

Supporting Information

Conformationally superarmed S-ethyl glycosyl donors as effective building blocks for chemoselective oligosaccharide synthesis in one pot

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Content

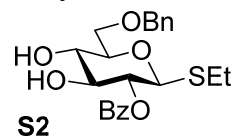
General Experimental	S1
Preparation of Glycosyl Donors	S2
Synthesis of Glycosyl Acceptors	S6
General Procedures for Competition Experiments	S7
Synthesis of Disaccharides	S7
One-Pot One-Addition Trisaccharide Synthesis	S9
X-ray Structure Determination of Disaccharide 10	S11
References	S12
NMR Spectra	S13
X-Ray Data for Disaccharide 10	S45

General Experimental

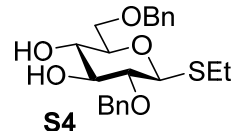
The reactions were performed using commercial reagents and the ACS grade solvents were purified and dried according to standard procedures. Column chromatography was performed on silica gel 60 (70-230 mesh), reactions were monitored by TLC on Kieselgel 60 F₂₅₄. The compounds were detected by examination under UV light and by charring with 10% sulfuric acid in methanol. Solvents were removed under reduced pressure at <40 °C. CH₂Cl₂ was distilled from CaH₂ directly prior to application. Acetonitrile was dried by refluxing with CaH₂ and then distilled and stored over molecular sieves (3 Å). Molecular sieves (3 or 4Å), used for reactions, were crushed and activated *in vacuo* at 390 °C during 8 h in the first instance and then for 2-3 h at 390 °C directly prior to application. Optical rotations were measured using a polarimeter. ¹H NMR spectra were recorded at 300 or 600 MHz, ¹³C NMR spectra were recorded at 75 MHz or 150 MHz. The ¹H chemical shifts are referenced to the signal of the residual CHCl₃ (δ_H = 7.24 ppm). The ¹³C chemical shifts are referenced to the central signal of CDCl₃ (δ_C = 77.23 ppm). HRMS determinations were made with the use of a mass spectrometer with FAB ionization and ion-trap detection.

Preparation of Glycosyl Donors

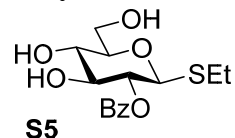
Ethyl 2-*O*-benzoyl-6-*O*-benzyl-1-thio- β -D-glucopyranoside (S2). A mixture of ethyl 2-*O*-benzoyl-4,6-*O*-benzylidene-1-thio- β -D-glucopyranoside (**S1**,¹ 2.56 g, 6.15 mmol) and molecular sieves (3 Å, 3.0 g) in dry THF (80 mL) was stirred under argon for 1 h at rt. NaCNBH₃ (5.15 g, 81.8 mmol) and a 2 M solution of HCl in diethyl ether (40.9 mL, 81.8 mmol) were added and the resulting mixture was stirred for 30 min at rt. After that, the volatiles were removed under reduced pressure and the residue was diluted with CH₂Cl₂ (~150 mL). The solid was filtered off through a pad of Celite and rinsed successively with CH₂Cl₂. The combined filtrate (~250 mL) was washed with water (50 mL), sat. aq. NaHCO₃ (50 mL), and water (3 x 50 mL). The organic phase was separated, dried over MgSO₄, and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (ethyl acetate – hexane gradient elution) to afford the title compound in 82% yield (2.11 g, 5.04 mmol) as a white amorphous solid. Analytical data for **S2**: R_f = 0.25 (ethyl acetate/hexane, 3/7, v/v); $[\alpha]_D^{27}$ -54.4 (c = 1, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ , 1.21 (t, 3H, J = 7.4 Hz, CH₂CH₃), 2.68 (m, 2H, CH₂CH₃), 3.48 (m, 1H, H-5), 3.65-3.78 (m, 3H, H-3, 4, 6b), 3.90 (s, 1H, OH), 3.82 (s, 1H, OH), 4.51 (d, 1H, $J_{1,2}$ = 9.5 Hz, H-1), 4.55 (br. s, 2H, CH₂Ph), 5.06 (dd, 1H, $J_{2,3}$ = 9.5 Hz, H-2), 7.29-8.04 (10H, aromatic) ppm; ¹³C NMR (75 MHz, CDCl₃): δ , 15.2, 24.1, 70.1, 71.8, 73.0, 73.7, 76.7, 78.7, 83.3, 127.9 (x3), 128.5 (x4), 129.7, 130.1 (x2), 133.4, 137.7, 166.4 ppm; HR-FAB MS $[M+Na]^+$ calcd for C₂₂H₂₆NaO₆S⁺ 441.1348, found 441.1334.



Ethyl 2,6-di-*O*-benzyl-1-thio- β -D-glucopyranoside (S4). A mixture of ethyl 2-*O*-benzyl-4,6-*O*-benzylidene-1-thio- β -D-glucopyranoside (**S3**,² 1.58 g, 3.92 mmol) and molecular sieves (3 Å, 2.0 g) in dry THF (65 mL) was stirred under argon for 1 h at rt. NaCNBH₃ (3.29 g, 52.0 mmol) and a 2 M solution of HCl in diethyl ether (26.1 mL, 52.0 mmol) were added and the resulting mixture was stirred for 30 min at rt. After that, the volatiles were removed under reduced pressure and the residue was diluted with CH₂Cl₂ (~50 mL). The solid was filtered off through a pad of Celite and rinsed successively with CH₂Cl₂. The combined filtrate (~100 mL) was washed with water (20 mL), sat. aq. NaHCO₃ (20 mL), and water (3 x 20 mL). The organic phase was separated, dried over MgSO₄, and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (ethyl acetate – hexane gradient elution) to afford the title compound in 98% yield (1.65 g, 4.07 mmol) as a white amorphous solid. Analytical data for **S4**: R_f = 0.25 (ethyl acetate/hexane, 3/7, v/v); $[\alpha]_D^{26.6}$ -28.7 (c = 1, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ , 1.32 (t, 3 H, J = 7.4 Hz, CH₂CH₃), 2.76 (m, 2H, CH₂CH₃), 3.05 (s, 1H, OH), 3.26 (dd, 1H, $J_{2,3}$ = 8.6 Hz, H-2), 3.41-3.62 (m, 3H, H-3, 4, 5), 3.67-3.79 (m, 2H, H-6a, 6b), 4.46 (d, 1H, $J_{1,2}$ = 9.6 Hz, H-1), 4.57 (dd, 2H, 2J = 13.7 Hz, CH₂Ph), 4.81 (dd, 2H, 2J = 10.9 Hz, CH₂Ph), 7.19-7.50 (m, 10H, aromatic) ppm; ¹³C NMR (75 MHz, CDCl₃): δ , 15.3, 25.3, 70.6, 72.0, 73.8, 75.3, 77.8, 78.2, 80.9, 85.0, 127.9 (x2), 128.0, 128.3, 128.5 (x2), 128.7 (x2), 128.8 (x2), 137.9, 138.1 ppm; HR-FAB MS $[M+Na]^+$ calcd for C₂₂H₂₈NaO₅S⁺ 427.1554, found 427.1555.

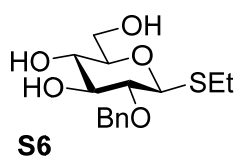


Ethyl 2-*O*-benzoyl-1-thio- β -D-glucopyranoside (S5). Compound **S1** (0.71 g, 1.70 mmol) was dissolved in a mixture of trifluoroacetic acid in wet CH₂Cl₂ (20 mL, 2/0.2/17.8, v/v/v) and the resulting mixture was stirred for 30 min at rt. After that, the reaction mixture was neutralized with trimethylamine (~3 mL) and



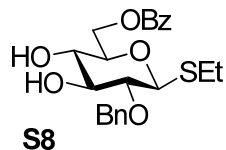
the volatiles were removed under reduced pressure. The residue was purified by column chromatography on silica gel (methanol - dichloromethane gradient elution) to afford the title compound in 86% yield (0.48 g, 1.46 mmol) as a white amorphous solid. Analytical data for **S5**: $R_f = 0.27$ (methanol/dichloromethane, 1/9, v/v); $[\alpha]_D^{19.9} -8.1$ ($c = 1$, MeOH); $^1\text{H NMR}$ (300 MHz, CD_3OD): δ , 1.18 (t, 3H, $J = 7.5$ Hz, CH_2CH_3), 2.70 (m, 2H, CH_2CH_3), 3.38-3.47 (m, 2H, H-5, 4), 3.64-3.73 (m, 2H, H-3, 6a), 3.89 (dd, 1H, $J_{6a,6b} = 12.1$ Hz, $J_{5,6b} = 1.8$ Hz, H-6b), 4.65 (d, 1H, $J_{1,2} = 10.0$ Hz, H-1), 4.97 (dd, 1H, $J_{2,3} = 9.3$ Hz, H-2), 7.45-8.00 (m, 5H, aromatic) ppm; $^{13}\text{C NMR}$ (75 MHz, CD_3OD): δ , 15.2, 24.9, 62.8, 71.6, 74.5, 77.4, 82.2, 84.6, 129.5 ($\times 2$), 130.7 ($\times 2$), 131.5, 134.3, 167.2 ppm; HR-FAB MS $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{15}\text{H}_{20}\text{NaO}_6\text{S}^+$ 351.0879, found 351.0875.

Ethyl 2-O-benzyl-1-thio- β -D-glucopyranoside (S6). Compound **S3** (0.55 g, 1.36 mmol) was dissolved in a mixture of trifluoroacetic acid in wet CH_2Cl_2 (20 mL, 1.5/0.2/14.8, v/v/v) and the resulting mixture was stirred for 30 min at rt. After that, the reaction mixture was neutralized with trimethylamine (~ 2.5 mL) and the volatiles were removed under reduced pressure. The residue was purified by column chromatography on silica gel (methanol - dichloromethane gradient elution) to afford the title compound in 85% yield (0.36 g, 1.16 mmol) as a white amorphous solid. Analytical data for **S6**: $R_f = 0.35$ (methanol/dichloromethane, 1/9, v/v); $[\alpha]_D^{21.5} -45.4$ ($c = 1$, MeOH); $^1\text{H NMR}$ (300 MHz, CD_3OD): δ , 1.17 (t, 3H, $J = 7.4$ Hz, CH_2CH_3), 2.65 (m, 2H, CH_2CH_3), 3.07 (dd, 1H, $J_{2,3} = 8.8$ Hz, H-2), 3.11-3.26 (m, 2H, H-4, 5), 3.39 (dd, 1H, $J_{3,4} = 8.8$ Hz, H-3), 3.54 (dd, 1H, $J_{5,6a} = 5.5$ Hz, $J_{6a,6b} = 12.0$ Hz, H-6a), 3.74 (dd, 1H, $J_{5,6b} = 2.1$ Hz, H-6b), 4.36 (d, 1H, $J_{1,2} = 9.7$ Hz, H-1), 4.70 (s, 2H, CH_2Ph), 7.10-7.32 (m, 5H, aromatic) ppm; $^{13}\text{C NMR}$ (75 MHz, CD_3OD): δ , 15.5, 25.5, 63.0, 71.8, 76.2, 79.8, 82.0, 82.9, 85.9, 128.8, 129.3 ($\times 2$), 129.5 ($\times 2$), 139.9 ppm; HR-FAB MS $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{15}\text{H}_{22}\text{NaO}_5\text{S}^+$ 337.1086, found 337.1082.



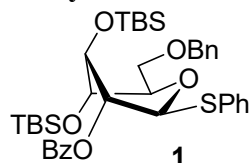
Ethyl 2,6-di-O-benzoyl-1-thio- β -D-glucopyranoside (S7). A solution of BzCN (0.08 g, 0.81 mmol) in dry CH_3CN (10.0 mL) was added dropwise to a solution of **S5** (0.19 g, 0.58 mmol) and triethyl amine (6.0 mL) in dry CH_3CN (10.0 mL), and the resulting mixture was stirred under argon for 3 h at -30 °C. MeOH (~ 1 mL) was added and the volatiles were removed under reduced pressure. The residue was purified by column chromatography on silica gel (ethyl acetate - hexane gradient elution) to afford the title compound in 70% yield (0.175 g, 0.41 mmol) as a white amorphous solid. Analytical data for **S7**: $R_f = 0.37$ (ethyl acetate/hexane, 1/1, v/v); $[\alpha]_D^{26.6} -7.7$ ($c = 1$, MeOH); $^1\text{H NMR}$ (300 MHz, CDCl_3): δ , 1.24 (t, 3H, $J = 7.4$ Hz, CH_2CH_3), 2.71 (m, 2H, CH_2CH_3), 3.16 (d, 1H, $J = 3.9$ Hz, OH), 3.52 (d, 1H, $J = 3.1$ Hz, OH), 3.57-3.72 (m, 2H, $J_{5,6b} = 4.2$ Hz, H-4, 5), 3.84 (ddd, 1H, $J_{3,4} = 9.3$ Hz, H-3), 4.57 (dd, 1H, $J_{6a,6b} = 12.2$ Hz, 6a), 4.63 (d, 1H, $J_{1,2} = 9.8$ Hz, H-1), 4.74 (dd, 1H, H-6b), 5.11 (dd, 1H, $J_{2,3} = 9.3$ Hz, H-2), 7.51-8.04 (m, 10H, aromatic) ppm; $^{13}\text{C NMR}$ (75 MHz, CDCl_3): δ , 15.2, 24.3, 63.9, 70.7, 73.0, 76.7, 78.2, 83.6, 128.7 ($\times 4$), 129.6, 129.7, 130.1 ($\times 2$), 130.2 ($\times 2$), 133.6, 133.7, 166.5, 167.5 ppm; HR-FAB MS $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{22}\text{H}_{24}\text{NaO}_7\text{S}^+$ 455.1141, found 455.1145.

Ethyl 6-O-benzoyl-2-O-benzyl-1-thio- β -D-glucopyranoside (S8). A solution of BzCN (0.138 g, 1.02 mmol) in dry CH_3CN (15.0 mL) was added dropwise to a solution of **S6** (0.305 g, 0.970 mmol) and triethylamine (10.0 mL) in dry CH_3CN (15.0 mL) at -30 °C and the resulting mixture



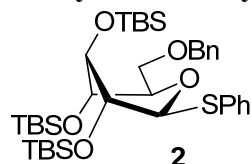
was stirred under argon for 3 h at that temperature. After that, MeOH (1.0 mL) was added to the reaction mixture and the volatiles were removed under reduced pressure. The residue was purified by column chromatography on silica gel (ethyl acetate – hexane gradient elution) to afford the title compound in 75% yield (0.30 g, 0.727 mmol) as a white amorphous solid. Analytical data for **S8**: $R_f = 0.34$ (ethyl acetate/hexane, 1/1, v/v); $[\alpha]_D^{21.5} -31.3$ ($c = 1$, CHCl_3); $^1\text{H NMR}$ (300 MHz, CDCl_3): δ , 1.30 (t, 3H, $J = 7.4$ Hz, CH_2CH_3), 2.75 (m, 2H, CH_2CH_3), 3.11 (br. s, 1H, OH), 3.26 (dd, 1H, $J_{2,3} = 9.7$ Hz, H-2), 3.39-3.56 (m, 3H, H-4, 5, OH), 3.58-3.64 (m, 1H, H-3), 4.48 (d, 1H, $J_{1,2} = 9.7$ Hz, H-1), 4.51-4.60 (m, 2H, H-6a, 6b), 4.80 (dd, $^2J = 10.9$ Hz, CH_2Ph), 8.73-7.89 (m, 10H, aromatic) ppm; $^{13}\text{C NMR}$ (75 MHz, CDCl_3): δ , 15.3, 25.3, 64.3, 70.2, 75.3, 77.8, 78.0, 80.9, 85.1, 128.3, 128.5 ($\times 2$), 128.6 ($\times 2$), 128.8 ($\times 2$), 129.8, 130.0 ($\times 2$), 138.0, 133.4, 167.2 ppm; HR-FAB MS $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{22}\text{H}_{24}\text{NaO}_7\text{S}^+$ 441.1347, found 441.1346.

Phenyl 2-O-benzoyl-6-O-benzyl-3,4-di-O-tert-butyldimethylsilyl-1-thio- β -D-lucopyranoside (1).



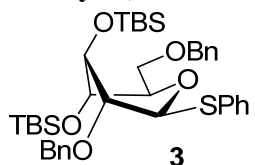
The synthesis of the title compound was performed in accordance with the reported procedure and its analytical data was in accordance with that previously described.³

Phenyl 6-O-benzyl-2,3,4-tri-O-tert-butyldimethylsilyl-1-thio- β -D-glucopyranoside (2).



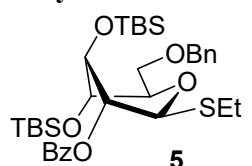
The synthesis of the title compound was performed in accordance with the reported procedure and its analytical data was in accordance with that previously described.⁴

Phenyl 2,6-di-O-benzyl-3,4-di-O-tert-butyldimethylsilyl-1-thio- β -D-glucopyranoside (3).



The synthesis of the title compound was performed in accordance with the reported procedure and its analytical data was in accordance with that previously described.³

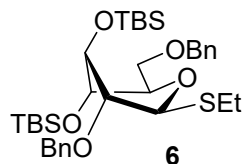
Ethyl 2-O-benzoyl-6-O-benzyl-3,4-di-O-tert-butyldimethylsilyl-1-thio- β -D-glucopyranoside (5).



TBSOTf (2.7 mL, 12.0 mmol) was added to a solution of **S2** (1.67 g, 4.0 mmol) in 2,6-lutidine (12.0 mL) and the resulting mixture was heated at 130 °C for 1 h. After that, the reaction mixture was allowed to cool to rt, diluted with ethyl acetate (~250 mL), and washed with 1 M aq. HCl (3 \times 50 mL), water (50 mL), sat. aq. NaHCO_3 (50 mL), and brine (2 \times 50 mL). The organic phase was separated, dried with MgSO_4 , and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (ethyl acetate – toluene gradient elution) to afford the title compound in 92% yield (2.35 g, 3.64 mmol) as a colorless syrup. Analytical data for **5**: $R_f = 0.71$ (ethyl acetate/ toluene, 1/9, v/v); $[\alpha]_D^{26.9} -3.7$ ($c = 1$, CHCl_3); $^1\text{H NMR}$ (300 MHz, CDCl_3): δ , 0.05, 0.06, 0.07, 0.08 (4 s, 12H, 2 \times SiMe_2), 0.83, 0.85 (2 s, 18H, 2 \times Si^tBu), 1.25 (t, 3H, $J = 7.4$ Hz, CH_2CH_3), 2.71 (m, 2H, CH_2CH_3), 3.65 (dd, 1H, $J_{6a,6b} = 9.7$ Hz, H-6a), 3.74-3.82 (m, 2H, H-4, 6b), 3.84-3.91 (m, 2H, $J_{5,6a} = 6.6$ Hz, H-3, 5), 4.58 (dd, 2H, $^2J = 12.0$ Hz, CH_2Ph), 4.85 (d, 1H, $J_{1,2} = 8.8$ Hz, H-1), 5.13 (dd, 1H, $J_{2,3} = 4.1$ Hz, H-2), 7.22-8.04 (m, 10H, aromatic) ppm; $^{13}\text{C NMR}$ (75 MHz, CDCl_3): δ , -4.2, -4.0, -3.6, -3.4, 0.2, 15.2, 18.2 ($\times 2$), 24.6

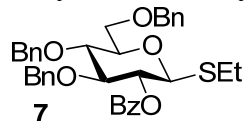
($\times 3$), 26.1 ($\times 3$), 71.0, 71.5, 73.6, 74.5, 75.8, 81.6, 81.7, 127.8, 127.8 ($\times 2$), 128.5 ($\times 4$), 130.2 ($\times 2$), 130.3, 133.3, 138.5, 165.7 ppm; HR-FAB MS $[M+Na]^+$ calcd for $C_{34}H_{54}NaO_6SSi_2^+$ 669.3078, found 669.3064.

Ethyl 2,6-di-*O*-benzyl-3,4-di-*O*-*tert*-butyldimethylsilyl-1-thio- β -D-glucopyranoside (6).



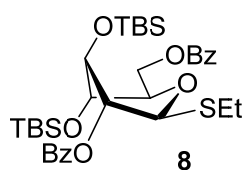
TBSOTf (1.05 mL, 4.60 mmol) was added to a solution of **S4** (0.85 g, 1.53 mmol) in 2,6-lutidine (5.0 mL) and the resulting mixture was heated at 130 °C for 1 h. After that, the reaction mixture was allowed to cool to rt, diluted with ethyl acetate (~100 mL) and washed with 1 M aq. HCl (3 \times 20 mL), water (20 mL), sat. aq. NaHCO₃ (20 mL) and brine (2 \times 20 mL). The organic phase was separated, dried with MgSO₄, and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (ethyl acetate – toluene gradient elution) to afford the title compound in 90% yield (0.87g, 1.37 mmol) as a colorless syrup. Analytical data for **6**: R_f = 0.75 (ethyl acetate/ toluene, 1/9, v/v); $[\alpha]_D^{26.8}$ -26.4 (c = 1, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ , -0.04, -0.01, 0.01, 0.03 (4 s, 12H, 2 \times SiMe₂), 0.80, 0.84 (2 s, 18H, 2 \times Si^{*t*}Bu), 1.26 (t, 3H, J = 7.4 Hz, CH₂CH₃), 2.69 (m, 2H, CH₂CH₃), 3.33 (dd, 1H, $J_{2,3}$ = 3.6 Hz, H-2), 3.56 (dd, 1H, $J_{5,6a}$ = 6.6 Hz, $J_{6a,6b}$ = 9.6 Hz, H-6a), 3.62-3.84 (m, 4H, H-3, 4, 5, 6b), 4.51 (dd, 2H, ² J = 12.0 Hz, CH₂Ph), 4.51 (dd, 2H, ² J = 10.9 Hz, CH₂Ph), 4.74 (d, 1H, $J_{1,2}$ = 8.6 Hz, H-1), 7.17-7.37 (m, 10H, aromatic) ppm; ¹³C NMR (75 MHz, CDCl₃): δ , -4.2, -3.9, -3.5, -3.4, 15.3, 18.2 ($\times 2$), 25.3, 26.1 ($\times 3$), 26.3 ($\times 3$), 71.3, 71.7, 73.4 ($\times 2$), 76.5, 81.3, 82.7, 82.8, 127.6, 127.7, 127.8 ($\times 4$), 128.3 ($\times 2$), 128.5 ($\times 2$), 138.6, 138.6 ppm; HR-FAB MS $[M+Na]^+$ calcd for $C_{34}H_{56}NaO_5SSi_2^+$ 655.3285, found 655.3291.

Ethyl 2-*O*-benzoyl-3,4,6-tri-*O*-benzyl-1-thio- β -D-glucopyranoside (7).



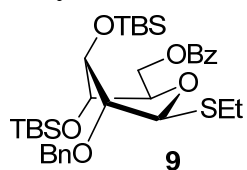
The synthesis of the title compound was performed in accordance with the reported procedure and its analytical data was in accordance with that previously described.⁵

Ethyl 2,6-di-*O*-benzoyl-3,4-di-*O*-*tert*-butyldimethylsilyl-1-thio- β -D-glucopyranoside (8).



TBSOTf (1.13 mL, 4.93 mmol) was added to a solution of **S7** (0.71 g, 1.64 mmol) in 2,6-lutidine (4.0 mL) and the resulting mixture was heated at 130 °C for 1 h. After that, the reaction mixture was allowed to cool to rt, diluted with ethyl acetate (~100 mL) and washed with 1 M aq. HCl (3 \times 15 mL), water (20 mL), sat. aq. NaHCO₃ (20 mL), and brine (2 \times 20 mL). The organic phase was separated, dried with MgSO₄, and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (ethyl acetate – toluene gradient elution) to afford the title compound in 89% yield (0.96 g, 1.46 mmol) as a colorless syrup. Analytical data for **8**: R_f = 0.75 (ethyl acetate/ toluene, 1/9, v/v); $[\alpha]_D^{26.9}$ -0.3 (c = 1, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ , -0.02, -0.01, -0.01, 0.00 (4 s, 12H, 2 \times SiMe₂), 0.75, 0.79 (2 s, 18H, 2 \times Si^{*t*}Bu), 1.12 (t, 3H, J = 7.4 Hz, CH₂CH₃), 2.58 (m, 2H, CH₂CH₃), 3.75 (dd, 1H, $J_{4,5}$ = 5.4 Hz, H-4), 3.84-3.91 (m, 2H, $J_{3,4}$ = 5.4 Hz, $J_{5,6a}$ = 7.0 Hz, $J_{5,6b}$ = 4.6 Hz, H-3, 5), 4.36 (dd, 1H, $J_{6a,6b}$ = 11.4 Hz, H-6a), 4.60 (dd, 1H, H-6b), 4.78 (d, 1H, $J_{1,2}$ = 8.6 Hz, H-1), 5.07 (dd, 1H, $J_{2,3}$ = 4.9 Hz, H-2), 7.40-8.05 (m, 10H, aromatic) ppm; ¹³C NMR (75 MHz, CDCl₃): δ , -4.2, -3.9, -3.4, -3.2, 15.2, 18.2 ($\times 2$), 24.8, 26.1 ($\times 3$), 26.2 ($\times 3$), 65.2, 71.5, 74.1, 75.6, 79.7, 81.9, 128.5 ($\times 2$), 128.6 ($\times 2$), 129.8 ($\times 4$), 130.2 ($\times 2$), 130.3, 133.3, 165.7, 166.5 ppm. HR-FAB MS $[M+Na]^+$ calcd for $C_{34}H_{52}NaO_7SSi_2^+$ 683.2870, found 683.2877.

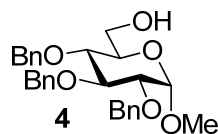
Ethyl 6-*O*-benzoyl-2-*O*-benzyl-3,4-di-*O*-*tert*-butyldimethylsilyl-1-thio- β -D-glucopyranoside (9).



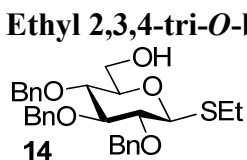
TBSOTf (0.30 mL, 1.28 mmol) was added to a solution of **S8** (0.18 g, 0.429 mmol) in 2,6-lutidine (3.0 mL) and the resulting mixture was heated at 130 °C for 1 h. After that, the reaction mixture was allowed to cool to rt, diluted with ethyl acetate (~60 mL) and washed with 1 M aq. HCl (3 x 5 mL), water (5 mL), sat. aq. NaHCO₃ (5 mL) and brine (2 x 5 mL). The

organic phase was separated, dried with MgSO₄, and concentrated in vacuo. The residue was purified by column chromatography on silica gel (ethyl acetate – toluene gradient elution) to afford the title compound in 86% yield (0.24g, 0.37 mmol) as a colorless syrup. Analytical data for **9**: R_f = 0.75 (ethyl acetate/ toluene, 1/9, v/v); [α]_D^{21.6} -0.8 (c = 1, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ , -0.05-0.02 (4 s, 12H, 2 x SiMe₂), 0.80, 0.82 (2 s, 18H, 2 x Si^tBu), 1.18 (t, 3H, J = 7.4 Hz, CH₂CH₃), 2.62 (m, 2H, CH₂CH₃), 3.32 (dd, 1H, J_{2,3} = 4.0 Hz, H-2), 3.71 (dd, 1H, H-4), 3.78-3.86 (m, 2H, J_{3,4} = 5.0 Hz, H-3, 5), 4.32 (dd, 1H, J_{5,6a} = 7.4 Hz, J_{6a,6b} = 11.3 Hz, H-6a), 4.52 (dd, 1H, J_{5,6b} = 4.8 Hz, H-6b), 4.65 (dd, ²J = 11.1 Hz, CH₂Ph), 4.74 (d, 1H, J_{1,2} = 8.4 Hz, H-1), 7.10 – 8.01 (m, 10H, aromatic) ppm; ¹³C NMR (75 MHz, CDCl₃): δ , -4.2, -3.8, -3.3, -3.2, 15.3, 18.2, 18.3, 25.4, 26.1 (x3), 26.4 (x3), 65.6, 71.7, 73.6, 76.5, 79.4, 82.5, 82.9, 127.6, 127.8 (x2), 128.3 (x2), 128.5 (x2), 129.8 (x2), 130.2, 133.2, 138.5, 166.4 ppm; HR-FAB MS [M+Na]⁺ calcd for C₃₄H₅₄NaO₆SSi₂⁺ 669.3077, found 669.3087.

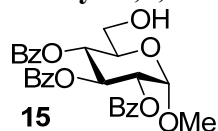
Synthesis of Glycosyl Acceptors



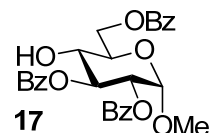
Methyl 2,3,4-tri-*O*-benzyl- α -D-glucopyranoside (4). The synthesis of the title compound was performed in accordance with the reported procedure and its analytical data was in accordance with that previously described.⁶



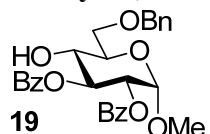
Ethyl 2,3,4-tri-*O*-benzyl-1-thio- β -D-glucopyranoside (14). The synthesis of the title compound was performed in accordance with the reported procedure and its analytical data was in accordance with that previously described.⁷



Methyl 2,3,4-tri-*O*-benzoyl- α -D-glucopyranoside (15). The synthesis of the title compound was performed in accordance with the reported procedure and its analytical data was in accordance with that previously described.⁸



Methyl 2,3,6-tri-*O*-benzoyl- α -D-glucopyranoside (17). The synthesis of the title compound was performed in accordance with the reported procedure and its analytical data was in accordance with that previously described.⁹



Methyl 2,3-di-*O*-benzoyl-6-*O*-benzyl- α -D-glucopyranoside (19). The synthesis of the title compound was performed in accordance with the reported procedure and its analytical data was in accordance with that previously described.¹⁰

General Procedures for Competition Experiments

Donor competition experiments. A mixture of two glycosyl donors (0.035 mmol each), glycosyl acceptor **4** (0.071 mmol), and freshly activated molecular sieves (3 Å, 150 mg) in CH₂Cl₂ (2.0 mL) was stirred under argon for 16 h at rt. The mixture was cooled to -78 °C, NIS (0.035 mmol) and TfOH (0.0035 mmol) were added, and the resulting mixture was stirred under argon for 1 h. During this time, the temperature of the reaction mixture was allowed to gradually increase to 0 °C. After that, triethylamine (~0.1 mL) was added, the solid was filtered off, and rinsed successively with CH₂Cl₂. The combined filtrate (~60 mL) was washed with sat. aq. NaHCO₃ (5 mL), 10% aq. Na₂S₂O₃ (5 mL) and water (3 x 10 mL). The organic layer was separated, dried with MgSO₄, and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (ethyl acetate – toluene gradient elution) and the amount of unreacted donors quantified.

Acceptor competition experiments. A mixture of two glycosyl acceptors (0.042 mmol each), glycosyl donor **5** (0.038 mmol), and freshly activated molecular sieves (3 Å, 150 mg) in CH₂Cl₂ (2.0 mL) was stirred under argon for 16 h at rt. The mixture was cooled to -78 °C, NIS (0.038 mmol) and TfOH (0.0038 mmol) were added, and the resulting mixture was stirred under argon for 1 h. During this time, the temperature of the reaction mixture was allowed to gradually increase to 0 °C. After that, triethylamine (~0.1 mL) was added, the solid was filtered off and rinsed successively with CH₂Cl₂. The combined filtrate (~60 mL) was washed with sat. aq. NaHCO₃ (5 mL), 10% aq. Na₂S₂O₃ (5 mL) and water (3 x 10 mL). The organic layer was separated, dried with MgSO₄, and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (ethyl acetate – toluene gradient elution) and the amount of unreacted acceptors quantified.

Synthesis of Disaccharides

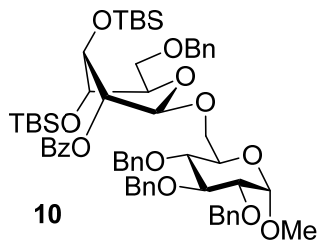
Method A. General glycosylation procedure in the presence of NIS/TfOH. A mixture of glycosyl donor (0.039 mmol), glycosyl acceptor (0.043 mmol), and freshly activated molecular sieves (3Å, 60 mg) in CH₂Cl₂ (2.0 mL) was stirred under argon for 16 h at rt. The mixture was cooled to -78 °C, NIS (0.051 mmol) and TfOH (0.0039 mmol) were added, and the resulting mixture was stirred under argon for 15-30 min (see Table 1 of the article). During this time, the temperature of the reaction mixture was allowed to increase gradually. After that, triethylamine (~0.1 mL) was added, the solid was filtered off and rinsed successively with CH₂Cl₂. The combined filtrate (~60 mL) was washed with sat. aq. NaHCO₃ (5 mL), 10% aq. Na₂S₂O₃ (5 mL) and water (3 x 10 mL). The organic layer was separated, dried with MgSO₄, and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (ethyl acetate – toluene gradient elution) to afford the corresponding disaccharide derivative.

Method B. General glycosylation procedure in the presence of DMTST. A mixture of glycosyl donor (0.039 mmol), glycosyl acceptor (0.043 mmol), and freshly activated molecular sieves (4Å, 60 mg) in CH₂Cl₂ (2.0 mL) was stirred under argon for 16 h at rt. The mixture was cooled to -78 °C, DMTST (0.051-0.078 mmol, see Table 1 of the article) was added, and the resulting mixture was stirred under argon for 10-20 min (see Table 1 of the article). During this

time, the temperature of the reaction mixture was allowed to increase gradually. After that, triethylamine (~0.1 mL) was added, the solid was filtered off and rinsed successively with CH₂Cl₂. The combined filtrate (~60 mL) was washed with sat. aq. NaHCO₃ (5 mL), 10% aq. Na₂S₂O₃ (5 mL) and water (3 x 10 mL). The organic layer was separated, dried with MgSO₄, and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (ethyl acetate – toluene gradient elution) to afford the corresponding disaccharide derivative.

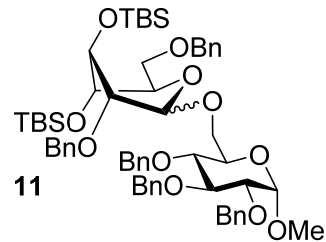
Methyl 6-O-(2-O-benzoyl-6-O-benzyl-3,4-di-O-tert-butyldimethylsilyl-β-D-glucopyranosyl)-2,3,4-tri-O-benzyl-α-D-glucopyranoside (10).

The title compound was prepared from donor **5** and acceptor **4** by Method A or B in 83 or 85% yield, respectively, as a colorless crystalline solid. Analytical data for **10**: *R_f* = 0.43 (ethyl acetate/ toluene, 1/9, v/v); m.p. 111-112.5 °C (methanol/water); [α]_D^{26.9} +3.2 (c = 1, CHCl₃); ¹H NMR (600 MHz, CDCl₃): δ, 0.01, 0.02, 0.04, 0.12 (4 s, 12H, 2 x SiMe₂), 0.84 (s, 18H, 2 x Si^tBu), 3.24 (s, 3H, OCH₃), 3.44-3.49 (m, 2H, *J*_{2,3} = 9.3 Hz, H-2, 4), 3.67-3.73 (m, 3H, H-5, 6a, 6a'), 3.77 (dd, 1H, *J*_{6a',6b'} = 9.7 Hz, H-6b'), 3.82 (dd, 1H, *J*_{4',5'} = 4.0 Hz, H-4'), 3.86 (dd, 1H, *J*_{3',4'} = 4.3 Hz, H-3'), 3.91 (dd, 1H, *J*_{3,4} = 9.3 Hz, H-3), 3.95 (m, 1H, *J*_{5',6a'} = 3.6 Hz, *J*_{5',6b'} = 6.4 Hz, H-5'), 4.16 (br. d, 1H, *J* = 8.9 Hz, H-6b), 4.49 (dd, 2H, ²*J* = 10.7 Hz, CH₂Ph), 4.50 (d, 1H, *J*_{1,2} = 3.5 Hz, H-1), 4.53 (dd, 2H, ²*J* = 10.8 Hz, CH₂Ph), 4.67 (dd, 2H, ²*J* = 12.1 Hz, CH₂Ph), 4.82 (dd, 2H, ²*J* = 11.0 Hz, CH₂Ph), 4.94 (d, 1H, *J*_{1',2'} = 6.3 Hz, H-1'), 5.11 (dd, 1H, *J*_{2',3'} = 3.3 Hz, H-2'), 7.07-7.99 (m, 25H, aromatic) ppm; ¹³C NMR (150 MHz, CDCl₃): δ, -4.3, -4.1 (x2), -3.8, 18.1, 18.2, 26.0 (x6), 55.2, 67.9, 69.8, 71.1, 71.2, 73.5, 73.6, 75.1 (x2), 75.7 (x2), 77.6, 79.5, 79.9, 82.2, 98.2, 100.2, 127.6, 127.7 (x4), 127.8, 128.0 (x5), 128.3 (x2), 128.4 (x3), 128.5 (x4), 128.6 (x2), 130.0 (x2), 130.1, 133.1, 138.4 (x2), 138.5, 139.1, 165.4 ppm; HR-FAB MS [M+Na]⁺ calcd for C₆₀H₈₀NaO₁₂Si₂⁺ 1071.5085, found 1071.5066.



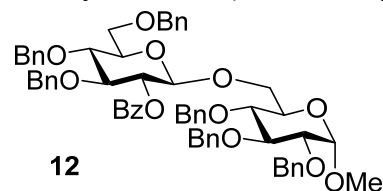
Methyl 6-O-(2,6-di-O-benzyl-3,4-di-O-tert-butyldimethylsilyl-α/β-D-glucopyranosyl)-2,3,4-tri-O-benzyl-α-D-glucopyranoside (11).

The title compound was prepared from donor **6** and acceptor **4** by Method A or B in 81 or 21% yield, respectively, as a colorless syrup. Selected analytical data for α-**11**: *R_f* = 0.50 (ethyl acetate/ toluene, 1/9, v/v); ¹H NMR (600 MHz, CDCl₃): δ, 3.28 (s, 3H, OCH₃), 3.29 (dd, 1H, H-2') ppm; ¹³C NMR (150 MHz, CDCl₃): δ, 96.7 (C-1), 98.1 (C-1') ppm; Selected analytical data for β-**11**: *R_f* = 0.50 (ethyl acetate/ toluene, 1/9, v/v); ¹H NMR (600 MHz, CDCl₃): δ, 3.36 (s, 3H, OCH₃) ppm; ¹³C NMR (150 MHz, CDCl₃): δ, 98.2 (C-1), 102.6 (C-1') ppm; HR-FAB MS [M+Na]⁺ calcd for C₆₀H₈₂NaO₁₁Si₂⁺ 1057.5293, found 1057.5288.

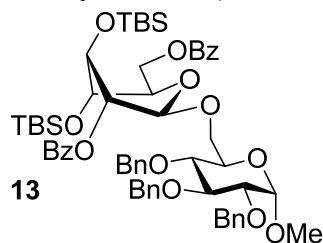


Methyl O-(2-O-Benzoyl-3,4,6-tri-O-benzyl-β-D-glucopyranosyl)-2,3,4-tri-O-benzyl-α-D-glucopyranoside (12).

The analytical data of **12** was in accordance with that previously described.¹¹



Methyl 6-O-(2,6-di-O-benzoyl-3,4-di-O-tert-butyldimethylsilyl-β-D-glucopyranosyl)-2,3,4-tri-O-benzyl-α-D-glucopyranoside (13).

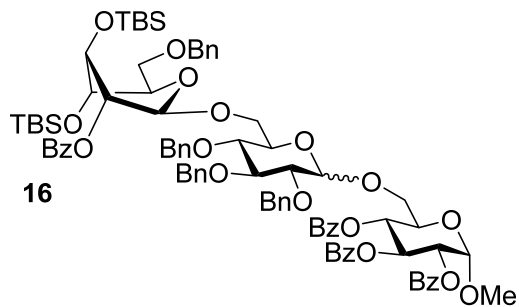


The title compound was prepared from donor **8** and acceptor **4** by Method A in 85% yield as a colorless syrup. Analytical data for **13**: $R_f = 0.60$ (ethyl acetate/toluene, 1/9, v/v); $[\alpha]_D^{26.9} +12.5$ ($c = 1$, CHCl_3); $^1\text{H NMR}$ (600 MHz, CDCl_3): δ , 0.01, 0.05, 0.10, 0.14 (4 s, 12H, 2 x SiMe_2), 0.84, 0.86 (2 s, 18H, 2 x Si^tBu), 3.16 (s, 3H, OCH_3), 3.44 (dd, 1H, $J_{2,3} = 9.7$ Hz, H-2), 3.46 (dd, 1H, $J_{4,5} = 9.4$ Hz, H-4), 3.64-3.67 (m, 2H, H-5, 6a), 3.85 (dd, 1H, $J_{4',5'} = 3.7$ Hz, H-4'), 3.88 (dd, 1H, $J_{3,4} = 9.3$ Hz, H-3), 3.93 (dd, 1H, $J_{3',4'} = 3.7$ Hz, H-3'), 4.08 (m, 1H, H-5), 4.14 (d, 1H, $J_{6a,6b} = 9.3$ Hz, H-6b), 4.49 (dd, 2H, $^2J = 10.9$ Hz, CH_2Ph), 4.50 (d, 1H, $J_{1,2} = 3.2$ Hz, H-1), 4.57-4.59 (m, 2H, H-6a', 6b'), 4.66 (dd, 2H, $^2J = 11.8$ Hz, CH_2Ph), 4.82 (dd, 2H, $^2J = 10.9$ Hz, CH_2Ph), 4.91 (d, 1H, $J_{1',2'} = 4.4$ Hz, H-1'), 5.06 (dd, 1H, $J_{2',3'} = 4.4$ Hz, H-2'), 7.11-8.05 (m, 25H, aromatic) ppm; $^{13}\text{C NMR}$ (150 MHz, CDCl_3): δ , -4.4, -4.1 ($\times 2$), -3.8, 18.2 ($\times 2$), 26.0 ($\times 6$), 29.9, 55.0, 65.6, 67.8, 69.7, 70.7, 73.5, 73.7, 74.6, 75.1, 75.7, 77.6, 79.9, 82.2, 98.0, 100.0, 127.6, 127.8, 128.0 ($\times 5$), 128.3 ($\times 2$), 128.4 ($\times 2$), 128.5 ($\times 4$), 128.6 ($\times 4$), 129.8 ($\times 2$), 130.0, 130.1 ($\times 2$), 130.4, 133.2 ($\times 2$), 138.4 ($\times 2$), 139.2, 165.5, 166.4 ppm; HR-FAB MS $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{60}\text{H}_{78}\text{NaO}_{13}\text{Si}_2^+$ 1085.4878, found 1085.4913.

One-Pot One-Addition Trisaccharide Synthesis

General procedure. A mixture of glycosyl donor **5** (0.038 mmol), glycosyl donor/acceptor **14** (0.038 mmol), glycosyl acceptor **15**, **17** or **19** (0.042 mmol), and freshly activated molecular sieves (3 Å, 150 mg) in CH_2Cl_2 (2.0 mL) was stirred under for 16 h at rt. The mixture was cooled to -78 °C, NIS (0.116 mmol) and TfOH (0.0116 mmol) were added, and the resulting mixture was stirred under argon for 5 h. During this time, the temperature of the reaction mixture was allowed to gradually increase to rt. After that, triethylamine (~ 0.1 mL) was added, the solid was filtered off and rinsed successively with CH_2Cl_2 . The combined filtrate (~ 60 mL) was washed with sat. aq. NaHCO_3 (5 mL), 10% aq. $\text{Na}_2\text{S}_2\text{O}_3$ (5 mL) and water (3 x 10 mL). The organic layer was separated, dried with MgSO_4 , and concentrated *in vacuo*. The residue was purified by column chromatography on Sephadex LH-20 (methanol – dichlorometane, 1/1, v/v) to afford the corresponding trisaccharide derivative **16**, **18** or **20**.

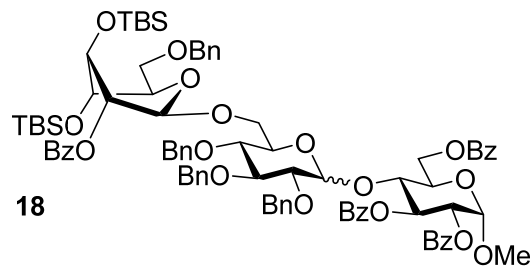
Methyl O-(2-O-benzoyl-6-O-benzyl-3,4-di-O-tert-butyldimethylsilyl-β-D-glucopyranosyl)-(1→6)-O-(2,3,4-tri-O-benzyl-α/β-D-glucopyranosyl)-(1→6)-2,3,4-tri-O-benzyl-α-D-glucopyranoside (16).



The title compound was prepared from building blocks **5**, **14**, and **15** in accordance with the general procedure in 42% yield as a colorless syrup. Analytical data for α -**16**: $R_f = 0.46$ (ethyl acetate/toluene, 1/9, v/v); $^1\text{H NMR}$ (600 MHz, CDCl_3): δ , -0.01, 0.03, 0.05, 0.12 (4 s, 12H, 2 x SiMe_2), 0.80, 0.83 (2 s, 18H, 2 x Si^tBu), 3.35-3.39 (m, 4H), 3.40 (d, 1H), 3.48 (dd, 1H), 3.56 (t, 1H), 3.60-3.66 (m, 2H), 3.72 (dd, 1H), 3.76-3.82 (m, 2H), 3.86-3.89 (m, 1H), 3.90-3.93 (m, 1H), 4.06 (dd, 1H), 4.15 (d, 1H), 4.23-4.28 (m, 1H), 4.39-4.47 (m, 3H), 4.52-4.58 (m, 2H), 4.67 (dd, 3H), 4.85 (d, 1H), 4.92 (d, 1H), 5.05 (dd, 2H), 5.20 (dd, 2H),

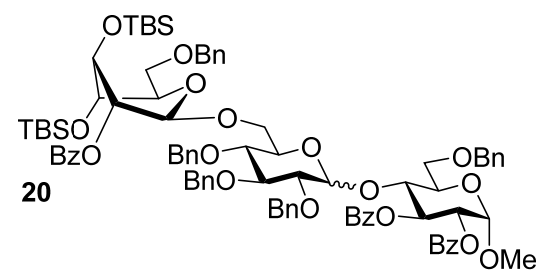
5.48 (t, 1H), 7.95-6.12 (m, 40H, aromatic) ppm; ^{13}C NMR (150 MHz, CDCl_3): δ , -4.3, -4.1 ($\times 2$), -3.8, 18.2 ($\times 2$), 26.1 ($\times 6$), 29.9, 55.9, 67.8, 68.7, 69.0, 70.1, 70.7, 70.9, 71.2, 72.4, 73.4, 74.5, 74.6, 75.0, 75.1, 75.2, 75.6, 79.0, 82.4, 84.7, 96.9, 100.1, 104.2, 127.8 ($\times 2$), 127.9 ($\times 2$), 128.2 ($\times 2$), 128.4 ($\times 7$), 128.5 ($\times 6$), 128.6 ($\times 3$), 129.2, 129.3, 129.5, 129.9 ($\times 2$), 130.1 ($\times 2$), 130.1 ($\times 2$), 130.1 ($\times 2$), 133.1, 133.23, 133.5, 138.2, 138.6, 138.8, 139.0, 165.4, 165.6, 166.0 ($\times 2$) ppm; HR-FAB MS $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{87}\text{H}_{104}\text{NaO}_{19}\text{Si}_2^+$ 1545.6400, found 1545.6368.

Methyl *O*-(2-*O*-benzoyl-6-*O*-benzyl-3,4-di-*O*-*tert*-butyldimethylsilyl- β -D-glucopyranosyl)-(1 \rightarrow 6)-*O*-(2,3,4-tri-*O*-benzyl- α / β -D-glucopyranosyl)-(1 \rightarrow 4)-2,3,6-tri-*O*-benzoyl- α -D-glucopyranoside (18).



The title compound was prepared from building blocks **5**, **14** and **17** in accordance with the general procedure in 37% yield as a colorless syrup. Analytical data for α -**18**: R_f = 0.55 (ethyl acetate/ toluene, 1/9, v/v); ^1H NMR (600 MHz, CDCl_3): δ , -0.03, 0.00, 0.03, 0.08 (4 s, 12H, 2 x SiMe_2), 0.81 (s, 18H, 2 x Si^tBu), 3.24 (dd, 1H), 3.40 (s, 3H), 3.45-3.50 (m, 1H), 3.64-3.56 (m, 2H), 3.70-3.76 (m, 1H), 3.79-3.89 (m, 4H), 3.95 (d, 1H), 4.05 (d, 1H), 4.19 (dd, 3H), 4.31 (d, 1H), 4.39 (d, 1H), 4.48-4.71 (m, 6H), 4.79 (d, 1H), 4.84 (d, 1H), 5.05 (m, 1H), 5.08 (d, 1H), 5.12 (d, 1H), 5.18 (dd, 1H), 6.18 (dd, 1H), 7.01-8.04 (m, 40H, aromatic) ppm; ^{13}C NMR (150 MHz, CDCl_3): δ , -4.4, -4.3, -4.2, -4.0, 18.1, 18.2, 26.0 ($\times 6$), 29.9, 55.5, 63.5, 67.6, 68.9, 70.8, 71.0, 71.4, 72.3, 72.4, 73.1, 73.5, 75.0, 75.3, 75.4, 75.5, 75.8, 79.1, 79.8, 81.4, 96.9, 98.9, 99.9, 127.4, 127.6, 127.7 ($\times 4$), 127.9 ($\times 4$), 128.0 ($\times 2$), 128.3 ($\times 4$), 128.4 ($\times 4$), 128.5 ($\times 2$), 128.6 ($\times 4$), 129.9 ($\times 2$), 130.00 ($\times 2$), 130.2 ($\times 2$), 133.0, 133.1, 133.3, 133.4, 138.2, 138.4, 138.6, 139.1, 165.3, 165.6, 166.1, 166.2 ppm. HR-FAB MS $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{87}\text{H}_{104}\text{NaO}_{19}\text{Si}_2^+$ 1545.6400, found 1545.6366.

Methyl *O*-(2-*O*-benzoyl-6-*O*-benzyl-3,4-di-*O*-*tert*-butyldimethylsilyl- β -D-glucopyranosyl)-(1 \rightarrow 6)-*O*-(2,3,4-tri-*O*-benzyl- α / β -D-glucopyranosyl)-(1 \rightarrow 4)-2,3-di-*O*-benzoyl-6-*O*-benzyl- α -D-glucopyranoside (20).



The title compound was prepared from building blocks **5**, **14** and **19** in accordance with the general procedure in 65% yield as a colorless syrup. Analytical data for α -**18**: R_f = 0.60 (ethyl acetate/ toluene, 1/9, v/v); ^1H NMR (600 MHz, CDCl_3): δ , 0.01, 0.05, 0.05, 0.12 (4 s, 12H, 2 x SiMe_2), 0.83, 0.84 (2s, 18H, 2 x Si^tBu), 3.18 (dd, 1H), 3.41 (s, 3H), 3.51 (dd, 1H), 3.65-3.73 (m, 2H), 3.76 (dd, 1H), 3.79-3.92 (m, 6H), 3.92-4.05 (m, 4H), 4.20-4.27 (m, 2H), 4.35 (d, 1H), 4.46 (s, 2H), 4.50 (dd, 2H), 4.54-4.63 (m, 3H), 4.87 (d, 1H), 4.97 (d, 1H), 5.07 (d, 1H), 5.07 (d, 1H), 5.15-5.22 (m, 2H), 6.11 (dd, 1H), 7.04-7.94 (m, 40H, aromatic) ppm; ^{13}C NMR (150 MHz, CDCl_3): δ , -4.4, -4.3, -4.2, -4.0, 18.1, 18.2, 26.0 ($\times 6$), 29.9, 55.5, 67.9, 68.7, 70.4, 70.8, 71.1 ($\times 2$), 72.3, 72.5, 72.7, 73.4, 73.5, 74.9, 75.0, 75.3, 75.4, 75.8, 79.4, 79.8, 81.5, 97.0, 98.4, 100.0, 127.7 ($\times 2$), 127.8 ($\times 3$), 127.9 ($\times 2$), 128.0 ($\times 2$), 128.1 ($\times 2$), 128.4 ($\times 11$), 128.5 ($\times 2$), 129.4, 129.9 ($\times 2$), 130.0 ($\times 2$), 130.1 ($\times 2$), 130.3, 133.0, 133.1, 133.4, 138.3, 138.4, 138.5, 138.6, 139.1, 165.3, 165.9, 166.2 ppm; HR-FAB MS $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{87}\text{H}_{106}\text{NaO}_{18}\text{Si}_2^+$ 1531.6608, found 1531.6576.

X-ray Structure Determination of Disaccharide 10

A crystal of approximate dimensions 0.324 x 0.121 x 0.098 mm³ was mounted on MiTeGen cryoloops in a random orientation. Preliminary examination and data collection were performed using a Bruker X8 Kappa Apex II Charge Coupled Device (CCD) Detector system single crystal X-Ray diffractometer equipped with an Oxford Cryostream LT device. All data were collected using graphite monochromated Mo K α radiation ($\lambda = 0.71073 \text{ \AA}$) from a fine focus sealed tube X-Ray source. Preliminary unit cell constants were determined with a set of 36 narrow frame scans. Typical data sets consist of combinations of ω and ϕ scan frames with typical scan width of 0.5° and counting time of 15 seconds/frame at a crystal to detector distance of 4.0 cm. The collected frames were integrated using an orientation matrix determined from the narrow frame scans. Apex II and SAINT software packages (*Bruker Analytical X-Ray, Madison, WI, 2010*) were used for data collection and data integration. Analysis of the integrated data did not show any decay. Final cell constants were determined by global refinement of reflections harvested from the complete data set. Collected data were corrected for systematic errors using SADABS (*Bruker Analytical X-Ray, Madison, WI, 2010*) based on the Laue symmetry using equivalent reflections.

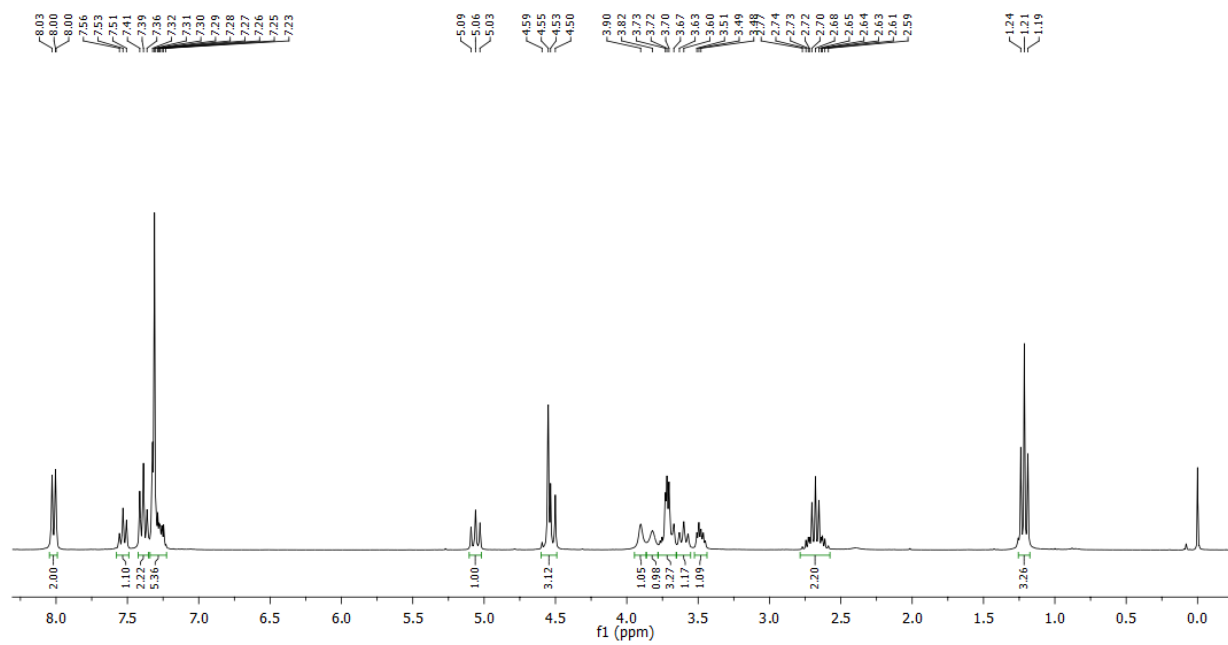
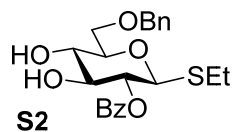
Crystal data and intensity data collection parameters are listed in Table 1S. Structure solution and refinement were carried out using the SHELXTL- PLUS software package.¹² The structure was solved by direct methods and refined successfully in the space group P2₁. Full matrix least-squares refinements were carried out by minimizing $\sum w(F_o^2 - F_c^2)^2$. The non-hydrogen atoms were refined anisotropically to convergence. All hydrogen atoms were treated using appropriate riding model (AFIX m³). The final residual values and structure refinement parameters are listed in Table 1S. Several motifs are disordered in this structure. The disorder was modeled with partial occupancy atoms and rigid body restraints (RIGU). Absolute structure determination was confirmed with a Flack x of -0.02(11).

Complete listings of positional and isotropic displacement coefficients for hydrogen atoms, anisotropic displacement coefficients for the non-hydrogen atoms are listed as supplementary material (Tables 2S and 4S). Table of calculated and observed structure factors are available in electronic format. The structural data have been deposited with Cambridge Crystallographic Data center with the CCDC number 1509777.

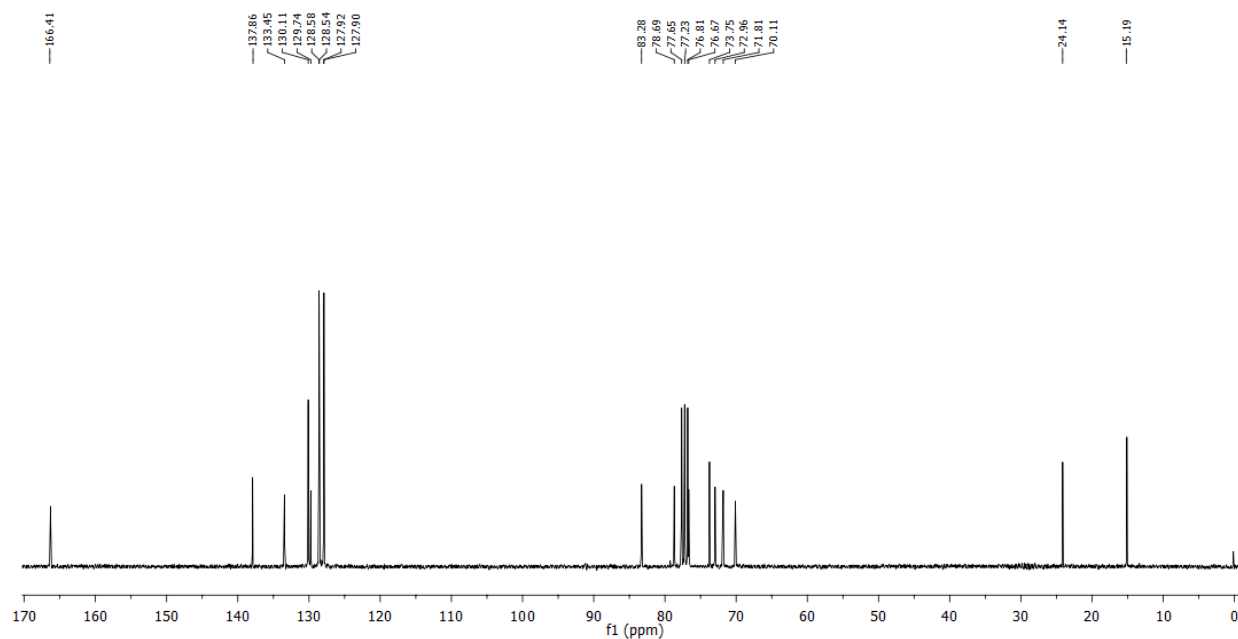
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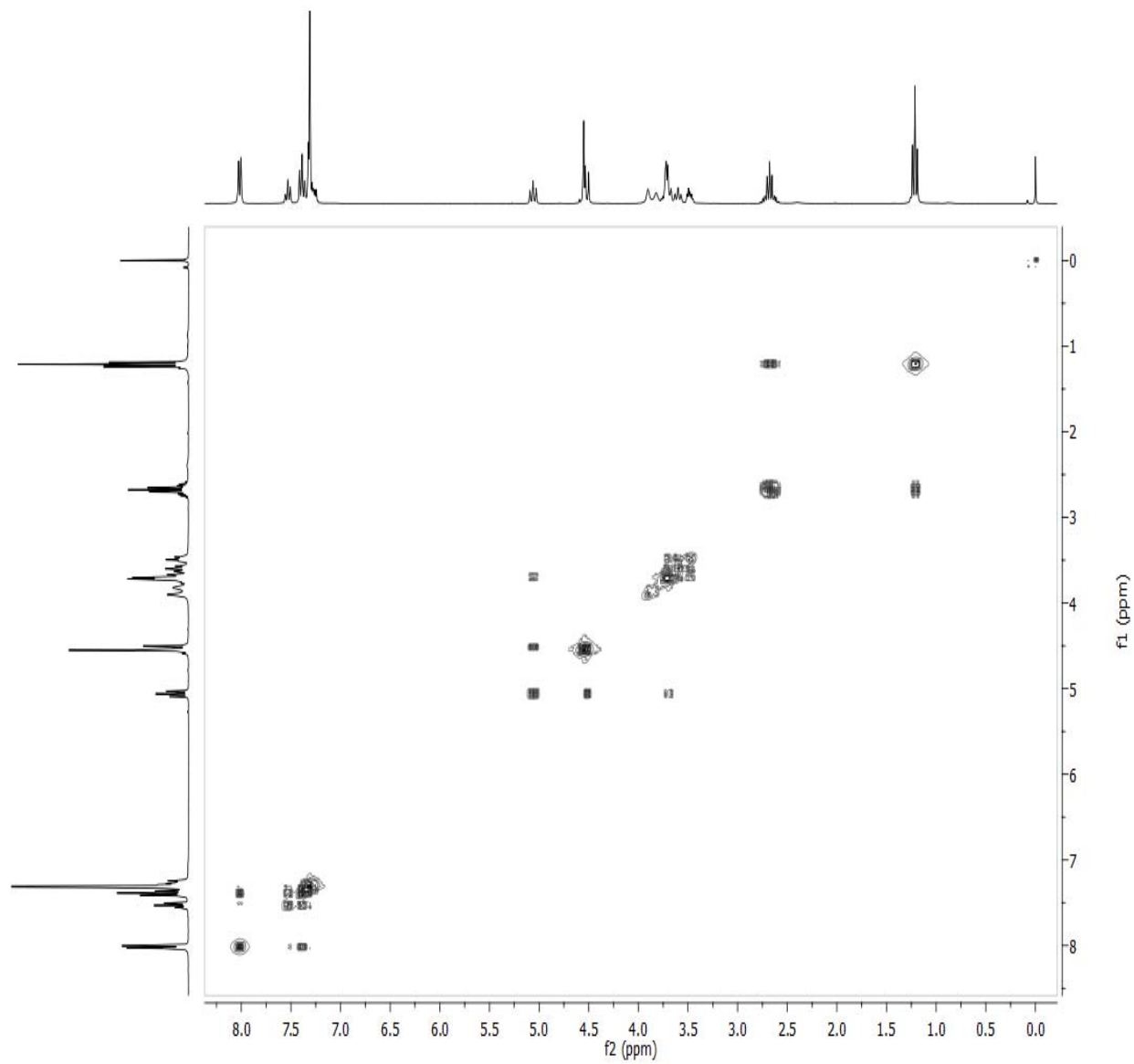
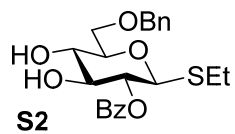
NMR Spectra



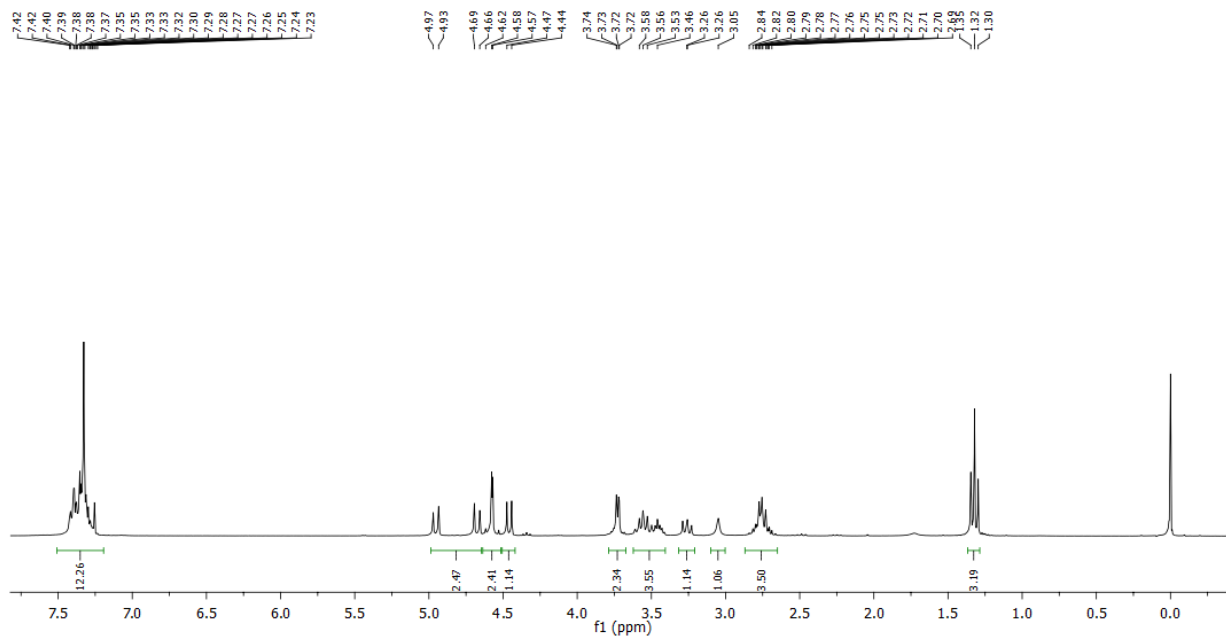
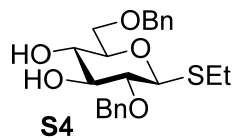
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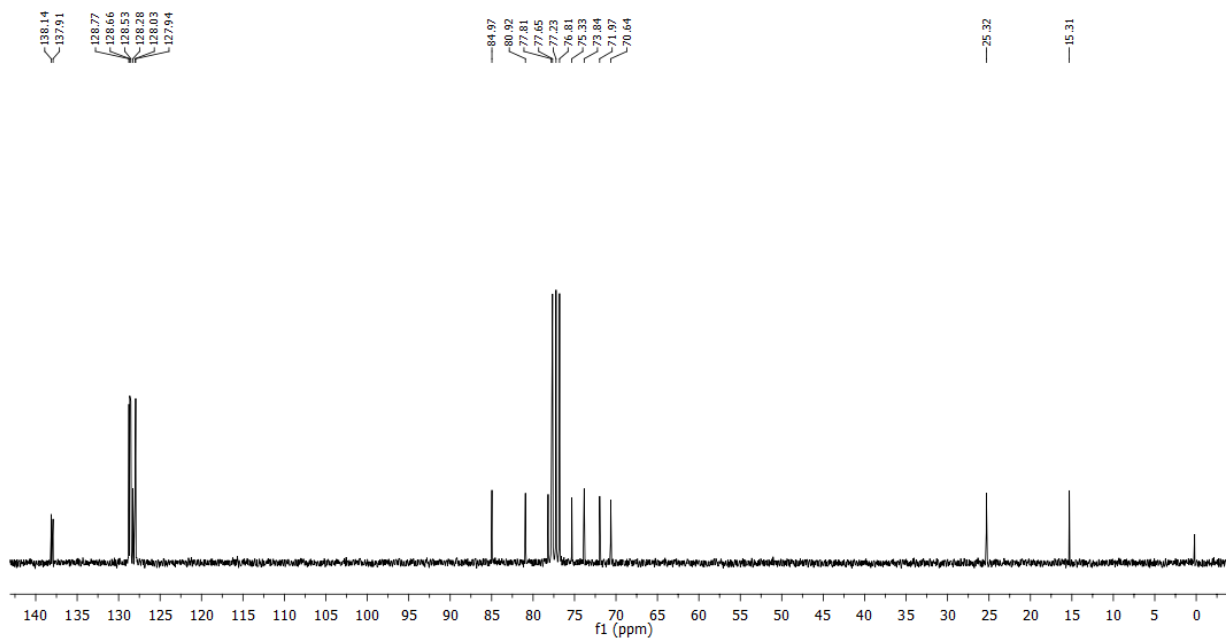
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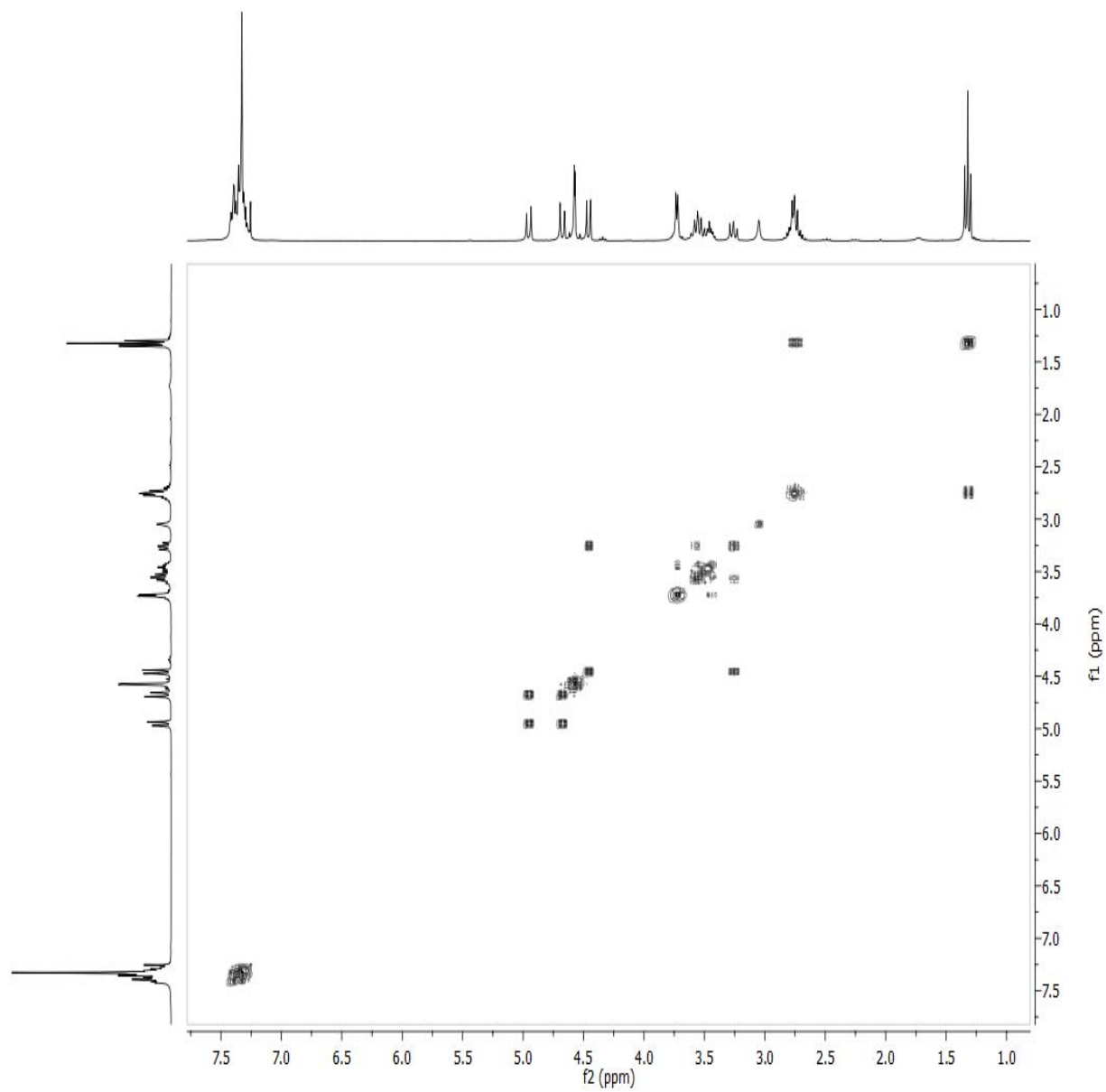
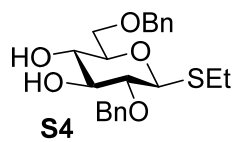
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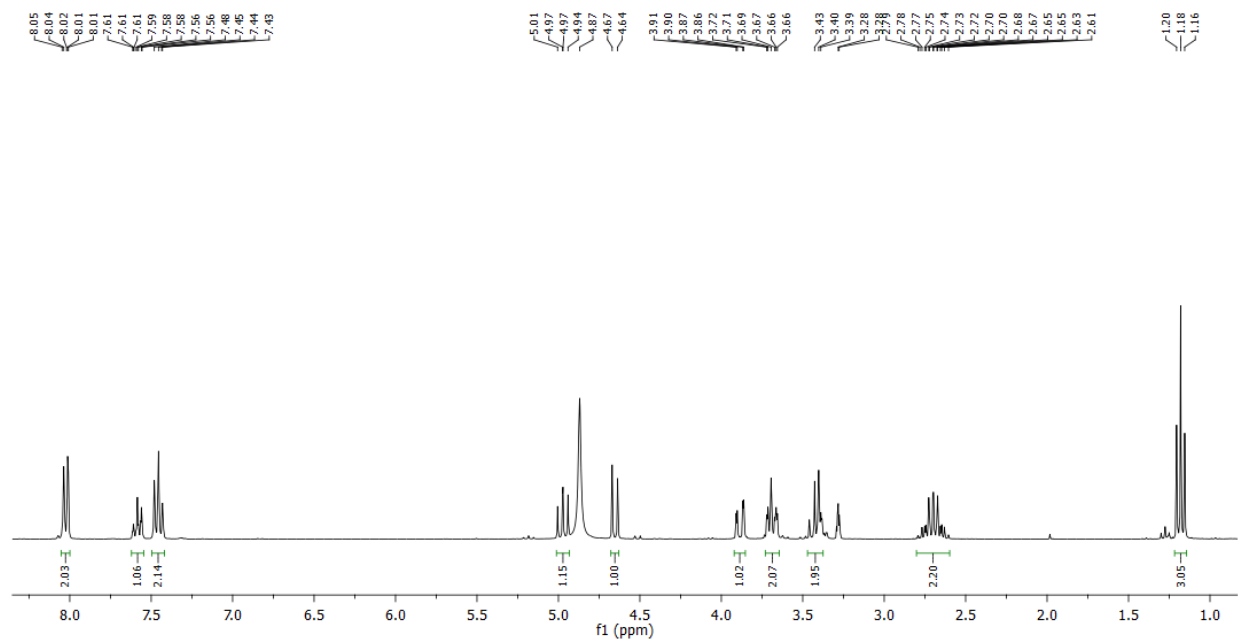
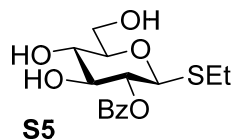
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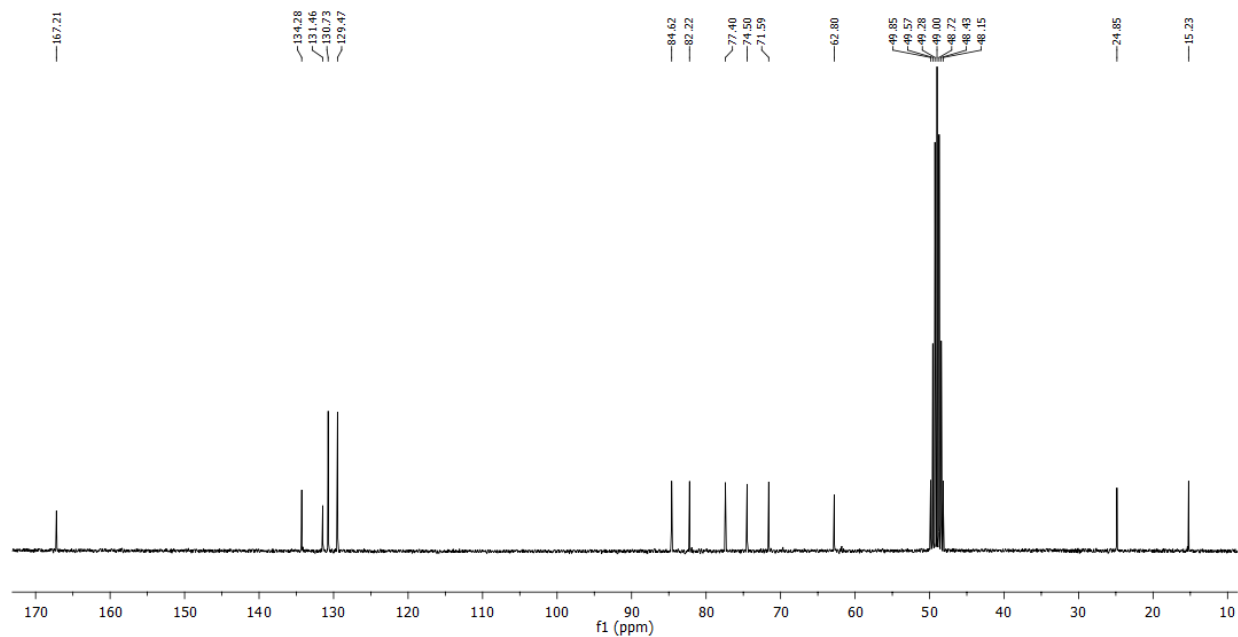
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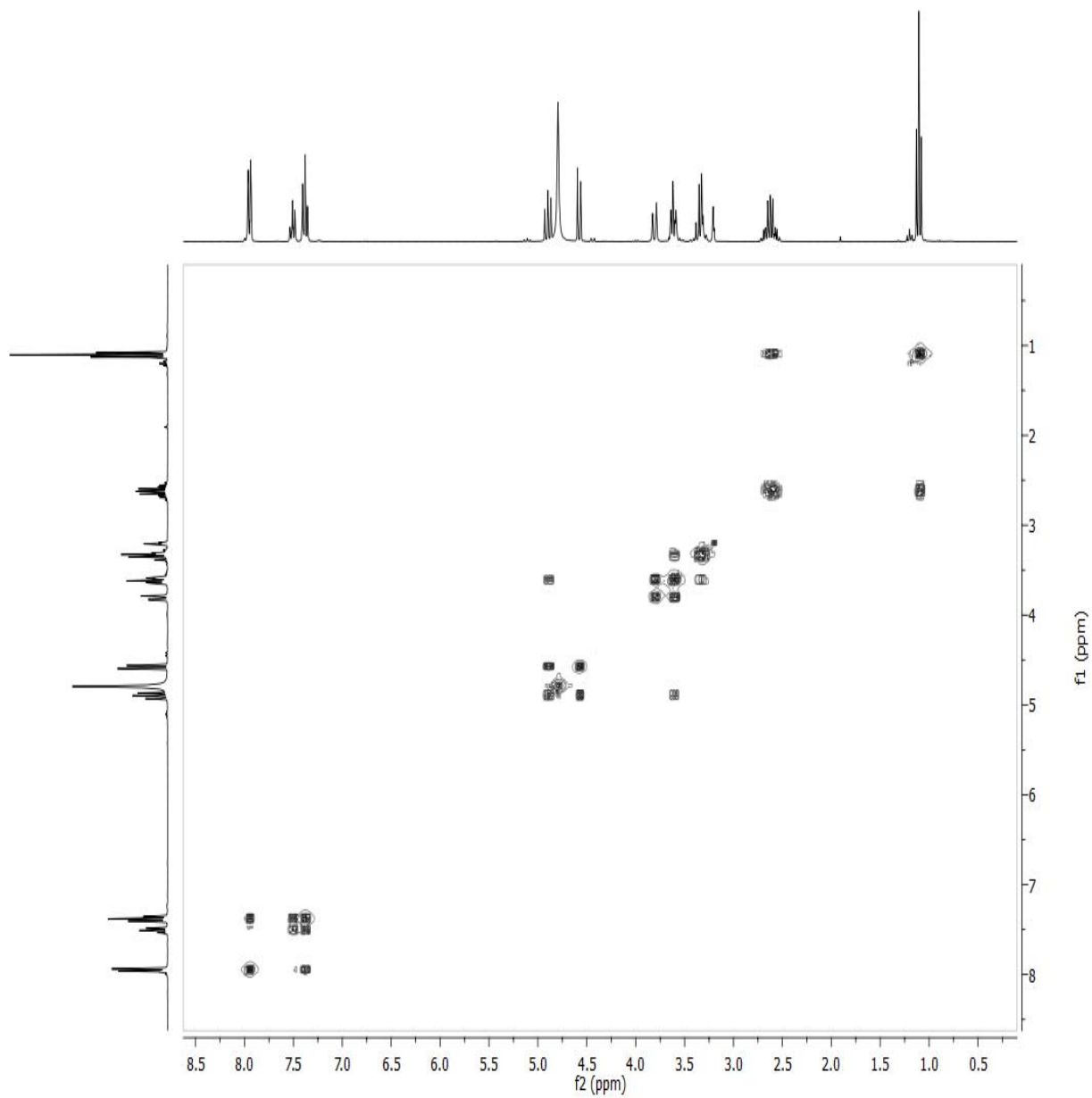
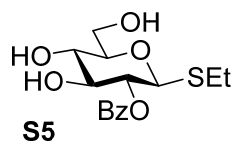
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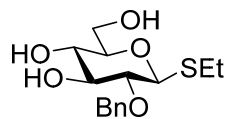


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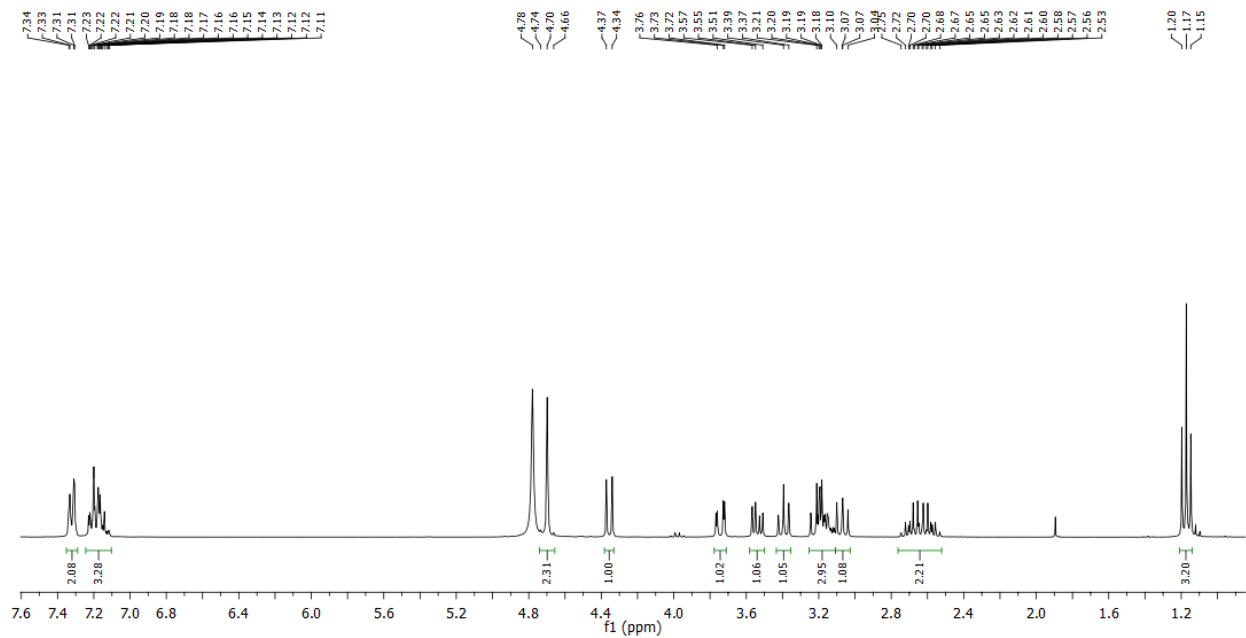


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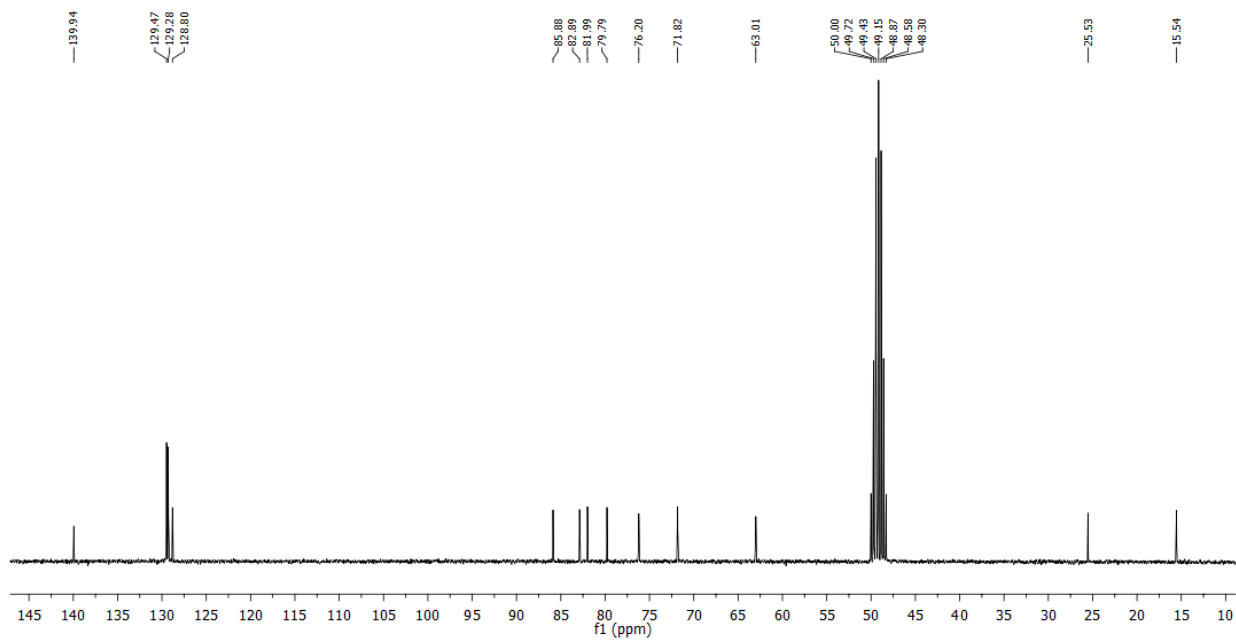




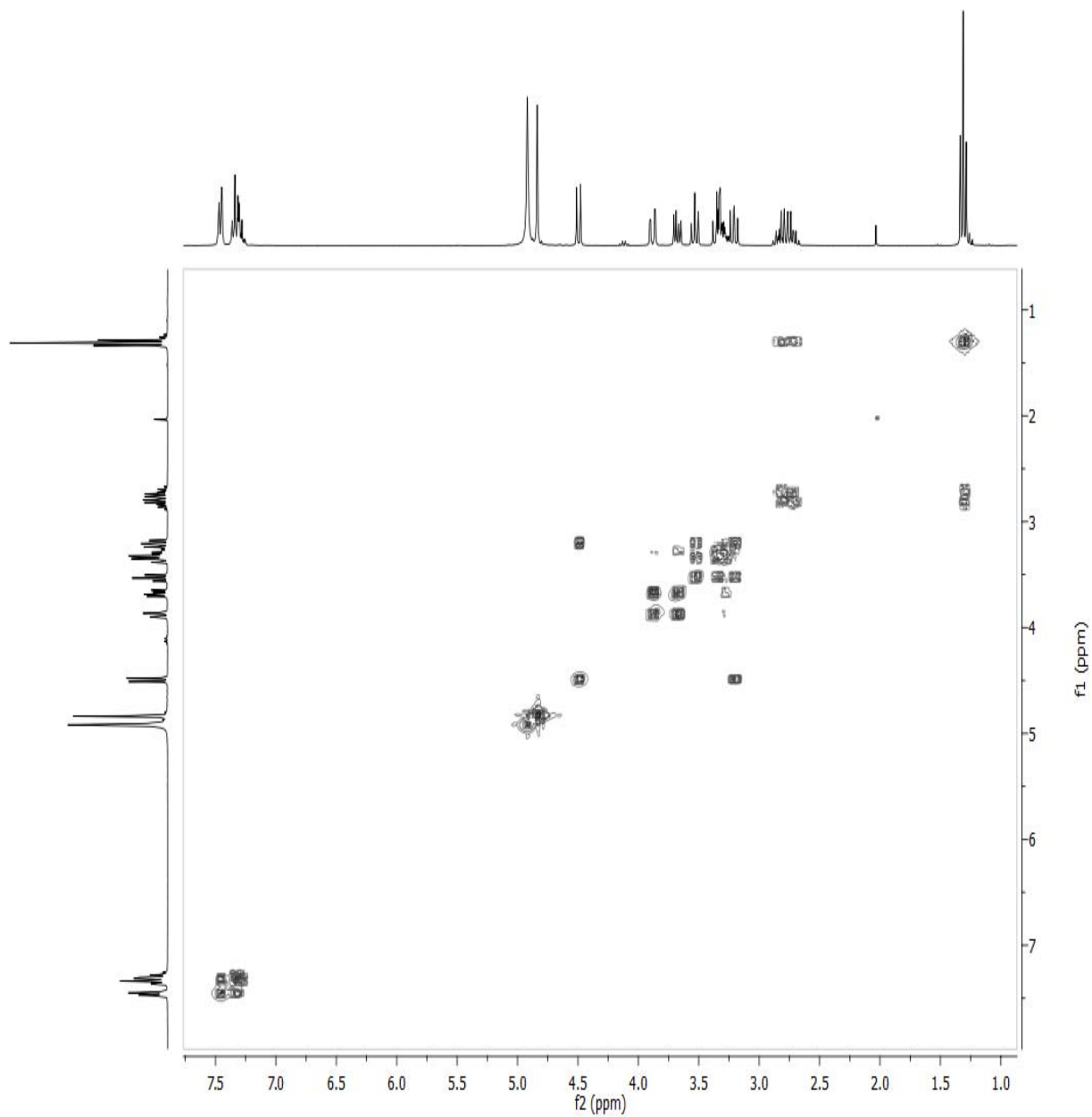
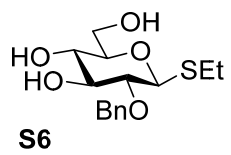
S6



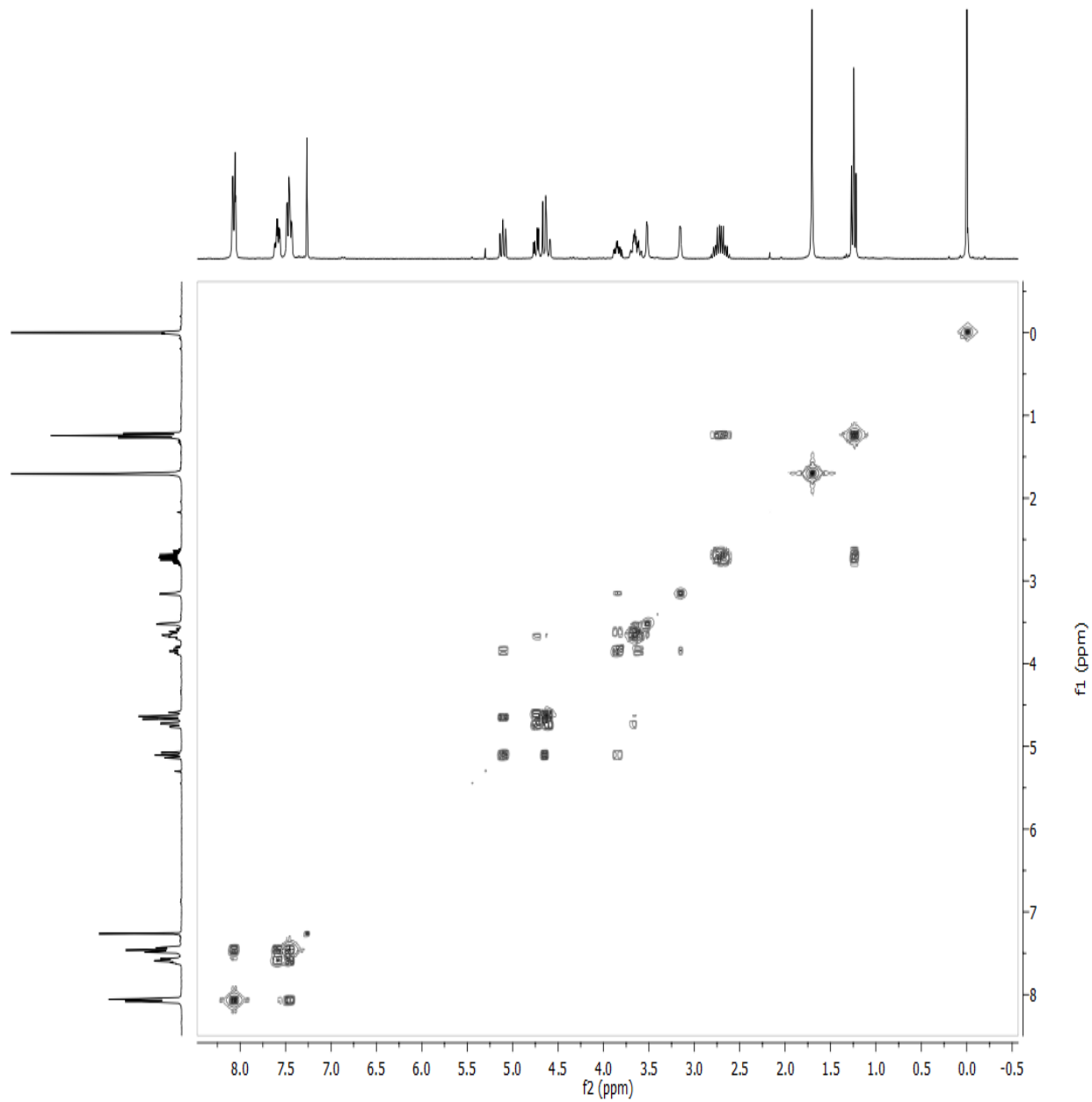
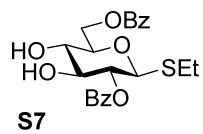
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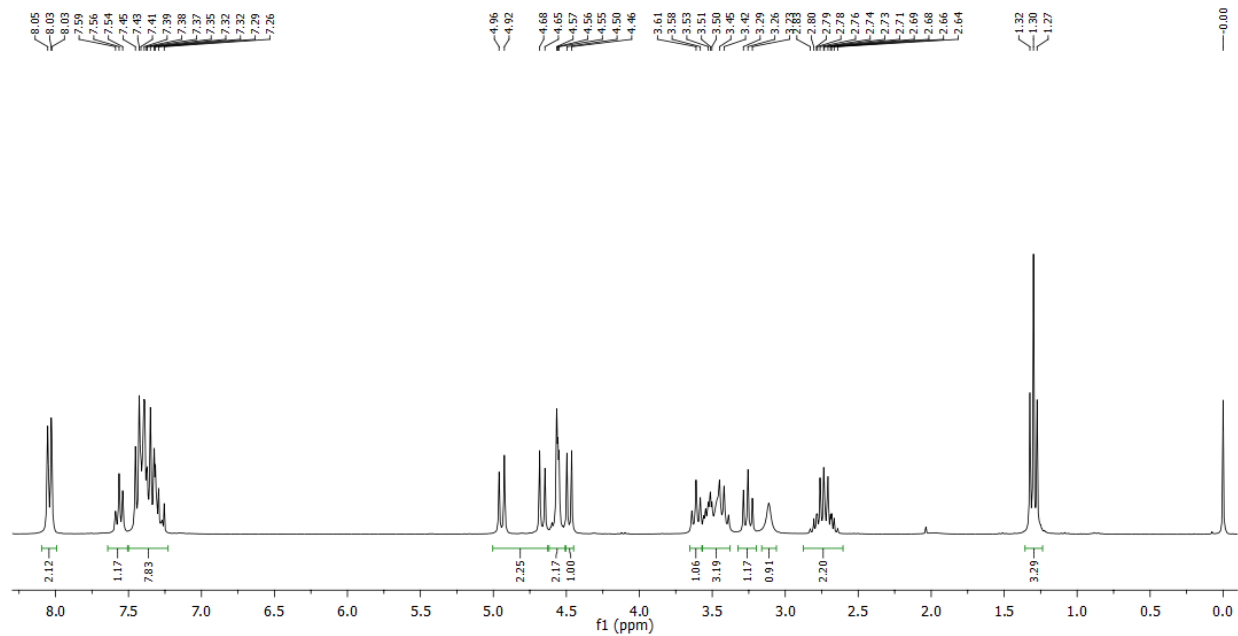
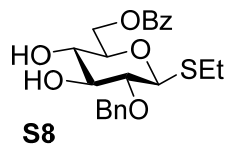
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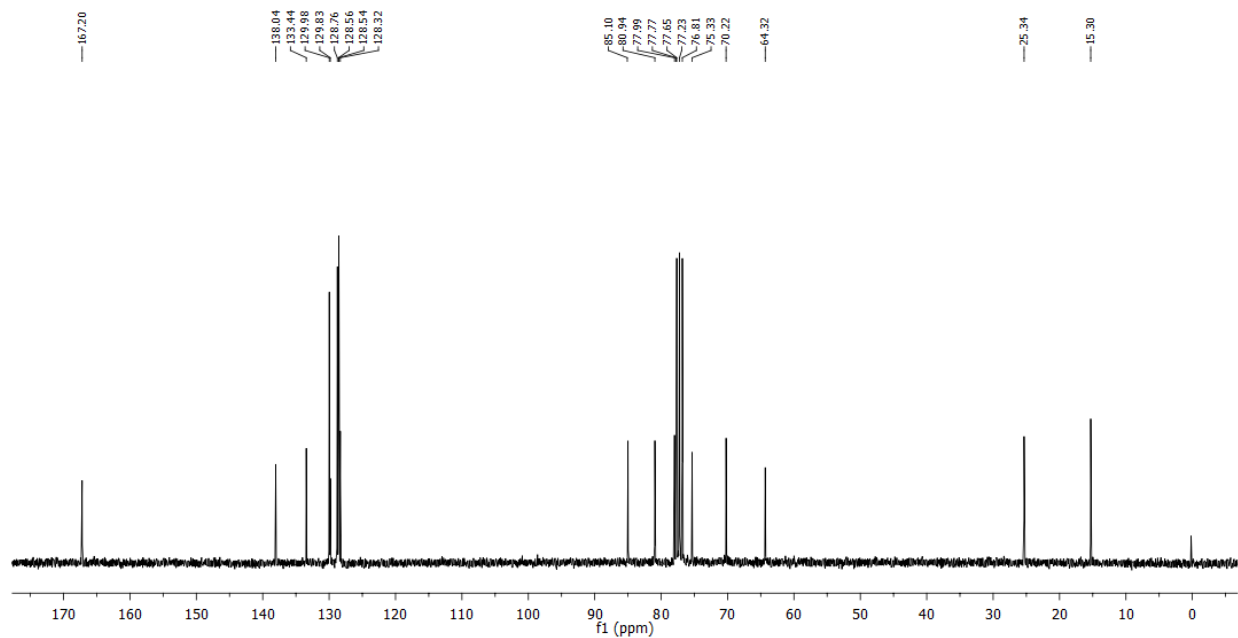
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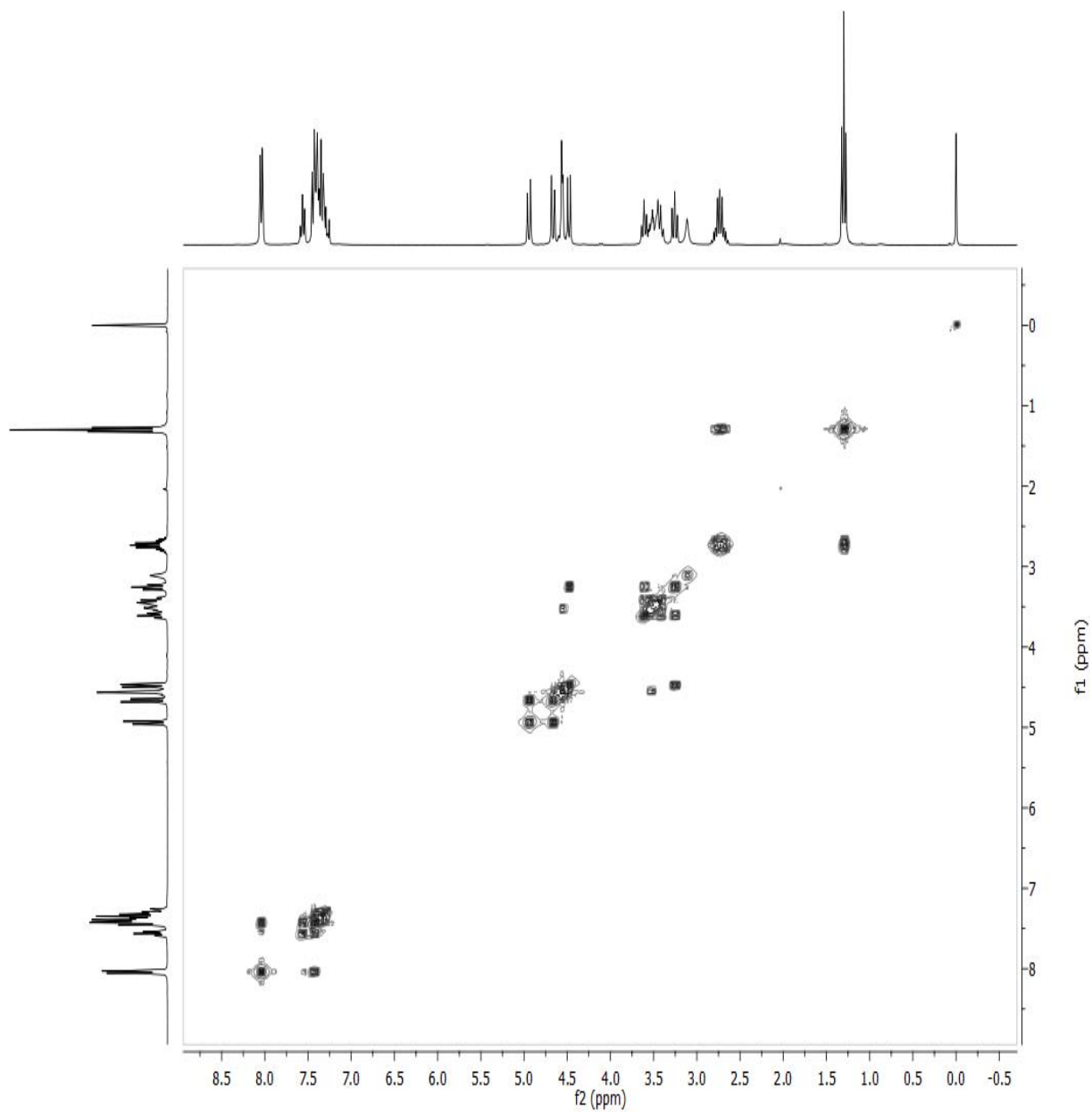
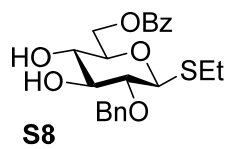
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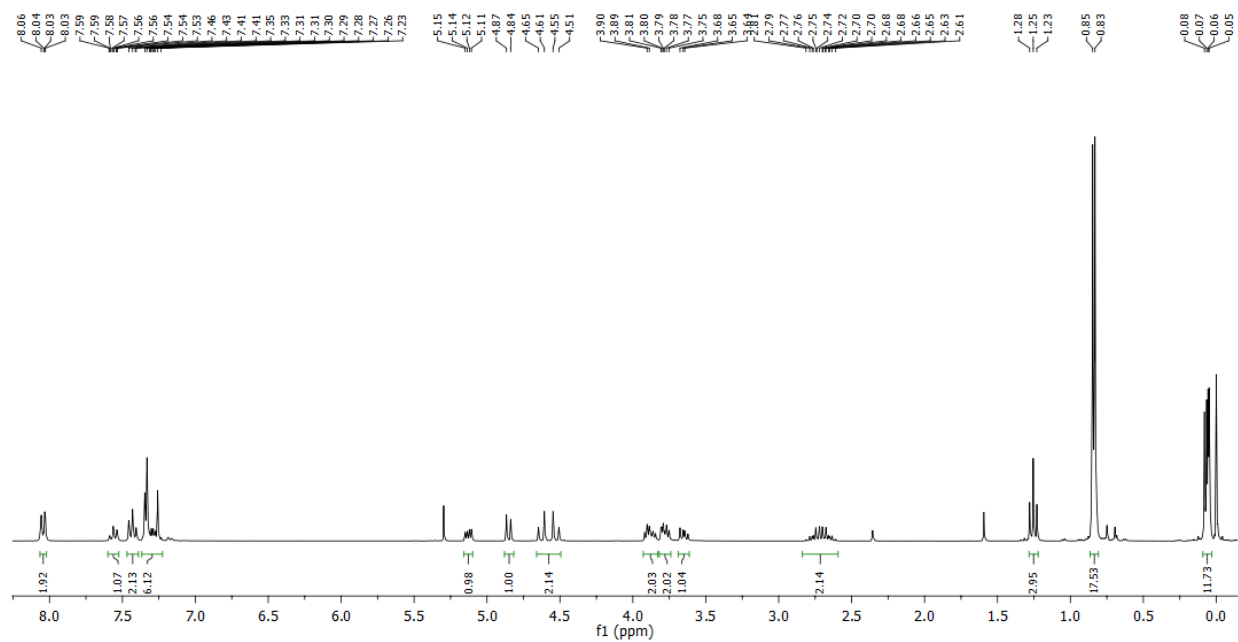
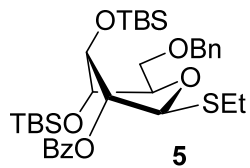
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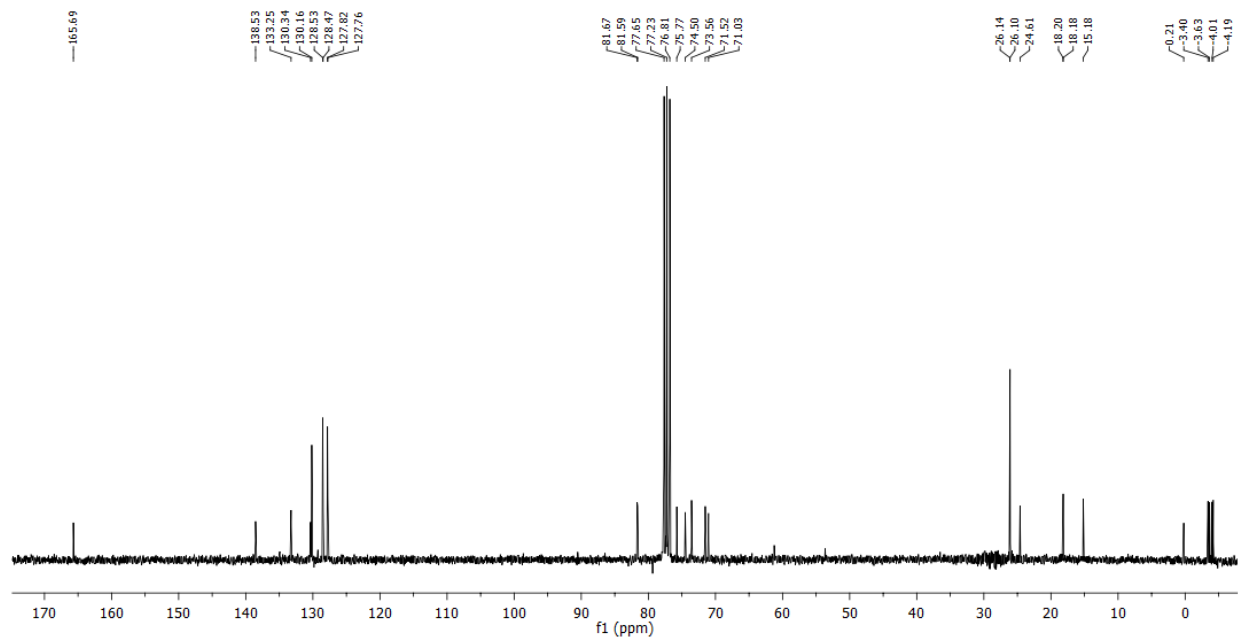
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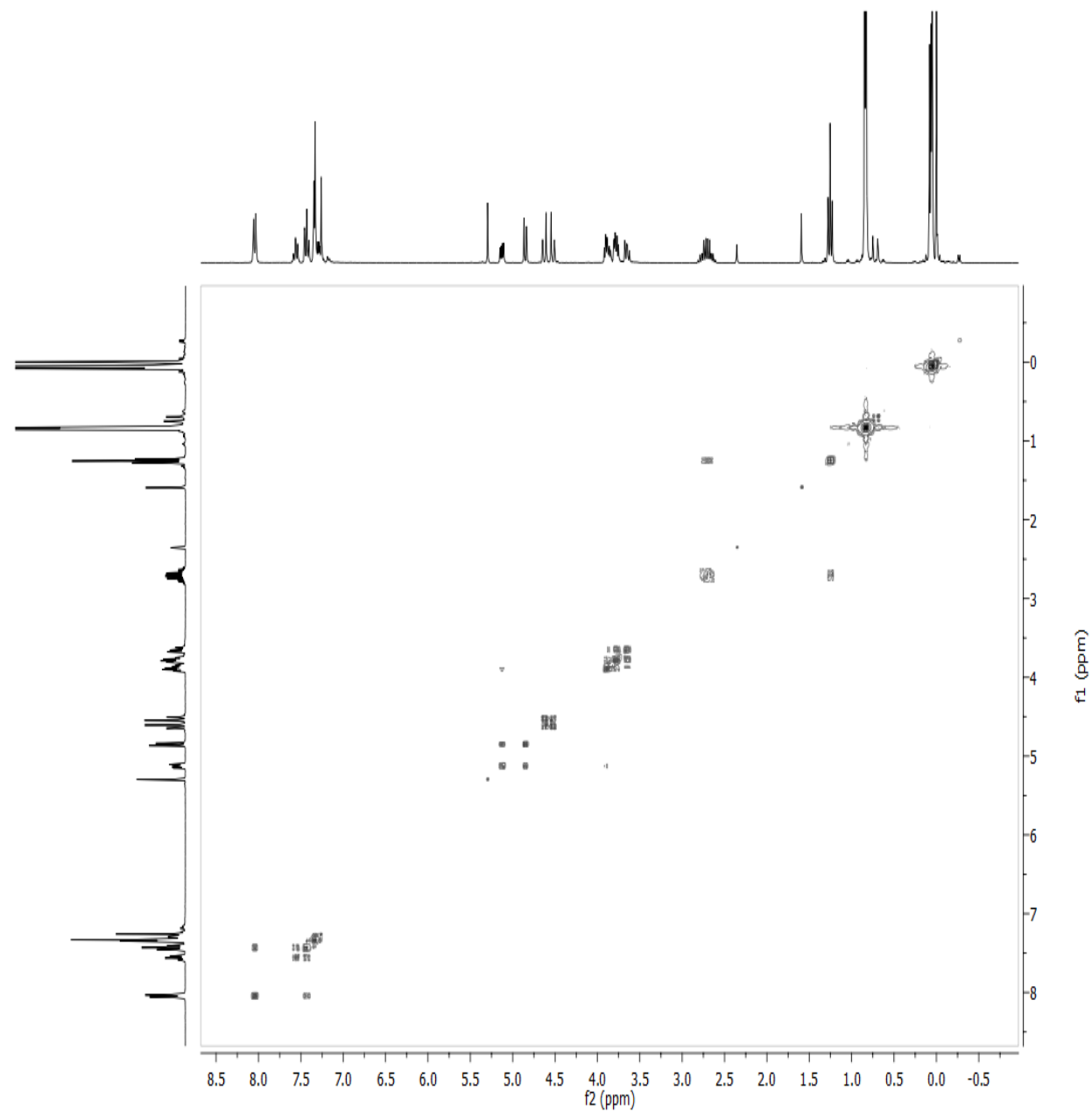
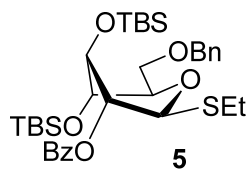
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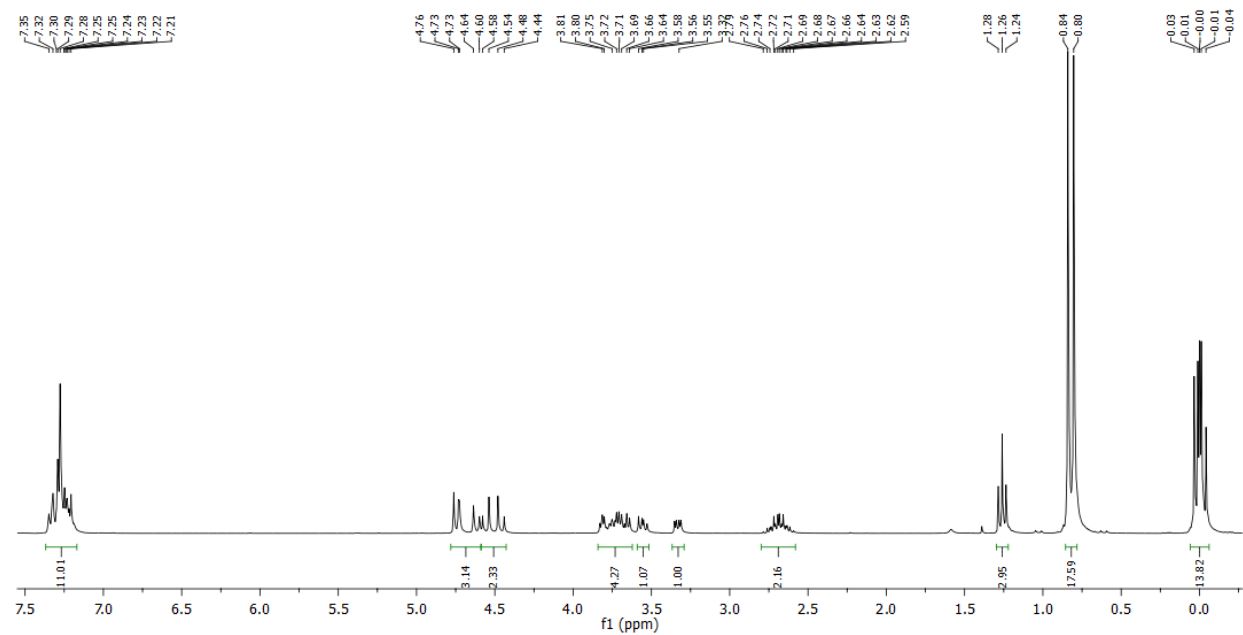
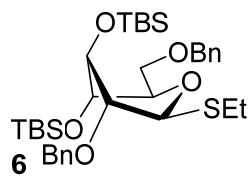
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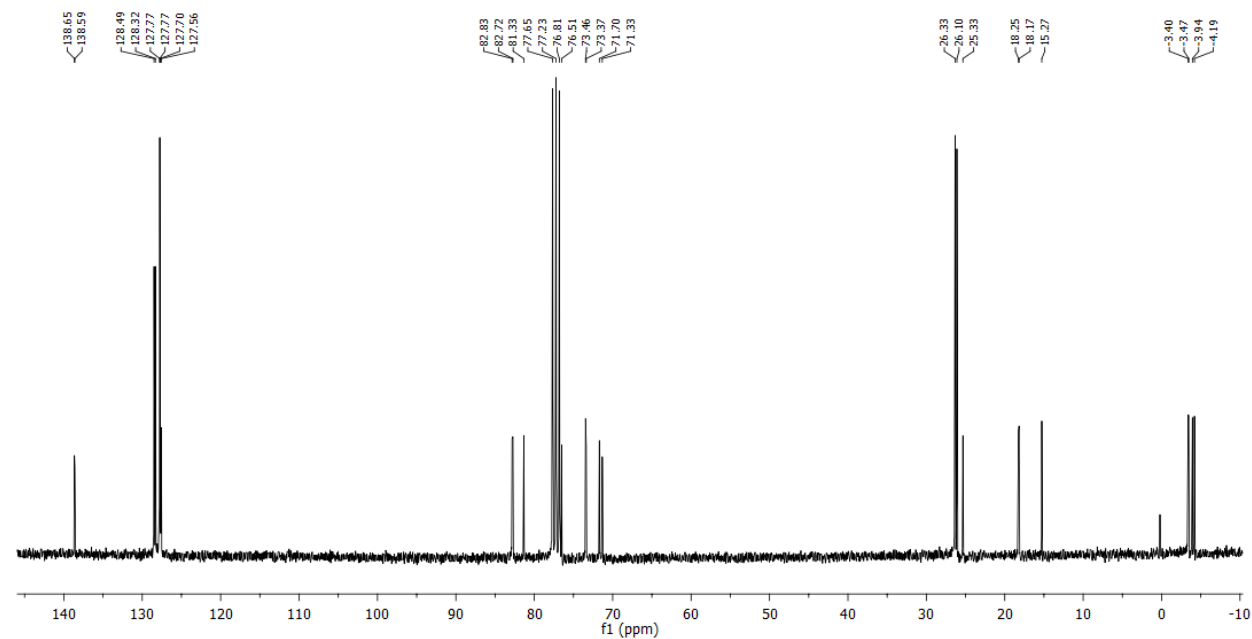
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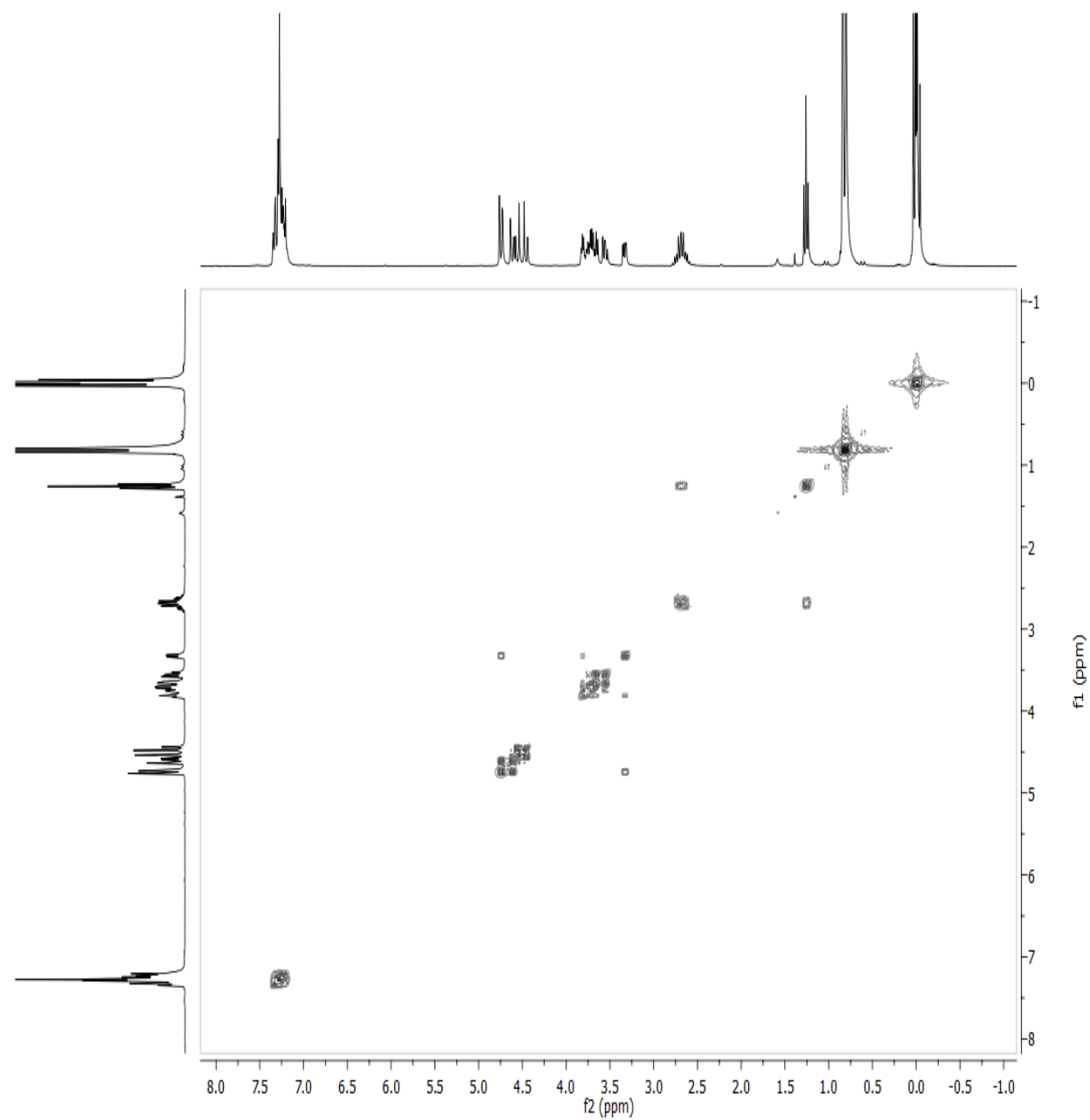
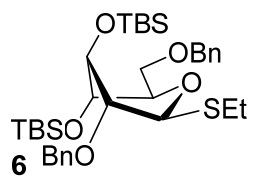
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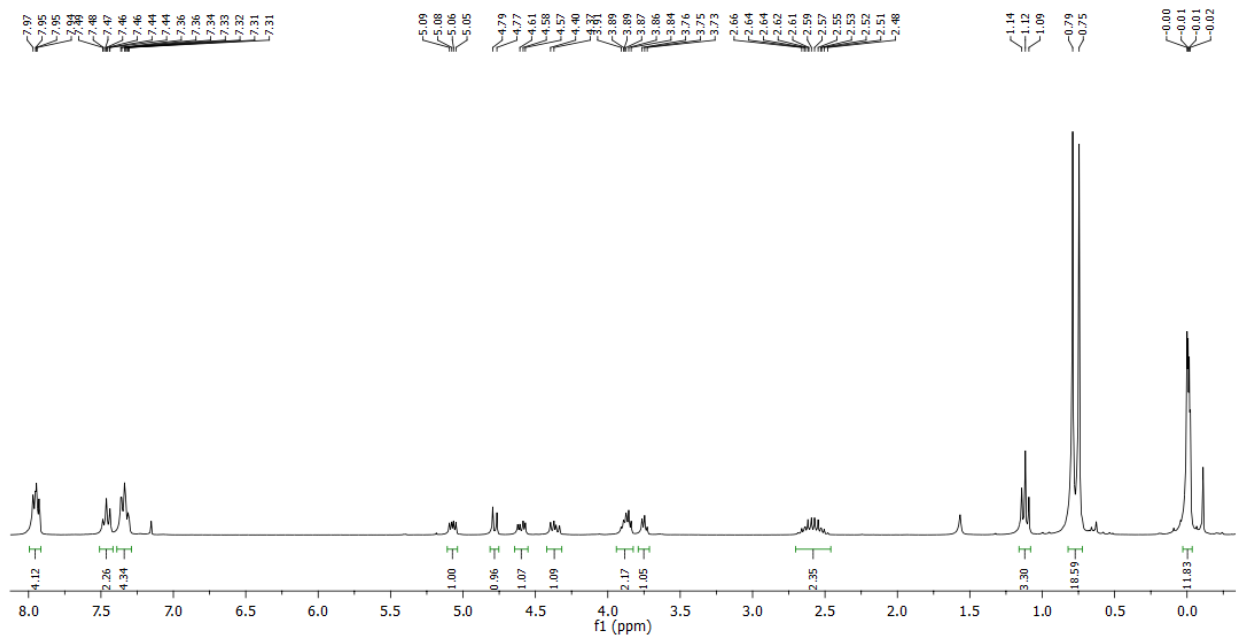
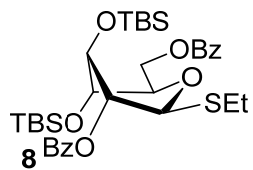
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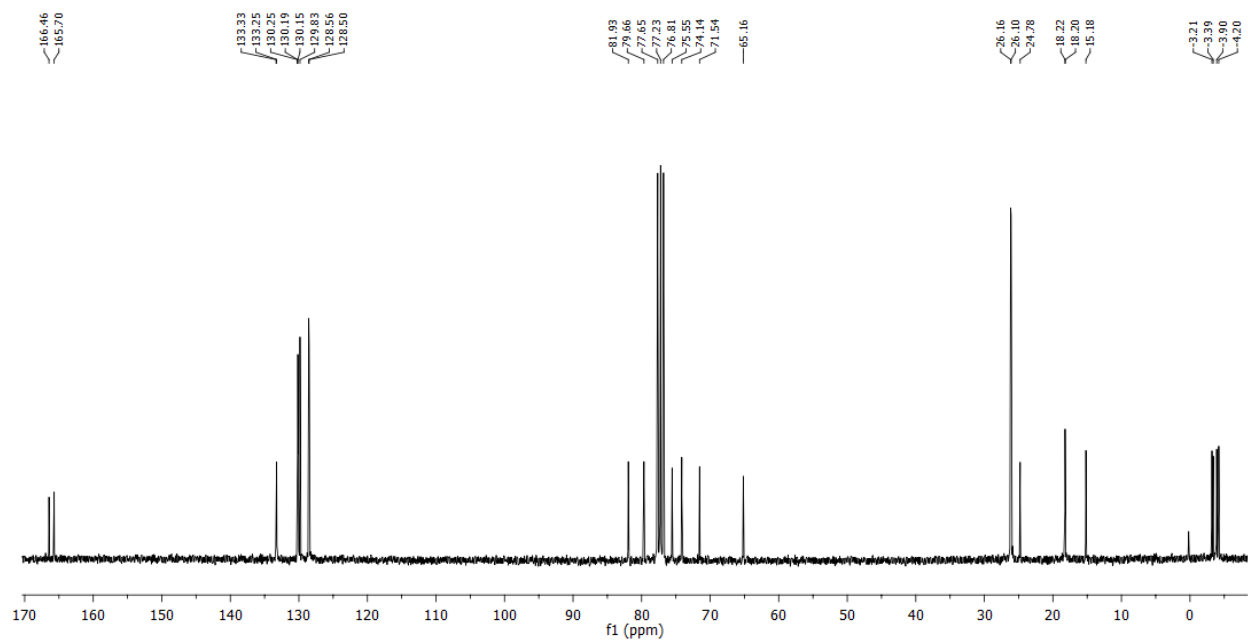
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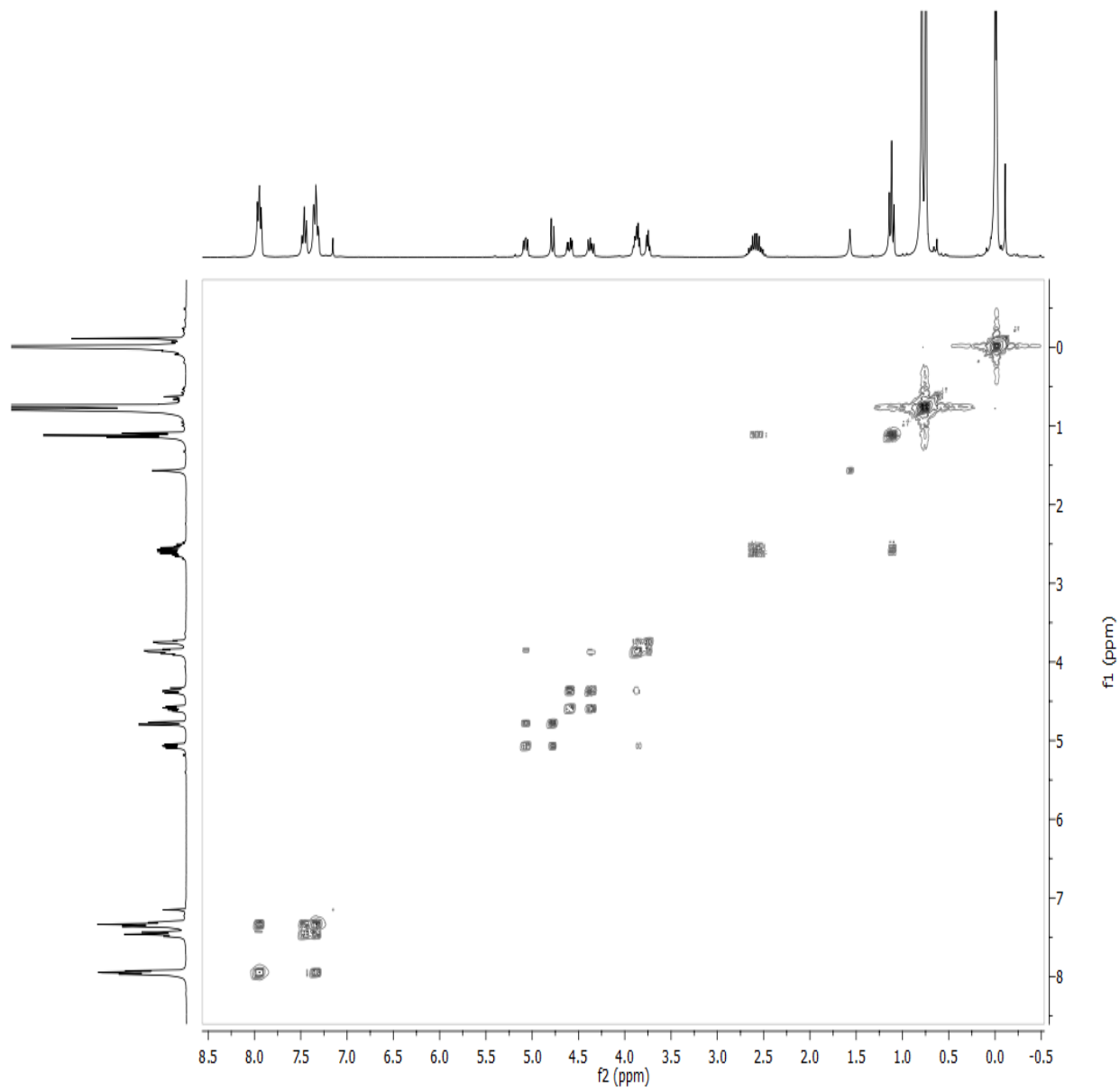
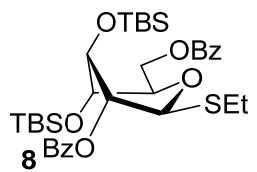
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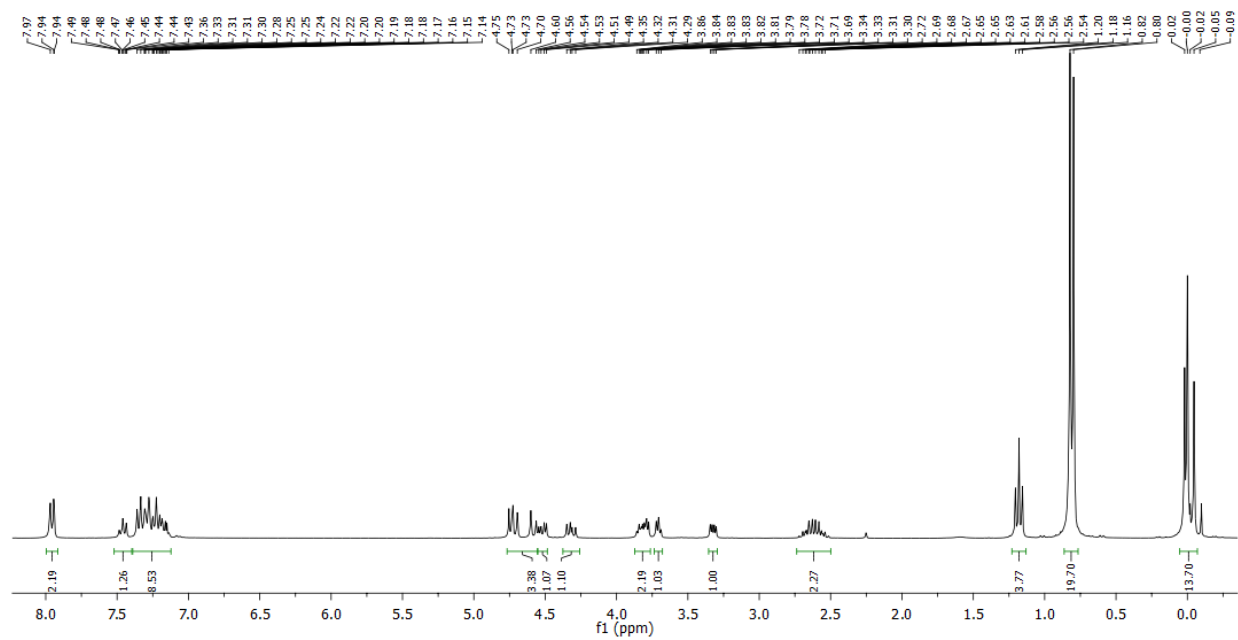
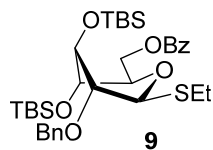
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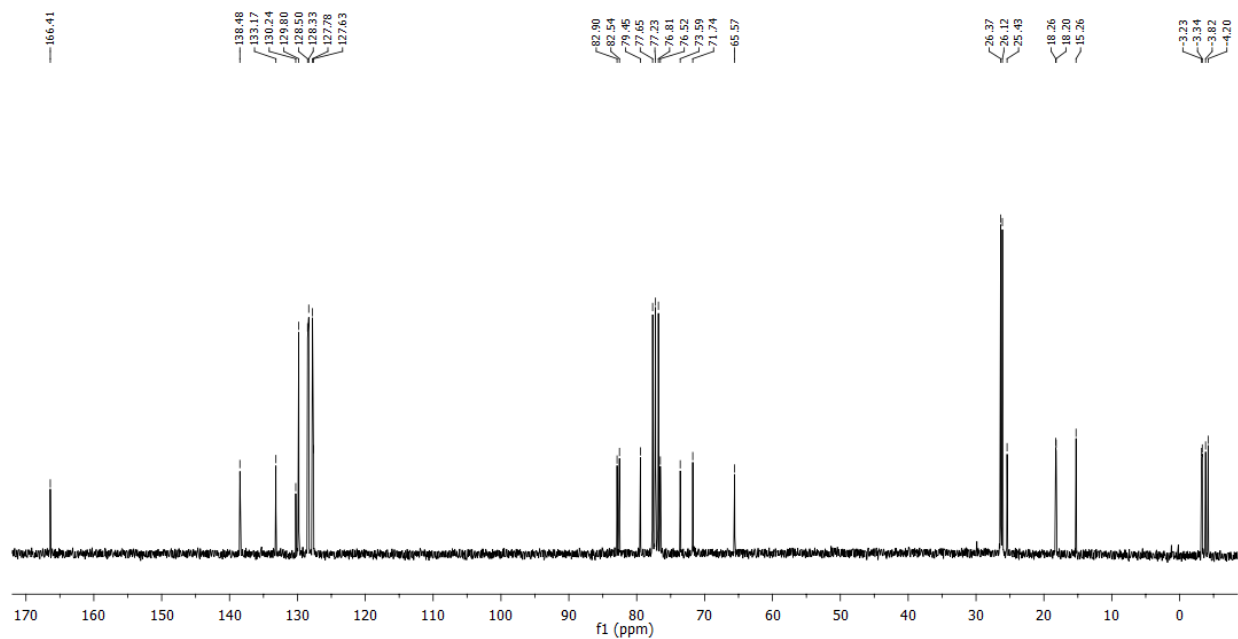
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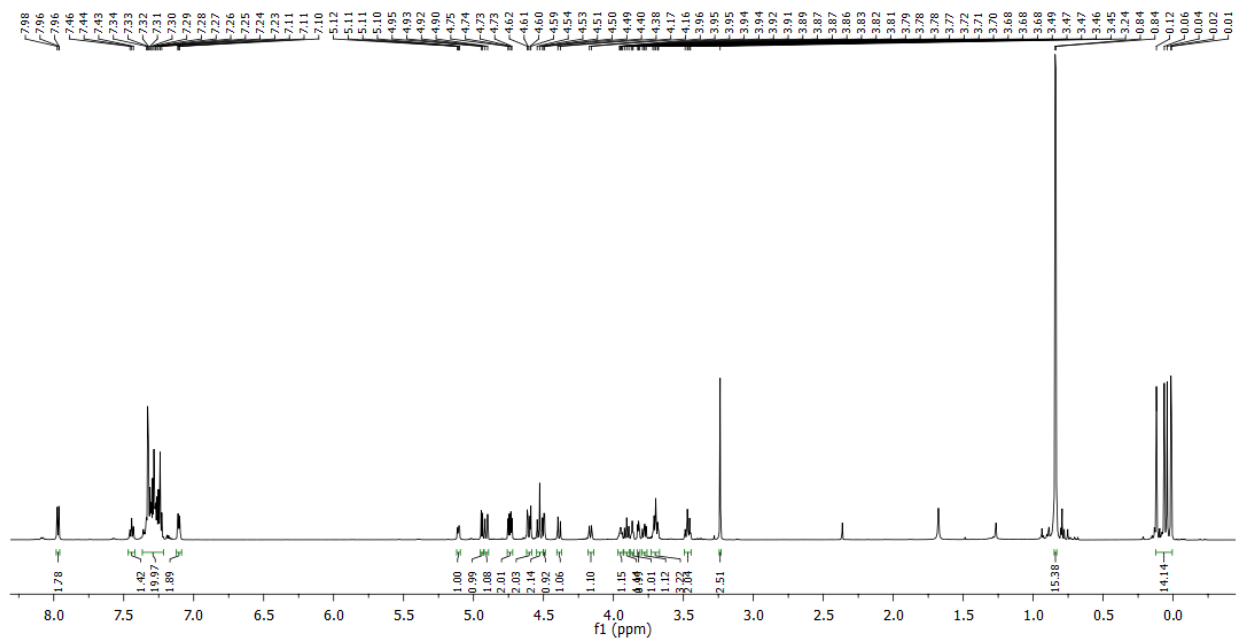
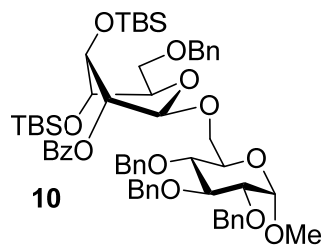
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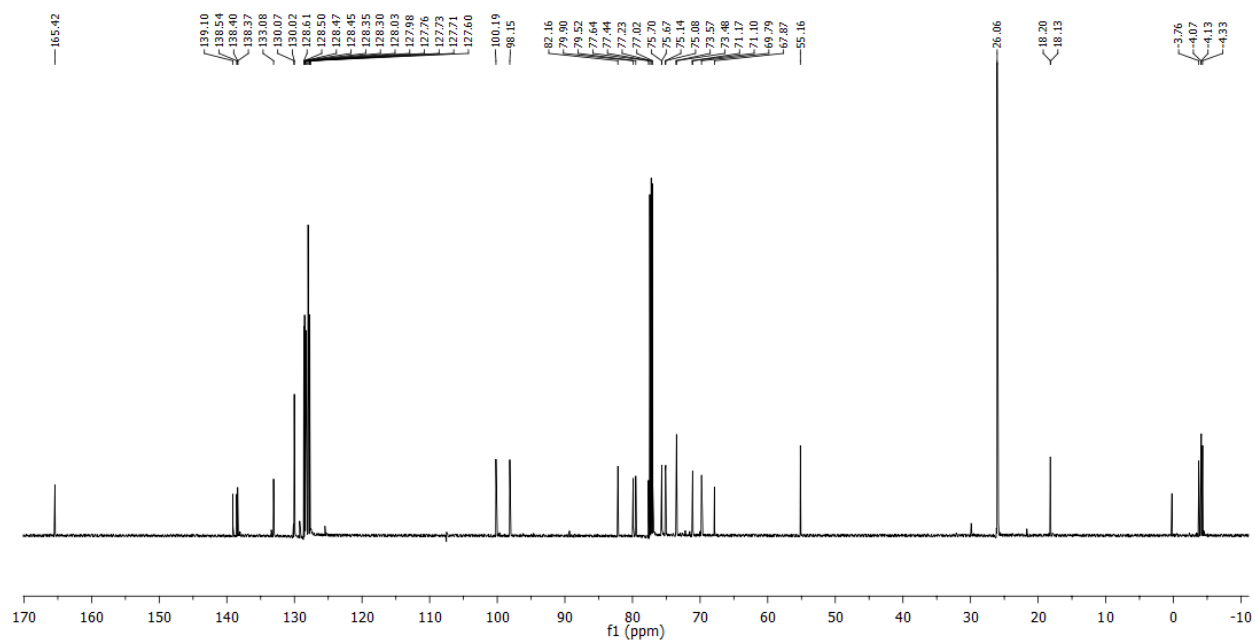
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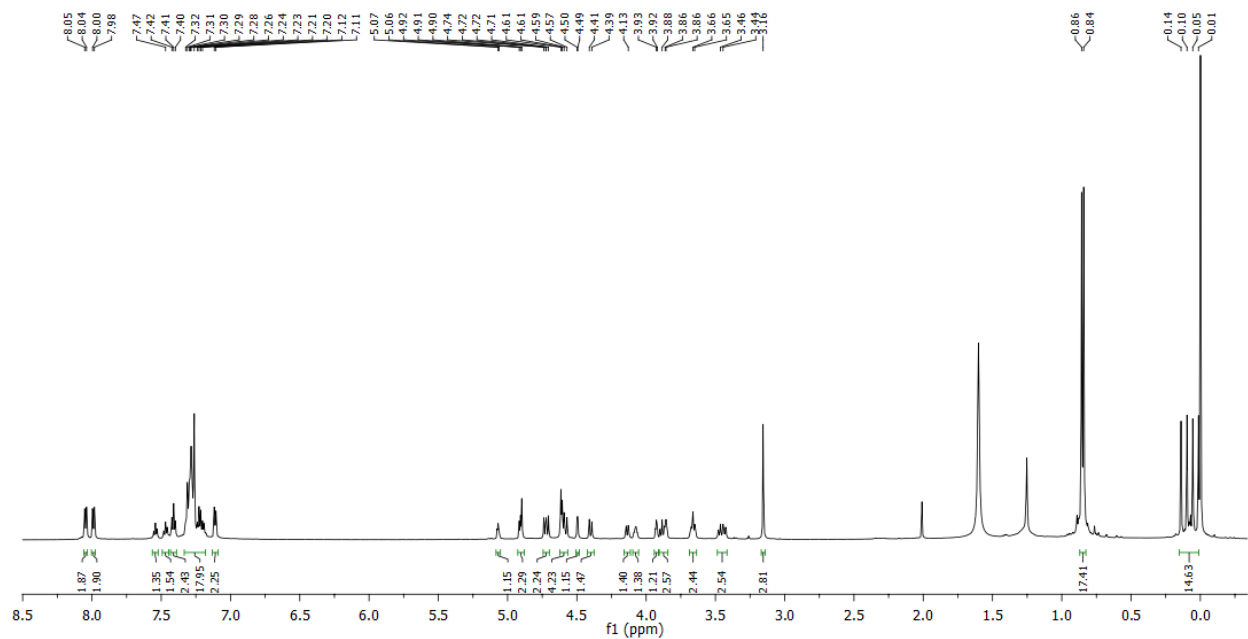
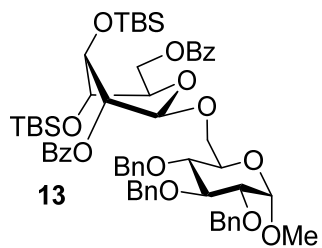
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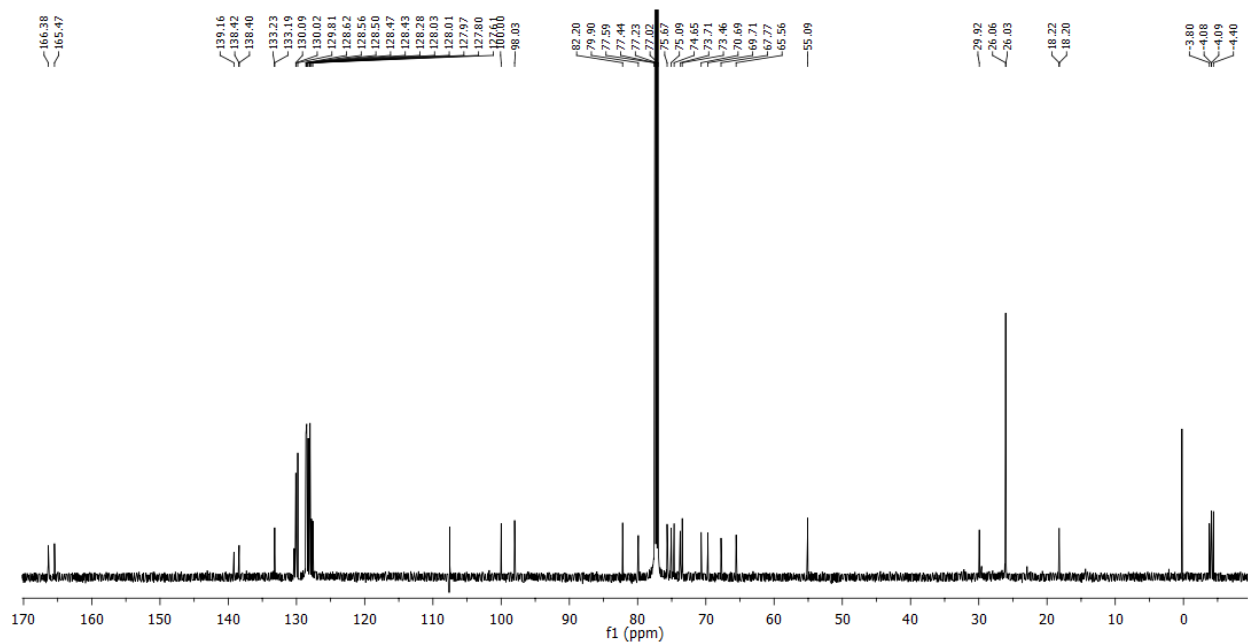
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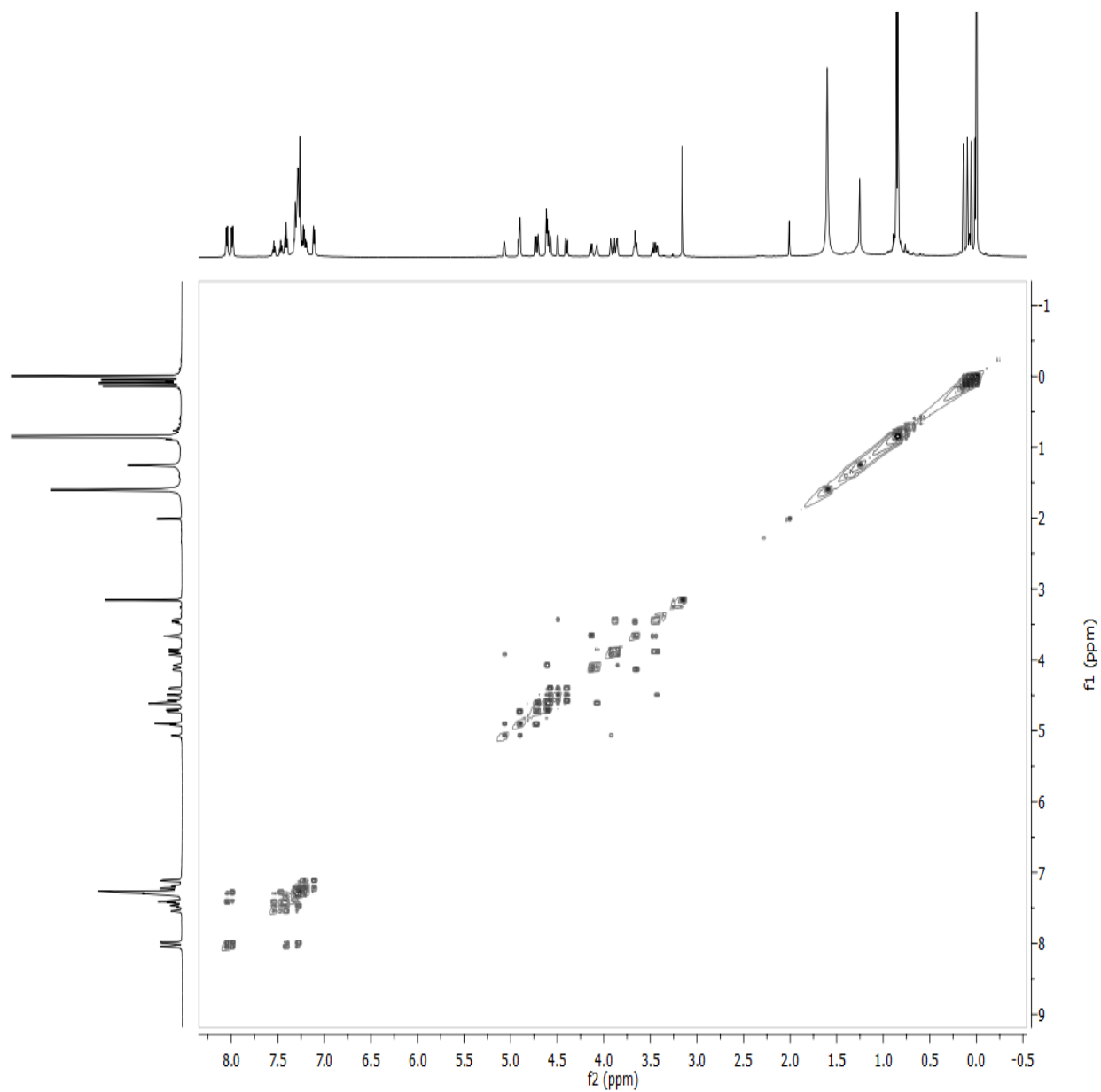
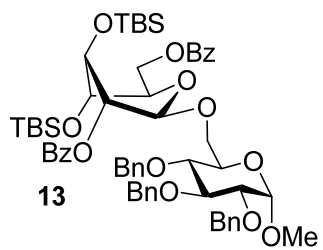
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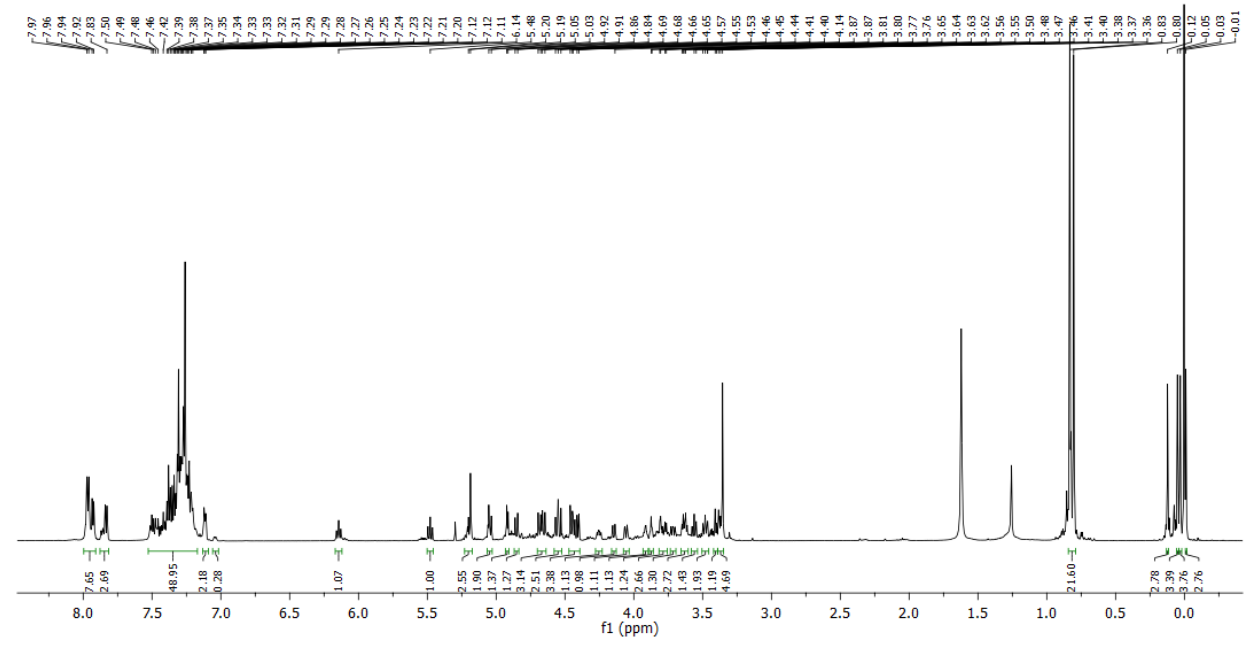
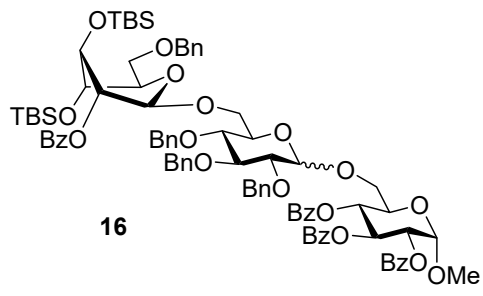
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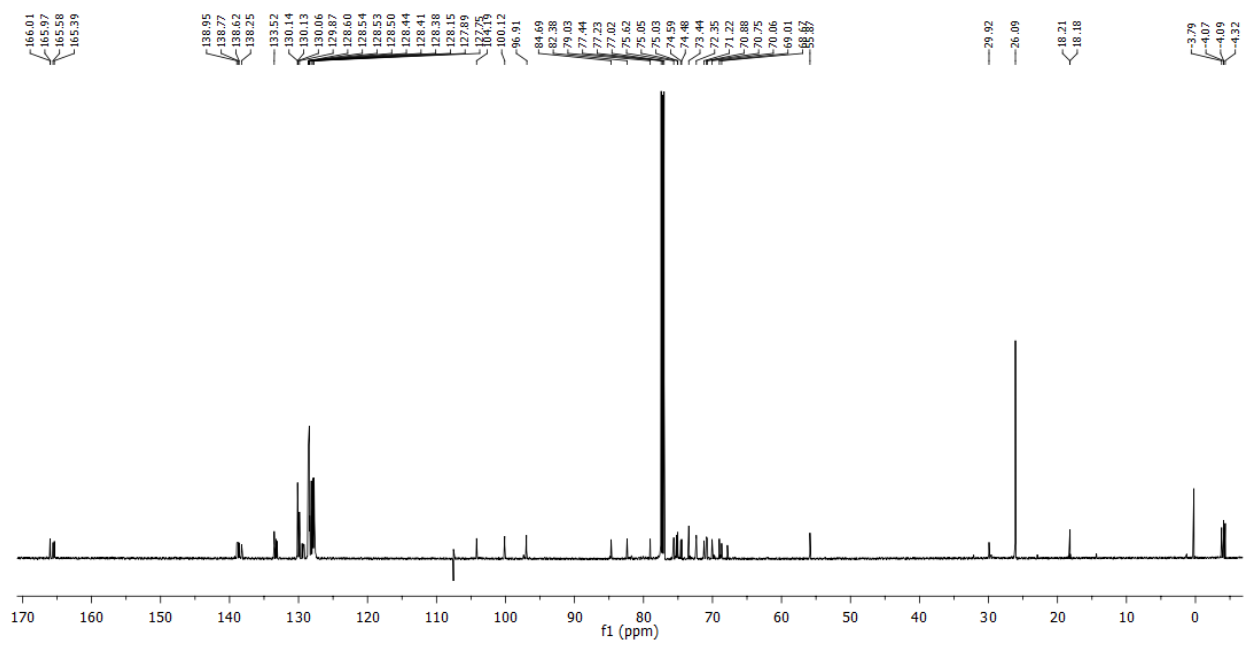
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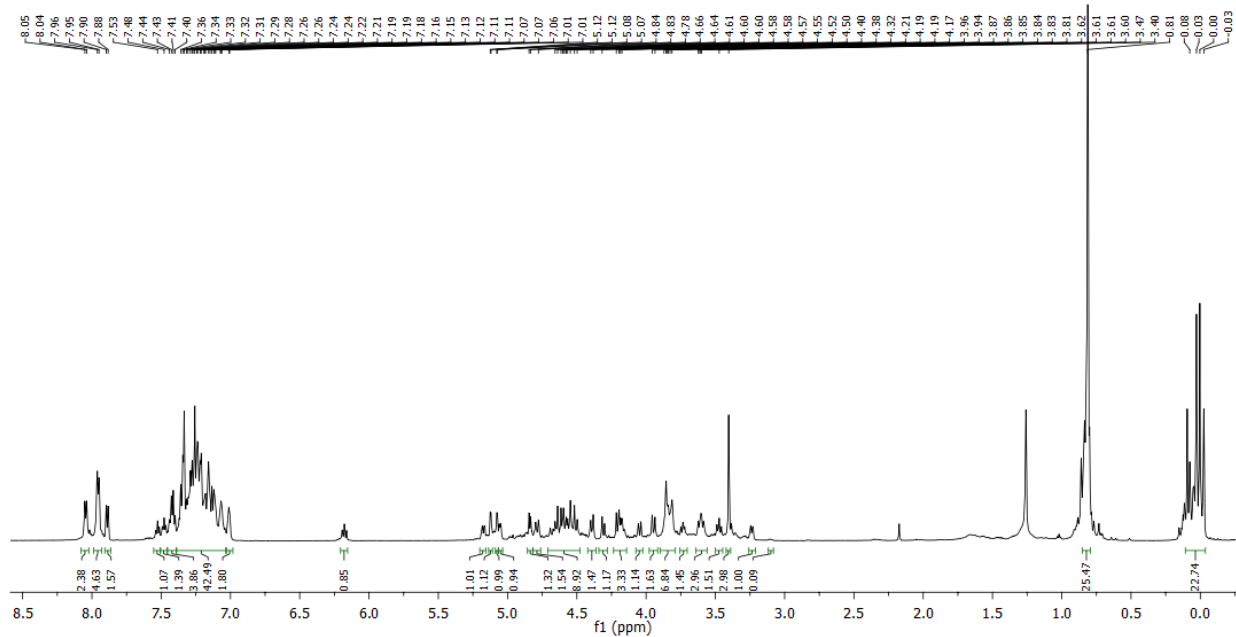
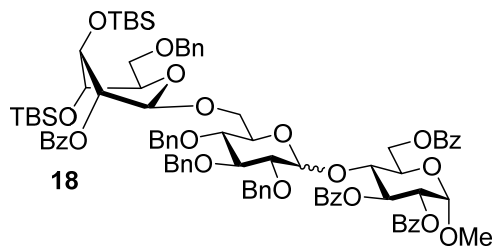
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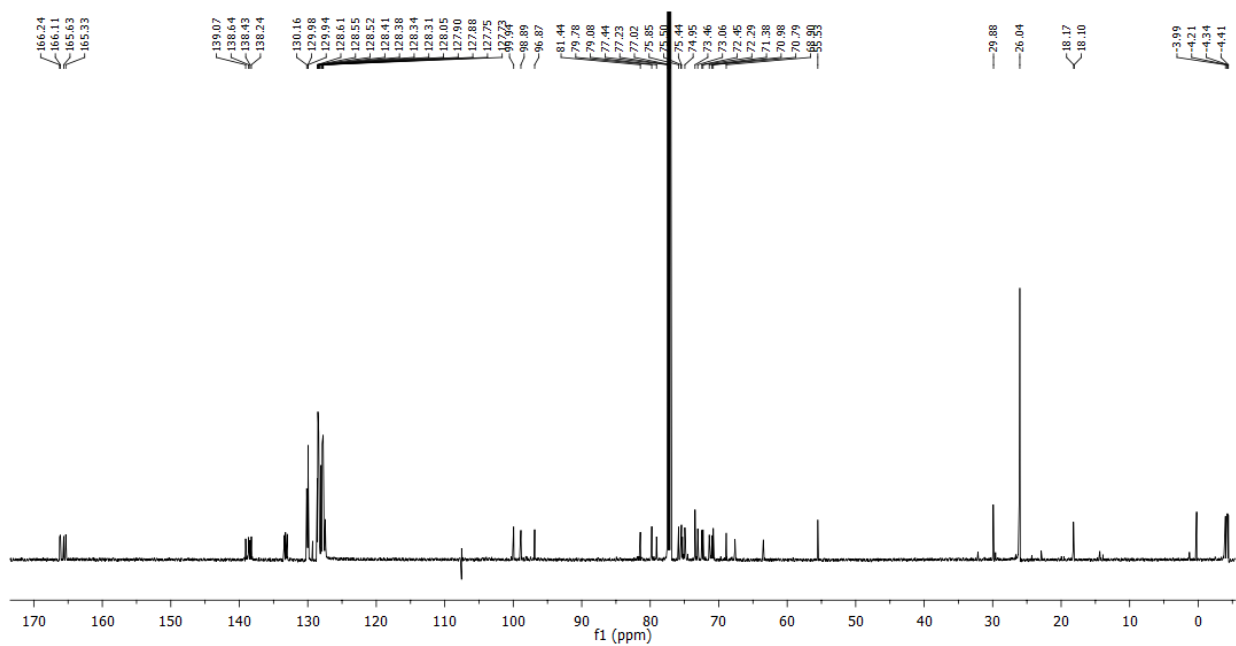
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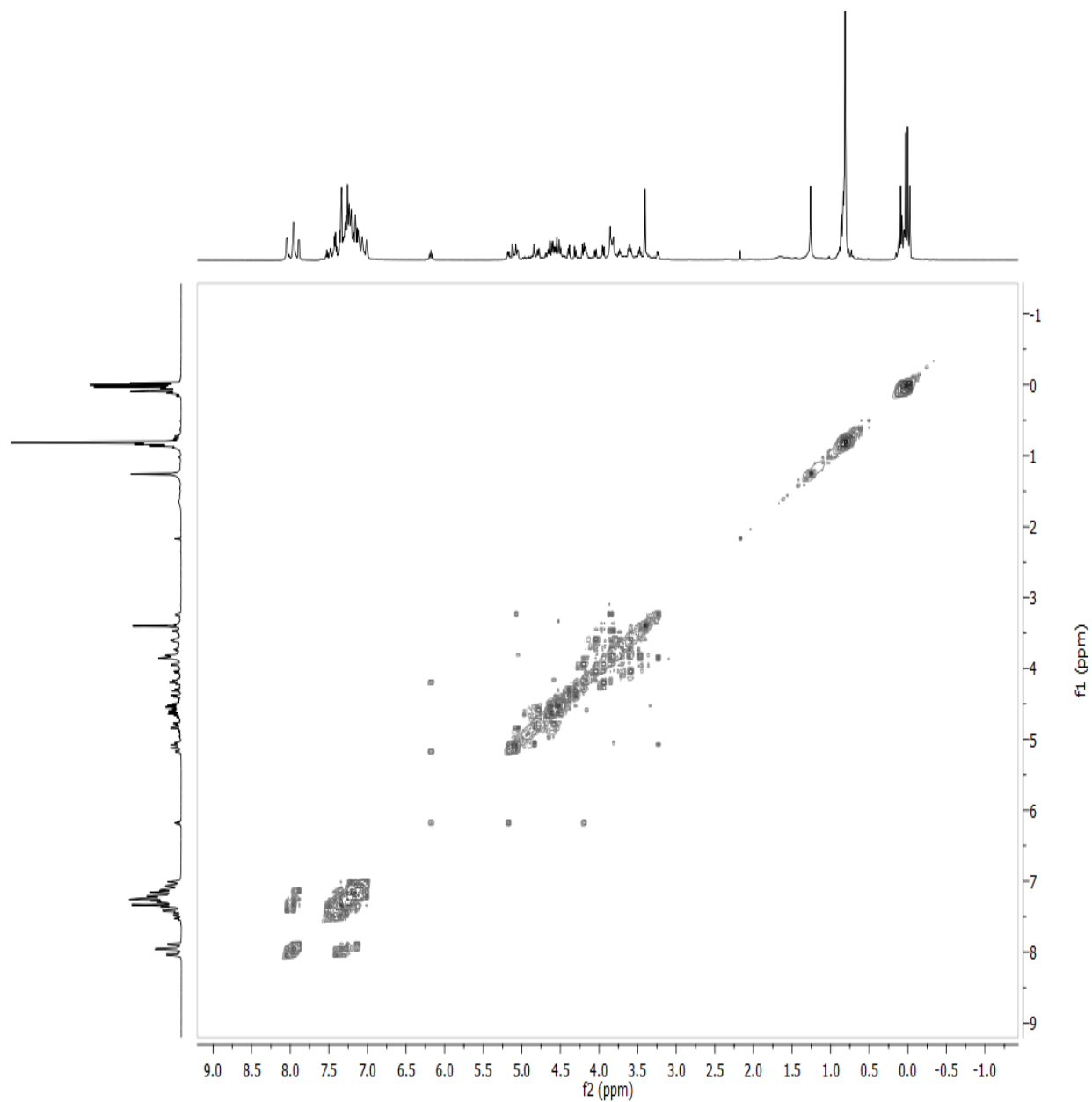
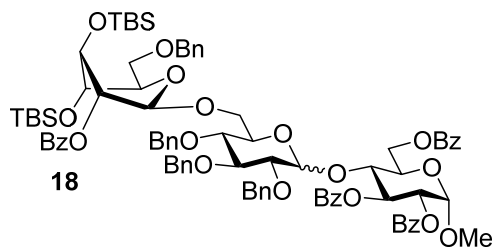


CDCl₃ 150MHz

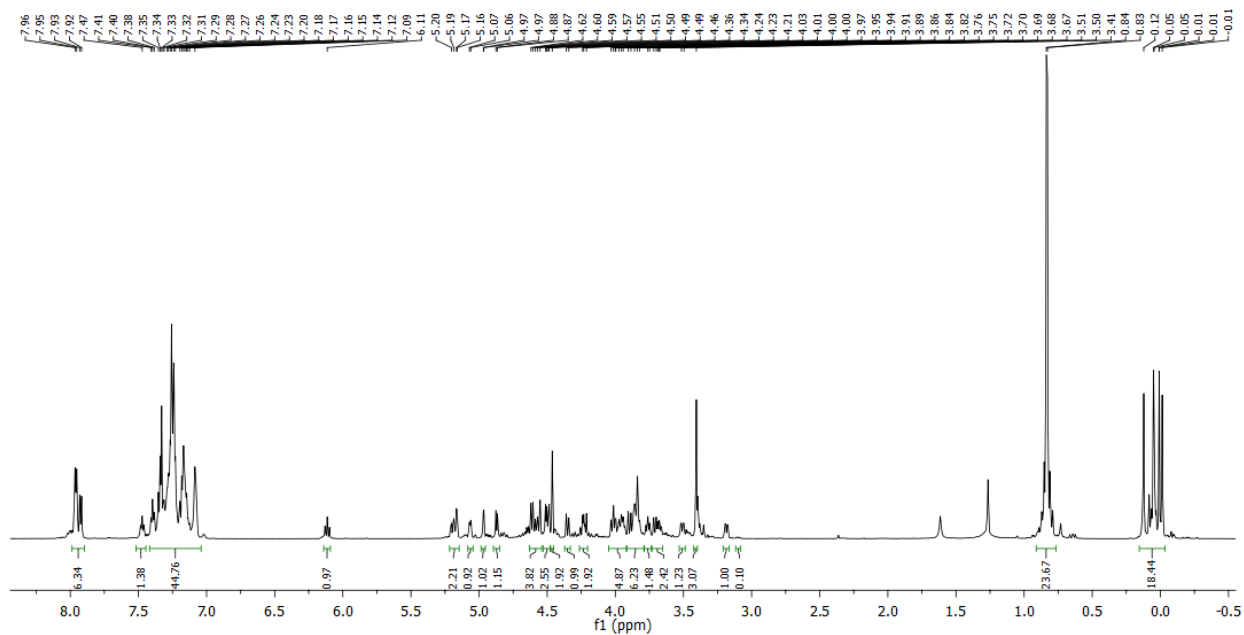
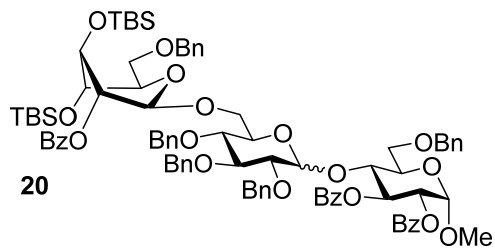


CDCl₃ 300 MHz

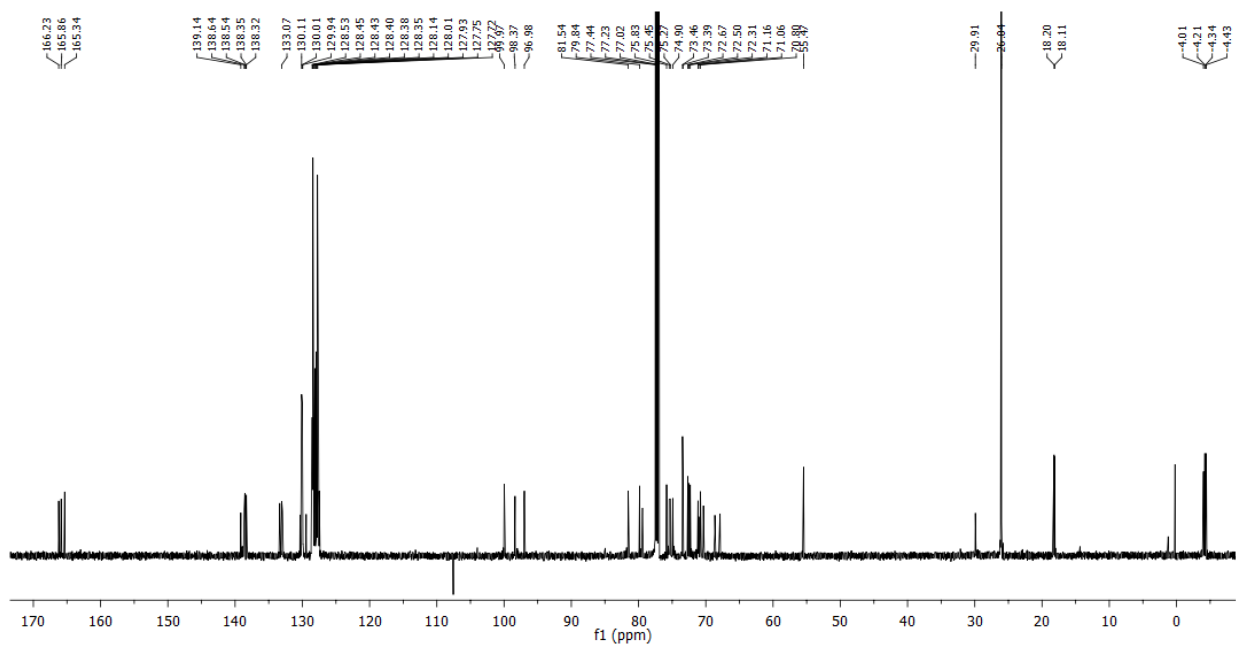




CDCl_3 600 MHz



CDCl₃ 300 MHz



CDCl₃ 150 MHz

X-Ray Data for Disaccharide 10

Table 1S. Crystal data and structure refinement for 10.

Identification code	a24115/lt/x8/100K/Mithila/M1	
Empirical formula	C ₆₀ H ₈₀ O ₁₂ Si ₂	
Formula weight	1049.42	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2 ₁	
Unit cell dimensions	a = 10.6177(6) Å	α = 90°.
	b = 12.3153(7) Å	β = 91.573(4)°.
	c = 22.9275(13) Å	γ = 90°.
Volume	2996.9(3) Å ³	
Z	2	
Density (calculated)	1.163 Mg/m ³	
Absorption coefficient	0.117 mm ⁻¹	
F(000)	1128	
Crystal size	0.324 x 0.121 x 0.098 mm ³	
Theta range for data collection	1.777 to 24.999°.	
Index ranges	-12 ≤ h ≤ 12, -14 ≤ k ≤ 14, -27 ≤ l ≤ 27	
Reflections collected	41545	
Independent reflections	10462 [R(int) = 0.0729]	
Completeness to theta = 24.999°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.8620 and 0.7753	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	10462 / 919 / 860	
Goodness-of-fit on F ²	1.017	
Final R indices [I > 2σ(I)]	R1 = 0.0616, wR2 = 0.1338	
R indices (all data)	R1 = 0.1417, wR2 = 0.1714	
Absolute structure parameter	-0.02(11)	
Largest diff. peak and hole	0.313 and -0.316 e.Å ⁻³	

Table 2S. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 10. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	864(4)	2589(4)	9027(2)	65(1)
O(2)	1542(4)	3465(4)	8183(2)	60(1)
O(3)	4620(4)	3987(4)	8878(2)	57(1)
O(4)	4610(4)	1689(4)	8816(2)	62(1)
O(5)	2131(4)	721(4)	8720(2)	58(1)
O(6)	3120(4)	5009(3)	7637(2)	52(1)
O(7)	2414(4)	6635(4)	7284(2)	67(1)
O(9)	4996(4)	7988(4)	7117(2)	54(1)
O(10)	3239(9)	6668(11)	5933(4)	43(3)
O(11)	5420(4)	5519(3)	6988(2)	44(1)
O(12)	5615(5)	4707(6)	6120(2)	86(2)
C(1)	1260(6)	2462(6)	8453(4)	61(2)
C(2)	2429(6)	1742(6)	8468(3)	59(2)
C(3)	3471(6)	2310(6)	8807(4)	59(2)
C(4)	3724(6)	3400(5)	8526(3)	56(2)
C(5)	2514(6)	4060(5)	8496(3)	57(2)
C(6)	2636(6)	5144(6)	8208(4)	62(2)
C(7)	3494(6)	5990(5)	7396(3)	48(2)
C(8)	4126(6)	5776(5)	6822(3)	50(2)
C(9)	4063(7)	6770(6)	6421(3)	65(2)
C(10)	3877(7)	7810(6)	6760(4)	66(2)
C(11)	2753(7)	7739(6)	7173(4)	71(2)
O(8)	1691(12)	9344(8)	7041(8)	47(4)
C(12)	1406(17)	8209(15)	7075(10)	46(5)
C(13)	822(15)	10024(12)	6730(9)	44(4)
C(14)	1565(10)	11005(7)	6566(7)	40(4)
C(15)	1790(10)	11821(8)	6974(6)	45(4)
C(16)	2569(10)	12688(8)	6841(7)	49(4)
C(17)	3125(10)	12739(9)	6300(7)	47(5)
C(18)	2900(9)	11923(10)	5892(7)	54(5)
C(19)	2120(10)	11056(8)	6024(7)	45(5)
O(8')	2108(10)	9402(7)	6686(7)	45(3)
C(12')	1708(16)	8331(14)	6790(11)	52(5)
C(13')	1150(14)	10013(13)	6372(11)	61(5)
C(14')	1704(10)	11091(7)	6198(8)	48(4)
C(15')	1861(10)	11893(8)	6620(7)	53(4)
C(16')	2464(10)	12859(7)	6485(7)	61(5)
C(17')	2909(9)	13023(7)	5927(8)	61(5)
C(18')	2751(8)	12220(9)	5505(7)	60(5)
C(19')	2149(9)	11254(8)	5641(8)	57(4)
C(20)	7315(15)	8260(13)	6365(6)	59(4)
C(21)	7323(16)	8526(17)	7686(7)	63(5)
C(22)	5998(10)	10214(8)	6911(5)	53(3)
C(23)	5445(12)	10498(12)	6308(6)	60(4)
C(24)	7194(11)	10905(11)	7011(7)	65(4)
C(25)	5025(12)	10553(13)	7358(7)	58(4)
C(26)	4173(12)	8126(9)	5076(6)	47(3)
C(27)	4561(12)	5714(11)	4978(6)	59(4)
C(28)	1868(10)	6637(10)	4878(5)	53(3)
C(29)	1317(15)	5539(12)	5056(7)	81(5)

C(30)	1965(14)	6658(14)	4211(5)	64(4)
C(31)	990(16)	7557(16)	5053(8)	74(5)
C(32)	6085(6)	4996(6)	6575(3)	54(2)
C(33)	7406(3)	4841(5)	6762(2)	53(2)
C(34)	7854(4)	5115(5)	7318(2)	69(2)
C(35)	9123(4)	4983(6)	7466(2)	89(3)
C(36)	9944(3)	4577(6)	7057(3)	84(3)
C(37)	9496(4)	4303(6)	6501(3)	116(4)
C(38)	8227(5)	4435(6)	6354(2)	108(4)
C(39)	5744(5)	4252(6)	8580(3)	56(2)
C(40)	6543(4)	4947(4)	8976(2)	53(2)
C(41)	7125(4)	4500(3)	9471(2)	60(2)
C(42)	7851(4)	5151(4)	9844(2)	71(2)
C(43)	7995(4)	6249(4)	9723(2)	80(2)
C(44)	7413(5)	6696(3)	9228(3)	86(3)
C(45)	6687(4)	6045(4)	8855(2)	73(2)
C(46)	4886(6)	1209(6)	9361(4)	77(2)
C(47)	6175(3)	680(4)	9377(2)	70(2)
C(48)	7149(4)	1079(4)	9045(3)	106(3)
C(49)	8344(4)	623(4)	9098(3)	106(3)
C(50)	8566(3)	-231(4)	9484(3)	84(3)
C(51)	7592(4)	-629(3)	9816(2)	64(2)
C(52)	6397(3)	-174(4)	9762(2)	59(2)
C(53)	2798(13)	-208(9)	8496(7)	50(4)
C(54)	2351(12)	-1226(9)	8778(6)	44(4)
C(55)	3131(9)	-1809(11)	9161(7)	71(5)
C(56)	2690(16)	-2748(9)	9422(6)	86(7)
C(57)	1468(17)	-3104(8)	9301(5)	73(5)
C(58)	688(9)	-2521(11)	8918(6)	52(5)
C(59)	1130(11)	-1582(11)	8656(6)	38(5)
C(53')	1940(20)	-101(10)	8251(6)	62(5)
C(59')	2018(14)	-1159(9)	8585(6)	38(4)
C(58')	937(9)	-1712(13)	8743(7)	44(6)
C(57')	1035(9)	-2634(11)	9091(7)	50(5)
C(56')	2213(13)	-3005(9)	9281(6)	58(5)
C(55')	3294(9)	-2452(12)	9123(6)	61(5)
C(54')	3196(10)	-1530(10)	8775(6)	51(4)
C(60)	-257(6)	3227(6)	9075(4)	67(2)
Si(1)	6389(6)	8727(5)	7001(3)	48(2)
Si(2)	3477(4)	6781(4)	5221(2)	44(1)
Si(1')	5875(6)	8968(5)	6978(3)	49(2)
Si(2')	2686(4)	6418(3)	5416(2)	46(1)
O(10')	2737(10)	6620(10)	6128(4)	41(3)
C(20')	5075(16)	10299(10)	6935(9)	68(5)
C(21')	6587(15)	8583(15)	6271(5)	60(4)
C(22')	7082(12)	8892(11)	7584(5)	47(4)
C(23')	7797(13)	7810(12)	7570(7)	57(4)
C(24')	8001(13)	9843(12)	7537(7)	68(4)
C(25')	6435(12)	8960(12)	8172(5)	49(3)
C(26')	2665(13)	4943(9)	5259(6)	58(4)
C(27')	4098(14)	7067(17)	5090(7)	67(5)
C(28')	1170(11)	7037(10)	5148(5)	53(4)
C(29')	1228(17)	8272(11)	5228(8)	76(5)
C(30')	985(16)	6803(15)	4494(6)	69(4)
C(31')	56(11)	6586(14)	5479(6)	66(4)

Table 3S. Bond lengths [Å] and angles [°] for 10.

O(1)-C(1)	1.403(9)	C(13)-H(13A)	0.9900
O(1)-C(60)	1.433(7)	C(13)-H(13B)	0.9900
O(2)-C(1)	1.417(8)	C(14)-C(15)	1.3900
O(2)-C(5)	1.441(8)	C(14)-C(19)	1.3900
O(3)-C(4)	1.427(8)	C(15)-C(16)	1.3900
O(3)-C(39)	1.430(7)	C(15)-H(15A)	0.9500
O(4)-C(46)	1.405(9)	C(16)-C(17)	1.3900
O(4)-C(3)	1.431(7)	C(16)-H(16A)	0.9500
O(5)-C(2)	1.422(8)	C(17)-C(18)	1.3900
O(5)-C(53)	1.448(11)	C(17)-H(17A)	0.9500
O(5)-C(53')	1.489(12)	C(18)-C(19)	1.3900
O(6)-C(7)	1.390(7)	C(18)-H(18A)	0.9500
O(6)-C(6)	1.429(8)	C(19)-H(19A)	0.9500
O(7)-C(7)	1.413(7)	O(8')-C(12')	1.408(18)
O(7)-C(11)	1.431(9)	O(8')-C(13')	1.442(15)
O(9)-C(10)	1.440(7)	C(12')-H(12C)	0.9900
O(9)-Si(1')	1.564(7)	C(12')-H(12D)	0.9900
O(9)-Si(1)	1.763(7)	C(13')-C(14')	1.510(17)
O(10)-C(9)	1.408(11)	C(13')-H(13C)	0.9900
O(10)-Si(2)	1.664(9)	C(13')-H(13D)	0.9900
O(11)-C(32)	1.359(8)	C(14')-C(15')	1.3900
O(11)-C(8)	1.450(7)	C(14')-C(19')	1.3900
O(12)-C(32)	1.199(8)	C(15')-C(16')	1.3900
C(1)-C(2)	1.525(9)	C(15')-H(15B)	0.9500
C(1)-H(1A)	1.0000	C(16')-C(17')	1.3900
C(2)-C(3)	1.507(9)	C(16')-H(16B)	0.9500
C(2)-H(2A)	1.0000	C(17')-C(18')	1.3900
C(3)-C(4)	1.516(10)	C(17')-H(17B)	0.9500
C(3)-H(3A)	1.0000	C(18')-C(19')	1.3900
C(4)-C(5)	1.521(9)	C(18')-H(18B)	0.9500
C(4)-H(4A)	1.0000	C(19')-H(19B)	0.9500
C(5)-C(6)	1.496(10)	C(20)-Si(1)	1.871(11)
C(5)-H(5A)	1.0000	C(20)-H(20A)	0.9800
C(6)-H(6A)	0.9900	C(20)-H(20B)	0.9800
C(6)-H(6B)	0.9900	C(20)-H(20C)	0.9800
C(7)-C(8)	1.517(9)	C(21)-Si(1)	1.851(12)
C(7)-H(7A)	1.0000	C(21)-H(21A)	0.9800
C(8)-C(9)	1.532(10)	C(21)-H(21B)	0.9800
C(8)-H(8A)	1.0000	C(21)-H(21C)	0.9800
C(9)-C(10)	1.514(10)	C(22)-C(23)	1.529(10)
C(9)-O(10')	1.555(12)	C(22)-C(25)	1.533(10)
C(9)-H(9)	1.0000	C(22)-C(24)	1.541(10)
C(10)-C(11)	1.545(11)	C(22)-Si(1)	1.888(10)
C(10)-H(10A)	1.0000	C(23)-H(23A)	0.9800
C(11)-C(12)	1.553(18)	C(23)-H(23B)	0.9800
C(11)-C(12')	1.574(17)	C(23)-H(23C)	0.9800
C(11)-H(11)	1.0000	C(24)-H(24A)	0.9800
O(8)-C(13)	1.424(16)	C(24)-H(24B)	0.9800
O(8)-C(12)	1.43(2)	C(24)-H(24C)	0.9800
C(12)-H(12A)	0.9900	C(25)-H(25A)	0.9800
C(12)-H(12B)	0.9900	C(25)-H(25B)	0.9800
C(13)-C(14)	1.497(17)	C(25)-H(25C)	0.9800
		C(26)-Si(2)	1.847(10)

C(26)-H(26A)	0.9800	C(50)-C(51)	1.3900
C(26)-H(26B)	0.9800	C(50)-H(50A)	0.9500
C(26)-H(26C)	0.9800	C(51)-C(52)	1.3900
C(27)-Si(2)	1.843(10)	C(51)-H(51A)	0.9500
C(27)-H(27A)	0.9800	C(52)-H(52A)	0.9500
C(27)-H(27B)	0.9800	C(53)-C(54)	1.494(12)
C(27)-H(27C)	0.9800	C(53)-H(53A)	0.9900
C(28)-C(31)	1.528(11)	C(53)-H(53B)	0.9900
C(28)-C(29)	1.534(10)	C(54)-C(55)	1.3900
C(28)-C(30)	1.535(10)	C(54)-C(59)	1.3900
C(28)-Si(2)	1.869(10)	C(55)-C(56)	1.3900
C(29)-H(29A)	0.9800	C(55)-H(55A)	0.9500
C(29)-H(29B)	0.9800	C(56)-C(57)	1.3900
C(29)-H(29C)	0.9800	C(56)-H(56A)	0.9500
C(30)-H(30A)	0.9800	C(57)-C(58)	1.3900
C(30)-H(30B)	0.9800	C(57)-H(57A)	0.9500
C(30)-H(30C)	0.9800	C(58)-C(59)	1.3900
C(31)-H(31A)	0.9800	C(58)-H(58A)	0.9500
C(31)-H(31B)	0.9800	C(59)-H(59A)	0.9500
C(31)-H(31C)	0.9800	C(53')-C(59')	1.513(13)
C(32)-C(33)	1.468(8)	C(53')-H(53C)	0.9900
C(33)-C(34)	1.3900	C(53')-H(53D)	0.9900
C(33)-C(38)	1.3900	C(59')-C(58')	1.3900
C(34)-C(35)	1.3900	C(59')-C(54')	1.3900
C(34)-H(34A)	0.9500	C(58')-C(57')	1.3900
C(35)-C(36)	1.3900	C(58')-H(58B)	0.9500
C(35)-H(35A)	0.9500	C(57')-C(56')	1.3900
C(36)-C(37)	1.3900	C(57')-H(57B)	0.9500
C(36)-H(36A)	0.9500	C(56')-C(55')	1.3900
C(37)-C(38)	1.3900	C(56')-H(56B)	0.9500
C(37)-H(37A)	0.9500	C(55')-C(54')	1.3900
C(38)-H(38A)	0.9500	C(55')-H(55B)	0.9500
C(39)-C(40)	1.495(8)	C(54')-H(54A)	0.9500
C(39)-H(39A)	0.9900	C(60)-H(60A)	0.9800
C(39)-H(39B)	0.9900	C(60)-H(60B)	0.9800
C(40)-C(41)	1.3900	C(60)-H(60C)	0.9800
C(40)-C(45)	1.3900	Si(1')-C(20')	1.847(11)
C(41)-C(42)	1.3900	Si(1')-C(22')	1.867(11)
C(41)-H(41A)	0.9500	Si(1')-C(21')	1.869(11)
C(42)-C(43)	1.3900	Si(2')-O(10')	1.651(9)
C(42)-H(42A)	0.9500	Si(2')-C(26')	1.852(11)
C(43)-C(44)	1.3900	Si(2')-C(28')	1.869(11)
C(43)-H(43A)	0.9500	Si(2')-C(27')	1.872(12)
C(44)-C(45)	1.3900	C(20')-H(20D)	0.9800
C(44)-H(44A)	0.9500	C(20')-H(20E)	0.9800
C(45)-H(45A)	0.9500	C(20')-H(20F)	0.9800
C(46)-C(47)	1.516(7)	C(21')-H(21D)	0.9800
C(46)-H(46A)	0.9900	C(21')-H(21E)	0.9800
C(46)-H(46B)	0.9900	C(21')-H(21F)	0.9800
C(47)-C(48)	1.3900	C(22')-C(24')	1.530(10)
C(47)-C(52)	1.3900	C(22')-C(25')	1.533(10)
C(48)-C(49)	1.3900	C(22')-C(23')	1.534(10)
C(48)-H(48A)	0.9500	C(23')-H(23D)	0.9800
C(49)-C(50)	1.3900	C(23')-H(23E)	0.9800
C(49)-H(49A)	0.9500	C(23')-H(23F)	0.9800

C(24')-H(24D)	0.9800	O(3)-C(4)-C(3)	109.4(6)
C(24')-H(24E)	0.9800	O(3)-C(4)-C(5)	107.7(5)
C(24')-H(24F)	0.9800	C(3)-C(4)-C(5)	109.5(5)
C(25')-H(25D)	0.9800	O(3)-C(4)-H(4A)	110.1
C(25')-H(25E)	0.9800	C(3)-C(4)-H(4A)	110.1
C(25')-H(25F)	0.9800	C(5)-C(4)-H(4A)	110.1
C(26')-H(26D)	0.9800	O(2)-C(5)-C(6)	107.6(6)
C(26')-H(26E)	0.9800	O(2)-C(5)-C(4)	110.1(5)
C(26')-H(26F)	0.9800	C(6)-C(5)-C(4)	114.5(5)
C(27')-H(27D)	0.9800	O(2)-C(5)-H(5A)	108.1
C(27')-H(27E)	0.9800	C(6)-C(5)-H(5A)	108.1
C(27')-H(27F)	0.9800	C(4)-C(5)-H(5A)	108.1
C(28')-C(31')	1.527(10)	O(6)-C(6)-C(5)	109.7(5)
C(28')-C(29')	1.533(11)	O(6)-C(6)-H(6A)	109.7
C(28')-C(30')	1.534(10)	C(5)-C(6)-H(6A)	109.7
C(29')-H(29D)	0.9800	O(6)-C(6)-H(6B)	109.7
C(29')-H(29E)	0.9800	C(5)-C(6)-H(6B)	109.7
C(29')-H(29F)	0.9800	H(6A)-C(6)-H(6B)	108.2
C(30')-H(30D)	0.9800	O(6)-C(7)-O(7)	108.8(5)
C(30')-H(30E)	0.9800	O(6)-C(7)-C(8)	109.3(5)
C(30')-H(30F)	0.9800	O(7)-C(7)-C(8)	108.4(5)
C(31')-H(31D)	0.9800	O(6)-C(7)-H(7A)	110.1
C(31')-H(31E)	0.9800	O(7)-C(7)-H(7A)	110.1
C(31')-H(31F)	0.9800	C(8)-C(7)-H(7A)	110.1
		O(11)-C(8)-C(7)	104.4(5)
C(1)-O(1)-C(60)	113.9(6)	O(11)-C(8)-C(9)	111.0(5)
C(1)-O(2)-C(5)	112.5(6)	C(7)-C(8)-C(9)	111.6(6)
C(4)-O(3)-C(39)	113.4(5)	O(11)-C(8)-H(8A)	109.9
C(46)-O(4)-C(3)	113.2(5)	C(7)-C(8)-H(8A)	109.9
C(2)-O(5)-C(53)	115.9(6)	C(9)-C(8)-H(8A)	109.9
C(2)-O(5)-C(53')	109.6(7)	O(10)-C(9)-C(10)	113.4(7)
C(7)-O(6)-C(6)	112.0(5)	O(10)-C(9)-C(8)	114.9(7)
C(7)-O(7)-C(11)	111.1(5)	C(10)-C(9)-C(8)	111.9(6)
C(10)-O(9)-Si(1')	119.2(5)	C(10)-C(9)-O(10')	101.2(7)
C(10)-O(9)-Si(1)	132.4(5)	C(8)-C(9)-O(10')	100.9(7)
C(9)-O(10)-Si(2)	131.7(8)	O(10)-C(9)-H(9)	105.2
C(32)-O(11)-C(8)	115.3(5)	C(10)-C(9)-H(9)	105.2
O(1)-C(1)-O(2)	112.7(5)	C(8)-C(9)-H(9)	105.2
O(1)-C(1)-C(2)	107.9(6)	O(9)-C(10)-C(9)	107.6(6)
O(2)-C(1)-C(2)	109.7(5)	O(9)-C(10)-C(11)	107.5(6)
O(1)-C(1)-H(1A)	108.8	C(9)-C(10)-C(11)	112.3(6)
O(2)-C(1)-H(1A)	108.8	O(9)-C(10)-H(10A)	109.8
C(2)-C(1)-H(1A)	108.8	C(9)-C(10)-H(10A)	109.8
O(5)-C(2)-C(3)	111.7(6)	C(11)-C(10)-H(10A)	109.8
O(5)-C(2)-C(1)	109.5(5)	O(7)-C(11)-C(10)	111.4(6)
C(3)-C(2)-C(1)	109.1(6)	O(7)-C(11)-C(12)	98.3(9)
O(5)-C(2)-H(2A)	108.8	C(10)-C(11)-C(12)	127.9(11)
C(3)-C(2)-H(2A)	108.8	O(7)-C(11)-C(12')	111.2(8)
C(1)-C(2)-H(2A)	108.8	C(10)-C(11)-C(12')	100.2(10)
O(4)-C(3)-C(2)	111.6(6)	O(7)-C(11)-H(11)	105.8
O(4)-C(3)-C(4)	108.6(5)	C(10)-C(11)-H(11)	105.8
C(2)-C(3)-C(4)	109.2(6)	C(12)-C(11)-H(11)	105.8
O(4)-C(3)-H(3A)	109.1	C(13)-O(8)-C(12)	117.8(12)
C(2)-C(3)-H(3A)	109.1	O(8)-C(12)-C(11)	100.1(12)
C(4)-C(3)-H(3A)	109.1	O(8)-C(12)-H(12A)	111.7

C(11)-C(12)-H(12A)	111.7	C(17')-C(18')-H(18B)	120.0
O(8)-C(12)-H(12B)	111.7	C(18')-C(19')-C(14')	120.0
C(11)-C(12)-H(12B)	111.7	C(18')-C(19')-H(19B)	120.0
H(12A)-C(12)-H(12B)	109.5	C(14')-C(19')-H(19B)	120.0
O(8)-C(13)-C(14)	105.2(11)	Si(1)-C(20)-H(20A)	109.5
O(8)-C(13)-H(13A)	110.7	Si(1)-C(20)-H(20B)	109.5
C(14)-C(13)-H(13A)	110.7	H(20A)-C(20)-H(20B)	109.5
O(8)-C(13)-H(13B)	110.7	Si(1)-C(20)-H(20C)	109.5
C(14)-C(13)-H(13B)	110.7	H(20A)-C(20)-H(20C)	109.5
H(13A)-C(13)-H(13B)	108.8	H(20B)-C(20)-H(20C)	109.5
C(15)-C(14)-C(19)	120.0	Si(1)-C(21)-H(21A)	109.5
C(15)-C(14)-C(13)	119.7(9)	Si(1)-C(21)-H(21B)	109.5
C(19)-C(14)-C(13)	120.0(9)	H(21A)-C(21)-H(21B)	109.5
C(14)-C(15)-C(16)	120.0	Si(1)-C(21)-H(21C)	109.5
C(14)-C(15)-H(15A)	120.0	H(21A)-C(21)-H(21C)	109.5
C(16)-C(15)-H(15A)	120.0	H(21B)-C(21)-H(21C)	109.5
C(17)-C(16)-C(15)	120.0	C(23)-C(22)-C(25)	107.0(11)
C(17)-C(16)-H(16A)	120.0	C(23)-C(22)-C(24)	107.6(10)
C(15)-C(16)-H(16A)	120.0	C(25)-C(22)-C(24)	108.6(11)
C(16)-C(17)-C(18)	120.0	C(23)-C(22)-Si(1)	113.4(8)
C(16)-C(17)-H(17A)	120.0	C(25)-C(22)-Si(1)	110.0(9)
C(18)-C(17)-H(17A)	120.0	C(24)-C(22)-Si(1)	110.0(8)
C(19)-C(18)-C(17)	120.0	C(22)-C(23)-H(23A)	109.5
C(19)-C(18)-H(18A)	120.0	C(22)-C(23)-H(23B)	109.5
C(17)-C(18)-H(18A)	120.0	H(23A)-C(23)-H(23B)	109.5
C(18)-C(19)-C(14)	120.0	C(22)-C(23)-H(23C)	109.5
C(18)-C(19)-H(19A)	120.0	H(23A)-C(23)-H(23C)	109.5
C(14)-C(19)-H(19A)	120.0	H(23B)-C(23)-H(23C)	109.5
C(12')-O(8')-C(13')	111.2(11)	C(22)-C(24)-H(24A)	109.5
O(8')-C(12')-C(11)	108.5(12)	C(22)-C(24)-H(24B)	109.5
O(8')-C(12')-H(12C)	110.0	H(24A)-C(24)-H(24B)	109.5
C(11)-C(12')-H(12C)	110.0	C(22)-C(24)-H(24C)	109.5
O(8')-C(12')-H(12D)	110.0	H(24A)-C(24)-H(24C)	109.5
C(11)-C(12')-H(12D)	110.0	H(24B)-C(24)-H(24C)	109.5
H(12C)-C(12')-H(12D)	108.4	C(22)-C(25)-H(25A)	109.5
O(8')-C(13')-C(14')	108.4(10)	C(22)-C(25)-H(25B)	109.5
O(8')-C(13')-H(13C)	110.0	H(25A)-C(25)-H(25B)	109.5
C(14')-C(13')-H(13C)	110.0	C(22)-C(25)-H(25C)	109.5
O(8')-C(13')-H(13D)	110.0	H(25A)-C(25)-H(25C)	109.5
C(14')-C(13')-H(13D)	110.0	H(25B)-C(25)-H(25C)	109.5
H(13C)-C(13')-H(13D)	108.4	Si(2)-C(26)-H(26A)	109.5
C(15')-C(14')-C(19')	120.0	Si(2)-C(26)-H(26B)	109.5
C(15')-C(14')-C(13')	118.8(10)	H(26A)-C(26)-H(26B)	109.5
C(19')-C(14')-C(13')	120.9(10)	Si(2)-C(26)-H(26C)	109.5
C(14')-C(15')-C(16')	120.0	H(26A)-C(26)-H(26C)	109.5
C(14')-C(15')-H(15B)	120.0	H(26B)-C(26)-H(26C)	109.5
C(16')-C(15')-H(15B)	120.0	Si(2)-C(27)-H(27A)	109.5
C(17')-C(16')-C(15')	120.0	Si(2)-C(27)-H(27B)	109.5
C(17')-C(16')-H(16B)	120.0	H(27A)-C(27)-H(27B)	109.5
C(15')-C(16')-H(16B)	120.0	Si(2)-C(27)-H(27C)	109.5
C(16')-C(17')-C(18')	120.0	H(27A)-C(27)-H(27C)	109.5
C(16')-C(17')-H(17B)	120.0	H(27B)-C(27)-H(27C)	109.5
C(18')-C(17')-H(17B)	120.0	C(31)-C(28)-C(29)	110.1(13)
C(19')-C(18')-C(17')	120.0	C(31)-C(28)-C(30)	107.9(12)
C(19')-C(18')-H(18B)	120.0	C(29)-C(28)-C(30)	108.5(11)

C(31)-C(28)-Si(2)	112.1(10)	C(43)-C(42)-H(42A)	120.0
C(29)-C(28)-Si(2)	108.8(8)	C(41)-C(42)-H(42A)	120.0
C(30)-C(28)-Si(2)	109.4(8)	C(44)-C(43)-C(42)	120.0
C(28)-C(29)-H(29A)	109.5	C(44)-C(43)-H(43A)	120.0
C(28)-C(29)-H(29B)	109.5	C(42)-C(43)-H(43A)	120.0
H(29A)-C(29)-H(29B)	109.5	C(45)-C(44)-C(43)	120.0
C(28)-C(29)-H(29C)	109.5	C(45)-C(44)-H(44A)	120.0
H(29A)-C(29)-H(29C)	109.5	C(43)-C(44)-H(44A)	120.0
H(29B)-C(29)-H(29C)	109.5	C(44)-C(45)-C(40)	120.0
C(28)-C(30)-H(30A)	109.5	C(44)-C(45)-H(45A)	120.0
C(28)-C(30)-H(30B)	109.5	C(40)-C(45)-H(45A)	120.0
H(30A)-C(30)-H(30B)	109.5	O(4)-C(46)-C(47)	111.6(6)
C(28)-C(30)-H(30C)	109.5	O(4)-C(46)-H(46A)	109.3
H(30A)-C(30)-H(30C)	109.5	C(47)-C(46)-H(46A)	109.3
H(30B)-C(30)-H(30C)	109.5	O(4)-C(46)-H(46B)	109.3
C(28)-C(31)-H(31A)	109.5	C(47)-C(46)-H(46B)	109.3
C(28)-C(31)-H(31B)	109.5	H(46A)-C(46)-H(46B)	108.0
H(31A)-C(31)-H(31B)	109.5	C(48)-C(47)-C(52)	120.0
C(28)-C(31)-H(31C)	109.5	C(48)-C(47)-C(46)	121.3(4)
H(31A)-C(31)-H(31C)	109.5	C(52)-C(47)-C(46)	118.5(4)
H(31B)-C(31)-H(31C)	109.5	C(47)-C(48)-C(49)	120.0
O(12)-C(32)-O(11)	122.4(6)	C(47)-C(48)-H(48A)	120.0
O(12)-C(32)-C(33)	126.0(7)	C(49)-C(48)-H(48A)	120.0
O(11)-C(32)-C(33)	111.6(6)	C(48)-C(49)-C(50)	120.0
C(34)-C(33)-C(38)	120.0	C(48)-C(49)-H(49A)	120.0
C(34)-C(33)-C(32)	122.5(4)	C(50)-C(49)-H(49A)	120.0
C(38)-C(33)-C(32)	117.5(4)	C(51)-C(50)-C(49)	120.0
C(33)-C(34)-C(35)	120.0	C(51)-C(50)-H(50A)	120.0
C(33)-C(34)-H(34A)	120.0	C(49)-C(50)-H(50A)	120.0
C(35)-C(34)-H(34A)	120.0	C(52)-C(51)-C(50)	120.0
C(34)-C(35)-C(36)	120.0	C(52)-C(51)-H(51A)	120.0
C(34)-C(35)-H(35A)	120.0	C(50)-C(51)-H(51A)	120.0
C(36)-C(35)-H(35A)	120.0	C(51)-C(52)-C(47)	120.0
C(37)-C(36)-C(35)	120.0	C(51)-C(52)-H(52A)	120.0
C(37)-C(36)-H(36A)	120.0	C(47)-C(52)-H(52A)	120.0
C(35)-C(36)-H(36A)	120.0	O(5)-C(53)-C(54)	110.2(9)
C(36)-C(37)-C(38)	120.0	O(5)-C(53)-H(53A)	109.6
C(36)-C(37)-H(37A)	120.0	C(54)-C(53)-H(53A)	109.6
C(38)-C(37)-H(37A)	120.0	O(5)-C(53)-H(53B)	109.6
C(37)-C(38)-C(33)	120.0	C(54)-C(53)-H(53B)	109.6
C(37)-C(38)-H(38A)	120.0	H(53A)-C(53)-H(53B)	108.1
C(33)-C(38)-H(38A)	120.0	C(55)-C(54)-C(59)	120.0
O(3)-C(39)-C(40)	107.9(5)	C(55)-C(54)-C(53)	121.1(11)
O(3)-C(39)-H(39A)	110.1	C(59)-C(54)-C(53)	118.9(11)
C(40)-C(39)-H(39A)	110.1	C(54)-C(55)-C(56)	120.0
O(3)-C(39)-H(39B)	110.1	C(54)-C(55)-H(55A)	120.0
C(40)-C(39)-H(39B)	110.1	C(56)-C(55)-H(55A)	120.0
H(39A)-C(39)-H(39B)	108.4	C(55)-C(56)-C(57)	120.0
C(41)-C(40)-C(45)	120.0	C(55)-C(56)-H(56A)	120.0
C(41)-C(40)-C(39)	120.1(4)	C(57)-C(56)-H(56A)	120.0
C(45)-C(40)-C(39)	119.9(4)	C(58)-C(57)-C(56)	120.0
C(40)-C(41)-C(42)	120.0	C(58)-C(57)-H(57A)	120.0
C(40)-C(41)-H(41A)	120.0	C(56)-C(57)-H(57A)	120.0
C(42)-C(41)-H(41A)	120.0	C(59)-C(58)-C(57)	120.0
C(43)-C(42)-C(41)	120.0	C(59)-C(58)-H(58A)	120.0

C(57)-C(58)-H(58A)	120.0	O(10')-Si(2')-C(27')	109.0(7)
C(58)-C(59)-C(54)	120.0	C(26')-Si(2')-C(27')	110.3(9)
C(58)-C(59)-H(59A)	120.0	C(28')-Si(2')-C(27')	112.8(7)
C(54)-C(59)-H(59A)	120.0	C(9)-O(10')-Si(2')	116.8(7)
O(5)-C(53')-C(59')	102.4(10)	Si(1')-C(20')-H(20D)	109.5
O(5)-C(53')-H(53C)	111.3	Si(1')-C(20')-H(20E)	109.5
C(59')-C(53')-H(53C)	111.3	H(20D)-C(20')-H(20E)	109.5
O(5)-C(53')-H(53D)	111.3	Si(1')-C(20')-H(20F)	109.5
C(59')-C(53')-H(53D)	111.3	H(20D)-C(20')-H(20F)	109.5
H(53C)-C(53')-H(53D)	109.2	H(20E)-C(20')-H(20F)	109.5
C(58')-C(59')-C(54')	120.0	Si(1')-C(21')-H(21D)	109.5
C(58')-C(59')-C(53')	121.2(12)	Si(1')-C(21')-H(21E)	109.5
C(54')-C(59')-C(53')	118.6(12)	H(21D)-C(21')-H(21E)	109.5
C(59')-C(58')-C(57')	120.0	Si(1')-C(21')-H(21F)	109.5
C(59')-C(58')-H(58B)	120.0	H(21D)-C(21')-H(21F)	109.5
C(57')-C(58')-H(58B)	120.0	H(21E)-C(21')-H(21F)	109.5
C(56')-C(57')-C(58')	120.0	C(24')-C(22')-C(25')	108.8(11)
C(56')-C(57')-H(57B)	120.0	C(24')-C(22')-C(23')	110.3(12)
C(58')-C(57')-H(57B)	120.0	C(25')-C(22')-C(23')	107.4(11)
C(57')-C(56')-C(55')	120.0	C(24')-C(22')-Si(1')	109.6(9)
C(57')-C(56')-H(56B)	120.0	C(25')-C(22')-Si(1')	109.7(9)
C(55')-C(56')-H(56B)	120.0	C(23')-C(22')-Si(1')	111.0(9)
C(56')-C(55')-C(54')	120.0	C(22')-C(23')-H(23D)	109.5
C(56')-C(55')-H(55B)	120.0	C(22')-C(23')-H(23E)	109.5
C(54')-C(55')-H(55B)	120.0	H(23D)-C(23')-H(23E)	109.5
C(55')-C(54')-C(59')	120.0	C(22')-C(23')-H(23F)	109.5
C(55')-C(54')-H(54A)	120.0	H(23D)-C(23')-H(23F)	109.5
C(59')-C(54')-H(54A)	120.0	H(23E)-C(23')-H(23F)	109.5
O(1)-C(60)-H(60A)	109.5	C(22')-C(24')-H(24D)	109.5
O(1)-C(60)-H(60B)	109.5	C(22')-C(24')-H(24E)	109.5
H(60A)-C(60)-H(60B)	109.5	H(24D)-C(24')-H(24E)	109.5
O(1)-C(60)-H(60C)	109.5	C(22')-C(24')-H(24F)	109.5
H(60A)-C(60)-H(60C)	109.5	H(24D)-C(24')-H(24F)	109.5
H(60B)-C(60)-H(60C)	109.5	H(24E)-C(24')-H(24F)	109.5
O(9)-Si(1)-C(21)	103.6(7)	C(22')-C(25')-H(25D)	109.5
O(9)-Si(1)-C(20)	114.9(7)	C(22')-C(25')-H(25E)	109.5
C(21)-Si(1)-C(20)	109.8(9)	H(25D)-C(25')-H(25E)	109.5
O(9)-Si(1)-C(22)	109.5(5)	C(22')-C(25')-H(25F)	109.5
C(21)-Si(1)-C(22)	109.4(8)	H(25D)-C(25')-H(25F)	109.5
C(20)-Si(1)-C(22)	109.4(7)	H(25E)-C(25')-H(25F)	109.5
O(10)-Si(2)-C(27)	110.4(7)	Si(2')-C(26')-H(26D)	109.5
O(10)-Si(2)-C(26)	108.9(6)	Si(2')-C(26')-H(26E)	109.5
C(27)-Si(2)-C(26)	109.2(7)	H(26D)-C(26')-H(26E)	109.5
O(10)-Si(2)-C(28)	104.1(5)	Si(2')-C(26')-H(26F)	109.5
C(27)-Si(2)-C(28)	112.1(7)	H(26D)-C(26')-H(26F)	109.5
C(26)-Si(2)-C(28)	112.0(6)	H(26E)-C(26')-H(26F)	109.5
O(9)-Si(1')-C(20')	114.8(7)	Si(2')-C(27')-H(27D)	109.5
O(9)-Si(1')-C(22')	102.1(5)	Si(2')-C(27')-H(27E)	109.5
C(20')-Si(1')-C(22')	112.9(8)	H(27D)-C(27')-H(27E)	109.5
O(9)-Si(1')-C(21')	103.8(7)	Si(2')-C(27')-H(27F)	109.5
C(20')-Si(1')-C(21')	112.1(10)	H(27D)-C(27')-H(27F)	109.5
C(22')-Si(1')-C(21')	110.3(7)	H(27E)-C(27')-H(27F)	109.5
O(10')-Si(2')-C(26')	109.8(7)	C(31')-C(28')-C(29')	109.3(13)
O(10')-Si(2')-C(28')	105.5(6)	C(31')-C(28')-C(30')	109.7(12)
C(26')-Si(2')-C(28')	109.3(6)	C(29')-C(28')-C(30')	107.9(12)

C(31')-C(28')-Si(2')	111.1(9)	H(30D)-C(30')-H(30E)	109.5
C(29')-C(28')-Si(2')	109.5(9)	C(28')-C(30')-H(30F)	109.5
C(30')-C(28')-Si(2')	109.3(10)	H(30D)-C(30')-H(30F)	109.5
C(28')-C(29')-H(29D)	109.5	H(30E)-C(30')-H(30F)	109.5
C(28')-C(29')-H(29E)	109.5	C(28')-C(31')-H(31D)	109.5
H(29D)-C(29')-H(29E)	109.5	C(28')-C(31')-H(31E)	109.5
C(28')-C(29')-H(29F)	109.5	H(31D)-C(31')-H(31E)	109.5
H(29D)-C(29')-H(29F)	109.5	C(28')-C(31')-H(31F)	109.5
H(29E)-C(29')-H(29F)	109.5	H(31D)-C(31')-H(31F)	109.5
C(28')-C(30')-H(30D)	109.5	H(31E)-C(31')-H(31F)	109.5
C(28')-C(30')-H(30E)	109.5		

Symmetry transformations used to generate equivalent atoms:

Table 4S. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 10. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
O(1)	40(3)	34(3)	122(4)	-10(3)	33(3)	0(2)
O(2)	42(3)	29(3)	112(4)	-11(2)	21(2)	-7(2)
O(3)	39(2)	41(3)	93(3)	-12(3)	24(2)	-8(2)
O(4)	43(2)	35(3)	111(4)	-5(3)	31(2)	4(2)
O(5)	46(3)	30(3)	99(4)	-11(2)	26(2)	-4(2)
O(6)	36(2)	28(3)	93(3)	-4(2)	9(2)	0(2)
O(7)	38(2)	30(3)	131(4)	-10(3)	-26(2)	8(2)
O(9)	65(3)	38(3)	57(3)	6(2)	-20(2)	-7(2)
O(10)	45(6)	44(6)	41(5)	3(5)	-8(4)	-9(5)
O(11)	45(2)	40(3)	45(3)	-1(2)	-8(2)	3(2)
O(12)	75(3)	123(5)	59(3)	-35(3)	8(3)	-37(3)
C(1)	43(4)	31(4)	111(5)	-15(4)	23(4)	-6(3)
C(2)	40(3)	34(4)	103(5)	-7(3)	27(3)	-6(3)
C(3)	39(4)	33(4)	106(5)	-10(4)	24(3)	0(3)
C(4)	38(3)	33(4)	98(5)	-8(3)	22(3)	-4(3)
C(5)	40(3)	32(4)	99(5)	-13(3)	27(3)	-9(3)
C(6)	41(4)	37(4)	109(5)	-13(4)	27(4)	-1(3)
C(7)	37(3)	26(4)	80(5)	-3(3)	-8(3)	0(3)
C(8)	50(4)	30(4)	68(4)	5(3)	-21(3)	3(3)
C(9)	92(5)	34(4)	66(4)	1(3)	-42(4)	1(3)
C(10)	77(4)	36(4)	82(5)	-4(4)	-44(4)	-1(3)
C(11)	64(4)	29(4)	118(6)	-4(4)	-44(4)	4(3)
O(8)	42(6)	29(5)	68(10)	6(5)	-17(6)	4(4)
C(12)	53(8)	25(6)	58(14)	6(7)	-18(7)	3(5)
C(13)	42(7)	29(6)	61(11)	5(7)	-14(7)	5(5)
C(14)	39(7)	33(7)	49(9)	1(6)	-11(6)	8(5)
C(15)	59(8)	33(7)	43(9)	5(6)	-8(7)	4(6)
C(16)	52(8)	33(8)	62(10)	-3(7)	-5(7)	3(6)
C(17)	44(9)	42(8)	53(10)	-2(6)	-9(7)	-1(7)
C(18)	64(9)	50(9)	47(10)	0(6)	-4(7)	-6(7)
C(19)	46(9)	36(8)	52(10)	-3(6)	-6(7)	8(6)
O(8')	40(5)	33(5)	63(8)	1(5)	-7(5)	1(4)
C(12')	43(8)	34(7)	78(14)	9(7)	-16(7)	5(5)
C(13')	37(7)	46(7)	98(13)	18(7)	-14(8)	10(5)
C(14')	38(8)	36(6)	69(11)	3(5)	-9(7)	6(5)
C(15')	50(8)	45(7)	64(10)	6(6)	-7(7)	3(6)
C(16')	64(10)	42(8)	77(11)	1(7)	-4(8)	-9(7)
C(17')	55(8)	50(8)	76(11)	21(6)	-6(7)	3(6)
C(18')	40(8)	61(8)	80(11)	12(7)	-1(7)	9(6)
C(19')	37(7)	53(8)	81(11)	8(6)	-6(7)	10(5)
C(20)	61(10)	41(9)	76(9)	9(7)	12(7)	9(8)
C(21)	53(10)	48(12)	86(10)	4(8)	-10(8)	-23(9)
C(22)	49(7)	35(7)	74(8)	-2(5)	4(6)	1(5)
C(23)	54(8)	45(9)	82(8)	13(6)	10(6)	3(6)
C(24)	57(7)	40(8)	99(11)	4(7)	0(7)	-9(6)
C(25)	52(8)	48(9)	73(9)	-5(8)	5(7)	-5(6)
C(26)	53(7)	46(7)	40(8)	5(6)	-6(6)	-14(6)
C(27)	69(8)	53(8)	56(9)	7(7)	7(7)	8(6)
C(28)	52(7)	56(8)	50(7)	16(6)	-12(6)	-10(5)
C(29)	82(10)	83(10)	77(10)	27(8)	-23(8)	-31(8)

C(30)	74(10)	68(11)	49(7)	6(7)	-18(6)	-4(8)
C(31)	69(9)	81(12)	70(12)	-3(9)	-18(8)	1(9)
C(32)	59(4)	53(5)	51(4)	-1(4)	13(3)	-8(3)
C(33)	56(4)	43(4)	61(4)	-4(3)	14(3)	-2(3)
C(34)	58(4)	90(6)	59(4)	0(4)	2(3)	32(4)
C(35)	51(4)	130(8)	87(6)	9(5)	5(4)	37(5)
C(36)	58(5)	79(7)	117(6)	1(5)	24(4)	13(4)
C(37)	64(5)	150(10)	137(7)	-64(7)	50(5)	-21(5)
C(38)	63(5)	145(10)	119(6)	-76(7)	37(4)	-29(5)
C(39)	36(3)	40(4)	94(5)	0(4)	18(3)	-3(3)
C(40)	34(3)	32(4)	94(5)	-8(3)	27(3)	-5(3)
C(41)	31(3)	40(4)	111(5)	-8(4)	10(3)	4(3)
C(42)	28(3)	62(5)	124(6)	-20(4)	9(4)	-1(3)
C(43)	59(5)	60(5)	122(7)	-37(4)	45(4)	-23(4)
C(44)	102(6)	47(5)	113(6)	-19(5)	55(5)	-29(4)
C(45)	80(5)	38(4)	103(6)	0(4)	40(4)	-10(3)
C(46)	46(4)	50(5)	138(7)	22(5)	41(4)	10(3)
C(47)	41(4)	34(4)	137(7)	2(4)	31(4)	2(3)
C(48)	53(3)	42(3)	227(7)	28(4)	70(4)	10(3)
C(49)	53(3)	42(3)	227(7)	28(4)	70(4)	10(3)
C(50)	45(4)	35(4)	174(8)	-25(5)	31(4)	5(3)
C(51)	46(4)	34(4)	114(6)	-23(4)	11(4)	2(3)
C(52)	38(3)	35(4)	106(6)	-14(4)	14(3)	-5(3)
C(53)	33(7)	29(6)	90(10)	-2(5)	25(7)	9(5)
C(54)	39(8)	26(6)	68(10)	-6(6)	1(6)	2(5)
C(55)	84(9)	37(9)	91(13)	1(8)	-29(8)	12(7)
C(56)	115(12)	36(9)	107(14)	11(9)	-13(10)	15(8)
C(57)	112(11)	41(9)	66(10)	-6(7)	7(9)	17(8)
C(58)	63(8)	41(9)	51(10)	-4(7)	24(7)	-4(6)
C(59)	36(7)	27(8)	51(10)	-8(7)	16(6)	3(5)
C(53')	79(12)	28(6)	79(9)	3(5)	8(7)	-3(6)
C(59')	38(8)	30(6)	47(8)	-11(6)	7(6)	-1(5)
C(58')	46(8)	36(10)	51(12)	6(8)	3(7)	1(6)
C(57')	61(8)	47(10)	42(11)	-2(7)	-1(7)	-9(6)
C(56')	65(9)	38(9)	69(11)	-1(8)	-20(8)	-1(7)
C(55')	55(8)	46(9)	81(12)	-6(8)	-12(7)	8(7)
C(54')	47(7)	45(8)	60(10)	-16(7)	0(6)	2(6)
C(60)	39(4)	41(5)	124(7)	-11(4)	26(4)	2(3)
Si(1)	51(4)	29(3)	62(3)	2(2)	1(3)	-3(2)
Si(2)	43(3)	47(3)	42(3)	5(2)	0(2)	-6(2)
Si(1')	56(4)	36(4)	53(3)	7(2)	-7(3)	-7(3)
Si(2')	46(3)	48(3)	44(3)	2(2)	-5(2)	-4(2)
O(10')	49(6)	38(6)	35(5)	6(5)	-8(4)	1(5)
C(20')	60(10)	49(8)	94(14)	9(8)	-2(10)	-4(7)
C(21')	65(10)	69(12)	47(8)	10(7)	0(7)	-21(8)
C(22')	42(7)	47(9)	50(7)	-4(6)	-7(5)	-7(6)
C(23')	54(8)	59(9)	58(9)	-4(7)	-9(7)	7(7)
C(24')	58(8)	63(9)	82(10)	9(8)	-13(7)	-22(7)
C(25')	51(7)	52(9)	44(6)	-1(6)	-8(5)	-11(6)
C(26')	59(8)	49(7)	66(9)	-7(6)	-12(7)	2(6)
C(27')	64(10)	97(14)	40(9)	17(9)	2(7)	-20(9)
C(28')	58(8)	43(8)	56(8)	-1(6)	-18(6)	3(6)
C(29')	102(12)	49(8)	76(11)	7(7)	-24(9)	1(7)
C(30')	83(10)	65(10)	58(8)	-4(7)	-20(7)	-6(9)
C(31')	53(7)	67(10)	79(9)	3(8)	-13(6)	4(7)

Table 5S. Hydrogen coordinates (x 10⁴) and isotropic displacement parameters (Å²x 10⁻³) for 10.

	x	y	z	U(eq)
H(1A)	578	2091	8219	73
H(2A)	2704	1621	8060	70
H(3A)	3202	2429	9217	71
H(4A)	4052	3292	8126	67
H(5A)	2231	4181	8903	68
H(6A)	1802	5503	8181	74
H(6B)	3211	5612	8444	74
H(7A)	4088	6375	7672	57
H(8A)	3722	5138	6622	60
H(9)	4923	6833	6257	78
H(10A)	3751	8431	6484	79
H(11)	3067	8038	7556	86
H(12A)	1003	7937	6709	55
H(12B)	860	8045	7407	55
H(13A)	113	10230	6979	53
H(13B)	480	9651	6378	53
H(15A)	1410	11787	7344	54
H(16A)	2723	13246	7121	59
H(17A)	3658	13332	6209	56
H(18A)	3280	11958	5522	64
H(19A)	1967	10499	5745	54
H(12C)	1574	7942	6416	62
H(12D)	903	8338	6997	62
H(13C)	416	10130	6622	73
H(13D)	862	9608	6020	73
H(15B)	1557	11782	7002	63
H(16B)	2571	13408	6773	73
H(17B)	3320	13683	5834	73
H(18B)	3055	12332	5124	73
H(19B)	2041	10706	5352	69
H(20A)	8070	8712	6332	89
H(20B)	7565	7501	6423	89
H(20C)	6796	8323	6006	89
H(21A)	8126	8913	7661	94
H(21B)	6853	8810	8015	94
H(21C)	7484	7750	7744	94
H(23A)	6043	10292	6009	90
H(23B)	4652	10103	6242	90
H(23C)	5285	11281	6286	90
H(24A)	7571	10740	7396	98
H(24B)	7800	10738	6709	98
H(24C)	6973	11677	6992	98
H(25A)	5347	10383	7752	87
H(25B)	4869	11336	7326	87
H(25C)	4236	10158	7282	87
H(26A)	4316	8200	4658	70
H(26B)	3595	8696	5201	70
H(26C)	4976	8194	5294	70
H(27A)	4691	5791	4559	89

H(27B)	5370	5784	5191	89
H(27C)	4197	4999	5056	89
H(29A)	1886	4955	4942	121
H(29B)	1218	5524	5480	121
H(29C)	493	5437	4860	121
H(30A)	2525	6074	4087	96
H(30B)	1126	6554	4031	96
H(30C)	2305	7360	4090	96
H(31A)	1351	8255	4937	111
H(31B)	166	7460	4857	111
H(31C)	891	7546	5477	111
H(34A)	7292	5393	7597	83
H(35A)	9429	5170	7846	107
H(36A)	10812	4487	7158	101
H(37A)	10058	4026	6222	139
H(38A)	7921	4248	5974	130
H(39A)	5533	4648	8214	68
H(39B)	6204	3581	8479	68
H(41A)	7027	3749	9554	72
H(42A)	8249	4845	10182	85
H(43A)	8491	6694	9978	96
H(44A)	7511	7447	9145	104
H(45A)	6289	6351	8517	87
H(46A)	4239	655	9443	92
H(46B)	4854	1770	9669	92
H(48A)	6998	1663	8781	127
H(49A)	9010	896	8872	127
H(50A)	9383	-542	9521	101
H(51A)	7744	-1213	10080	77
H(52A)	5731	-446	9989	71
H(53A)	3713	-119	8574	60
H(53B)	2653	-256	8068	60
H(55A)	3966	-1566	9244	85
H(56A)	3223	-3146	9684	104
H(57A)	1167	-3746	9480	88
H(58A)	-146	-2765	8835	62
H(59A)	597	-1184	8395	45
H(53C)	2603	-54	7958	74
H(53D)	1103	-15	8052	74
H(58B)	132	-1459	8614	53
H(57B)	297	-3012	9199	60
H(56B)	2280	-3635	9518	69
H(55B)	4099	-2705	9252	73
H(54A)	3934	-1152	8667	61
H(60A)	-46	3999	9040	101
H(60B)	-629	3095	9455	101
H(60C)	-862	3025	8764	101
H(20D)	5691	10865	6847	102
H(20E)	4418	10280	6627	102
H(20F)	4693	10459	7310	102
H(21D)	7159	9159	6148	91
H(21E)	7057	7903	6320	91
H(21F)	5917	8485	5972	91
H(23D)	7198	7208	7600	86
H(23E)	8243	7751	7203	86

H(23F)	8406	7781	7899	86
H(24D)	8420	9808	7162	102
H(24E)	7537	10529	7564	102
H(24F)	8633	9802	7856	102
H(25D)	5843	8354	8207	74
H(25E)	7071	8921	8489	74
H(25F)	5976	9648	8197	74
H(26D)	2635	4828	4836	87
H(26E)	3428	4606	5428	87
H(26F)	1921	4614	5431	87
H(27D)	4075	6950	4667	101
H(27E)	4092	7848	5172	101
H(27F)	4866	6742	5261	101
H(29D)	1942	8564	5016	114
H(29E)	444	8599	5076	114
H(29F)	1335	8443	5644	114
H(30D)	1701	7093	4283	103
H(30E)	930	6017	4433	103
H(30F)	206	7148	4350	103
H(31D)	17	5796	5428	99
H(31E)	164	6758	5895	99
H(31F)	-726	6914	5326	99

Table 6S. Torsion angles [°] for 10.

C(60)-O(1)-C(1)-O(2)	60.6(7)
C(60)-O(1)-C(1)-C(2)	-178.2(5)
C(5)-O(2)-C(1)-O(1)	58.7(7)
C(5)-O(2)-C(1)-C(2)	-61.5(7)
C(53)-O(5)-C(2)-C(3)	-92.0(10)
C(53')-O(5)-C(2)-C(3)	-137.9(10)
C(53)-O(5)-C(2)-C(1)	147.0(10)
C(53')-O(5)-C(2)-C(1)	101.1(11)
O(1)-C(1)-C(2)-O(5)	59.0(7)
O(2)-C(1)-C(2)-O(5)	-177.9(6)
O(1)-C(1)-C(2)-C(3)	-63.5(7)
O(2)-C(1)-C(2)-C(3)	59.6(8)
C(46)-O(4)-C(3)-C(2)	-107.0(7)
C(46)-O(4)-C(3)-C(4)	132.6(7)
O(5)-C(2)-C(3)-O(4)	61.2(7)
C(1)-C(2)-C(3)-O(4)	-177.6(6)
O(5)-C(2)-C(3)-C(4)	-178.7(5)
C(1)-C(2)-C(3)-C(4)	-57.5(7)
C(39)-O(3)-C(4)-C(3)	120.1(6)
C(39)-O(3)-C(4)-C(5)	-121.0(6)
O(4)-C(3)-C(4)-O(3)	-63.9(7)
C(2)-C(3)-C(4)-O(3)	174.2(5)
O(4)-C(3)-C(4)-C(5)	178.3(6)
C(2)-C(3)-C(4)-C(5)	56.4(8)
C(1)-O(2)-C(5)-C(6)	-174.3(5)
C(1)-O(2)-C(5)-C(4)	60.3(7)
O(3)-C(4)-C(5)-O(2)	-175.5(5)
C(3)-C(4)-C(5)-O(2)	-56.7(8)
O(3)-C(4)-C(5)-C(6)	63.1(8)
C(3)-C(4)-C(5)-C(6)	-178.1(7)
C(7)-O(6)-C(6)-C(5)	-167.7(5)
O(2)-C(5)-C(6)-O(6)	-67.2(6)
C(4)-C(5)-C(6)-O(6)	55.6(8)
C(6)-O(6)-C(7)-O(7)	-68.0(7)
C(6)-O(6)-C(7)-C(8)	173.8(5)
C(11)-O(7)-C(7)-O(6)	165.8(6)
C(11)-O(7)-C(7)-C(8)	-75.4(7)
C(32)-O(11)-C(8)-C(7)	161.8(5)
C(32)-O(11)-C(8)-C(9)	-77.9(7)
O(6)-C(7)-C(8)-O(11)	-83.9(6)
O(7)-C(7)-C(8)-O(11)	157.7(5)
O(6)-C(7)-C(8)-C(9)	156.2(5)
O(7)-C(7)-C(8)-C(9)	37.7(7)
Si(2)-O(10)-C(9)-C(10)	106.4(12)
Si(2)-O(10)-C(9)-C(8)	-123.1(11)
O(11)-C(8)-C(9)-O(10)	135.3(7)
C(7)-C(8)-C(9)-O(10)	-108.7(8)
O(11)-C(8)-C(9)-C(10)	-93.5(7)
C(7)-C(8)-C(9)-C(10)	22.5(8)
O(11)-C(8)-C(9)-O(10')	159.7(6)
C(7)-C(8)-C(9)-O(10')	-84.3(7)
Si(1')-O(9)-C(10)-C(9)	112.1(7)
Si(1)-O(9)-C(10)-C(9)	92.6(7)

Si(1')-O(9)-C(10)-C(11)	-126.7(6)
Si(1)-O(9)-C(10)-C(11)	-146.1(6)
O(10)-C(9)-C(10)-O(9)	-161.6(7)
C(8)-C(9)-C(10)-O(9)	66.4(8)
O(10')-C(9)-C(10)-O(9)	173.1(6)
O(10)-C(9)-C(10)-C(11)	80.3(9)
C(8)-C(9)-C(10)-C(11)	-51.7(8)
O(10')-C(9)-C(10)-C(11)	55.0(8)
C(7)-O(7)-C(11)-C(10)	44.0(8)
C(7)-O(7)-C(11)-C(12)	-179.5(10)
C(7)-O(7)-C(11)-C(12')	154.9(11)
O(9)-C(10)-C(11)-O(7)	-99.2(6)
C(9)-C(10)-C(11)-O(7)	19.0(8)
O(9)-C(10)-C(11)-C(12)	140.7(11)
C(9)-C(10)-C(11)-C(12)	-101.1(12)
O(9)-C(10)-C(11)-C(12')	143.1(9)
C(9)-C(10)-C(11)-C(12')	-98.7(9)
C(13)-O(8)-C(12)-C(11)	155.3(13)
O(7)-C(11)-C(12)-O(8)	172.5(11)
C(10)-C(11)-C(12)-O(8)	-62.0(17)
C(12)-O(8)-C(13)-C(14)	-158.2(14)
O(8)-C(13)-C(14)-C(15)	-80.5(13)
O(8)-C(13)-C(14)-C(19)	94.0(12)
C(19)-C(14)-C(15)-C(16)	0.0
C(13)-C(14)-C(15)-C(16)	174.4(11)
C(14)-C(15)-C(16)-C(17)	0.0
C(15)-C(16)-C(17)-C(18)	0.0
C(16)-C(17)-C(18)-C(19)	0.0
C(17)-C(18)-C(19)-C(14)	0.0
C(15)-C(14)-C(19)-C(18)	0.0
C(13)-C(14)-C(19)-C(18)	-174.4(11)
C(13')-O(8')-C(12')-C(11)	-175.9(13)
O(7)-C(11)-C(12')-O(8')	-178.7(11)
C(10)-C(11)-C(12')-O(8')	-60.8(14)
C(12')-O(8')-C(13')-C(14')	-171.9(13)
O(8')-C(13')-C(14')-C(15')	-76.2(14)
O(8')-C(13')-C(14')-C(19')	98.5(13)
C(19')-C(14')-C(15')-C(16')	0.0
C(13')-C(14')-C(15')-C(16')	174.7(10)
C(14')-C(15')-C(16')-C(17')	0.0
C(15')-C(16')-C(17')-C(18')	0.0
C(16')-C(17')-C(18')-C(19')	0.0
C(17')-C(18')-C(19')-C(14')	0.0
C(15')-C(14')-C(19')-C(18')	0.0
C(13')-C(14')-C(19')-C(18')	-174.6(10)
C(8)-O(11)-C(32)-O(12)	-4.5(10)
C(8)-O(11)-C(32)-C(33)	175.7(5)
O(12)-C(32)-C(33)-C(34)	-174.2(6)
O(11)-C(32)-C(33)-C(34)	5.6(8)
O(12)-C(32)-C(33)-C(38)	7.8(9)
O(11)-C(32)-C(33)-C(38)	-172.4(5)
C(38)-C(33)-C(34)-C(35)	0.0
C(32)-C(33)-C(34)-C(35)	-177.9(6)
C(33)-C(34)-C(35)-C(36)	0.0
C(34)-C(35)-C(36)-C(37)	0.0

C(35)-C(36)-C(37)-C(38)	0.0
C(36)-C(37)-C(38)-C(33)	0.0
C(34)-C(33)-C(38)-C(37)	0.0
C(32)-C(33)-C(38)-C(37)	178.0(5)
C(4)-O(3)-C(39)-C(40)	174.9(5)
O(3)-C(39)-C(40)-C(41)	70.4(5)
O(3)-C(39)-C(40)-C(45)	-108.5(5)
C(45)-C(40)-C(41)-C(42)	0.0
C(39)-C(40)-C(41)-C(42)	-179.0(4)
C(40)-C(41)-C(42)-C(43)	0.0
C(41)-C(42)-C(43)-C(44)	0.0
C(42)-C(43)-C(44)-C(45)	0.0
C(43)-C(44)-C(45)-C(40)	0.0
C(41)-C(40)-C(45)-C(44)	0.0
C(39)-C(40)-C(45)-C(44)	179.0(4)
C(3)-O(4)-C(46)-C(47)	-172.7(6)
O(4)-C(46)-C(47)-C(48)	30.1(8)
O(4)-C(46)-C(47)-C(52)	-154.2(5)
C(52)-C(47)-C(48)-C(49)	0.0
C(46)-C(47)-C(48)-C(49)	175.6(6)
C(47)-C(48)-C(49)-C(50)	0.0
C(48)-C(49)-C(50)-C(51)	0.0
C(49)-C(50)-C(51)-C(52)	0.0
C(50)-C(51)-C(52)-C(47)	0.0
C(48)-C(47)-C(52)-C(51)	0.0
C(46)-C(47)-C(52)-C(51)	-175.8(6)
C(2)-O(5)-C(53)-C(54)	-177.4(9)
O(5)-C(53)-C(54)-C(55)	-111.5(12)
O(5)-C(53)-C(54)-C(59)	67.9(14)
C(59)-C(54)-C(55)-C(56)	0.0
C(53)-C(54)-C(55)-C(56)	179.3(12)
C(54)-C(55)-C(56)-C(57)	0.0
C(55)-C(56)-C(57)-C(58)	0.0
C(56)-C(57)-C(58)-C(59)	0.0
C(57)-C(58)-C(59)-C(54)	0.0
C(55)-C(54)-C(59)-C(58)	0.0
C(53)-C(54)-C(59)-C(58)	-179.4(12)
C(2)-O(5)-C(53')-C(59')	164.0(9)
O(5)-C(53')-C(59')-C(58')	99.1(12)
O(5)-C(53')-C(59')-C(54')	-75.7(14)
C(54')-C(59')-C(58')-C(57')	0.0
C(53')-C(59')-C(58')-C(57')	-174.8(13)
C(59')-C(58')-C(57')-C(56')	0.0
C(58')-C(57')-C(56')-C(55')	0.0
C(57')-C(56')-C(55')-C(54')	0.0
C(56')-C(55')-C(54')-C(59')	0.0
C(58')-C(59')-C(54')-C(55')	0.0
C(53')-C(59')-C(54')-C(55')	174.9(13)
C(10)-O(9)-Si(1)-C(21)	179.7(9)
C(10)-O(9)-Si(1)-C(20)	-60.5(9)
C(10)-O(9)-Si(1)-C(22)	63.1(8)
C(23)-C(22)-Si(1)-O(9)	-77.9(9)
C(25)-C(22)-Si(1)-O(9)	41.9(10)
C(24)-C(22)-Si(1)-O(9)	161.5(9)
C(23)-C(22)-Si(1)-C(21)	169.2(10)

C(25)-C(22)-Si(1)-C(21)	-71.0(11)
C(24)-C(22)-Si(1)-C(21)	48.6(12)
C(23)-C(22)-Si(1)-C(20)	48.8(11)
C(25)-C(22)-Si(1)-C(20)	168.7(10)
C(24)-C(22)-Si(1)-C(20)	-71.8(11)
C(9)-O(10)-Si(2)-C(27)	63.9(14)
C(9)-O(10)-Si(2)-C(26)	-56.0(14)
C(9)-O(10)-Si(2)-C(28)	-175.6(11)
C(31)-C(28)-Si(2)-O(10)	63.8(12)
C(29)-C(28)-Si(2)-O(10)	-58.2(12)
C(30)-C(28)-Si(2)-O(10)	-176.6(10)
C(31)-C(28)-Si(2)-C(27)	-176.9(11)
C(29)-C(28)-Si(2)-C(27)	61.1(12)
C(30)-C(28)-Si(2)-C(27)	-57.2(11)
C(31)-C(28)-Si(2)-C(26)	-53.7(12)
C(29)-C(28)-Si(2)-C(26)	-175.7(10)
C(30)-C(28)-Si(2)-C(26)	65.9(11)
C(10)-O(9)-Si(1')-C(20')	53.6(10)
C(10)-O(9)-Si(1')-C(22')	176.1(6)
C(10)-O(9)-Si(1')-C(21')	-69.2(8)
C(10)-C(9)-O(10')-Si(2')	128.6(8)
C(8)-C(9)-O(10')-Si(2')	-116.2(8)
C(26')-Si(2')-O(10')-C(9)	93.7(9)
C(28')-Si(2')-O(10')-C(9)	-148.6(8)
C(27')-Si(2')-O(10')-C(9)	-27.3(12)
O(9)-Si(1')-C(22')-C(24')	-177.0(9)
C(20')-Si(1')-C(22')-C(24')	-53.1(13)
C(21')-Si(1')-C(22')-C(24')	73.1(12)
O(9)-Si(1')-C(22')-C(25')	-57.6(10)
C(20')-Si(1')-C(22')-C(25')	66.2(12)
C(21')-Si(1')-C(22')-C(25')	-167.5(10)
O(9)-Si(1')-C(22')-C(23')	60.9(10)
C(20')-Si(1')-C(22')-C(23')	-175.2(11)
C(21')-Si(1')-C(22')-C(23')	-49.0(12)
O(10')-Si(2')-C(28')-C(31')	-54.0(11)
C(26')-Si(2')-C(28')-C(31')	64.0(12)
C(27')-Si(2')-C(28')-C(31')	-172.9(11)
O(10')-Si(2')-C(28')-C(29')	66.8(11)
C(26')-Si(2')-C(28')-C(29')	-175.1(11)
C(27')-Si(2')-C(28')-C(29')	-52.0(13)
O(10')-Si(2')-C(28')-C(30')	-175.2(10)
C(26')-Si(2')-C(28')-C(30')	-57.1(11)
C(27')-Si(2')-C(28')-C(30')	66.0(12)

Symmetry transformations used to generate equivalent atoms:

Table 7S. Hydrogen bonds for 10 [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(10)-H(10A)...O(8')	1.00	2.17	2.717(12)	112.3
C(12')-H(12C)...O(10')	0.99	2.16	2.83(2)	124.1
C(53)-H(53A)...O(4)	0.99	2.48	3.102(14)	120.6
C(53)-H(53B)...O(8)#1	0.99	2.59	3.55(2)	163.6

Symmetry transformations used to generate equivalent atoms:

#1 x,y-1,z

Projection view with 30% probability ellipsoids- disorder components and H atoms omitted for clarity:

