

Convergent synthesis of isomeric heterosaccharides related to the fragments of galactomannan from *Aspergillus fumigatus*

D.A. Argunov, V.B. Krylov, N.E. Nifantiev*

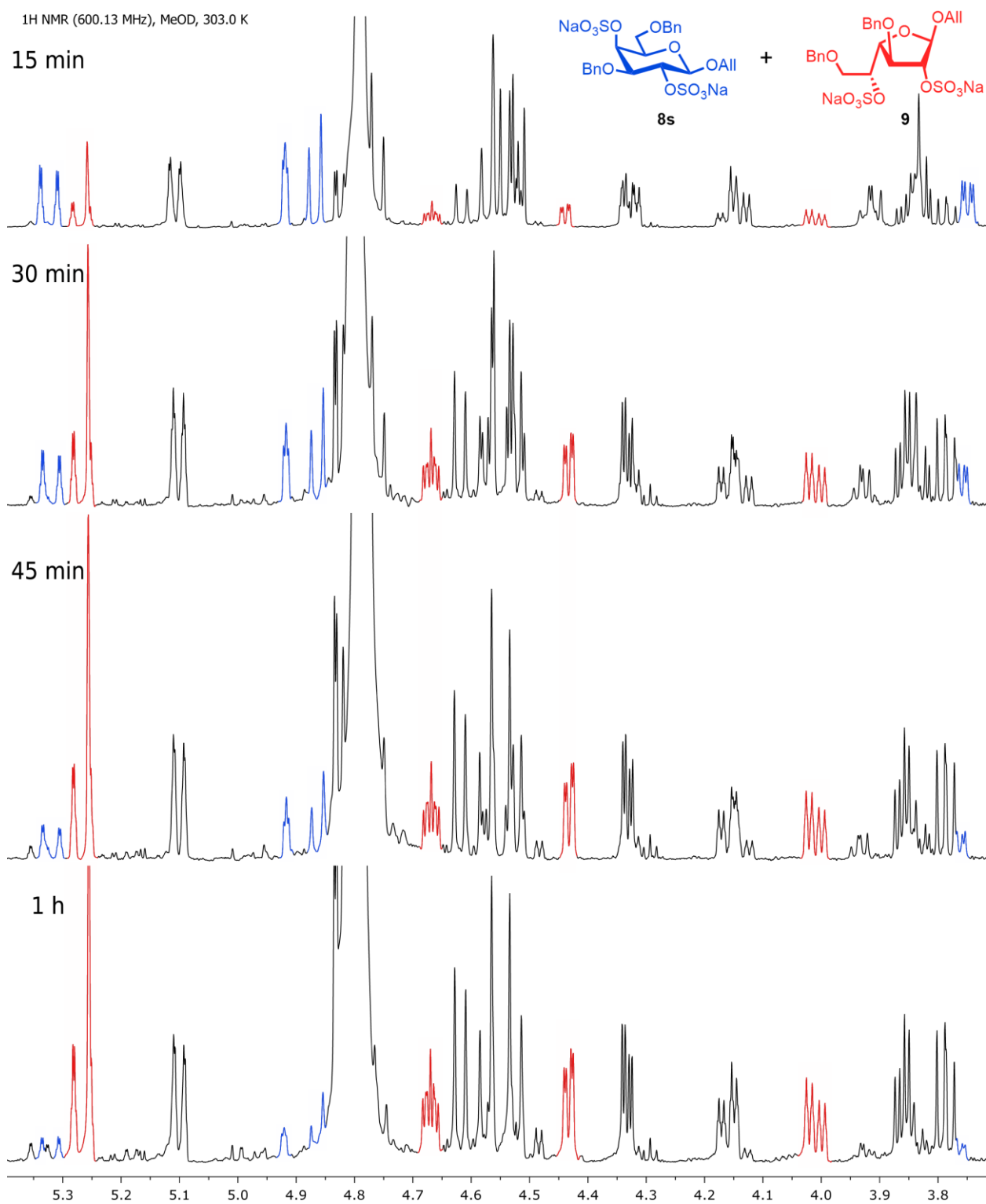
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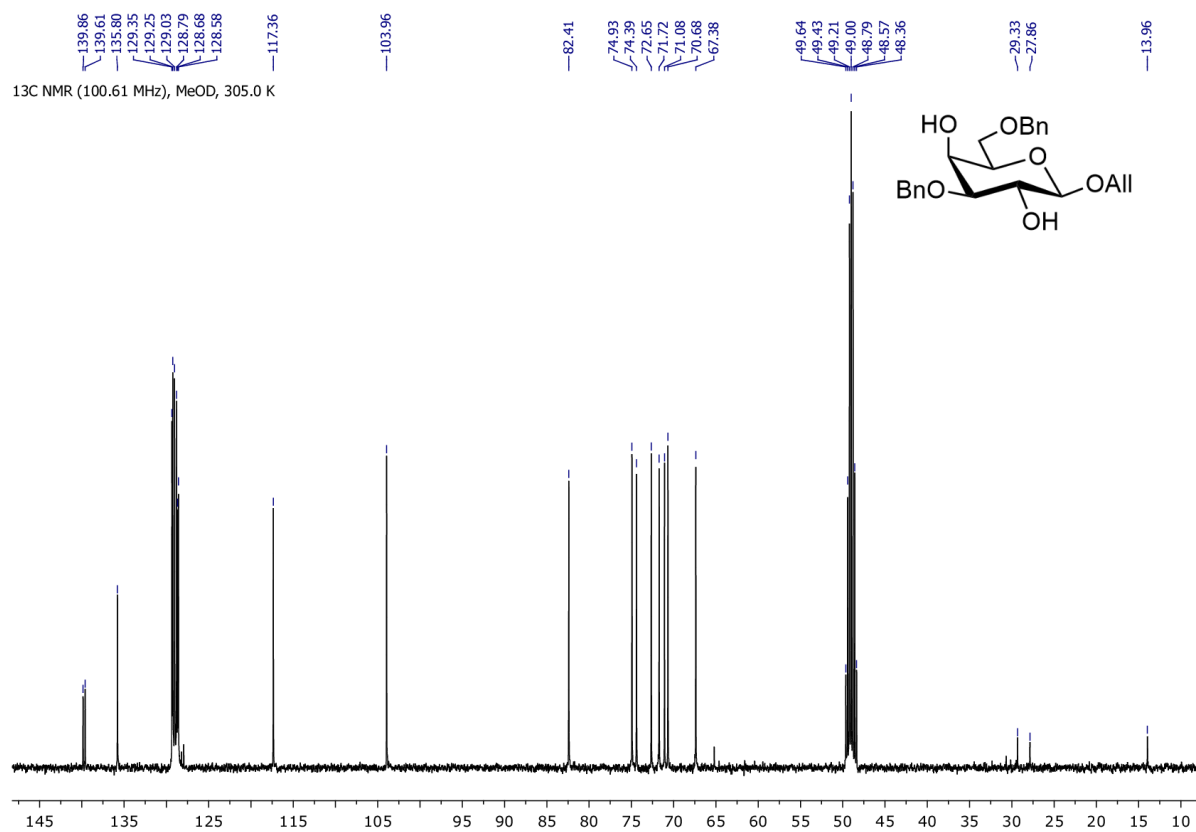
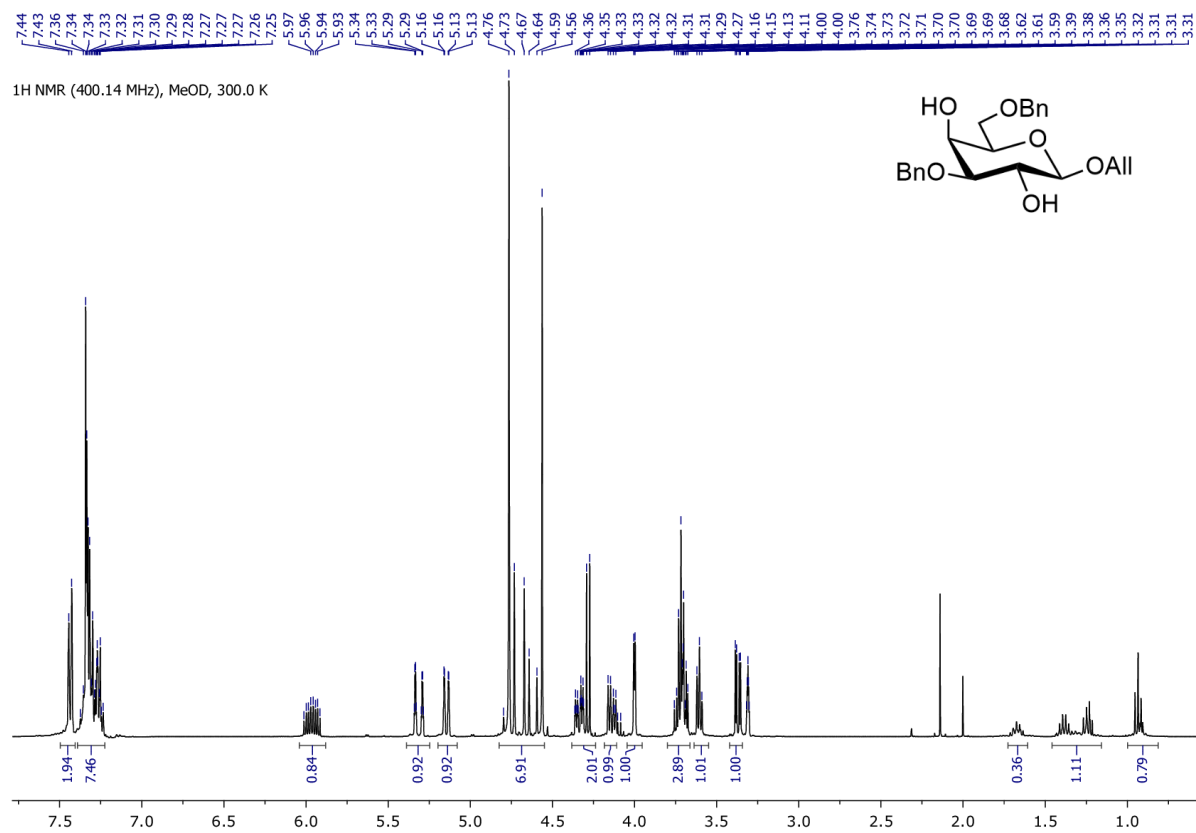
NMR-monitoring of rearrangement of pyranoside **8***



* Conditions: **8** (1 eq.), Py·SO₃ (8 eq.), HSO₃Cl (3.2 eq.), DMF (0.8 mL/1 mmol Py·SO₃), 40 °C. Probes were treated by excess of aq. NaHCO₃, concentrated *in vacuo* and extracted from solid by MeOH.

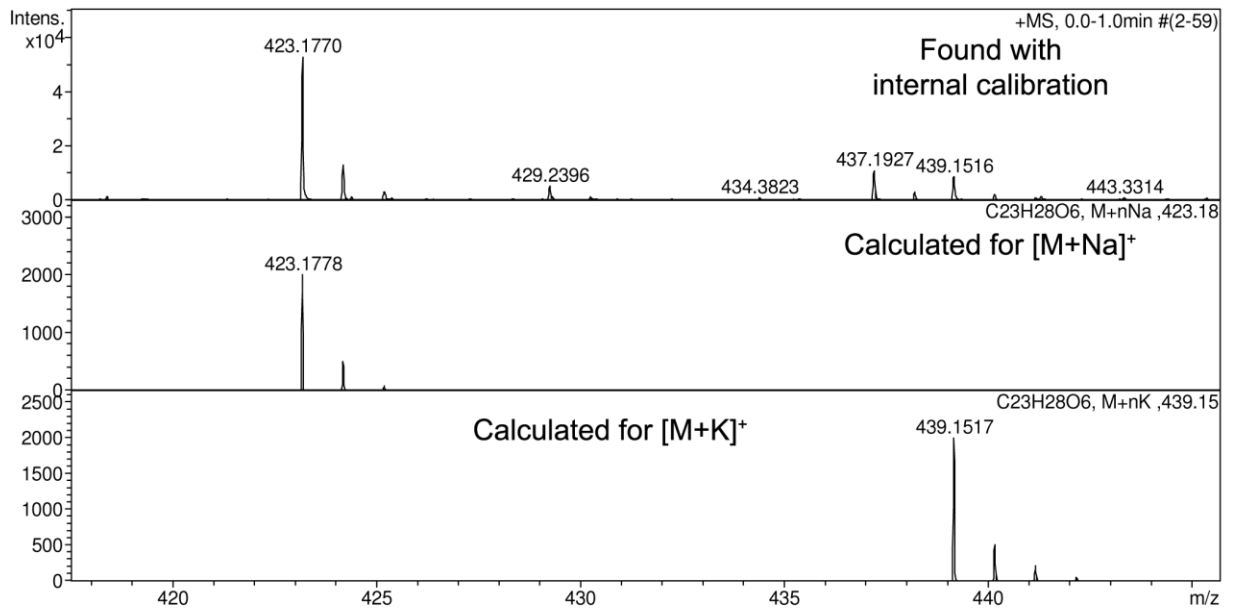
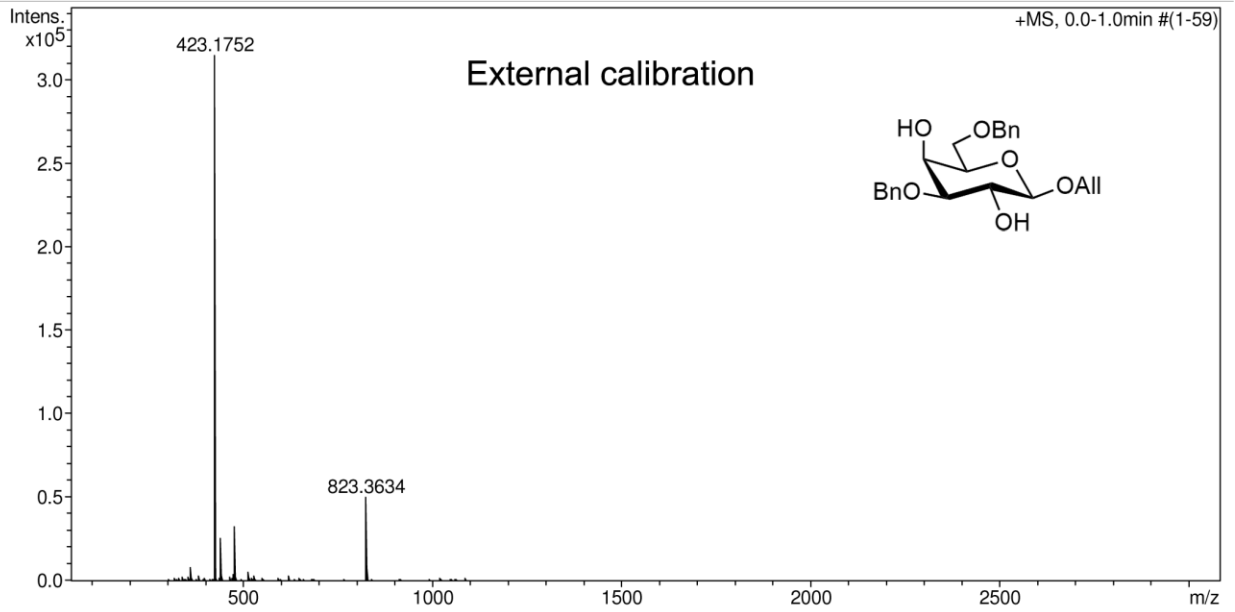
NMR and ESI-mass spectra of compounds

Allyl 3,6-di-O-benzyl-β-D-galactopyranoside (8)

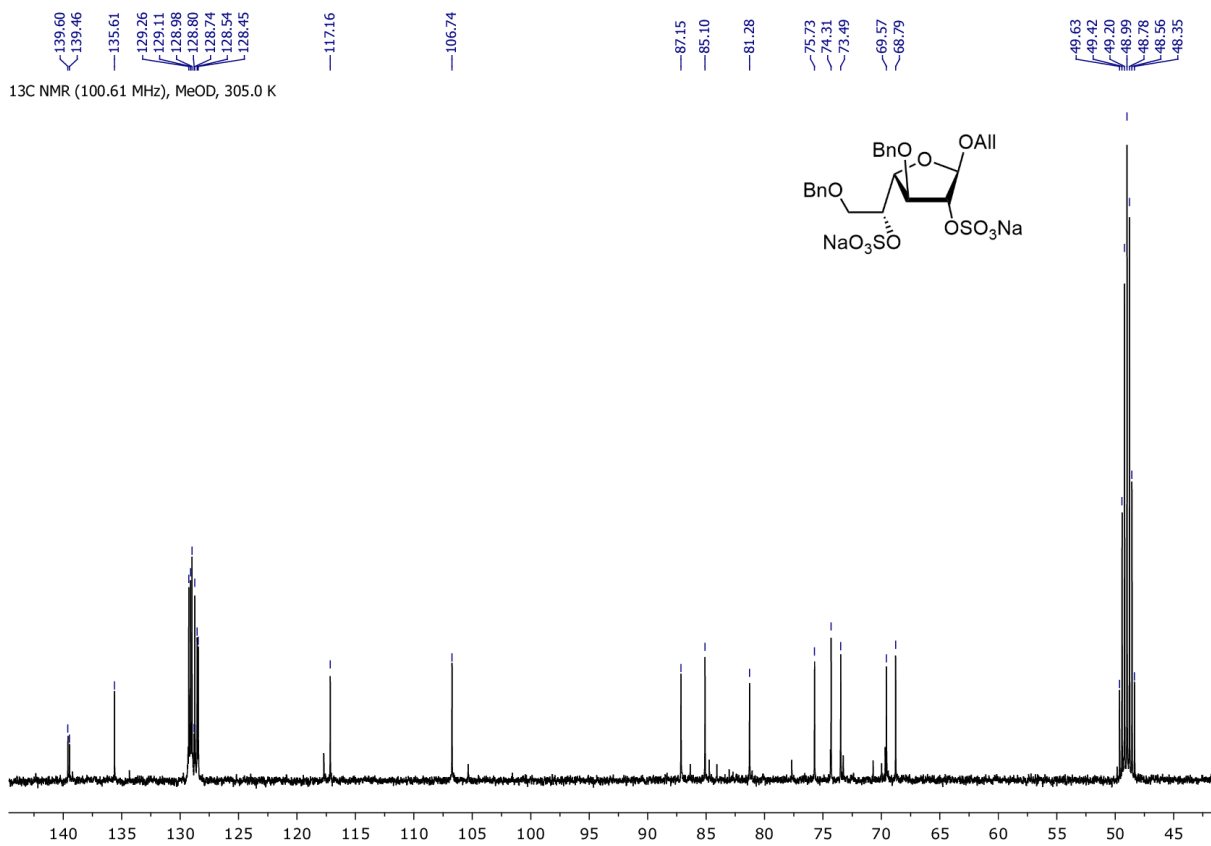
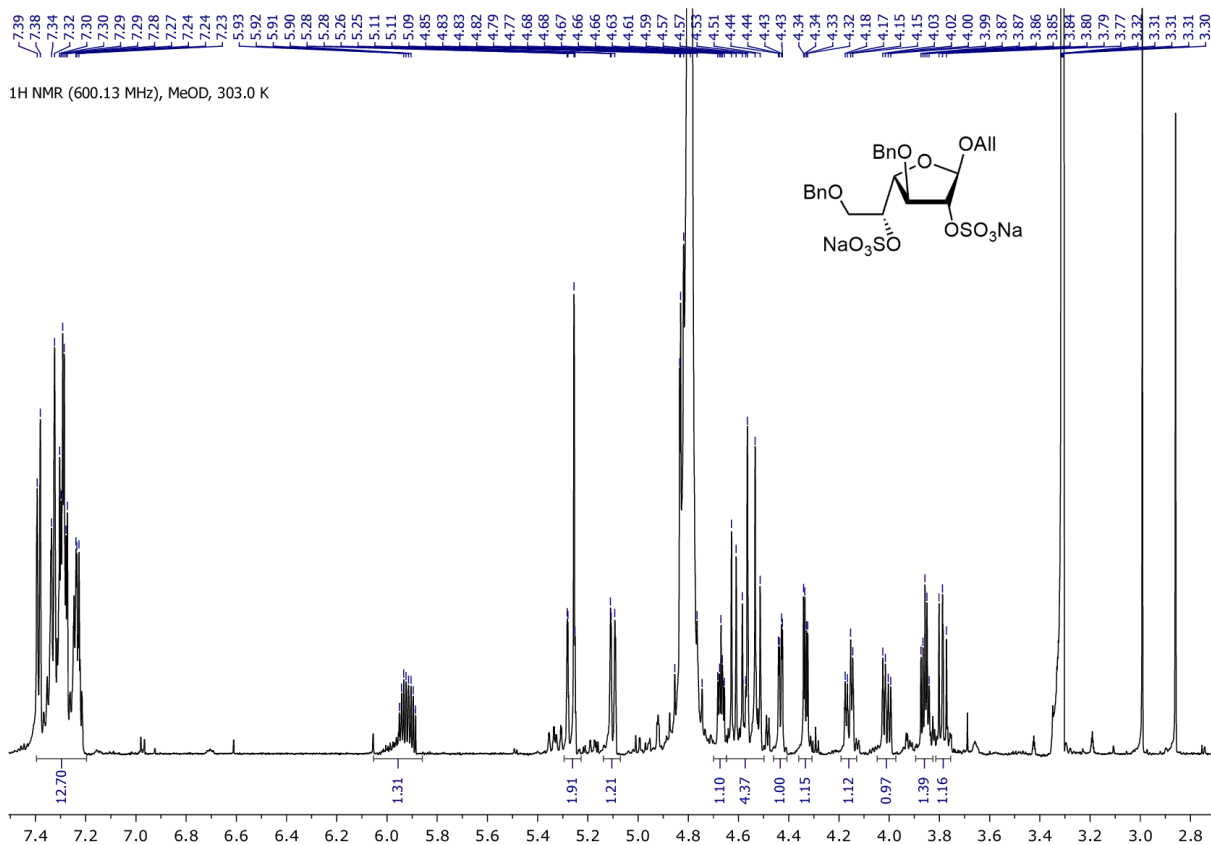


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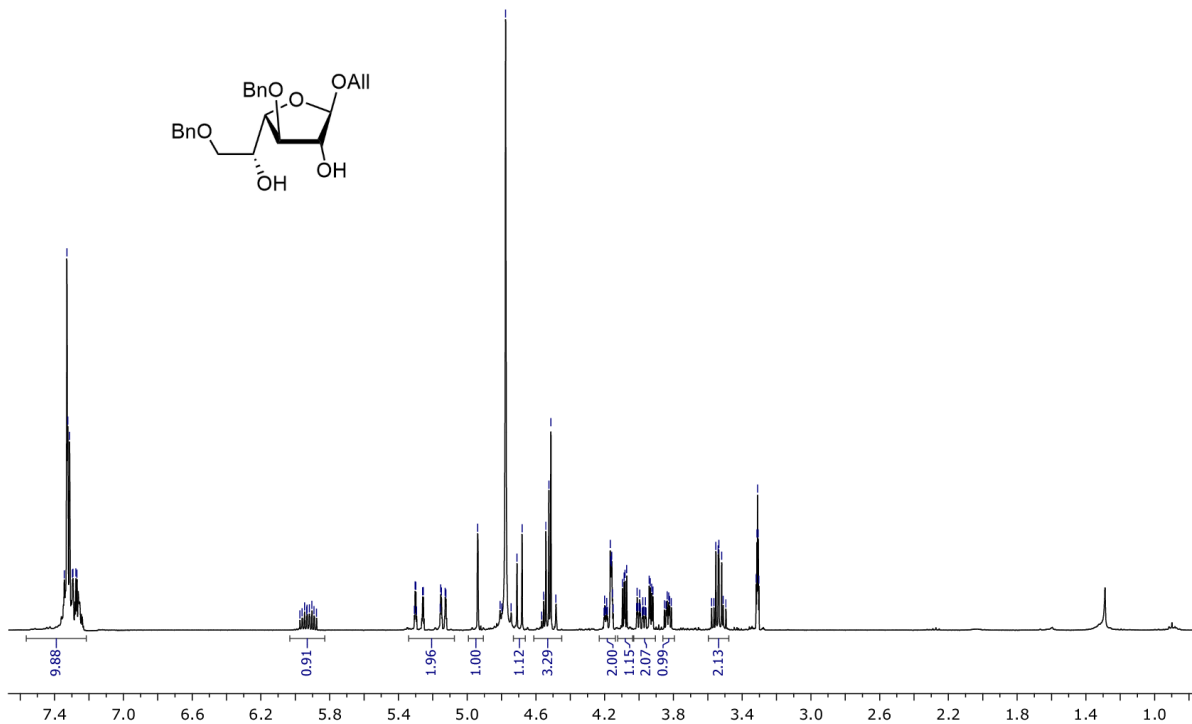
Disodium salt of allyl 3,6-di-O-benzyl-2,5-di-O-sulfo-β-D-galactofuranoside (9)



Allyl 3,6-di-O-benzyl-β-D-galactofuranoside (10)

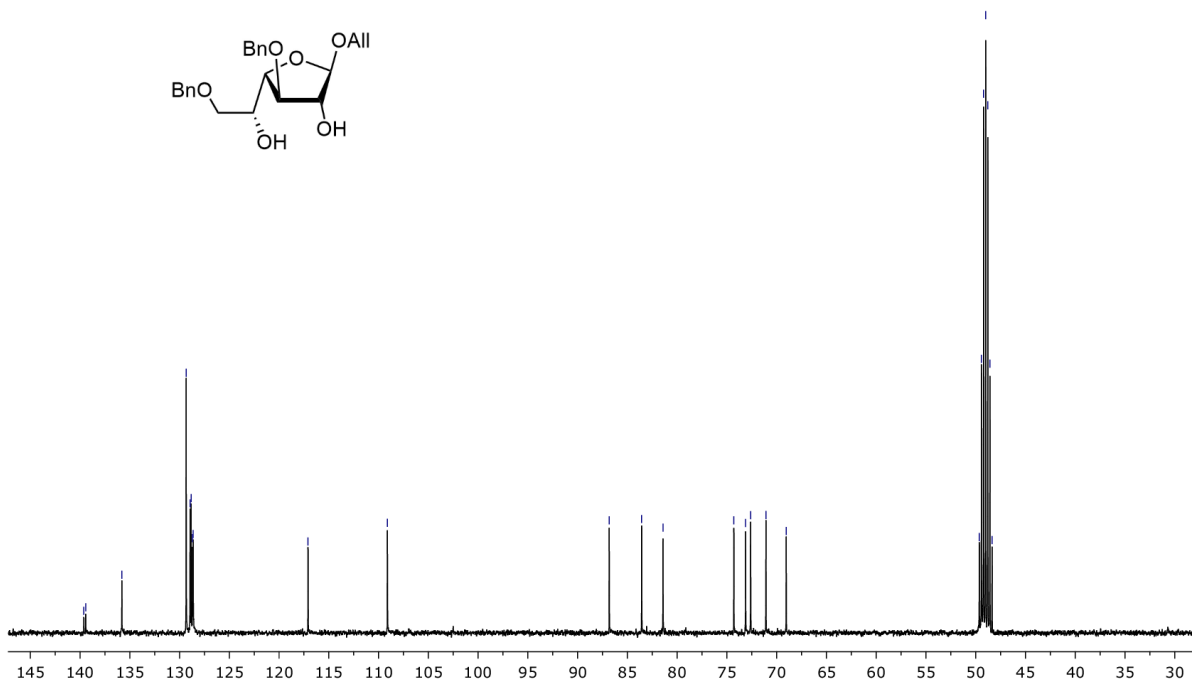
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¹H NMR (400.14 MHz), MeOD, 305.0 K



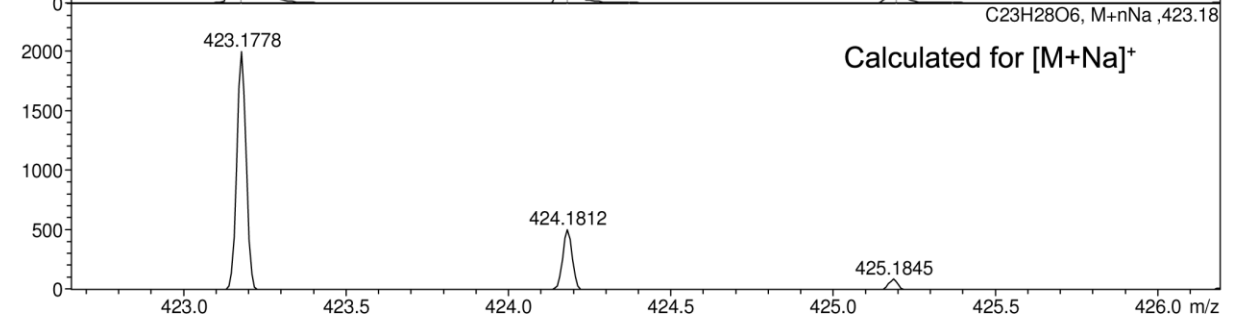
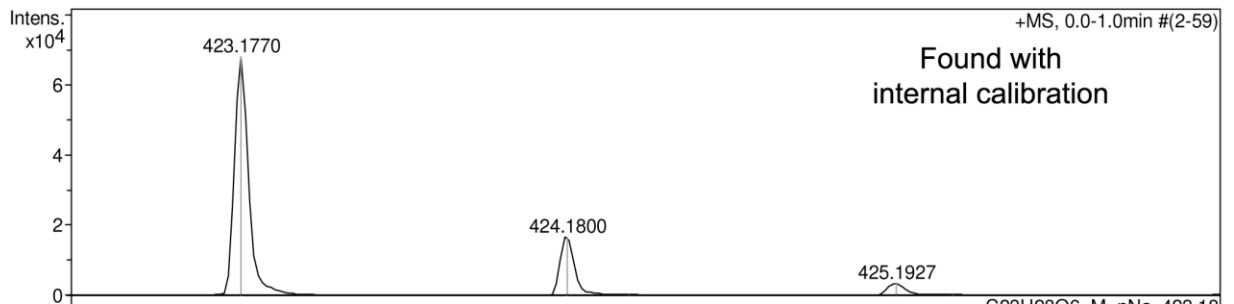
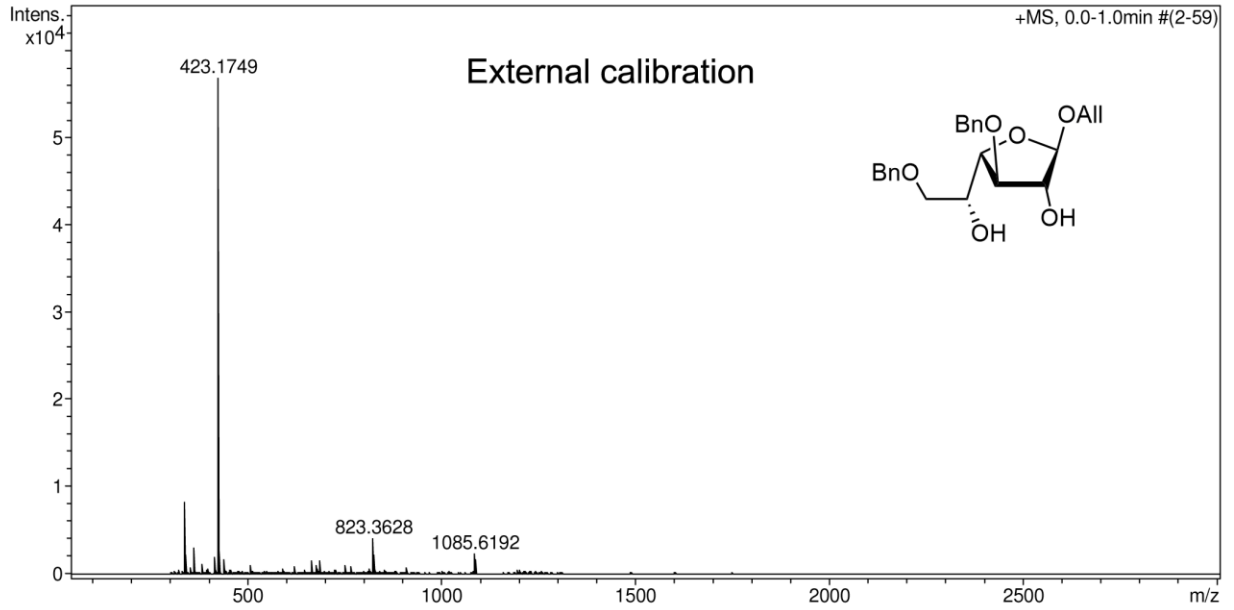
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¹³C NMR (100.61 MHz), MeOD, 305.0 K

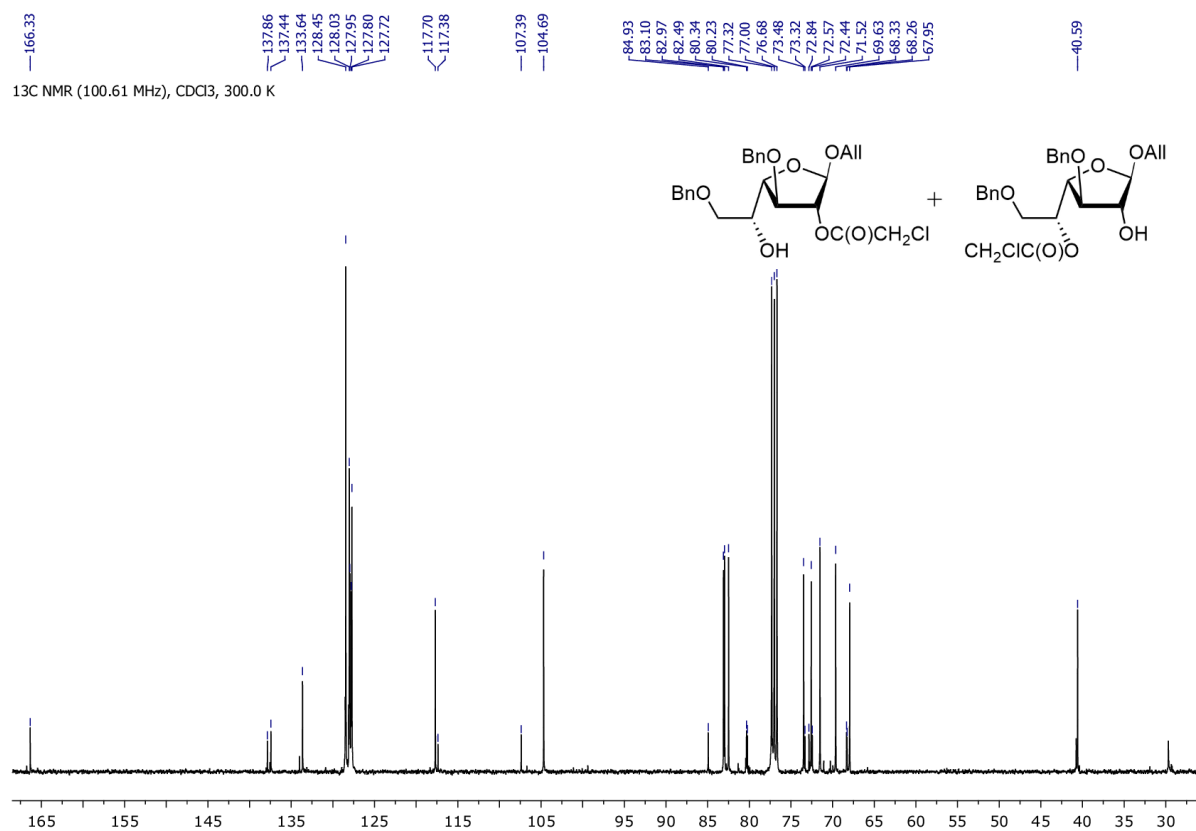
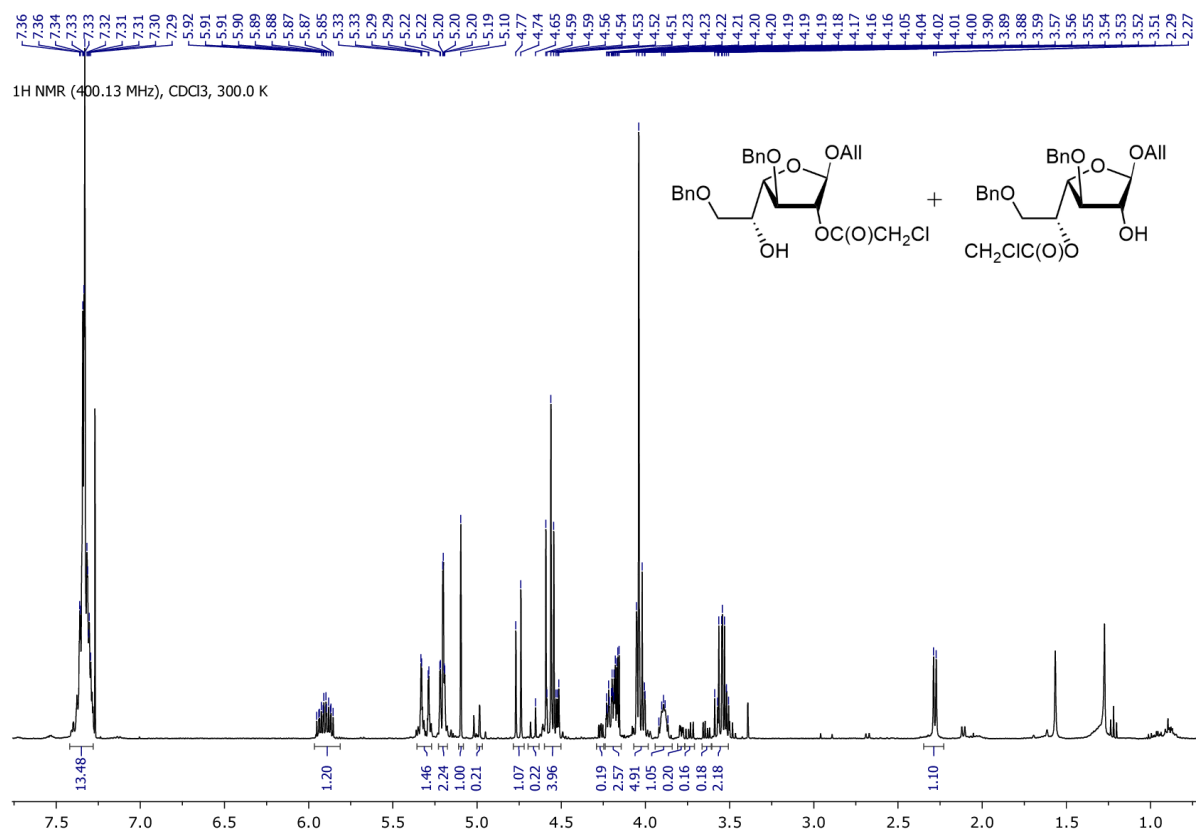


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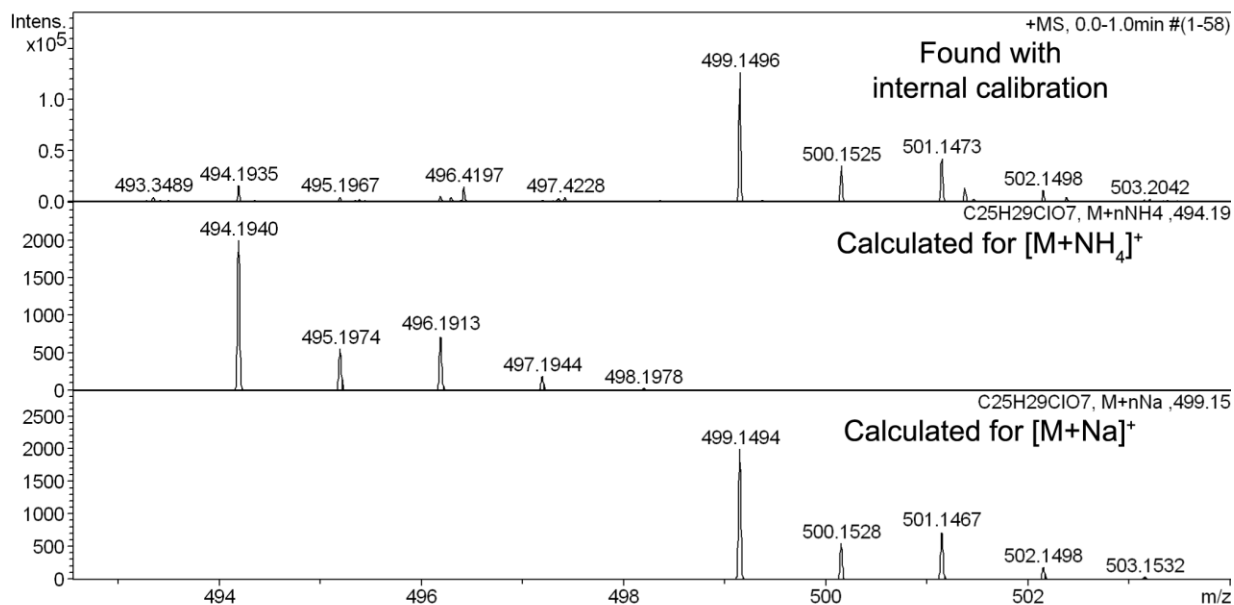
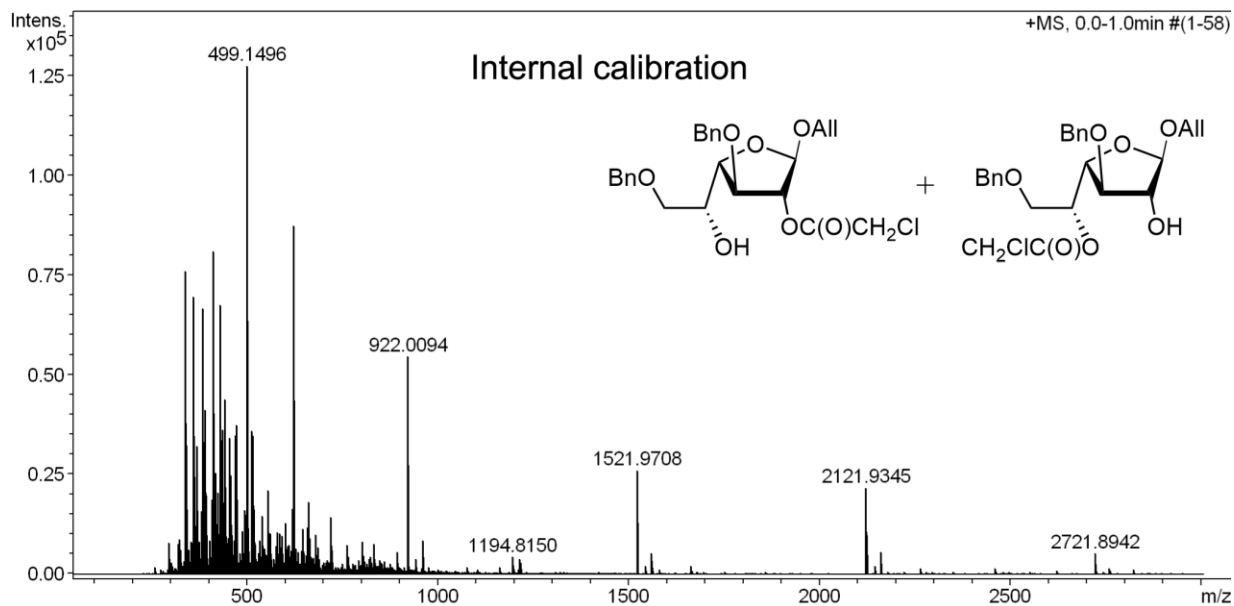


Allyl 3,6-di-O-benzyl-2-O-chloroacetyl-β-D-galactofuranoside (11)



Acquisition Parameter

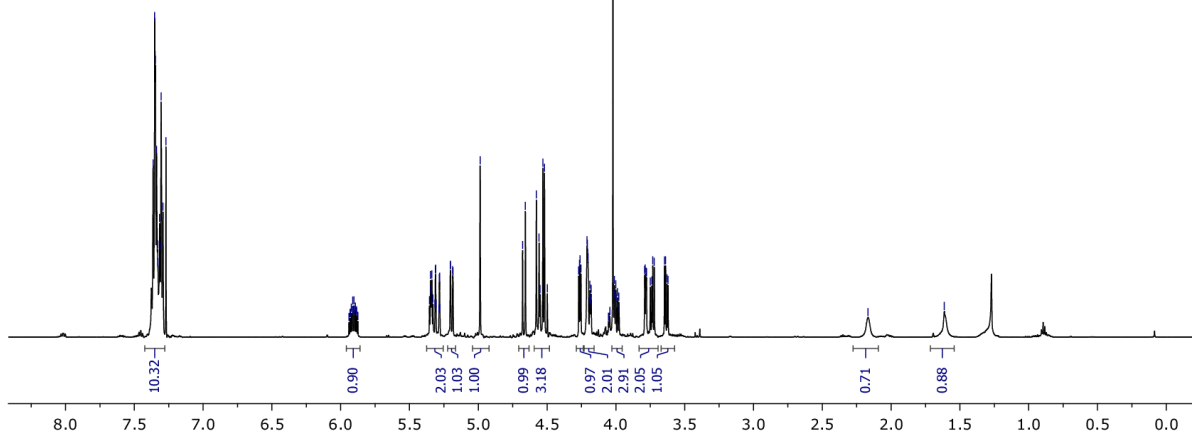
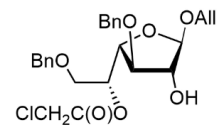
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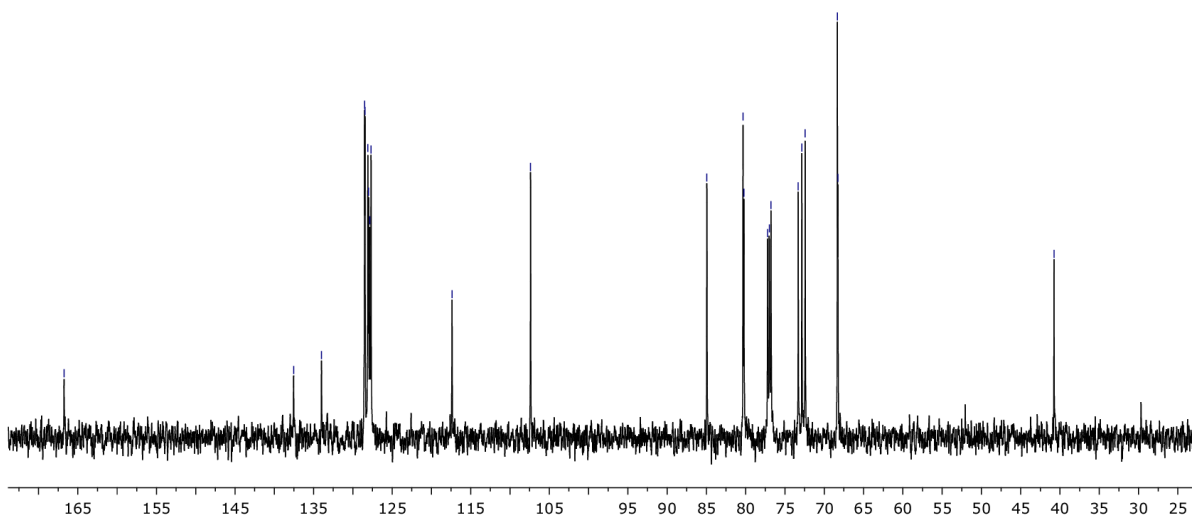
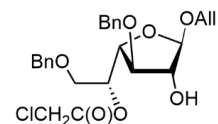
Allyl 3,6-di-O-benzyl-5-O-chloroacetyl-β-D-galactofuranoside (12)



¹H NMR (600.13 MHz), CDCl₃, 300.3 K

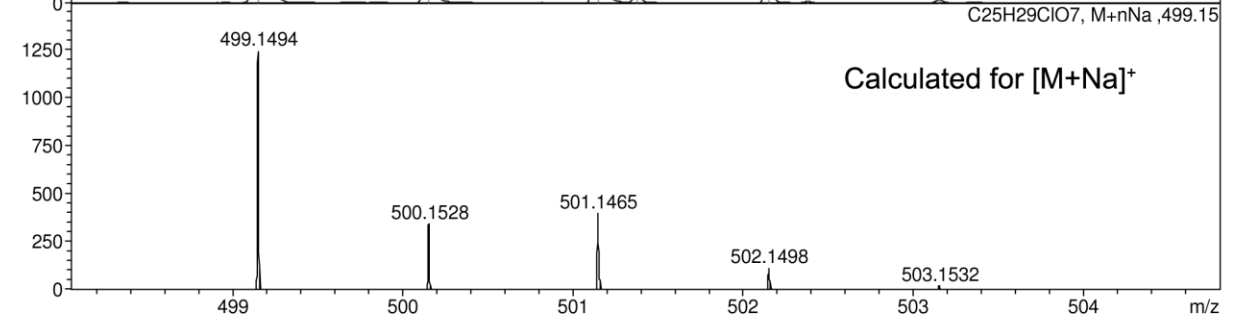
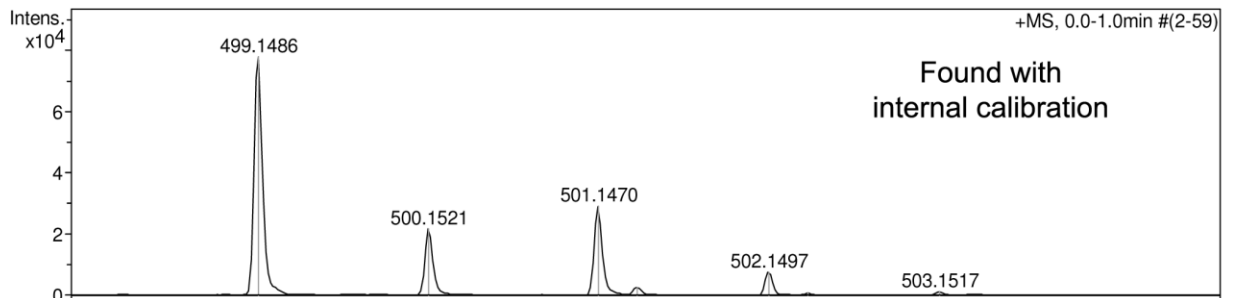
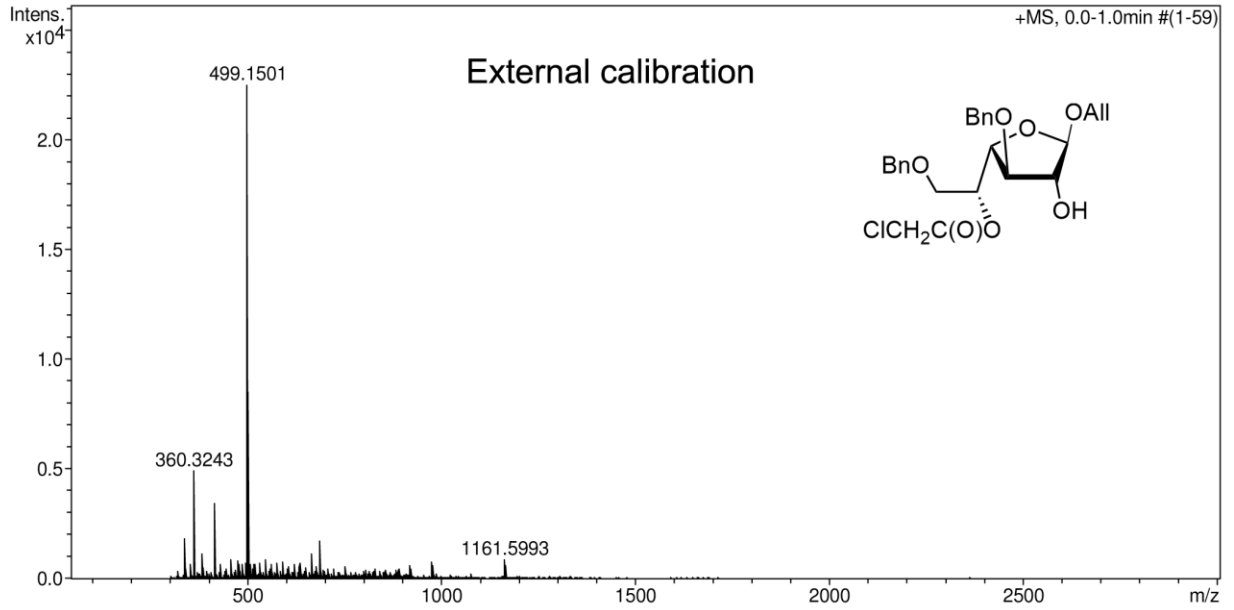


¹³C NMR (150.92 MHz), CDCl₃, 300.6 K

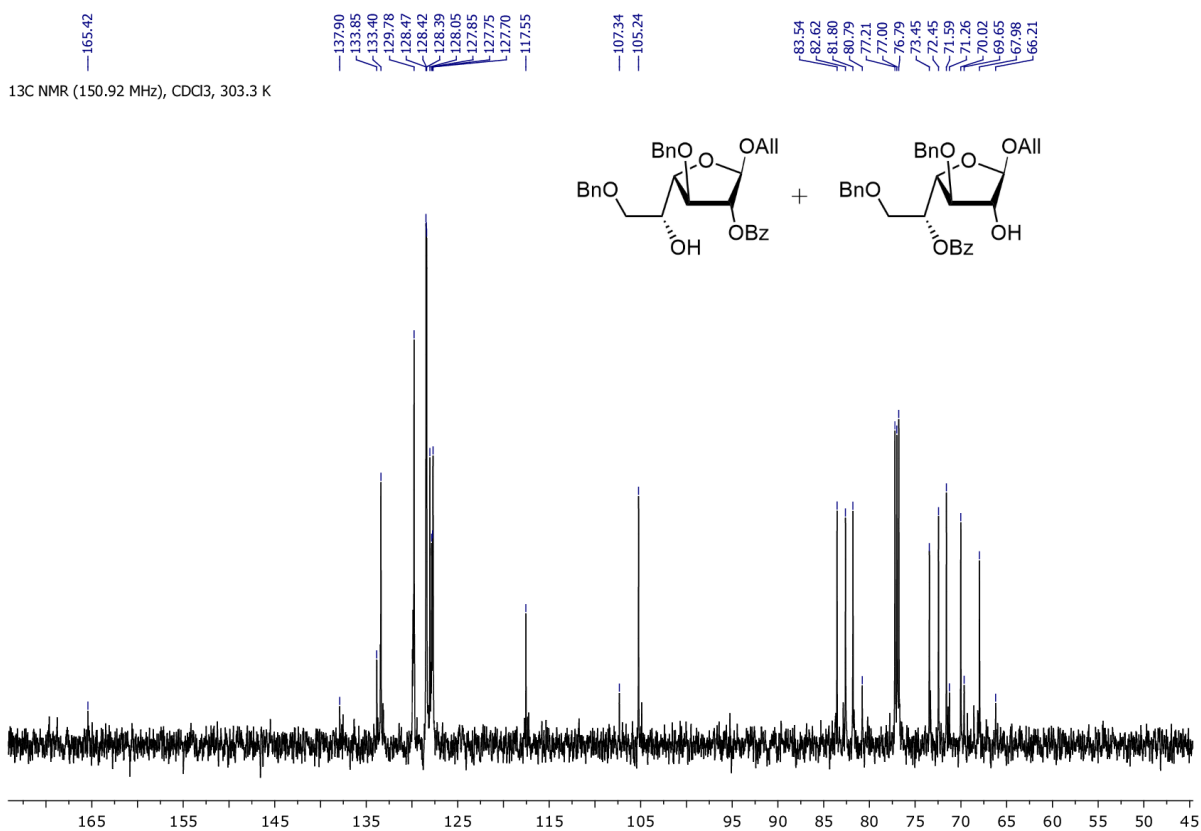
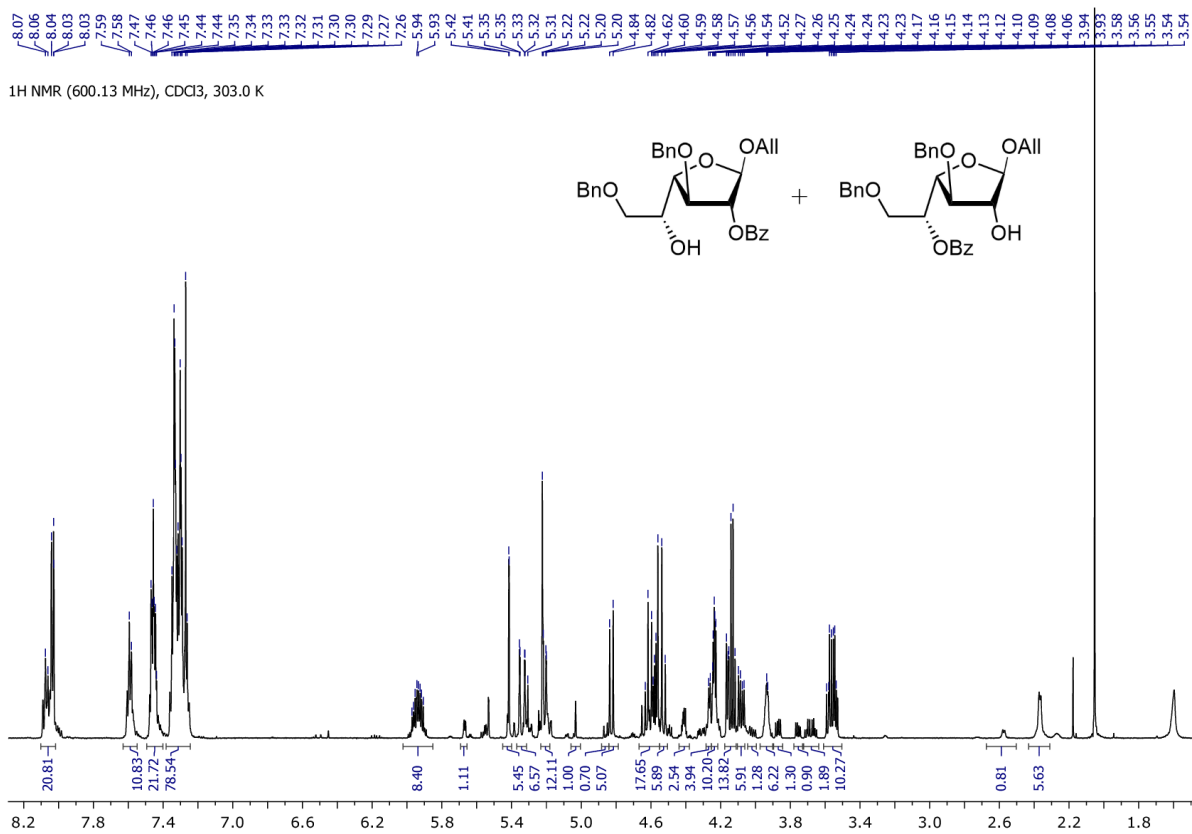


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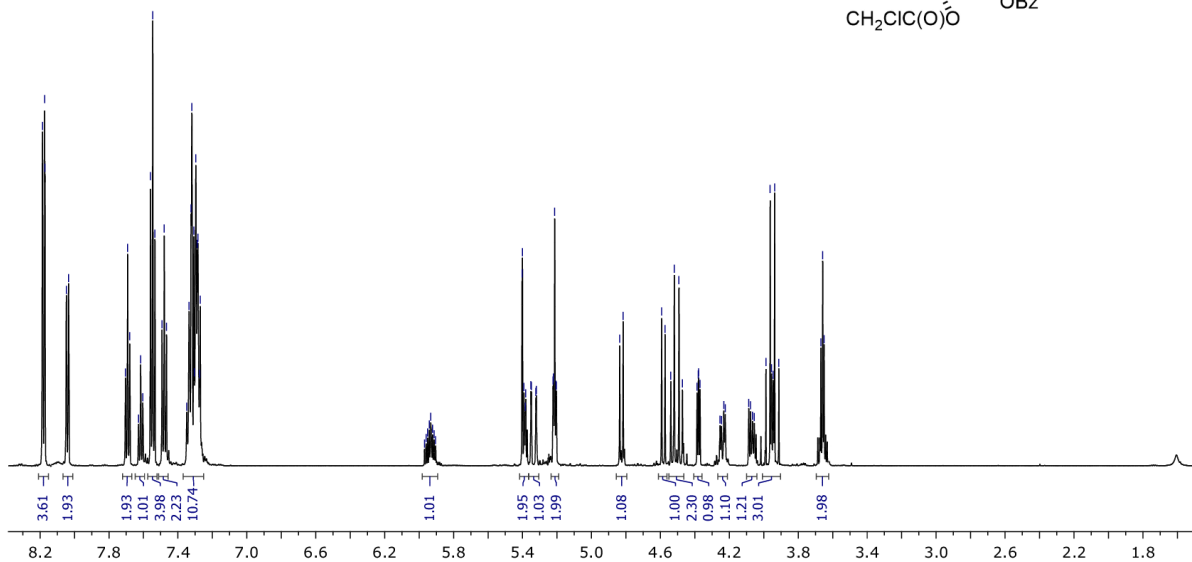
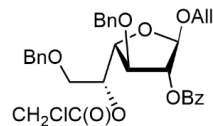
Allyl 2-O-benzoyl-3,6-di-O-benzyl-β-D-galactofuranoside (13) from diol 10



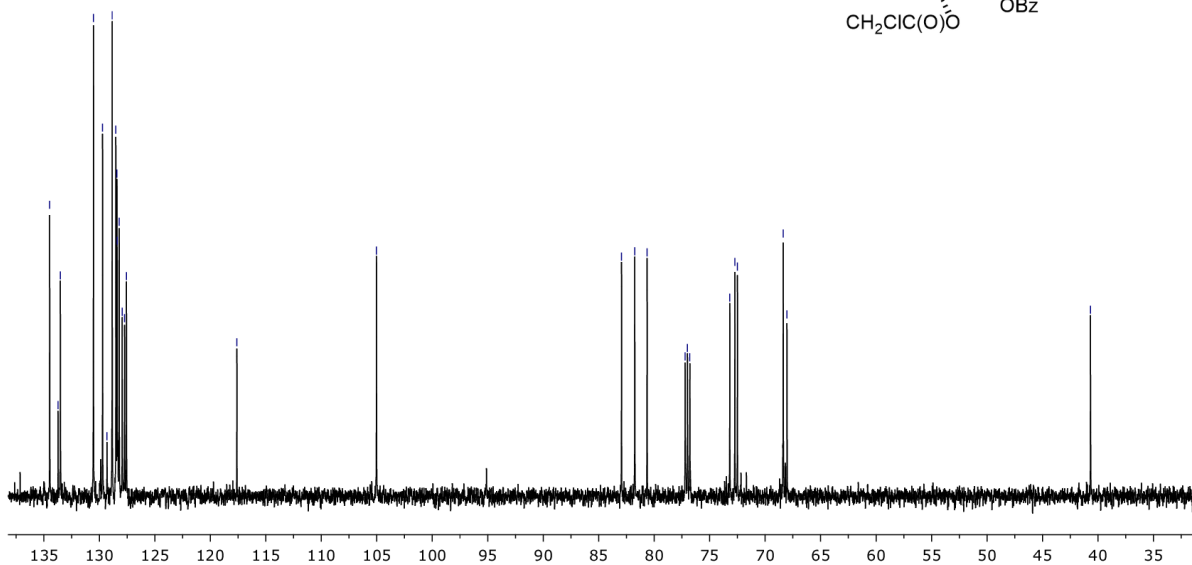
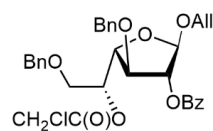
Allyl 2-O-benzoyl-3,6-di-O-benzyl-5-O-chloroacetyl-β-D-galactofuranoside (15)



¹H NMR (600.13 MHz), CDCl₃, 303.0 K

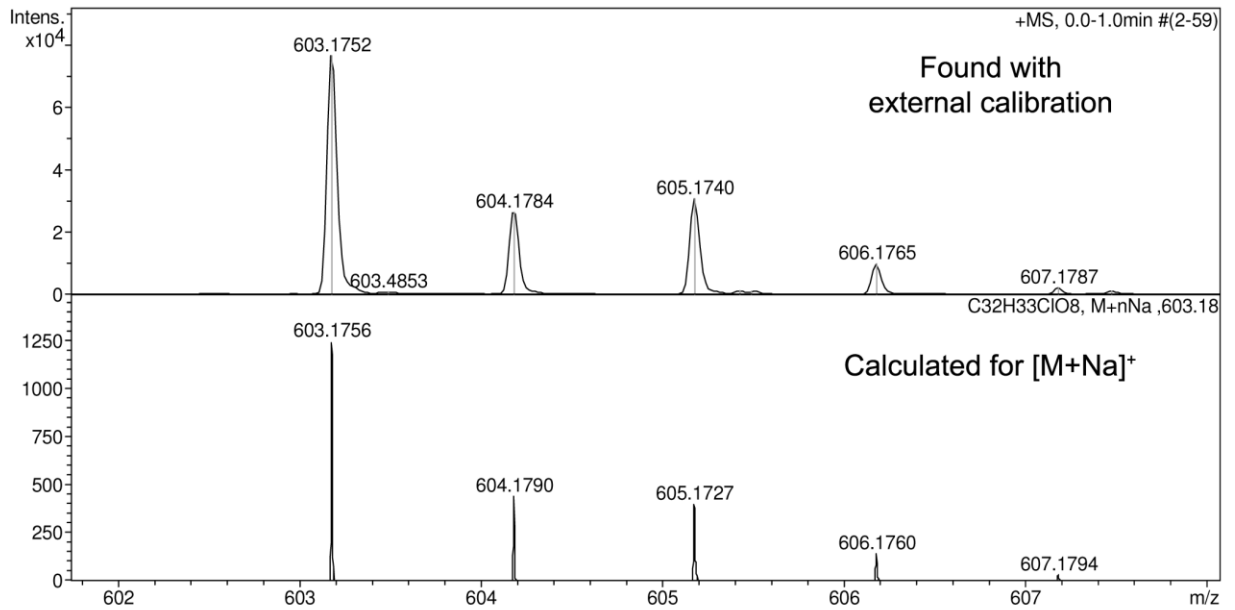
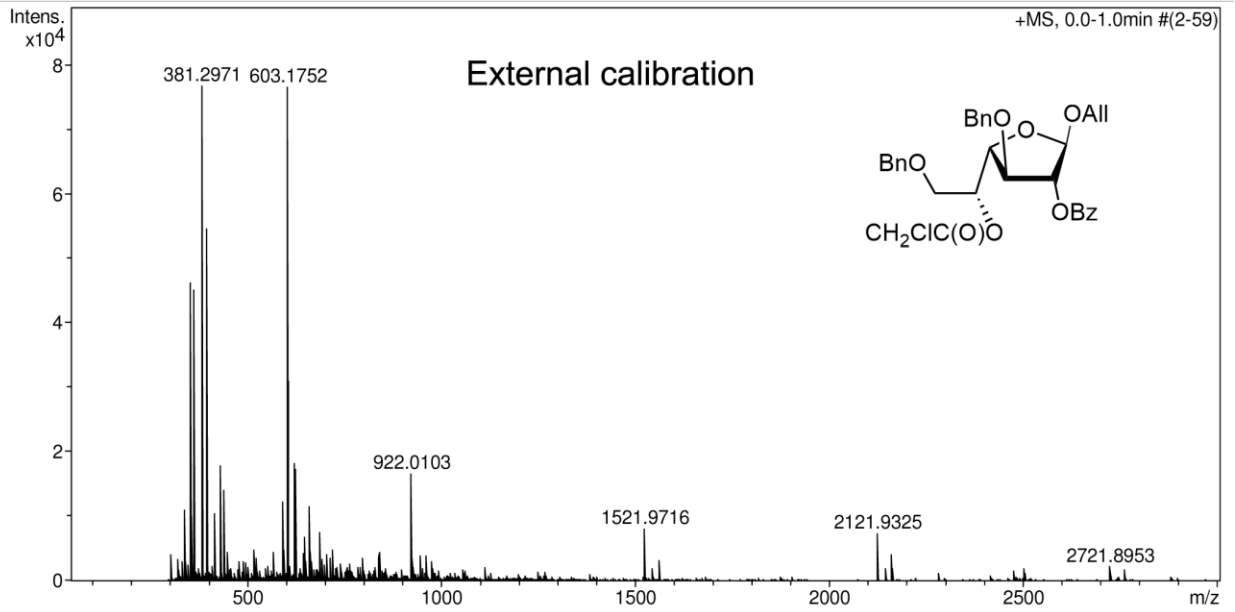


¹³C NMR (150.91 MHz), CDCl₃, 303.1 K

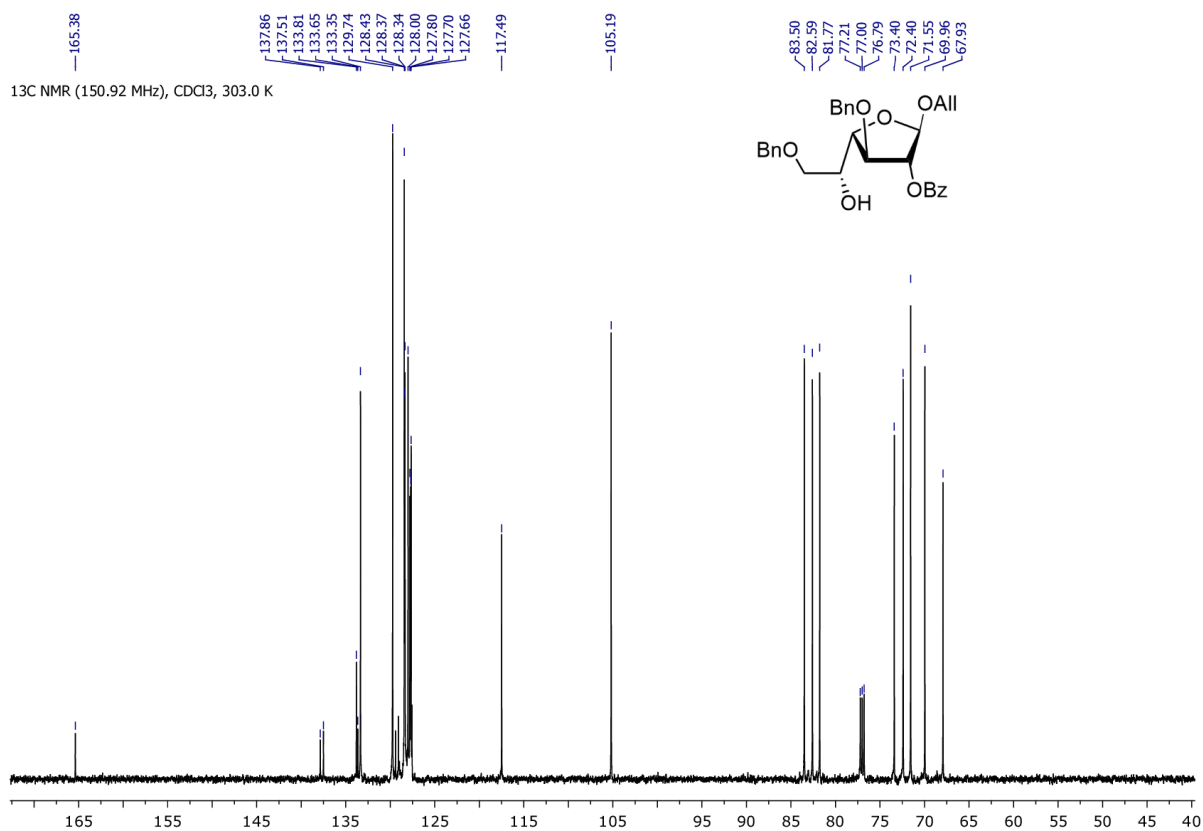
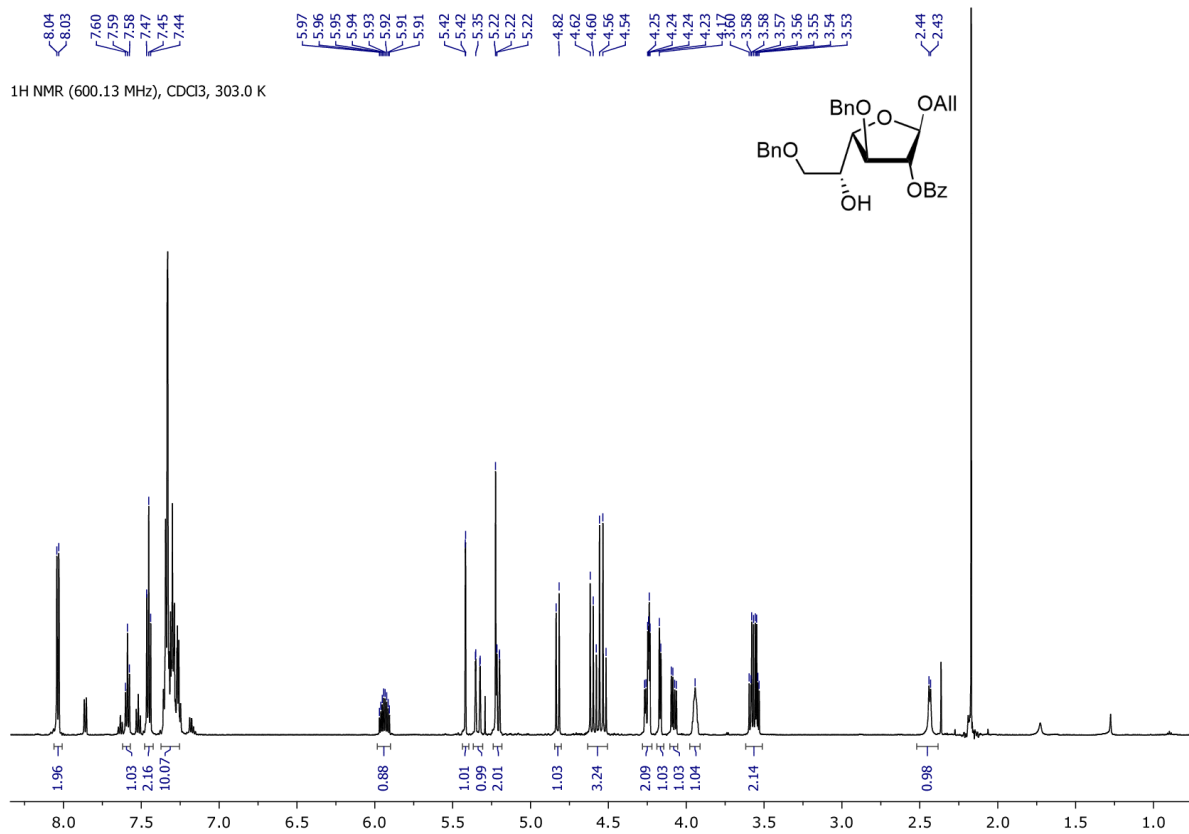


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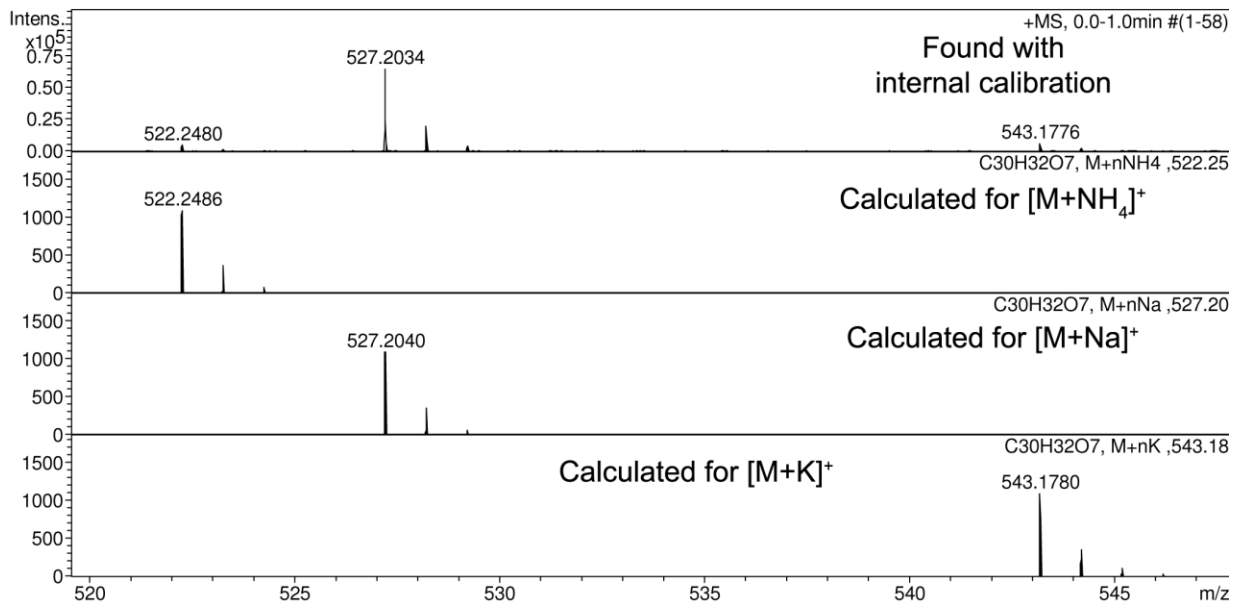
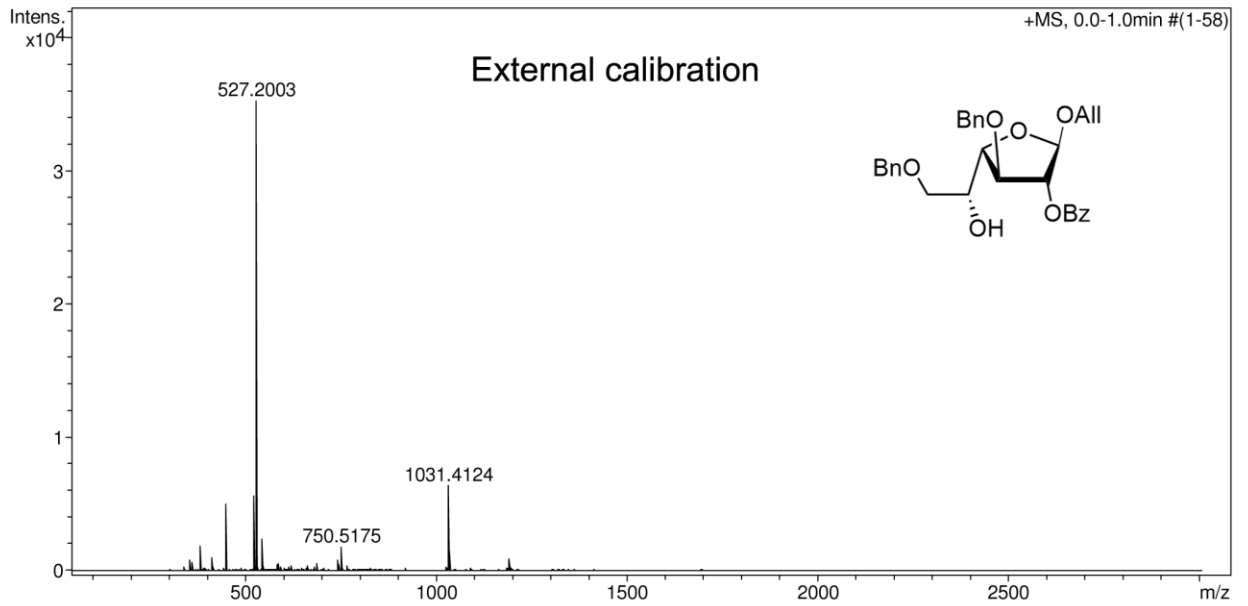


Allyl 2-O-benzoyl-3,6-di-O-benzyl- β -D-galactofuranoside (13) (pure, from 15)

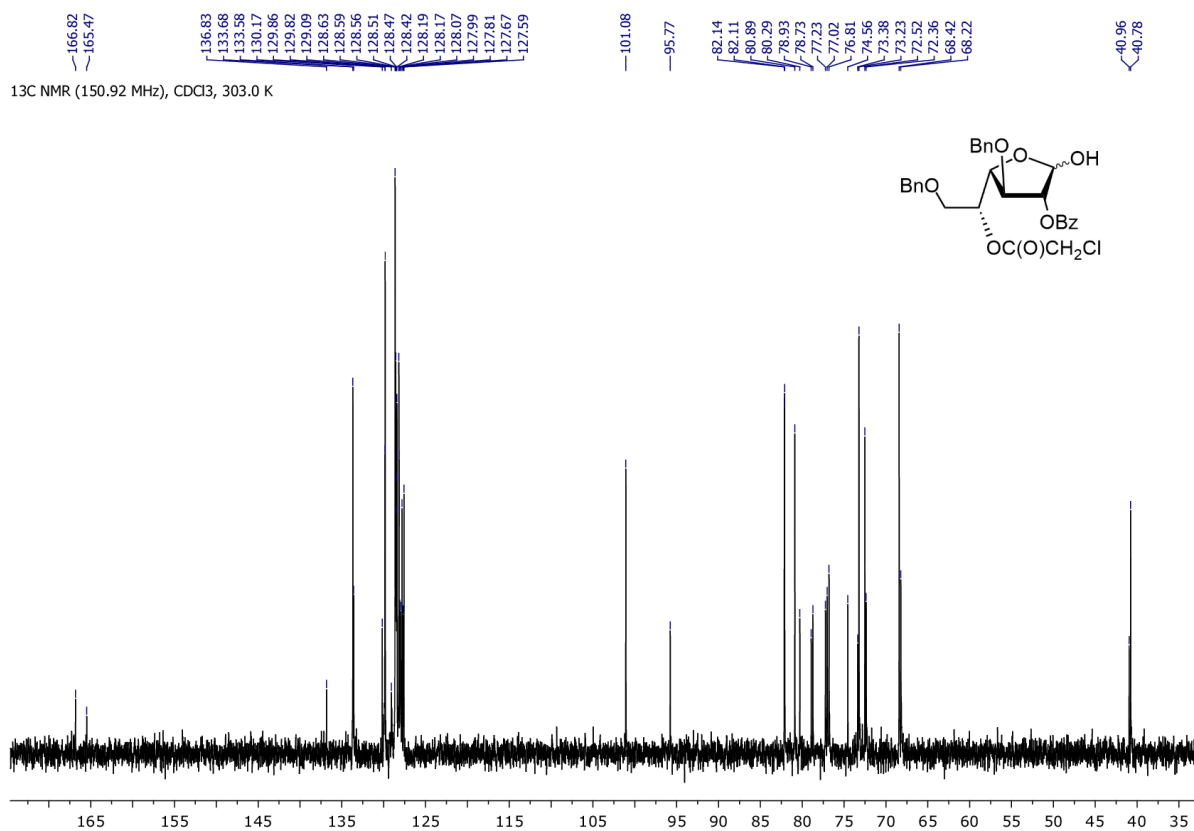
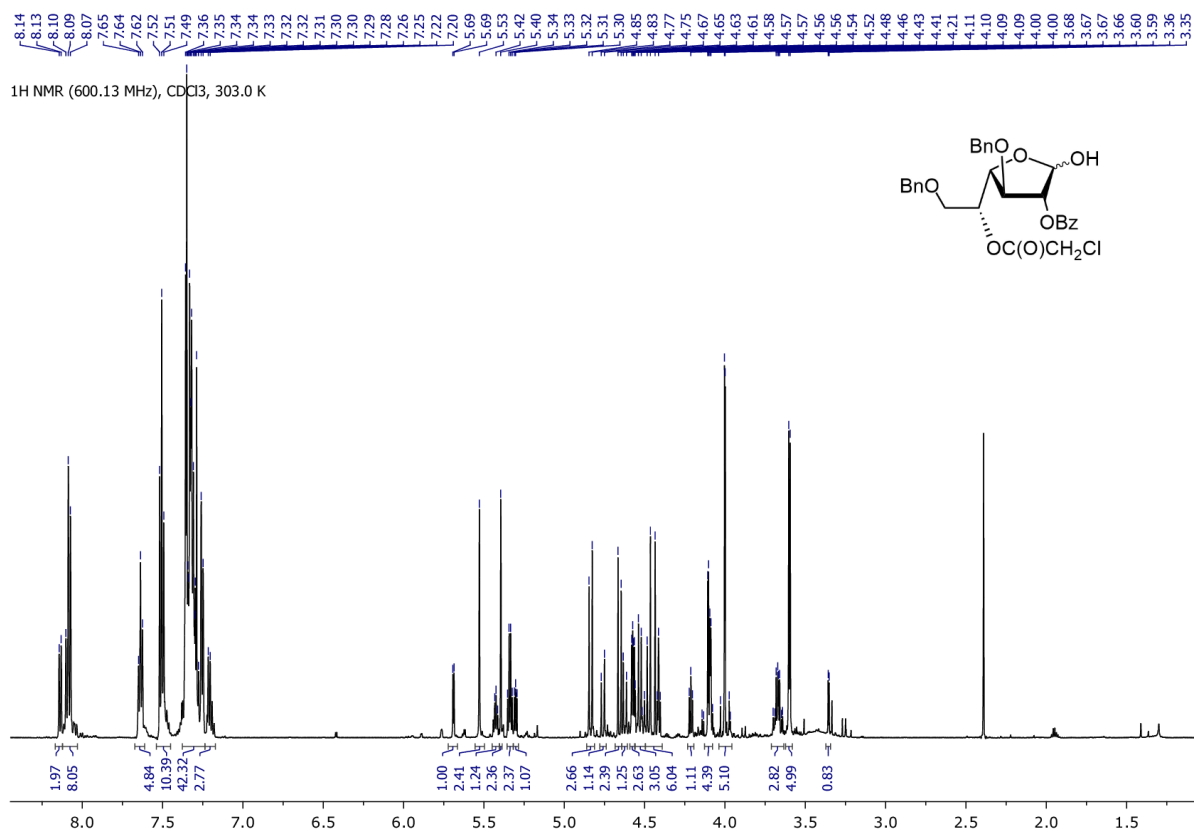


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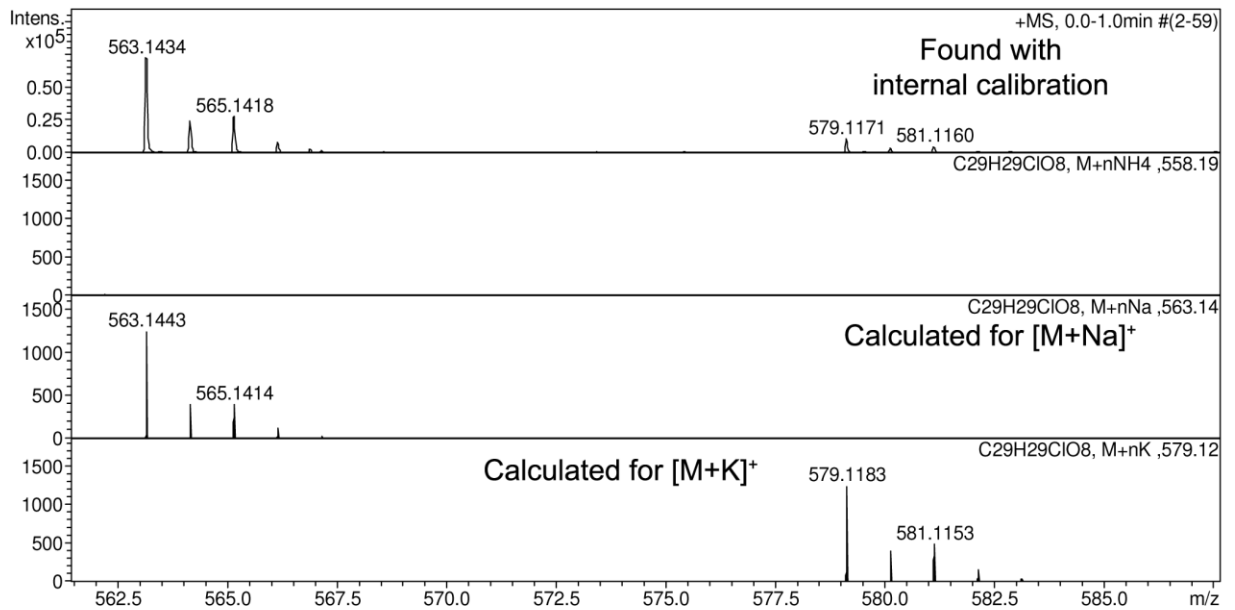
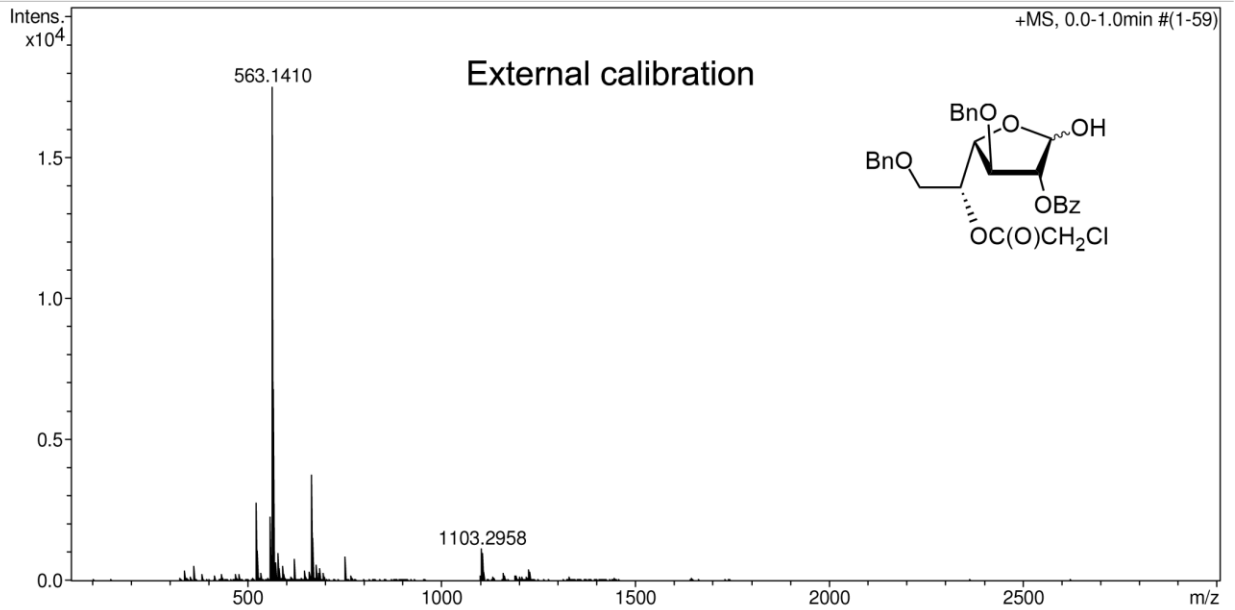


2-O-benzoyl-3,6-di-O-benzyl-5-O-chloroacetyl-D-galactofuranose (16)

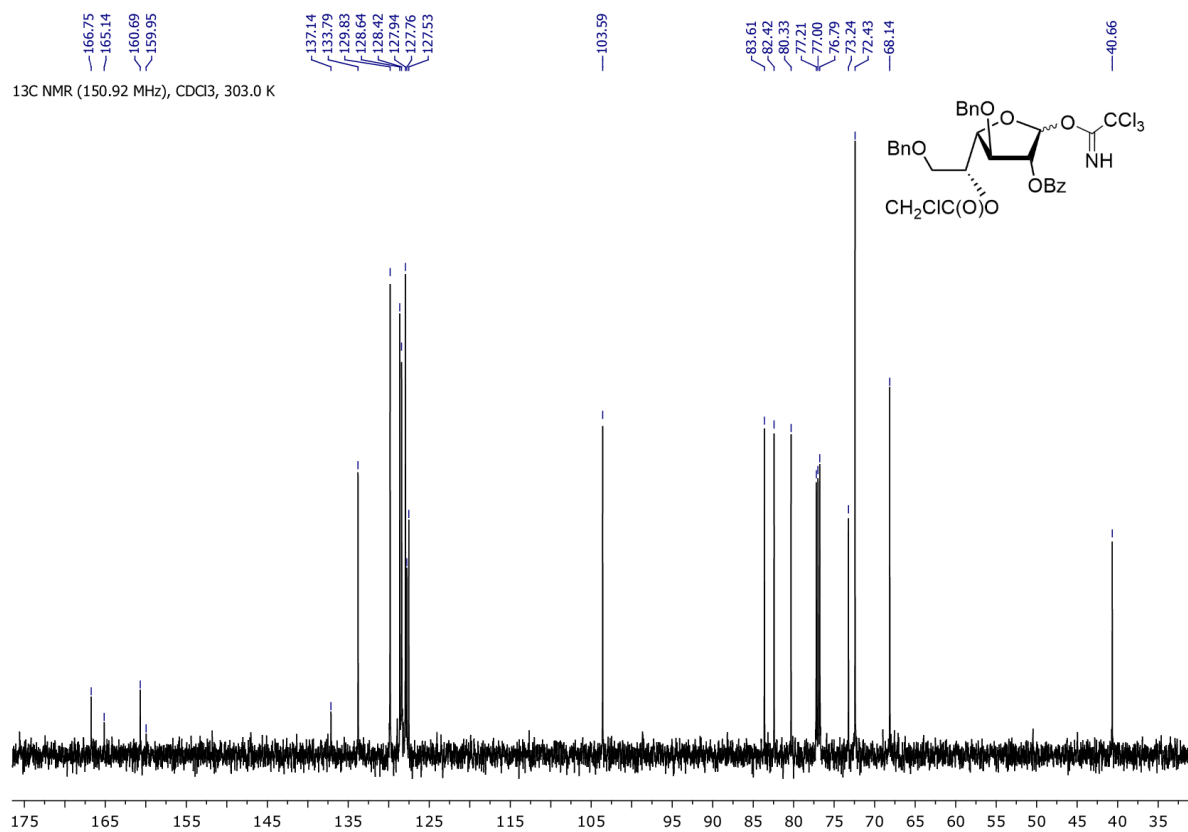
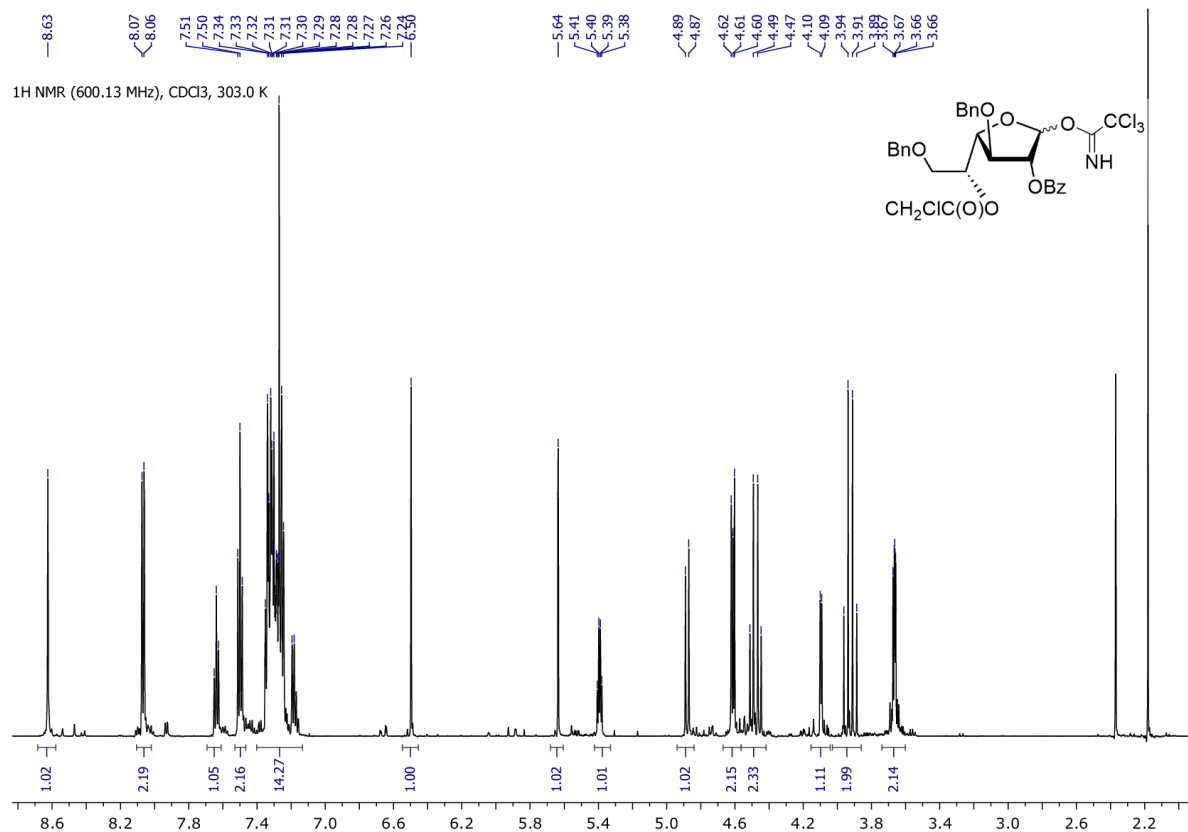


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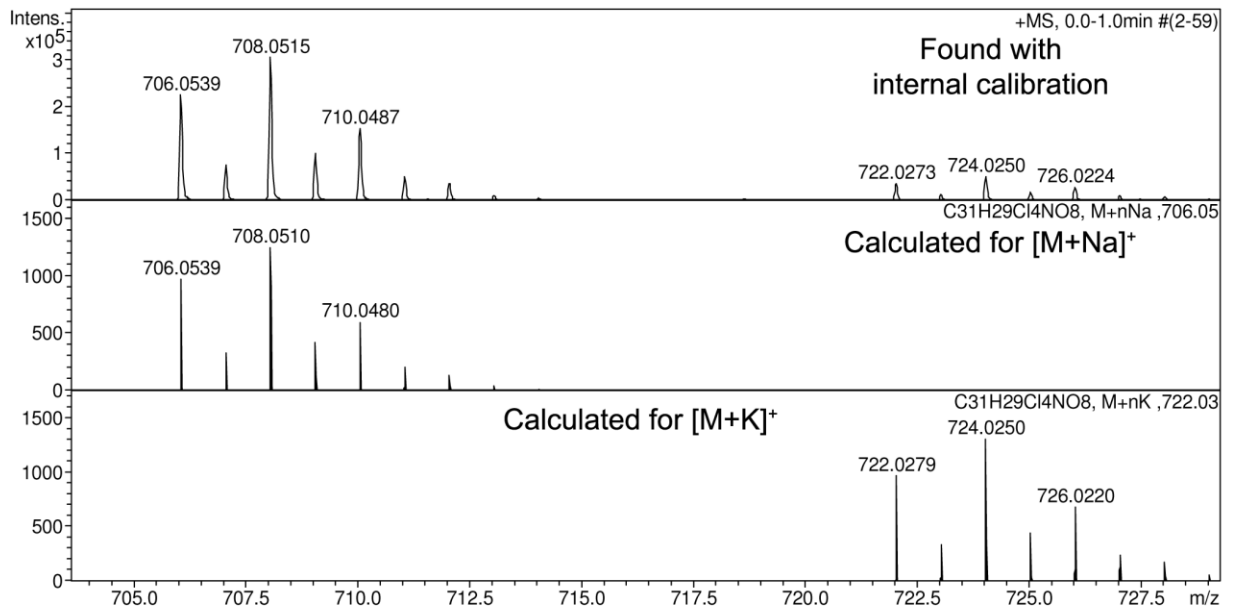
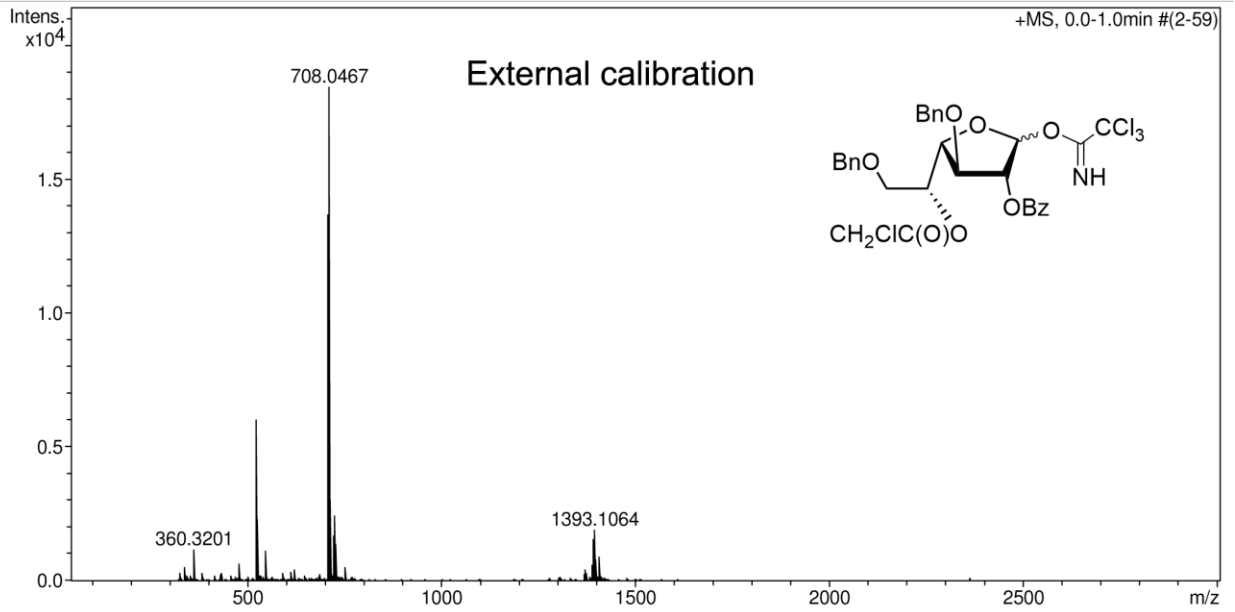


2-O-benzoyl-3,6-di-O-benzyl-5-O-chloroacetyl- β -D-galactofuranoside trichloroacetimidate (17)

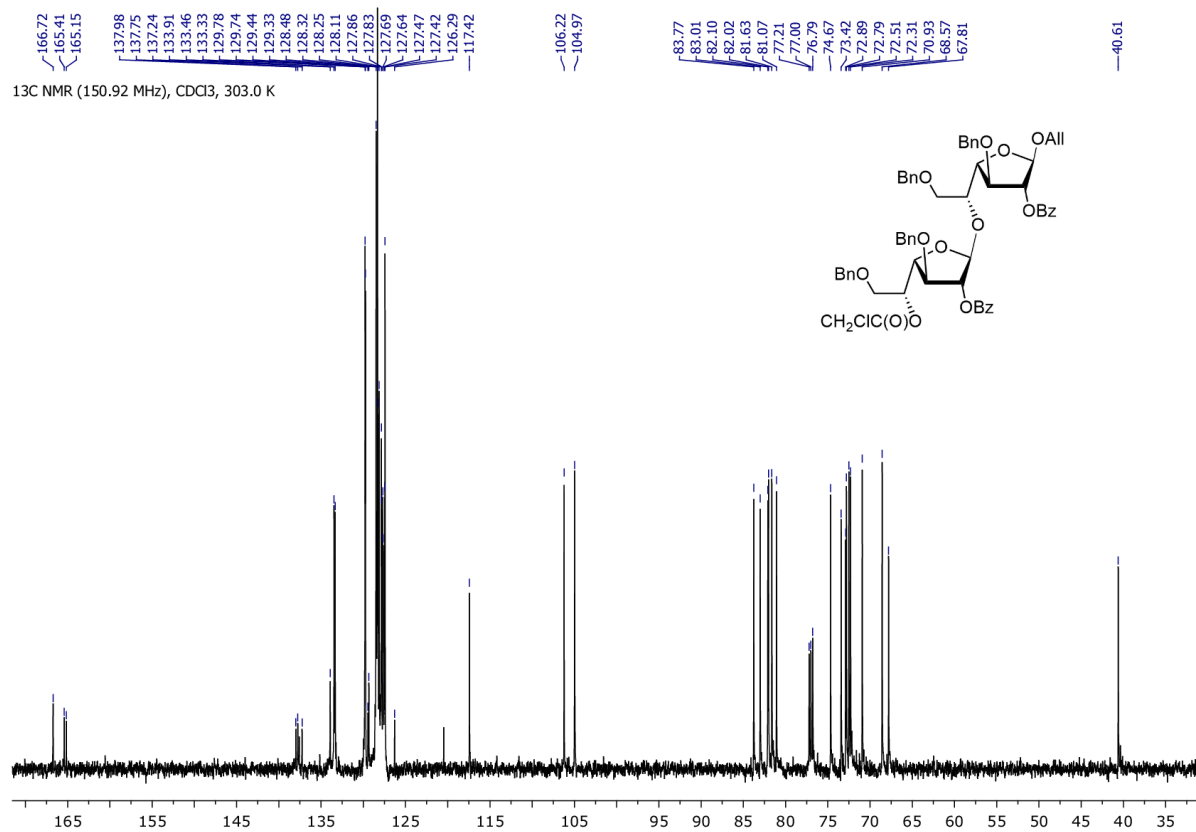
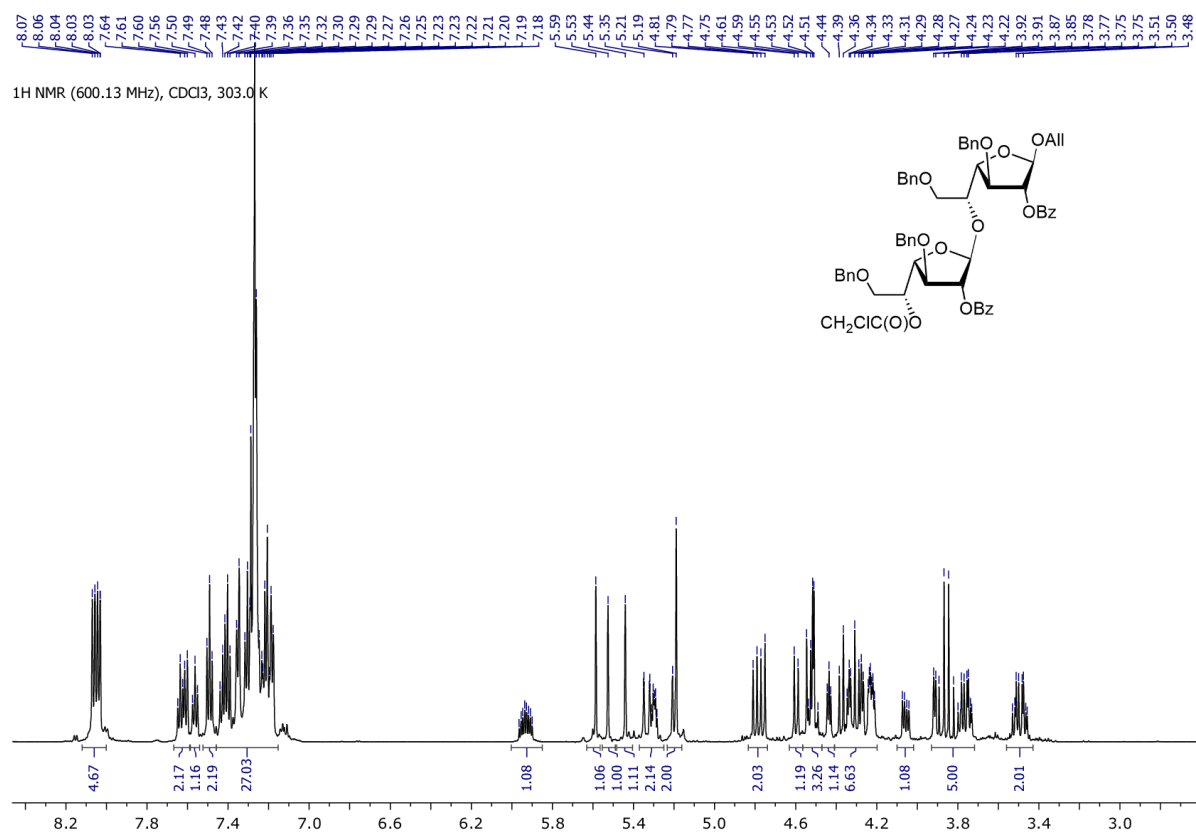


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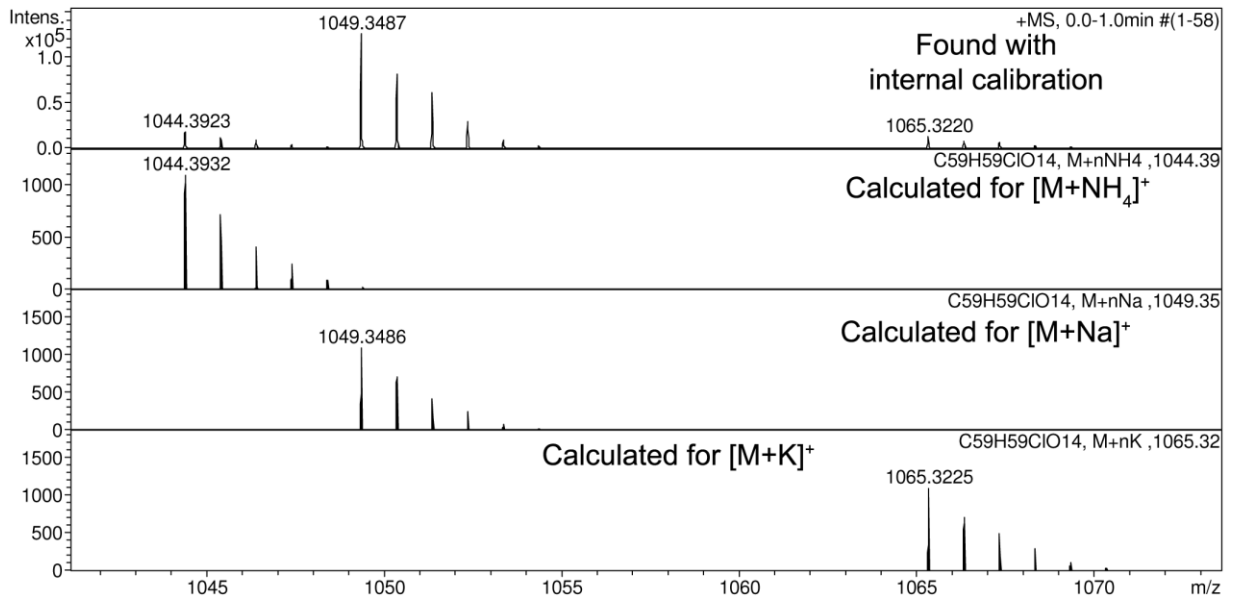
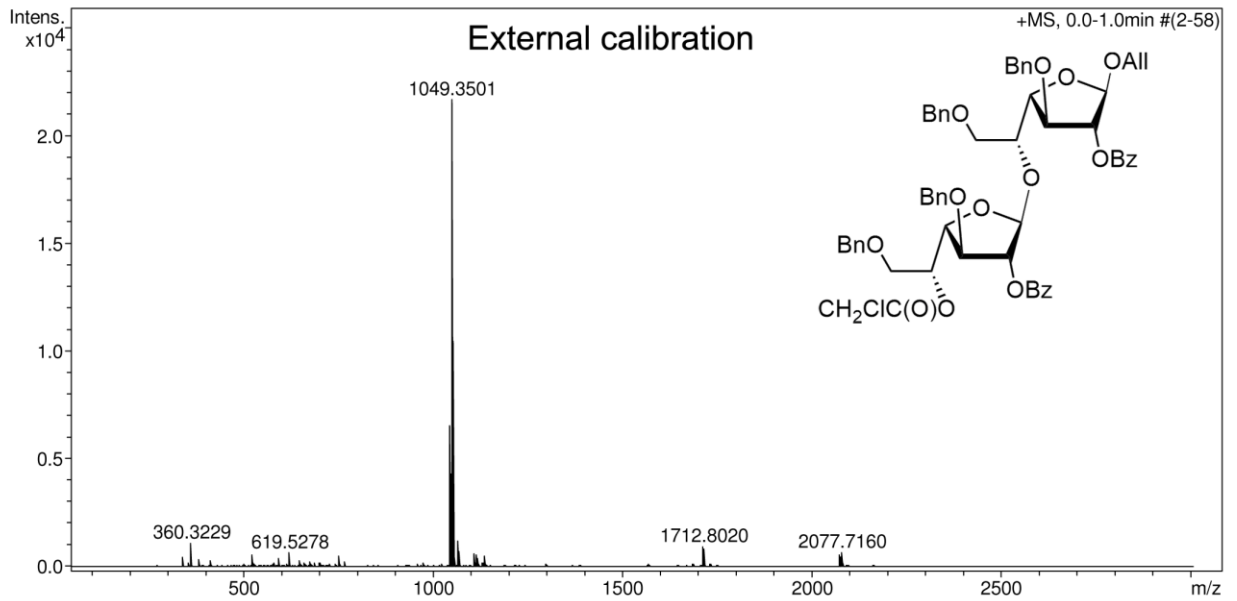


Allyl 2-O-benzoyl-3,6-di-O-benzyl-5-O-chloroacetyl-β-D-galactofuranosyl-(1→5)-2-O-benzoyl-3,6-di-O-benzyl-β-D-galactofuranoside (18)



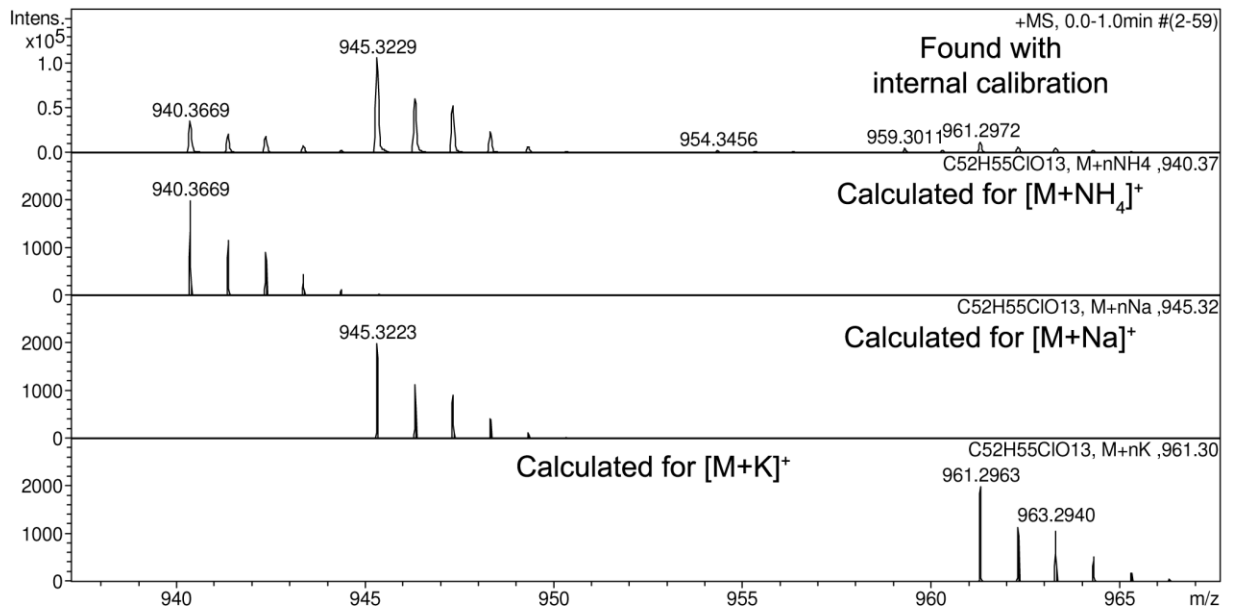
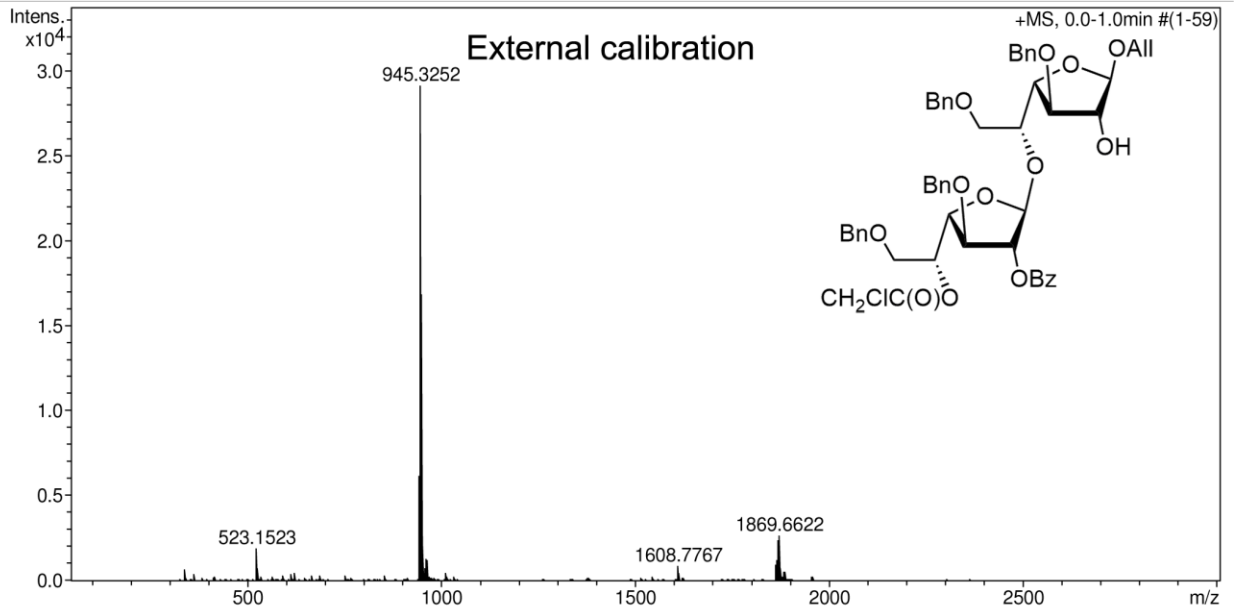
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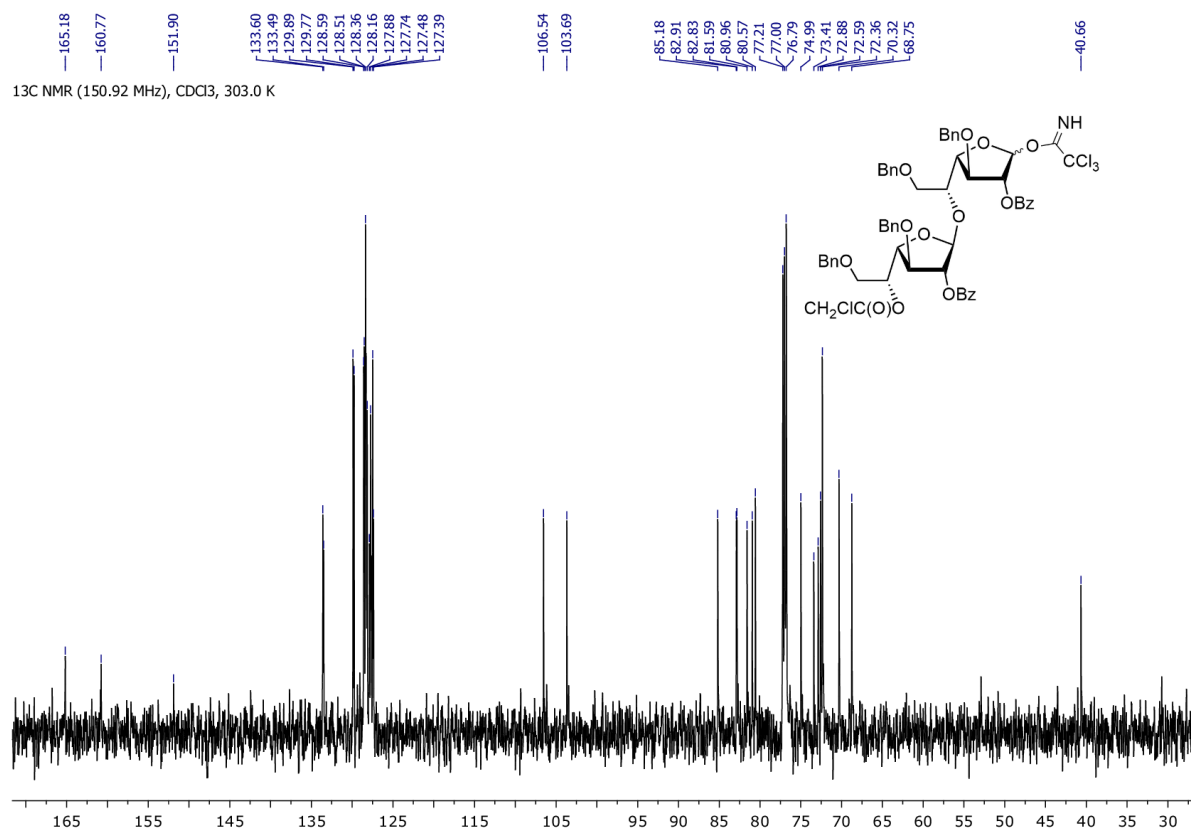
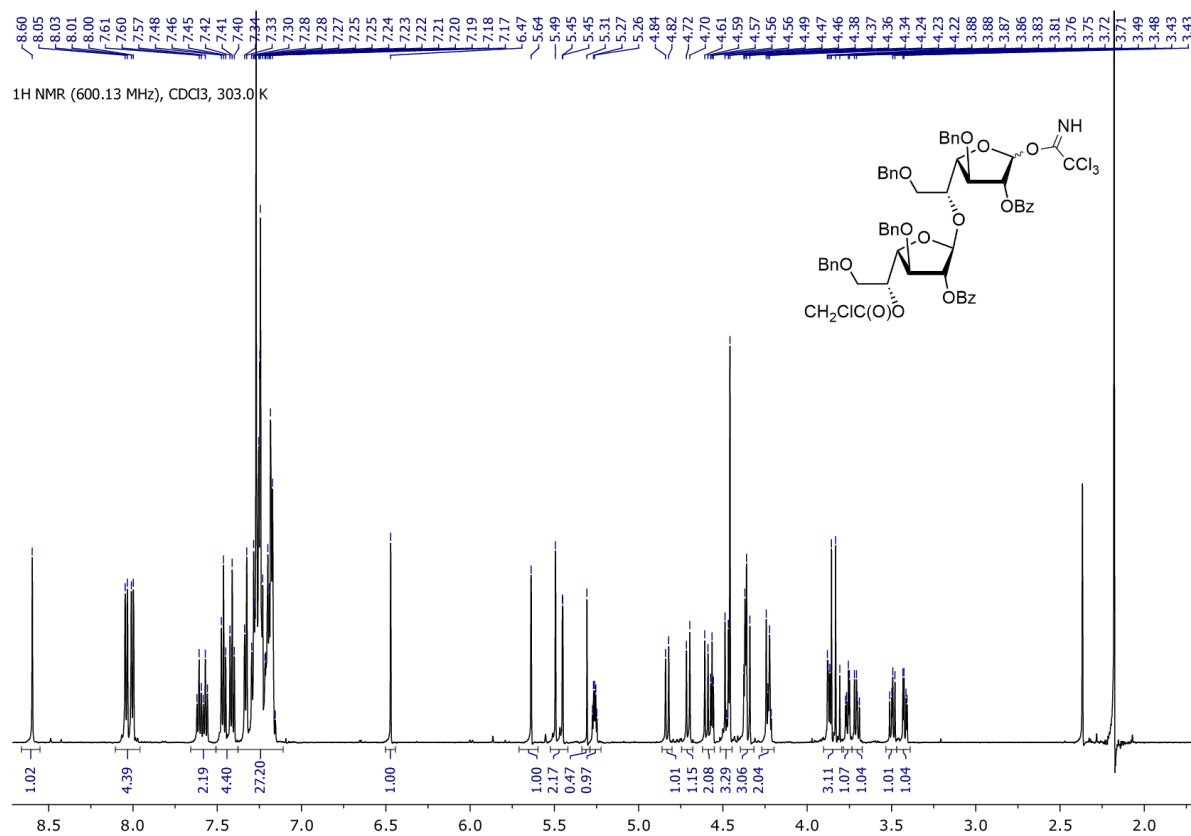
Calculated for $[M+Na]^+$

C52H55ClO13, M+nNa, 945.32

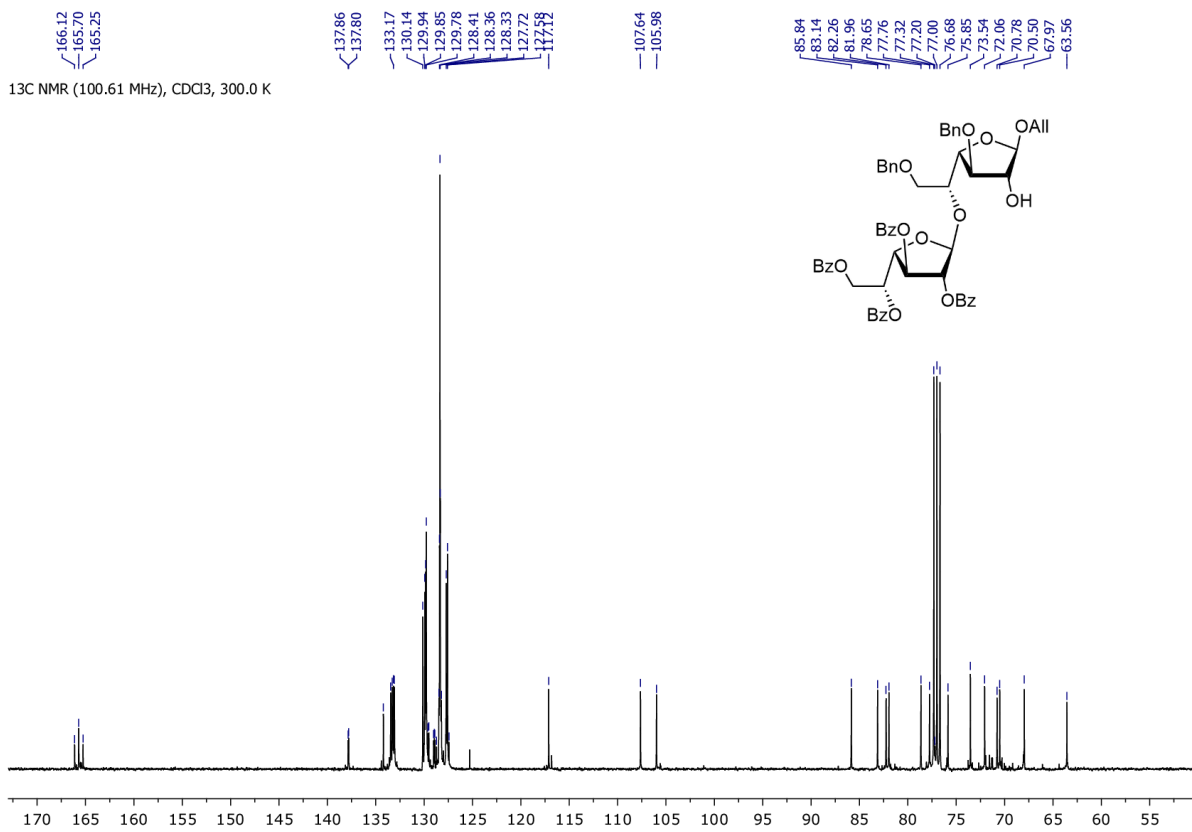
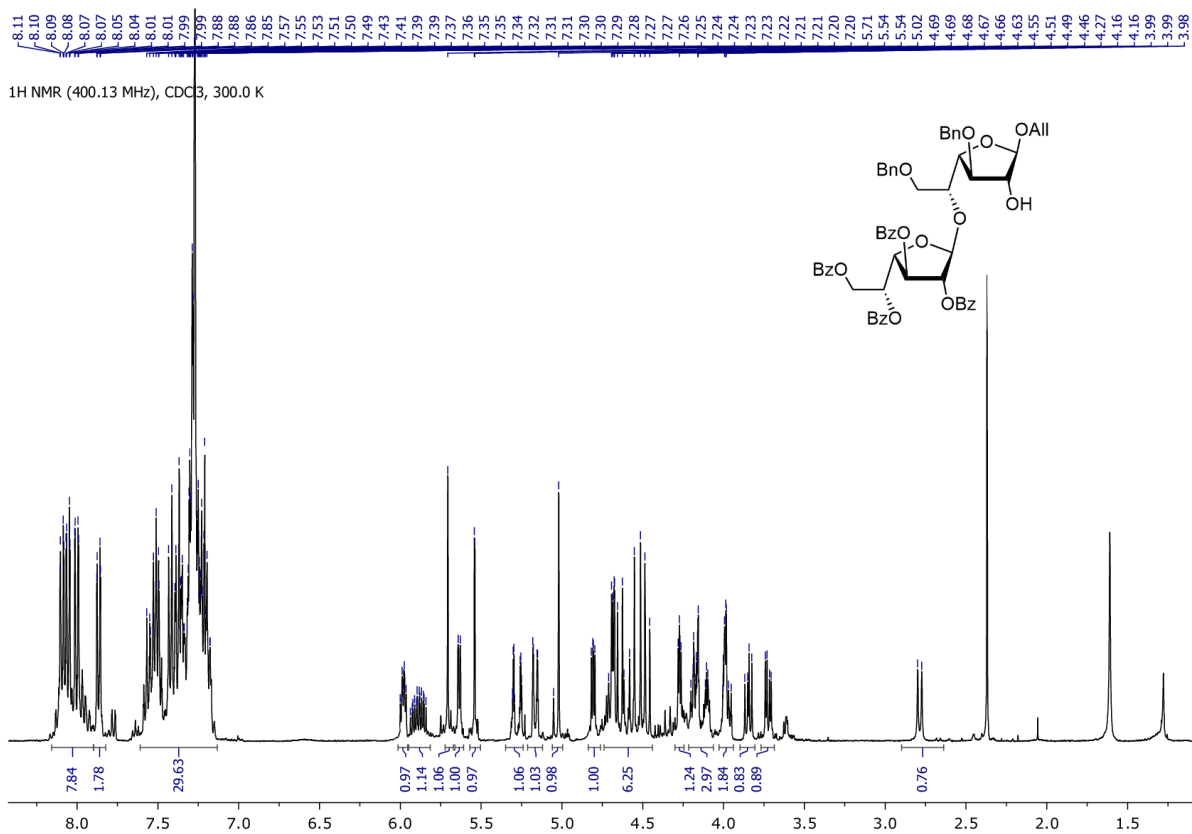
Calculated for $[M+K]^+$

C52H55ClO13, M+nK, 961.30

2-O-benzoyl-3,6-di-O-benzyl-5-O-chloroacetyl-β-D-galactofuranosyl-(1→5)-2-O-benzoyl-3,6-di-O-benzyl-β-D-galactofuranoside trichloroacetimidate (5)

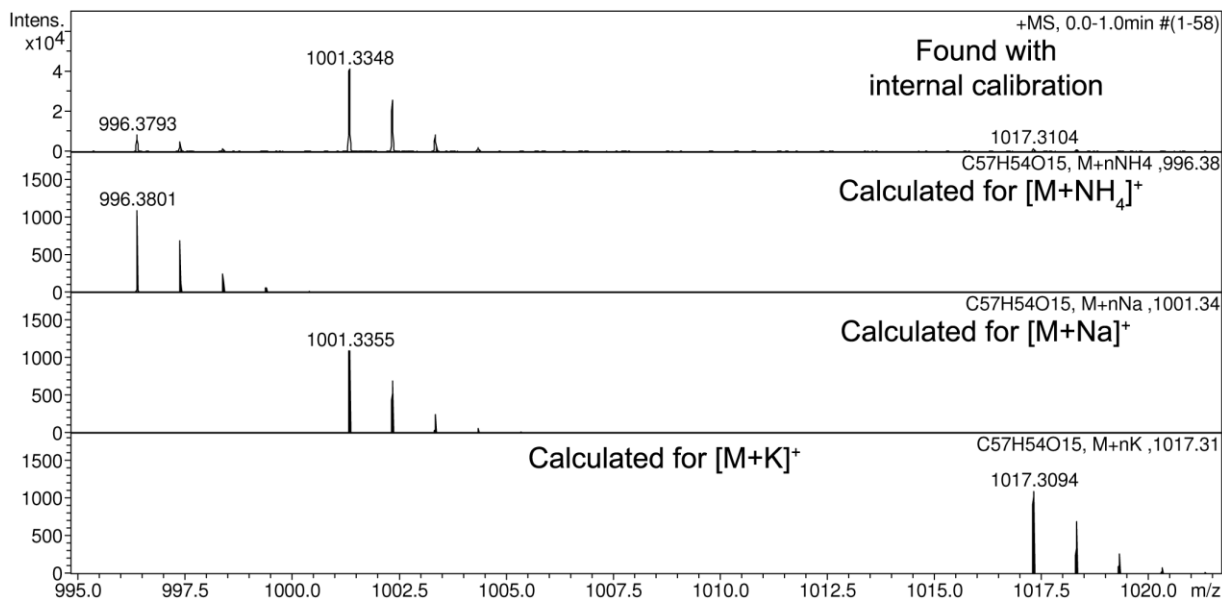
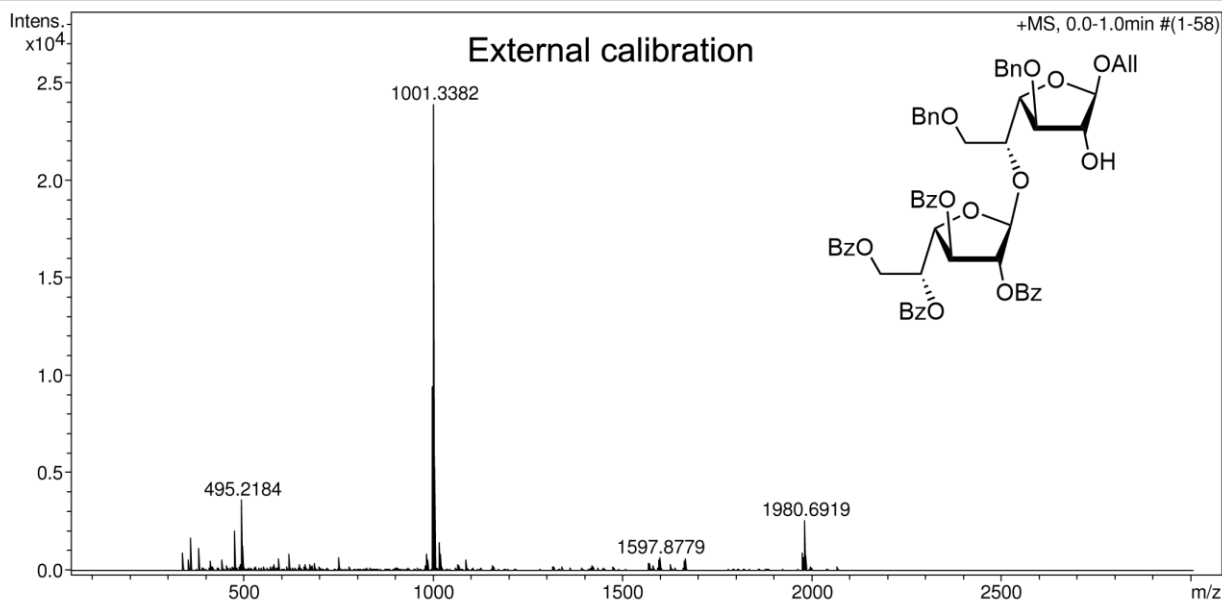


Allyl 2,3,5,6-tetra-O-benzoyl-β-D-galactofuranosyl-(1→5)-3,6-di-O-benzyl-β-D-galactofuranoside (21)

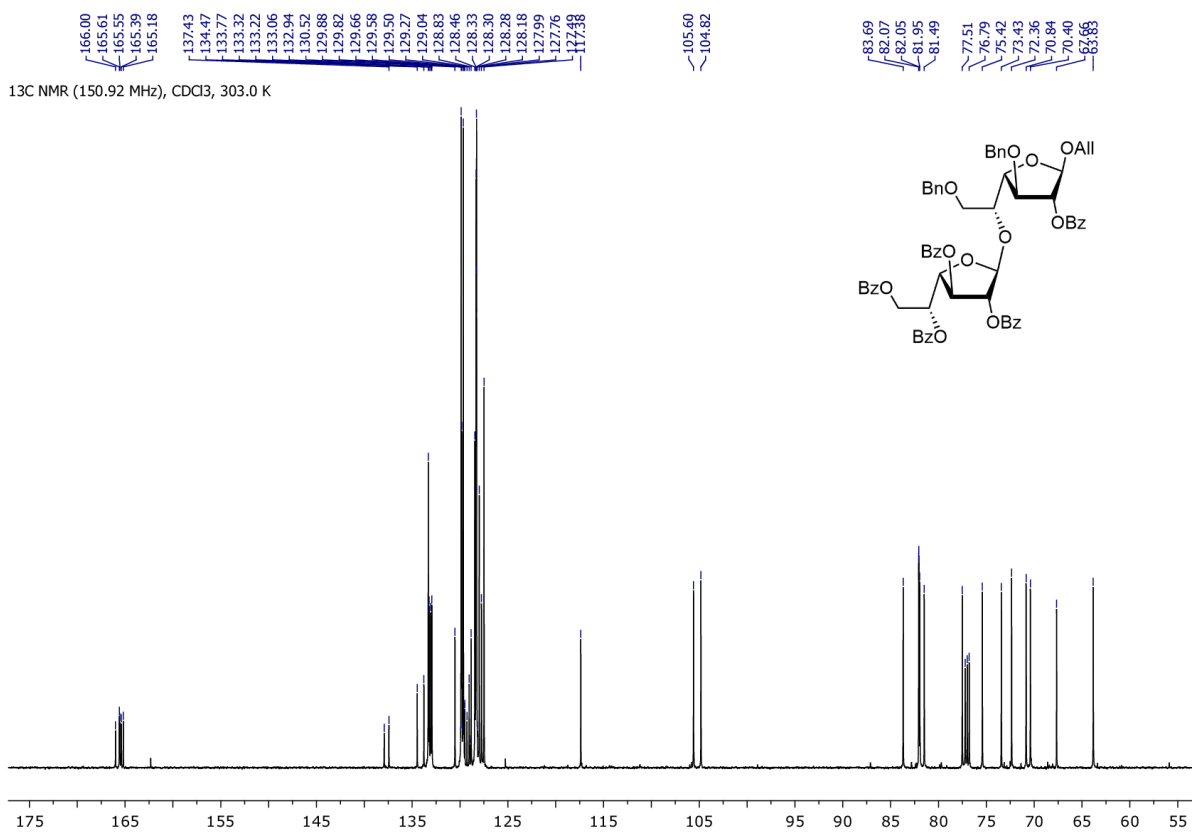
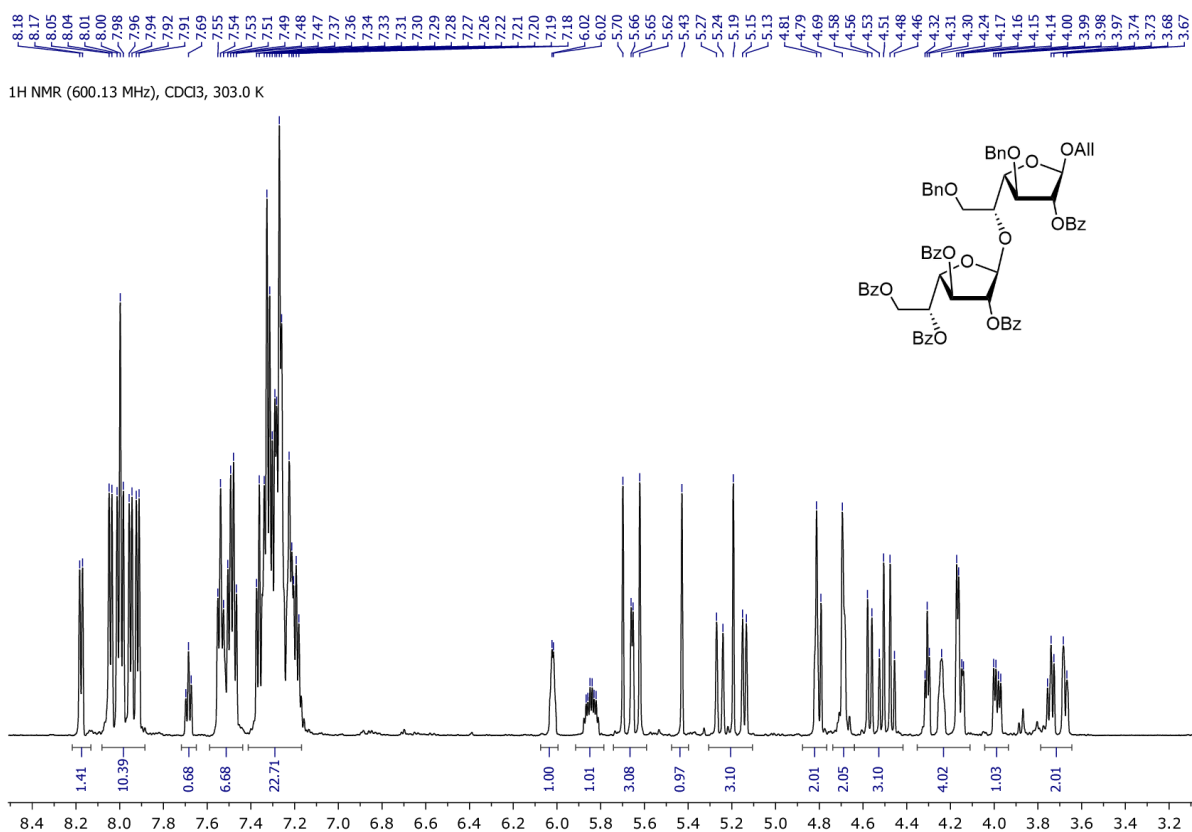


Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	1200.0 Vpp	Set Divert Valve	Waste

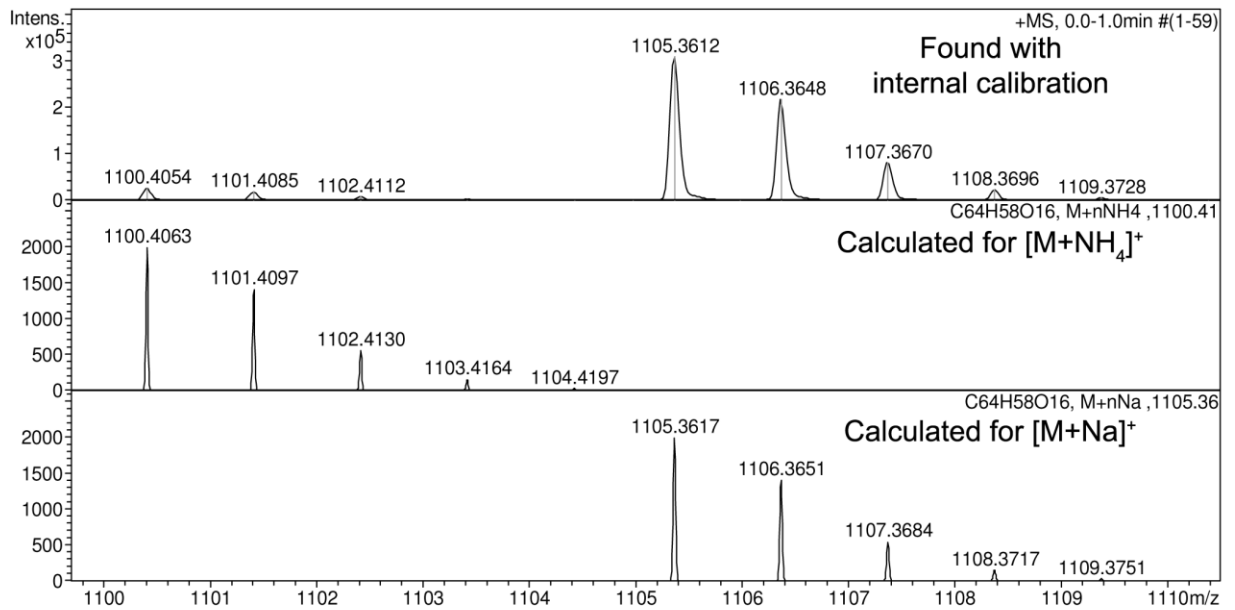
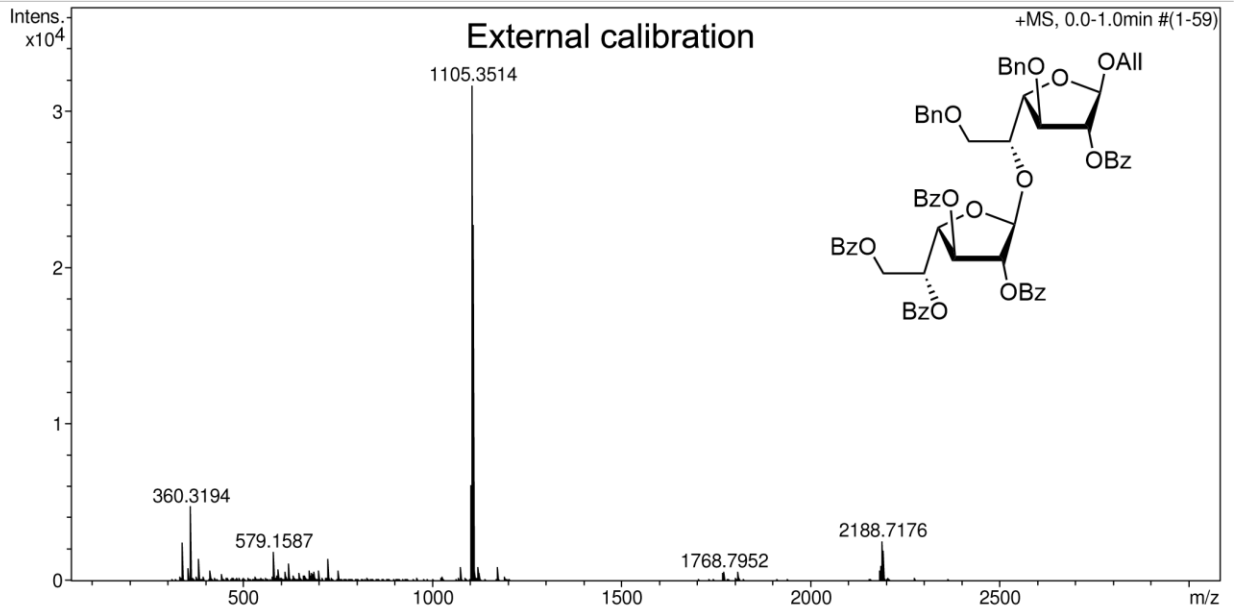


Allyl 2,3,5,6-tetra-O-benzoyl-β-D-galactofuranosyl-(1→5)-2-O-benzoyl-3,6-di-O-benzyl-β-D-galactofuranoside (22)

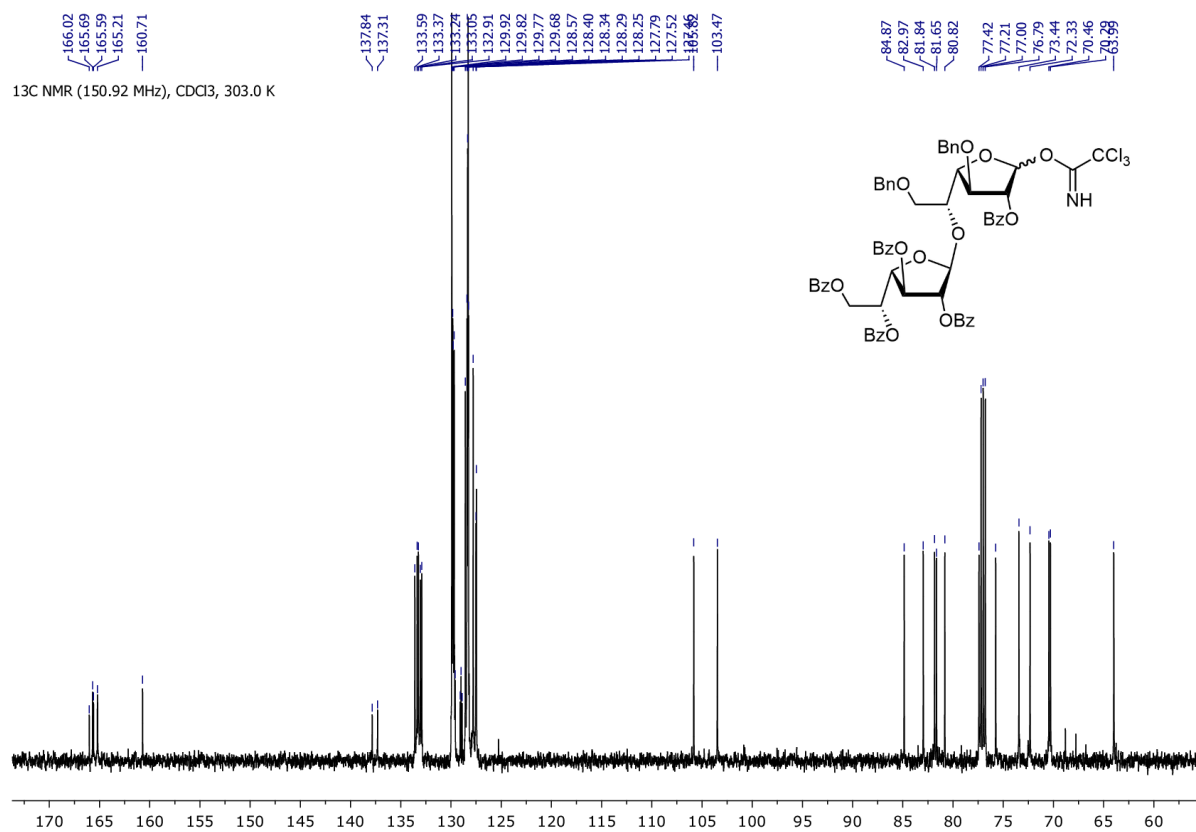
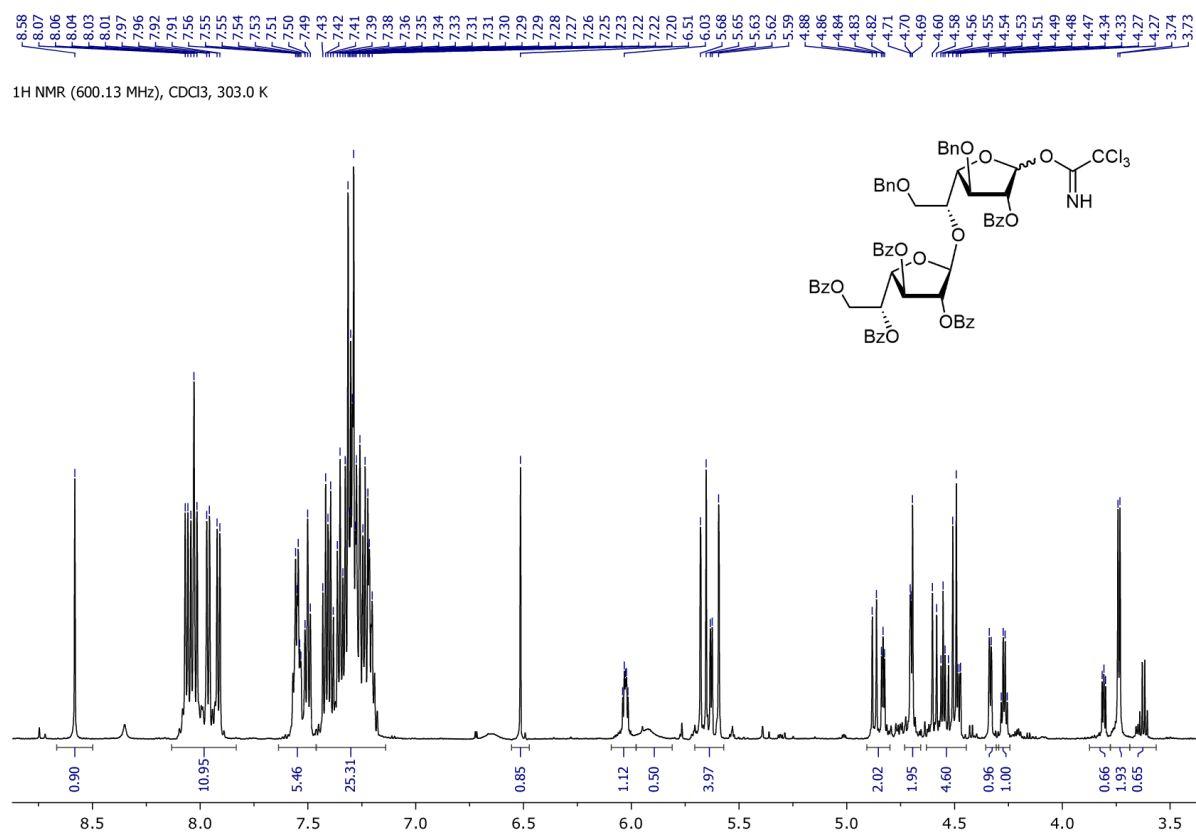


Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

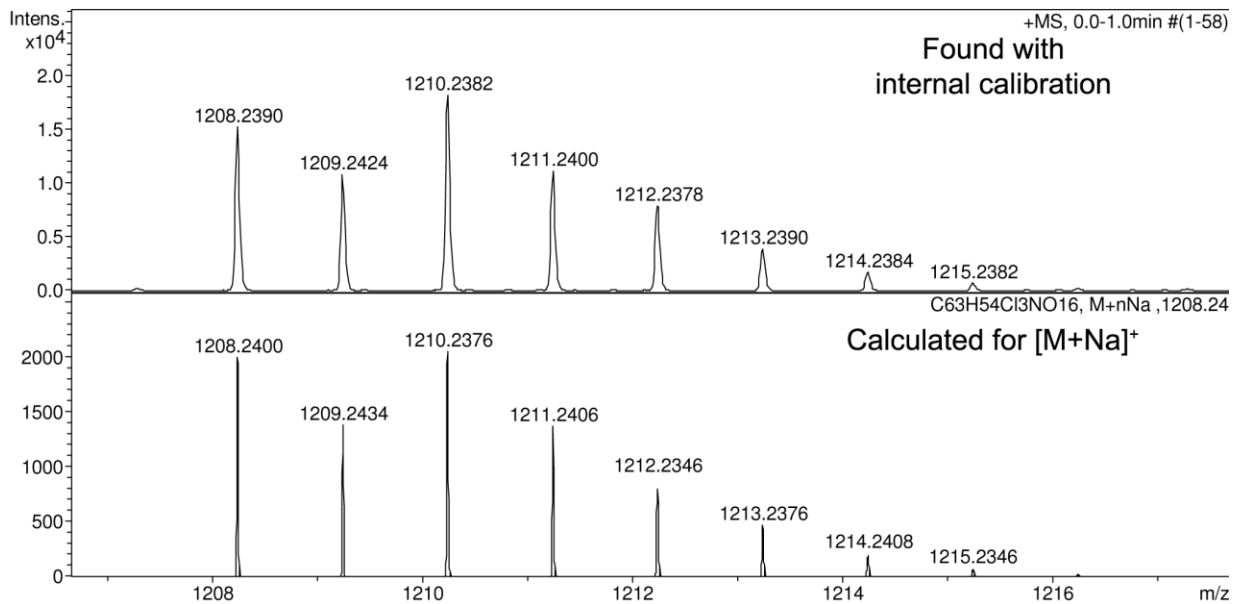
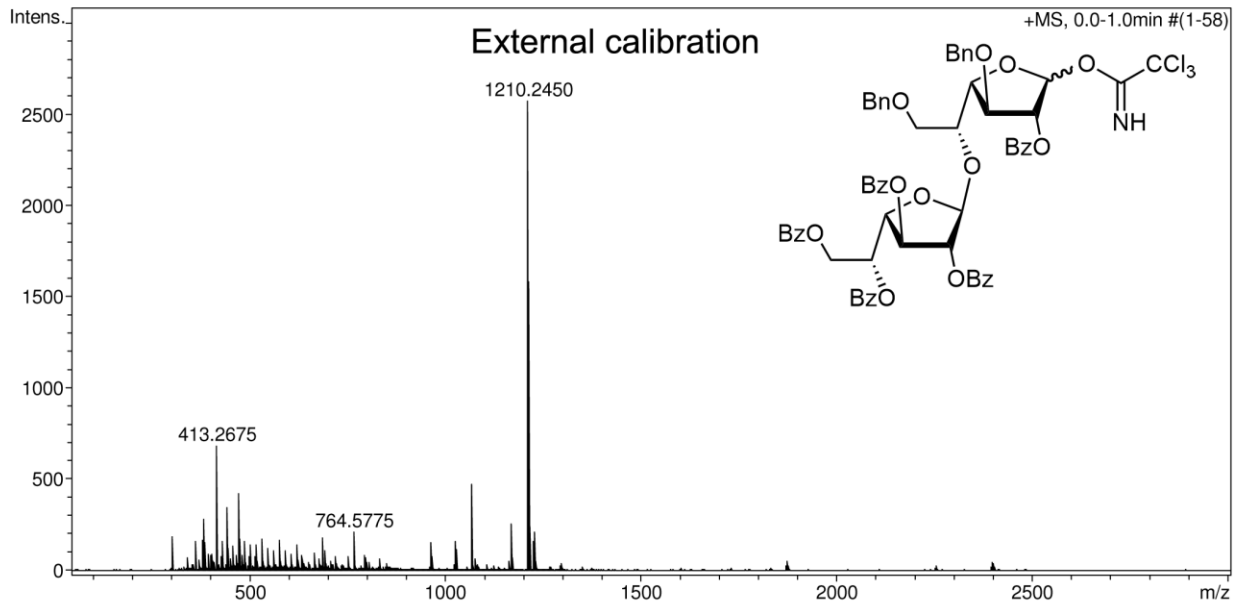


2,3,5,6-tetra-O-benzoyl-β-D-galactofuranosyl-(1→5)-2-O-benzoyl-3,6-di-O-benzyl-β-D-galactofuranoside trichloroacetimidate (6)



Acquisition Parameter

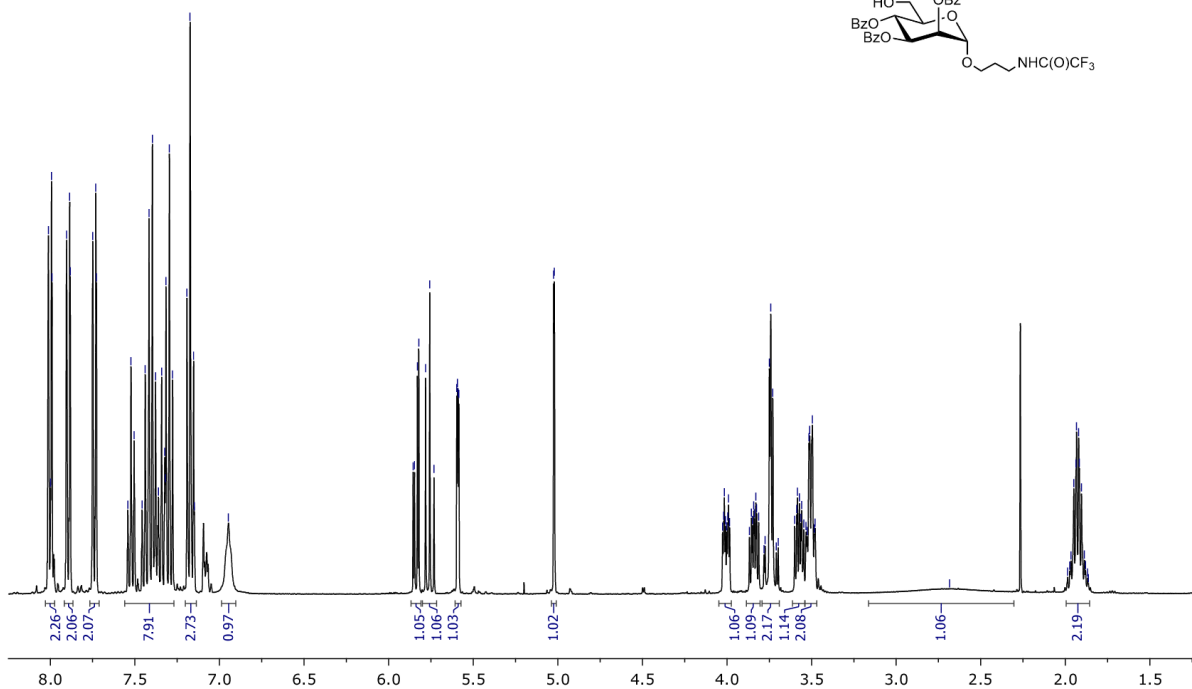
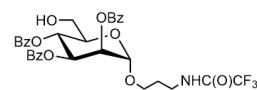
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	1200.0 Vpp	Set Divert Valve	Waste



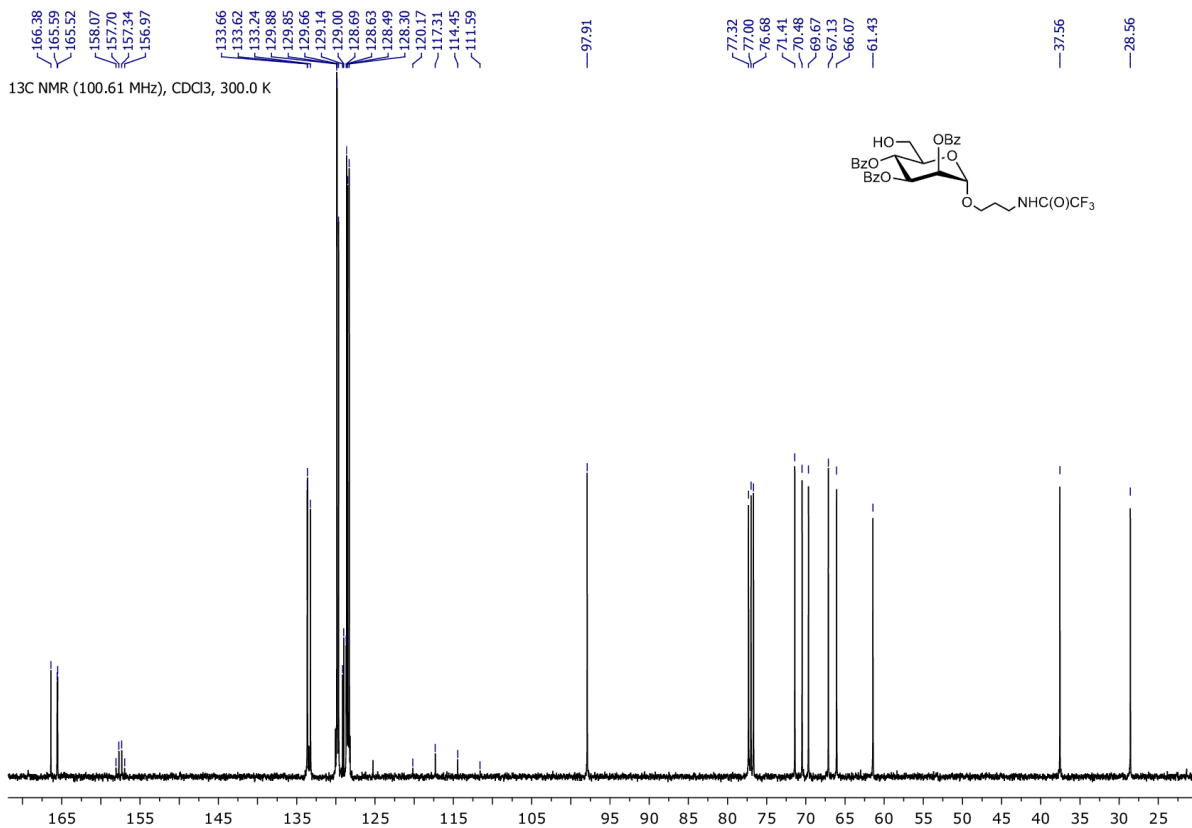
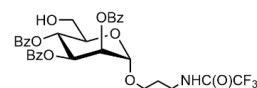
3-trifluoroacetamidopropyl 2,3,4-tri-O-benzoyl- α -D-mannopyranoside (3)



^1H NMR (400.13 MHz), CDCl_3 , 300.0 K

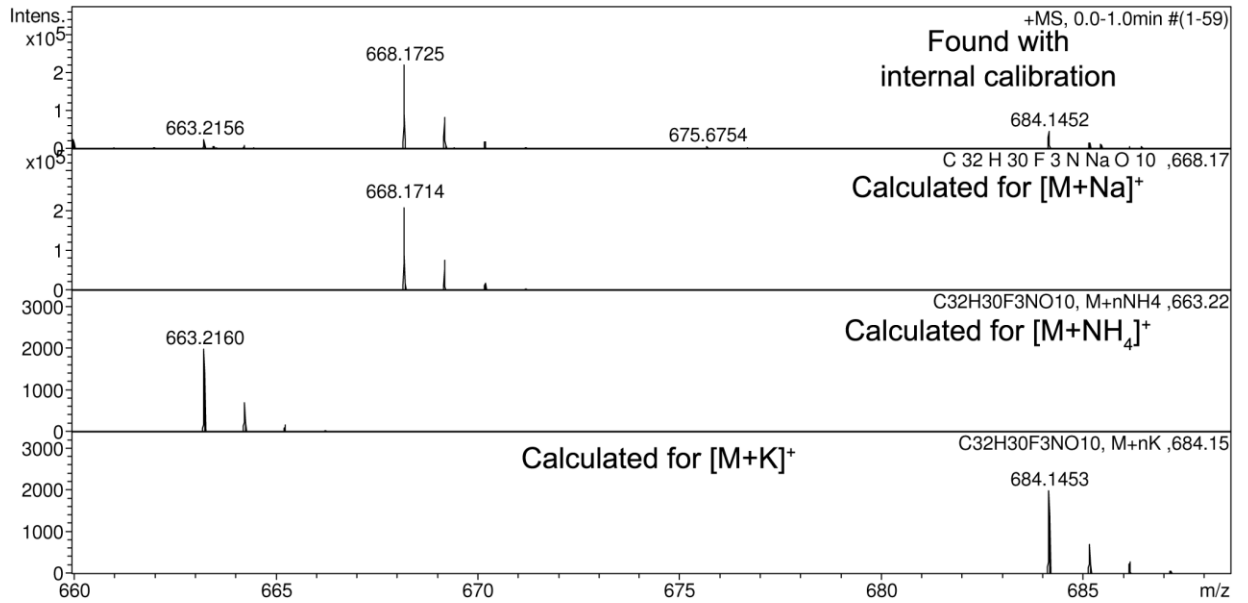
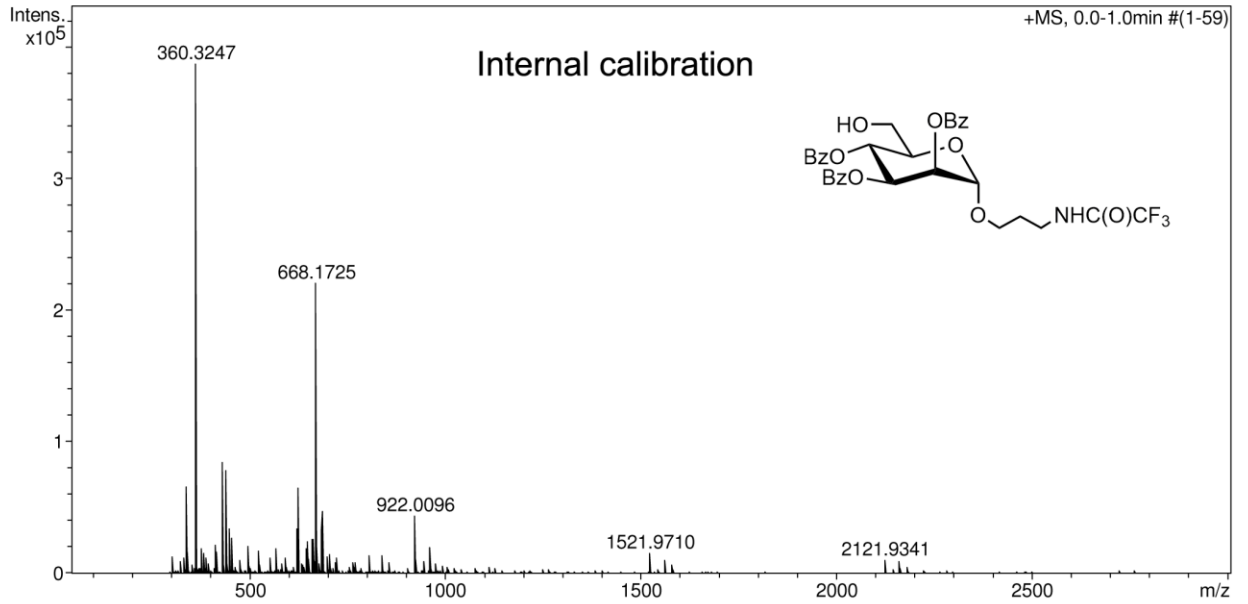


^{13}C NMR (100.61 MHz), CDCl_3 , 300.0 K



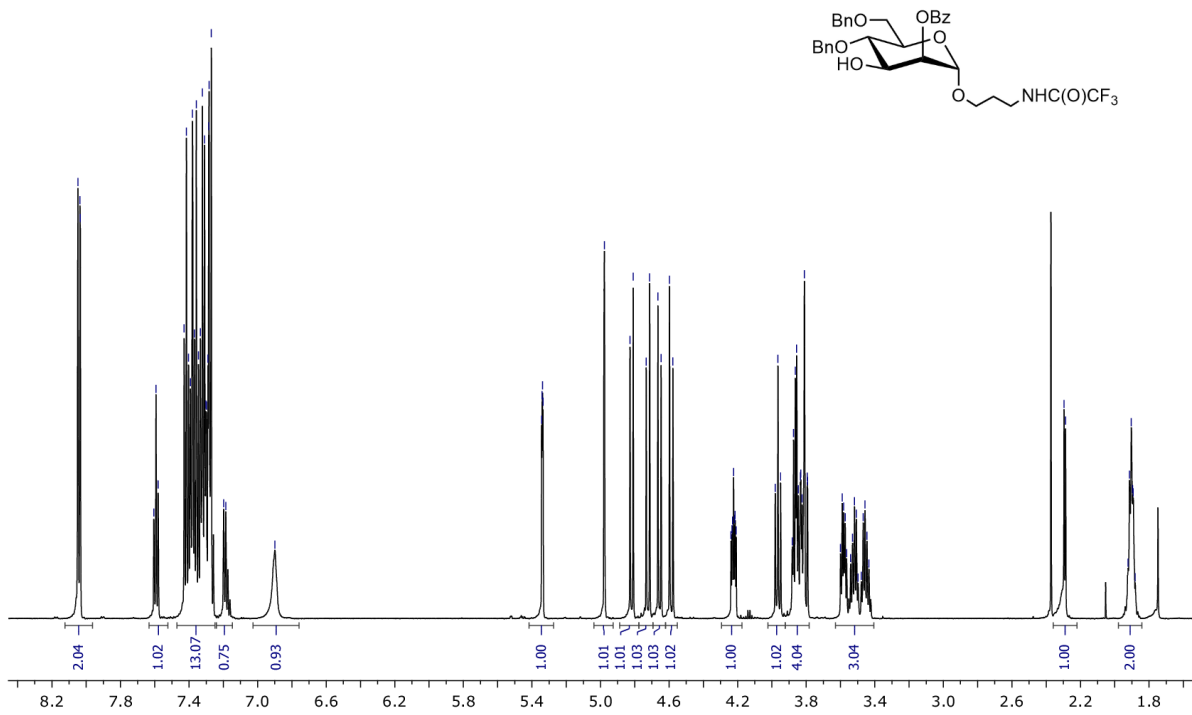
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	1200.0 Vpp	Set Divert Valve	Waste

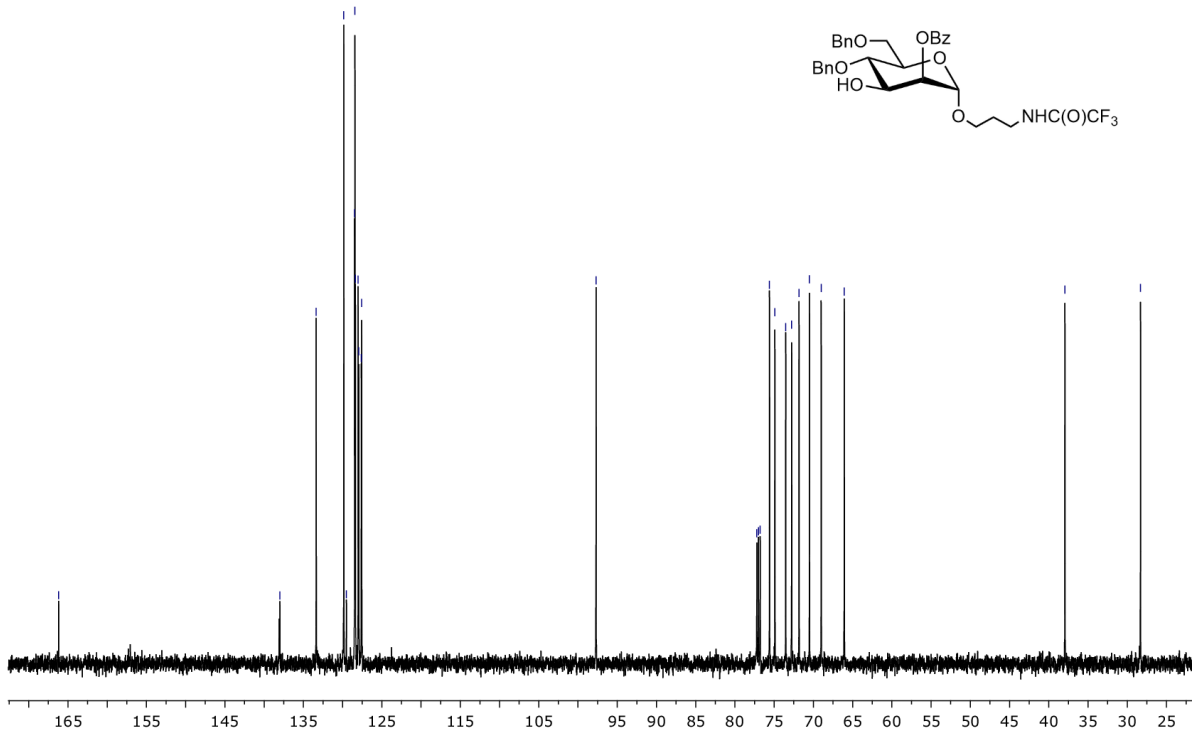


3-trifluoroacetamidopropyl 2-O-benzoyl-4,5-di-O-benzyl- α -D-mannopyranoside (4)

¹H NMR (600.13 MHz), CDCl₃, 303.0 K

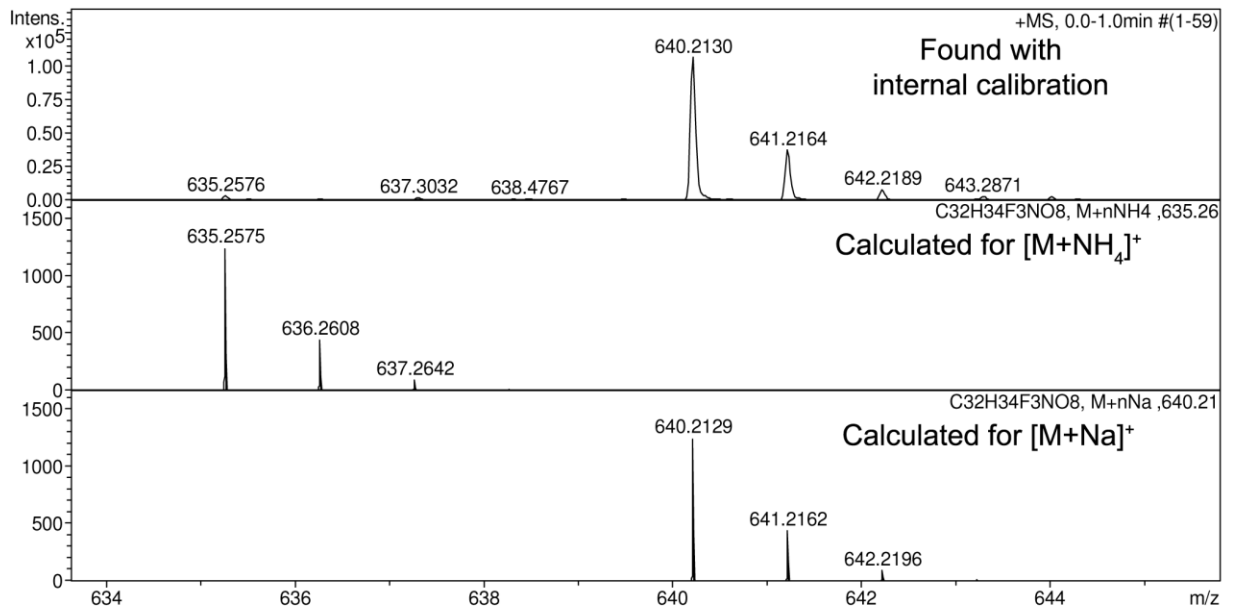
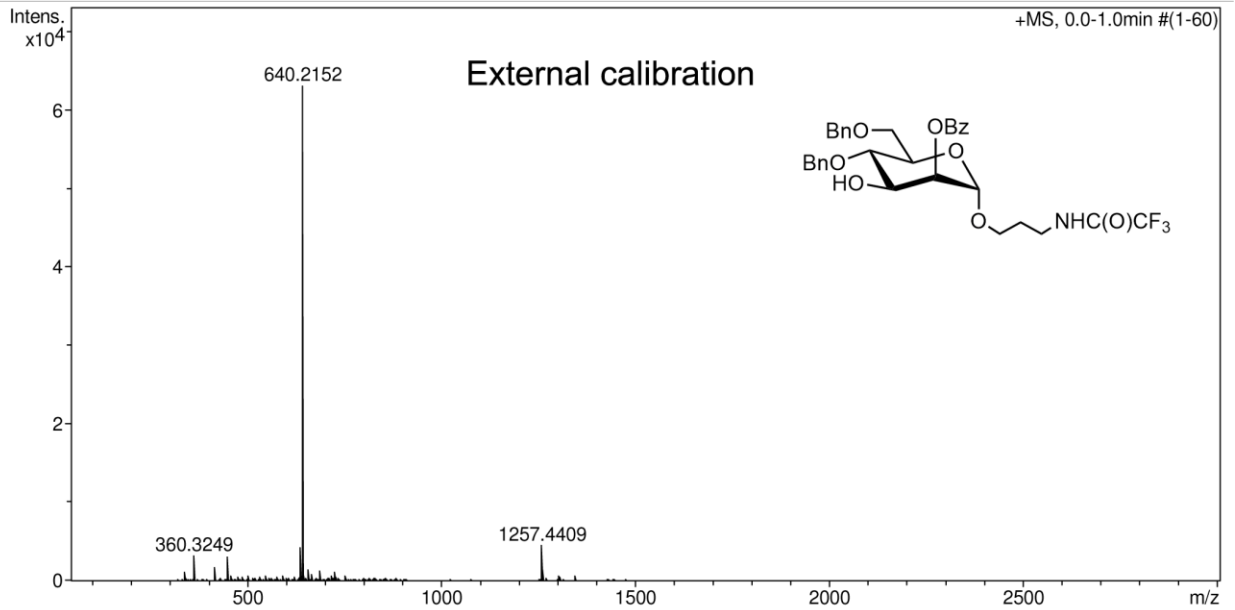


¹³C NMR (150.92 MHz), CDCl₃, 303.2 K



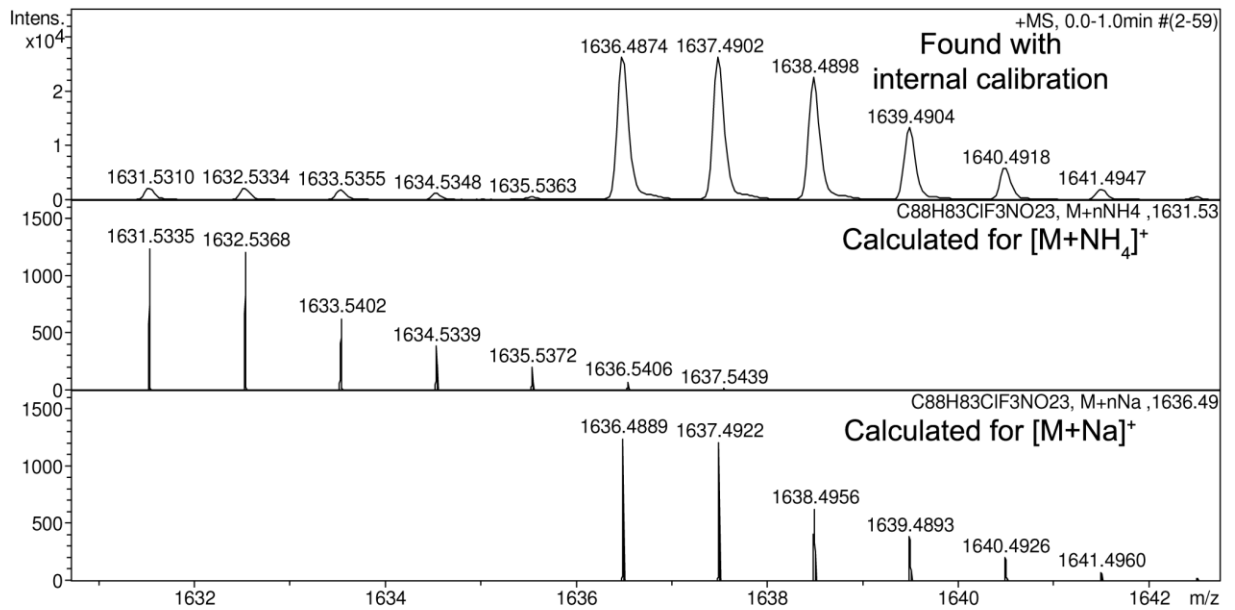
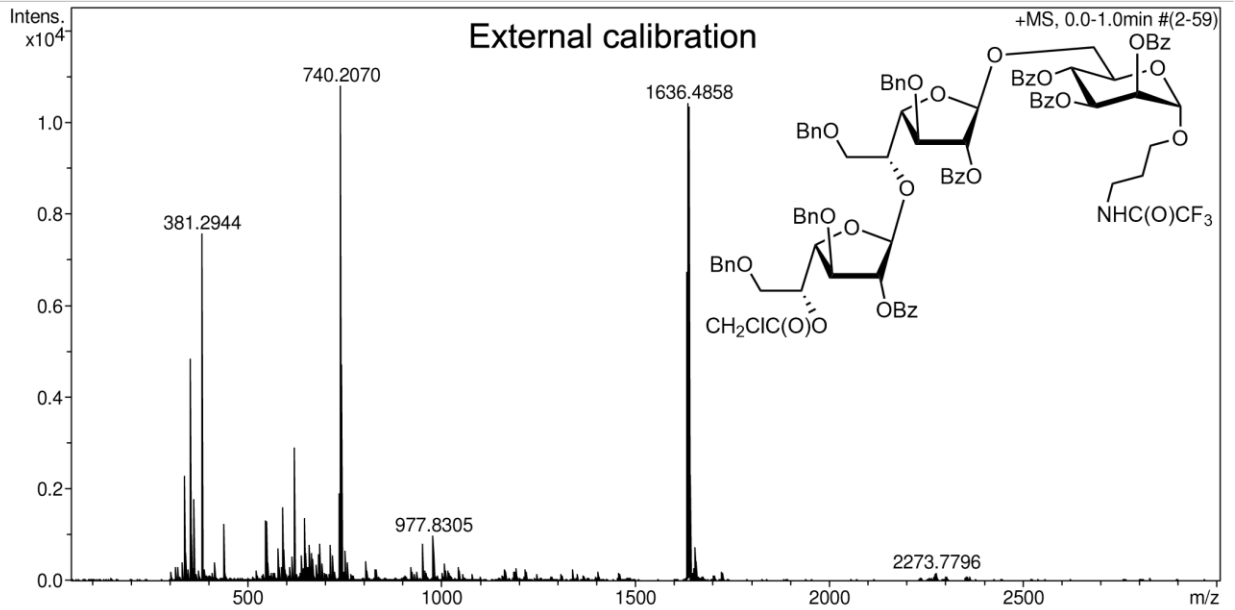
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

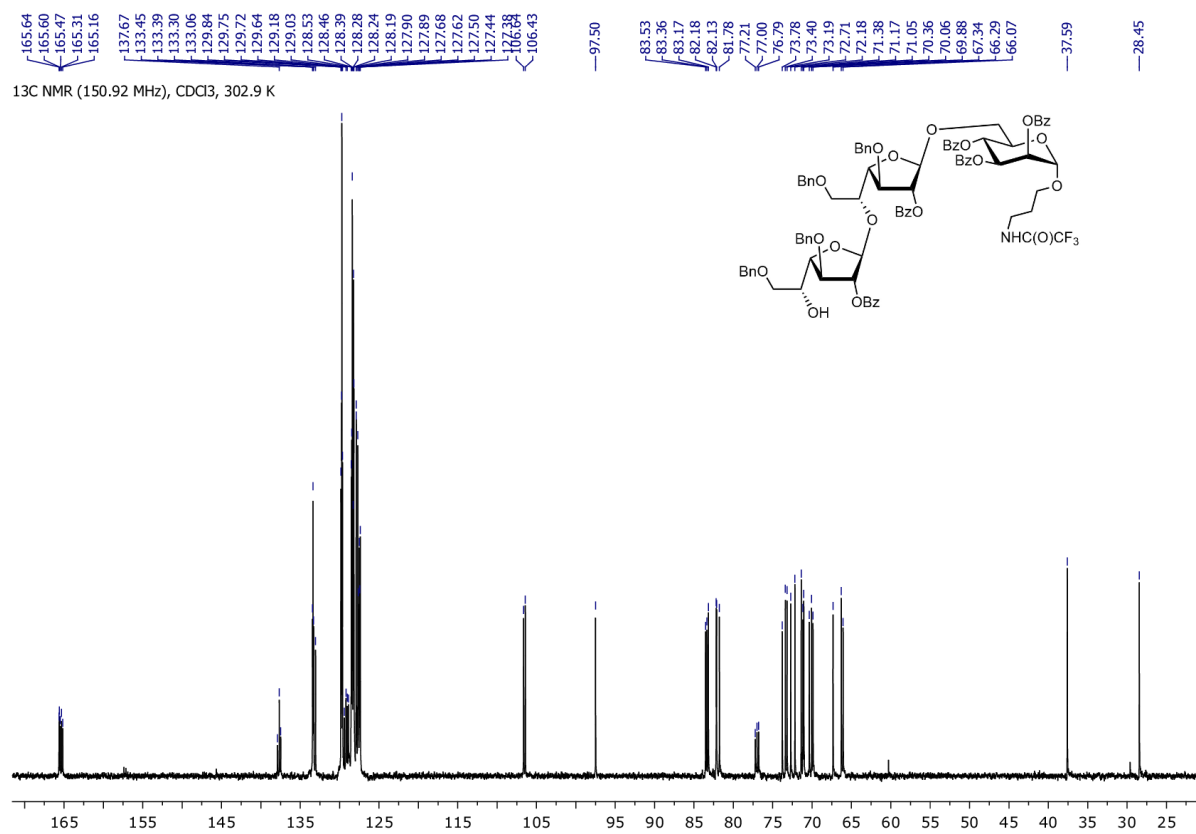
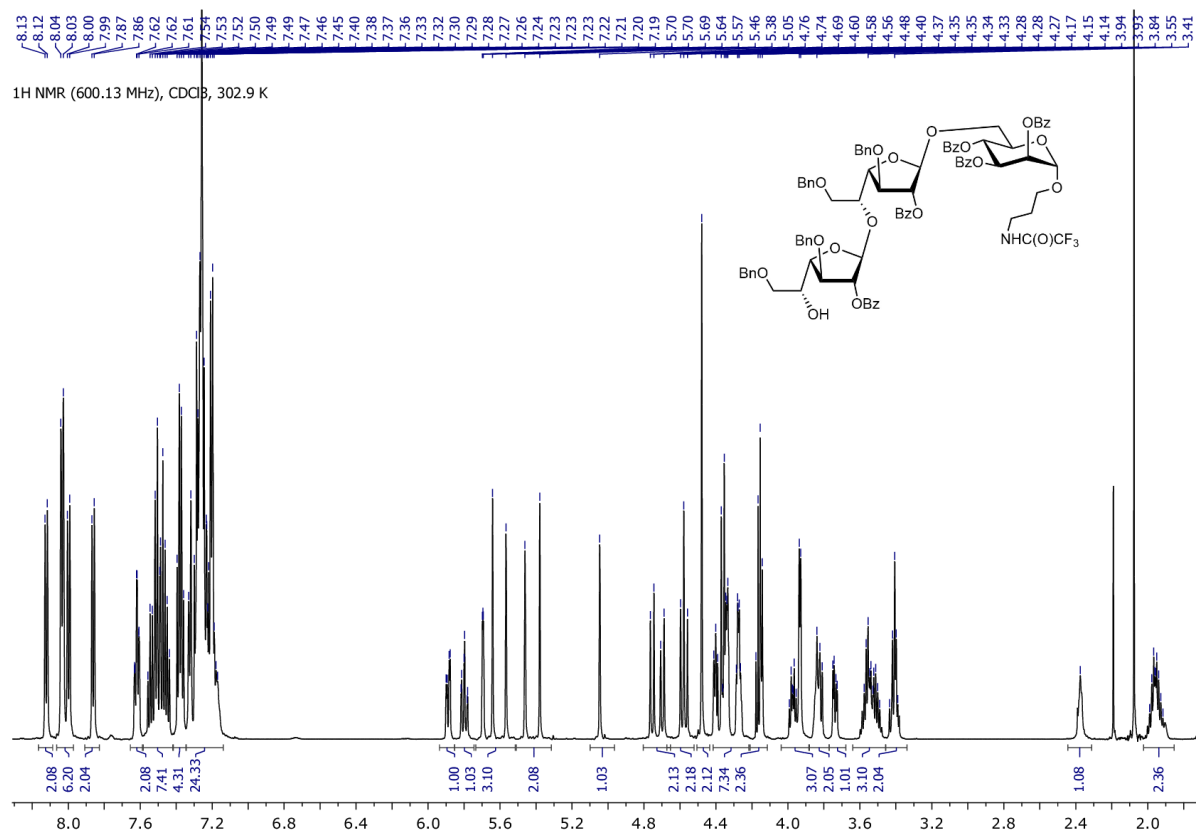


Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

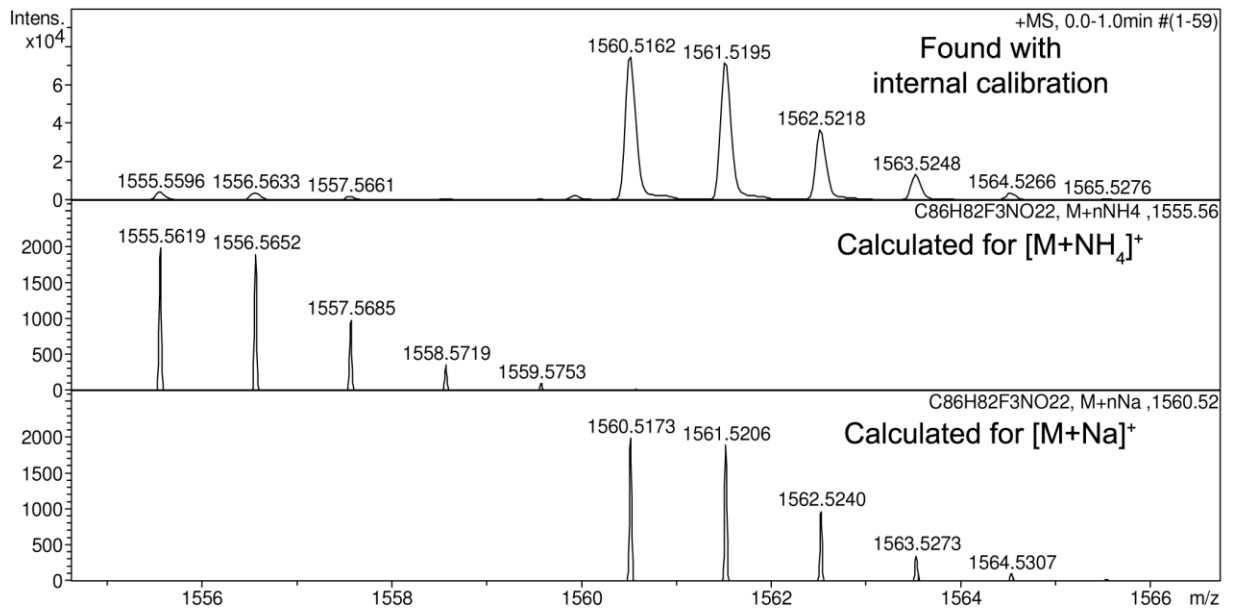
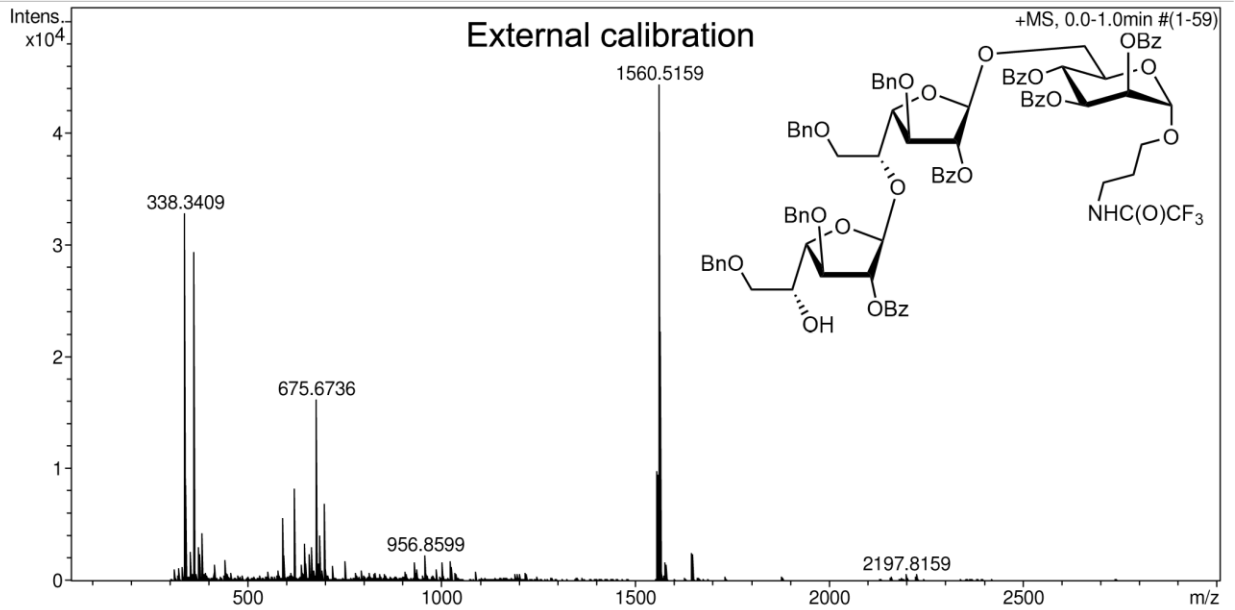


3-trifluoroacetamidopropyl 2-O-benzoyl-3,6-di-O-benzyl-β-D-galactofuranosyl-(1→5)-2-O-benzoyl-3,6-di-O-benzyl-β-D-galactofuranosyl-(1→6)-2,3,4-tri-O-benzoyl-α-D-mannopyranoside (26)

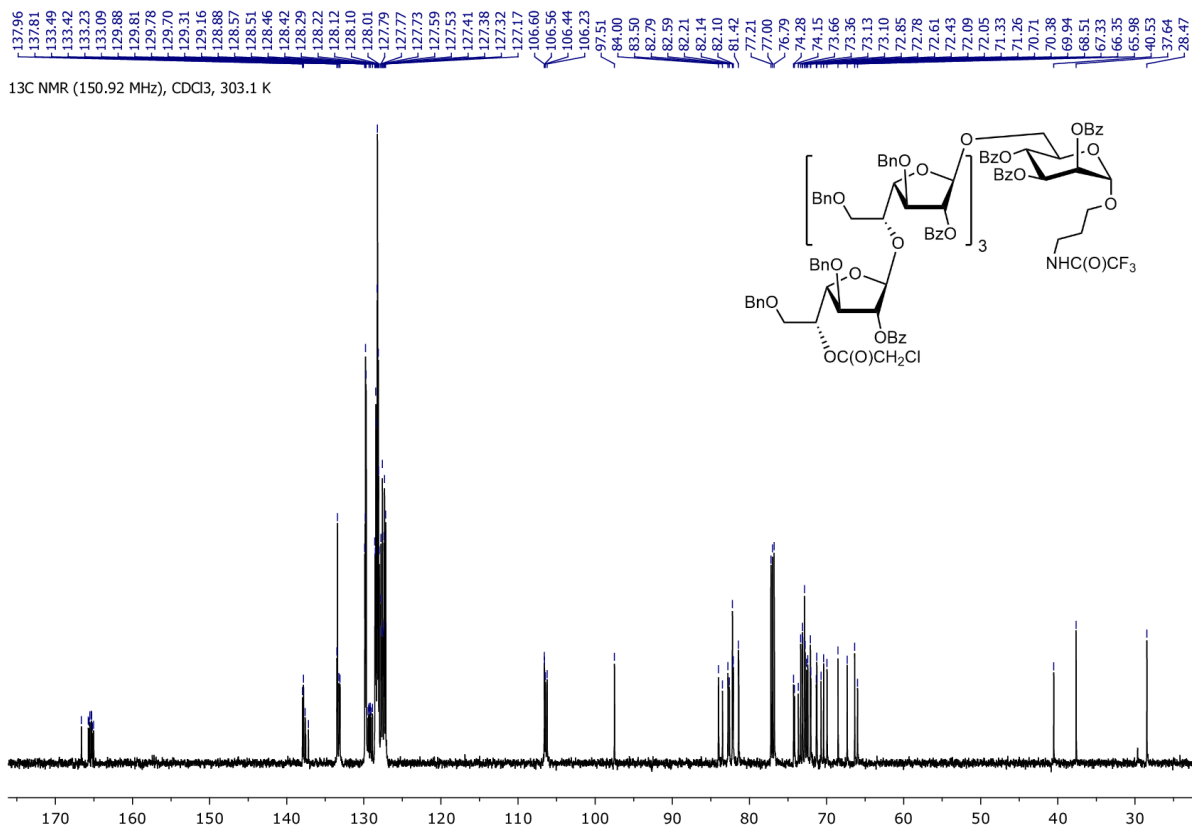
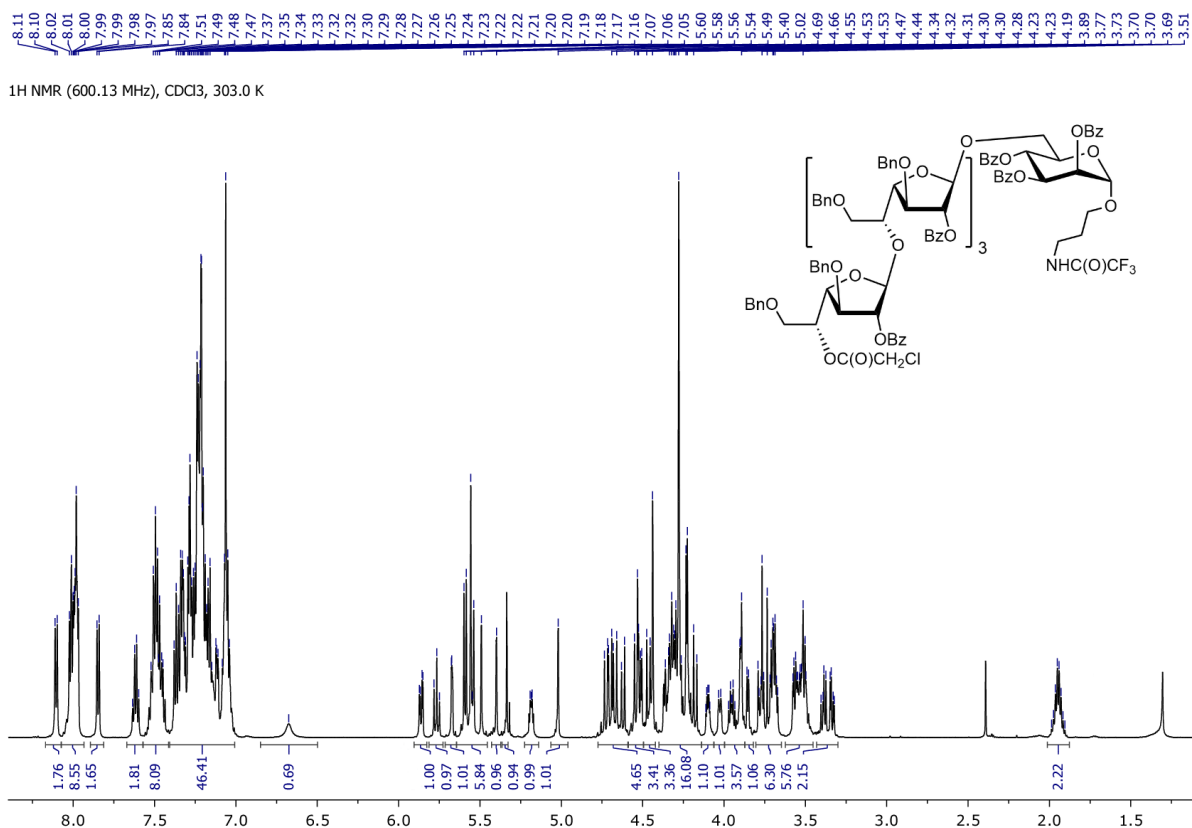


Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

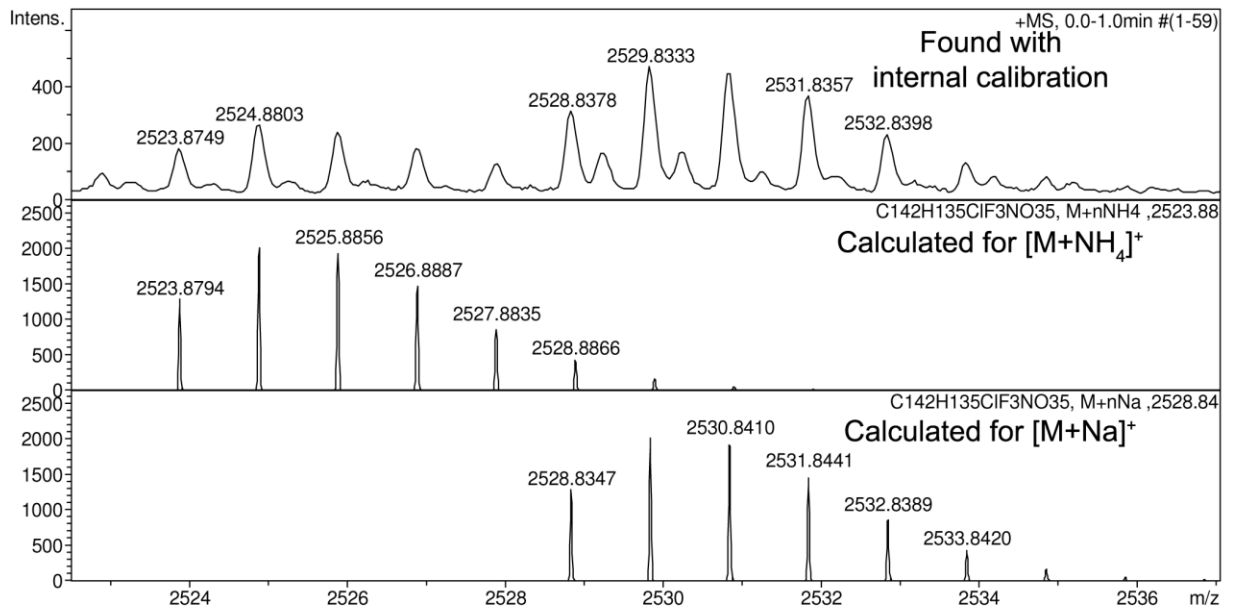
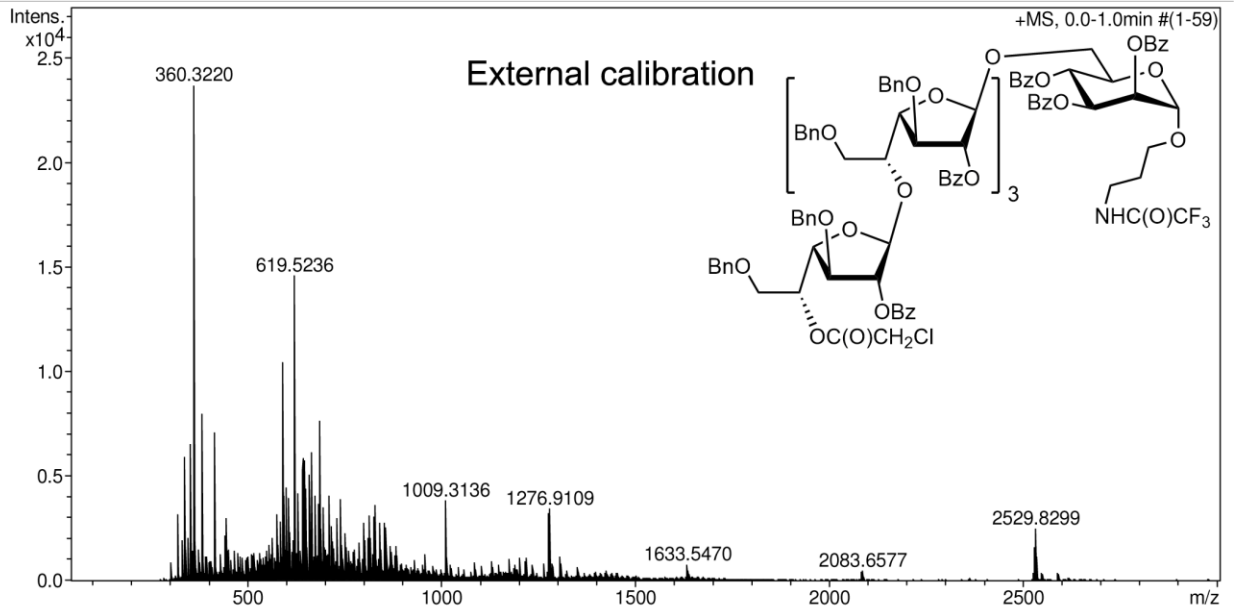


3-trifluoroacetamidopropyl 2-O-benzoyl-3,6-di-O-benzyl-5-O-chloroacetyl- β -D-galactofuranosyl-(1 \rightarrow 5)-2-O-benzoyl-3,6-di-O-benzyl- β -D-galactofuranosyl-(1 \rightarrow 5)-2-O-benzoyl-3,6-di-O-benzyl- β -D-galactofuranosyl-(1 \rightarrow 5)-2-O-benzoyl-3,6-di-O-benzyl- β -D-galactofuranosyl-(1 \rightarrow 6)-2,3,4-tri-O-benzoyl- α -D-mannopyranoside (27)

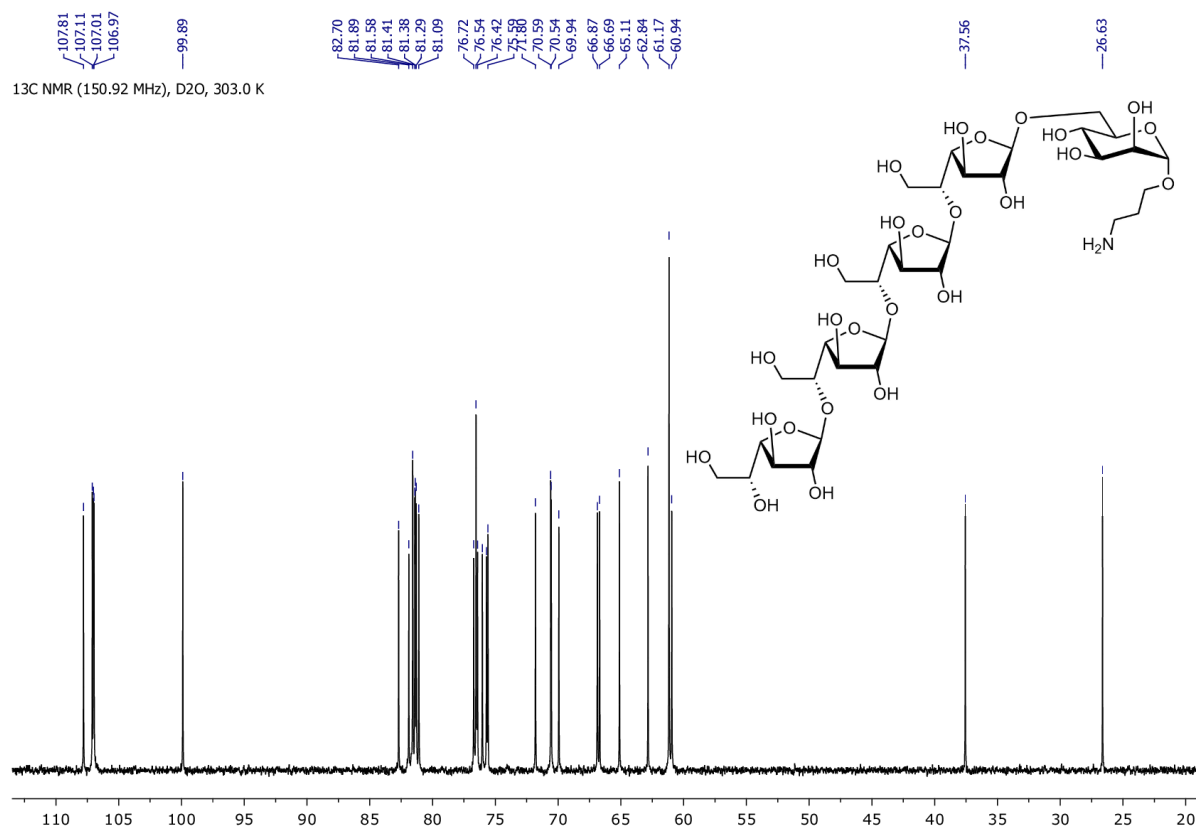
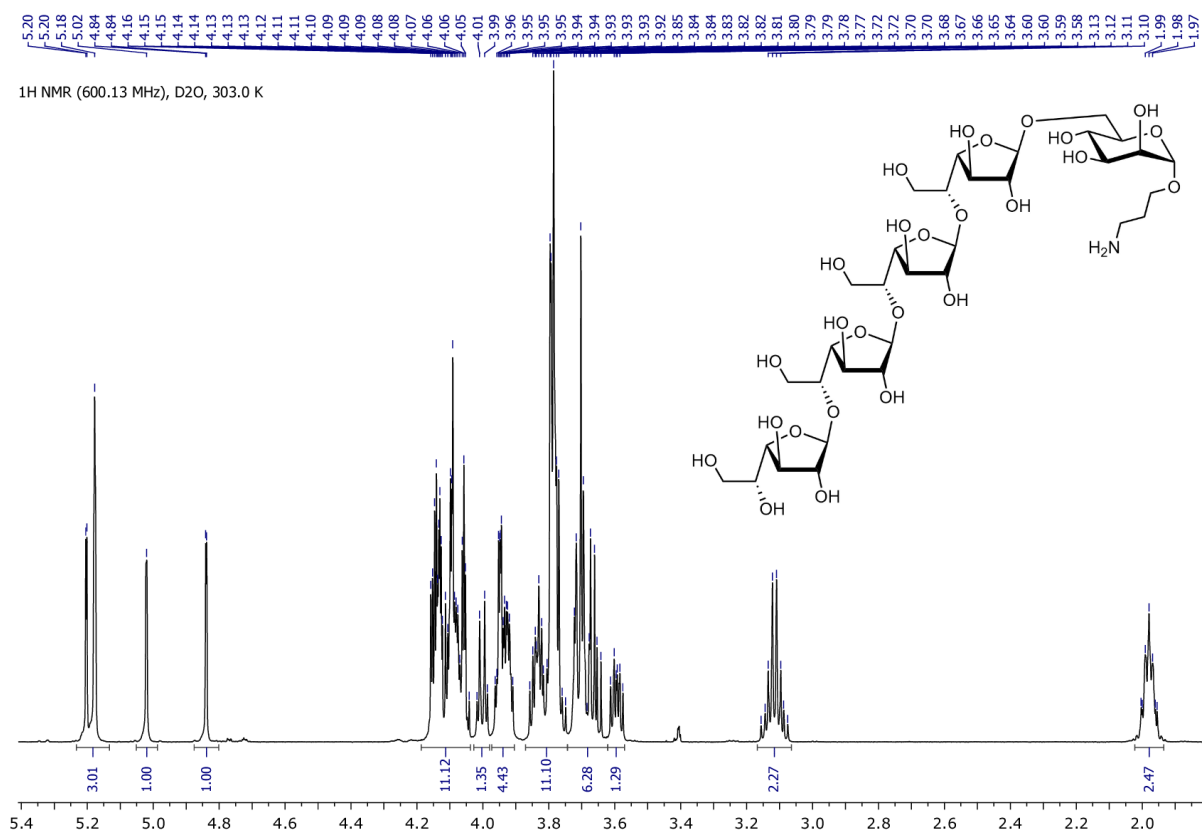


Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

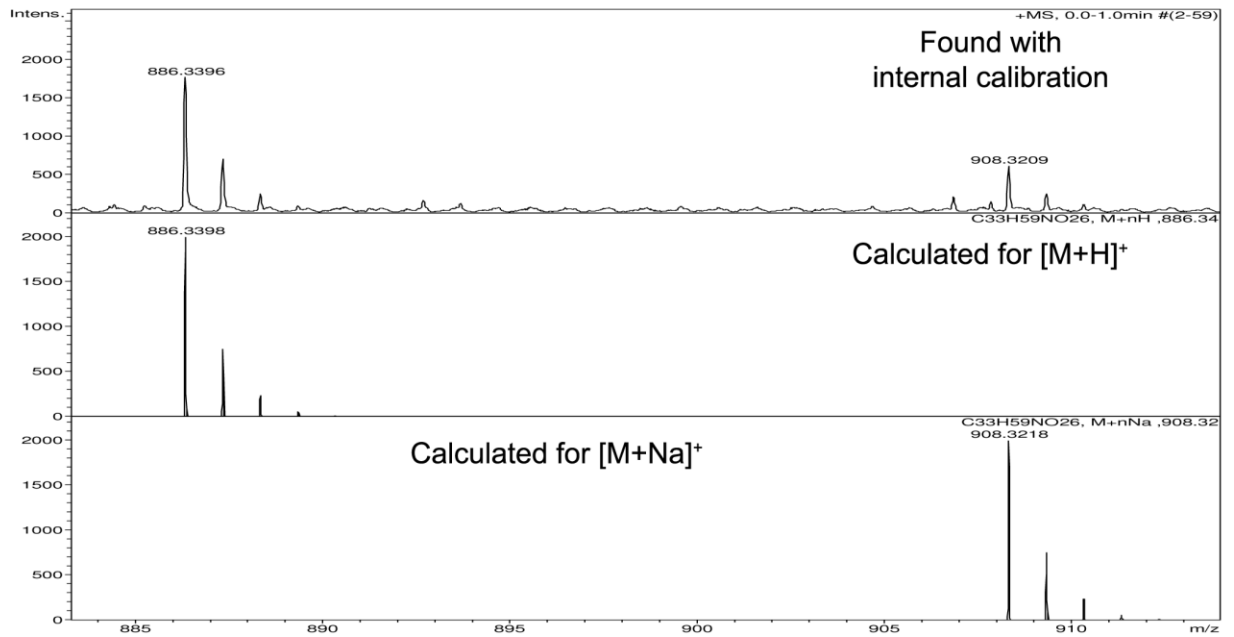
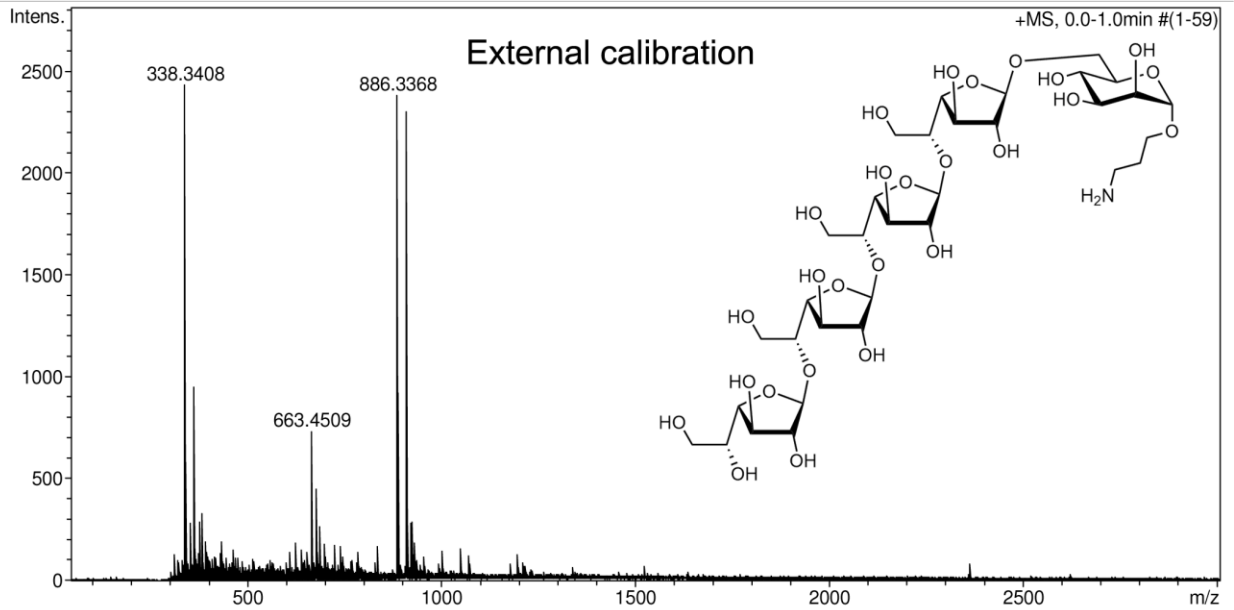


3-aminopropyl β -D-galactofuranosyl-(1 \rightarrow 5)- β -D-galactofuranosyl-(1 \rightarrow 5)- β -D-galactofuranosyl-(1 \rightarrow 5)- β -D-galactofuranosyl-(1 \rightarrow 6)- α -D-mannopyranoside (1)

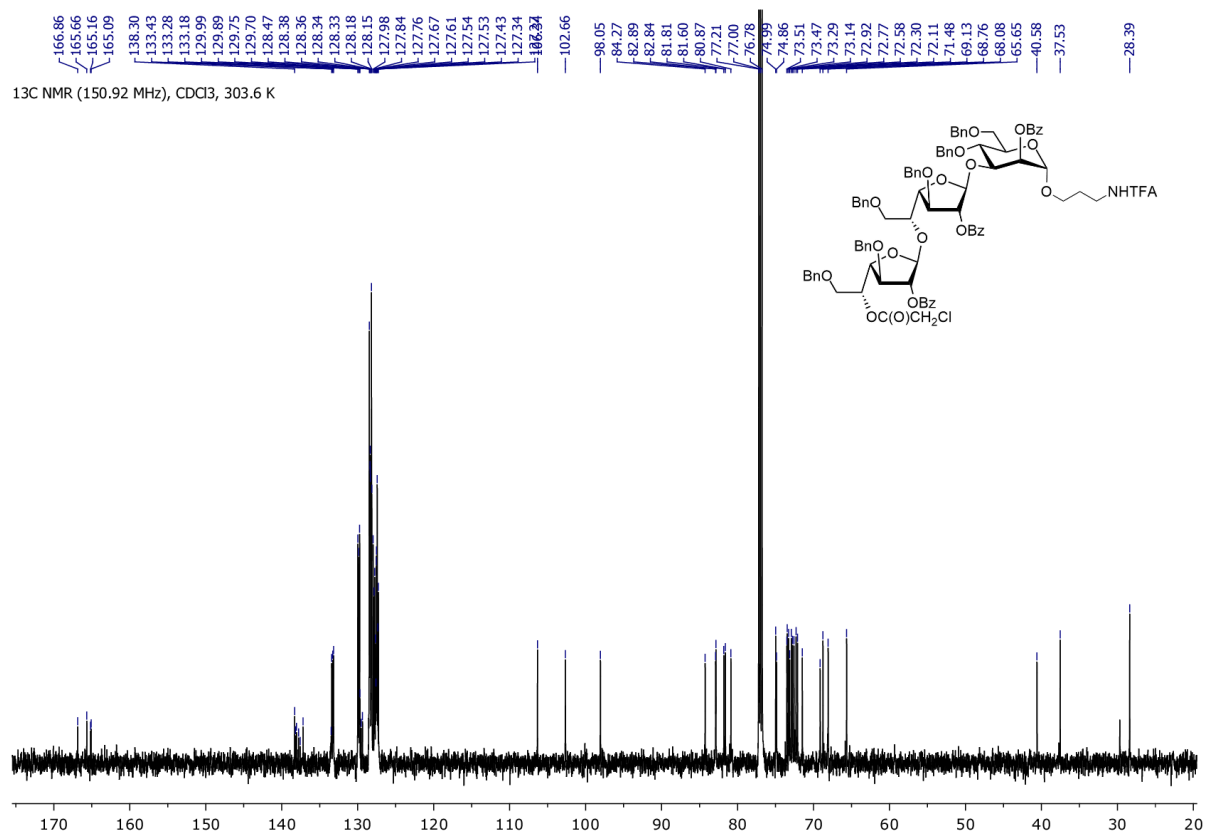
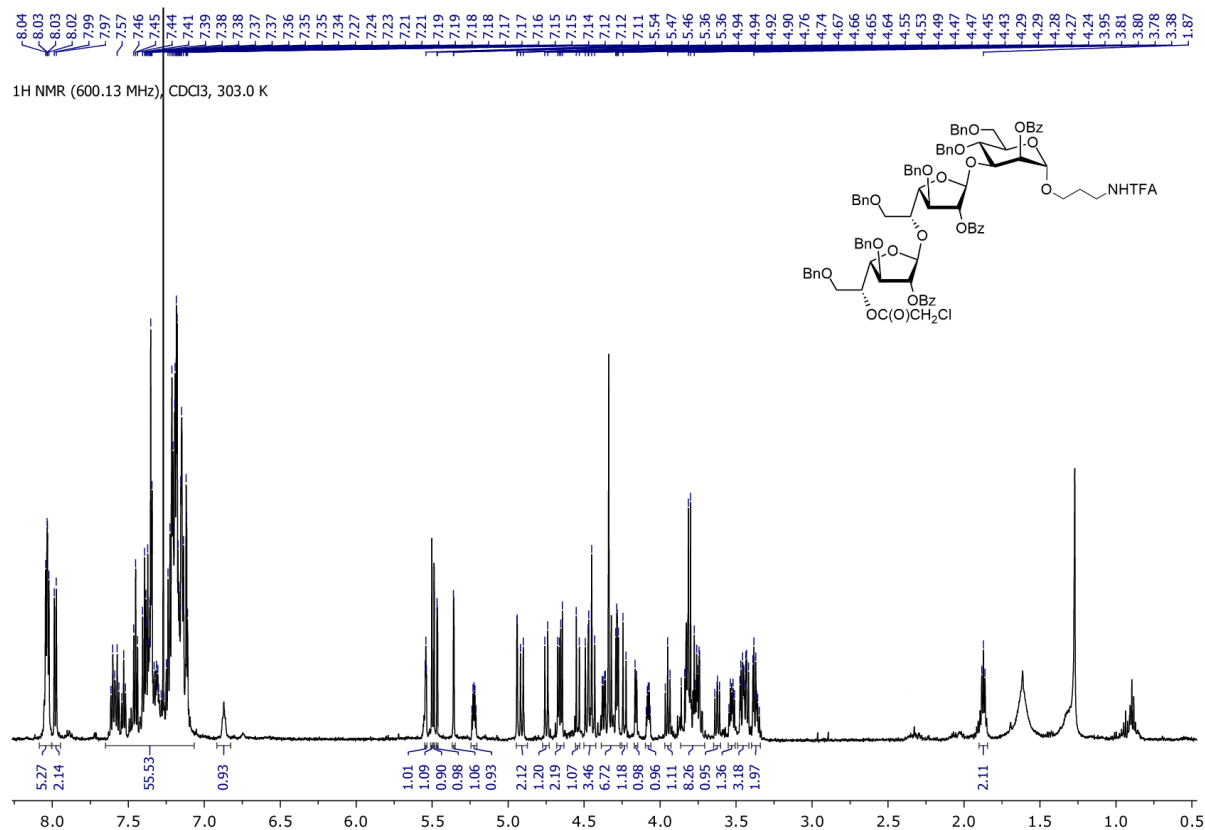


Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

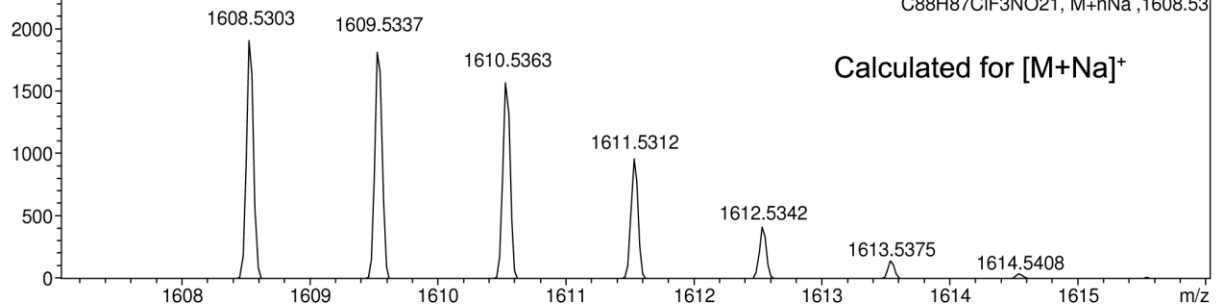
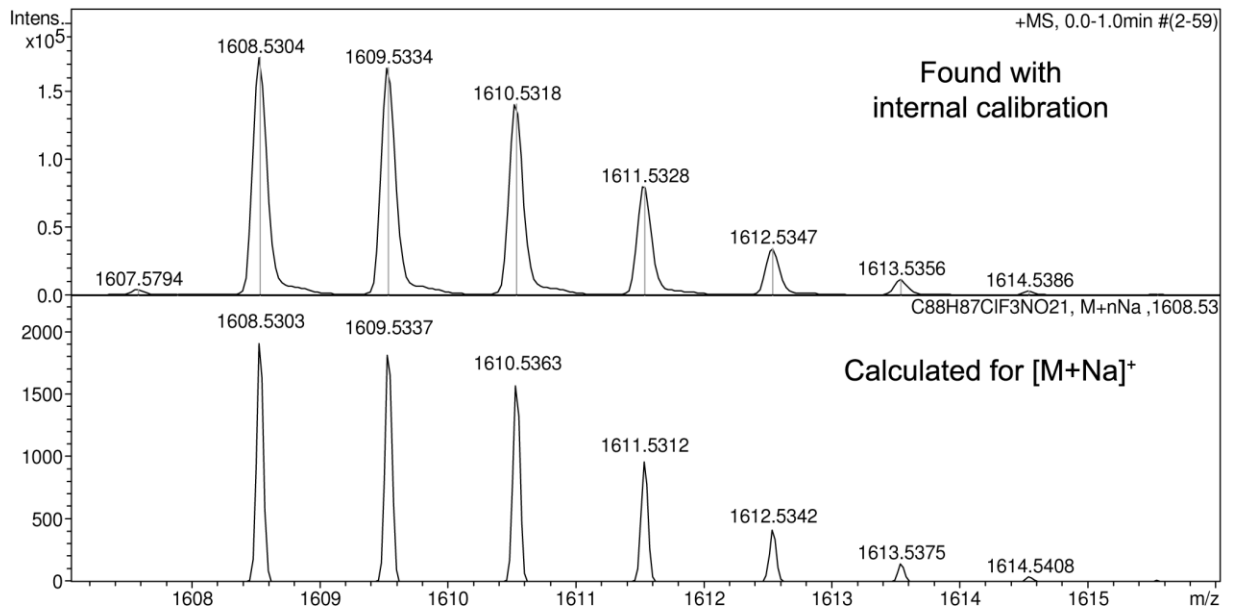
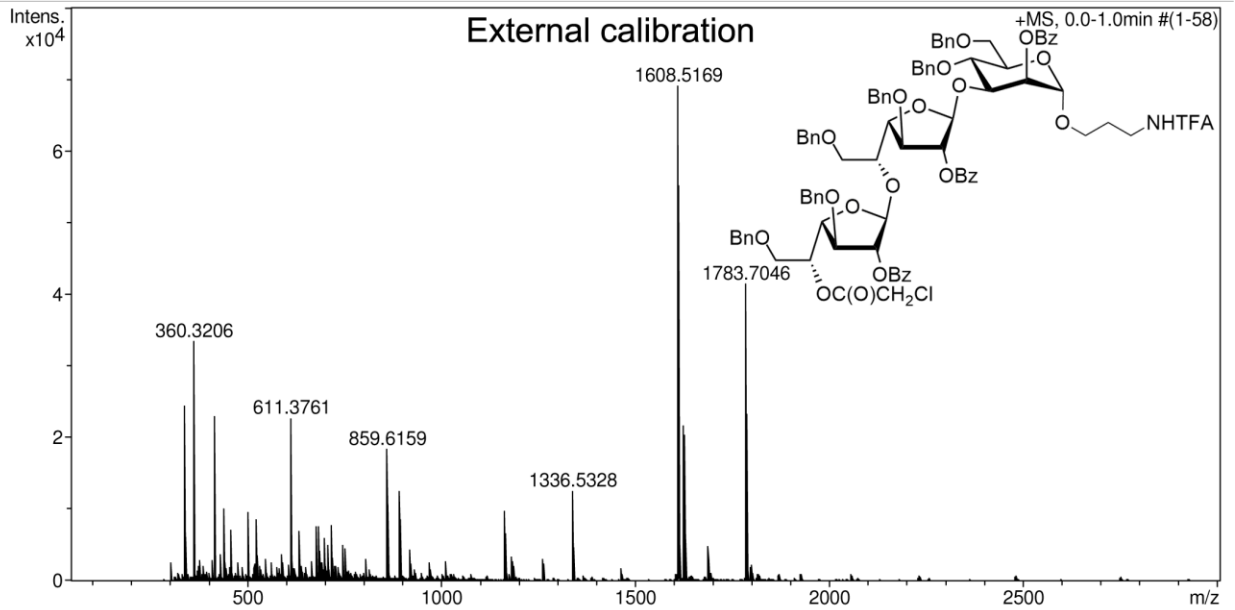


3-trifluoroacetamidopropyl 2-O-benzoyl-3,6-di-O-benzyl-5-O-chloroacetyl- β -D-galactofuranosyl-(1 \rightarrow 5)-2-O-benzoyl-3,6-di-O-benzyl- β -D-galactofuranosyl-(1 \rightarrow 3)-2-O-benzoyl-4,6-di-O-benzyl- α -D-mannopyranoside (28)

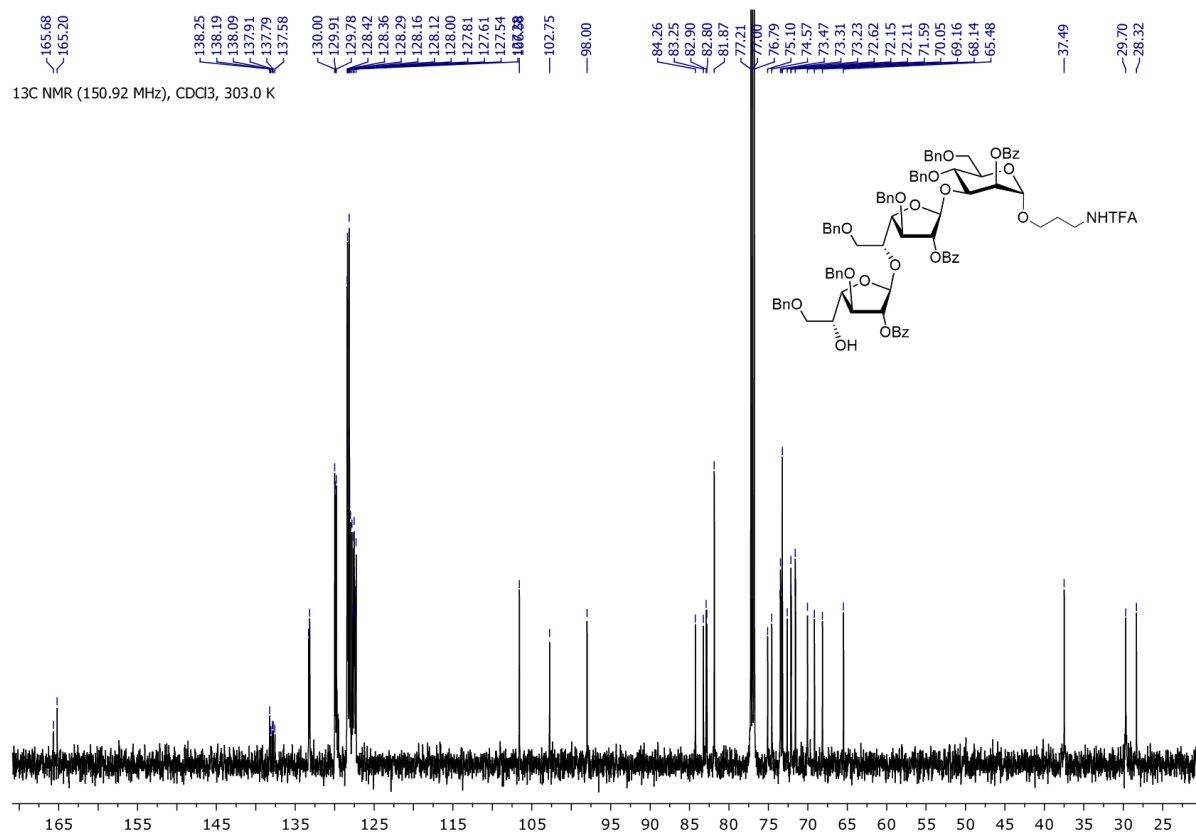
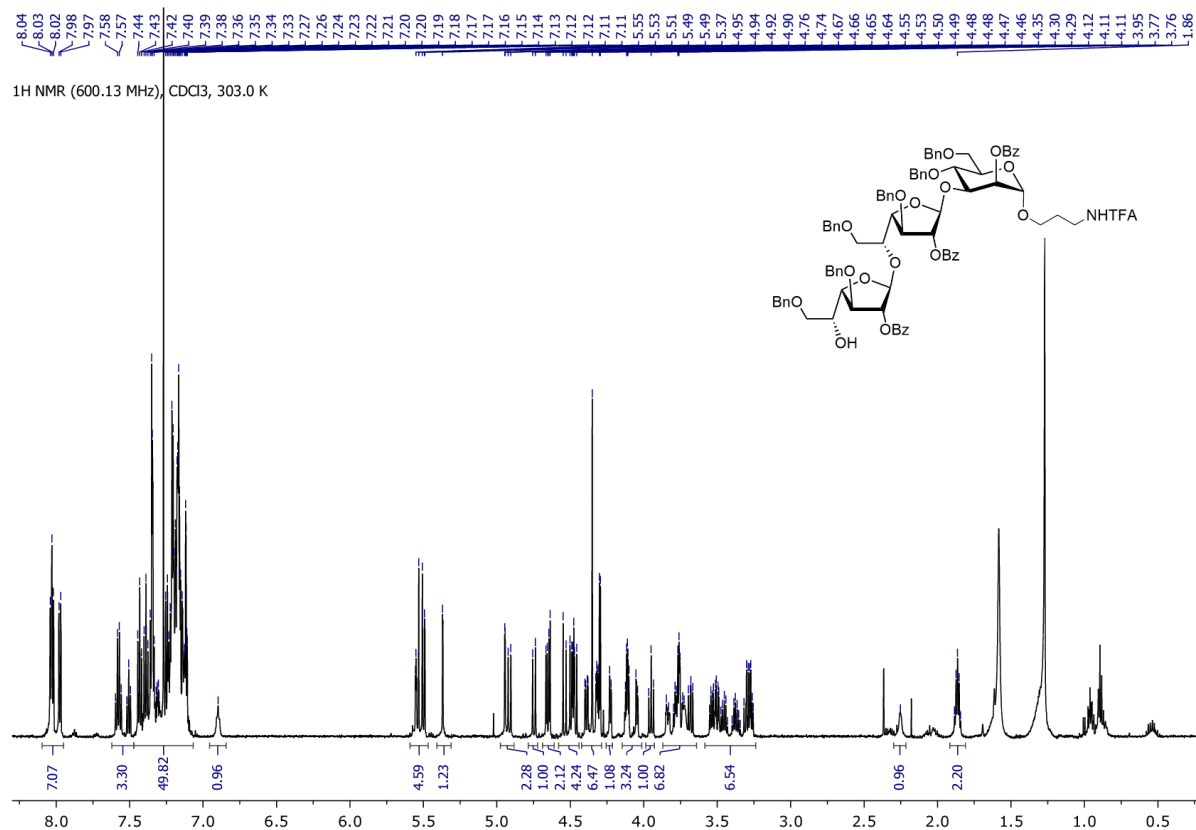


Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

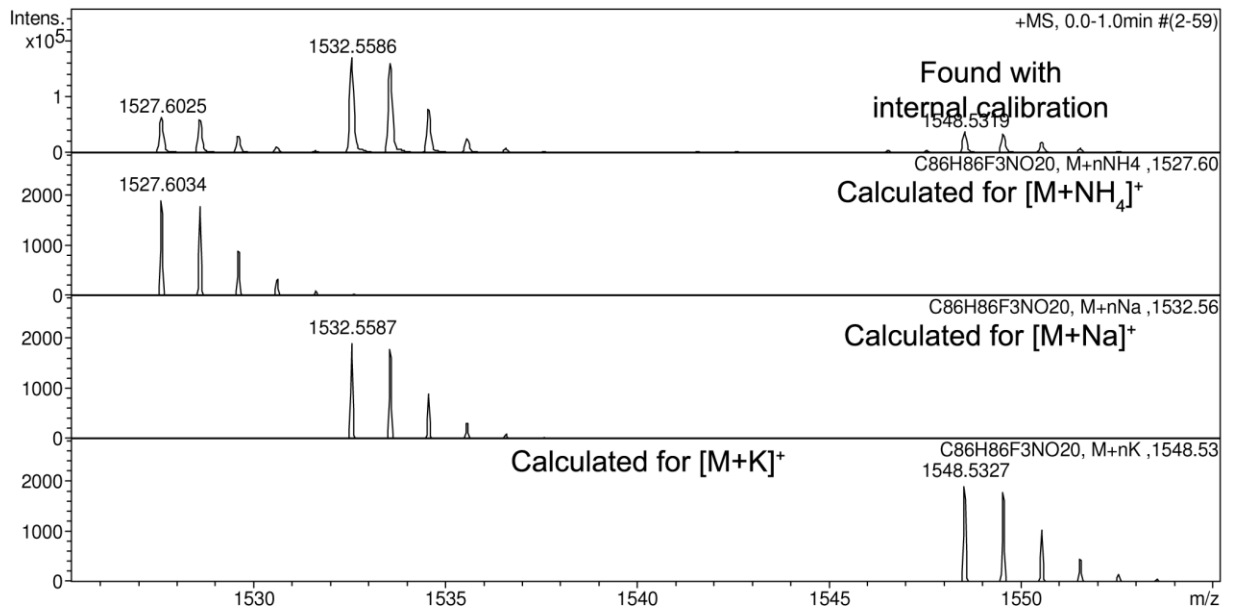
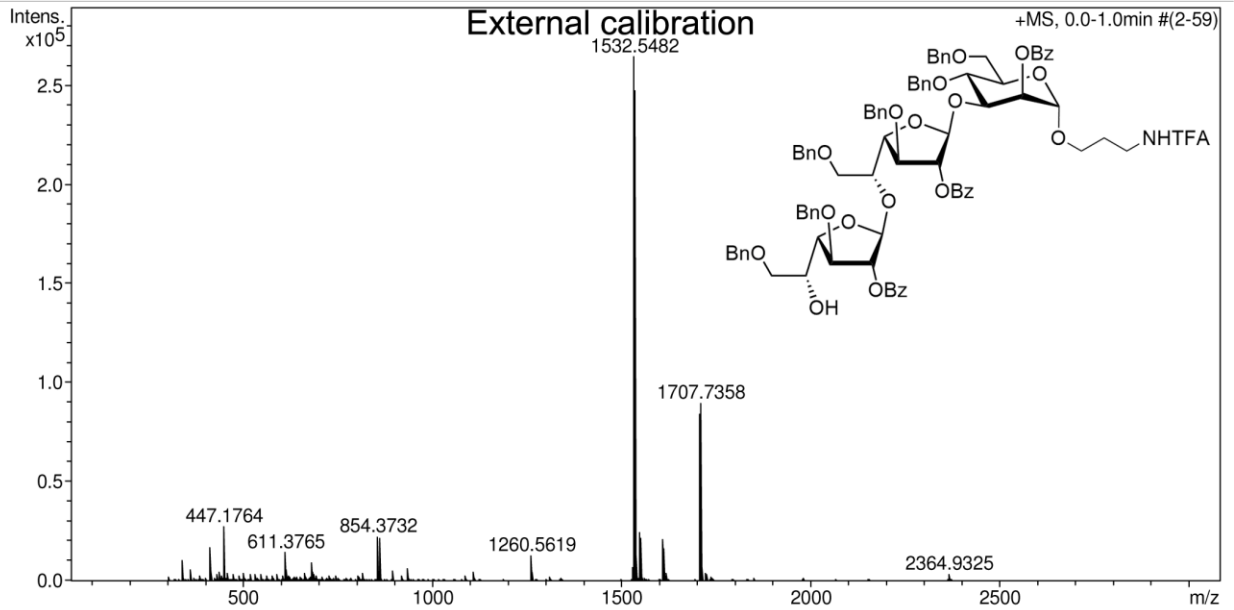


3-trifluoroacetamidopropyl 2-O-benzoyl-3,6-di-O-benzyl-β-D-galactofuranosyl-(1→5)-2-O-benzoyl-3,6-di-O-benzyl-β-D-galactofuranosyl-(1→3)-2-O-benzoyl-4,6-di-O-benzyl-α-D-mannopyranoside (29)

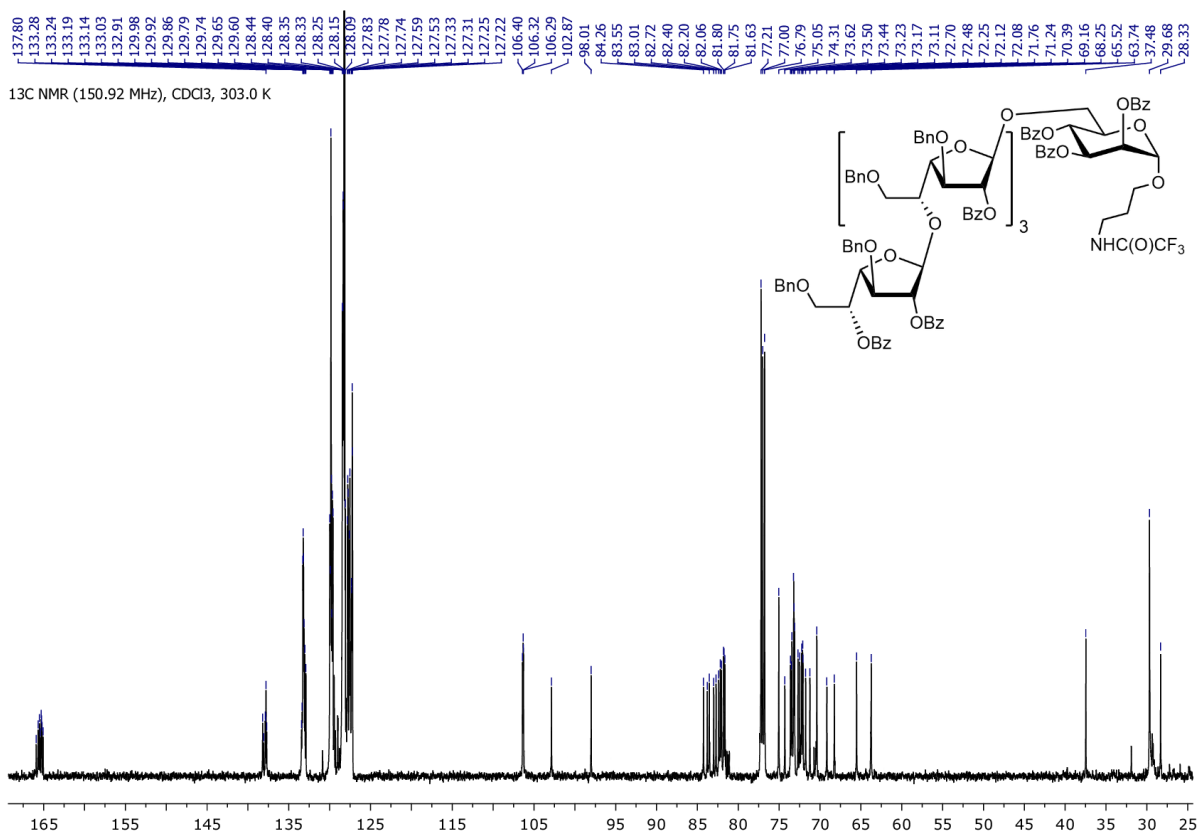
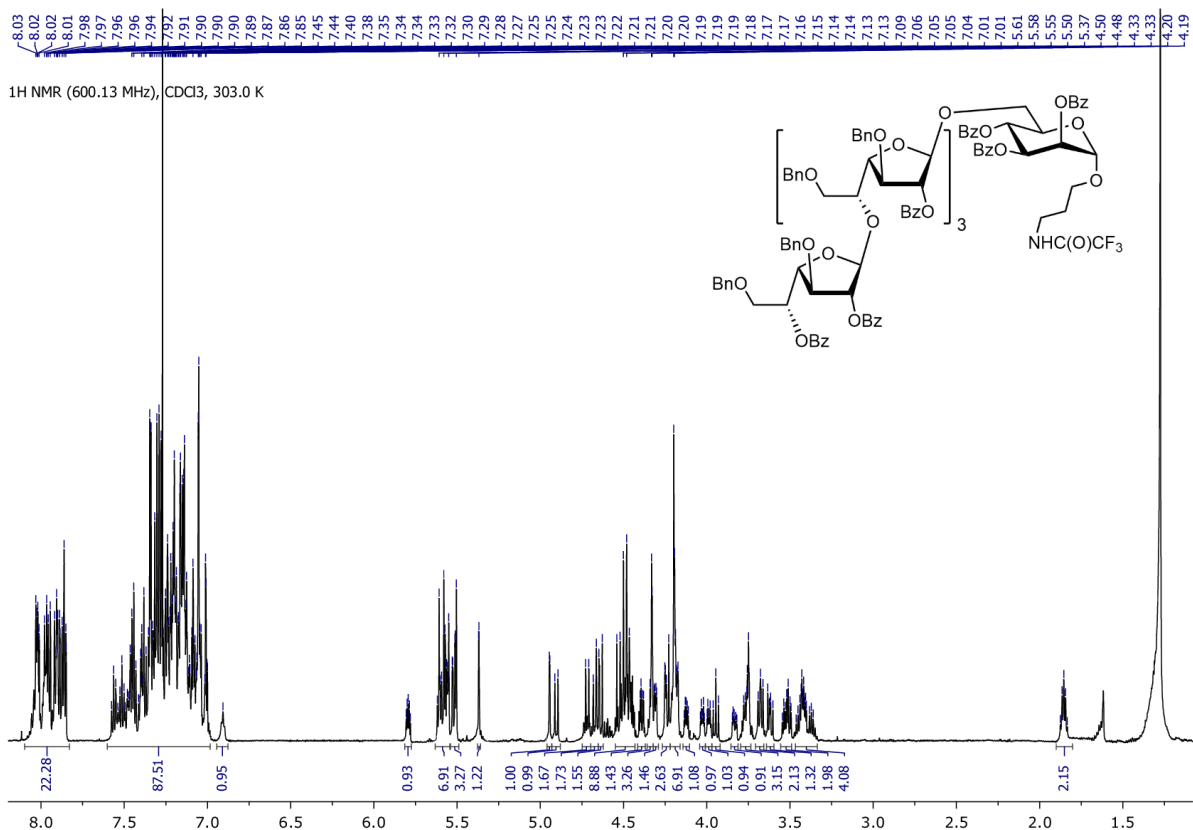


Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

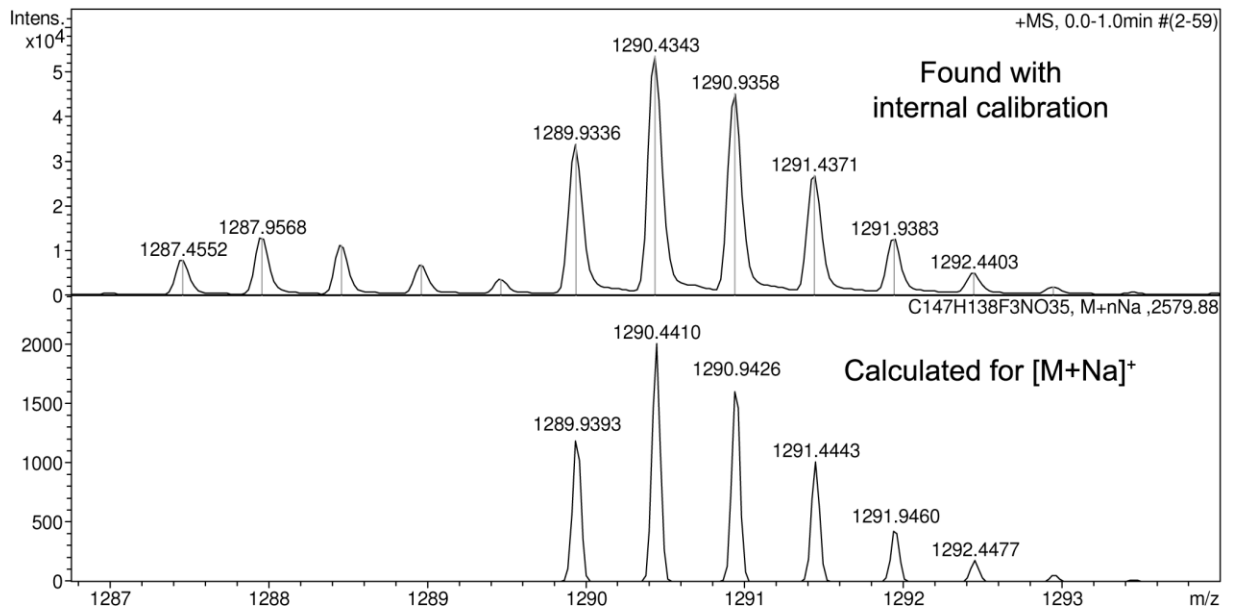
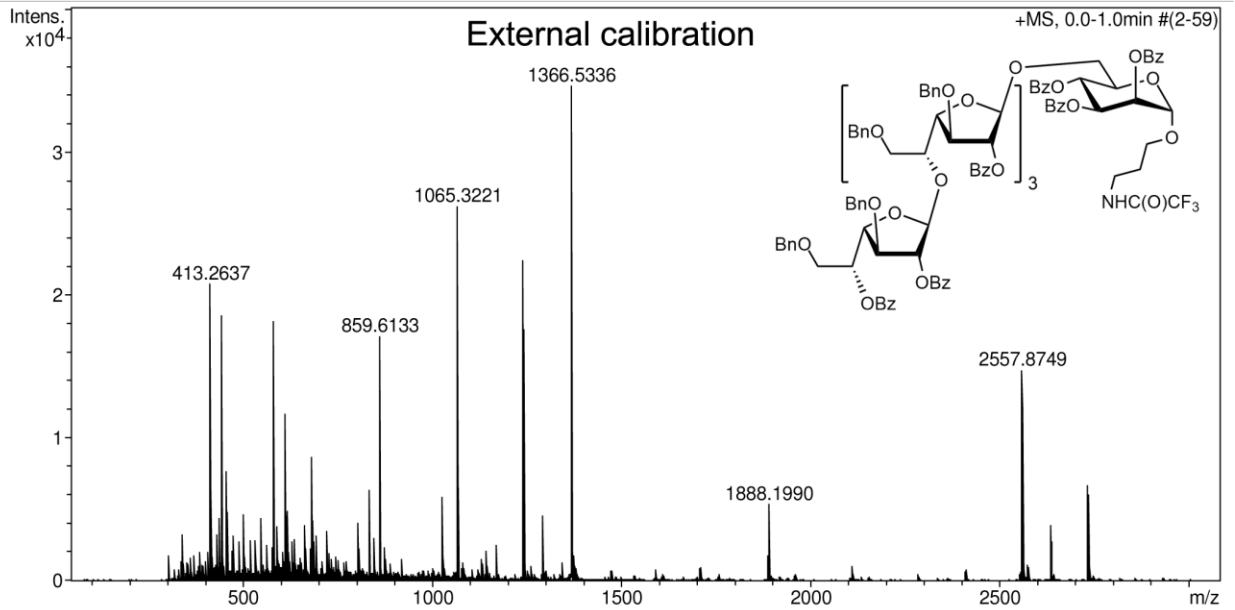


3-trifluoroacetamidopropyl 2,3,5,6-tetra-O-benzoyl-β-D-galactofuranosyl-(1→5)-2-O-benzoyl-3,6-di-O-benzyl-β-D-galactofuranosyl-(1→5)-2-O-benzoyl-3,6-di-O-benzyl-β-D-galactofuranosyl-(1→5)-2-O-benzoyl-3,6-di-O-benzyl-β-D-galactofuranosyl-(1→3)-2-O-benzoyl-4,6-di-O-benzyl-α-D-mannopyranoside (30)

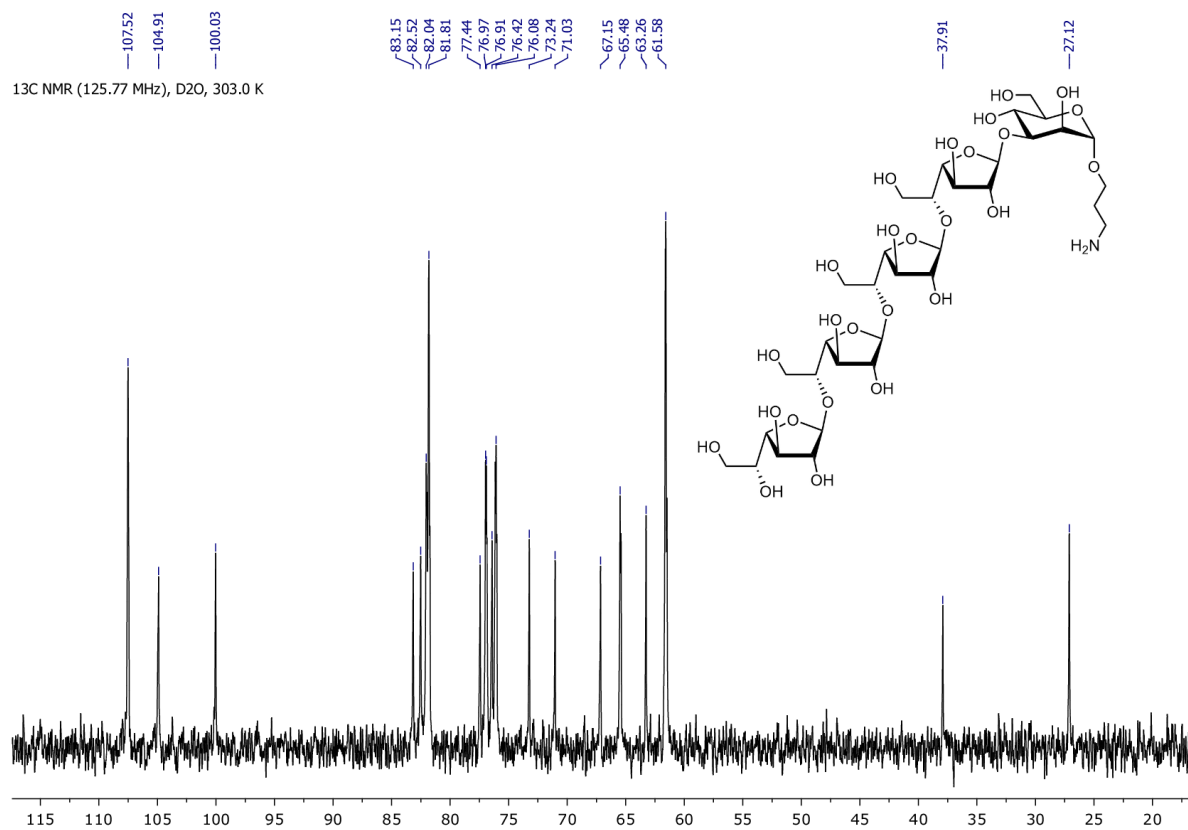
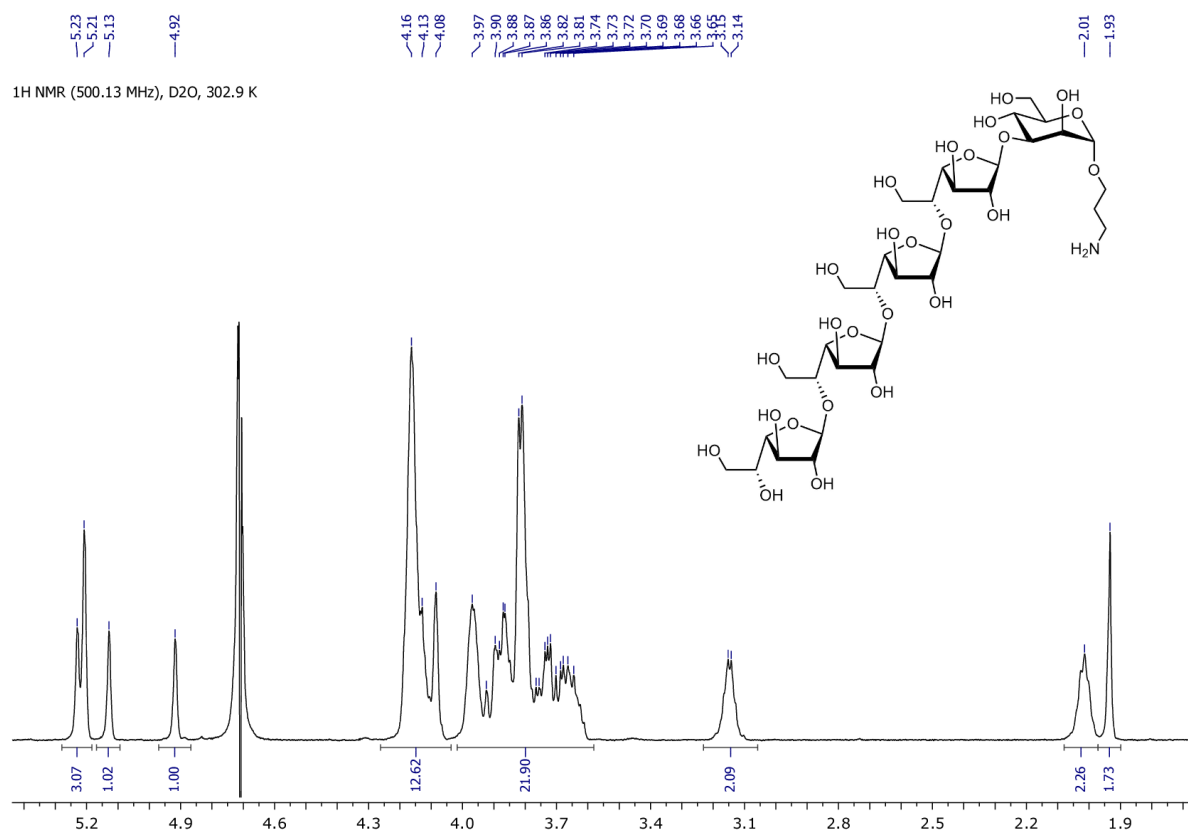


Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



3-aminopropyl β -D-galactofuranosyl-(1 \rightarrow 5)- β -D-galactofuranosyl-(1 \rightarrow 5)- β -D-galactofuranosyl-(1 \rightarrow 5)- β -D-galactofuranosyl-(1 \rightarrow 3)- α -D-mannopyranoside (2)



Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	1200.0 Vpp	Set Divert Valve	Waste

