Isothiourea - Catalyzed α - Selective Glycosylations

Table of Contents

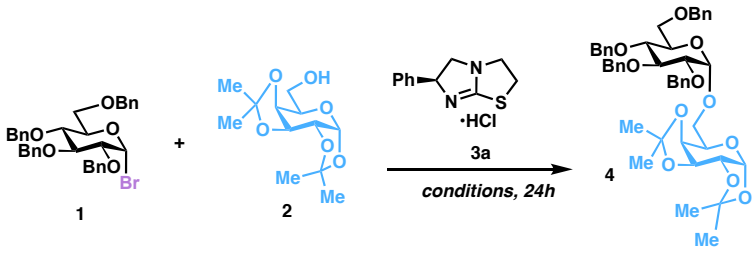
1. General Information

All reactions were performed in flame dried round bottom or modified Schlenk (Kjedahl shape) flasks fitted with rubber septa or a yellow PTFE cap under a positive pressure of argon, unless otherwise indicated. Air-and moisture-sensitive liquids and solutions were transferred by syringe or canula. Dry THF, Et_2O and CH_2Cl_2 were obtained from a PureSolv MD-5 Solvent Purification System (Inert). Unless otherwise noted, all other reagents were used as obtained from commercial sources without further purification. Analytical thin-layer chromatography (TLC) was carried out using 0.2 mm commercial silica gel plates (silica gel 60, F254, Merck) and visualized using a UV lamp and or ceric ammonium molybdate (CAM) or aqueous potassium permanganate ($KMnO_4$) stain. Preparatory TLC was also carried out on the same silica gel plates. Organic solutions were concentrated using a Heidolph rotary evaporator at ~ 10 torr.

NMR spectra (1D and 2D experiments) were recorded on a Bruker AV-III-HD spectrometer (^1H at 500 MHz and ^{13}C at 125 MHz). Chemical shifts (δ) are given in ppm with reference to residual proton signals in the solvent [^1H NMR – $CHCl_3$ (7.26); ^{13}C NMR: $CDCl_3$ (77.00). Data are presented as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, and bs = broad singlet), integration, and coupling constant in hertz (Hz). High-resolution mass (HRMS) measurements for compound characterization were carried out using a Quan TOF analyzer or an Agilent 6550 QTOF system and are reported as m/z (relative intensity). Infrared spectra were recorded on ThermoFisher Nicolet iS50 FT IR using neat thin film technique.

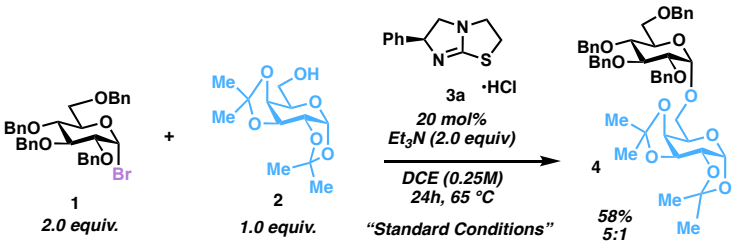
2. Additional Optimization Data

Figure S1. Reaction Optimization



Entry	Solvent (M)	Temp (°C)	Donor (equiv.)	Acceptor (equiv.)	Catalyst mol %	Base (equiv.)	Yield, α/β
1	PhMe [0.2M]	50	1	1	10	$(i\text{Pr})_2\text{NEt}$ (2.0)	64%, 2:1
2	MTBE [0.2M]	50	1	1	10	$(i\text{Pr})_2\text{NEt}$ (2.0)	56%, 1.6:1
3	DCE [0.2M]	50	1	1	10	$(i\text{Pr})_2\text{NEt}$ (2.0)	54%, 2.5:1
4	DCE [0.2M]	50	1	1	20	$(i\text{Pr})_2\text{NEt}$ (2.0)	52%, 2:1
5	DCE [0.2M]	50	1	1	20	NEt_3 (2.0)	42%, 3:1
6	DCE [0.2M]	65	1	1	20	NEt_3 (2.0)	47%, 3.5:1
7	DCE [0.2M]	65	2	1	20	NEt_3 (2.0)	80%, 2.7:1
8	DCE [0.2M]	65	1	2	20	NEt_3 (2.0)	61%, 3:1
9	DCE [0.25M]	65	2	1	20	NEt_3 (2.0)	58%, 5:1
10	DCE [0.25M]	65	2	1	0	NEt_3 (2.0)	51%, 2:1
11	DCE [0.25M]	65	2	1	20	NEt_3 (0)	messy

Figure S2. Catalyst Optimization



Entry	*Deviation from Standard Conditions*	Yield, α/β
1	catalyst 3b	**57%, 4.5:1**
2	catalyst 3c	**67%, 5.2:1**
3	catalyst 3d	**75%, 5.2:1**
4	catalyst 3e	**64%, 4.8:1**
5	catalyst 3f	**55%, 6:1**
6	catalyst 3g	**71%, 6:1**
7	**catalyst 3h**	**83%, 9.5:1**
8	catalyst 3i	**74%, 4.5:1**
9	catalyst 3h, 10 mol%	**65%, 10:1**

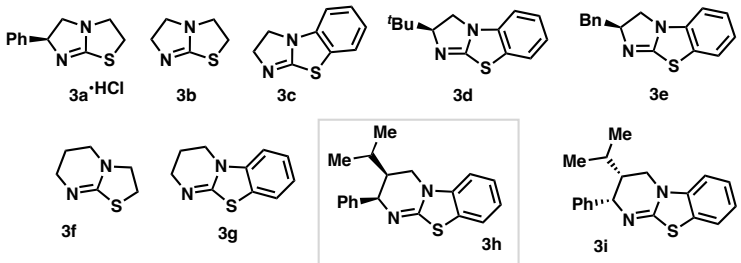


Figure S3. Bromide and Chloride Comparison

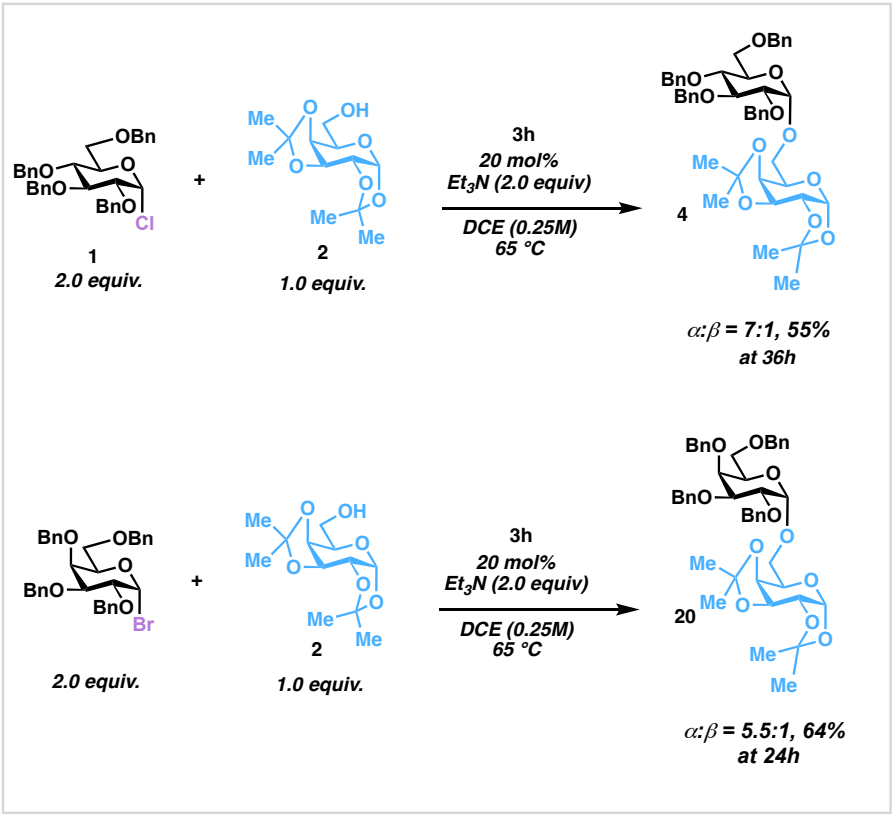


Figure S4. NMR Study

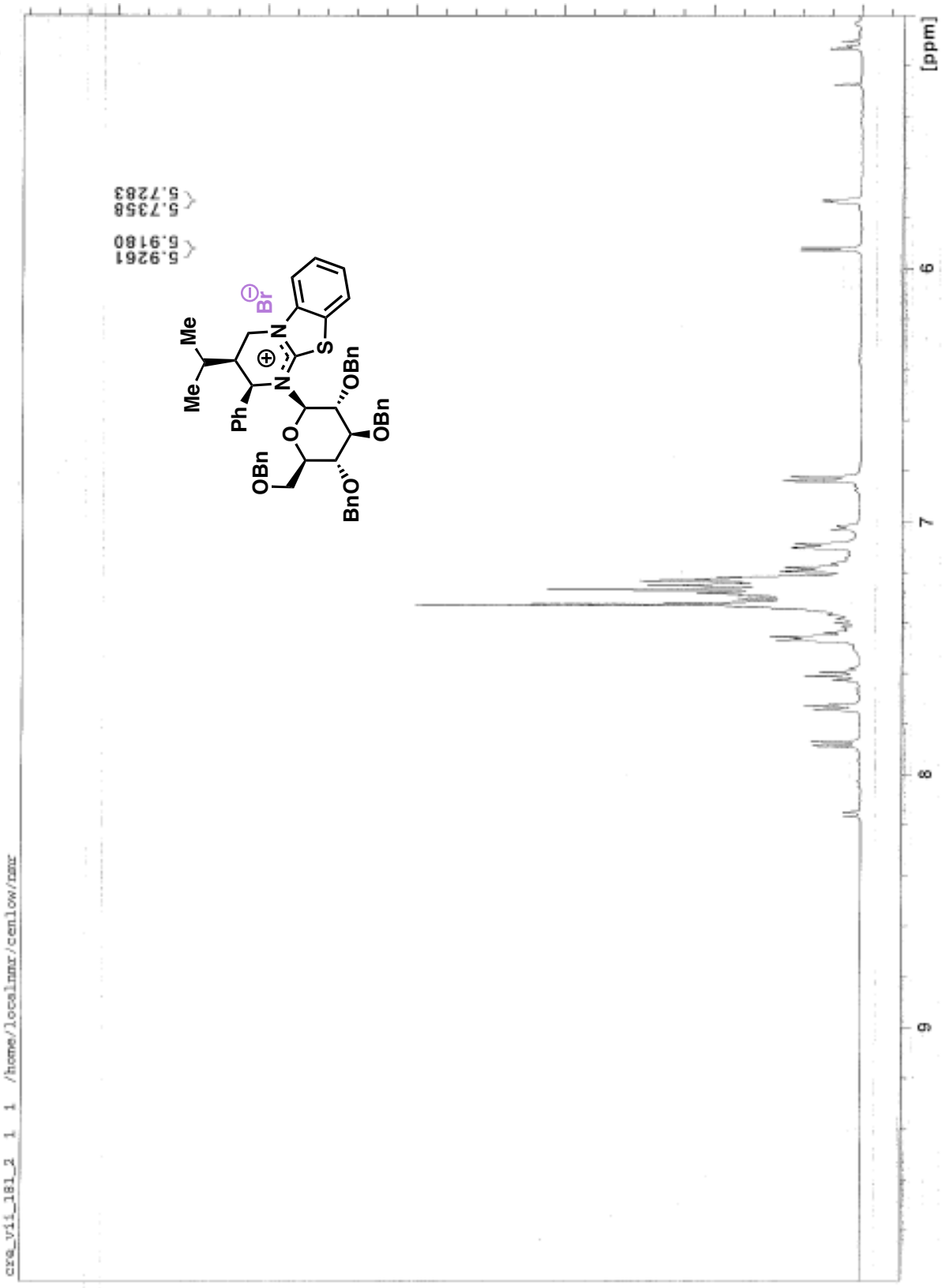
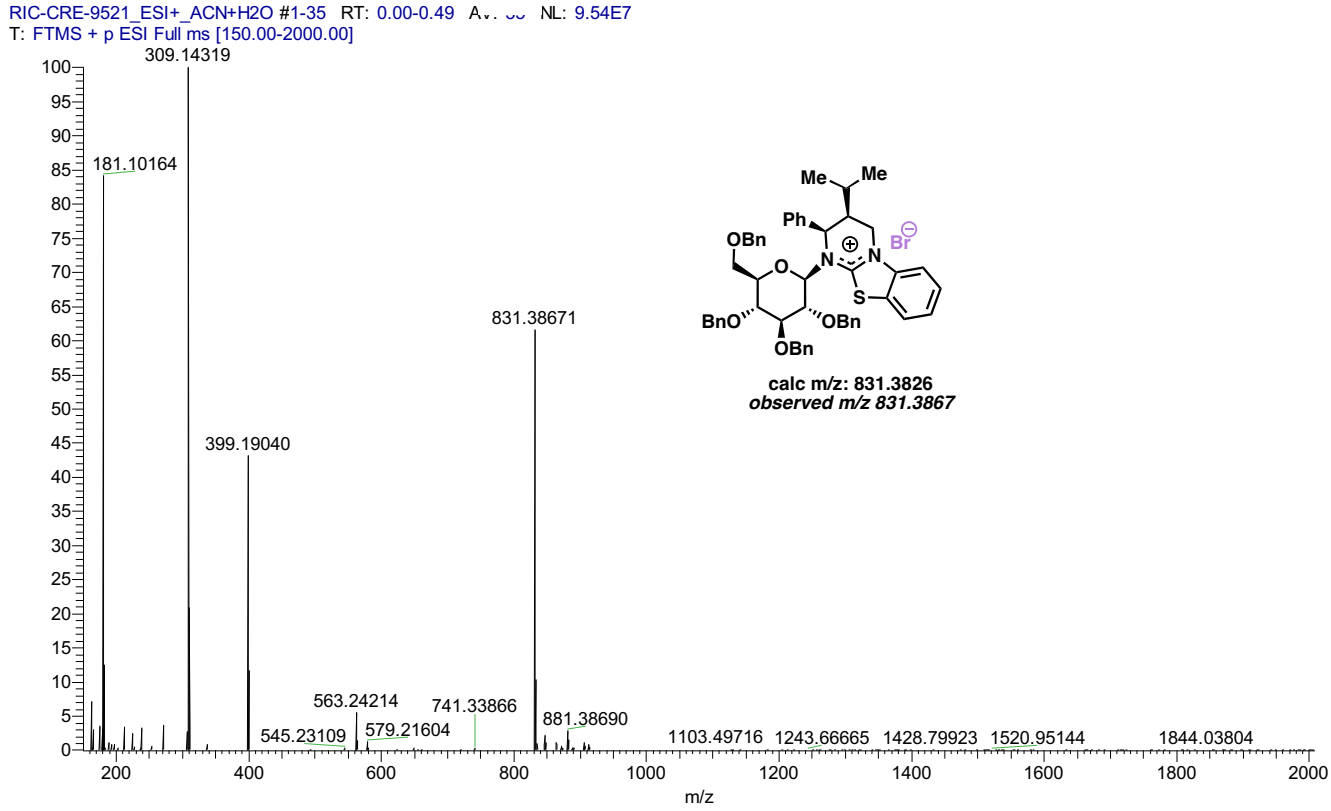
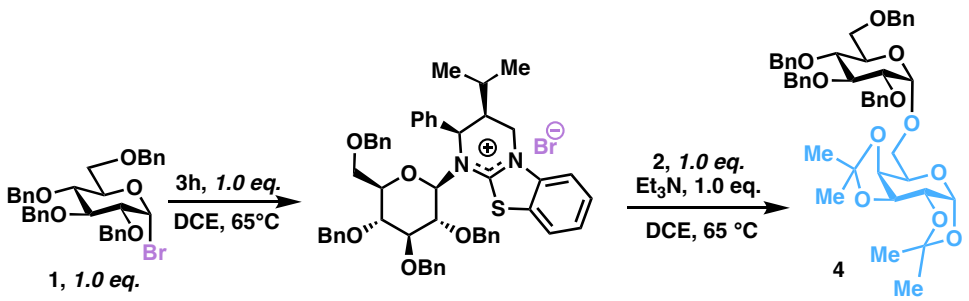

cre_vii_181_2 1 1 /home/localnmr/cen1ow/nmr
5.9261
5.9180
5.7358
5.7283
Me
Me
Ph
N
S
Br
OBn
OBn
BnO
OBn
O
[ppm]
6
7
8
9

Figure S5. Mass Spectrometry Study



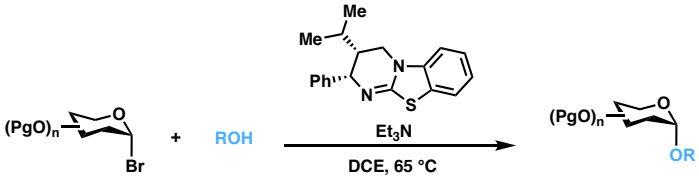


A mixture of **1** (40 mg, 0.066 mmol, 1.0 equiv.) and **3h** (30 mg, 0.09 mmol, 1.5 equiv.) were heated in 1,2 DCE (0.4 mL) at 65 °C for 12 h. The reaction was concentrated to dryness and a small amount dissolved in MeCN. The cationic adduct **5**, was confirmed through electrospray ionization (ESI) with an *m/z* observed at 831.3867 (*vide supra*). The dried material was mixed with **2** (17.2 mg, 0.066 mmol, 1.0 equiv.), and Et_3N (6.7 μL, 0.066 mmol, 1.0 equiv.) in 1,2 DCE (0.4 mL) 65 °C for 24h. Following concentration and purification on silica gel (hexanes/ethyl acetate), **4** was obtained (14 mg, 27% yield).

*Note – The adduct is very hygroscopic and has limited stability, reflected in the low yield of the stoichiometric reaction.

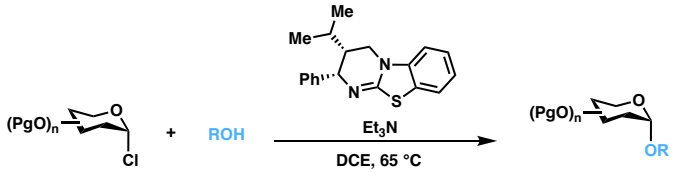
3. Experimental Procedures

General Procedure A



A 10 mL flame-dried round bottle flask was charged with glucosyl bromide (0.4 mmol, 2.0 equiv.), alcohol (0.2 mmol, 1.0 equiv.), catalyst **3h** (0.04 mmol, 20 mol%), Et_3N (0.4 mmol, 2.0 equiv.) and DCE (0.8 mL). The reaction was stirred at 65 °C for 20-24 h, concentrated, and purified by silica gel flash chromatography (hexanes/ethyl acetate) to give the desired product.

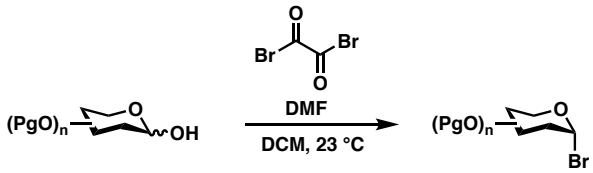
General Procedure B



A 10 mL flame-dried round bottle flask was charged with galactosyl or rhamnosyl chloride (0.4 mmol, 2.0 equiv), alcohol (0.2 mmol, 1.0 equiv), catalyst **3h** (0.04 mmol, 20 mol%), Et_3N (0.4 mmol, 2.0 equiv.) and DCE (0.4 mL). The resulting solution was stirred at 65 °C for 18-24 h, concentrated, and purified by silica gel flash chromatography (hexanes/ethyl acetate) to give the desired product.

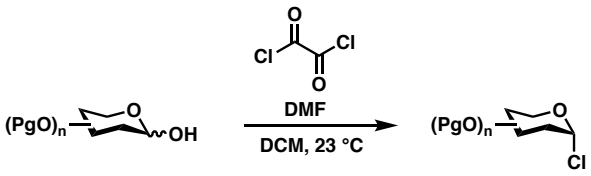
The α:β ratio of the desired products were determined by ^{1}H NMR analysis based on the ratio of the anomeric protons of both α- and β-anomers. When the anomeric protons are overlapped, other diagnostic protons of both anomers were analyzed.

General Procedure C – Preparation of Glucosyl Bromides



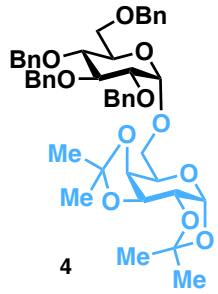
To a stirred (0.1M DCM) solution of hemiacetal at 23°C was added DMF (1.5 equiv.). To this was added a solution of oxalyl bromide (2.0M DCM, 4.0 equiv.), dropwise. Upon consumption of starting material, the reaction was poured into saturated aqueous $NaHCO_3$, and extracted three times with 1:1 hexanes:ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate and concentrated, *in vacuo*. The product was used immediately and was sufficiently pure.

General Procedure D – Preparation of Galactosyl and Rhamnosyl Chlorides



To a stirred (0.1M DCM) solution of hemiacetal at 23°C was added DMF (1.5 equiv.). To this was added neat oxalyl chloride (4.0 equiv.), dropwise. Upon consumption of starting material, the reaction was poured into saturated aqueous $NaHCO_3$, and extracted three times with 1:1 hexanes:ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate and concentrated, *in vacuo*. Silica gel chromatography (hexanes:ethyl acetate) furnished the glycosyl chlorides.

4. Experimental Data



4

General Procedure A: 130 mg, 83%, $\alpha:\beta$ = 9.5:1

Data for α anomer **4**:

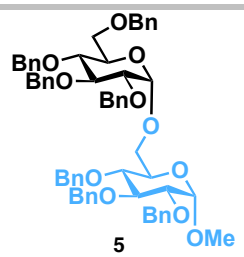
R_f = 0.39 (hexane/AcOEt 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ 7.40-7.23 (m, 18H), 7.17-7.12 (m, 2H), 5.53 (d, *J* = 5.0 Hz, 1H), 5.01 (d, *J* = 3.7 Hz, 1H), 4.99 (d, *J* = 10.9 Hz, 1H), 4.82 (dd, *J* = 12.2, 10.8 Hz, 2H), 4.75 (d, *J* = 11.9 Hz, 1H), 4.70 (d, *J* = 11.9 Hz, 1H), 4.63 (d, *J* = 12.1 Hz, 1H), 4.60 (dd, *J* = 7.9, 2.4 Hz, 1H), 4.48 (dd, *J* = 11.5, 8.4 Hz, 2H), 4.36 (dd, *J* = 7.9, 1.9 Hz, 1H), 4.32 (dd, *J* = 5.0, 2.4 Hz, 1H), 4.05 (ddd, *J* = 7.9, 6.1, 1.9 Hz, 1H), 3.99 (t, *J* = 9.3 Hz, 1H), 3.83 (dt, *J* = 9.9, 2.7 Hz, 1H), 3.81-3.73 (m, 3H), 3.68 (dd, *J* = 10.1, 9.0 Hz, 1H), 3.65 (dd, *J* = 10.7, 2.1 Hz, 1H), 3.59 (dd, *J* = 9.6, 3.6 Hz, 1H), 1.53 (s, 3H), 1.45 (s, 3H), 1.33 (s, 3H), 1.31 (s, 3H)

^{13}C NMR (125 MHz, $CDCl_3$):
δ 138.90, 138.31, 137.98, 128.61, 128.32, 127.90, 127.85, 127.79, 127.66, 127.61, 127.48, 109.17, 108.57, 97.01, 96.27, 81.93, 79.76, 77.53, 75.60, 74.96, 73.43, 72.31, 70.77, 70.61, 70.18, 68.31, 66.16, 65.65, 29.67, 26.14, 26.04, 24.89, 24.61.

IR (cm^{-1}): 2922, 1724, 1496, 1453, 1371, 1255, 1209, 1163, 1066, 1027, 999, 918, 889, 735, 696, 511.

HRMS-ESI (m/z): $[M+H]^+$ calcd. for $[C_{46}H_{54}O_{11}]^+$ 783.3666, found 805.3556, $[M+Na]^+$.



5

General Procedure A: 144 mg, 73%, $\alpha:\beta$ = 8:1

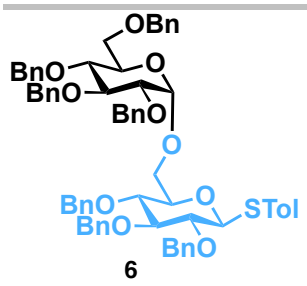
Data for α anomer **5**:

R_f = 0.45 (hexane/AcOEt = 4:1)
^{1}H NMR (500 MHz, $CDCl_3$):
δ 7.44-7.16 (m, 35H), 5.08-4.97 (m, 4H), 4.93-4.83 (m, 3H), 4.81-4.70 (m, 4H), 4.67-4.61 (m, 3H), 4.56-4.46 (m, 2H), 4.10-4.01 (m, 2H), 3.93-3.83 (m, 3H), 3.82-3.66 (m, 4H), 3.65-3.58 (m, 2H), 3.52 (dd, *J* = 9.6, 3.6 Hz, 1H), 3.43 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ 138.78, 138.42, 138.39, 138.14, 137.94, 128.37, 128.32, 128.29, 127.97, 127.84, 127.69, 127.58, 97.92, 97.21, 82.10, 81.64, 80.10, 79.94, 77.73, 77.57, 75.68, 75.46, 74.93, 73.34, 72.31, 70.32, 70.19, 78.42, 66.00, 66.1, 55.11.

IR (cm^{-1}): 3030, 2920, 1496, 1452, 1366, 1153, 1071, 1039, 1026, 996, 937, 735, 694, 611, 466.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{62}H_{66}NaO_{11}]^+$ 1009.4497, found 1009.4494.



6

General Procedure A: 139 mg, 73%, $\alpha:\beta$ = 20:1

Data for α anomer **6**:

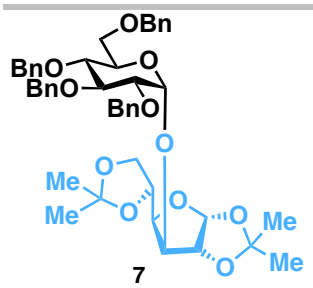
R_f = 0.45 (hexane/AcOEt = 5:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ 7.46 (d, *J* = 10.0 Hz, 2H), 7.41-7.23 (m, 33H), 7.14-7.12 (m, 2H), 7.07 (d, *J* = 10.0 Hz, 2H), 5.04 (d, *J* = 5.0 Hz, 1H), 5.00-4.52 (m, 14H), 3.99 (t, *J* = 10.0 Hz, 1H), 3.89-3.83 (m, 2H), 3.78-3.58 (m, 8H), 3.49-3.46 (m, 1H), 3.25 (t, *J* = 10.0 Hz, 1H), 2.23 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ 138.87, 138.54, 138.48, 138.44, 138.18, 138.08, 138.04, 137.99, 137.75, 132.88, 129.76, 128.43, 128.38, 128.34, 128.22, 127.95, 127.88, 127.80, 127.72, 127.65, 127.62, 127.51, 97.34, 88.45, 86.64, 81.75, 81.09, 80.13, 78.72, 77.64, 75.63, 75.43, 74.94, 73.38, 72.41, 70.18, 68.49, 66.23, 21.07.

IR (cm^{-1}): 3011, 2917, 1637, 1496, 1453, 1360, 1215, 1067, 1027, 803, 696, 665, 471, 422.

HRMS-ESI (m/z):$[M+Na]^+$ calcd. for $[C_{68}H_{70}NaSO_{10}]^+$, 1101.4582, found 1101.4580.



7

General Procedure A: 135mg, 86%, $\alpha:\beta$ = 1:0

Data for α anomer **7**:

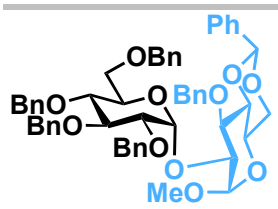
TLC: R_f = 0.45 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ 7.40-7.24 (m, 18H), 7.14 (d, *J* = 6.8 Hz, 2H), 5.89 (d, *J* = 3.6 Hz, 1H), 5.26 (d, *J* = 3.5 Hz, 1H), 4.98 (d, *J* = 10.8 Hz, 1H), 4.83 (dd, *J* = 15.9, 10.7 Hz, 2H), 4.79-4.70 (m, 2H), 4.68 (d, *J* = 3.6 Hz, 1H), 4.63 (d, *J* = 12.1 Hz, 1H), 4.53-4.45 (m, 3H), 4.25 (d, *J* = 2.8 Hz, 1H), 4.15 (dd, *J* = 8.0, 2.8 Hz, 1H), 4.06 (d, *J* = 5.6 Hz, 2H), 3.96 (t, *J* = 9.4 Hz, 1H), 3.84-3.79 (m, 1H), 3.73 (d, *J* = 4.2 Hz, 2H), 3.63 (t, *J* = 9.6 Hz, 1H), 3.58 (dd, *J* = 9.8, 3.5 Hz, 1H), 1.50 (s, 3H, C*H*$_3$), 1.43 (s, 3H), 1.26 (s, 3H), 1.26 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ 138.63, 138.14, 137.92, 137.81, 128.43, 128.37, 128.04, 127.92, 127.86, 127.73, 127.68, 127.54, 111.76, 109.04, 105.17, 97.92, 83.68, 81.48, 81.17, 80.60, 79.94, 77.64, 75.62, 75.29, 73.54, 73.03, 72.32, 71.17, 68.56, 67.03, 26.98, 26.77, 26.12, 25.46.

IR (cm^{-1}): 2986, 2925, 1497, 1454, 1369, 1254, 1209, 1162, 1064, 1041, 1014, 957, 941, 850, 737, 696, 641, 607, 514, 469.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{46}H_{54}NaO_{11}]^+$, 805.3558, found 805.3548.



8

General Procedure A: 124mg, 69%, α:β = 10:1

Data for α anomer **8**:

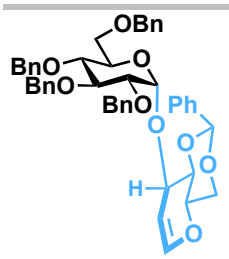
TLC: R_f = 0.40 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ 7.53-6.98 (m, 30H), 5.55 (s, 1H), 4.93 (d, *J* = 3.5 Hz, 1H), 4.88 (d, *J* = 3.5 Hz, 1H), 4.87-4.75 (m, 5H), 4.70 (d, *J* = 12.0 Hz, 1H), 4.52 (d, *J* = 12.0 Hz, 1H), 4.45 (d, *J* = 11.0 Hz, 1H), 4.32-4.26 (m, 2H), 4.15-4.07 (m, 3H), 3.89-3.83 (m, 2H), 3.73-3.57 (m, 4H), 3.50-3.44 (m, 1H), 3.44 (s, 3H), 3.41-3.36 (m, 1H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ 138.82, 138.65, 138.40, 137.97, 137.91, 137.38, 128.89, 128.77, 128.37, 128.27, 128.23, 128.18, 128.01, 127.95, 127.84, 127.76, 127.60, 127.55, 127.43, 125.95, 101.20, 97.20, 94.41, 82.35, 82.10, 79.09, 77.65, 75.66, 75.7, 74.91, 74.26, 73.19, 73.01, 69.89, 69.02, 67.99, 62.25, 54.98.

IR (cm^{-1}): 3031, 2917, 1496, 1451, 1368, 1160, 1100, 1073, 1052, 1025, 998, 969, 902, 850, 781, 730, 694, 643, 611, 574, 539, 491, 459.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{55}H_{58}NaO_{11}]^+$, 917.3871, found 917.3870.



9

General Procedure A: 95 mg, 63%, $\alpha:\beta$ = 7:1

Data for α anomer **9**:

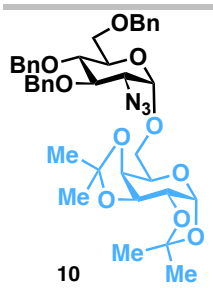
TLC: R_f = 0.40 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ 7.52-7.45 (m, 2H), 7.43-7.30 (m, 18 H), 7.25-7.17 (m, 5H), 6.40 (dd, *J* = 6.0, 1.5 Hz, 1H), 5.59 (s, 1H), 5.49 (d, *J* = 4.0 Hz, 1H), 5.07 (d, *J* = 10.5 Hz, 1H), 4.90 (t, *J* = 6.5 Hz, 2H), 4.80 (dd, *J* = 6.0, 2.0 Hz, 1H), 4.77-4.60 (m, 4H), 4.58-4.51 (m, 2H), 4.41-4.36 (m, 1H), 4.22 (dd, *J* = 8.0, 2.5 Hz, 1H), 4.07 (t, *J* = 9.0 Hz, 1H), 4.03-4.98 (m, 2H), 3.83 (t, *J* = 10.5, Hz, 1H), 3.77 (dd, *J* = 7.0, 3.5 Hz, 1H), 3.70-3.66 (m, 2H), 3.60 (dd, *J* = 9.5, 3.5 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ 144.91, 138.78, 138.12, 137.85, 137.77, 136.99, 129.12, 128.18, 128.10, 127.93, 127.85, 127.82, 127.62, 127.43, 126.11, 102.26, 101.51, 96.38, 81.68, 79.48, 78.95, 77.48, 75.58, 75.04, 73.43, 71.72, 71.28, 70.46, 68.63, 68.38, 68.30.

IR (cm^{-1}): 3029, 2919, 1729, 1637, 1496, 1453, 1374, 1352, 1236, 1095, 1045, 1026, 1010, 955, 916, 877, 836, 736, 694.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{47}H_{48}NaO_9]^+$, 779.3191, found 779.3190.



10

General Procedure A: 104 mg, 73%, $\alpha:\beta$ = 6:1

Data for α anomer **10**:

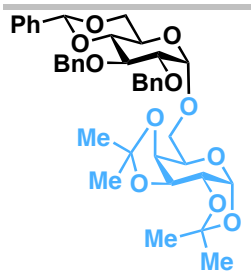
TLC: R_f = 0.45 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ 7.39-7.18 (m, 20H), 5.52 (d, *J* = 5.0 Hz, 1H), 4.99 (d, *J* = 5.0 Hz, 1H, H-1), 4.87 (s, 2H), 4.80 (d, *J* = 11.0 Hz, 1H), 4.70 (s, 1H), 4.65-4.61 (m, 2H), 4.54-4.47 (m, 2H), 4.33-4.30 (m, 2H), 4.02-3.98 (m, 2H), 3.90-3.87 (m, 1H), 3.84-3.70 (m, 4H), 3.67 (dd, *J* = 11.0 Hz, 2.0, 1H), 3.35 (dd, *J* = 11.0, 2.0, 1H), 1.54 (s, 3H), 1.44 (s, 3H), 1.34 (s, 3H), 1.33 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ 138.00, 137.81, 128.53, 128.41, 128.37, 127.96, 127.90, 127.78, 127.72, 127.62, 126.94, 109.24, 108.57, 98.24, 96.21, 79.85, 78.19, 75.24, 74.91, 73.47, 70.79, 70.63, 70.61, 70.53, 68.10, 66.80, 66.19, 63.33, 26.08, 25.93, 24.91, 24.34.

IR (cm^{-1}): 2923, 2105, 1496, 1454, 1381, 1309, 1255, 1209, 1152, 1066, 1044, 1001, 918, 889, 862, 735, 696, 510, 459.

HRMS-ESI (m/z) $[M+Na]^+$ calcd. for $[C_{39}H_{47}NaN_3O_{10}]^+$, 740.3154, found 740.3157.



11

General Procedure A: 117 mg, 85%, $\alpha:\beta$ = 20:1

Data for α anomer **11**:

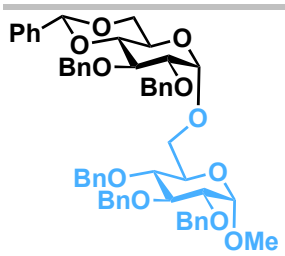
TLC: R_f = 0.4 (hexane/AcOEt = 5:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ 7.52-7.50 (m, 2H), 7.40-7.25 (m, 13H), 5.59 (s, 1H), 5.55 (d, *J* = 5.0 Hz, 1H), 4.96-4.75 (m, 5H), 4.62 (dd, *J* = 7.5, 2.0 Hz, 1H), 4.39-4.29 (m, 3H), 4.08-4.04 (m, 2H), 3.93-3.90 (m, 1H), 3.83-3.77 (m, 2H), 3.73 (t, *J* = 10.5 Hz, 1H), 3.64-3.58 (m, 2H), 1.56 (s, 3H), 1.44 (s, 3H), 1.34 (s, 3H), 1.33 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ 138.82, 138.26, 137.46, 128.82, 128.48, 128.32, 128.22, 128.16, 127.99, 127.89, 127.73, 127.69, 127.47, 126.00, 109.17, 108.61, 101.14, 98.31, 96.28, 82.06, 79.21, 78.51, 75.21, 72.84, 70.77, 70.61, 68.98, 66.84, 65.86, 62.41, 26.13, 26.02, 24.89, 24.57.

IR (cm^{-1}): 2986, 2933, 1496, 1454, 1371, 1255, 1209, 1165, 1087, 1068, 1028, 994, 916, 888, 733, 696, 511, 437.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{39}H_{46}NaO_{11}]^+$, 713.2932, found 713.2926.



12

General Procedure A: 112 mg, 65%, $\alpha:\beta$ = 18:1

Data for α anomer **12**:

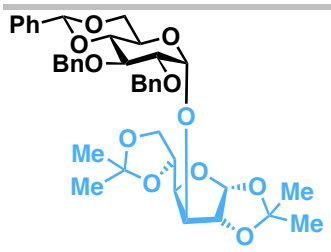
TLC: R_f = 0.45 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ 7.48-7.23 (m, 30H), 5.55 (s, 1H), 4.98-4.87 (m, 5H), 4.82 (d, *J* = 2.0 Hz, 1H, H-1), 4.80 (m, 1H), 4.74-4.62 (5H), 4.58-4.56 (m, 2H), 4.21 (q, *J* = 5.0 Hz, 1H), 4.02-3.96 (m, 2H), 3.89 (td, *J* = 10.0, 4.5 Hz, 1H), 3.80-3.52 (m, 7H), 3.46-3.42 (m, 2H), 3.34 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ 138.81, 138.68, 138.36, 138.14, 137.49, 128.88, 128.41, 128.36, 128.20, 128.01, 127.97, 127.88, 127.73, 127.61, 127.50, 126.05, 101.29, 98.20, 97.97, 82.17, 82.09, 80.06, 79.31, 77.91, 77.72, 75.70, 75.03, 73.35, 72.84, 70.34, 69.08, 66.34, 62.52, 55.19.

IR (cm^{-1}): 3031, 2918, 1497, 1452, 1367, 1327, 1213, 1159, 1088, 1069, 1025, 916, 737, 694, 617, 596, 461.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{55}H_{58}NaO_{11}]^+$, 917.3871, found 917.3865.



13

General Procedure A: 77%, $\alpha:\beta$ > 20:1

Data for α anomer **13**:

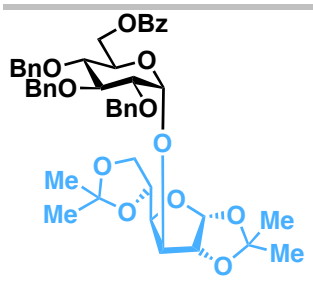
TLC: R_f = 0.45 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ 7.52-7.50 (m, 2H), 7.42-7.27 (m, 13H), 5.93 (d, *J* = 3.5 Hz, 1H), 5.59 (s, 1H), 5.26 (d, *J* = 3.5 Hz, 1H), 4.94 (d, *J* = 11.0 Hz, 1H), 4.84 (d, *J* = 11.0 Hz, 1H), 4.80 (s, 2H,), 4.59 (d, *J* = 3.5 Hz, 1H), 4.54-4.50 (m, 1H), 4.34 (q, *J* = 4.5 Hz, 1H), 4.25 (d, *J* = 2.5 Hz, 1H), 4.10 (dd, *J* = 8.5, 3.0 Hz, 1H), 4.08-3.99 (m, 3H), 3.89-3.84 (m, 1H), 3.76 (t, *J* = 10.5 Hz, 1H), 3.66 (t, *J* = 10.5 Hz, 1H), 3.60 (dd, *J* = 11.5, 3.5 Hz, 1H), 1.51 (s, 3H), 1.43 (s, 3H), 1.32 (s, 3H), 1.27 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ 138.56, 138.05, 137.16, 128.95, 128.34, 128.29, 127.92, 127.71, 127.60, 125.86, 111.93, 109.13, 105.16, 101.2, 98.78, 84.05, 82.20, 81.17, 80.29, 79.20, 78.11, 75.22, 73.68, 72.06, 68.94, 67.12, 63.34, 27.08, 26.81, 26.31, 25.46.

IR (cm^{-1}): 2986, 2934, 1732, 1497, 1454, 1371, 1255, 1212, 1150, 1071, 1017, 913, 841, 734, 696, 678, 655, 507, 457.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{39}H_{46}NaO_{11}]^+$, 713.2932, found 713.2935.



14

General Procedure A: 131 mg, 82%, $\alpha:\beta$ > 20:1

Data for α anomer **14**:

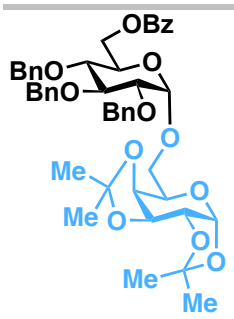
TLC R_f = 0.45 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ 7.98-7.96 (m, 2H), 7.48 (t, *J* = 6.0 Hz, 1H), 7.36-7.17 (m, 16H), 5.82 (d, *J* = 4.0 Hz, 1H), 5.20 (d, *J* = 3.5 Hz, 1H), 4.92 (d, *J* = 10.5 Hz, 1H), 4.85 (d, *J* = 10.5 Hz, 1H), 4.75 (t, *J* = 11.0 Hz, 1H), 4.64-4.62 (m, 1H), 4.55-4.51 (m, 3H), 4.44-4.38 (m, 2H), 4.20 (d, *J* = 3.0 Hz, 1H), 4.03-3.92 (m, 5H), 3.53-3.48 (m, 2H), 1.41 (s, 3H), 1.32 (s, 3H), 1.16 (s, 3H), 1.14 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ 166.30, 138.38, 138.00, 137.48, 133.08, 129.78, 129.58, 128.45, 128.41, 128.35, 128.23, 128.10, 127.97, 127.73, 127.45, 111.87, 109.19, 105.17, 97.55, 84.02, 81.41, 81.22, 80.21, 80.08, 77.82, 75.80, 75.59, 73.09, 72.18, 69.88, 67.16, 63.76, 27.07, 26.82, 26.18, 25.48.

IR (cm^{-1}): 2986, 2932, 1719, 1602, 1497, 1453, 1371, 1335, 1273, 1212, 1145, 1065, 1026, 841, 735, 711, 696, 459, 430.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{46}H_{52}NaO_{12}]^+$, 819.3351, found 819.3351.



15

General Procedure A: 127 mg, 80%, $\alpha:\beta$ > 20:1

Data for α anomer **15**:

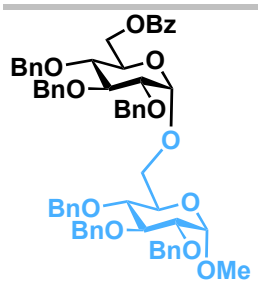
TLC: R_f = 0.40 (hexane/AcOEt = 5:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ 8.03-8.02 (m, 2H), 7.56-7.22 (18H), 5.55 (d, *J* = 5.0 Hz, 1H), 5.08 (d, *J* = 10.5 Hz, 1H), 5.01 (d, *J* = 3.5 Hz, 1H), 4.92 (d, *J* = 11.0 Hz, 1H), 4.85 (d, *J* = 11.0 Hz, 1H), 4.79 (d, *J* =11.0 Hz, 1H), 4.73 (d, *J* = 11.0 Hz, 1H), 4.64-4.59 (m, 2H), 4.55 (d, *J* = 2.5 Hz, 1H), 4.33-4.32 (m, 2H), 4.14-4.07 (m, 3H), 3.86-3.77 (m, 2H), 3.68 (m, 2H), 1.56 (s, 3H), 1.45 (s, 3H), 1.34 (s, 3H), 1.32 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ 166.19, 138.58, 138.17, 137.83, 132.94, 129.89, 129.64, 128.39, 128.32, 128.06, 127.99, 127.80, 127.74, 127.65, 109.20, 108.56, 96.72, 96.27, 81.89, 79.96, 77.47, 75.80, 75.00, 72.35, 70.87, 70.62, 70.53, 68.77, 66.55, 65.78, 63.45, 26.10, 26.01, 24.88, 24.59.

IR (cm^{-1}): 2917, 1719, 1602, 1497, 1453, 1381, 1337, 1273, 1207, 1164, 1066, 1026, 998, 918, 888, 735, 711, 696, 672, 512, 486, 463, 420.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{46}H_{52}NaO_{12}]^+$, 819.3351, found 819.3352.



16

General Procedure A: 132 mg, 66%, $\alpha:\beta$ = 16:1

Data for α anomer **16**:

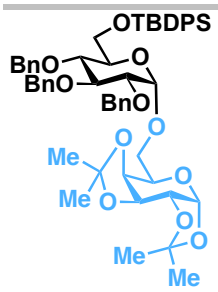
TLC: R_f = 0.40 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ 7.90-7.88 (m, 2H), 7.47-7.44 (m, 1H), 7.33-7.14 (m, 32H), 4.90-4.81 (m, 5H), 4.73 (dd, *J* = 10.5, 4.5 Hz, 1H), 4.63-4.40 (m, 8H), 4.31 (dd, *J* = 12.0, 4.5 Hz, 1H), 3.95-3.88 (m, 3H), 3.74-3.68 (m, 2H), 3.63-3.61 (m, 1H), 3.54-3.50 (m, 2H), 3.47 (dd, *J* = 10.0, 4.0 Hz, 1H), 3.33 (dd, *J* = 9.5, 3.5 Hz, 1H), 3.27 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ 166.16, 138.79, 138.49, 138.30, 138.12, 137.96, 132.99, 129.95, 129.61, 128.39, 128.17, 127.96, 127.90, 127.82, 127.76, 127.71, 127.67, 127.57, 97.88, 96.87, 82.07, 81.67, 80.15, 80.12, 77.81, 77.52, 75.71, 75.00, 73.31, 72.41, 70.34, 68.85, 65.98, 63.41, 55.15.

IR (cm^{-1}): 3030, 2916, 1719, 1602, 1496, 1452, 1358, 1272, 1159, 1088, 1068, 1025, 912, 733, 711, 695, 611, 531, 460.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{62}H_{64}NaO_{12}]^+$, 1023.4290, found 1023.4288.



17

General Procedure A 115 mg: 62%, $\alpha:\beta$ = 7:1

Data for α anomer **17**:

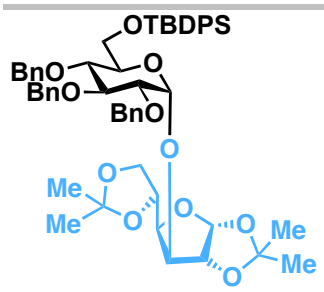
R_f = 0.45 (hexane/AcOEt = 5:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ 7.74-7.70 (m, 4H), 7.45-7.40 (m, 4H), 7.39-7.29 (m, 16H), 7.23-7.20 (m, 1H), 5.54 (d, *J* = 5.0 Hz, 1H), 5.09 (d, *J* = 3.5 Hz, 1H), 5.02 (d, *J* = 10.5 Hz, 1H), 4.94 (d, *J* = 10.5 Hz, 1H), 4.86-4.83 (m, 2H), 4.75-4.69 (m, 2H), 4.63 (dd, *J* = 8.0, 2.5 Hz, 1H), 4.38 (dd, *J* = 8.0, 2.0 Hz, 1H), 4.33 (q, *J* = 2.5 Hz, 1H), 4.08-4.04 (m, 2H), 3.99-3.90 (m, 2H), 3.83-3.70 (m, 3H), 3.63 (dd, *J* = 9.5, 3.5 Hz, 1H), 1.54 (s, 3H), 1.49 (s, 3H), 1.34 (s, 6H), 1.08 (s, 9H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ 138.85, 138.48, 138.37, 135.89, 135.80, 135.59, 133.67, 133.23, 129.54, 129.50, 128.34, 128.31, 128.21, 128.09, 127.83, 127.69, 127.62, 127.57, 127.49, 109.13, 108.49, 96.28, 82.04, 80.21, 77.58, 75.9, 75.79, 75.07, 72.13, 71.48, 70.77, 70.64, 70.62, 65.46, 65.32, 62.64, 26.80, 26.11, 26.05, 24.88, 24.60, 19.30.

IR (cm^{-1}): 2930, 1496, 1454, 1428, 1381, 1255, 1210, 1162, 1067, 998, 918, 823, 735, 696, 648, 612, 504, 466.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{55}H_{66}NaSiO_{11}]^+$, 953.4267, found 953.4278.



18

General Procedure A: 116 mg, 63%, $\alpha:\beta$ > 20:1

Data for α anomer **18**:

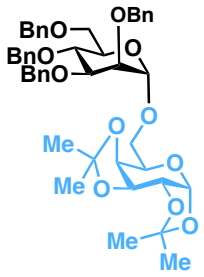
TLC: R_f = 0.45 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ 7.73-7.70 (m, 4H), 7.43-7.27 (m, 19H), 7.15-7.13 (m, 2H), 5.84 (d, *J* = 3.5 Hz, 1H, H-1), 5.26 (d, *J* = 3.0 Hz, 1H), 4.97 (d, *J* = 11.0 Hz, 1H), 4.90 (d, *J* = 10.5 Hz, 1H), 4.84-4.80 (m, 2H), 4.74 (d, *J* = 12.0 Hz, 1H), 4.61-4.58 (m, 2H), 4.52-4.51 (m, 1H), 4.25 (d, *J* = 3.0 Hz, 1H), 4.13 (dd, *J* = 8.0, 2.5 Hz, 1H), 4.08-4.01 (m, 2H), 3.99 (t, *J* = 9.0 Hz, 1H), 3.91 (d, *J* = 3.0 Hz, 1H), 3.76-3.74 (m, 1H), 3.67 (t, *J* = 9.0 Hz, 1H), 3.57 (dd, *J* = 10.0, 3.5 Hz, 1H), 1.47 (s, 3H), 1.45 (s, 3H), 1.27 (s, 3H), 1.20 (s, 3H), 1.07 (s, 9H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ 138.59 138.27, 137.97, 135.80, 135.66, 133.43, 133.17, 129.65, 129.60, 128.45, 128.41, 128.38, 128.04, 128.02, 127.82, 127.69, 127.64, 127.59, 127.41, 111.82, 105.13, 97.78, 83.83, 81.49, 81.22, 80.43, 80.27, 77.69, 75.77, 75.40, 73.16, 72.31, 67.07, 62.91, 29.68, 27.06, 26.84, 26.26, 25.47, 19.27.

IR (cm^{-1}): 2930, 1497, 1454, 1427, 1371, 1255, 1213, 1145, 1066, 1036, 957, 882, 842, 823, 736, 696, 613, 503, 489.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{55}H_{66}NaSiO_{11}]^+$, 953.4267, found 953.4277.



19

General Procedure B: mg, %, $\alpha:\beta$ = 2:1

Data for α anomer: **19**

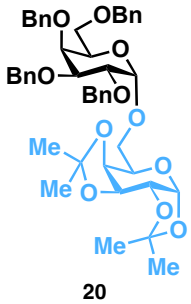
R_f = 0.40 (hexane/AcOEt 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ 7.41 – 7.23 (m, 18H), 7.19 – 7.14 (m, 2H), 5.53 (d, J = 4.9 Hz, 1H), 5.03 (d, J = 1.9 Hz, 1H), 4.88 (d, J = 10.7 Hz, 1H), 4.75 (d, J = 3.6 Hz, 2H), 4.70 (d, J = 12.2 Hz, 1H), 4.61 (dd, J = 8.1, 2.6 Hz, 3H), 4.53 (t, J = 11.0 Hz, 2H), 4.32 (dd, J = 5.1, 2.4 Hz, 1H), 4.17 (dd, J = 7.9, 1.9 Hz, 1H), 4.03 (t, J = 9.1 Hz, 1H), 3.98 (td, J = 6.7, 1.9 Hz, 1H), 3.92 (dd, J = 9.4, 3.1 Hz, 1H), 3.87 – 3.77 (m, 4H), 3.76 – 3.68 (m, 2H), 1.52 (s, 3H), 1.44 (s, 3H), 1.34 (s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ 138.74, 138.64, 138.59, 138.52, 128.43, 128.41, 128.15, 127.92, 127.75, 127.69, 127.65, 127.60, 127.55, 109.48, 108.69, 97.40, 96.48, 80.19, 75.23, 74.99, 74.71, 73.46, 72.46, 72.22, 71.05, 70.81, 70.74, 69.25, 65.50, 65.39, 26.28, 26.12, 25.05, 24.72.

IR (cm^{-1}): 3029, 2986, 2910, 1496, 1453, 1381, 1370, 1254, 1209, 1167, 1097, 1066, 1027, 1000, 901, 862, 804, 733, 696, 646, 602, 511, 459

HRMS-ESI (m/z): $[M+H]^+$ calcd. for $[C_{46}H_{55}O_{11}]^+$ 783.3700, found 783.3649.



20

General Procedure B: 137 mg, 88%, $\alpha:\beta$ = 20:1

Data for α anomer **20**:

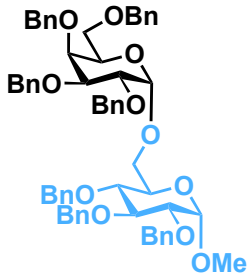
TLC: R_f = 0.45 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ 7.38 (ddd, *J* = 7.9, 3.5, 1.5 Hz, 3H), 7.35-7.24 (m, 17H), 5.51 (d, *J* = 5.0 Hz, 1H), 5.01 (d, *J* = 3.7 Hz, 1H), 4.94 (d, *J* = 11.5 Hz, 1H), 4.83 (d, *J* = 11.7 Hz, 1H), 4.79-4.70 (m, 3H), 4.61-4.54 (m, 2H), 4.48 (d, *J* = 11.8 Hz, 1H), 4.42 (d, *J* = 11.8 Hz, 1H), 4.34-4.27 (m, 2H), 4.09-3.99 (m, 4H), 3.96 (dd, *J* = 10.1, 2.8 Hz, 1H), 3.79 (dd, *J* = 10.5, 6.4 Hz, 1H), 3.74 (dd, *J* = 10.5, 7.1 Hz, 1H), 3.58 (dd, *J* = 9.2, 7.5 Hz, 1H), 3.52 (dd, *J* = 9.2, 5.7 Hz, 1H), 1.52 (s, 3H), 1.43 (s, 3H), 1.33 (s, 3H), 1.30 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ 138.91, 138.73, 138.05, 128.35, 128.28, 128.20, 128.16, 127.79, 127.70, 127.63, 127.46, 127.44, 127.39, 109.15, 108.48, 97.53 96.30, 78.96, 76.39, 74.91, 74.74, 73.36, 73.02, 72.63, 70.85, 70.61, 69.12, 68.65, 66.30, 65.78, 26.13, 26.02, 24.91, 24.57.

IR (cm^{-1}): 2917, 1496, 1453, 1370, 1308, 1254, 1209, 1165, 1095, 1066, 998, 917, 889, 865, 734, 695, 609, 511, 461.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{46}H_{54}NaO_{11}]^+$, 805.3558, found 805.3549.



21

General Procedure B: 146 mg, 74%, $\alpha:\beta$ > 20:1

Data for α anomer **21**:

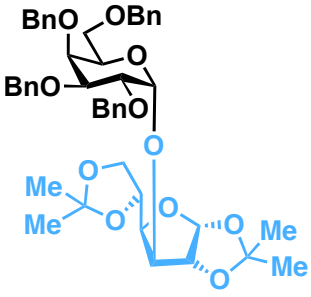
TLC: R_f = 0.40 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ 7.35-7.22 (m, 35H), 4.98 (d, *J* = 3.5 Hz, 1H), 4.94 (t, *J* = 11.5 Hz, 2H), 4.84 (d, *J* = 11.0 Hz, 1H), 4.81-4.77 (m, 2H), 4.74-4.68 (m, 4H), 4.58 (d, *J* = 11.0 Hz, 2H), 4.55-4.52 (m, 2H), 4.42 (d, *J* = 11.5 Hz, 1H), 4.35 (d, *J* = 11.5 Hz, 1H), 4.02 (dd, *J* = 9.5, 3.5 Hz, 1H), 3.98-3.88 (m, 4H), 3.81-3.74 (m, 2H), 3.72 (d, *J* = 11.5 Hz, 1H), 3.58 (t, *J* = 9.5 Hz, 1H), 3.51-3.47 (m, 2H), 3.41 (dd, *J* = 9.5, 3.5 Hz, 1H), 3.29 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ 138.86, 138.75, 138.70, 138.40, 138.20, 138.04, 128.38, 128.33, 128.28, 128.22, 128.19, 127.95, 127.79, 127.68, 127.63, 127.49, 127.40, 127.34, 97.91, 97.86, 82.06, 80.15, 78.24, 77.98, 76.52, 75.67, 75.08, 74.98, 74.73, 73.32, 72.79, 72.51, 70.28, 69.36, 68.91, 66.39, 55.01.

IR (cm^{-1}): 3030, 2917, 1721, 1496, 1453, 1355, 1278, 1208, 1132, 1091, 1025, 911, 732, 694, 607, 458.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{62}H_{66}NaO_{11}]^+$, 1009.4497, found 1009.4494.



22

General Procedure B: 127 mg, 81%, $\alpha:\beta$ > 20:1

Data for α anomer **22**:

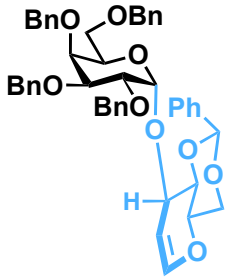
R_f = 0.40 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ 7.40-7.24 (m, 20H), 5.87 (d, *J* = 3.5 Hz, 1H), 5.21 (d, *J* = 4.0 Hz, 1H), 4.96 (d, *J* = 11.5 Hz, 1H), 4.84 (d, *J* = 11.5 Hz, 1H), 4.79 (d, *J* = 11.5 Hz, 1H), 4.76-4.70 (m, 3H), 4.57 (d, *J* = 11.5 Hz, 1H), 4.53-4.44 (m, 2H), 4.42 (d, *J* = 12.0 Hz, 1H), 4.18 (d, *J* = 5.0 Hz, 2H), 4.04 (tdd, *J* = 13.5, 8.5, 4.5 Hz, 3H), 3.94 (d, *J* = 4.0 Hz, 2H), 3.87 (dd, *J* = 10.0, 2.5 Hz, 1H), 3.58 (dd, *J* = 9.5, 6.5 Hz, 1H), 3.47 (dd, *J* = 9.5, 5.5 Hz, 1H), 1.48 (s, 3H), 1.42 (s, 3H), 1.24 (s, 3H), 1.19 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ 138.23, 137.91, 137.81, 137.16, 128.64, 128.58, 128.55, 128.42, 128.30, 128.16, 127.82, 127.75, 127.65, 127.55, 111.79, 105.64, 101.30, 86.00, 82.82, 80.24, 79.07, 75.62, 75.07, 74.86, 74.67, 74.37, 73.54, 72.41, 70.86, 69.83, 69.55, 64.70, 26.79, 26.06.

IR (cm^{-1}): 2915, 1496, 1453, 1355, 1276, 1209, 1132, 1090, 1049, 1025, 912, 781, 733, 694, 633, 620, 605, 508, 455, 430.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{46}H_{54}NaO_{11}]^+$, 805.3558, found 805.3548.



23

General Procedure B: 104 mg, 69%, $\alpha:\beta$ > 20:1

Data for α anomer **23**:

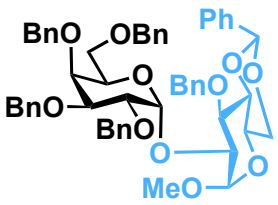
TLC: R_f = 0.45 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ7.42-7.11 (m, 25H), 6.33 (dd, *J* = 6.0, 1.0 Hz, 1H), 5.53 (s, 1H), 5.42 (d, *J* = 3.5 Hz, 1H), 5.29 (s, 1H), 4.93 (d, *J* = 11.5 Hz, 1H), 4.86 (*J* = 11.5 Hz, 1H), 4.76-4.62 (m, 4H), 4.55 (d, *J* = 11.5 Hz, 1H), 4.44 (d, *J* = 11.5 Hz, 1H), 4.37-4.30 (m, 2H), 4.23-4.20 (m, 1H), 4.14-3.89 (m, 6H), 3.78 (t, *J* = 10.0 Hz, 1H), 3.56-3.51 (m, 1H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ144.97, 138.91, 138.63, 138.45, 137.96, 137.12, 129.09, 128.37, 128.30, 128.18, 127.89, 127.78, 127.71, 127.52, 127.46, 127.38, 127.33, 126.10, 102.52, 101.35, 97.09, 79.68, 79.42, 78.91, 78.68, 75.92, 75.08, 74.76, 74.48, 73.50, 73.23, 72.28, 71.52, 69.67, 69.17, 68.81, 68.36.

IR (cm^{-1}): 3029, 2919, 1729, 1637, 1496, 1453, 1374, 1352, 1236, 1095, 1045, 1026, 1010, 955, 916, 877, 836, 736, 694.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{47}H_{48}NaO_9]^+$, 779.3191, found 779.3219.



24

General Procedure B: 130 mg, 73%, $\alpha:\beta$ > 20:1

Data for α anomer **24**:

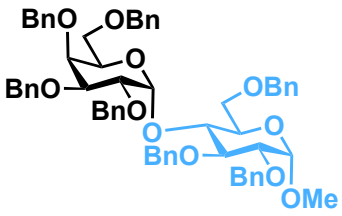
TLC: R_f = 0.35 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ 7.59-7.08 (m, 30H), 5.52 (s, 1H), 4.96 (d, *J* = 3.2 Hz, 1H), 4.93-4.77 (m, 5H), 4.73-4.66 (m, 3H), 4.52 (d, *J* = 11.4 Hz, 1H), 4.32-4.23 (m, 4H), 4.09 (t, *J* = 9.4 Hz, 1H), 4.03 (dd, *J* = 10.5, 2.9 Hz, 1H), 3.95 (dd, *J* = 10.1, 2.4 Hz, 1H), 3.88-3.75 (m, 3H), 3.70 (t, *J* = 10.1 Hz, 1H), 3.54 (t, *J* = 9.5 Hz, 1H), 3.48 (dd, *J* = 9.5, 6.5 Hz, 1H), 3.42 (s, 3H), 3.34 (dd, *J* = 9.5, 6.8 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ 138.86, 138.68, 138.42, 137.40, 128.87, 128.35, 128.25, 128.15, 128.09, 127.88, 127.75, 127.56, 127.48, 127.44, 127.38, 125.98, 101.17, 97.36, 94.88, 82.39, 78.83, 75.94, 75.34, 74.98, 74.72, 73.94, 73.08, 72.89, 72.78, 69.04, 68.95, 68.84, 62.29, 55.06.

IR (cm^{-1}): 3031, 2917, 1496, 1451, 1368, 1347, 1329, 1213, 1160, 1131, 1100, 1073, 1052, 1025, 998, 969, 902, 850, 781, 694, 611, 459.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{55}H_{58}NaO_{11}]^+$, 917.3871, found 917.3869.



25

General Procedure B: 62%, α:β = 20:1

Data for α anomer **25**:

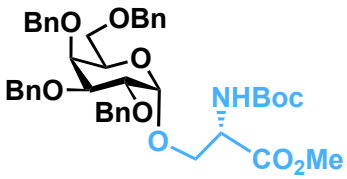
TLC: R_f = 0.45 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ7.31-7.17 (m, 35H), 5.76 (d, *J* = 3.5 Hz, 1H), 4.97 (d, *J* = 11.5 Hz, 1H), 4.86 (d, *J* = 11.5 Hz, 1H), 4.81 (d, *J* = 11.5 Hz, 1H), 4.71-4.65 (m, 4H), 4.60 (d, *J* = 11.5 Hz, 1H), 4.57-4.51 (m, 4H), 4.41 (d, *J* = 11.5 Hz, 1H), 4.29 (d, *J* = 11.5 Hz, 1H), 4.22 (d, *J* = 11.5 Hz, 1H), 4.07 (t, *J* = 9.5 Hz, 1H), 4.00-3.96 (m, 2H), 3.93-3.84 (m, 3H), 3.81 (dd, *J* = 10.0, 2.5 Hz, 1H), 3.70 (d, *J* = 11.0, 3.5 Hz, 1H), 3.64 (dd, *J* = 11.0, 2.5 Hz, 1H), 3.55 (dd, *J* = 9.5, 3.5 Hz, 1H), 3.49-3.39 (m, 2H), 3.37 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ138.94, 128.57, 138.32, 138.23, 137.96, 128.35, 128.28, 128.22, 128.16, 127.83, 127.72, 127.64, 127.53, 127.46, 127.40, 127.32, 126.98, 126.68, 97.68, 97.43, 81.97, 80.12, 79.12, 75.59, 74.72, 74.60, 74.29, 73.74, 73.37, 73.32, 73.02, 72.72, 69.82, 69.43, 69.39, 68.64, 55.04.

IR (cm^{-1}): 3029, 2918, 1723, 1496, 1453, 1361, 1273, 1207, 1093, 1040, 1027, 911, 732, 694, 607, 548, 461.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{62}H_{66}NaO_{11}]^+$, 1009.4497, found 1009.4498.



26

General Procedure B: 109 mg, 74%, α:β = 20:1

Data for α anomer **26**:

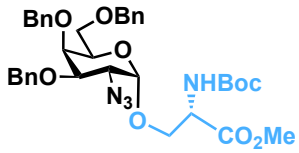
TLC: R_f = 0.3 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ 7.33-7.27 (m, 20H), 5.68 (d, *J* = 8.5 Hz, 1H), 4.84 (d, *J* = 11.0 Hz, 1H), 4.73-4.62 (m, 4H), 4.54 (d, *J* = 12.0 Hz, 1H), 4.48-4.42 (m, 2H), 4.34-4.32 (m, 2H), 4.03 (dd, *J* = 11.0, 3.5 Hz, 1H), 3.94 (dd, *J* = 10.5, 4.0 Hz, 1H), 3.88-3.85 (m, 2H), 3.78 (dd, *J* = 10.5, 3.0 Hz, 1H), 3.71 (dd, *J* = 11.0, 2.5 Hz, 1H), 3.56 (s, 3H), 3.46-3.45 (m, 2H), 1.34 (s, 9H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ170.87, 155.55, 138.65, 138.51, 138.49, 137.88, 128.33, 128.28, 128.18, 127.8, 127.73, 127.64, 127.57, 127.4, 127.42, 127.39, 99.23, 79.82, 78.61, 76.34, 74.72, 74.67, 73.41, 73.12, 72.97, 70.26, 69.71, 68.60, 54.13, 52.30, 28.27.

IR (cm^{-1}): 3029, 2928, 1748, 1713, 1496, 1453, 1391, 1365, 1348, 1300, 1246, 1209, 1157, 1093, 1054, 1027, 911, 734, 696, 601, 460.

HRMS-ESI (m/z): $[M+H]^+$ calcd. for $[C_{43}H_{52}NO_{10}]^+$, 742.3591, found 742.3588.



27

General Procedure B: 112 mg, 83%, α:β > 20:1

Data for α anomer **27**:

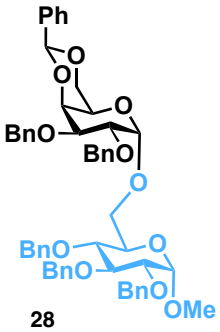
TLC: R_f = 0.45 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ 7.35-7.24 (m, 15H), 5.56 (d, *J* = 8.5 Hz, 1H), 4.86 (dd, *J* = 7.5, 4.0 Hz, 1H), 4.74 (d, *J* = 11.5 Hz, 1H), 4.67 (d, *J* = 11.5 Hz, 1H), 4.54-4.51 (m, 3H), 4.44 (d, *J* = 12.0 Hz, 1H), 4.06-4.02 (m, 2H), 3.96 (t, *J* = 6.5 Hz, 1H), 3.93-3.88 (m, 3H), 3.82 (dd, *J* = 11.0, 3.5 Hz, 1H), 3.76 (s, 3H), 3.61-3.54 (m, 2H), 1.45 (s, 9H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ170.60, 155.42, 138.15, 137.73, 137.42, 128.50, 128.42, 128.27, 128.09, 127.91, 127.82, 127.72, 99.46, 80.07, 74.82, 73.51, 73.06, 72.11, 69.90, 69.78, 68.35, 59.54, 54.03, 52.59, 28.28.

IR (cm^{-1}): 3367, 2930, 2162, 2151, 2108, 1747, 1713, 1496, 1454, 1365, 1348, 1248, 1210, 1157, 1095, 1051, 984, 735, 696, 654, 461.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{36}H_{44}NaN_4O_9]^+$, 699.3001, found 699.2992.



28

General Procedure B: 134 mg, 75%, α:β = 20:1

Data for α anomer **28**:

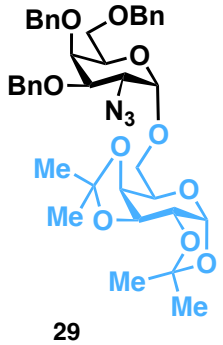
TLC: R_f = 0.35 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ7.53-7.51(m, 2H), 7.39-7.21 (m, 28H), 5.46 (s, 1H), 5.06 (d, *J* = 3.5 Hz, 1H), 4.99 (d, *J* = 10.5 Hz, 1H), 4.90 (d, *J* = 11.5 Hz, 1H), 4.82-4.68 (m, 7H), 4.61-4.53 (m, 3H), 4.13-4.10 (m, 2H), 4.07 (dd, *J* = 10.0, 3.5 Hz, 1H), 3.99 (t, *J* = 9.5 Hz, 1H), 3.95 (dd, *J* = 10.5, 7.5 Hz, 1H), 3.88 (d, *J* = 10.0, 3.5 Hz, 1H), 3.78-3.69 (m, 3H), 3.58 (t, *J* = 10.0, 3.5 Hz, 1H), 3.49 (s, 1H), 3.45 (dd, *J* = 9.5, 3.5 Hz, 1H), 3.3 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ138.85, 138.78, 138.66, 138.50, 138.15, 137.83, 128.82, 128.39, 128.34, 128.25, 128.06, 127.96, 127.82, 127.73, 127.56, 127.50, 127.48, 127.37, 126.34, 101.03, 98.35, 97.86, 82.09, 80.12, 77.99, 75.66, 75.61, 74.90, 74.80, 73.32, 72.81, 71.84, 70.11, 69.34, 66.43, 62.58, 54.97.

IR (cm^{-1}): 3030, 2917, 1496, 1453, 1358, 1249, 1211, 1130, 1069, 1049, 1026, 996, 917, 831, 795, 734, 694, 614, 558, 505, 461.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{55}H_{58}NaO_{11}]^+$, 917.3871, found 917.3867.



29

General Procedure B: 116 mg, 81%, α:β > 20:1

Data for α anomer **29**:

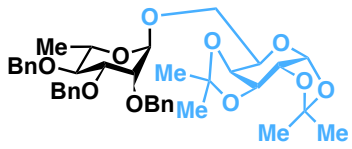
TLC: R_f = 0.40 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ7.36-7.28 (m, 15H), 5.52 (d, *J* = 5.0 Hz, 1H), 4.97 (d, *J* = 4.0 Hz, 1 H), 4.88 (d, *J* =11.5 Hz, 1H), 4.74 (d, *J* = 11.5 Hz, 1H), 4.68 (d, *J* = 11.0 Hz, 1H), 4.61 (dd, *J* = 7.5, 2.5 Hz, 1H), 4.56-4.43 (m, 3H), 4.32-4.29 (m, 2H), 4.10-3.99 (m, 4H), 3.85-3.81 (m, 2H), 3.70 (dd, *J* = 10, 5.5 Hz, 1H), 3.62 (t, *J* = 9.5 Hz, 1H), 3.55 (dd, *J* = 9.0, 5.5 Hz, 1H), 1.54 (s, 3H), 1.42 (s, 3H), 1.34 (s, 3H), 1.33 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ138.32, 137.88, 137.60, 128.75, 128.44, 128.36, 128.21, 128.07, 127.80, 127.70, 127.61, 109.24, 108.53, 98.33, 96.22, 74.77, 73.32, 72.08, 70.93, 70.56, 69.29, 68.33, 66.87, 66.45, 59.67, 26.03, 25.90, 24.93, 24.34.

IR (cm^{-1}): 2923, 2107, 1725, 1496, 1454, 1381, 1309, 1255, 1209, 1165, 1115, 1095, 1066, 1003, 918, 889, 865, 734, 696, 650, 599, 506, 462, 429.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{39}H_{47}NaN_3O_{10}]^+$, 740.3154, found 740.3143.



30

General Procedure B: 111 mg, 82%, α:β > 20:1

Data for α anomer **30**:

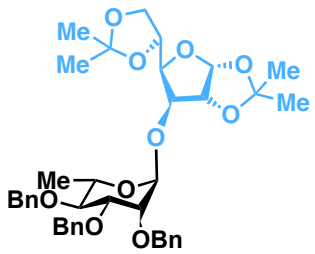
TLC: R_f = 0.45 (hexane/AcOEt = 5:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ7.43–7.28 (m, 15H), 5.55 (d, *J* = 5.0 Hz, 1 H), 4.96 (d, *J* = 11Hz, 1 H), 4.93 (d, *J* = 1.5 Hz, 1H), 4.77 (s, 2H), 4.69-4.60 (m, 4H), 4.33 (q, *J* = 2.5 Hz, 1H), 4.19 (dd, *J* = 7.5, 1.5 Hz, 1H), 3.95-3.78 (m, 5H), 3.66 (t, *J* = 9.5 Hz, 1H), 3.61 (dd, *J* = 7.0, 3.5 Hz, 1H), 1.55 (s, 3H), 1.47 (s, 3H), 1.36 (s, 6H), 1.35 (d, *J* = 6.0 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ138.65, 138.50, 138.33, 128.19, 127.96, 127.80, 127.52, 127.46, 127.35, 109.18, 108.43, 97.90, 96.14, 79.85, 75.11, 74.63, 72.48, 71.82, 71.04, 70.50, 70.43, 67.96, 67.14, 65.81, 26.05, 25.88, 24.87, 24.30.

IR (cm^{-1}): 2986, 2932, 1496, 1454, 1381, 1254, 1209, 1167, 1063, 1027, 1000, 917, 900, 801, 734, 696, 647, 511, 457.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{39}H_{48}NaO_{10}]^+$, 699.3140, found 699.3129.



31

General Procedure B: 105 mg, 78%, α:β > 20:1

Data for α anomer **31**:

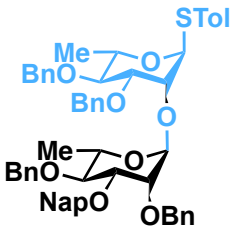
TLC: R_f = 0.45 (hexane/AcOEt = 5:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ7.40-7.27 (m, 15H), 5.77 (d, *J* = 4.0 Hz, 1H), 4.93 (d, *J* = 11.0 Hz, 1H), 4.86 (d, *J* = 12.0 Hz, 1H), 4.76 (d, *J* = 1.5 Hz, 1H), 4.67-4.64 (m, 4H), 4.28 (d, *J* = 3.5 Hz, 1H), 4.19-4.15 (m, 2H), 4.09-4.06 (m, 2H), 3.99-3.96 (m, 1H), 3.90 (dd, *J* = 8.5, 6.0 Hz, 1H), 3.75 (dd, *J* = 9.0, 3.0 Hz, 1H), 3.69 (m, 1H), 3.60 (t, *J* = 10.0 Hz, 1H), 1.49 (s, 3H), 1.38 (s, 3H), 1.31 (s, H), 1.28 (d, *J* = 6.0 Hz, 3H), 1.28 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ138.94, 138.37, 138.18, 128.37, 128.32, 128.18, 128.16, 127.78, 127.71, 127.58, 127.49, 127.35, 111.87, 109.16, 105.14, 95.86, 81.68, 80.90, 80.22, 79.78, 76.61, 75.20, 74.82, 73.30, 72.48, 71.89, 68.50, 67.76, 26.70, 26.15, 25.19, 17.62.

IR (cm^{-1}): 3029, 2916, 1496, 1453, 1371, 1355, 1208, 1131, 1091, 1072, 1025, 911, 847, 733, 695, 532, 459.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{39}H_{48}NaO_{10}]^+$, 699.3140, found 699.3124.



32

General Procedure B: 141 mg, 78%, α:β = 20:1

Data for α anomer **32**:

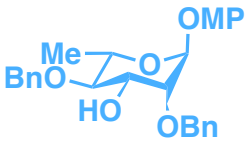
TLC: R_f = 0.45 (hexane/AcOEt = 5:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ7.69-7.60 (m, 4H), 7.34-7.30 (m, 2H), 7.25-7.09 (m, 23H), 6.98 (d, *J* = 10.0 Hz, 2H), 5.23 (d, *J* = 1.0 Hz, 1H), 5.00 (d, *J* = 1.0 Hz, 1H), 4.86 (m, 1H), 4.69-4.62 (m, 3H), 4.55-4.51 (m, 4H), 4.46 (t, *J* = 12.5 Hz, 1H), 4.37-4.35 (m, 1H), 4.13 (s, 1H), 3.98-3.94 (m, 1H), 3.82 (dd, *J* = 9.5, 3.0 Hz, 1H), 3.74-3.70 (m, 2H), 3.67-3.64 (m, 1H), 3.54 (t, *J* = 9.0 Hz, 1H), 3.17 (t, *J* = 9.0 Hz, 1H), 2.2 (s, 3H), 1.17 (d, *J* = 6.0 Hz, 3H), 1.07 (d, *J* = 6.0 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ138.51, 138.34, 138.22, 137.91, 137.40, 136.02, 133.20, 132.86, 131.71, 130.62, 129.78, 128.48, 128.35, 128.31, 128.19, 127.91, 127.68, 127.72, 127.60, 127.57, 127.51, 126.31, 125.97, 125.76, 99.41, 87.61, 80.58, 80.30, 80.10, 79.05, 75.91, 75.29, 75.19, 75.00, 74.87, 72.70, 72.46, 72.20, 69.16, 68.81, 68.57, 68.24, 21.03, 17.91, 17.71.

IR (cm^{-1}): 3029, 2910, 1731, 1602, 1494, 1453, 1364, 1282, 1208, 1086, 1060, 1026, 911, 843, 810, 733, 696, 620, 474.

HRMS-ESI (m/z): $[M+Na]^+$ calcd. for $[C_{58}H_{60}NaSO_8]^+$, 939.3901, found 939.3904.



33

Prepared according to the method of P.B. Mukhopadhyay[1]

1.35g, 68% yield

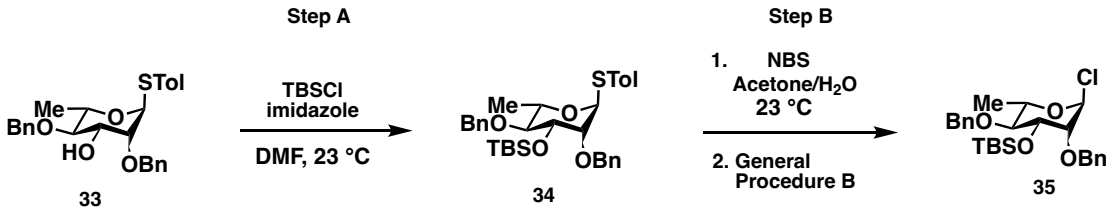
Data for compound **33**:

TLC: R_f = 0.45 (hexane/AcOEt = 1:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ7.38-7.27 (m, 5H), 6.99-6.97 (m, 2H), 6.85-6.82 (m, 2H), 5.40 (s, 1H), 4.79-4.74 (m, 2H), 4.14-4.10 (m, 2H), 3.90-3.86 (dq, 1H, *J* = 9.4, 6.1 Hz), 3.78 (s, 3H), 3.43 (t, *J* = 9.4 Hz, 1H), 2.50 (br s, 1H), 2.38 (br s, 1H), 1.33 (d, *J* = 6.1 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ154.90, 150.19, 138.15, 128.67, 128.07, 127.95, 117.54, 114.59, 98.07, 81.58, 75.10, 71.26, 71.04, 67.86, 55.63, 18.02.

IR (cm^{-1}): 3011, 2917, 1637, 1496, 1453, 1360, 1215, 1067, 1027, 803, 696, 665, 471, 422.



Step A: To a stirred solution of **33** (2.5g, 5.6 mmol, 1.0 equiv.) in DMF at 23°C was added imidazole (1.13 g, 16.8 mmol, 3.0 equiv.) followed by TBSCl (1.25g, 8.3 mmol, 1.5 equiv.). After 12 hours, the reaction was diluted with brine, and extracted three times with ethyl acetate. The combined organic layers were dried over sodium sulfate and concentrated *in vacuo*. Silica gel chromatography eluting with 5% EtOAc in hexanes furnished compound **34** (2.49g) 78% yield.

Data for **34**:

TLC: R_f = 0.50 (hexane/AcOEt = 9:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ7.39-7.29 (m, 12H), 7.10 (d, *J* = 8.0 Hz, 2H), 5.40 (s, 1H), 4.93 (d, *J* = 11.0 Hz, 1H), 4.78-4.61 (m, 3H), 4.16-4.13 (m, 1H), 4.08-4.05 (m, 1H), 3.88 (s, 1H), 3.60-3.57 (m, 1H), 2.33 (s, 3H), 1.30 (d, *J* = 6.0 Hz, 3H), 0.98 (s, 9H), 0.21 (s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ138.58, 138.26, 137.39, 131.85, 131.01, 129.76, 128.30, 128.27, 127.75, 127.67, 127.59, 127.47, 86.63, 81.42, 80.96, 75.35, 73.53, 72.87, 69.47, 25.99, 21.07, 18.06, 17.84, -4.39, -4.66.

IR (cm^{-1}): 3030, 2915, 1496, 1453, 1362, 1208, 1141, 1070, 1026, 911, 841, 788, 733, 694, 655, 612, 545, 484, 458.

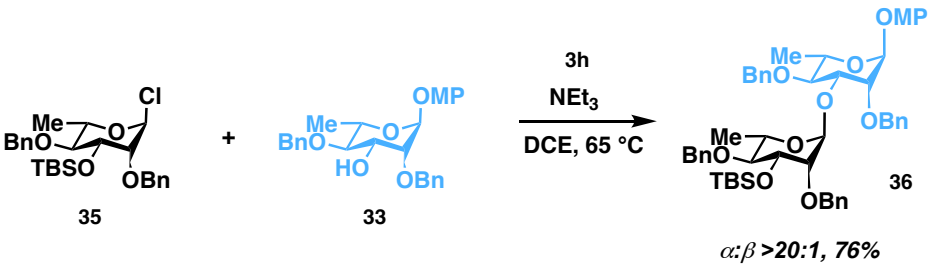
Step B: To a stirred solution of **34** (2.49g, 4.4 mmol, 1.0 equiv.) in 5:1 acetone:water (24 ml) at 23°C was added NBS (2.36g, 13.2 mmol, 3.0 equiv.) portion-wise over 10 minutes. At 2 hours the reaction was quenched with saturated aqueous $Na_2S_2O_3$ and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate and concentrated *in vacuo*. The crude material (1.7 g, 3.7 mmol, 1.0 equiv.) was subjected to General Procedure B. Following silica gel plug filtration – 10:1 hexanes:ethyl acetate, **35** was obtained (1.5 g), in 85% yield. This compound had limited stability and was used immediately following NMR analysis.

Data for **35**:

TLC: R_f = 0.50 (hexane/AcOEt = 12:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ7.44-7.32 (m, 10H), 6.05 (s, 1H), 5.00-4.88 (m, 2H), 4.73-4.66 (m, 2H), 4.45-4.42 (m, 1H), 4.03-3.99 (m, 1H), 3.85-3.84 (m, 1H), 3.66-3.61 (m, 1H), 1.35 (d, J = 6.5 Hz, 3H), 1.03 (s, 9H), 0.19 (s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ138.24, 137.88, 128.41, 128.26, 128.08, 127.98, 127.84, 127.64, 127.54, 92.08, 82.15, 80.47, 75.37, 73.79, 71.57, 71.33, 25.92, 17.54, -4.49, -4.74.



General Procedure B: 1.11 mmol scale, 711 mg, 76%, $\alpha:\beta$ > 20:1

Data for **36**:

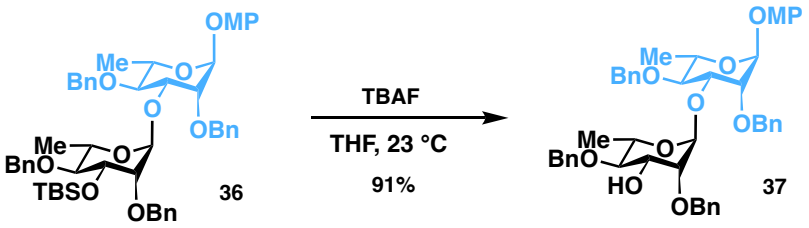
TLC: R_f = 0.45 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ7.49 (d, *J* = 7.5 Hz, 2H), 7.39-7.29 (m, 18H), 7.01 (d, *J* = 9.0 Hz, 2H), 6.86 (d, *J* = 9.0 Hz, 2H), 5.46 (d, *J* = 1.5 Hz, 1H), 5.26 (s, 1H), 4.98 (d, *J* = 11.0 Hz, 1H), 4.84-4.66 (m, 6H), 4.45 (d, *J* = 11.0 Hz, 1H), 4.36 (dd, *J* = 9.5, 2.0 Hz, 1H), 4.27 (dd, *J* = 9.5, 2.0 Hz, 1H), 4.00 (t, *J* = 2.0 Hz, 1H), 3.95-3.89 (m, 1H), 3.80 (s, 3H), 3.75-3.73 (m, 1H), 3.61 (t, *J* = 9.0 Hz, 1H), 1.32 (d, *J* = 6.0 Hz, 3H), 1.31 (d, *J* = 6.0 Hz, 3H), 1.00 (s, 9H), -0.15 (s, 3H), -0.14 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ154.78, 150.27, 138.84, 138.62, 138.33, 137.96, 128.46, 128.39, 128.12, 127.70, 127.63, 127.57, 127.30, 127.23, 127.02, 117.50, 114.51, 100.43, 96.56, 81.28, 80.66, 80.00, 78.07, 74.97, 74.85, 73.22, 73.18, 72.86, 69.94, 68.81, 55.56, 25.96, 18.03, 17.93, -4.36, -4.71.

IR (cm^{-1}): 2927, 2854, 1506, 1453, 1386, 1360, 1266, 1249, 1214, 1124, 1095, 1049, 1028, 1003, 927, 870, 835, 796, 776, 732, 695, 611, 520, 459, 427.

HRMS-ESI (m/z): $[M+NH_4]^+$ calcd. for $[C_{53}H_{70}NO_{10}Si]^+$, 908.4769, found 908.4763.



To a stirred solution of **36** (700 mg, 0.79 mmol, 1.0 equiv.) in THF (5 mL) at 23 °C was added a solution of TBAF (1.0 M in THF, 1.58 mL, 2.0 equiv.) dropwise. After 6 h, the reaction was diluted with ethyl acetate and sequentially washed with water then brine. The organic layer was dried over sodium sulfate and concentrated *in vacuo*. Silica gel chromatography (5:1 hexanes:ethyl acetate) furnished **37** (550 mg) in 92% yield.

Data for **37**:

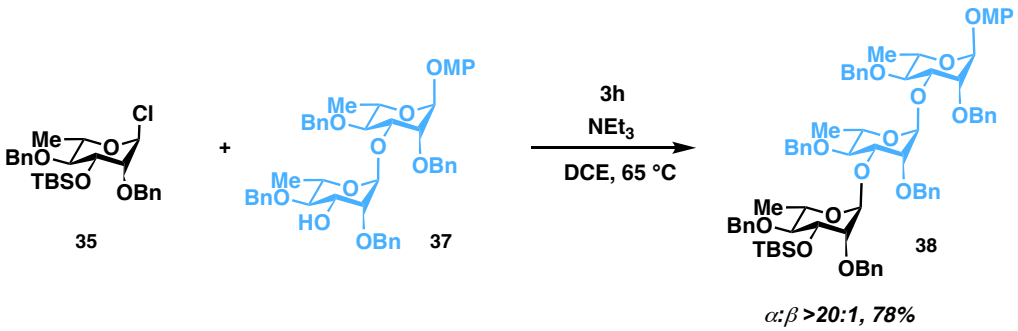
TLC: R_f = 0.30 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ7.46-7.30 (m, 16H), 7.29-7.28 (m, 2H), 7.21 (d, *J* = 7.0 Hz, 2H), 7.01 (d, *J* = 9.0 Hz, 2H), 6.86 (d, *J* = 9.0 Hz, 2H), 5.43 (s, 1H), 5.32 (s, 1H, H-1), 4.97 (d, *J* = 11.0 Hz, 1H), 4.87-4.78 (m, 4H), 4.70 (d, *J* = 11.0 Hz, 1H), 4.42-4.36 (m, 2H), 4.20-4.18 (m, 1H), 4.08 (bs, 1H), 3.96-3.88 (m, 3H), 3.80 (s, 3H), 3.77-3.73 (m, 2H), 3.40 (t, *J* = 9.5 Hz, 1H), 1.36 (d, *J* = 6.0 Hz, 3H), 1.35 (d, *J* = 6.0 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ154.78, 150.21, 138.68, 138.37, 137.77, 137.69, 128.42, 128.37, 128.22, 128.23, 127.71, 127.67, 127.53, 127.48, 126.74, 117.50, 114.50, 98.79, 96.66, 82.11, 80.83, 79.04, 77.61, 77.47, 74.77, 72.84, 72.41, 71.56, 68.90, 67.79, 55.53, 17.99, 17.94.

IR (cm^{-1}): 2932, 1735, 1506, 1453, 1372, 1215, 1135, 1093, 1027, 920, 827, 733, 696, 634, 607, 518, 459, 436.

HRMS-ESI (m/z): $[M+NH_4]^+$ calcd. for $[C_{47}H_{56}NO_{10}]^+$, 794.3904, found 794.3895.



General Procedure B: 0.64 mmol scale, 305 mg, 78% yield. α:β > 20:1

Data for **38**:

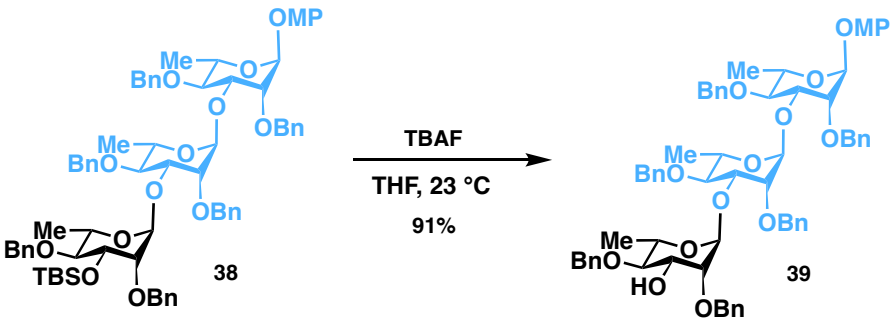
TLC: R_f = 0.30 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ7.47 (d, *J* = 7.0 Hz, 2H), 7.37-725 (m, 28H), 6.98 (d, *J* = 9.5 Hz, 2H), 6.84 (d, *J* = 9.5 Hz, 2H), 5.43 (d, *J* = 1.5 Hz, 1H), 5.28 (s, 1H), 5.16 (d, *J* = 1.0 Hz, 1H), 4.93 (d, *J* = 11.5 Hz, 1H), 4.82-4.61 (m, 8H), 4.44 (s, 2H), 4.40-4.37 (d, *J* = 12.0 Hz, 1H), 4.33 (dd, *J* = 9.5, 3.0 Hz, 1H), 4.23 (dd, *J* = 9.5, 3.0 Hz, 1H), 4.20 (dd, *J* = 9.5, 3.0 Hz, 1H), 3.96 (t, *J* = 2.0 Hz, 1H), 3.90-3.83 (m, 4H), 3.79 (s, 3H), 3.73-3.63 (m, 3H), 3.55 (t, *J* = 9.0 Hz, 1H), 1.28 (d, *J* = 6.5 Hz, 6H), 1.20 (d, *J* = 6.5 Hz, 3H), 0.95 (s, 9H), 0.09 (s, 3H), 0.08 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ154.81, 150.28, 138.89, 138.65, 138.62, 138.30, 138.13, 137.89, 128.49, 128.40, 128.31, 128.12, 128.09, 127.74, 127.72, 127.59, 127.43, 127.31, 127.23, 127.19, 127.07, 126.83, 117.52, 114.53, 100.51, 99.62, 96.50, 81.28, 80.66, 80.58, 80.01, 78.93, 78.56, 78.19, 77.94, 74.88, 74.63, 73.24, 72.84, 72.35, 68.87, 68.4, 68.73, 55.60, 25.96, 18.12, 17.97, 17.93, 18.0, -4.40, -4.73.

IR (cm^{-1}): 2928, 2856, 1508, 1497, 1453, 1386, 1360, 1288, 1252, 1213, 1094, 1041, 1027, 921, 868, 835, 801, 776, 732, 695, 613, 562, 518, 489, 478, 459.

HRMS-ESI (m/z): $[M+NH_4]^+$ calcd. for $[C_{73}H_{92}NO_{14}Si]^+$, 1234.6287, found 1234.6280.



To a stirred solution of **38** (230 mg, 0.19 mmol, 1.0 equiv.) in THF (3 mL) at 23 °C was added a solution of TBAF (1.0 M THF, 0.38 mL, 0.38 mmol, 2.0 equiv.) dropwise. After 6 h, the reaction was diluted with ethyl acetate and sequentially washed with water then brine. The organic layer was dried over sodium sulfate and concentrated *in vacuo*. Silica gel chromatography (7:1 hexanes:ethyl acetate) furnished **39** (190 mg) in 91% yield.

Data for **39**:

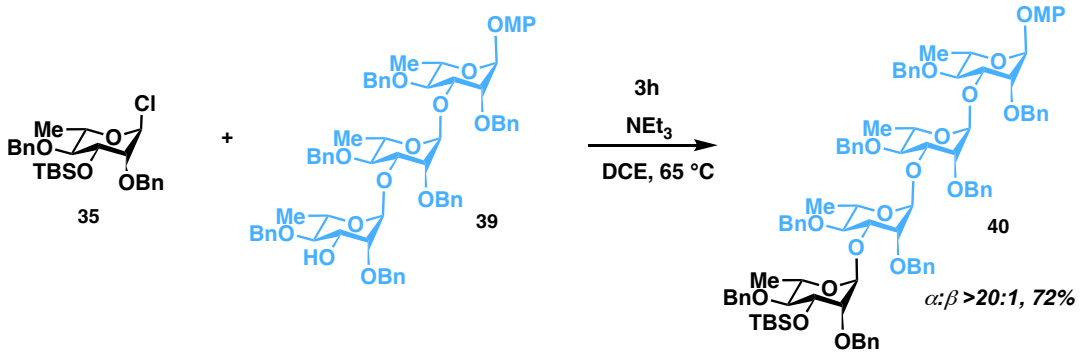
TLC: R_f = 0.25 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ7.45 (d, *J* = 7.5 Hz, 2H), 7.35-7.18 (m, 26H), 7.13 (d, *J* = 6.5 Hz, 2H), 6.95 (d, *J* = 9.5 Hz, 2H), 6.81 (d, *J* = 9.5 Hz, 2H), 5.40 (d, *J* = 1.5 Hz, 2H), 5.22 (s, 1H), 5.19 (s, 1H), 4.87 (d, *J* = 12.0 Hz, 1H), 4.82-60 (m, 7H), 4.44-4.42 (m, 2H), 4.39-4.27 (m, 2H), 4.22 (dd, *J* = 10.0, 3.0, Hz, 1H), 4.10-4.08 (m, 1H), 3.98-3.78 (m, 5H), 3.77-3.75 (m, 4H), 3.69-3.61 (m, 3H), 3.31 (t, *J* = 9.0 Hz, 1H), 2.25 (d, *J* = 9.5 Hz, 1H), 1.28 (d, *J* = 6.5 Hz, 3H), 1.24 (d, *J* = 6.5 Hz, 3H), 1.20 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ154.82, 150.30, 138.75, 138.65, 138.24, 137.98, 137.76, 128.49, 128.42, 128.36, 128.29, 128.26, 127.75, 127.66, 127.50, 127.33, 127.12, 126.61, 117.54, 114.55, 99.70, 98.85, 96.44, 82.17, 80.89, 80.57, 79.12, 78.54, 78.26, 78.07, 74.89, 74.69, 74.48, 72.82, 72.45, 72.36, 71.51, 68.83, 67.71, 55.61, 18.13, 18.03, 17.94.

IR (cm^{-1}): 2917, 1506, 1497, 1453, 1386, 1360, 1288, 1252, 1213, 1094, 1041, 1027, 921, 868, 835, 801, 776, 732, 695, 613, 562, 518, 489, 478, 459.

HRMS-ESI (m/z): $[M+NH_4]^+$ calcd. for $[C_{67}H_{78}NO_{14}]^+$, 1120.5422, found 1120.5413.



General Procedure B: 0.06 mmol scale, 70 mg, 72% yield. α:β > 20:1

Data for **40**:

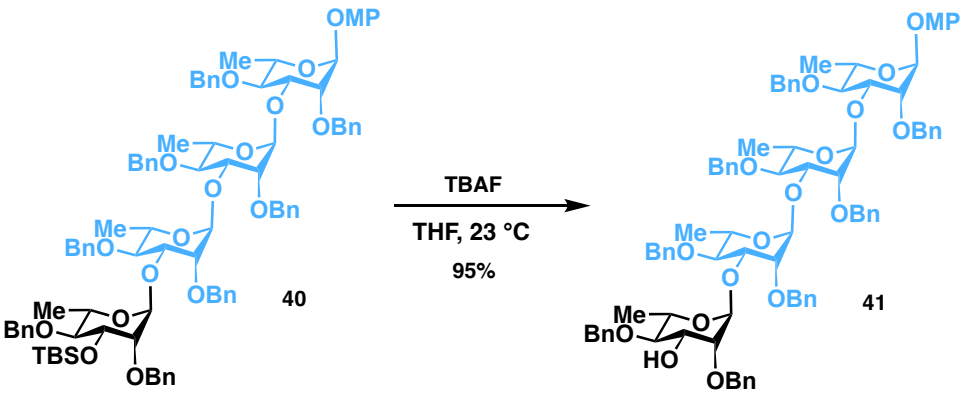
TLC: R_f = 0.25 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ 7.45 (d, *J* = 7.5 Hz, 2H), 7.35-7.20 (m, 38H), 6.96 (d, *J* = 9.5 Hz, 2H), 6.82 (d, *J* = 9.5 Hz, 2H), 5.40 (s, 1H), 5.25 (s, 1H), 5.18 (s, 1H), 5.11 (s, 1H), 4.90 (d, *J* = 12.0 Hz, 1H), 4.82-4.55 (m, 9H), 4.48-4.45 (m, 1H), 4.39-4.29 (m, 5H), 4.20 (dd, *J* = 9.5, 3.0 Hz, 1H), 4.17-4.13 (m, 2H), 3.94 (bs, 1H), 3.88-3.84 (m, 3H), 3.81-3.76 (m, 5H), 3.70-3.59 (m, 4H), 3.50 (t, *J* = 9.0, 1H), 1.27 (d, *J* = 6.5 Hz, 3H), 1.26 (d, *J* = 6.5 Hz, 3H), 1.15 (d, *J* = 6.5 Hz, 6H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ 154.82, 150.31, 138.92, 138.67, 138.57, 138.30, 138.18, 138.05, 137.92, 128.49, 128.49, 128.42, 128.32, 128.28, 128.11, 128.09, 127.75, 127.72, 127.62, 127.46, 127.40, 127.34, 127.29, 127.25, 127.19, 127.06, 126.88, 126.76, 117.54, 114.55, 100.44, 99.75, 99.54, 96.46, 81.29, 80.70, 80.57, 80.03, 78.95, 78.80, 78.67, 78.47, 78.22, 78.02, 74.88, 74.66, 74.52, 73.19, 73.14, 72.85, 72.36, 72.30, 68.84, 68.81, 68.61, 55.62, 25.96, 18.10, 18.02, 17.98, 17.95, -4.40, -4.73.

IR (cm^{-1}): 3030, 2920, 1726, 1702, 1493, 1453, 1365, 1314, 1274, 1206, 1085, 1026, 910, 844, 808, 795, 733, 695, 616, 485, 462, 426.

HRMS-ESI (m/z): $[M+NH_4]^+$ calcd. for $[C_{93}H_{114}NO_{18}Si]^+$, 1560.7805, found 1560.7800.



To a stirred solution of **40** (65 mg, 0.04 mmol, 1.0 equiv.) in THF (0.4 mL) at 23 °C was added a solution of TBAF (1.0 M THF, 0.08 mL, 0.08 mmol, 2.0 equiv.) dropwise. After 6 h, the reaction was diluted with ethyl acetate and sequentially washed with water then brine. The organic layer was dried over sodium sulfate and concentrated *in vacuo*. Silica gel chromatography (5:1 hexanes:ethyl acetate) furnished **41** (54 mg) in 95% yield.

Data for **41**:

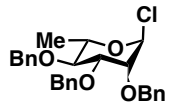
TLC: R_f = 0.60 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ7.44 (d, *J* = 7.5 Hz, 2H), 7.34-7.21 (m, 36H), 7.12 (d, *J* = 6.0 Hz, 2H), 6.96 (d, *J* = 9.0 Hz, 2H), 6.81 (d, *J* = 9.0 Hz, 2H), 5.40 (d, *J* = 1.5 Hz, 1H), 5.26 (s, 1H), 5.17 (s, 1H), 5.16 (s, 1H), 4.87 (d, *J* = 11.0 Hz, 1H), 4.81-4.60 (m, 7H), 4.47-4.45 (m, 1H), 4.39-4.27 (m, 5H,), 4.19 (dd, *J* = 9.5, 2.5 Hz, 2H), 4.07-4.04 (m, 1H), 3.97-3.93 (m, 2H), 3.87-3.3.80 (m, 4H), 3.79-3.3.70 (m, 5H), 3.67-3.60 (m, 4H), 3.28 (t, *J* = 9.0 Hz, 1H), 2.25 (d, *J* = 9.5 Hz, 1H), 1.26 (d, *J* = 6.5 Hz, 3H), 1.25 (d, *J* = 6.5 Hz, 3H), 1.18 (d, *J* = 6.5 Hz, 3H), 1.17 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ154.82, 150.30, 138.76, 138.67, 138.50, 138.33, 138.10, 138.00, 137.90, 137.77, 128.50, 128.42, 128.33, 128.27, 127.77, 127.71, 127.66, 127.49, 127.32, 127.30, 127.02, 126.93, 126.53, 117.54, 114.55, 99.83, 99.45, 98.79, 96.46, 82.18, 80.91, 80.59, 79.12, 78.92, 78.57, 78.20, 78.02, 77.80, 74.86, 74.69, 74.37, 72.86, 72.39, 72.24, 71.50, 68.85, 68.73, 67.68, 60.37 55.63, 18.10, 17.99, 17.95.

IR (cm^{-1}): 2931, 1506, 1453, 1386, 1283, 1214, 1094, 1040, 1027, 918, 827, 733, 695, 606, 519, 459.

HRMS-ESI (m/z): $[M+NH_4]^+$ calcd. for $[C_{87}H_{100}NO_{18}]^+$, 1446.6940, found 1446.6934.



42

General Procedure D: 3.68 mmol scale, 1.51 g, 91% yield.

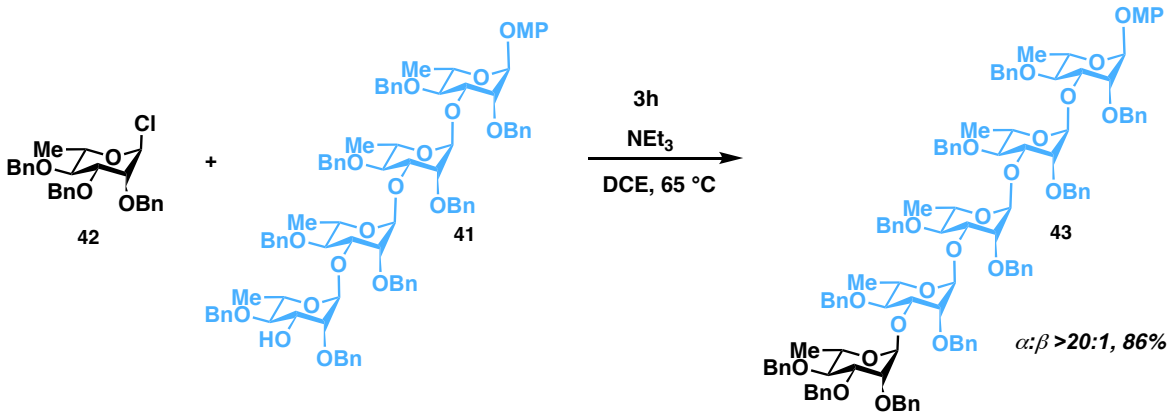
Data for **42:**

TLC: R_f = 0.60 (hexane/AcOEt = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):
δ7.39-7.34 (m, 15H), 6.05 (d, *J* = 1.0 Hz, 1H), 5.00 (d, *J* = 11.0 Hz, 1H), 4.77-4.63 (m, 5H), 4.19 (dd, *J* = 9.5, 3.0 Hz, 1H), 4.02 (p, *J* = 3.0 Hz, 1H), 3.92 (d, *J* = 2.0 Hz, 1H), 3.71 (d, *J* = 9.5 Hz, 1H), 1.39 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$):
δ138.22, 138.05, 137.55, 128.43, 128.37, 128.34, 127.94, 127.88, 127.74, 127.69, 91.58, 79.62, 78.23, 77.98, 75.44, 73.01, 72.40, 71.04, 53.36, 46.10, 17.56.

Characterization matches literature precedent[2]



General Procedure B: 0.036 mmol, 57 mg, 86%, $\alpha:\beta$ > 20:1

Data for **43**:

TLC: R_f = 0.30 (hexane/AcOEt = 4:1)

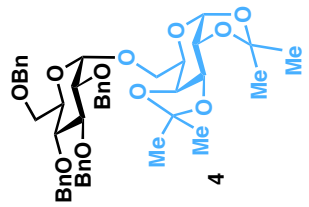
^{1}H NMR (500 MHz, $CDCl_3$):
δ7.46 (d, *J* = 7.5 Hz, 2H), 7.36-7.18 (m, 53H), 6.97 (d, *J* = 9.0 Hz, 2H), 6.83 (d, *J* = 9.0 Hz, 2H), 5.41 (d, *J* = 1.5 Hz, 1H), 5.26 (s, 1H), 5.18 (s, 1H), 5.14 (s, 1H), 5.13 (s, 1H), 4.95 (d, *J* = 11.0 Hz, 1H), 4.80-4.74 (m, 4H), 4.72-4.59 (m, 6H), 4.56-4.50 (m, 3H), 4.47-4.42(m, 4H), 4.41-4.30 (m, 4H), 4.21 (dd, *J* = 9.5, 3.0 Hz, 1H), 4.17-4.13 (m, 2H), 3.95 (t, *J* = 2.5 Hz, 1H), 3.88-3.86 (m, 4H), 3.85-3.76 (m, 9H), 3.73-3.59 (m, 4H), 3.57-3.51 (m, 1H), 1.27 (d, *J* = 6.5 Hz, 6H, 2*C*H*$_3$), 1.20 (d, *J* = 6.5 Hz, 3H), 1.16 (d, *J* = 6.5 Hz, 3H), 1.12 (d, *J* = 6.5 Hz, 3H).

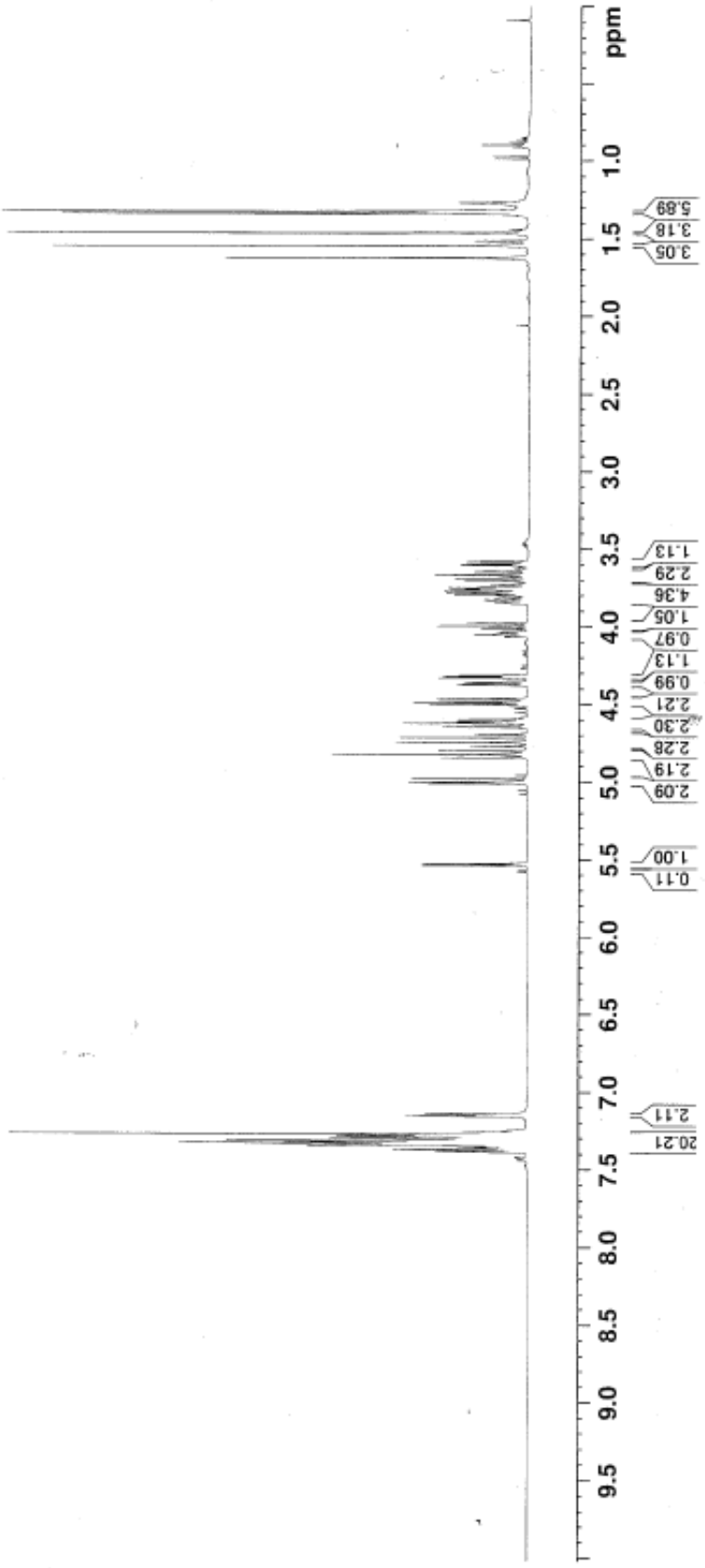
^{13}C NMR (125 MHz, $CDCl_3$):
δ154.82, 150.29, 138.92, 138.63, 138.54, 138.34, 138.30, 138.13, 138.06, 137.90, 128.49, 128.41, 128.30, 128.25, 128.21, 128.18, 128.14, 127.76, 127.70, 127.63, 127.56, 127.46, 127.37, 127.31, 127.27, 127.03, 126.84, 126.72, 117.53, 114.54, 99.64, 99.47, 96.45, 80.78, 80.62, 80.57, 80.43, 79.77, 78.83, 78.60, 78.22, 78.04, 75.70, 74.97, 74.86, 74.64, 74.56, 74.37, 72.85, 72.43, 72.27, 72.04, 68.85, 68.74, 68.65, 68.59, 55.62, 18.14, 18.03, 17.99, 17.95.

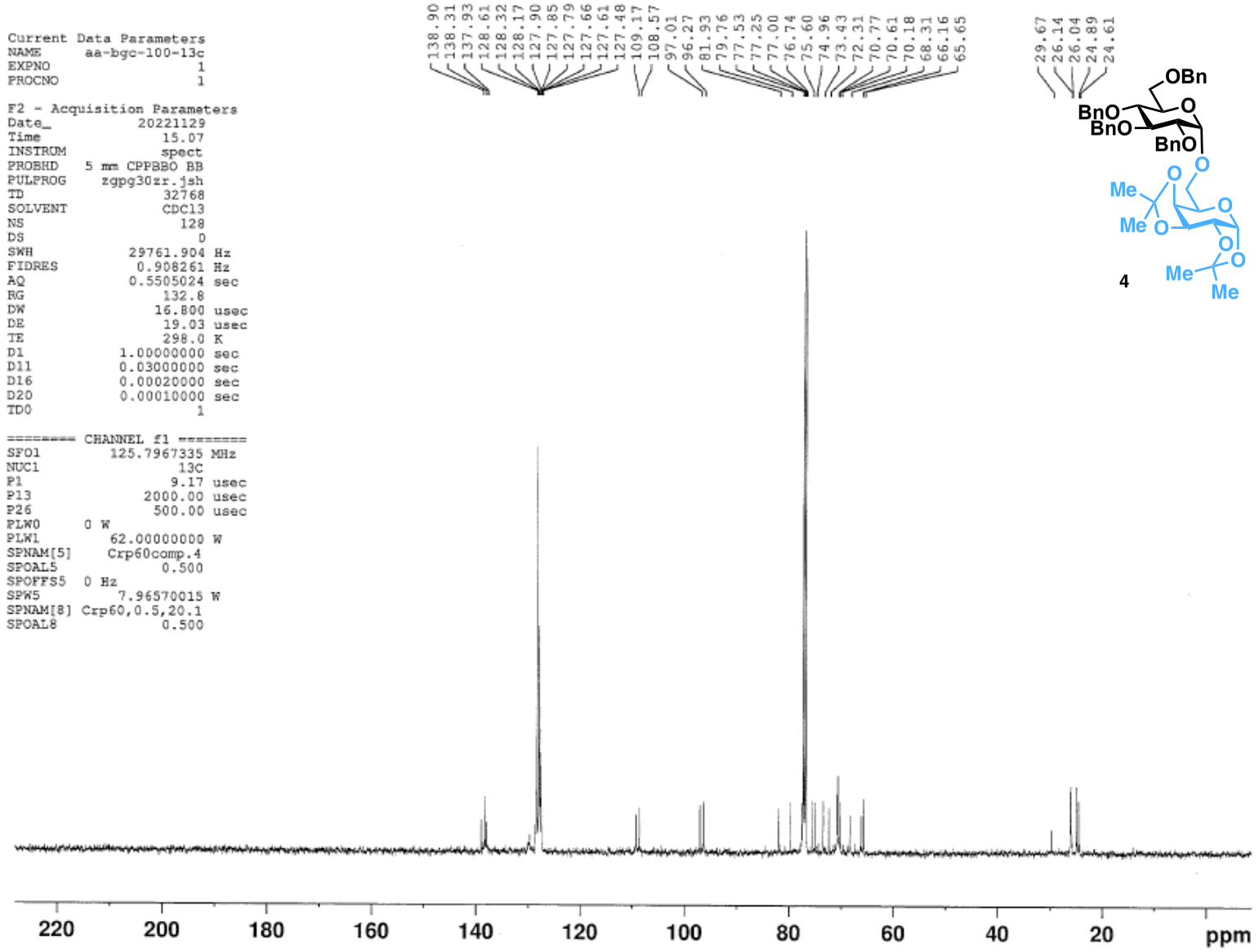
IR (cm^{-1}): 2930, 1496, 1453, 1387, 1356, 1282, 1213, 1175, 1097, 1041, 1027, 1005, 919, 828, 731, 694, 611, 540, 477, 456, 432.

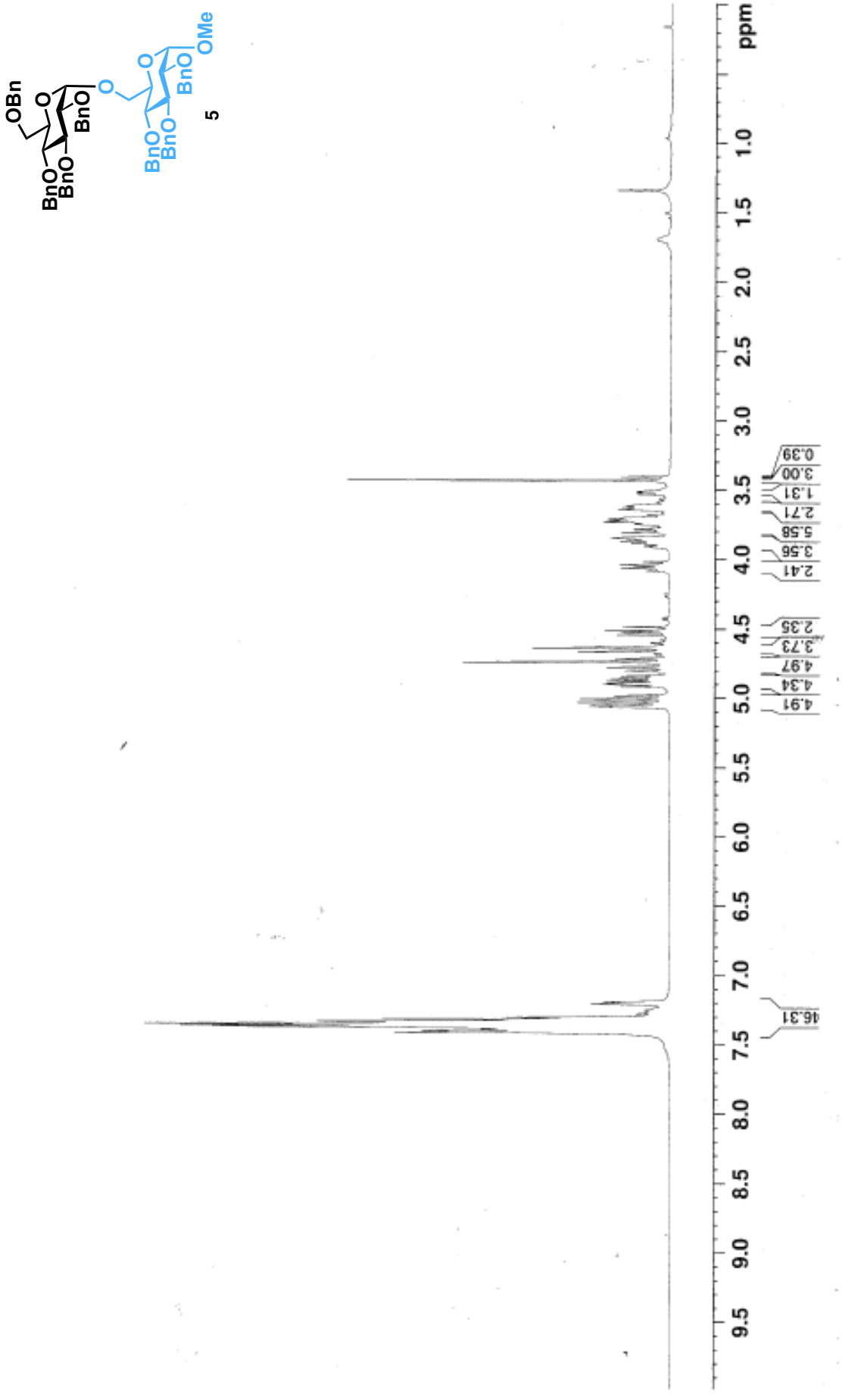
HRMS-ESI (m/z): $[M+NH_4]^+$ calcd. for $[C_{114}H_{128}NO_{22}]^+$, 1862.8923, found 1862.8911.

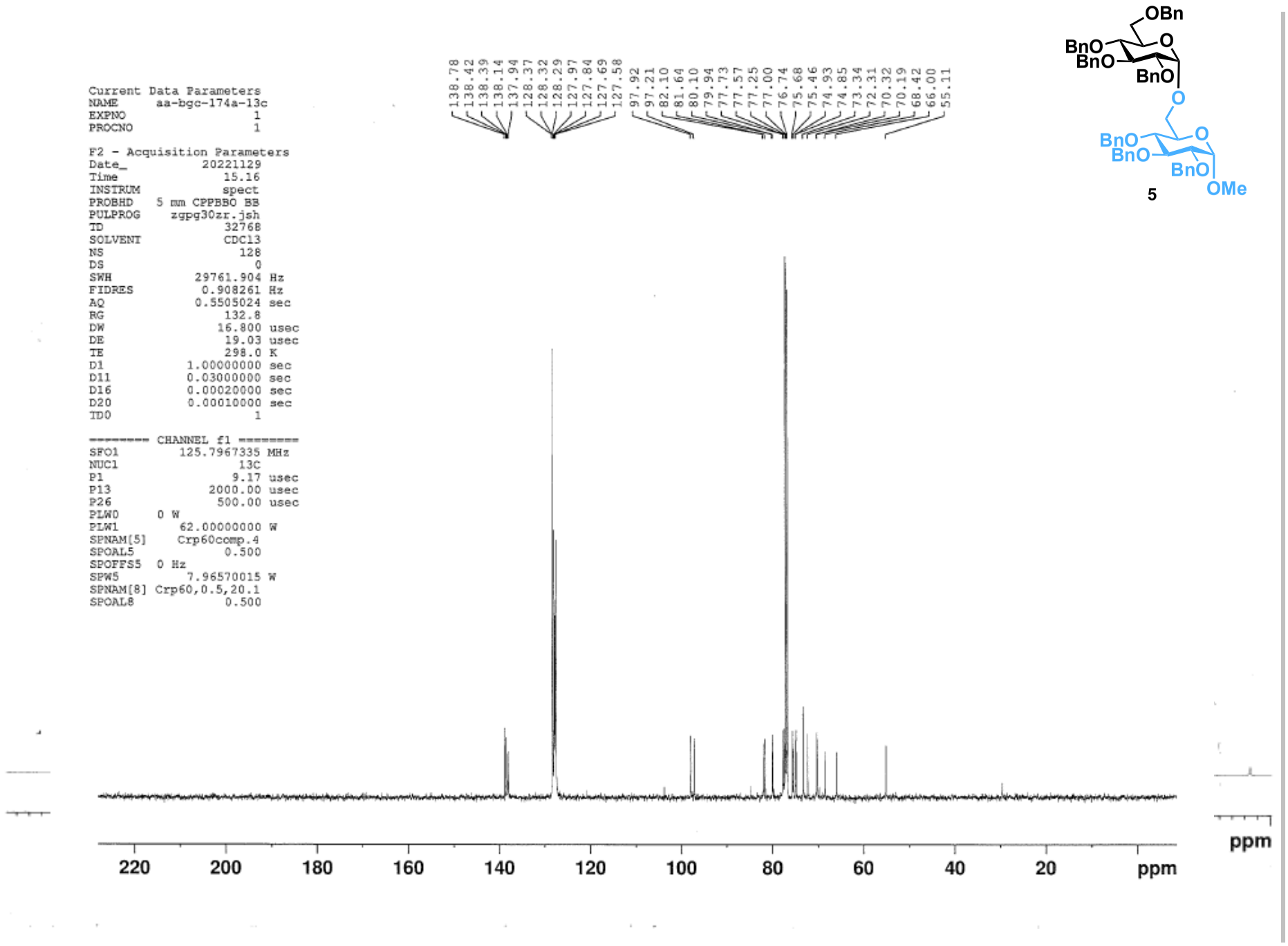
5. NMR Spectra

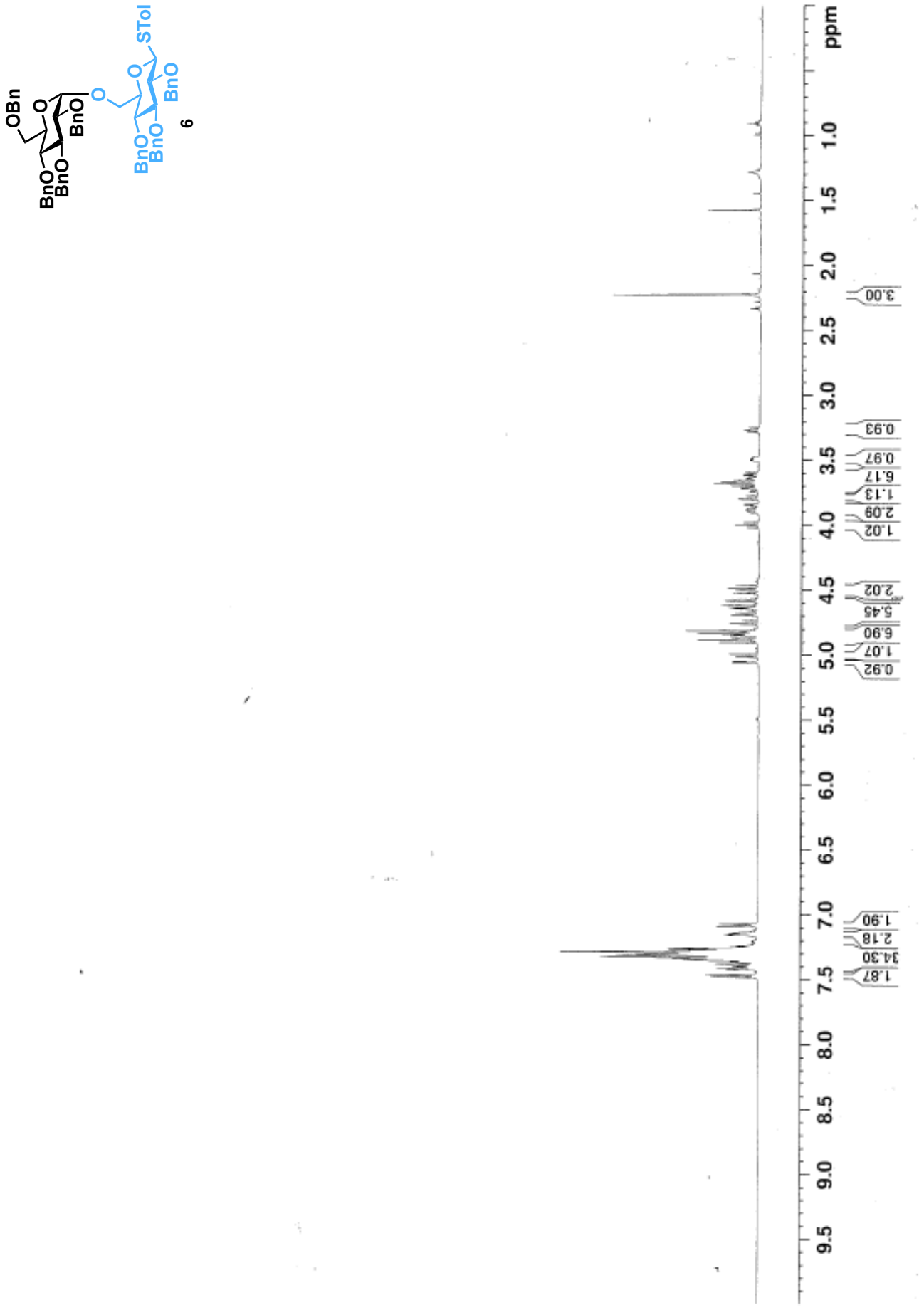


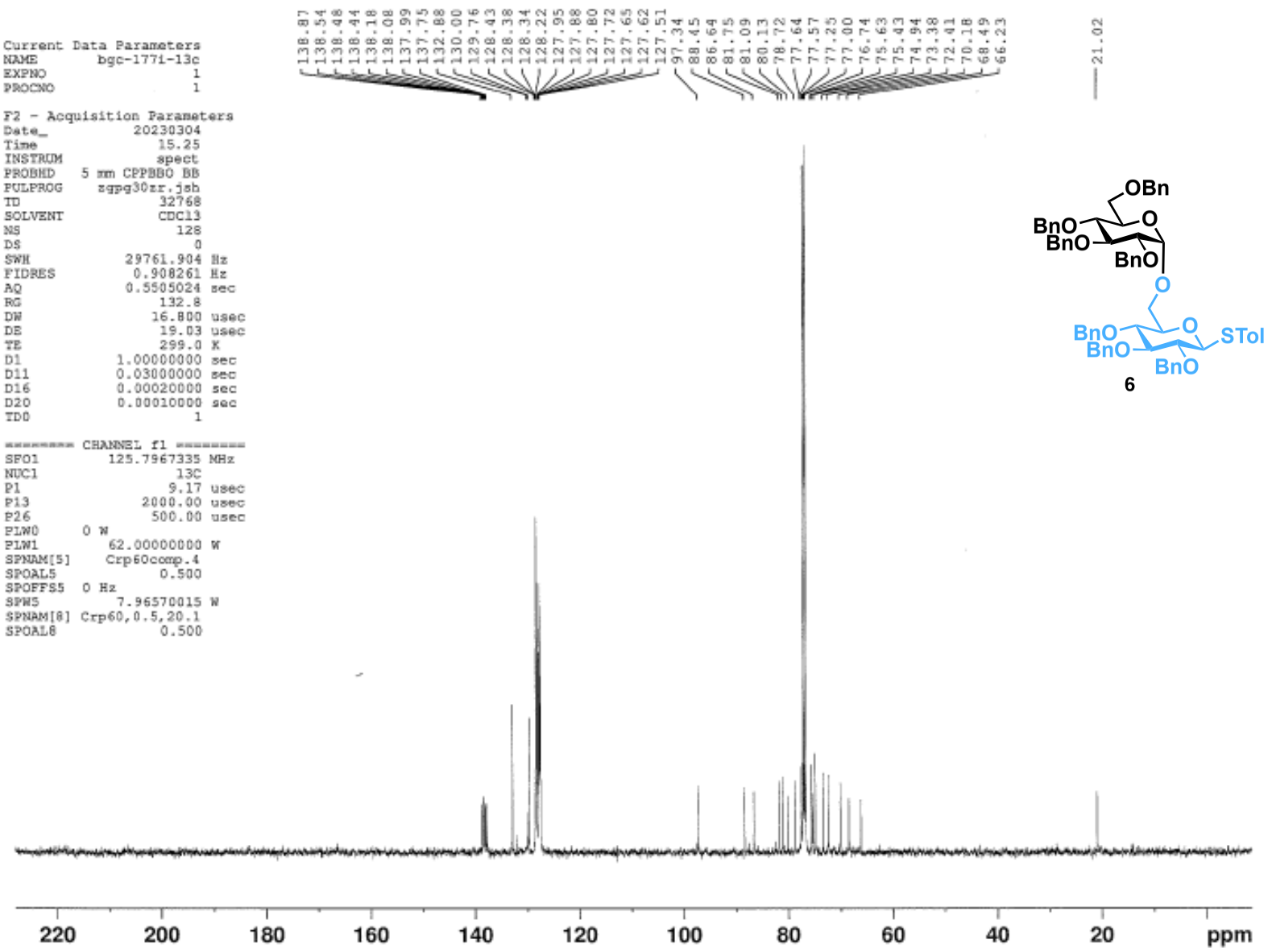


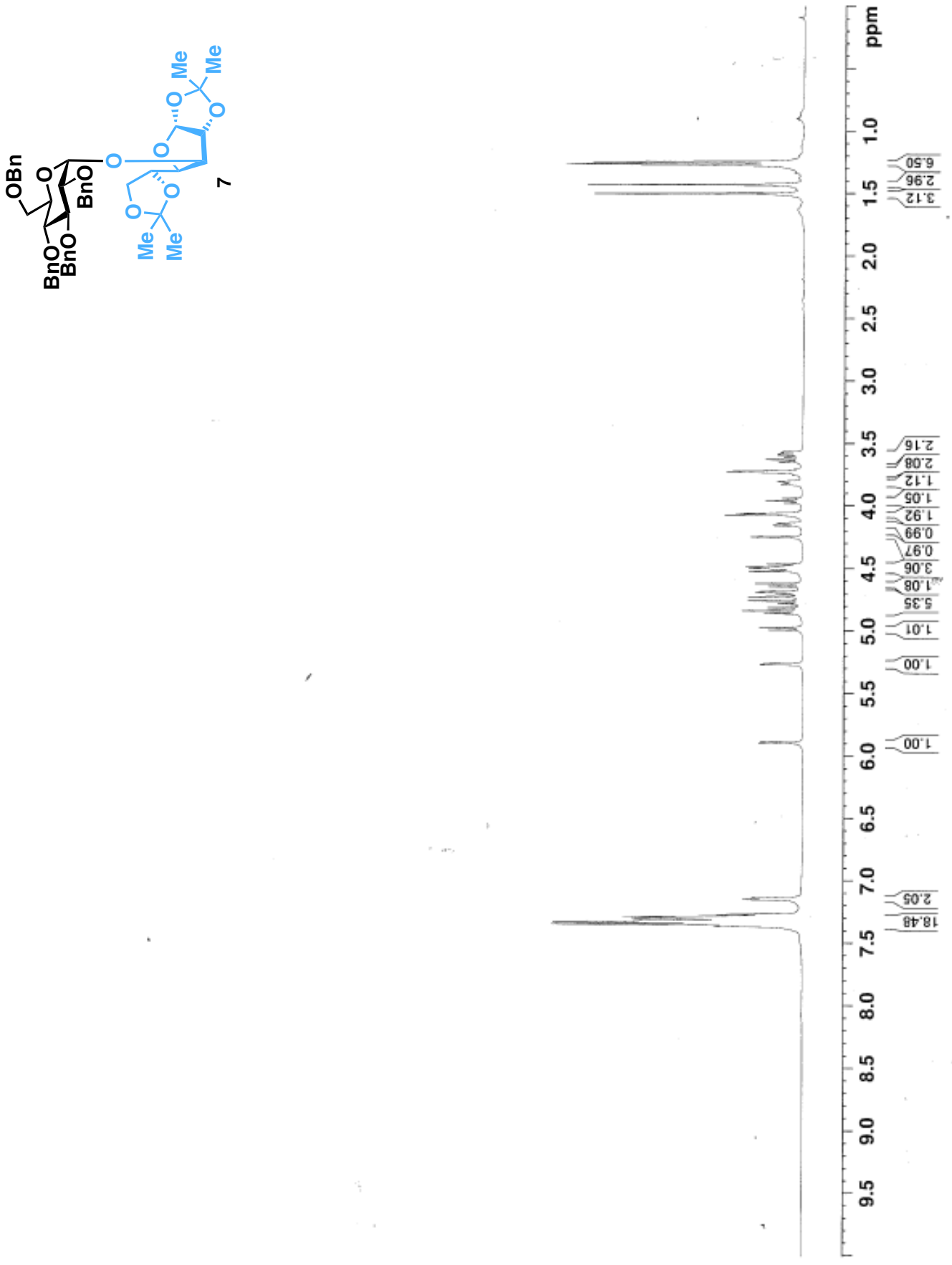

Current Data Parameters
NAME aa-bgc-100-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221129
Time 15.07
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
138.90
138.31
137.93
128.61
128.32
128.17
127.90
127.85
127.79
127.66
127.61
127.48
109.17
108.57
97.01
96.27
81.93
79.76
77.53
77.25
77.00
76.74
75.60
74.96
73.43
72.31
70.77
70.61
70.18
68.31
66.16
65.65
29.67
26.14
26.04
24.89
24.61
OBn
BnO
BnO
BnO
Me
Me
Me
Me
4
220
200
180
160
140
120
100
80
60
40
20
ppm

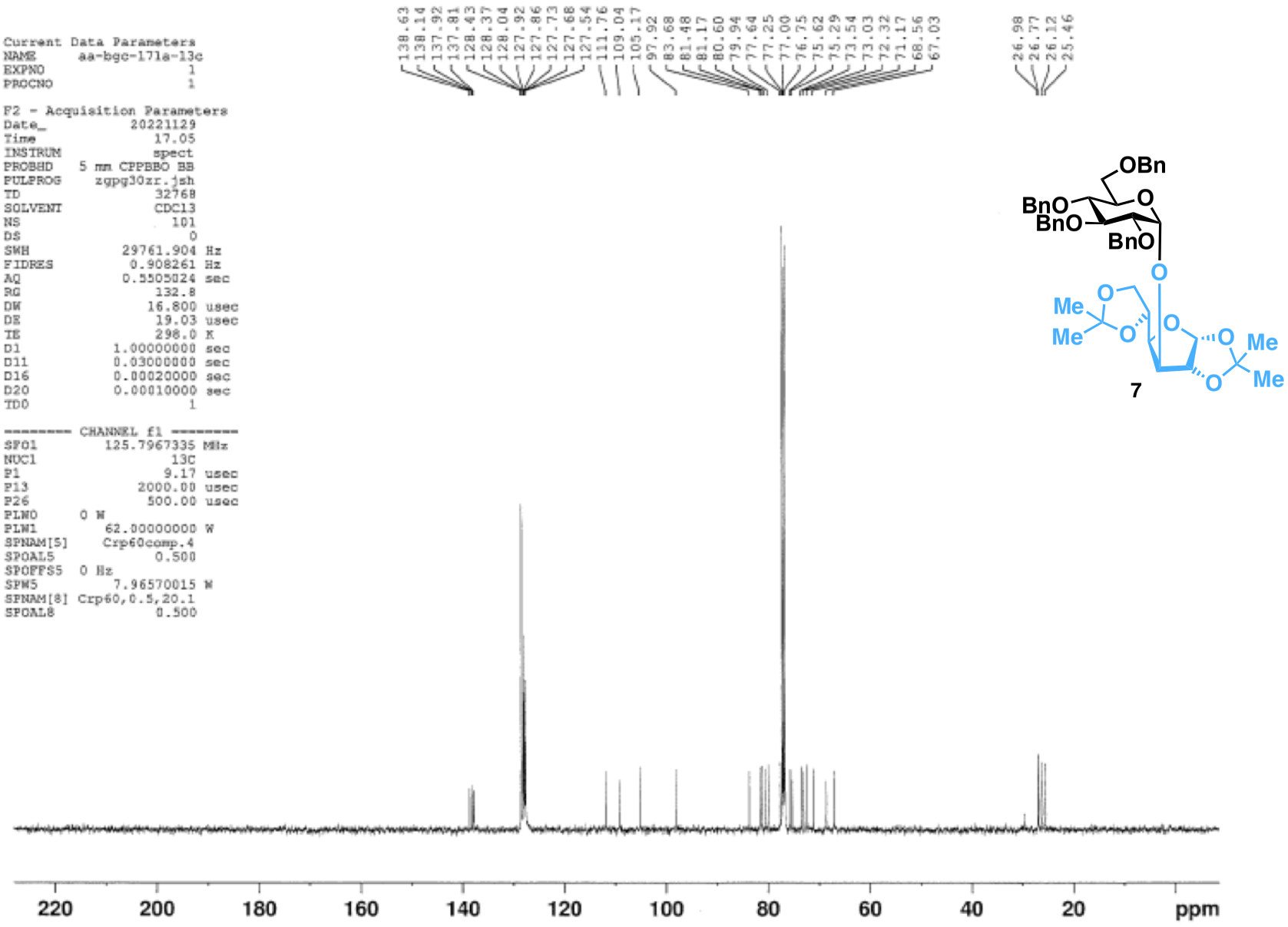

OBn
BnO
BnO
BnO
BnO
BnO
BnO
OMe
5
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
16.31
4.91
4.34
4.97
3.73
2.35
2.41
3.56
5.58
2.71
1.31
3.00
0.39


138.78
138.42
138.39
138.14
137.94
128.37
128.32
128.29
127.97
127.84
127.69
127.58
97.92
97.21
82.10
81.64
80.10
79.94
77.73
77.57
77.25
77.00
76.74
75.68
75.46
74.93
74.85
73.34
72.31
70.32
70.19
68.42
66.00
55.11
OBn
BnO
BnO
BnO
O
O
BnO
BnO
BnO
OMe
5
Current Data Parameters
NAME aa-bgc-174a-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221129
Time 15.16
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
220
200
180
160
140
120
100
80
60
40
20
ppm
ppm

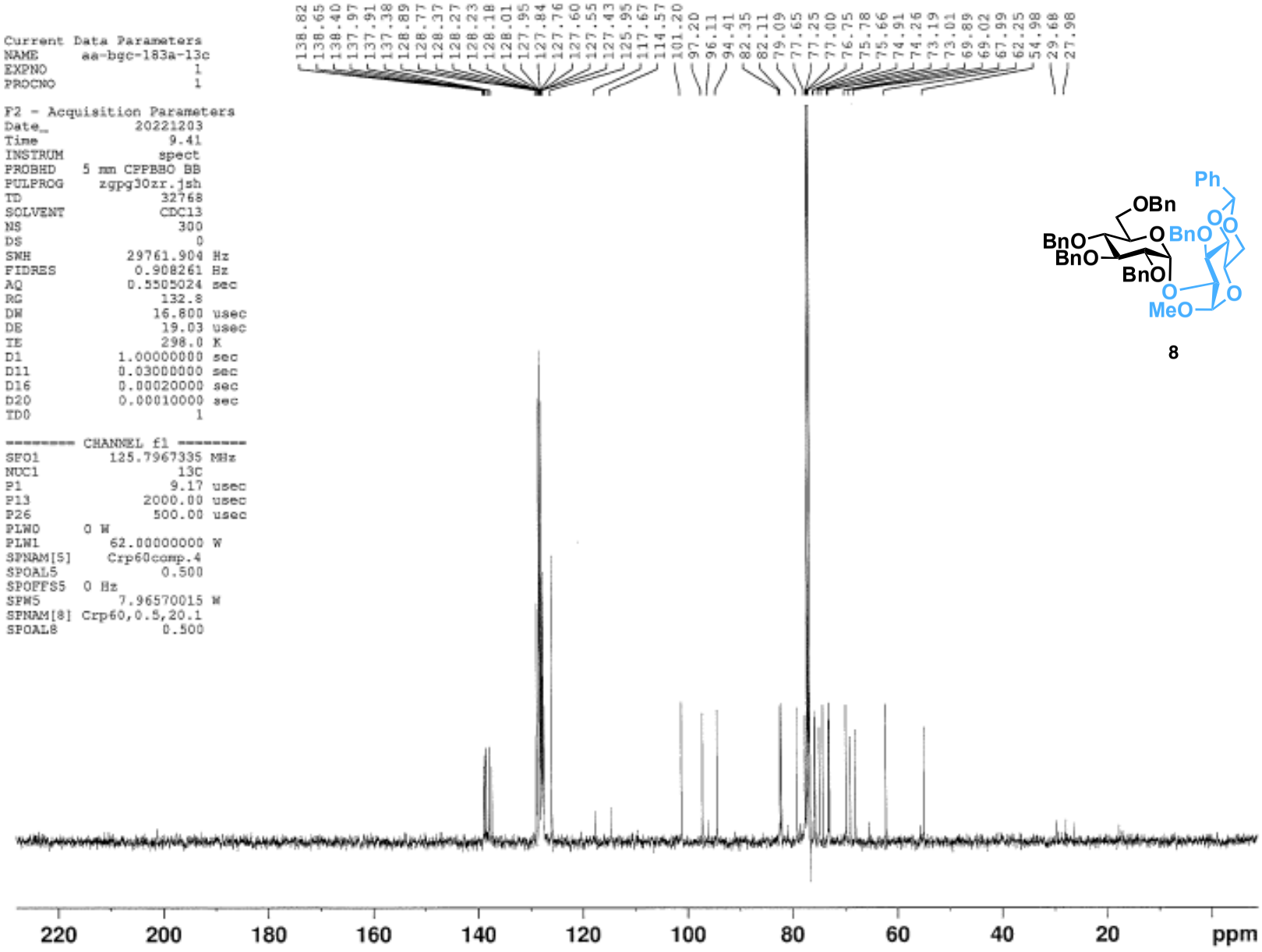

OBn
BnO
BnO
BnO
O
O
BnO
BnO
BnO
O
STol
BnO
6
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
3.00
0.93
0.97
6.17
1.13
2.09
1.02
2.02
5.45
6.90
1.07
0.92
1.87
34.30
2.18
1.90


Current Data Parameters
NAME bgc-1771-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20230304
Time 15.25
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsb
TD 32768
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 299.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
138.87
138.54
138.48
138.44
138.18
138.08
137.99
137.75
132.88
130.00
129.76
128.43
128.38
128.34
128.22
127.95
127.88
127.80
127.72
127.65
127.62
127.51
97.34
88.45
86.64
81.75
81.09
80.13
78.72
77.64
77.57
77.25
77.00
76.74
75.63
75.43
74.94
73.38
72.41
70.18
68.49
66.23
21.02
OBn
BnO
BnO
BnO
O
O
BnO
BnO
BnO
O
STol
6
220
200
180
160
140
120
100
80
60
40
20
ppm

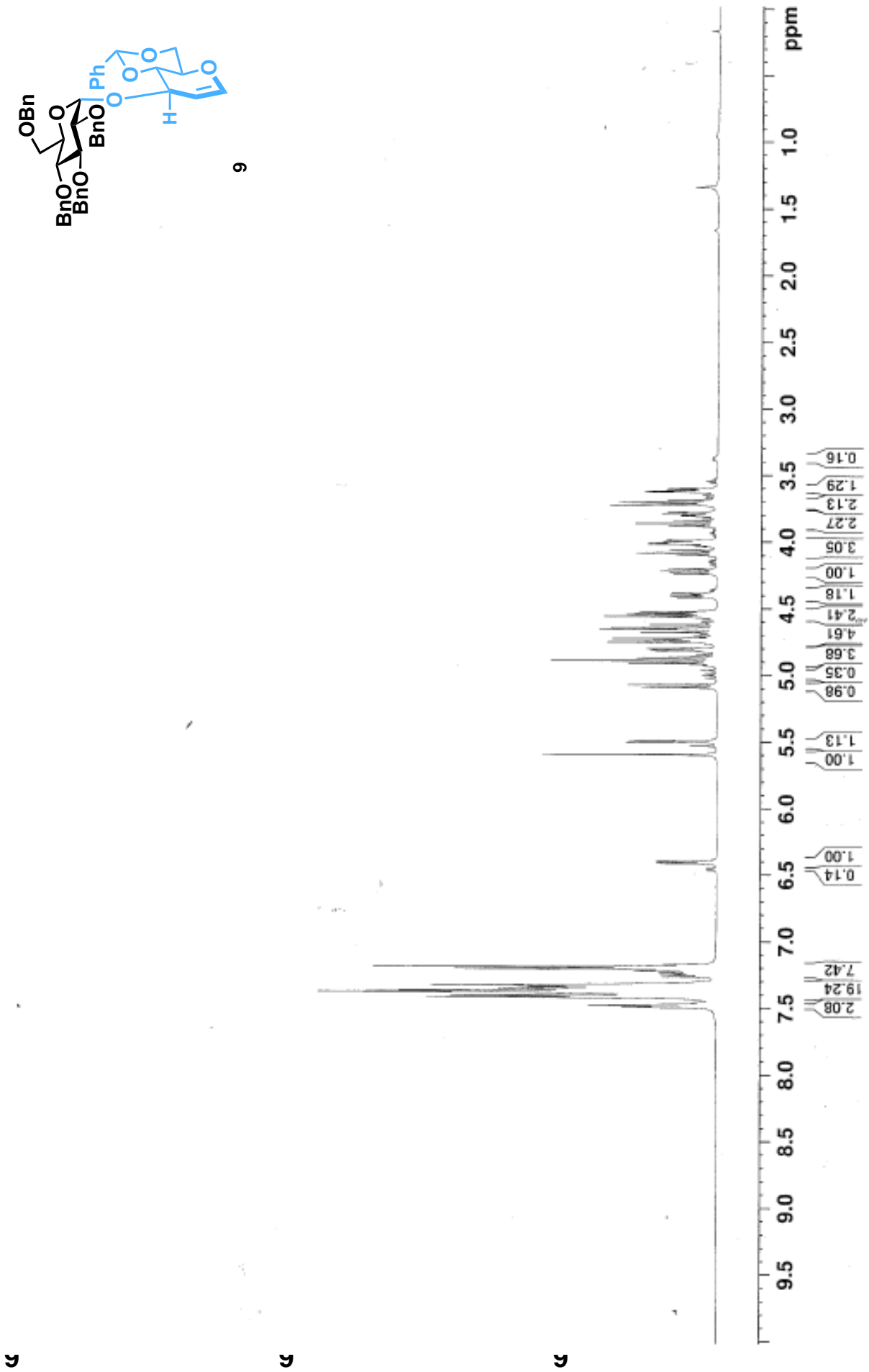

OBn
BnO
BnO
BnO
O
O
O
O
O
Me
Me
Me
Me
7
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
6.50
2.96
3.12
2.16
2.08
1.12
1.05
1.92
0.99
0.97
3.06
1.08
5.35
1.01
1.00
1.00
2.05
18.48

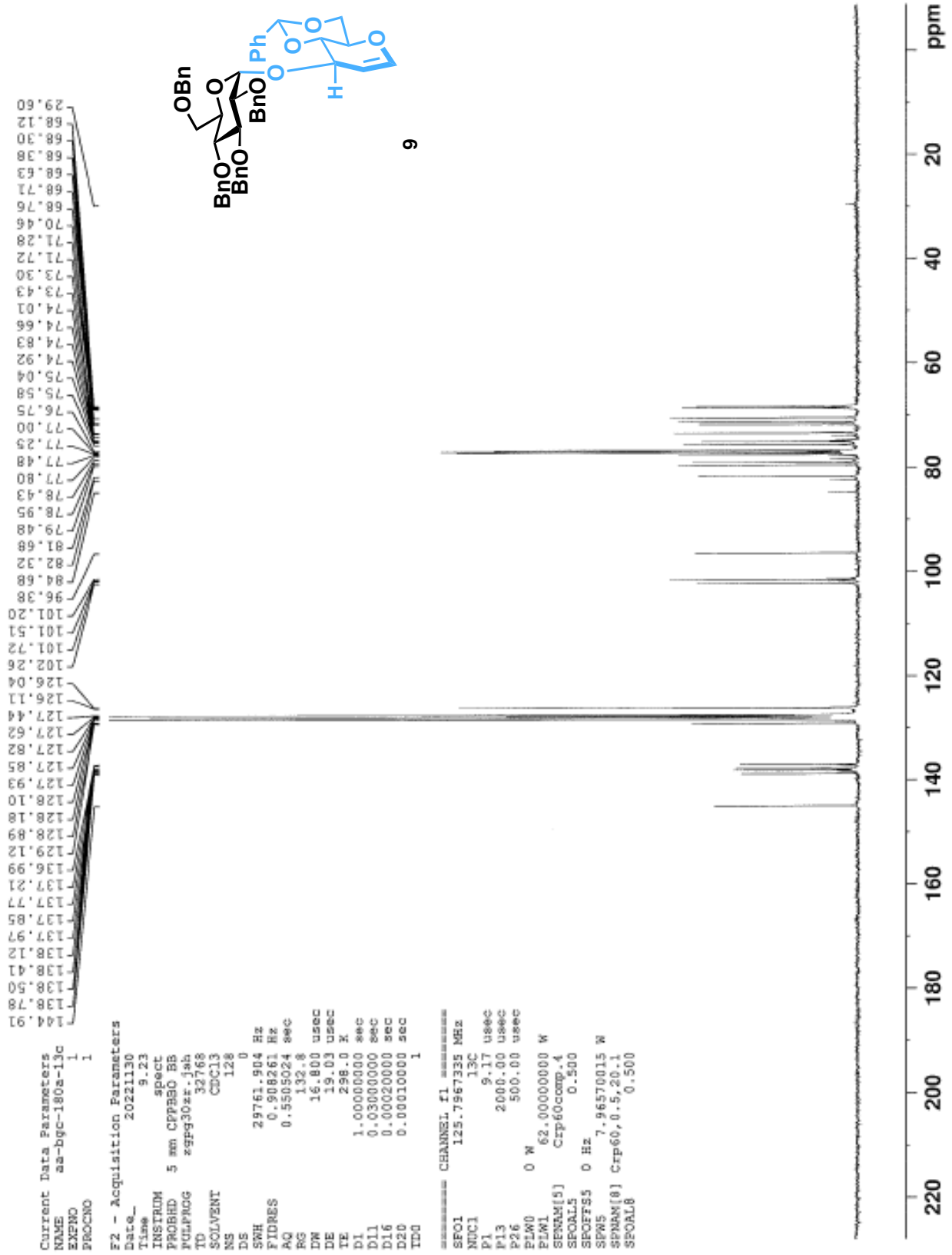

Current Data Parameters
NAME aa-bgc-171a-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221129
Time 17.05
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 101
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
138.63
138.14
137.92
137.81
128.43
128.37
128.04
127.92
127.86
127.73
127.68
127.54
111.76
109.04
105.17
97.92
83.68
81.48
81.17
80.60
79.94
77.64
77.25
77.00
76.75
75.62
75.29
73.54
73.03
72.32
71.17
68.56
67.03
26.98
26.77
26.12
25.46
OBn
BnO
BnO
BnO
O
O
Me
Me
O
O
O
O
Me
Me
7
220
200
180
160
140
120
100
80
60
40
20
ppm

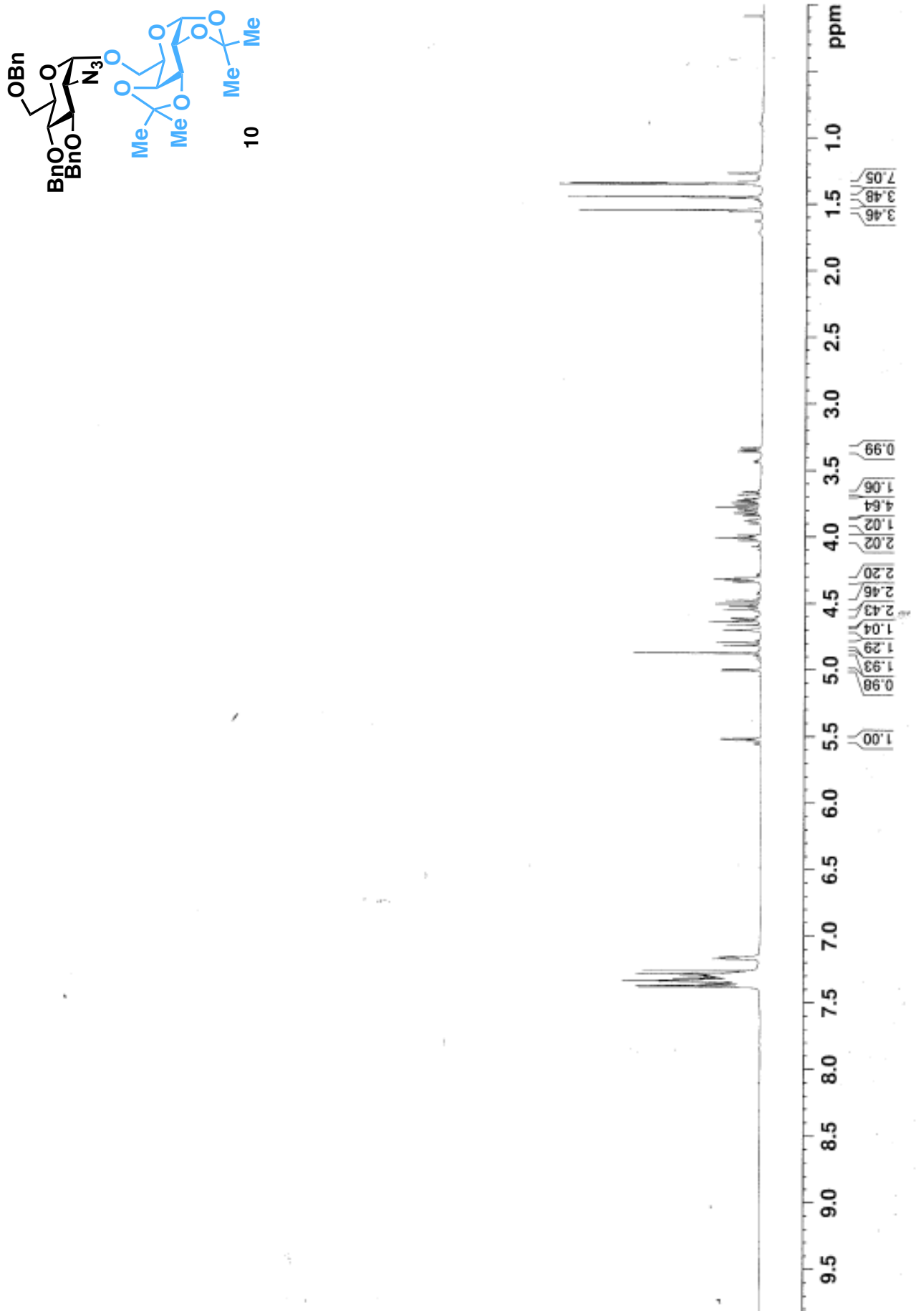
Ph
O
O
BnO
O
MeO
OBn
O
BnO
BnO
BnO
8
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
2.00
26.25
3.23
2.04
0.10
1.00
1.07
2.18
4.10
2.19
1.06
1.11
2.15
3.19
2.26
4.97
3.44
1.08


Current Data Parameters
NAME aa-bgc-183a-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221203
Time 9.41
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDC13
NS 300
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
138.82
138.65
138.40
137.97
137.91
137.38
128.89
128.77
128.37
128.27
128.23
128.18
128.01
127.95
127.84
127.76
127.60
127.55
127.43
125.95
117.67
114.57
101.20
97.20
96.11
94.41
82.35
82.11
79.09
77.65
77.25
77.00
76.75
75.78
75.66
74.91
74.26
73.19
73.01
69.89
69.02
67.99
62.25
54.98
29.68
27.98
Ph
OBn
BnO
BnO
BnO
BnO
O
O
O
O
MeO
220
200
180
160
140
120
100
80
60
40
20
ppm

8

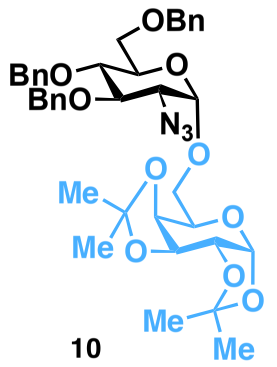

OBn
BnO
BnO
BnO
Ph
H
9
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.16
1.29
2.13
2.27
3.05
1.00
1.18
2.41
4.61
3.68
0.35
0.98
1.13
1.00
1.00
0.14
7.42
19.24
2.08


OBn
BnO
BnO
BnO
Ph
H
9
ppm

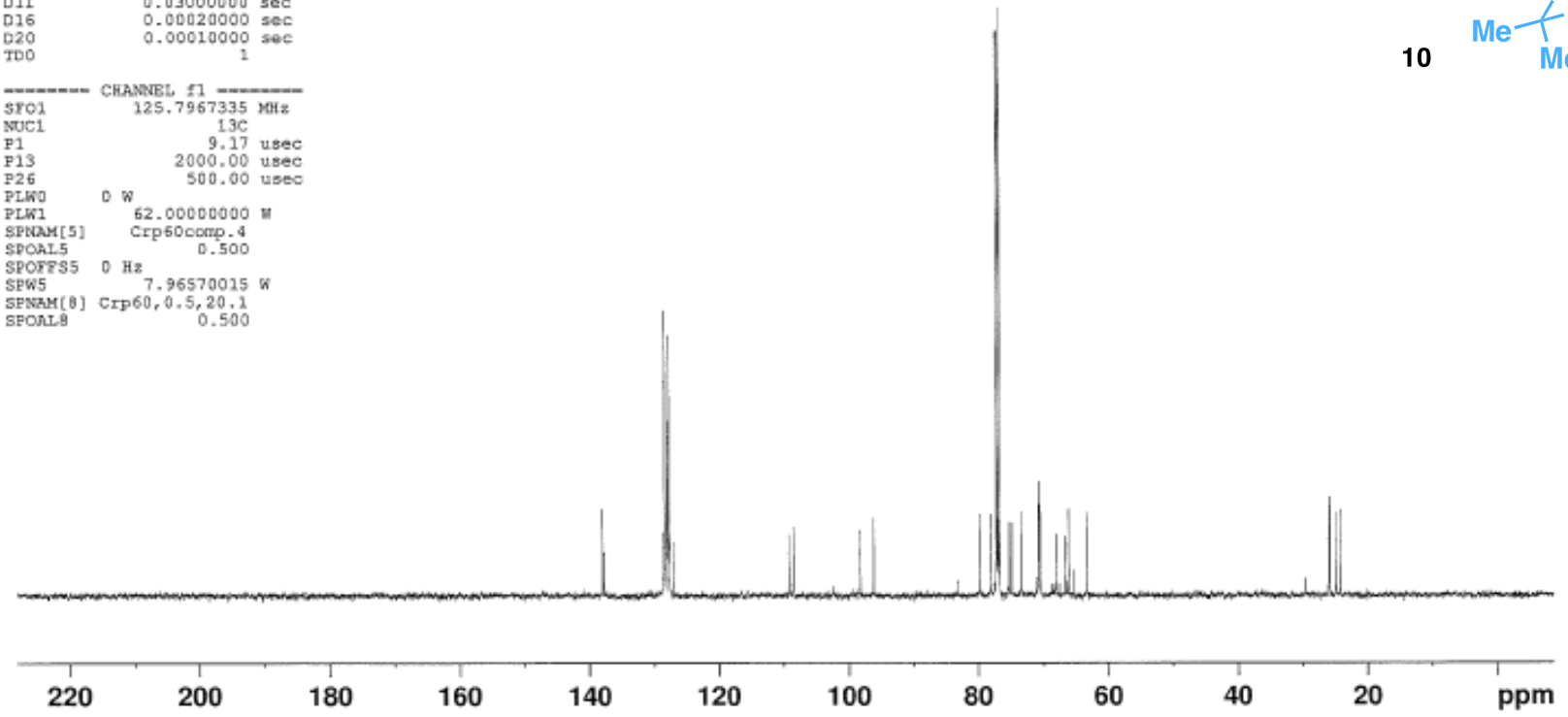

OBn
BnO
BnO
N3
Me
Me
Me
Me
10
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
7.05
3.48
3.46
0.99
1.06
4.64
1.02
2.02
2.20
2.46
2.43
1.04
1.29
1.93
0.98
1.00

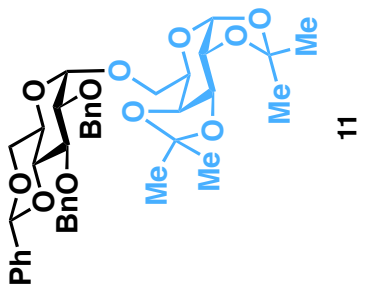
```
Current Data Parameters
NAME      aa-bgc-287-13c
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20221118
Time               8.52
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG   zgpg30zr.jsh
TD               32768
SOLVENT          CDCl3
NS                 116
DS                   0
SWH          29761.904 Hz
FIDRES        0.908261 Hz
AQ            0.5505024 sec
RG                132.8
DW               16.800 usec
DE               19.03 usec
TE               298.0 K
D1            1.00000000 sec
D11           0.03000000 sec
D16           0.00020000 sec
D20           0.00010000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1       125.7967335 MHz
NUC1               13C
P1                9.17 usec
P13            2000.00 usec
P26             500.00 usec
PLW0      0 W
PLW1        62.00000000 W
SPNAM[5]    Crp60comp.4
SPOAL5            0.500
SPOFFS5   0 Hz
SPW5         7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8            0.500
```

138.01
137.81
128.53
128.41
128.37
127.96
127.90
127.78
127.72
127.62
126.94
109.24
108.57
98.24
96.21
79.85
78.19
77.25
77.00
76.74
75.24
74.91
73.47
71.20
70.79
70.63
70.61
70.53
68.79
68.10
67.56
66.80
66.39
66.19
65.34
63.33
29.67
26.08
25.93
24.91
24.35



10




Ph
BnO
BnO
O
O
O
O
O
O
O
O
O
Me
Me
Me
Me

11

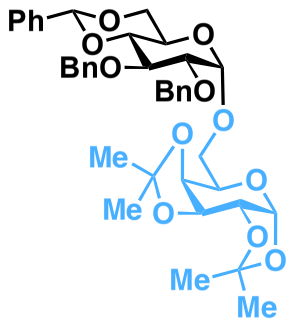
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
6.00
3.00
2.99
2.04
1.04
2.13
1.10
1.99
2.12
0.88
1.12
2.13
1.05
1.90
2.00
17.57

11

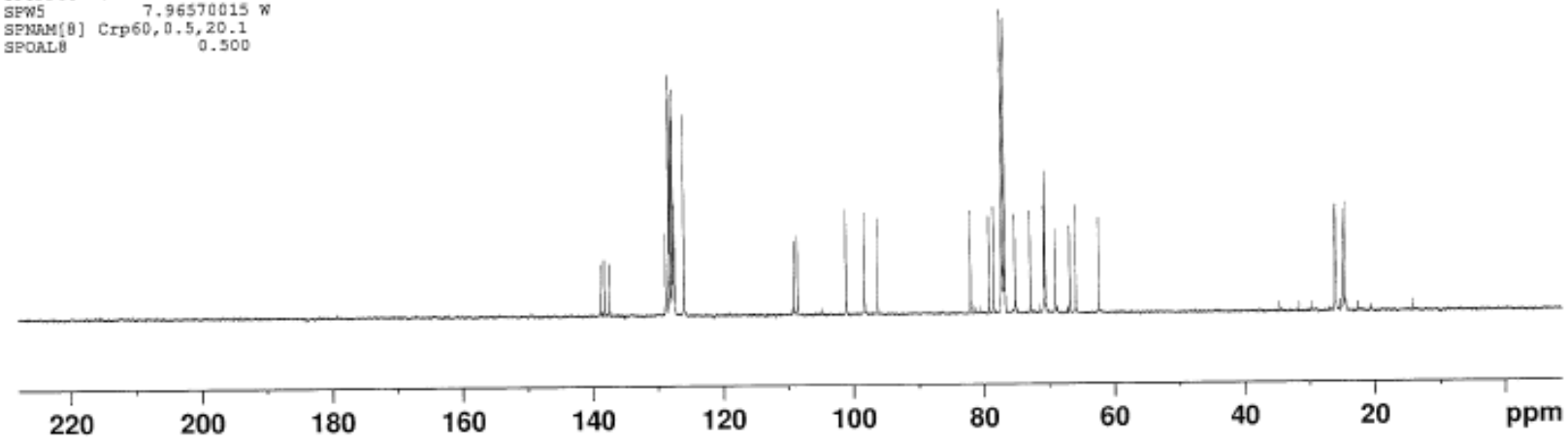
```
Current Data Parameters
NAME      aa-bgc-266b-13c
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20221118
Time               9.09
INSTRUM           spect
PROBHD    5 mm CPPBBO BB
PULPRDG     zgpg30zr.jsh
TD                32768
SOLVENT           CDCl3
NS                  183
DS                    0
SWH           29761.904 Hz
FIDRES         0.908261 Hz
AQ             0.5505024 sec
RG                132.8
DW               16.800 usec
DE                19.03 usec
TE                298.0 K
D1            1.00000000 sec
D11           0.03000000 sec
D16           0.00020000 sec
D20           0.00010000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         125.7967335 MHz
NUC1                 13C
P1                  9.17 usec
P13               2000.00 usec
P26                500.00 usec
PLW0      0 W
PLW1        62.00000000 W
SPNAM[5]     Crp60comp.4
SPOAL5             0.500
SPOFFS5   0 Hz
SPW5          7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8             0.500
```

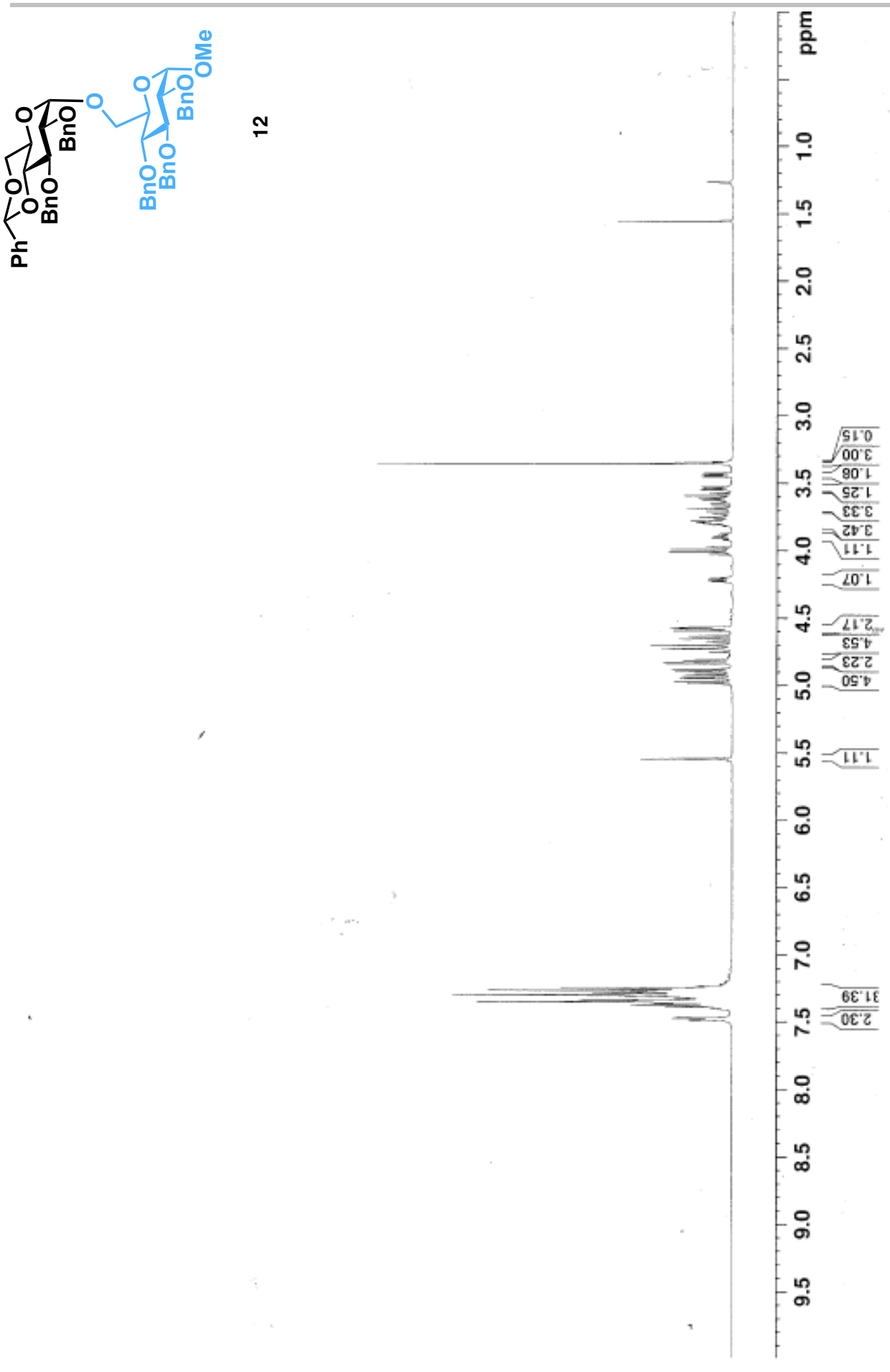
138.82
138.26
137.46
128.82
128.48
128.32
128.22
128.16
127.99
127.89
127.73
127.69
127.47
126.00
109.17
108.61
101.14
98.31
96.28
82.06
79.21
78.51
77.26
77.00
76.75
75.21
72.84
70.77
70.61
68.98
66.84
65.86
62.41

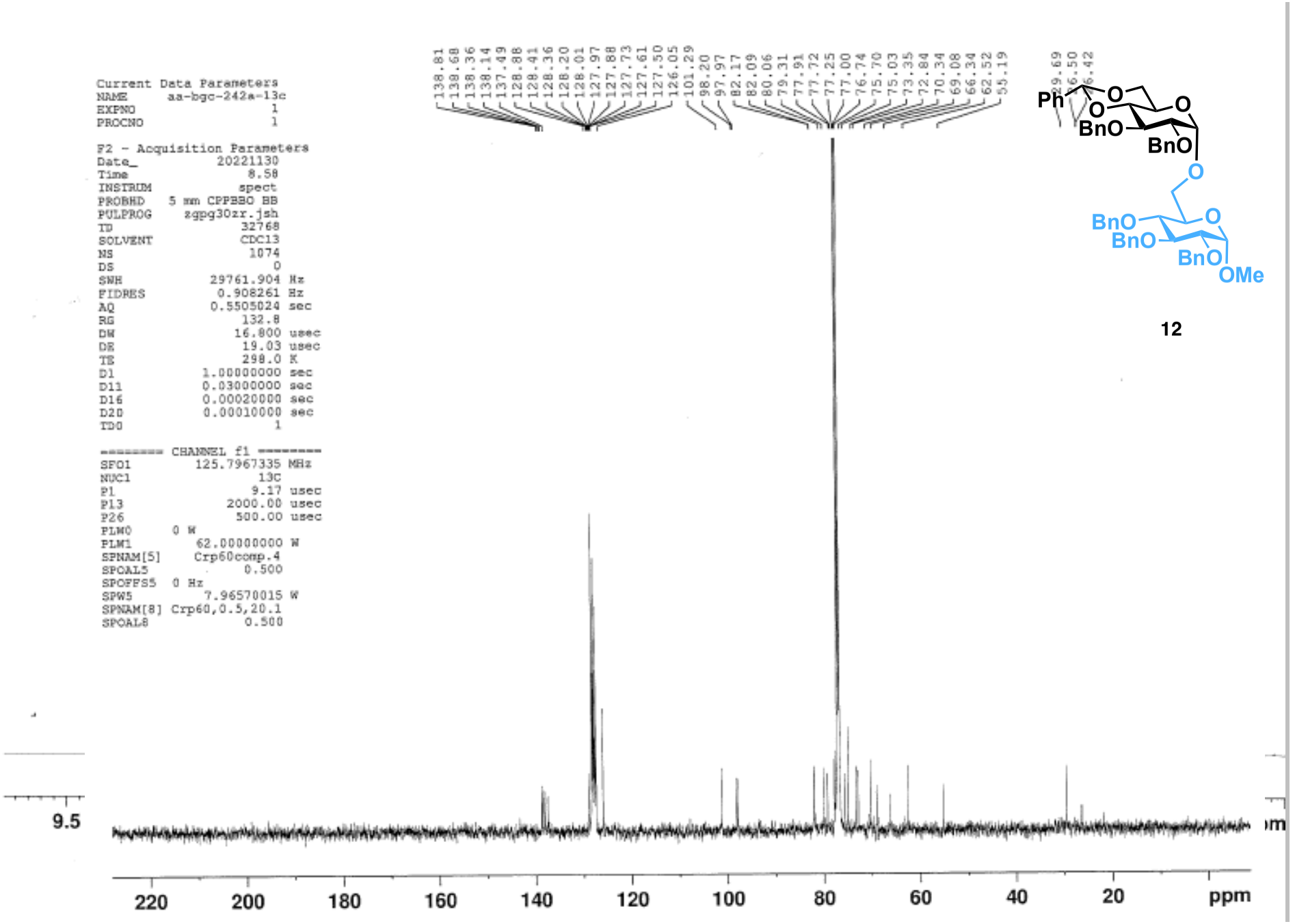
26.13
26.02
24.89
24.57

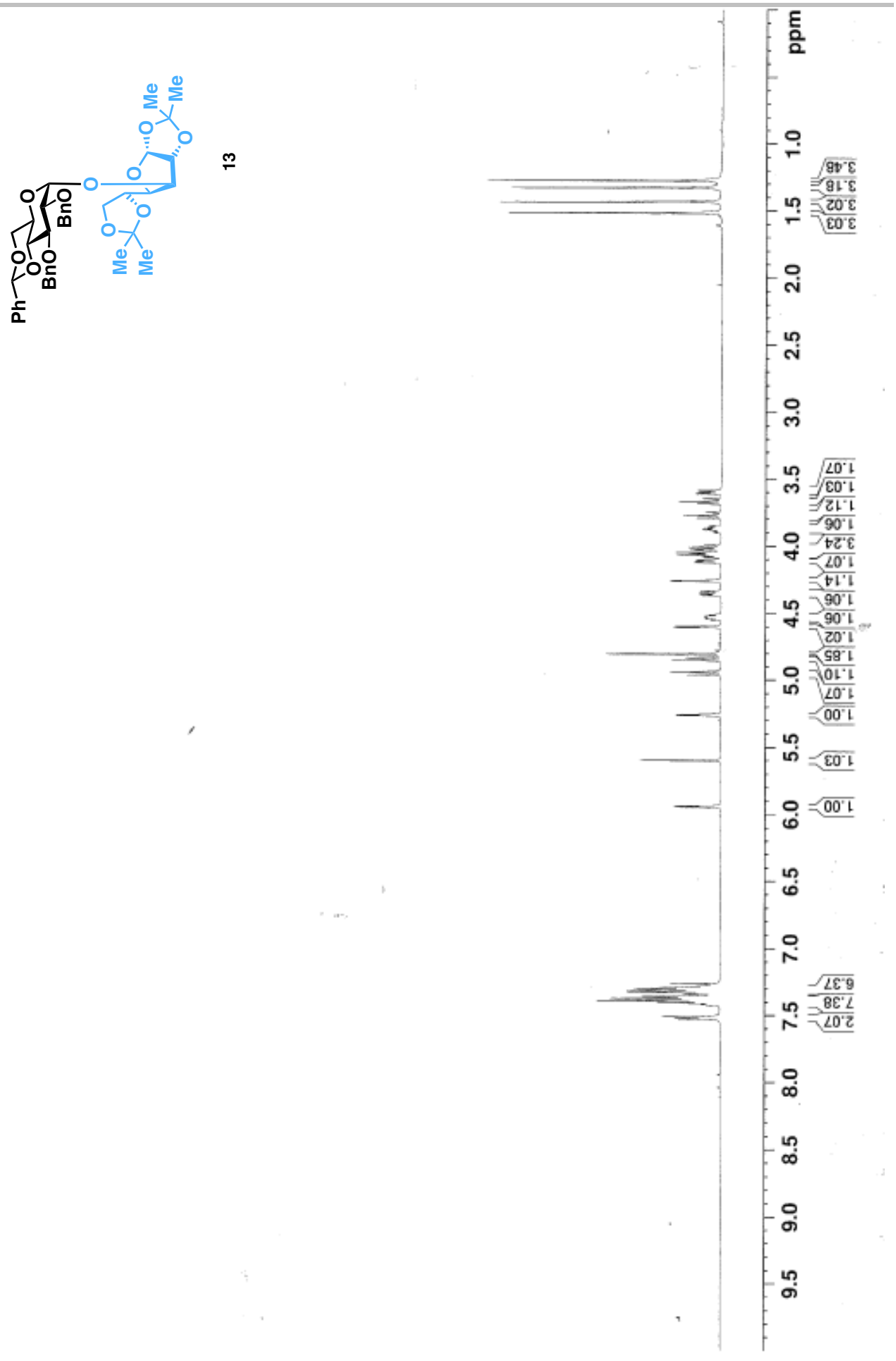


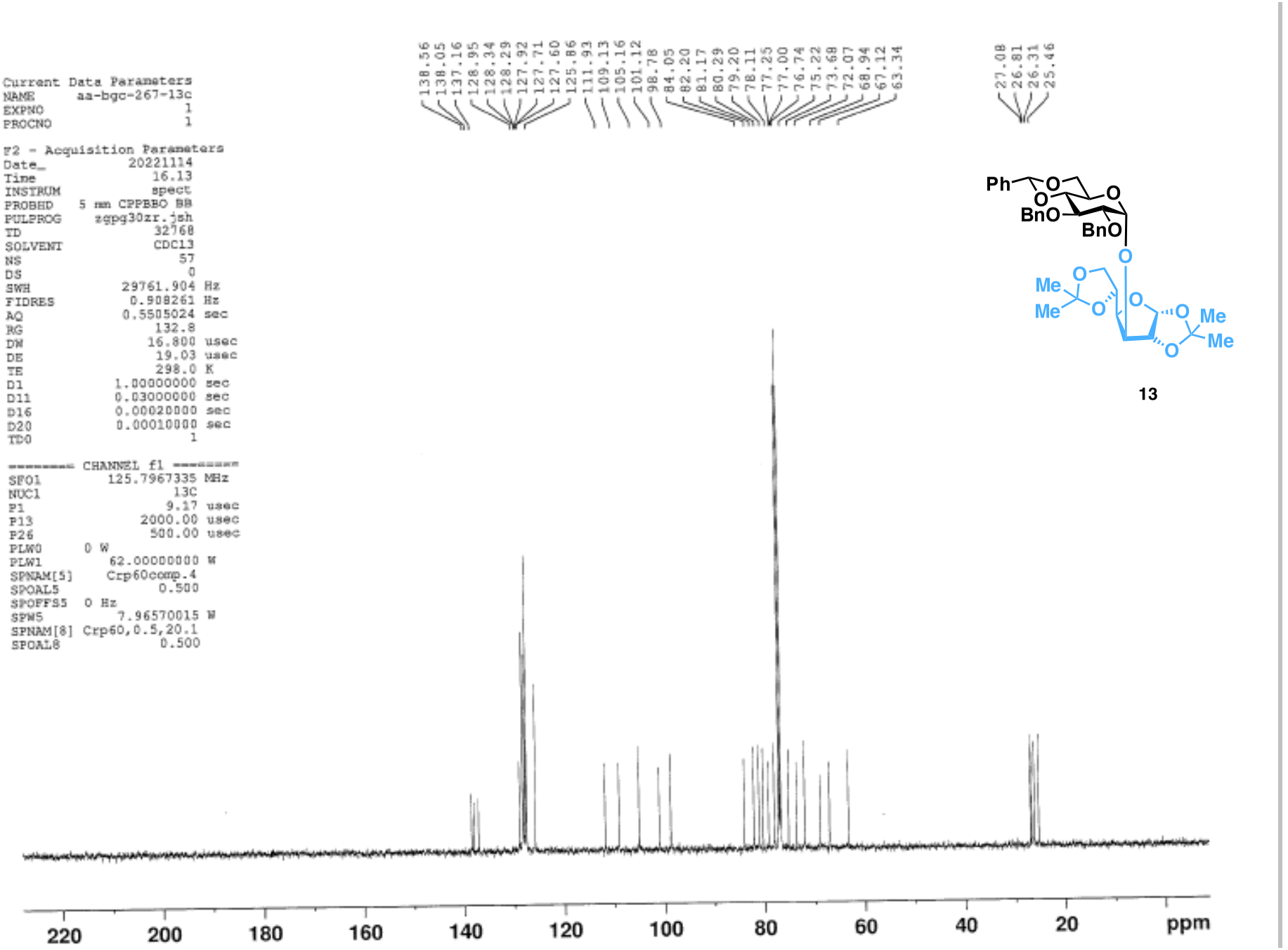
11

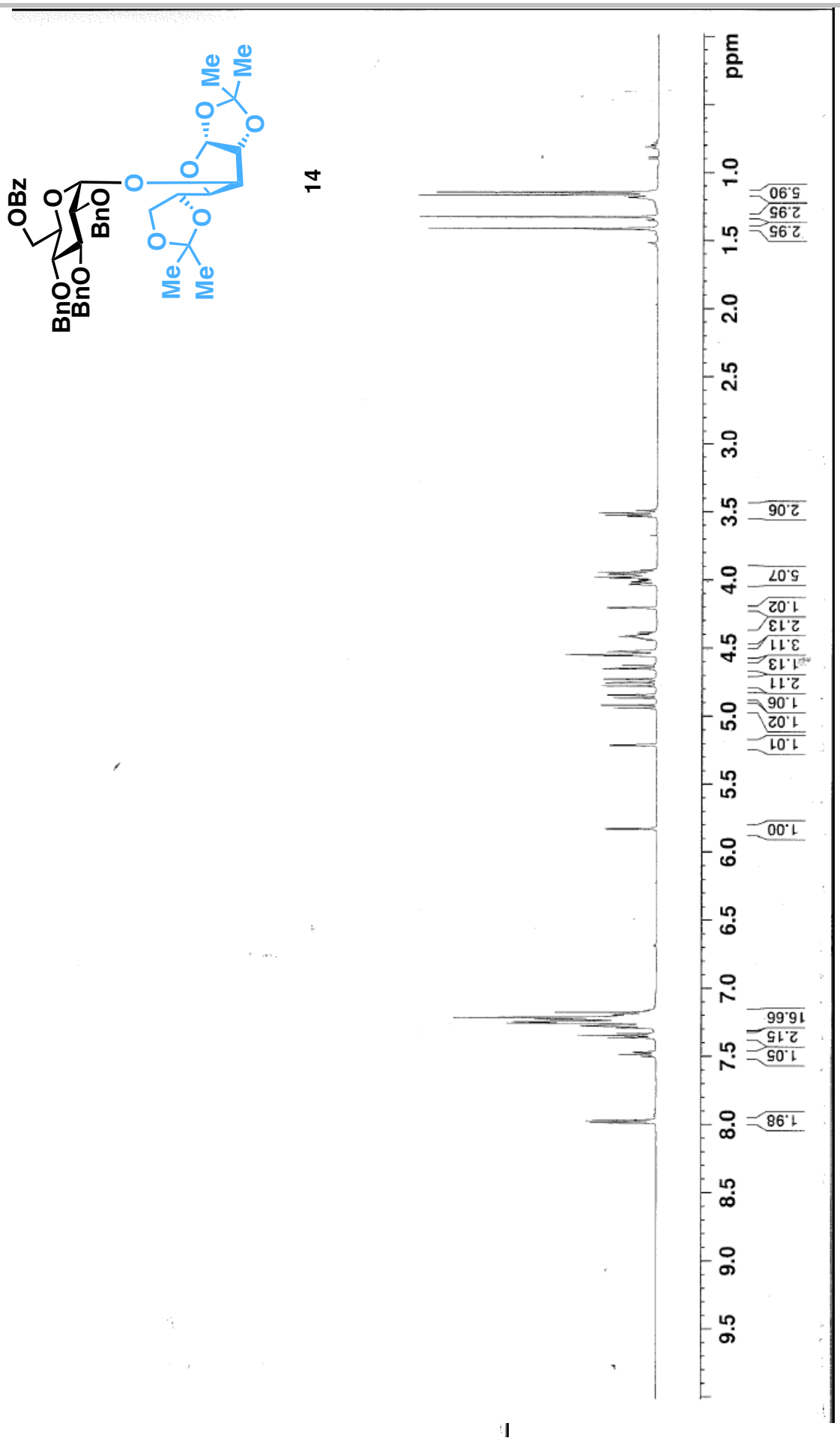


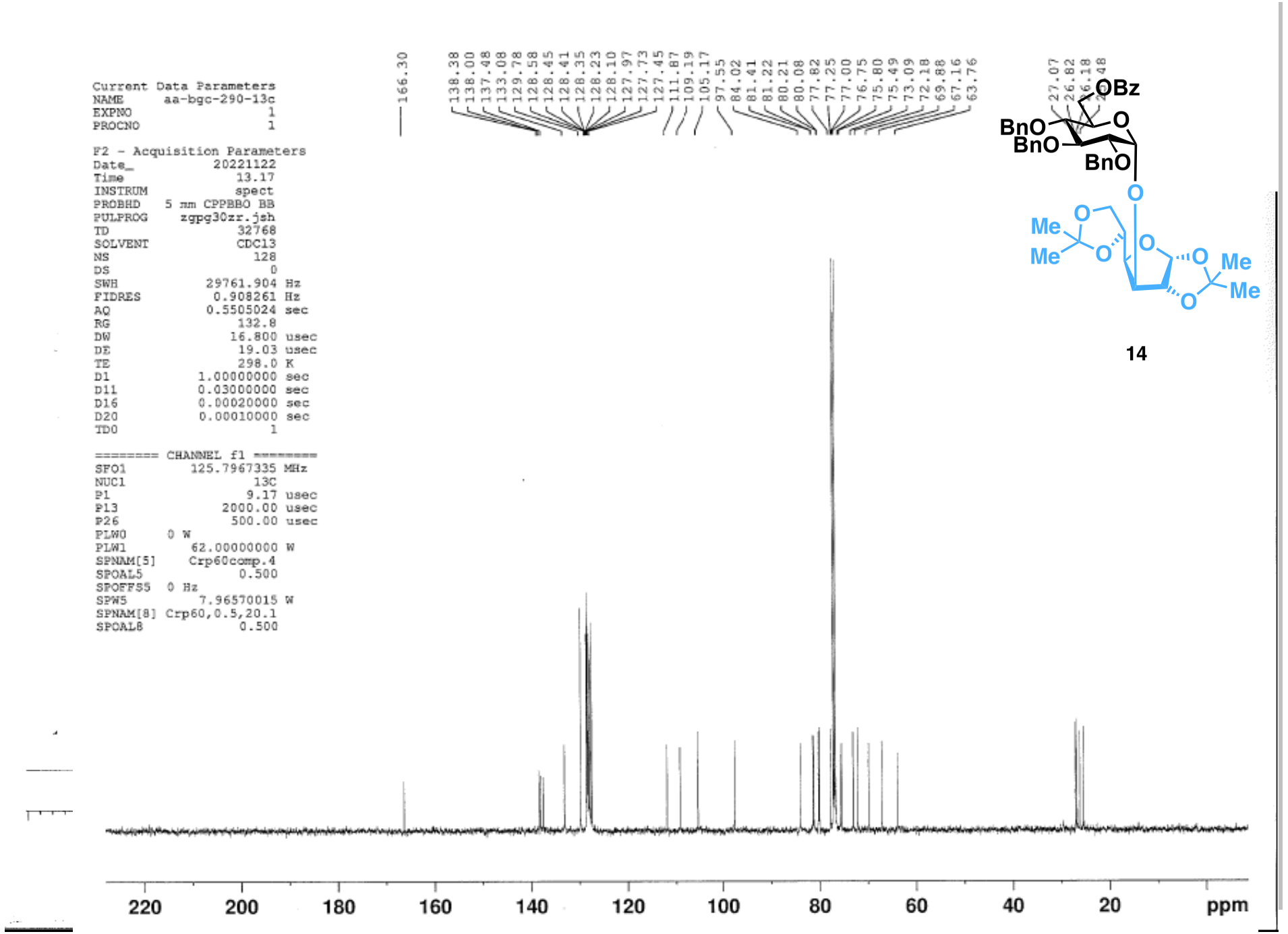

Ph
O
O
O
O
BnO
BnO
O
O
BnO
BnO
BnO
OMe
12
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
31.39
2.30
1.11
4.50
2.23
4.53
2.17
1.07
1.11
3.42
3.33
1.25
1.08
3.00
0.15


Current Data Parameters
NAME aa-bgc-242a-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221130
Time 8.58
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 1074
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
138.81
138.68
138.36
138.14
137.49
128.88
128.41
128.36
128.20
128.01
127.97
127.88
127.73
127.61
127.50
126.05
101.29
98.20
97.97
82.17
82.09
80.06
79.31
77.91
77.72
77.25
77.00
76.74
75.70
75.03
73.35
72.84
70.34
69.08
66.34
62.52
55.19
29.69
26.50
26.42
Ph
O
BnO
BnO
O
BnO
BnO
O
BnO
OMe
12
9.5
220
200
180
160
140
120
100
80
60
40
20
ppm

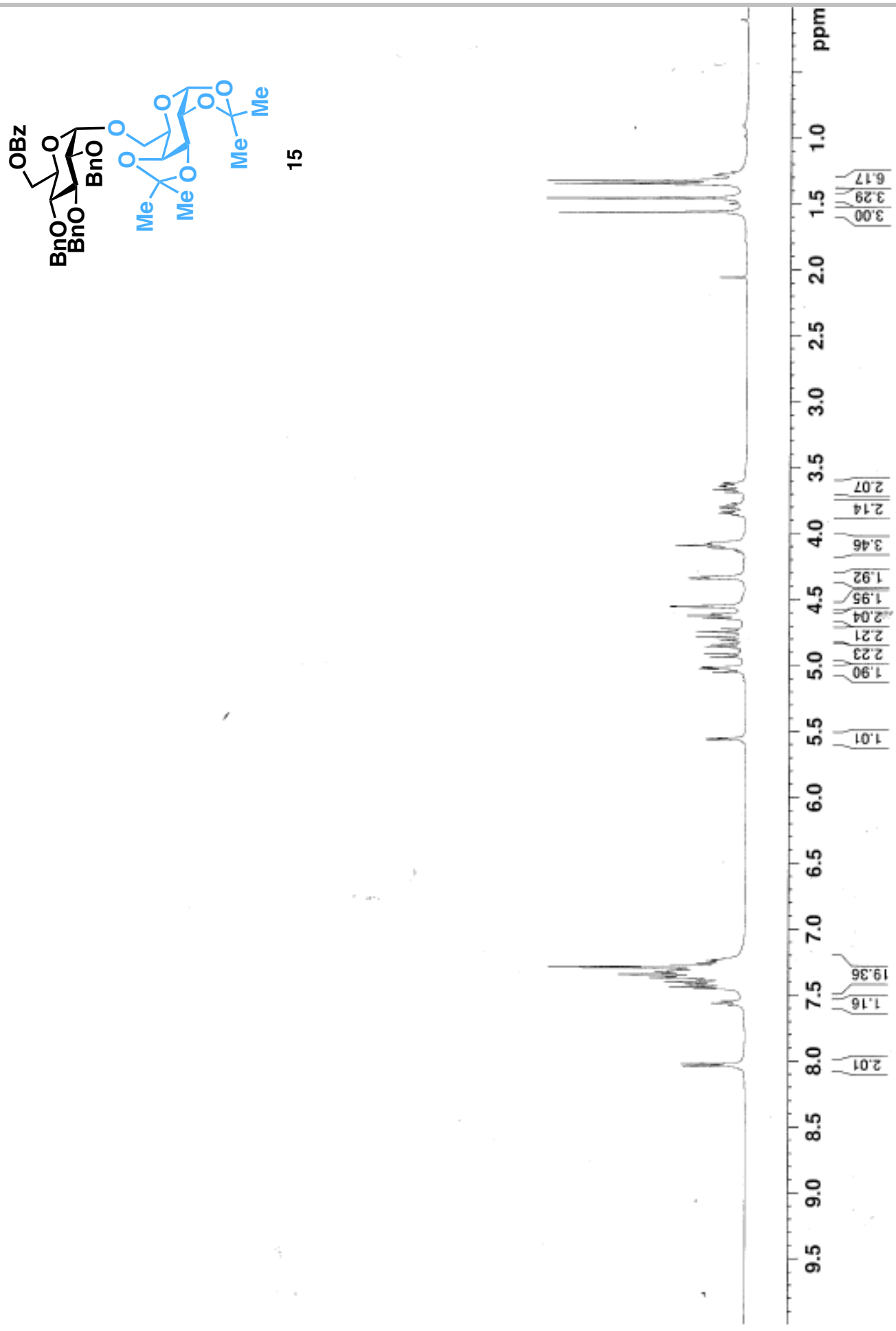

Ph
BnO
BnO
O
O
O
O
O
O
O
Me
Me
Me
Me
13
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
3.03
3.02
3.18
3.48
1.07
1.03
1.12
1.06
3.24
1.07
1.14
1.06
1.06
1.02
1.85
1.10
1.07
1.00
1.03
1.00
2.07
7.38
6.37

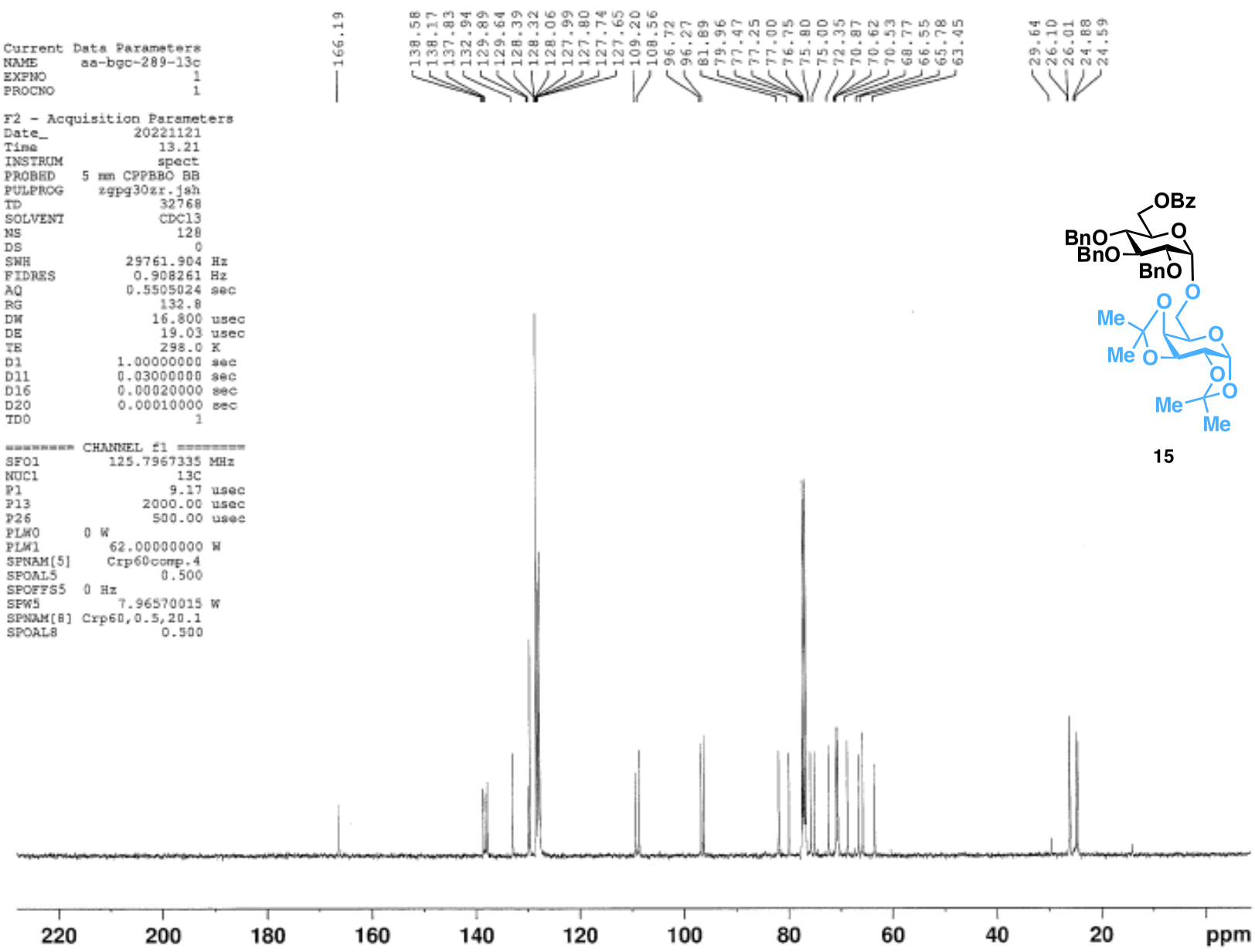

Current Data Parameters
NAME aa-bgc-267-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221114
Time 16.13
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 57
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
138.56
138.05
137.16
128.95
128.34
128.29
127.92
127.71
127.60
125.86
111.93
109.13
105.16
101.12
98.78
84.05
82.20
81.17
80.29
79.20
78.11
77.25
77.00
76.74
75.22
73.68
72.07
68.94
67.12
63.34
27.08
26.81
26.31
25.46
Ph
BnO
BnO
Me
Me
Me
Me
13
220
200
180
160
140
120
100
80
60
40
20
ppm


OBz
BnO
BnO
BnO
O
O
O
O
O
O
Me
Me
Me
Me
14
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
2.95
2.95
5.90
2.06
5.07
1.02
2.13
3.11
1.13
2.11
1.06
1.02
1.01
1.00
16.66
2.15
1.05
1.98

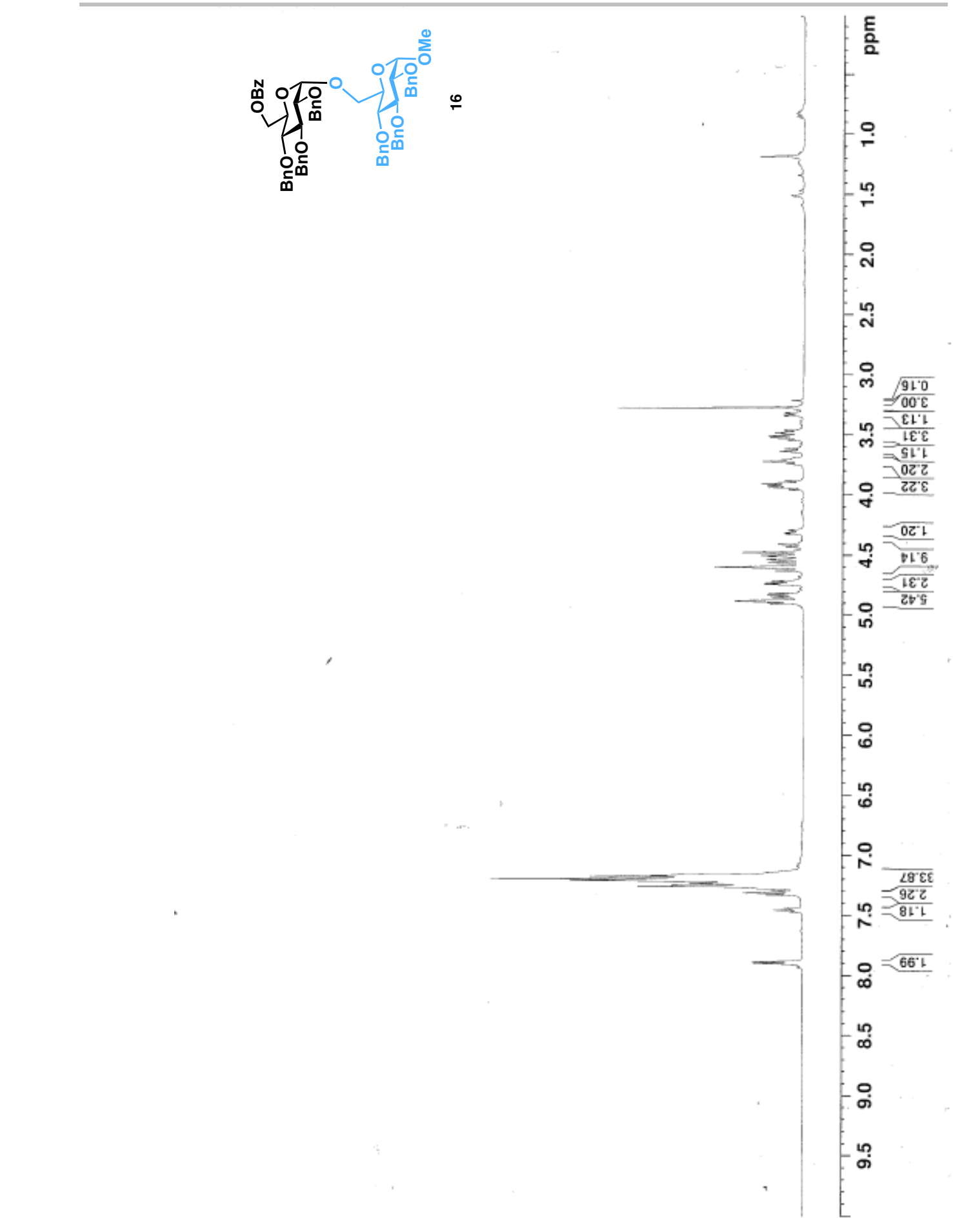

Current Data Parameters
NAME aa-bgc-290-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221122
Time 13.17
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
166.30
138.38
138.00
137.48
133.08
129.78
128.58
128.45
128.41
128.35
128.23
128.10
127.97
127.73
127.45
111.87
109.19
105.17
97.55
84.02
81.41
81.22
80.21
80.08
77.82
77.25
77.00
76.75
75.80
75.49
73.09
72.18
69.88
67.16
63.76
27.07
26.82
26.18
24.48
OBz
BnO
BnO
BnO
O
O
Me
Me
O
O
O
O
Me
Me
O
220
200
180
160
140
120
100
80
60
40
20
ppm

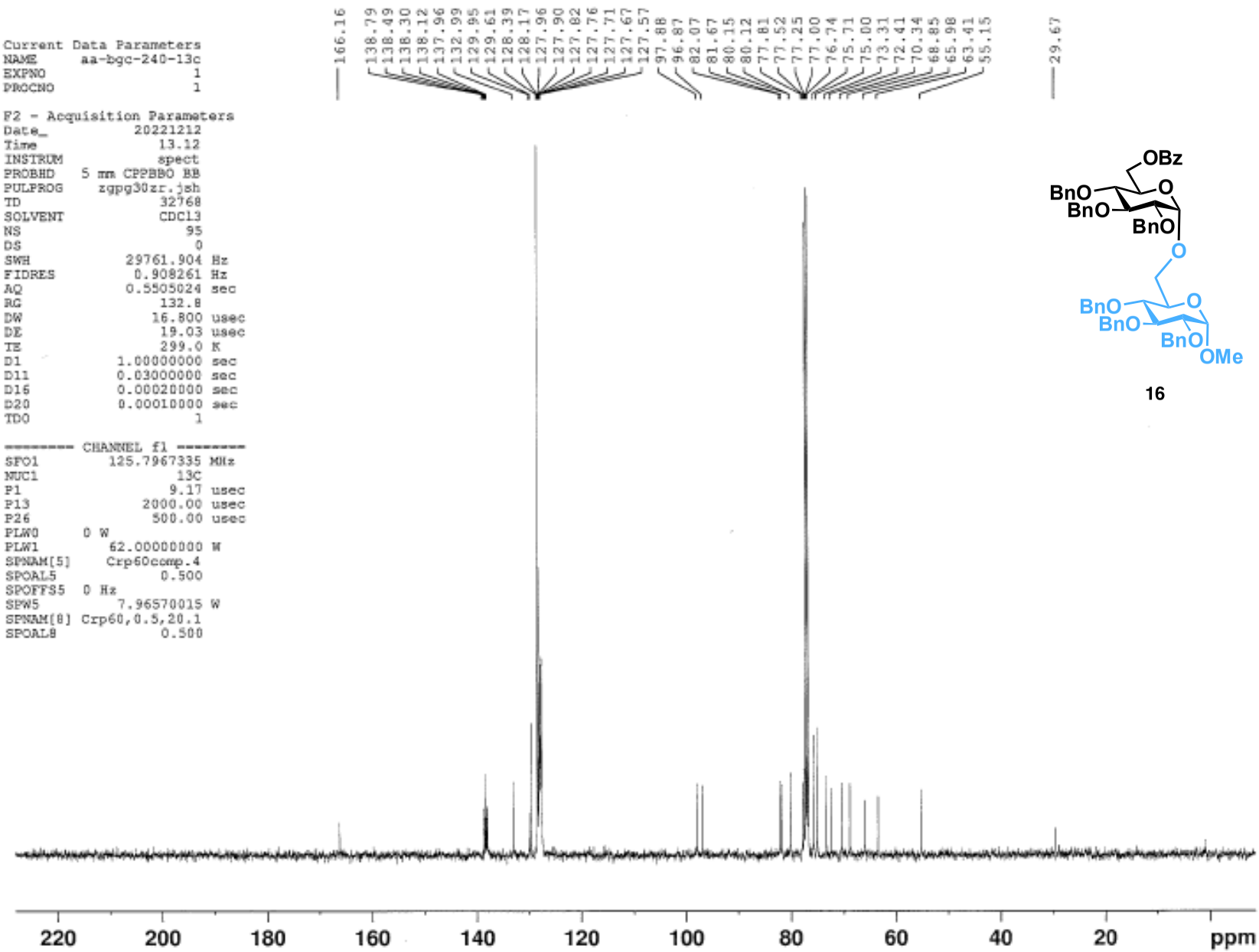
14

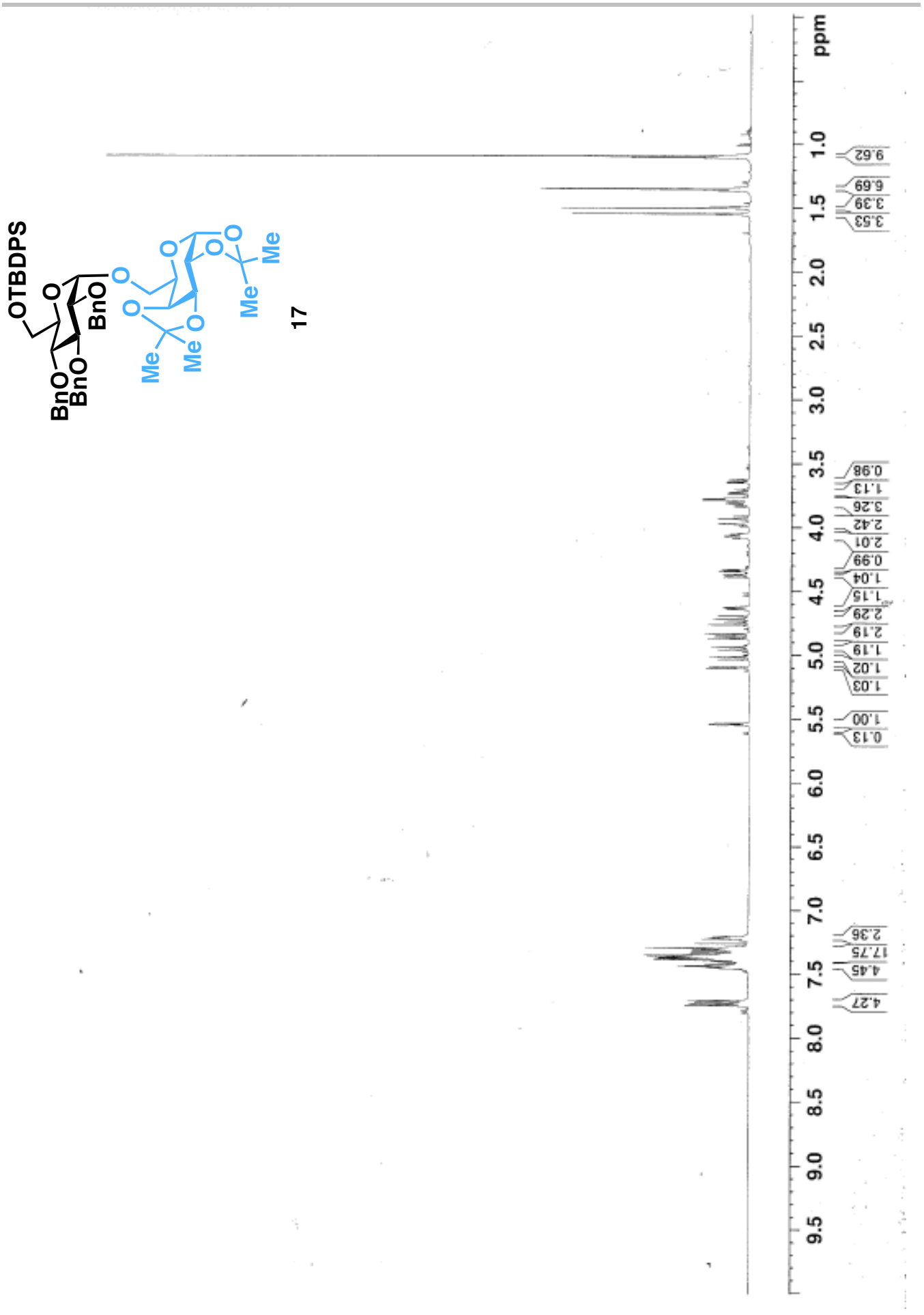

OBz
BnO
BnO
BnO
O
O
O
O
O
O
O
Me
Me
Me
Me
15
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
6.17
3.29
3.00
2.07
2.14
3.46
1.92
1.95
2.04
2.21
2.23
1.90
1.01
19.36
1.16
2.01

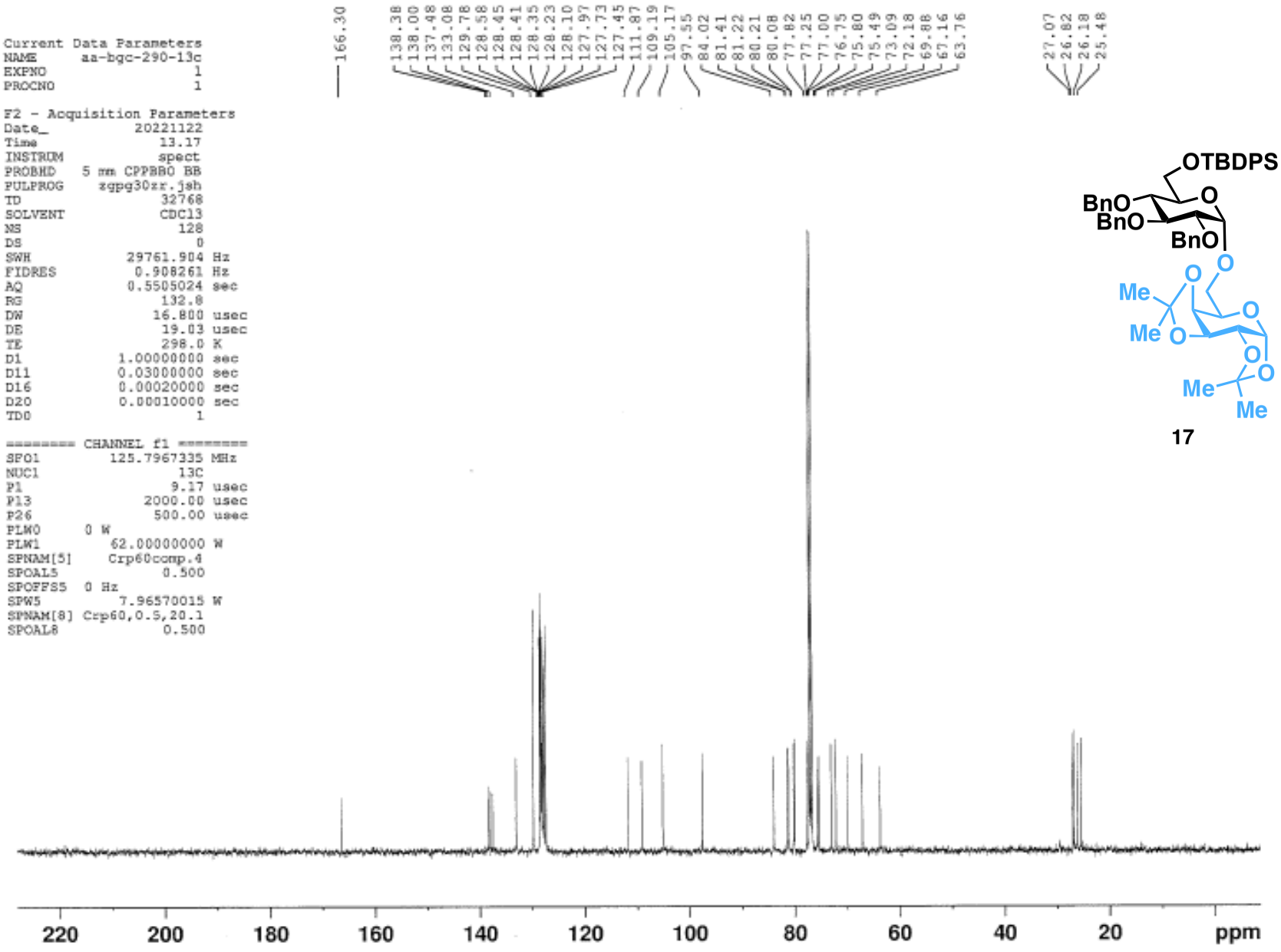

Current Data Parameters
NAME aa-bgc-289-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221121
Time 13.21
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jah
TD 32768
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
166.19
138.58
138.17
137.83
132.94
129.89
129.64
128.39
128.32
128.06
127.99
127.80
127.74
127.65
109.20
108.56
96.72
96.27
81.89
79.96
77.47
77.25
77.00
76.75
75.80
75.00
72.35
70.87
70.62
70.53
68.77
66.55
65.78
63.45
29.64
26.10
26.01
24.88
24.59
OBz
BnO
BnO
BnO
O
O
O
Me
Me
O
O
O
Me
O
Me
220
200
180
160
140
120
100
80
60
40
20
ppm

15

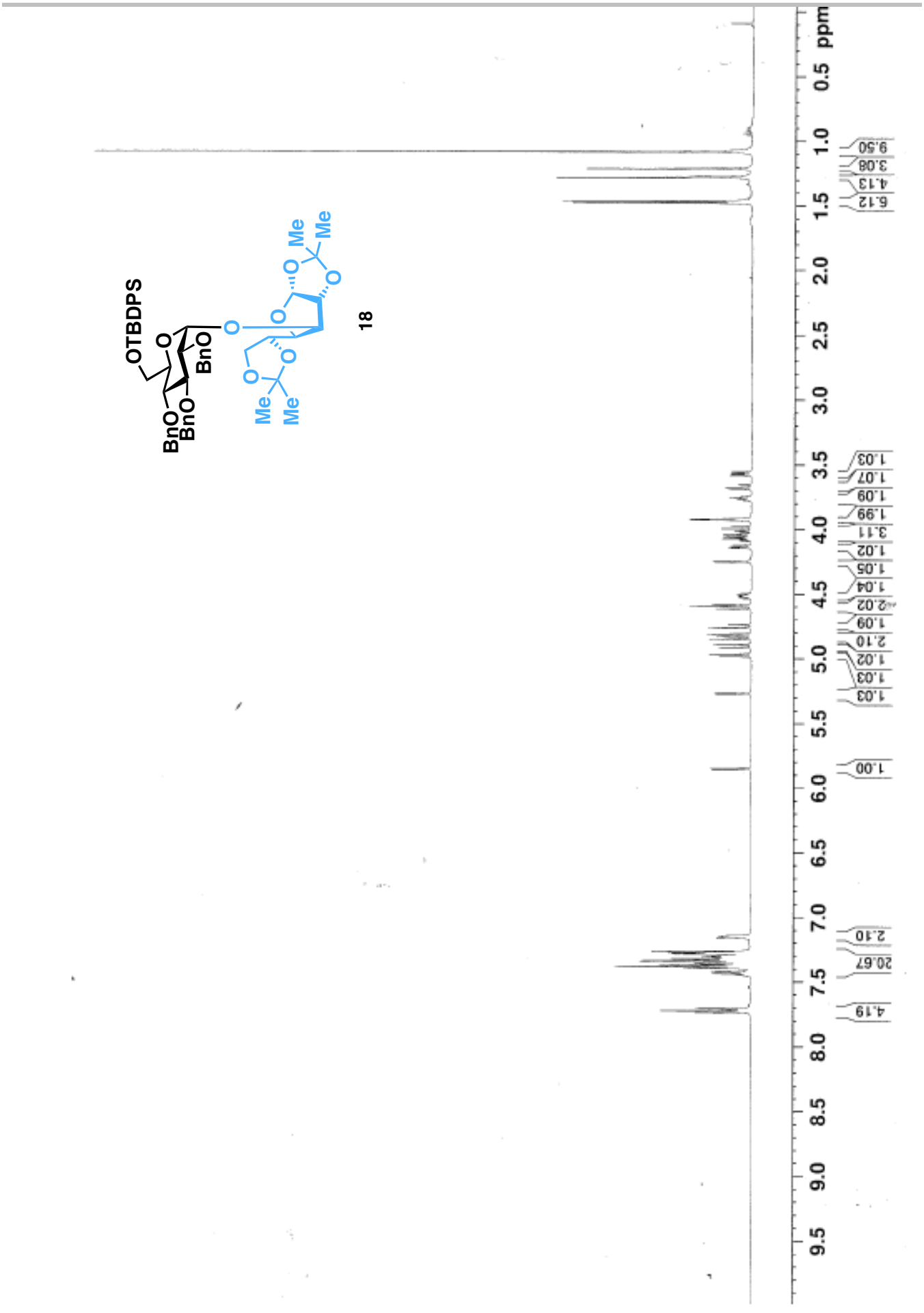

OBz
BnO
BnO
BnO
BnO
BnO
BnO
OMe
16
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.16
3.00
1.13
3.31
1.15
2.20
3.22
1.20
9.14
2.31
5.42
1.99
33.87
2.26
1.18

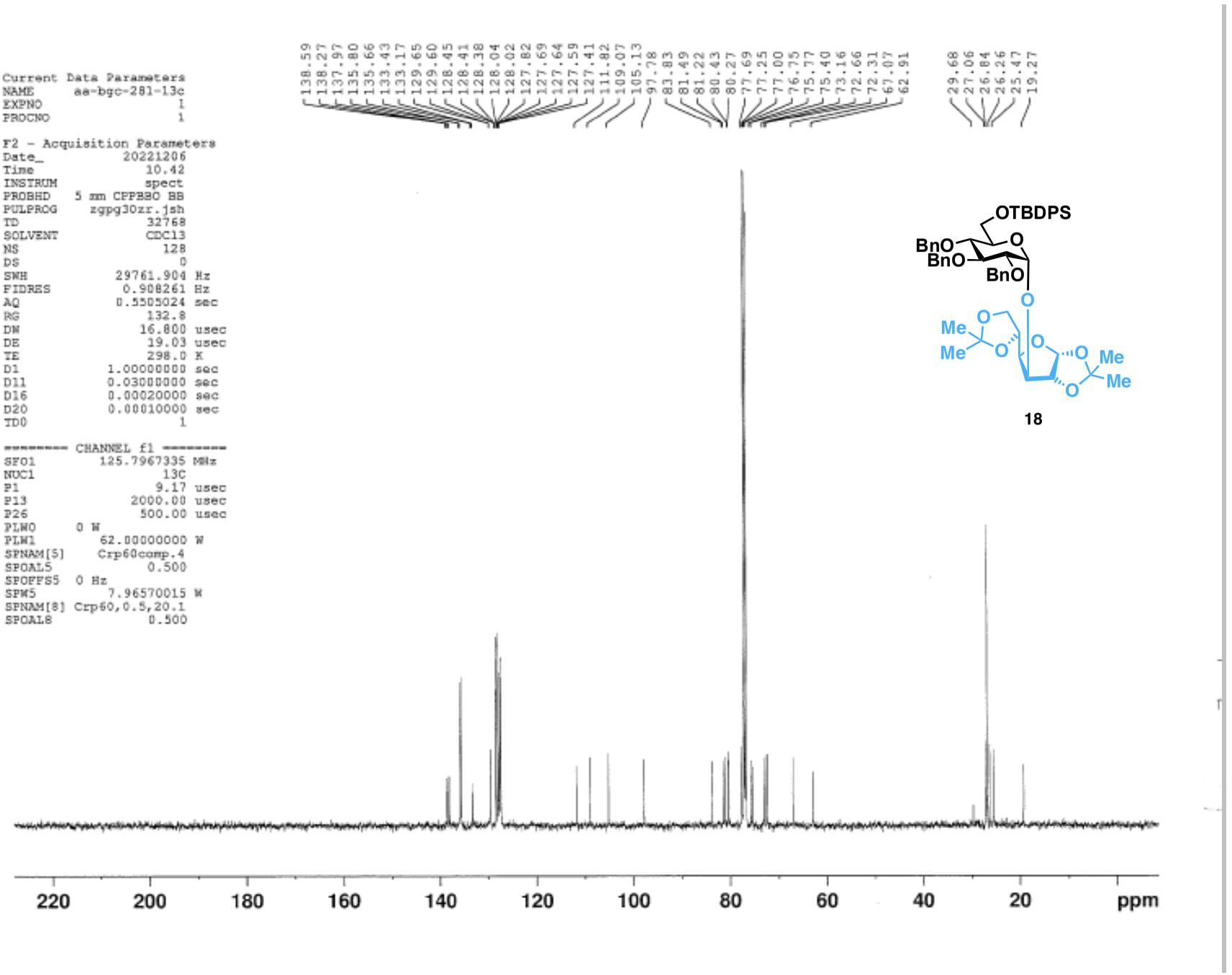

Current Data Parameters
NAME aa-bgc-240-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221212
Time 13.12
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 95
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 299.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
166.16
138.79
138.49
138.30
138.12
137.96
132.99
129.95
129.61
128.39
128.17
127.96
127.90
127.82
127.76
127.71
127.67
127.57
97.88
96.87
82.07
81.67
80.15
80.12
77.81
77.52
77.25
77.00
76.74
75.71
75.00
73.31
72.41
70.34
68.85
65.98
63.41
55.15
29.67
OBz
BnO
BnO
BnO
O
BnO
BnO
BnO
O
OMe
16
220
200
180
160
140
120
100
80
60
40
20
ppm

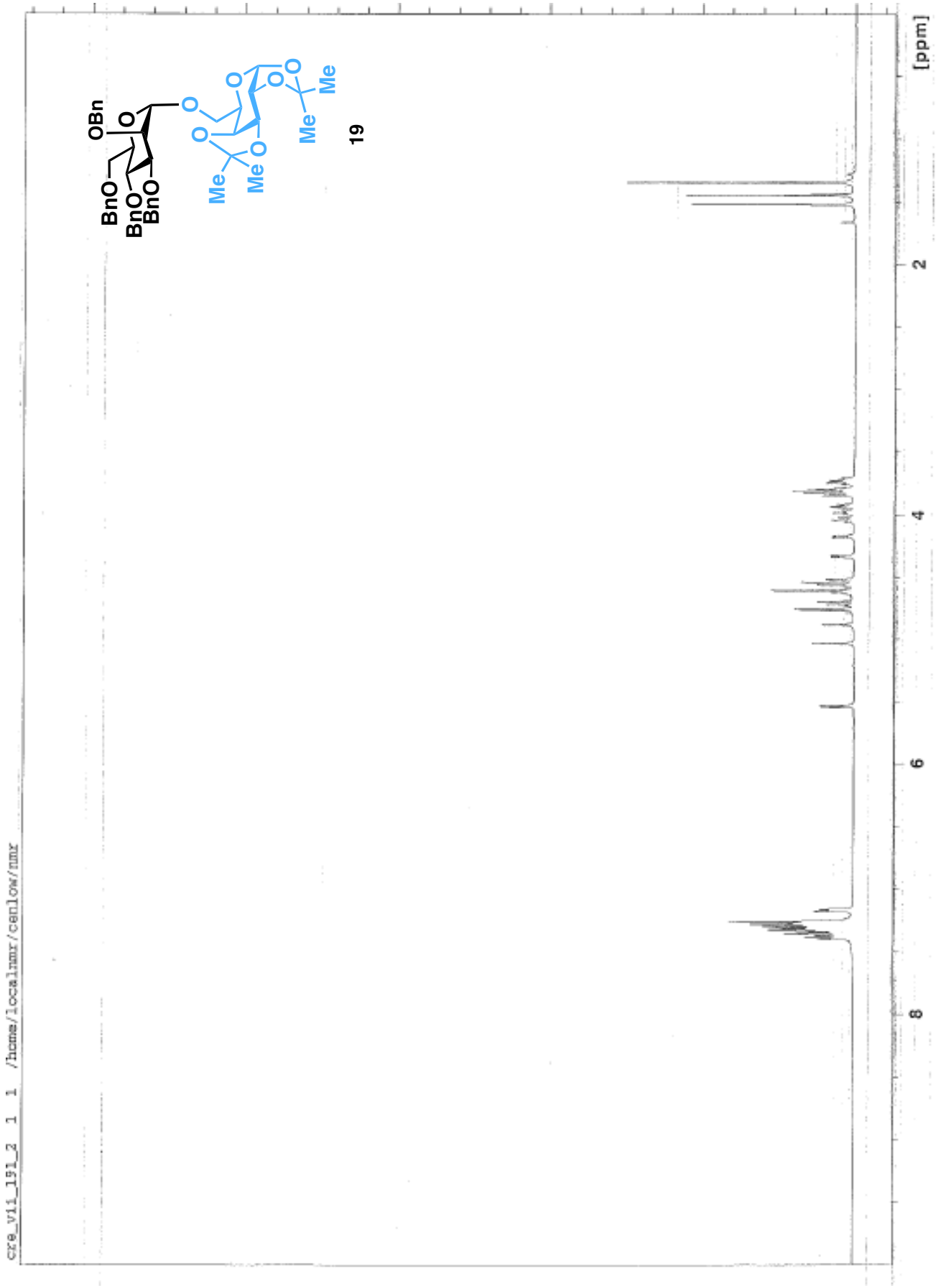

OTBDPS
BnO
BnO
BnO
O
O
O
O
O
O
O
Me
Me
Me
Me
17
ppm
9.62
6.69
3.39
3.53
0.98
1.13
3.26
2.42
2.01
0.99
1.04
1.15
2.29
2.19
1.19
1.02
1.03
0.13
1.00
2.36
17.75
4.45
4.27

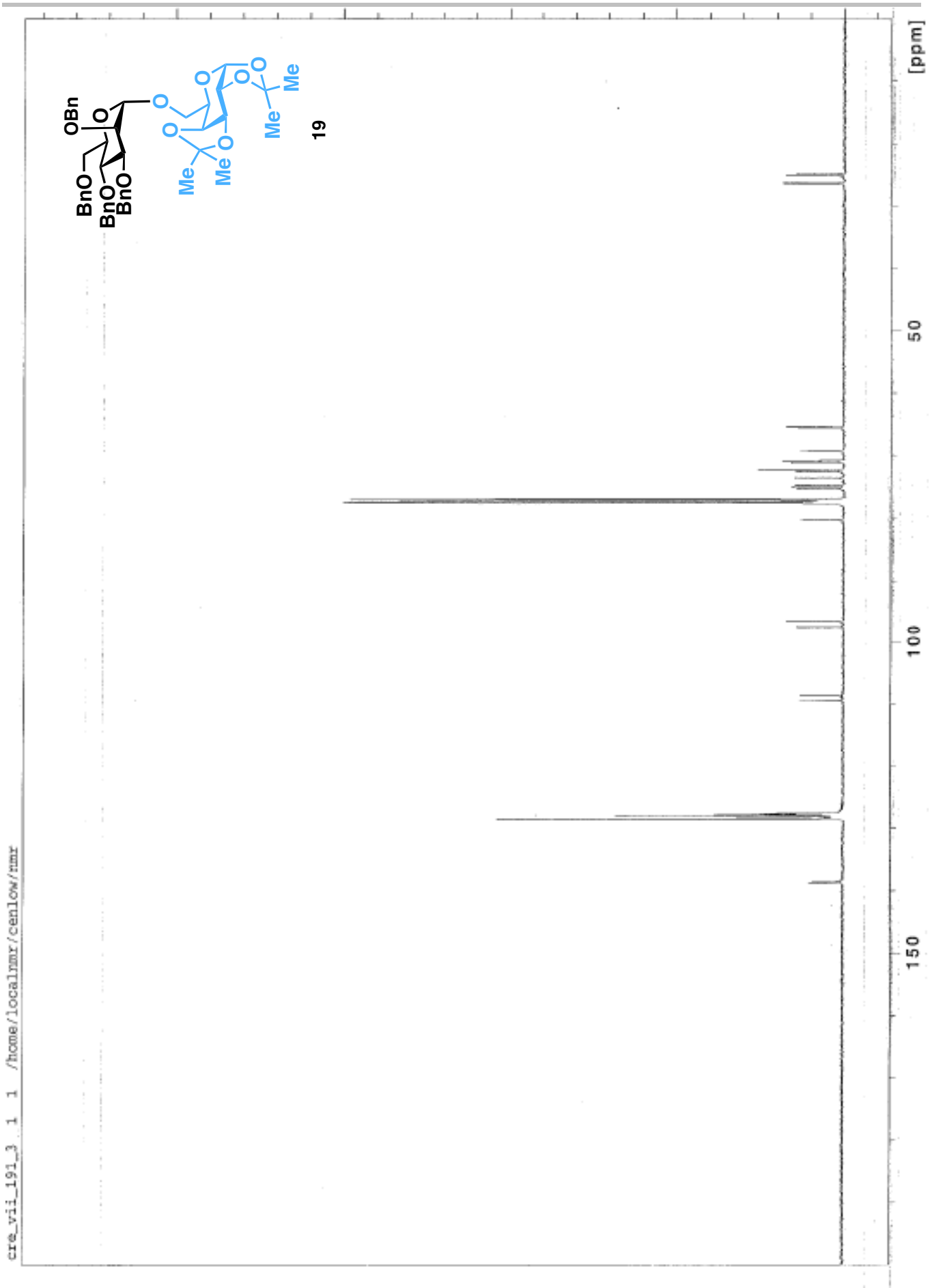

Current Data Parameters
NAME aa-bgc-290-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221122
Time 13.17
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
166.30
138.38
138.00
137.48
133.08
129.78
128.58
128.45
128.41
128.35
128.23
128.10
127.97
127.73
127.45
111.87
109.19
105.17
97.55
84.02
81.41
81.22
80.21
80.08
77.82
77.25
77.00
76.75
75.80
75.49
73.09
72.18
69.88
67.16
63.76
27.07
26.82
26.18
25.48
OTBDPS
BnO
BnO
BnO
O
Me
Me
Me
Me
220
200
180
160
140
120
100
80
60
40
20
ppm

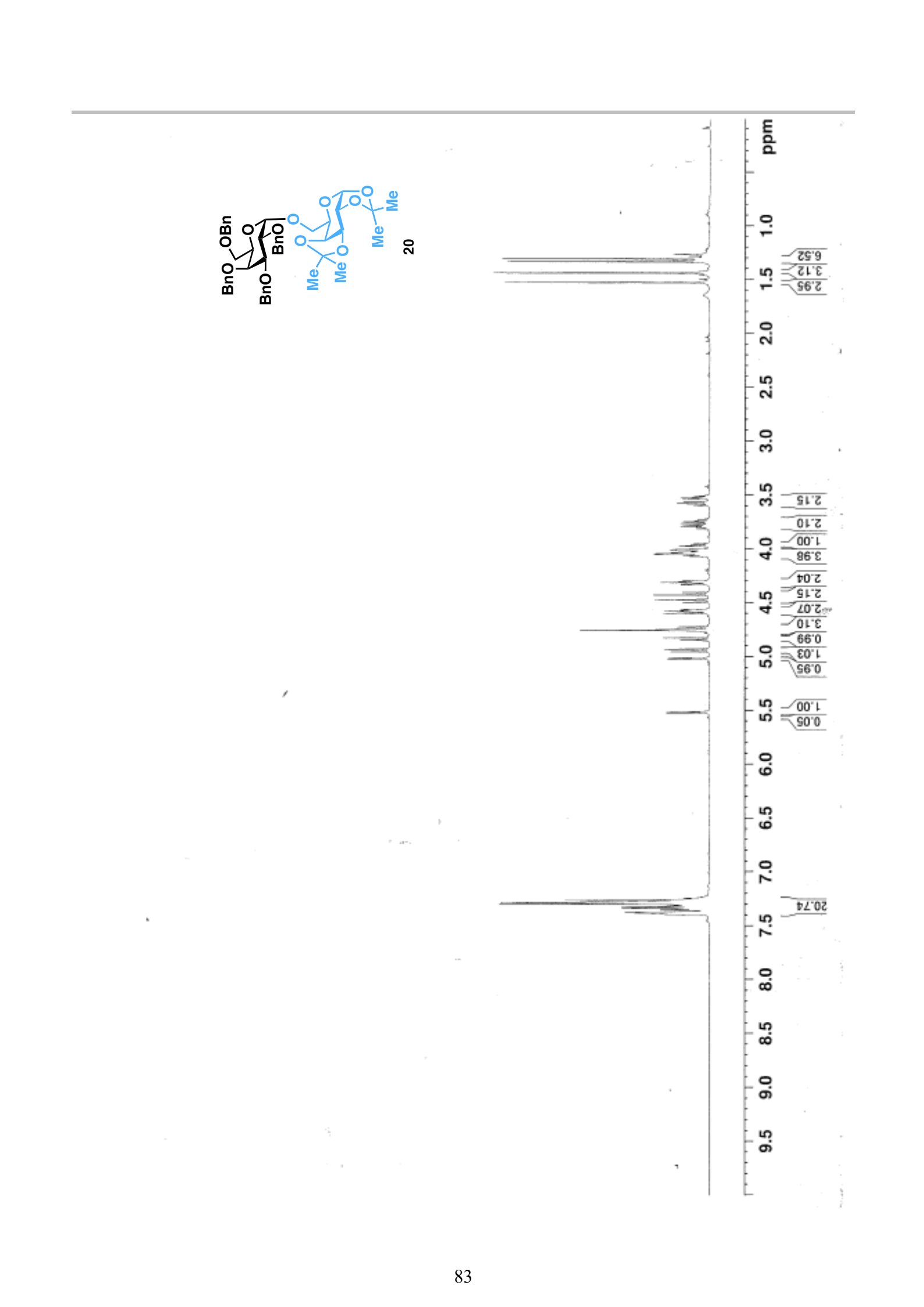
17

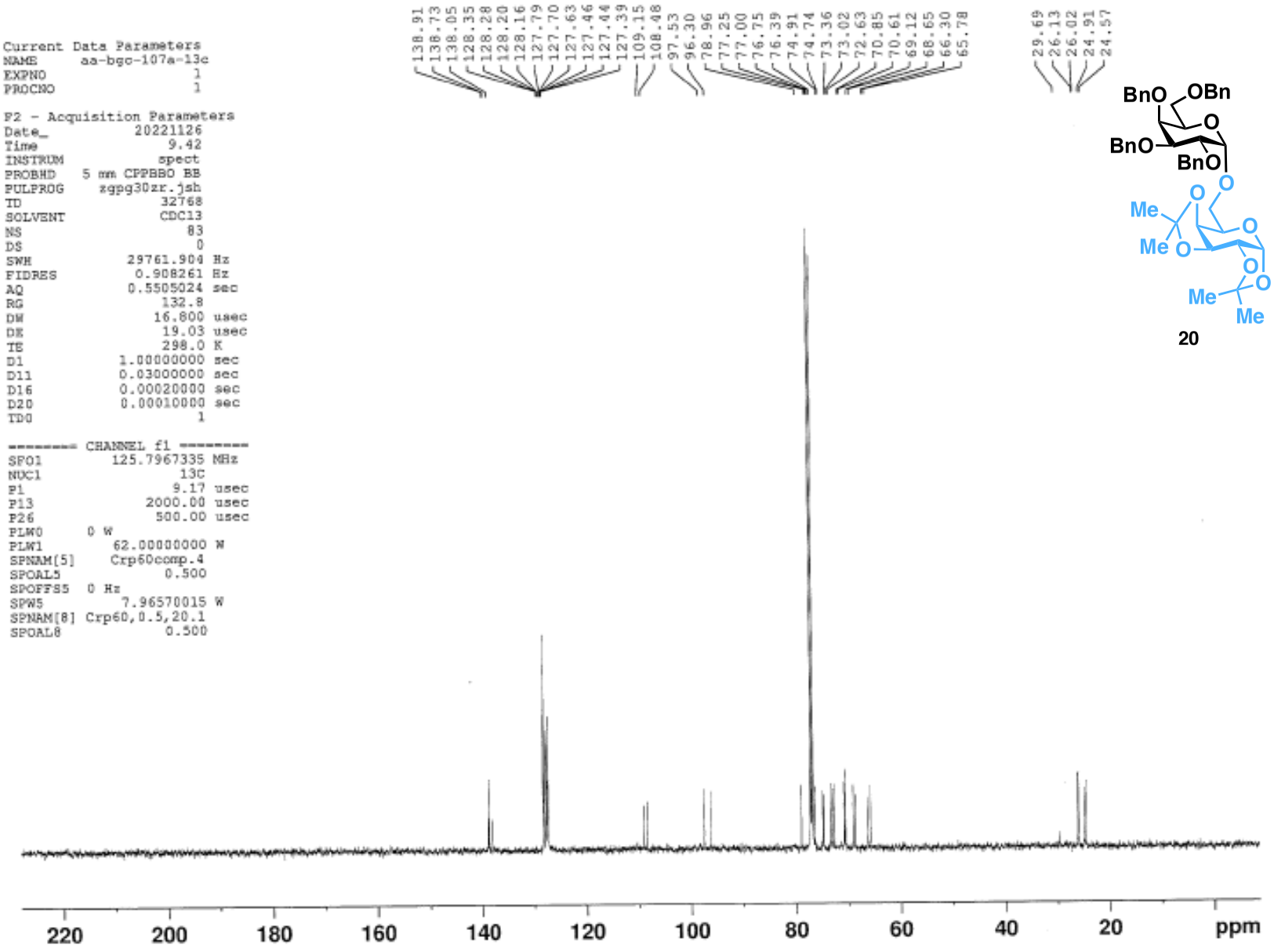

OTBDPS
BnO
BnO
BnO
O
O
O
O
O
O
O
Me
Me
Me
Me
18
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
9.50
3.08
4.13
5.12
1.03
1.07
1.09
1.99
3.11
1.02
1.05
1.04
2.02
1.09
2.10
1.02
1.03
1.03
1.00
2.10
20.67
4.19


Current Data Parameters
NAME aa-bgc-281-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221206
Time 10.42
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
PIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
138.59
138.27
137.97
135.80
135.66
133.43
133.17
129.65
129.60
128.45
128.41
128.38
128.04
128.02
127.82
127.69
127.64
127.59
127.41
111.82
109.07
105.13
97.78
83.83
81.49
81.22
80.43
80.27
77.69
77.25
77.00
76.75
75.77
75.40
73.16
72.66
72.31
67.07
62.91
29.68
27.06
26.84
26.26
25.47
19.27
OTBDPS
BnO
BnO
BnO
O
O
Me
Me
O
O
O
O
Me
Me
18
220
200
180
160
140
120
100
80
60
40
20
ppm

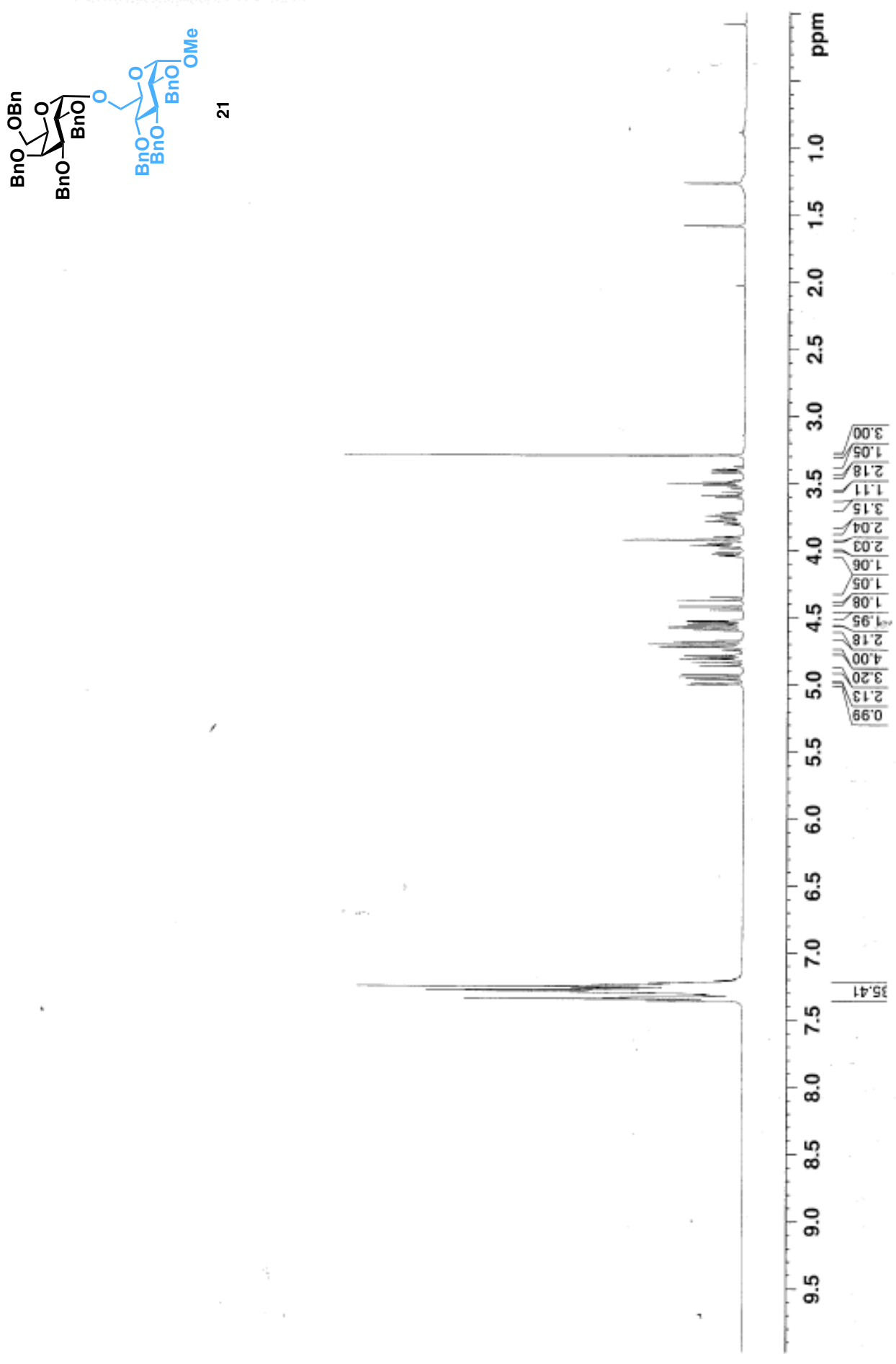

cre_v11_191_2 1 1 /home/localnmr/cenlow/nmr
OBn
BnO
BnO
BnO
Me
Me
Me
Me
19
8
6
4
2
[ppm]

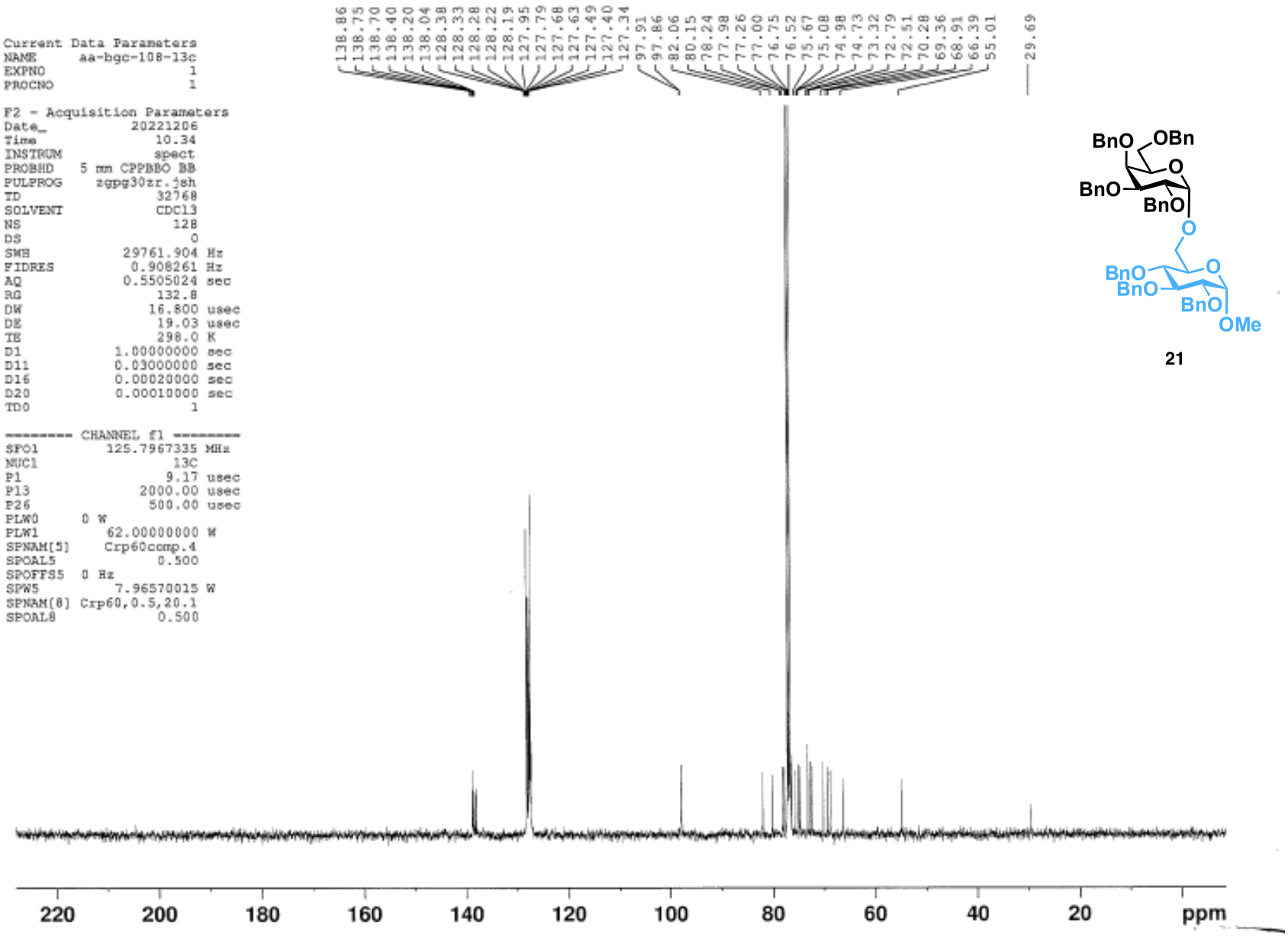

cre_vii_191_3 1 1 /home/localnmr/cenlow/nmr
19
OBn
BnO
BnO
BnO
O
O
O
O
O
O
Me
Me
Me
Me
150
100
50
[ppm]

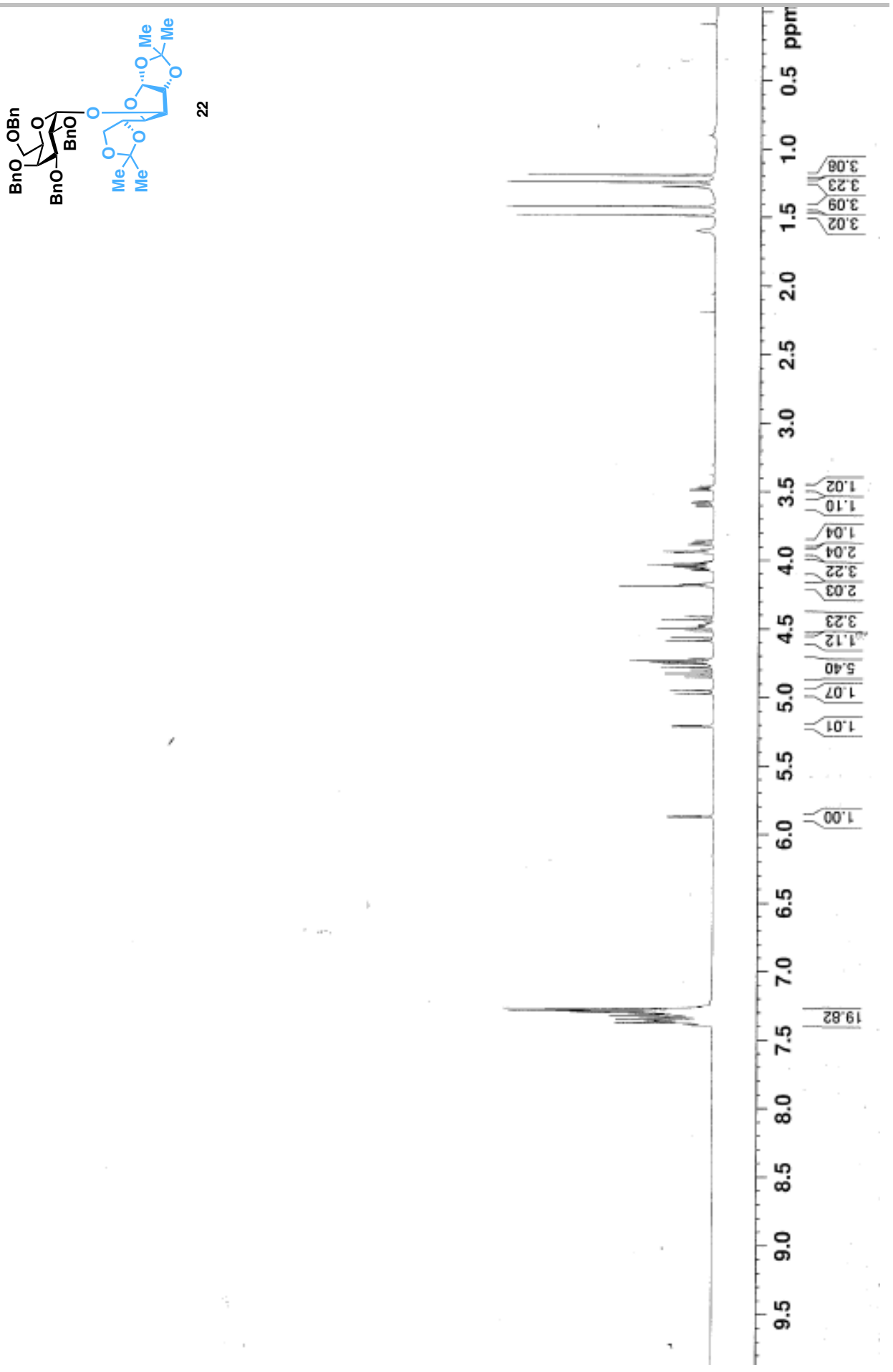

BnO
OBn
BnO
BnO
O
O
O
O
O
Me
Me
Me
Me
20
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
6.52
3.12
2.95
2.15
2.10
1.00
3.98
2.04
2.15
2.07
3.10
0.99
1.03
0.95
1.00
0.05
20.74

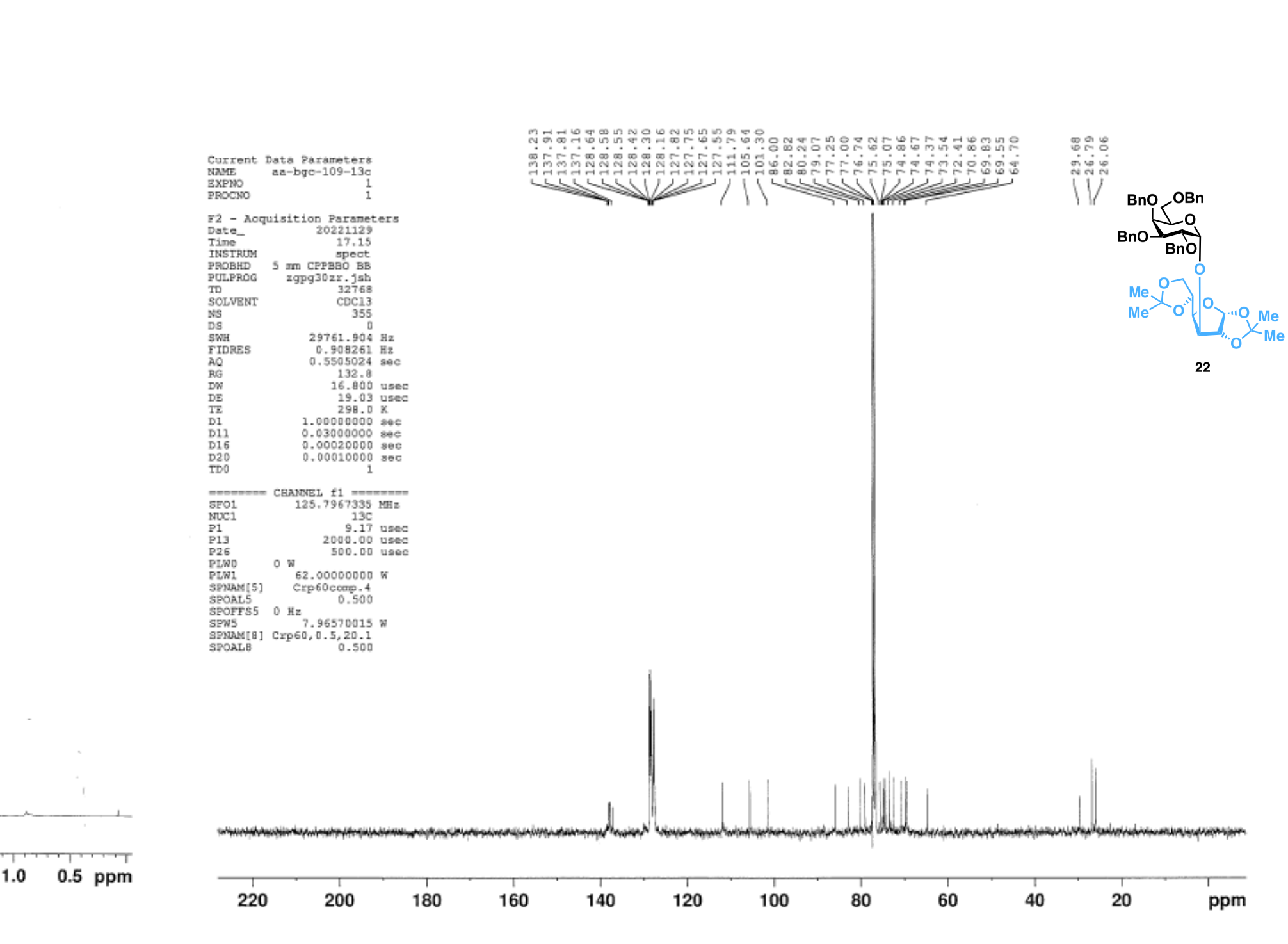

Current Data Parameters
NAME aa-bgc-107a-13c
EXPNO 1
PROCNO 1
P2 - Acquisition Parameters
Date_ 20221126
Time 9.42
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 83
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
138.91
138.73
138.05
128.35
128.28
128.20
128.16
127.79
127.70
127.63
127.46
127.44
127.39
109.15
108.48
97.53
96.30
78.96
77.25
77.00
76.75
76.39
74.91
74.74
73.36
73.02
72.63
70.85
70.61
69.12
68.65
66.30
65.78
29.69
26.13
26.02
24.91
24.57
BnO
OBn
O
BnO
BnO
O
O
Me
Me
O
O
O
Me
Me
220
200
180
160
140
120
100
80
60
40
20
ppm

20

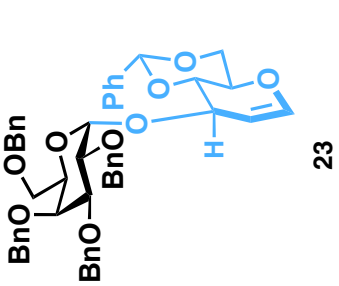

BnO
OBn
O
BnO
BnO
O
BnO
BnO
BnO
OMe
21
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
35.41
0.99
2.13
3.20
4.00
2.18
1.95
1.08
1.05
1.06
2.03
2.04
3.15
1.11
2.18
1.05
3.00


Current Data Parameters
NAME aa-bgc-108-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221206
Time 10.34
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
138.86
138.75
138.70
138.40
138.20
138.04
128.38
128.33
128.28
128.22
128.19
127.95
127.79
127.68
127.63
127.49
127.40
127.34
97.91
97.86
82.06
80.15
78.24
77.98
77.26
77.00
76.75
76.52
75.67
75.08
74.98
74.73
73.32
72.79
72.51
70.28
69.36
68.91
66.39
55.01
29.69
BnO
OBn
O
BnO
BnO
O
BnO
O
BnO
BnO
OMe
21
220
200
180
160
140
120
100
80
60
40
20
ppm

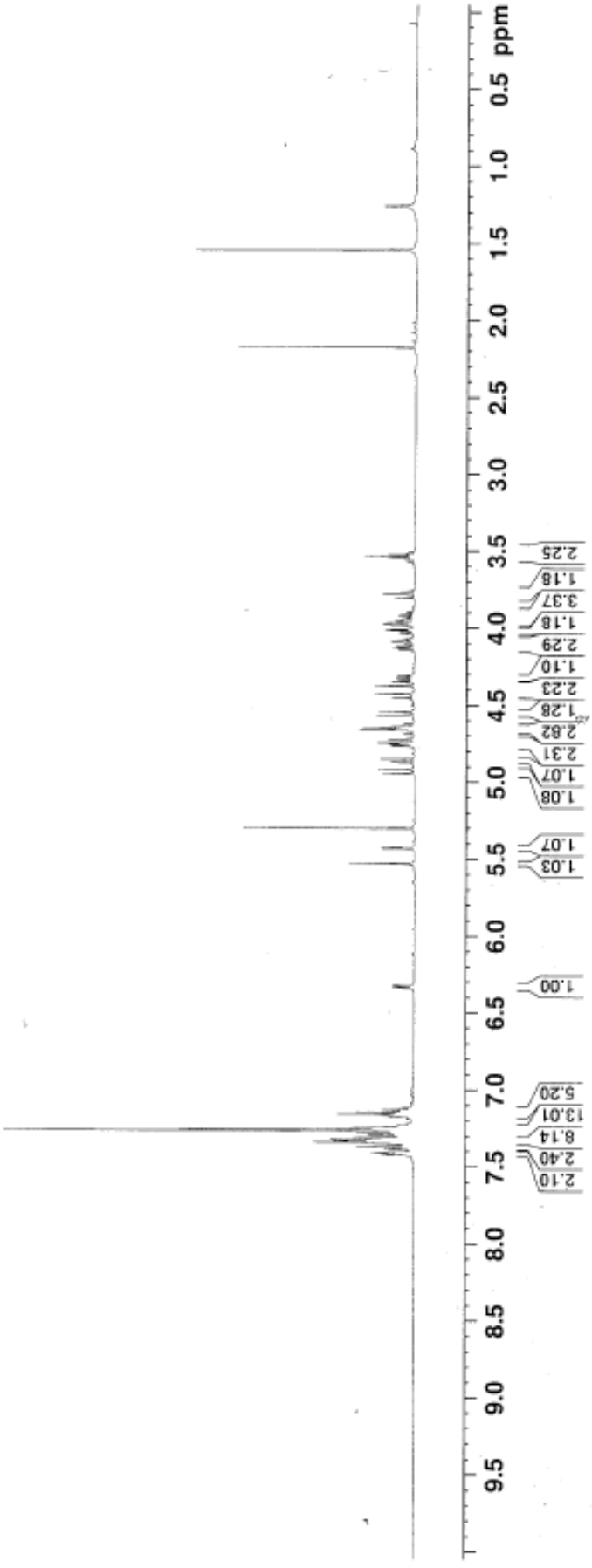

BnO
OBn
BnO
BnO
O
O
O
O
O
O
Me
Me
Me
Me
22
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
3.08
3.23
3.09
3.02
1.02
1.10
1.04
2.04
3.22
2.03
3.23
1.12
5.40
1.07
1.01
1.00
19.82

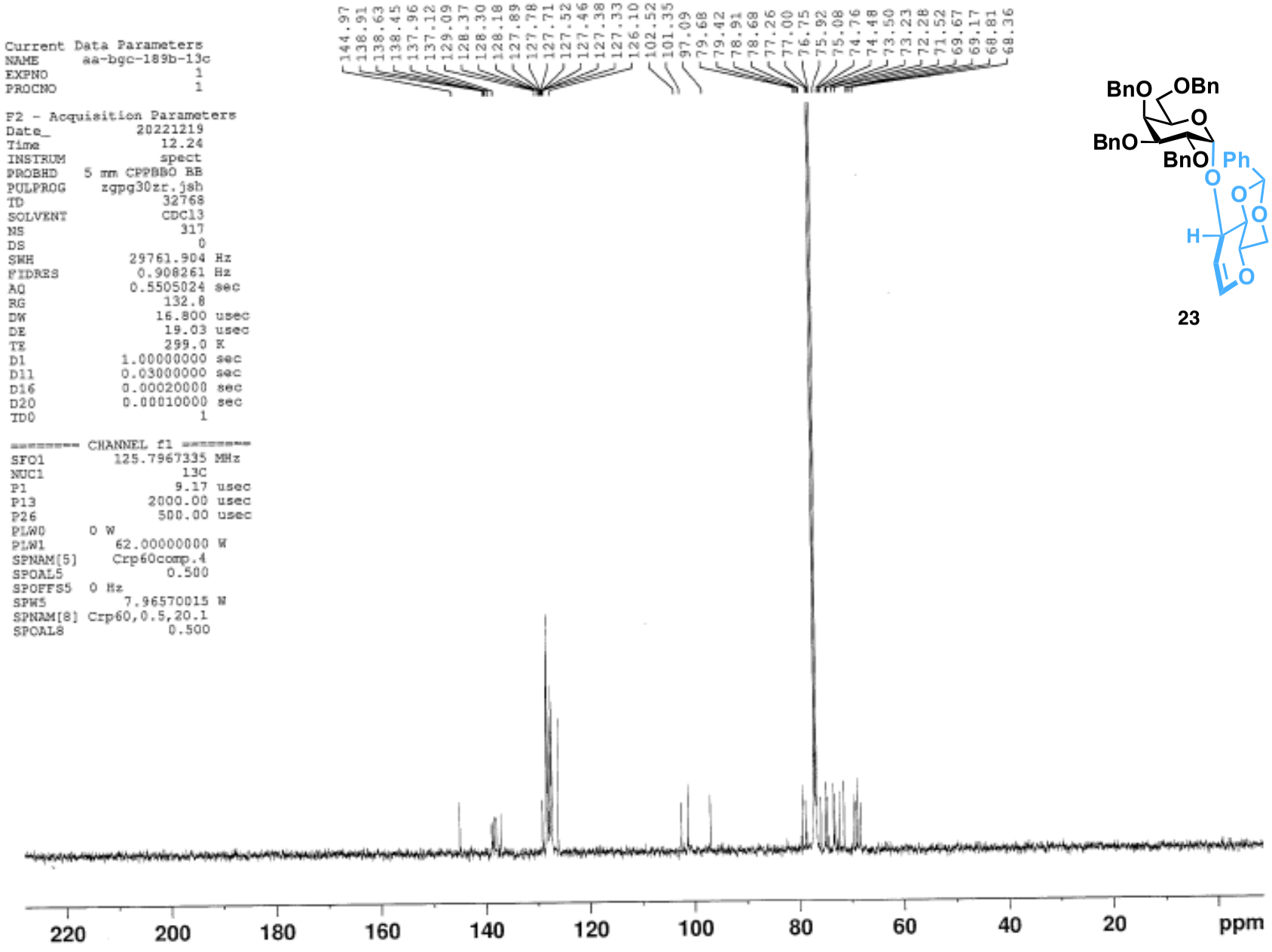

Current Data Parameters
NAME aa-bgc-109-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221129
Time 17.15
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 355
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
138.23
137.91
137.81
137.16
128.64
128.58
128.55
128.42
128.30
128.16
127.82
127.75
127.65
127.55
111.79
105.64
101.30
86.00
82.82
80.24
79.07
77.25
77.00
76.74
75.62
75.07
74.86
74.67
74.37
73.54
72.41
70.86
69.83
69.55
64.70
29.68
26.79
26.06
BnO
OBn
O
BnO
BnO
O
Me
Me
O
O
O
Me
Me
O
220
200
180
160
140
120
100
80
60
40
20
ppm
1.0
0.5
ppm

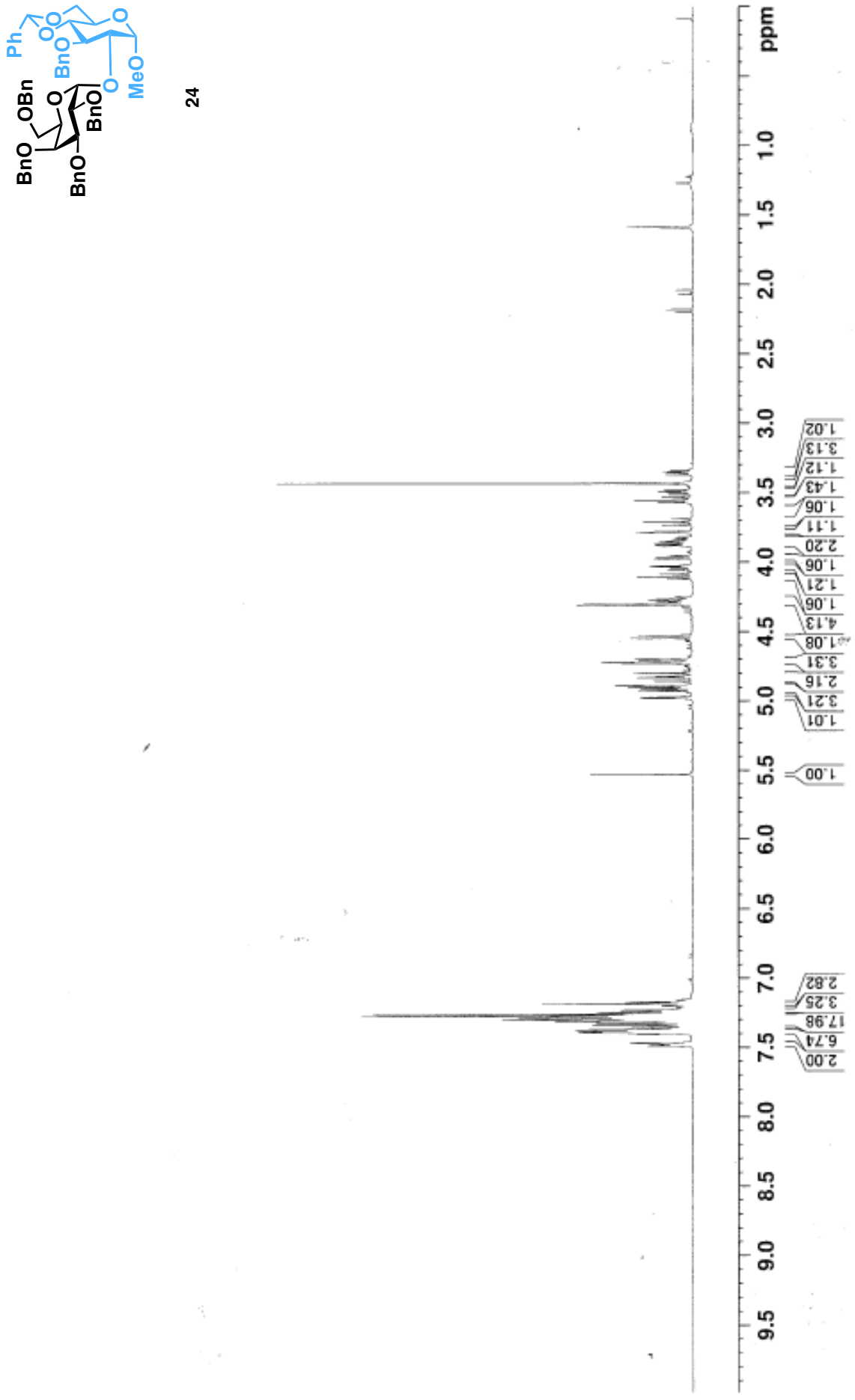
22


BnO
OBn
O
BnO
BnO
Ph
O
O
O
O
H

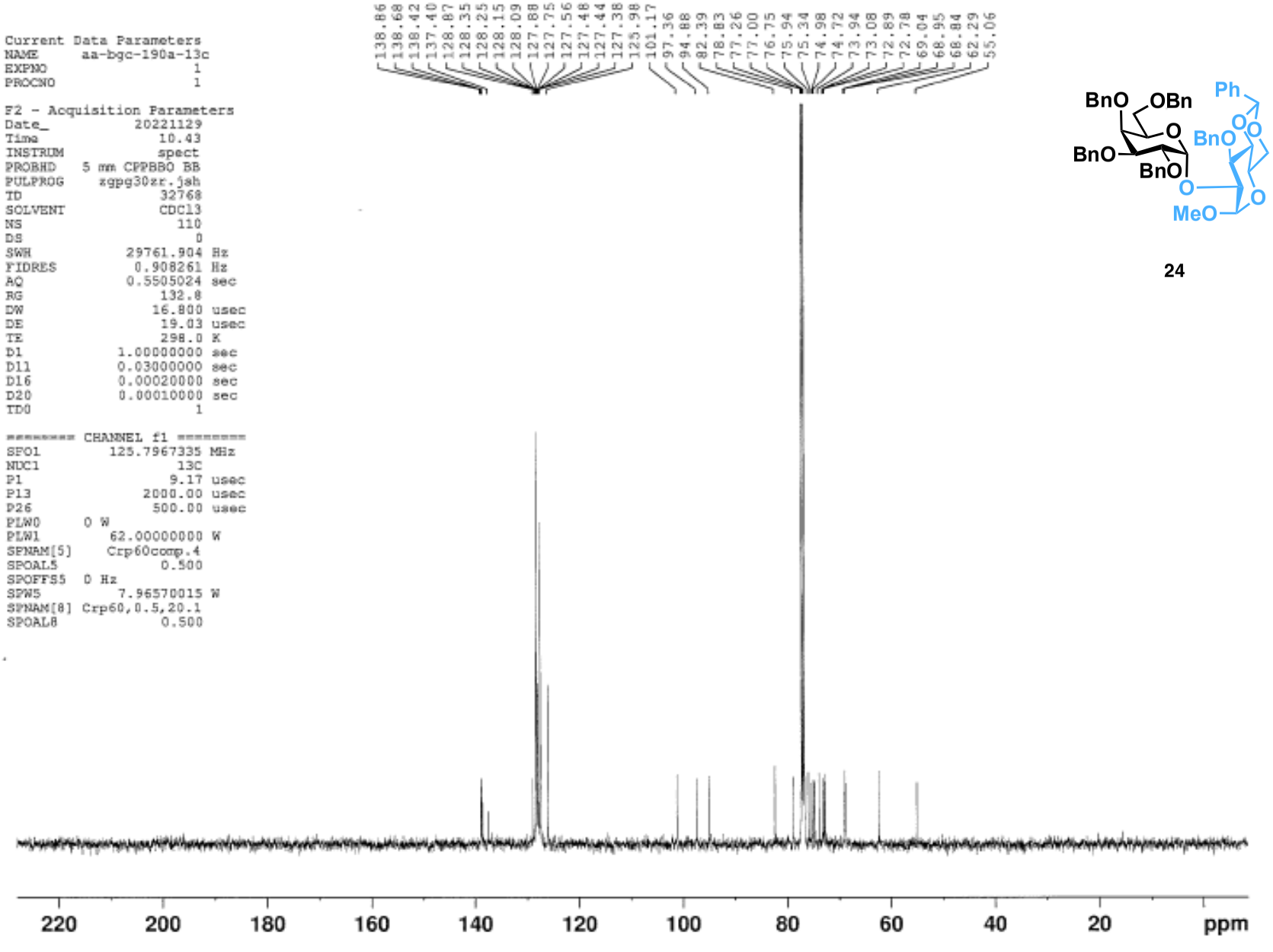
23


9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.10
2.40
8.14
13.01
5.20
1.00
1.03
1.07
1.08
1.07
2.31
2.82
1.28
2.23
1.10
2.29
1.18
3.37
1.18
2.25

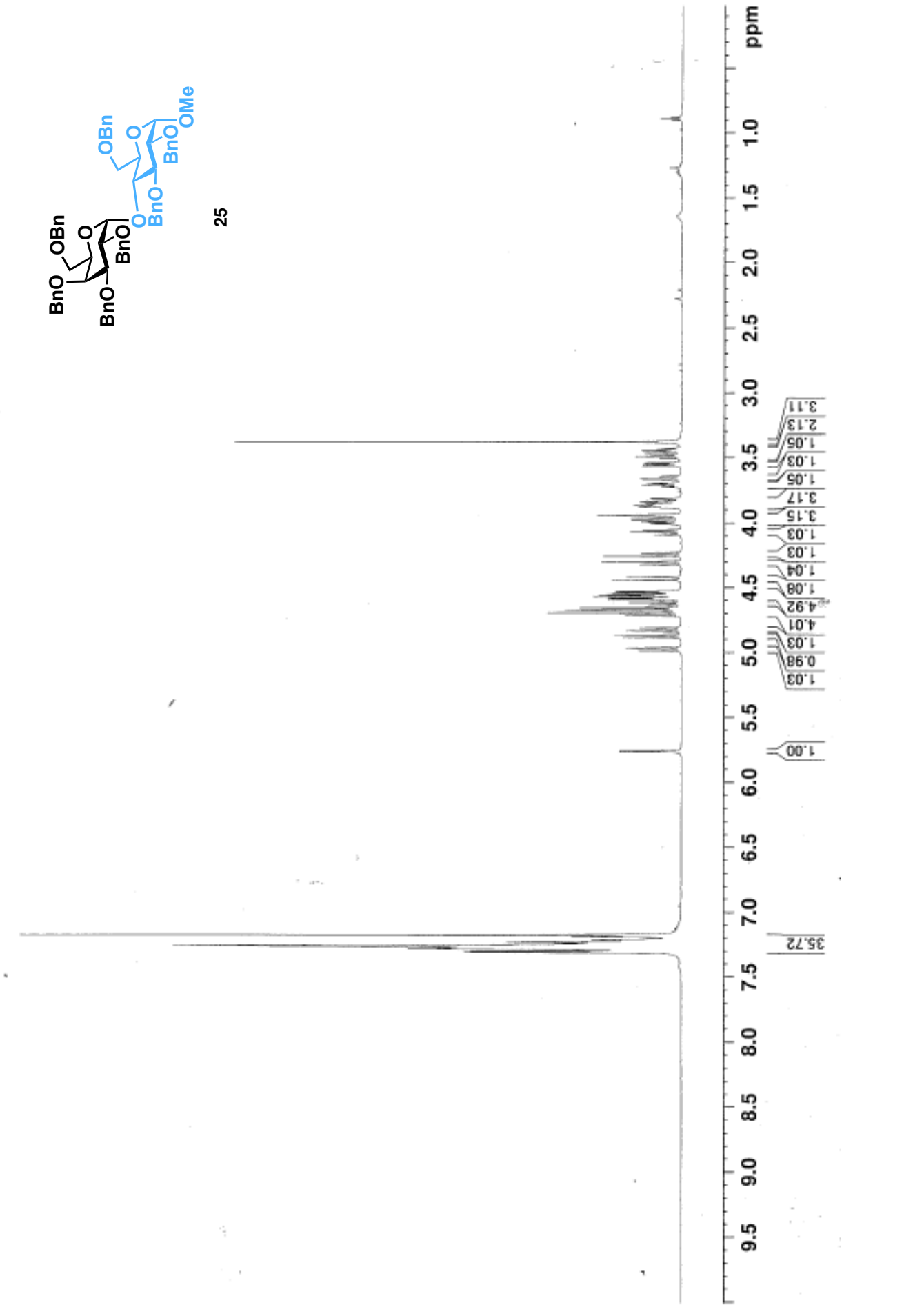

144.97
138.91
138.63
138.45
137.96
137.12
129.09
128.37
128.30
128.18
127.89
127.78
127.71
127.52
127.46
127.38
127.33
126.10
102.52
101.35
97.09
79.68
79.42
78.91
78.68
77.26
77.00
76.75
75.92
75.08
74.76
74.48
73.50
73.23
72.28
71.52
69.67
69.17
68.81
68.36
Current Data Parameters
NAME aa-bgc-189b-13c
EXPNO 1
PROCNO 1
P2 - Acquisition Parameters
Date_ 20221219
Time 12.24
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 317
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 299.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
BnO
OBn
O
BnO
BnO
Ph
O
O
O
H
O
23
220
200
180
160
140
120
100
80
60
40
20
ppm

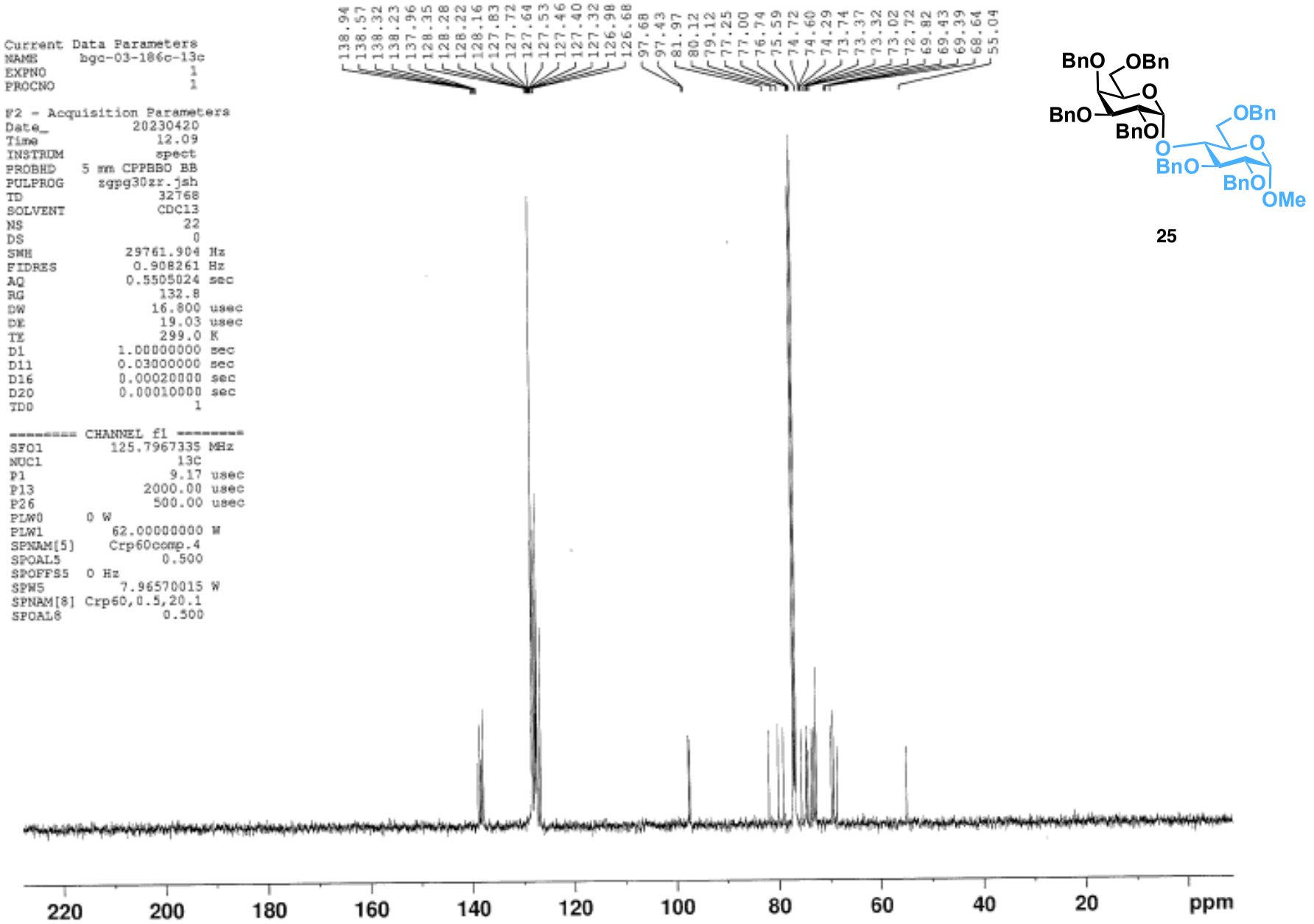

Ph
BnO
MeO
OBn
BnO
BnO
BnO
24
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
2.00
6.74
17.98
3.25
2.82
1.00
1.01
3.21
2.16
3.31
1.08
4.13
1.06
1.21
1.06
2.20
1.11
1.06
1.43
1.12
3.13
1.02

```
Current Data Parameters
NAME      aa-bgc-190a-13c
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20221129
Time              10.43
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG    zgpg30zr.jsh
TD               32768
SOLVENT          CDCl3
NS                 110
DS                   0
SWH          29761.904 Hz
FIDRES        0.908261 Hz
AQ           0.5505024 sec
RG               132.8
DW              16.800 usec
DE              19.03 usec
TE              298.0 K
D1           1.00000000 sec
D11          0.03000000 sec
D16          0.00020000 sec
D20          0.00010000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        125.7967335 MHz
NUC1               13C
P1                9.17 usec
P13            2000.00 usec
P26             500.00 usec
PLW0     0 W
PLW1       62.00000000 W
SPNAM[5]    Crp60comp.4
SPOAL5            0.500
SPOFFS5  0 Hz
SPW5        7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8            0.500
```

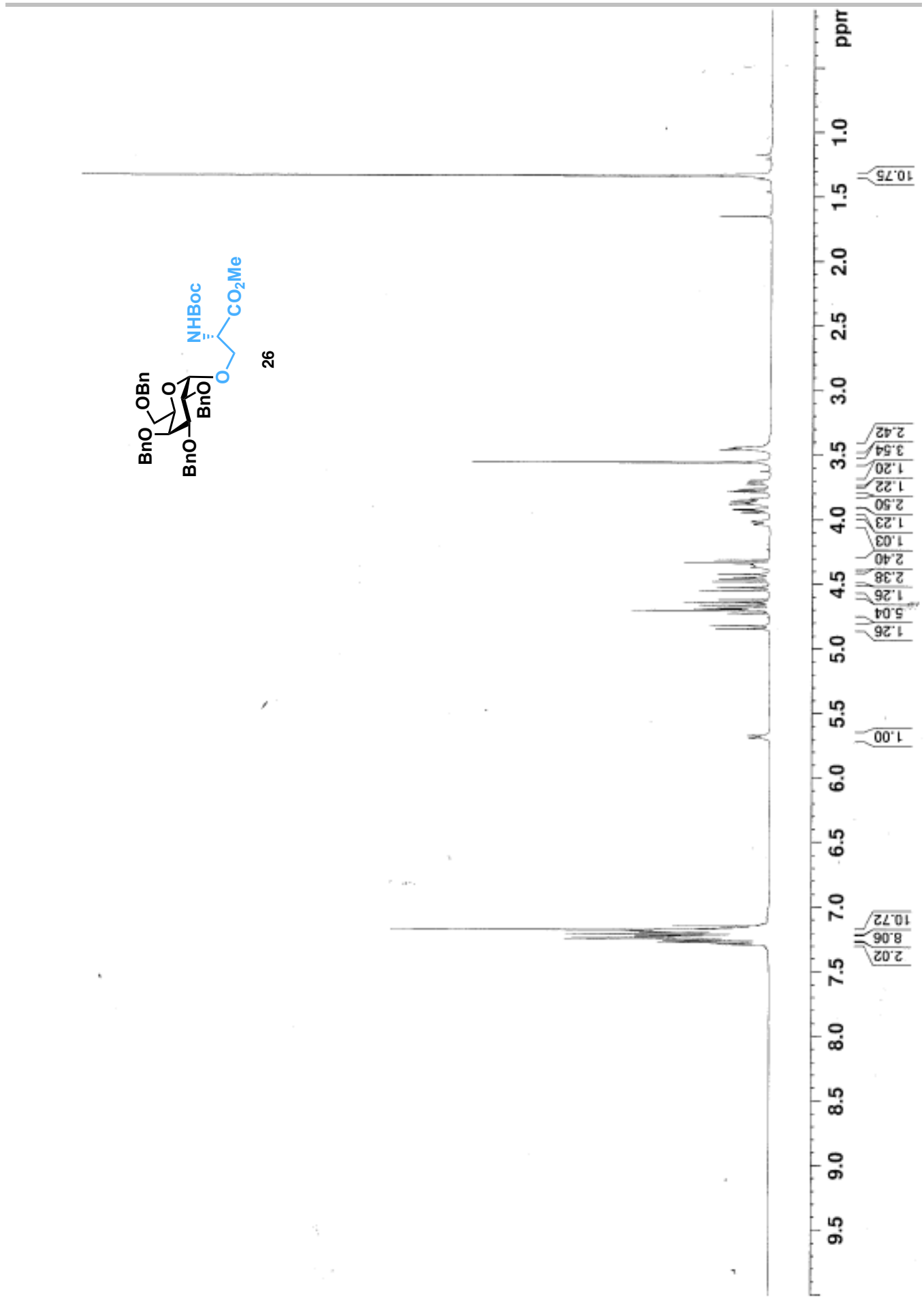


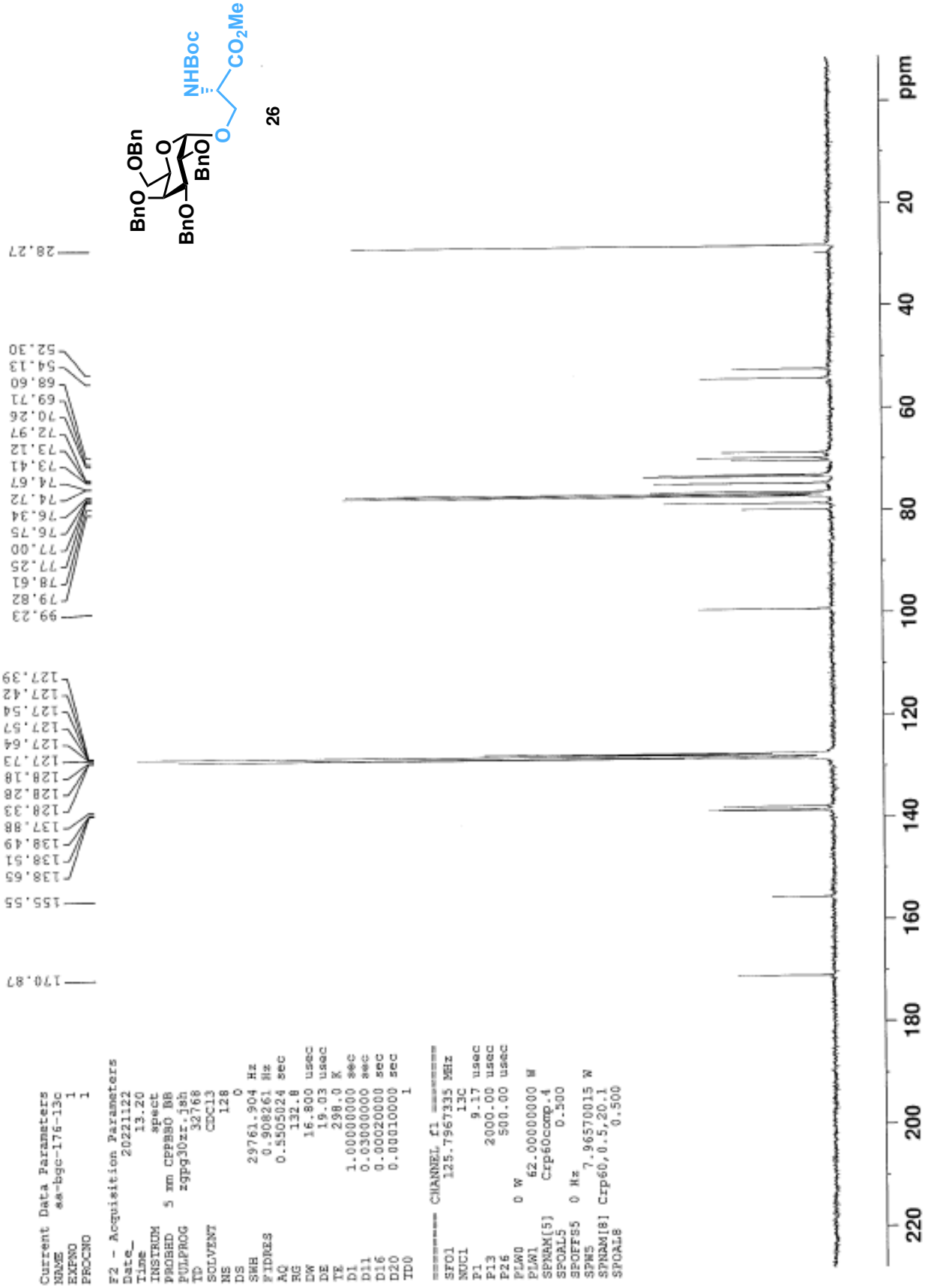
24


BnO
OBn
O
BnO
BnO
OBn
O
BnO
BnO
OMe
25
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
35.72
1.00
1.03
0.98
1.03
4.01
4.92
1.08
1.04
1.03
1.03
3.15
3.17
1.05
1.03
1.05
2.13
3.11


Current Data Parameters
NAME bgc-03-186c-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20230420
Time 12.09
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 22
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 299.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
138.94
138.57
138.32
138.23
137.96
128.35
128.28
128.22
128.16
127.83
127.72
127.64
127.53
127.46
127.40
127.32
126.98
126.68
97.68
97.43
81.97
80.12
79.12
77.25
77.00
76.74
75.59
74.72
74.60
74.29
73.74
73.37
73.32
73.02
72.72
69.82
69.43
69.39
68.64
55.04
BnO
OBn
O
BnO
BnO
O
OBn
O
BnO
BnO
OMe
220
200
180
160
140
120
100
80
60
40
20
ppm

25

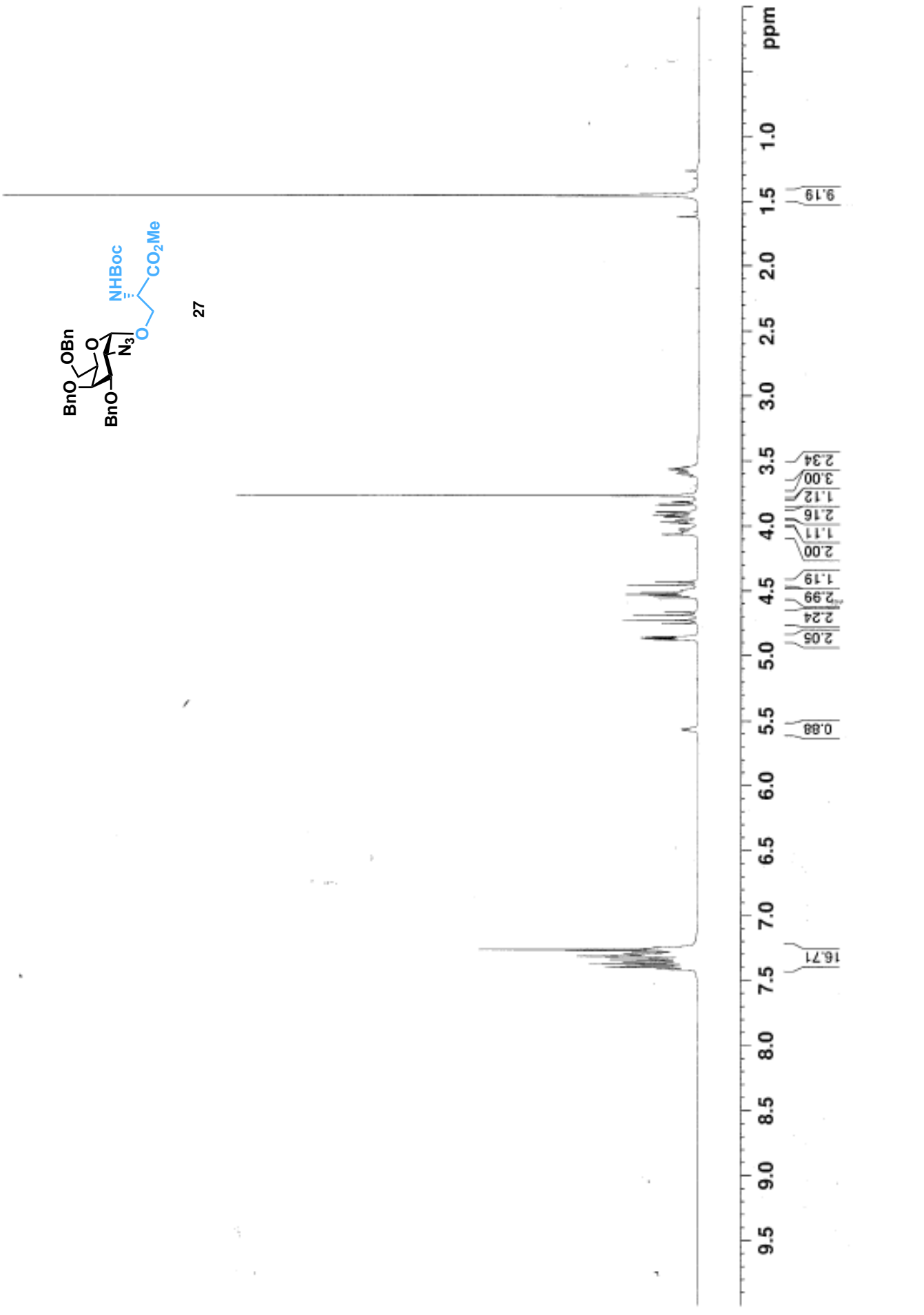

NHBoc
CO2Me
O
BnO
OBn
BnO
BnO
O
26
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
10.75
2.42
3.54
1.20
1.22
2.50
1.23
1.03
2.40
2.38
1.26
5.04
1.26
1.00
10.72
8.06
2.02

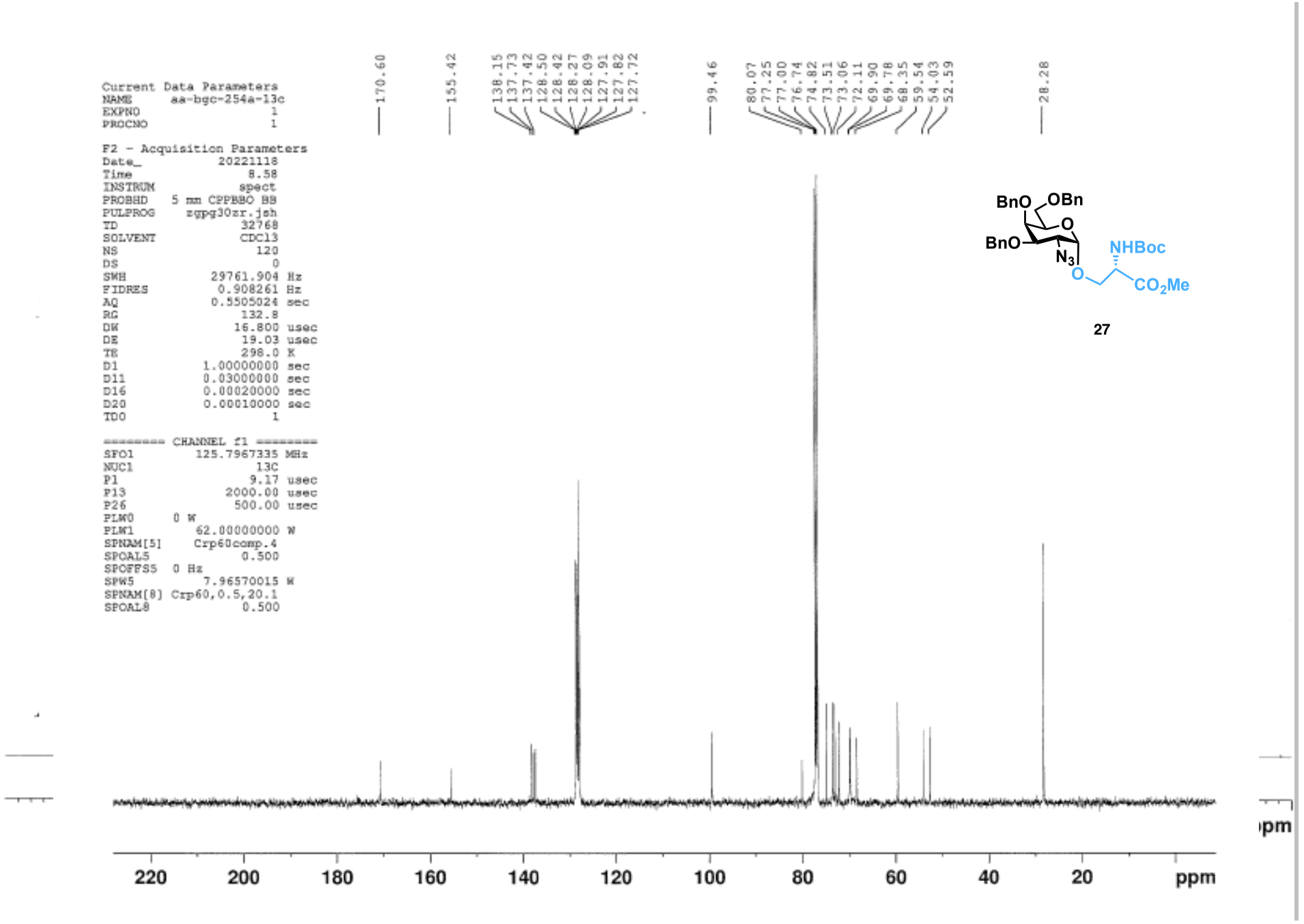


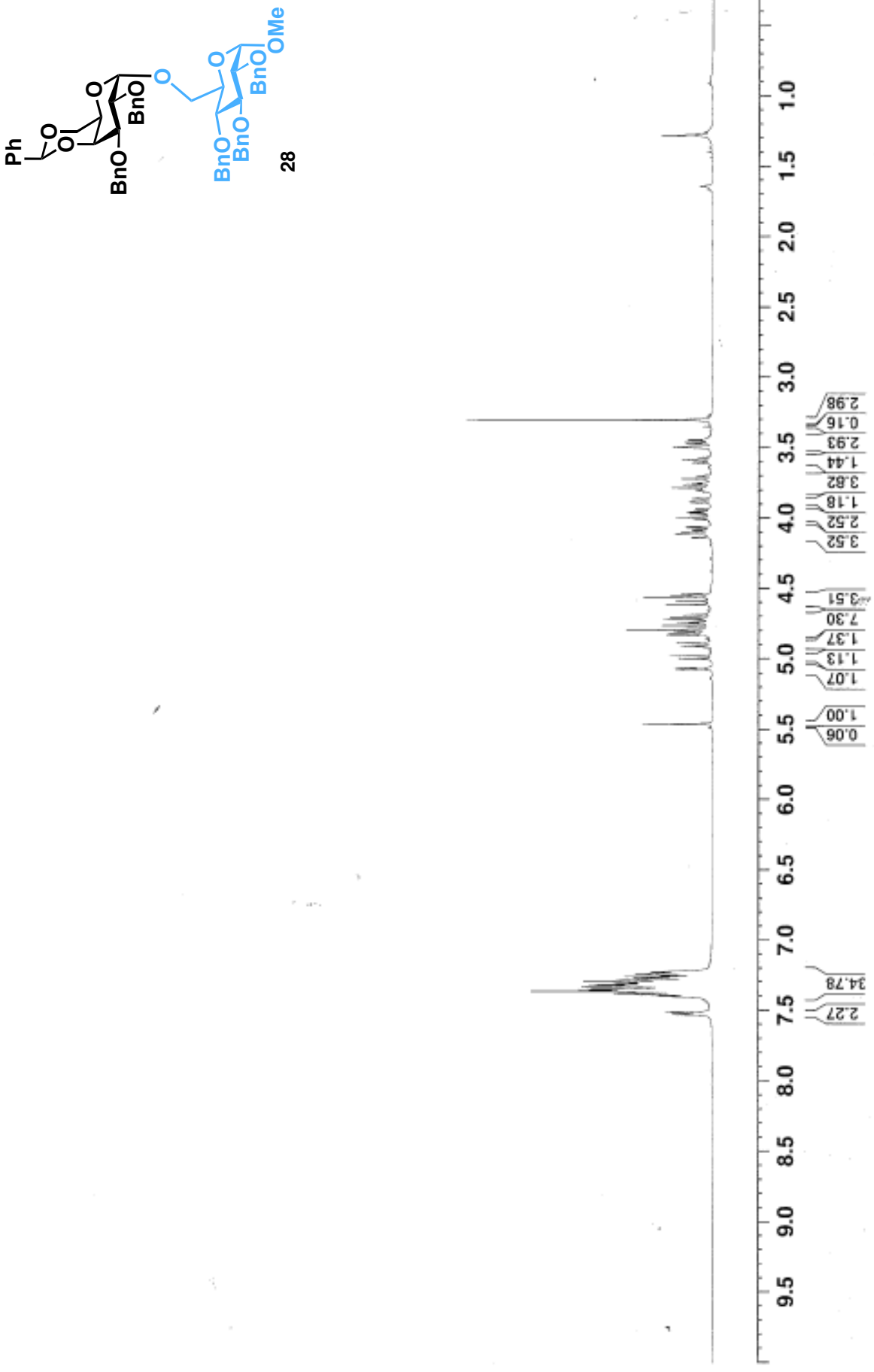
Current Data Parameters
NAME aa-bgc-176-13c
EXPNO 1
PROCNO 1

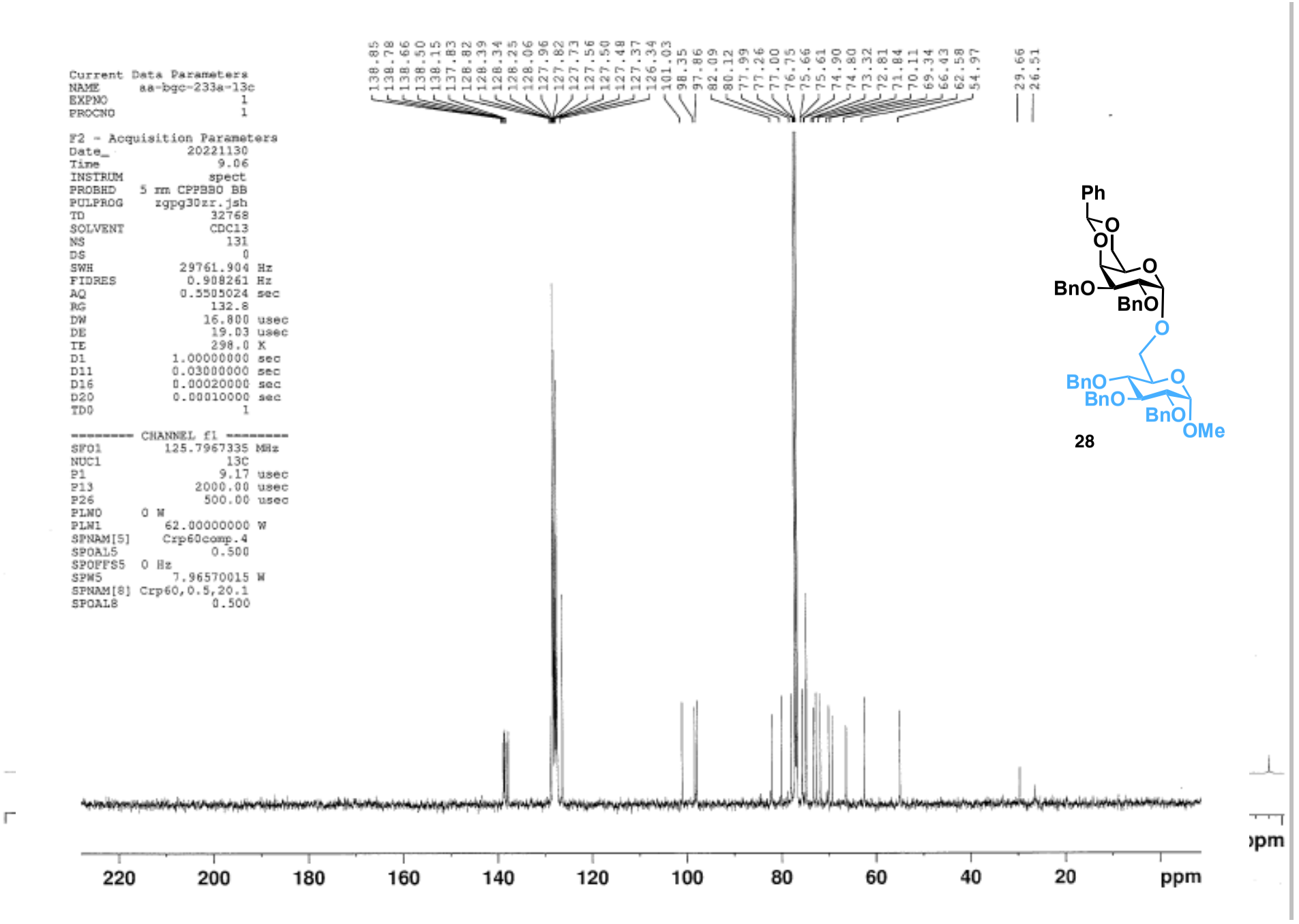
F2 - Acquisition Parameters
Date_ 20221122
Time 13.20
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jbh
TD 32768
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1

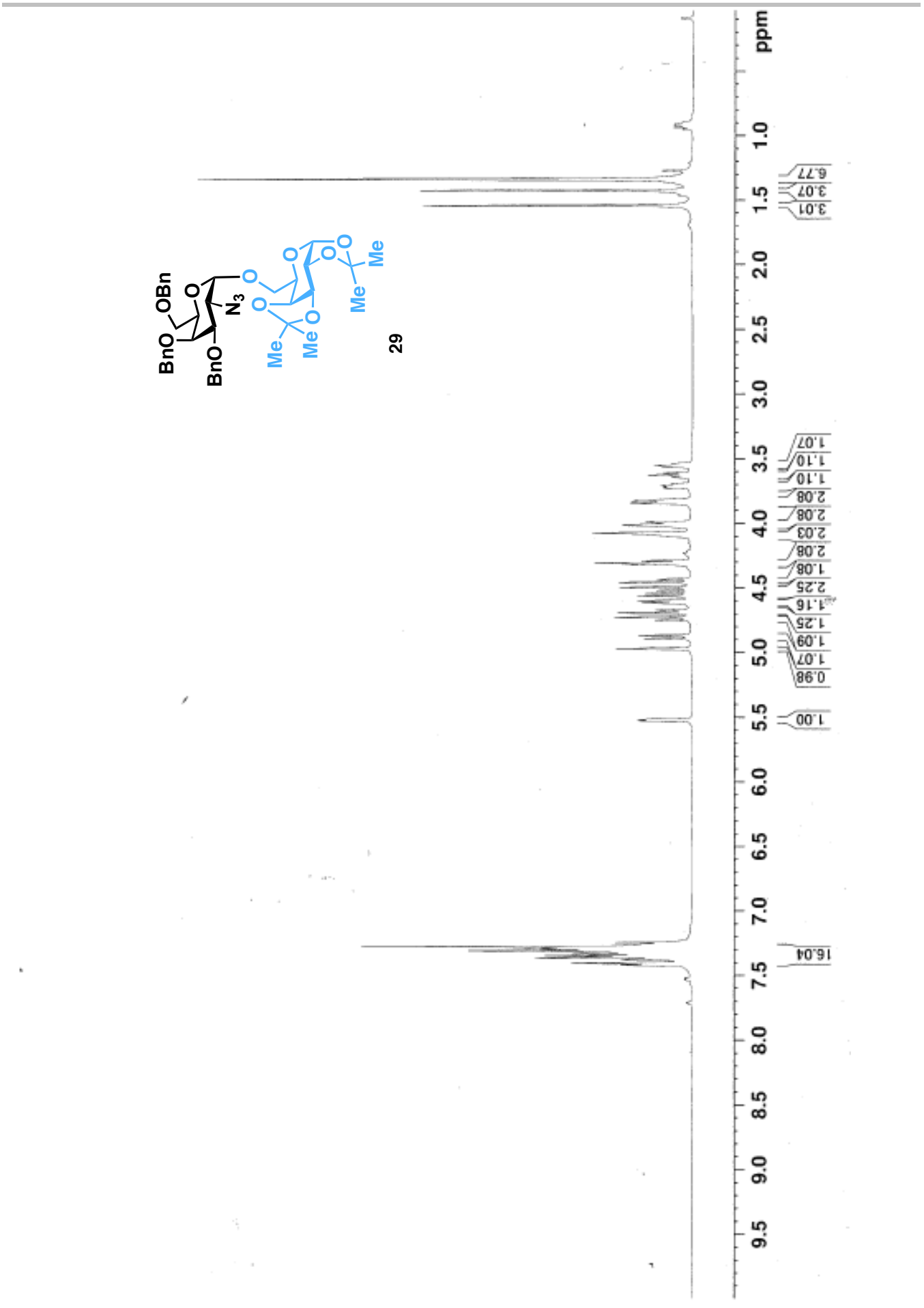
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W 62.00000000 W
PLW1 Crp60comp.4
SPNAM[5] 0.500
SPOAL5
SPOFFS5 0 Hz 7.96570015 W
SPW5
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500

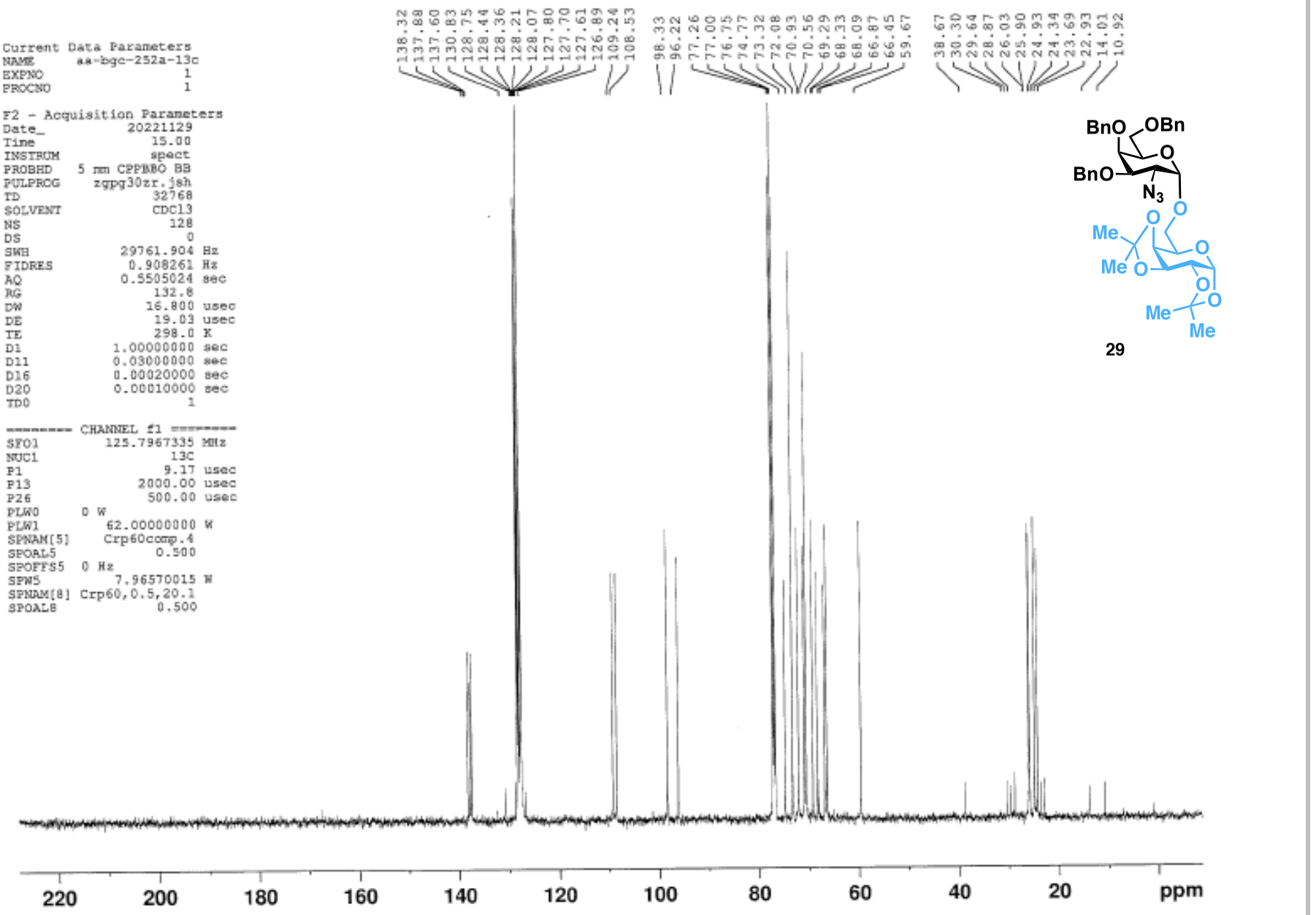

BnO
OBn
BnO
O
N3
O
NHBoc
CO2Me
27
ppm
9.19
2.34
3.00
1.12
2.16
1.11
2.00
1.19
2.99
2.24
2.05
0.88
16.71


Current Data Parameters
NAME aa-bgc-254a-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221118
Time 8.58
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 120
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
170.60
155.42
138.15
137.73
137.42
128.50
128.42
128.27
128.09
127.91
127.82
127.72
99.46
80.07
77.25
77.00
76.74
74.82
73.51
73.06
72.11
69.90
69.78
68.35
59.54
54.03
52.59
28.28
BnO
OBn
O
BnO
N3
O
NHBoc
CO2Me
27
220
200
180
160
140
120
100
80
60
40
20
ppm

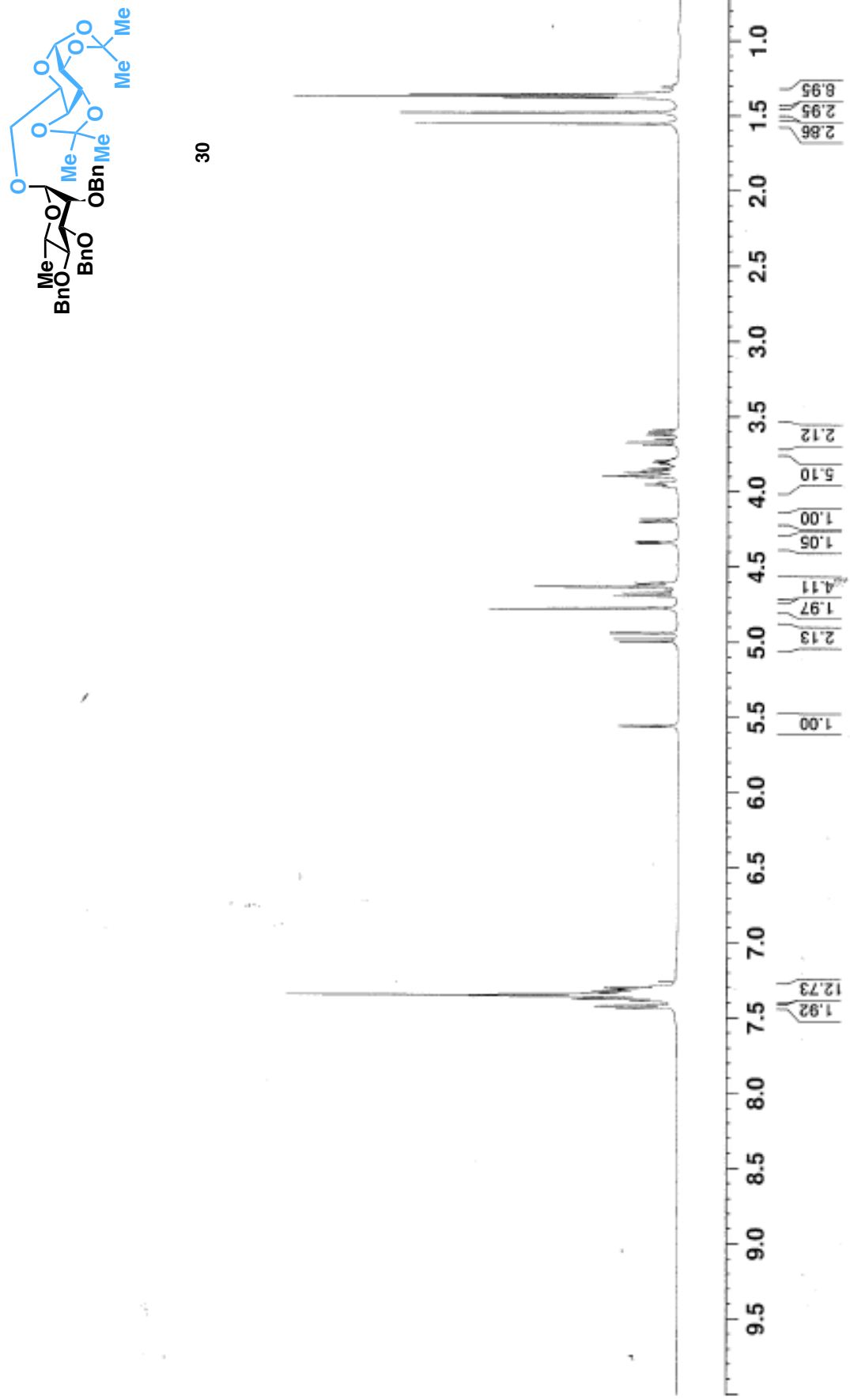

Ph
BnO
BnO
BnO
BnO
BnO
OMe
28
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
34.78
2.27
1.00
0.06
1.07
1.13
1.37
7.30
3.51
3.52
2.52
1.18
3.82
1.44
2.93
0.16
2.98


Current Data Parameters
NAME aa-bgc-233a-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221130
Time 9.06
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 131
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
138.85
138.78
138.66
138.50
138.15
137.83
128.82
128.39
128.34
128.25
128.06
127.96
127.82
127.73
127.56
127.50
127.48
127.37
126.34
101.03
98.35
97.86
82.09
80.12
77.99
77.26
77.00
76.75
75.66
75.61
74.90
74.80
73.32
72.81
71.84
70.11
69.34
66.43
62.58
54.97
29.66
26.51
Ph
O
O
O
BnO
BnO
O
BnO
O
BnO
BnO
OMe
28
220
200
180
160
140
120
100
80
60
40
20
ppm

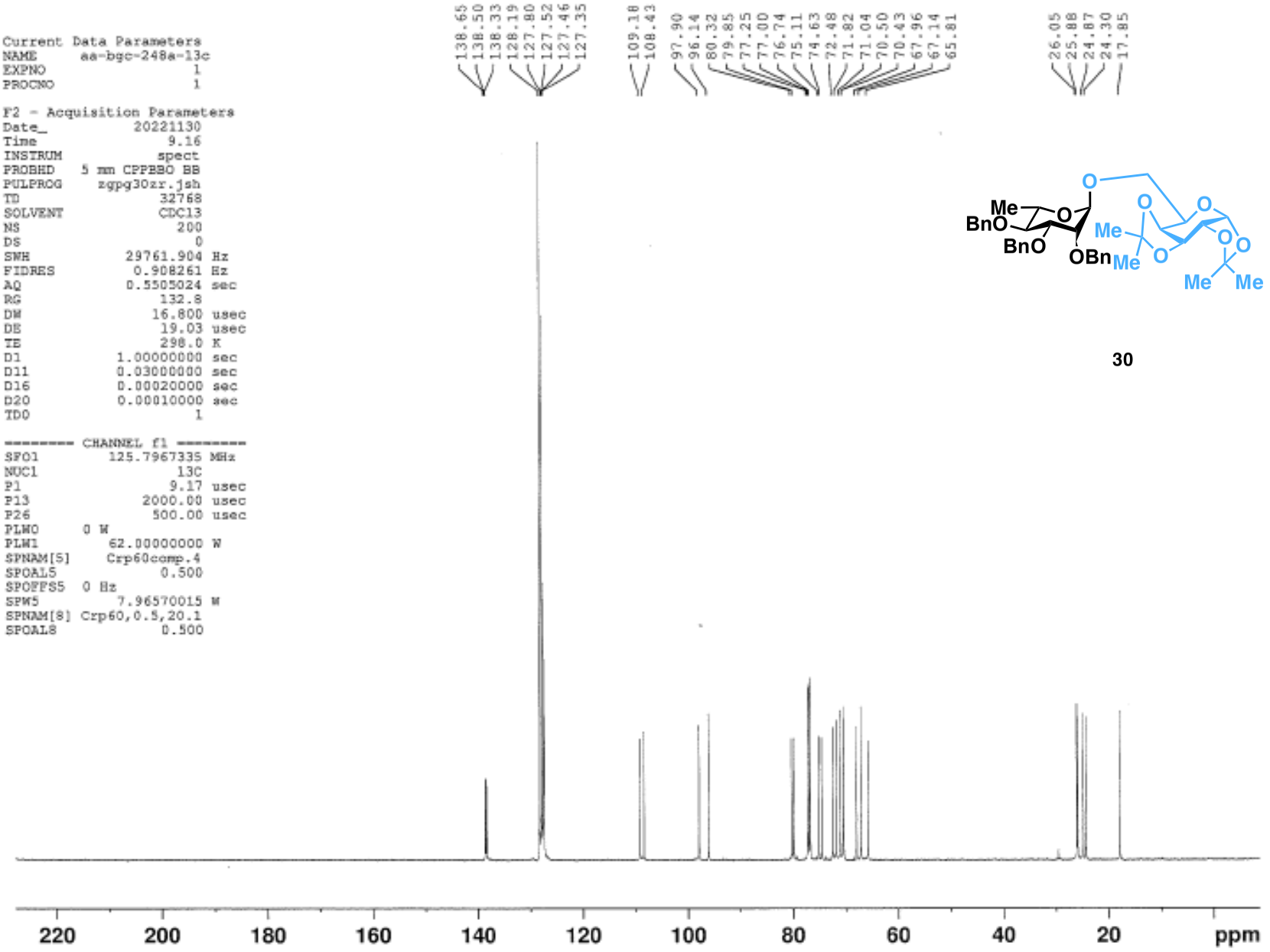

BnO
OBn
O
N3
BnO
O
O
O
O
O
Me
Me
Me
Me
29
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
6.77
3.07
3.01
1.07
1.10
1.10
2.08
2.08
2.03
2.08
1.08
2.25
1.16
1.25
1.09
1.07
0.98
1.00
16.04


Current Data Parameters
NAME aa-bgc-252a-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221129
Time 15.00
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
138.32
137.88
137.60
130.83
128.75
128.44
128.36
128.21
128.07
127.80
127.70
127.61
126.89
109.24
108.53
98.33
96.22
77.26
77.00
76.75
74.77
73.32
72.08
70.93
70.56
69.29
68.33
68.09
66.87
66.45
59.67
38.67
30.30
29.64
28.87
26.03
25.90
24.93
24.34
23.69
22.93
14.01
10.92
BnO
OBn
BnO
O
N3
O
O
Me
Me
O
O
O
Me
O
Me
220
200
180
160
140
120
100
80
60
40
20
ppm

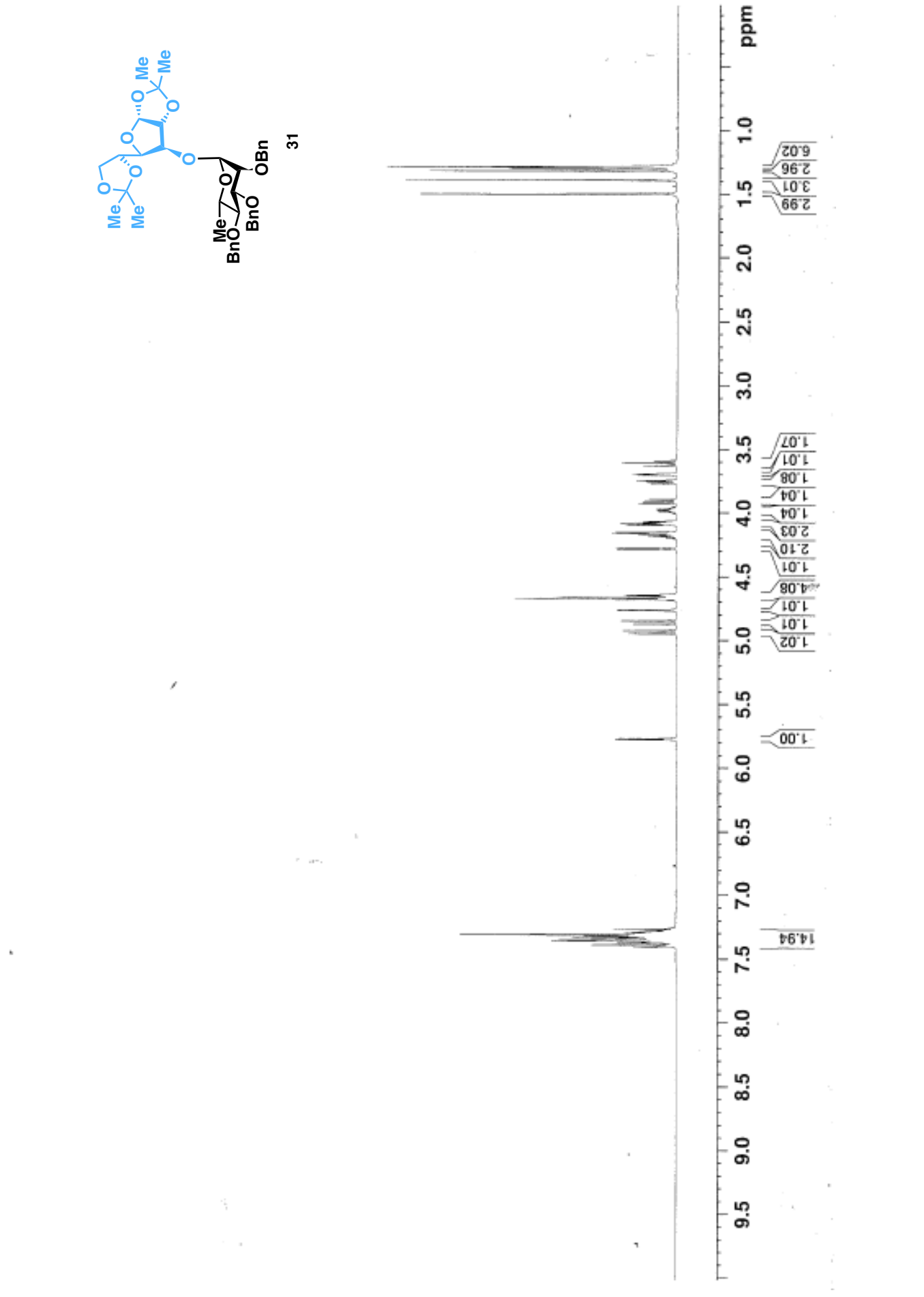
29

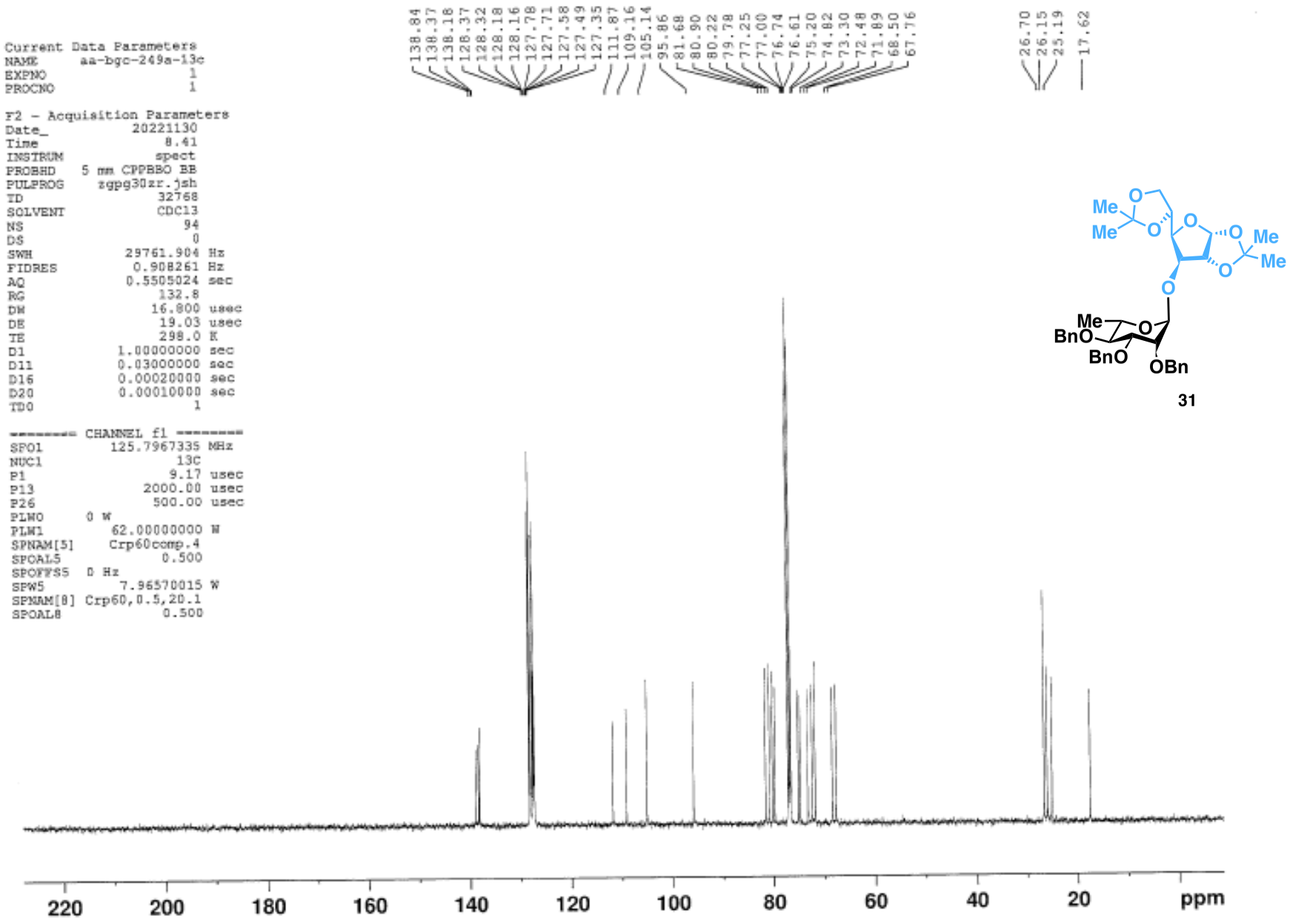

30
Me
BnO
BnO
OBn
Me
Me
Me
Me
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
8.95
2.95
2.86
2.12
5.10
1.00
1.05
4.11
1.97
2.13
1.00
12.73
1.92

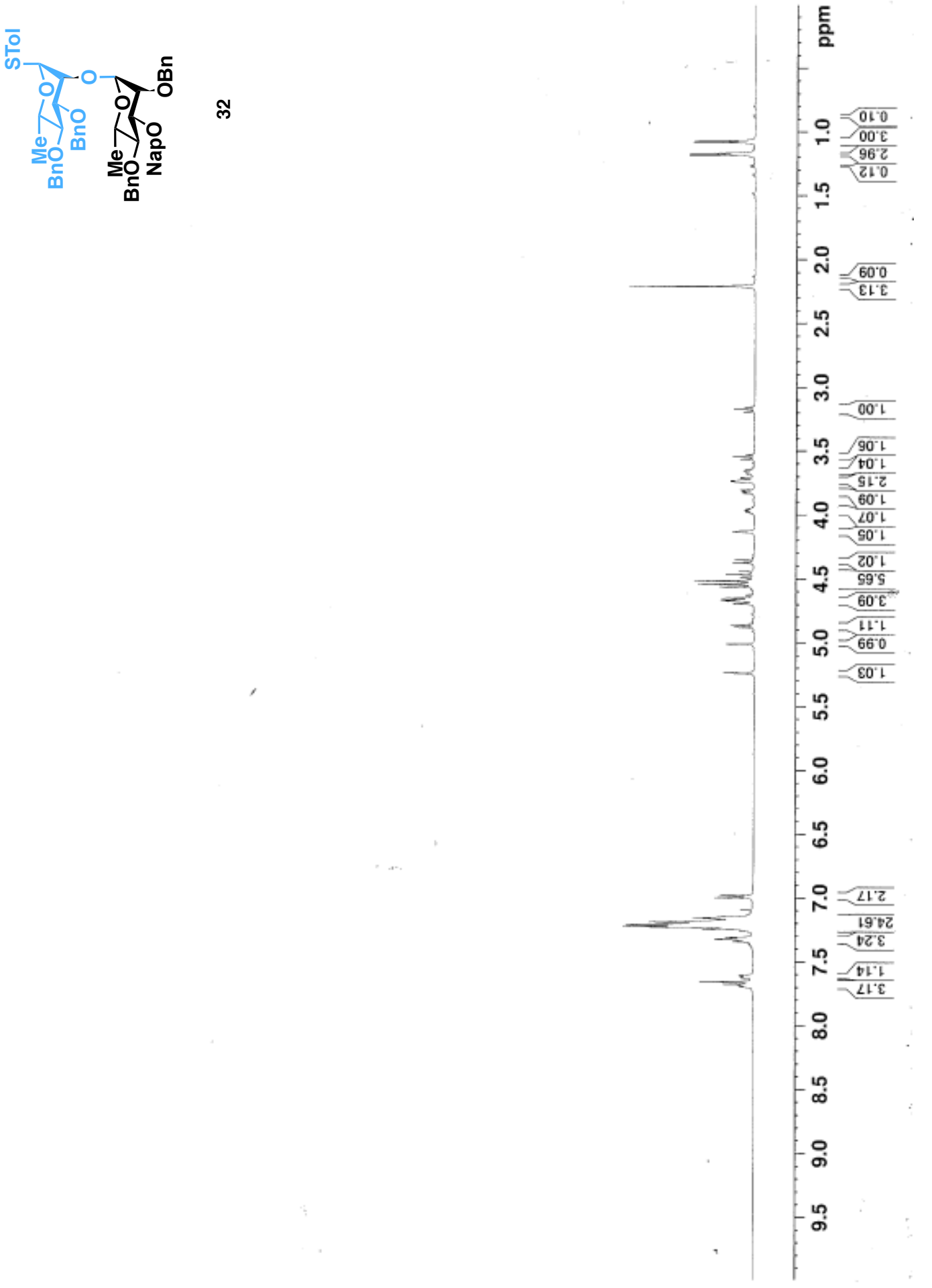
```
Current Data Parameters
NAME      aa-bgc-248a-13c
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20221130
Time                9.16
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG     zgpg30zr.jsh
TD                 32768
SOLVENT            CDCl3
NS                   200
DS                     0
SWH            29761.904 Hz
FIDRES          0.908261 Hz
AQ             0.5505024 sec
RG                 132.8
DW                16.800 usec
DE                19.03 usec
TE                298.0 K
D1            1.00000000 sec
D11           0.03000000 sec
D16           0.00020000 sec
D20           0.00010000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         125.7967335 MHz
NUC1                 13C
P1                  9.17 usec
P13              2000.00 usec
P26               500.00 usec
PLW0      0 W
PLW1        62.00000000 W
SPNAM[5]     Crp60comp.4
SPOAL5             0.500
SPOFFS5   0 Hz
SPW5          7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8             0.500
```

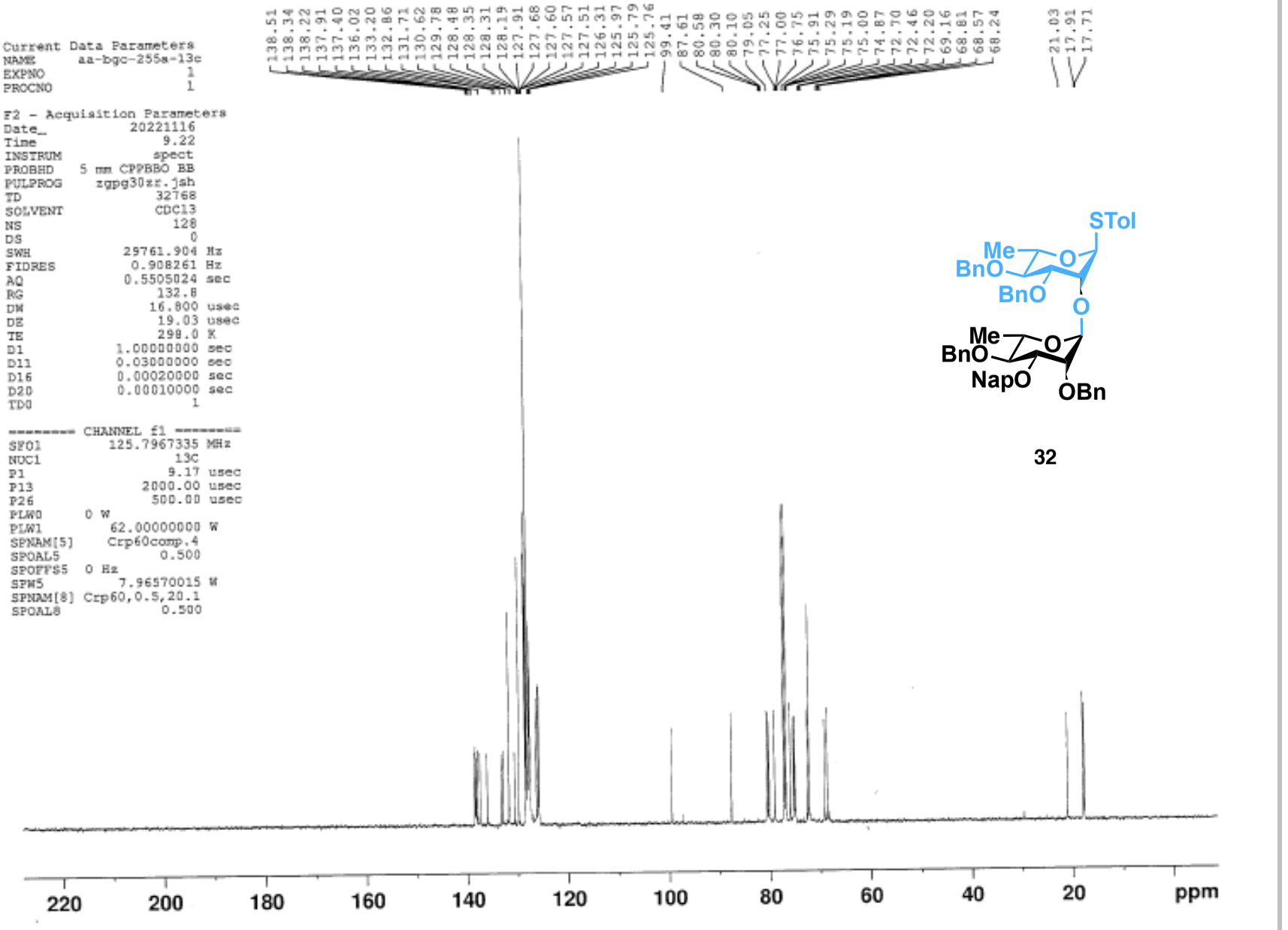


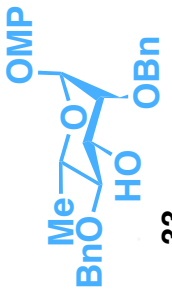
30


Me
Me
Me
Me
BnO
Me
BnO
OBn
31
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
6.02
2.96
3.01
2.99
1.07
1.01
1.08
1.04
1.04
2.03
2.10
1.01
4.08
1.01
1.01
1.02
1.00
14.94

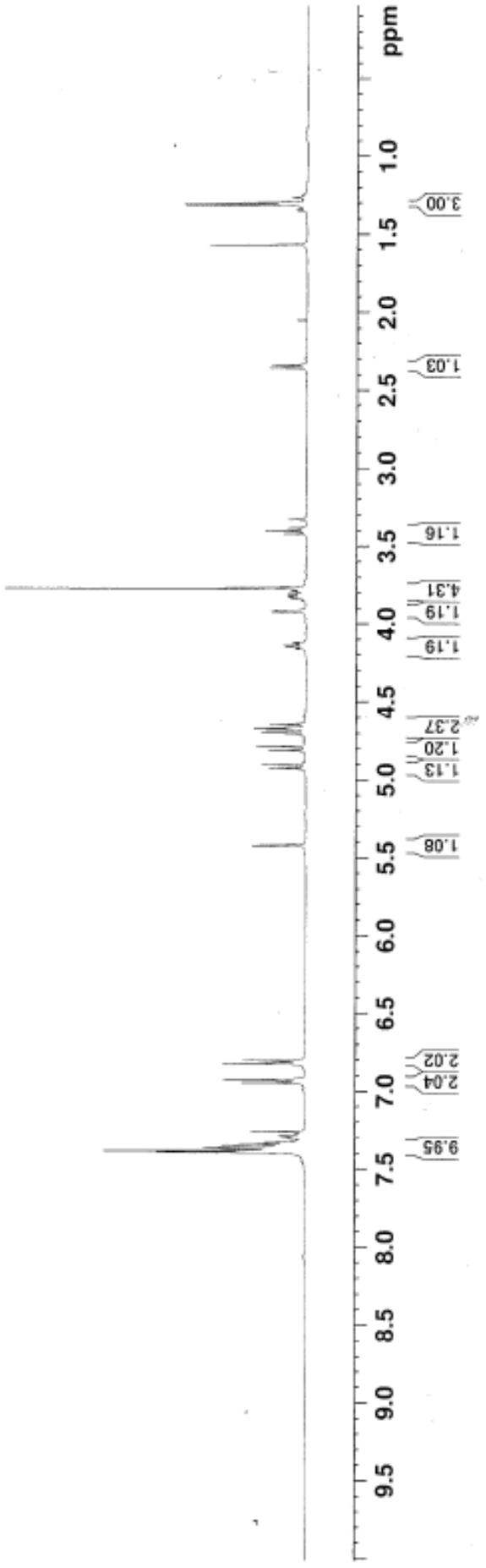

Current Data Parameters
NAME aa-bgc-249a-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221130
Time 8.41
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 94
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
138.84
138.37
138.18
128.37
128.32
128.18
128.16
127.78
127.71
127.58
127.49
127.35
111.87
109.16
105.14
95.86
81.68
80.90
80.22
79.78
77.25
77.00
76.74
76.61
75.20
74.82
73.30
72.48
71.89
68.50
67.76
26.70
26.15
25.19
17.62
Me
Me
O
O
O
O
Me
Me
O
Me
BnO
O
BnO
OBn
31
220
200
180
160
140
120
100
80
60
40
20
ppm

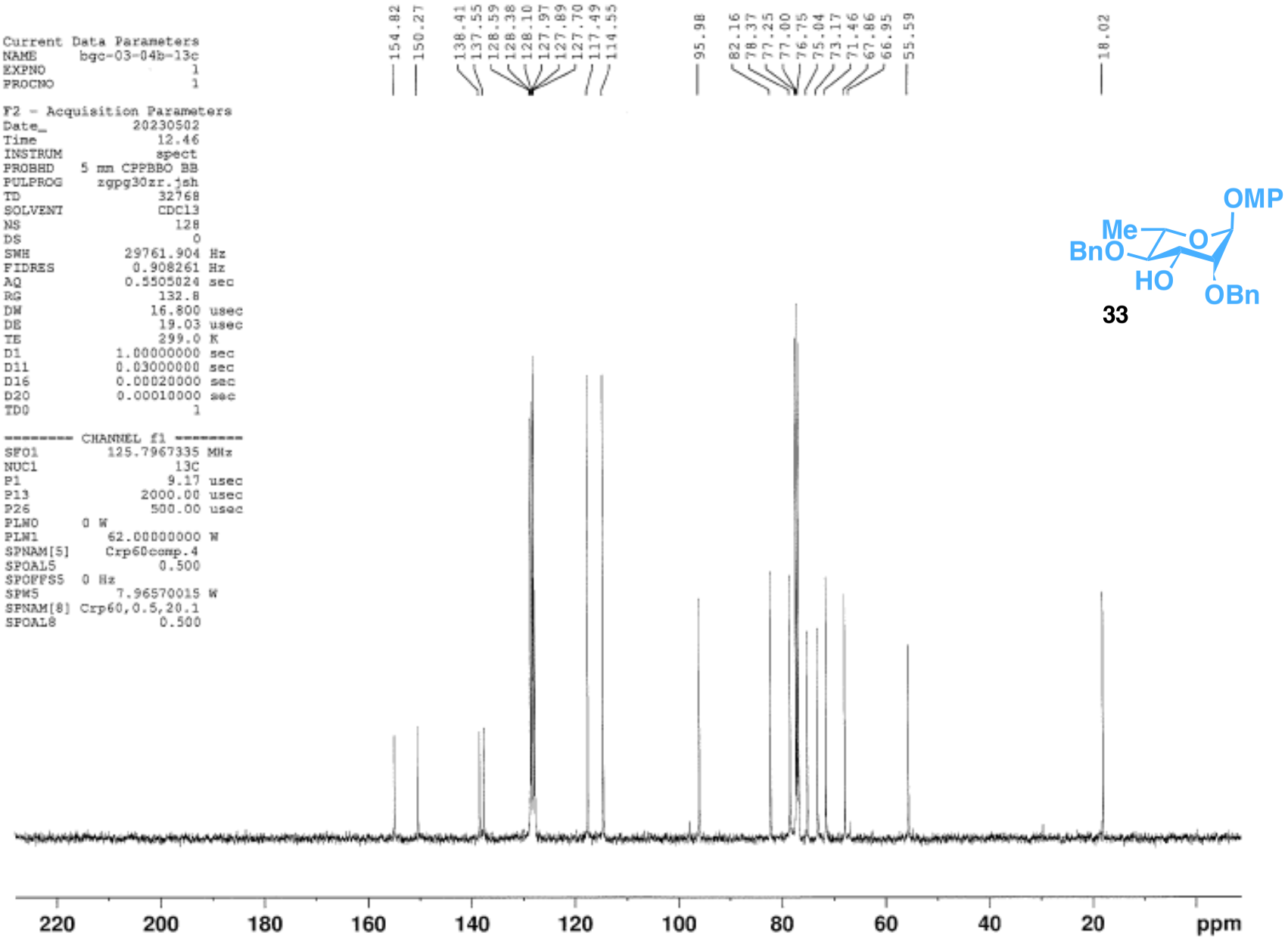

STol
Me
BnO
BnO
O
Me
BnO
O
NapO
OBn
32
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.10
3.00
2.96
0.12
0.09
3.13
1.00
1.06
1.04
2.15
1.09
1.07
1.05
1.02
5.65
3.09
1.11
0.99
1.03
2.17
24.61
3.24
1.14
3.17

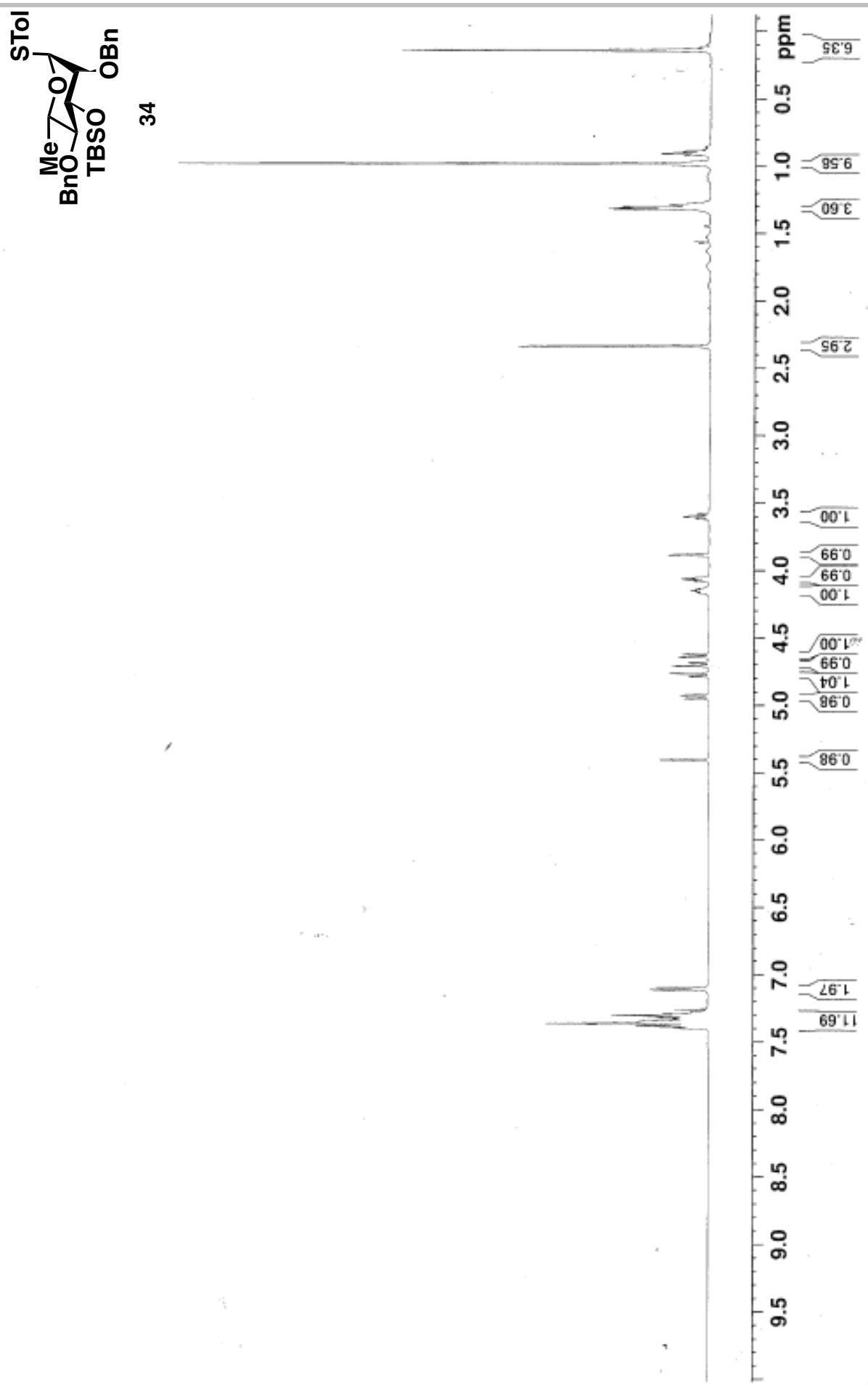

138.51
138.34
138.22
137.91
137.40
136.02
133.20
132.86
131.71
130.62
129.78
128.48
128.35
128.31
128.19
127.91
127.68
127.60
127.57
127.51
126.31
125.97
125.79
125.76
99.41
87.61
80.58
80.30
80.10
79.05
77.25
77.00
76.75
75.91
75.29
75.19
75.00
74.87
72.70
72.46
72.20
69.16
68.81
68.57
68.24
21.03
17.91
17.71
Current Data Parameters
NAME aa-bgc-255a-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221116
Time 9.22
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
STol
Me
BnO
BnO
O
Me
BnO
O
NapO
OBn
32
220
200
180
160
140
120
100
80
60
40
20
ppm


OMP
OBn
O
HO
Me
BnO

33


ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
3.00
1.03
1.16
4.31
1.19
1.19
2.37
1.20
1.13
1.08
2.02
2.04
9.95


Current Data Parameters
NAME bgc-03-04b-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20230502
Time 12.46
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 299.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
154.82
150.27
138.41
137.55
128.59
128.38
128.10
127.97
127.89
127.70
117.49
114.55
95.98
82.16
78.37
77.25
77.00
76.75
75.04
73.17
71.46
67.86
66.95
55.59
18.02
OMP
Me
BnO
O
HO
OBn
33
220
200
180
160
140
120
100
80
60
40
20
ppm


STol
OBn
O
Me
BnO
TBSO
34
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
6.35
9.56
3.60
2.95
1.00
0.99
0.99
1.00
1.00
0.99
1.04
0.98
0.98
1.97
11.69

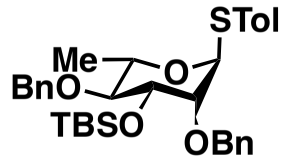
```
Current Data Parameters
NAME       bgc-03-12-13c
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230322
Time               17.45
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG     zgpg30zr.jsh
TD                32768
SOLVENT           CDCl3
NS                  128
DS                    0
SWH           29761.904 Hz
FIDRES         0.908261 Hz
AQ            0.5505024 sec
RG                132.8
DW               16.800 usec
DE               19.03 usec
TE               299.0 K
D1           1.00000000 sec
D11          0.03000000 sec
D16          0.00020000 sec
D20          0.00010000 sec
TD0                   1
```

138.62
138.30
137.43
131.89
131.05
129.80
128.34
128.31
127.79
127.71
127.63
127.51

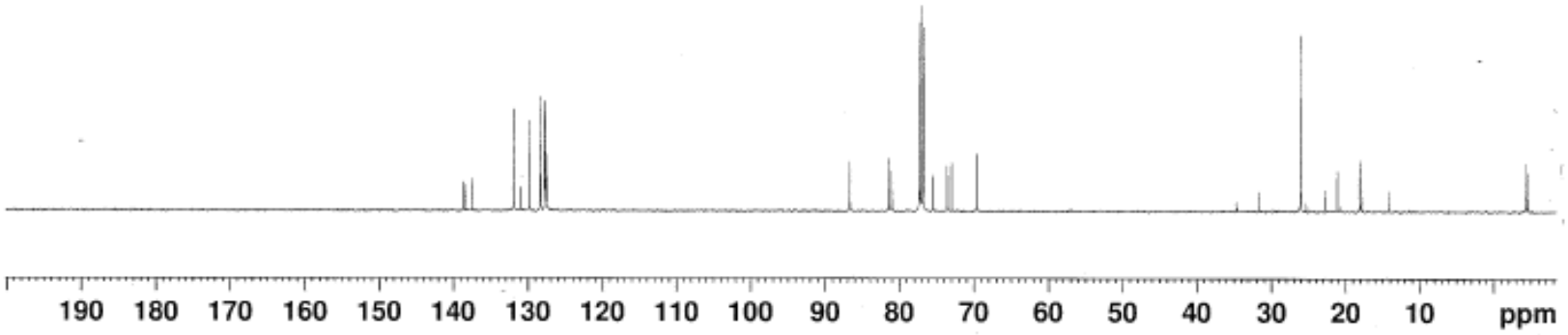
86.67
81.46
81.00
77.29
77.04
76.78
75.39
73.57
72.91
69.51

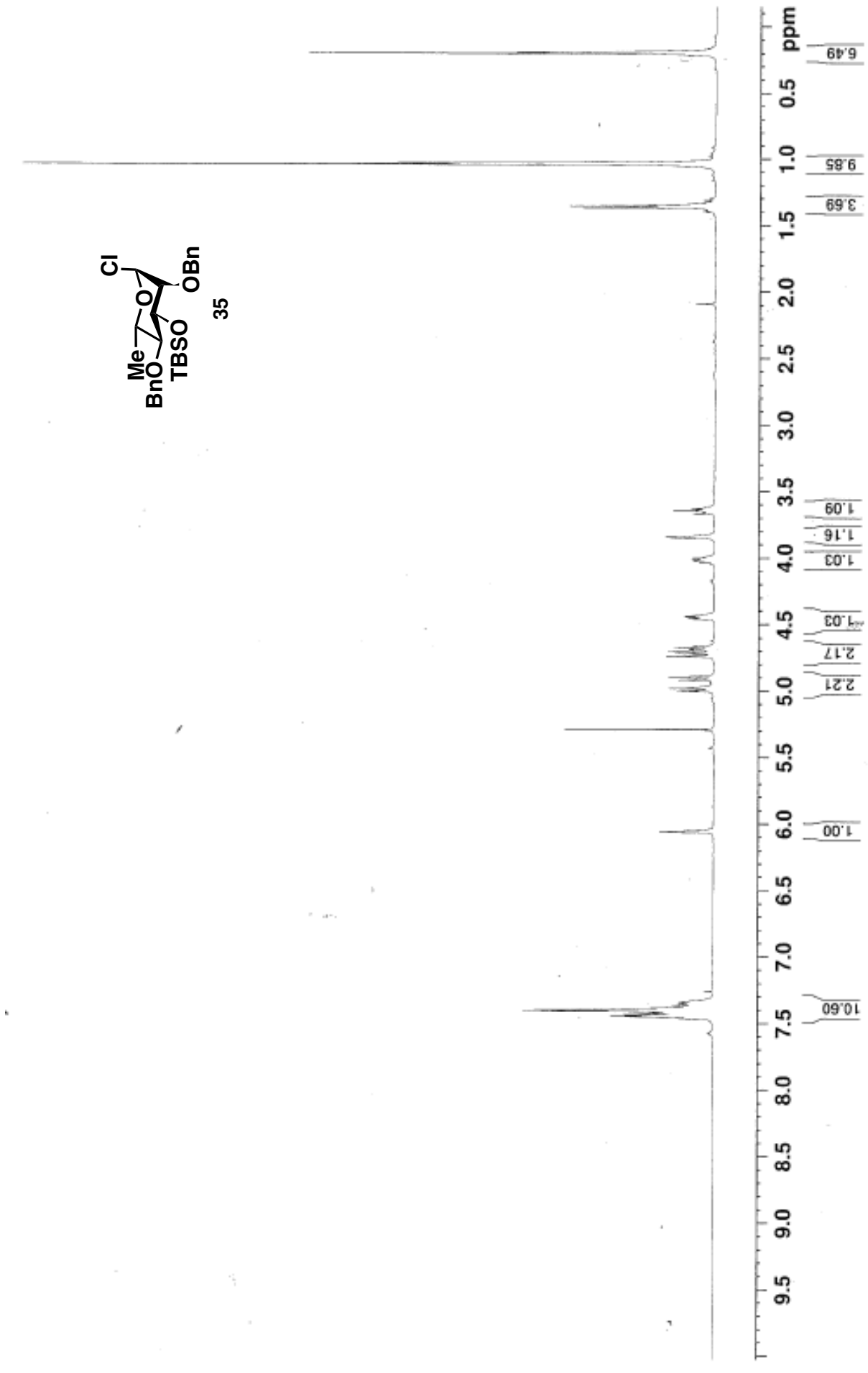
26.03
21.11
18.10
17.88

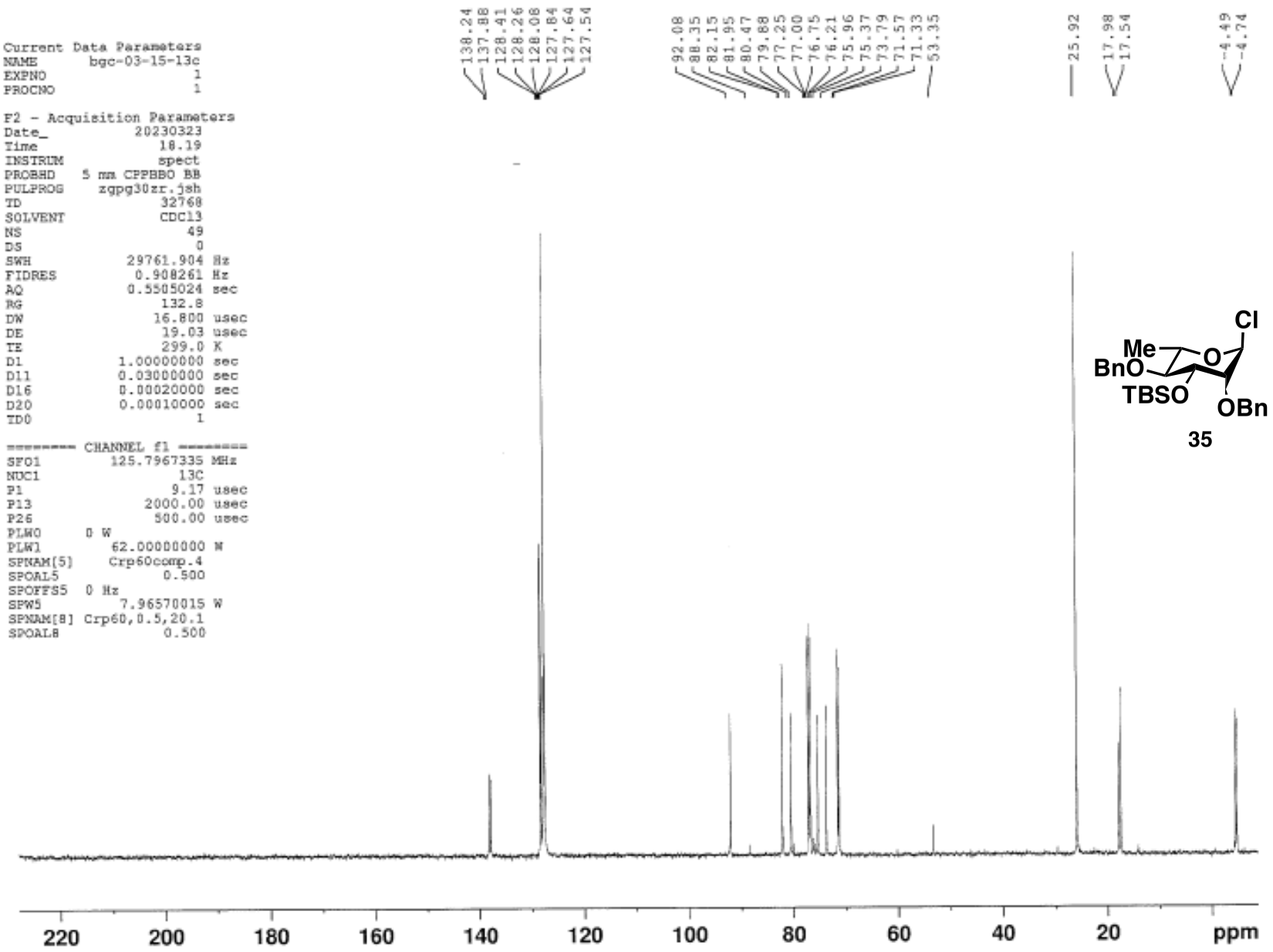
-4.35
-4.62

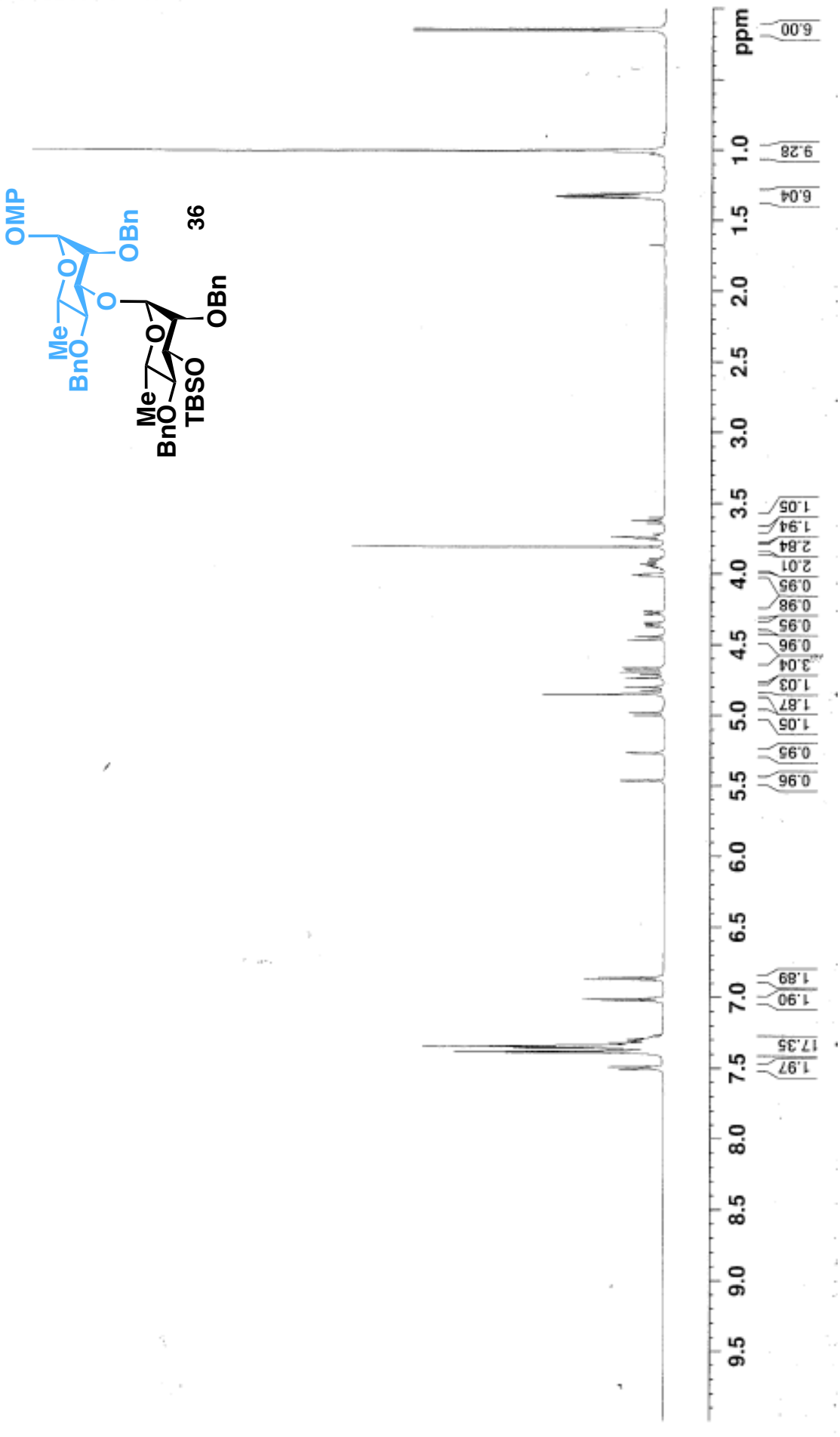


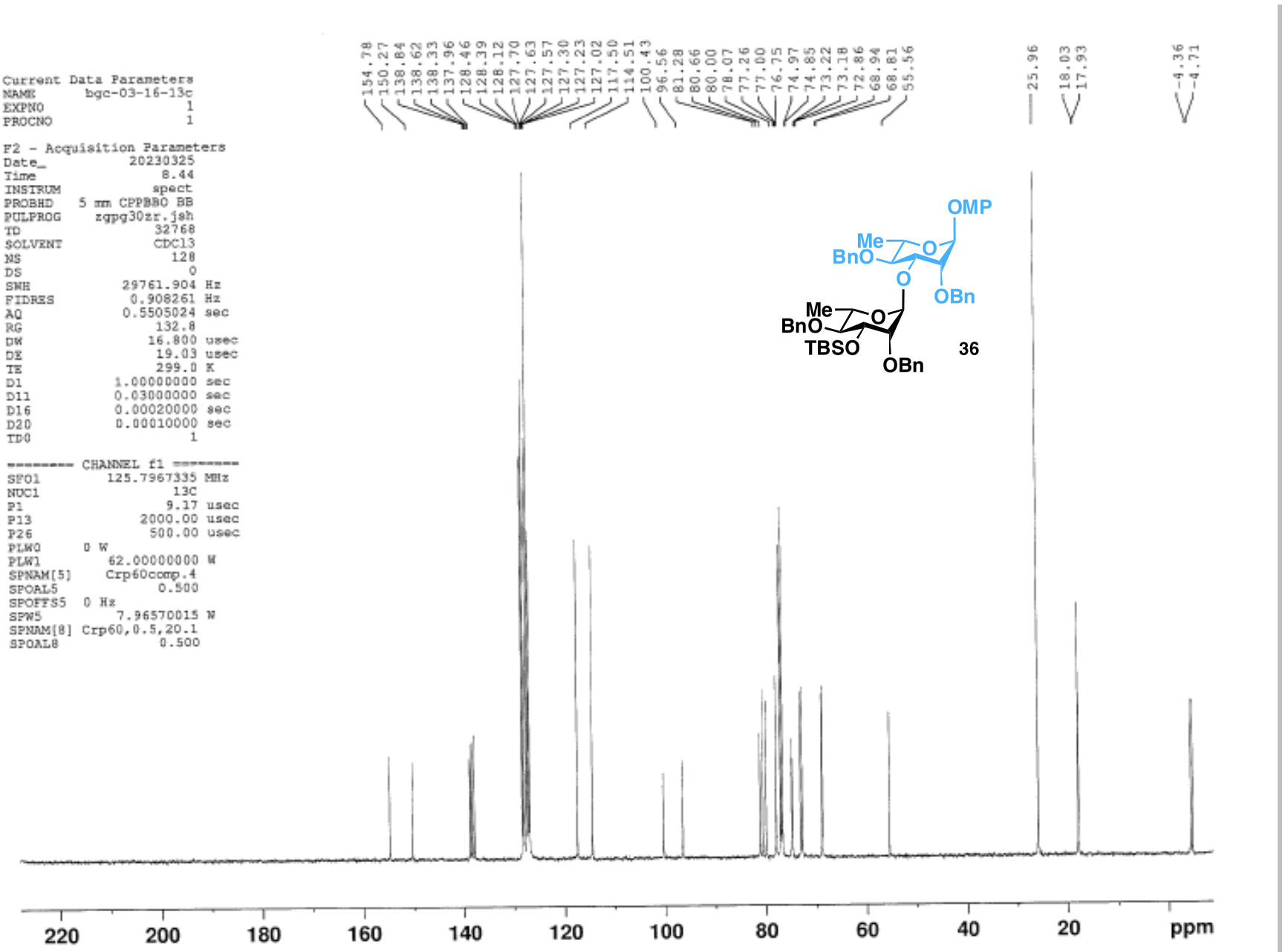
34

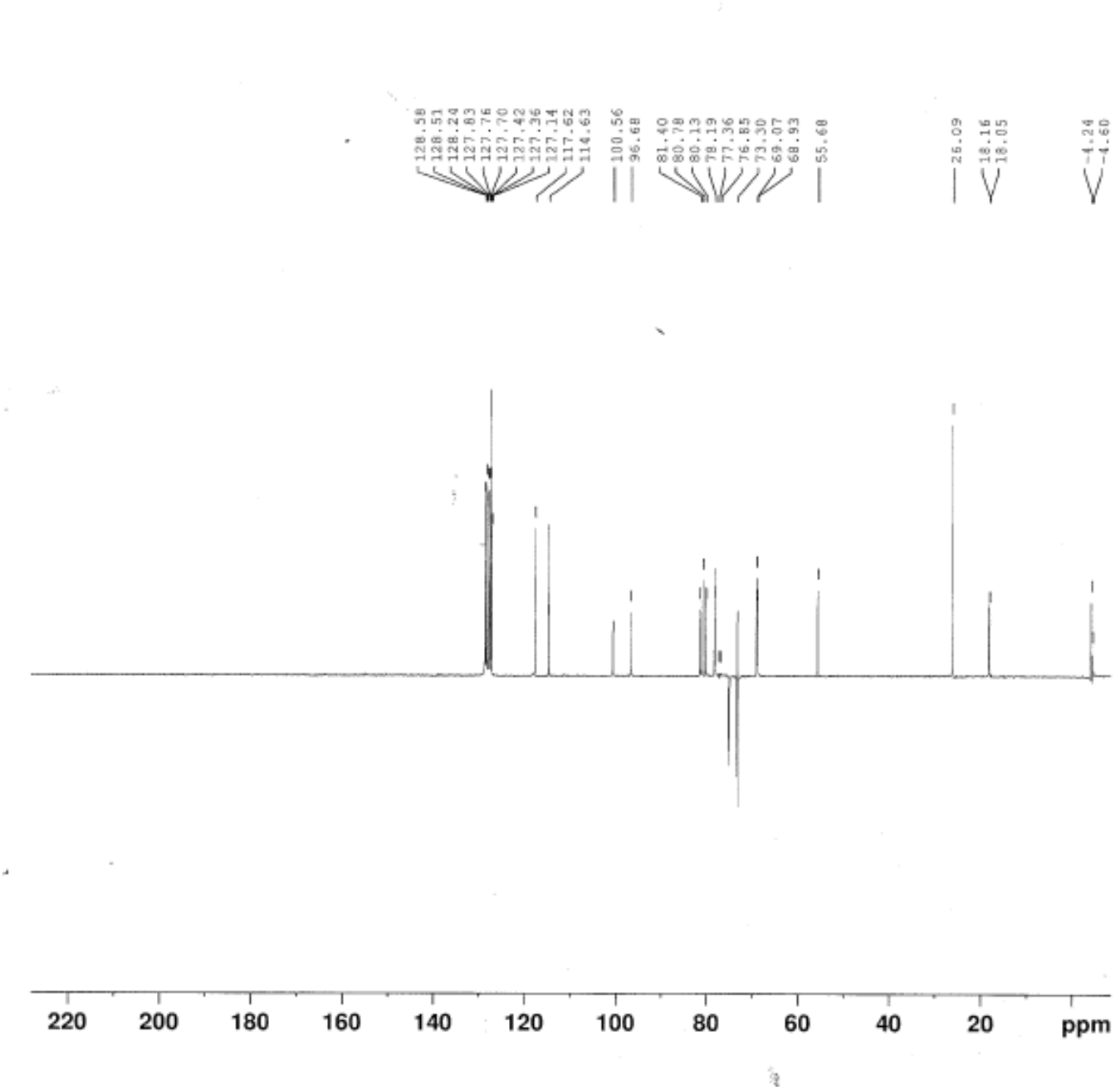



Cl
O
OBn
Me
BnO
TBSO
35
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
6.49
9.85
3.69
1.09
1.16
1.03
1.03
2.17
2.21
1.00
10.60

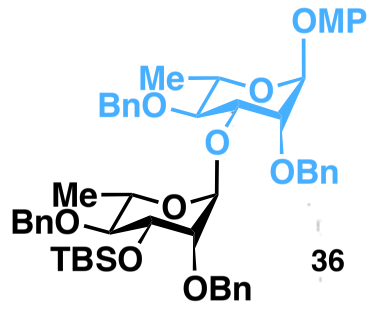

Current Data Parameters
NAME bgc-03-15-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20230323
Time 18.19
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 49
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 299.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
138.24
137.88
128.41
128.26
128.08
127.84
127.64
127.54
92.08
88.35
82.15
81.95
80.47
79.88
77.25
77.00
76.75
76.21
75.96
75.37
73.79
71.57
71.33
53.35
25.92
17.98
17.54
-4.49
-4.74
Cl
Me
BnO
O
TBSO
OBn
35
220
200
180
160
140
120
100
80
60
40
20
ppm


OMP
OBn
Me
BnO
O
O
36
OBn
Me
BnO
TBSO
ppm
6.00
9.28
6.04
1.05
1.94
2.84
2.01
0.95
0.98
0.95
0.96
3.04
1.03
1.87
1.05
0.95
0.96
1.89
1.90
17.35
1.97


Current Data Parameters
NAME bgc-03-16-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20230325
Time 8.44
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 299.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
154.78
150.27
138.84
138.62
138.33
137.96
128.46
128.39
128.12
127.70
127.63
127.57
127.30
127.23
127.02
117.50
114.51
100.43
96.56
81.28
80.66
80.00
78.07
77.26
77.00
76.75
74.97
74.85
73.22
73.18
72.86
68.94
68.81
55.56
25.96
18.03
17.93
-4.36
-4.71
OMP
Me
BnO
O
O
OBn
Me
BnO
O
TBSO
OBn
36
220
200
180
160
140
120
100
80
60
40
20
ppm

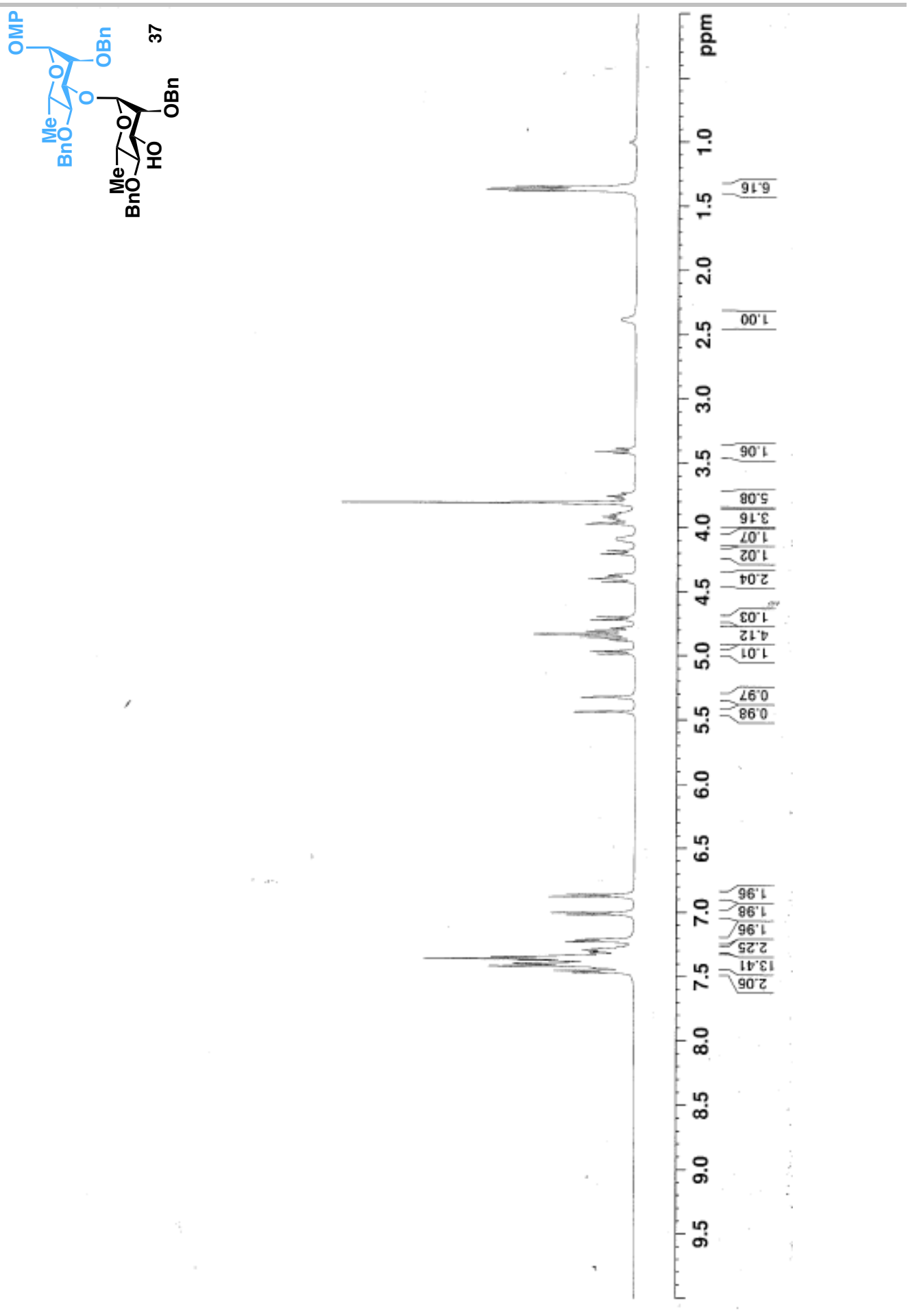


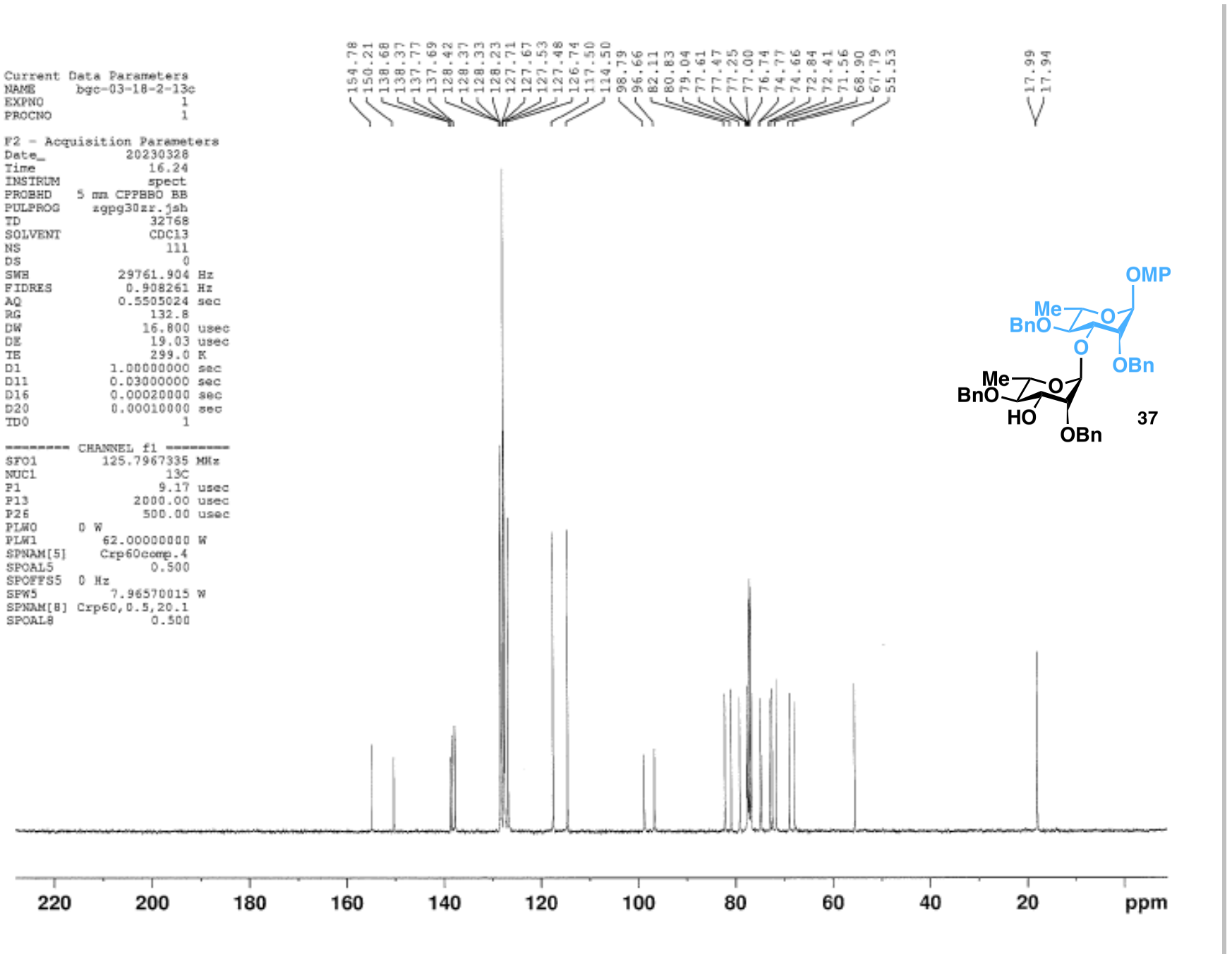

```
Current Data Parameters
NAME      bgc-03-16-dept
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230325
Time                8.48
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             dept
TD                 32768
SOLVENT            CDCl3
NS                    50
DS                     0
SWH            29761.904 Hz
FIDRES         0.908261 Hz
AQ             0.5505024 sec
RG                 132.8
DW                16.800 usec
DE                18.00 usec
TE                299.0 K
CNST2         145.0000000
CNST12          1.5000000
D1              1.00000000 sec
D2              0.00344828 sec
D12             0.00002000 sec
TD0                    1
```

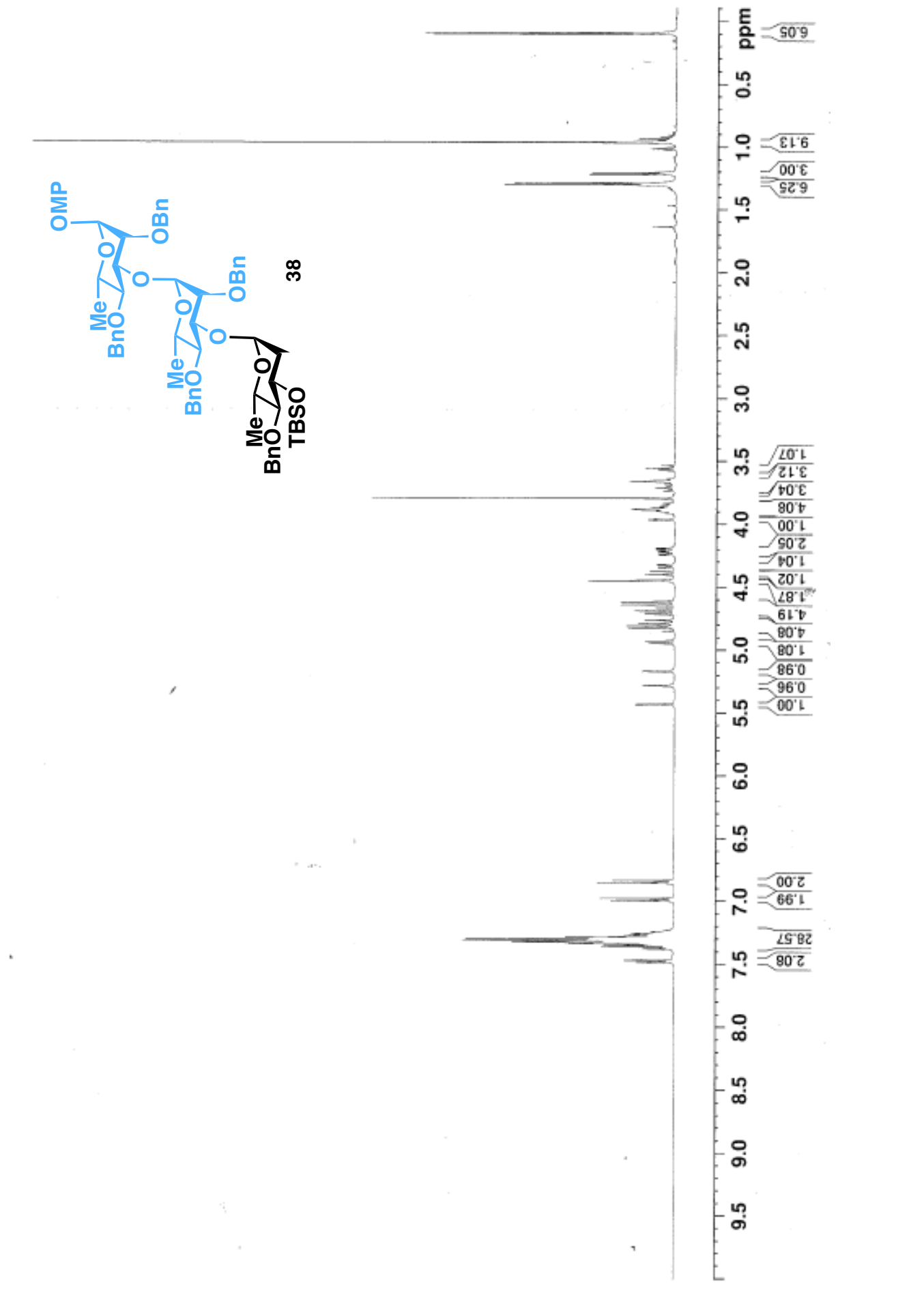


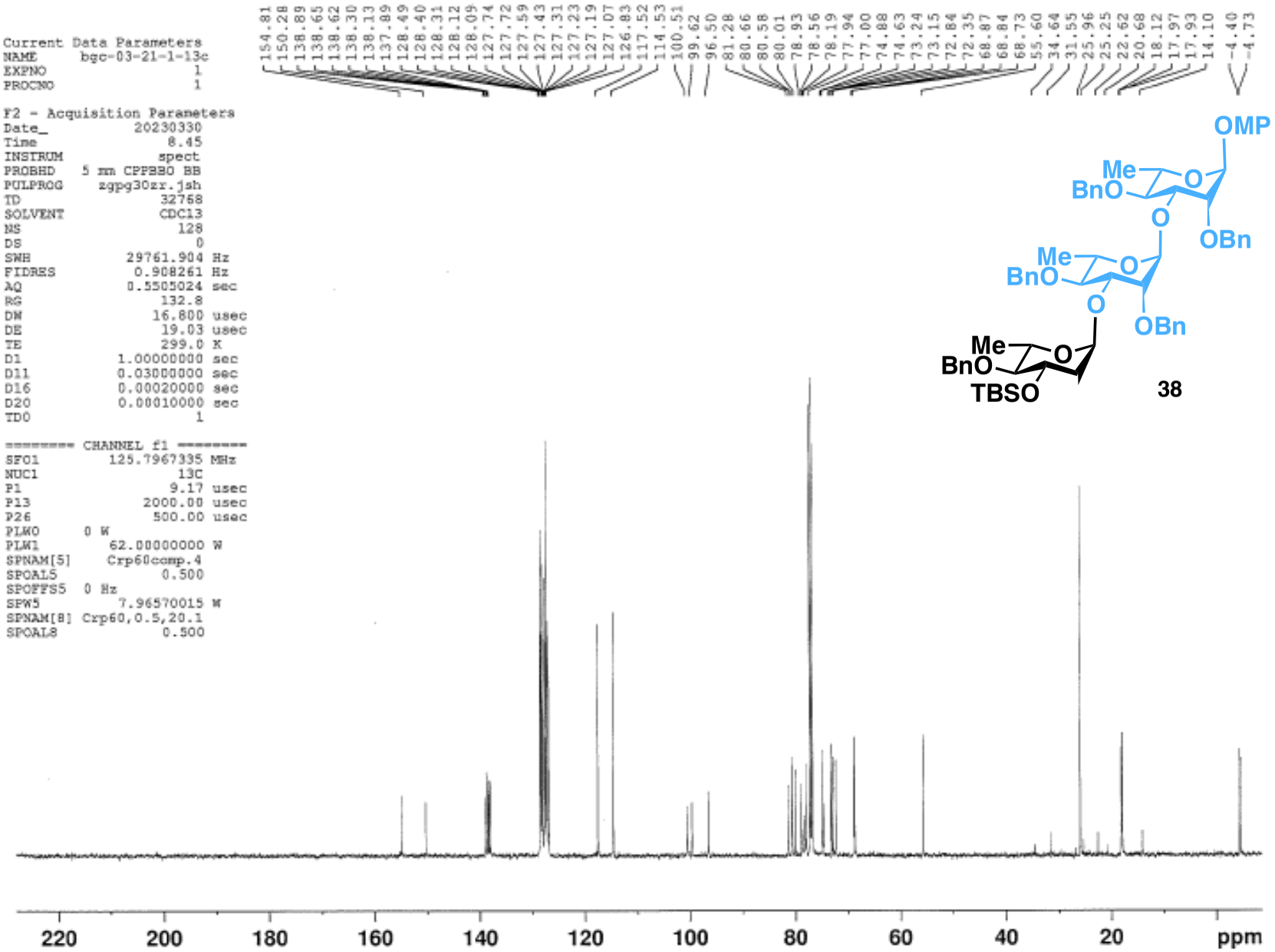
36

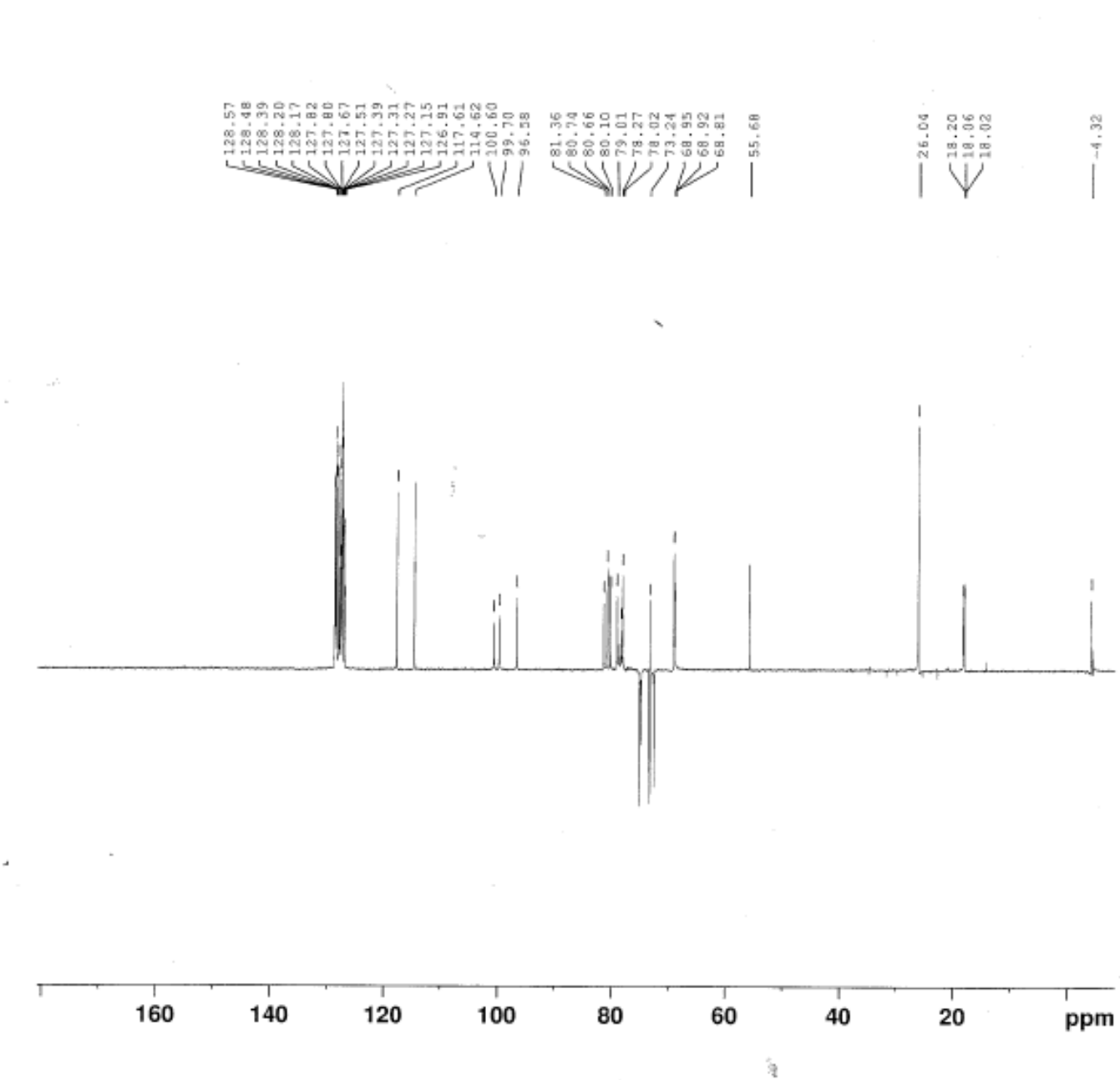
bgc-03-16-hsqc 1 1 /home/localnmr/cen1ow/nmr
H1 standard parameters, cryoprobe prodigy,
General
DATE= 2023/03/25
TIME= 08:49
INSTRUM = spect
PULPROG = hsqcetgpsi
F2 (1H)
SI = 1024
SF = 500.23
SW_p = 4746.835
F1 (13C)
SI = 1024
SF = 125.703
SW_p = 20833.333
F1 [ppm]
F2 [ppm]
OMP
OBn
Me
BnO
TBSO
36


OMP
OBn
Me
BnO
O
O
37
OBn
O
HO
Me
BnO
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
6.16
1.00
1.06
5.08
3.16
1.07
1.02
2.04
1.03
4.12
1.01
0.97
0.98
1.96
1.98
1.96
2.25
13.41
2.06


Current Data Parameters
NAME bgc-03-18-2-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20230328
Time 16.24
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 111
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 299.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
154.78
150.21
138.68
138.37
137.77
137.69
128.42
128.37
128.33
128.23
127.71
127.67
127.53
127.48
126.74
117.50
114.50
98.79
96.66
82.11
80.83
79.04
77.61
77.47
77.25
77.00
76.74
74.77
74.66
72.84
72.41
71.56
68.90
67.79
55.53
17.99
17.94
OMP
Me
BnO
O
O
OBn
Me
BnO
O
HO
OBn
37
220
200
180
160
140
120
100
80
60
40
20
ppm

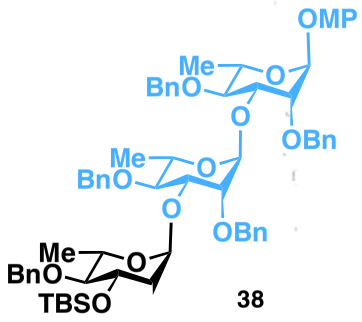

OMP
OBn
Me
BnO
O
OBn
Me
BnO
O
Me
BnO
TBSO
38
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
6.05
9.13
3.00
6.25
1.07
3.12
3.04
4.08
1.00
2.05
1.04
1.02
1.87
4.19
4.08
1.08
0.98
0.96
1.00
1.99
2.00
2.08
28.57


154.81
150.28
138.89
138.65
138.62
138.30
138.13
137.89
128.49
128.40
128.31
128.12
128.09
127.74
127.72
127.59
127.43
127.31
127.23
127.19
127.07
126.83
117.52
114.53
100.51
99.62
96.50
81.28
80.66
80.58
80.01
78.93
78.56
78.19
77.94
77.00
74.88
74.63
73.24
73.15
72.84
72.35
68.87
68.84
68.73
55.60
34.64
31.55
25.96
25.25
22.62
20.68
18.12
17.97
17.93
14.10
-4.40
-4.73
Current Data Parameters
NAME bgc-03-21-1-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20230330
Time 8.45
INSTRUM spect
PROBHD 5 mm CPFBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 299.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
OMP
Me
BnO
O
OBn
Me
BnO
O
OBn
Me
BnO
TBSO
38
220
200
180
160
140
120
100
80
60
40
20
ppm

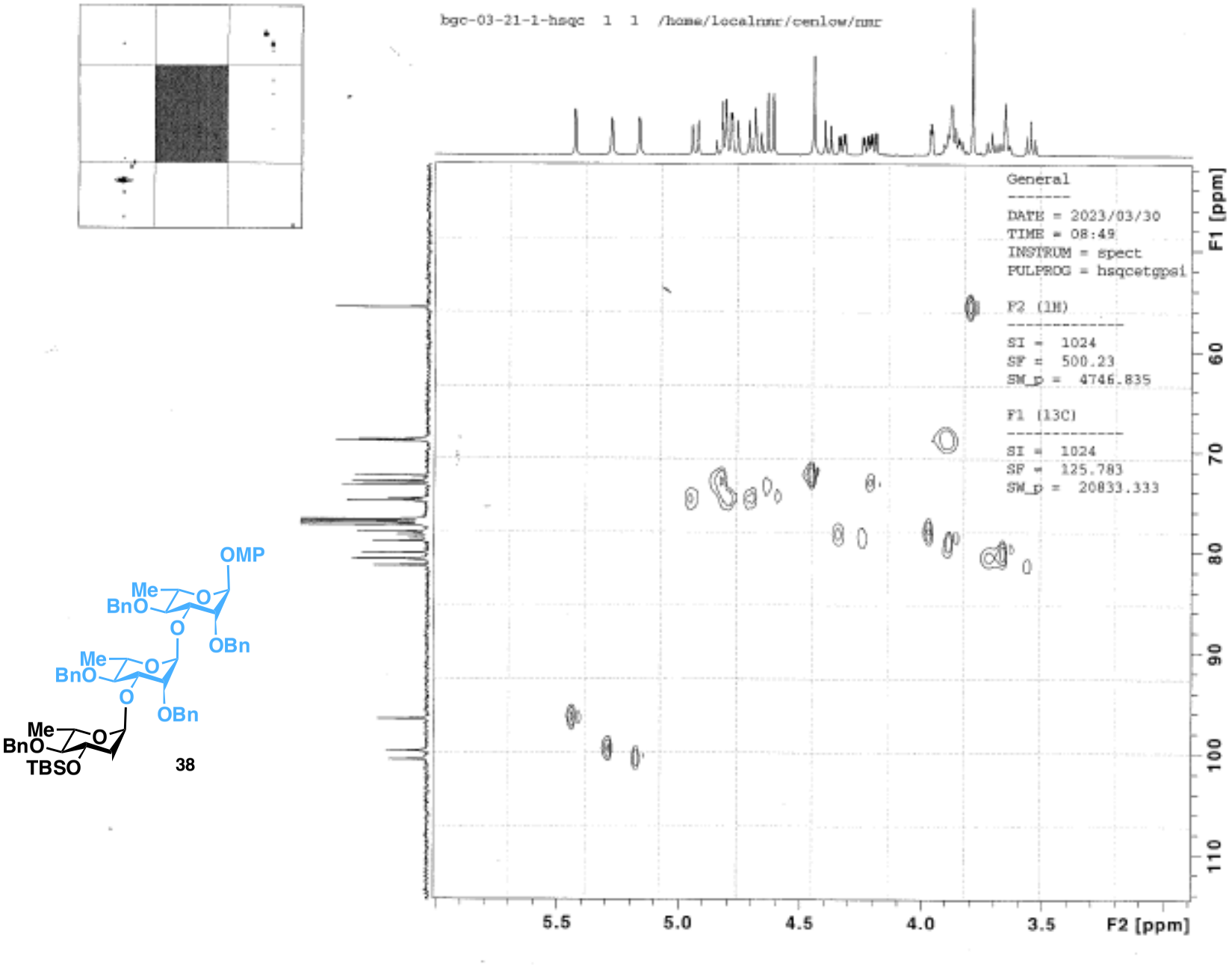


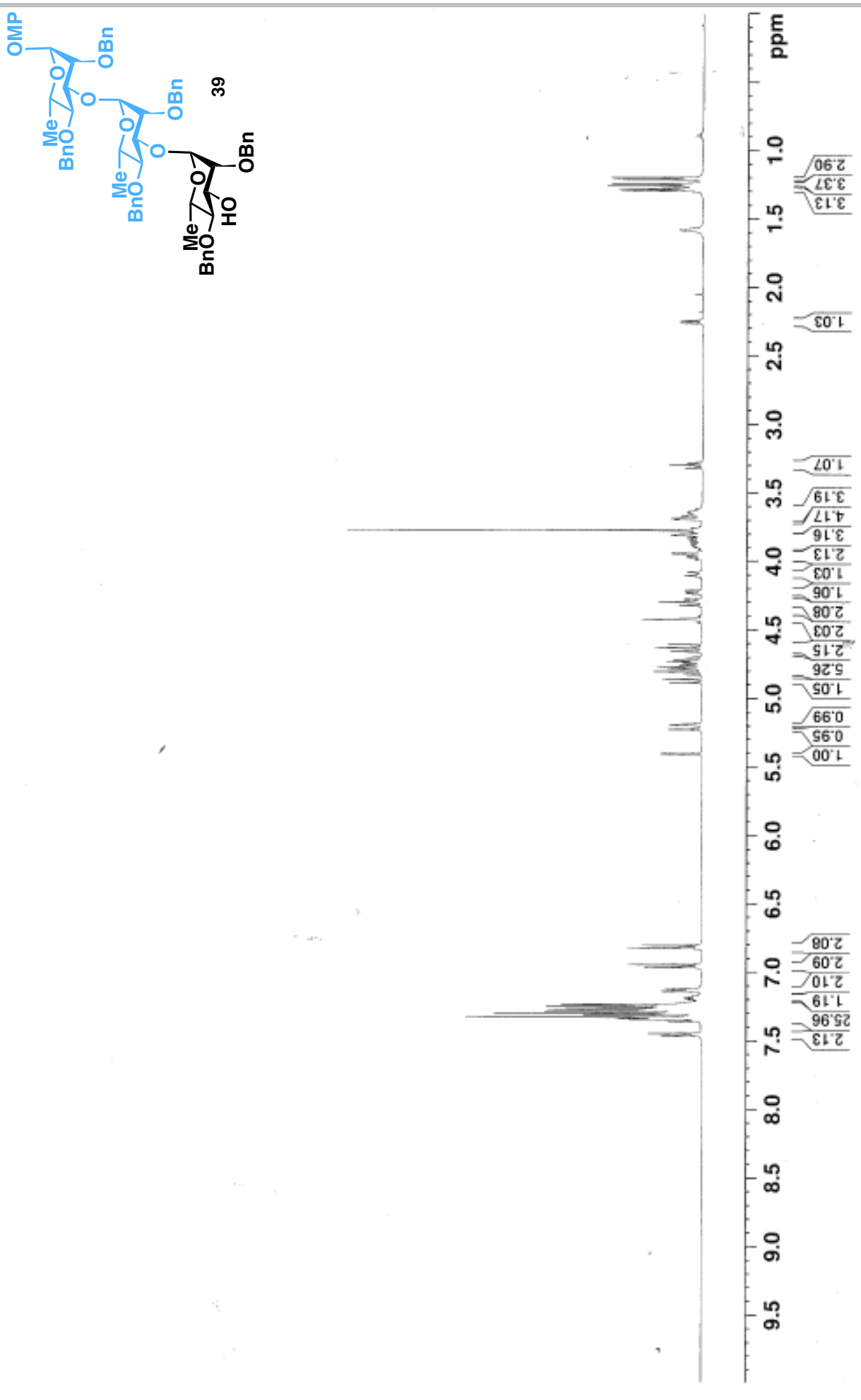
BRUKER

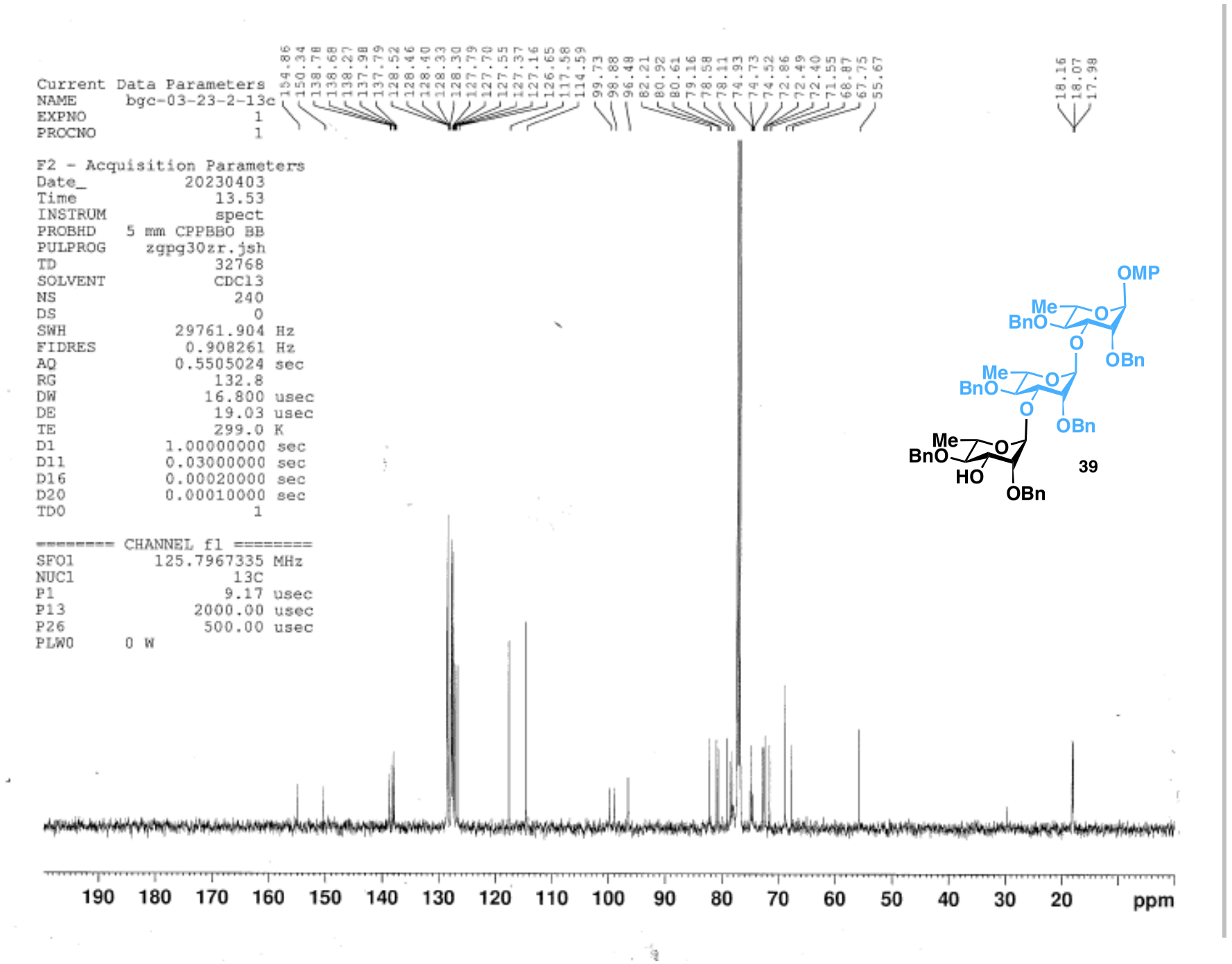
```
Current Data Parameters
NAME      bgc-03-21-1-dept
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20230330
Time                8.46
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG            dept
TD                32768
SOLVENT           CDCl3
NS                  128
DS                    0
SWH           29761.904 Hz
FIDRES         0.908261 Hz
AQ            0.5505024 sec
RG                132.8
DW               16.800 usec
DE                18.00 usec
TE                299.0 K
CNST2        145.0000000
CNST12         1.5000000
D1             1.00000000 sec
D2             0.00344828 sec
D12            0.00002000 sec
TD0                   1
```

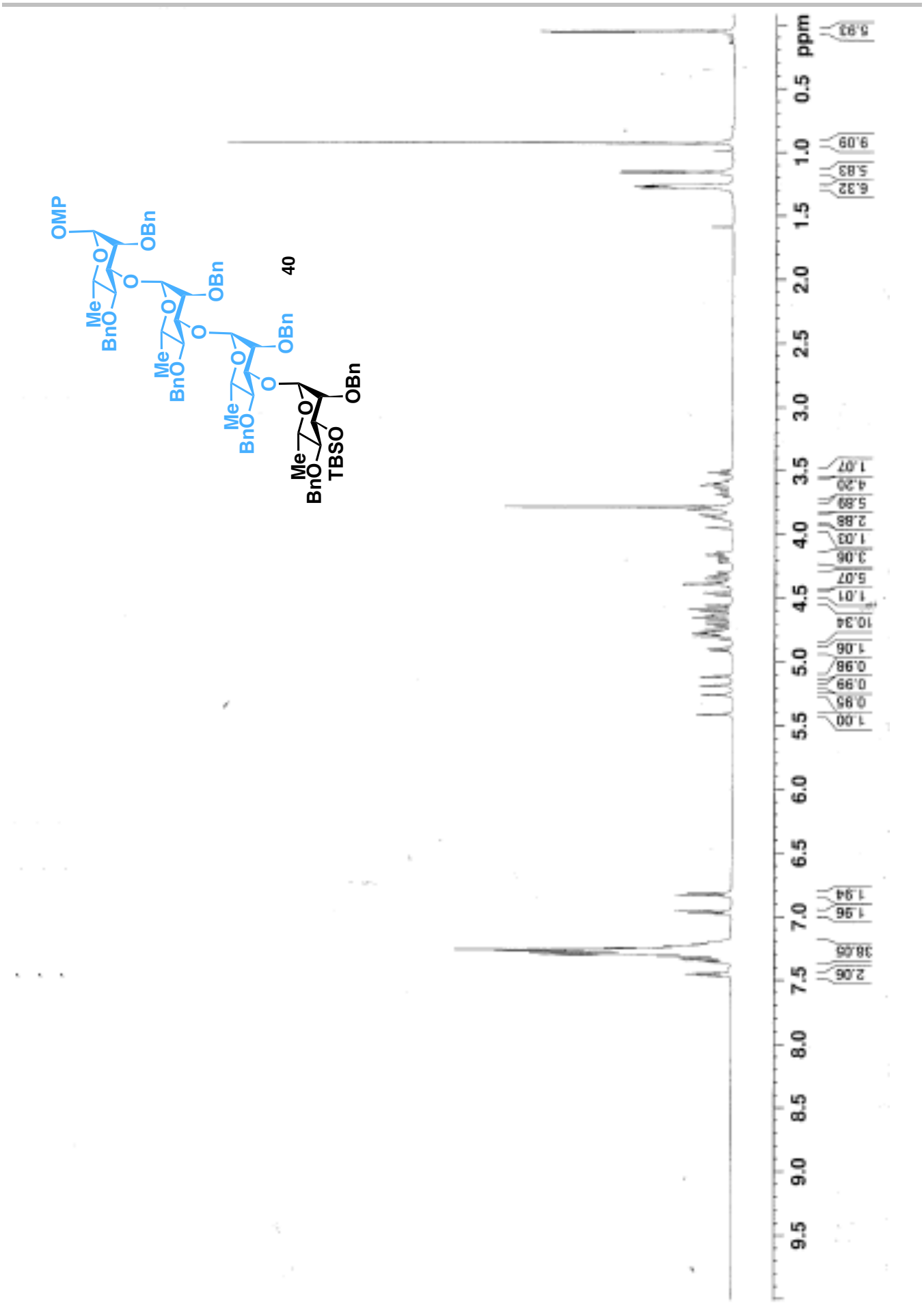


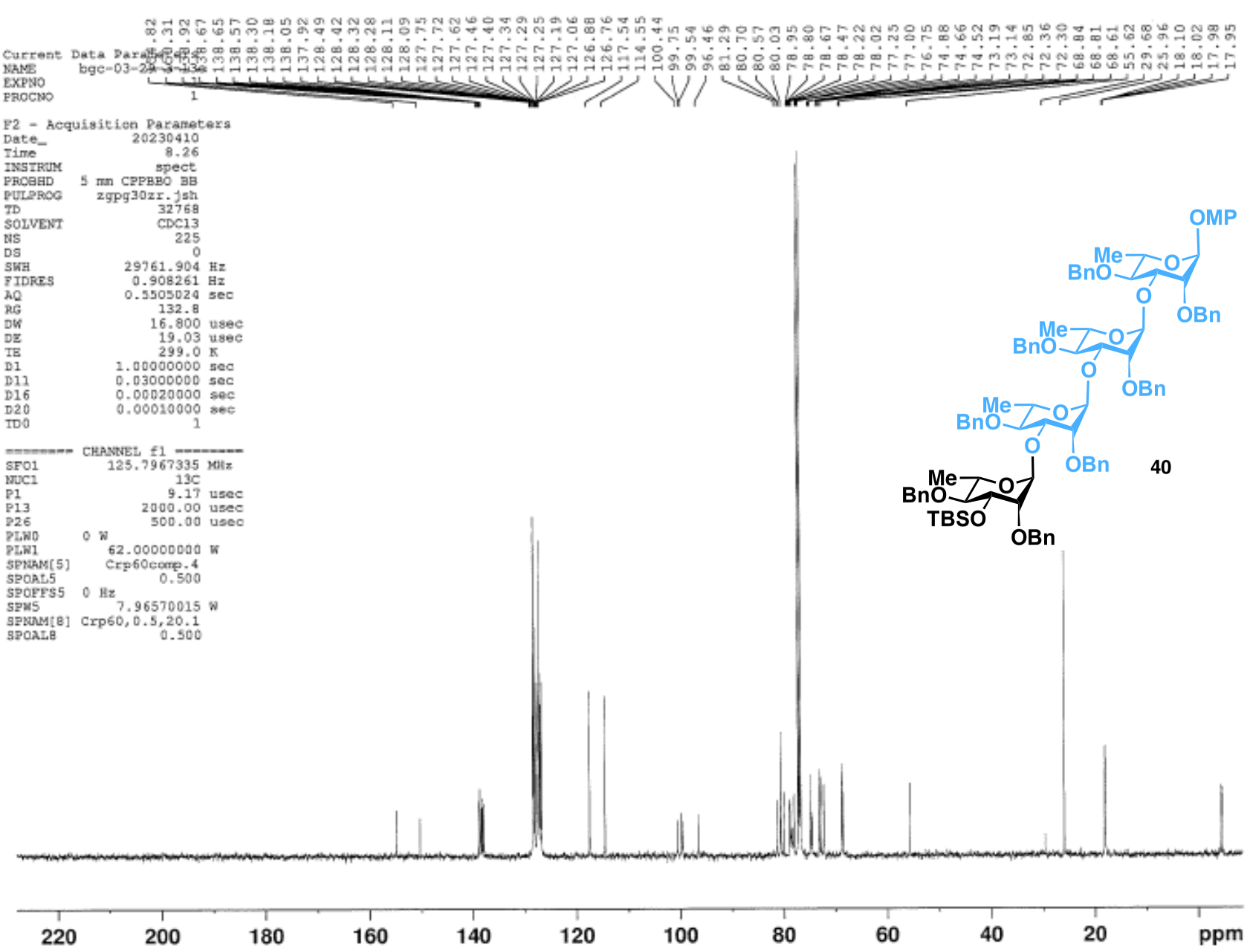
38

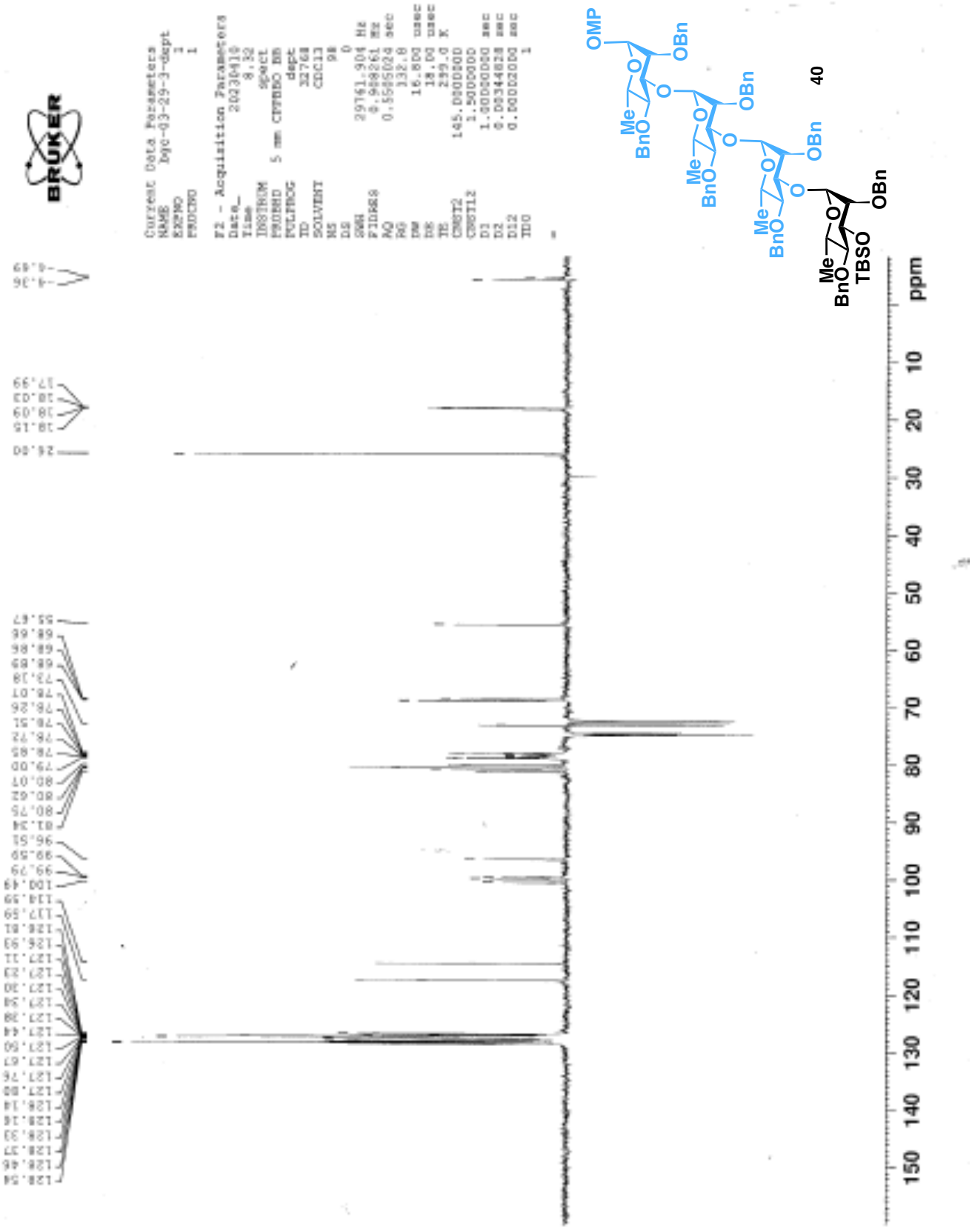

bgc-03-21-1-hsqc 1 1 /home/localnmr/cenlow/nmr
General
DATE = 2023/03/30
TIME = 08:49
INSTRUM = spect
PULPROG = hsqcetgpsi
F2 (1H)
SI = 1024
SF = 500.23
SW_p = 4746.835
F1 (13C)
SI = 1024
SF = 125.783
SW_p = 20833.333
F1 [ppm]
F2 [ppm]
5.5
5.0
4.5
4.0
3.5
60
70
80
90
100
110
OMP
Me
BnO
O
OBn
Me
BnO
O
OBn
Me
BnO
O
TBSO
38

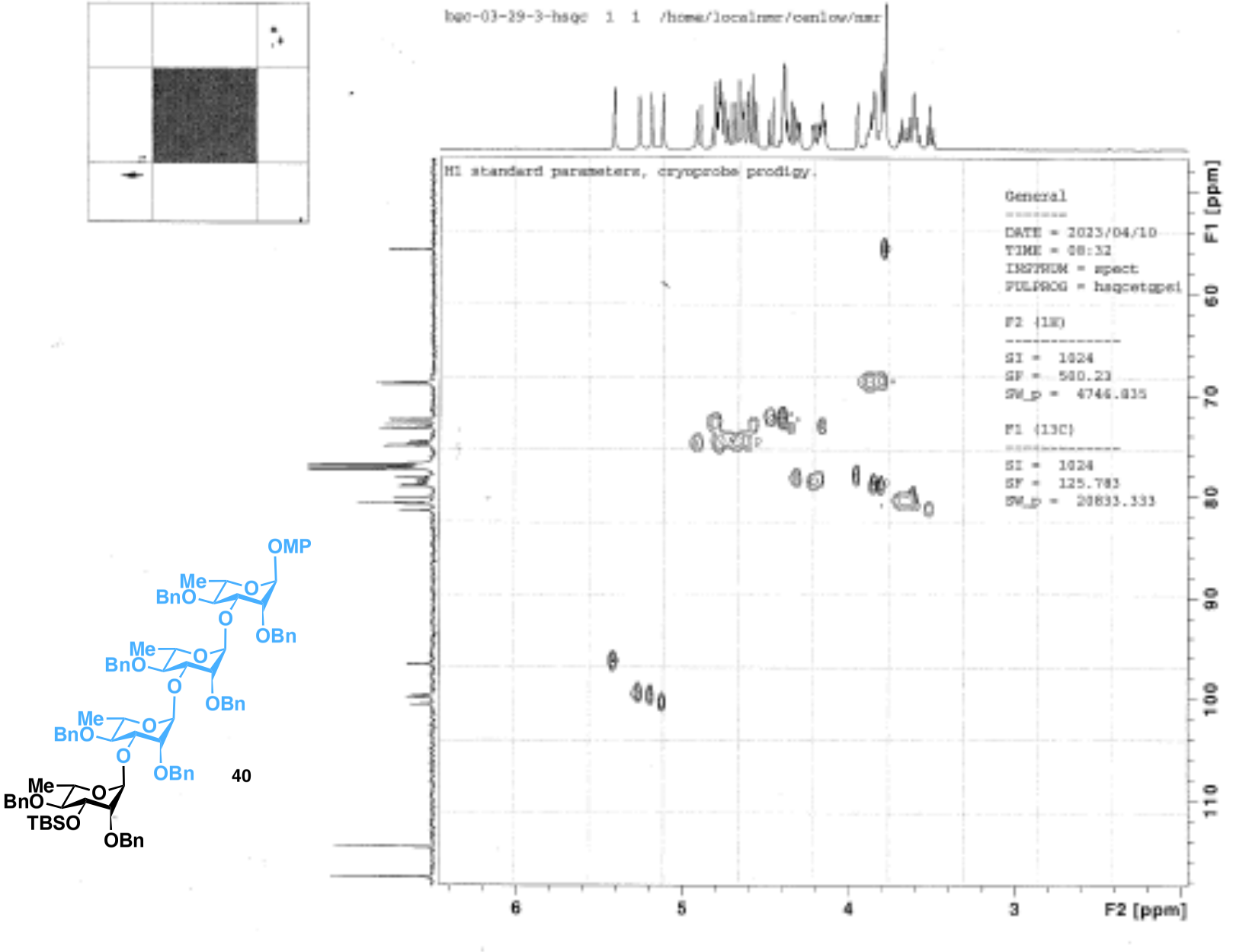

OMP
OBn
Me
BnO
O
OBn
Me
BnO
O
OBn
Me
BnO
O
HO
39
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
2.90
3.37
3.13
1.03
1.07
3.19
4.17
3.16
2.13
1.03
1.06
2.08
2.03
2.15
5.26
1.05
0.99
0.95
1.00
2.08
2.09
2.10
1.19
25.96
2.13

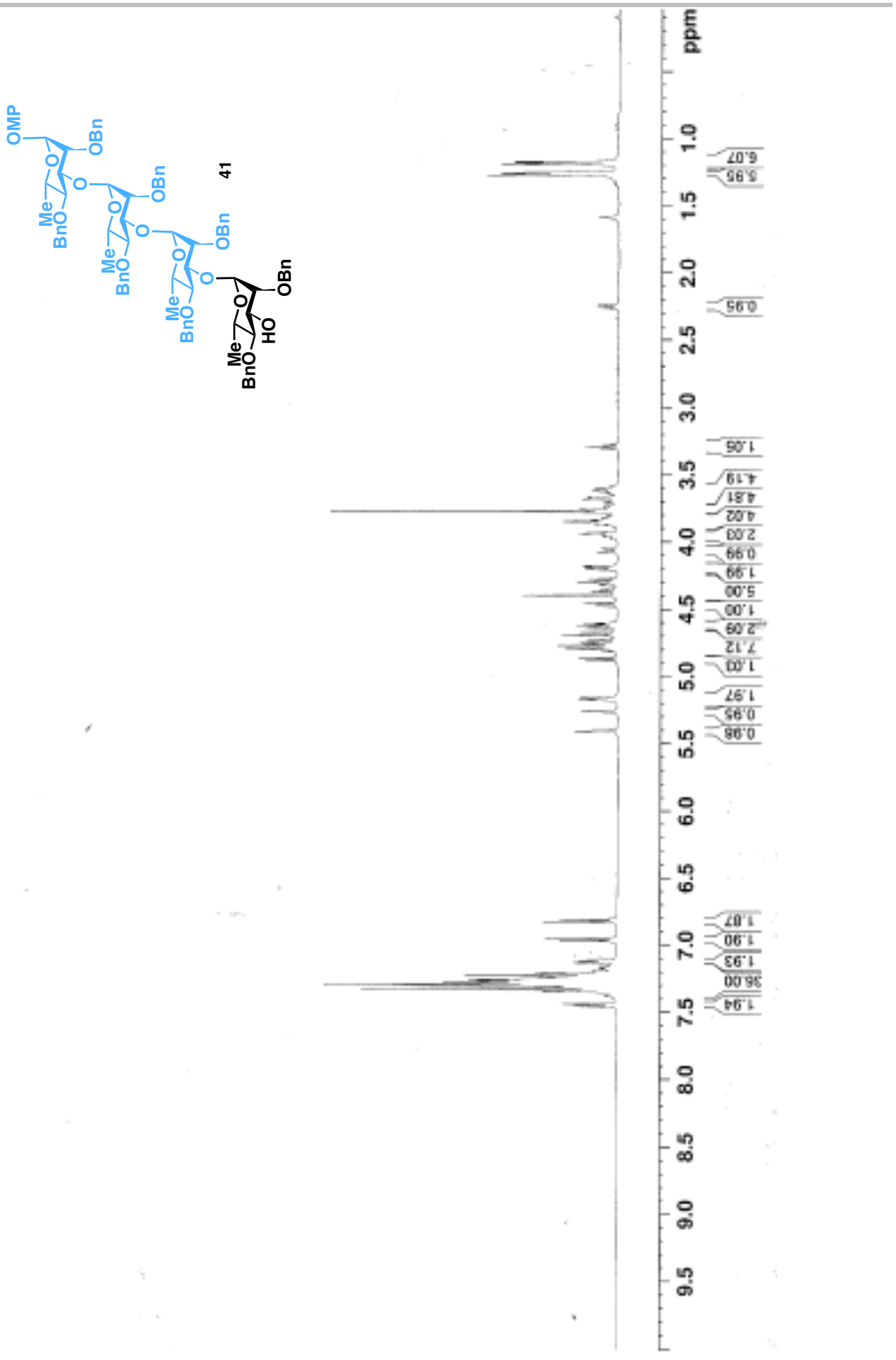

Current Data Parameters
NAME bgc-03-23-2-13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20230403
Time 13.53
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 240
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 299.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
154.86
150.34
138.78
138.68
138.27
137.98
137.79
128.52
128.46
128.40
128.33
128.30
127.79
127.70
127.55
127.37
127.16
126.65
117.58
114.59
99.73
98.88
96.48
82.21
80.92
80.61
79.16
78.58
78.11
74.93
74.73
74.52
72.86
72.49
72.40
71.55
68.87
67.75
55.67
18.16
18.07
17.98
OMP
Me
BnO
O
OBn
Me
BnO
O
OBn
Me
BnO
O
HO
OBn
39
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

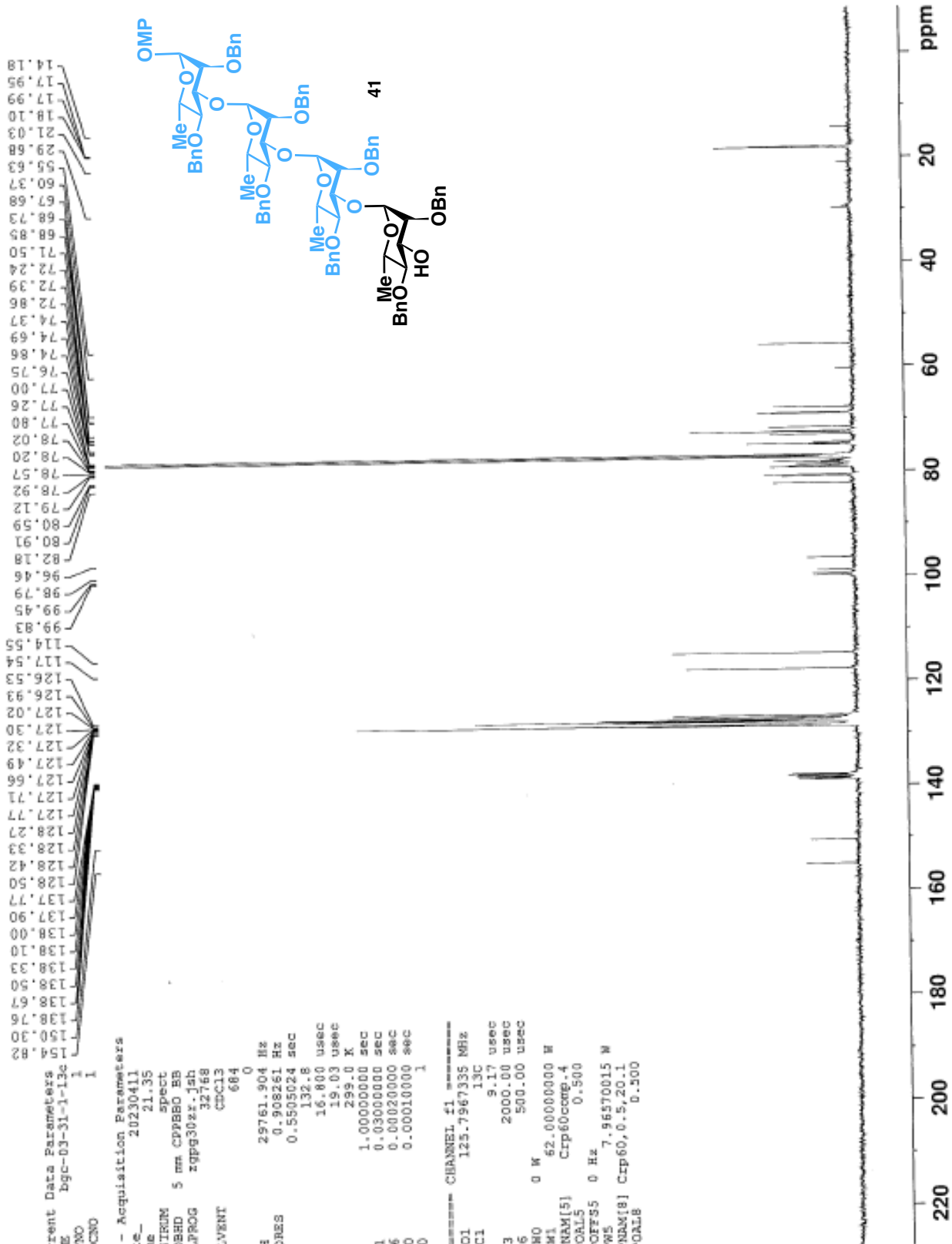

OMP
OBn
Me
BnO
O
OBn
Me
BnO
O
OBn
Me
BnO
O
OBn
Me
BnO
TBSO
OBn
40
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
5.83
9.09
5.83
6.32
1.07
4.20
5.89
2.88
1.03
3.06
5.07
1.01
10.34
1.06
0.98
0.99
0.85
1.00
1.94
1.96
38.05
2.06

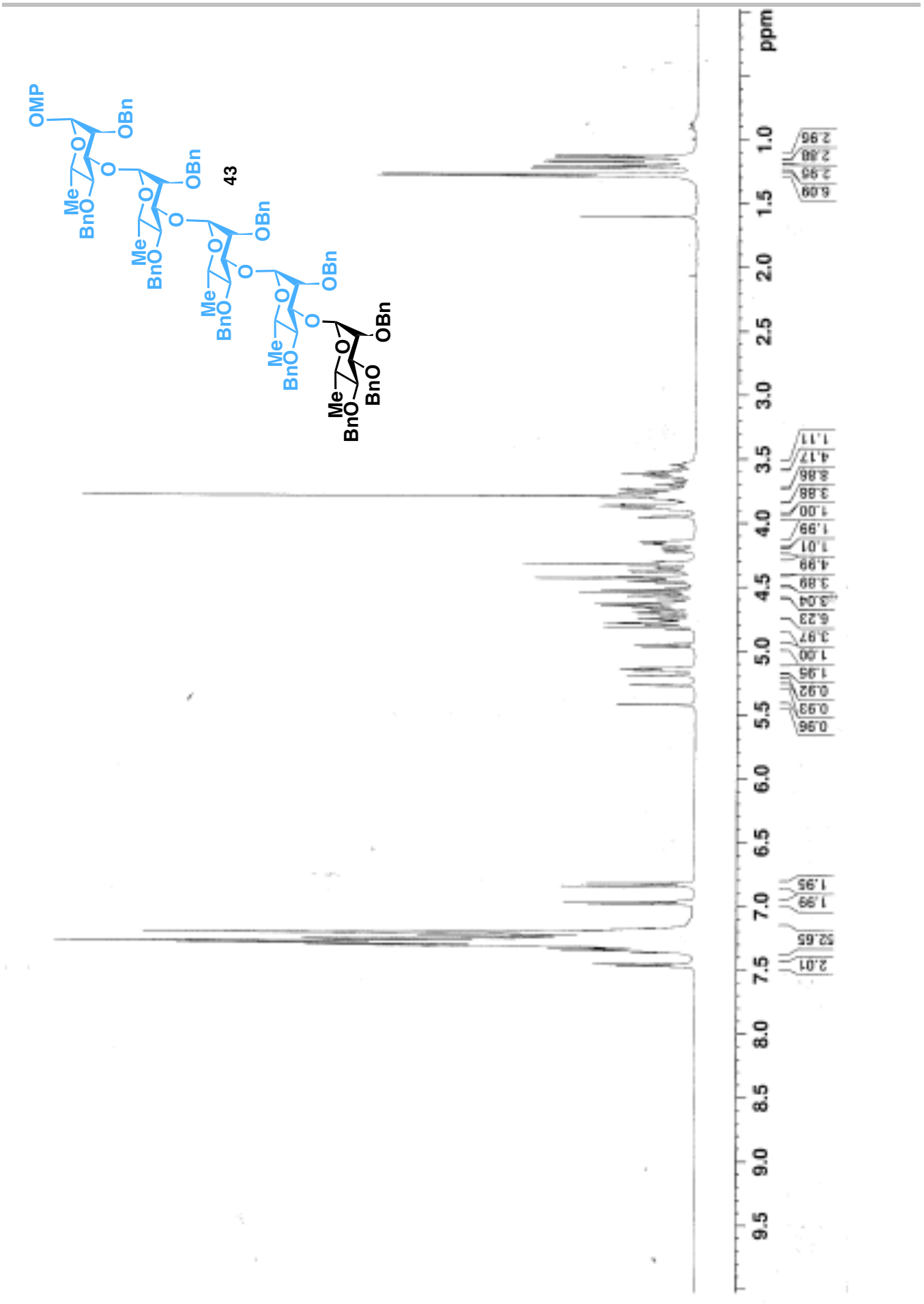

Current Data Parameters
NAME bgc-03-29-a-13c
EXPNO
PROCNO 1
F2 - Acquisition Parameters
Date_ 20230410
Time 8.26
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDC13
NS 225
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 299.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
OMP
Me
BnO
O
OBn
TBSO
40
220
200
180
160
140
120
100
80
60
40
20
ppm

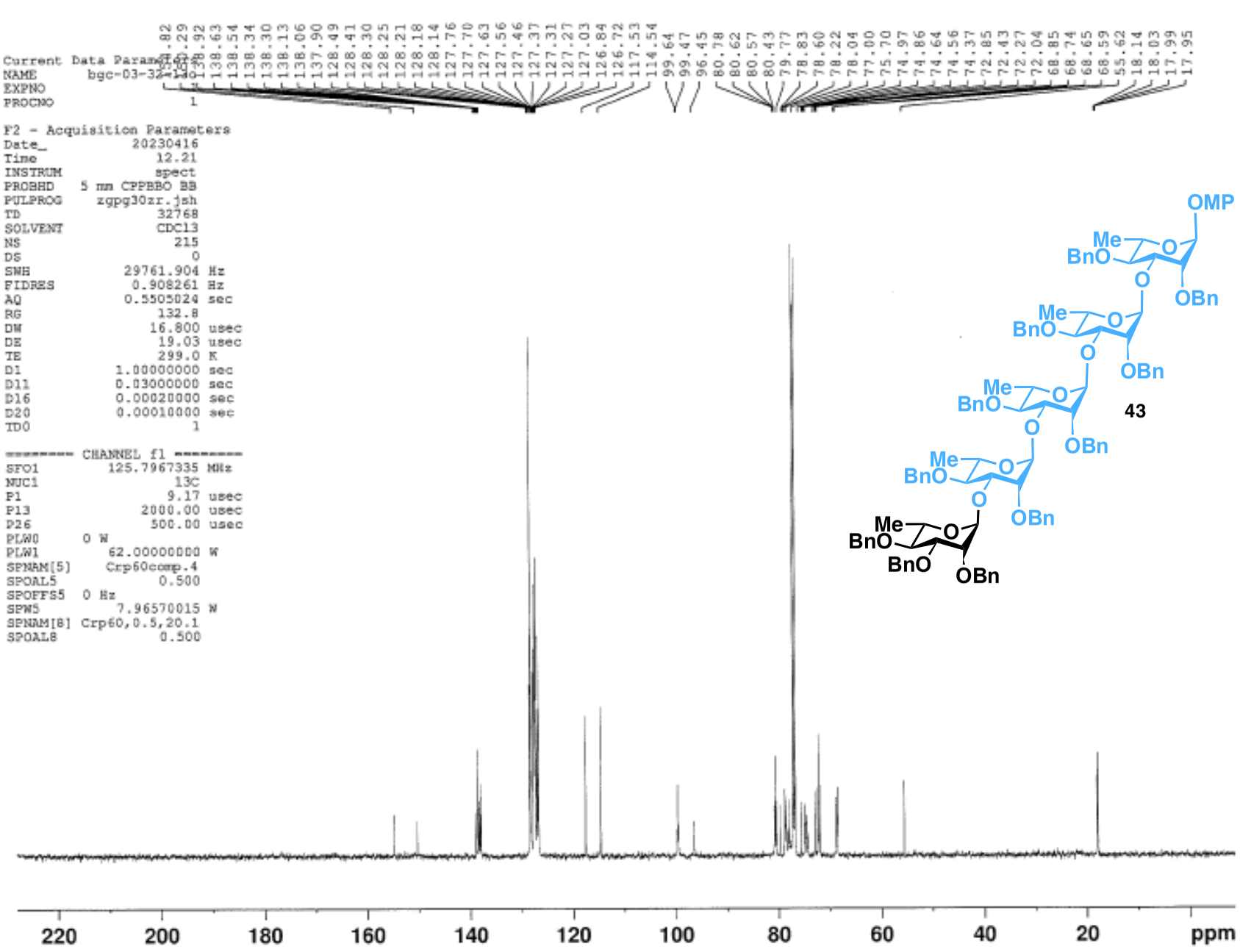

OMP
OBn
Me
BnO
O
OBn
Me
BnO
O
OBn
Me
BnO
O
OBn
Me
BnO
TBSO
40
ppm

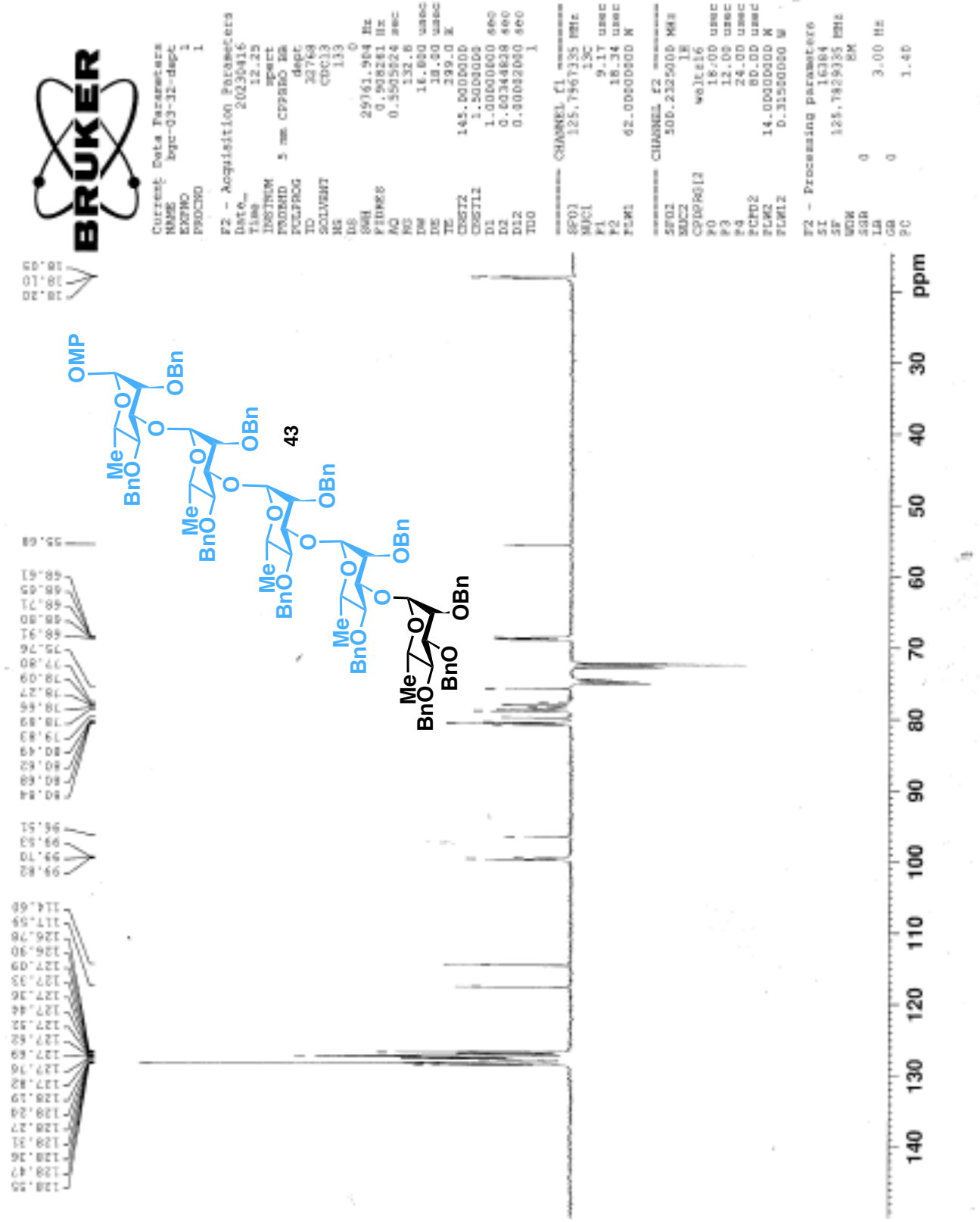

hqc-03-29-3-hsqc 1 1 /home/localnmr/cenlow/nmr
H1 standard parameters, cryoprobe prodigy
General
DATE = 2023/04/10
TIME = 08:32
INSTRUM = spect
PULPROG = hsqcetgpsi
F2 (1H)
SI = 1024
SF = 500.23
SW_p = 4746.835
F1 (13C)
SI = 1024
SF = 125.783
SW_p = 20833.333
F1 [ppm]
F2 [ppm]
OMP
Me
BnO
OBn
TBSO
40

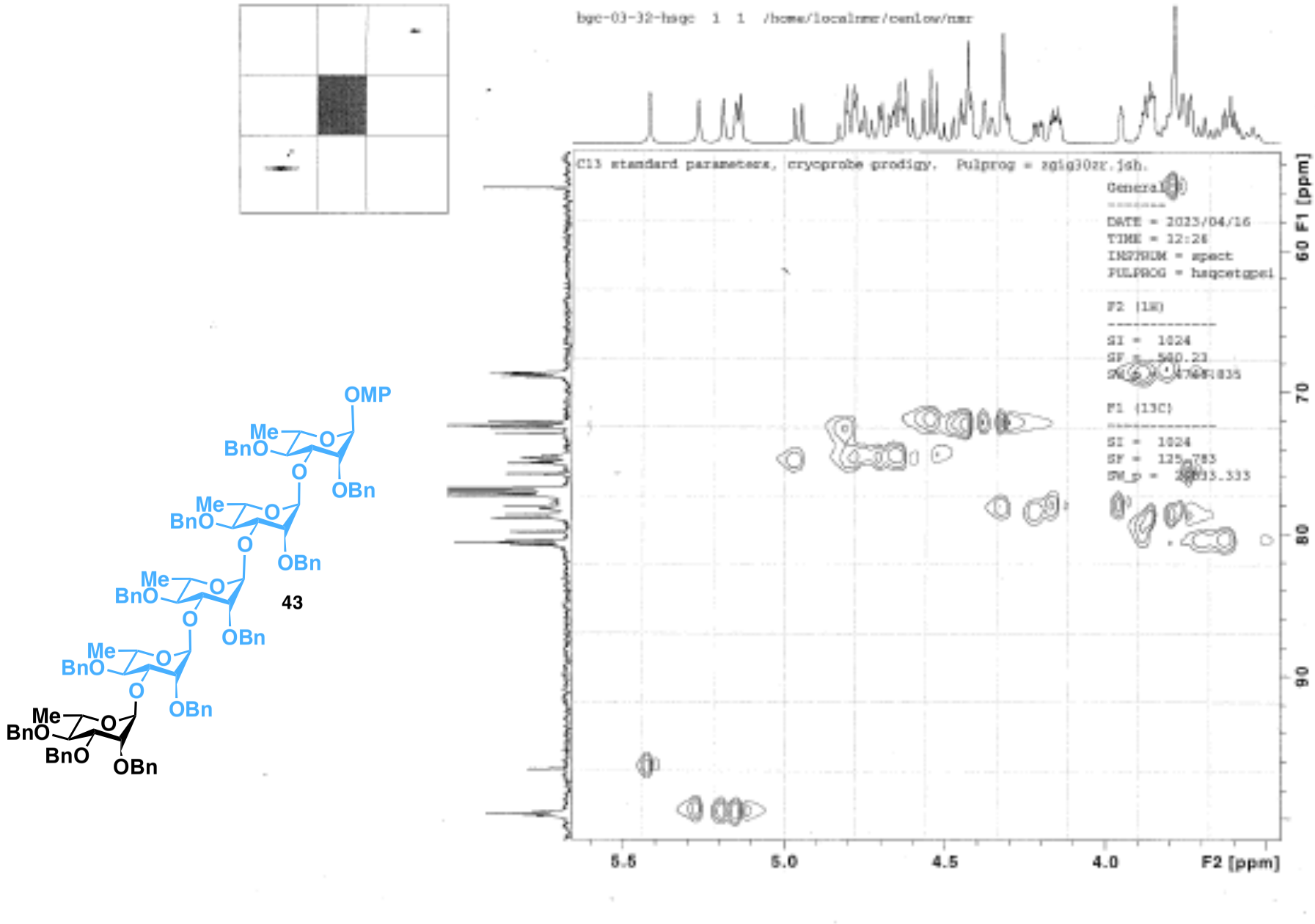

OMP
OBn
Me
BnO
O
41
OBn
OBn
OBn
Me
BnO
HO
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
6.07
5.95
0.95
1.05
4.19
4.81
4.02
2.03
0.99
1.99
5.00
1.00
2.09
7.12
1.03
1.97
0.95
0.98
1.94
36.00
1.93
1.90
1.87


OMP
OBn
Me
BnO
O
41
HO
ppm
220
200
180
160
140
120
100
80
60
40
20


OMP
OBn
Me
BnO
O
43
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
2.96
2.88
2.93
6.09
1.11
4.17
8.86
3.88
1.00
1.99
1.01
4.99
3.89
3.04
6.23
3.97
1.00
1.95
0.92
0.93
0.96
1.95
1.99
52.65
2.01


Current Data Parameters
NAME bgc-03-32-1H-13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20230416
Time 12.21
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30zr.jsh
TD 32768
SOLVENT CDCl3
NS 215
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 132.8
DW 16.800 usec
DE 19.03 usec
TE 299.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00010000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7967335 MHz
NUC1 13C
P1 9.17 usec
P13 2000.00 usec
P26 500.00 usec
PLW0 0 W
PLW1 62.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 7.96570015 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
138.82
138.29
138.92
138.63
138.54
138.34
138.30
138.13
138.06
137.90
128.49
128.41
128.30
128.25
128.21
128.18
128.14
127.76
127.70
127.63
127.56
127.46
127.37
127.31
127.27
127.03
126.84
126.72
117.53
114.54
99.64
99.47
96.45
80.78
80.62
80.57
80.43
79.77
78.83
78.60
78.22
78.04
77.00
75.70
74.97
74.86
74.64
74.56
74.37
72.85
72.43
72.27
72.04
68.85
68.74
68.65
68.59
55.62
18.14
18.03
17.99
17.95
OMP
Me
BnO
O
OBn
43
BnO
220
200
180
160
140
120
100
80
60
40
20
ppm


OMP
OBn
Me
BnO
43
ppm


bgc-03-32-hsqc 1 1 /home/localnmr/cenlow/nmr
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
General
DATE = 2023/04/16
TIME = 12:26
INSTRUM = spect
PULPROG = hsqcetgpsi
F2 (1H)
SI = 1024
F1 (13C)
SI = 1024
SF = 125.783
OMP
Me
BnO
O
OBn
43
5.5
5.0
4.5
4.0
F2 [ppm]
60
70
80
90
F1 [ppm]

6. References

[1] P.R. Verma, P.B. Mukhopadhyay. *Carbohydrate Res.,* **2010**, *345*, 432-436.
[2] P.J. Garegg, T. Nordberg, P. Konradsson, S.C.T. Svensson. *Carbohydrate Res.,* **1983**, *116*, 308-311.