

Supporting Information for:

**A Glycosylation Protocol Based on Activation of Glycosyl 2-Pyridyl
Sulfones with Samarium Triflate**

Grace X. Chang and Todd L. Lowary

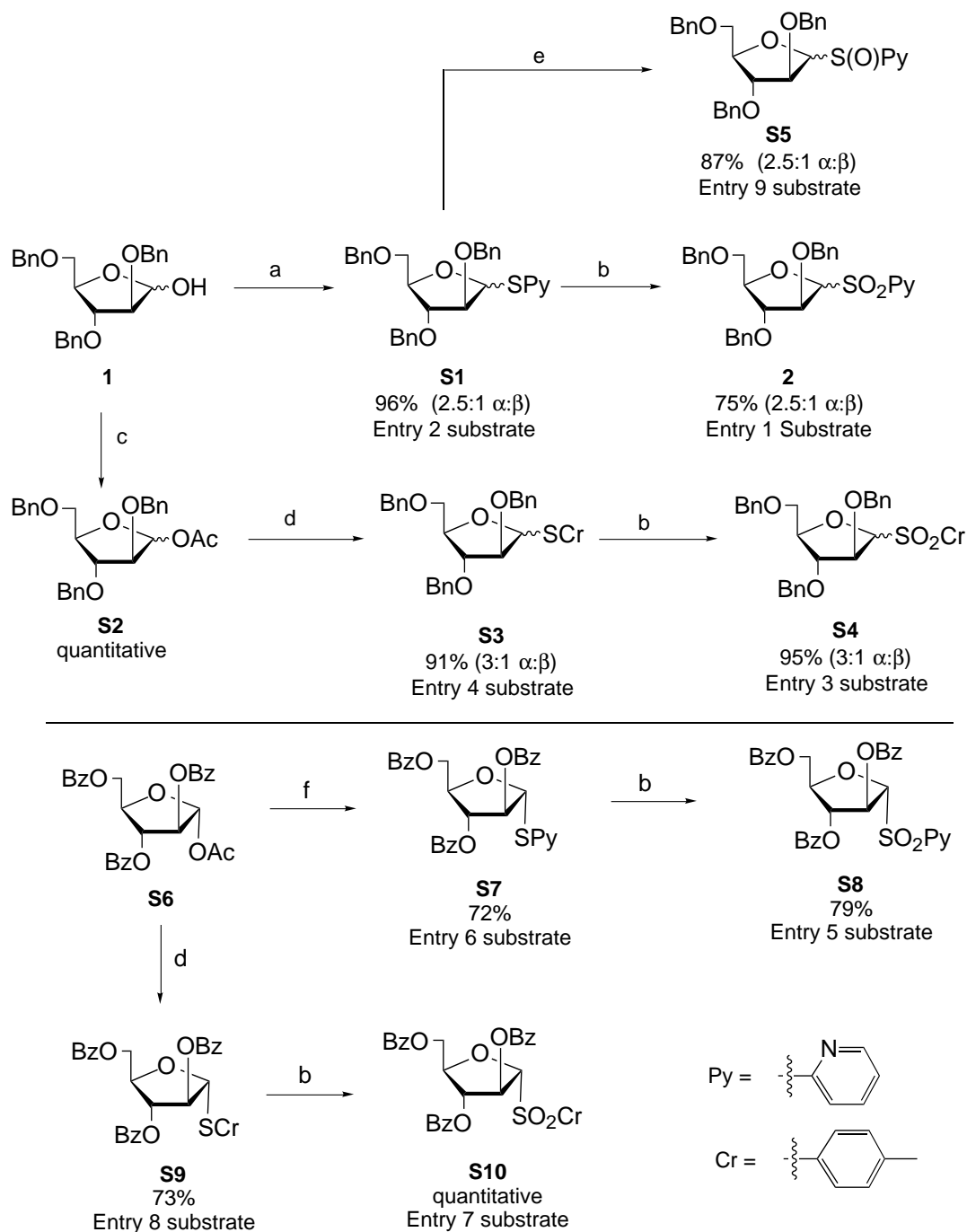
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General Procedure

Methyl 2-O-[2,3,4,6-tetra-O-benzyl- α -D-mannopyranosyl]-3,4,6-tri-O-benzyl- α/β -D-mannopyranoside (**15**). Pyridyl sulfone **3** (148 mg, 0.22 mmol) and acceptor **6** (99 mg, 0.148 mmol) were dissolved in dry toluene (10 mL) and the flask was purged with argon. Sm(OTf)₃ (88 mg, 0.14 mmol) was added to the reaction mixture in one portion under argon and the reaction mixture was heated at 70 °C for 18 h. The reaction mixture was then cooled, evaporated and the residue was partitioned between CH₂Cl₂ and H₂O. The organic layer was washed with brine, dried over Na₂SO₄, and then concentrated to afford a light yellow oil. The oil was purified by column chromatography (6:1 hexane- EtOAc) to yield **15** (133 mg, 89%) as a clear syrup.

Preparation of Substrates Used in Table 1



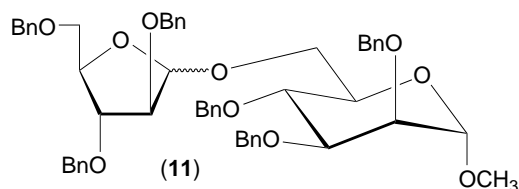
(a) PySSPy, $n\text{-Bu}_3\text{P}$, CH_2Cl_2 , rt, 2 h; (b) $m\text{-CPBA}$ (4.0 equiv), NaHCO_3 , CH_2Cl_2 , rt, 4 h; (c) Ac_2O , pyridine, 0°C , 1 h; (d) CrSH , $\text{BF}_3\text{-OEt}_2$, CH_2Cl_2 , 0°C , 1 h; (e) $m\text{-CPBA}$ (1.0 equiv), CH_2Cl_2 , -78°C , 0.5 h; (f) 2-mercaptopyridine, $\text{BF}_3\text{-OEt}_2$, CH_2Cl_2 , 0°C to rt, overnight.

Experimental Data

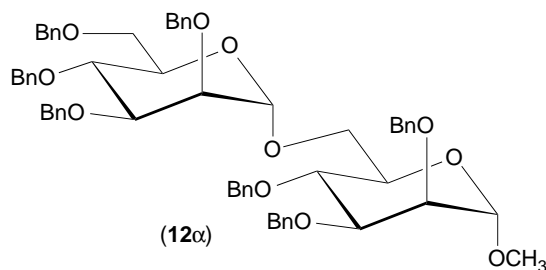
Known Compounds:

- 7** Dourtoglou, V.; Gross, B. *J. Carbohydr. Chem.* **1983**, 2, 57.
8 Subramaniam, V.; Lowary, T. L. *Tetrahedron* **1999**, 55, 5965
9 Szarek, W. A.; Jarell, H. C.; Jones, J. K. N. *Carbohydr. Res.* **1977**, 57, C13.
10 Dourtoglou, V.; Gross, B. *J. Carbohydr. Chem.* **1983**, 2, 57.

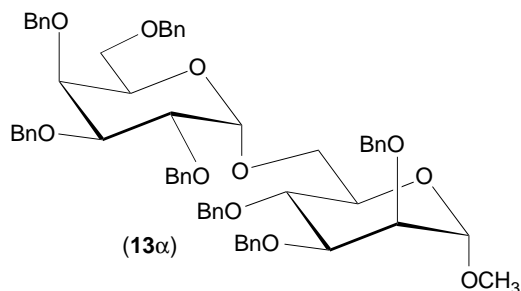
New Compounds:



¹H NMR (400 MHz, CDCl₃) δ_H 7.35–7.23 (m, 30 H), 5.30 (s, 0.3 H), 5.11 (d, 0.7 H, *J* = 4.1 Hz), 4.90–4.42 (m, 13 H), 4.13–3.60 (m, 11 H), 3.28 (s, 0.9 H), 3.18 (s, 2.1 H); ¹³C NMR (100.623 MHz, CDCl₃) δ_C 139.08, 138.83, 138.76, 138.74, 128.79, 128.75, 128.53, 128.39, 128.32, 128.25, 128.20, 128.15, 128.10, 128.05, 128.01, 107.15, 101.60, 99.66, 99.37, 88.39, 84.65, 84.20, 84.11, 81.08, 80.74, 75.71, 75.53, 75.31, 75.14, 73.77, 73.70, 73.22, 72.65, 72.59, 72.51, 72.33, 72.23, 72.15, 70.20, 67.66, 55.10. ESIMS: calcd [M + Na]⁺ C₅₄H₅₈O₁₀Na 889.39221; found 889.38607.

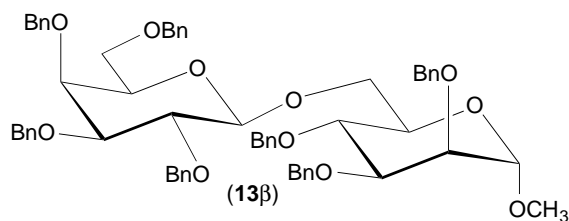


¹H NMR (400 MHz, CDCl₃) δ_H 7.36–7.16 (m, 35 H), 4.89–4.45 (m, 15 H), 3.92–3.65 (m, 13 H), 3.23 (s, 3 H); ¹³C NMR (100.623 MHz, CDCl₃) δ_C 138.47, 128.30, 128.27, 128.24, 128.16, 127.83, 127.76, 127.69, 127.66, 127.63, 127.57, 127.53, 127.44, 127.42, 127.29, 98.89, 98.04, 80.26, 79.26, 74.94, 74.94, 74.94, 74.87, 74.78, 74.74, 74.53, 73.21, 72.79, 72.32, 72.06, 71.80, 71.44, 71.40, 69.17, 54.59. ESIMS: calcd [M + Na]⁺ C₆₂H₆₆O₁₁Na 1009.44973; found 1009.45691.

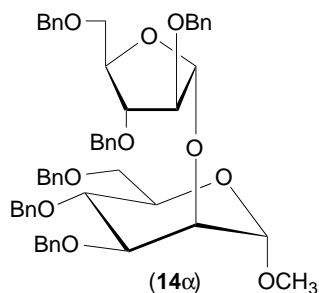


¹H NMR (400 MHz, CDCl₃) δ_H 7.37–7.17 (m, 35 H), 5.11 (d, 1 H, *J* = 5.2 Hz), 4.97–4.93 (m, 15 H), 4.07–3.76 (m, 10 H), 3.57–3.52 (m, 2 H), 3.20 (s, 3 H); ¹³C NMR (100.623 MHz,

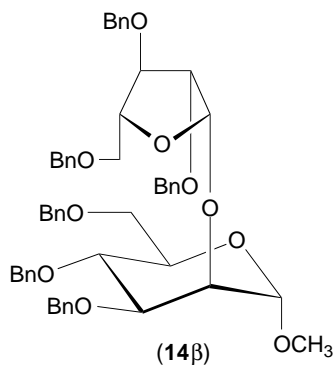
CDCl₃) δ_C 139.39, 139.36, 128.83, 128.80, 128.76, 128.68, 128.61, 128.51, 128.36, 128.33, 128.26, 128.11, 127.96, 127.84, 127.80, 99.25, 97.69, 80.76, 78.84, 77.01, 75.67, 75.67, 75.52, 75.25, 75.24, 73.82, 73.30, 73.29, 73.18, 72.61, 72.09, 69.62, 69.29, 66.88, 55.08. ESIMS: calcd [M + Na]⁺ C₆₂H₆₆O₁₁Na 1009.44973; found 1009.45409.



¹H NMR (400 MHz, CDCl₃) δ_H 7.37–7.20 (m, 35 H), 5.03–4.92 (m, 2 H), 4.80–4.21 (m, 15 H), 3.90–3.47 (m, 11 H), 3.21 (s, 3 H); ¹³C NMR (100.623 MHz, CDCl₃) δ_C 139.35, 139.20, 139.02, 138.73, 138.36, 128.84, 128.73, 128.66, 128.59, 128.55, 128.29, 128.20, 127.98, 127.92, 127.85, 127.70, 104.87, 99.21, 82.53, 80.69, 79.88, 75.64, 75.49, 75.38, 74.98, 74.94, 74.07, 73.94, 73.67, 73.44, 73.12, 72.46, 71.85, 69.59, 69.09, 55.11. calcd [M + Na]⁺ C₆₂H₆₆O₁₁Na 1009.444973; found 1009.44130.

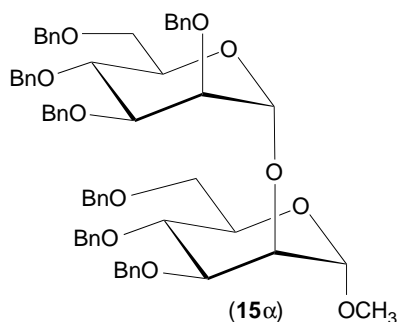


¹H NMR (400 MHz, CDCl₃) δ_H 7.31–7.18 (m, 30 H), 5.37 (d, 1 H, *J* = 1.3 Hz), 4.88 (d, 1 H, *J* = 10.8 Hz), 4.80 (d, 1 H, *J* = 1.6 Hz), 4.70–4.36 (m, 11 H), 4.23–4.22 (m, 1 H), 4.14–4.13 (m, 2 H), 3.96–3.92 (m, 3 H), 3.80–3.66 (m, 4 H), 3.58 (dd, 1 H, *J* = 5.1, 11.0 Hz), 3.33 (s, 3 H); ¹³C NMR (100.623 MHz, CDCl₃) δ_C 138.91, 138.06, 128.92, 128.81, 128.78, 128.76, 128.73, 128.39, 128.30, 128.21, 128.19, 128.10, 128.07, 127.90, 108.18, 101.20, 88.67, 83.44, 80.70, 80.07, 75.62, 75.56, 74.39, 73.88, 73.81, 73.20, 72.56, 72.23, 72.18, 69.81, 69.74, 55.15. ESIMS: calcd [M + Na]⁺ C₅₄H₅₈O₁₀Na 889.39221; found 889.38737.

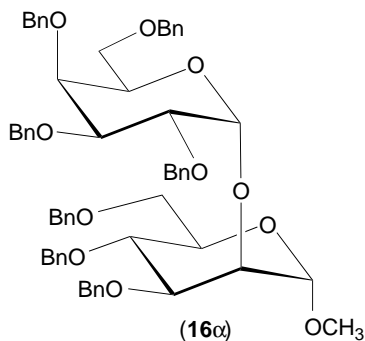


¹H NMR (400 MHz, CDCl₃) δ_H 7.29–7.10 (m, 30 H), 5.25 (d, 1 H, *J* = 3.6 Hz), 4.87 (d, 1 H, *J* = 1.7 Hz), 4.81 (d, 1 H, *J* = 10.8 Hz), 4.76–4.72 (m, 2 H), 4.66–4.11 (m, 14 H), 3.89–3.55 (m, 6 H), 3.34 (s, 3 H); ¹³C NMR (100.623 MHz, CDCl₃) δ_C 139.18, 138.84, 138.75, 138.67,

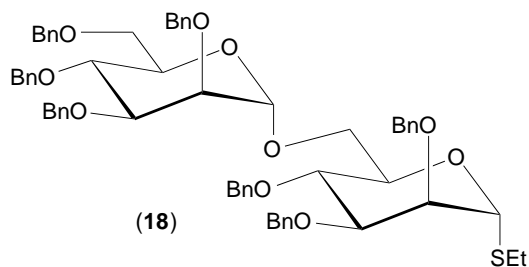
137.81, 128.74, 128.70, 128.35, 128.28, 128.20, 128.18, 128.16, 128.11, 128.02, 127.96, 127.93, 127.90, 127.87, 127.77, 121.53, 99.20, 98.16, 84.55, 84.33, 80.97, 79.22, 76.61, 75.39, 75.26, 73.64, 73.30, 73.01, 72.63, 72.28, 72.20, 71.93, 69.94, 55.38. ESIMS: calcd $[M + Na]^+ C_{54}H_{58}O_{10}Na$ 889.39221; found 889.40110.



1H NMR (400 MHz, $CDCl_3$) δ_H 7.31–7.14 (m, 35 H), 5.19 (d, 1 H, $J = 1.6$ Hz), 4.85–4.47 (m, 15 H), 3.92–3.71 (m, 12 H), 3.24 (s, 3 H); ^{13}C NMR (100.623 MHz, $CDCl_3$) δ_C 139.07, 138.88, 128.92, 128.84, 128.74, 128.65, 128.36, 128.34, 128.25, 128.19, 128.11, 127.97, 127.94, 127.91, 127.86, 100.38, 100.03, 80.50, 80.21, 75.57, 75.51, 75.46, 75.40, 75.33, 74.77, 73.83, 73.78, 72.88, 72.70, 72.66, 72.64, 72.23, 70.04, 69.85, 55.15. ESIMS: calcd $[M + Na]^+ C_{62}H_{66}O_{11}Na$ 1009.44973; found 1009.44014.

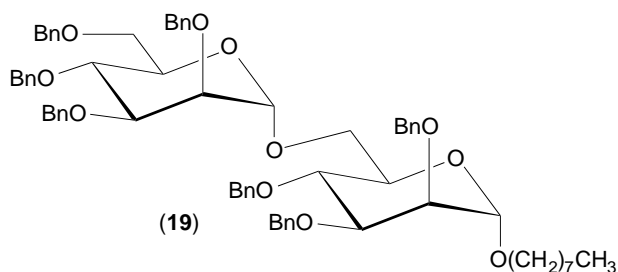


1H NMR (400 MHz, $CDCl_3$) δ_H 7.33–7.13 (m 35 H), 5.46 (d, 1 H, $J = 3.6$ Hz), 4.92 (d, 1 H, $J = 11.5$ Hz), 4.81–4.78 (m, 3 H), 4.69–4.29 (m, 11 H), 4.16–4.15 (m, 1 H), 4.07–3.89 (m, 6 H), 3.75–3.70 (m, 3 H), 3.58–3.48 (m, 2 H), 3.25 (s, 3 H); ^{13}C NMR (100.623 MHz, $CDCl_3$) δ_C 138.66, 128.86, 128.80, 128.78, 128.73, 128.69, 128.67, 128.52, 128.41, 128.20, 128.12, 128.06, 128.03, 127.86, 127.83, 127.57, 100.57, 98.44, 80.92, 78.94, 76.92, 75.93, 75.53, 75.31, 75.17, 73.83, 73.78, 73.74, 73.74, 73.37, 73.32, 72.55, 71.64, 70.51, 70.19, 69.91, 55.12. ESIMS: calcd $[M + Na]^+ C_{62}H_{66}O_{11}Na$ 1009.44973; found 1009.45106.

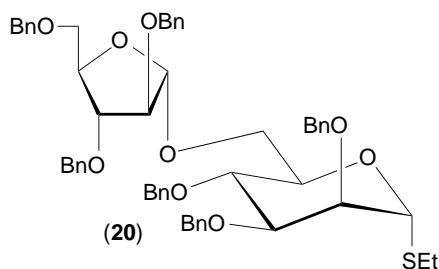


1H NMR (400 MHz, $CDCl_3$) δ_H 7.37–7.13 (m, 35 H), 5.30 (s, 1 H), 5.10 (s, 1 H), 4.90–4.86 (m, 2 H), 4.67–4.41 (m, 12 H), 4.06–3.57 (m, 12 H), 2.60–2.43 (m, 2 H), 1.20–1.17 (m, 3 H);

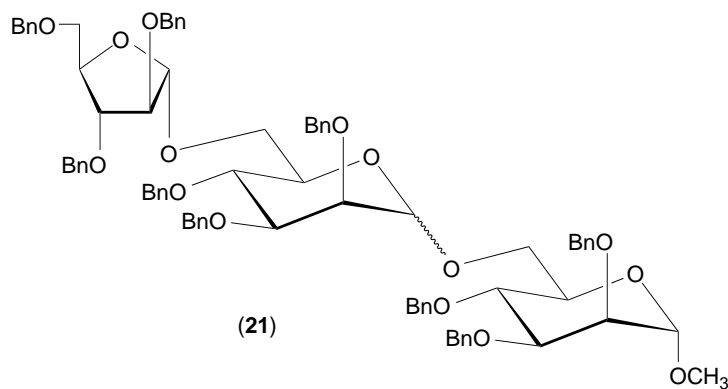
^{13}C NMR (100.623 MHz, CDCl_3) δ_{C} 128.40, 128.35, 128.32, 128.24, 128.20, 127.90, 127.84, 127.76, 127.70, 127.57, 127.53, 127.49, 127.38, 98.01, 81.90, 79.59, 76.55, 75.31, 75.04, 74.98, 74.91, 74.83, 74.71, 73.28, 73.51, 72.41, 72.25, 72.08, 71.84, 71.58, 69.20, 66.10, 25.30, 15.03. ESIMS: calcd $[\text{M} + \text{Na}]^+ \text{C}_{63}\text{H}_{68}\text{O}_{10}\text{SNa}$ 1039.44310; found 1039.45059.



^1H NMR (400 MHz, CDCl_3) δ_{H} 7.37–7.15 (m, 35 H), 5.15 (s, 1 H), 4.89–4.45 (m, 15 H), 4.03–3.53 (m, 13 H), 3.33–3.28 (m, 1 H), 1.48–0.85 (m, 15 H); ^{13}C NMR (100.623 MHz, CDCl_3) δ_{C} 138.83, 138.62, 138.50, 128.36, 128.21, 127.88, 127.81, 127.75, 127.68, 127.55, 127.33, 98.22, 97.88, 80.49, 79.56, 77.23, 76.52, 75.26, 75.12, 75.09, 74.99, 74.73, 73.31, 72.88, 72.43, 72.23, 71.96, 71.73, 71.57, 69.34, 67.66, 31.86, 31.61, 29.72, 29.43, 29.26, 26.22, 22.68, 14.11. ESIMS: calcd $[\text{M} + \text{Na}]^+ \text{C}_{69}\text{H}_{80}\text{O}_{11}\text{Na}$ 1107.55981; found 1107.54000.

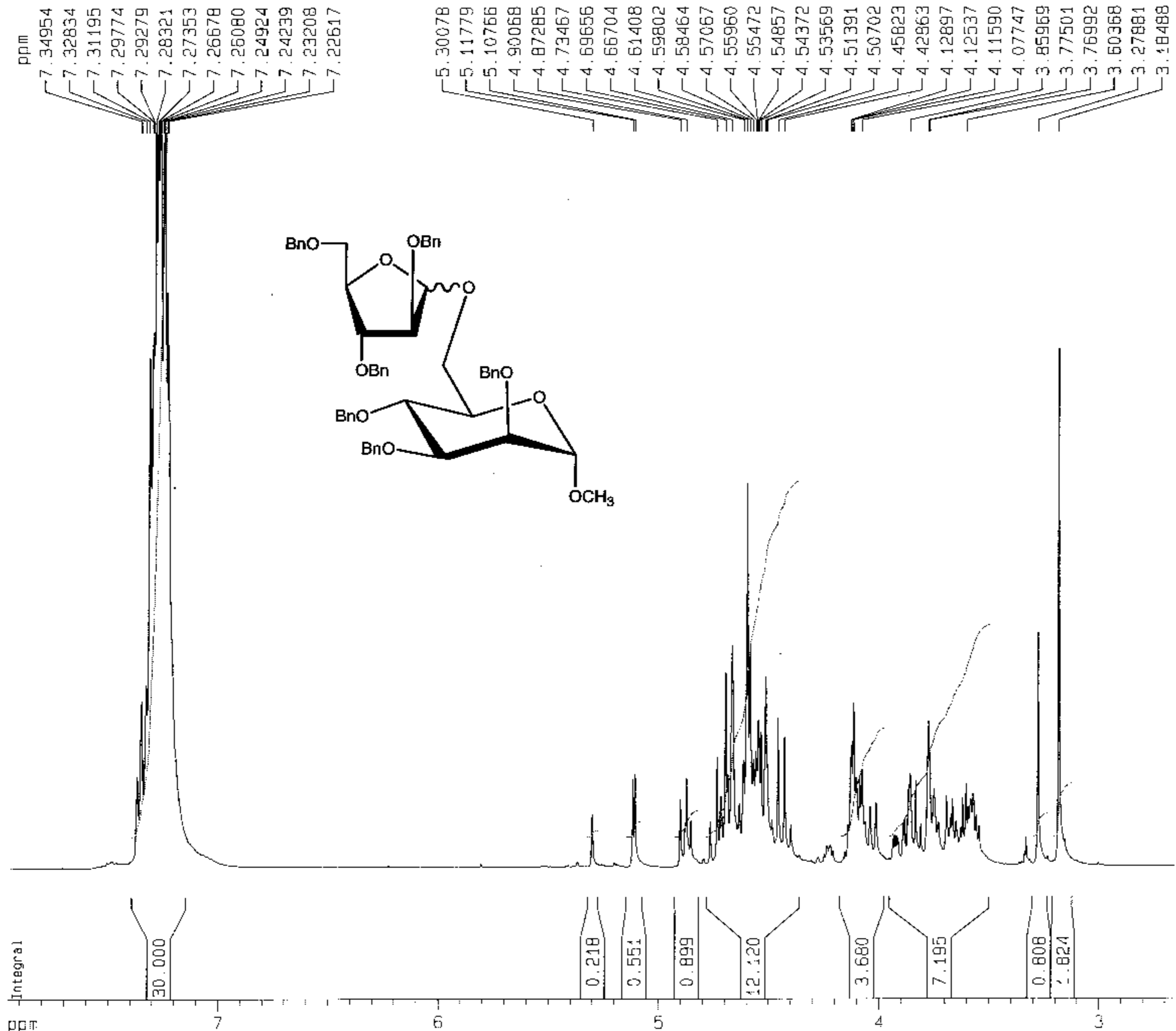


^1H NMR (400 MHz, CDCl_3) δ_{H} 7.33–7.19 (m, 30 H), 5.34 (s, 1 H), 5.27 (s, 1 H), 4.85–4.40 (m, 11 H), 4.13–3.57 (m, 12 H), 2.61–2.48 (m, 2 H), 1.20 (t, 3 H, $J = 7.4$ Hz); ^{13}C NMR (100.623 MHz, CDCl_3) δ_{C} 139.22, 138.45, 138.29, 128.90, 128.84, 128.80, 128.54, 128.44, 128.42, 128.30, 128.23, 128.14, 128.08, 128.02, 107.10, 88.49, 84.13, 82.65, 81.26, 80.92, 77.10, 75.61, 75.40, 73.90, 73.80, 72.66, 72.60, 72.55, 72.26, 70.20, 66.43, 25.79, 15.48. ESIMS: calcd $[\text{M} + \text{Na}]^+ \text{C}_{55}\text{H}_{60}\text{O}_9\text{SNa}$ 919.38555; found 919.39438.



^1H NMR (400 MHz, CDCl_3) δ_{H} 7.41–7.20 (m, 45 H), 5.29 (s, 1 H), 5.08 (s, 1 H), 4.87–4.38 (m, 19 H), 4.23–3.57 (m, 17 H), 3.22–3.21 (m, 3 H); ^{13}C NMR (100.623 MHz, CDCl_3) δ_{C} 128.84, 128.76, 128.70, 128.66, 128.58, 128.52, 128.37, 128.34, 128.29, 128.20, 128.13,

128.10, 128.04, 127.97, 107.54, 107.20, 102.96, 99.423, 99.34, 98.81, 88.36, 88.22, 84.36, 84.14, 81.18, 80.79, 75.51, 75.22, 75.08, 74.98, 73.76, 73.29, 73.07, 72.59, 72.48, 72.42, 72.19, 72.09, 71.96, 71.88, 70.23, 66.57, 55.14, 55.06. ESIMS: calcd $[M + Na]^+$ $C_{81}H_{86}O_{15}Na$ 1321.58641; found 1321.59404.



Current Data Parameters
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 PROCNO 1

F2 - Acquisition Parameters
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 TD 65536
 SOLVENT CDCl3
 NS 25
 DS 0
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 57
 DW 50.400 usec.
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec

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 P1 7.50 usec
 PL1 -6.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
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 SF 400.1300288 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
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 F1 3182.26 Hz
 F2P 2.655 ppm
 F2 1062.47 Hz
 PPMCM 0.27168 ppm/cm
 HZCM 108.70717 Hz/cm

Integral

30.000

0.218

0.551

0.899

12.120

3.680

7.195

0.808

1.824

ppm

GXC-III-61-C13

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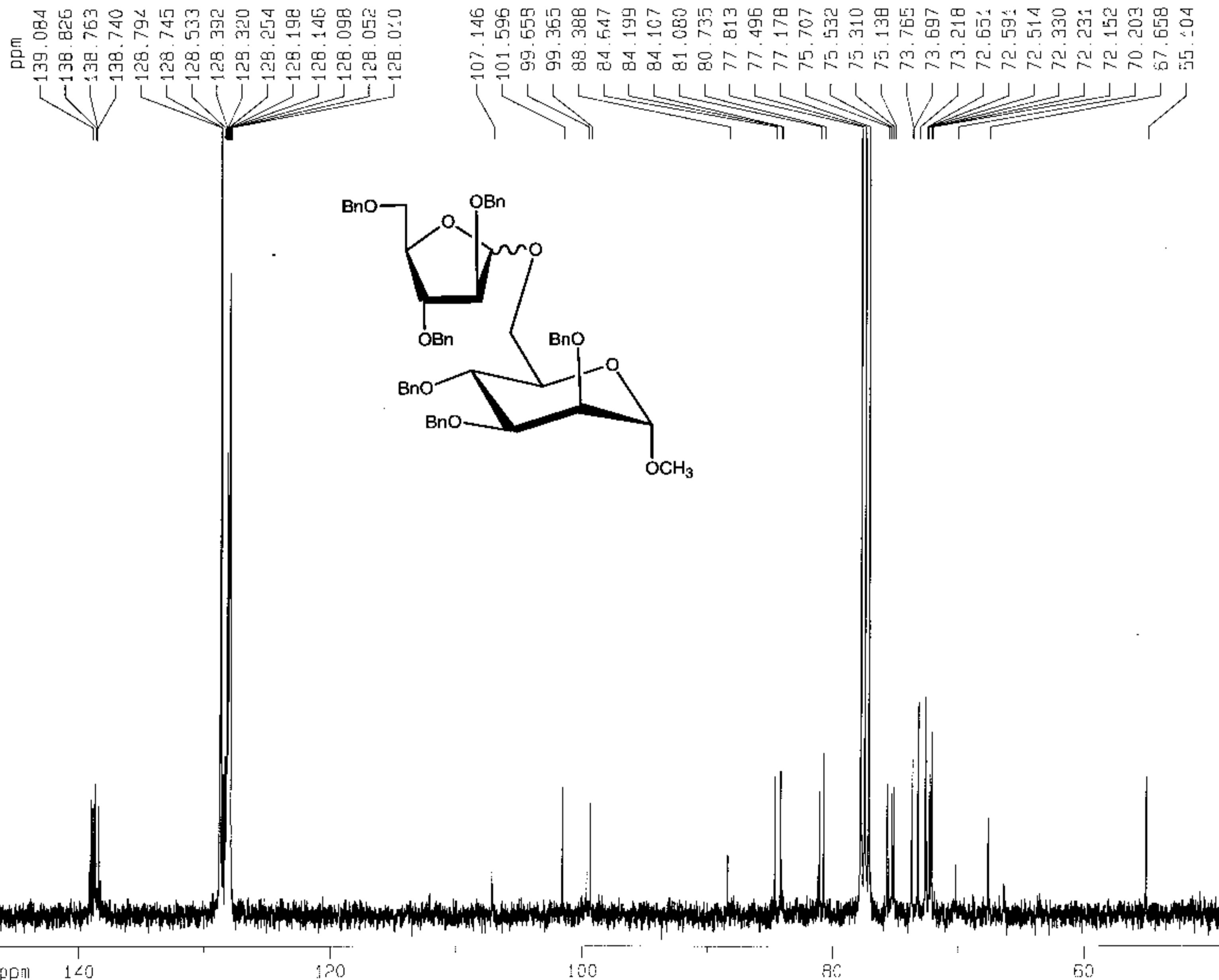
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 FIDRES 0.383387 Hz
 AQ 1.3042164 sec
 RG 8192
 JW 19.900 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 d12 0.00002000 sec

----- CHANNEL f1 -----
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 P1 5.90 usec
 PL1 -6.00 dB
 SF01 100.6237959 MHz

----- CHANNEL f2 -----
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 PCPD2 80.00 usec
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 PL12 15.80 dB
 PL13 15.80 dB
 SF02 400.1316005 MHz

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 SSB 0
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 GB 0
 PC 1.40

1D NMR plot parameters
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 F1 14727.60 Hz
 F2P 48.528 ppm
 F2 4882.57 Hz
 FPMCK 5.01799 ppm/cm
 HZCM 504.67344 Hz/cm



GXC-IV-16-B-H1

Current Data Parameters

NAME gxciv16b
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

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 PULPROG zg30
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 SOLVENT CDC13
 NS 80
 DS 0
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
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 TE 300.0 K
 D1 2.0000000 sec

===== CHANNEL f1 =====

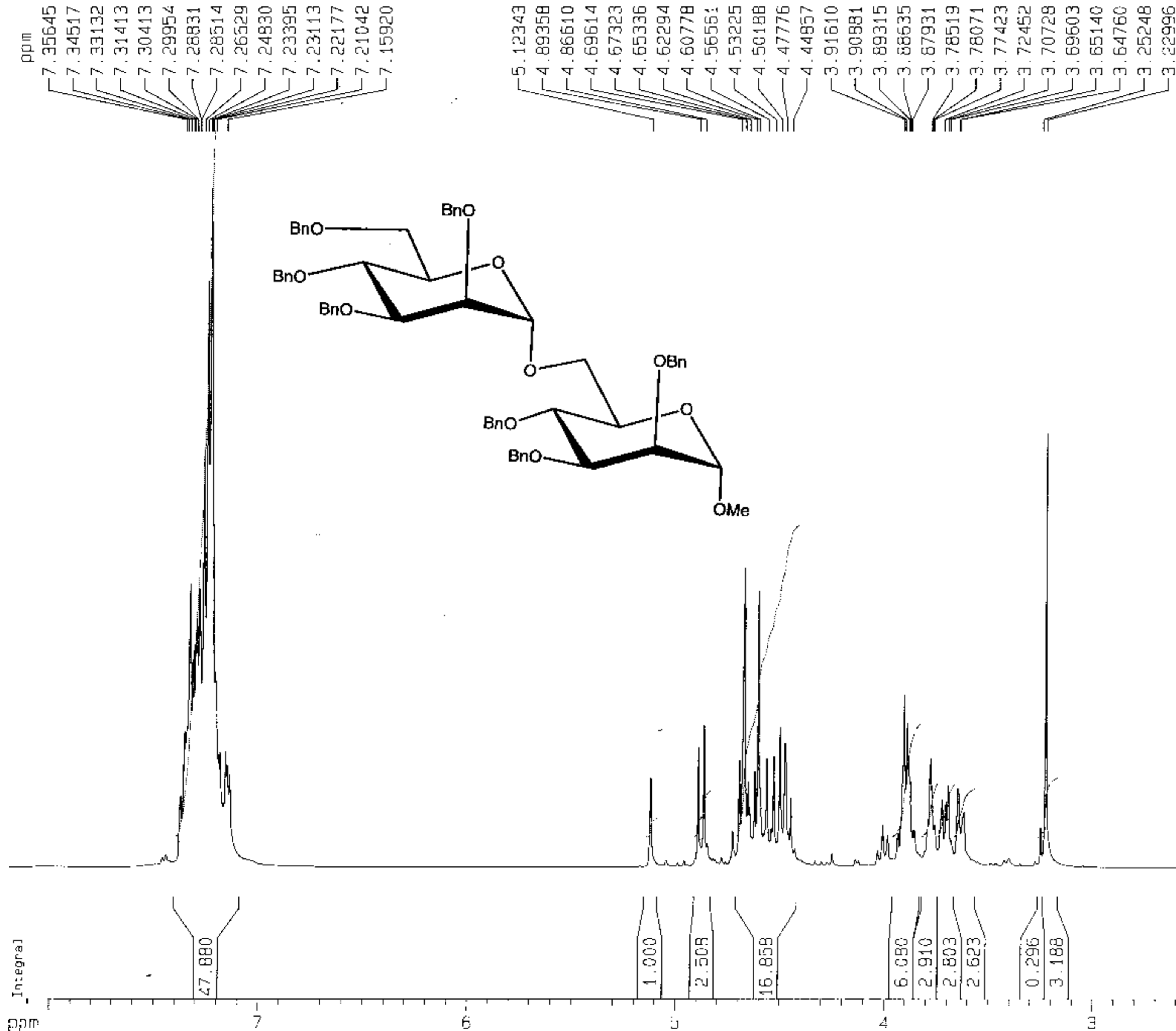
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F2 - Processing parameters

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 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 19.50 cm
 F1P B.194 ppm
 F1 3278.50 Hz
 F2P 2.599 ppm
 F2 1039.93 Hz
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 HZCM 114.79854 Hz/cm



GXC-IV-16-B-C13

Current Data Parameters
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 PROCNO 1

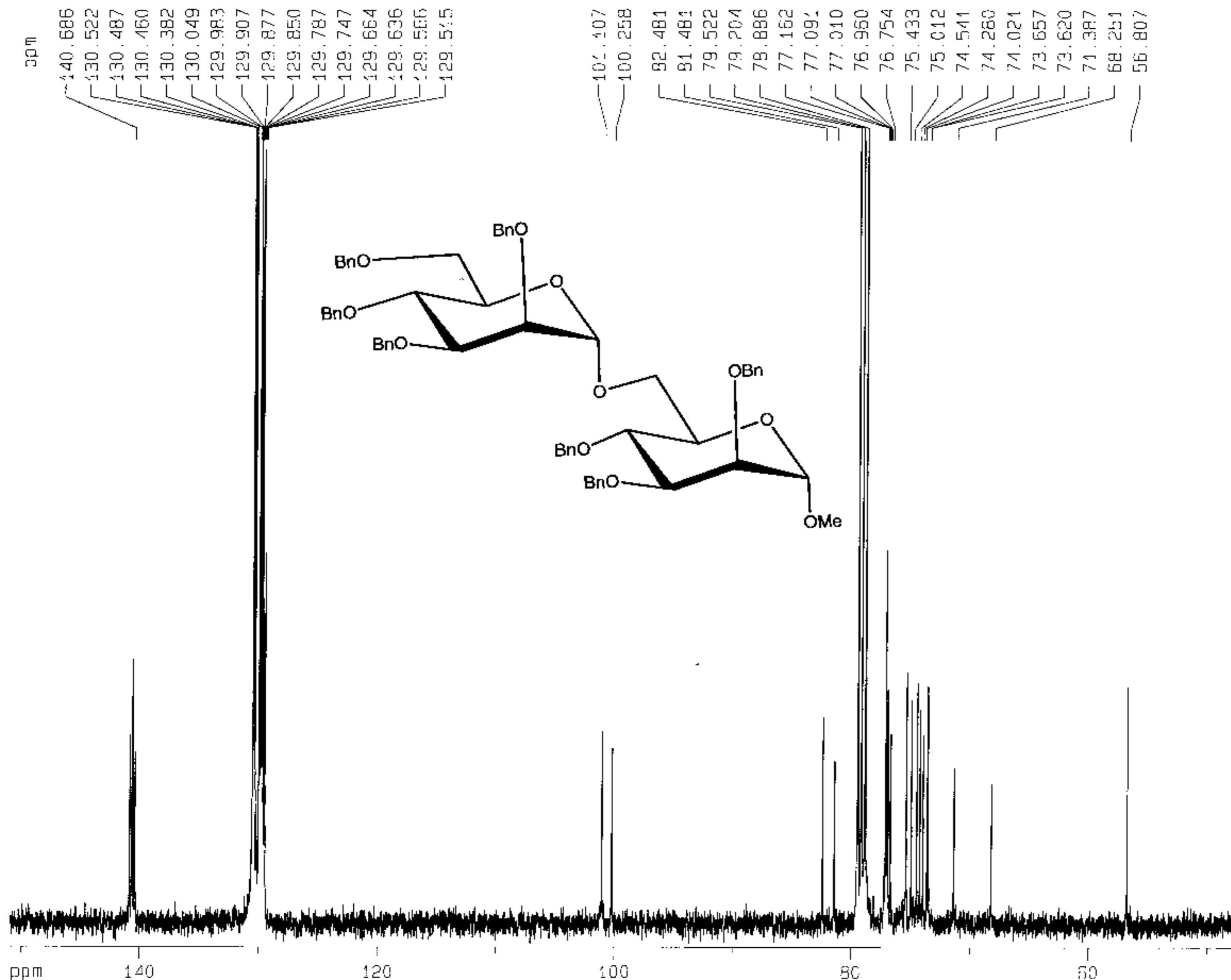
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 NS 408
 DS 4
 SWH 25125.629 Hz
 FIDRES 0.363387 Hz
 AQ 1.3042164 sec
 RG 8192
 DW 19.900 usec
 DE 6.00 usec
 TC 300.0 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 d12 0.00002000 sec

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 P1 5.90 usec
 PL1 -6.00 dB
 SF01 100.6237959 MHz

----- CHANNEL f2 -----
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 NUC2 1H
 PCPD2 80.00 usec
 PL2 -6.00 dB
 PL12 15.80 dB
 PL13 15.80 dB
 SF02 400.1316005 MHz

F2 - Processing parameters
 SI 32/68
 SF 100.6125573 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 19.50 cm
 F1P 150.842 ppm
 F1 15176.61 Hz
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 F2 4749.19 Hz
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 HZCM 534.73914 Hz/cm



GXC-IV-06-A-H1

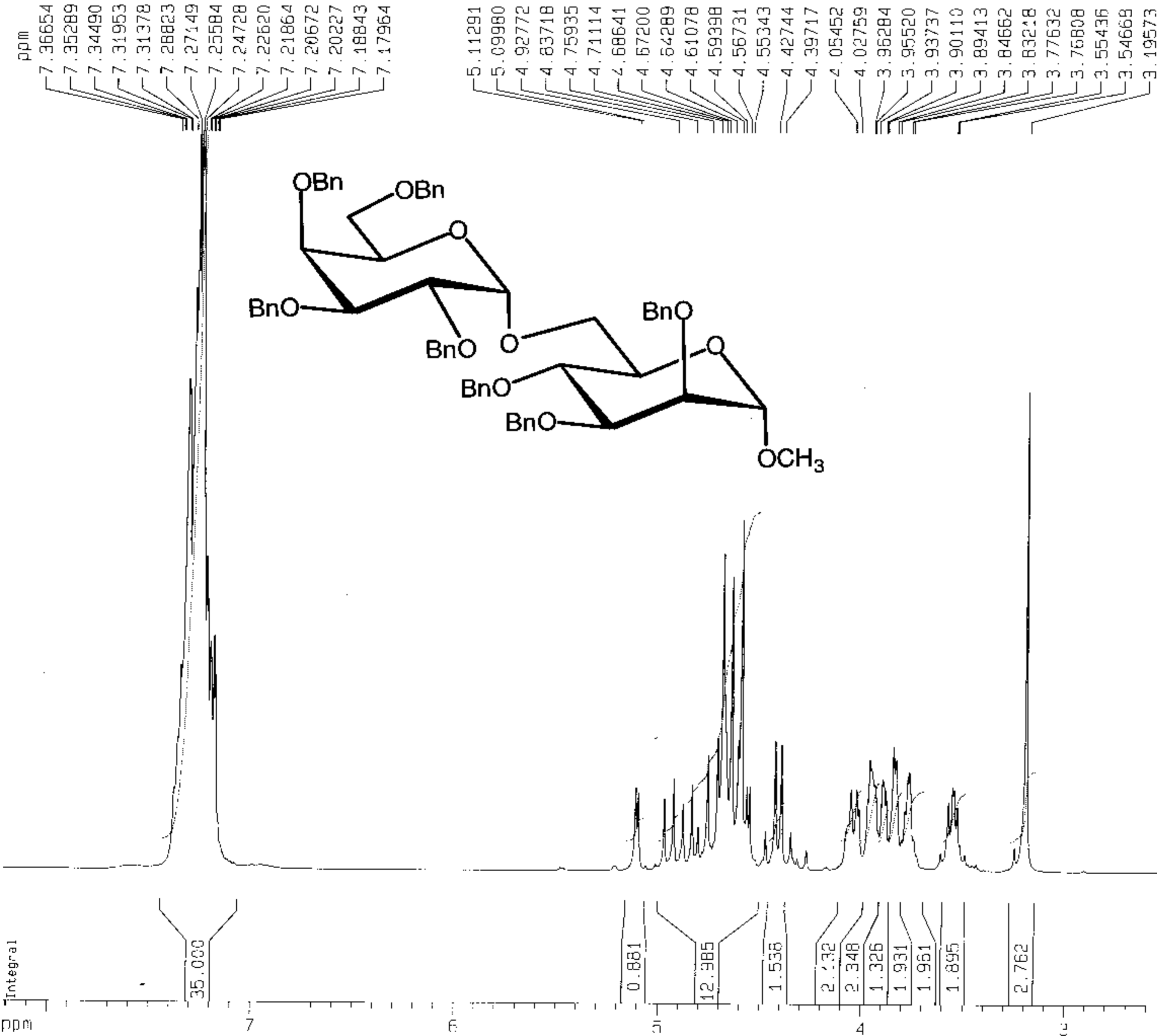
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 PULPROG zg30
 TD 16384
 SOLVENT CDCl3
 NS 37
 DS 0
 SWH 5175.983 Hz
 FIDRES 0.315917 Hz
 AQ 1.5827444 sec
 RG 71.8
 DW 96.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec

===== CHANNEL f1 =====
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 P1 7.20 usec
 PL1 -4.00 dB
 SF01 250.1315447 MHz

F2 - Processing parameters
 SI 32768
 SF 250.1300266 MHz
 WDW EM
 SSR 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 19.50 cm
 F1P 8.216 ppm
 F1 2055.10 Hz
 F2P 2.460 ppm
 F2 615.42 Hz
 PPMCM 0.29516 ppm/cm
 HZCM 73.82961 Hz/cm



GXC-IV-06-A-C13

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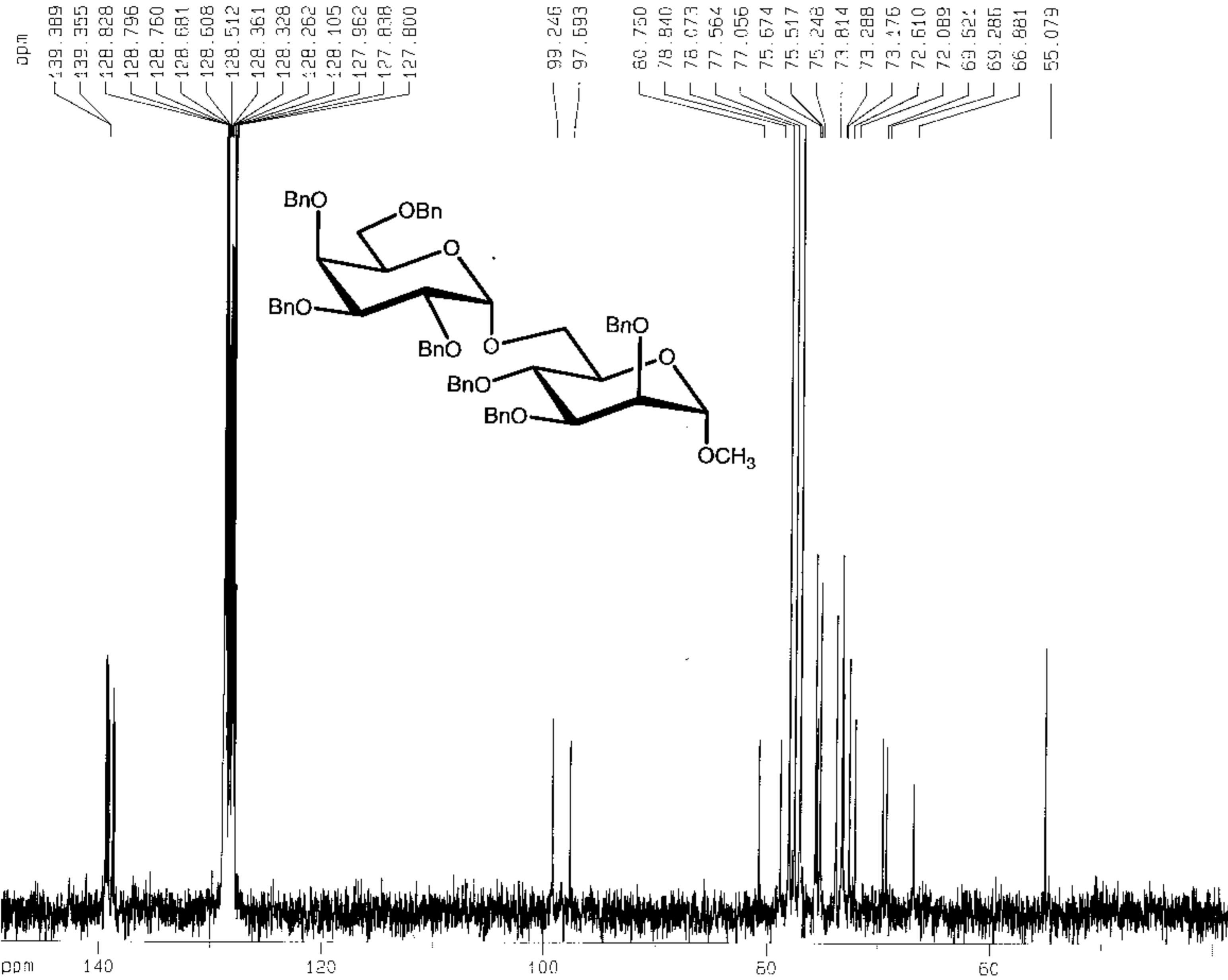
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 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 69
 DS 4
 SWH 15723.271 Hz
 FIDRES 0.239918 Hz
 AQ 2.0840948 sec
 RG 11585.2
 DW 31.800 usec
 DE 30.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

----- CHANNEL f1 -----
 NUC1 13C
 P1 8.00 usec
 PL1 0.00 dB
 SF01 62.8952140 MHz

----- CHANNEL f2 -----
 CPDPRG2 wa1t716
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -4.00 dB
 PL12 16.00 dB
 PL13 17.00 dB
 SF02 250.1310005 MHz

F2 - Processing parameters
 SI 32768
 SF 62.8952140 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 19.50 cm
 F1P 148.622 ppm
 F1 9347.63 Hz
 F2P 38.527 ppm
 F2 2423.14 Hz
 PPMCM 5.64593 ppm/cm
 HZCM 355.10190 Hz/cm



GXC-IV-10-B-H1

Current Data Parameters

NAME gxciv10b
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 991207
 Time 11.37
 INSTRUM spect
 PROBHD 5 mm BBO BB-1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 30
 DS 0
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 32
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec

==== CHANNFI f1 =====

NUC1 1H
 P1 7.50 usec
 PL1 -6.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters

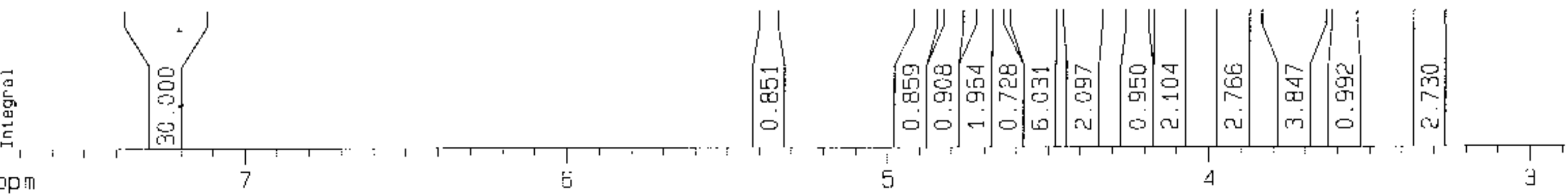
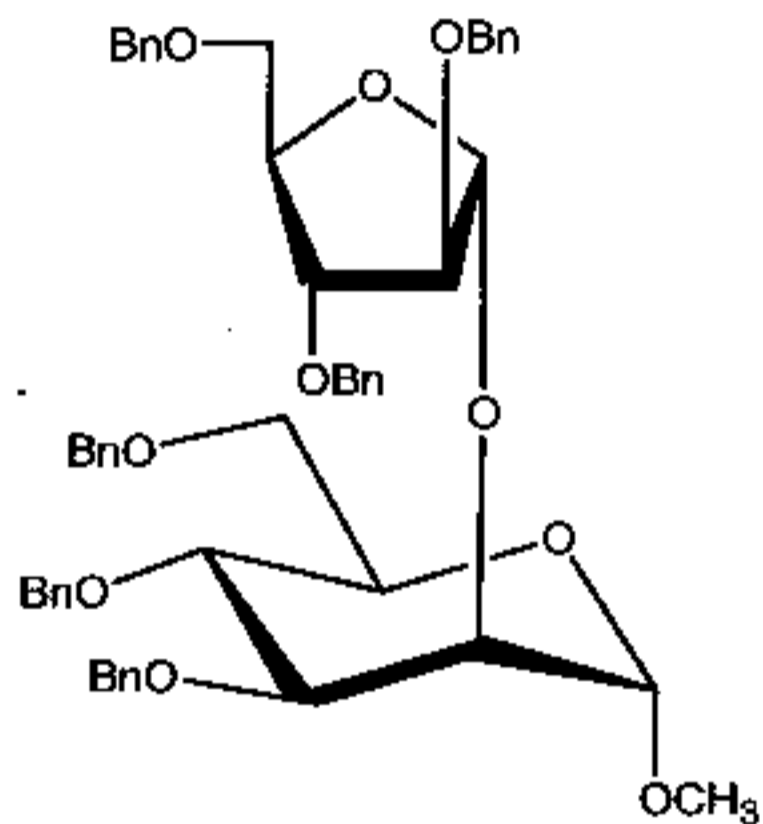
SF 400.1300437 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 19.50 cm
 F1P 7.779 ppm
 F1 3112.51 Hz
 F2P 2.892 ppm
 F2 1157.19 Hz
 PPMCM 0.25060 ppm/cm
 HZCM 100.27300 Hz/cm

7.31123
 7.30002
 7.27206
 7.25952
 7.25194
 7.24541
 7.23639
 7.22414
 7.21840
 7.20856
 7.19389
 7.18818

5.37634
 5.37298
 4.89618
 4.86920
 4.80300
 4.79903
 4.70183
 4.68887
 4.65840
 4.57595
 4.56948
 4.56430
 4.54786
 4.53935
 4.53046
 4.50996
 4.49984
 4.42959
 4.39972
 4.39363
 4.36405
 4.14136
 4.13317
 4.12895
 3.93581
 3.92340
 3.77128
 3.74143
 3.33005



GXC-IV-10-B-C13

Current Data Parameters
 NAME gxciv10b
 EXPNO 2
 PROCNO 1

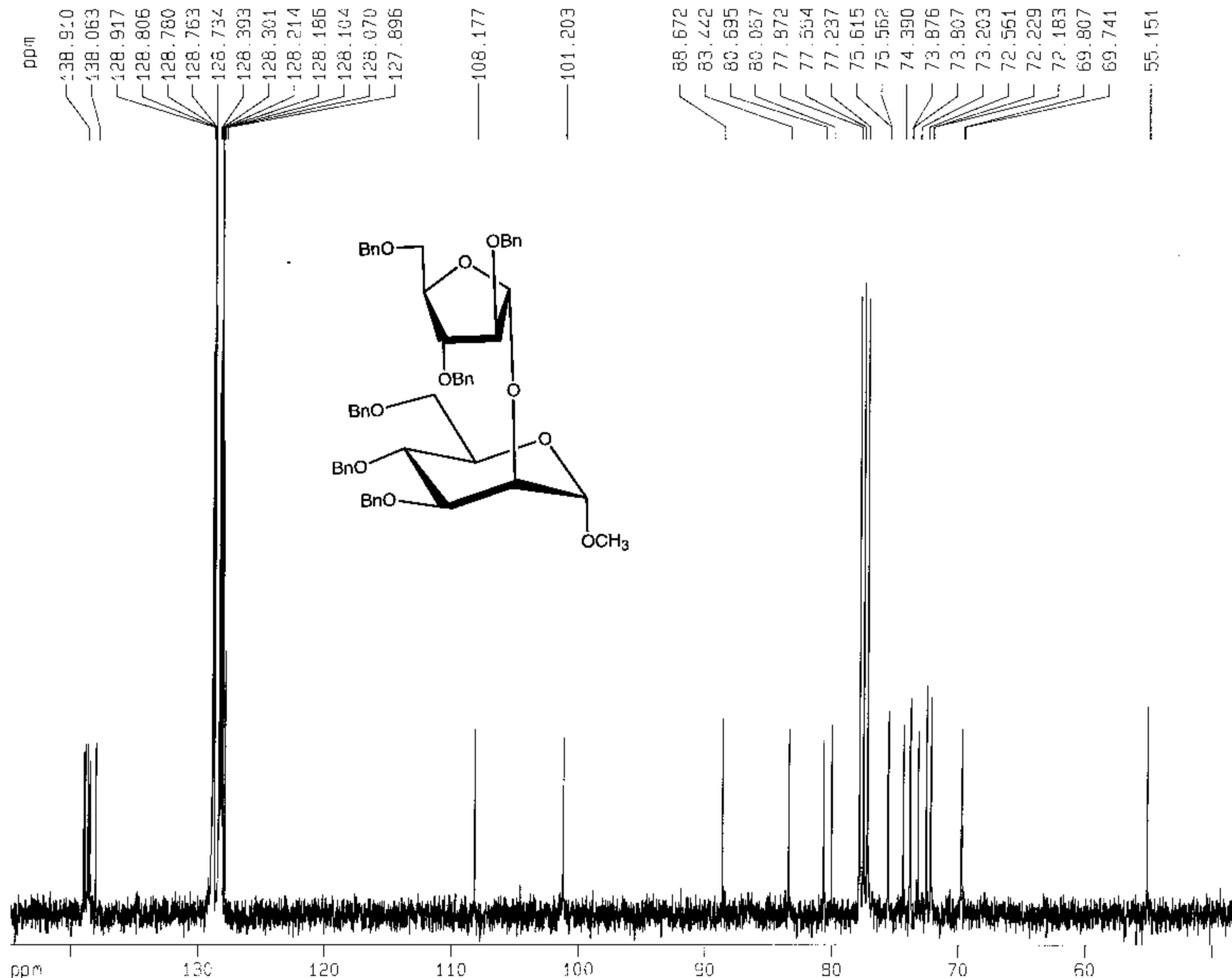
F2 - Acquisition Parameters
 Date_ 991207
 Time 11.48
 INSTRUM spect
 PROBHD 5 mm BBO BB-1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 41
 DS 4
 SWH 25125.629 Hz
 FIDRES 0.383367 Hz
 AQ 1.3042164 sec
 RG 8192
 DW 19.900 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 5.90 usec
 PL1 6.00 dB
 SF01 100.6237959 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPUP 80.00 usec
 PL2 -6.00 dB
 PL12 15.80 dB
 PL13 15.80 dB
 SF02 400.1315005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127290 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 19.50 cm
 F1P 144.725 ppm
 F1 14561.20 Hz
 F2P 47.977 ppm
 F2 4827.10 Hz
 PPMCM 4.96145 ppm/cm
 HZCM 499.18463 Hz/cm



GXC-IV-10-C-H1

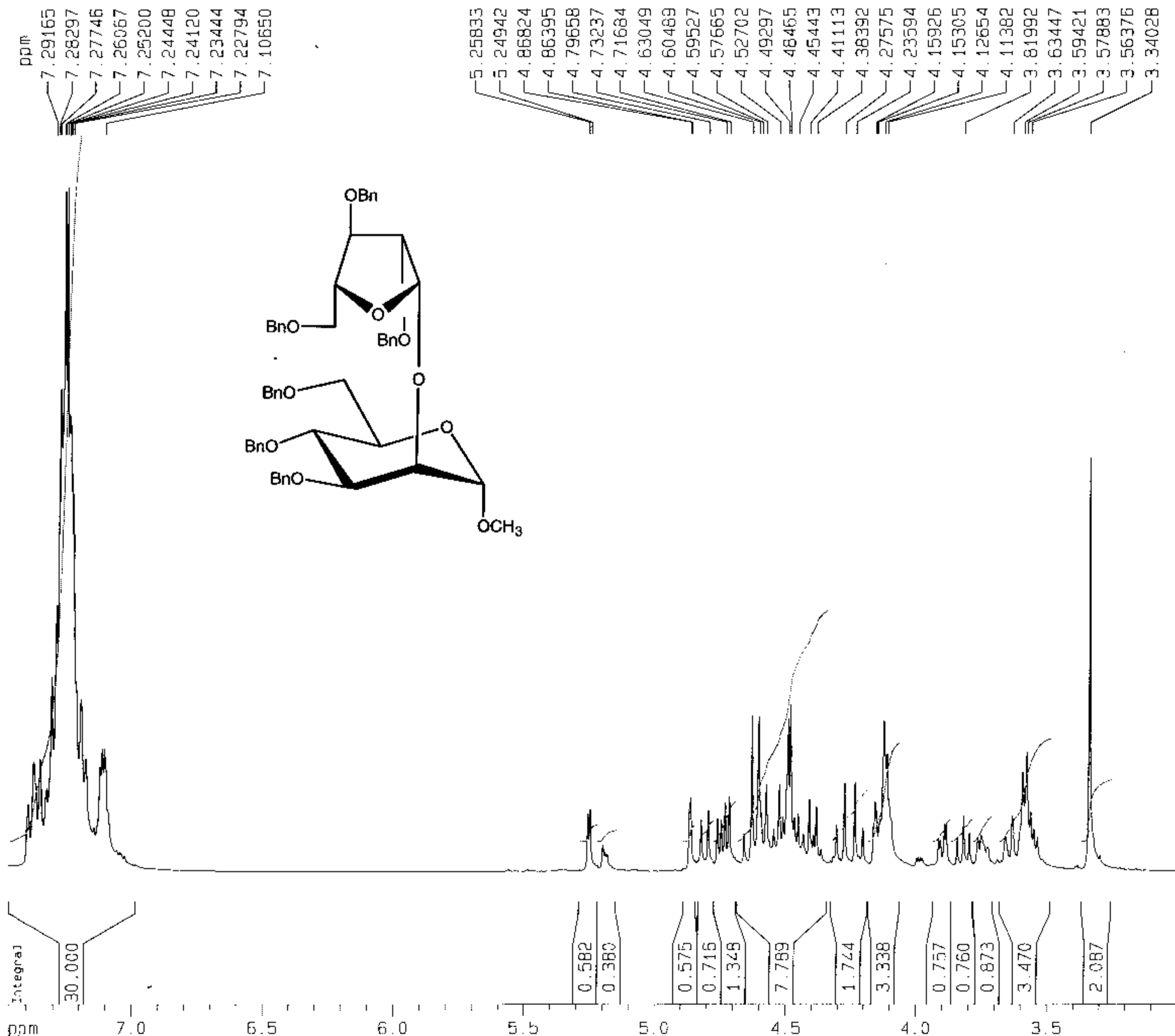
Current Data Parameters
 NAME gxciv10c
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 991207
 Time 18.16
 INSTRUM spect
 PROBHD 5 mm BBO BB-1
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 33
 DS 0
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 161.3
 DW 60.400 usec
 DE 6.00 usec
 IE 300.0 K
 D1 2.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.50 usec
 PL1 -6.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300207 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 19.50 cm
 F1P 7.471 ppm
 F1 2989.37 Hz
 F2P 2.995 ppm
 F2 1198.52 Hz
 PPMCM 0.22952 ppm/cm
 HZCM 91.83883 Hz/cm



GXC-IV-10-C-C13

Current Data Parameters
 NAME gxciv10c
 EXPNO 2
 PROCNO 1

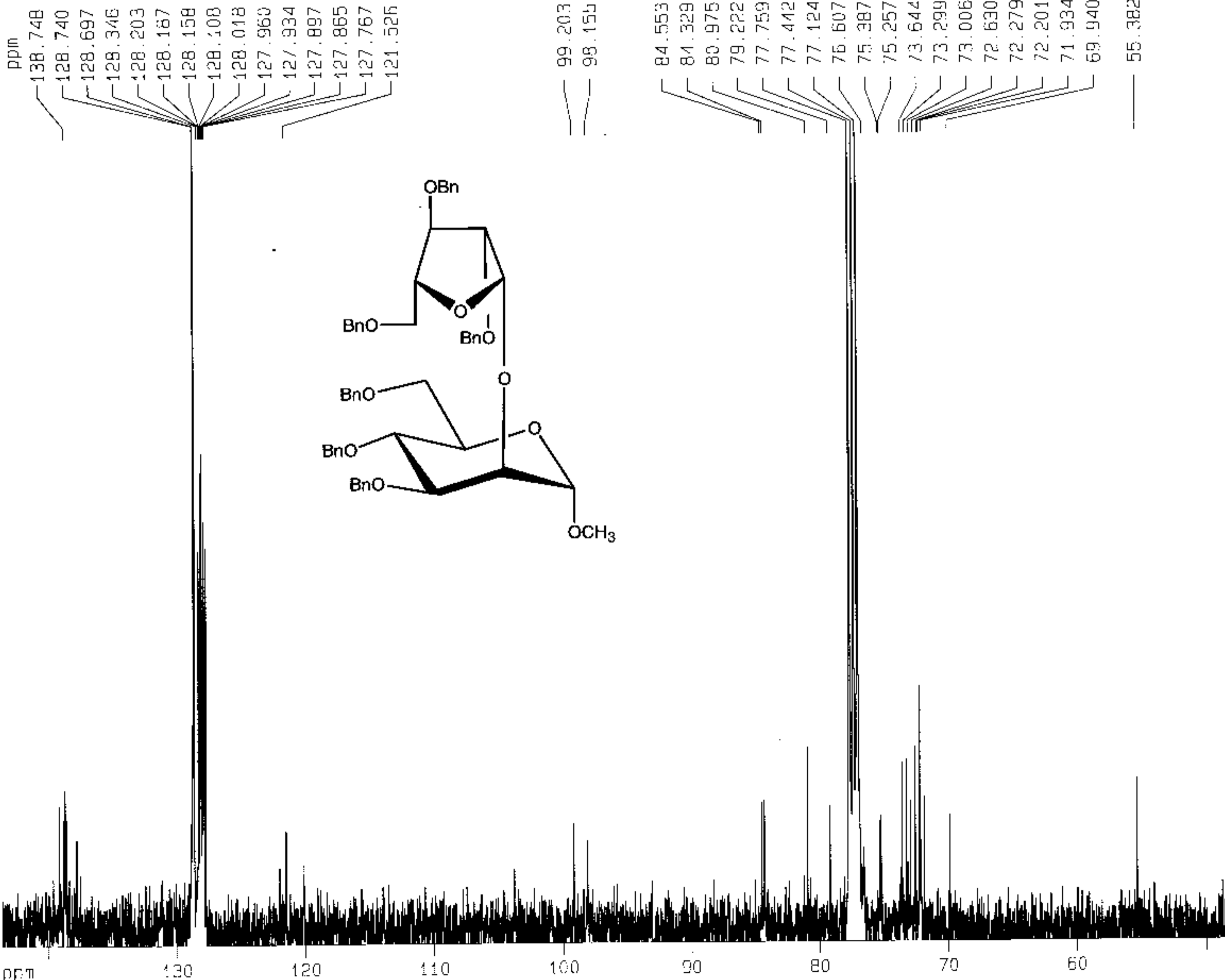
F2 - Acquisition Parameters
 Date_ 991207
 Time 18.55
 INSTRUM spect
 PROBHD 5 mm BBO BB-1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 649
 DS 4
 SWH 25125.629 Hz
 FIDRES 0.393387 Hz
 AQ 1.3042164 sec
 RG 2048
 JW 19.900 usec
 JE 6.00 usec
 FE 300.0 K
 SI 2.0000000 sec
 d11 0.0300000 sec
 d12 0.00002000 sec

----- CHANNEL f1 -----
 NUC1 13C
 P1 5.90 usec
 PL1 -6.00 dB
 SFO1 100.6237959 MHz

----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -6.00 dB
 PL12 15.80 dB
 PL13 15.80 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127290 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 19.50 cm
 F1P 143.623 ppm
 F1 14450.27 Hz
 F2P 47.977 ppm
 F2 4827.10 Hz
 PPMCM 4.90491 ppm/cm
 HZCM 493.49597 Hz/cm



GXC-IV-11-B-H1

Current Data Parameters

NAME gxciv11b
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 991206
 Time 12.09
 INSTRUM spect
 PROBHD 5 mm BBO BB-1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 32
 DS 0
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 35.9
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec

===== CHANNEL f1 =====

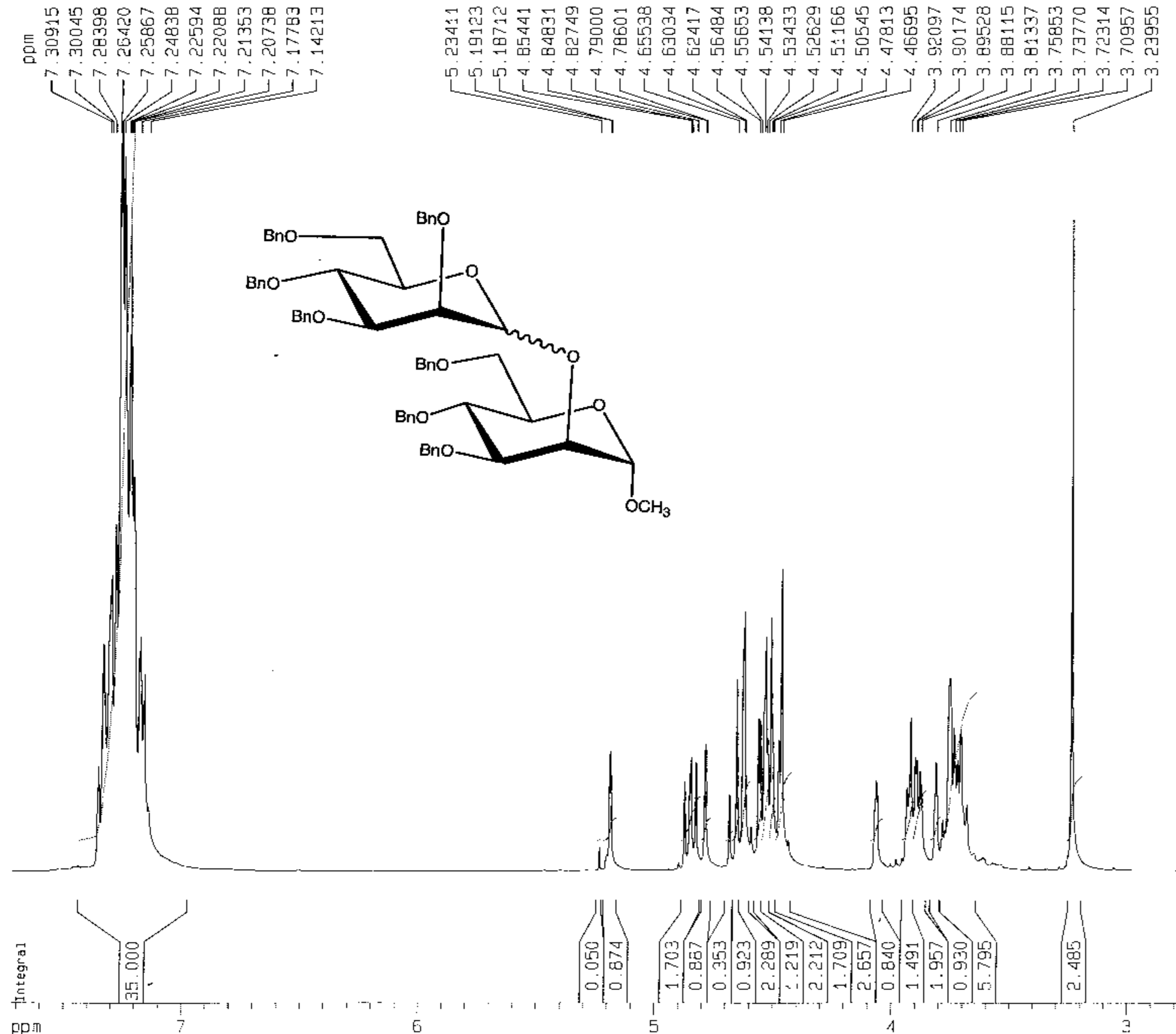
NUC1 1H
 P1 7.50 usec
 PL1 -6.00 dB
 SF01 400.1324710 MHz

F2 Processing parameters

SI 32768
 SF 400.1300406 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 19.50 cm
 -1P 7.718 ppm
 F1 3088.21 Hz
 F2P 2.763 ppm
 F2 1105.47 Hz
 PPMCM 0.25411 ppm/cm
 HZCM 101.67870 Hz/cm



GXC-IV-11-B-C13

Current Data Parameters

NAME gxciv11b
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

Date_ 991206
 Time 12.22
 INSTRUM spect
 PROBHD 5 mm BBO BB-1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 209
 DS 4
 SWH 25125.629 Hz
 FIDRES 0.383387 Hz
 AQ 1.3042154 sec
 RG 16384
 JW 19.900 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

----- CHANNEL f1 -----

NUC1 13C
 P1 5.90 usec
 PL1 -6.00 dB
 SFG1 100.6237959 MHz

----- CHANNEL f2 -----

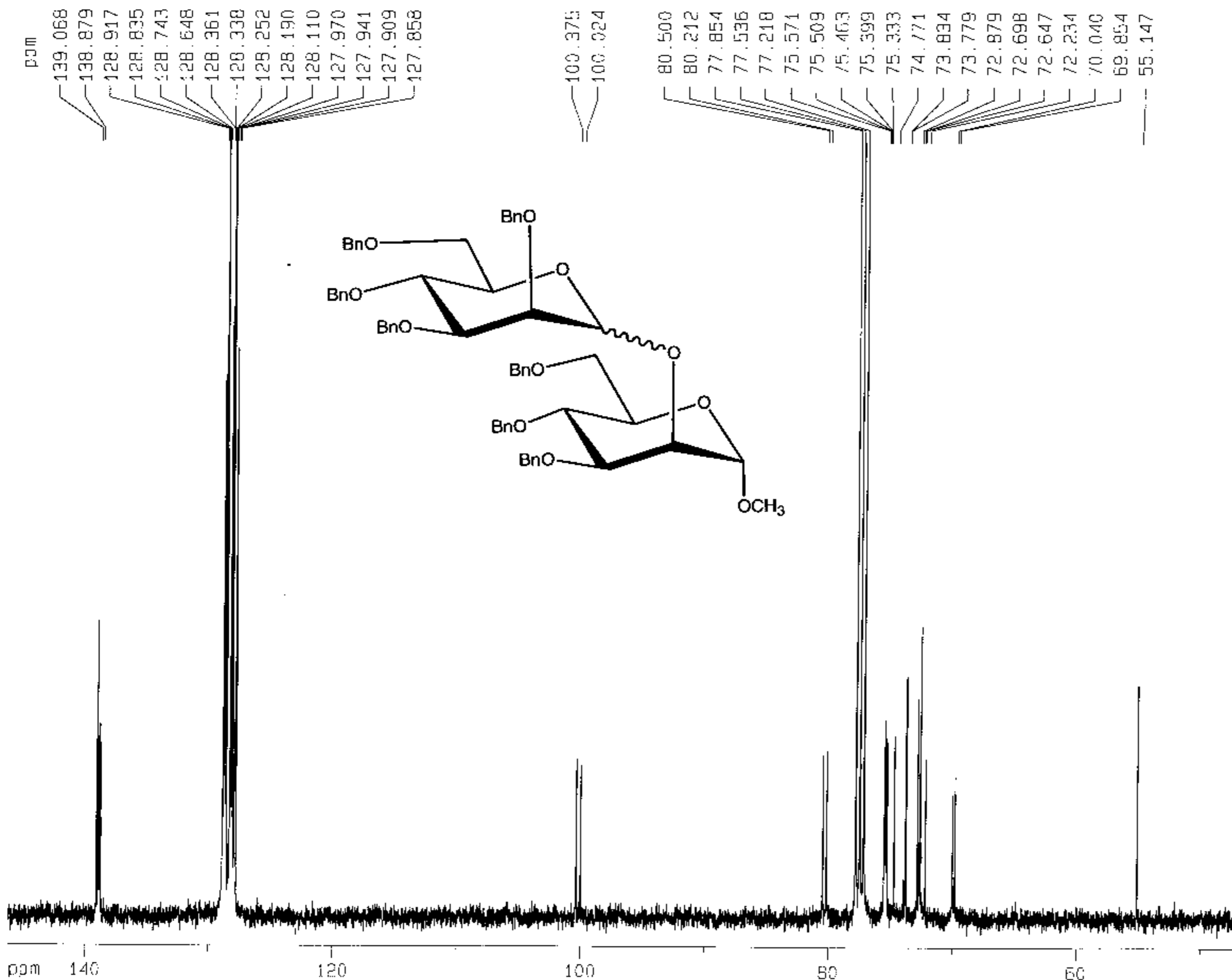
PCPD2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -6.00 dB
 PL12 15.80 dB
 PL13 15.80 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters

SI 32768
 SF 100.6127290 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

F2 NMR plot parameters

CX 19.50 cm
 F1P 146.103 ppm
 F1 14699.87 Hz
 F2P 47.150 ppm
 F2 4743.90 Hz
 PPMCM 5.07453 ppm/cm
 HZCM 510.56213 Hz/cm



GXC-IV-12-A-H1

Current Data Parameters

NAME gxciv12a
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date.. 991208
 Time 9.57
 INSTRUM spect
 PROBHD 5 mm BBO BB-1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 45.3
 JW 60.400 usec
 DF 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec

==== CHANNEL f1 =====

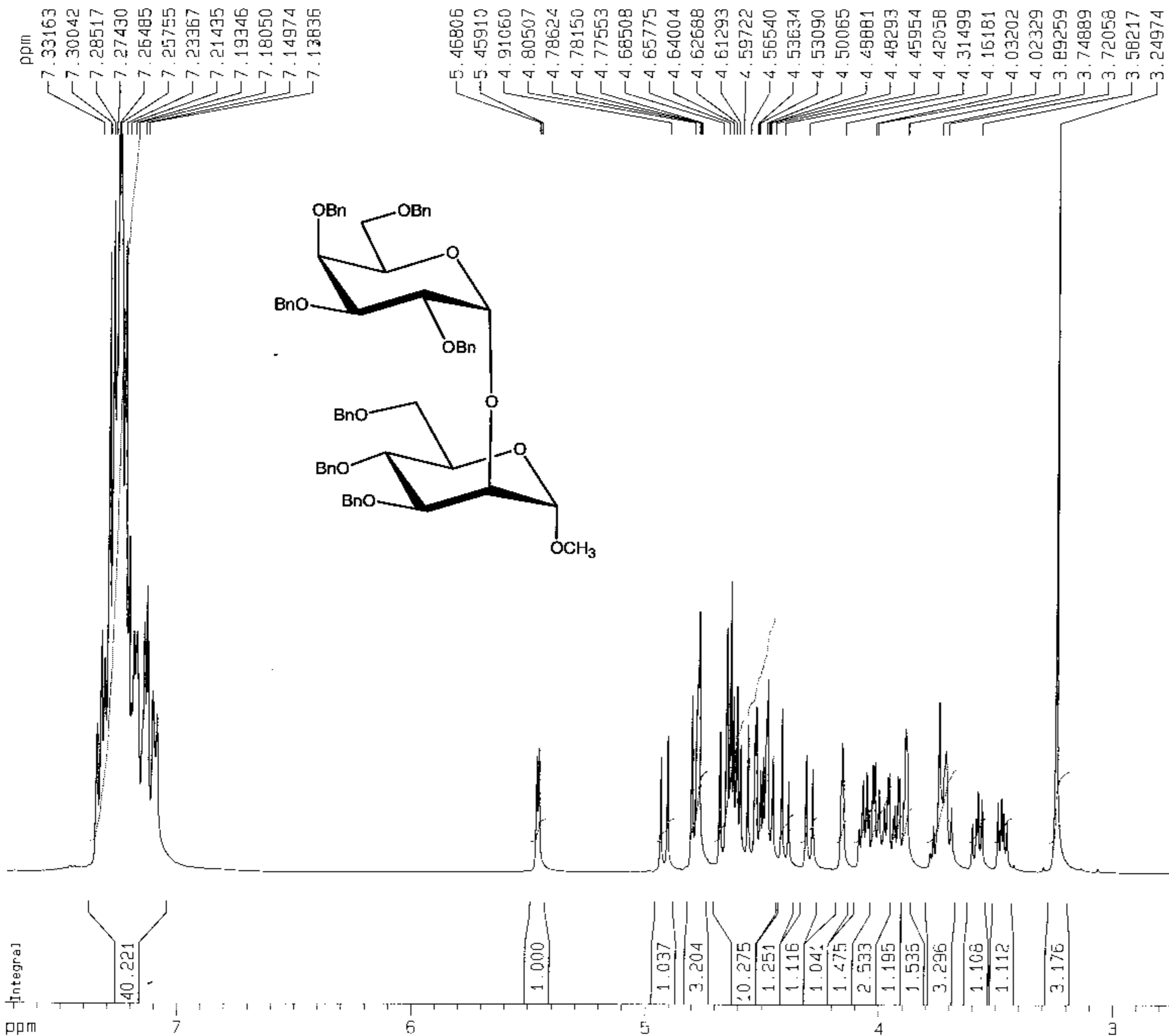
NUC1 1H
 P1 7.50 usec
 PL1 -6.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters

SI 32768
 SF 400.1300360 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 19.50 cm
 F1P 7.730 ppm
 F1 3092.85 Hz
 F2P 2.729 ppm
 F2 1091.84 Hz
 PPMCM 0.25646 ppm/cm
 HZCM 102.61584 Hz/cm



GXC-IV-12-A-C13

Current Data Parameters
 NAME gxciv12a
 EXPNO 2
 PROCNO 1

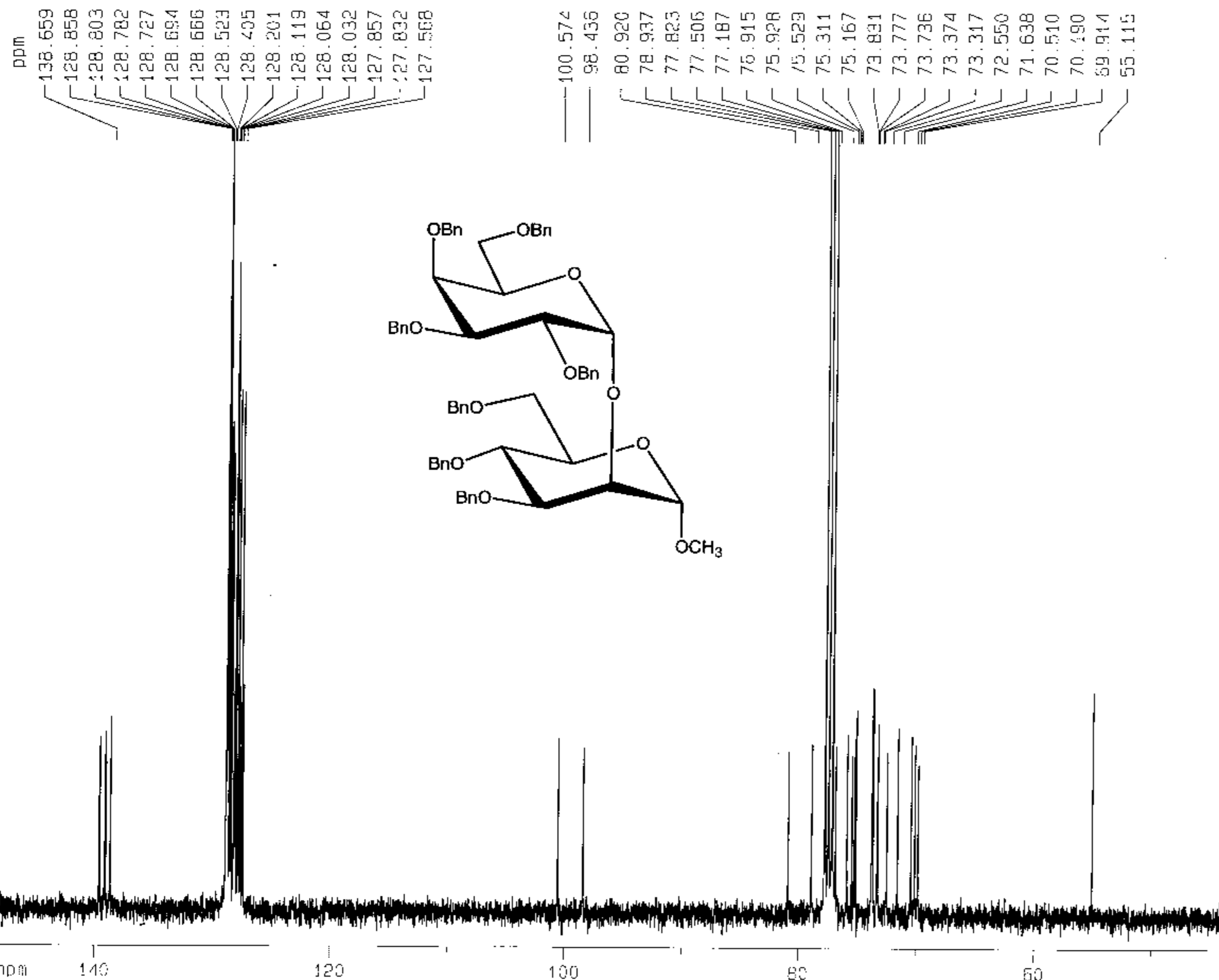
F2 - Acquisition Parameters
 Date_ 991208
 Time 10.06
 INSTRUM spect
 PROBHD 5 mm 860 BB-1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 213
 DS 4
 SWH 25125.629 Hz
 FIDRES 0.383387 Hz
 AQ 1.3042164 sec
 RG 4096
 DW 19.900 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 5.90 usec
 PL1 -6.00 dB
 SF01 100.6237959 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -6.00 dB
 PL12 15.80 dB
 PL13 15.80 dB
 SF02 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127290 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

10 NMR plot parameters
 CX 19.50 cm
 F1P 148.309 ppm
 F1 14321.73 Hz
 F2P 43.843 ppm
 F2 4411.11 Hz
 PPMCM 5.35723 ppm/cm
 HZCM 539.00568 Hz/cm



138.659
 128.858
 128.803
 128.782
 128.727
 128.694
 128.666
 128.523
 128.495
 128.201
 128.119
 128.064
 128.032
 127.857
 127.832
 127.568

100.574
 98.436
 80.920
 78.937
 77.823
 77.506
 77.187
 76.915
 75.928
 75.529
 75.311
 75.167
 73.831
 73.777
 73.736
 73.374
 73.317
 72.550
 71.638
 70.510
 70.190
 69.914
 55.115

ppm 140 120 100 80 60

GXC-IV-1-H1

Current Data Parameters

NAME gxciv1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 991115
 Time 21.11
 INSTRUM spect
 PROBHD 5 mm 680 BB-1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 33
 DS 0
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 57
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec

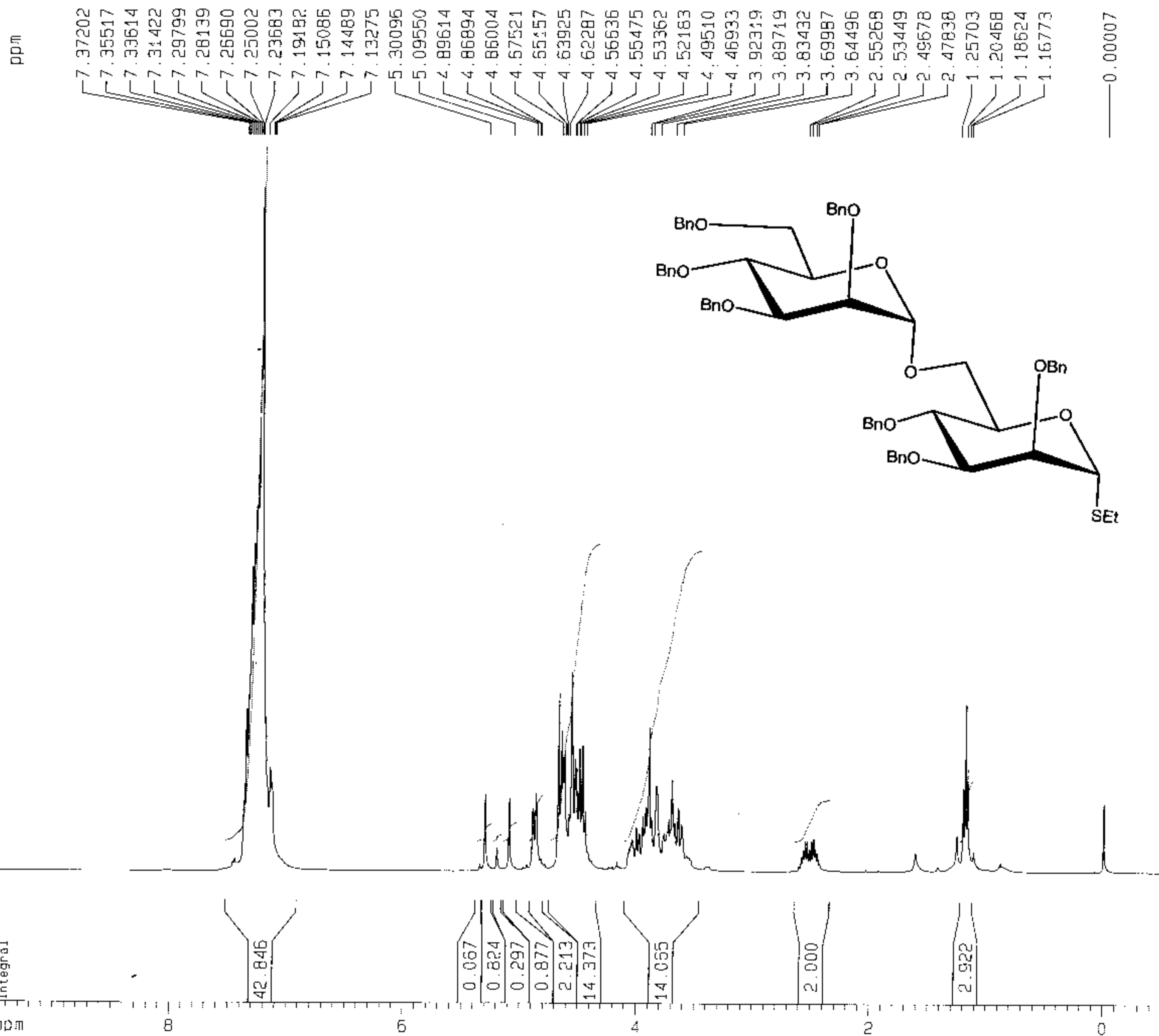
===== CHANNEL f1 =====
 NUC1 1H
 P1 7.50 usec
 PL1 -6.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters

SI 32768
 SF 400.1300299 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 19.50 cm
 F1P 9.500 ppm
 F1 3801.24 Hz
 F2P -0.500 ppm
 F2 -200.06 Hz
 PPMCM 0.51282 ppm/cm
 HZCM 205.19489 Hz/cm



7.37202
 7.35517
 7.33614
 7.31422
 7.29799
 7.28139
 7.26690
 7.25002
 7.23683
 7.19182
 7.15086
 7.14489
 7.13275
 5.30096
 5.09550
 4.89614
 4.86894
 4.86004
 4.67521
 4.55157
 4.63925
 4.62287
 4.56636
 4.55475
 4.53362
 4.52163
 4.49510
 4.46933
 3.92319
 3.89719
 3.83432
 3.69987
 3.64496
 2.55268
 2.53449
 2.49678
 2.47838
 1.25703
 1.20468
 1.18624
 1.16773
 0.00007

42.846
 0.067
 0.824
 0.297
 0.877
 2.213
 14.373
 14.055
 2.000
 2.922

GXC-TV-1-C13

Current Data Parameters

NAME gxciv1
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

Date_ 991115
 Time 21.19
 INSTRUM spect
 PROBHD 5 mm BBO BB-1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 588
 DS 4
 SWH 25125.629 Hz
 FIDRES 0.383387 Hz
 AQ 1.3042164 sec
 RG 3649.1
 DW 19.900 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====

NUC1 13C
 P1 5.90 usec
 PL1 -6.00 dB
 SFO1 100.6237959 MHz

===== CHANNEL f2 =====

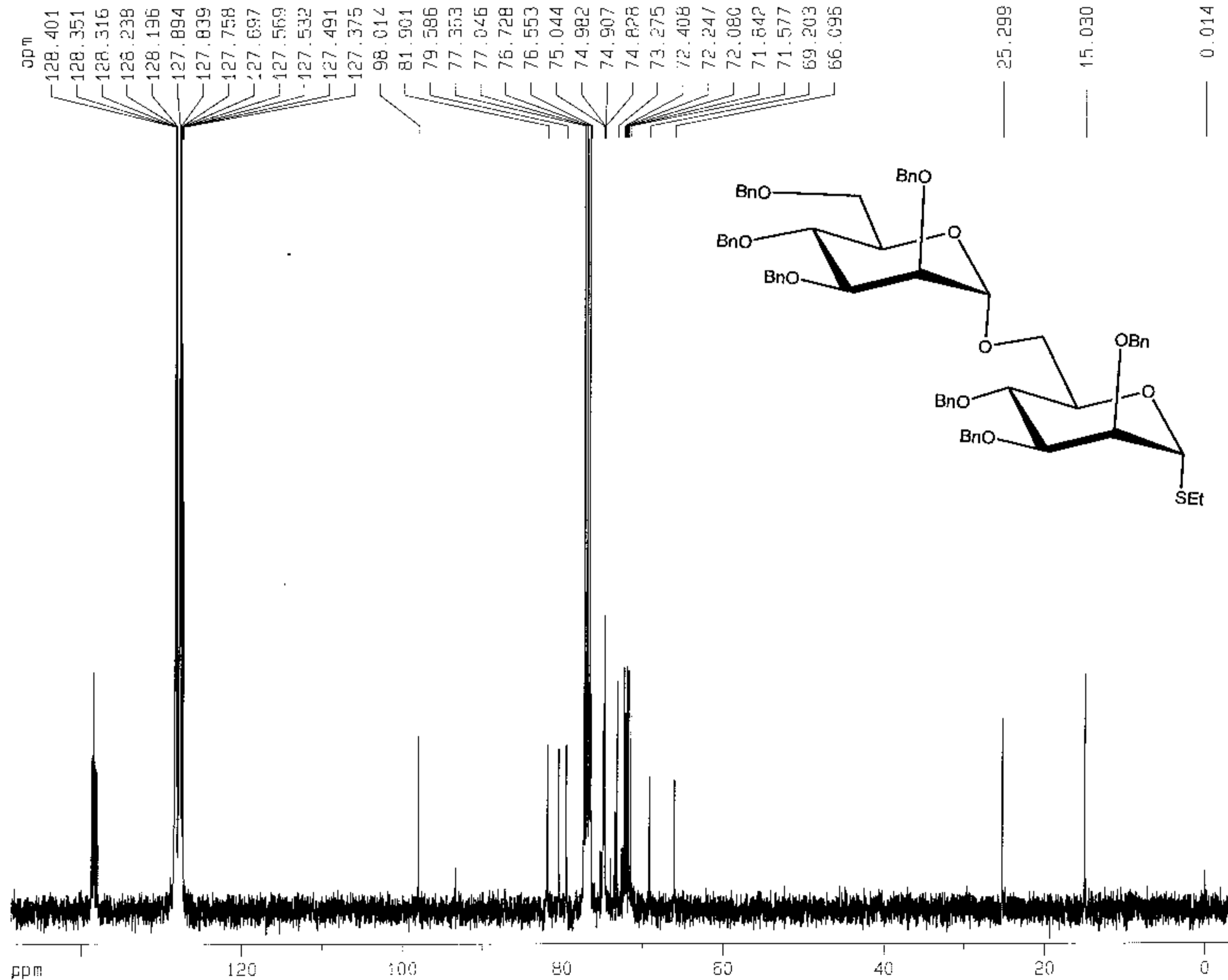
CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -6.00 dB
 PL12 15.80 dB
 PL15 15.80 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters

SI 32768
 SF 100.6127728 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters

CX 19.50 cm
 F1P 148.700 ppm
 F1 14961.15 Hz
 F2F 4.278 ppm
 F2 -430.38 Hz
 PRMCM 7.84502 ppm/cm
 HZCM 789.30936 Hz/cm



GXC-IV-17-A-H1

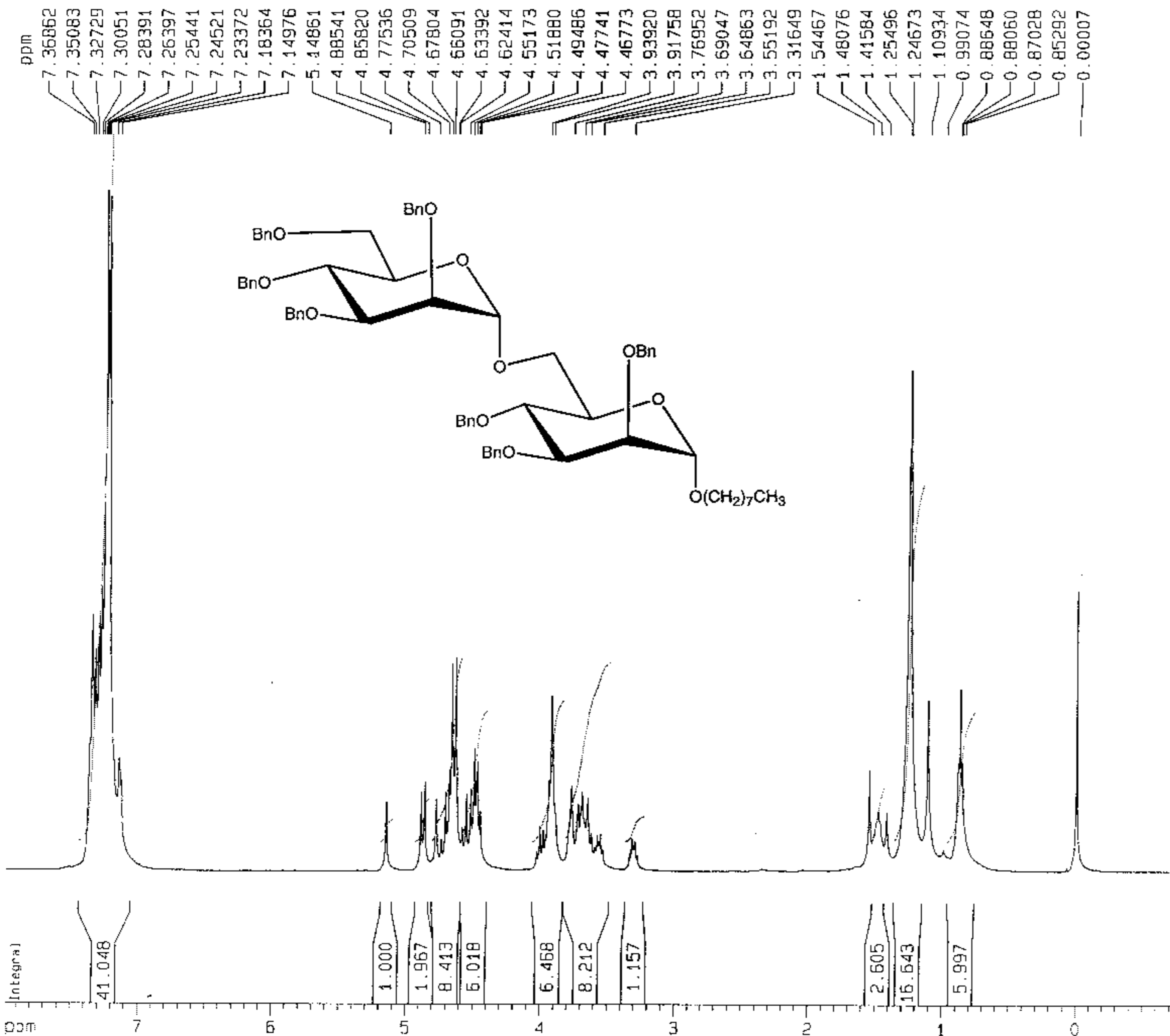
Current Data Parameters
 NAME gxc0417a
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 991221
 Time 18.17
 INSTRUM spect
 PROBHD 5 mm SB0 BB-1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 57
 DS 0
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 203.2
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.50 usec
 PL1 -6.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300195 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 19.50 cm
 F1P 7.976 ppm
 F1 3191.56 Hz
 F2P -0.747 ppm
 F2 -298.78 Hz
 PPMCM 0.44733 ppm/cm
 HZCM 178.99202 Hz/cm



GXC-IV-17-A-C13

Current Data Parameters

NAME gxciv17a
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters

Date_ 991221
 Time 22.39
 INSTRUM spect
 PROBHD 5 mm GNP 1H/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 9234
 DS 4
 SWH 15723.271 Hz
 FIDRES 0.239918 Hz
 AQ 2.0840948 sec
 RG 14596.5
 DW 31.800 usec
 DE 30.00 usec
 TE 300.0 K
 D1 2.0000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

----- CHANNEL f1 -----

NUC1 13C
 P1 8.00 usec
 PL1 0.00 dB
 SF01 62.9021320 MHz

----- CHANNEL f2 -----

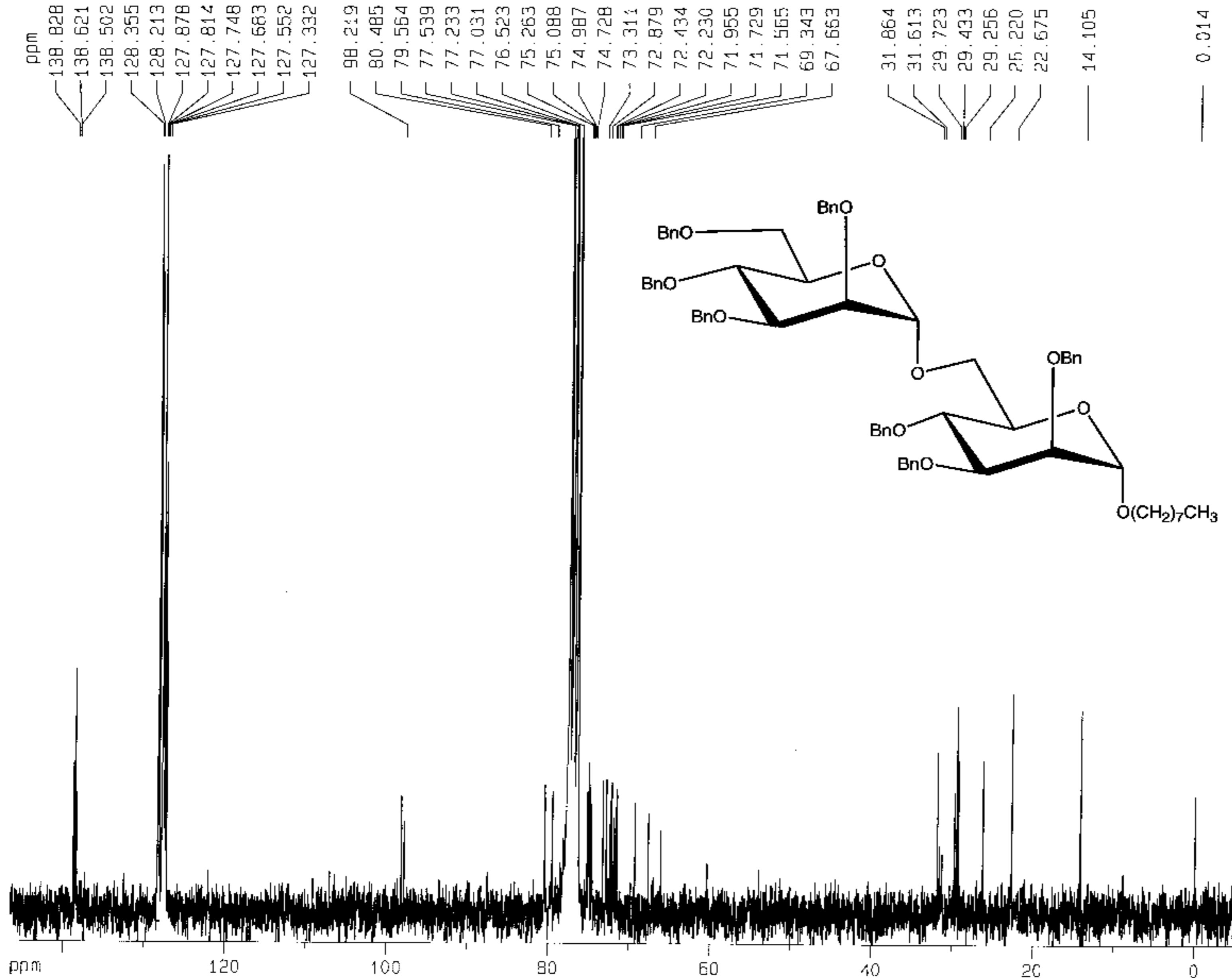
CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -4.00 dB
 PL12 16.00 dB
 PL13 17.00 dB
 SF02 250.1310005 MHz

F2 Processing parameters

SI 32768
 SF 62.8952369 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters

CX 19.50 cm
 F1P 146.603 ppm
 F1 9220.65 Hz
 F2P -5.433 ppm
 F2 -341.73 Hz
 PPMCM 7.79676 ppm/cm
 HzCM 490.37978 Hz/cm



GXC-III-94-H1-TOP

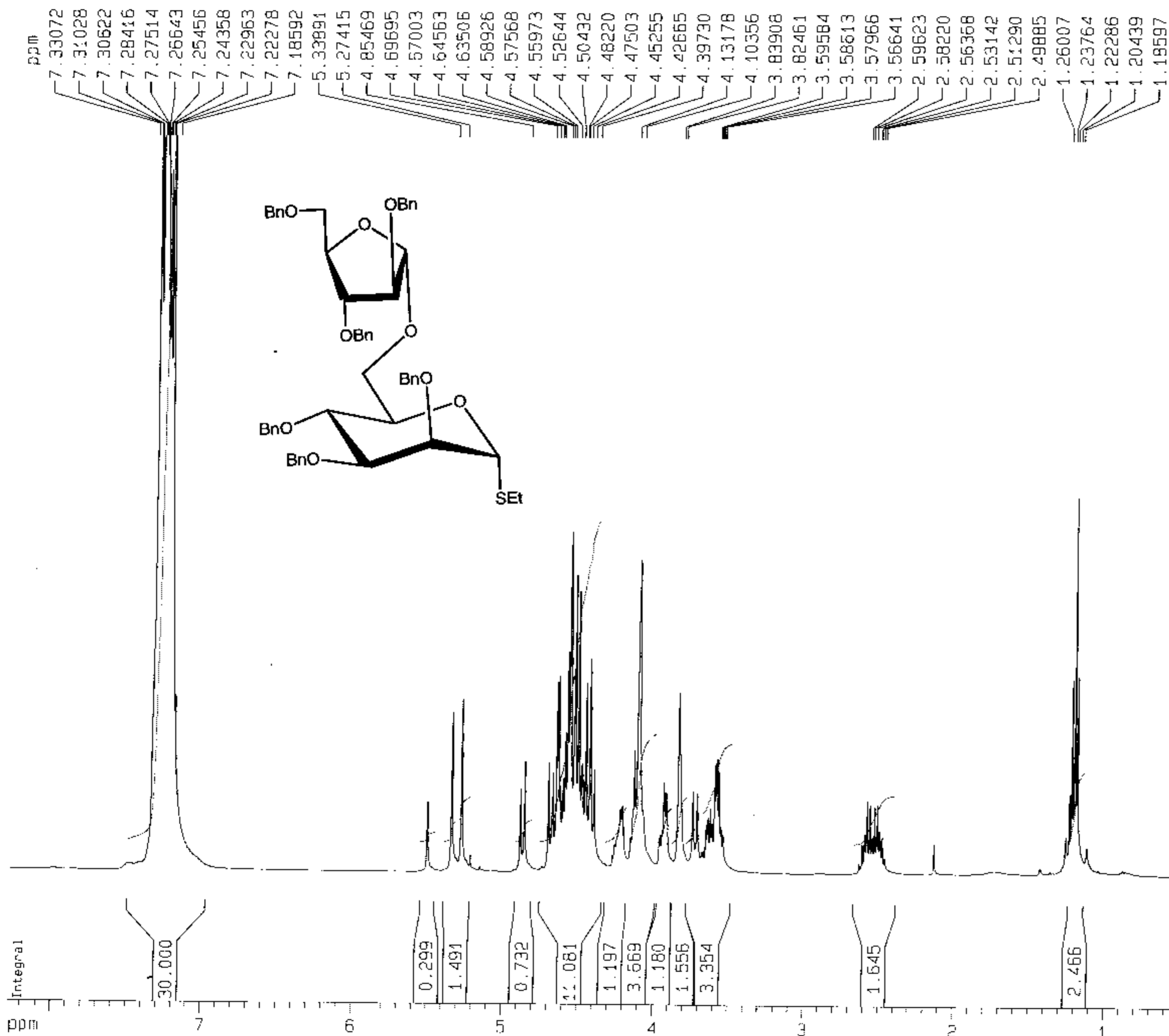
Current Data Parameters
 NAME gxciii94t
 EXPNO 1
 PROCNO 1

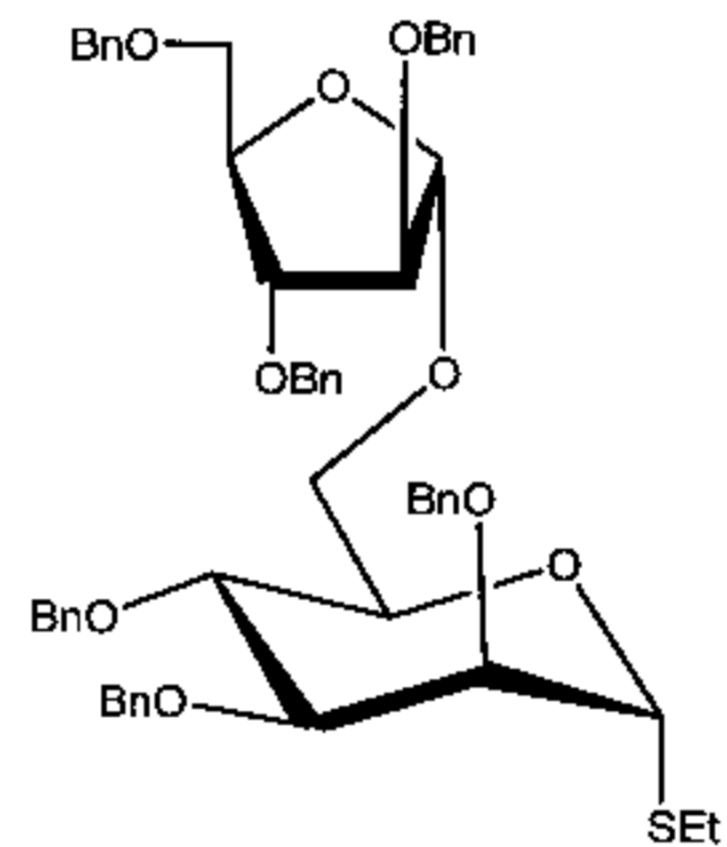
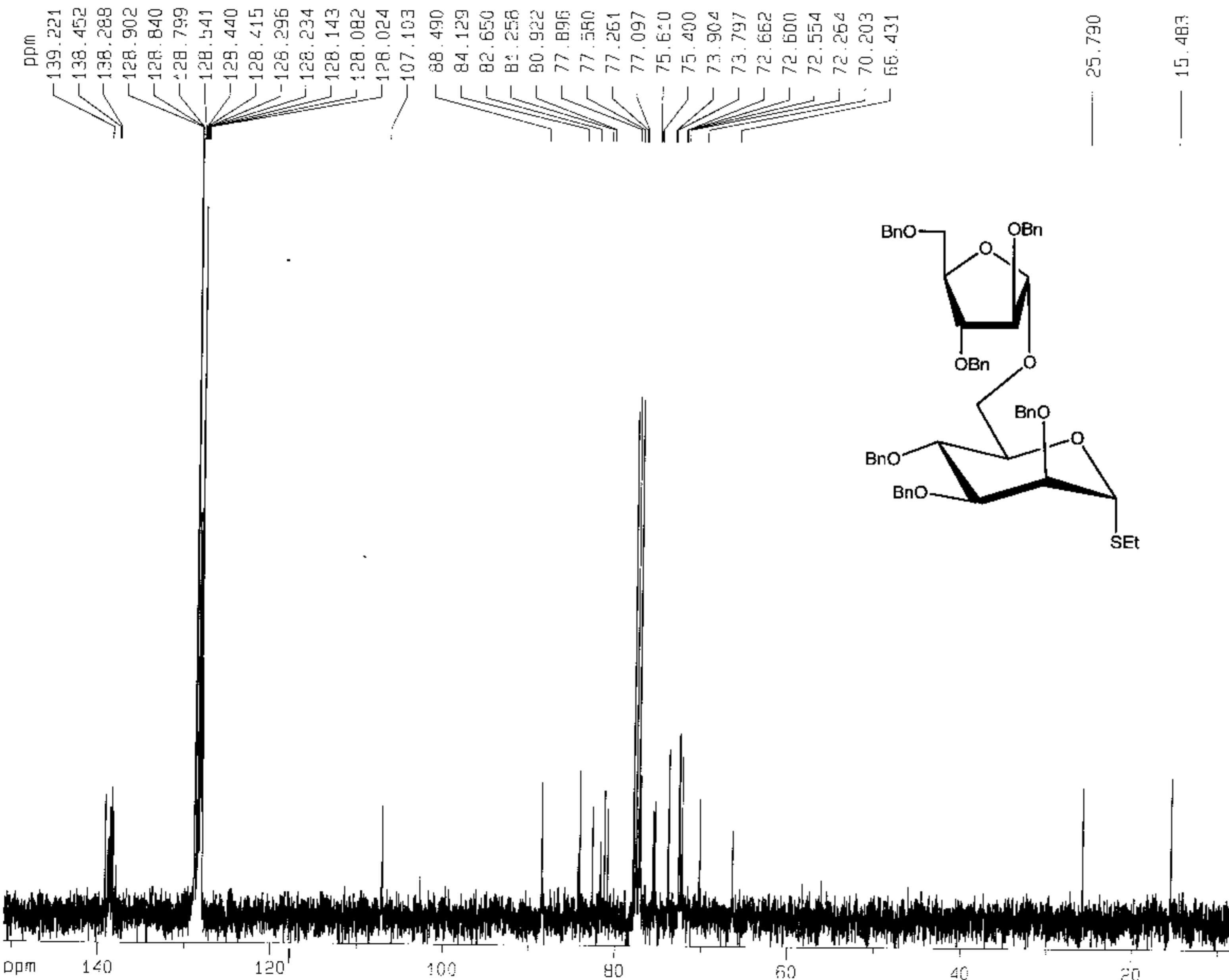
F2 - Acquisition Parameters
 Date_ 991020
 Time 17.47
 INSTRUM spect
 PROBHD 5 mm BBO BB-1
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 26
 DS 0
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 32
 JW 60.400 usec
 DE 6.00 usec
 IE 300.0 K
 D1 2.00000000 sec

==== CHANNEL f1 =====
 NUC1 1H
 P1 7.50 usec
 PL1 -6.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
 ST 32768
 SF 400.1300473 MHz
 WDW EM
 SSB 0
 _B 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 19.50 cm
 F1P 8.272 ppm
 F1 3309.95 Hz
 F2P 0.531 ppm
 F2 212.50 Hz
 PPMCM 0.39698 ppm/cm
 HZCM 158.84369 Hz/cm





Current Data Parameters

NAME gxc11194t
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 991020
Time 17.51
INSTRUM spect
PROBHD 5 mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 19
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 5160.6
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec
d11 0.0300000 sec
d12 0.00002000 sec

----- CHANNEL f1 -----

NUC1 13C
P1 5.90 usec
PL1 -6.00 dB
SFO1 100.6237959 MHz

----- CHANNEL f2 -----

CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 15.80 dB
PL13 15.80 dB
SFO2 400.1316005 MHz

F2 - Processing parameters

SI 32768
SF 100.6127990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 19.50 cm
F1P 150.789 ppm
F1 15171.32 Hz
F2P 8.561 ppm
F2 861.36 Hz
PPMCM 7.29375 ppm/cm
HZCM 733.84418 Hz/cm

GXC-III-99-H1

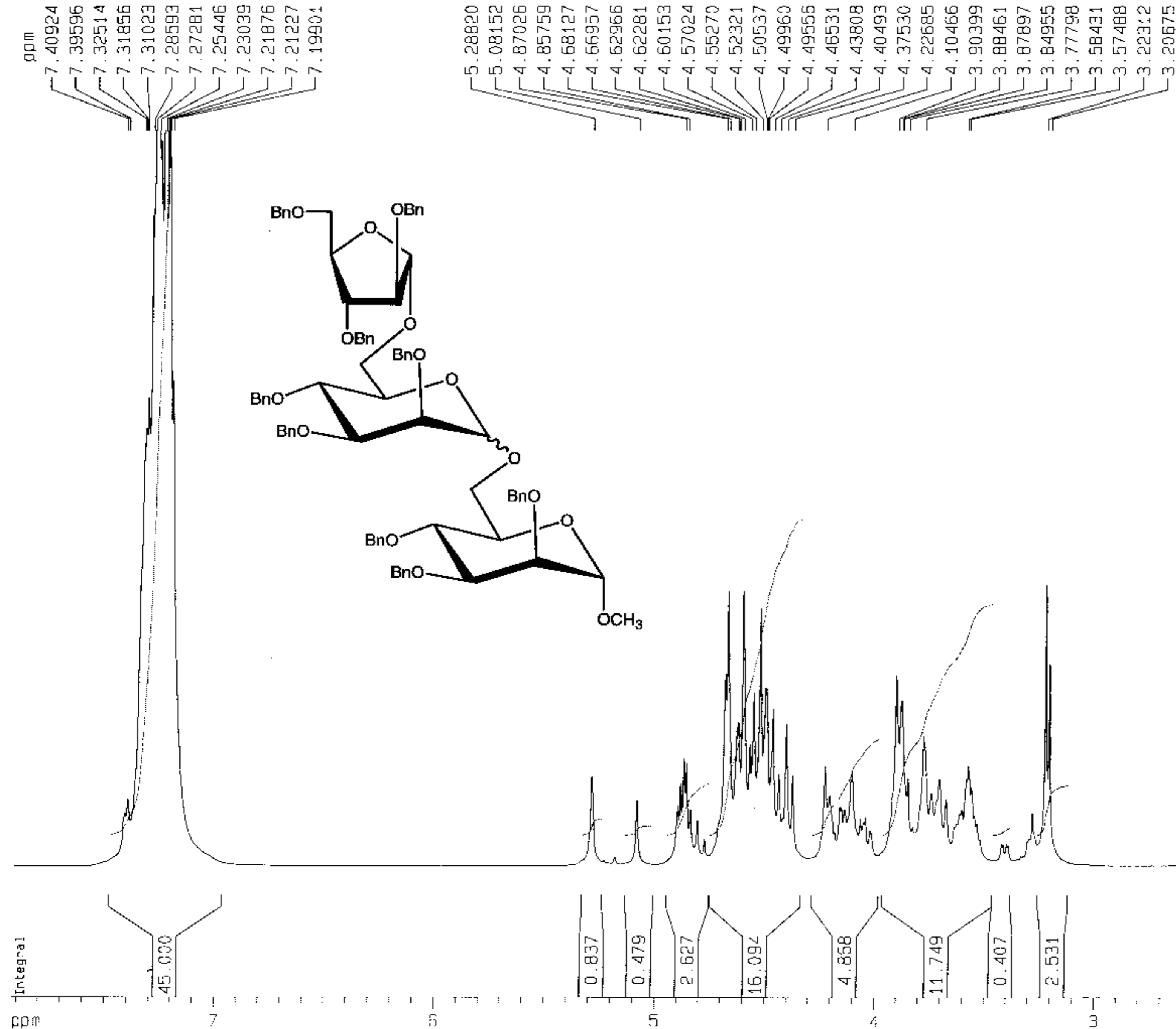
Current Data Parameters
 NAME gxciii99
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 991105
 Time 13.21
 INSTRUM spect
 PROBHD 5 mm BBO BB-1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 46
 DS 0
 SWH 8278.146 Hz
 FTDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 35.9
 DW 60.400 usec
 DE 6.00 usec
 TC 300.0 K
 D1 2.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.50 usec
 PL1 -6.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300417 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 19.50 cm
 F1P 7.921 ppm
 F1 3169.40 Hz
 F2P 2.623 ppm
 F2 1049.61 Hz
 PPMCM 0.27168 ppm/cm
 HZCM 108.70717 Hz/cm



GXC-111-99-C13

Current Data Parameters
 NAME gxc11199
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 991105
 Time 13.29
 INSTRUM spect
 PROBD 5 mm BBO BB-1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 147
 DS 4
 SWH 25125.629 Hz
 FIDRES 0.383387 Hz
 AQ 1.3042164 sec
 RG 16384
 JW 19.900 usec
 DE 6.00 usec
 TE 300.0 K
 O1 2.0000000 sec
 d11 0.0300000 sec
 d12 0.0000200 sec

----- CHANNEL f1 -----
 NUC1 13C
 P1 5.90 usec
 PL1 -6.00 dB
 SFO1 100.6237959 MHz

----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -6.00 dB
 PL12 15.80 dB
 PL13 15.80 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127290 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 19.50 cm
 F1P 142.562 ppm
 F1 14343.57 Hz
 F2P 50.299 ppm
 F2 5060.72 Hz
 PCMC 4.73145 ppm/cm
 HZCM 476.04370 Hz/cm

