Supporting Information

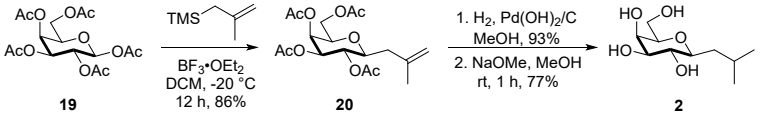
Materials and Reagents Unless otherwise noted, all commercially available reagents were used as obtained from suppliers without further purification. Chemicals were purchased from Sigma–Aldrich (Merck), Fisher–Scientific, Alfa–Aeser, Fluorochem, Carbosynth, or TCI. Solvents used in work up procedures and chromatography were either reagent or HPLC grade and were used without any further purification. Dichloromethane, methanol, toluene, acetonitrile, and tetrahydrofuran were obtained from commercial sources and dried using a Puresolv™ dry solvent purification system before performing reactions. Deuterated solvents $CDCl_3$, CD_3OD, Acetone–d_6 were obtained from suppliers and used without purification.

General Methods for Synthesis and Compound Analysis All reactions were performed under an inert atmosphere. Air and/or moisture sensitive liquids were transferred via cannula or plastic syringes fitted with stainless steel needles. Reactions were monitored by thin-layer chromatography (TLC) performed on aluminium sheets precoated with silica gel using the solvents indicated. Spots were visualized by UV light (254 nm) and charred with H_2SO_4–MeOH (1:20). During work–up, solutions in organic solvents were dried over Na_2SO_4. Removal of volatile organic solvents was carried out under reduced pressure on a Heidolph and/or Buchi rotary evaporator with the water bath temperature set between 40–50 °C. Chromatography was carried out with silica gel 60 (0.040–0.630 mm) using the solvent indicated via a stepwise solvent polarity gradient correlated with TLC mobility. Proton nuclear magnetic resonance (^{1}H–NMR) spectra and proton-decoupled carbon nuclear magnetic resonance (^{13}C–NMR) spectra were recorded at 25 °C with 400 Jeol and 500 MHz Agilent spectrometers. Data are reported in the following order: Chemical shift (δ) in ppm relative to internal standard Me_4Si in $CDCl_3$ (δ 0.0) for ^{1}H and $CDCl_3$ (δ 77.0) or CD_3OD (δ 47.59) for ^{13}C; multiplicities indicated as b (broad), s (singlet), d (doublet), t (triplet), q (quartet), p (pentet), hept (heptet), m (multiplet); Coupling constants (*J*) are given in Hertz (Hz). ^{1}H–NMR spectral signals were assigned with the aid of COSY, ^{13}C–NMR spectral signals using DEPT, gHSQCAD and/or gHMBCAD. Assignments of sugar protons are reported from H–1 to H–6a/b, with H–1 as the anomeric proton and H–6a & H–6b as the C-6 protons. Carbon assignments follow the same numbering pattern. H–1' refers to protons α to the anomeric carbon in the aglycon moiety, H–2' refers to protons β to the anomeric carbon, i.e. the iPr CH. Carbon assignments follow the same numbering pattern. NMR data for known compounds was in good agreement with previously published data. High resolution mass spectra were measured in positive and/or negative mode as indicated using an Agilent Accurate Mass Q–TOF LC/MS Mass Spectrometry instrument using MeCN (acetonitrile) or MeOH as solvent. All HRMS data reported is within 5 ppm of the calculated mass.

Isopropyl β-D-thiogalactopyranose (IPTG, 1) IPTG was purchased from Carbosynth and used without further purification. ^{1}H-NMR (500 MHz, CD_3OD) δ 4.39 (d, *J* = 9.4 Hz, 1H, H-1), 3.87 (dd, *J* = 3.3, 1.1 Hz, 1H, H-4), 3.72 (dd, *J* = 11.4, 6.8 Hz, 1H, H-6a), 3.68 (dd, *J* = 11.4, 5.5 Hz, 1H, H-6b), 3.53 – 3.48 (overlapping signals, 2H, H-2 & H-5), 3.45 (dd, *J* = 9.3, 3.3 Hz, 1H, H-3), 3.23 (hept, *J* = 6.8 Hz, 1H, isopropyl CH), 1.31 (d, *J* = 7.1 Hz, 3H), 1.30 (d, *J* = 7.2 Hz, 3H) (each CH_3); ^{13}C-NMR (126 MHz, CD_3OD) δ 85.71 (C-1), 79.08 (C-5), 74.92 (C-3), 70.20 (C-2), 69.04 (C-4), 61.18 (C-6), 34.39 (C-1'), 22.98, 22.88 (each CH_3).

3,4,6-Tri-*O*-benzyl-D-galactal (7) D-Galactal (2.00 g, 13.7 mmol) was dissolved in DMF and cooled to 0°C. NaH (2.18 g, 54.5 mmol) was added slowly, causing liberation of H_2 and fizzing. The reaction was stirred for 10 mins followed by addition of BnBr (6.47 mL, 54.5 mmol). The reaction was allowed to attain room temperature and stirred overnight and was eventually quenched with MeOH following indication of product formation via TLC. The reaction mixture was extracted with EtOAc and the combined organic layers were washed with water, dried with Na_2SO_4 and the solvent was removed under reduced pressure. Chromatography (cyclohexane-EtOAc 4:1) afforded the product as a white solid (4.85 g, 85%). R_f 0.51 (cyclohexane-EtOAc 4:1); ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.44 – 7.16 (overlapping signals, 16H, each Ar-H), 6.37 (dd, *J* = 6.3, 1.5 Hz, 1H, H-1), 4.88 (d, *J* = 12.0 Hz, 1H, Bn-

CH_2), 4.86 (ddd, *J* = 6.3, 2.9, 1.3 Hz, 1H, H-2), 4.66 (d, *J* = 12.2 Hz, 1H), 4.65 (d, *J* = 12.0 Hz, 1H), 4.62 (d, *J* = 12.2 Hz, 1H), 4.51 (d, *J* = 11.9 Hz, 1H), 4.43 (d, *J* = 11.9 Hz, 1H) (each Bn-CH_2), 4.23 – 4.16 (overlapping signals, 2H, H-3 & H-5), 3.95 (bt, *J* = 4.1 Hz, 1H, H-4), 3.79 (dd, *J* = 10.3, 7.2 Hz, 1H, H-6a), 3.66 (dd, *J* = 10.3, 5.1 Hz, 1H, H-6b); ^{13} C-NMR (126 MHz, $CDCl_3$) δ 144.2 (C-1), 138.5, 138.4, 138.0, 128.4, 128.3, 128.1, 127.9, 127.7, 127.6 127.4 (each Ar-C/ C-H) 100.0 (C-2), 75.7 (C-5), 73.4 (CH_2), 73.3 (CH_2), 71.3 (C-4), 70.9 (C-3), 70.7 (CH_2), 68.4 (C-6); HRMS calcd. for $C_{27}H_{32}NO_4$ $[M+NH_4]^+$ 434.2331, found m/z 434.2323. The NMR data obtained was in good agreement with the literature. [2]

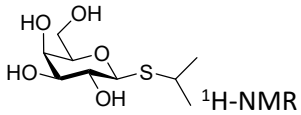


Scheme S1: Synthesis of compound **2**.

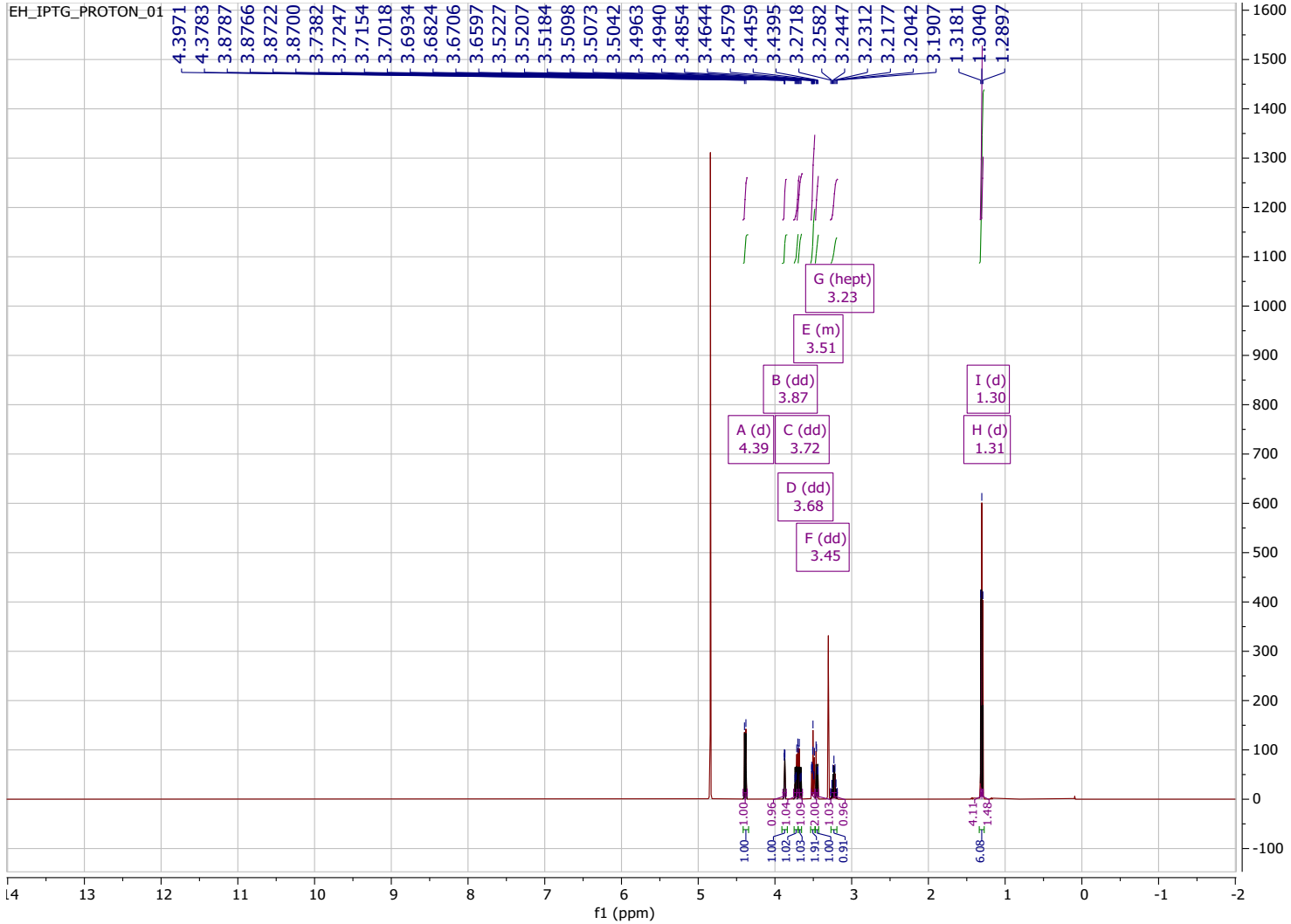
1,2,3,4,6-Penta-*O*-acetyl-β-D-galactopyranose (19) D-Galactose (15.00 g, 83.26 mmol) was dissolved in acetic anhydride (100 mL) and heated to reflux. NaOAc (6.86 g, 83.6 mmol) was added portion wise to the reaction mixture, which was then allowed to heat under reflux for 3 h. The reaction was poured onto ice and stirred for 15 mins. The mixture was filtered and the solid collected. This solid was recrystallized from MeOH to give a white solid as the title compound (28.6 g, 88%). NMR data obtained was in good agreement with the literature. [4] HRMS calcd. for $C_{16}H_{22}O_{11}Na$ $[M+Na]^+$ 413.1054, found m/z 413.1054.

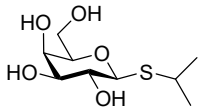
1-Deoxy-1-(2'-Methylallyl)-2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranose (20) Galactopyranose 19 (1.00 g, 2.56 mmol) was dissolved in CH_2Cl_2 and cooled to -20 °C. Methylallyl trimethylsilane (1.80 mL, 10.3 mmol) was added followed by the dropwise addition of $BF_3{\cdot}OEt_2$ (0.81 mL, 6.57 mmol). The reaction mixture was stirred overnight under an inert atmosphere. The reaction mixture was then washed with a satd. $NaHCO_3$ solution, dried over $MgSO_4$ and the solvent removed under reduced pressure. Flash chromatography (cyclohexane-EtOAc 4:1) provided 20 (851 mg, 86%) as a white solid.; R_f 0.32 (cyclohexane-EtOAc 3:2); ^{1} H-NMR (500 MHz, $CDCl_3$) δ 5.40 (dd, *J* = 3.4, 1.2 Hz, 1H, H-4), 5.10 (t, *J* = 9.8 Hz, 1H, H-2), 5.02 (dd, *J* = 10.1, 3.4 Hz, 1H, H-3), 4.79 (t, *J* = 1.8 Hz, 1H, C=C*H*H), 4.73 (q, *J* = 1.2 Hz, 1H, C=CH*H*), 4.13 (dd, *J* = 11.3, 6.9 Hz, 1H, H-6a), 4.04 (dd, *J* = 11.3, 6.5 Hz, 1H, H-6b), 3.84 (td, *J* = 6.8, 1.3 Hz, 1H, H-5), 3.57 (td, *J* = 9.5, 8.1, 4.0 Hz, 1H, H-1), 2.27 (dd, *J* = 14.9, 8.1 Hz, 1H, C*H*H), 2.22 (dd, *J* = 15.1, 4.1 Hz, 1H, CH*H*), 2.15 (s, 3H), 2.02 (s, 3H), 2.02 (s, 3H), 1.97 (s, 3H), 1.75 (s, 3H, CH_3).; ^{13} C-NMR (126 MHz, $CDCl_3$) δ 170.43, 170.32, 170.23, 169.79 (each C=O), 141.41 (*C*=CH_2), 112.67 (C=*C*H_2), 77.07 (C-1), 74.12 (C-5), 72.25 (C-3), 69.52 (C-2), 67.73 (C-4), 61.64 (C-6), 39.80 (*C*HH), 22.77 (CH_3), 20.79, 20.69, 20.64, 20.60 (each OCH_3). The NMR data for **20** obtained was in good agreement with the data reported previously. [1]

Supporting Information



^{1}H-NMR

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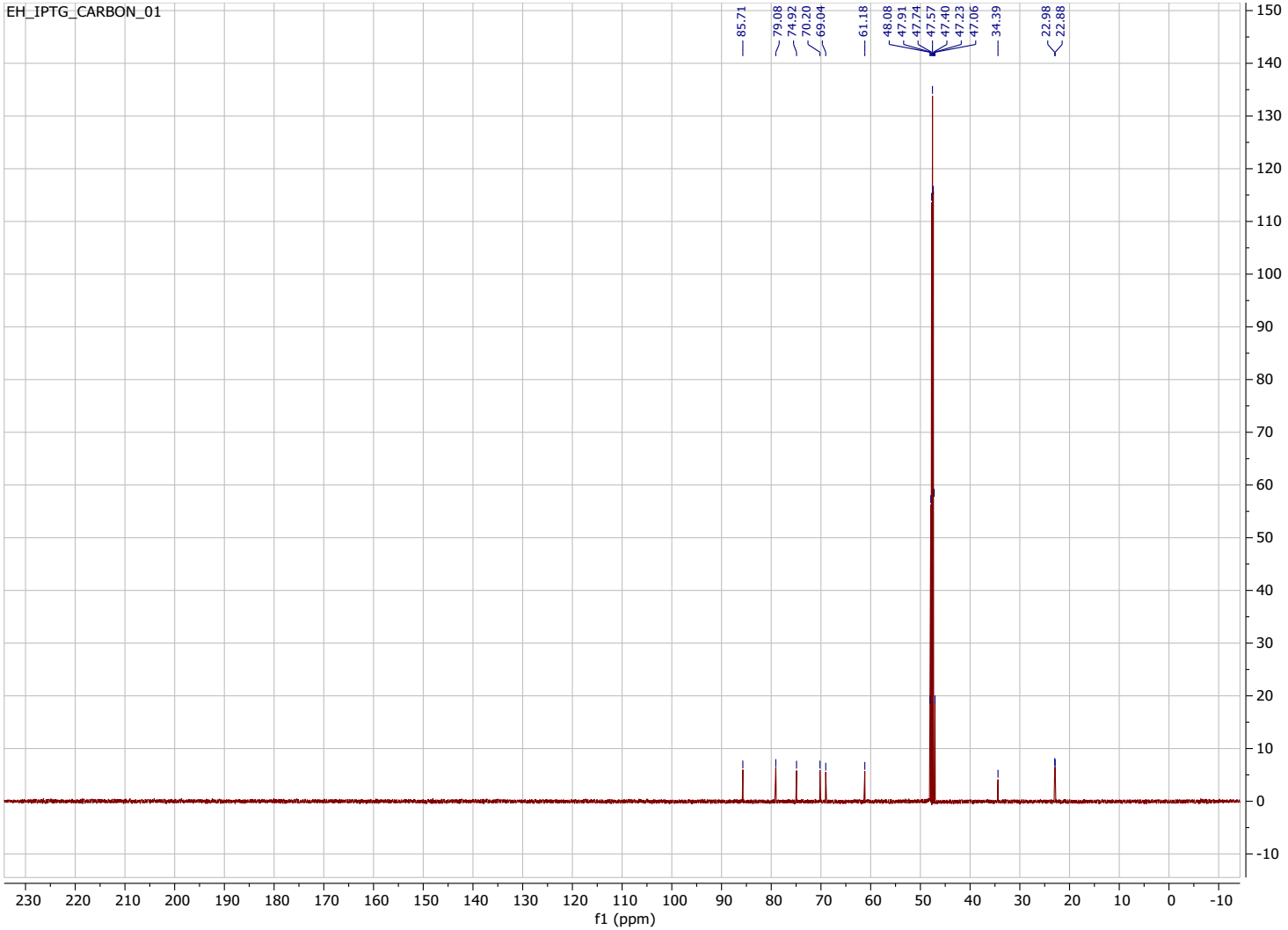
OH
O
HO
S
OH

^{13}C-NMR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70.20
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61.18
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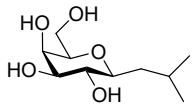
47.23
47.06
34.39
22.98
22.88
f1 (ppm)

Supporting Information



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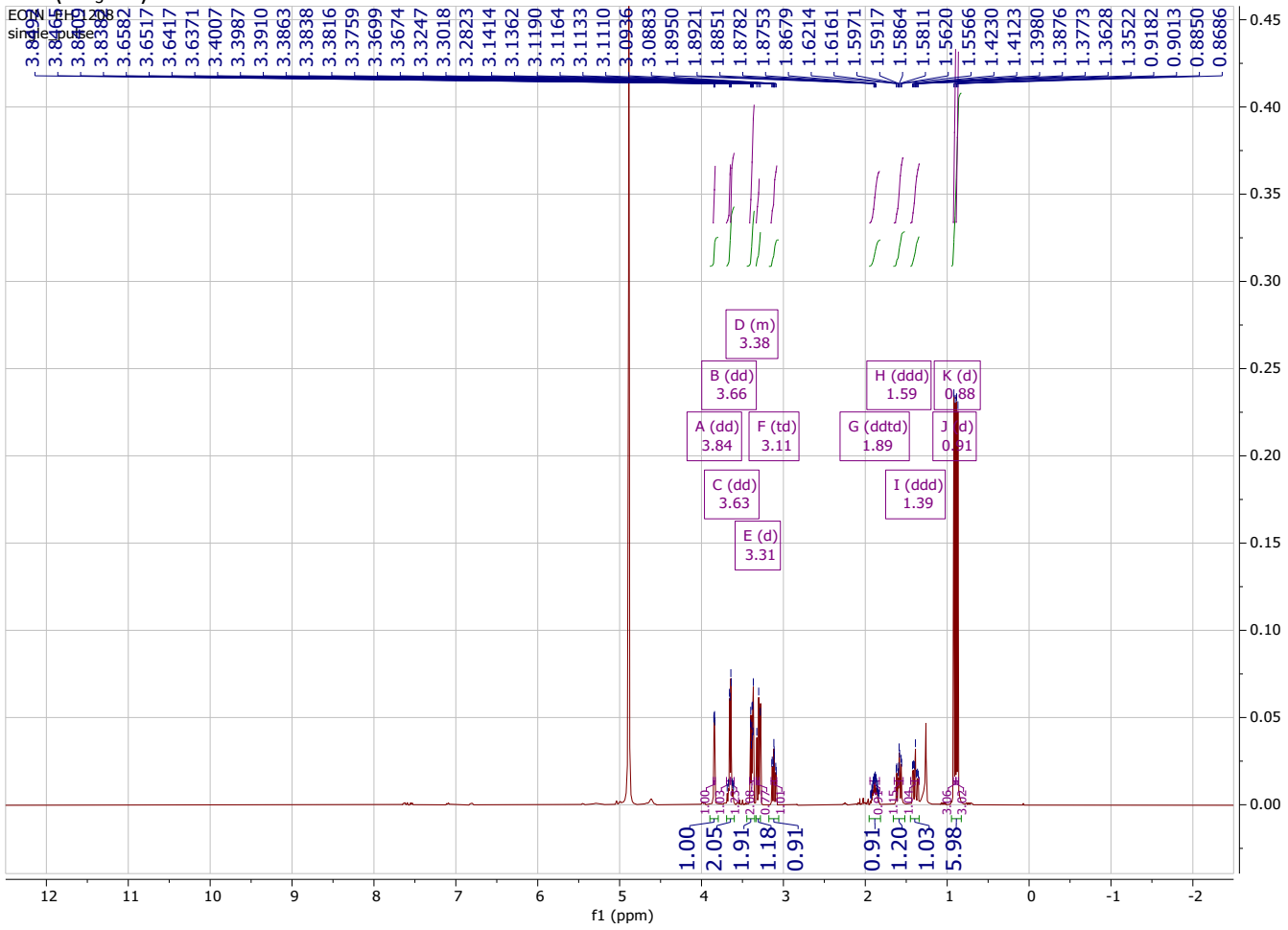

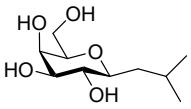

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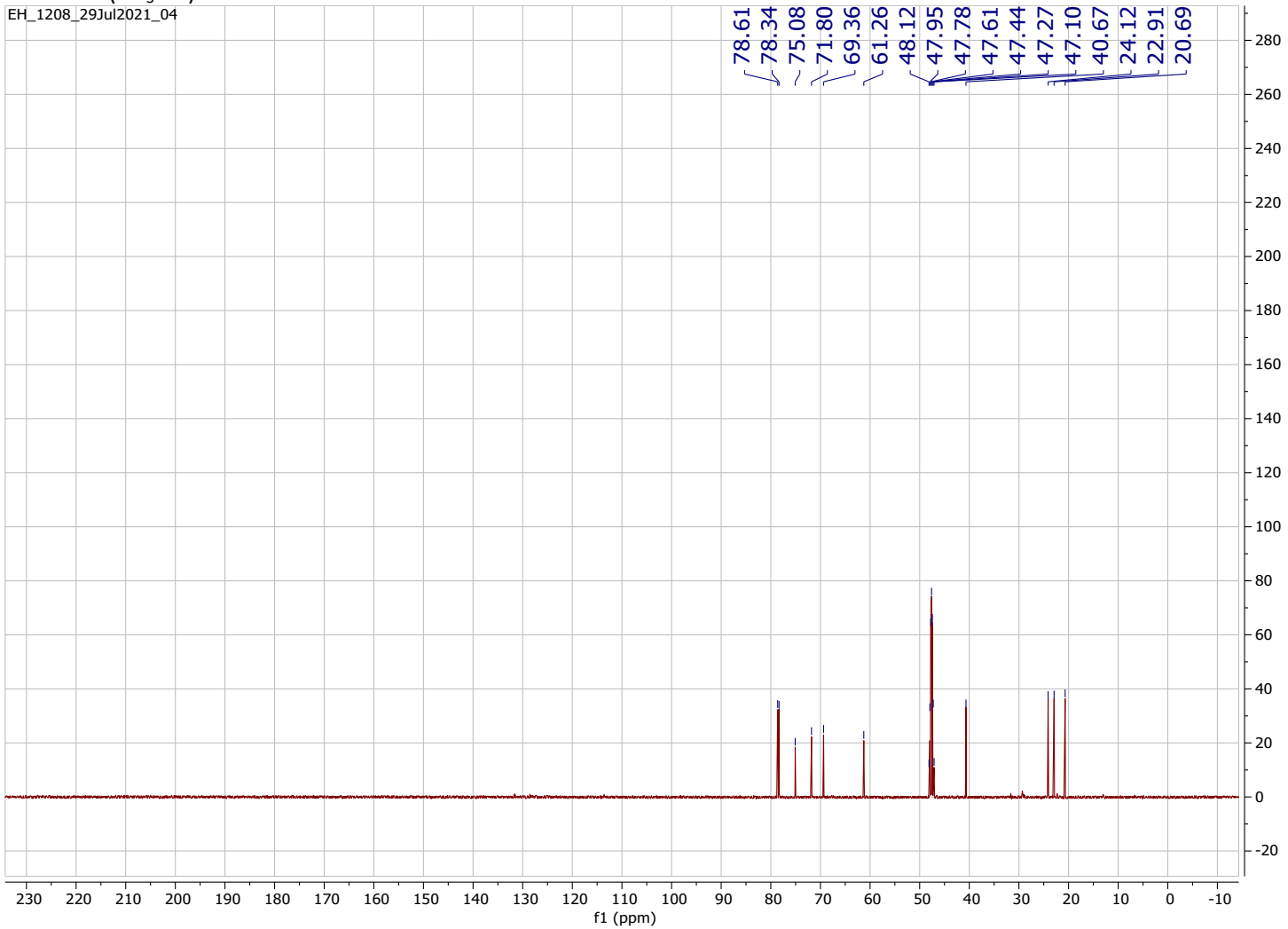
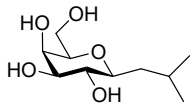
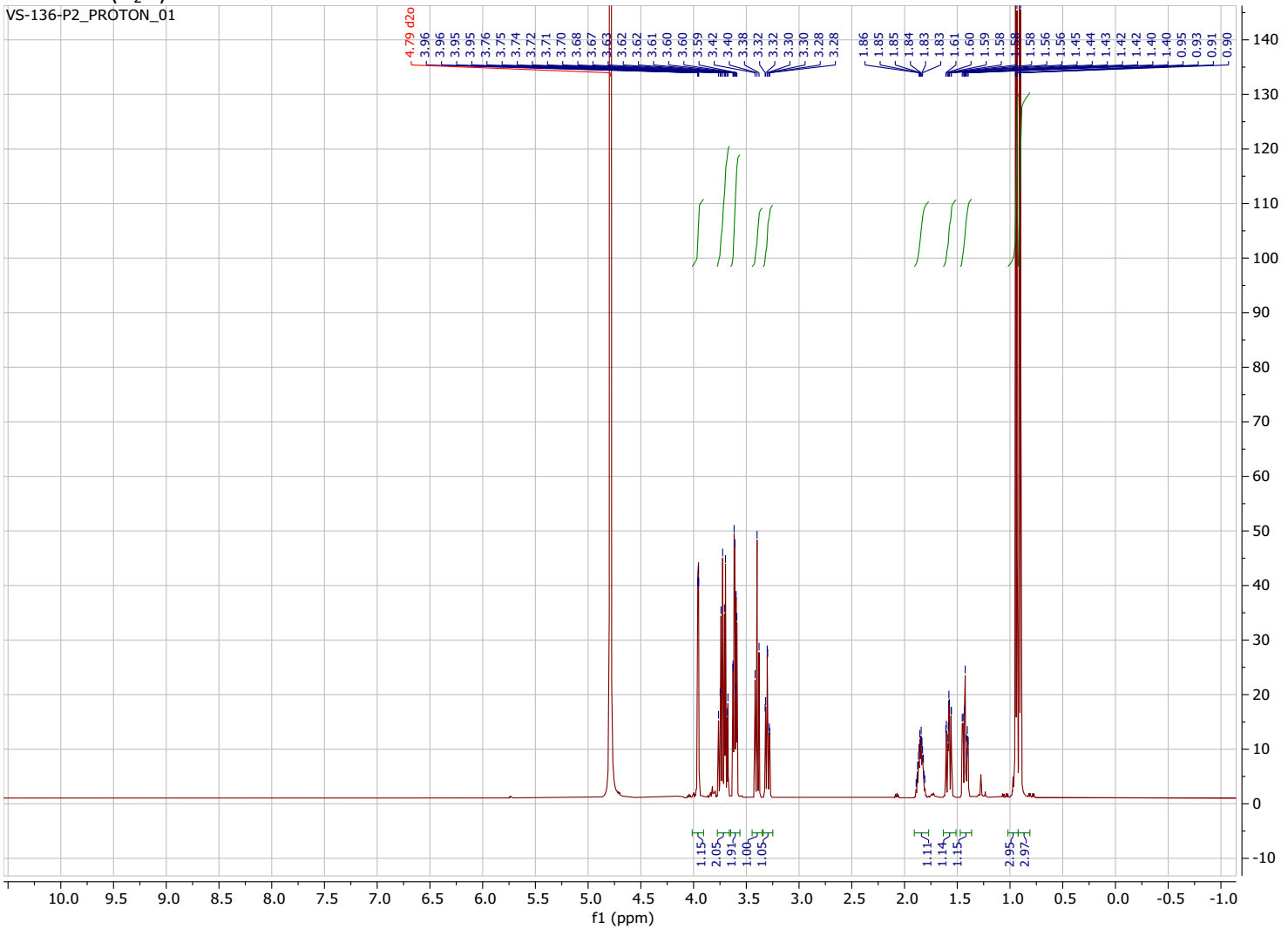
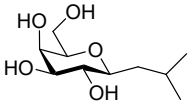


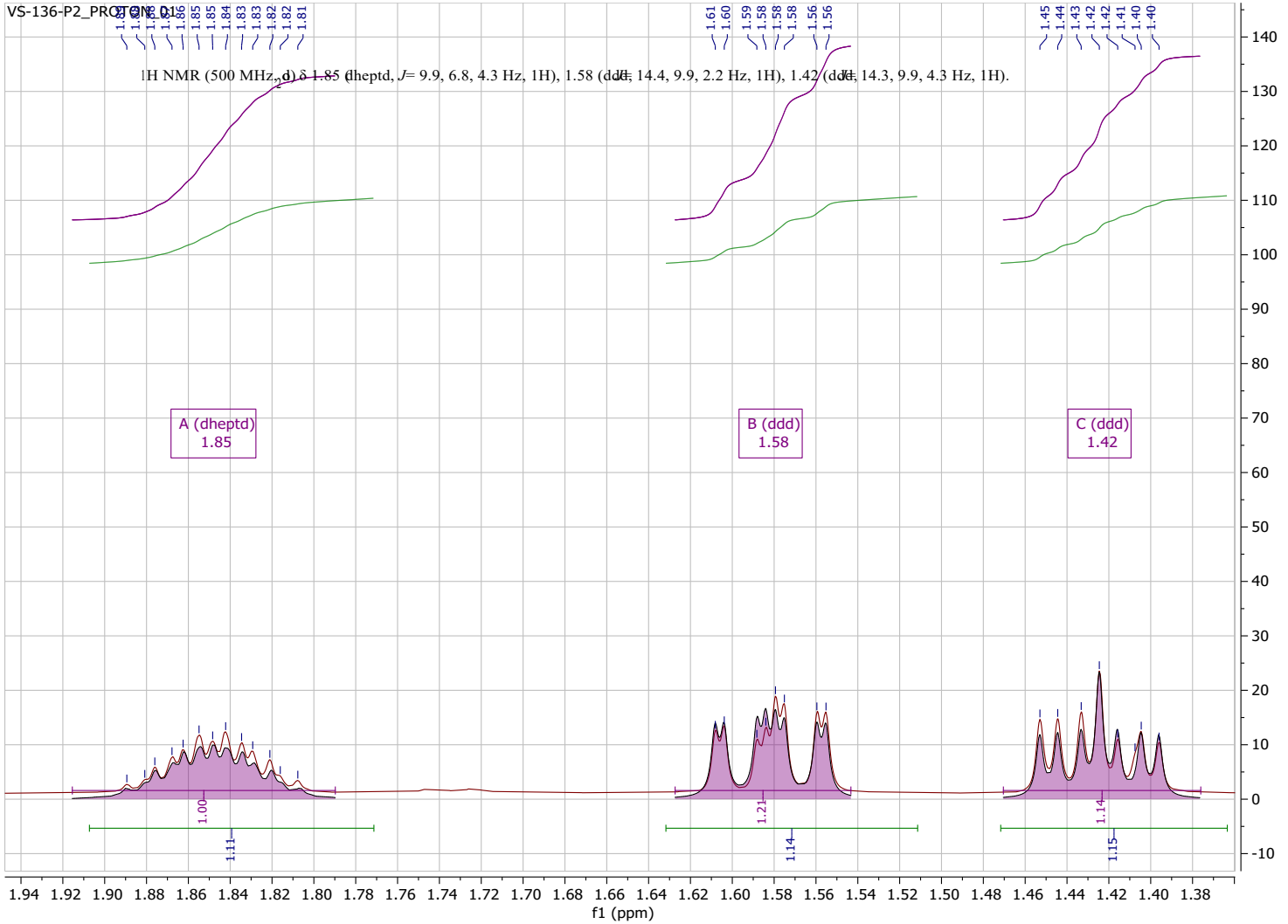


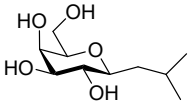
^{1}H-NMR (D_2O)





^{1}H-NMR (D_2O) with simulation of signals using multiplet manager in MNova shown

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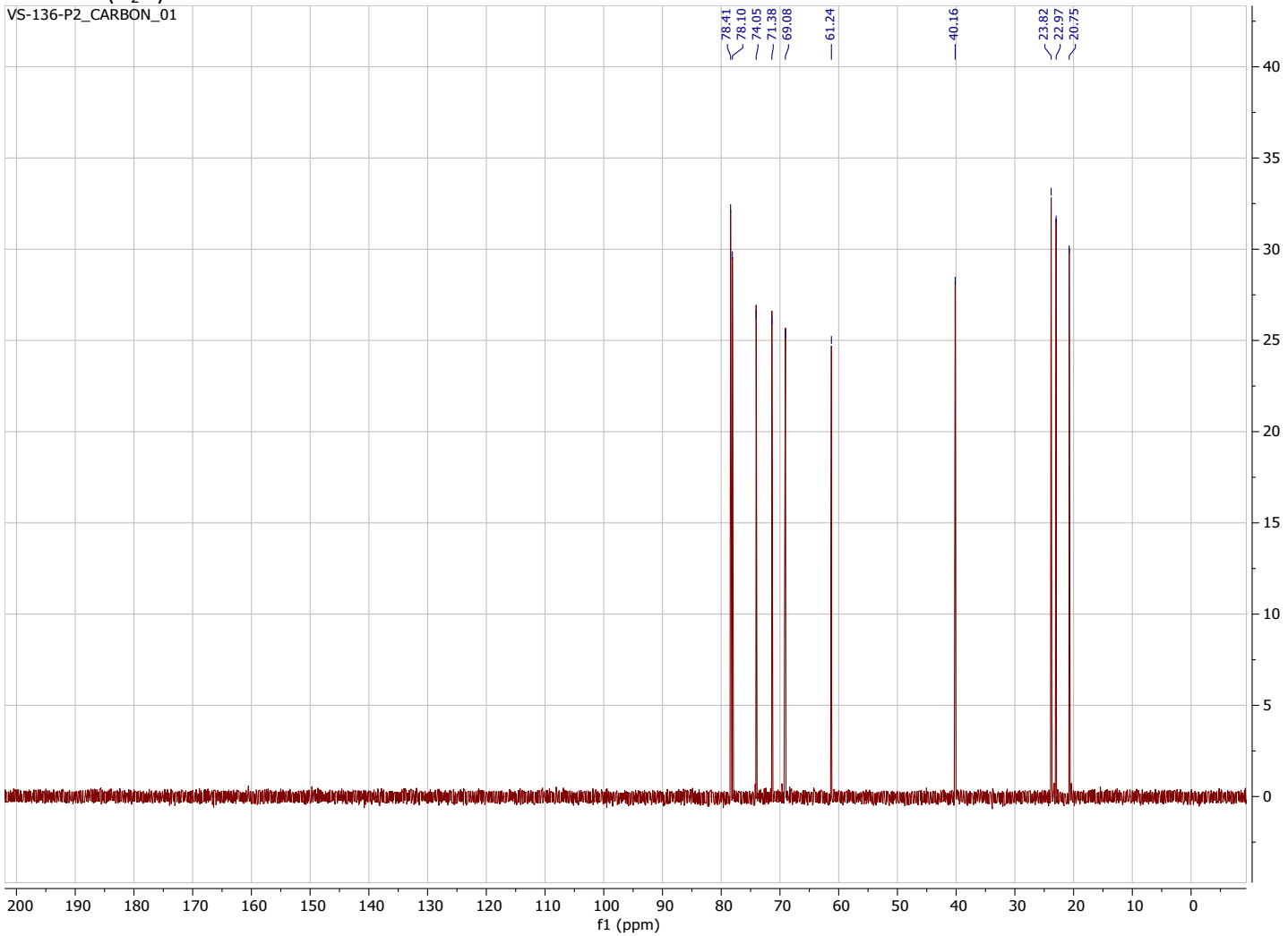
OH
O
HO
OH

^{13}C-NMR (D_2O)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69.08
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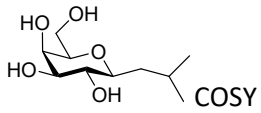
20.75
f1 (ppm)



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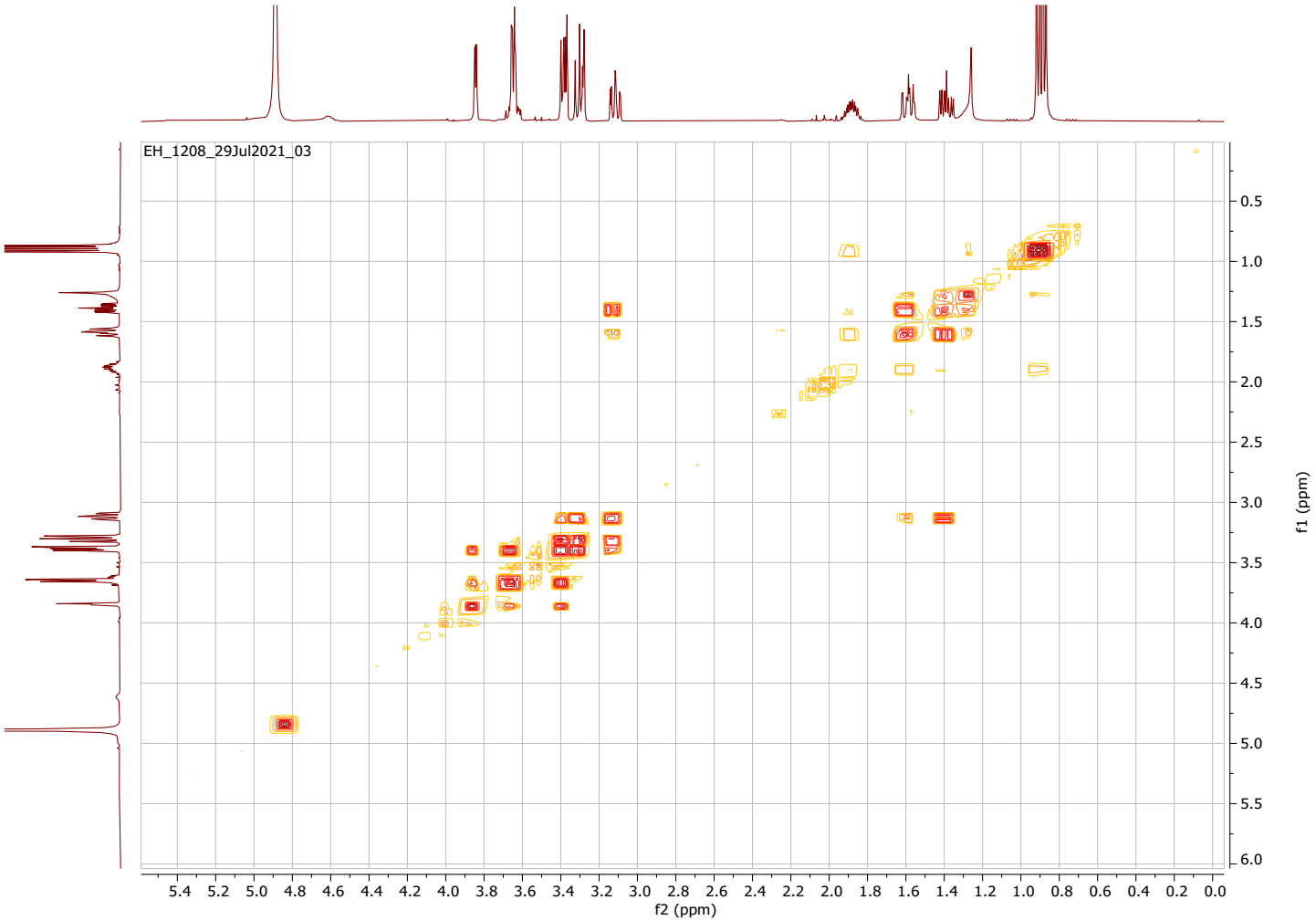

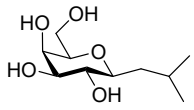

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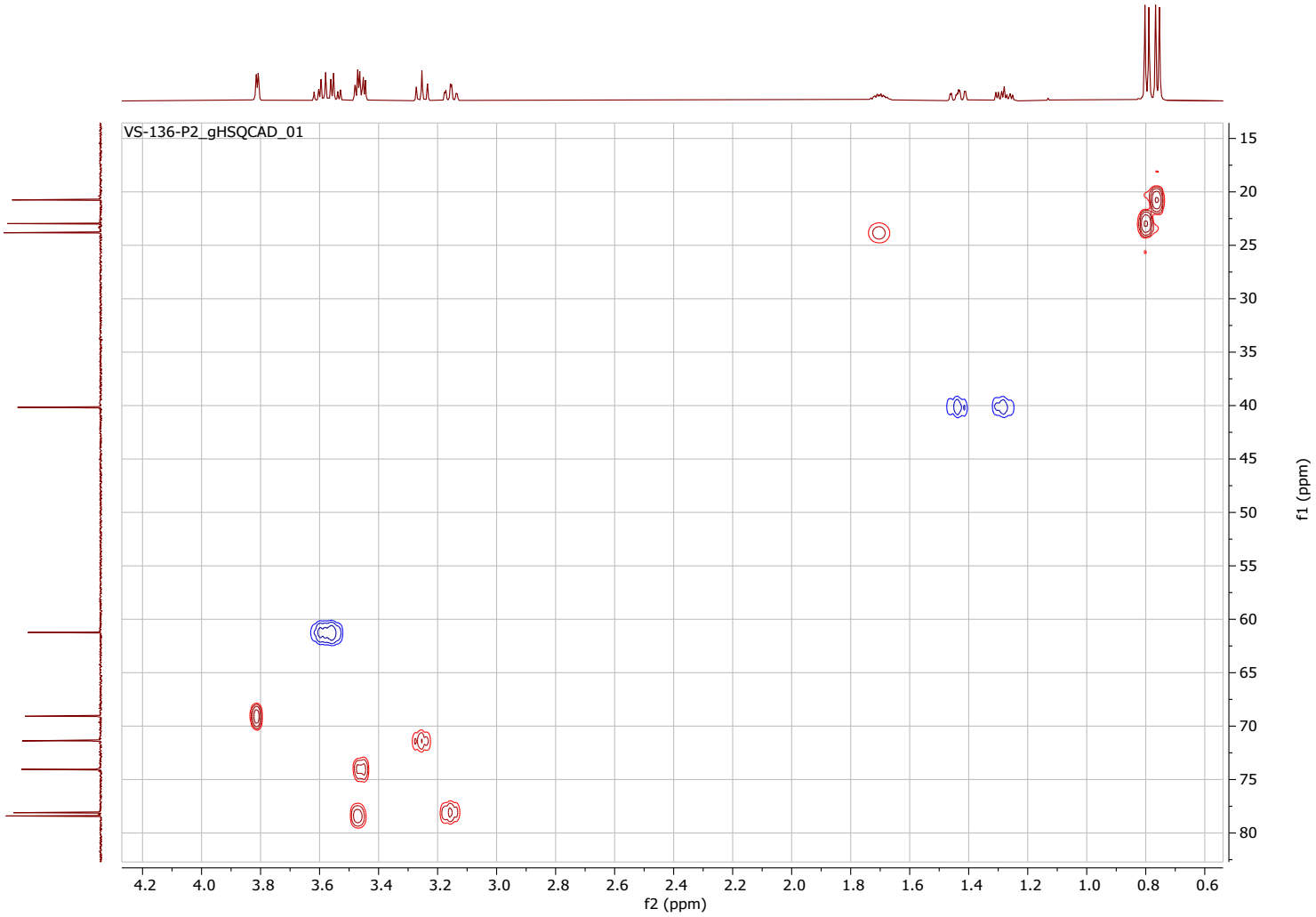

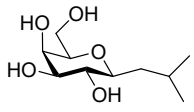

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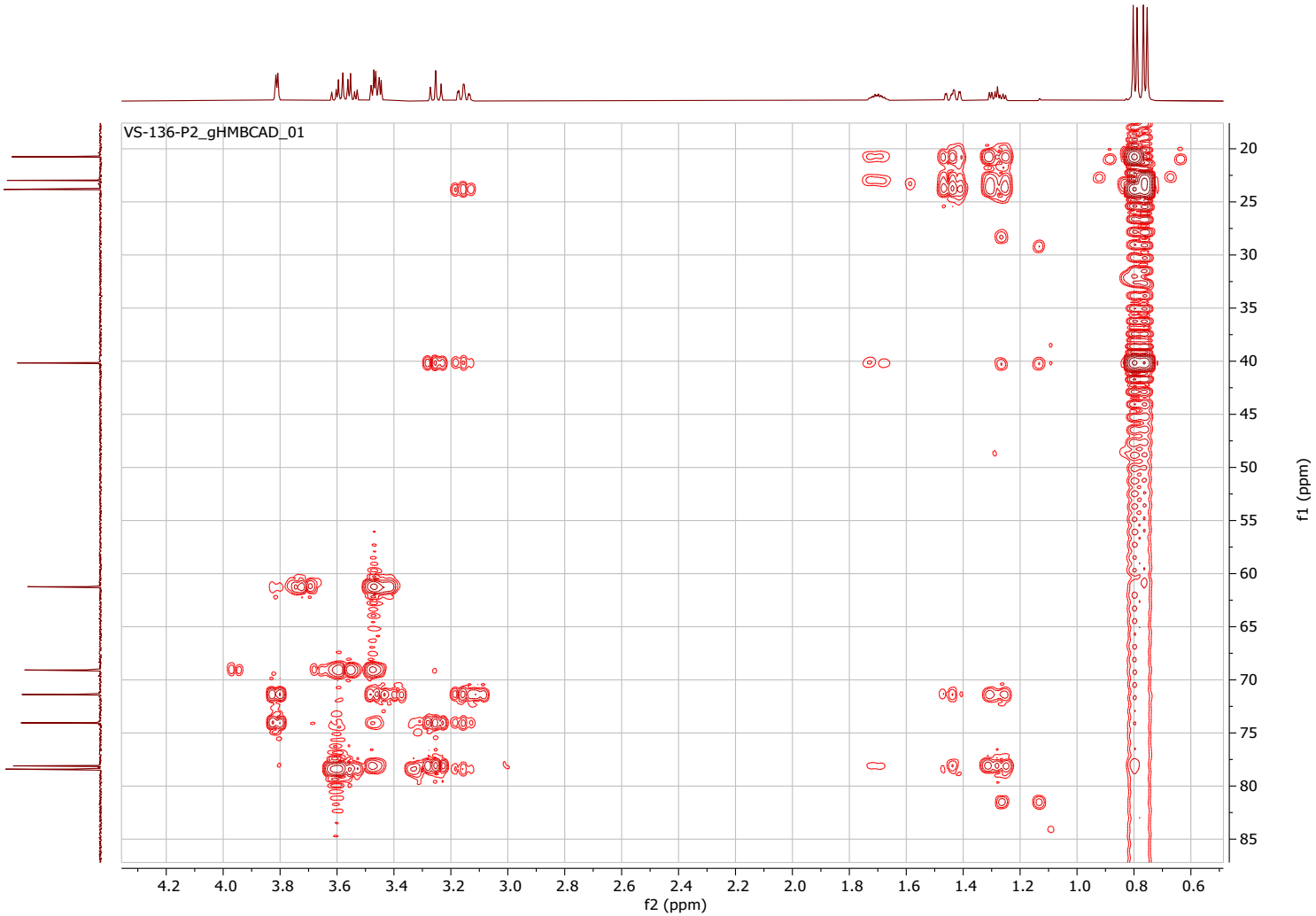

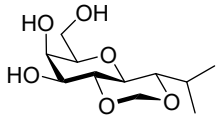
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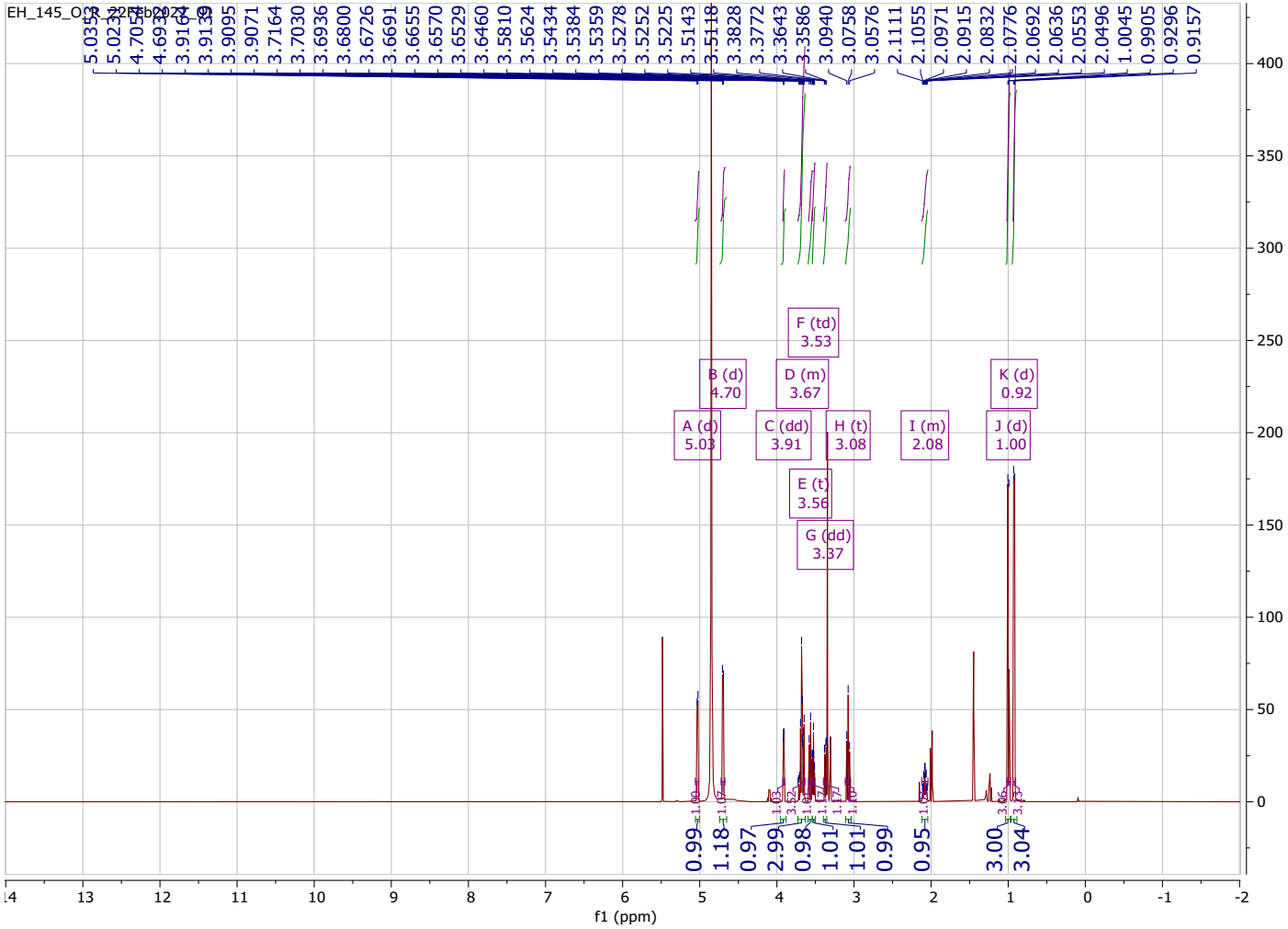
O
HO
O
O

^{1}H-NMR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3.6726
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3.6655
3.6570
3.6529
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3.5624
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3.5359
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3.5225
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B (d) 4.70
D (m) 3.67
K (d) 0.92
A (d) 5.03
C (dd) 3.91
H (t) 3.08
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J (d) 1.00
E (t) 3.56
G (dd) 3.37
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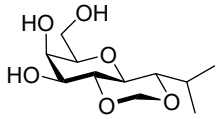
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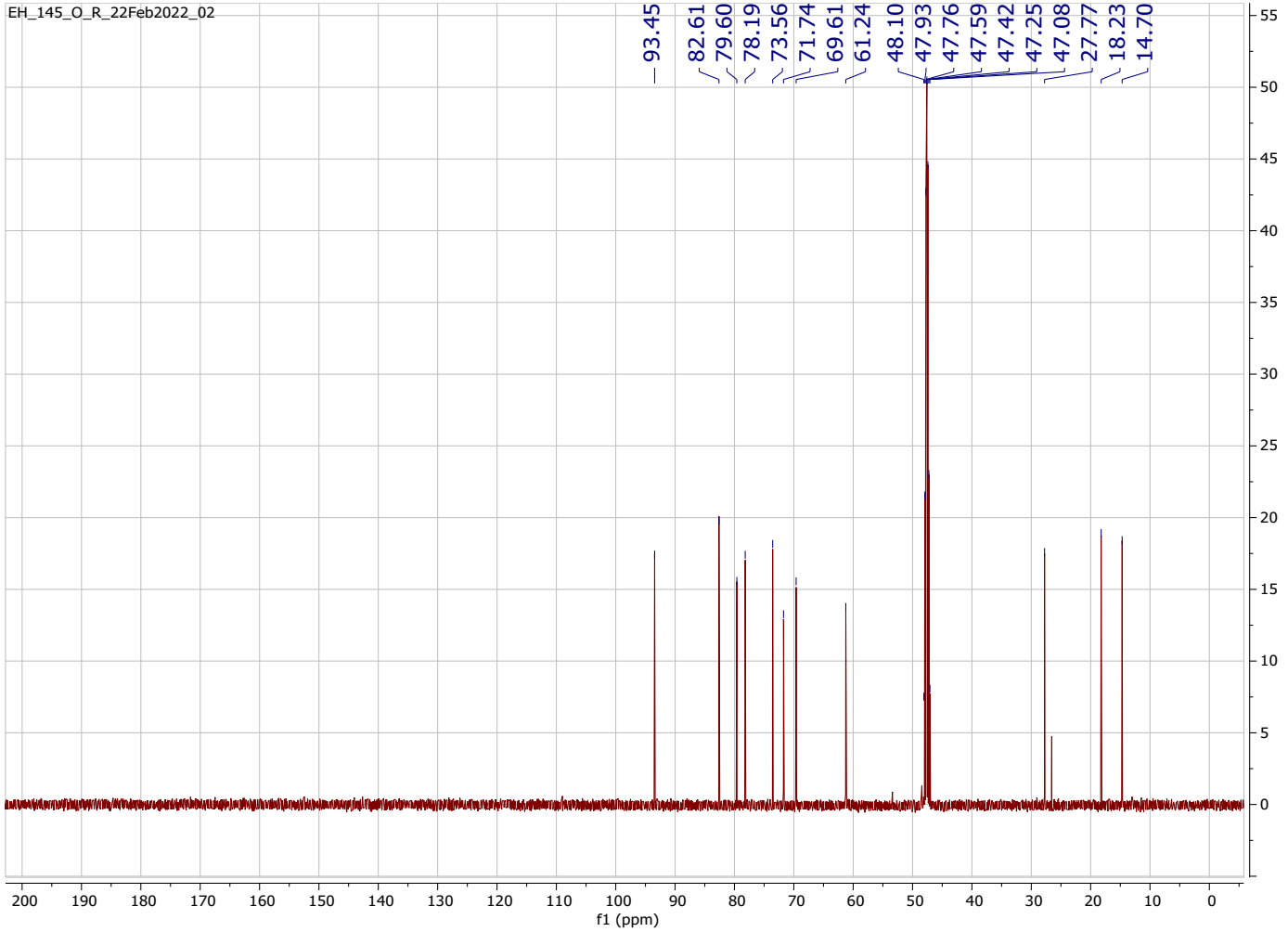
O
HO
O
O

^{13}C-NMR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71.74
69.61
61.24
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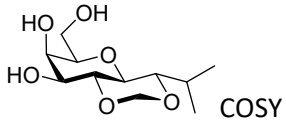
f1 (ppm)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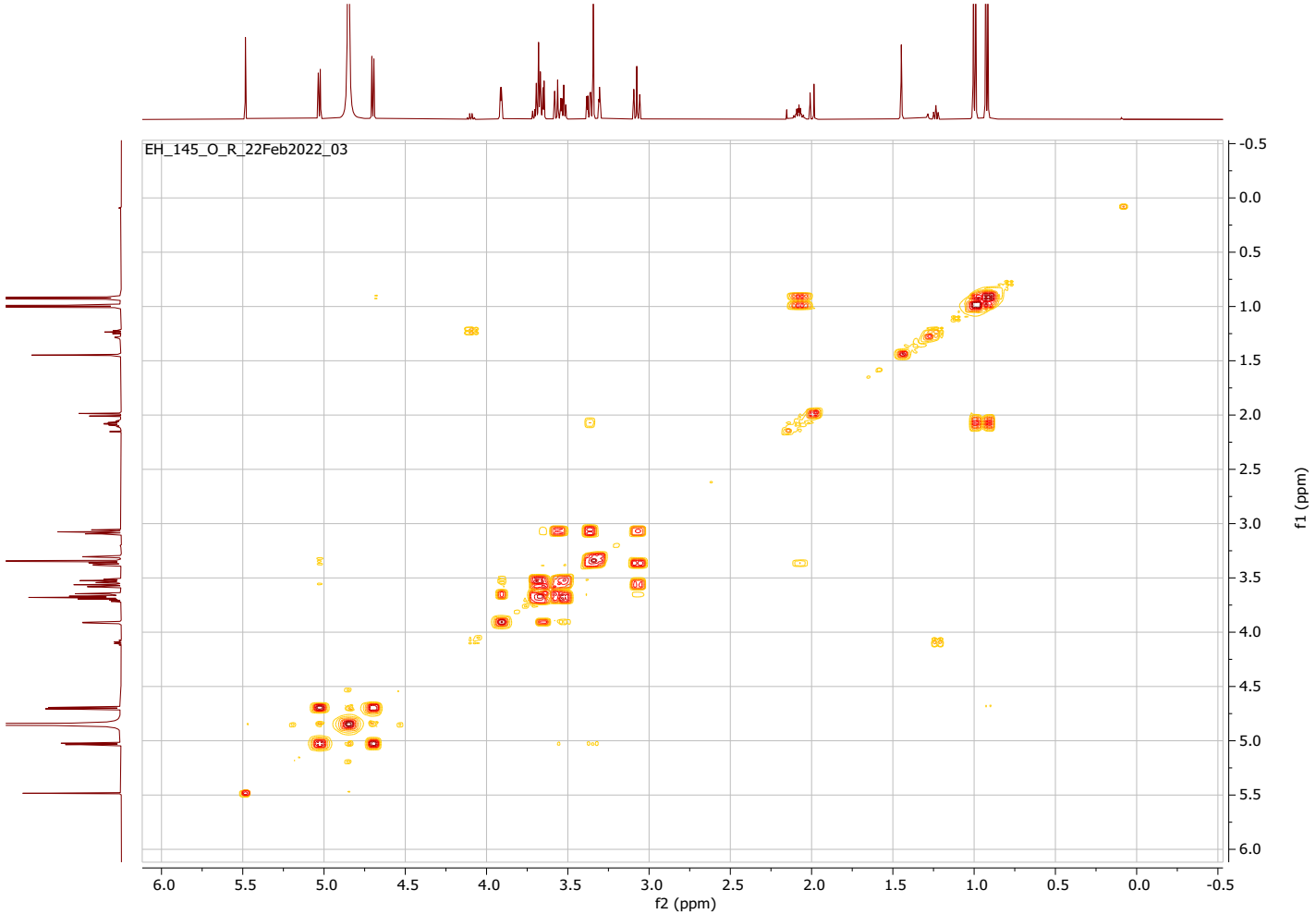
O
HO
O
O

COSY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f2 (ppm)
f1 (ppm)
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5.0
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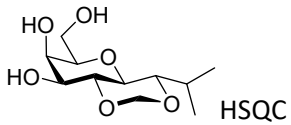
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5



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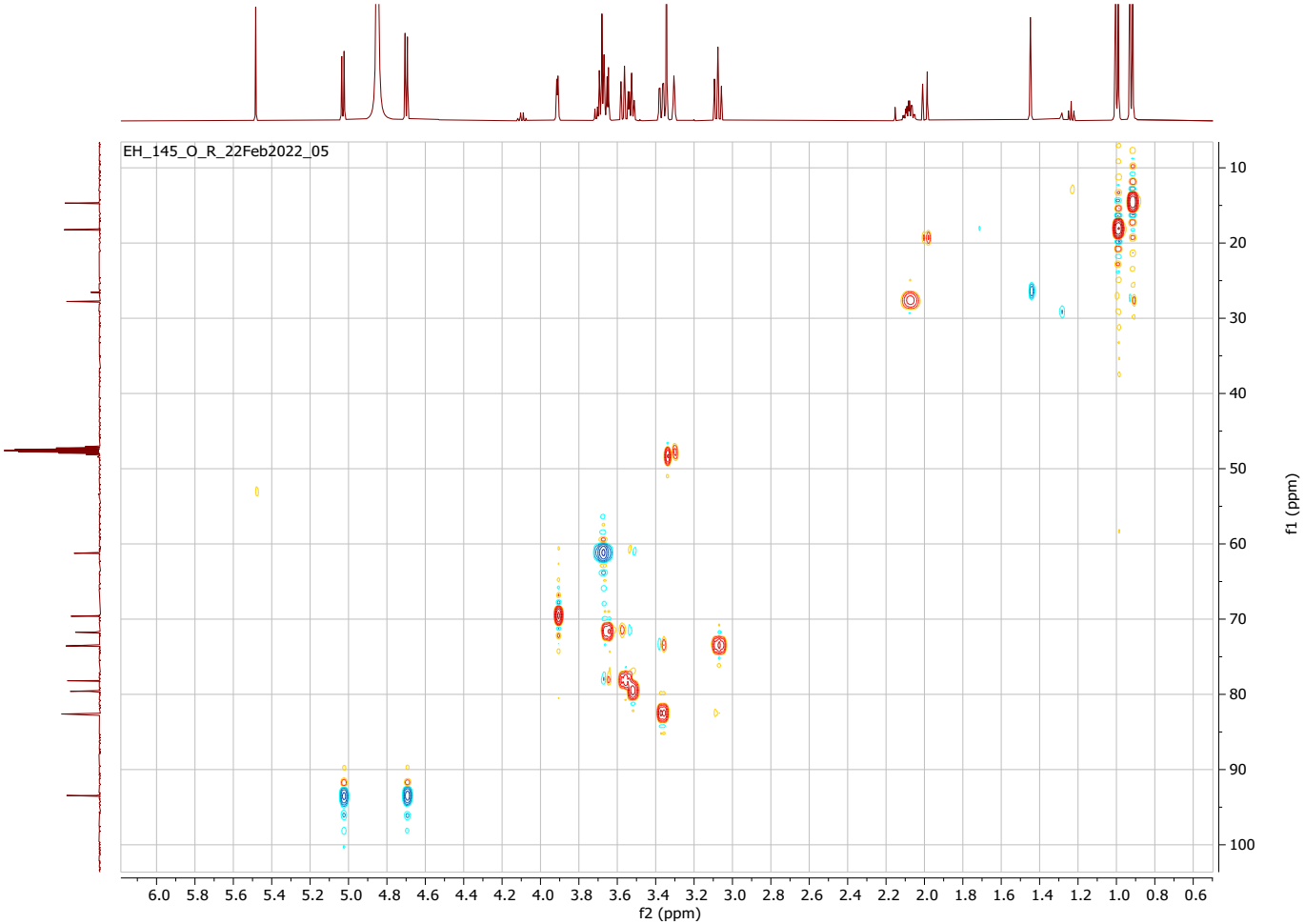
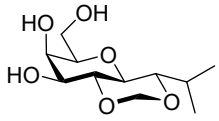
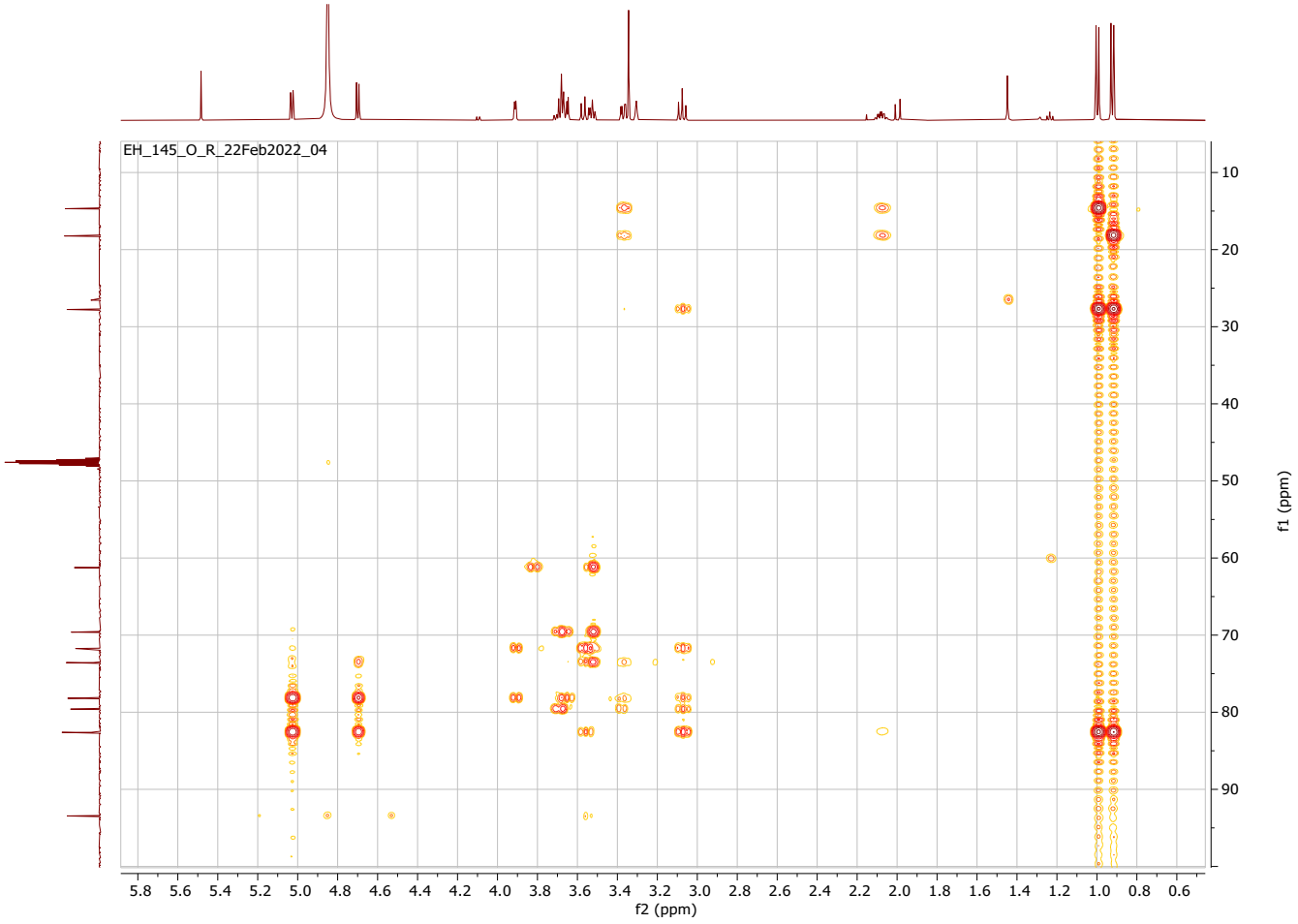



HO
OH
O
HO
O
O

HMBC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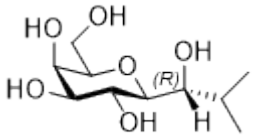
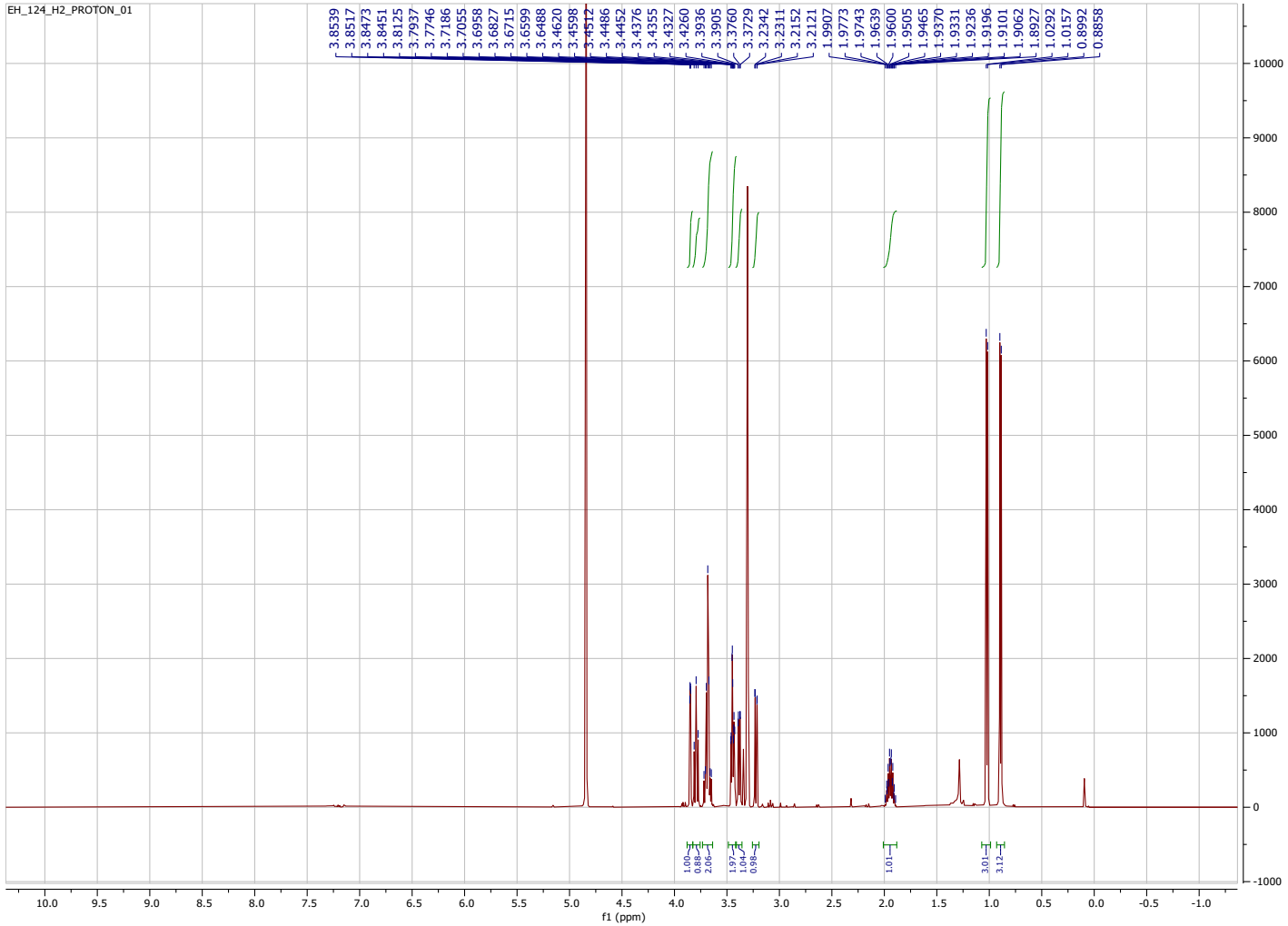
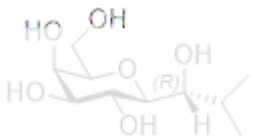
20
30
40
50
60
70
80
90
f1 (ppm)
5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8 1.6 1.4 1.2 1.0 0.8 0.6
f2 (ppm)

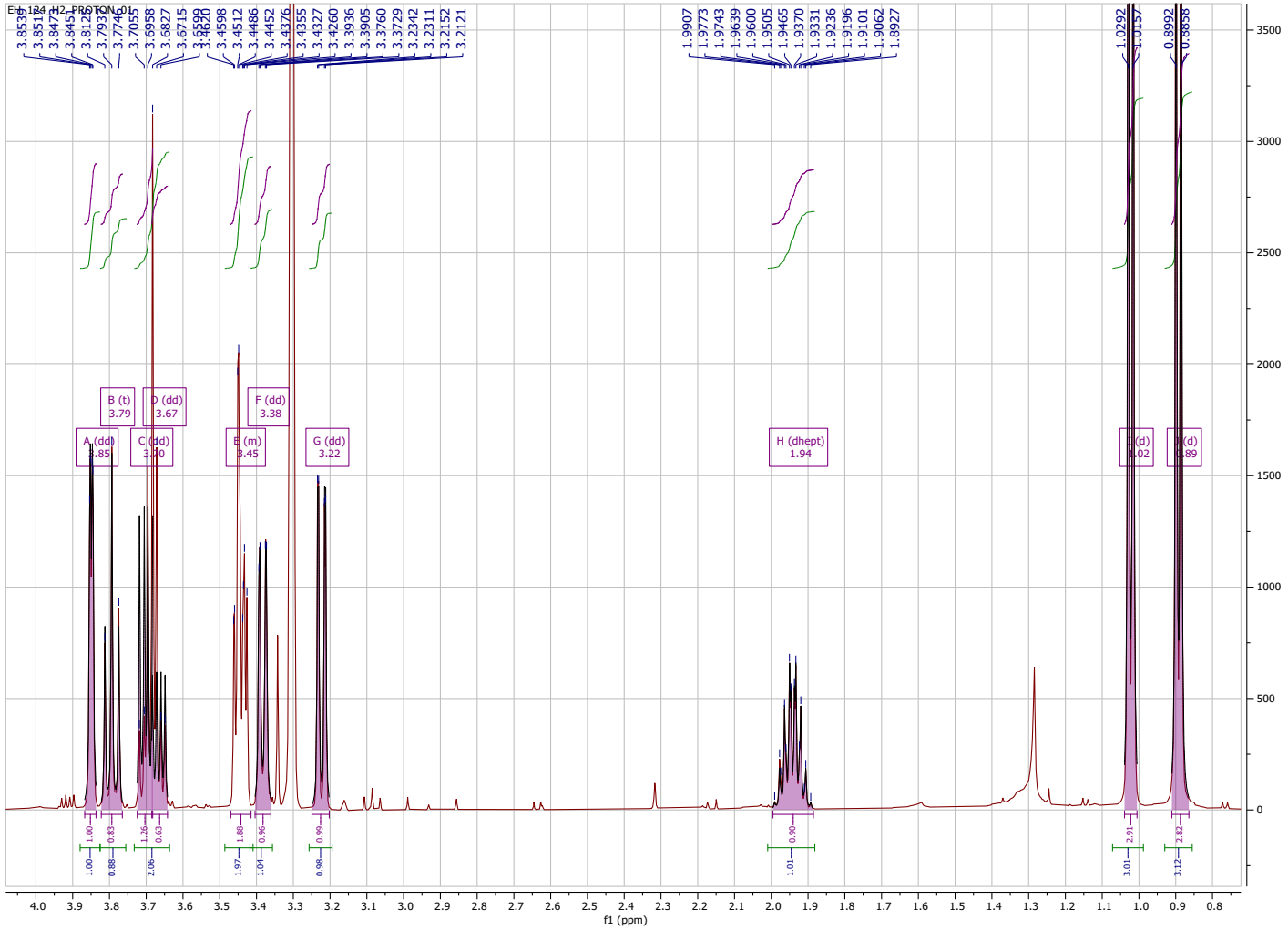

HO
OH
O
OH
(R)
HO
OH
H

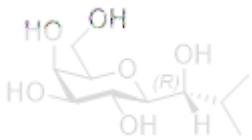
^{1}H-NMR **4**


EH_124_H2_PROTON_01
3.8539
3.8517
3.8473
3.8451
3.8125
3.7937
3.7746
3.7186
3.7055
3.6958
3.6827
3.6715
3.6599
3.6488
3.4620
3.4598
3.4512
3.4486
3.4452
3.4376
3.4355
3.4327
3.4260
3.3936
3.3905
3.3760
3.3729
3.2342
3.2311
3.2152
3.2121
1.9907
1.9773
1.9743
1.9639
1.9600
1.9505
1.9465
1.9370
1.9331
1.9236
1.9196
1.9101
1.9062
1.8927
1.0292
1.0157
0.8992
0.8858
1.00
0.88
2.06
1.97
1.04
0.98
1.01
3.01
3.12
f1 (ppm)

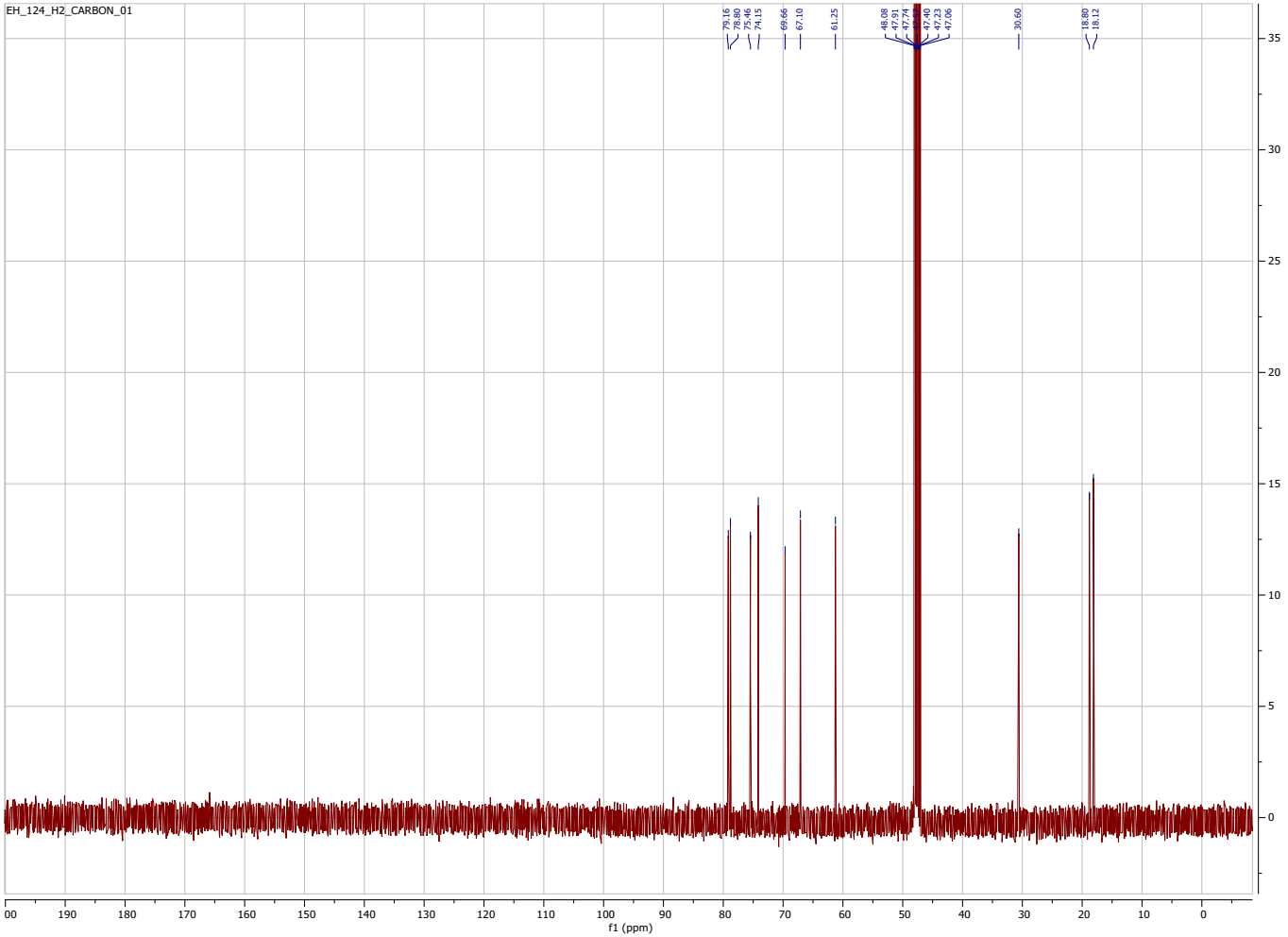

HO
OH
O
OH
HO
(R)
OH
H

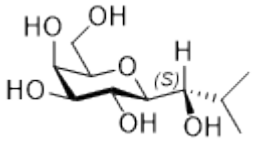
^{1}H-NMR **4**


B (t) 3.79
D (dd) 3.67
F (dd) 3.38
G (dd) 3.22
H (dhept) 1.94
f1 (ppm)

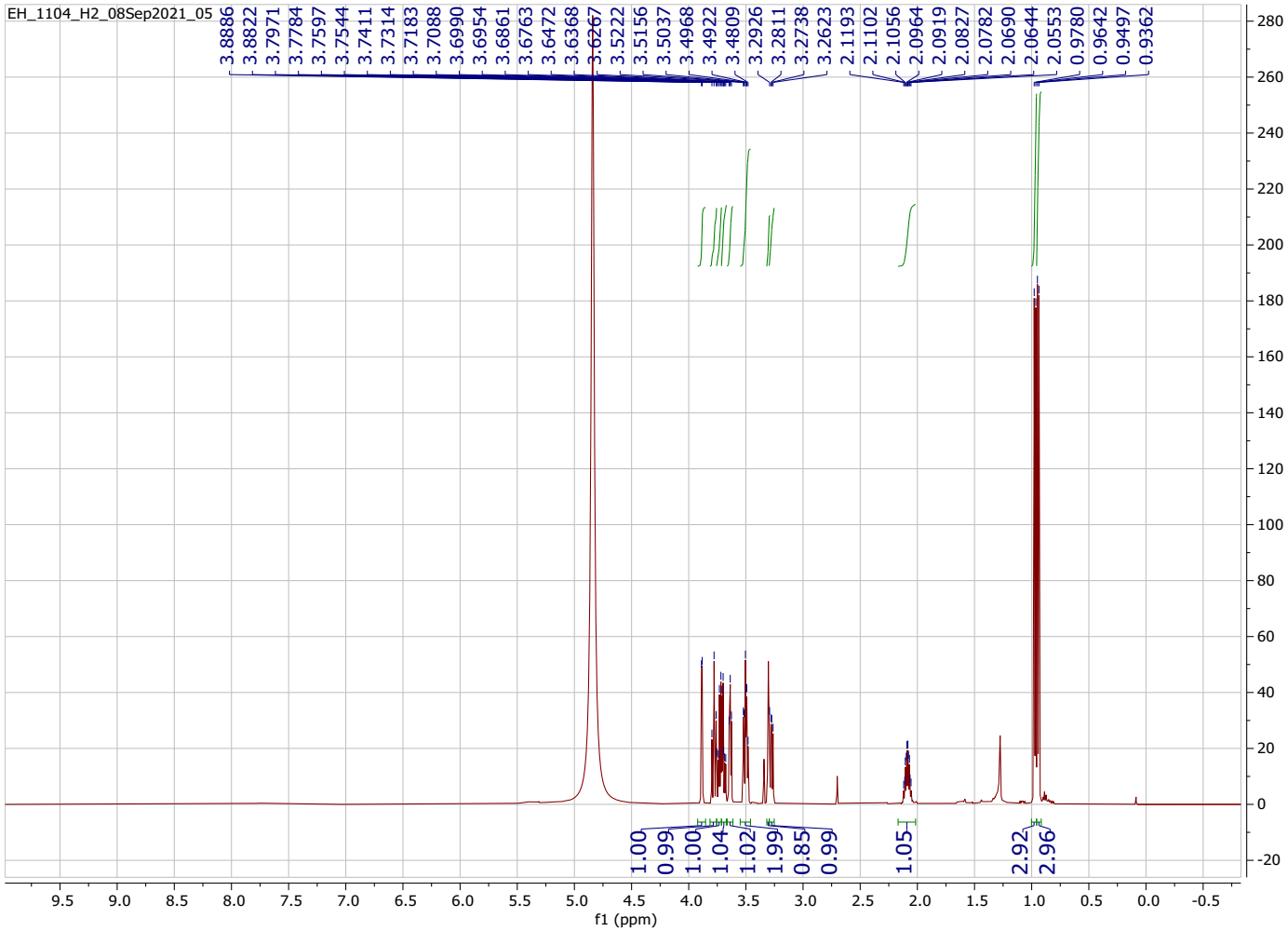

HO
OH
O
OH
HO
(R)
OH
H

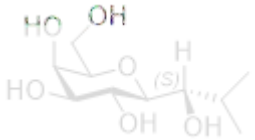
^{13}C-NMR **4**


EH_124_H2_CARBON_01
79.16
78.86
75.46
74.15
69.66
67.10
61.25
48.08
47.91
47.74
47.40
47.23
47.06
30.60
18.80
18.12
35
30
25
20
15
10
5
0
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

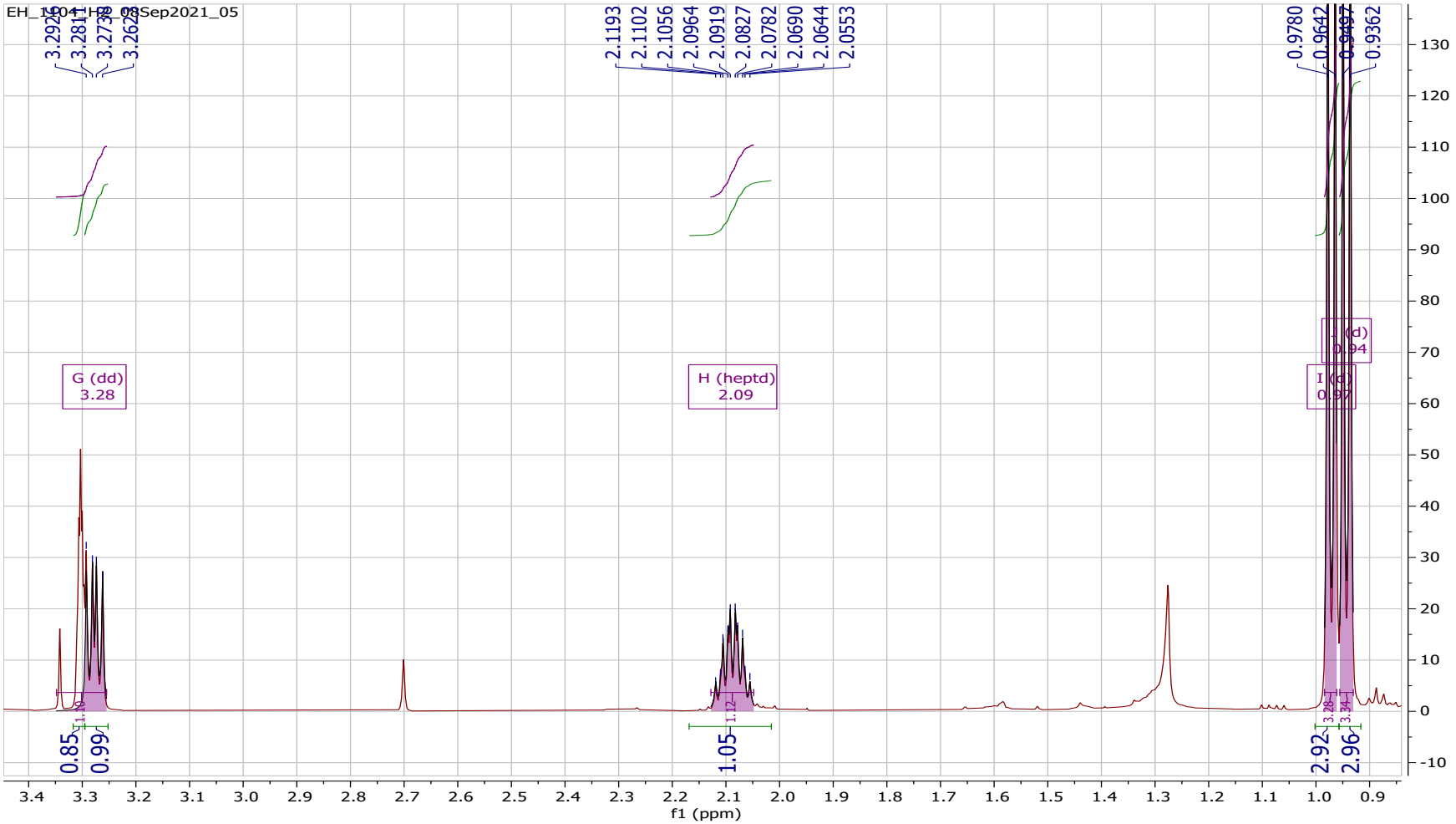


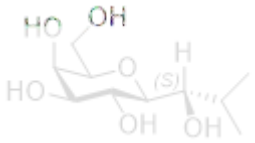
^{1}H-NMR **5** (CD_3OD)



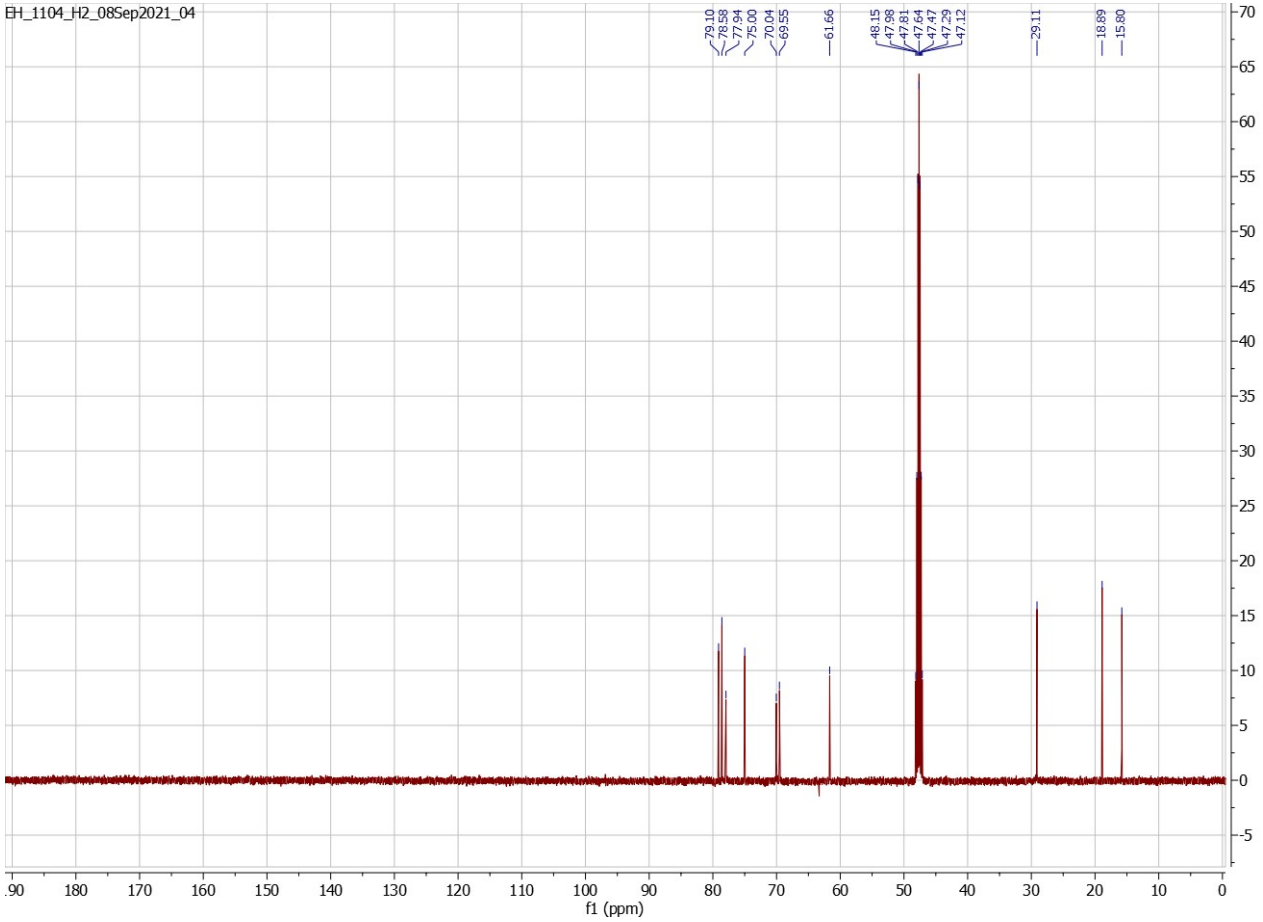


^{1}H-NMR **5**

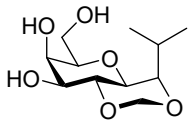

3.2924
3.2814
3.2738
3.2622
2.1193
2.1102
2.1056
2.0964
2.0919
2.0827
2.0782
2.0690
2.0644
2.0553
0.9780
0.9642
0.9497
0.9362
G (dd)
3.28
H (heptd)
2.09
0.85
0.99
1.05
2.92
2.96
f1 (ppm)



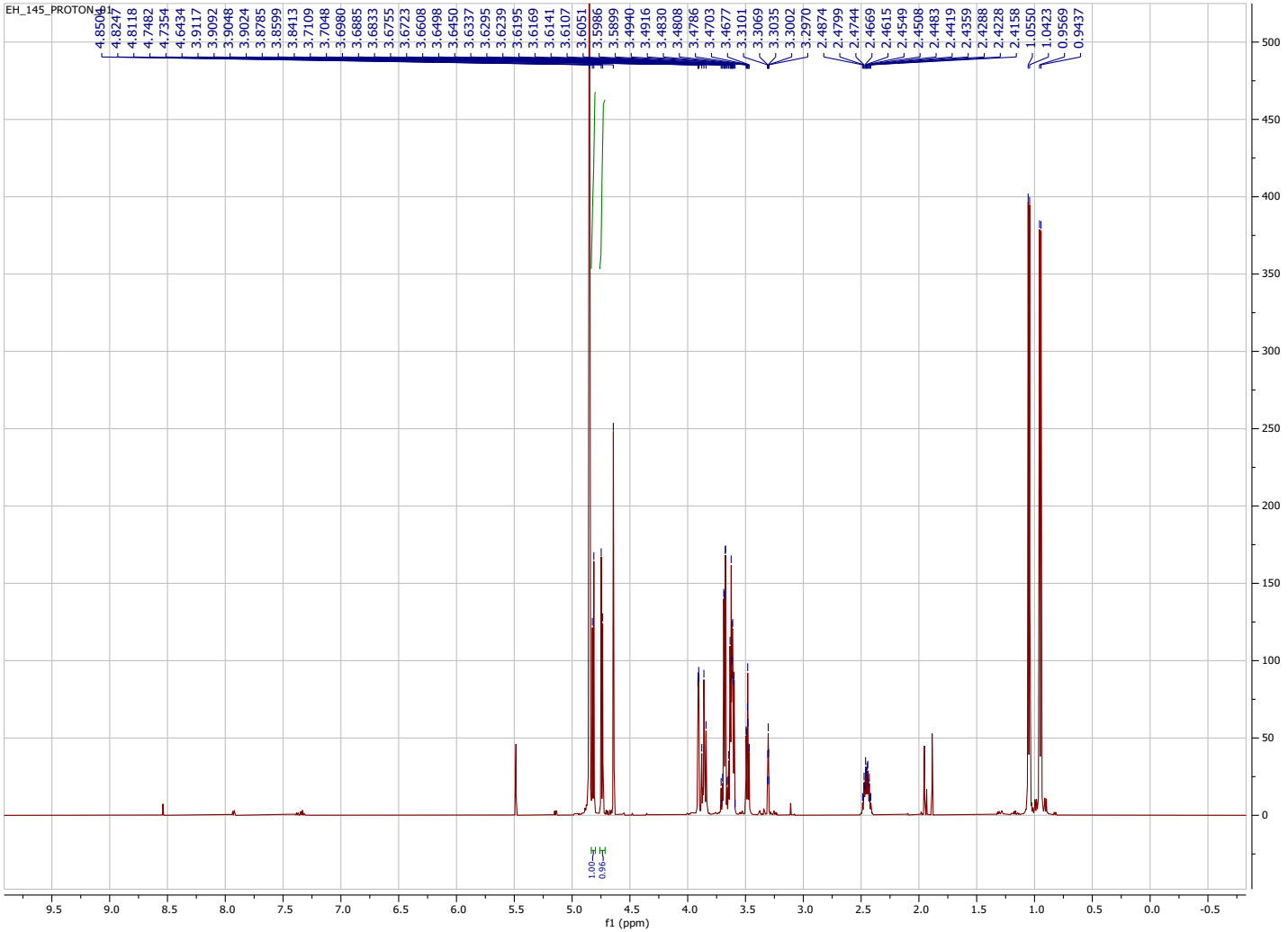
^{13}C-NMR **5**

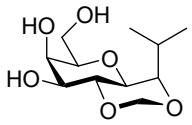

EH_1104_H2_08Sep2021_04
79.10
78.58
77.94
75.00
70.04
69.55
61.66
48.15
47.98
47.81
47.64
47.47
47.29
47.12
29.11
18.89
15.80
f1 (ppm)

Supporting Information

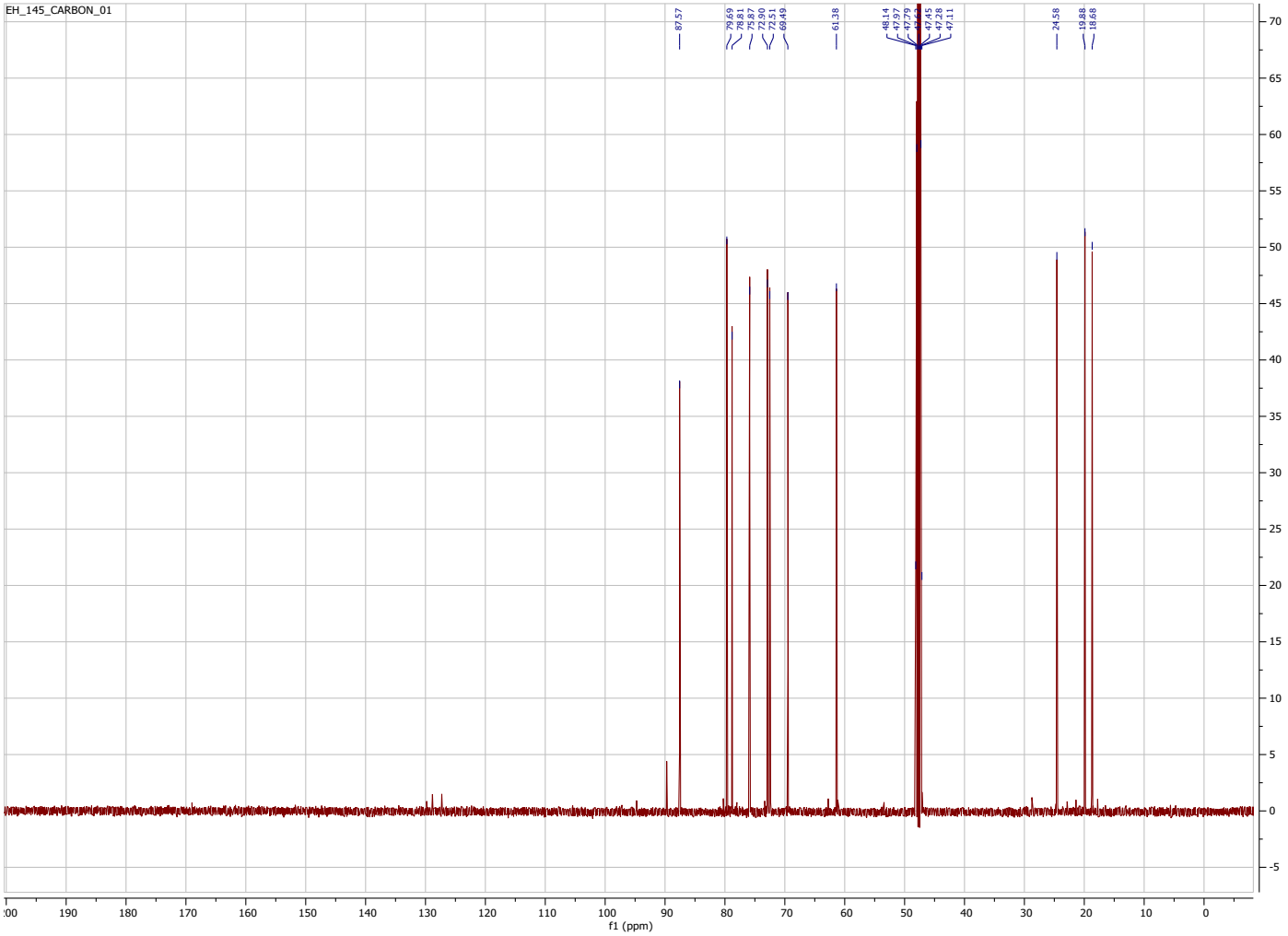


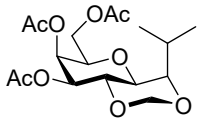
^{1}H-NMR **6**



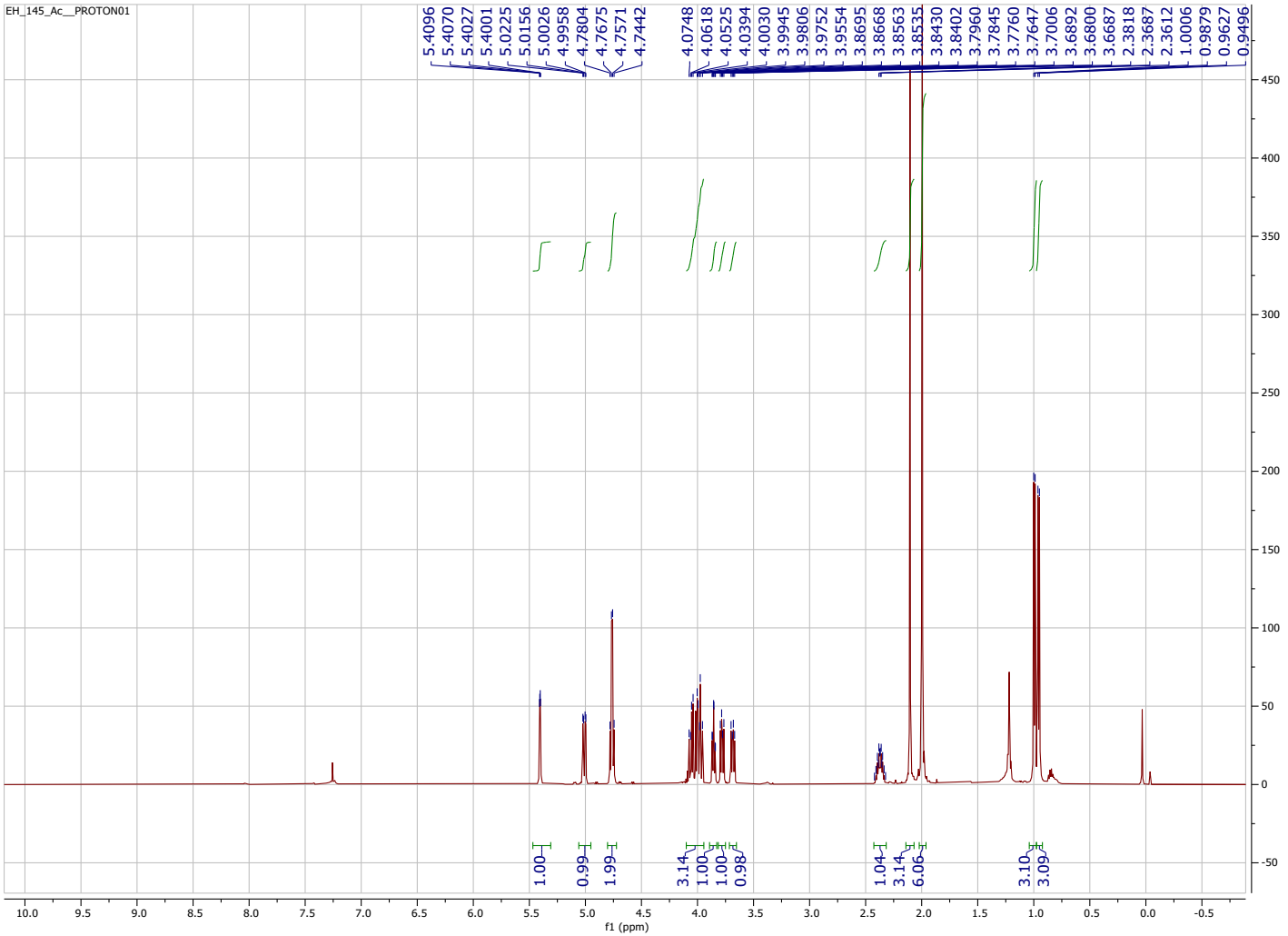

HO
OH
O
HO
O
O

^{13}C-NMR **6**

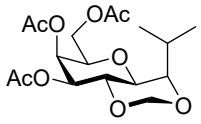

EH_145_CARBON_01
87.57
79.69
78.81
75.87
72.90
72.51
69.49
61.38
48.14
47.97
47.79
47.45
47.28
47.11
24.58
19.88
18.68
f1 (ppm)


AcO
OAc
O
AcO
O
O

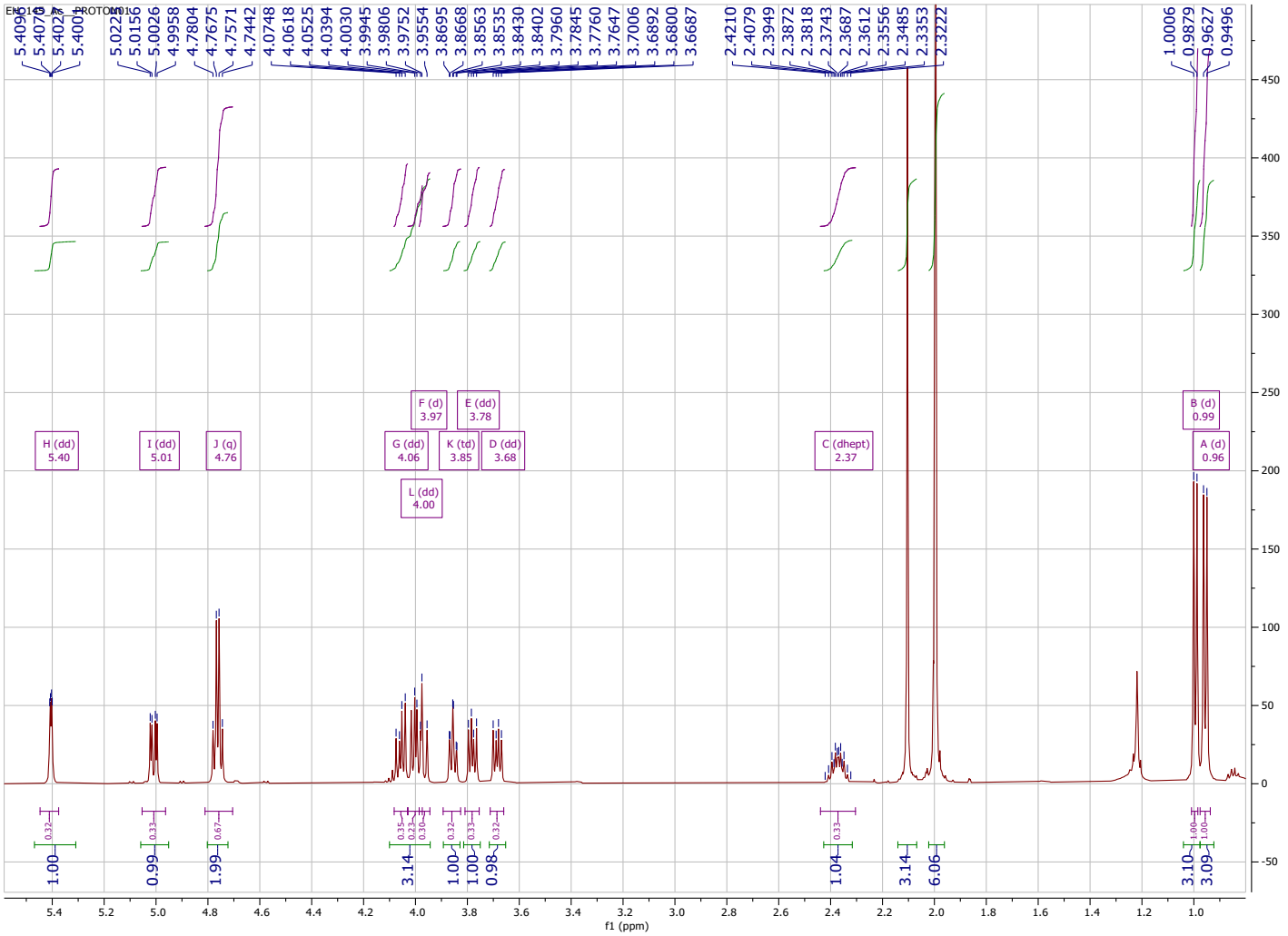
^{1}H-NMR

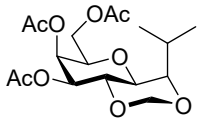

EH_145_Ac__PROTON01
5.4096
5.4070
5.4027
5.4001
5.0225
5.0156
5.0026
4.9958
4.7804
4.7675
4.7571
4.7442
4.0748
4.0618
4.0525
4.0394
4.0030
3.9945
3.9806
3.9752
3.9554
3.8695
3.8668
3.8563
3.8535
3.8430
3.8402
3.7960
3.7845
3.7760
3.7647
3.7006
3.6892
3.6800
3.6687
2.3818
2.3687
2.3612
1.0006
0.9879
0.9627
0.9496
1.00
0.99
1.99
3.14
1.00
1.00
0.98
1.04
3.14
6.06
3.10
3.09
f1 (ppm)

Supporting Information

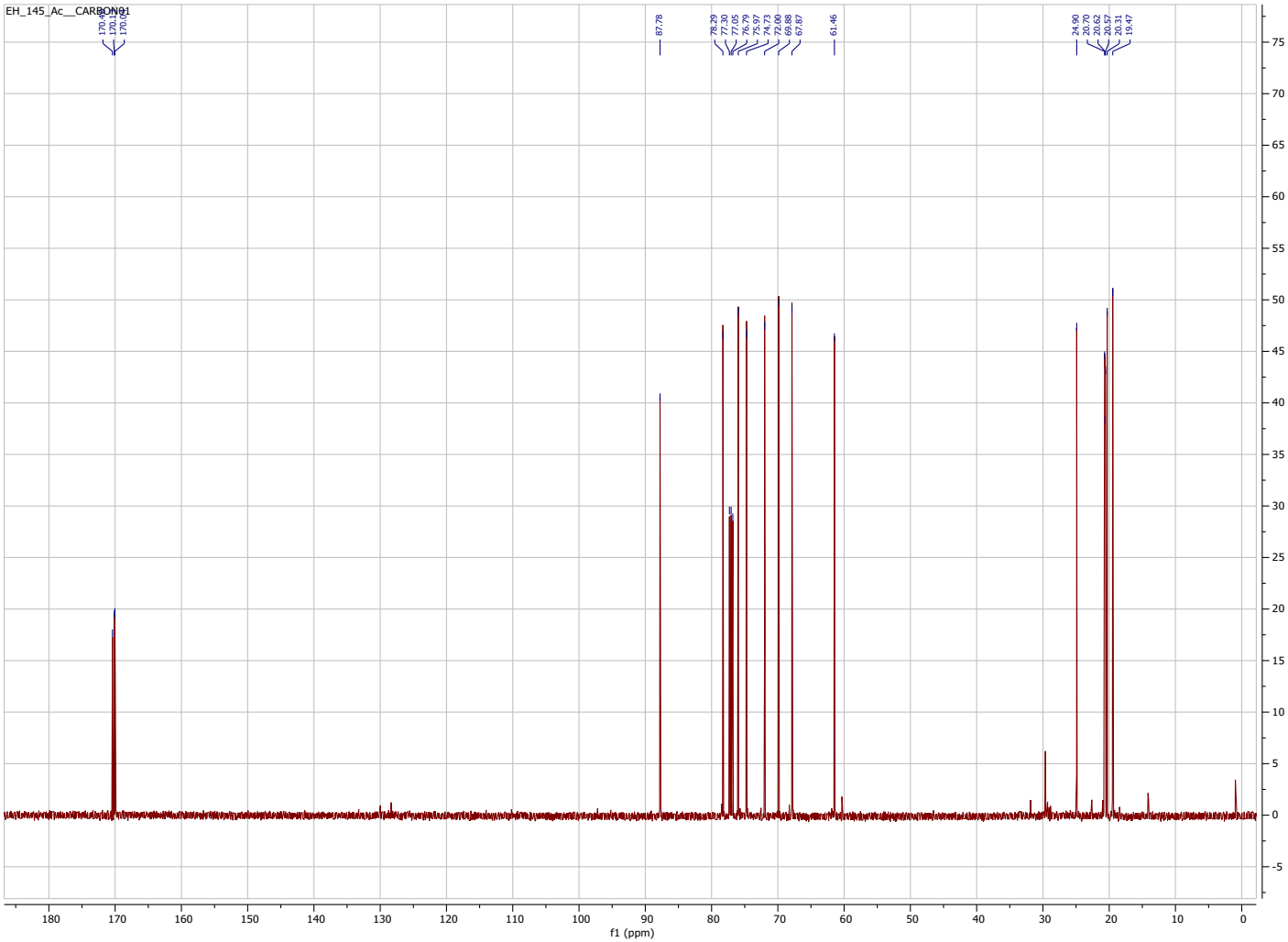


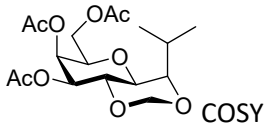
^{1}H-NMR



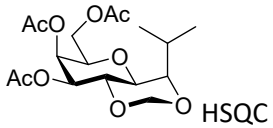

AcO
OAc
O
AcO
O
O

^{13}C-NMR

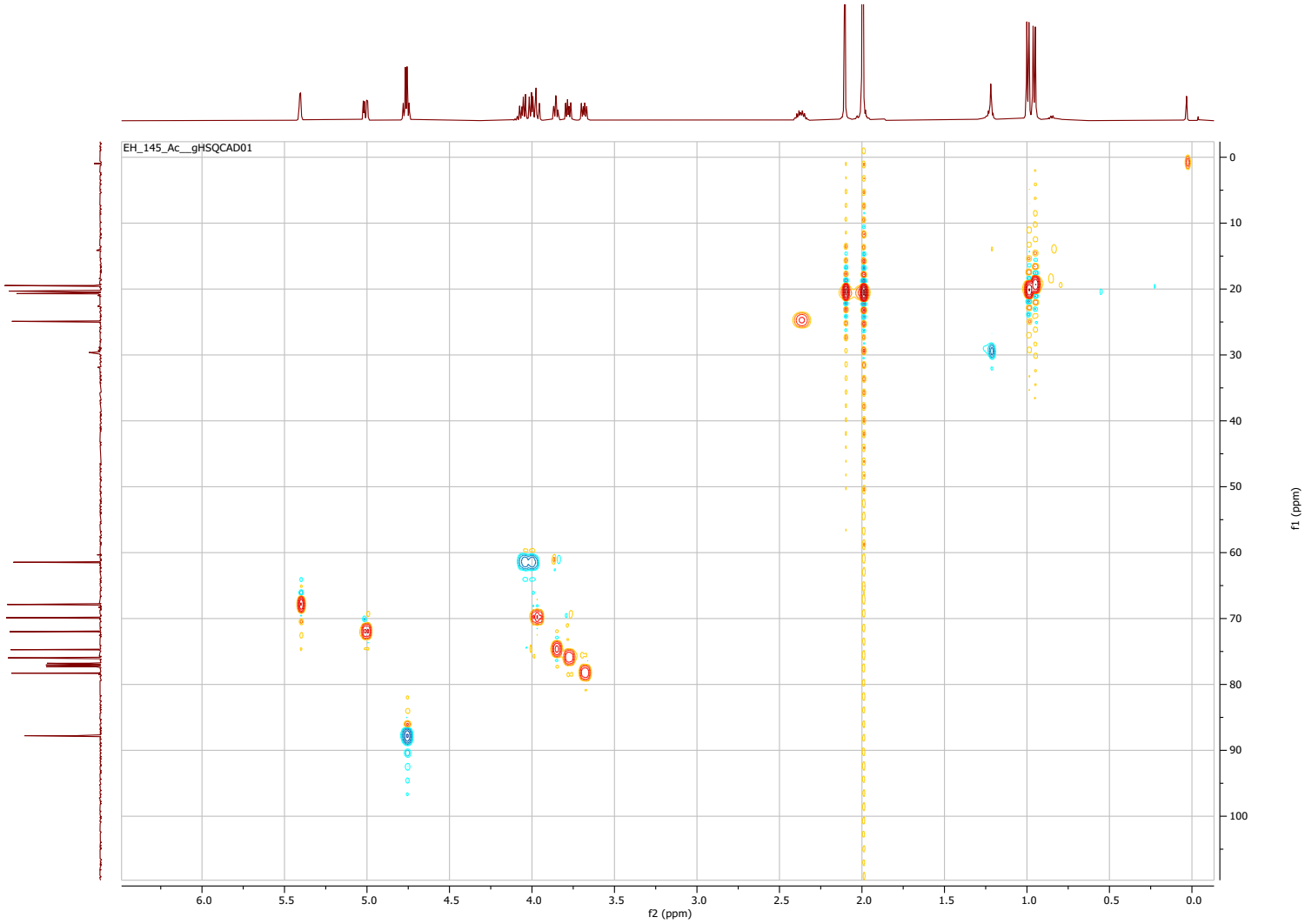

EH_145_Ac__CARBON_01
170.48
170.12
170.01
87.78
78.29
77.30
77.05
76.79
75.97
74.73
72.00
69.88
67.87
61.46
24.90
20.70
20.62
20.57
20.31
19.47
f1 (ppm)

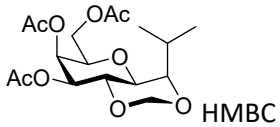


COSY

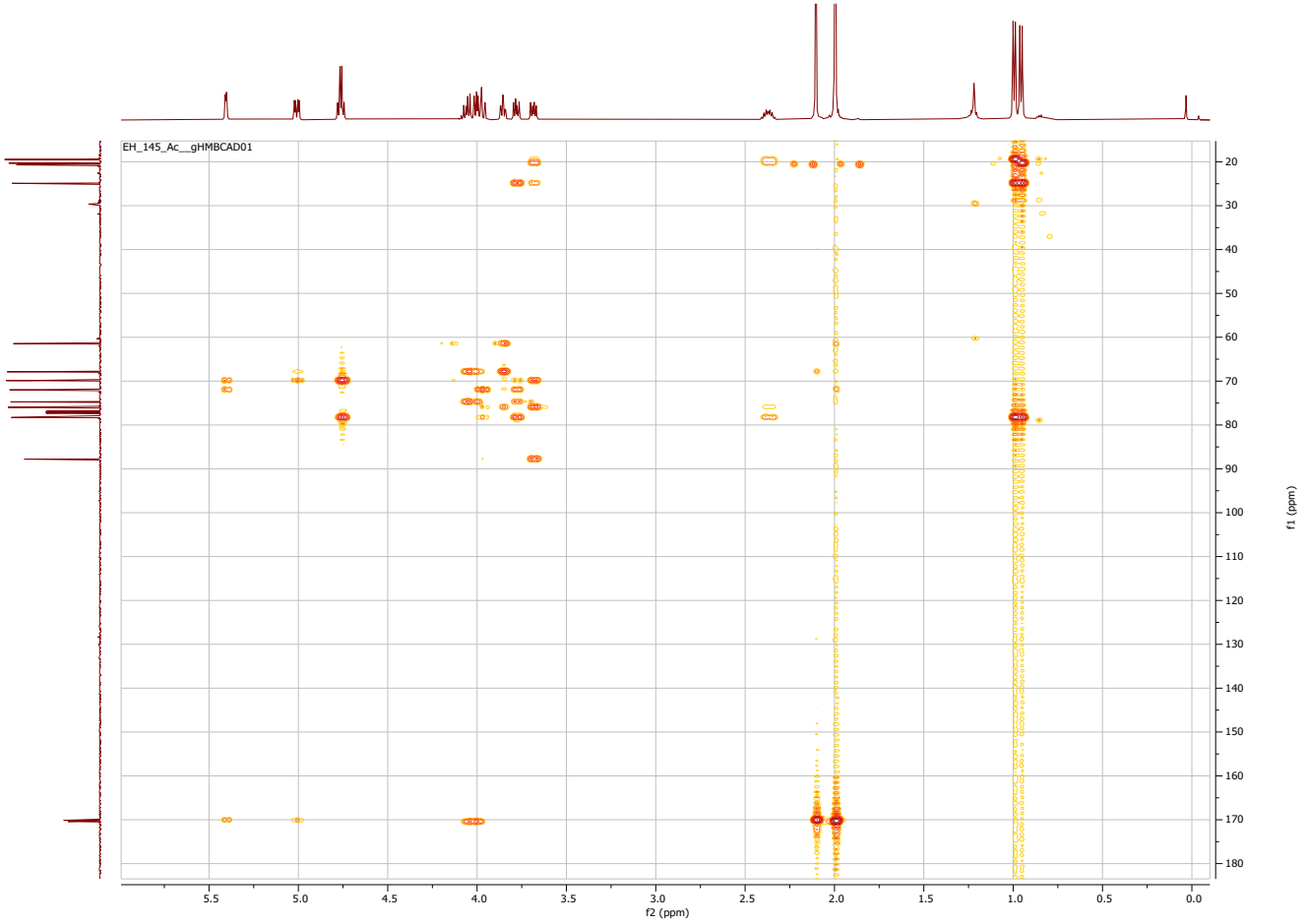


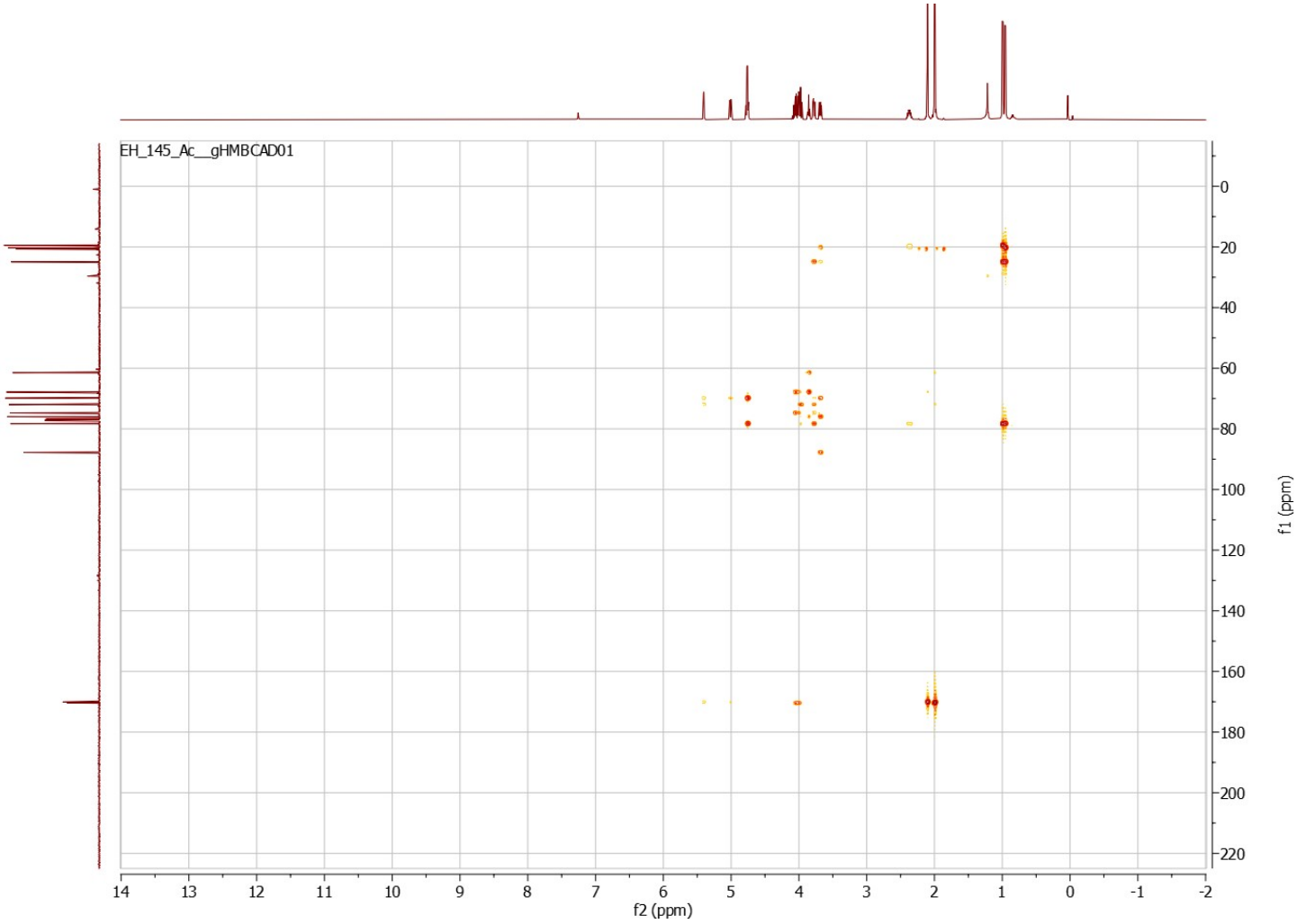
HSQC

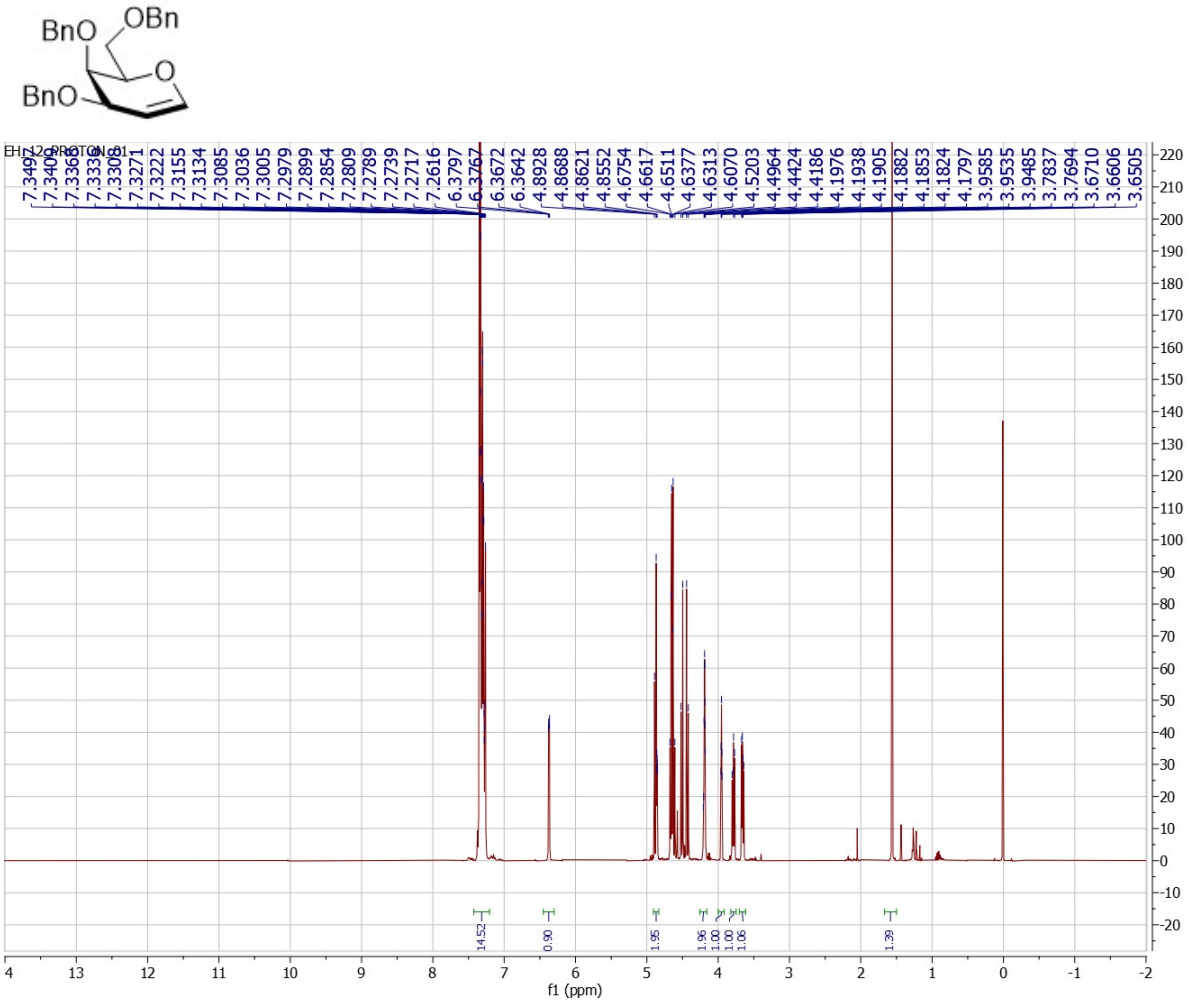


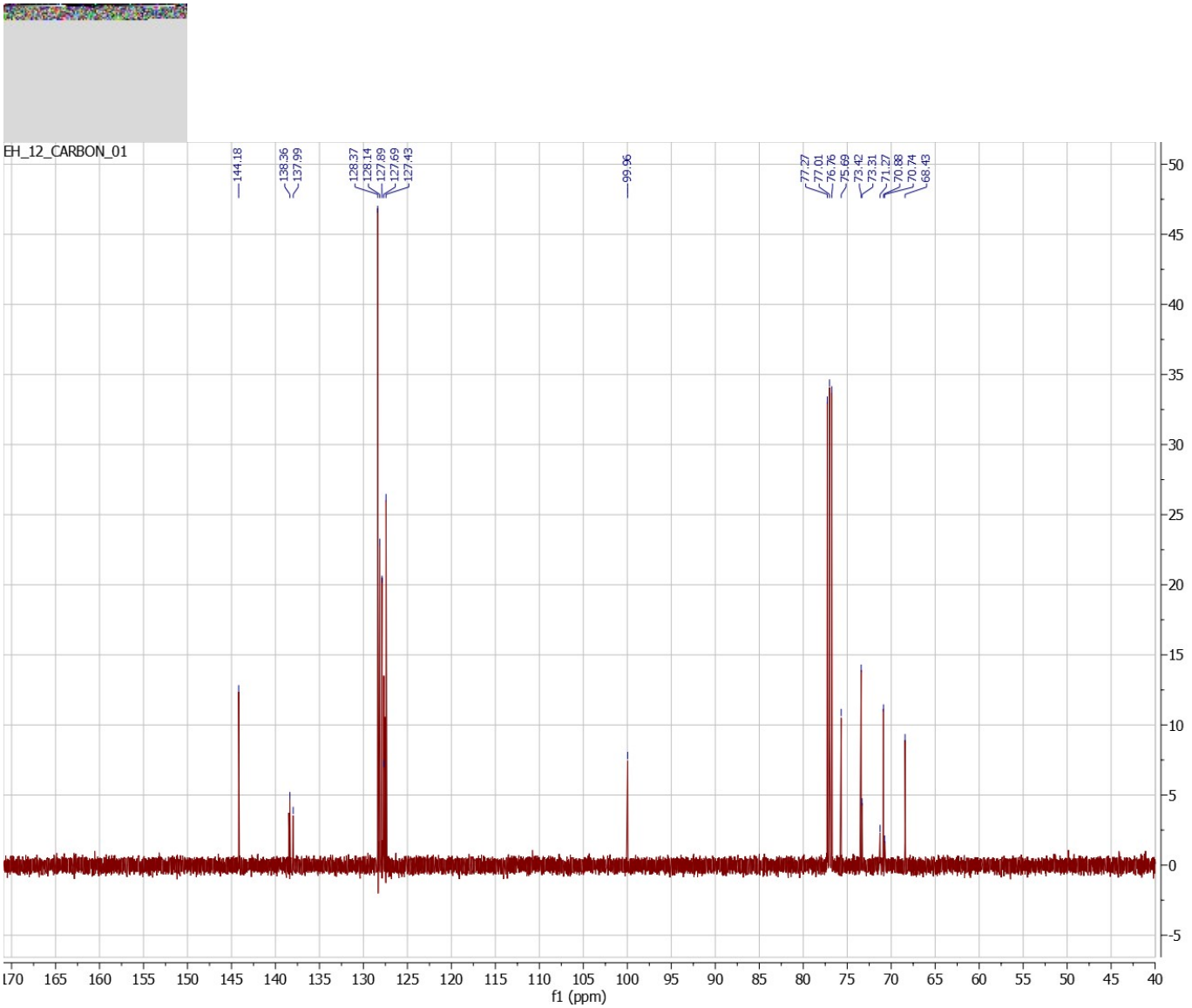


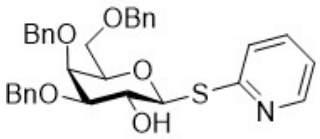
HMBC

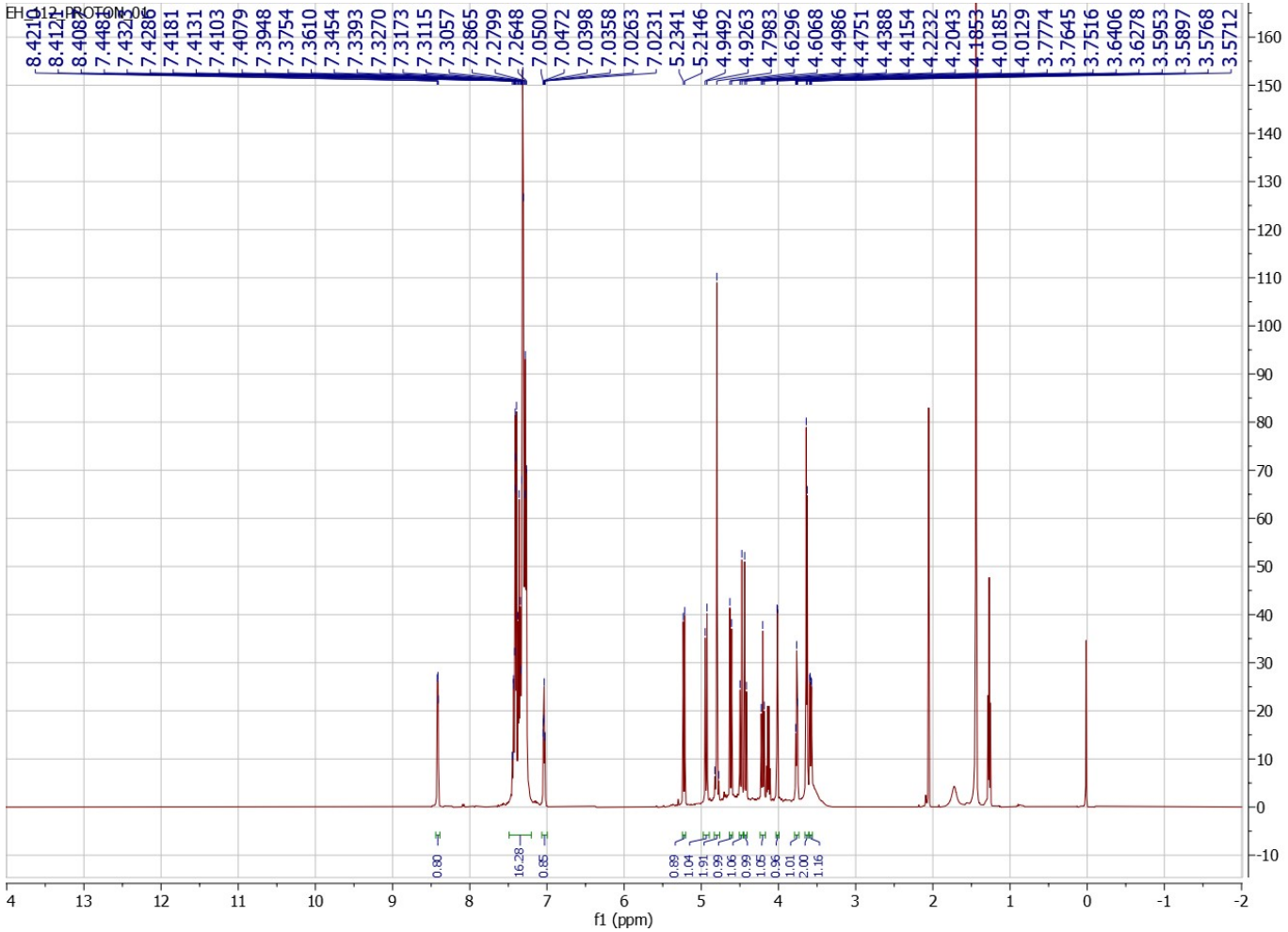


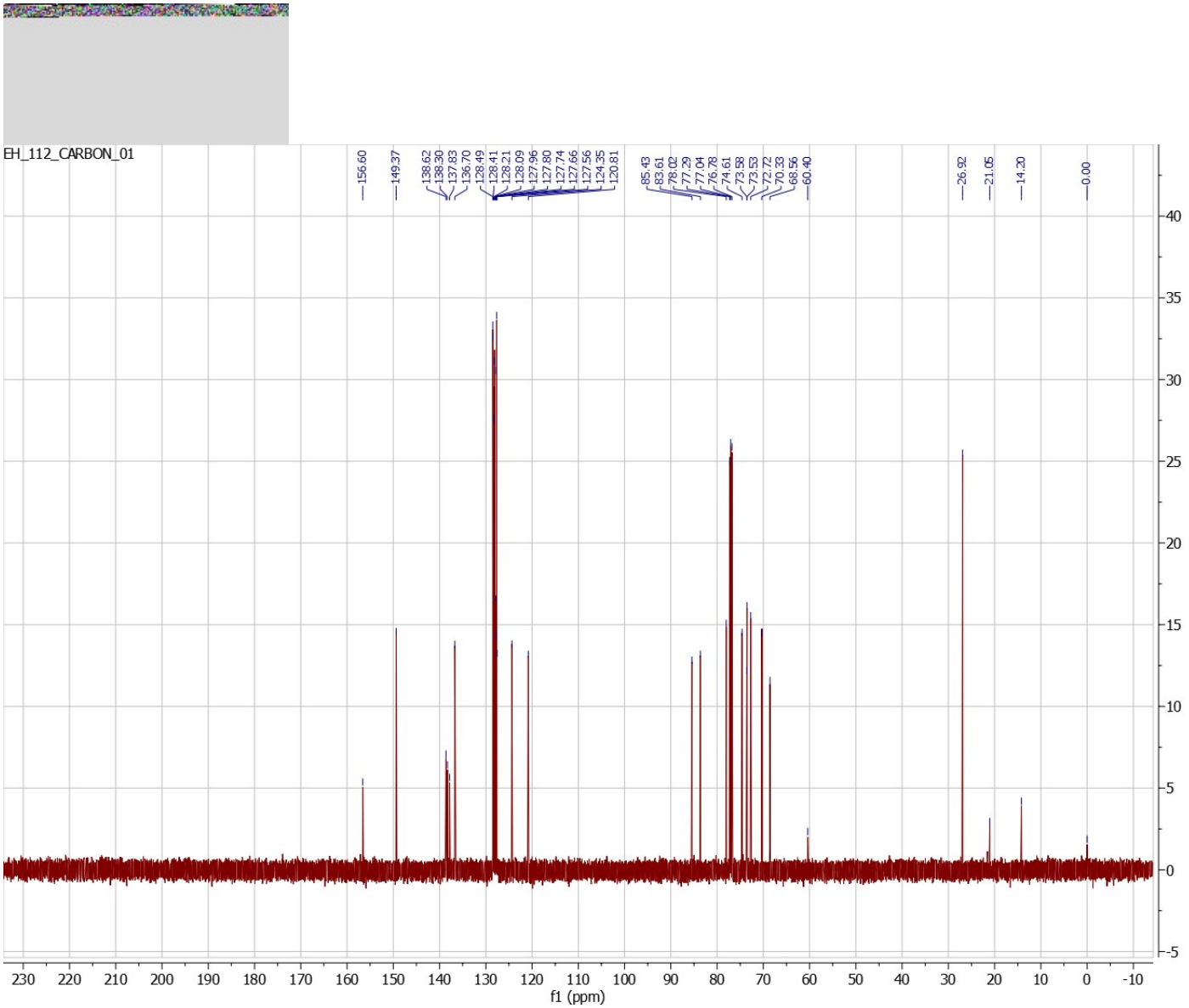

EH_145_Ac__gHMBCAD01
f2 (ppm)
f1 (ppm)

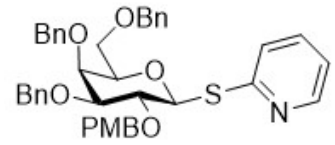

BnO
OBn
O
BnO
f1 (ppm)

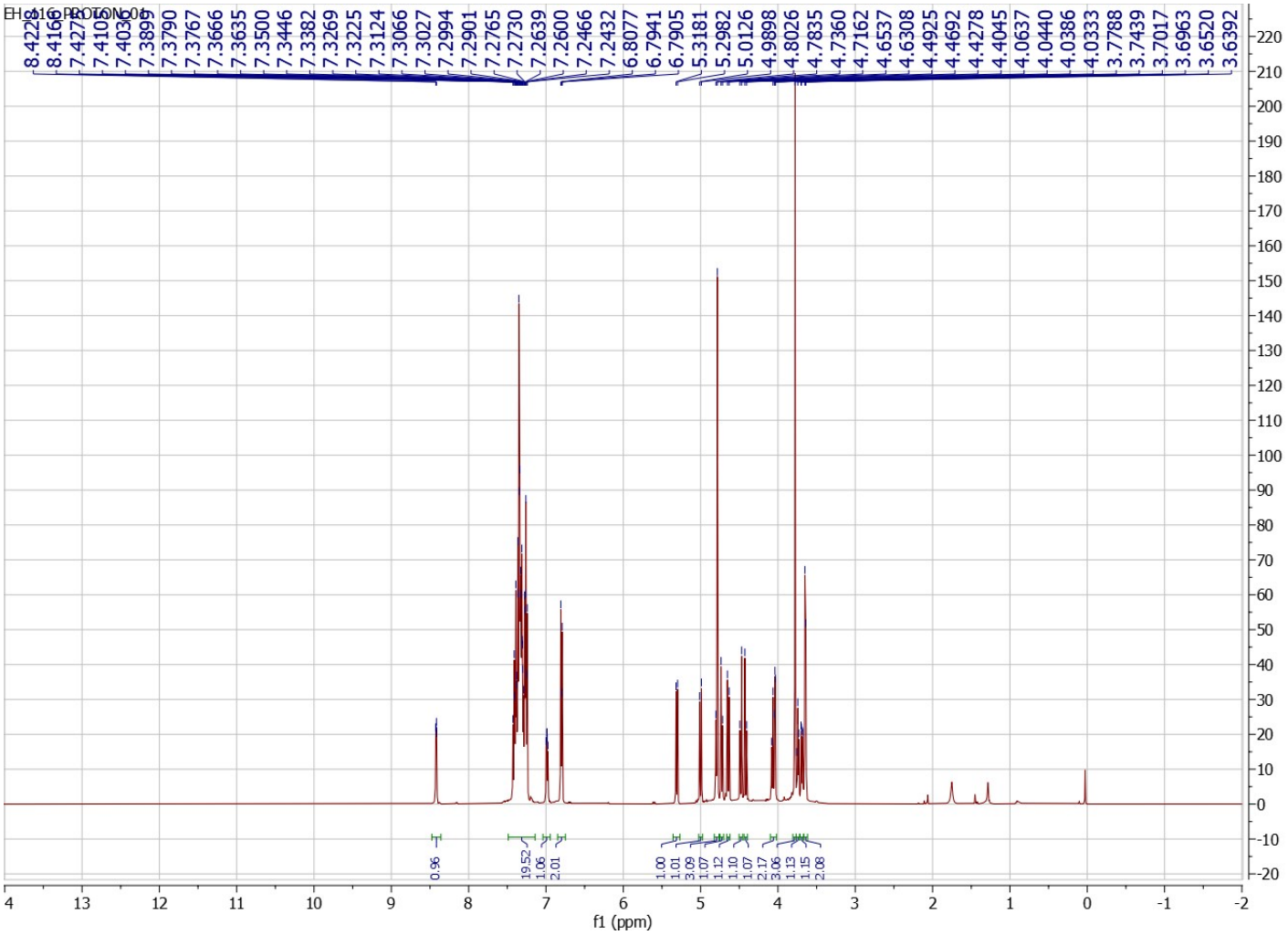

EH_12_CARBON_01
144.18
138.36
137.99
128.37
128.14
127.89
127.69
127.43
99.96
77.27
77.01
76.76
75.69
73.42
73.31
71.27
70.88
70.74
68.43
f1 (ppm)

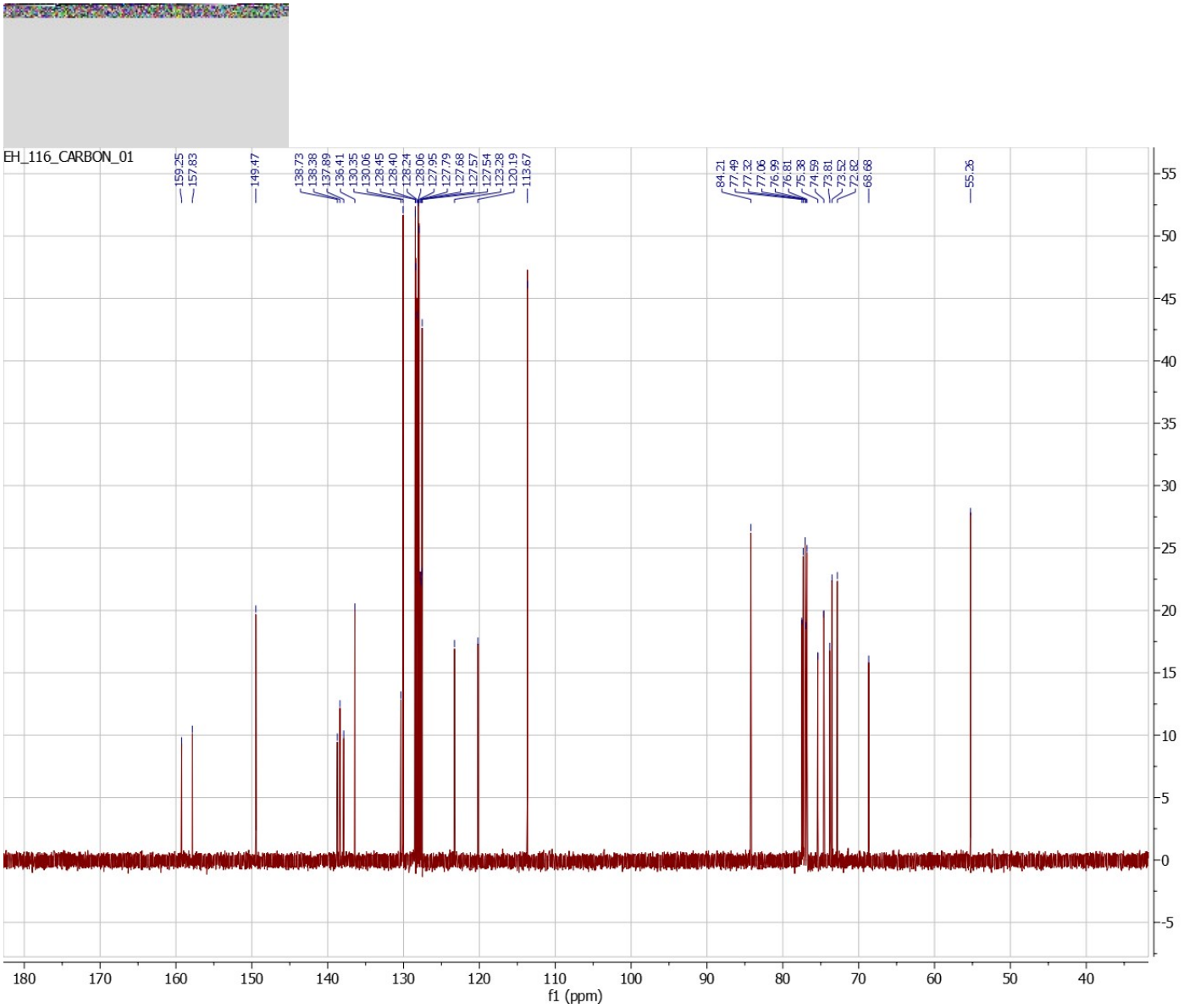

BnO
OBn
O
BnO
S
N
OH


EH-112-PROTON-01
8.4210
8.4121
8.4087
7.4480
7.4325
7.4286
7.4181
7.4131
7.4103
7.4079
7.3948
7.3754
7.3610
7.3454
7.3393
7.3270
7.3173
7.3115
7.3057
7.2865
7.2799
7.2648
7.0500
7.0472
7.0398
7.0358
7.0263
7.0231
5.2341
5.2146
4.9492
4.9263
4.7983
4.6296
4.6068
4.4986
4.4751
4.4388
4.4154
4.2232
4.2043
4.1853
4.0185
4.0129
3.7774
3.7645
3.7516
3.6406
3.6278
3.5953
3.5897
3.5768
3.5712
0.80
16.28
0.85
0.89
1.04
1.91
0.99
1.06
0.99
1.05
0.96
1.01
2.00
1.16
f1 (ppm)

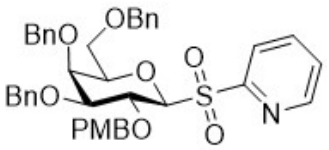

EH_112_CARBON_01
156.60
149.37
138.62
138.30
137.83
136.70
128.49
128.41
128.21
128.09
127.96
127.80
127.74
127.66
127.56
124.35
120.81
85.43
83.61
78.02
77.29
77.04
76.78
74.61
73.58
73.53
72.72
70.33
68.56
60.40
26.92
21.05
14.20
0.00
f1 (ppm)

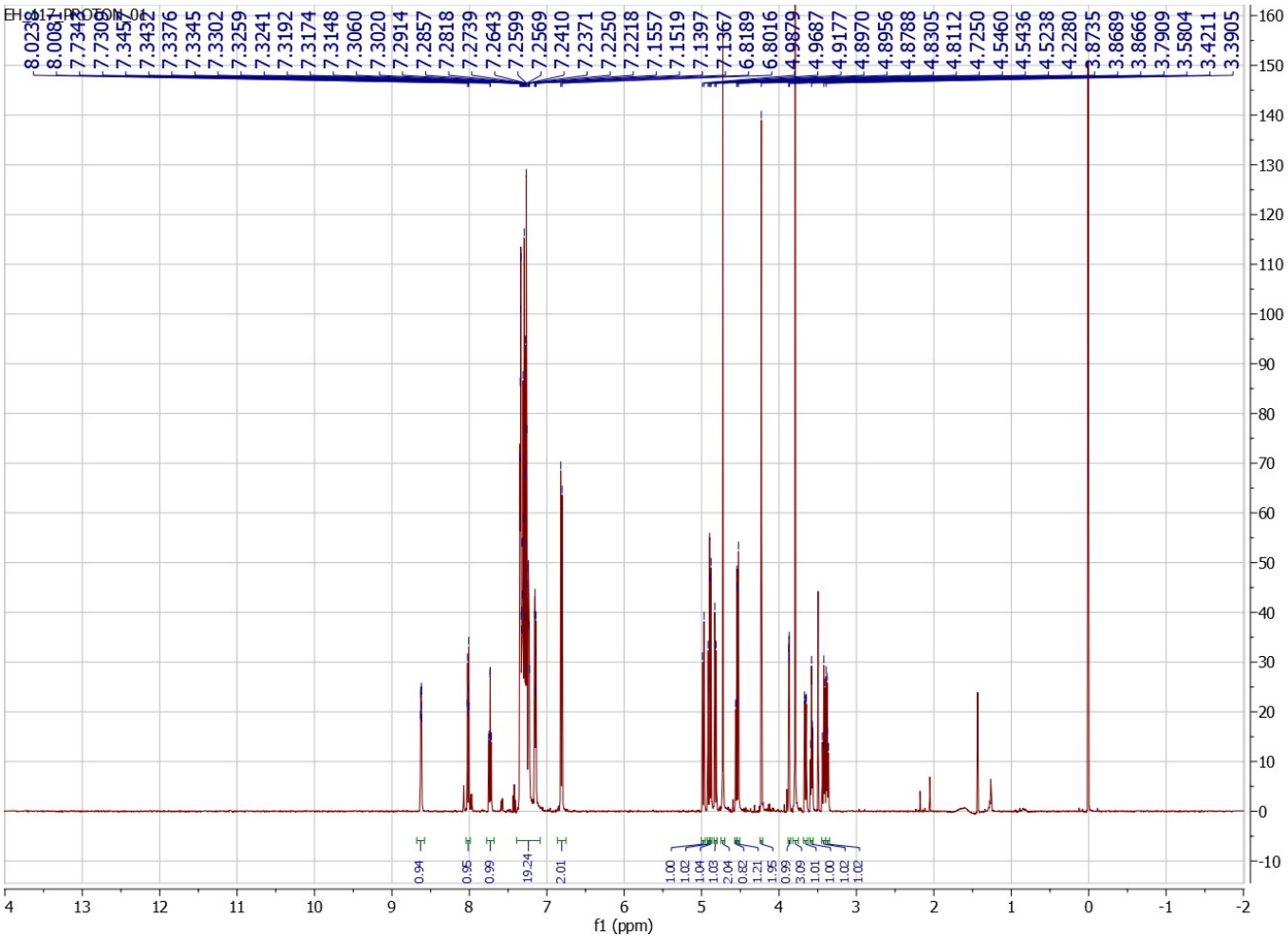

BnO
OBn
O
BnO
S
N
PMBO

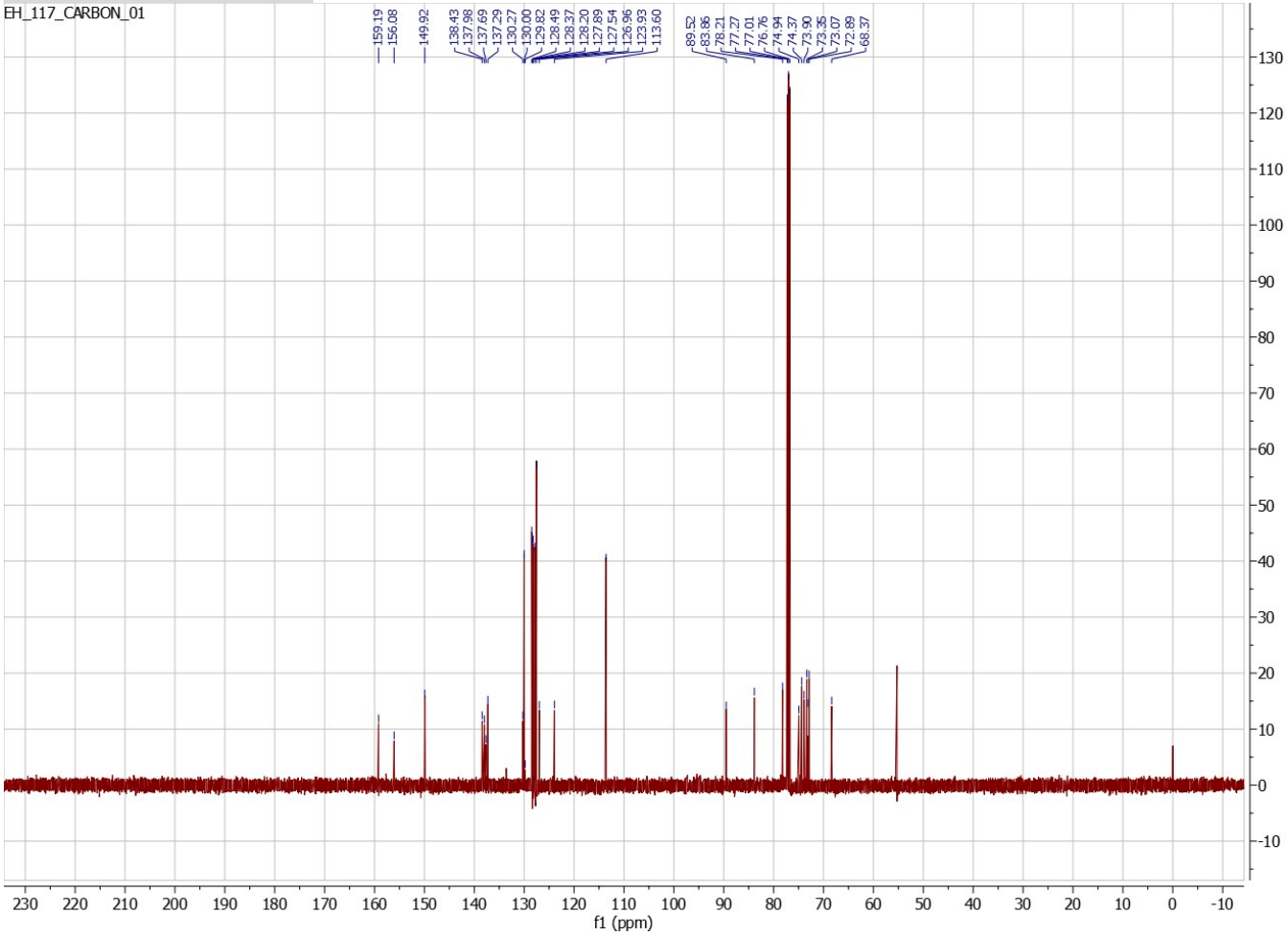

EH-316-PROTON-01
8.4225
8.4166
7.4278
7.4105
7.4030
7.3899
7.3790
7.3767
7.3666
7.3635
7.3500
7.3446
7.3382
7.3269
7.3225
7.3124
7.3066
7.3027
7.2994
7.2901
7.2765
7.2730
7.2639
7.2600
7.2466
7.2432
6.8077
6.7941
6.7905
5.3181
5.2982
5.0126
4.9898
4.8026
4.7835
4.7360
4.7162
4.6537
4.6308
4.4925
4.4692
4.4278
4.4045
4.0637
4.0440
4.0386
4.0333
3.7788
3.7439
3.7017
3.6963
3.6520
3.6392
0.96
19.52
1.06
2.01
1.00
1.01
3.09
1.07
1.12
1.10
1.07
2.17
3.06
1.13
1.15
2.08
f1 (ppm)


EH_116_CARBON_01
159.25
157.83
149.47
138.73
138.38
137.89
136.41
130.55
130.06
128.45
128.40
128.24
128.06
127.95
127.79
127.68
127.57
127.54
123.28
120.19
113.67
84.21
77.49
77.32
77.06
76.99
76.81
75.38
74.59
73.81
73.52
72.82
68.68
55.26
f1 (ppm)

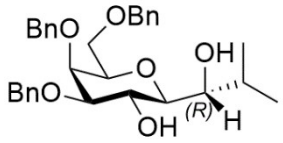
Supporting Information

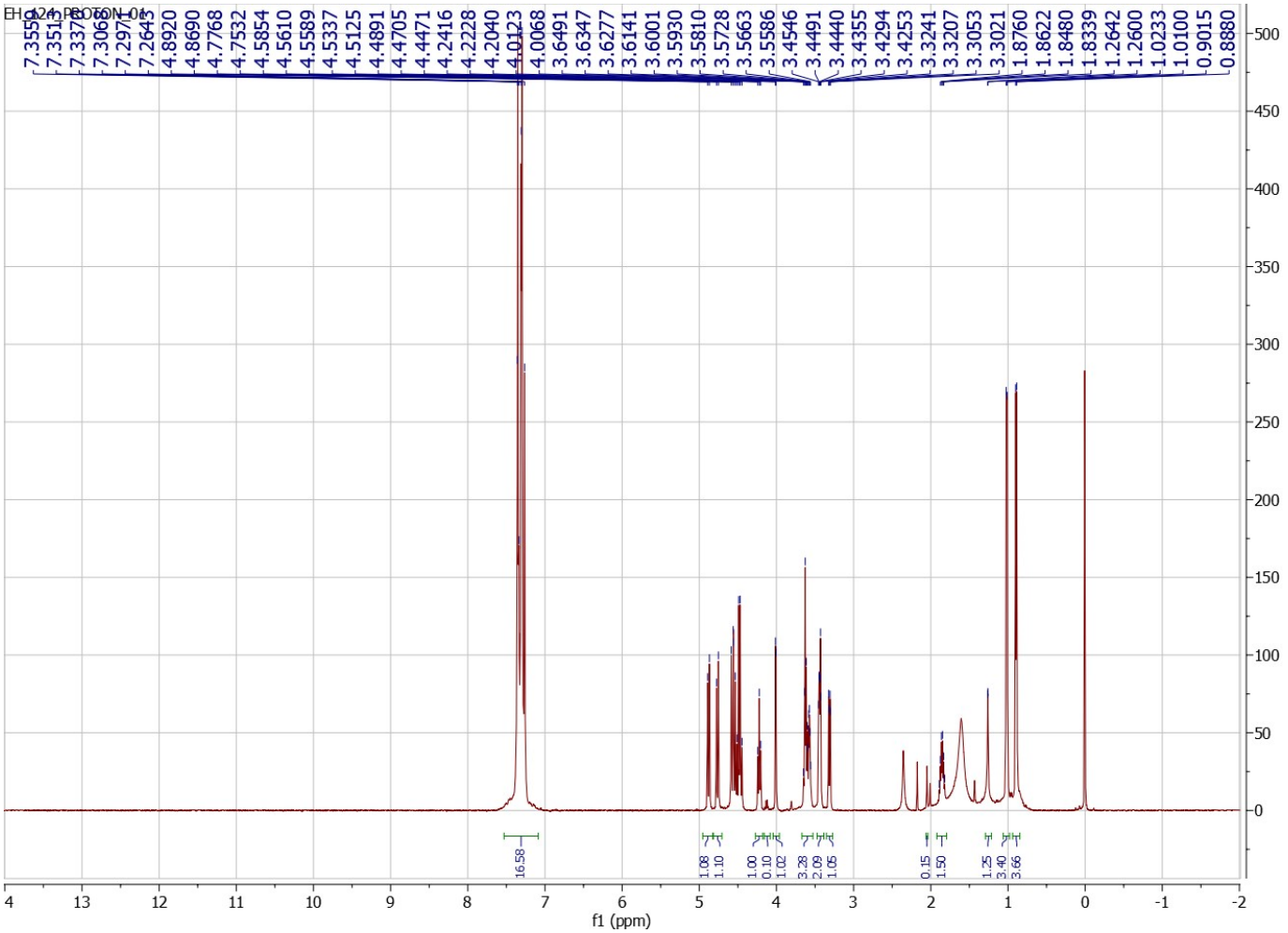


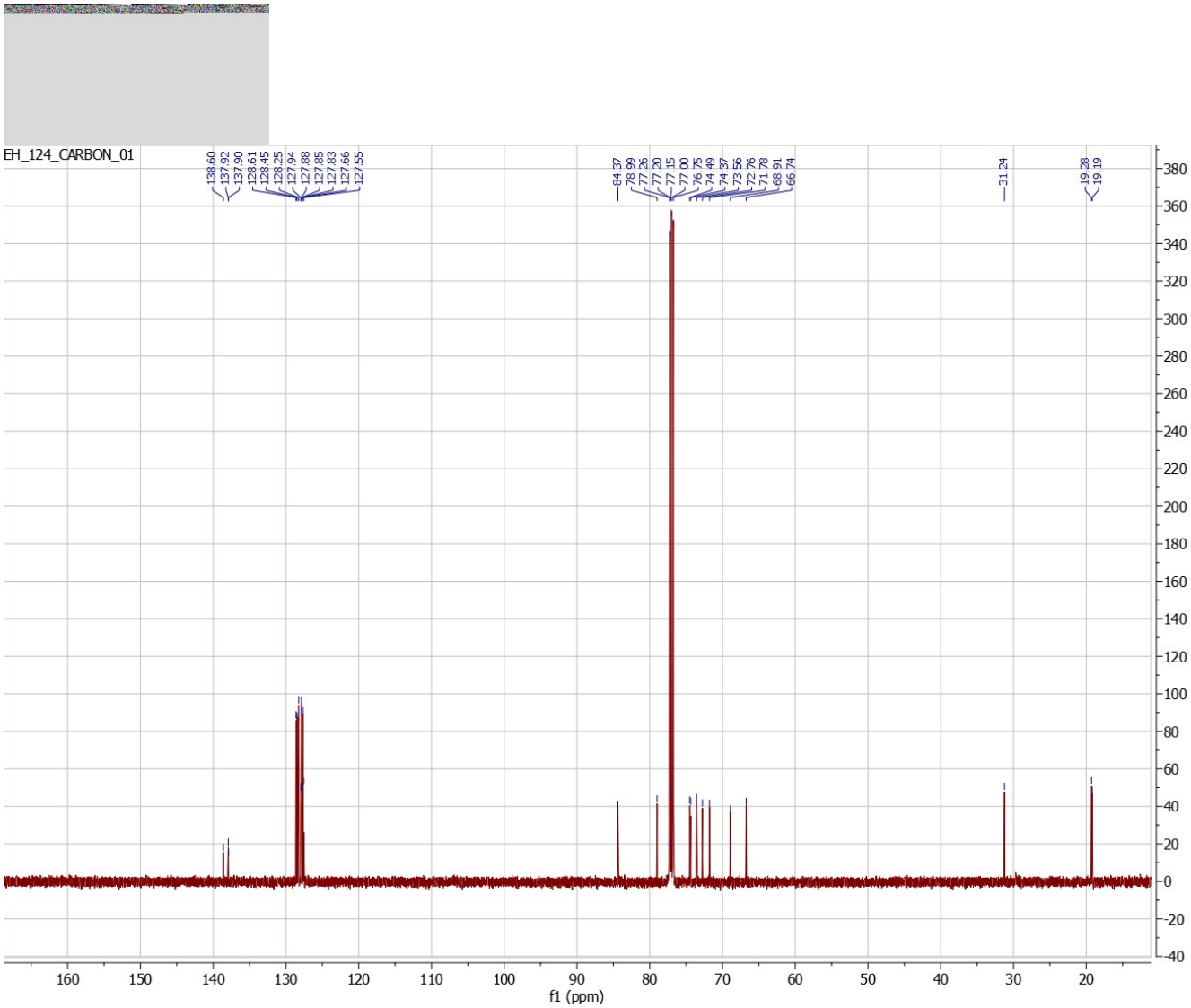


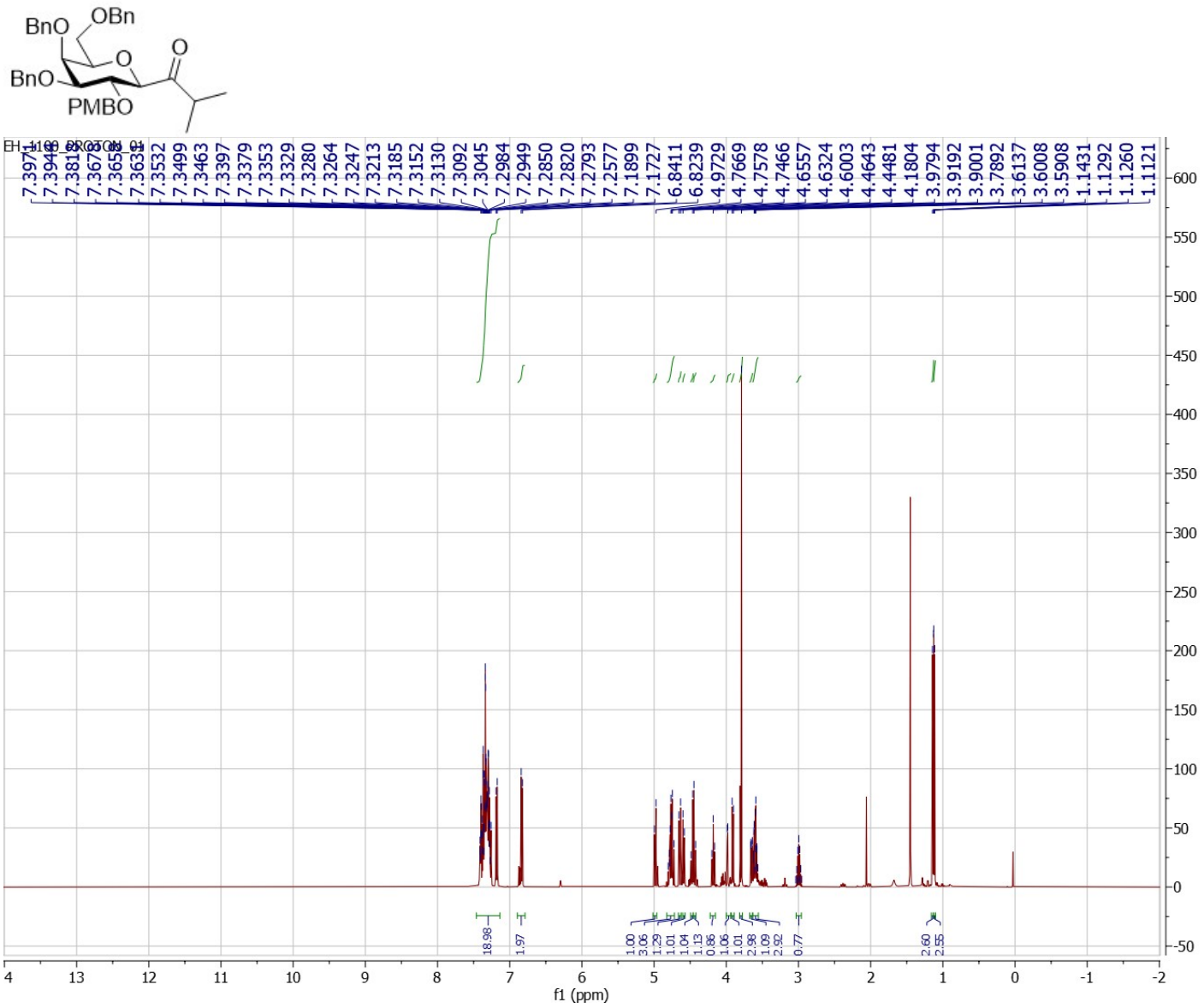

EH_117_CARBON_01
159.19
156.08
149.92
138.43
137.98
137.69
137.29
130.27
130.00
129.82
128.49
128.37
128.20
127.89
127.54
126.96
123.93
113.60
89.52
83.86
78.21
77.27
77.01
76.76
74.94
74.37
73.90
73.35
73.07
72.89
68.37
f1 (ppm)

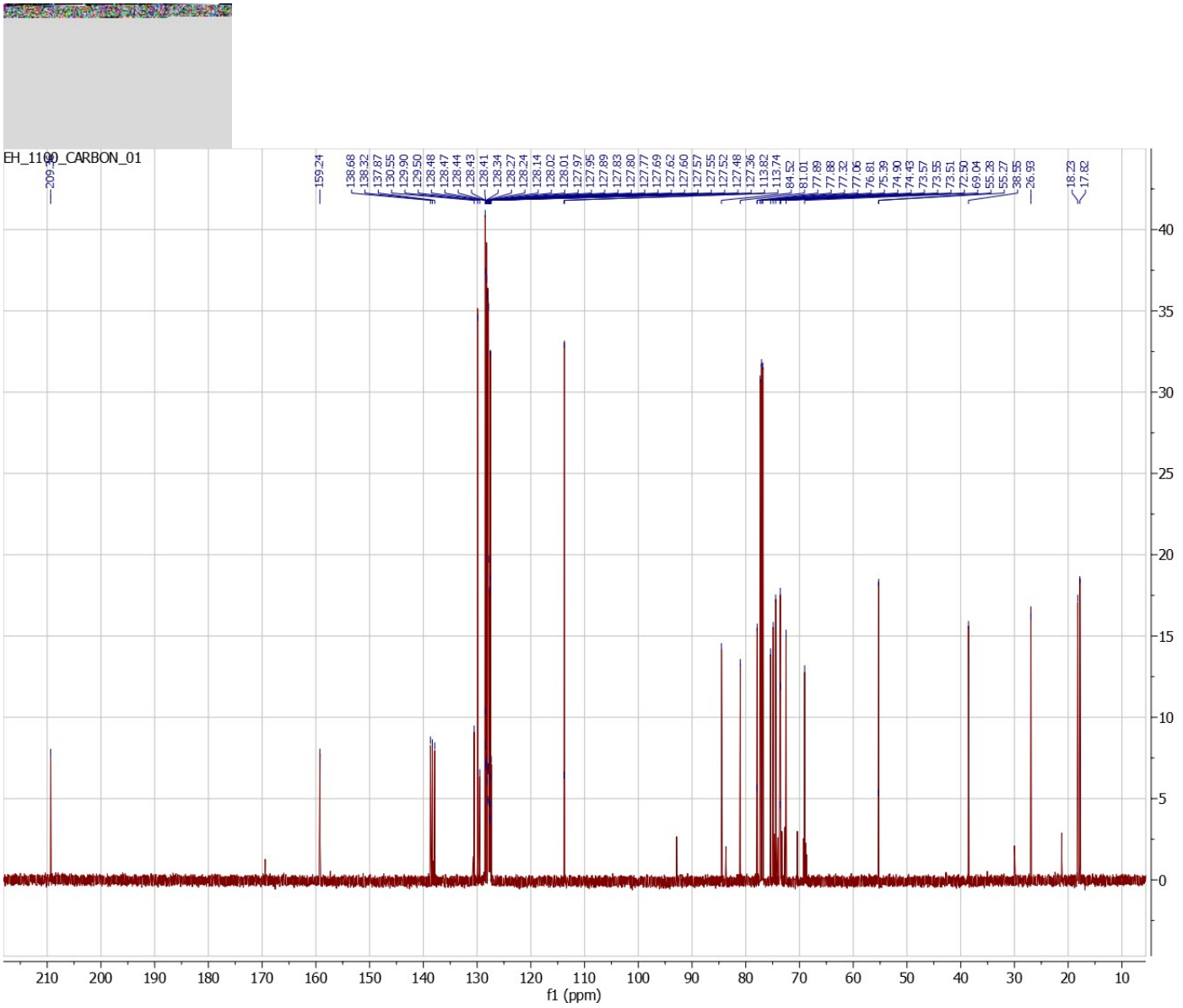
Supporting Information

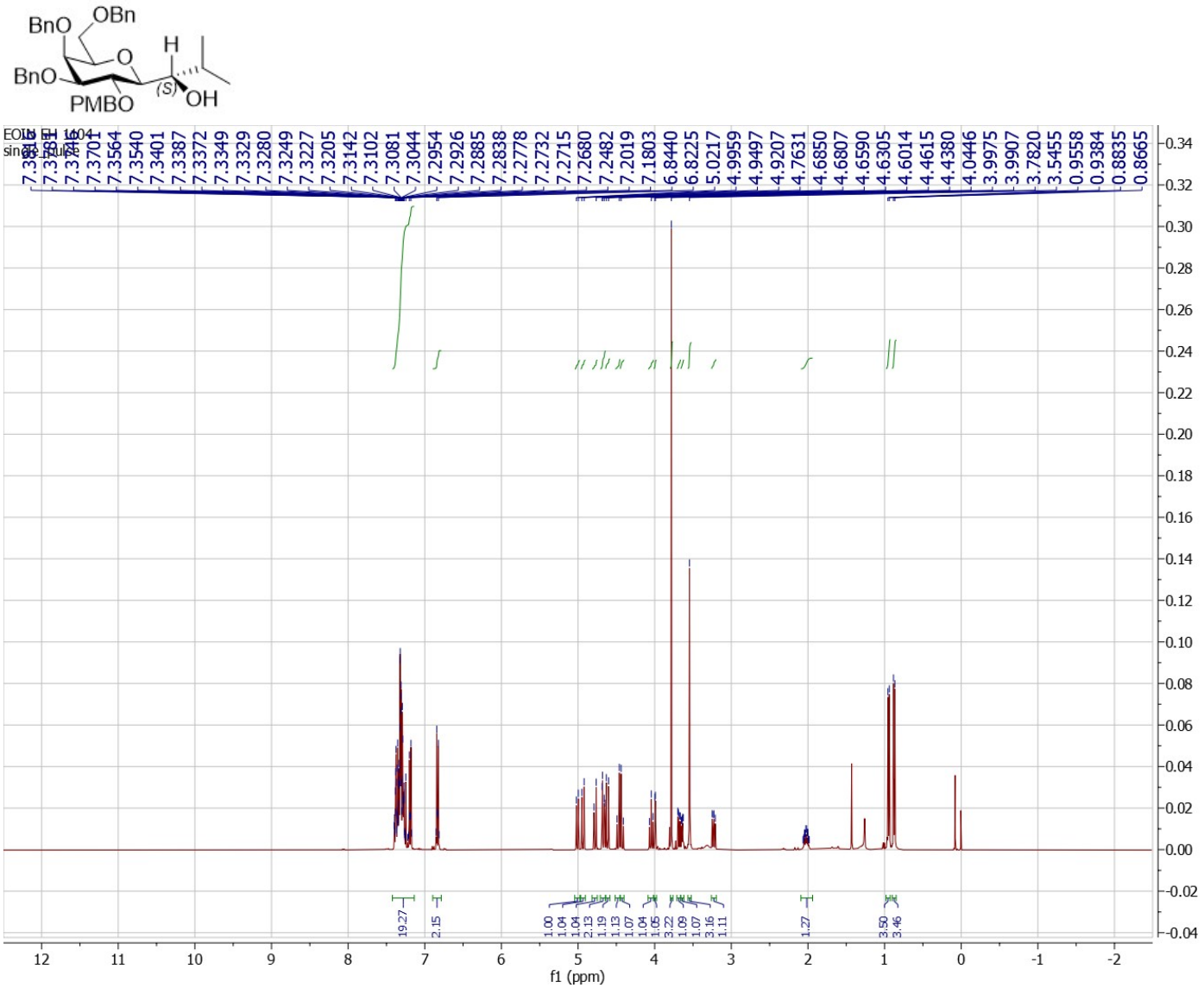


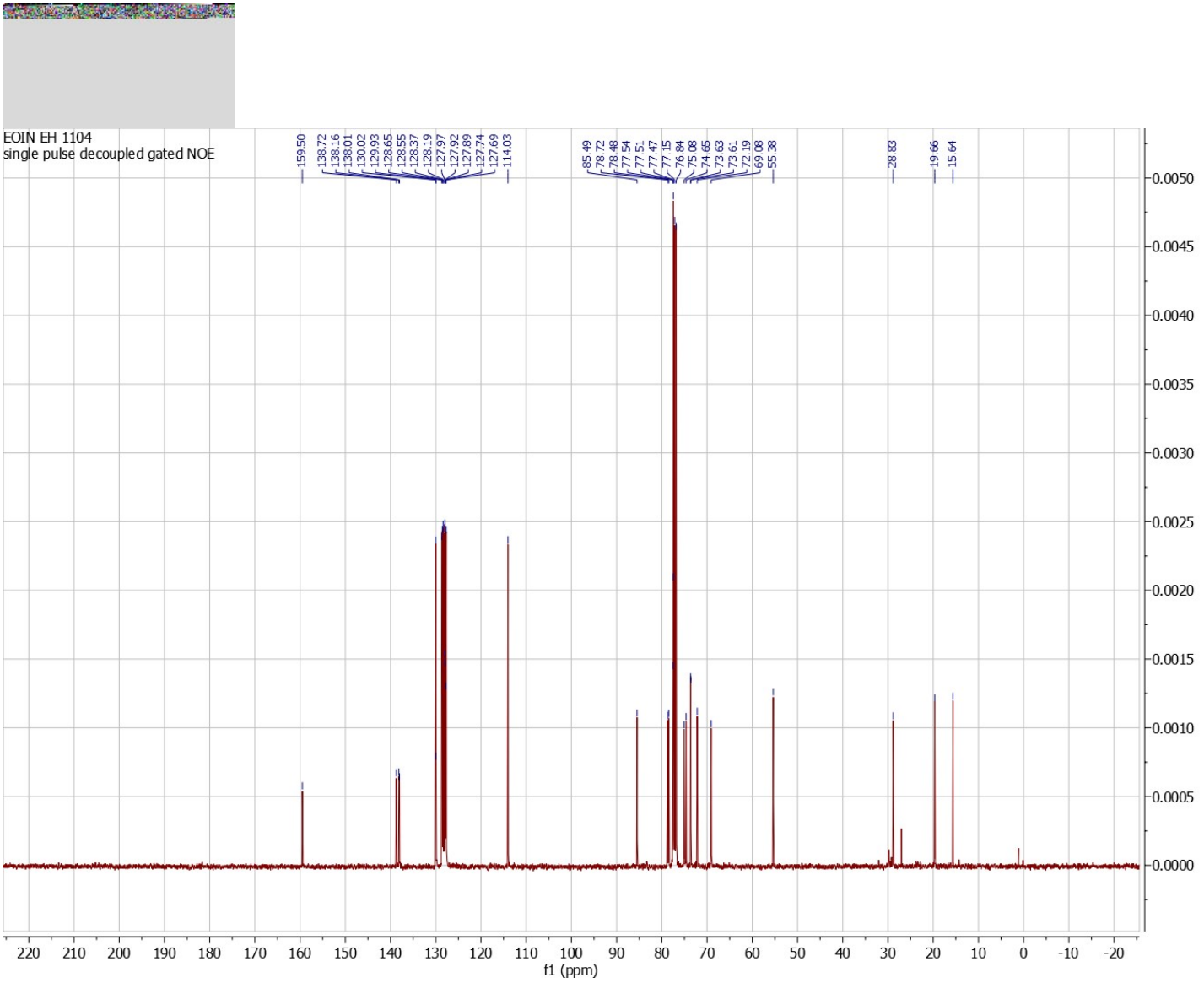



EH_124_CARBON_01
138.60
137.92
137.90
128.61
128.45
128.25
127.94
127.88
127.85
127.83
127.66
127.55
84.37
78.99
77.26
77.20
77.15
77.00
76.75
74.49
74.37
73.56
72.76
71.78
68.91
66.74
31.24
19.28
19.19
f1 (ppm)

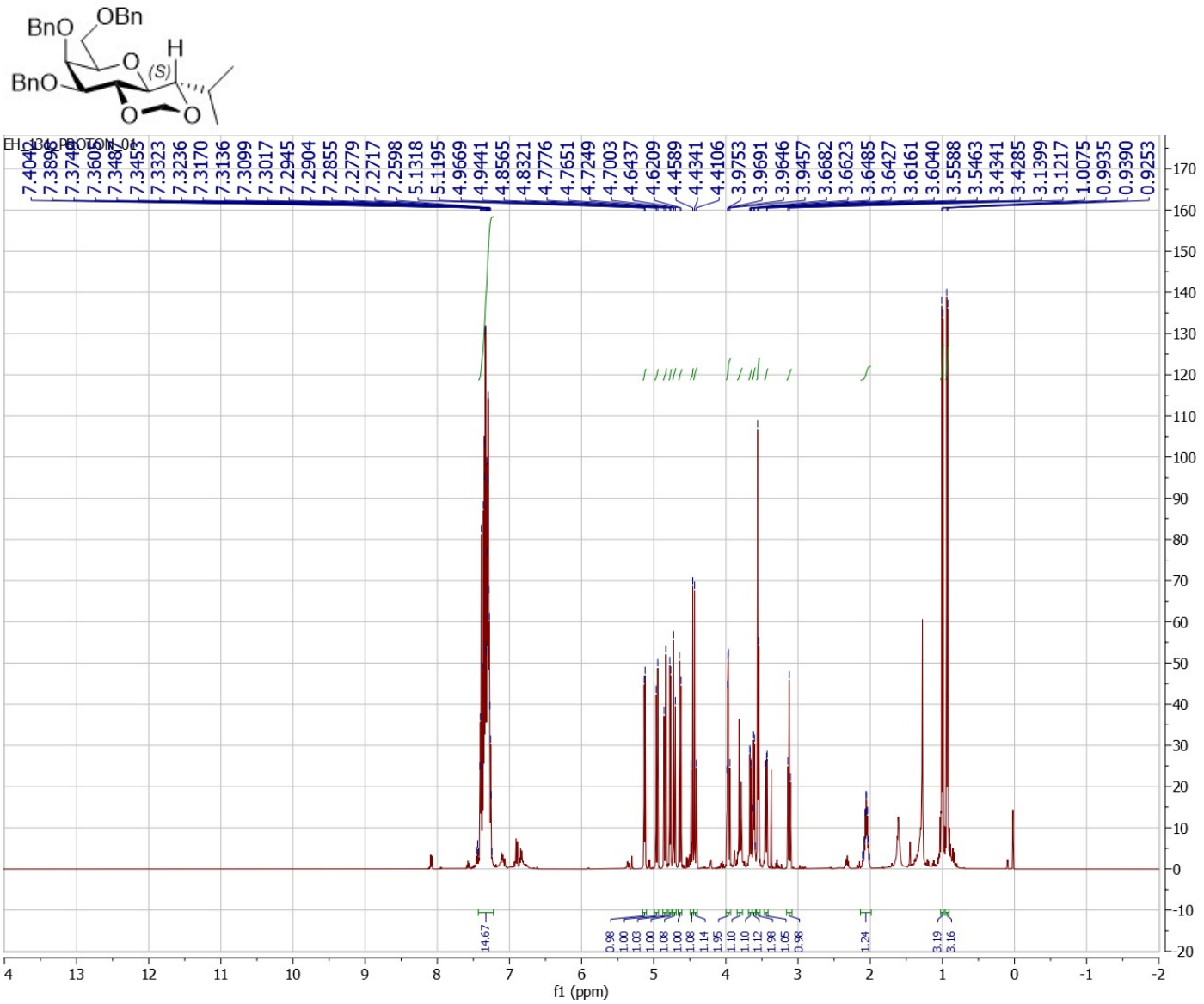

BnO
OBn
O
O
BnO
PMBO
f1 (ppm)

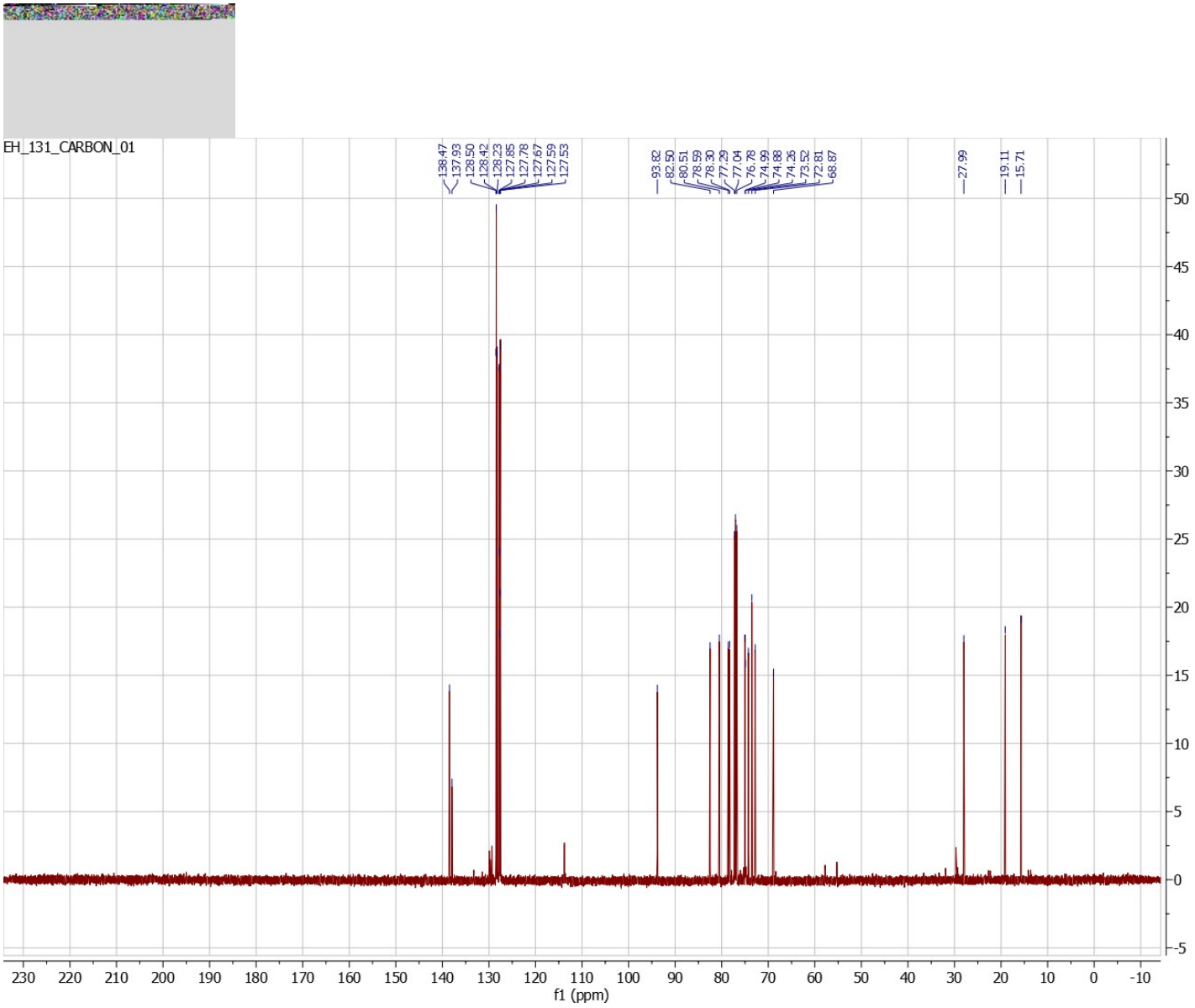

EH_1100_CARBON_01
209.36
159.24
138.68
138.32
137.87
130.55
129.90
129.50
128.48
128.47
128.44
128.43
128.41
128.34
128.27
128.24
128.14
128.02
128.01
127.97
127.95
127.89
127.83
127.80
127.77
127.69
127.62
127.60
127.57
127.55
127.52
127.48
127.36
113.82
113.74
84.52
81.01
77.89
77.88
77.32
77.06
76.81
75.39
74.90
74.43
73.57
73.55
73.51
72.50
69.04
55.28
55.27
38.55
26.93
18.23
17.82
40
35
30
25
20
15
10
5
0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


BnO
OBn
O
H
BnO
(S)
OH
PMBO
f1 (ppm)


EOIN EH 1104
single pulse decoupled gated NOE
159.50
138.72
138.16
138.01
130.02
129.93
128.65
128.55
128.37
128.19
127.97
127.92
127.89
127.74
127.69
114.03
85.49
78.72
78.48
77.54
77.51
77.47
77.15
76.84
75.08
74.65
73.63
73.61
72.19
69.08
55.38
28.83
19.66
15.64
0.0050
0.0045
0.0040
0.0035
0.0030
0.0025
0.0020
0.0015
0.0010
0.0005
0.0000
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

Supporting Information




EH_131_CARBON_01
138.47
137.93
128.50
128.42
128.23
127.85
127.78
127.67
127.59
127.53
93.82
82.50
80.51
78.59
78.30
77.29
77.04
76.78
74.99
74.88
74.26
73.52
72.81
68.87
27.99
19.11
15.71
f1 (ppm)