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Preparative synthesis of selectively substituted 1,6-anhydro-α-D-galactofuranose derivatives

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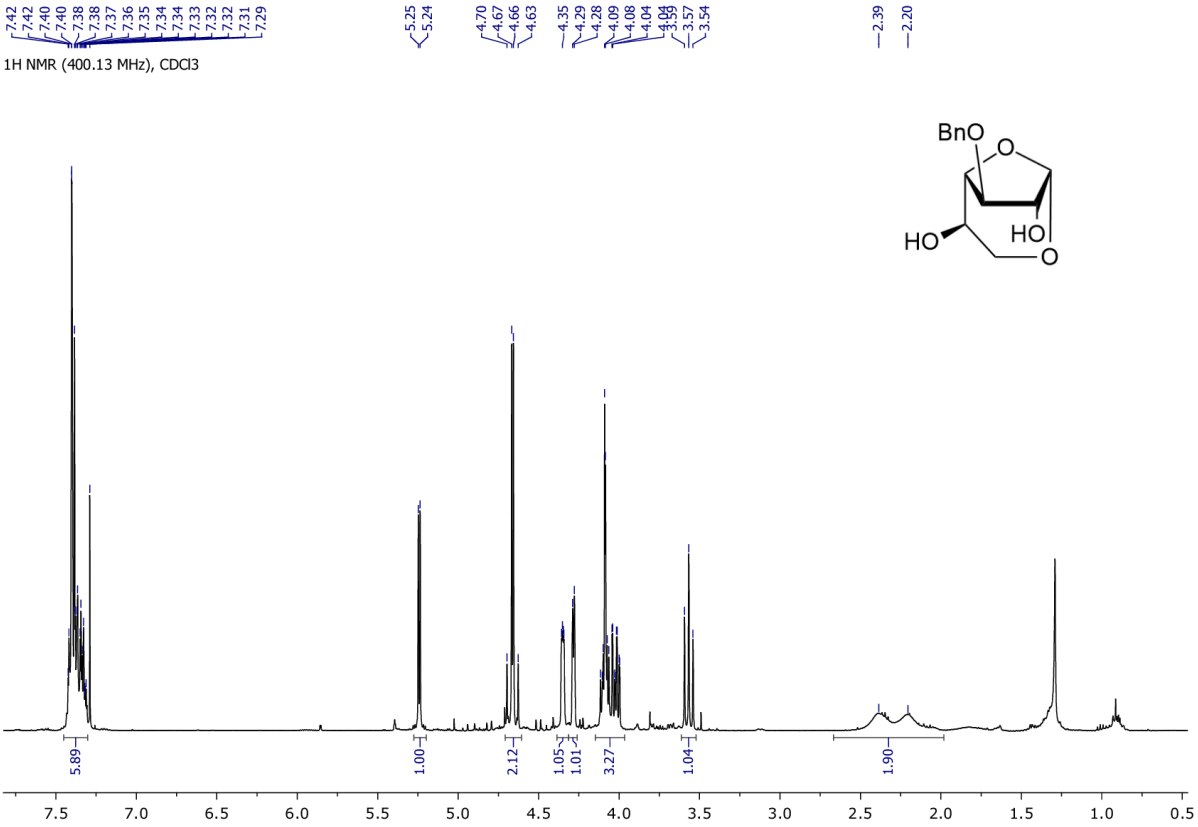
Materials and methods

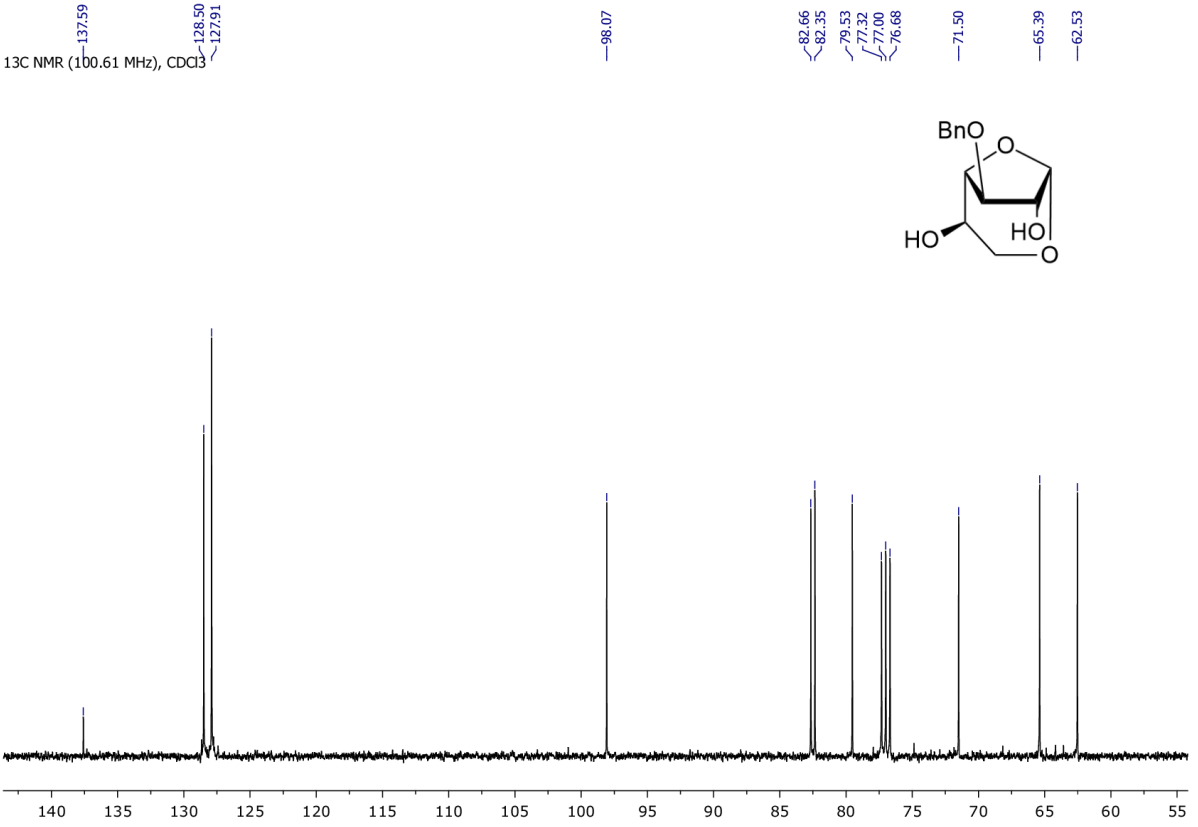
All reagents for synthesis were commercial and used without further purification. Solvents were distilled over CaH_2 (CH_2Cl_2) or purchased as dry. All reactions involving air- or moisture-sensitive reagents were carried out using dry solvents under dry argon. Thin-layer chromatography (TLC) was carried out on aluminum sheets coated with silica gel 60 F_{254} (Merck). Analysis TLC plates were inspected by UV light (λ = 254 nm) and developed by treatment with a mixture of 15% H_3PO_4 and orcinol (1.8 g/l) in EtOH/H_2O (95:5, v/v) followed by heating. Silica gel column chromatography was performed with Silica Gel 60 (40-63 μm, E. Merck).

NMR spectra were recorded at 293-305 K using Bruker AMX400 (400 MHz) or Bruker AV600 (600 MHz) spectrometer. Chemical shifts are reported relative to chloroform (δ 7.27) for ^{1}H NMR and (δ 77.0) for ^{13}C NMR. Optical rotations were measured using a JASCO DIP-360 polarimeter at the ambient temperature in solvents specified. High resolution mass spectra (HR MS) were measured on a Bruker micrOTOF II instrument using electrospray ionization (ESI).[1] The measurements were done in a positive ion mode (interface capillary voltage –4500 V) or in a negative ion mode (3200 V); mass range from m/z 50 to m/z 3000 Da; external or internal calibration was done with Electrospray Calibrant Solution (Fluka). A syringe injection was used for solutions in a mixture of acetonitrile and water (50:50 v/v flow rate 3 lL/min). Nitrogen was applied as a dry gas; interface temperature was set at 180 °C.

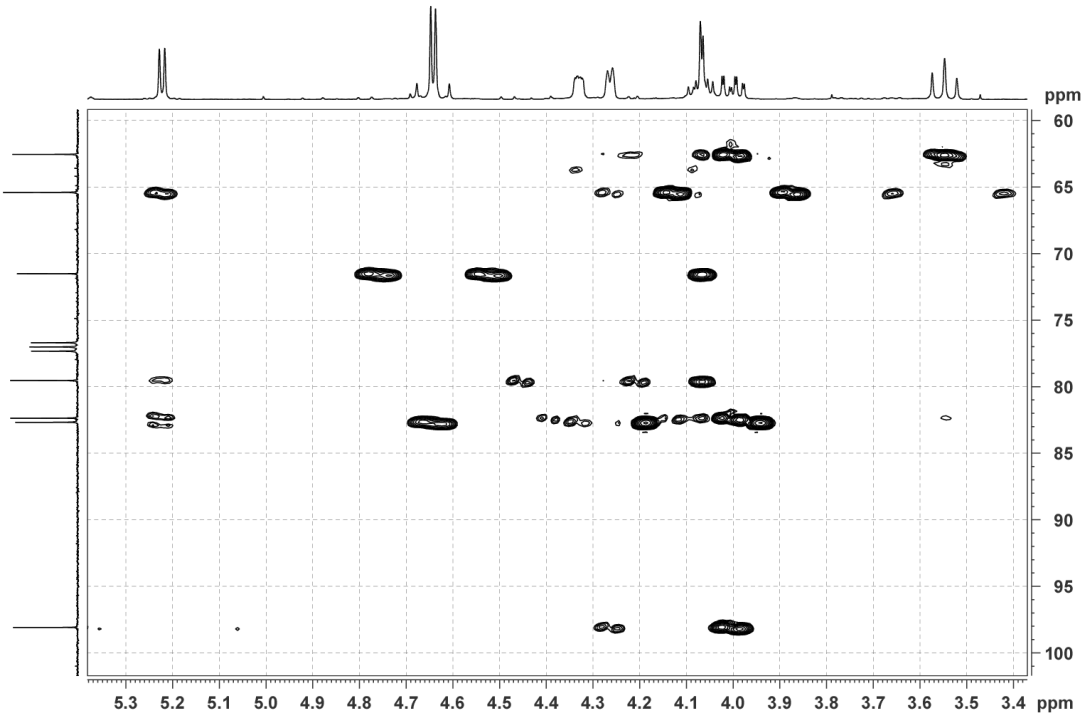
1 P.A. Belyakov, V.I. Kadentsev, A.O. Chizhov, N.G. Kolotyrkina, A.S. Shashkov and V.P. Ananikov, *Mendeleev Commun.*, 2010, **20**, 125.

1,6-Anhydro-3-O-benzyl-α-D-galactofuranose 4



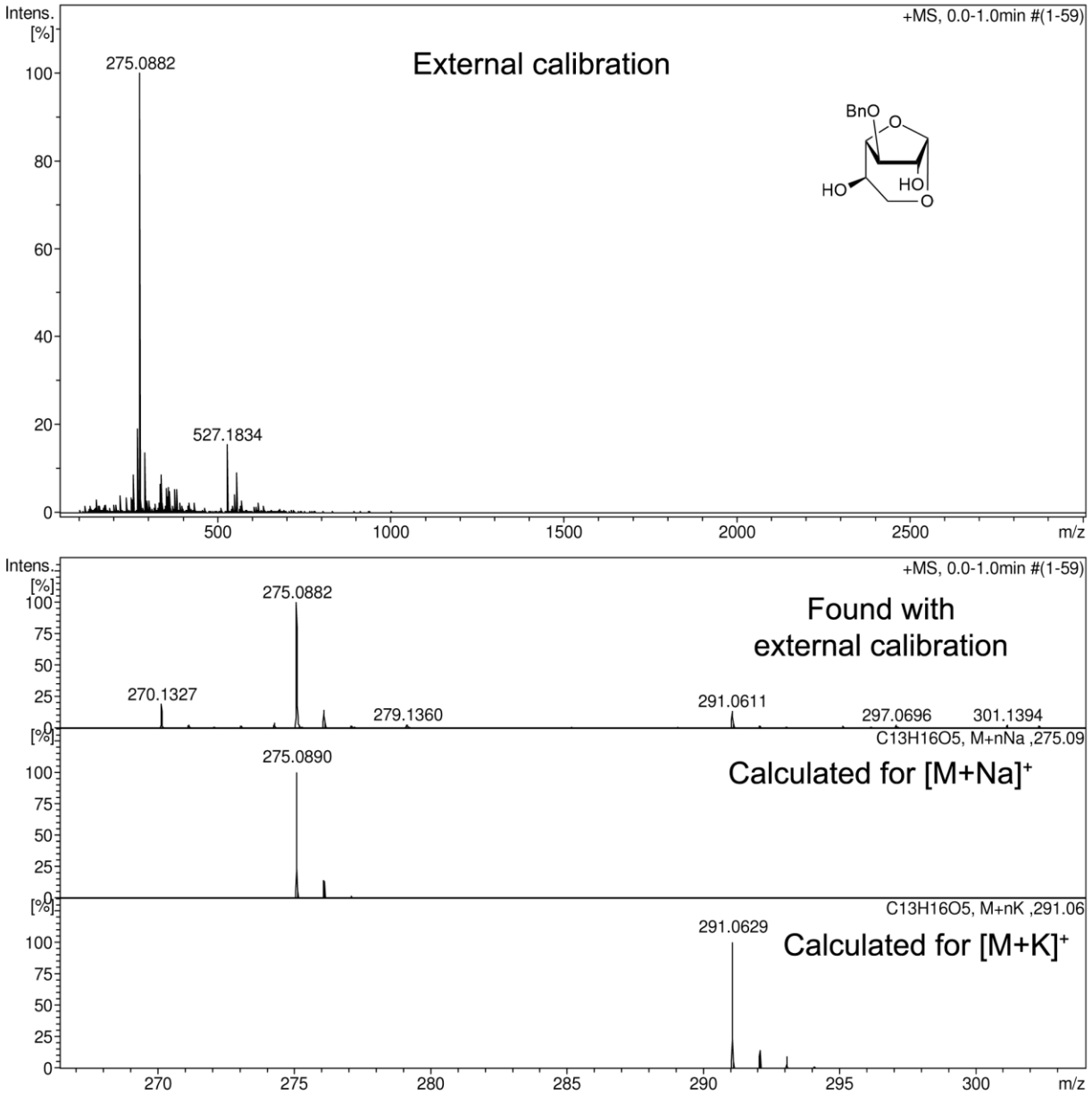


HMBC (600 MHz, $CDCl_3$)

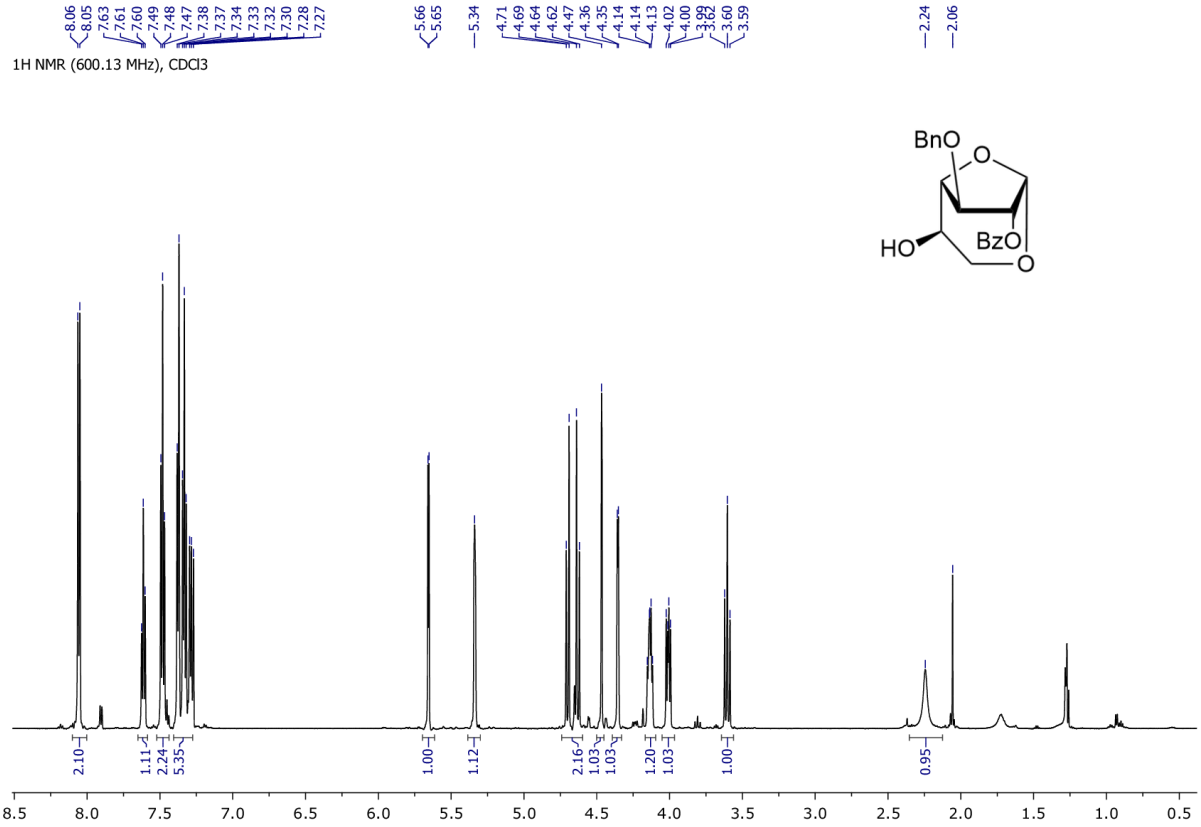


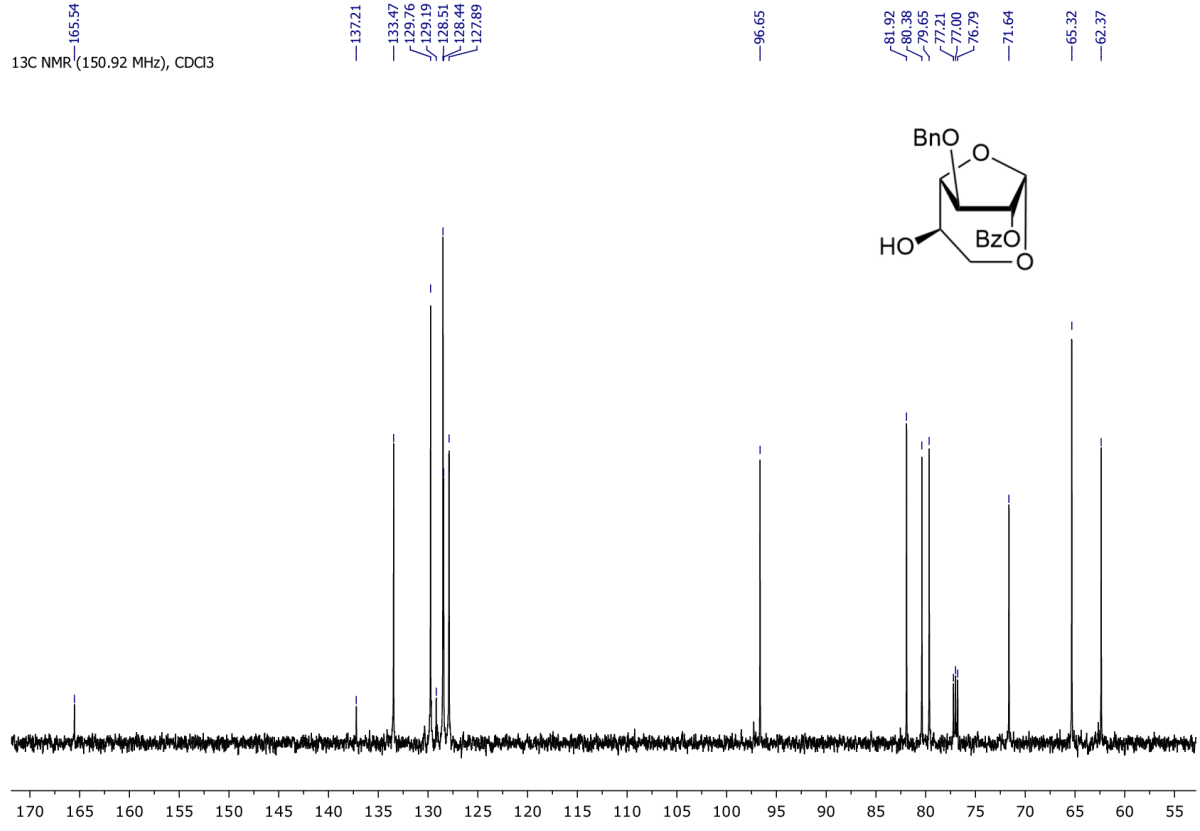
Acquisition Parameter

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

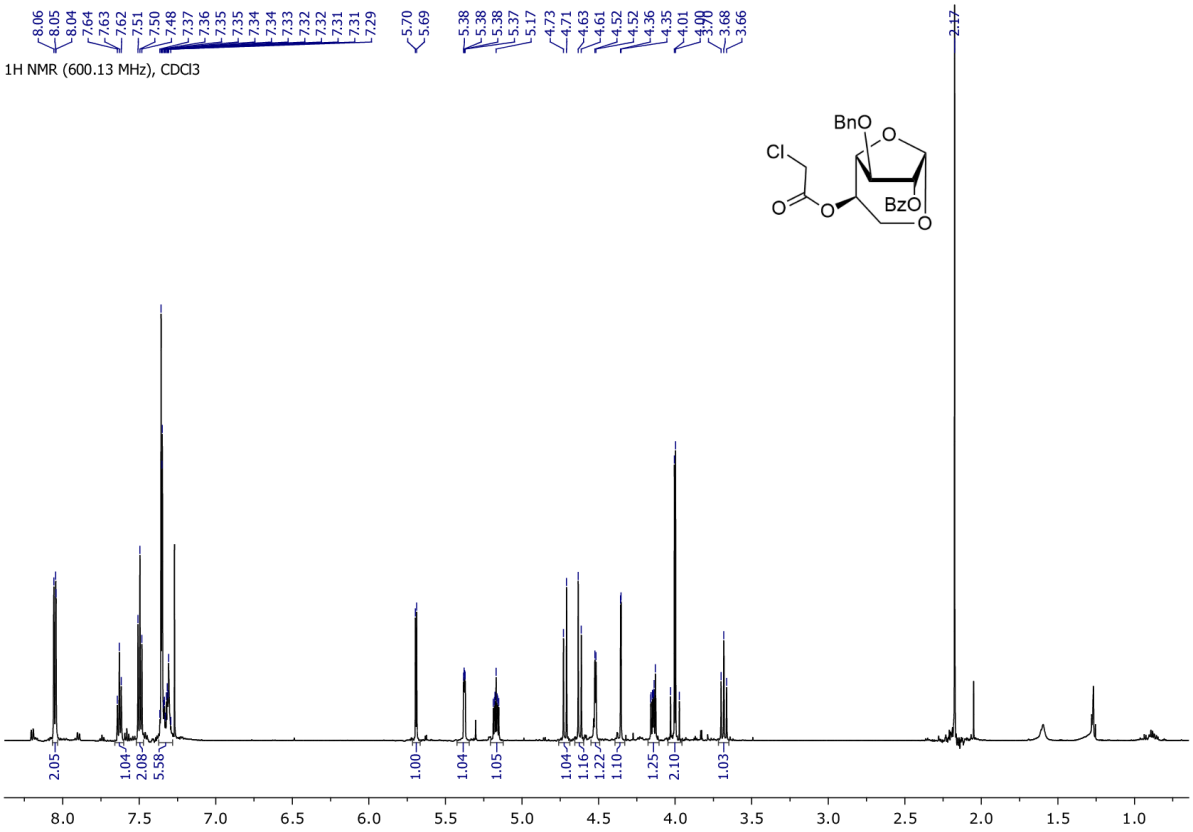


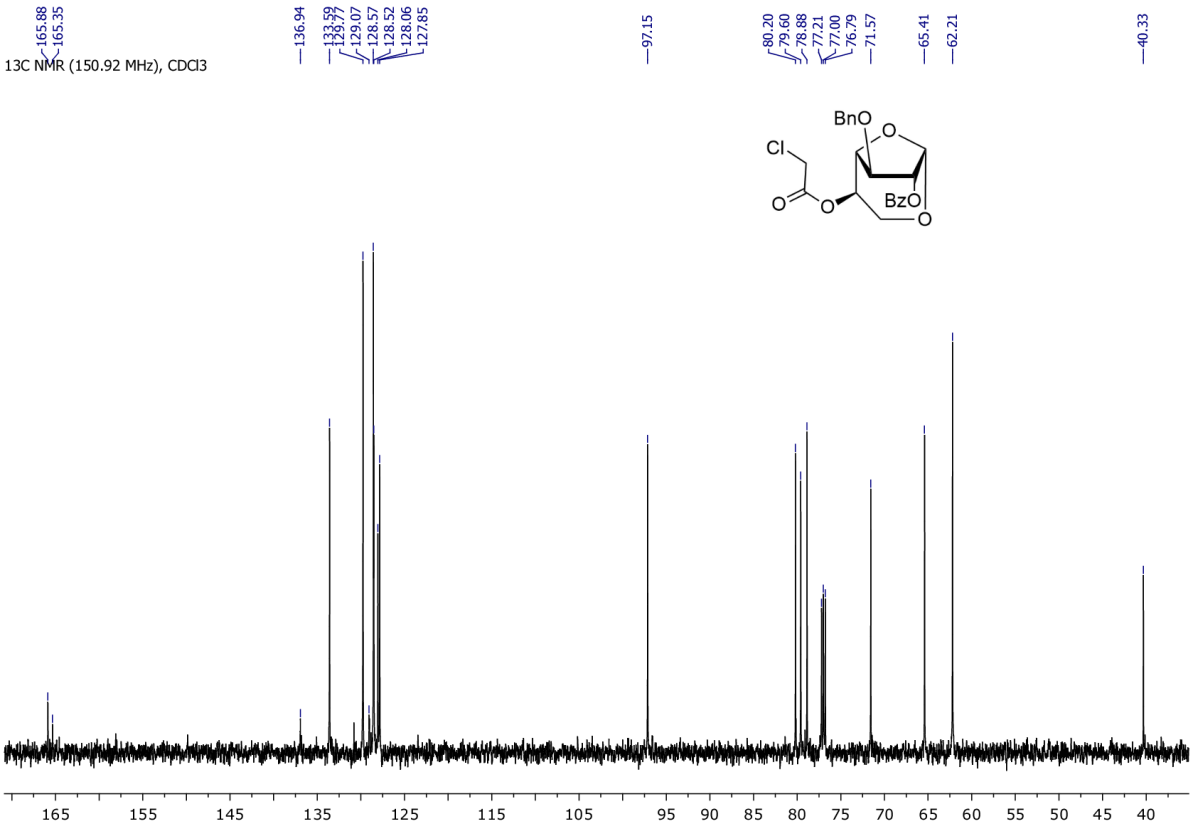
1,6-Anhydro-2-O-benzoyl-3-O-benzyl-α-D-galactofuranose 4





1,6-Anhydro-2-O-benzoyl-3-O-benzyl-5-O-chloroacetyl-α-D-galactofuranose 6





Acquisition Parameter

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