0 mg) in 52% yield as a white solid. **Cyclohexakis-(1→4)-(2-acetamido-2-deoxy-α-D-glucopyranosyl)** (**1**);
$[\alpha]_D$ = 30.3 (*c* = 1.1, H_2O, 20 °C); ^1H NMR (D_2O, 600 MHz) δ 4.93 (d, *J* = 3.0 Hz, 1 H), 3.91 (dd, *J* = 10.8, 3.0 Hz, 1 H), 3.87–3.82 (m, 3 H), 3.79–3.77 (m, 1 H), 3.72 (d, *J* = 9.0 Hz, 1 H), 3.62 (*pseudo*-t, *J* = 9.6 Hz, 1 H), 1.95–1.92 (m, 3 H); ^{13}C NMR (D_2O, 150 MHz, with acetone as internal standard) δ 174.3, 100.8, 81.1, 72.4, 71.7, 60.2, 54.6, 22.1; HRMS (MALDI-TOF MS, 2,5-dihyroxy benzoic acid as matrix) *m/z* calculated for $C_{48}H_{78}N_6NaO_{30}$ $[M+Na]^+$, 1241.465; found, 1241.470.

6. Reference

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3 S. Manabe, K. Ishii and Y. Ito, *J. Am. Chem. Soc.*, 2006, **128**, 10666.

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5 K. Yano, T. Itoh and T. Nokami, *Carbohydr. Res.*, 2020, **492**, 108018.

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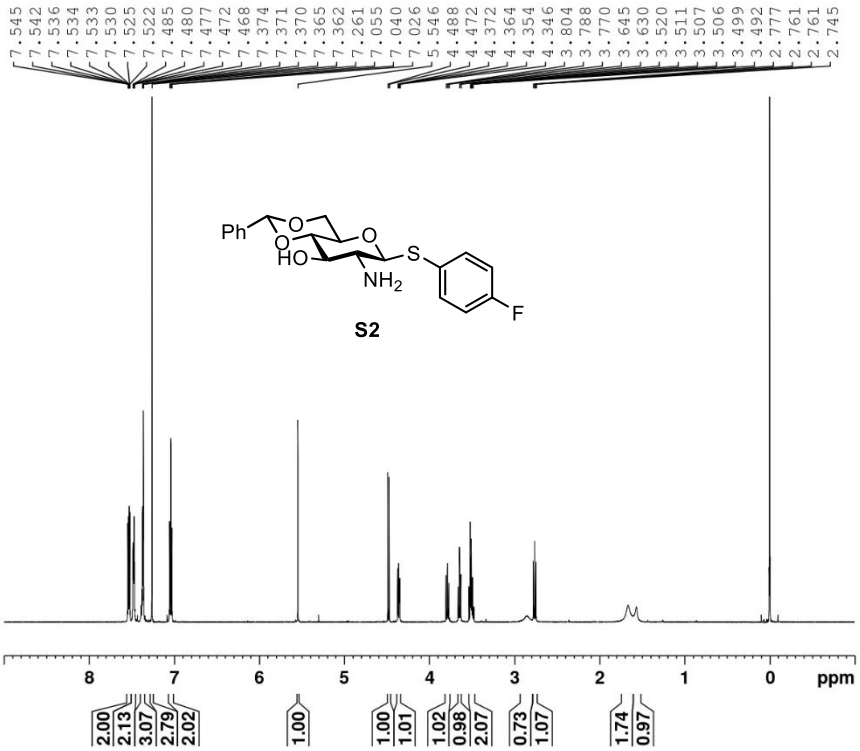
7. ^{1}H and ^{13}C NMR spectra of building blocks

^{1}H NMR

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Current Data Parameters
NAME                 end-113
EXPNO                     10
PROCNO                     1

F2 - Acquisition Parameters
Date_               20210318
Time                    9.13
INSTRUM                spect
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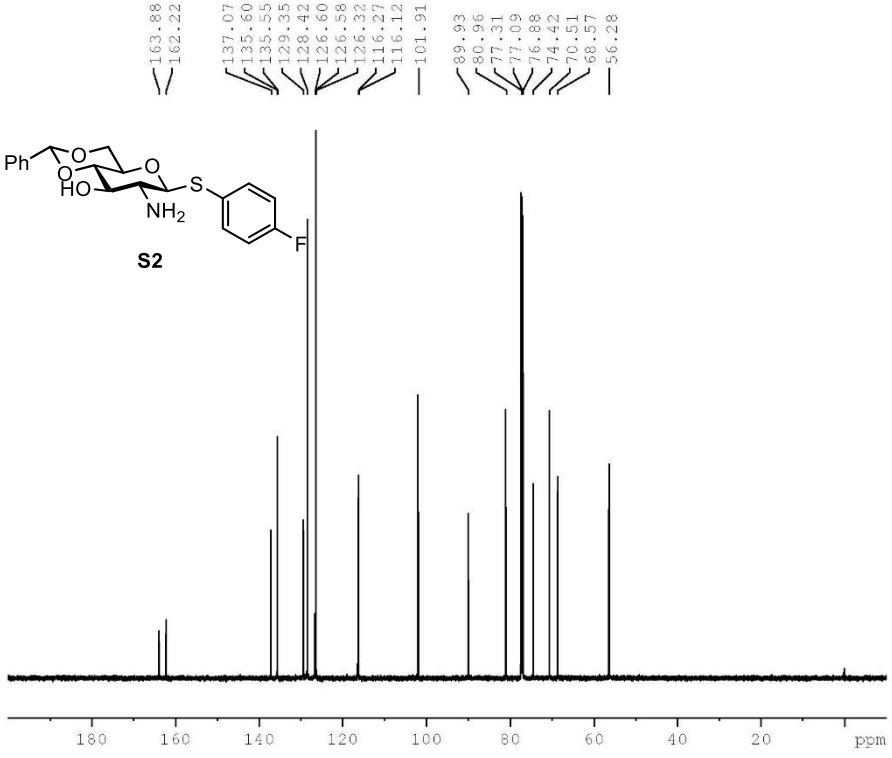
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                12335.526 Hz
FIDRES             0.188225 Hz
AQ                 2.6563926 sec
RG                       203
DW                    40.533 usec
DE                     6.50 usec
TE                    294.5 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.00 usec
PL1                    -2.00 dB
PL1W              18.91009140 W
SFO1              600.1337060 MHz

F2 - Processing parameters
SI                        32768
SF                600.1300157 MHz
WDW                        EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```

^{13}C NMR




```
Current Data Parameters
NAME      END-113 carbon
EXPNO                 10
PROCNO                 1

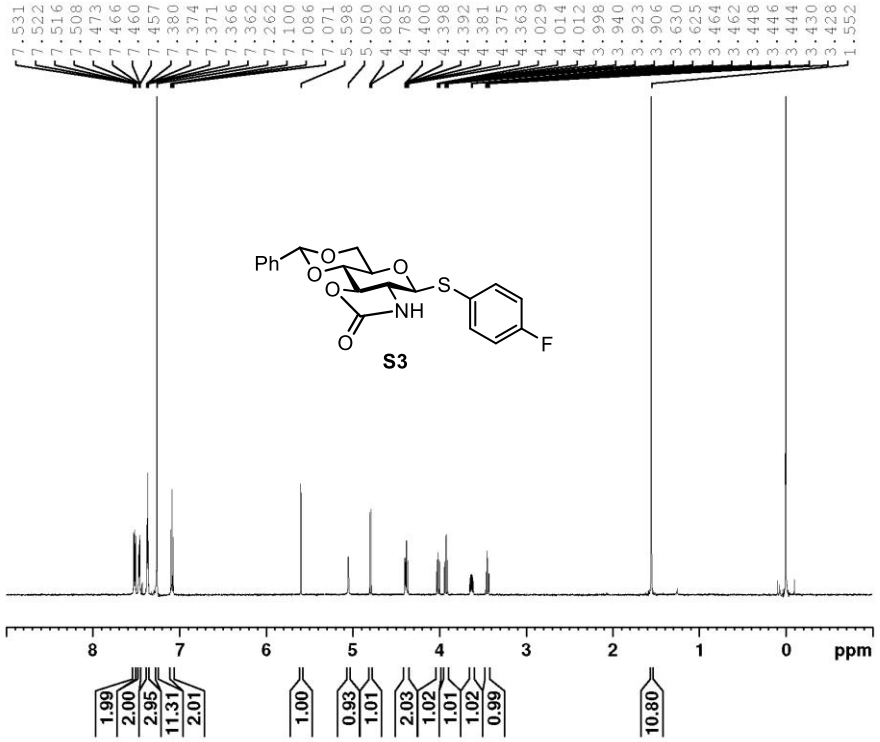
F2 - Acquisition Parameters
Date_           20210319
Time                9.27
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  500
DS                    2
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ             0.9087659 sec
RG                  203
DW                13.867 usec
DE                 6.50 usec
TE                296.5 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.00 usec
PL1                -1.00 dB
PL1W          125.22619629 W
SFO1          150.9178988 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               13.78 dB
PL13               14.00 dB
PL2W          18.91009140 W
PL12W          0.49968192 W
PL13W          0.47499999 W
SFO2          600.1324005 MHz

F2 - Processing parameters
SI                    32768
SF            150.9028090 MHz
WDW                    EM
SSB          0
LB                 1.00 Hz
GB           0
PC                 1.40
```

^{1}H NMR

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NAME         END-115 crude
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_              20210322
Time                  9.22
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH             12335.526 Hz
FIDRES          0.188225 Hz
AQ              2.6563926 sec
RG                     203
DW                40.533 usec
DE                 6.50 usec
TE                294.5 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                  13.00 usec
PL1                 -2.00 dB
PL1W         18.91009140 W
SFO1        600.1337060 MHz

F2 - Processing parameters
SI                   32768
SF           600.1300153 MHz
WDW                     EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```
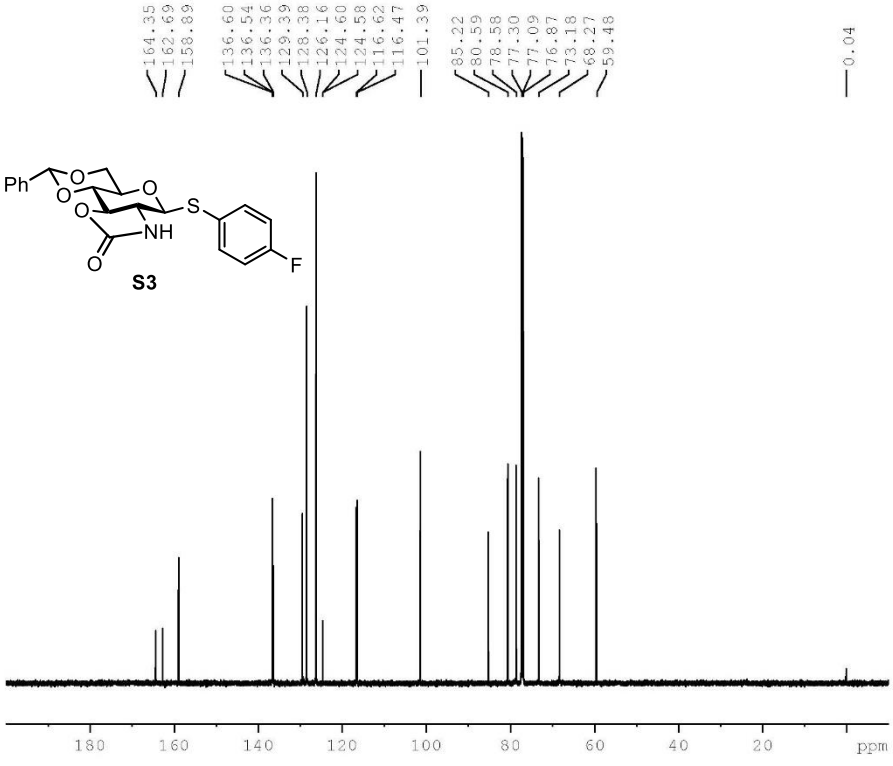

^{13}C NMR

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Current Data Parameters
NAME         END-115 carbon
EXPNO                   10
PROCNO                   1

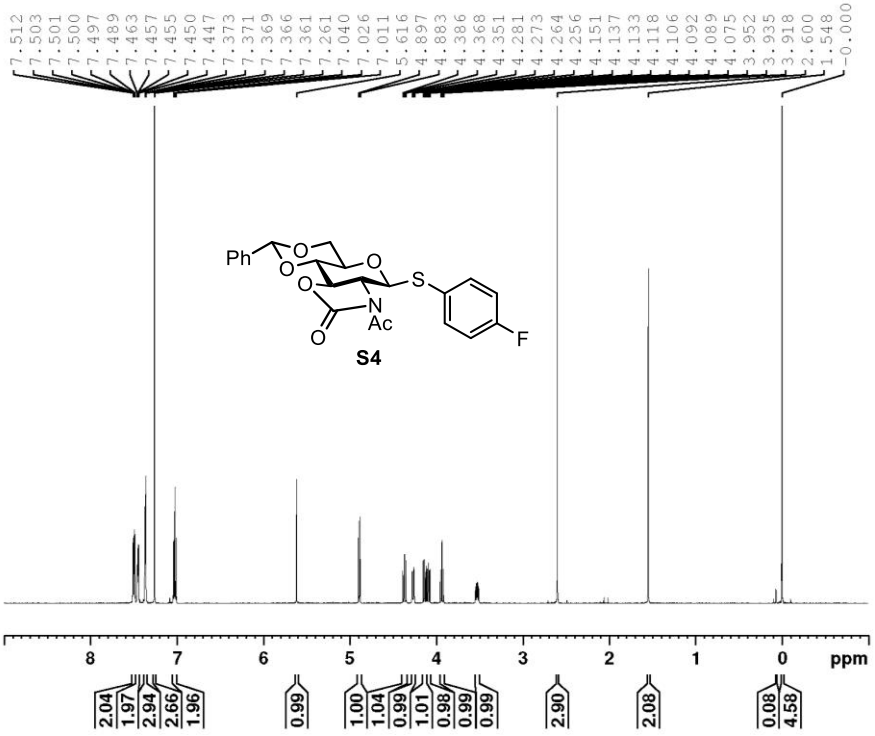
F2 - Acquisition Parameters
Date_              20210322
Time                 16.47
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                  65536
SOLVENT              CDCl3
NS                     500
DS                       2
SWH             36057.691 Hz
FIDRES          0.550197 Hz
AQ              0.9087659 sec
RG                     203
DW                13.867 usec
DE                 6.50 usec
TE                296.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                  10.00 usec
PL1                 -1.00 dB
PL1W        125.22619629 W
SFO1        150.9178988 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                    1H
PCPD2               80.00 usec
PL2                 -2.00 dB
PL12                13.78 dB
PL13                14.00 dB
PL2W         18.91009140 W
PL12W         0.49968192 W
PL13W         0.47499999 W
SFO2        600.1324005 MHz

F2 - Processing parameters
SI                   32768
SF           150.9028090 MHz
WDW                     EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```

^{1}H NMR

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Current Data Parameters
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EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20210326
Time              10.38
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           12335.526 Hz
FIDRES        0.188225 Hz
AQ            2.6563926 sec
RG                  203
DW               40.533 usec
DE                6.50 usec
TE               295.7 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                13.00 usec
PL1               -2.00 dB
PL1W        18.91009140 W
SFO1        600.1337060 MHz

F2 - Processing parameters
SI                32768
SF          600.1300159 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```
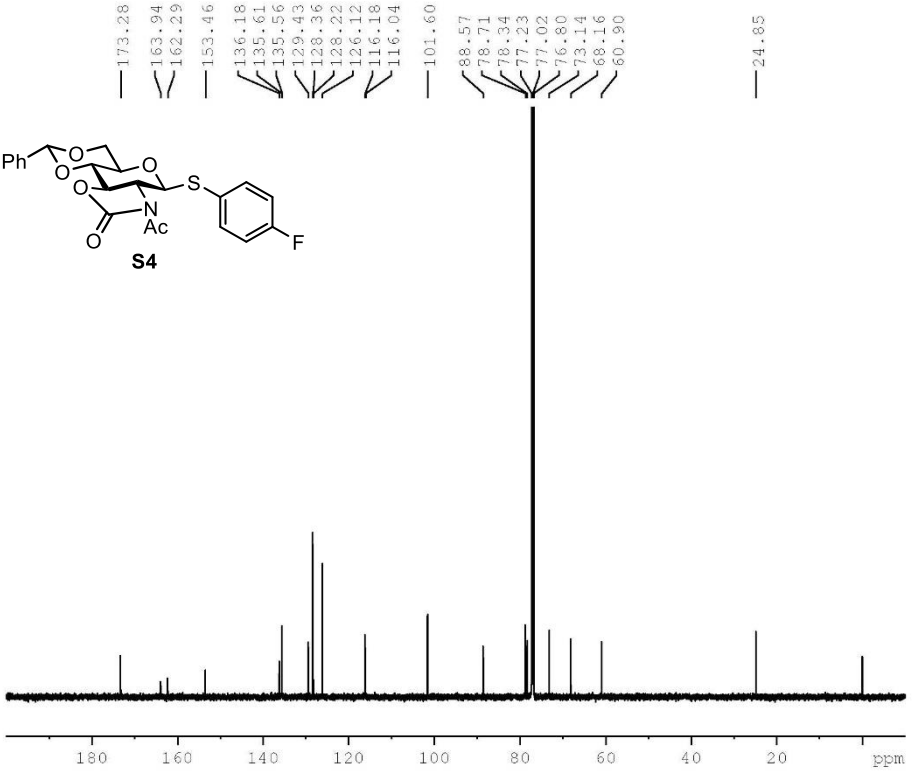
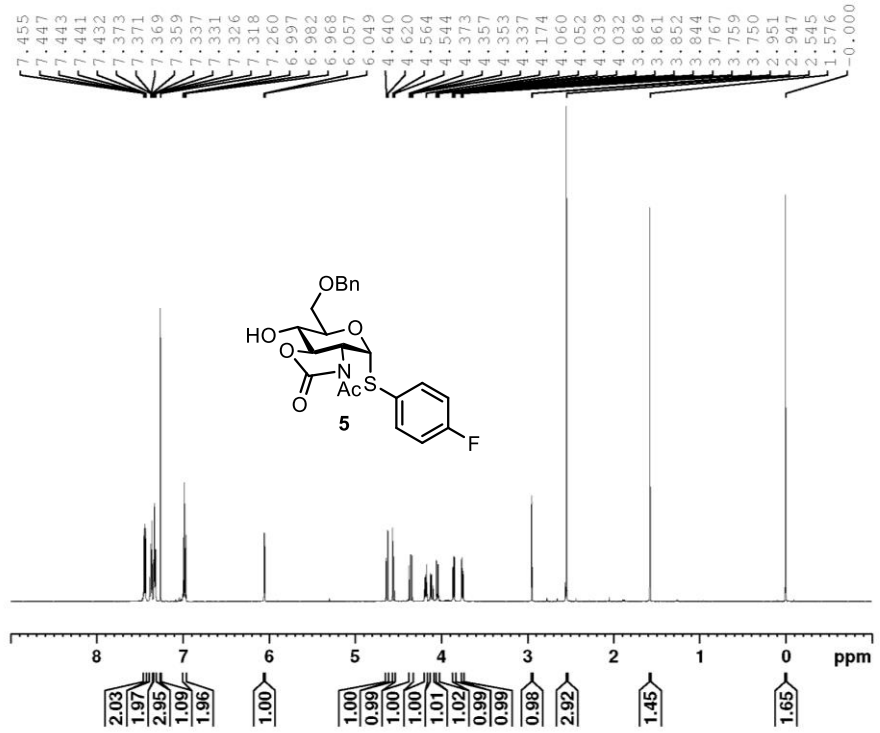

^{13}C NMR




```
Current Data Parameters
NAME             END-194 C
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_            20210930
Time                15.48
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1900
DS                     2
SWH            36057.691 Hz
FIDRES          0.550197 Hz
AQ              0.9087659 sec
RG                   203
DW                13.867 use
DE                 6.50 use
TE                300.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.00 use
PL1                -1.00 dB
PL1W        125.22619629 W
SFO1         150.9178988 MHz

======== CHANNEL f2 =======
CPDPRG[2          waltz16
NUC2                   1H
PCPD2              80.00 use
PL2                -2.00 dB
PL12               13.78 dB
PL13               14.00 dB
PL2W         18.91009140 W
PL12W         0.49968192 W
PL13W         0.47499999 W
SFO2         600.1324005 MHz

F2 - Processing parameters
SI                 32768
SF           150.9028091 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{1}H NMR

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Current Data Parameters
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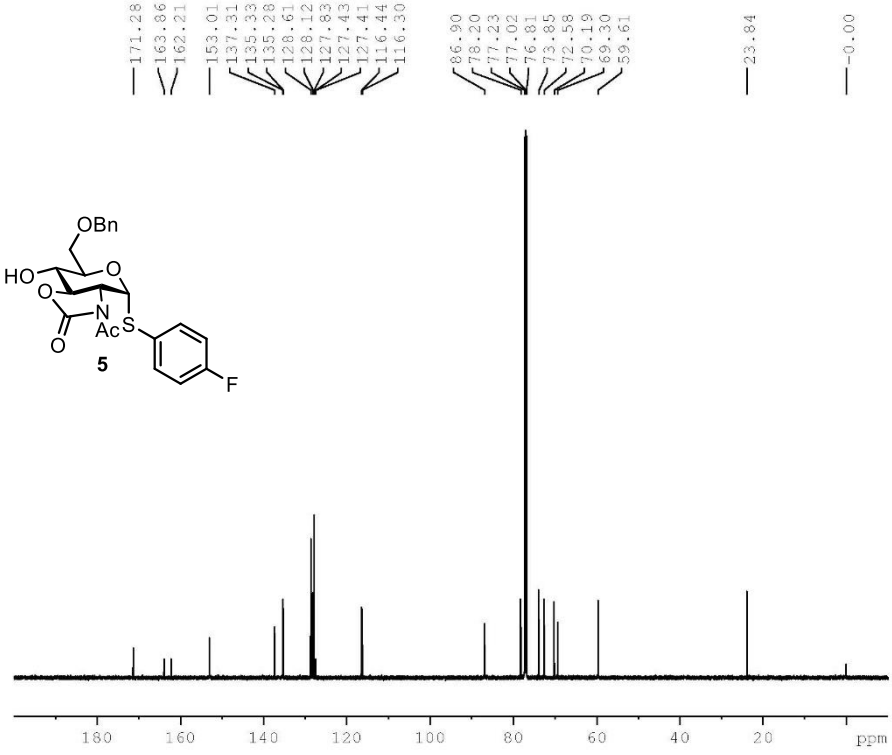
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20210402
Time              14.46
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH          12335.526 Hz
FIDRES        0.188225 Hz
AQ           2.6563926 sec
RG                 203
DW              40.533 usec
DE               6.50 usec
TE              296.9 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1              13.00 usec
PL1             -2.00 dB
PL1W       18.91009140 W
SFO1       600.1337060 MHz

F2 - Processing parameters
SI                32768
SF          600.1300161 MHz
WDW                  EM
SSB       0
LB              0.30 Hz
GB        0
PC              1.00
```

^{13}C NMR




```
Current Data Parameters
NAME          RA-133 13C
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20210402
Time              15.47
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                1024
DS                   2
SWH          36057.691 Hz
FIDRES        0.550197 Hz
AQ           0.9087659 sec
RG                 203
DW              13.867 usec
DE               6.50 usec
TE              298.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

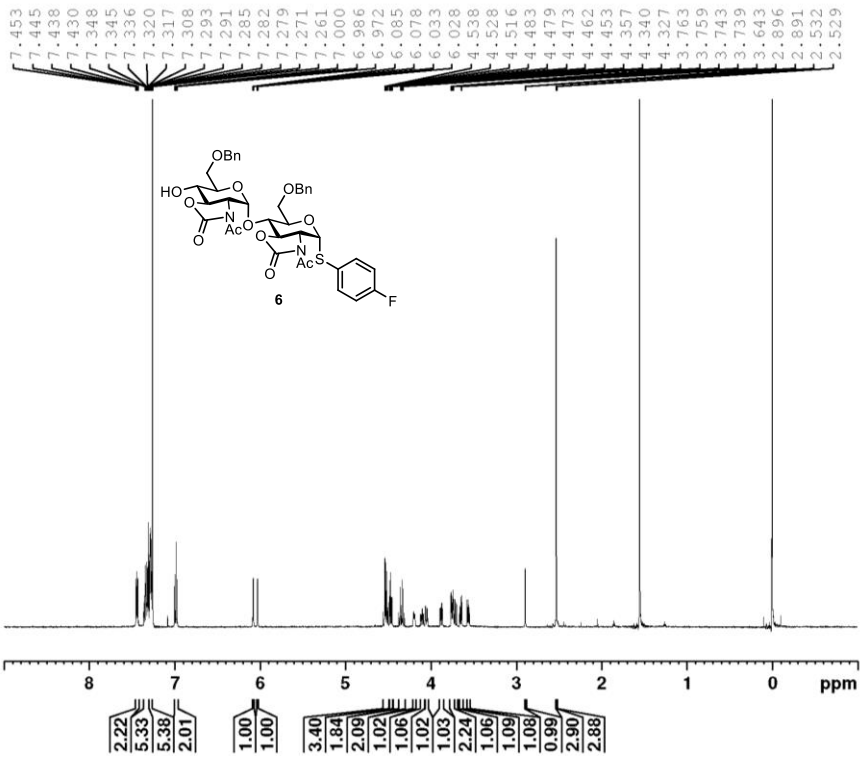
======== CHANNEL f1 ========
NUC1                13C
P1              10.00 usec
PL1             -1.00 dB
PL1W       125.22619629 W
SFO1       150.9178988 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                1H
PCPD2           80.00 usec
PL2             -2.00 dB
PL12            13.78 dB
PL13            14.00 dB
PL2W       18.91009140 W
PL12W       0.49968192 W
PL13W       0.47499999 W
SFO2       600.1324005 MHz

F2 - Processing parameters
SI                32768
SF          150.9028103 MHz
WDW                  EM
SSB       0
LB              1.00 Hz
GB        0
PC              1.40
```

9. ^{1}H, ^{13}C NMR, H-H COSY, and HMQC spectra of linear oligosaccharides

^{1}H NMR



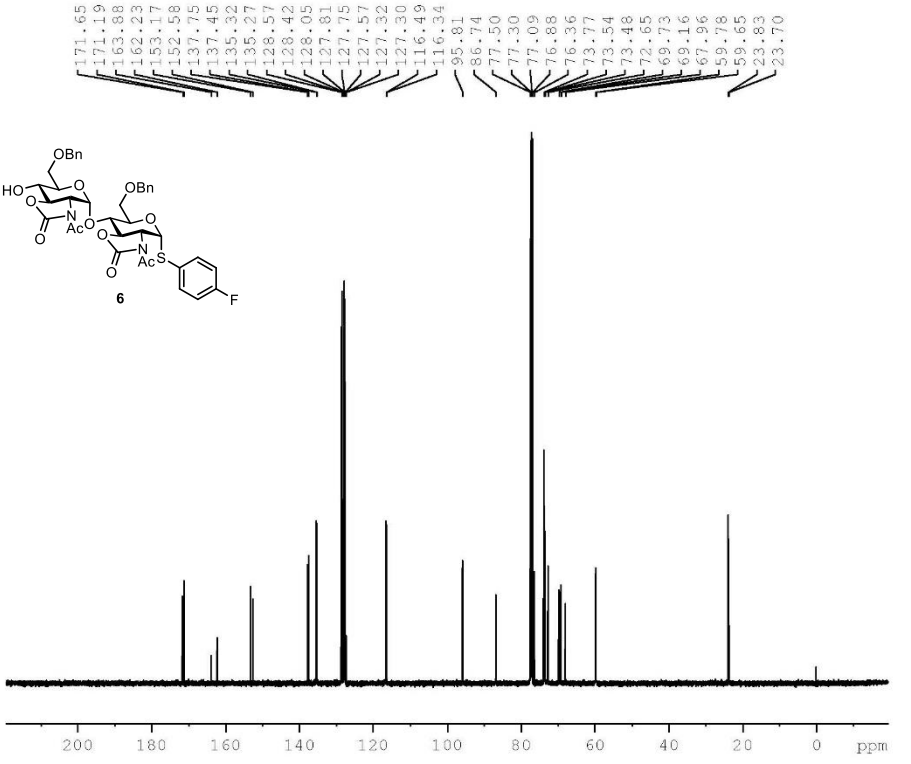

```
Current Data Parameters
NAME                 END-1n2 kore
EXPNO                          10
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20210903
Time                        16.47
INSTRUM                     spect
PROBHD     5 mm PABBO BB-
PULPROG                      zg30
TD                          65536
SOLVENT                     CDCl3
NS                             16
DS                              2
SWH                     12335.526 Hz
FIDRES                   0.188225 Hz
AQ                       2.6563926 sec
RG                            203
DW                         40.533 usec
DE                          6.50 usec
TE                         297.8 K
D1                    1.00000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                           1H
P1                          13.00 usec
PL1                         -2.00 dB
PL1W                18.91009140 W
SFO1                600.1337060 MHz

F2 - Processing parameters
SI                          32768
SF                  600.1300159 MHz
WDW                            EM
SSB                0
LB                           0.30 Hz
GB                 0
PC                           1.00
```

^{13}C NMR




```
Current Data Parameters
NAME      END-SPhF 1n2 C
EXPNO                  10
PROCNO                  1

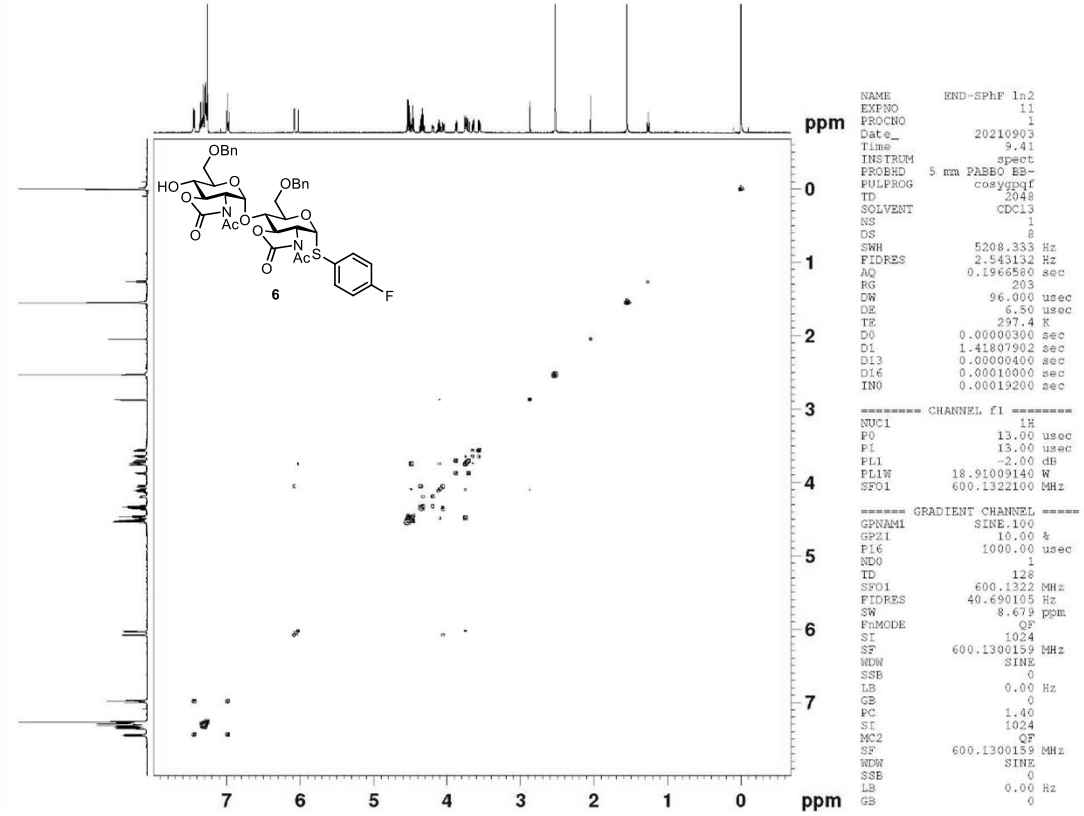
F2 - Acquisition Parameters
Date_            20210906
Time                20.29
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  500
DS                    2
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ             0.9087659 sec
RG                  203
DW               13.867 usec
DE                6.50 usec
TE                298.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.00 usec
PL1                -1.00 dB
PL1W       125.22619629 W
SFO1        150.9178988 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               13.78 dB
PL13               14.00 dB
PL2W        18.91009140 W
PL12W        0.49968192 W
PL13W        0.47499999 W
SFO2        600.1324005 MHz

F2 - Processing parameters
SI                  32768
SF          150.9028090 MHz
WDW                    EM
SSB                0
LB                   1.00 Hz
GB                 0
PC                   1.40
```

H-H cosy




```
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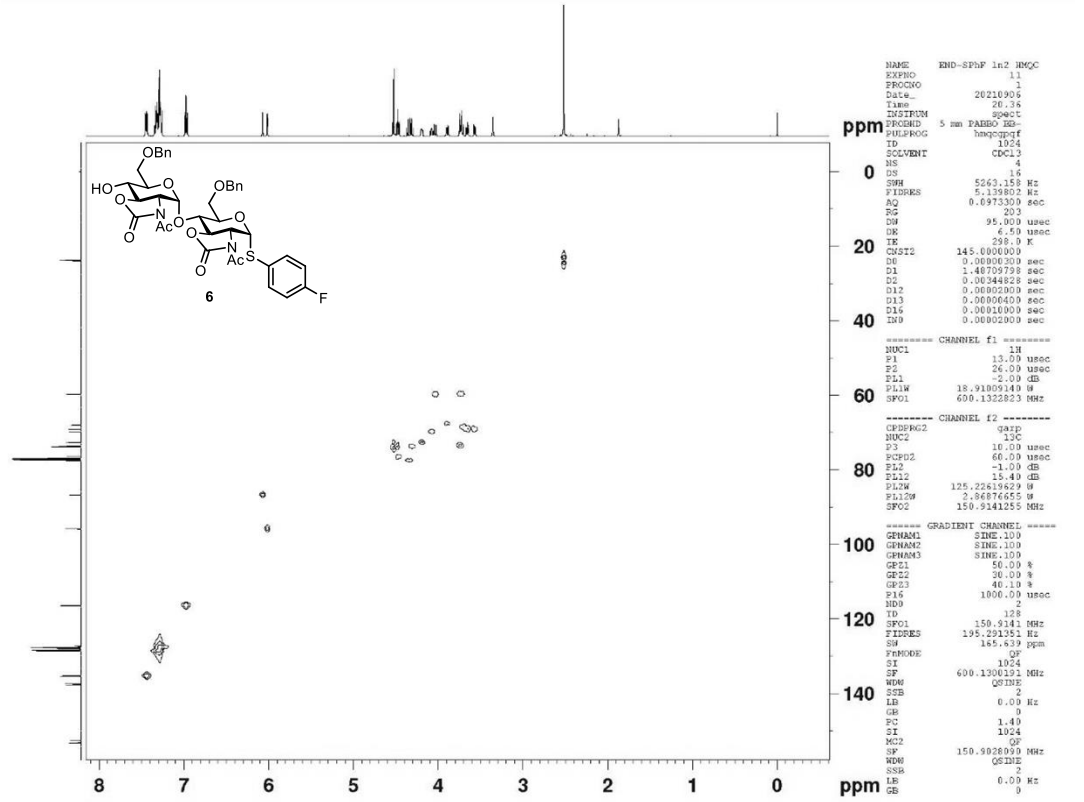
EXPNO              11
PROCNO              1
Date_          20210903
Time               9.41
INSTRUM           spect
PROBHD    5 mm PABBO BB-
PULPROG        cosygpqf
TD                2048
SOLVENT           CDCl3
NS                   1
DS                   8
SWH           5208.333 Hz
FIDRES        2.543132 Hz
AQ            0.1966580 sec
RG                 203
DW              96.000 usec
DE               6.50 usec
TE              297.4 K
D0            0.00000300 sec
D1            1.41807902 sec
D13           0.00000400 sec
D16           0.00010000 sec
IN0           0.00019200 sec

======== CHANNEL f1 ========
NUC1               1H
P0              13.00 usec
P1              13.00 usec
PL1             -2.00 dB
PL1W       18.91009140 W
SFO1      600.1322100 MHz

====== GRADIENT CHANNEL =====
GPNAM1         SINE.100
GPZ1             10.00 %
P16            1000.00 usec
ND0                  1
TD                 128
SFO1        600.1322 MHz
FIDRES     40.690105 Hz
SW             8.679 ppm
FnMODE            QF
SI                1024
SF         600.1300159 MHz
WDW             SINE
SSB                 0
LB               0.00 Hz
GB                  0
PC               1.40
SI                1024
MC2               QF
SF         600.1300159 MHz
WDW             SINE
SSB                 0
LB               0.00 Hz
GB                  0
```

HMQC



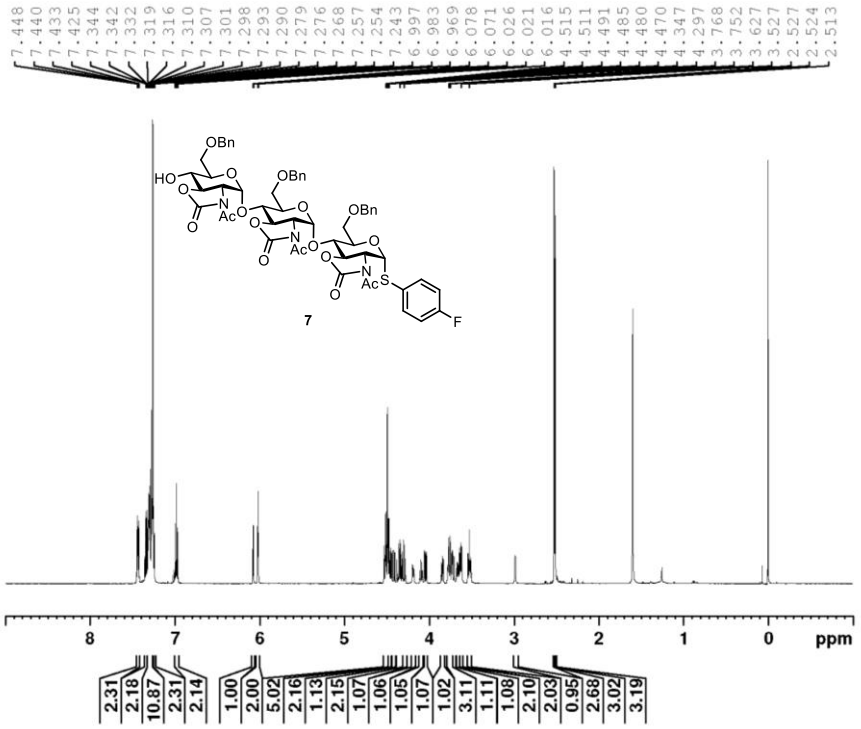

```
NAME      END-SPhF 1n2 HMQC
EXPNO              11
PROCNO              1
Date_          20210906
Time               20.36
INSTRUM           spect
PROBHD    5 mm PABBO BB-
PULPROG        hmqcgpqf
TD                1024
SOLVENT           CDCl3
NS                   4
DS                  16
SWH           5263.158 Hz
FIDRES        5.139802 Hz
AQ            0.0973300 sec
RG                 203
DW              95.000 usec
DE               6.50 usec
TE              298.0 K
CNST2      145.0000000
D0            0.00000300 sec
D1            1.48709798 sec
D2            0.00344828 sec
D12           0.00002000 sec
D13           0.00000400 sec
D16           0.00010000 sec
IN0           0.00002000 sec

======== CHANNEL f1 ========
NUC1               1H
P1              13.00 usec
P2              26.00 usec
PL1             -2.00 dB
PL1W       18.91009140 W
SFO1      600.1322823 MHz

======== CHANNEL f2 ========
CPDPRG2         garp
NUC2               13C
P3              10.00 usec
PCPD2           60.00 usec
PL2             -1.00 dB
PL12            15.40 dB
PL2W       125.22619629 W
PL12W        2.86876655 W
SFO2      150.9141255 MHz

====== GRADIENT CHANNEL =====
GPNAM1         SINE.100
GPNAM2         SINE.100
GPNAM3         SINE.100
GPZ1             50.00 %
GPZ2             30.00 %
GPZ3             40.10 %
P16            1000.00 usec
ND0                  2
TD                 128
SFO1      150.9141 MHz
FIDRES    195.291351 Hz
SW            165.639 ppm
FnMODE            QF
SI                1024
SF         600.1300191 MHz
WDW             QSINE
SSB                 2
LB               0.00 Hz
GB                  0
PC               1.40
SI                1024
MC2               QF
SF         150.9028090 MHz
WDW             QSINE
SSB                 2
LB               0.00 Hz
GB                  0
```

^{1}H NMR

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Current Data Parameters
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EXPNO                20
PROCNO                1

F2 - Acquisition Parameters
Date_          20210917
Time              20.37
INSTRUM           spect
PROBHD  5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH          12335.526 Hz
FIDRES        0.188225 Hz
AQ           2.6563926 sec
RG                 203
DW              40.533 usec
DE               6.50 usec
TE              297.3 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1               13.00 usec
PL1              -2.00 dB
PL1W       18.91009140 W
SFO1      600.1337060 MHz

F2 - Processing parameters
SI                32768
SF          600.1300159 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```
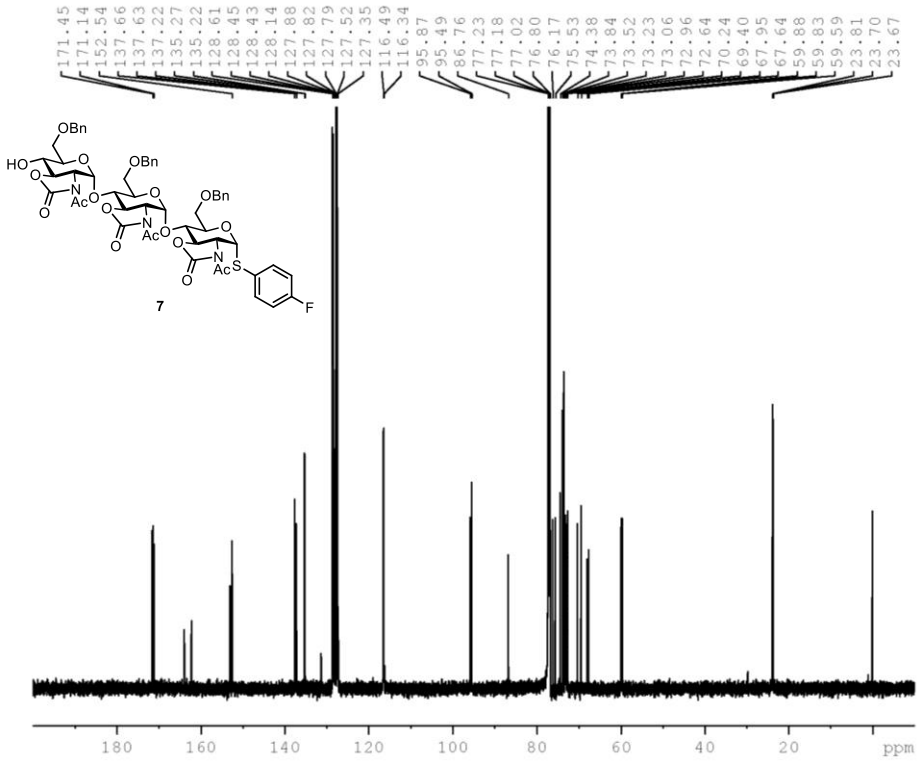
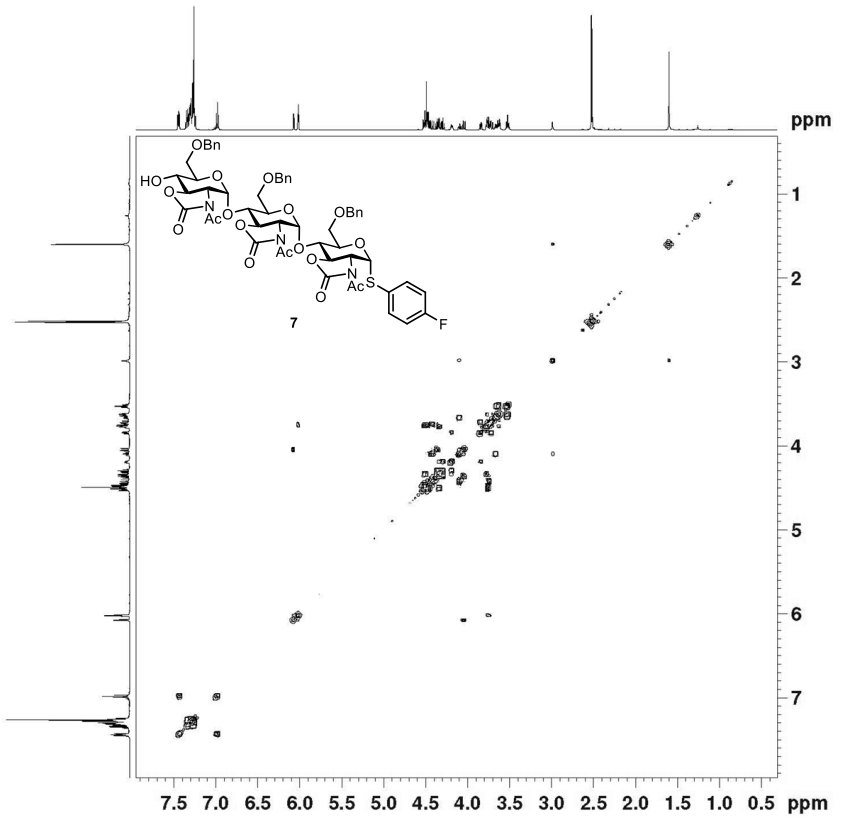

^{13}C NMR



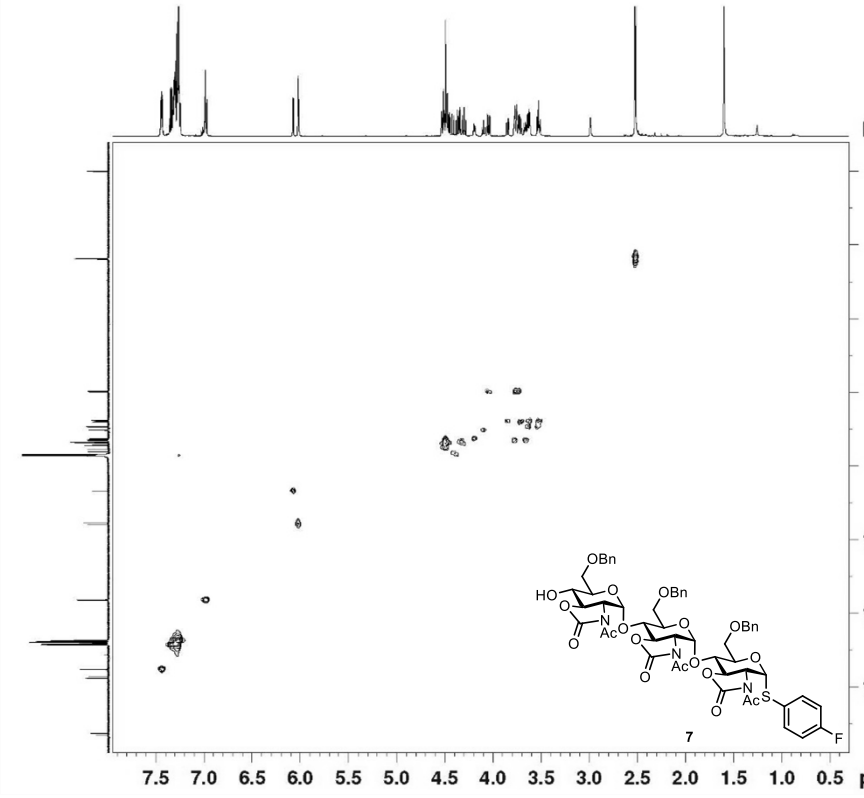

```
Current Data Parameters
NAME      END-SPhF 1n3 C
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20210918
Time               8.41
INSTRUM           spect
PROBHD  5 mm PABBO BB-
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS               14000
DS                   2
SWH          36057.691 Hz
FIDRES        0.550197 Hz
AQ           0.9087659 sec
RG                 203
DW              13.867 usec
DE               6.50 usec
TE              298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1               10.00 usec
PL1              -1.00 dB
PL1W       125.22619629 W
SFO1       150.9178988 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -2.00 dB
PL12             13.78 dB
PL13             14.00 dB
PL2W       18.91009140 W
PL12W       0.49968192 W
PL13W       0.47499999 W
SFO2      600.1324005 MHz

F2 - Processing parameters
SI                32768
SF          150.9028101 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

H-H cosy



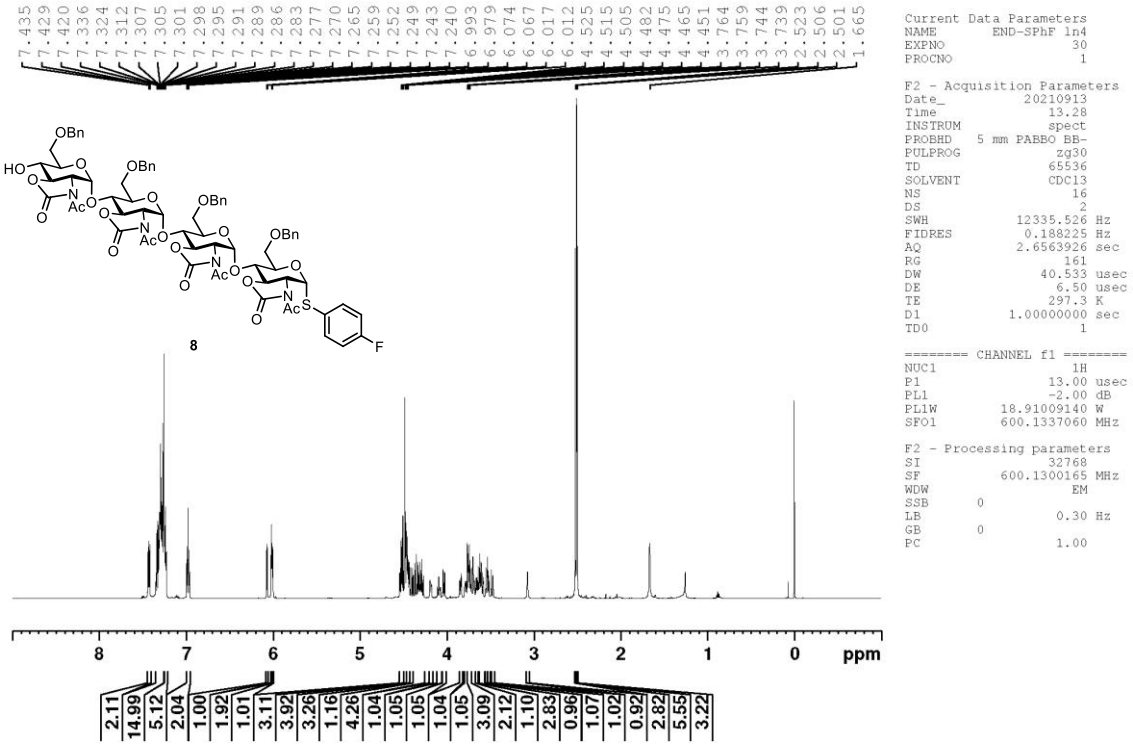

```
NAME      END-SPhF ln3 cosy
EXPNO               11
PROCNO               1
Date_         20210917
Time             20.59
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG       cosygpqf
TD               2048
SOLVENT          CDCl3
NS                  1
DS                  8
SWH           4587.156 Hz
FIDRES        2.239822 Hz
AQ            0.2232820 sec
RG                144
DW            109.000 usec
DE               6.50 usec
TE              297.4 K
D0         0.00000300 sec
D1         1.39145505 sec
D13        0.00000400 sec
D16        0.00010000 sec
IN0        0.00021800 sec

======== CHANNEL f1 ========
NUC1                1H
P0              13.00 usec
P1              13.00 usec
PL1             -2.00 dB
PL1W       18.91009140 W
SFO1       600.1324953 MHz

====== GRADIENT CHANNEL =====
GPNAM1         SINE.100
GPZ1              10.00 %
P16             1000.00 usec
ND0                  1
TD                 128
SFO1          600.1325 MHz
FIDRES        35.837154 Hz
SW             7.644 ppm
FnMODE              QF
SI                1024
SF         600.1300159 MHz
WDW               SINE
SSB                  0
LB                0.00 Hz
GB                   0
PC                1.40
SI                1024
MC2                 QF
SF         600.1300159 MHz
WDW               SINE
SSB                  0
LB                0.00 Hz
GB                   0
```

HMQC



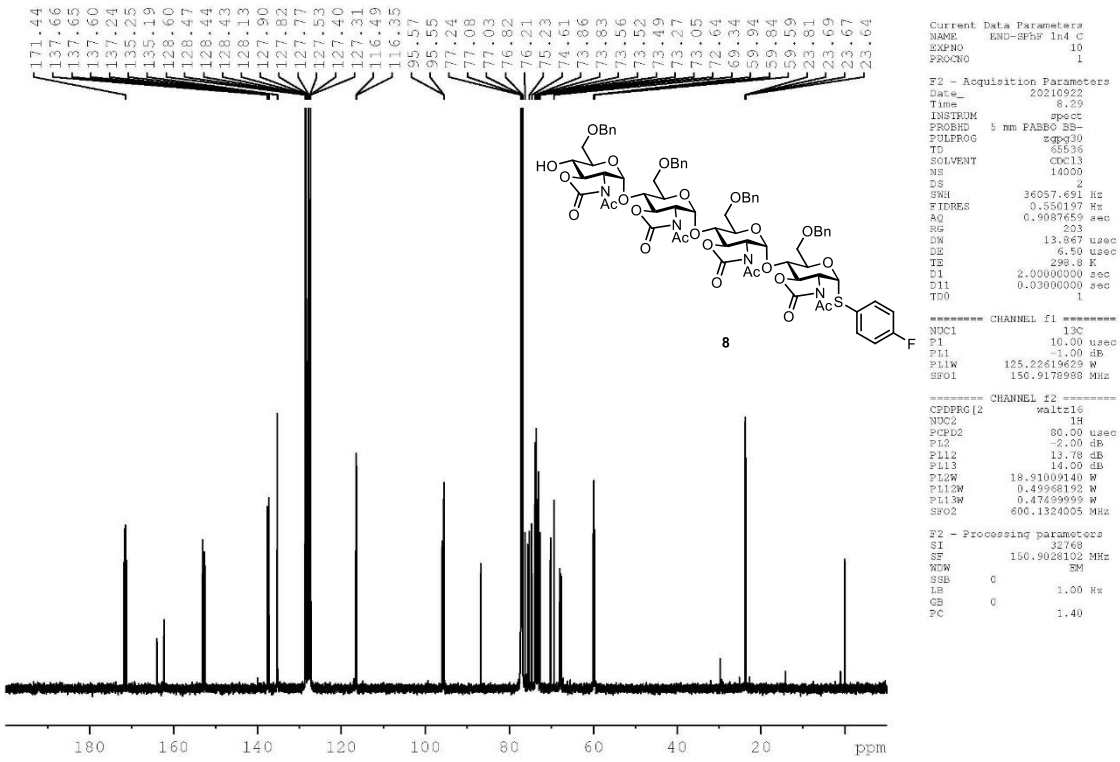

```
NAME      END-SPhF ln3
EXPNO               21
PROCNO               1
Date_         20210917
Time             20.39
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG       hmqcgpqf
TD               1024
SOLVENT          CDCl3
NS                  4
DS                 16
SWH           4587.156 Hz
FIDRES        4.479644 Hz
AQ            0.1116860 sec
RG                203
DW            109.000 usec
DE               6.50 usec
TE              297.4 K
CNST2       145.0000000
D0         0.00000300 sec
D1         1.47276199 sec
D2         0.00344828 sec
D12        0.00002000 sec
D13        0.00000400 sec
D16        0.00010000 sec
IN0        0.00002000 sec

-------- CHANNEL f1 --------
NUC1                1H
P1              13.00 usec
P2              26.00 usec
PL1             -2.00 dB
PL1W       18.91009140 W
SFO1       600.1324914 MHz

======== CHANNEL f2 ========
CPDPRG2          garp
NUC2               13C
P3              10.00 usec
PCPD2           60.00 usec
PL2             -1.00 dB
PL12            15.40 dB
PL2W       125.22619629 W
PL12W        2.86876655 W
SFO2       150.9141255 MHz

------ GRADIENT CHANNEL -----
GPNAM1         SINE.100
GPNAM2         SINE.100
GPNAM3         SINE.100
GPZ1              50.00 %
GPZ2              30.00 %
GPZ3              40.10 %
P16             1000.00 usec
ND0                  2
TD                 128
SFO1          159.9141 MHz
FIDRES        195.291351 Hz
SW             165.639 ppm
FnMODE              QF
SI                1024
SF         600.1300159 MHz
WDW              QSINE
SSB                  2
LB                0.00 Hz
GB                   0
PC                1.40
SI                1024
MC2                 QF
SF         150.9028090 MHz
WDW              QSINE
SSB                  2
LB                0.00 Hz
GB                   0
```

^{1}H NMR



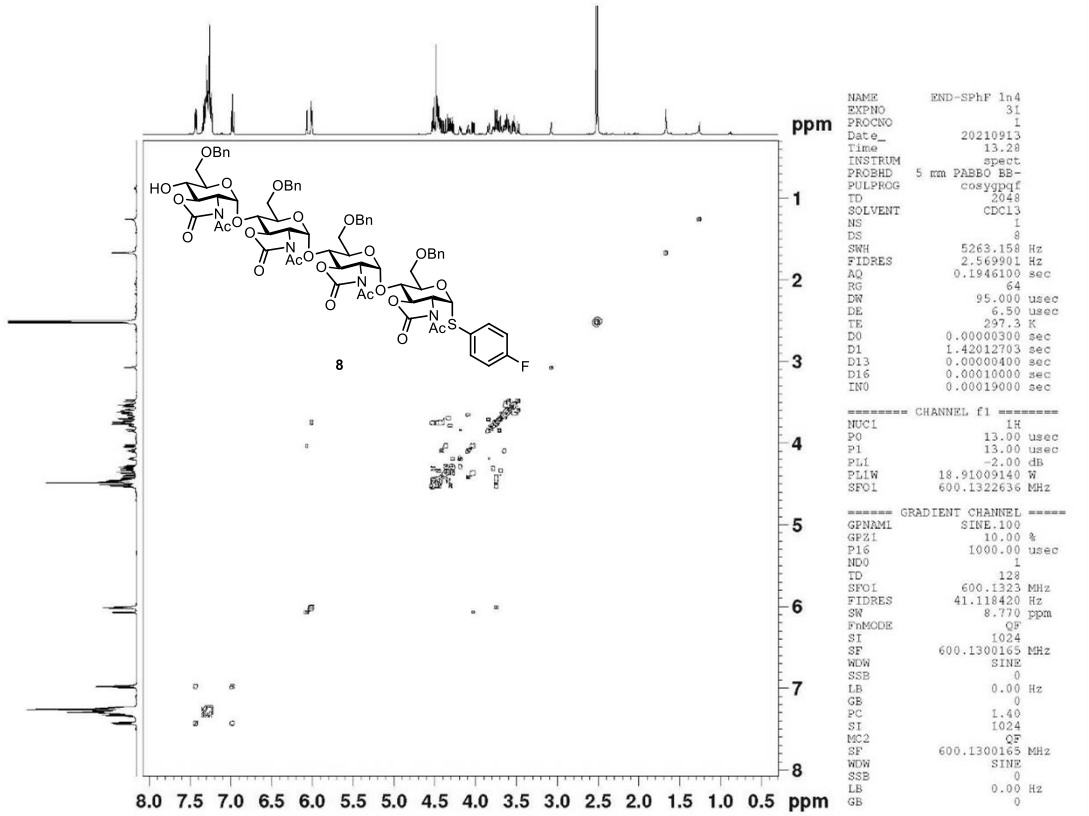

```
Current Data Parameters
NAME        END-SPhF 1n4
EXPNO                30
PROCNO                1

F2 - Acquisition Parameters
Date_          20210913
Time              13.28
INSTRUM           spect
PROBHD    5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH          12335.526 Hz
FIDRES        0.188225 Hz
AQ            2.6563926 sec
RG                 161
DW              40.533 usec
DE                6.50 usec
TE               297.3 K
D1          1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1               13.00 usec
PL1              -2.00 dB
PL1W       18.91009140 W
SFO1       600.1337060 MHz

F2 - Processing parameters
SI                32768
SF         600.1300165 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

^{13}C NMR

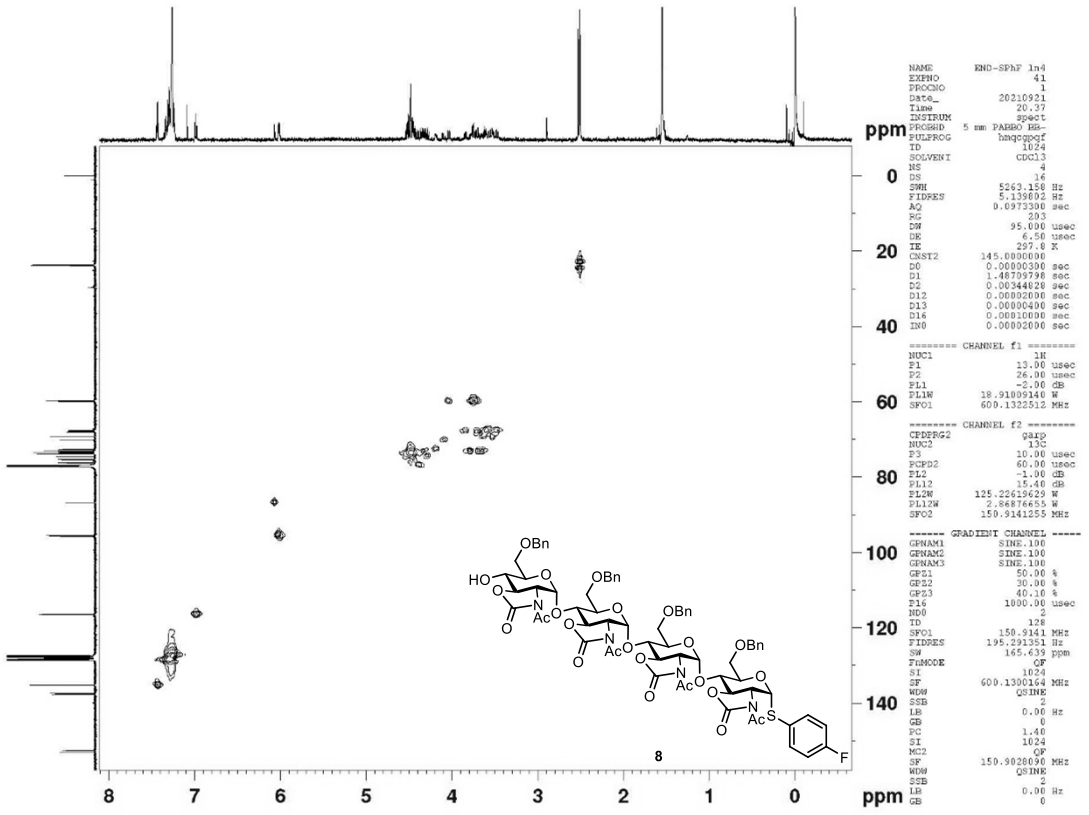



```
Current Data Parameters
NAME      END-SPhF 1n4 C
EXPNO                10
PROCNO0               1

F2 - Acquisition Parameters
Date_          20210922
Time               8.29
INSTRUM           spect
PROBHD    5 mm PABBO BB-
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS               14000
DS                   2
SWH          36057.691 Hz
FIDRES        0.550197 Hz
AQ            0.9087659 sec
RG                 203
DW              13.867 usec
DE                6.50 usec
TE               298.8 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1               10.00 usec
PL1              -1.00 dB
PL1W      125.22619629 W
SFO1       150.9178988 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -2.00 dB
PL12             13.78 dB
PL13             14.00 dB
PL2W       18.91009140 W
PL12W       0.49968192 W
PL13W       0.47499999 W
SFO2       600.1324005 MHz

F2 - Processing parameters
SI                32768
SF         150.9028102 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

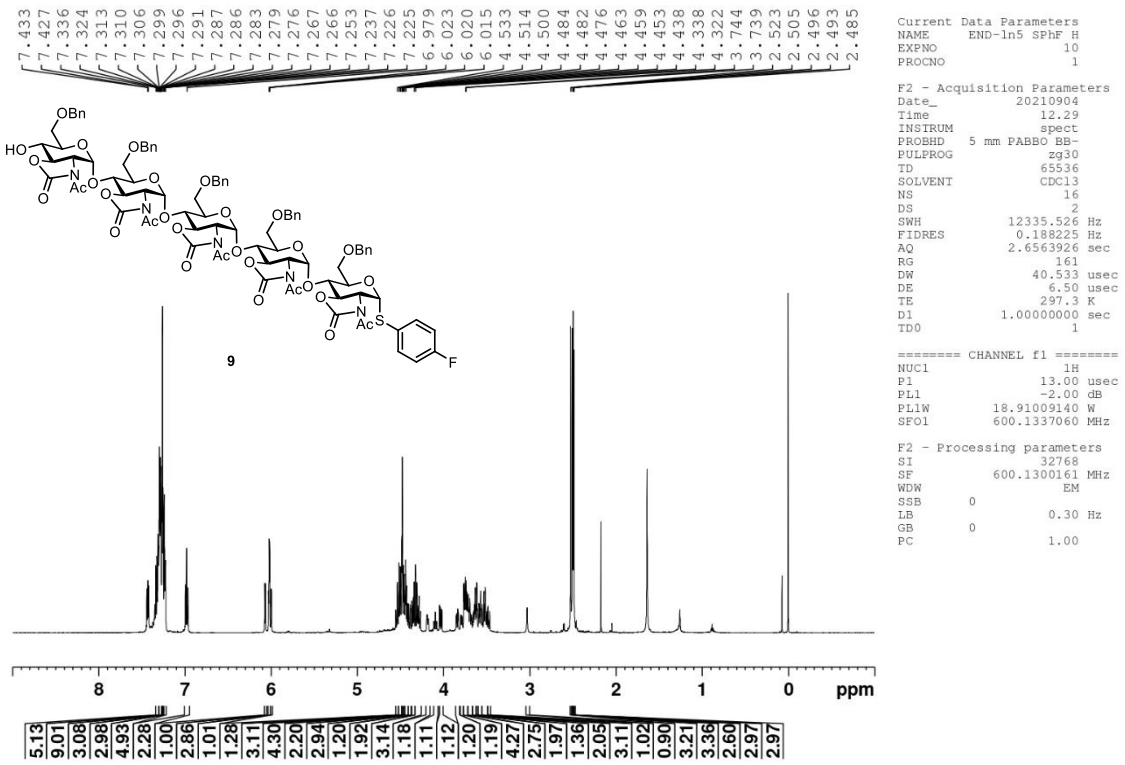
H-H cosy


ppm
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
8
NAME END-SPhF 1n4
EXPNO 31
PROCNO 1
Date_ 20210913
Time 13.28
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG cosygpqf
TD 2048
SOLVENT CDCl3
NS 1
DS 8
SWH 5263.158 Hz
FIDRES 2.569901 Hz
AQ 0.1946100 sec
RG 64
DW 95.000 usec
DE 6.50 usec
TE 297.3 K
D0 0.00000300 sec
D1 1.42012703 sec
D13 0.00000400 sec
D16 0.00010000 sec
IN0 0.00019000 sec
======== CHANNEL f1 ========
NUC1 1H
P0 13.00 usec
P1 13.00 usec
PL1 -2.00 dB
PL1W 18.91009140 W
SFO1 600.1322636 MHz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPZ1 10.00 %
P16 1000.00 usec
ND0 1
TD 128
SFO1 600.1323 MHz
FIDRES 41.118420 Hz
SW 8.770 ppm
FnMODE QF
SI 1024
SF 600.1300165 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
SI 1024
MC2 QF
SF 600.1300165 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

HMQC

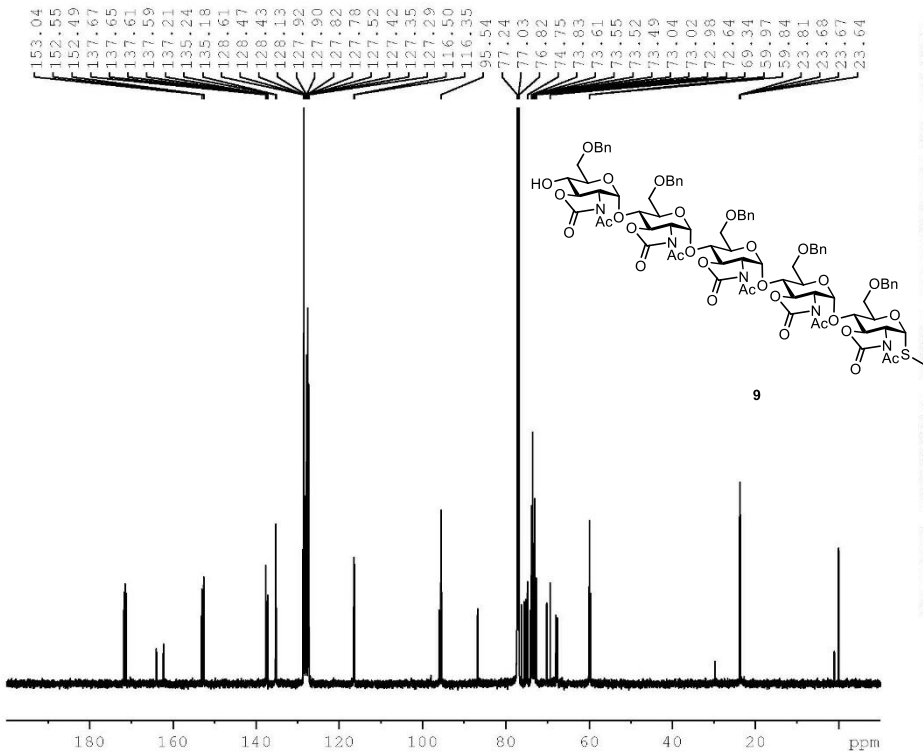

ppm
8 7 6 5 4 3 2 1 0 ppm
8
NAME END-SPhF 1n4
EXPNO 41
PROCNO 1
Date_ 20210921
Time 20.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG hmqcgpqf
TD 1024
SOLVENT CDCl3
NS 4
DS 16
SWH 5263.158 Hz
FIDRES 5.139602 Hz
AQ 0.0973300 sec
RG 203
DW 95.000 usec
DE 6.50 usec
TE 297.0 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.48709798 sec
D2 0.00344828 sec
D12 0.00002000 sec
D13 0.00000400 sec
D16 0.00010000 sec
IN0 0.00002000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
P2 26.00 usec
PL1 -2.00 dB
PL1W 18.91009140 W
SFO1 600.1322512 MHz
======== CHANNEL f2 ========
CPDPRG2 garp
NUC2 13C
P3 10.00 usec
PCPD2 60.00 usec
PL2 -1.00 dB
PL12 15.40 dB
PL2W 125.22619629 W
PL12W 2.86876655 W
SFO2 150.9141255 MHz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
D16 1000.00 usec
ND0 2
TD 128
SFO1 150.9141 MHz
FIDRES 195.291351 Hz
SW 165.639 ppm
FnMODE QF
SI 1024
SF 600.1300164 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40
SI 1024
MC2 QF
SF 150.9028090 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

^{1}H NMR



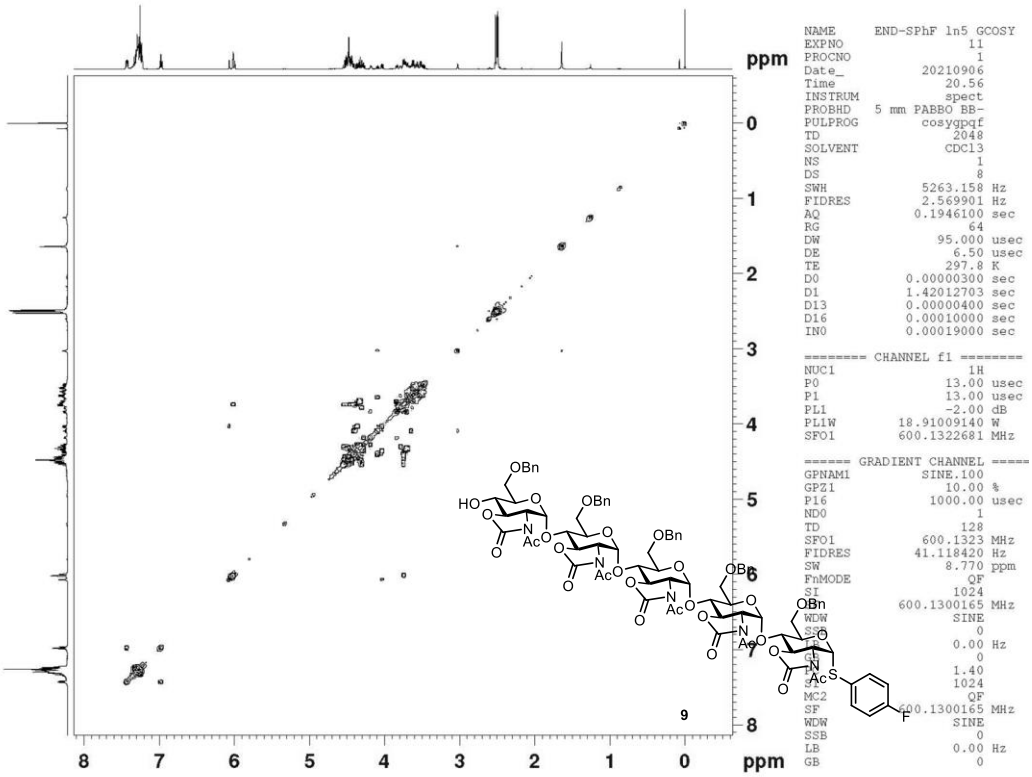

```
Current Data Parameters
NAME      END-1n5 SPhF H
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20210904
Time              12.29
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6563926 sec
RG                  161
DW               40.533 usec
DE                6.50 usec
TE              297.3 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                1H
P1               13.00 usec
PL1              -2.00 dB
PL1W        18.91009140 W
SFO1       600.1337060 MHz

F2 - Processing parameters
SI                32768
SF          600.1300161 MHz
WDW                  EM
SSB       0
LB             0.30 Hz
GB        0
PC             1.00
```

^{13}C NMR

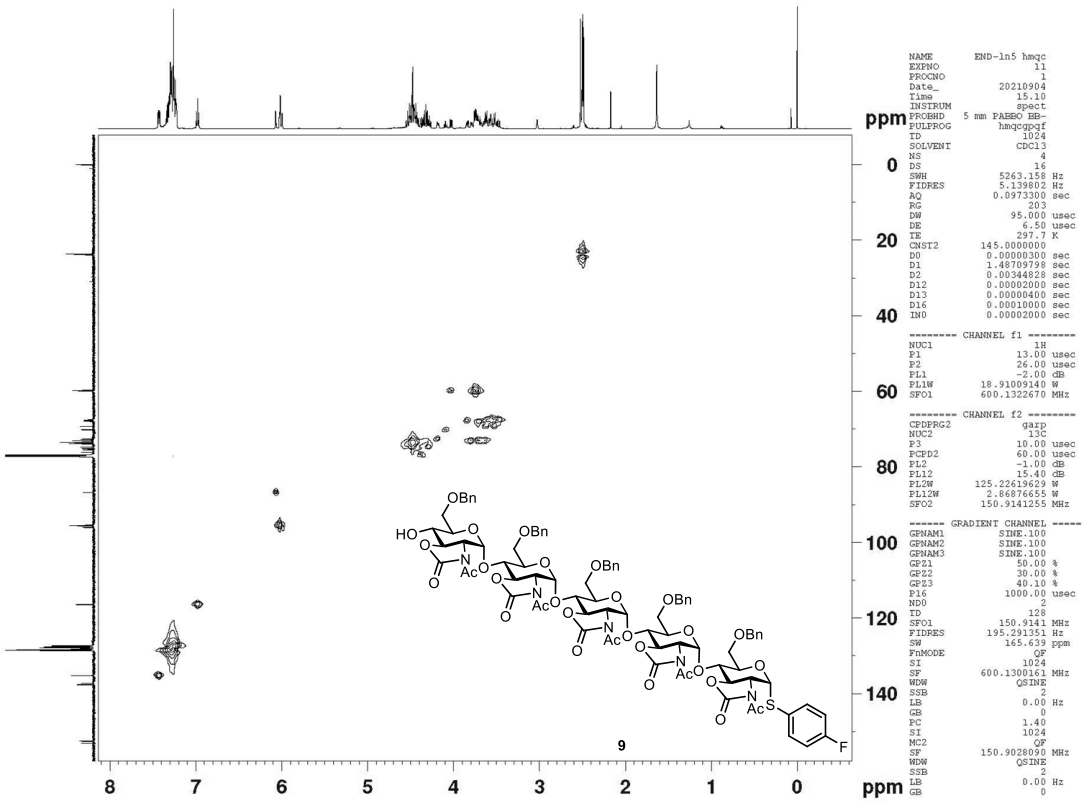



```
Current Data Parameters
NAME      END-SPhF 1n5 C 13
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20210907
Time              8.38
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                14000
DS                    2
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9087659 sec
RG                  203
DW               13.867 usec
DE                6.50 usec
TE              298.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1               13C
P1               10.00 usec
PL1              -1.00 dB
PL1W       125.22619629 W
SFO1       150.9178988 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -2.00 dB
PL12             13.78 dB
PL13             14.00 dB
PL2W        18.91009140 W
PL12W        0.49968192 W
PL13W        0.47499999 W
SFO2       600.1324005 MHz

F2 - Processing parameters
SI                32768
SF          150.9028102 MHz
WDW                  EM
SSB       0
LB             1.00 Hz
GB        0
PC             1.40
```

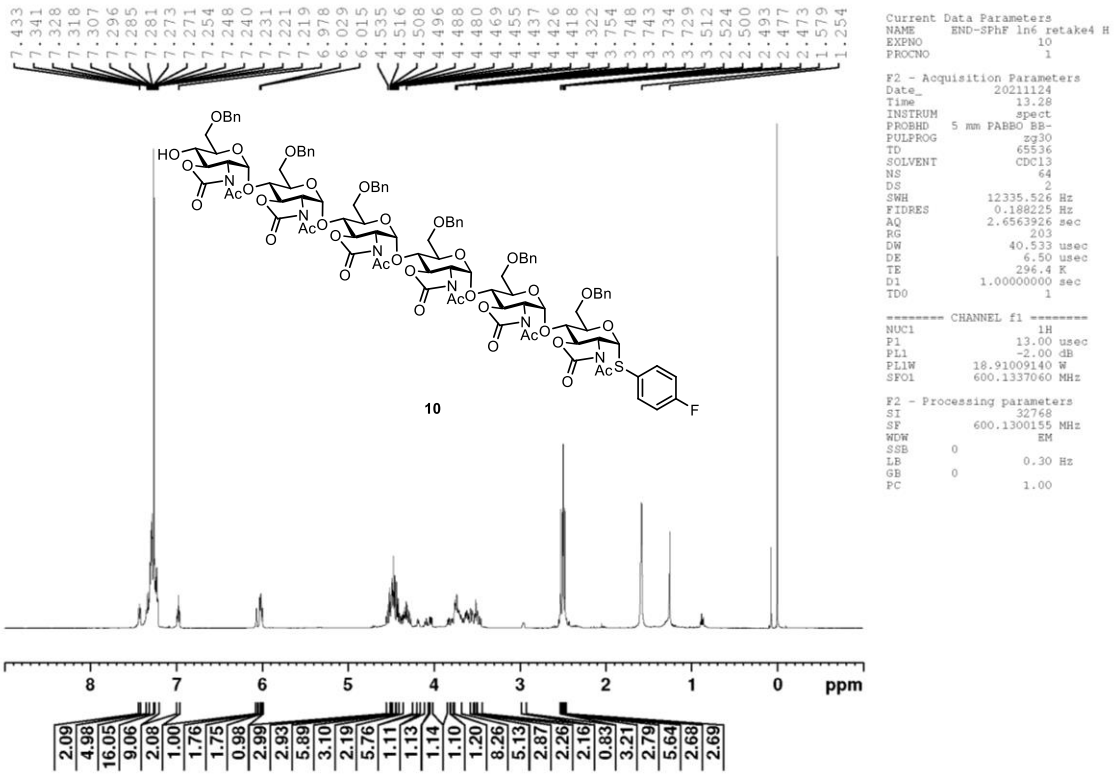
H-H cosy


ppm
9
NAME END-SPhF ln5 GCOSY
EXPNO 11
PROCNO 1
Date_ 20210906
Time 20.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG cosygpqf
TD 2048
SOLVENT CDCl3
NS 1
DS 8
SWH 5263.158 Hz
FIDRES 2.569901 Hz
AQ 0.1946100 sec
RG 64
DW 95.000 usec
DE 6.50 usec
TE 297.8 K
D0 0.00000300 sec
D1 1.42012703 sec
D13 0.00000400 sec
D16 0.00010000 sec
IN0 0.00019000 sec
======== CHANNEL f1 ========
NUC1 1H
P0 13.00 usec
P1 13.00 usec
PL1 -2.00 dB
PL1W 18.91009140 W
SFO1 600.1322681 MHz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPZ1 10.00 %
P16 1000.00 usec
ND0 1
TD 128
SFO1 600.1323 MHz
FIDRES 41.118420 Hz
SW 8.770 ppm
FnMODE QF
SI 1024
SF 600.1300165 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
SI 1024
MC2 QF
SF 600.1300165 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

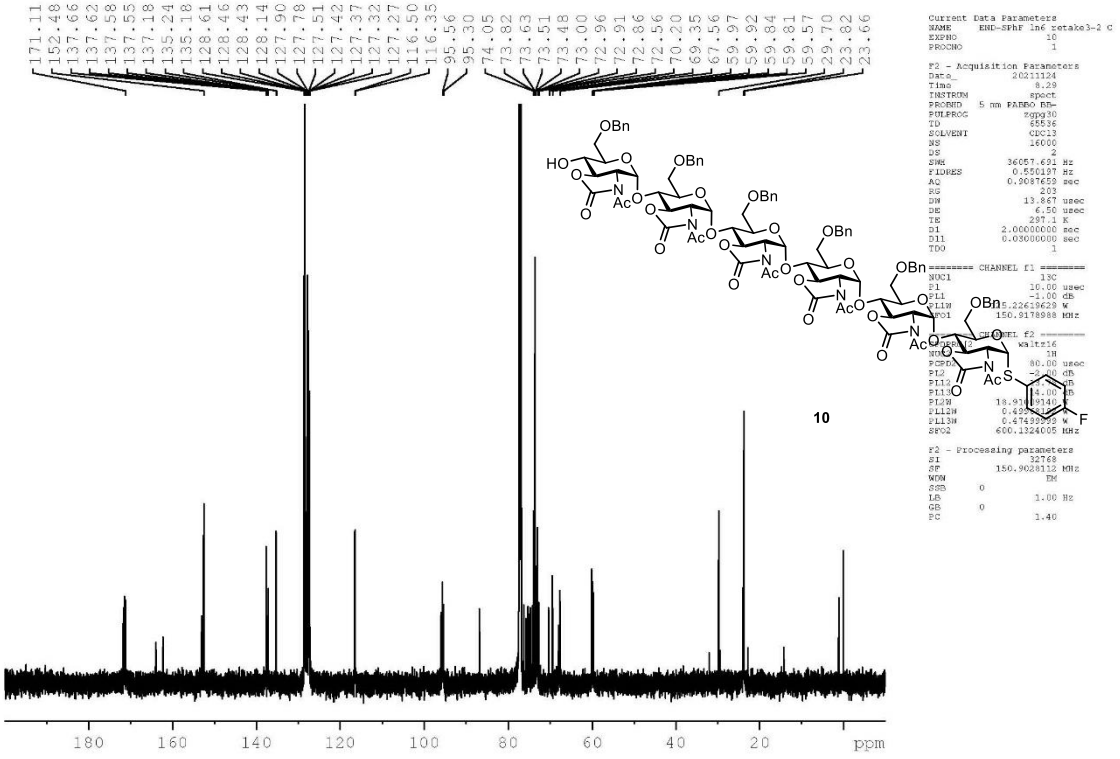
HMQC


ppm
9
NAME END-ln5 hmqc
EXPNO 11
PROCNO 1
Date_ 20210904
Time 15.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG hmqcgpqf
TD 1024
SOLVENT CDCl3
NS 4
DS 16
SWH 5263.158 Hz
FIDRES 5.139802 Hz
AQ 0.0973300 sec
RG 203
DW 95.000 usec
DE 6.50 usec
TE 297.7 K
CNST2 145.00000000
D0 0.00000300 sec
D1 1.48709798 sec
D2 0.00344828 sec
D12 0.00002000 sec
D13 0.00000400 sec
D16 0.00010000 sec
IN0 0.00002000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
P2 26.00 usec
PL1 -2.00 dB
PL1W 18.91009140 W
SFO1 600.1322670 MHz
======== CHANNEL f2 ========
CPDPRG2 garp
NUC2 13C
P3 10.00 usec
PCPD2 60.00 usec
PL2 -1.00 dB
PL12 15.40 dB
PL2W 125.22619629 W
PL12W 2.86876655 W
SFO2 150.9141255 MHz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec
ND0 2
TD 128
SFO1 150.9141 MHz
FIDRES 195.291351 Hz
SW 165.639 ppm
FnMODE QF
SI 1024
SF 600.1300161 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40
SI 1024
MC2 QF
SF 150.9028090 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

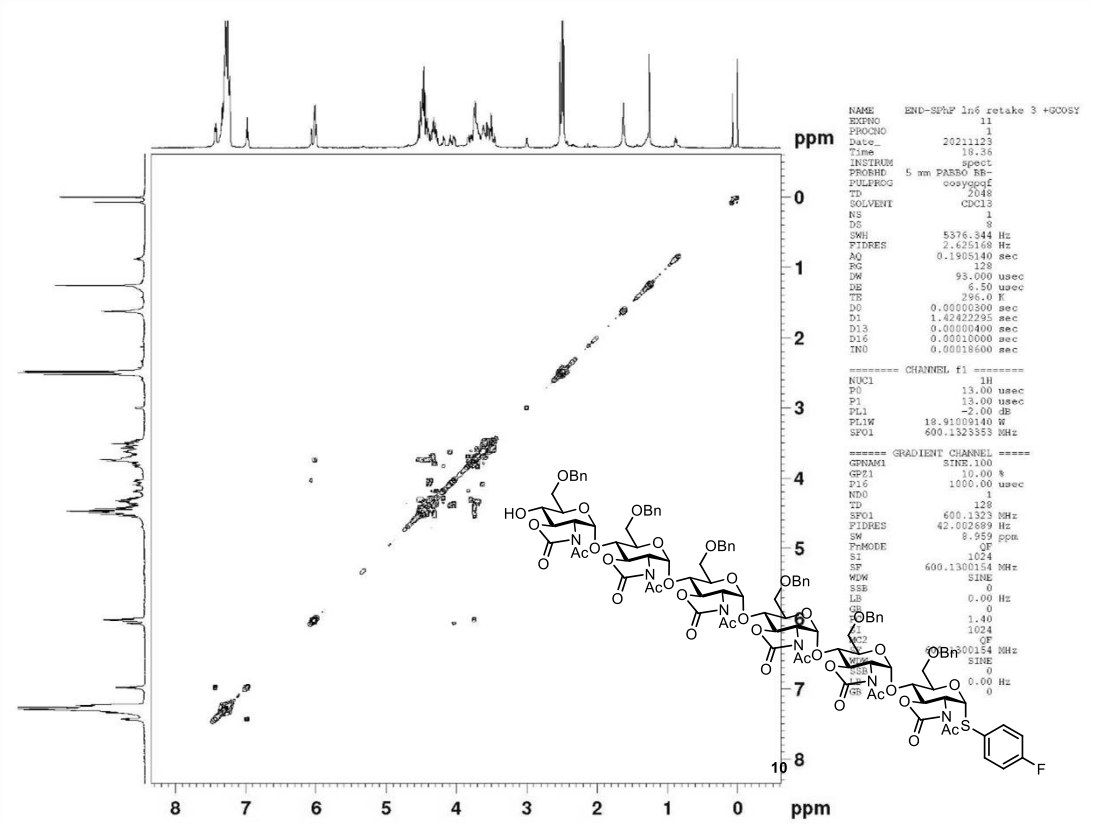
^{1}H NMR



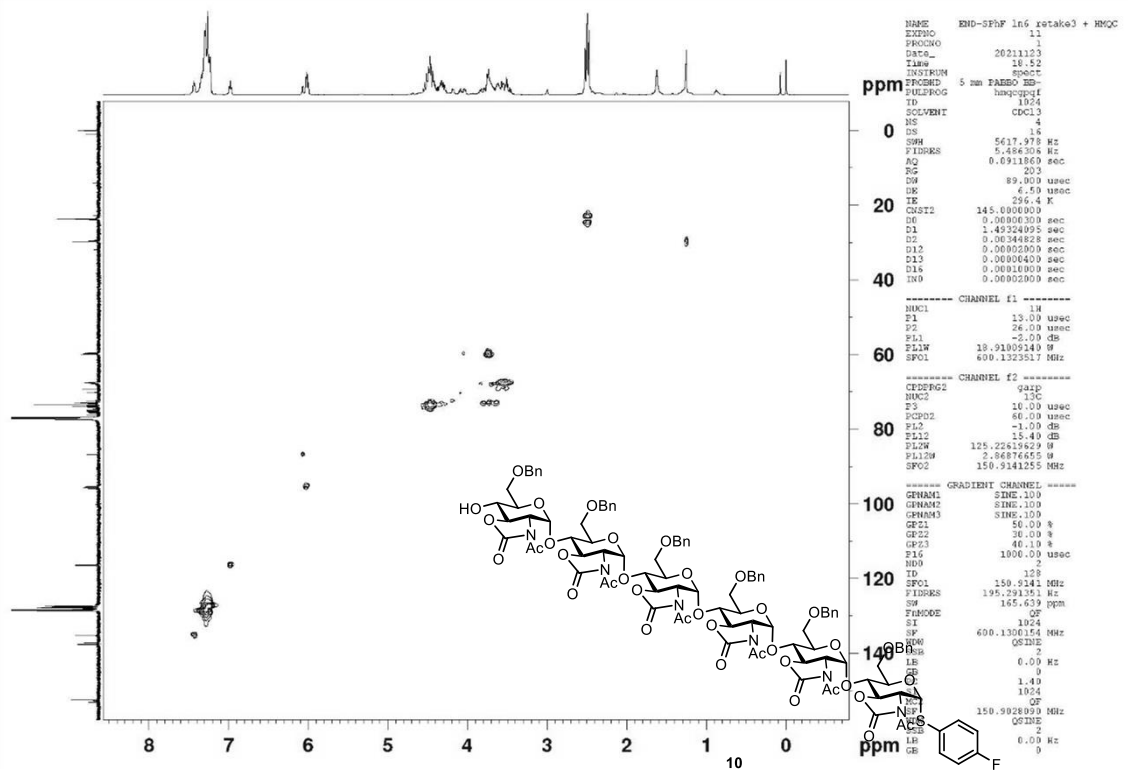
^{13}C NMR



H-H cosy

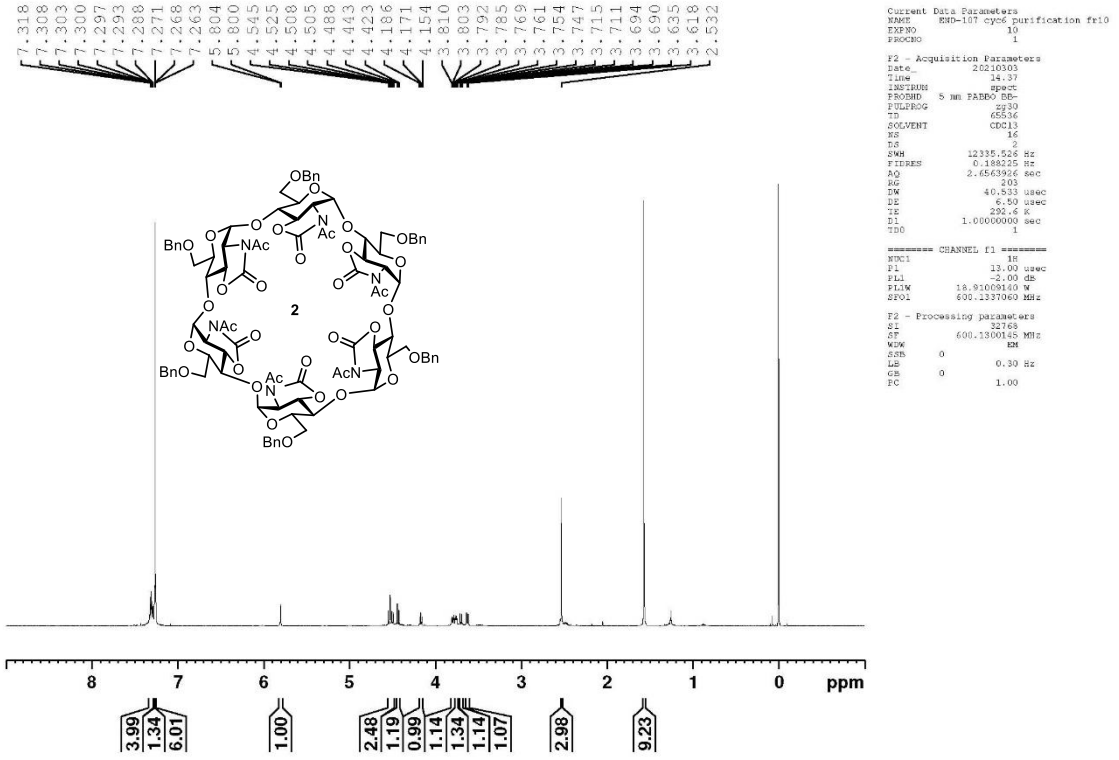


HMQC



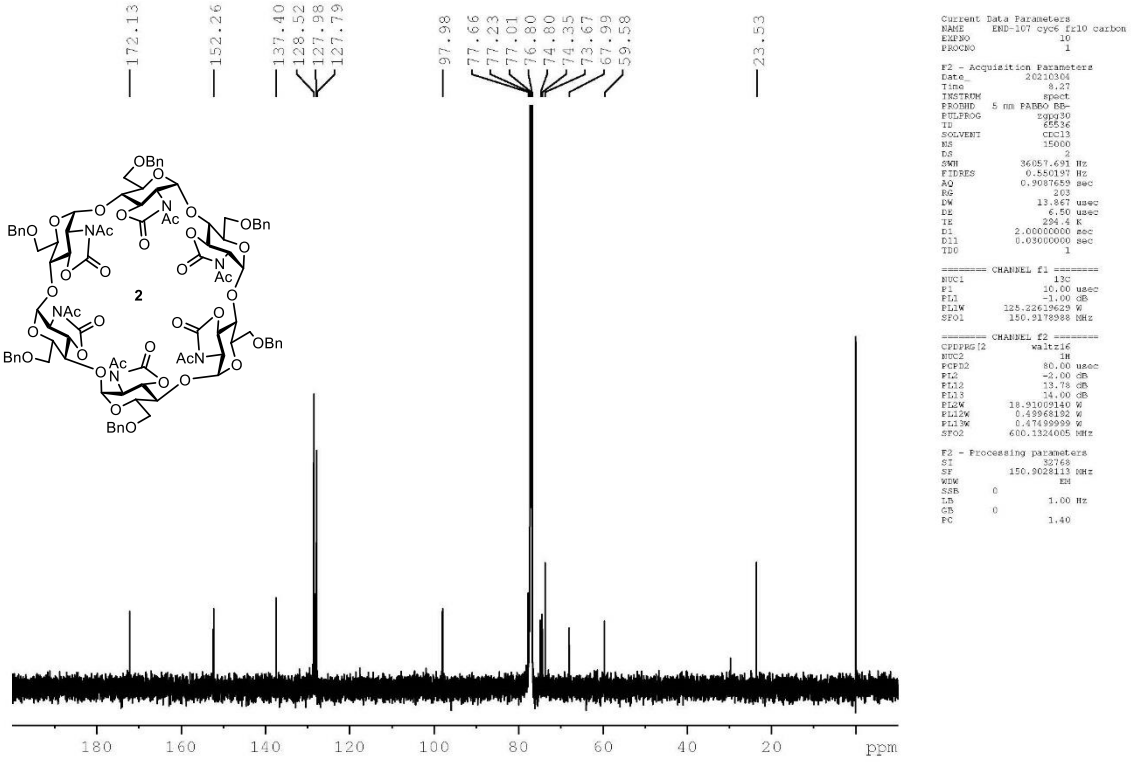
9. ^{1}H, ^{13}C NMR, H-H COSY, and HMQC spectra of cyclic oligosaccharides

^{1}H NMR



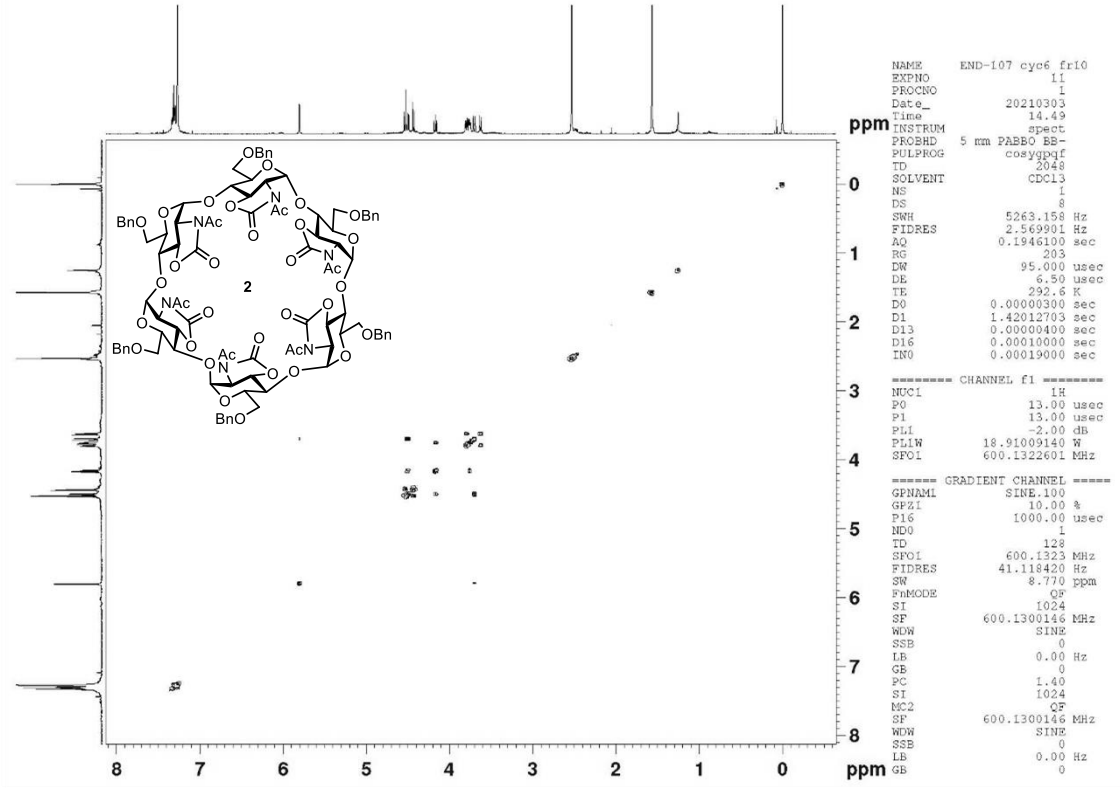

```
Current Data Parameters
NAME      END-107 cyc6 purification fr10
EXPNO              10
PROCNO              1

F2 - Acquisition Parameters
Date_         20210303
Time              14.37
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zg30
TD              65536
SOLVENT          CDCl3
NS                 16
DS                  2
SWH          12335.526 Hz
FIDRES        0.188225 Hz
AQ            2.6563926 sec
RG                203
DW             40.533 usec
DE              6.50 usec
TE             292.6 K
D1          1.00000000 sec
TD0                 1

======== CHANNEL f1 ========
NUC1               1H
P1              13.00 usec
PL1             -2.00 dB
PL1W       18.91009140 W
SFO1       600.1337060 MHz

F2 - Processing parameters
SI              32768
SF         600.1300145 MHz
WDW                EM
SSB                0
LB              0.30 Hz
GB                 0
PC              1.00
```

^{13}C NMR



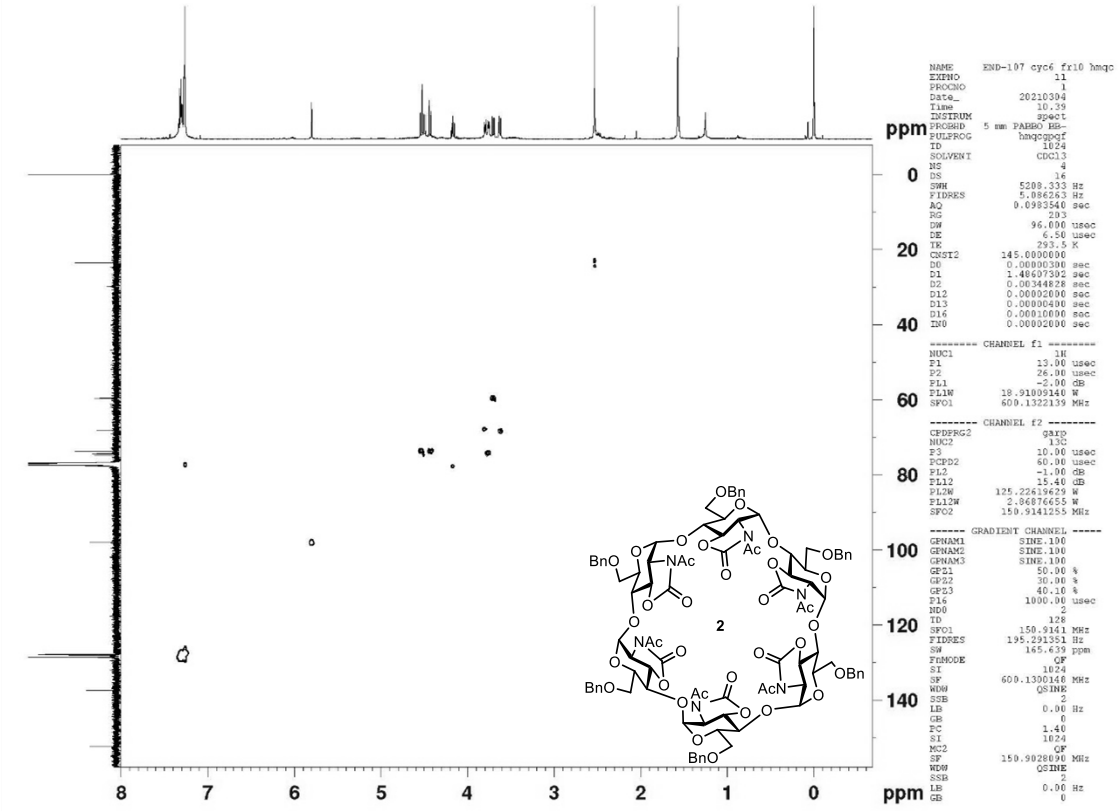

```
Current Data Parameters
NAME      END-107 cyc6 fr10 carbon
EXPNO              10
PROCNO              1

F2 - Acquisition Parameters
Date_         20210304
Time               8.27
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD              65536
SOLVENT          CDCl3
NS              15000
DS                  2
SWH          36057.691 Hz
FIDRES        0.550197 Hz
AQ            0.9087659 sec
RG                203
DW             13.867 usec
DE              6.50 usec
TE             294.4 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                 1

======== CHANNEL f1 ========
NUC1              13C
P1              10.00 usec
PL1             -1.00 dB
PL1W       125.22619629 W
SFO1       150.9178988 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2               1H
PCPD2           80.00 usec
PL2             -2.00 dB
PL12            13.76 dB
PL13            14.00 dB
PL2W       18.91009140 W
PL12W       0.49968192 W
PL13W       0.47499999 W
SFO2       600.1324005 MHz

F2 - Processing parameters
SI              32768
SF         150.9028113 MHz
WDW                EM
SSB                0
LB              1.00 Hz
GB                 0
PC              1.40
```

H-H cosy



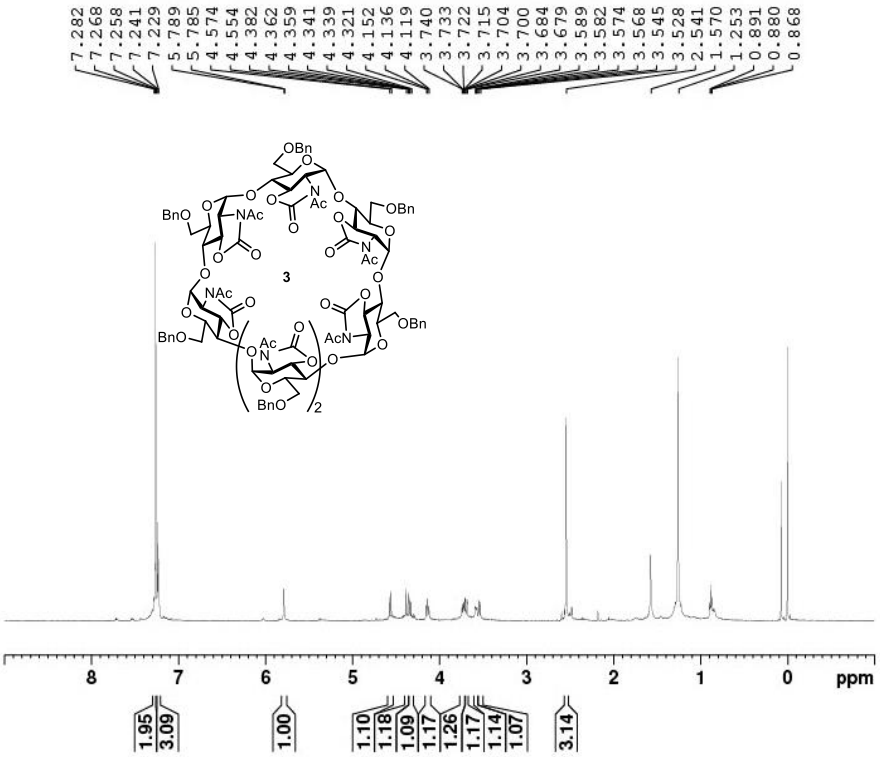

```
NAME        END-107 cyc6 fr10
EXPNO               11
PROCNO               1
Date_         20210303
Time             14.49
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG      cosygpqf
TD                2048
SOLVENT          CDCl3
NS                   1
DS                   8
SWH           5263.158 Hz
FIDRES        2.569901 Hz
AQ            0.1946100 sec
RG                 203
DW               95.000 usec
DE                6.50 usec
TE               292.6 K
D0            0.00000300 sec
D1            1.42012703 sec
D13           0.00000400 sec
D16           0.00010000 sec
IN0           0.00019000 sec

======== CHANNEL f1 ========
NUC1                1H
P0                13.00 usec
P1                13.00 usec
PL1               -2.00 dB
PL1W         18.91009140 W
SFO1         600.1322601 MHz

====== GRADIENT CHANNEL =====
GPNAM1          SINE.100
GPZ1               10.00 %
P16              1000.00 usec
ND0                   1
TD                  128
SFO1           600.1323 MHz
FIDRES        41.118420 Hz
SW                8.770 ppm
FnMODE              QF
SI                1024
SF           600.1300146 MHz
WDW               SINE
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.40
SI                1024
MC2                 QF
SF           600.1300146 MHz
WDW               SINE
SSB                   0
LB                 0.00 Hz
GB                    0
```

HMQC



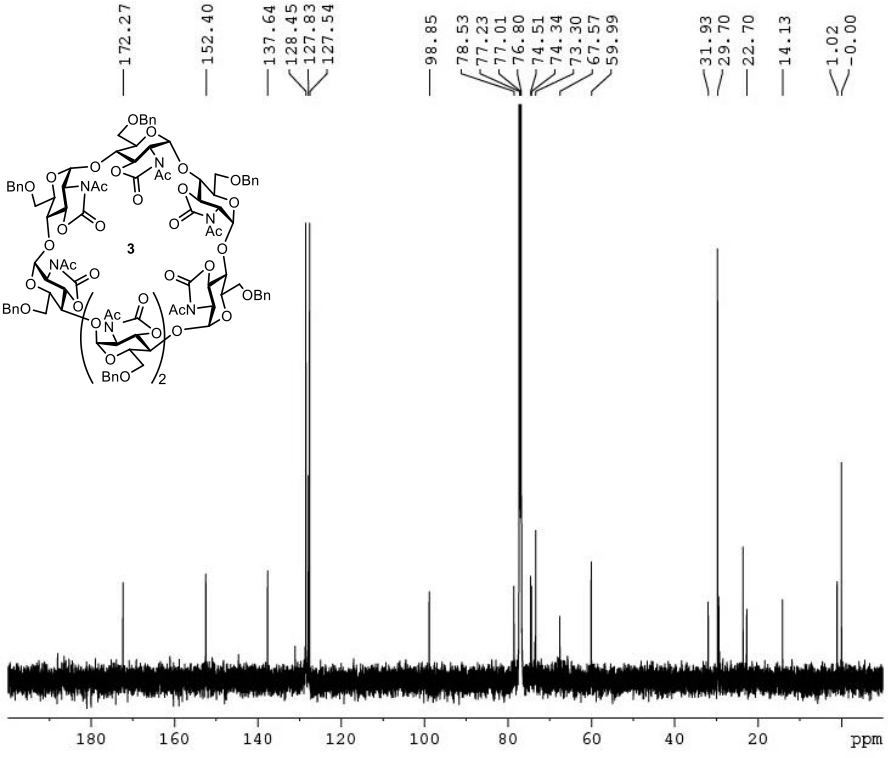

```
NAME        END-107 cyc6 fr10 hmqc
EXPNO               11
PROCNO               1
Date_         20210304
Time             10.39
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG      hmqcgpqf
TD                1024
SOLVENT          CDCl3
NS                   4
DS                  16
SWH           5208.333 Hz
FIDRES        5.086263 Hz
AQ            0.0983540 sec
RG                 203
DW               96.000 usec
DE                6.50 usec
TE               293.5 K
CNST2        145.0000000
D0            0.00000300 sec
D1            1.48607302 sec
D2            0.00344828 sec
D12           0.00002000 sec
D13           0.00000400 sec
D16           0.00010000 sec
IN0           0.00002000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                13.00 usec
P2                26.00 usec
PL1               -2.00 dB
PL1W         18.91009140 W
SFO1         600.1322139 MHz

======== CHANNEL f2 ========
CPDPRG2          garp
NUC2                13C
P3                10.00 usec
PCPD2             60.00 usec
PL2               -1.00 dB
PL12              15.40 dB
PL2W         125.22619629 W
PL12W         2.86876655 W
SFO2         150.9141255 MHz

====== GRADIENT CHANNEL =====
GPNAM1          SINE.100
GPNAM2          SINE.100
GPNAM3          SINE.100
GPZ1               50.00 %
GPZ2               30.00 %
GPZ3               40.10 %
P16              1000.00 usec
ND0                   2
TD                  128
SFO1           150.9141 MHz
FIDRES       195.291351 Hz
SW               165.639 ppm
FnMODE              QF
SI                1024
SF           600.1300148 MHz
WDW              QSINE
SSB                   2
LB                 0.00 Hz
GB                    0
PC                 1.40
SI                1024
MC2                 QF
SF           150.9028098 MHz
WDW              QSINE
SSB                   2
LB                 0.00 Hz
GB                    0
```

^{1}H NMR



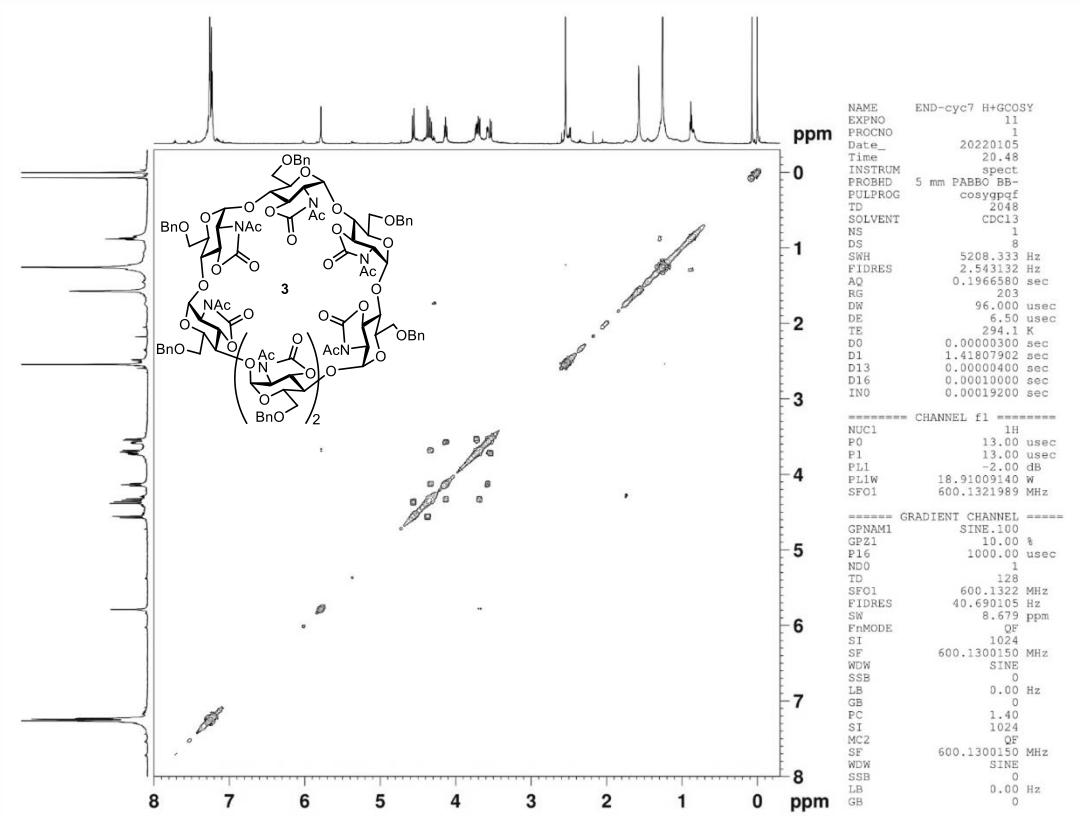

```
Current Data Parameters
NAME      END-cyc7 H+GCOSY
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20220105
Time                20.47
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH             12335.526 Hz
FIDRES          0.188225 Hz
AQ              2.6563926 sec
RG                    203
DW                 40.533 usec
DE                  6.50 usec
TE                 294.1 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  13.00 usec
PL1                 -2.00 dB
PL1W          18.91009140 W
SFO1          600.1337060 MHz

F2 - Processing parameters
SI                  32768
SF            600.1300150 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

^{13}C NMR

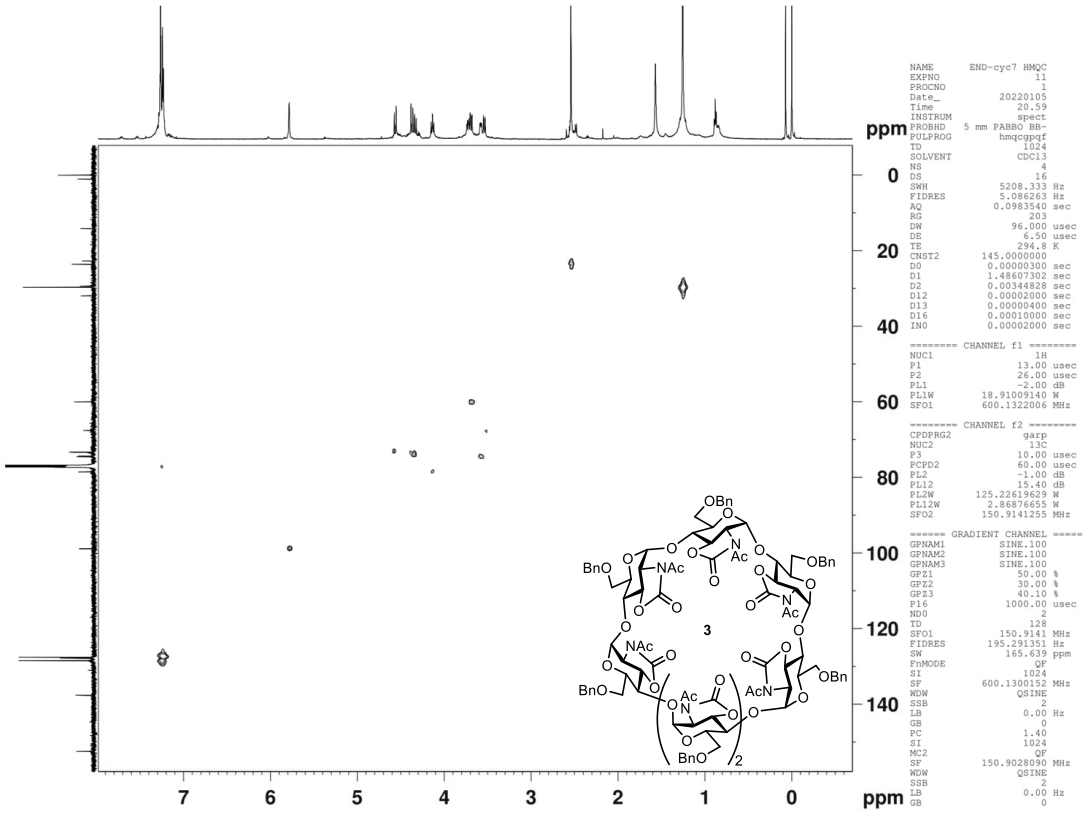



```
Current Data Parameters
NAME          END-cyc7 C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_            20220106
Time                8.54
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                  14000
DS                      2
SWH             36057.691 Hz
FIDRES          0.550197 Hz
AQ              0.9087659 sec
RG                    203
DW                 13.867 usec
DE                  6.50 usec
TE                 295.3 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.00 usec
PL1                 -1.00 dB
PL1W          125.22619629 W
SFO1          150.9178988 MHz

======== CHANNEL f2 ========
CPDPRG[2            waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                 -2.00 dB
PL12                13.78 dB
PL13                14.00 dB
PL2W          18.91009140 W
PL12W          0.49968192 W
PL13W          0.47499999 W
SFO2          600.1324005 MHz

F2 - Processing parameters
SI                  32768
SF            150.9028113 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

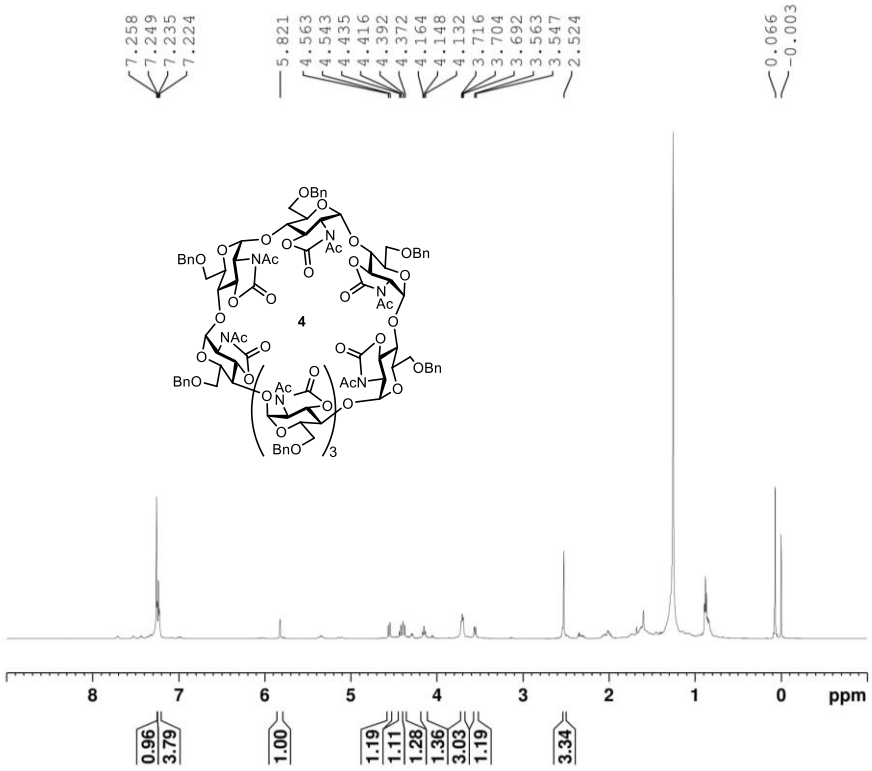
H-H cosy


ppm
8 7 6 5 4 3 2 1 0 ppm
3
NAME END-cyc7 H+GCOSY
EXPNO 11
PROCNO 1
Date_ 20220105
Time 20.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG cosygpqf
TD 2048
SOLVENT CDC13
NS 1
DS 8
SWH 5208.333 Hz
FIDRES 2.543132 Hz
AQ 0.1966580 sec
RG 203
DW 96.000 usec
DE 6.50 usec
TE 294.1 K
D0 0.00000300 sec
D1 1.41807902 sec
D13 0.00000400 sec
D16 0.00010000 sec
IN0 0.00019200 sec
======== CHANNEL f1 ========
NUC1 1H
P0 13.00 usec
P1 13.00 usec
PL1 -2.00 dB
PL1W 18.91009140 W
SFO1 600.1321989 MHz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPZ1 10.00 %
P16 1000.00 usec
ND0 1
TD 128
SFO1 600.1322 MHz
FIDRES 40.690105 Hz
SW 8.679 ppm
FnMODE QF
SI 1024
SF 600.1300150 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
SI 1024
MC2 QF
SF 600.1300150 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

HMQC

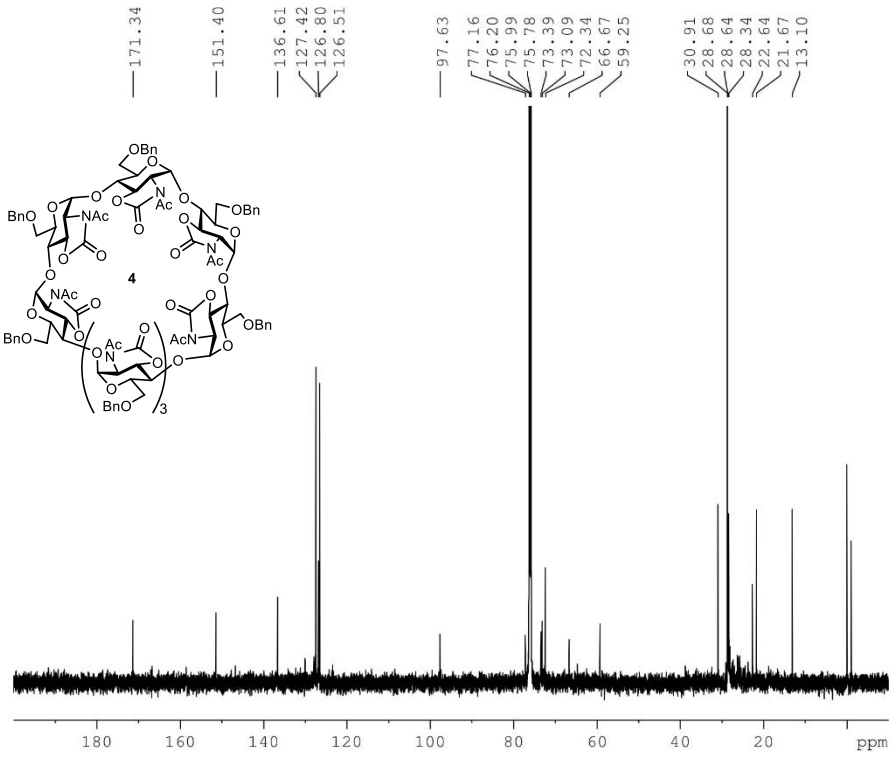

ppm
7 6 5 4 3 2 1 0 ppm
3
NAME END-cyc7 HMQC
EXPNO 11
PROCNO 1
Date_ 20220105
Time 20.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG hmqcgpqf
TD 1024
SOLVENT CDC13
NS 4
DS 16
SWH 5208.333 Hz
FIDRES 5.086263 Hz
AQ 0.0983540 sec
RG 203
DW 96.000 usec
DE 6.50 usec
TE 294.8 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.48607302 sec
D2 0.00344828 sec
D12 0.00002000 sec
D13 0.00000400 sec
D16 0.00010000 sec
IN0 0.00002000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
P2 26.00 usec
PL1 -2.00 dB
PL1W 18.91009140 W
SFO1 600.1322006 MHz
======== CHANNEL f2 ========
CPDPRG2 garp
NUC2 13C
P3 10.00 usec
PCPD2 60.00 usec
PL2 -1.00 dB
PL12 15.40 dB
PL2W 125.22619629 W
PL12W 2.86876655 W
SFO2 150.9141255 MHz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec
ND0 2
TD 128
SFO1 150.9141 MHz
FIDRES 195.291351 Hz
SW 165.639 ppm
FnMODE QF
SI 1024
SF 600.1300152 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40
SI 1024
MC2 QF
SF 150.9028090 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

^{1}H NMR



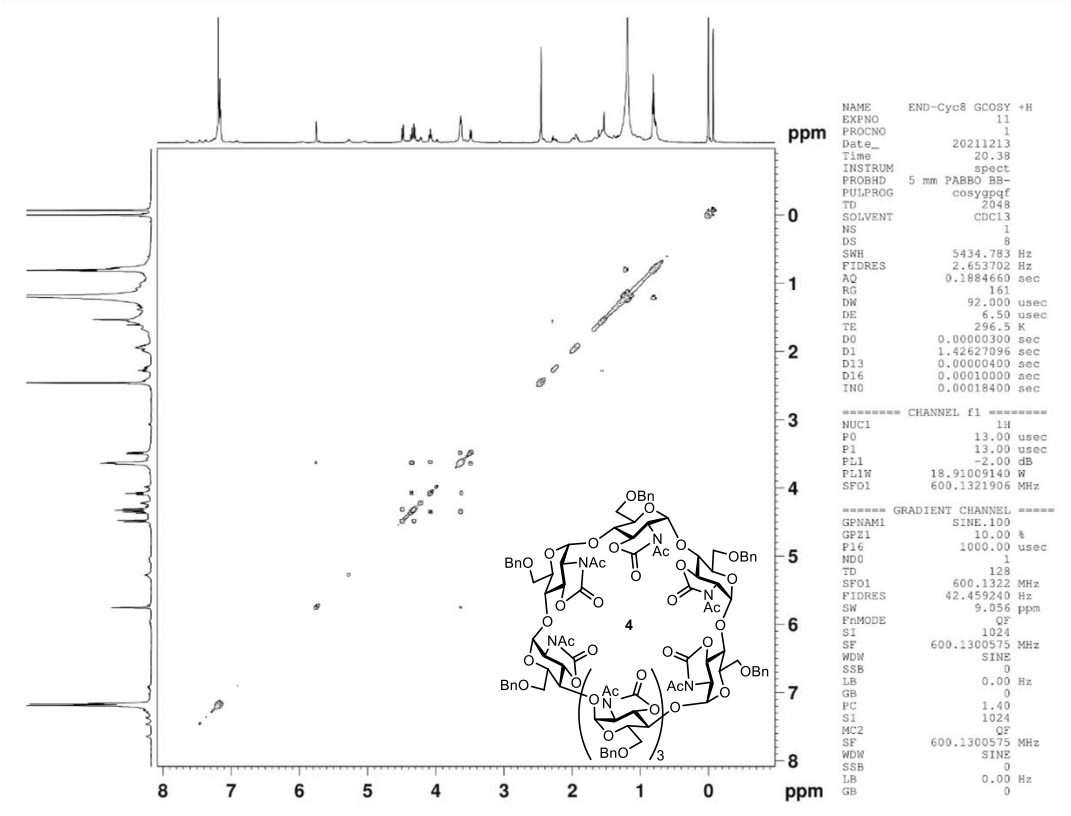

```
Current Data Parameters
NAME      END-Cyc8 GCOSY +H
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20211213
Time              20.37
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    64
DS                     2
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ             2.6563926 sec
RG                   203
DW               40.533 usec
DE                6.50 usec
TE                296.5 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 13.00 usec
PL1                -2.00 dB
PL1W        18.91009140 W
SFO1       600.1337060 MHz

F2 - Processing parameters
SI                 32768
SF           600.1300175 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{13}C NMR



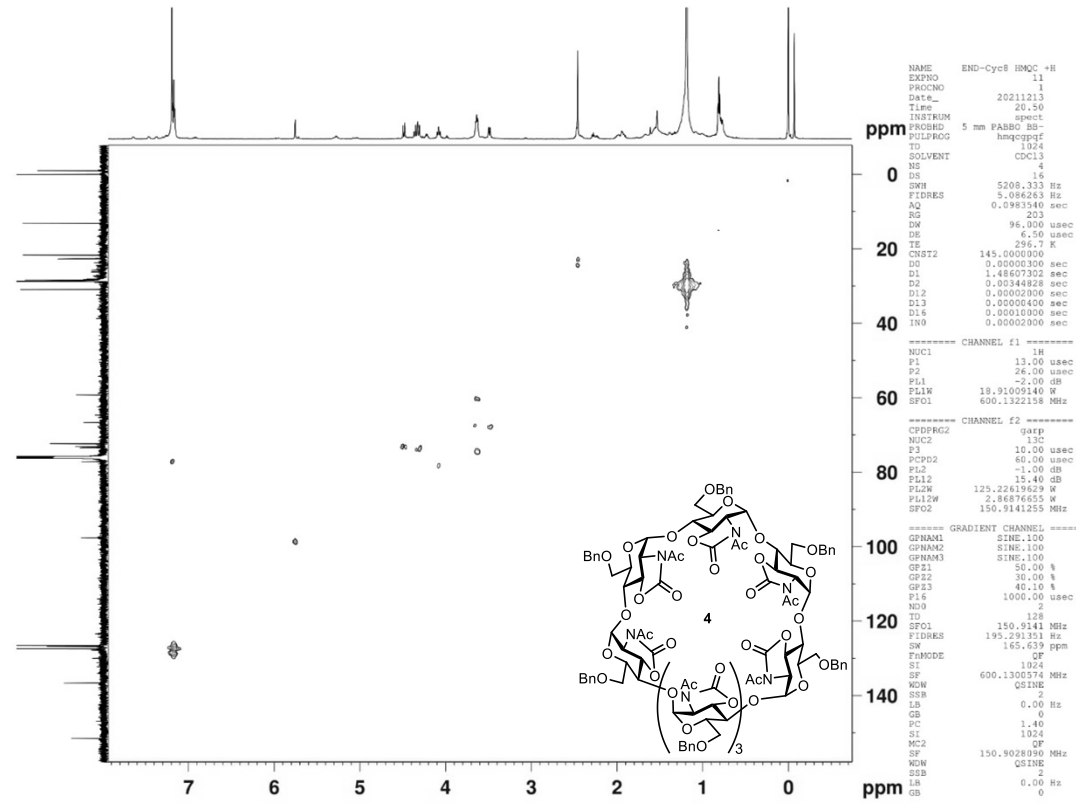

```
Current Data Parameters
NAME          END-Cyc8 C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211214
Time               8.45
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                 14000
DS                     2
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ             0.9087659 sec
RG                   203
DW               13.867 usec
DE                6.50 usec
TE                296.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.00 usec
PL1                -1.00 dB
PL1W       125.22619629 W
SFO1       150.9178988 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               13.78 dB
PL13               14.00 dB
PL2W        18.91009140 W
PL12W        0.49968192 W
PL13W        0.47499999 W
SFO2       600.1324005 MHz

F2 - Processing parameters
SI                 32768
SF           150.9029654 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

H-H cosy




```
NAME      END-Cyc8 GCOSY +H
EXPNO              11
PROCNO              1
Date_        20211213
Time            20.38
INSTRUM         spect
PROBHD   5 mm PABBO BB-
PULPROG      cosygpqf
TD              2048
SOLVENT          CDCl3
NS                  1
DS                  8
SWH         5434.783 Hz
FIDRES      2.653702 Hz
AQ          0.1884660 sec
RG                161
DW             92.000 usec
DE              6.50 usec
TE             296.5 K
D0          0.00000300 sec
D1          1.42627096 sec
D13         0.00000400 sec
D16         0.00010000 sec
IN0         0.00018400 sec

======== CHANNEL f1 ========
NUC1               1H
P0              13.00 usec
P1              13.00 usec
PL1             -2.00 dB
PLW        18.91009140 W
SFO1      600.1321906 MHz

====== GRADIENT CHANNEL =====
GPNAM1        SINE.100
GPZ1             10.00 %
P16            1000.00 usec
ND0                 1
TD                128
SFO1        600.1322 MHz
FIDRES      42.459240 Hz
SW           9.056 ppm
FnMODE             QF
SI               1024
SF        600.1300575 MHz
WDW              SINE
SSB                 0
LB              0.00 Hz
GB                  0
PC               1.40
SI               1024
MC2                QF
SF        600.1300575 MHz
WDW              SINE
SSB                 0
LB              0.00 Hz
GB                  0
```

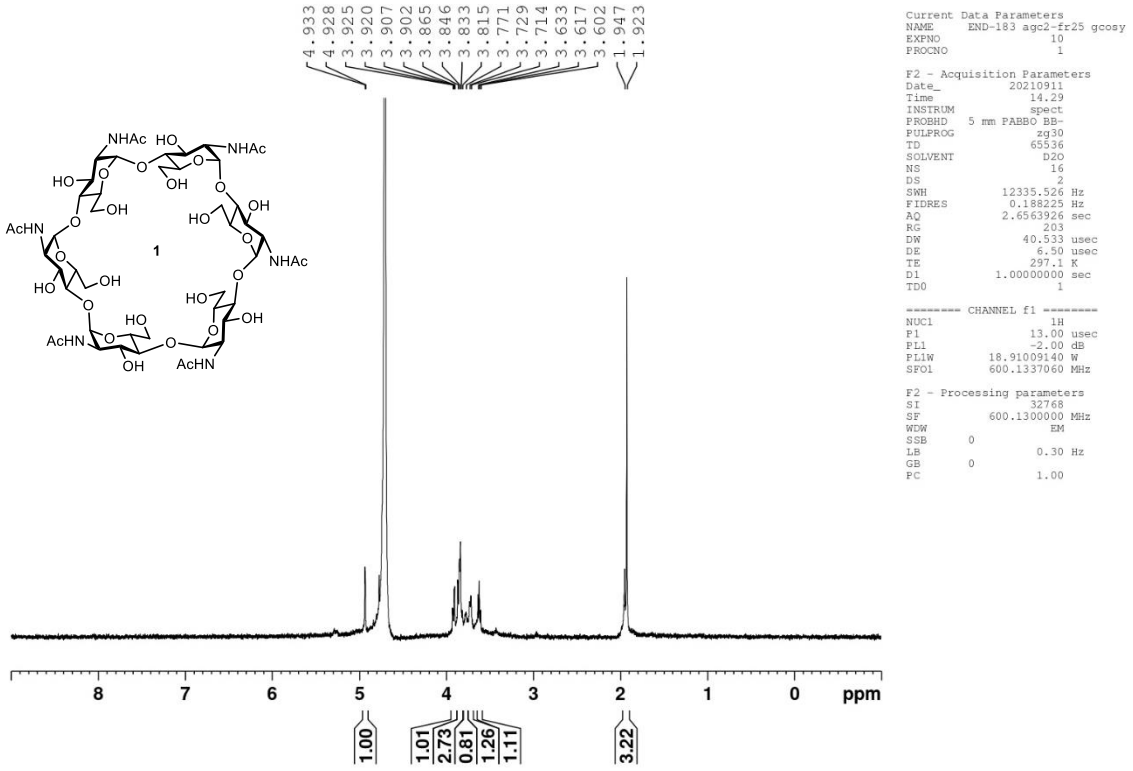
HMQC




```
NAME      END-Cyc8 HMQC +H
EXPNO              11
PROCNO              1
Date_        20211213
Time            20.50
INSTRUM         spect
PROBHD   5 mm PABBO BB-
PULPROG      hmqcgpqf
TD                1024
SOLVENT          CDCl3
NS                  4
DS                 16
SWH          5208.333 Hz
FIDRES       5.086263 Hz
AQ           0.0983540 sec
RG                203
DW              96.000 usec
DE               6.50 usec
TE              296.7 K
CNST2       145.0000000
D0          0.00000300 sec
D1          1.48607302 sec
D2          0.00344828 sec
D12         0.00002000 sec
D13         0.00000400 sec
D16         0.00010000 sec
IN0         0.00002000 sec

======== CHANNEL f1 ========
NUC1               1H
P1              13.00 usec
P2              26.00 usec
PL1             -2.00 dB
PLW        18.91009140 W
SFO1      600.1322158 MHz

======== CHANNEL f2 ========
CPDPRG2          garp
NUC2              13C
P3              10.00 usec
PCPD2           60.00 usec
PL2             -1.00 dB
PL12            15.40 dB
PLW2       125.22619629 W
PL12W        2.86876655 W
SFO2      150.9141255 MHz

====== GRADIENT CHANNEL =====
GPNAM1        SINE.100
GPNAM2        SINE.100
GPNAM3        SINE.100
GPZ1             50.00 %
GPZ2             30.00 %
GPZ3             40.10 %
P16            1000.00 usec
ND0                 2
TD                128
SFO1        150.9141 MHz
FIDRES      195.291351 Hz
SW           165.639 ppm
FnMODE             QF
SI               1024
SF        600.1300574 MHz
WDW             QSINE
SSB                 2
LB              0.00 Hz
GB                  0
PC               1.40
SI               1024
MC2                QF
SF        150.9028090 MHz
WDW             QSINE
SSB                 2
LB              0.00 Hz
GB                  0
```

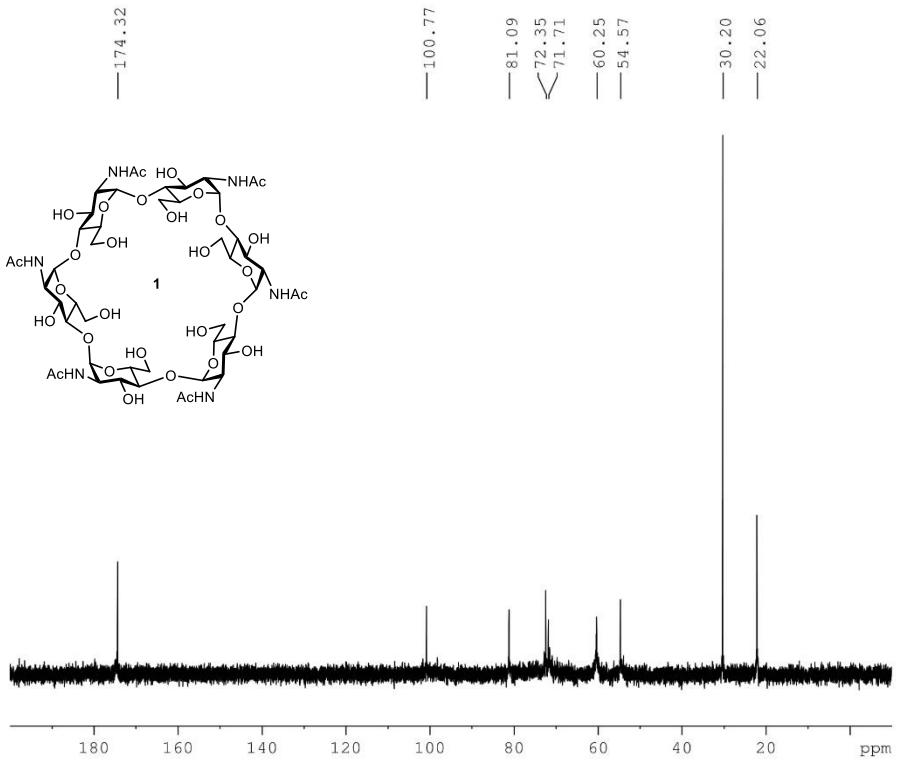
10. ^{1}H, ^{13}C NMR, H-H COSY and HMQC spectra of deprotected cyclic hexasaccharide

^{1}H NMR



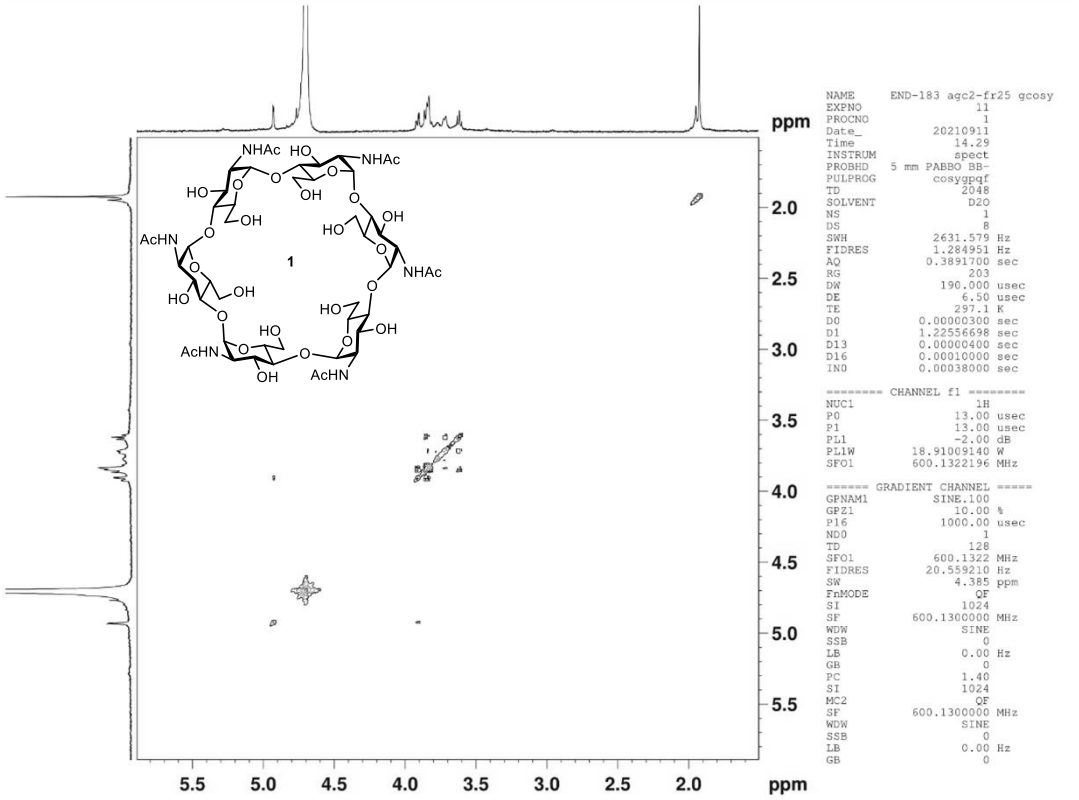

```
Current Data Parameters
NAME      END-183 agc2-fr25 gcosy
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210911
Time              14.29
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT              D2O
NS                    16
DS                     2
SWH            12335.526 Hz
FIDRES          0.188225 Hz
AQ              2.6563926 sec
RG                   203
DW                40.533 usec
DE                 6.50 usec
TE                297.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 13.00 usec
PL1                -2.00 dB
PL1W         18.91009140 W
SFO1         600.1337060 MHz

F2 - Processing parameters
SI                 32768
SF            600.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{13}C NMR

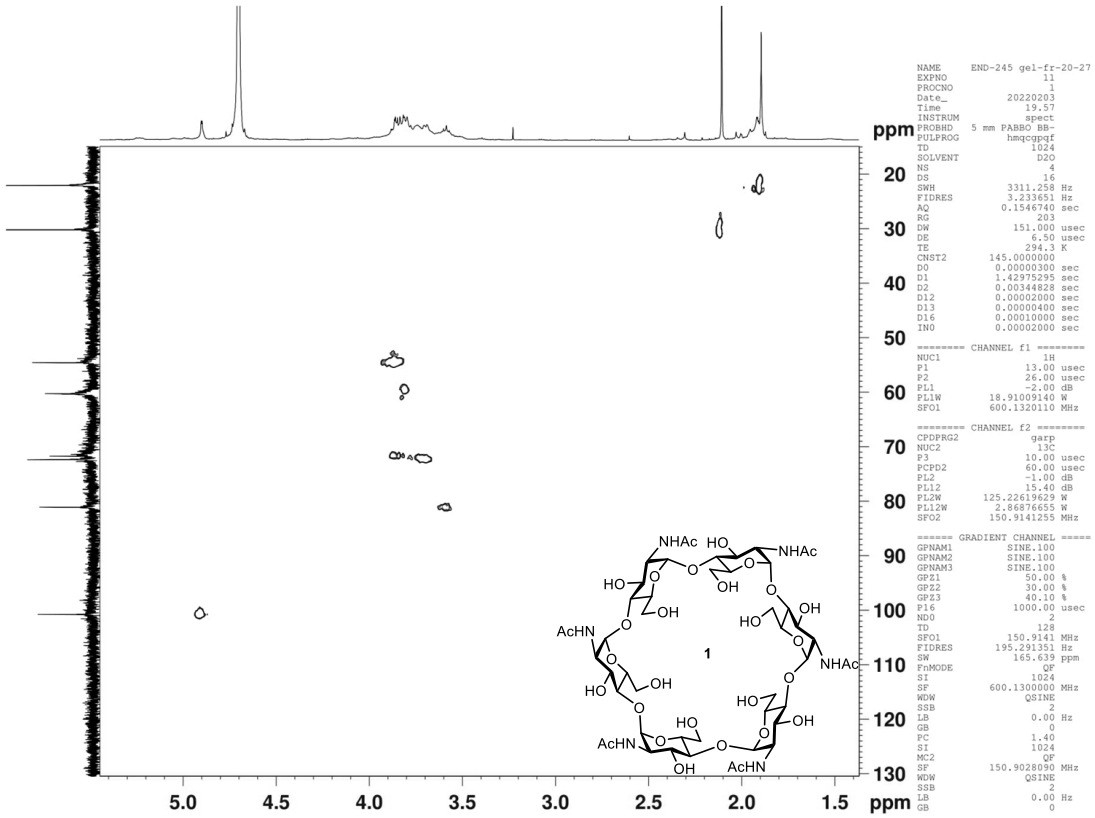



```
Current Data Parameters
NAME      END-245 gel-fr-20-27 13C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20220204
Time              7.49
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT              D2O
NS                 14000
DS                     2
SWH            36057.691 Hz
FIDRES          0.550197 Hz
AQ              0.9087659 sec
RG                   203
DW                13.867 usec
DE                 6.50 usec
TE                295.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1               -1.00 dB
PL1W        125.22619629 W
SFO1         150.9178988 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               13.78 dB
PL13               14.00 dB
PL2W         18.91009140 W
PL12W         0.49968192 W
PL13W         0.47499999 W
SFO2         600.1324005 MHz

F2 - Processing parameters
SI                 32768
SF            150.9028090 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

H-H cosy


NAME END-183 agc2-fr25 gcosy
EXPNO 11
PROCNO 1
Date_ 20210911
Time 14.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG cosygpqf
TD 2048
SOLVENT D2O
NS 1
DS 8
SWH 2631.579 Hz
FIDRES 1.284951 Hz
AQ 0.3891700 sec
RG 203
DW 190.000 usec
DE 6.50 usec
TE 297.1 K
D0 0.00000300 sec
D1 1.22556698 sec
D13 0.00000400 sec
D16 0.00010000 sec
IN0 0.00038000 sec
======== CHANNEL f1 ========
NUC1 1H
P0 13.00 usec
P1 13.00 usec
PL1 -2.00 dB
PL1W 18.91009140 W
SFO1 600.1322196 MHz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPZ1 10.00 %
P16 1000.00 usec
ND0 1
TD 128
SFO1 600.1322 MHz
FIDRES 20.559210 Hz
SW 4.385 ppm
FnMODE QF
SI 1024
SF 600.1300000 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
SI 1024
MC2 QF
SF 600.1300000 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
ppm
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 ppm
NHAc HO NHAc
HO OH
HO OH
AcHN
NHAc
HO OH
HO OH
HO
AcHN
AcHN
OH
1

HMQC


NAME END-245 gel-fr-20-27
EXPNO 11
PROCNO 1
Date_ 20220203
Time 19.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG hmqcgpqf
TD 1024
SOLVENT D2O
NS 4
DS 16
SWH 3311.258 Hz
FIDRES 3.233651 Hz
AQ 0.1546740 sec
RG 203
DW 151.000 usec
DE 6.50 usec
TE 294.3 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.42975295 sec
D2 0.00344828 sec
D12 0.00002000 sec
D13 0.00000400 sec
D16 0.00010000 sec
IN0 0.00002000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
P2 26.00 usec
PL1 -2.00 dB
PL1W 18.91009140 W
SFO1 600.1320110 MHz
======== CHANNEL f2 ========
CPDPRG2 garp
NUC2 13C
P3 10.00 usec
PCPD2 60.00 usec
PL2 -1.00 dB
PL12 15.40 dB
PL2W 125.22619629 W
PL12W 2.86876655 W
SFO2 150.9141255 MHz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec
ND0 2
TD 128
SFO1 150.9141 MHz
FIDRES 195.291351 Hz
SW 165.639 ppm
FnMODE QF
SI 1024
SF 600.1300000 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40
SI 1024
MC2 QF
SF 150.9028090 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
ppm
20
30
40
50
60
70
80
90
100
110
120
130
5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 ppm
NHAc HO NHAc
HO OH
HO OH
AcHN
NHAc
HO OH
HO OH
HO
AcHN
AcHN
OH
1