Supplementary Materials

Borylated 5-Membered Ring Iminosugars: Detailed Nuclear Magnetic Resonance Spectroscopic Characterisation, and Method for Analysis of Anomeric and Boron Equilibria

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Table S1. Structures of the reference compounds analysed by ^{11}B-NMR and their corresponding signals at 128 MHz, dissolved in the deuterated solvent stated in brackets. **4**

Figure S1. ^{1}H- (400 MHz), ^{13}C-NMR (100 MHz), DEPT, ^{11}B-NMR (128 MHz), COSY, HSQC and HMBC spectra of *N*-(3-methylphenyl boronic acid pinacol ester)-3,6-dideoxy-3,6-imino-1,2-*O*-isopropylidene-α-D-gulofuranose **meta 2** in $CDCl_3$. **6**

Figure S2. ^{1}H- (400 MHz), ^{13}C-NMR (100 MHz), DEPT, ^{11}B-NMR (128 MHz), COSY, HSQC and HMBC spectra of *N*-(3-methylphenyl boronic acid)-3,6-dideoxy-3,6-imino-D-gulofuranose **meta 3** in $CDCl_3$. **25**

Figure S3. ^{1}H-NMR spectrum (400 MHz, D_2O) of intermediate **meta 3** with colour-coded signals, highlighting the furanose anomeric forms they belong to, with interpretation of the isolated signals and tentative interpretation of the overlapping ones. Namely, the orange designates the *α-fur* form and indigo designates the *β-fur* form. A) section 7.87 ppm to 7.20 ppm; B) section 5.50 ppm to 4.87 ppm; C) section 4.61 ppm to 4.12 ppm; D) section 4.00 ppm to 3.15 ppm. Highlighted are also the principal COSY correlations to hydrogen atoms within the same spin systems. **57**

Figure S4. ^{13}C-NMR spectrum (100 MHz, D_2O) sections of intermediate **meta 3** with colour-coded signals, highlighting the furanose anomeric forms they belong to, with interpretation of the isolated signals and tentative interpretation of the overlapping ones. Namely, the orange designates the *α-fur* form and indigo designates the *β-fur* form. A) section 141.5 ppm to 127.2 ppm; B) section 104.0 ppm to 76.0 ppm; C) section 75.5 ppm to 56.7 ppm. Highlighted are also the principal HSQC and HMBC correlations to hydrogen atoms within the same spin systems. **61**

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Figure S6. ^{1}H- (400 MHz), ^{13}C-NMR (100 MHz), DEPT, ^{11}B-NMR (128 MHz), COSY, HSQC and HMBC spectra of *N*-(2-methylphenyl boronic acid pinacol ester)-3,6-dideoxy-3,6-imino-1,2-*O*-isopropylidene-α-D-gulofuranose **ortho 2** in $CDCl_3$. **78**

Figure S7. ^{1}H- (400 MHz), ^{13}C-NMR (100 MHz), DEPT, ^{11}B-NMR (128 MHz), COSY, HSQC and HMBC spectra of *N*-(2-methylphenyl boronic acid)-3,6-dideoxy-3,6-imino-D-gulofuranose **ortho 3** in D_2O. **99**

Figure S8. ^{1}H-NMR spectrum (400 MHz, D_2O) of intermediate **ortho 3** with colour-coded signals, highlighting the furanose anomeric forms they belong to, with interpretation of the isolated signals and tentative interpretation of the overlapping ones. Namely, the orange designates the *α-fur* form and indigo designates the *β-fur* form. A) section 7.93 ppm to 7.47 ppm; B) section 5.57 ppm to 4.54 ppm; C) section 4.54 ppm to 3.40 ppm. Highlighted are also the principal COSY correlations to hydrogen atoms within the same spin systems. **122**

Figure S9. ^{13}C-NMR spectrum (100 MHz, D_2O) sections of intermediate **ortho 3** with colour-coded signals, highlighting the furanose anomeric forms they belong to, with interpretation of the isolated **125**

signals and tentative interpretation of the overlapping ones. Namely, the orange designates the *α-fur* form and indigo designates the *β-fur* form. A) section 100.5 ppm to 79 ppm; B) section 105.0 ppm to 90.0 ppm ; C) section 78.0 ppm to 58.0 ppm. Highlighted are also the principal HSQC correlations to hydrogen atoms within the same spin systems.

Figure S10. ^{1}H- (400 MHz), ^{13}C-NMR (100 MHz), DEPT, ^{11}B-NMR (128 MHz), COSY, HSQC and HMBC spectra of *N*-(2-methylphenyl boronic acid)-1,4-dideoxy-1,4-imino-L-gulitol **ortho 4** in D_2O. **127**

Figure S11. ^{1}H-NMR spectrum (400 MHz, D_2O) of final compound **ortho 4** with colour-coded signals, highlighting the boronic acid and boronate forms they belong to, with interpretation of the isolated signals and tentative interpretation of the overlapping ones. Namely, the orange designates the boronic acid form and indigo designates the boronate form. A) section 8.00 ppm to 6.90 ppm; B) section 4.65 ppm to 3.00 ppm; C) section 1.80 ppm to 1.10 ppm. Highlighted are also the principal COSY correlations to hydrogen atoms within the same spin systems. **154**

Figure S12. ^{13}C-NMR spectrum (100 MHz, D_2O) sections of final compound **ortho 4** with colour-coded signals, highlighting the boronic acid and boronate forms they belong to, with interpretation of the isolated signals and tentative interpretation of the overlapping ones. Namely, the orange designates the boronic acid form and indigo designates the boronate form. A) section 144 ppm to 109 ppm; B) section 76 ppm to 44 ppm. **157**

Figure S13. ^{1}H- (400 MHz), ^{13}C-NMR (100 MHz), ^{11}B-NMR (128 MHz), COSY and HSQC spectra of *N*-(3-methylphenyl boronic acid)-3,6-dideoxy-3,6-imino-1,2-*O*-isopropylidene-*α*-D-gulofuranose **meta 5** in MeOD. **159**

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Figure S15. ^{13}C-NMR spectrum (100 MHz, MeOD) of compound **meta 5** with colour-coded signals, highlighting the boronic acid form they belong to, with interpretation of the isolated signals and tentative interpretation of the overlapping ones. Namely, the orange designates the boronic acid form. A) section 141 ppm to 25 ppm; B) section 140 ppm to 120 ppm. **175**

Figure S16. ^{1}H- (400 MHz) and COSY spectra of the minor species for *N*-(3-methylphenyl boronic acid)-3,6-dideoxy-3,6-imino-1,2-*O*-isopropylidene-*α*-D-gulofuranose **meta 5** in MeOD. **177**

Figure S17. ^{1}H- (400 MHz), ^{13}C-NMR (100 MHz), DEPT, HSQC and HMBC spectra of *N*-(3-methylphenyl boronic acid)-3,6-dideoxy-3,6-imino-1,2-*O*-isopropylidene-*α*-D-gulofuranose **meta 5** in MeOD. **187**

Figure S18. ^{1}H- (400 MHz), ^{13}C-NMR (100 MHz), DEPT, ^{11}B-NMR (128 MHz), COSY and HSQC spectra of *N*-(2-methylphenyl boronic acid)-3,6-dideoxy-3,6-imino-1,2-*O*-isopropylidene-*α*-D-gulofuranose **ortho 5** in D_2O. **206**

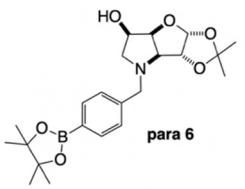
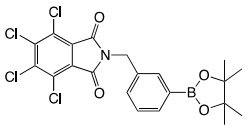
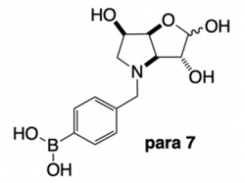
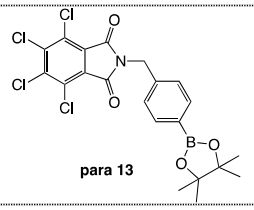
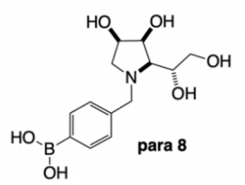
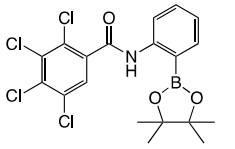
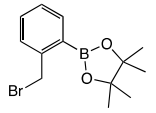
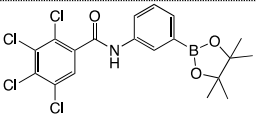
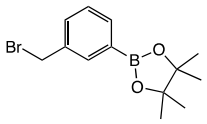
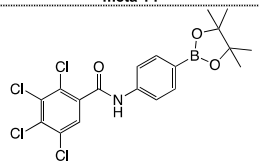
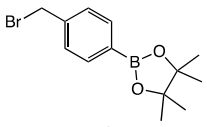
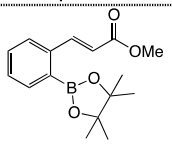
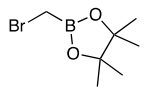
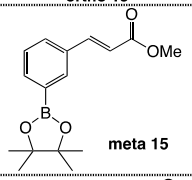
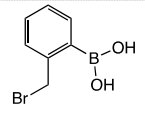
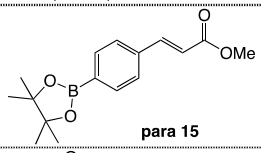
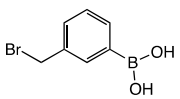
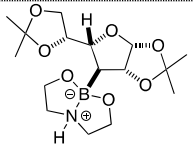
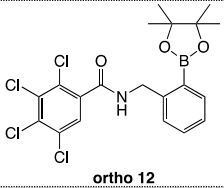
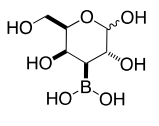
Figure S19. ^{1}H-NMR spectrum (400 MHz, D_2O) of compound **ortho 5** with colour-coded signals, highlighting the boronic acid and boronate forms they belong to, with interpretation of the isolated signals and tentative interpretation of the overlapping ones. Namely, the orange designates the boronic acid form and indigo designates the boronate form. A) section 7.80 ppm to 6.80 ppm; B) section 6.10 ppm to 4.20 ppm; C) section 3.90 ppm to 2.70 ppm; D) section 1.60 ppm to 1.25 ppm. Highlighted are also the principal COSY correlations to hydrogen atoms within the same spin systems. **221**

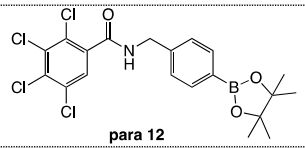
Figure S20. ^{13}C-NMR spectrum (100 MHz, D_2O) of compound **ortho 5** with colour-coded signals, highlighting the boronic acid and boronate forms they belong to, with interpretation of the isolated signals and tentative interpretation of the overlapping ones. Namely, the orange designates the boronic acid form and indigo designates the boronate form. A) section 140 ppm to 23 ppm. **225**

NMR Experimental details **226**

References **259**

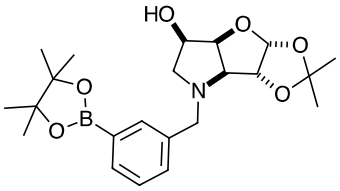
Table S1. Structures of the reference compounds analysed by ^{11}B-NMR and their corresponding signals at 128 MHz, dissolved in the deuterated solvent stated in brackets.

Structure	Chemical shift, ppm	Reference	Structure	Chemical shift, ppm	Ref
 para 6	30.6, 22.3 (integration ratio 3.7 : 1.0) ($CDCl_3$)	§	 meta 13	31.5 ($CDCl_3$)	[1]
 para 7	28.7, 19.4 (integration ratio 9.3 : 1.0) (D_2O)	§	 para 13	31.1 ($CDCl_3$)	[1]
 para 8	28.0, 19.3 (integration ratio 3.9 : 1.0) (D_2O)	§	 ortho 14	31.1 ($CDCl_3$)	[1]
 ortho 9	31 (D_2O)	Inventory (Boron Molecular)[2]	 meta 14	31.7 ($CDCl_3$)	[1]
 meta 9	31 (D_2O)	Inventory (Boron Molecular)[2]	 para 14	31.8 ($CDCl_3$)	[1]
 para 9	31 (D_2O)	Inventory (Boron Molecular)[2]	 ortho 15	31.5 ($CDCl_3$)	[3]
 10	Rapidly decomposed (D_2O)	Inventory (Boron Molecular)[2]	 meta 15	30.7 ($CDCl_3$)	[3]
 ortho 11	30 (D_2O)	Inventory (Boron Molecular)[2]	 para 15	31.2 ($CDCl_3$)	[3]
 meta 11	29 (D_2O)	Inventory (Boron Molecular)[2]	 16 D-*Galacto*	30.9, 17.2, 10.3 (integration ratio: 0.02 : 0.03 : 1.0) (CD_3OD)	[4,5]
 ortho 12	31.8 ($CDCl_3$)	[1]	 17 D-*Galacto*	31.7, 19.4 (integration ratio: 4.4 : 1.0) (D_2O)	[4,5]

 para 12	31.6 ($CDCl_3$)	[1]			

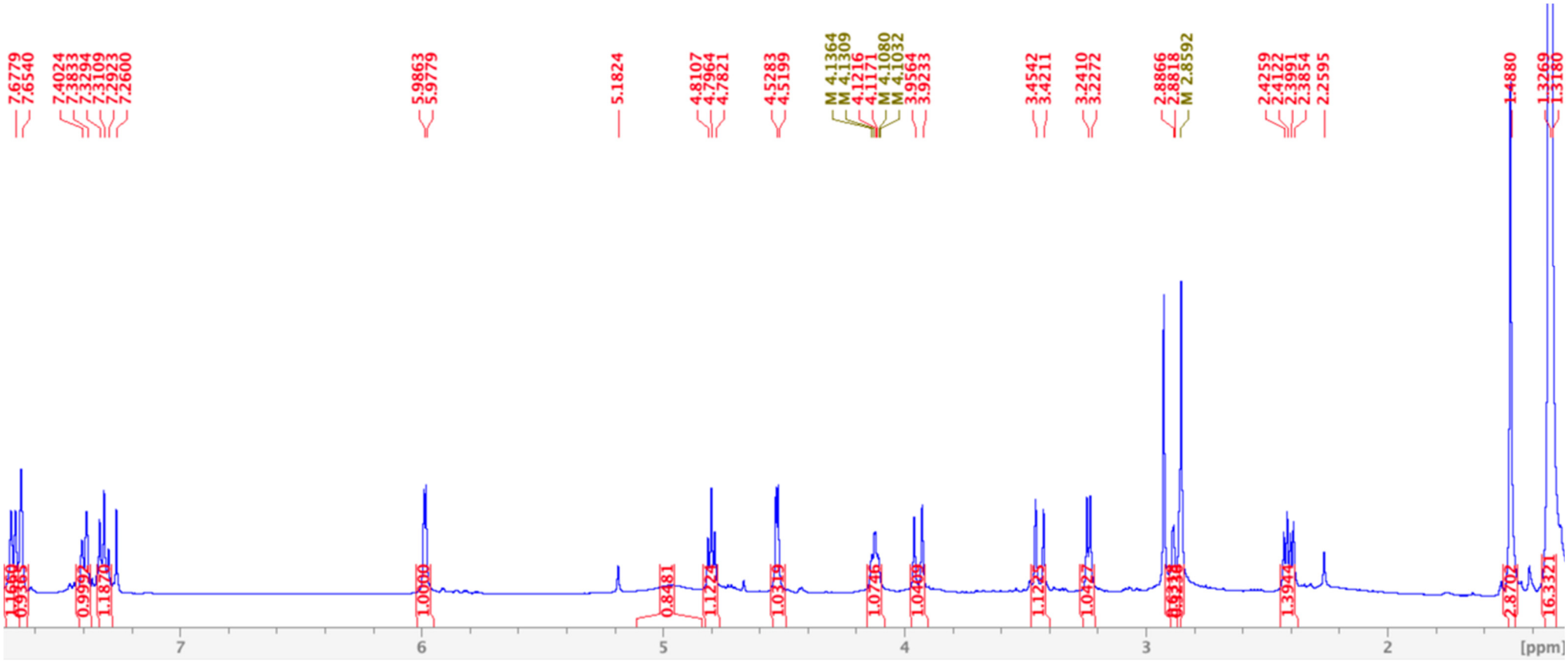
§ The following data will be shortly published.

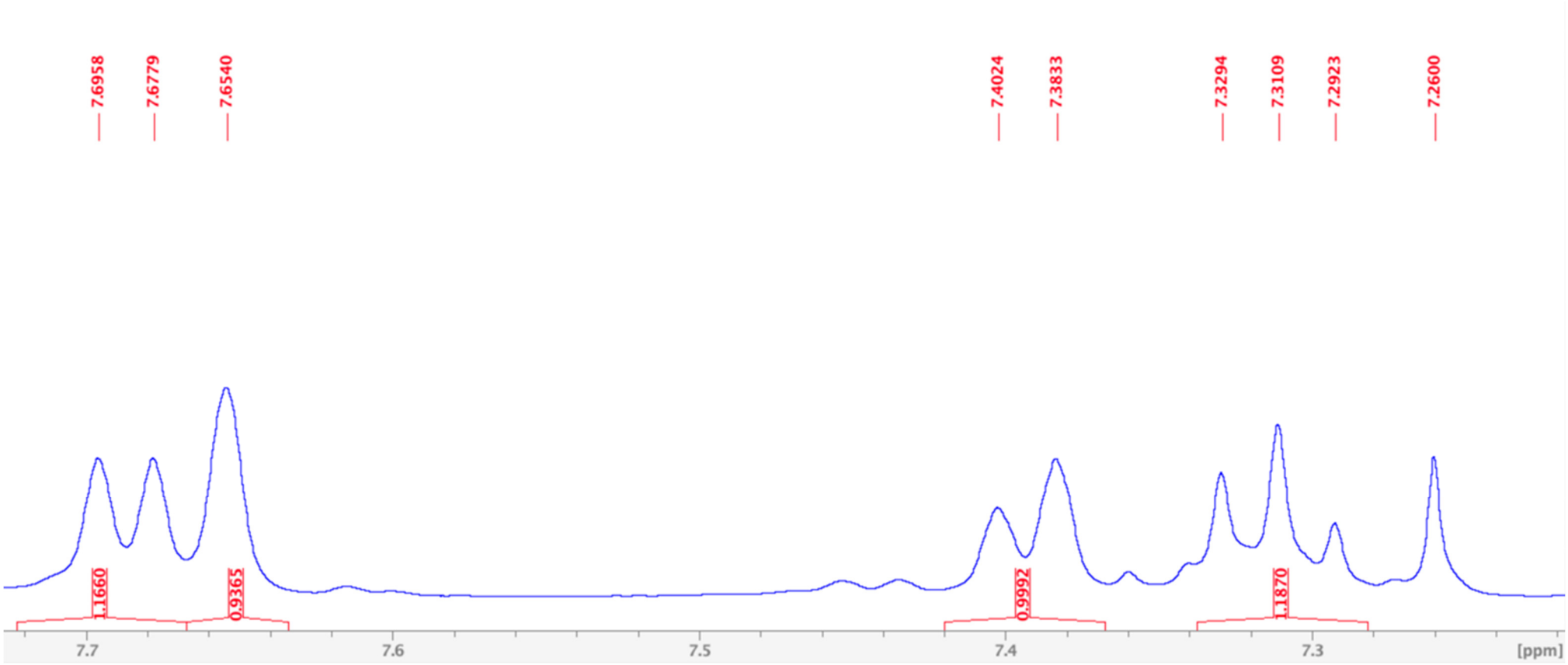
Figure S1. ^{1}H- (400 MHz), ^{13}C-NMR (100 MHz), DEPT, ^{11}B-NMR (128 MHz), COSY, HSQC and HMBC spectra of *N*-(3-methylphenyl boronic acid pinacol ester)-3,6-dideoxy-3,6-imino-1,2-*O*-isopropylidene-α-D-gulofuranose **meta 2** in $CDCl_3$.

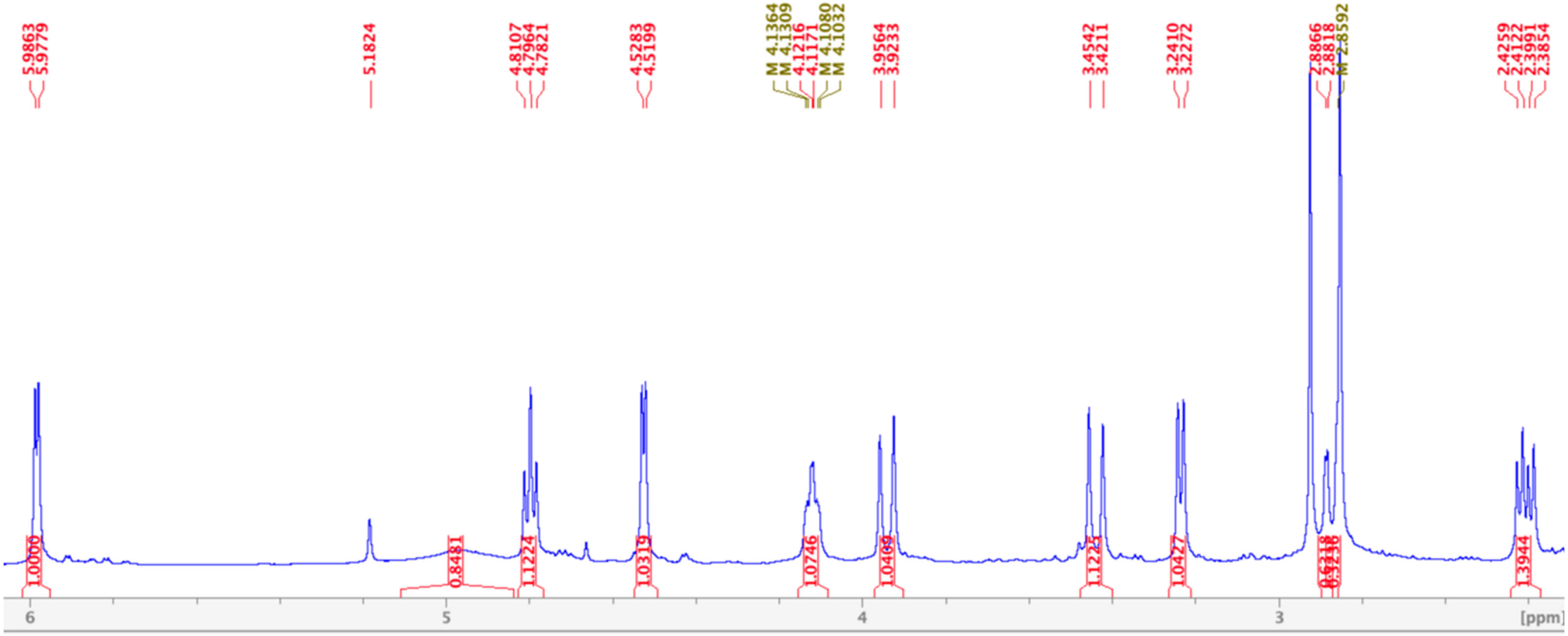


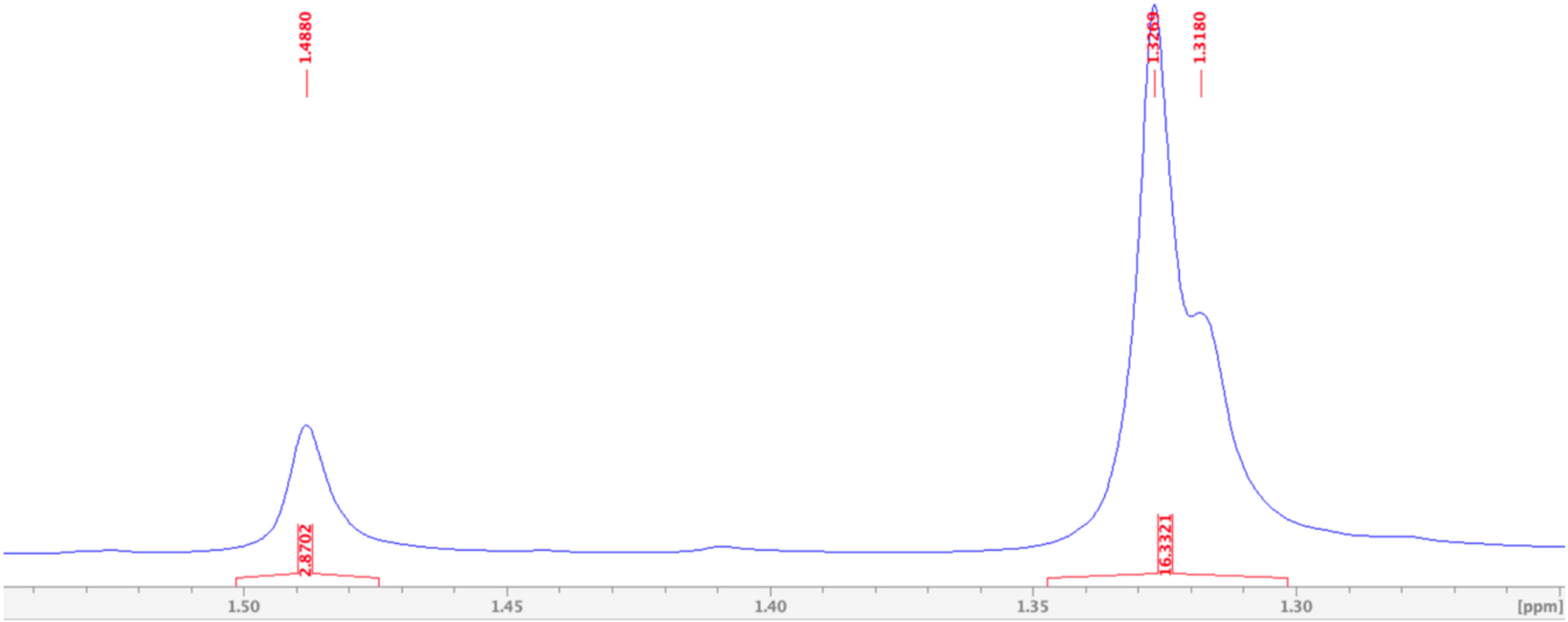
δH: 7.69 (1H, d, $J_{HC,HD}$ 7.2 Hz, ArH^{C}), 7.65 (1H, s, ArH^{A}), 7.39 (1H, d, $J_{HE,HD}$ 7.7 Hz, ArH^{E}), 7.31 (1H, app-t, $J_{HD,HC/HE}$ 7.4 Hz, ArH^{D}), 5.98 (1H, d, $J_{H\text{-}1,H\text{-}2}$ 3.4 Hz, H-1), 5.10-4.85 (1H, broad s, OH), 4.80 (1H, t, $J_{H\text{-}4,H\text{-}3/H\text{-}5}$ 5.7 Hz, H-4), 4.52 (1H, d, $J_{H\text{-}2,H\text{-}1}$ 3.3, H-2), 4.12 (1H, td, $J_{H\text{-}5,H\text{-}4}$ 5.7 Hz, $J_{H\text{-}5,H\text{-}6'}$ 2.1 Hz, H-5), 3.94 (1H, d, $J_{Ha,Hb}$ 13.2 Hz, ArCH^{a}H^{b}), 3.43 (1H, d, $J_{Hb,Ha}$ 13.1 Hz, ArCH$^{a}H^{b}$), 3.23 (1H, d, $J_{H\text{-}3,H\text{-}4}$ 5.5 Hz, H-3), 2.87 (1H, partially obscured dd, $J_{H\text{-}6',H\text{-}6}$ 11.0 Hz, $J_{H\text{-}6',H\text{-}5}$ 1.9 Hz, H-6'), 2.40 (1H, dd, $J_{H\text{-}6,H\text{-}6'}$ 10.8 Hz, $J_{H\text{-}6,H\text{-}5}$ 5.5 Hz, H-6), 1.49 (3H, s, CH_3 acetonide), 1.33 (12H, s, 4 x CH_3 pinacol), 1.32 (3H, s, CH_3 acetonide); **δC**: 137.0 (ArC$_{quat}$), 135.1 (ArC^{A}), 133.9 (ArC^{C}), 131.9 (ArC^{E}), 127.9 (ArC^{D}), 129.5-128.6 (broad, ArC^{B}-B), 112.6 (C$_{quat}$ acetonide), 107.6 (C-1), 84.0 (2 x C$_{quat}$ pinacol) 83.9 (C-2), 83.1 (C-4), 72.7 (C-3), 69.5 (C-5), 61.1 (C-6), 58.2 (CH_2Ar), 27.6, 26.9 (2 x CH_3, acetonide), 24.9 (4 x CH_3 pinacol); **δB**: 30.8.

^{1}H-NMR

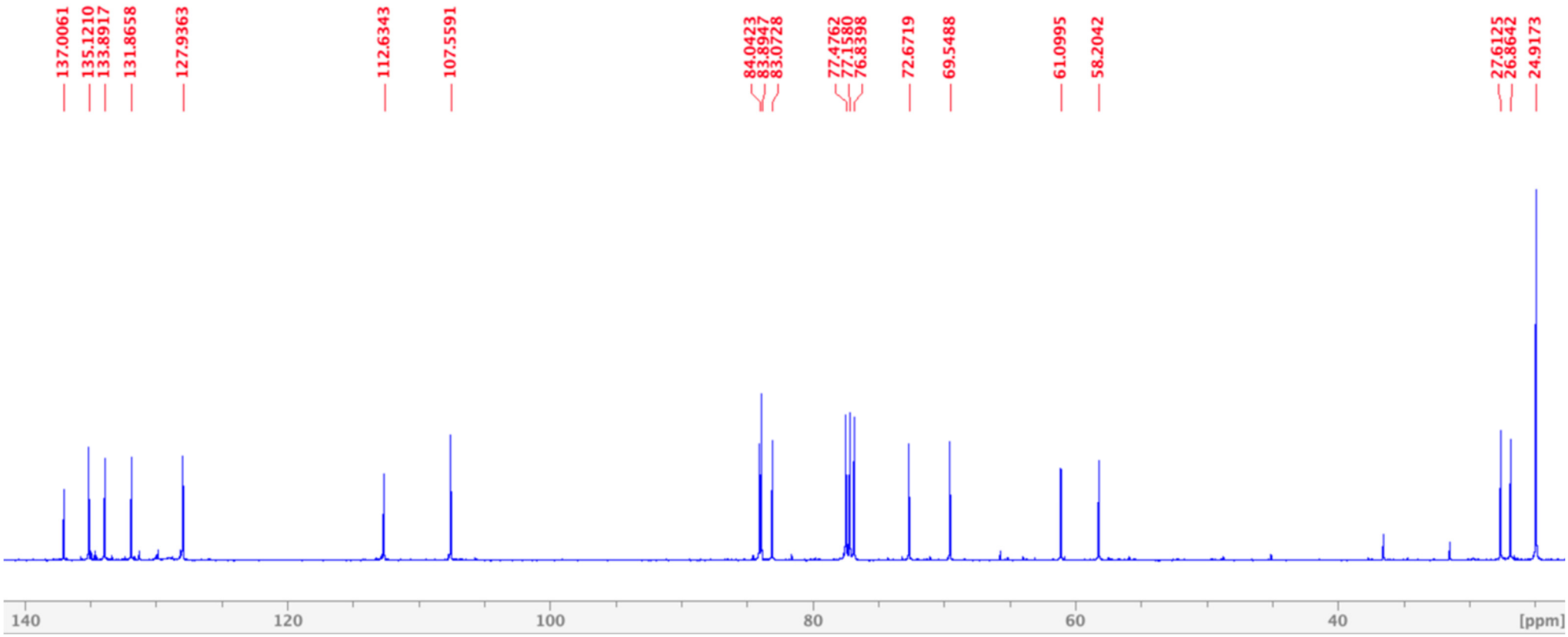

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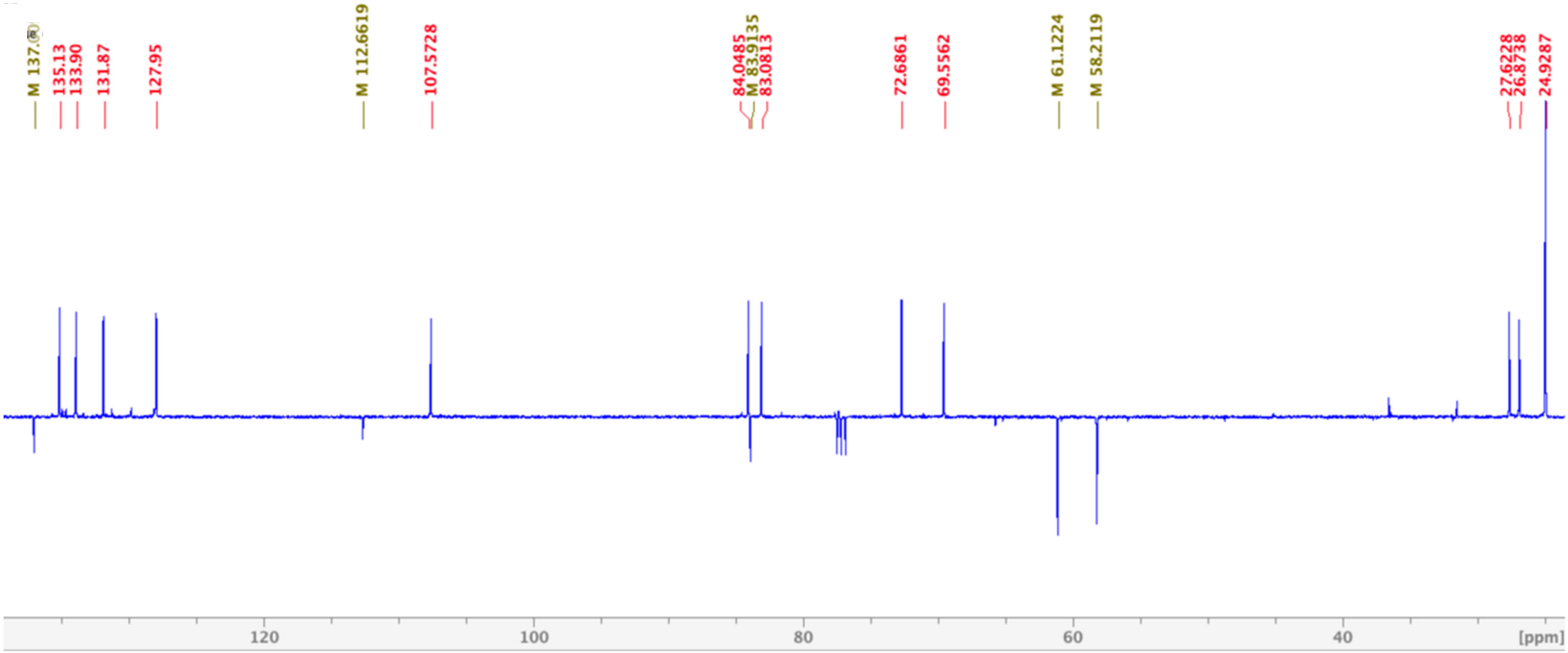

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1.35
1.30
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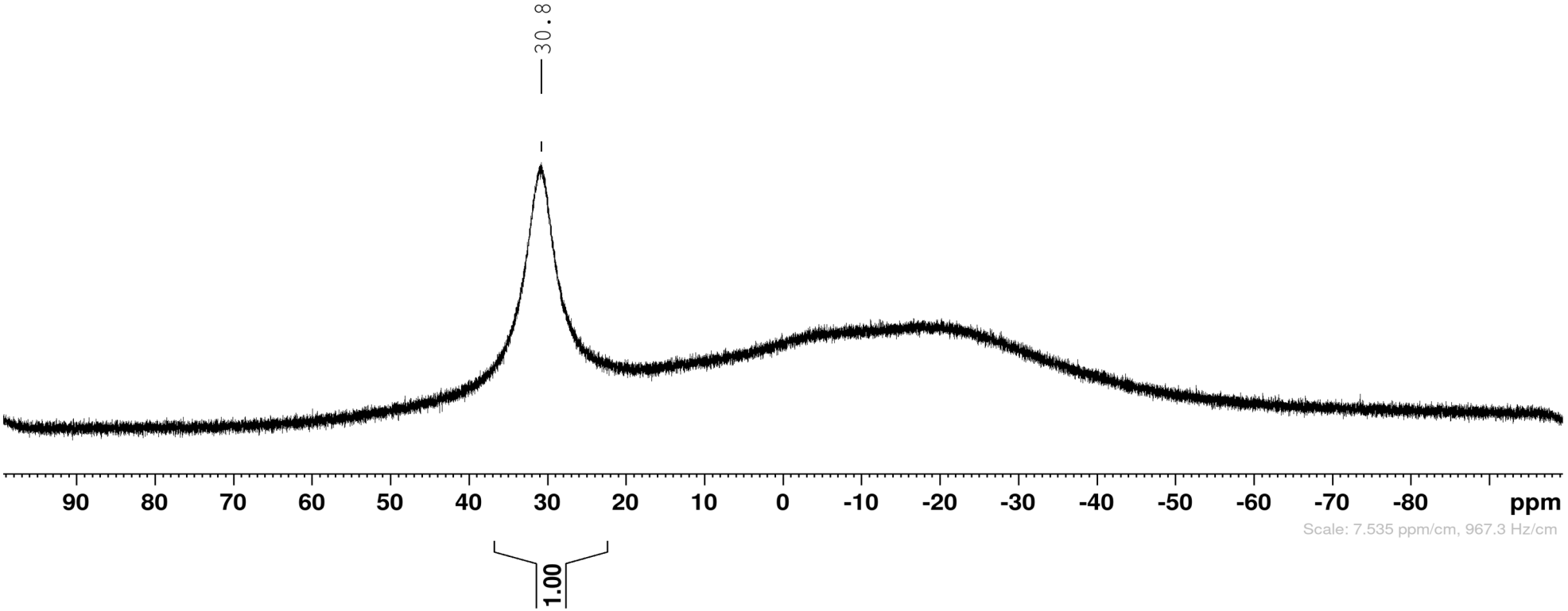
^{13}C-NMR


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131.8658
127.9363
112.6343
107.5591
84.0423
83.8947
83.0728
77.4762
77.1580
76.8398
72.6719
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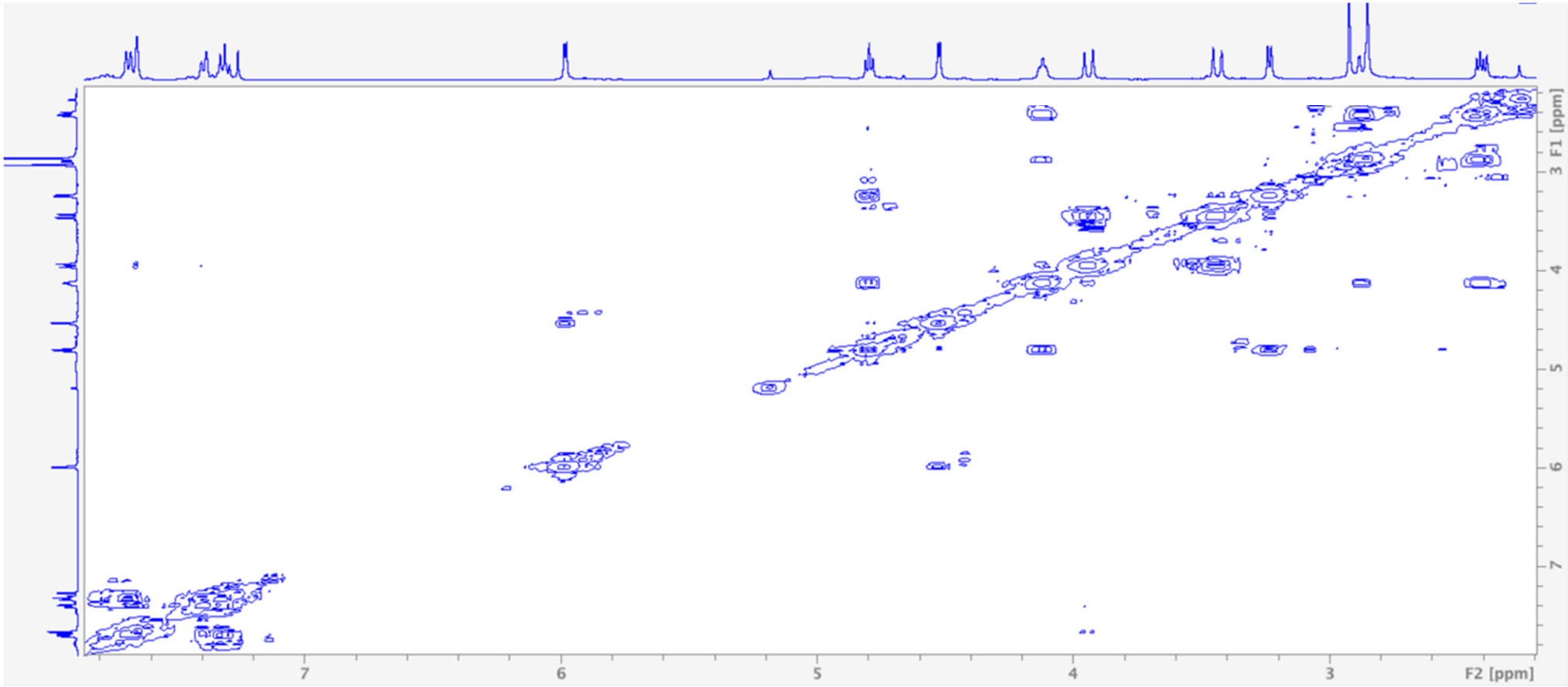
DEPT

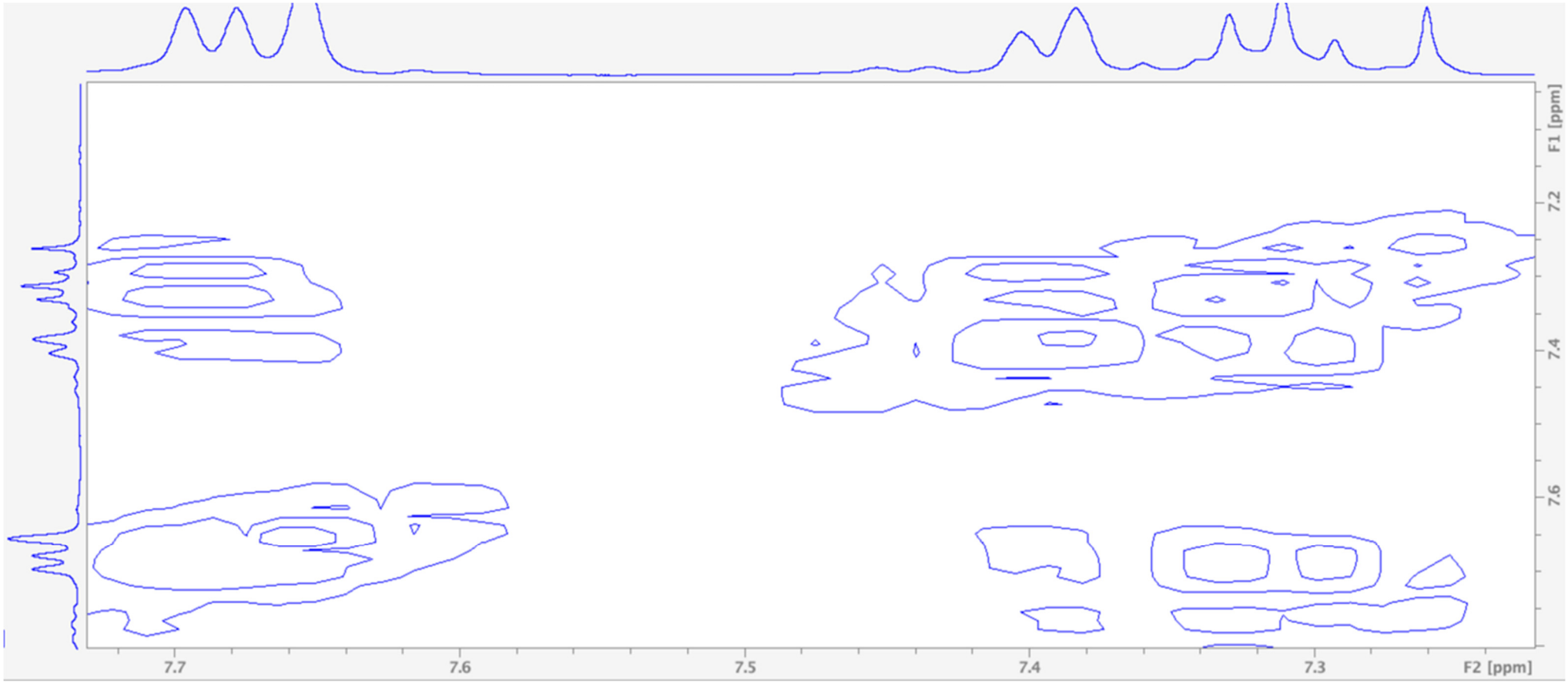

M 137.(R)
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133.90
131.87
127.95
M 112.6619
107.5728
84.0485
M 83.9135
83.0813
72.6861
69.5562
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M 58.2119
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24.9287
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100
80
60
40
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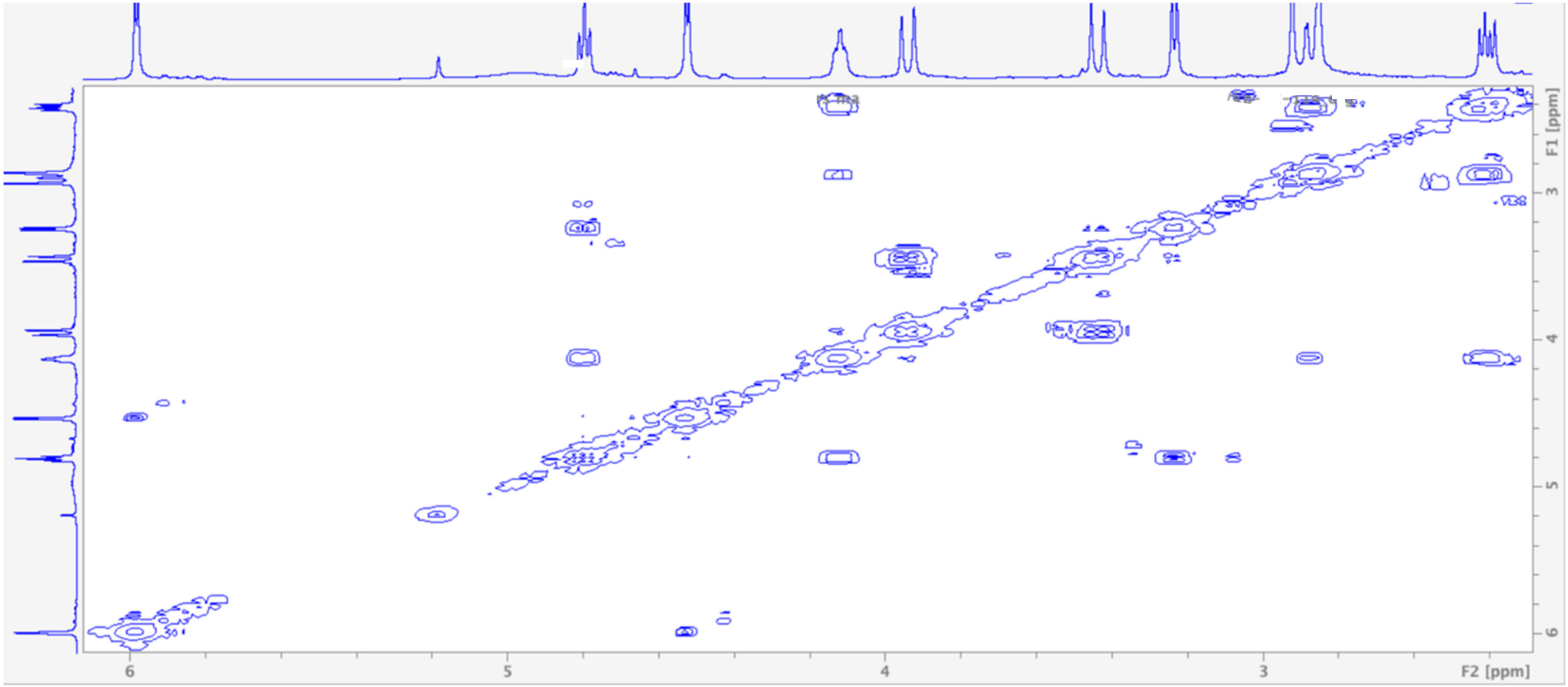
^{11}B-NMR


30.8
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
ppm
Scale: 7.535 ppm/cm, 967.3 Hz/cm
1.00

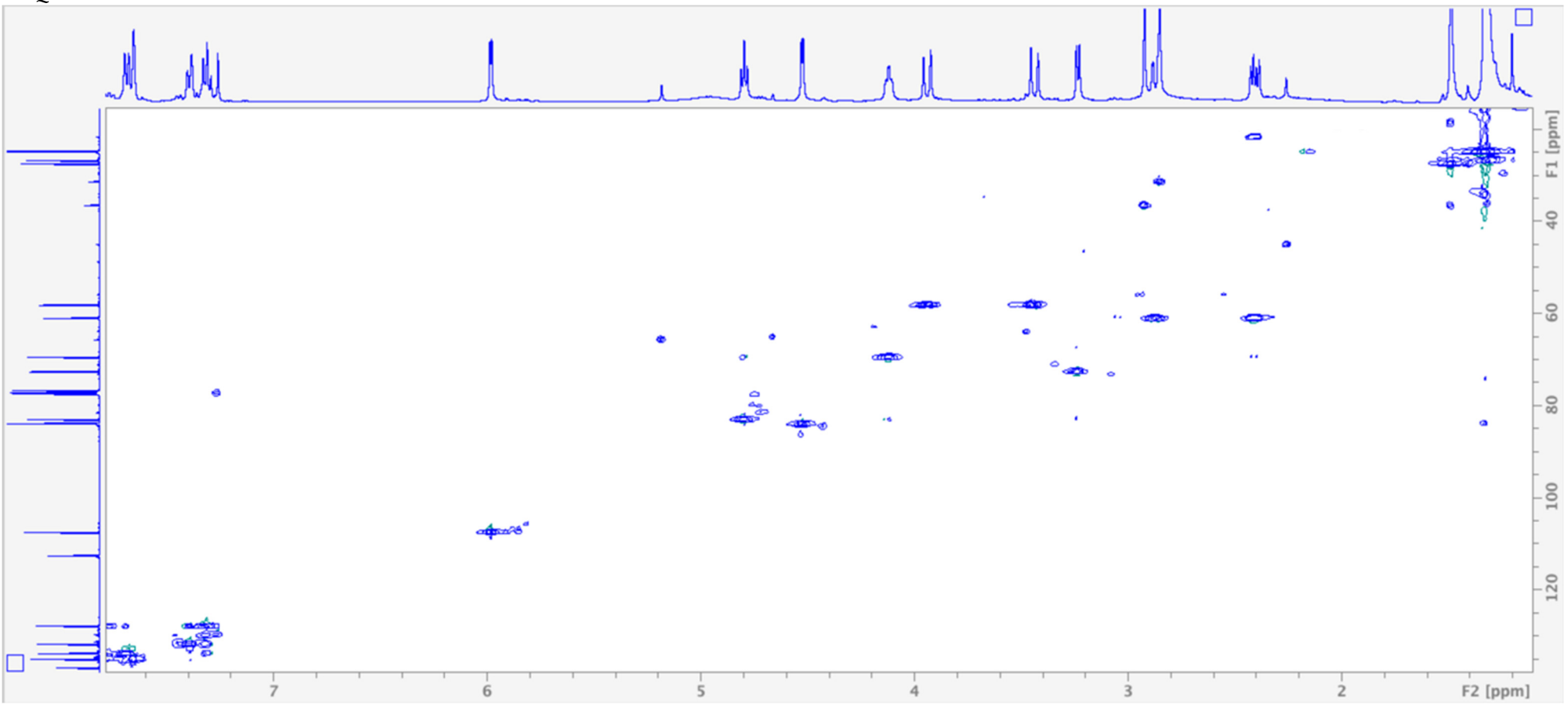
COSY

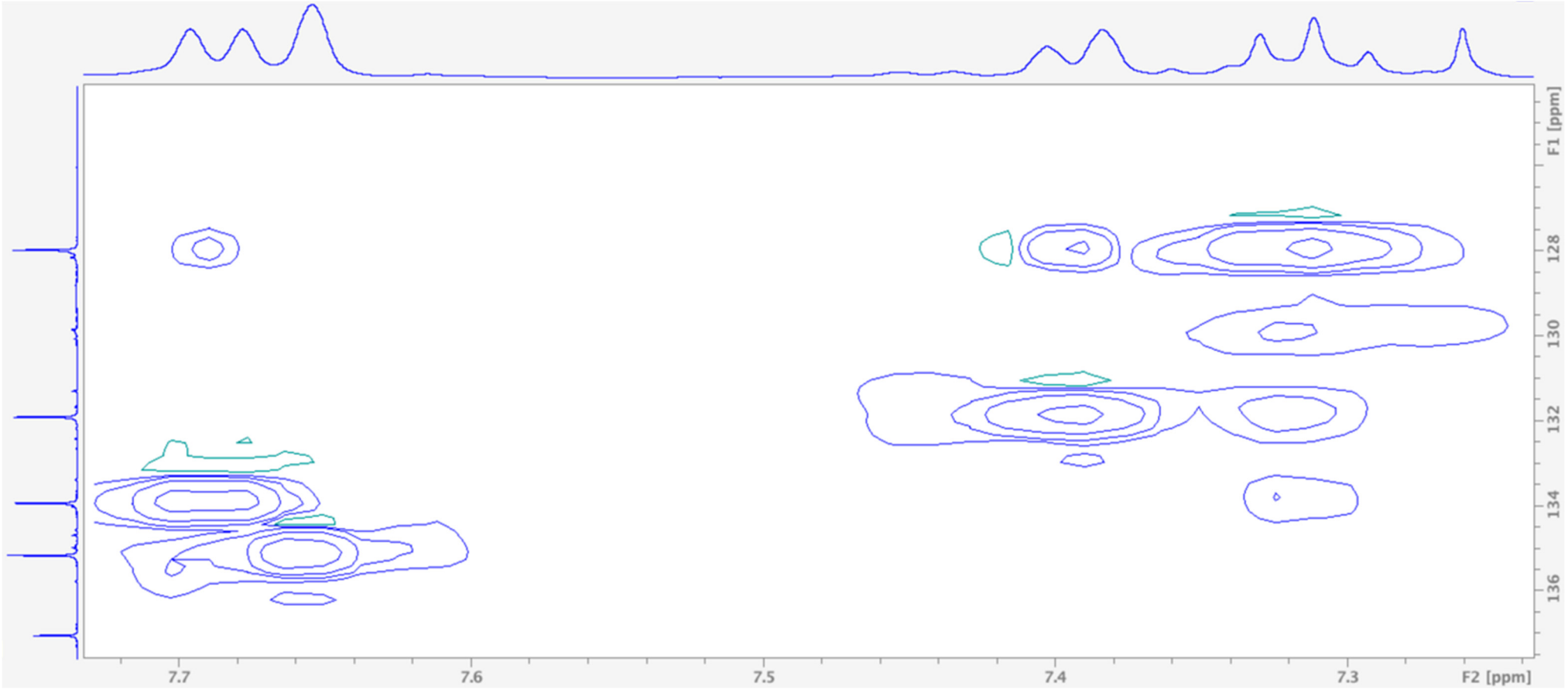



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7.4
7.6
7.7
7.6
7.5
7.4
7.3
F2 [ppm]

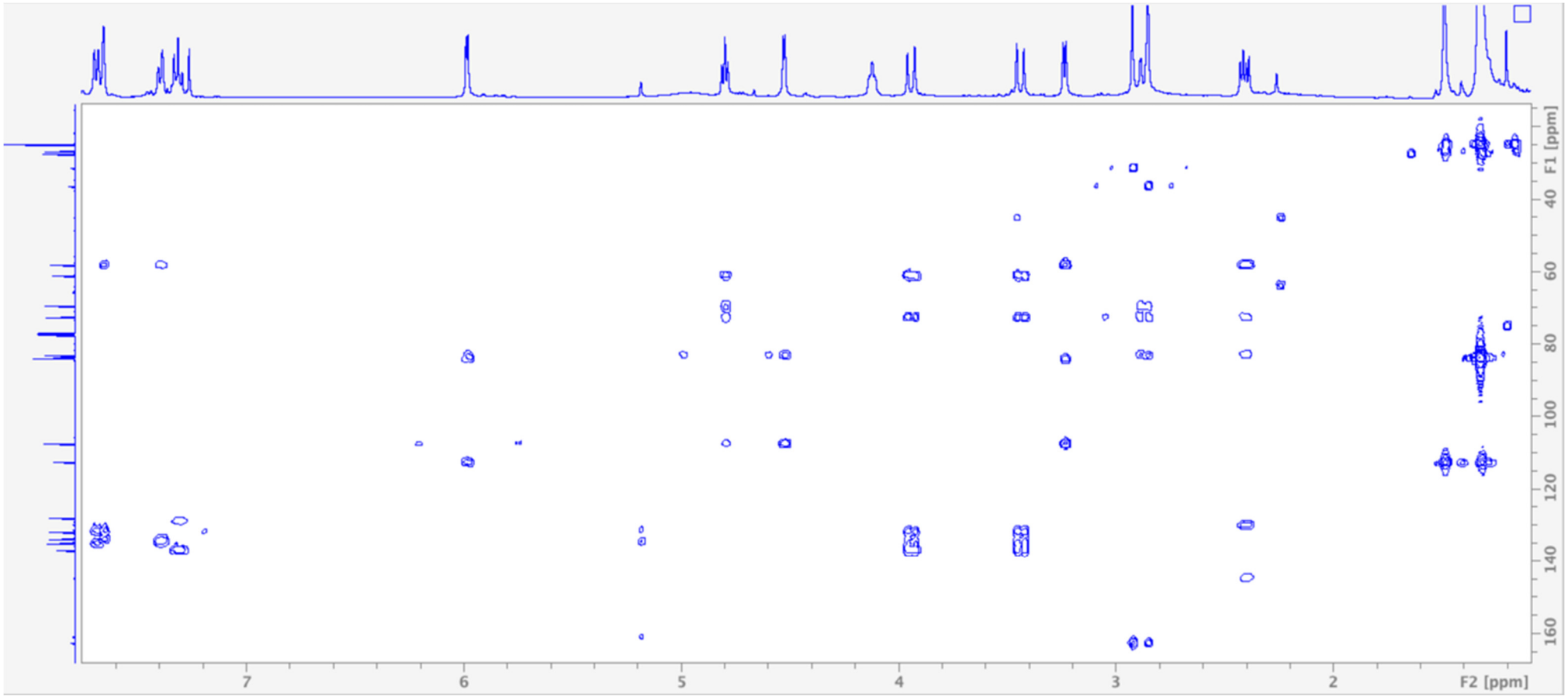

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4
5
6
6
5
4
3
F2 [ppm]

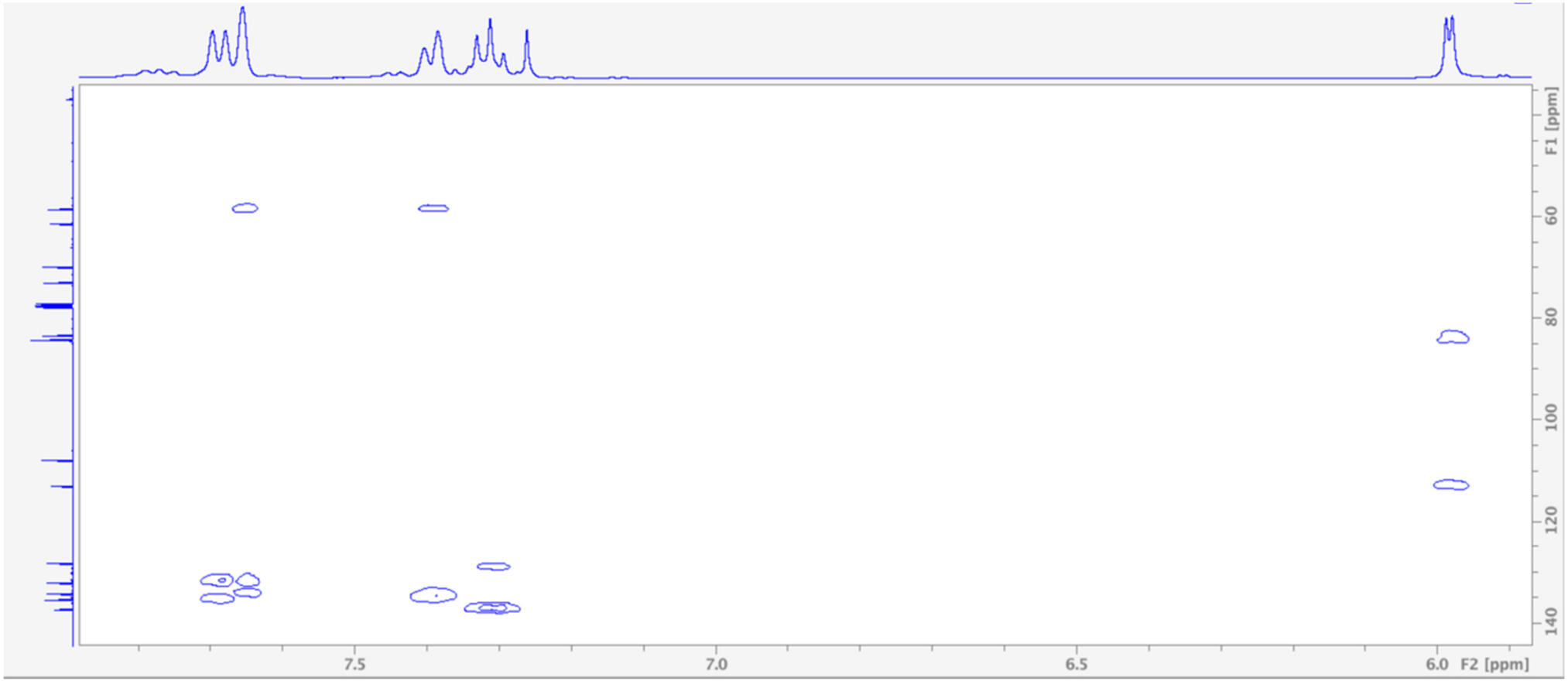
HSQC

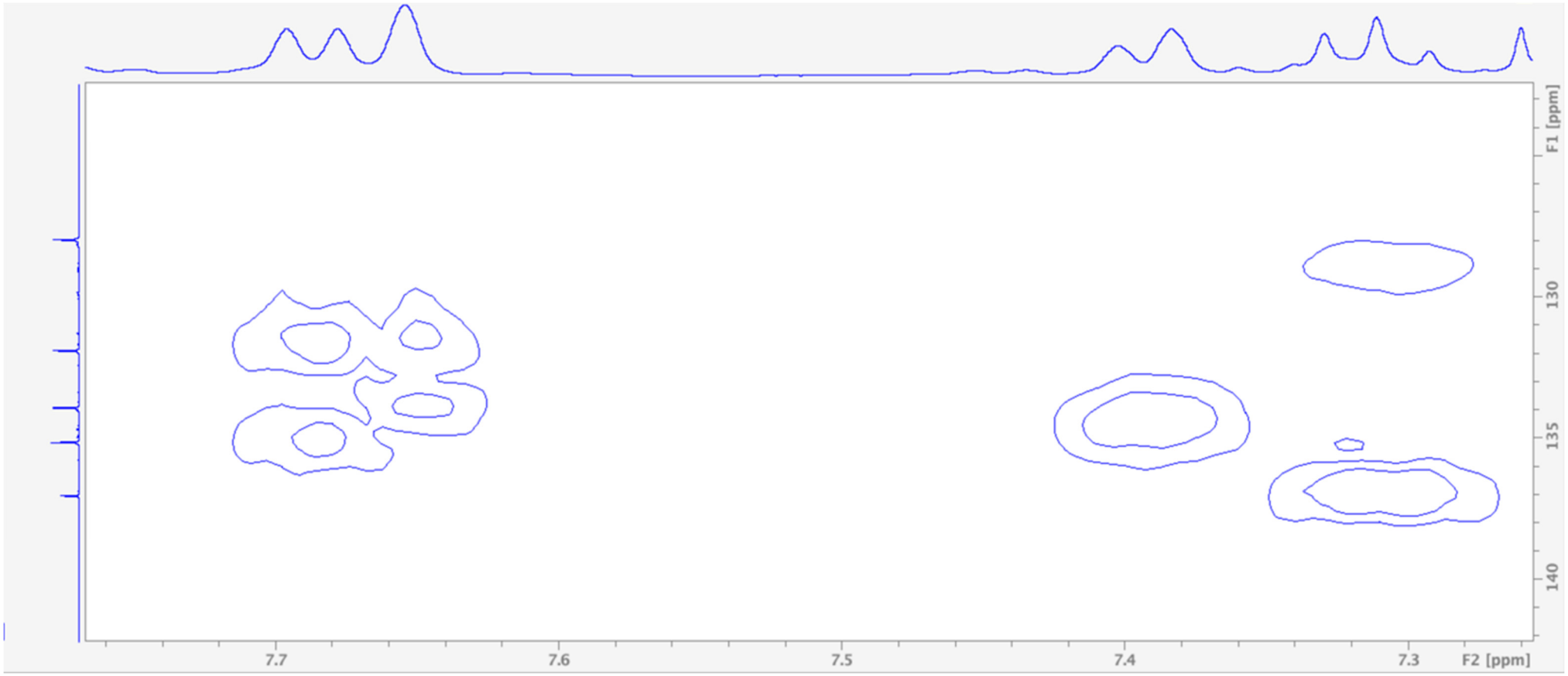


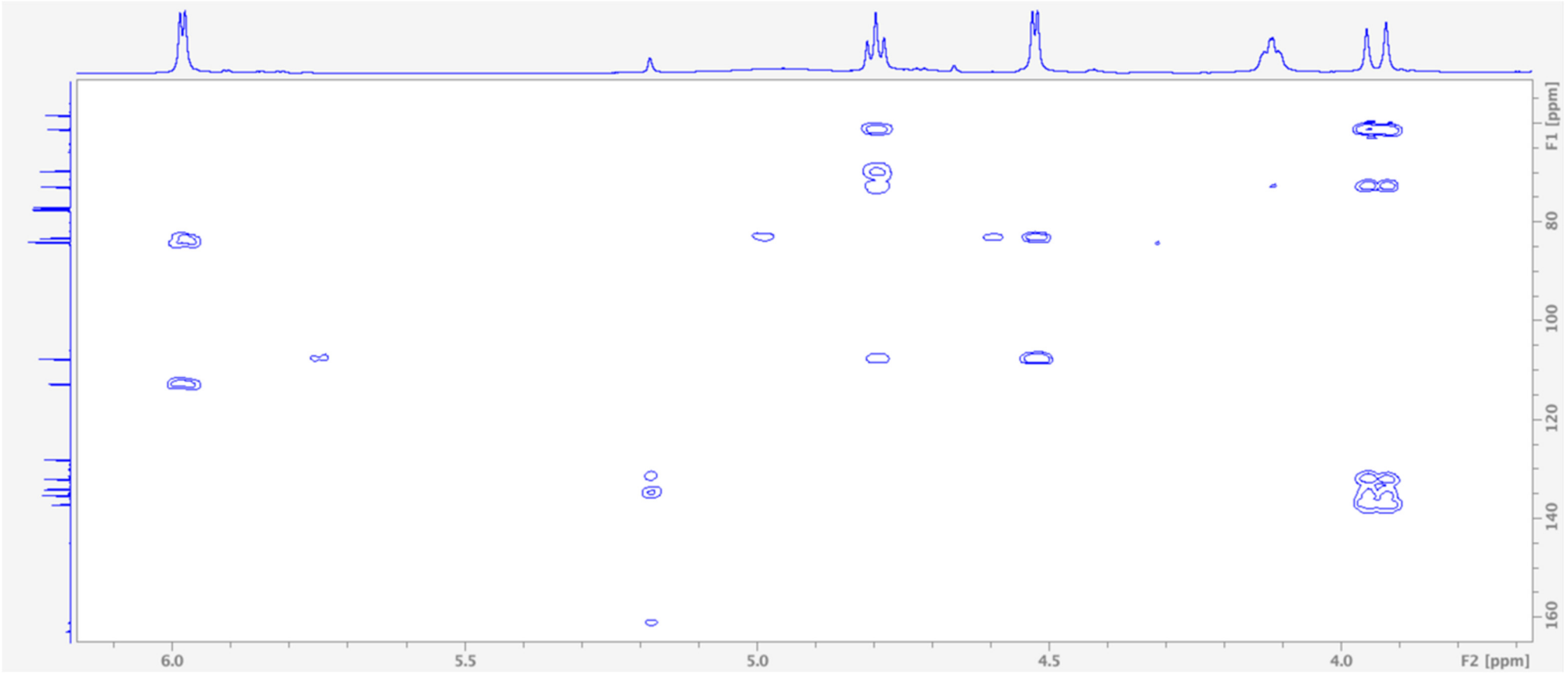

F1 [ppm]
128
130
132
134
136
7.7
7.6
7.5
7.4
7.3
F2 [ppm]

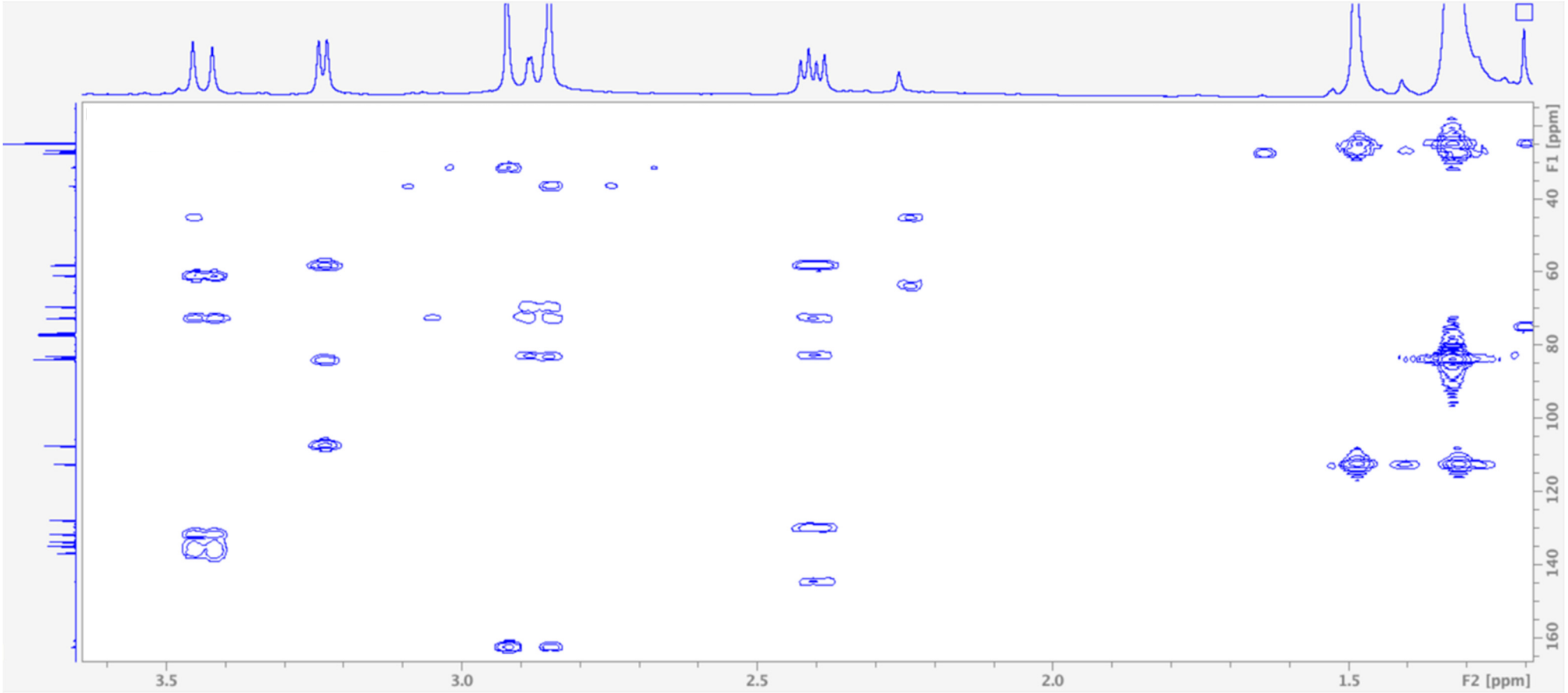
HMBC




F1 [ppm]
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80
100
120
140
7.5
7.0
6.5
6.0
F2 [ppm]


F1 [ppm]
130
135
140
7.7
7.6
7.5
7.4
7.3
F2 [ppm]


F1 [ppm]
80
100
120
140
160
6.0
5.5
5.0
4.5
4.0
F2 [ppm]


F1 [ppm]
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60
80
100
120
140
160
3.5
3.0
2.5
2.0
1.5
F2 [ppm]

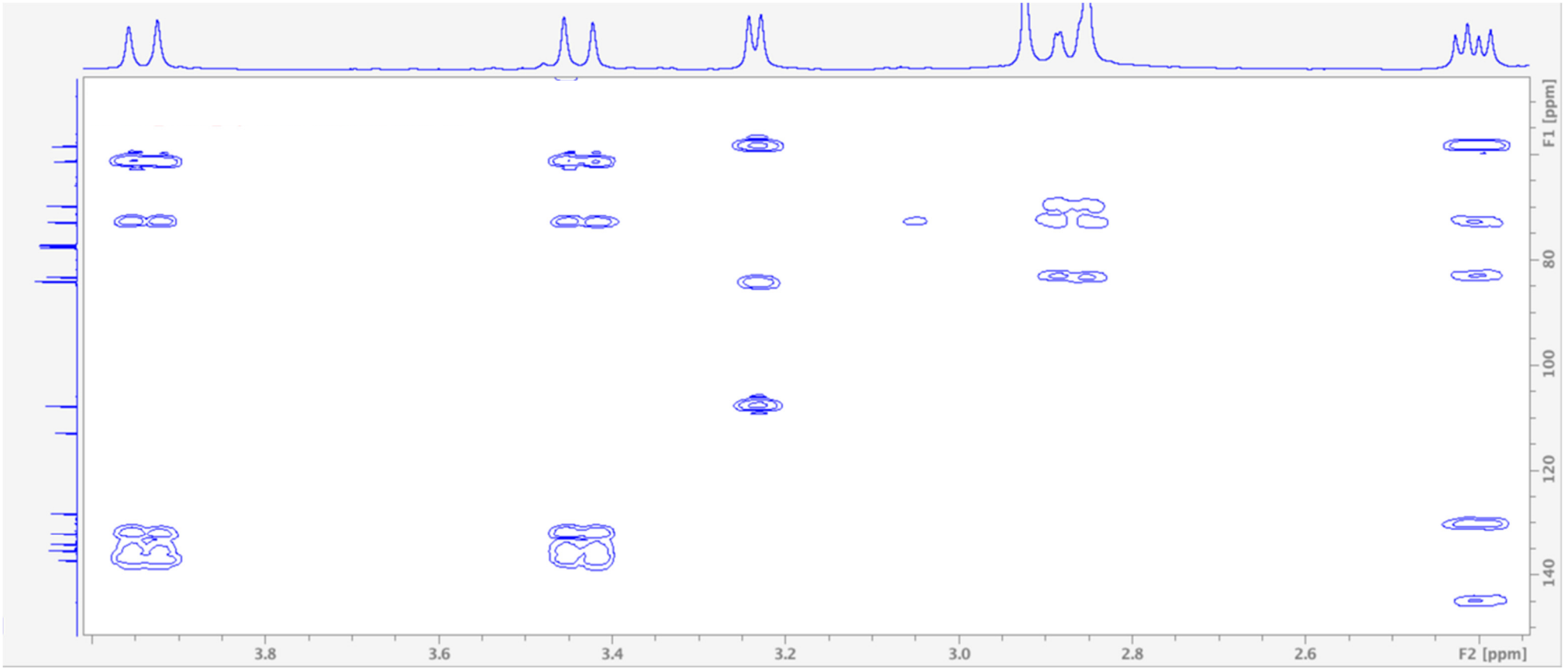
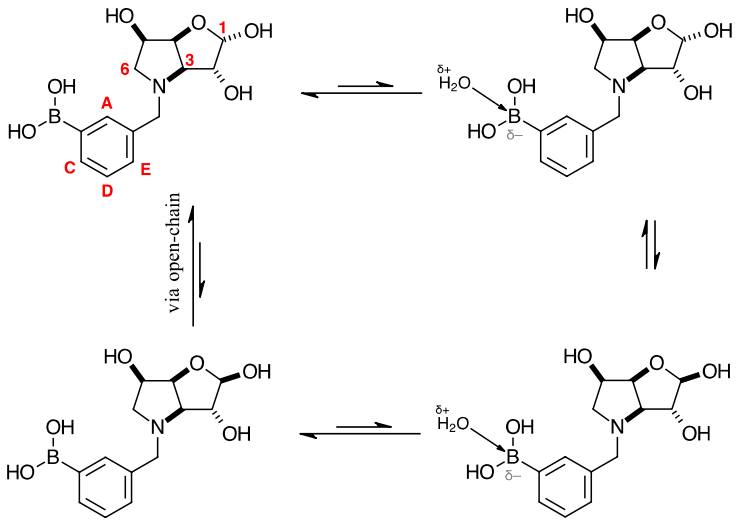

F1 [ppm]
80
100
120
140
3.8
3.6
3.4
3.2
3.0
2.8
2.6
F2 [ppm]

Figure S2. ^{1}H- (400 MHz), ^{13}C-NMR (100 MHz), DEPT, ^{11}B-NMR (128 MHz), COSY, HSQC and HMBC spectra of *N*-(3-methylphenyl boronic acid)-3,6-dideoxy-3,6-imino-D-gulofuranose **meta 3** in D_2O.



Ratio *α-fur* anomer : *β-fur* anomer : open-chain, 1.0 : 0.5 : 0.002.

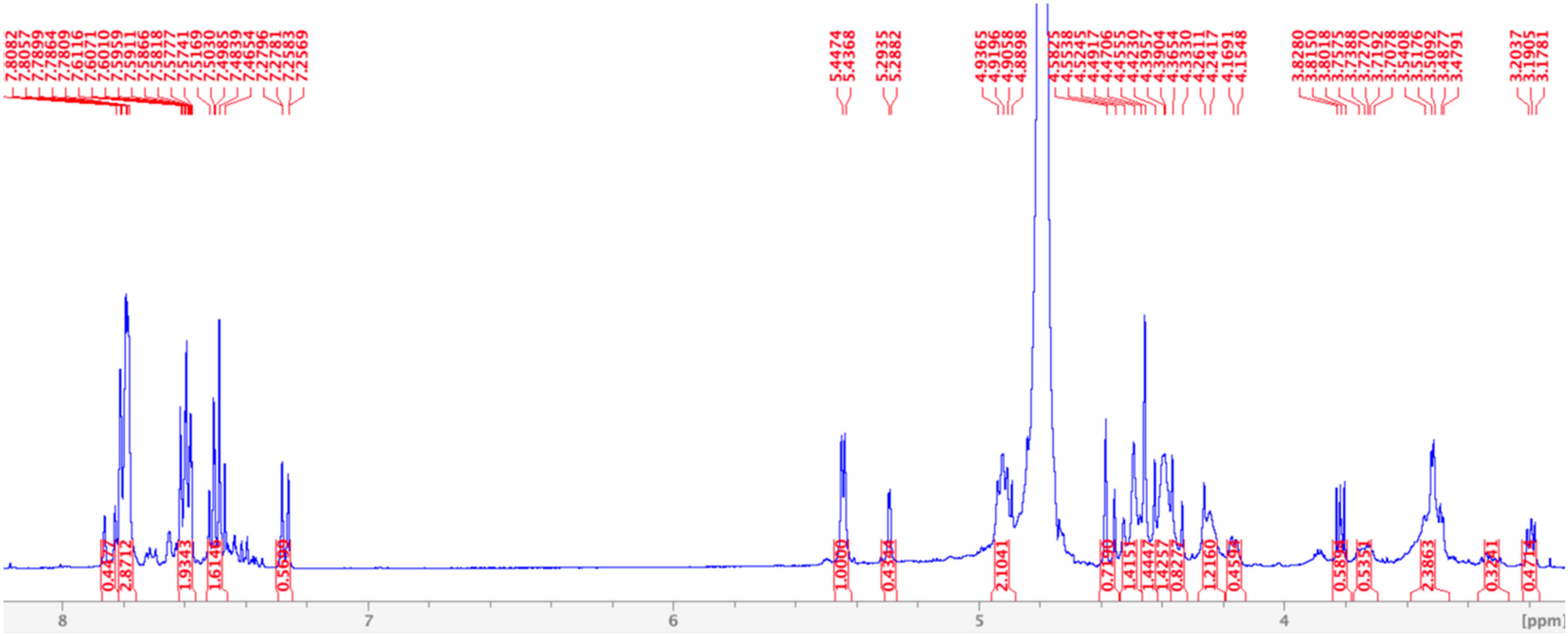
***α-fur* anomer**. **δH**: 7.80 (1H, dd, $J_{ArHC,ArHD}$ 7.6 Hz, $J_{ArHC,ArHA}$ 1.2 Hz, ArH^{C}), 7.78 (1H, d, $J_{ArHA,ArHC}$ 1.6 Hz, ArH^{A}), 7.58 (1H, dt, $J_{ArHE,ArHD}$ 7.5 Hz, $J_{ArHE,ArHA}$ 1.9 Hz, ArH^{E}), 7.48 (1H, t, $J_{ArHD,ArHC/HE}$ 7.6 Hz, ArH^{D}), 5.44 (1H, d, $J_{H\text{-}1,H\text{-}2}$ 4.3 Hz, H-1), 4.93 (1H, app-d, *J* 5.6 Hz, H-4), 4.51 (1H, d, $J_{Ha,Hb}$ 13.1 Hz, H^{a}), 4.44 (1H, d, $J_{Hb,Ha}$ 13.1 Hz, H^{b}), 4.42-4.35 (2H, m, H-2 and H-5), 4.27-4.20 (1H, m, H-3), 3.60-3.52 (1H, m, H-6), 3.49 (1H, dd, $J_{H\text{-}6',H\text{-}6}$ 12.2 Hz, $J_{H\text{-}6',H\text{-}5}$ 3.5 Hz, H-6'); **δC**: 142.4 (ArC_{quat}), 136.2 (ArC^{A}), 135.4 (ArC^{C}), 133.5 (ArC^{E}), 128.9 (ArC^{D}), 99.0 (C-1, broadened, 89 Hz), 78.9 (C-4, broadened, 88 Hz), 72.8 (C-2 and C-3, broadened, 68 Hz), 67.4 (C-5), 61.9-59.7 (C-6, broadened, 226 Hz), 59.7-58.4 (CH_2Ar broadened, 170 Hz); Not discernible: ArC^{B}-B.

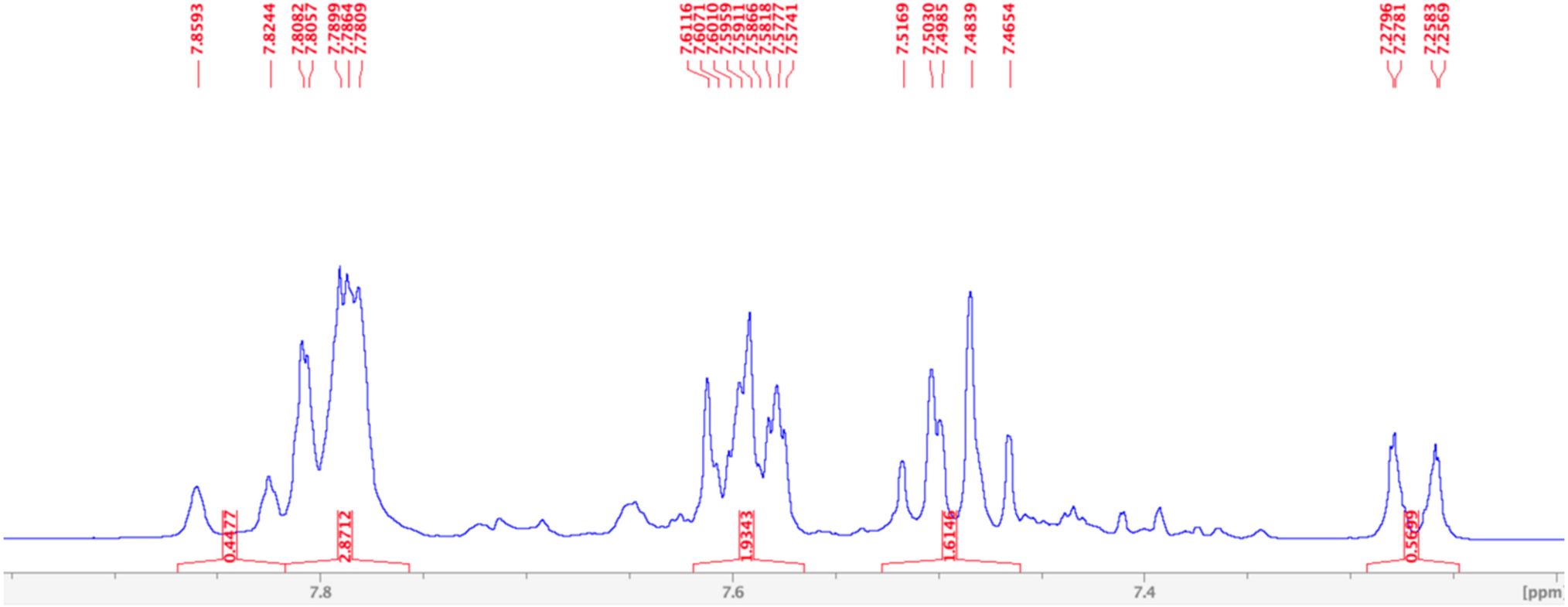
***β-fur* anomer**. **δH**: 7.82-7.76 (1H, obscured, ArH^{A}), 7.60 (1H, dd, $J_{ArHC,ArHD}$ 5.5 Hz, $J_{ArHC,ArHA}$ 1.9 Hz, ArH^{C}), 7.50 (1H, t, $J_{ArHD,ArHC/HE}$ 7.3 Hz, ArH^{D}), 7.27 (1H, dd, $J_{ArHE,ArHD}$ 8.5 Hz, $J_{ArHE,ArHA}$ 0.6 Hz, ArH^{E}), 5.29 (1H, d, $J_{H\text{-}1,H\text{-}2}$ 2.1 Hz, H-1), 4.91 (1H, app-d, $J_{H\text{-}4,H\text{-}3}$ 5.6 Hz, H-4), 4.57 (1H, d, $J_{Ha,Hb}$ 13.0 Hz, H^{a}), 4.35 (1H, d, $J_{Hb,Ha}$ 13.0 Hz, H^{b}), 4.49-4.44 (1H, obscured, H-5), 4.42-4.35 (2H, m, H-2), 4.16 (1H, dd, $J_{H\text{-}3,H\text{-}4}$ 5.8 Hz, $J_{H\text{-}3,H\text{-}2}$ 1.8 Hz, H-3), 3.74 (1H, app-dd, $J_{H\text{-}6,H\text{-}6'}$ 12.3 Hz, $J_{H\text{-}6,H\text{-}5}$ 4.4 Hz, H-6), 3.52-3.47 (1H, obscured, H-6'); **δC**: 139.6 (ArC_{quat}), 135.9 (ArC^{A}), 135.5 (ArC^{C}), 129.4 (ArC^{E}), 129.0 (ArC^{D}), 103.1 (C-1, broadened, 29 Hz), 80.7 (C-4, slightly broadened, 30 Hz), 72.7 (C-2 and C-3, broadened, 68 Hz), 67.5 (C-5), 61.9-59.7 (C-6, broadened, 226 Hz), 59.7-58.4 (CH_2Ar broadened, 170 Hz); Not discernible: ArC^{B}-B.

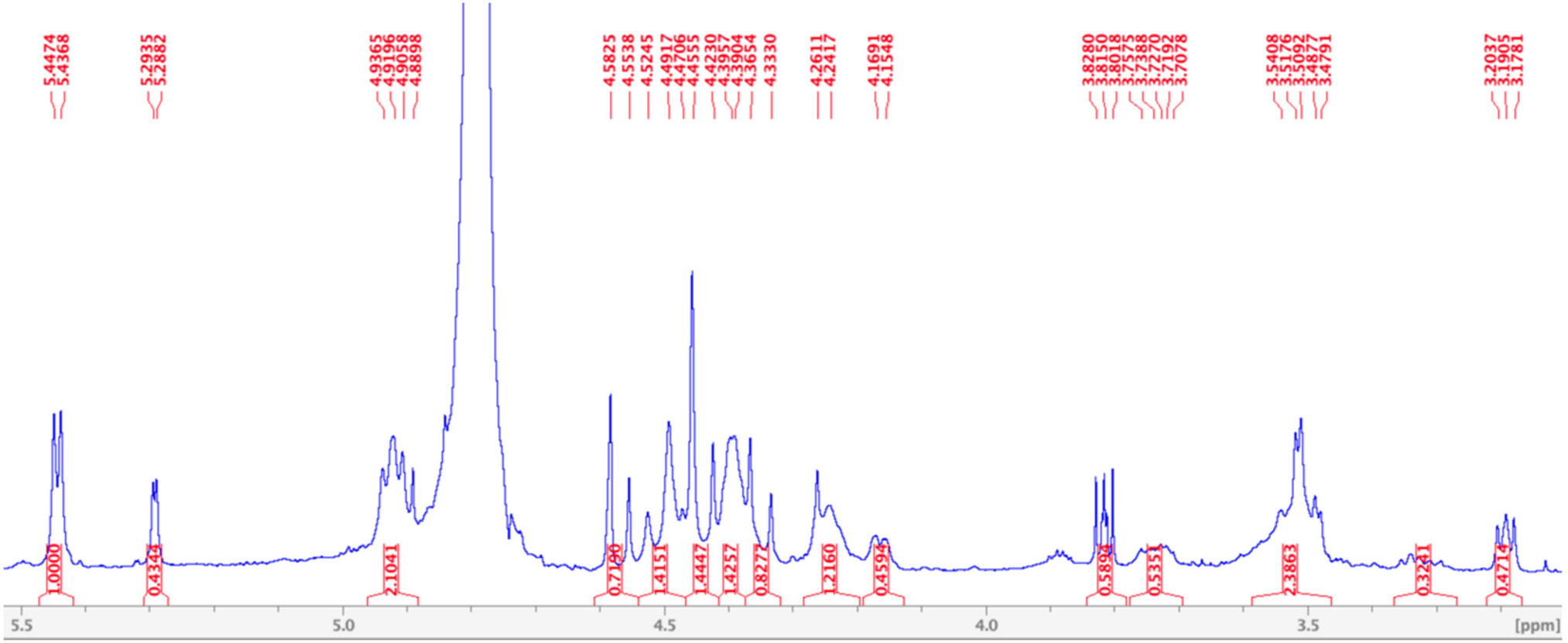
Open-chain. **δH**: 9.86 (1H, s, CHO).

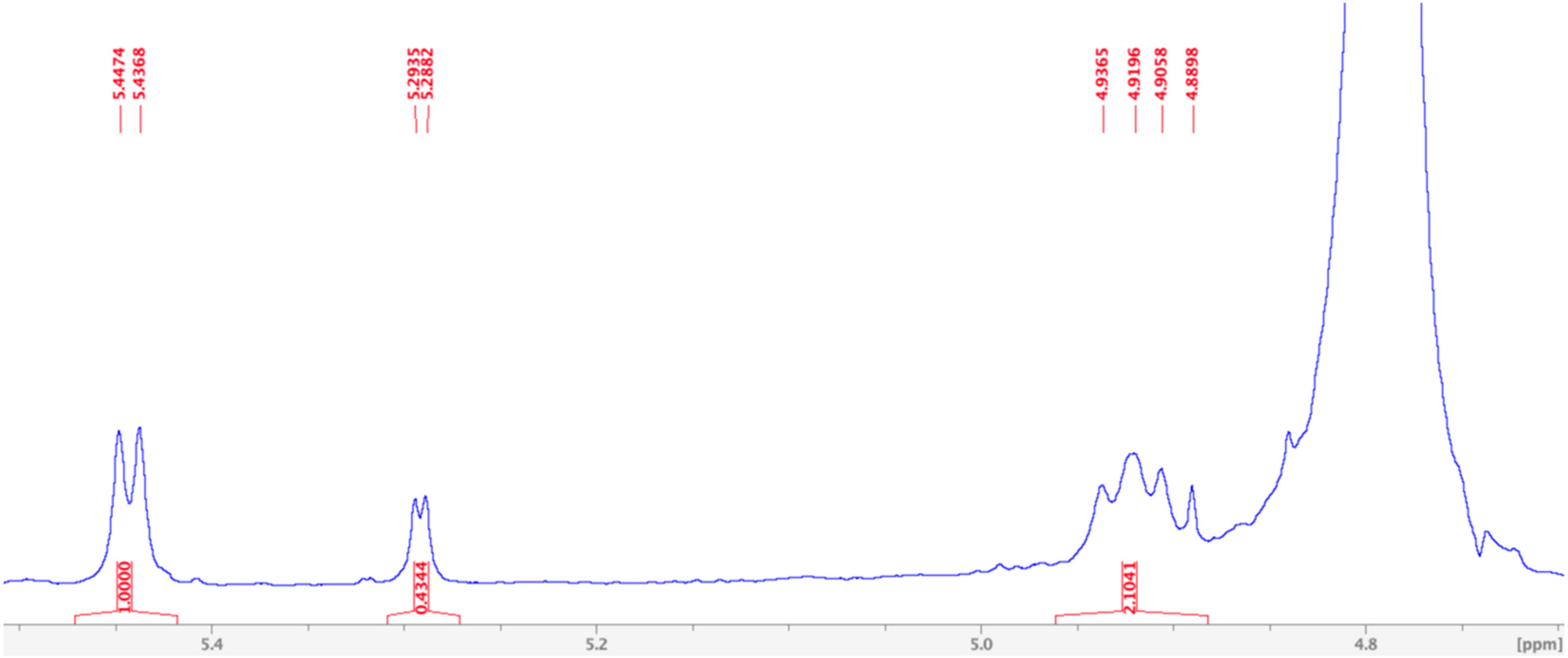
δB: 28.6 (broad, integration: 6.5), 19.2 (sharp, integration: 1.0).

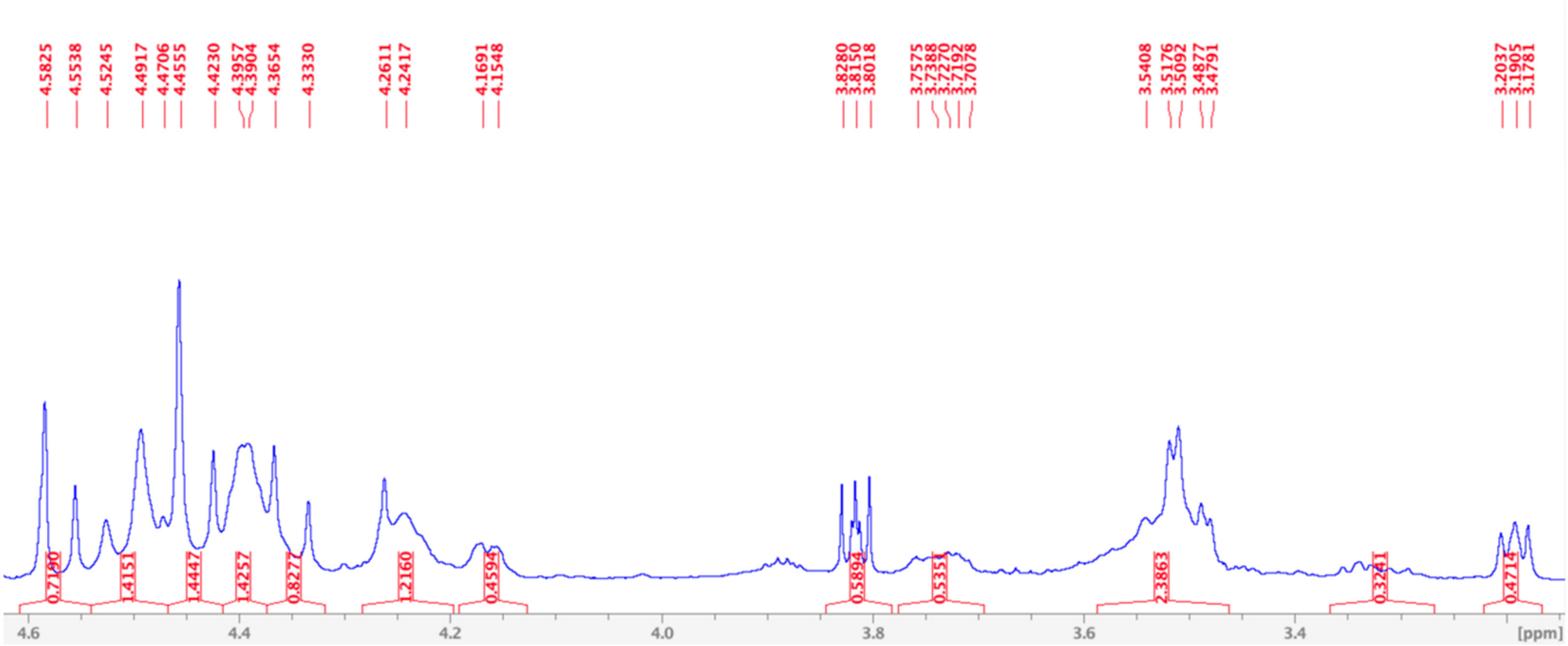
^{1}H-NMR

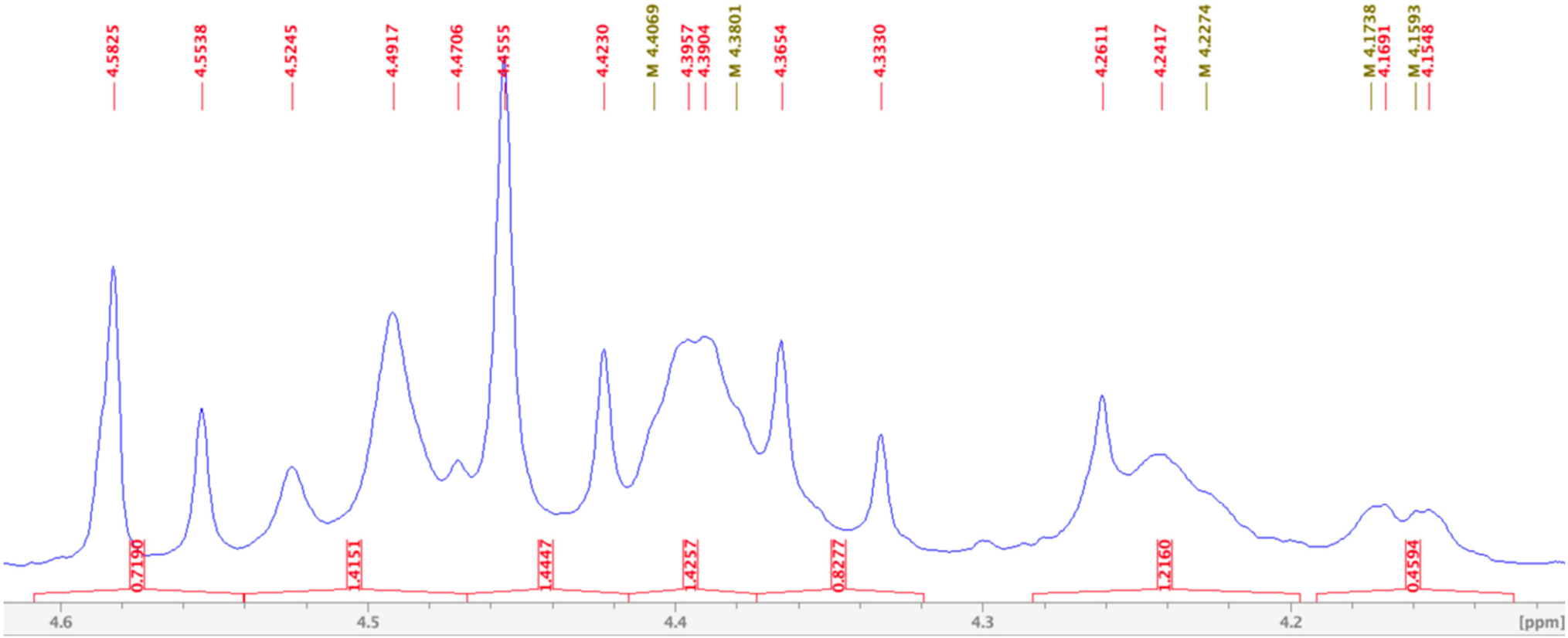

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7.5866
7.5818
7.5777
7.5741
7.5169
7.5030
7.4985
7.4839
7.4654
7.2796
7.2781
7.2583
7.2569
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5.2935
5.2882
4.9365
4.9196
4.9058
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4.5825
4.5538
4.5245
4.4917
4.4706
4.4555
4.4230
4.3957
4.3904
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4.2611
4.2417
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4.1548
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3.8018
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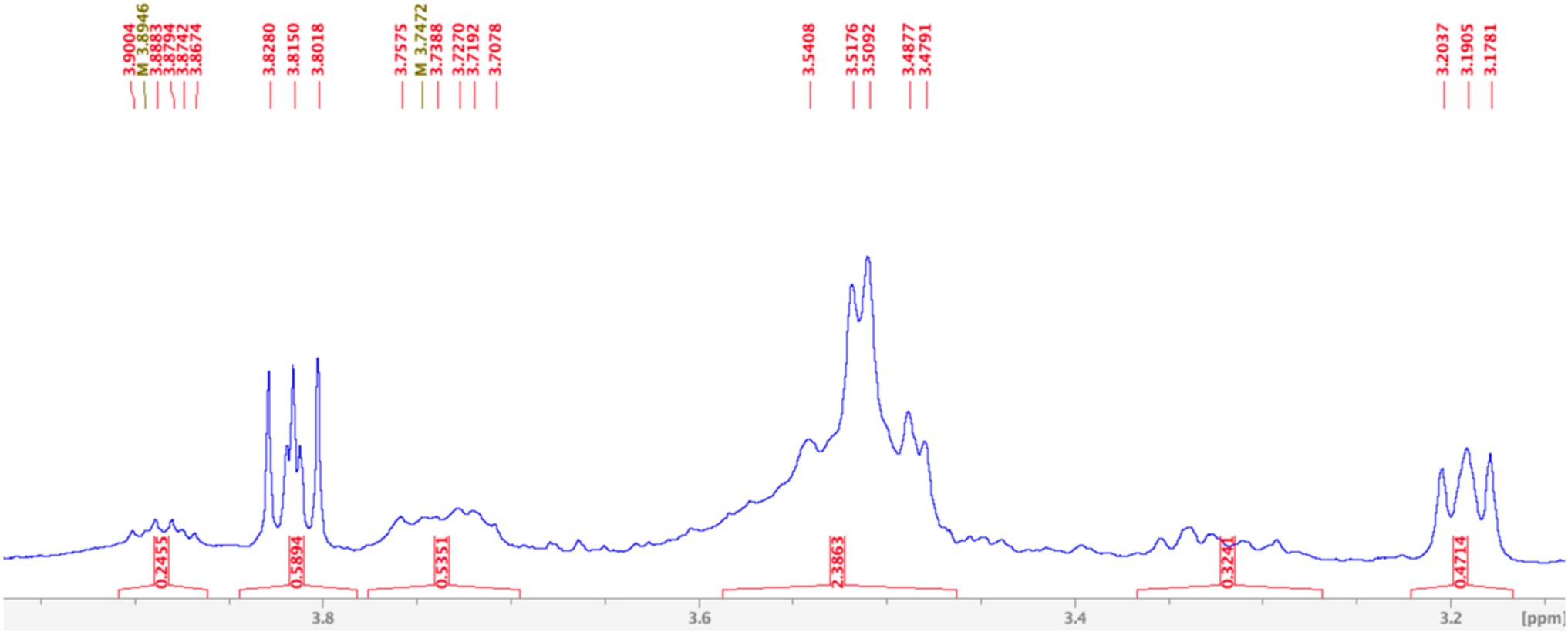

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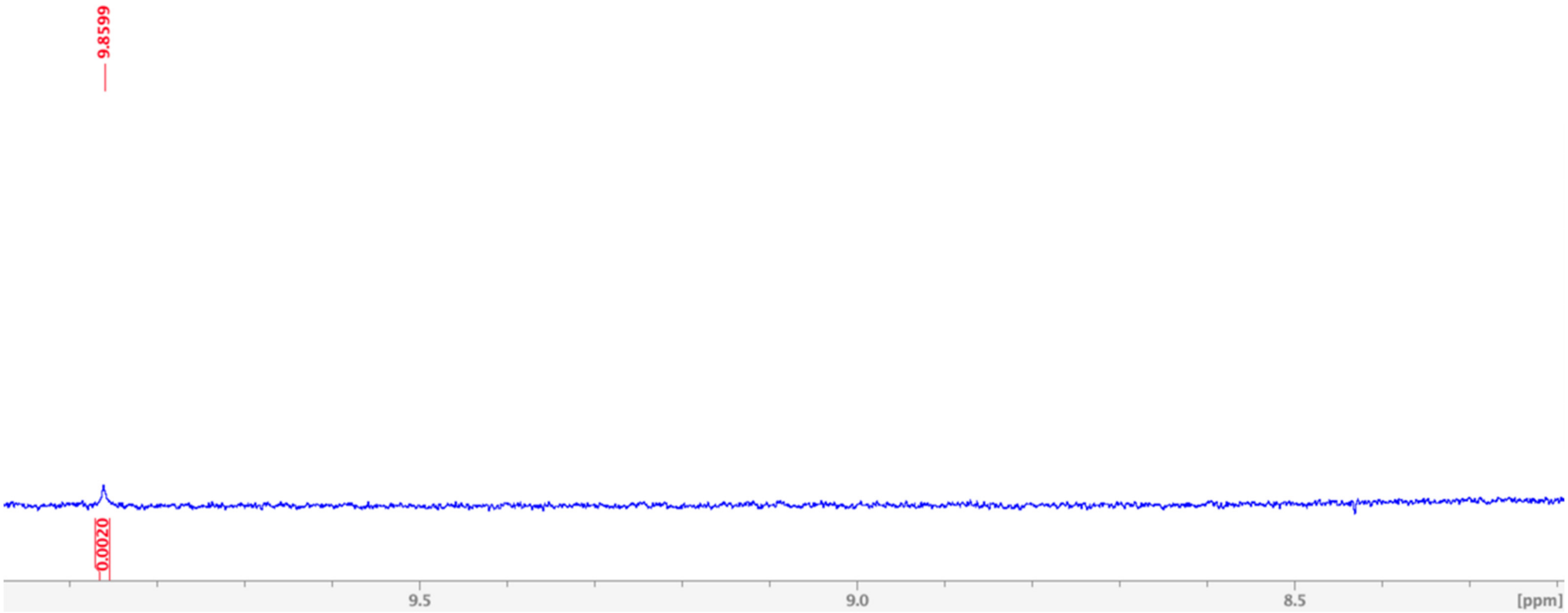

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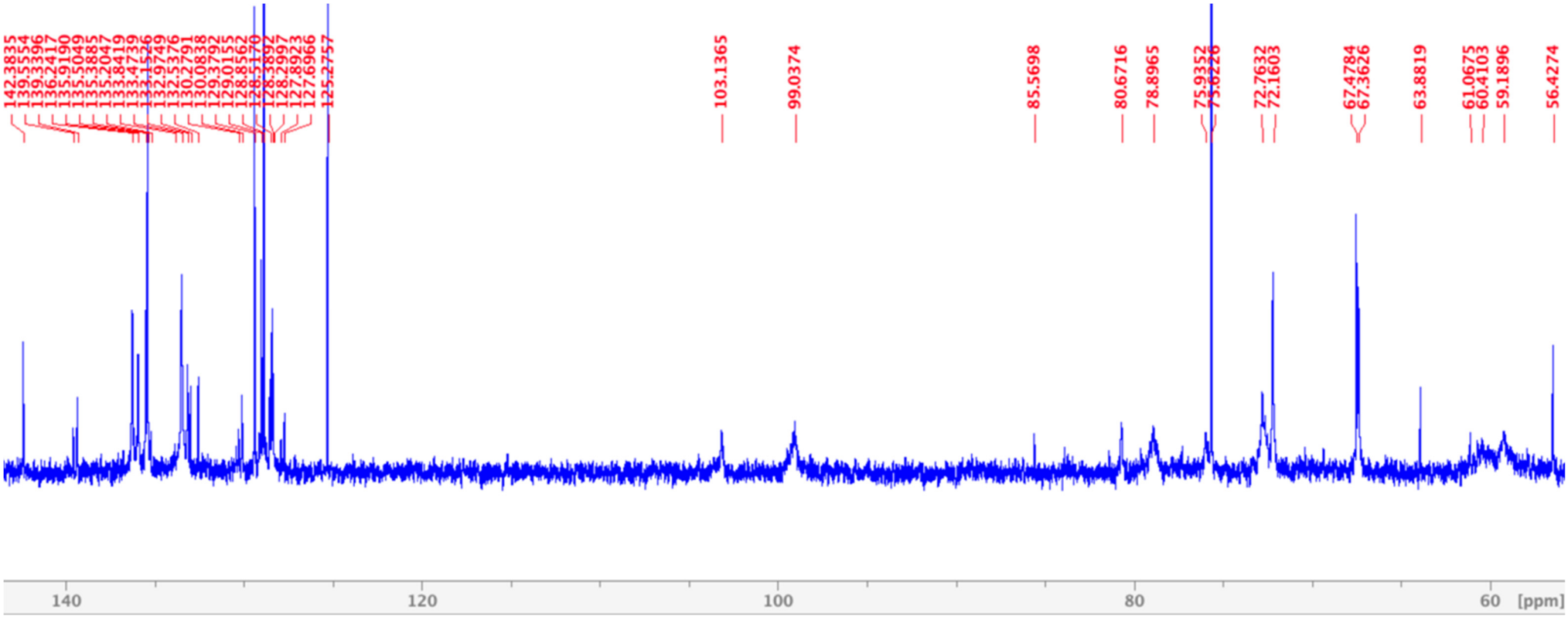

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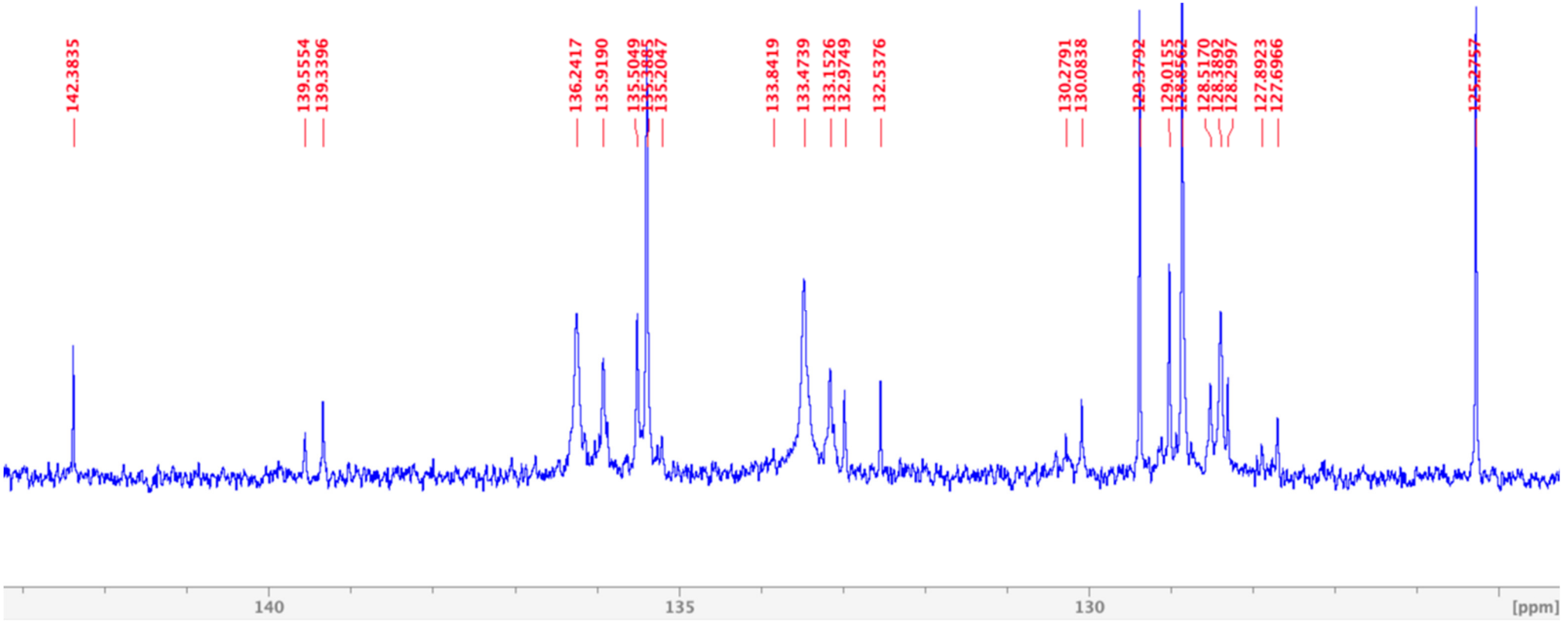

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M 4.3801
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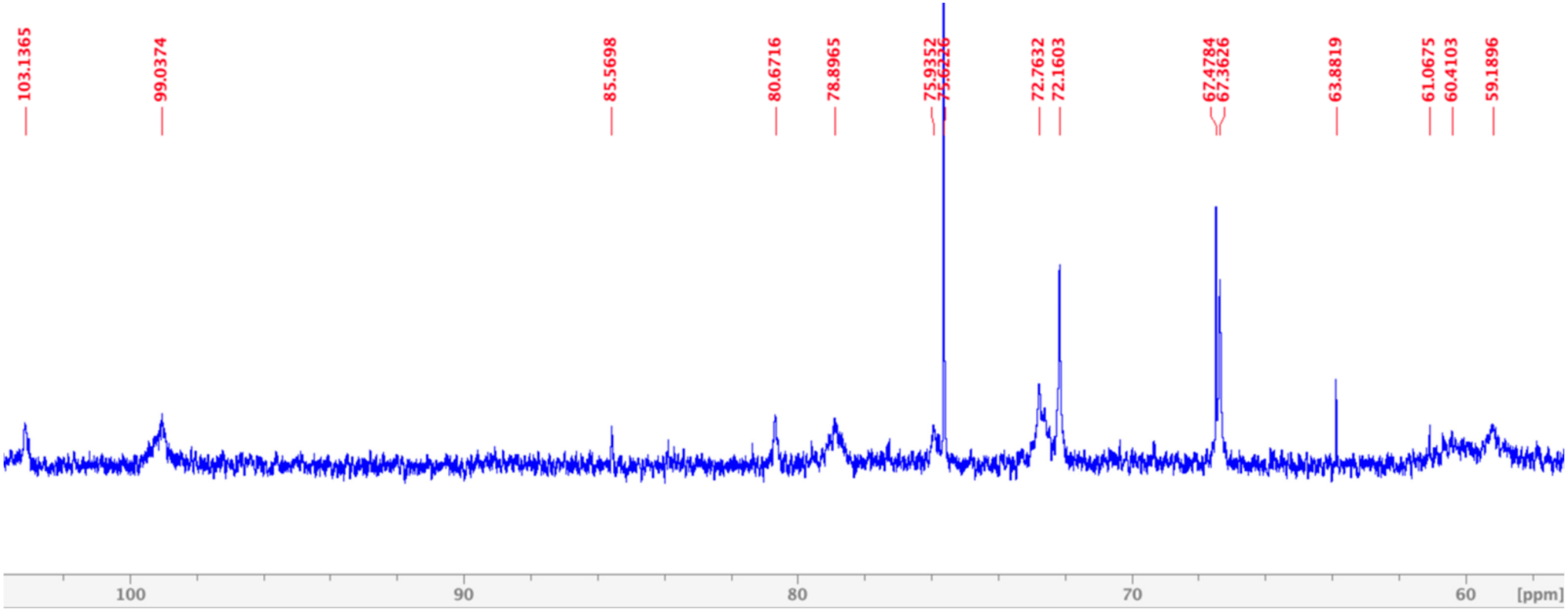

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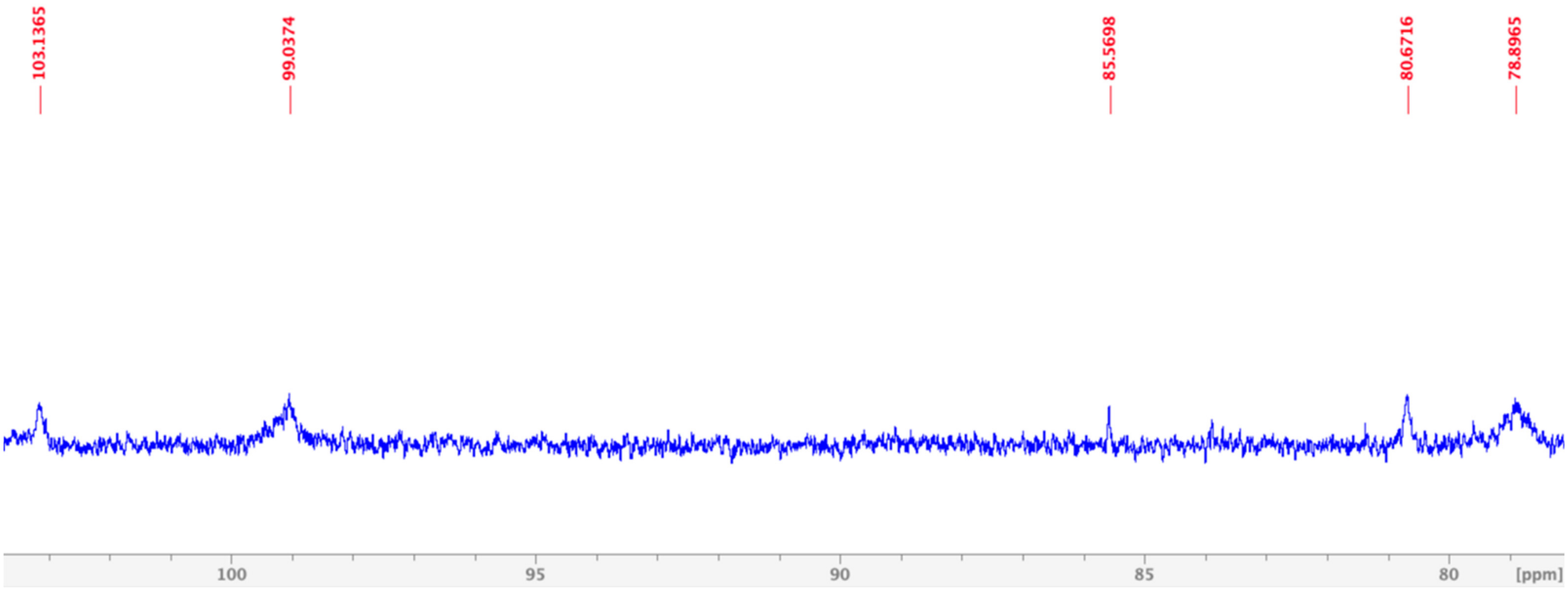

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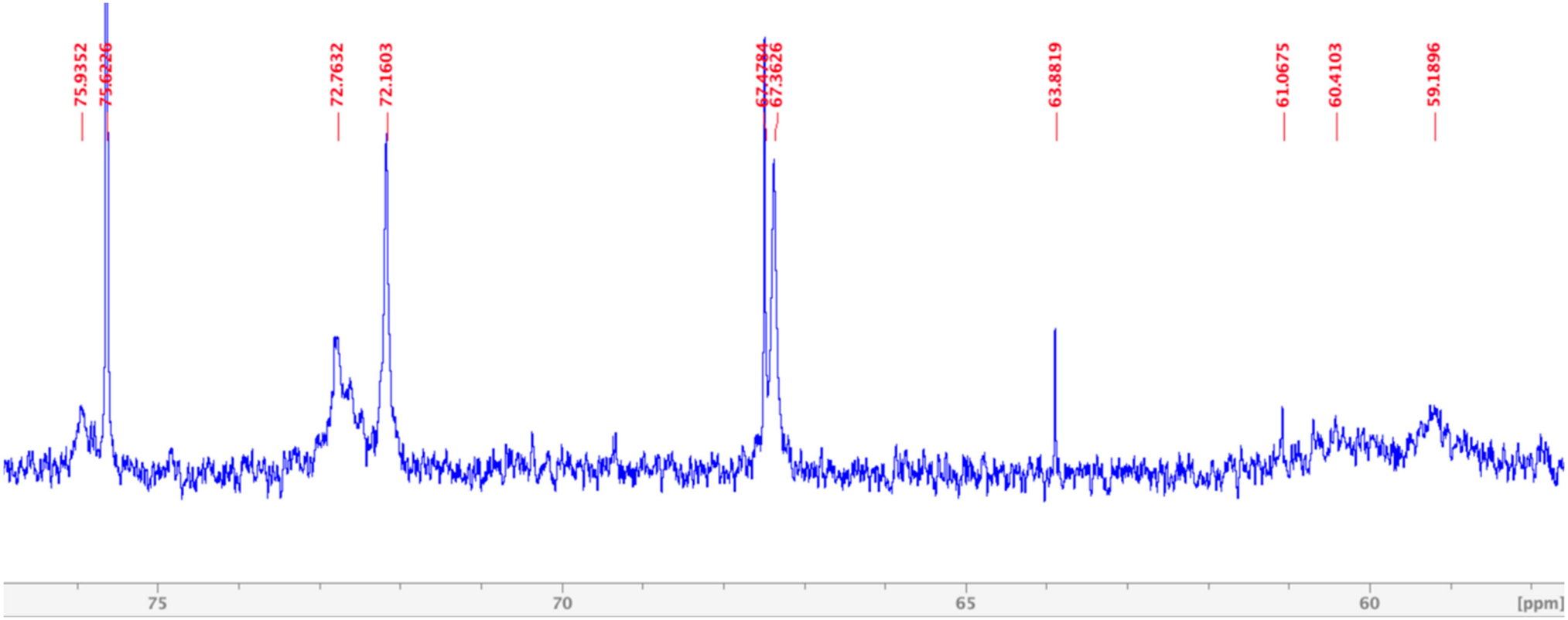
^{13}C-NMR


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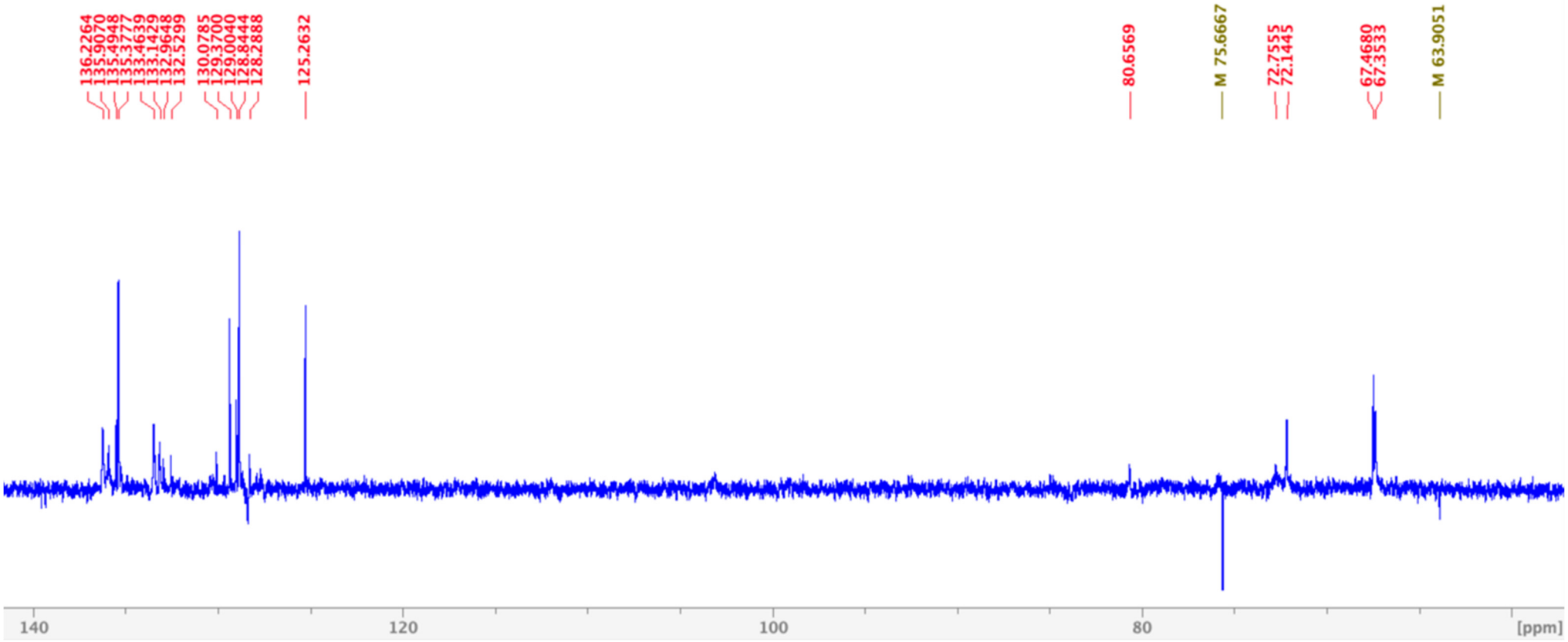

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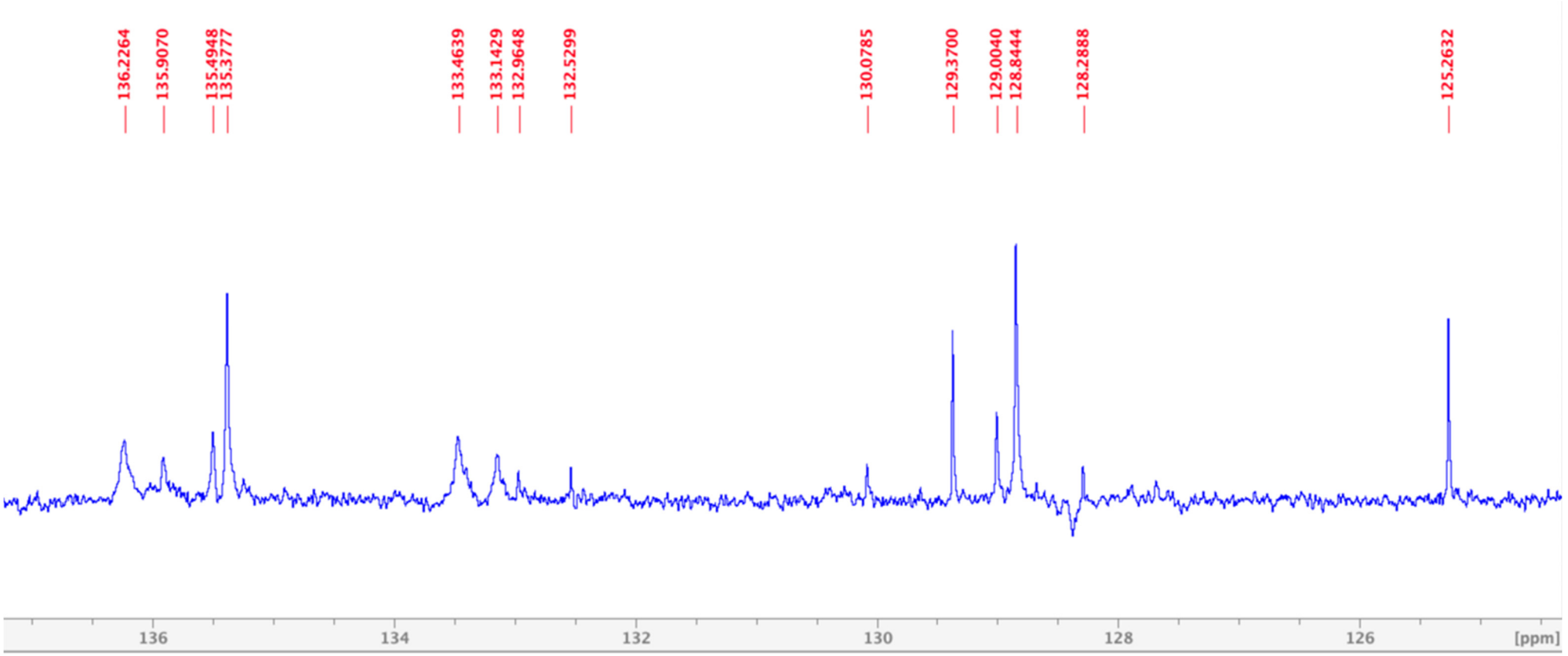

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63.8819
61.0675
60.4103
59.1896
100
90
80
70
60
[ppm]

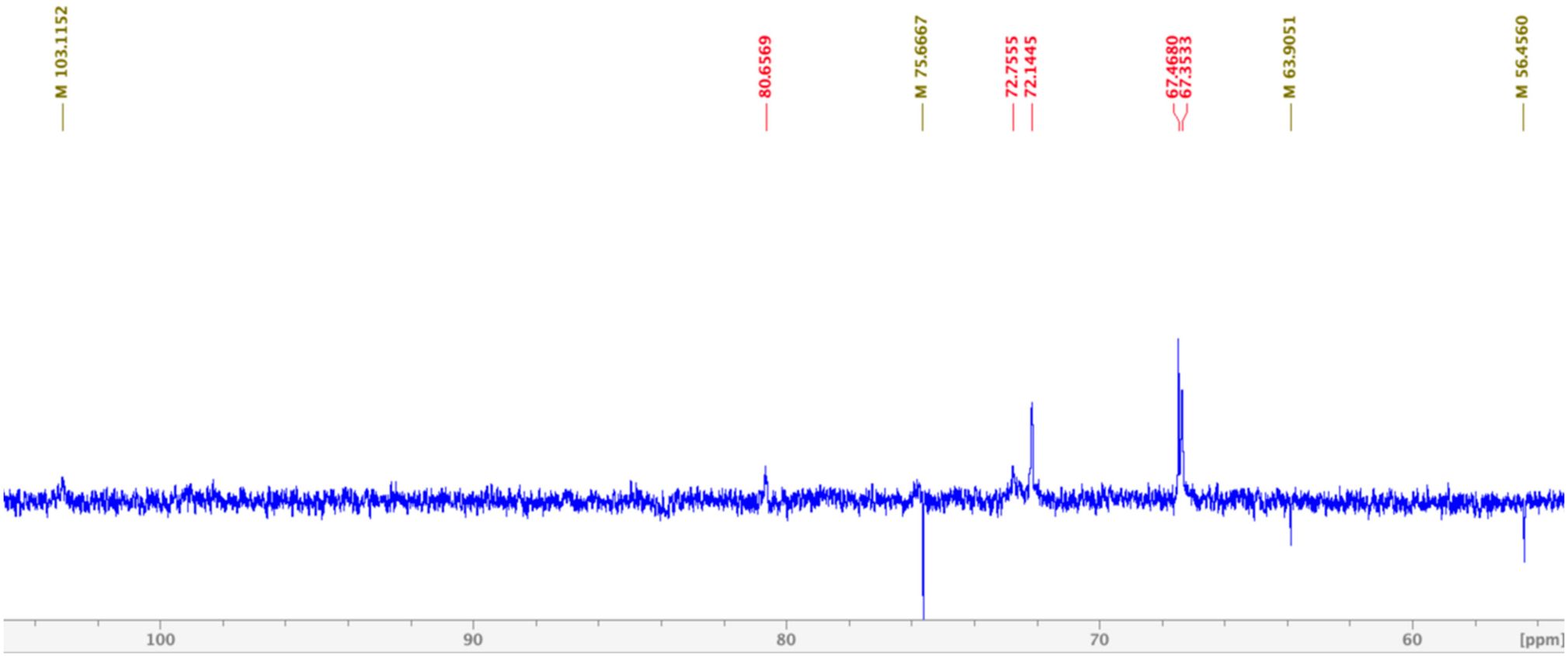

103.1365
99.0374
85.5698
80.6716
78.8965
100
95
90
85
80
[ppm]


75.9352
75.6226
72.7632
72.1603
67.4784
67.3626
63.8819
61.0675
60.4103
59.1896
75
70
65
60
[ppm]

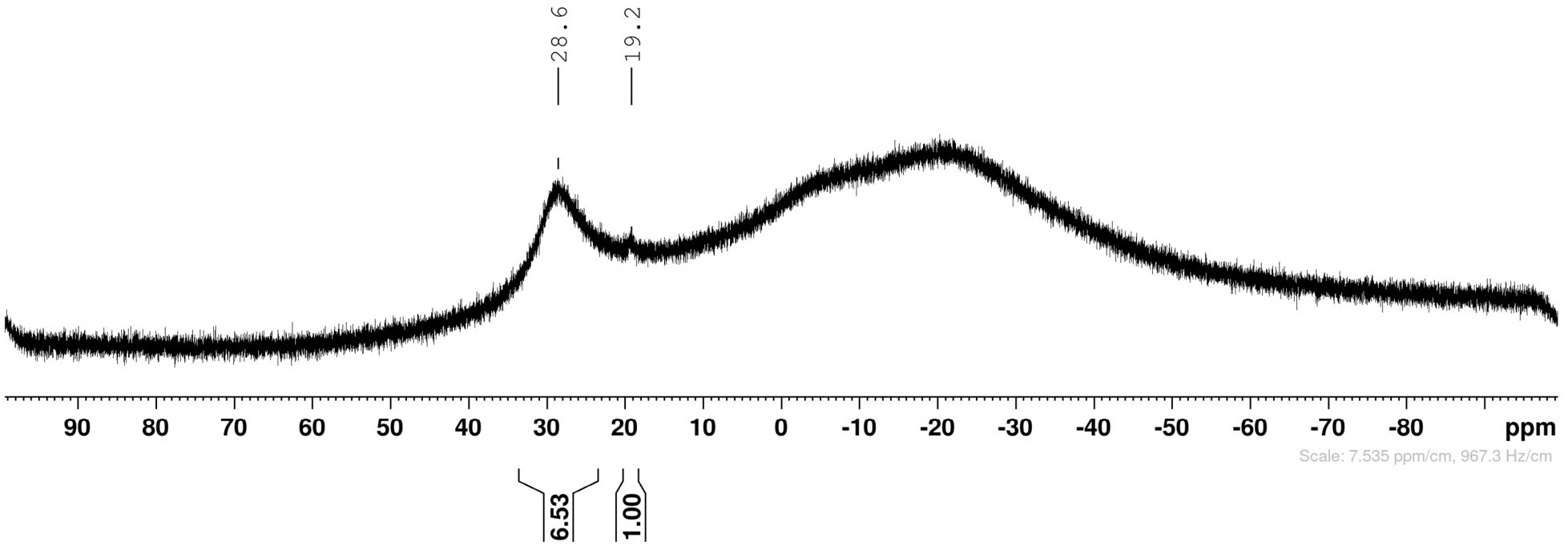
DEPT


136.2264
135.9070
135.4948
135.3777
133.4639
133.1429
132.9648
132.5299
130.0785
129.3700
129.0040
128.8444
128.2888
125.2632
80.6569
M 75.6667
72.7555
72.1445
67.4680
67.3533
M 63.9051
140
120
100
80
[ppm]

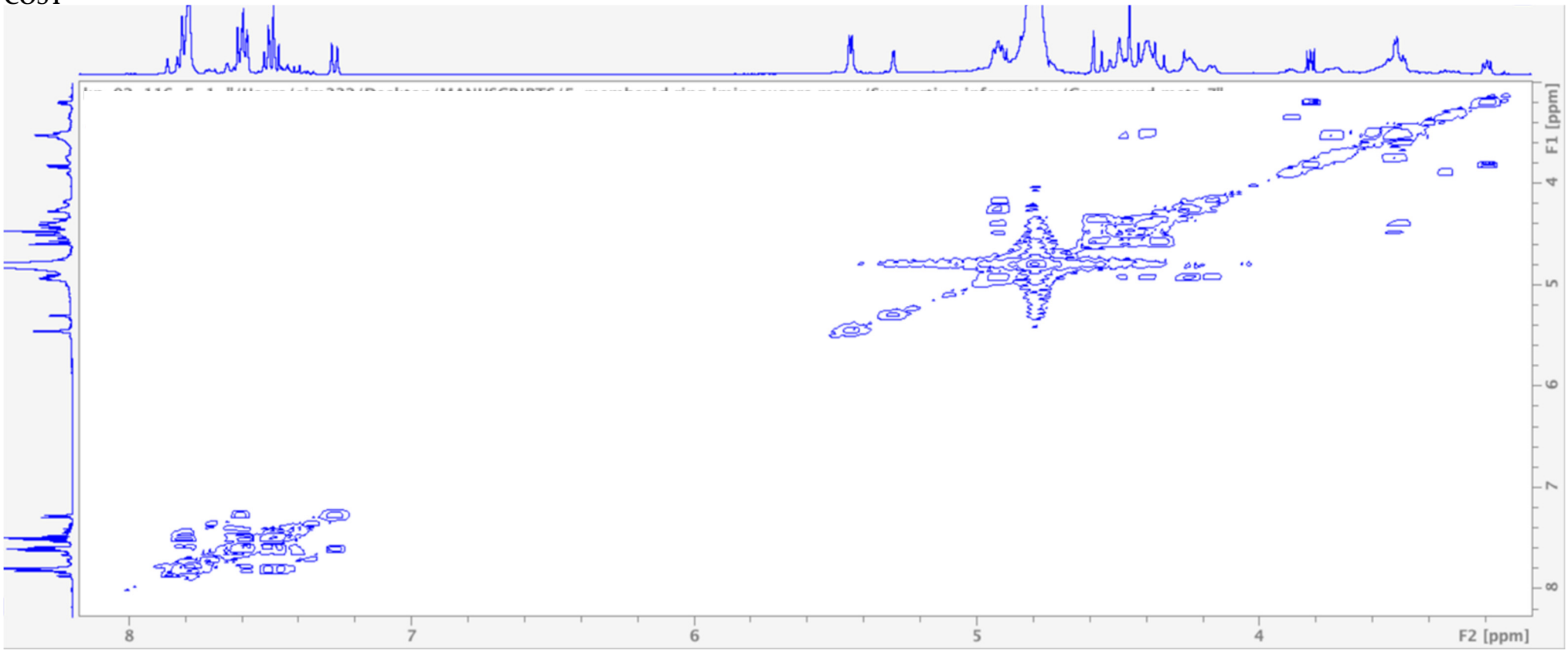

136.2264
135.9070
135.4948
135.3777
133.4639
133.1429
132.9648
132.5299
130.0785
129.3700
129.0040
128.8444
128.2888
125.2632
136
134
132
130
128
126
[ppm]

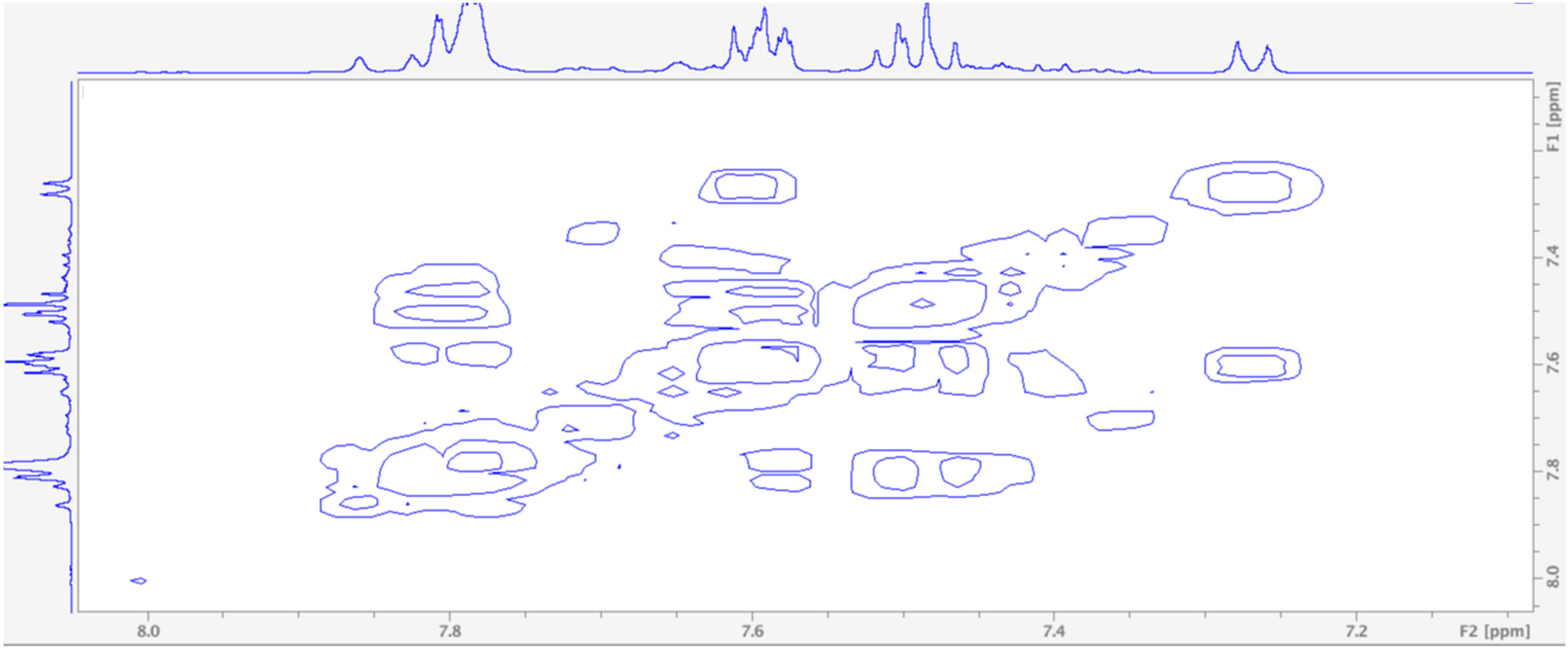

M 103.1152
80.6569
M 75.6667
72.7555
72.1445
67.4680
67.3533
M 63.9051
M 56.4560
100
90
80
70
60
[ppm]

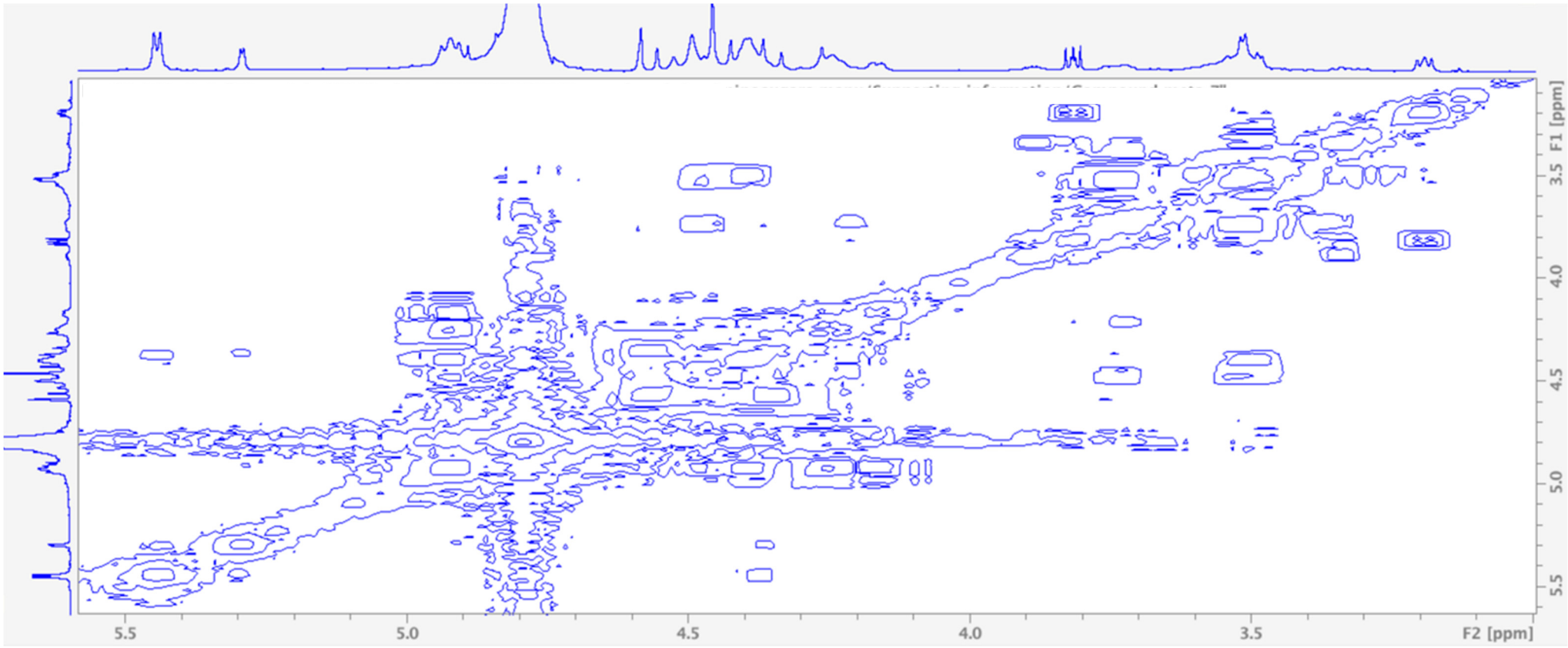
^{11}B-NMR

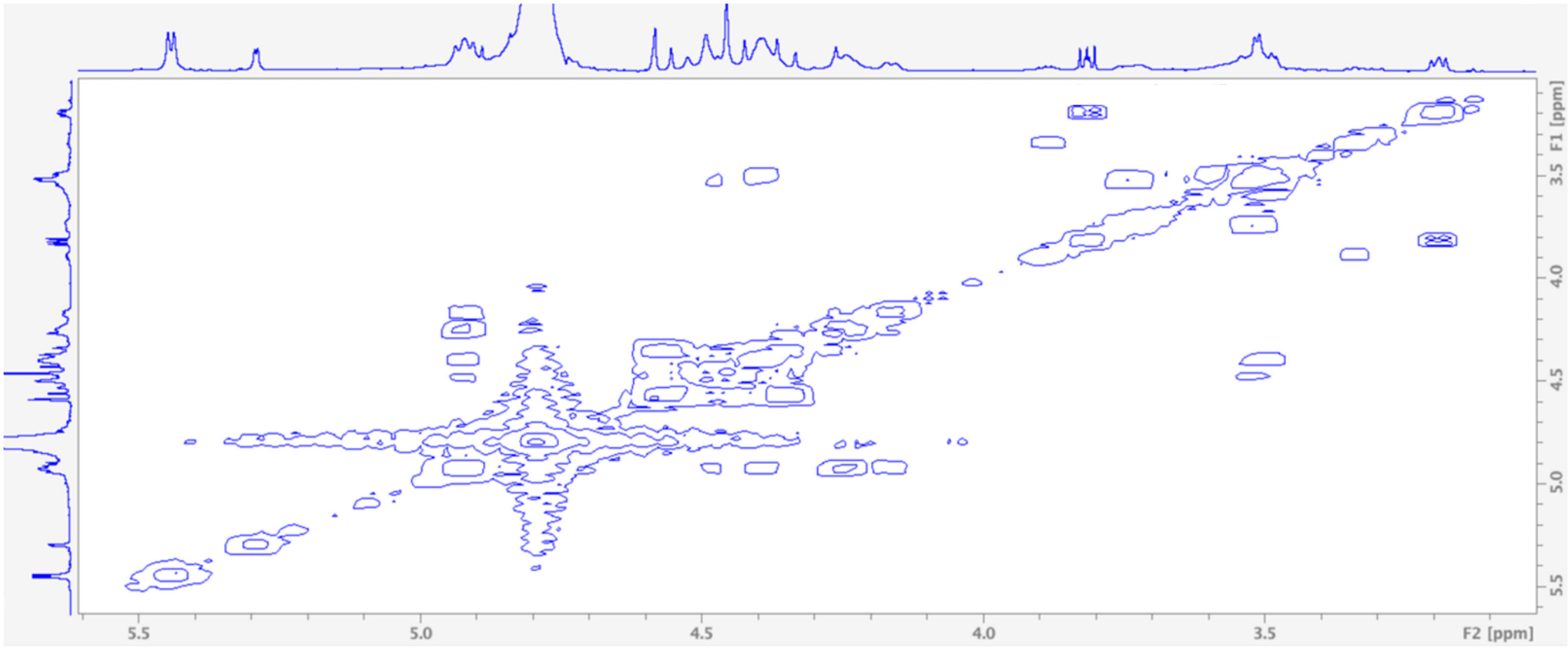

28.6
19.2
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
ppm
Scale: 7.535 ppm/cm, 967.3 Hz/cm
6.53
1.00

COSY

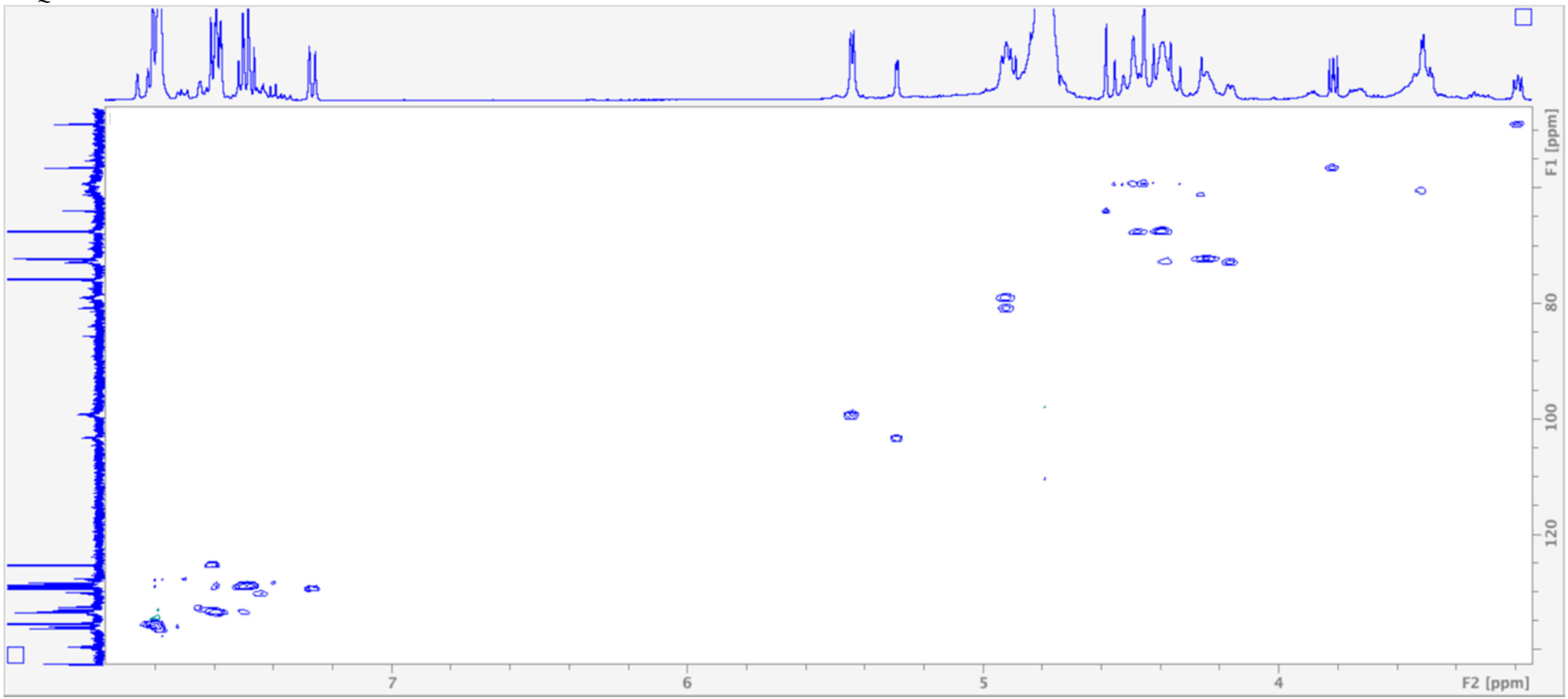


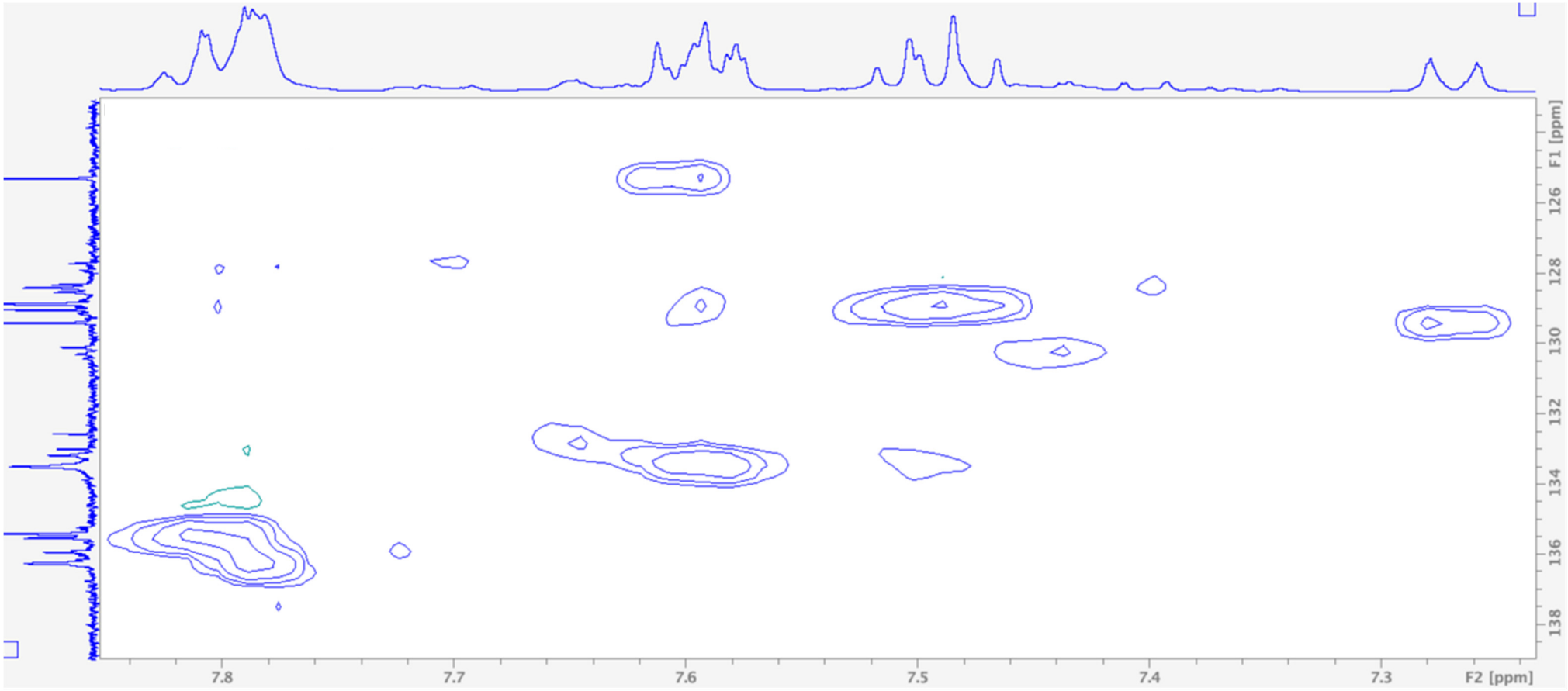

F1 [ppm]
7.4
7.6
7.8
8.0
8.0
7.8
7.6
7.4
7.2
F2 [ppm]

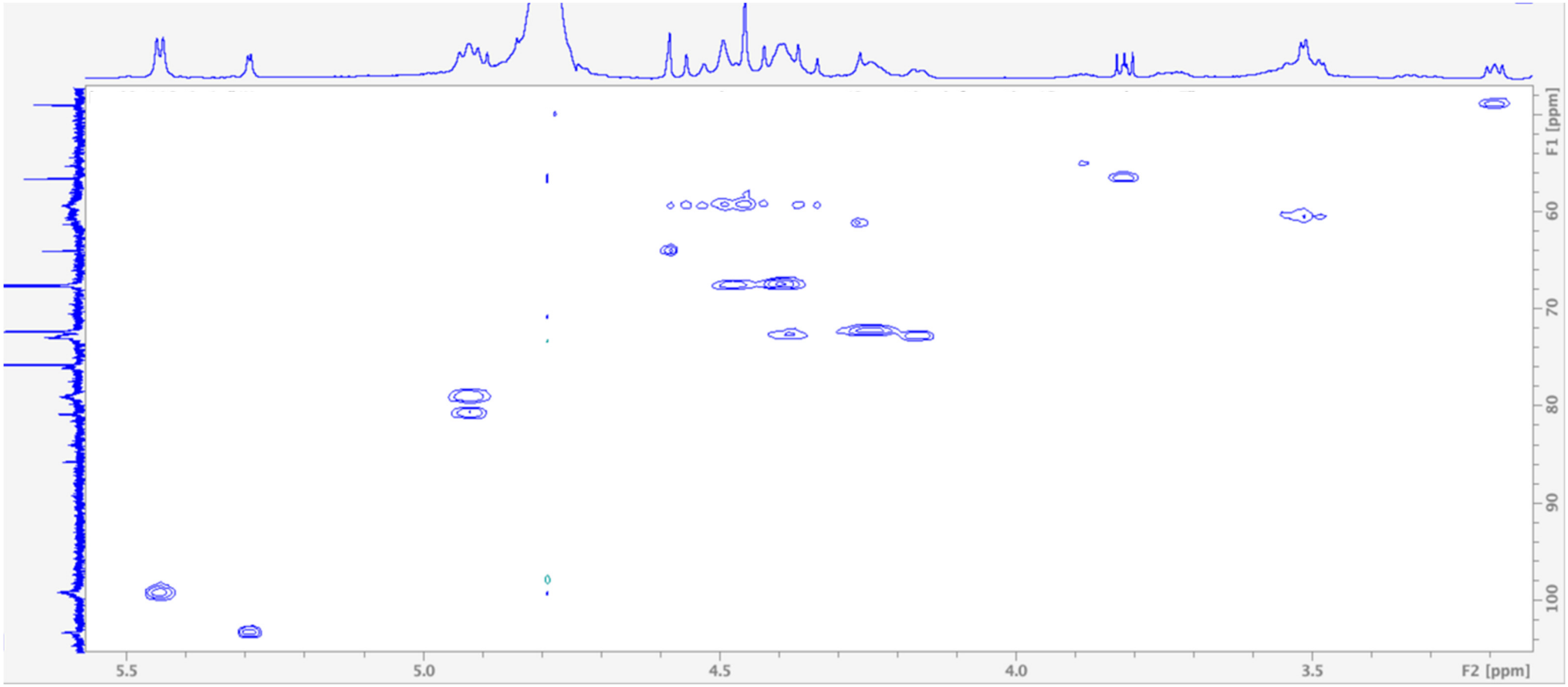

5.5
5.0
4.5
4.0
3.5
F2 [ppm]
F1 [ppm]
3.5
4.0
4.5
5.0
5.5

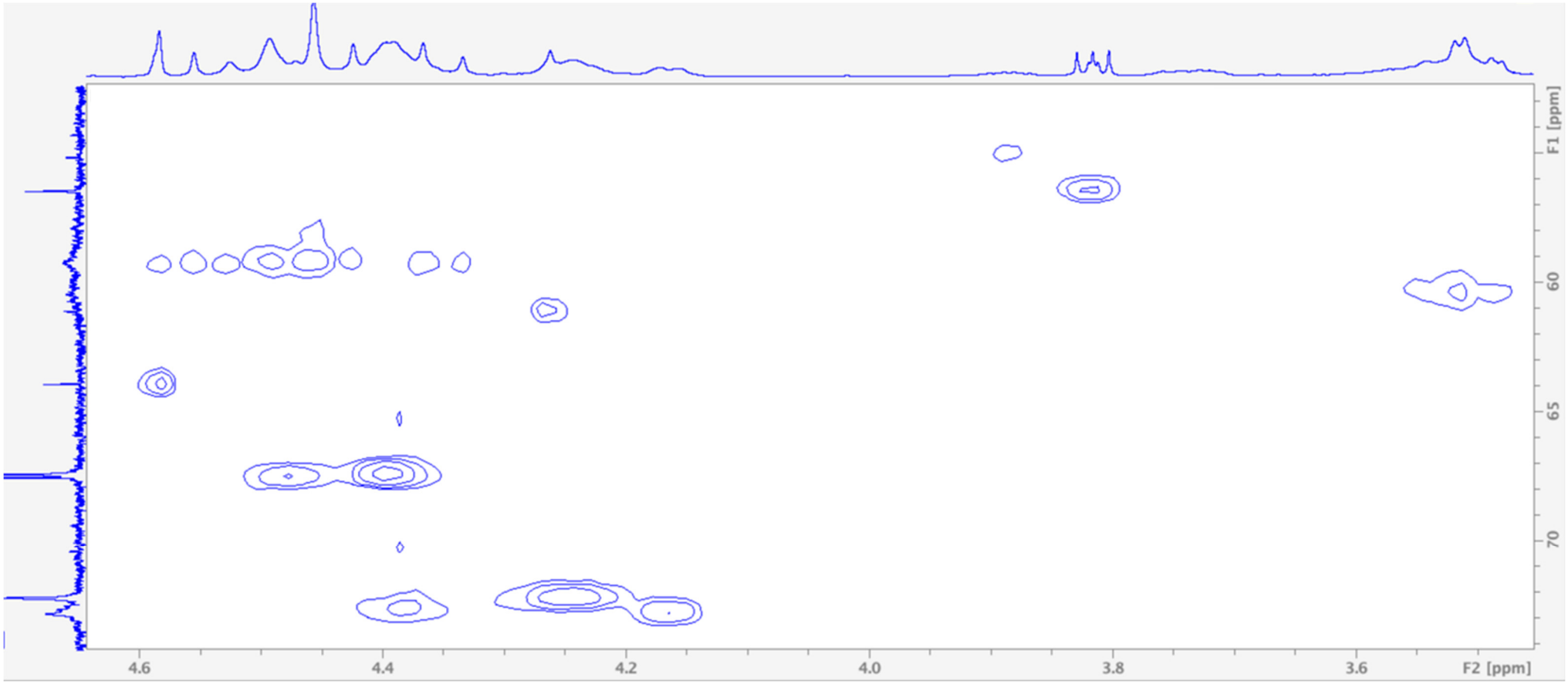

5.5
5.0
4.5
4.0
3.5
F2 [ppm]
3.5
4.0
4.5
5.0
5.5
F1 [ppm]

HSQC

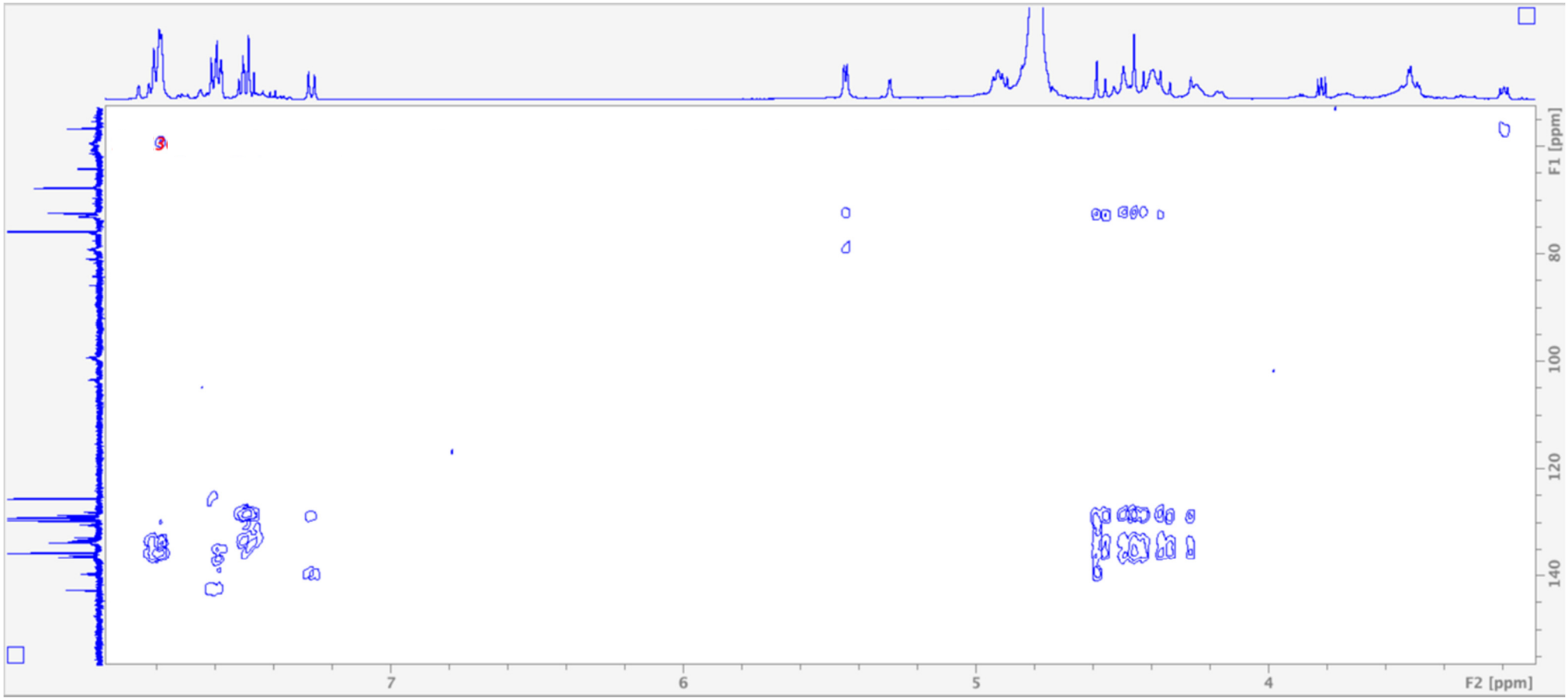


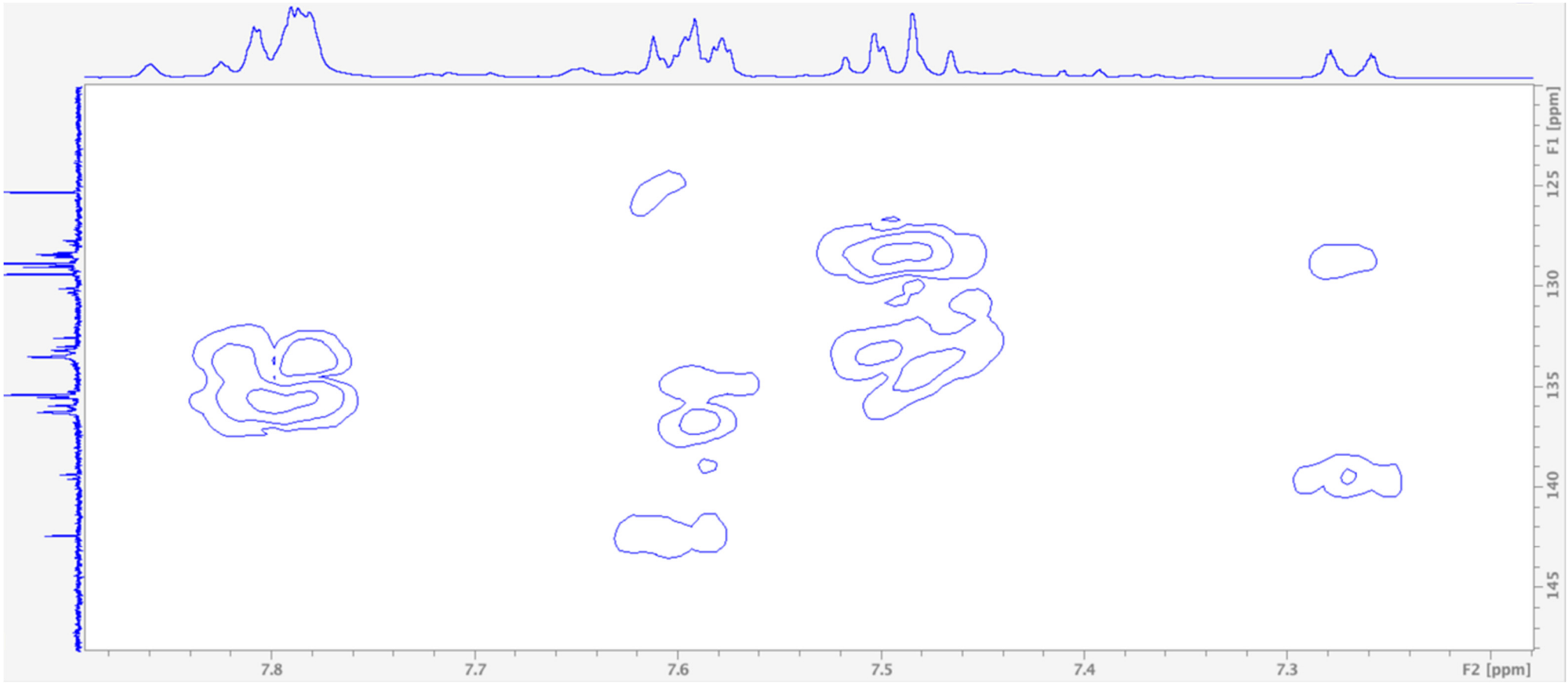

F1 [ppm]
126
128
130
132
134
136
138
7.8
7.7
7.6
7.5
7.4
7.3
F2 [ppm]

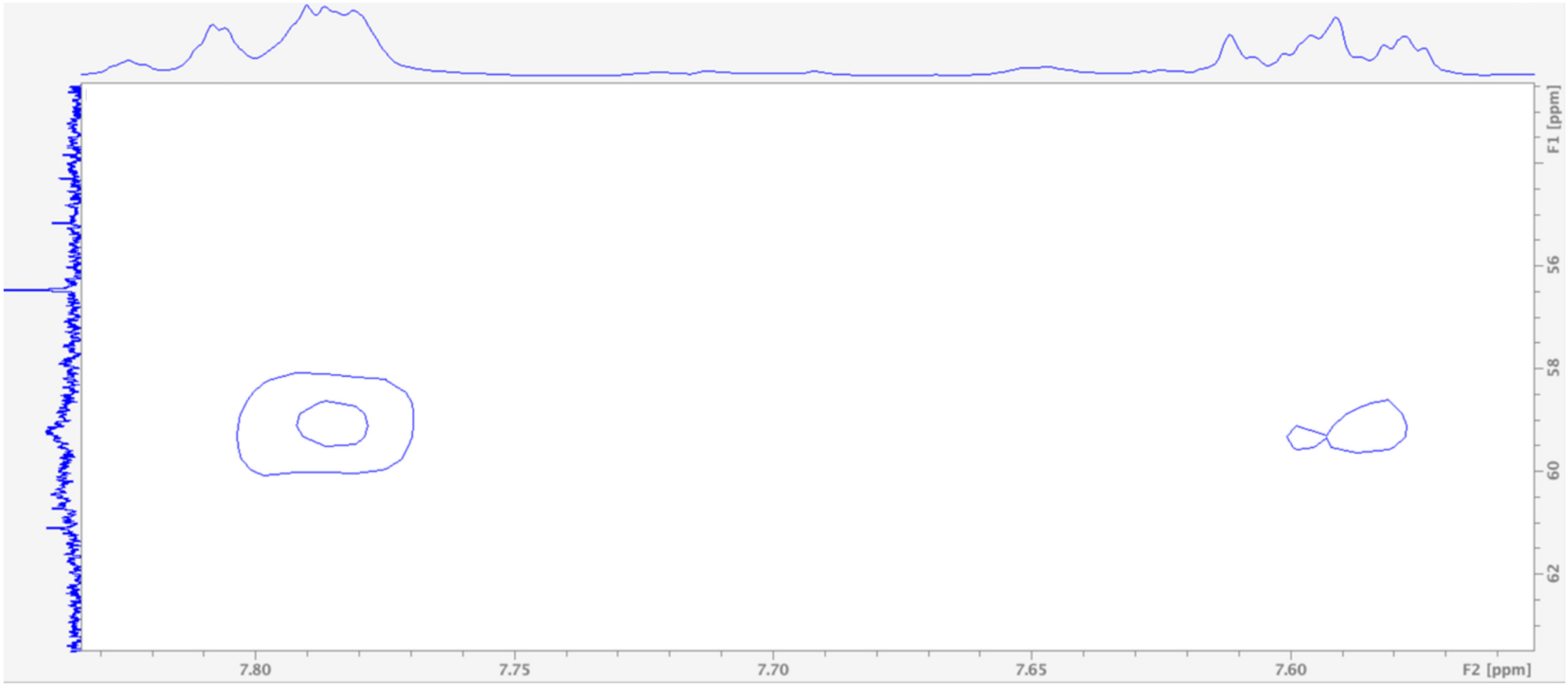

F1 [ppm]
60
70
80
90
100
5.5
5.0
4.5
4.0
3.5
F2 [ppm]

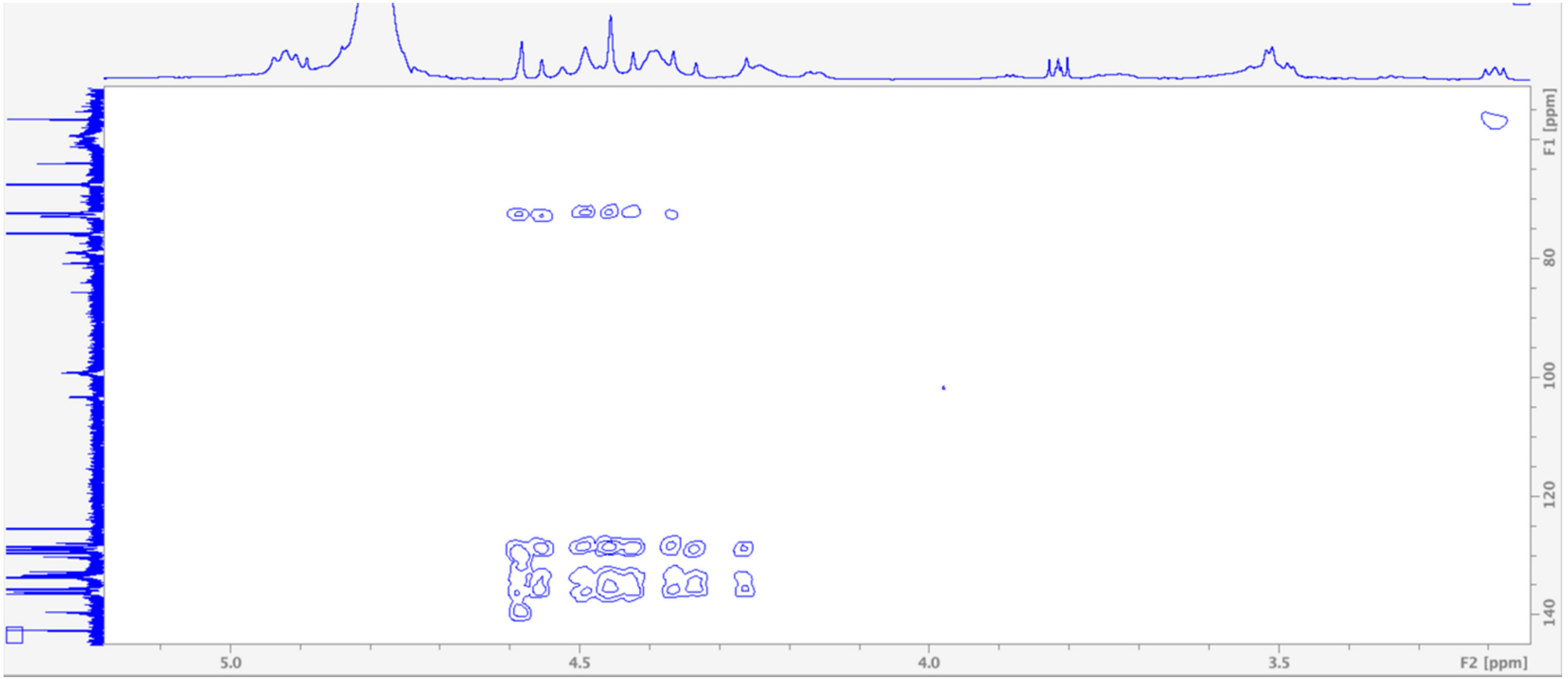

F1 [ppm]
60
65
70
4.6
4.4
4.2
4.0
3.8
3.6
F2 [ppm]

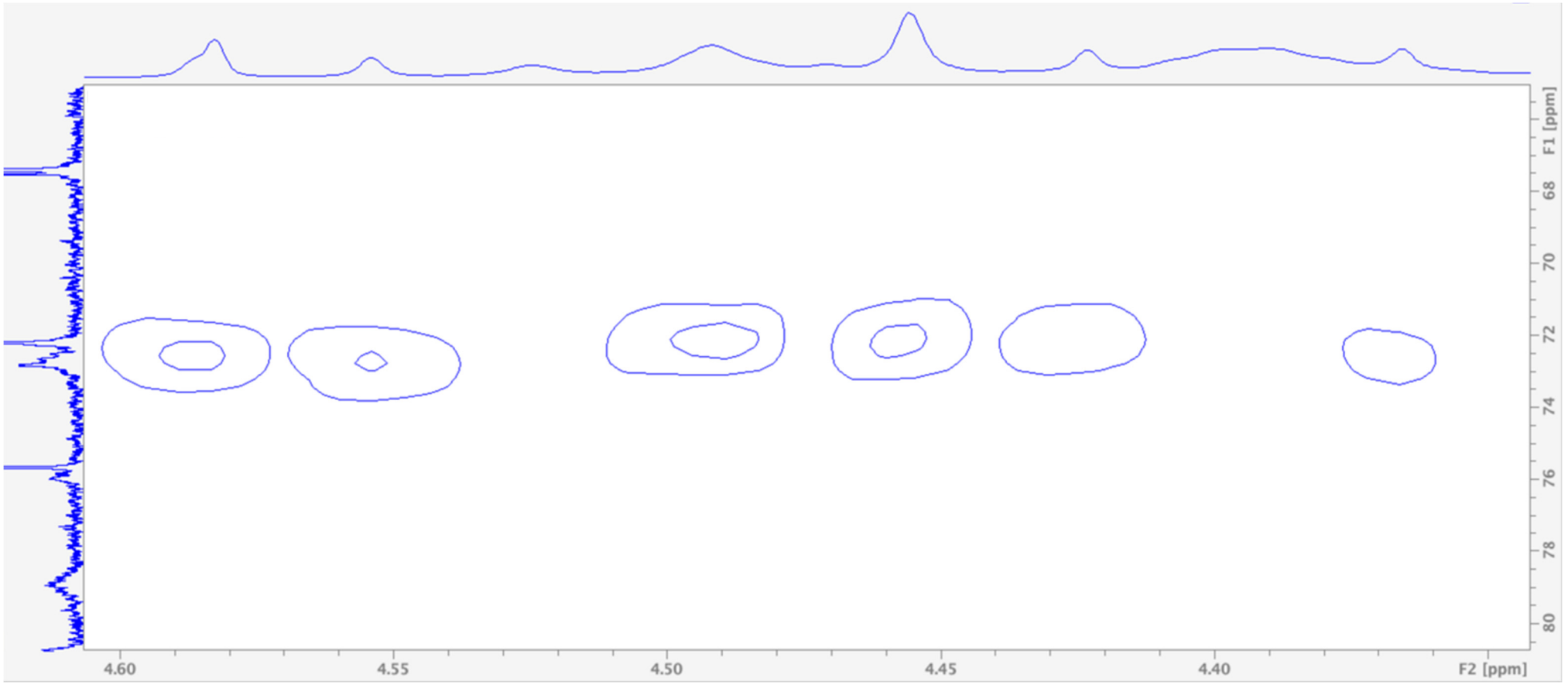
HMBC




F1 [ppm]
125
130
135
140
145
7.8
7.7
7.6
7.5
7.4
7.3
F2 [ppm]


F1 [ppm]
56
58
60
62
7.80
7.75
7.70
7.65
7.60
F2 [ppm]


F1 [ppm]
80
100
120
140
5.0
4.5
4.0
3.5
F2 [ppm]


F1 [ppm]
68
70
72
74
76
78
80
4.60
4.55
4.50
4.45
4.40
F2 [ppm]

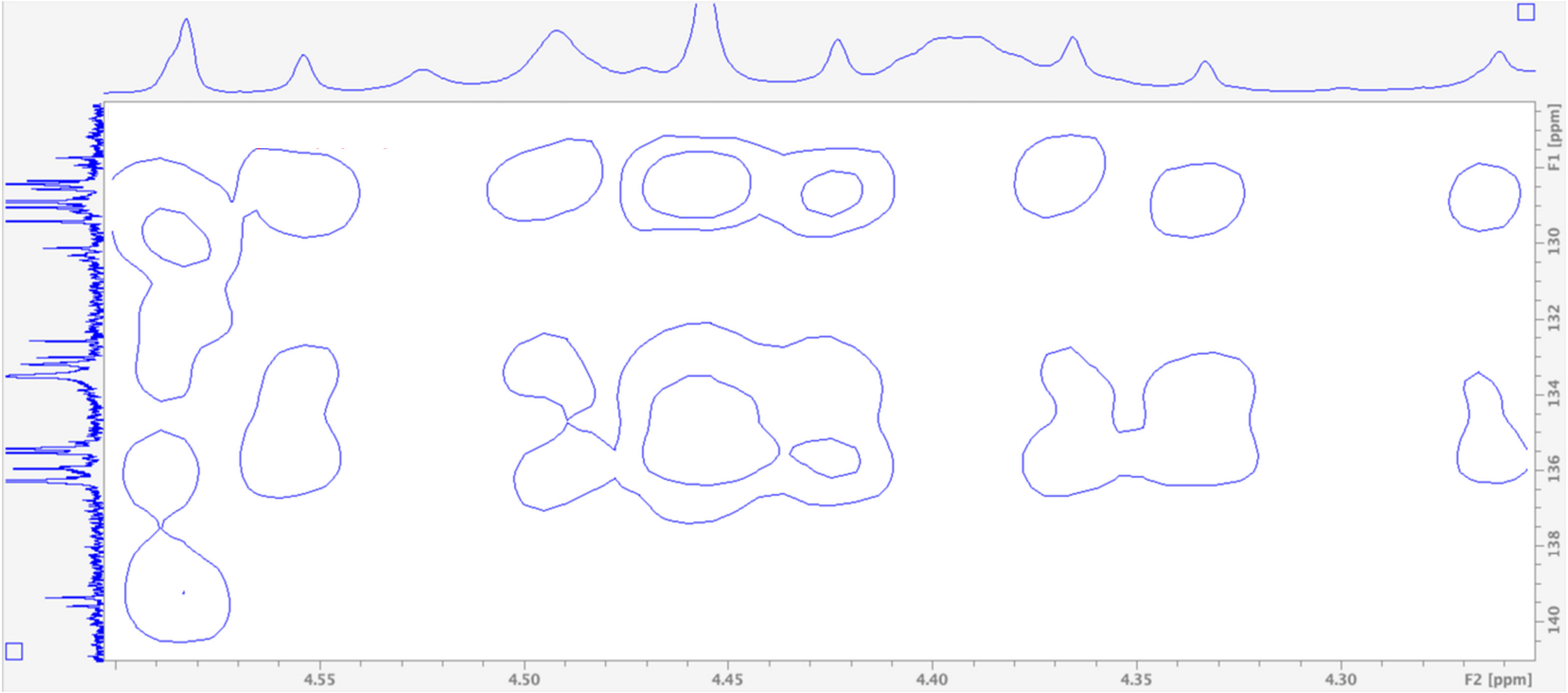
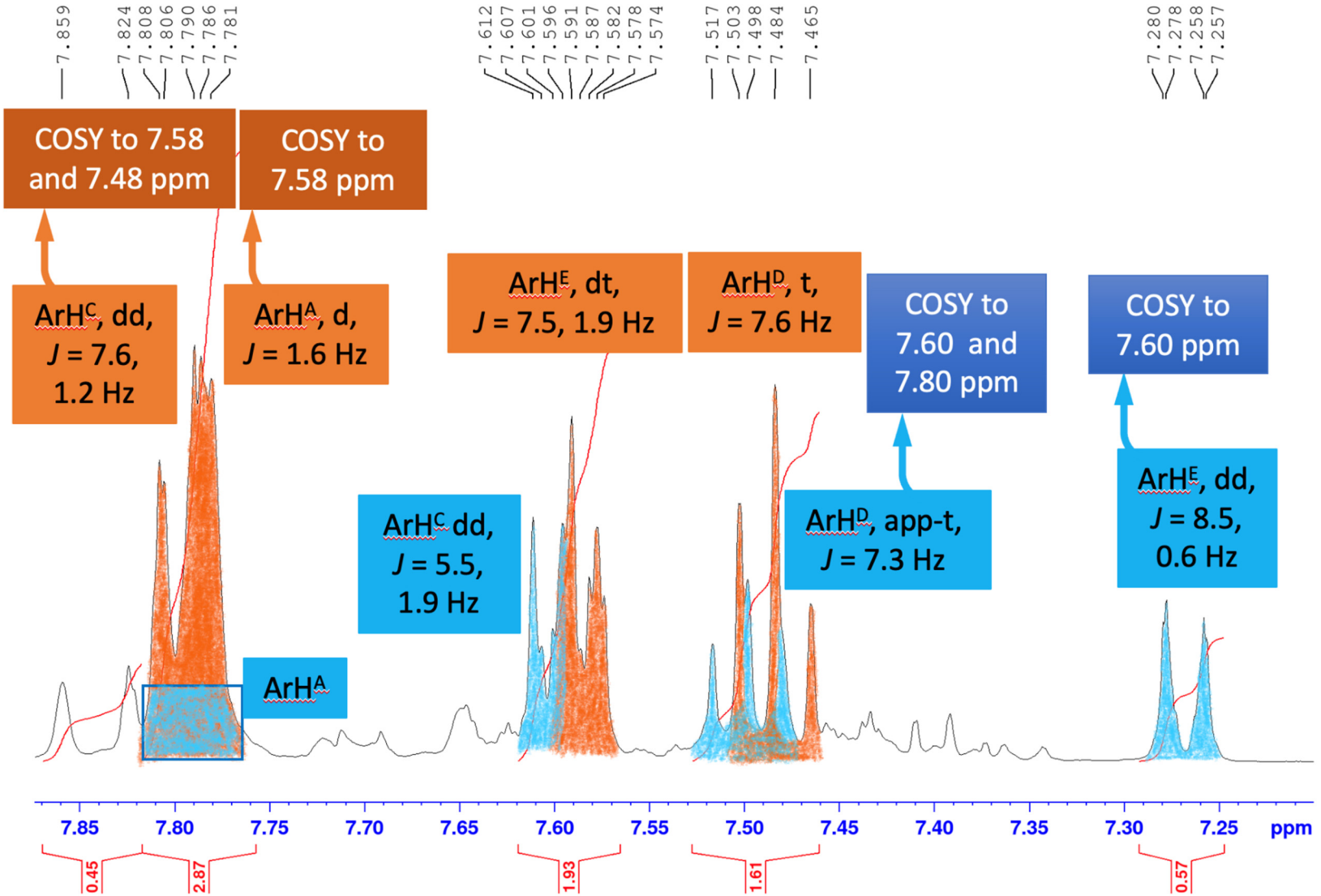
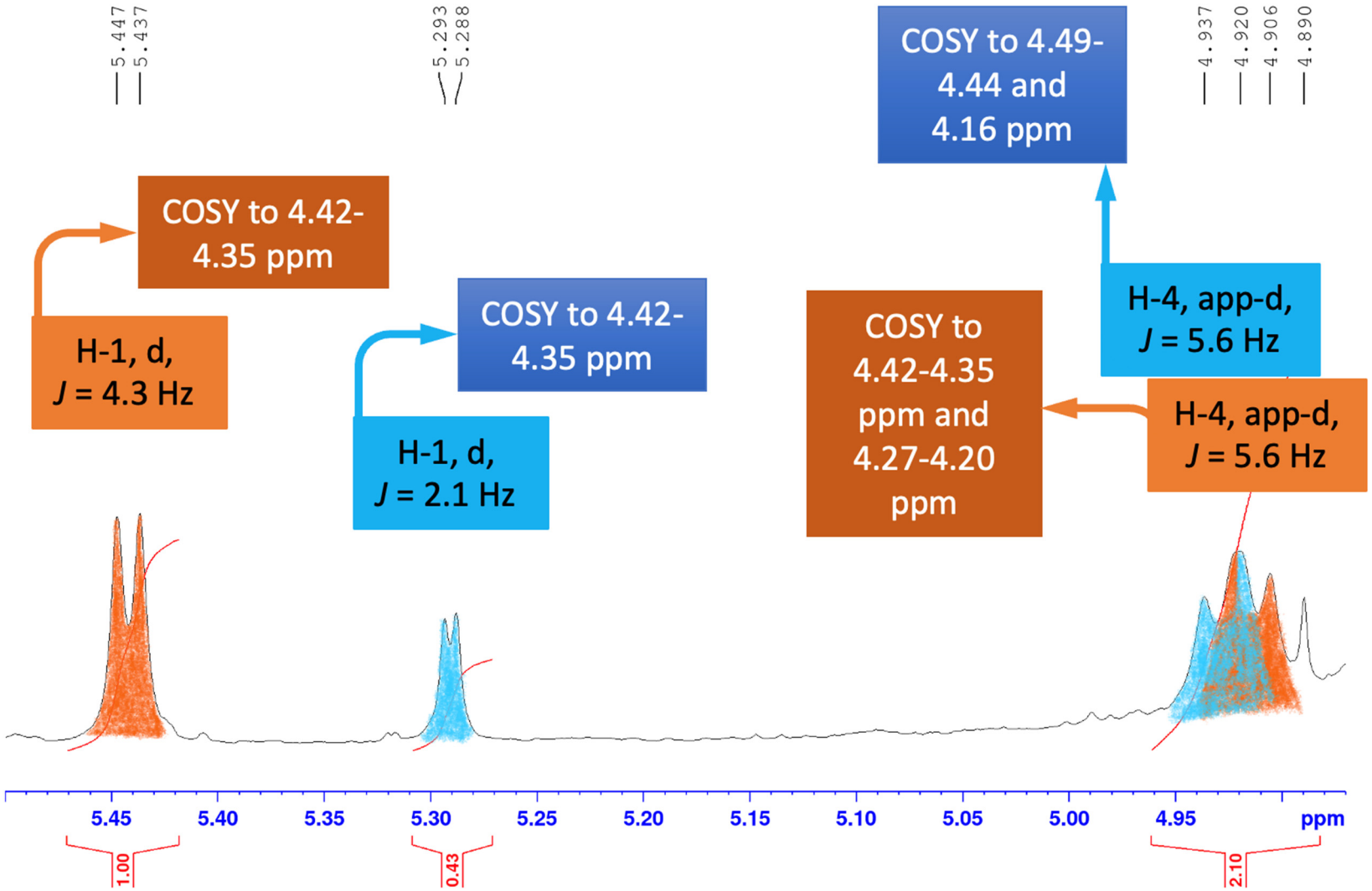

F1 [ppm]
130
132
134
136
138
140
4.55
4.50
4.45
4.40
4.35
4.30
F2 [ppm]

Figure S3. ^{1}H-NMR spectrum (400 MHz, D_2O) of intermediate **meta 3** with colour-coded signals, highlighting the furanose anomeric forms they belong to, with interpretation of the isolated signals and tentative interpretation of the overlapping ones. Namely, the orange designates the *α-fur* form and indigo designates the *β-fur* form. A) section 7.87 ppm to 7.20 ppm; B) section 5.50 ppm to 4.87 ppm; C) section 4.61 ppm to 4.12 ppm; D) section 4.00 ppm to 3.15 ppm. Highlighted are also the principal COSY correlations to hydrogen atoms within the same spin systems.

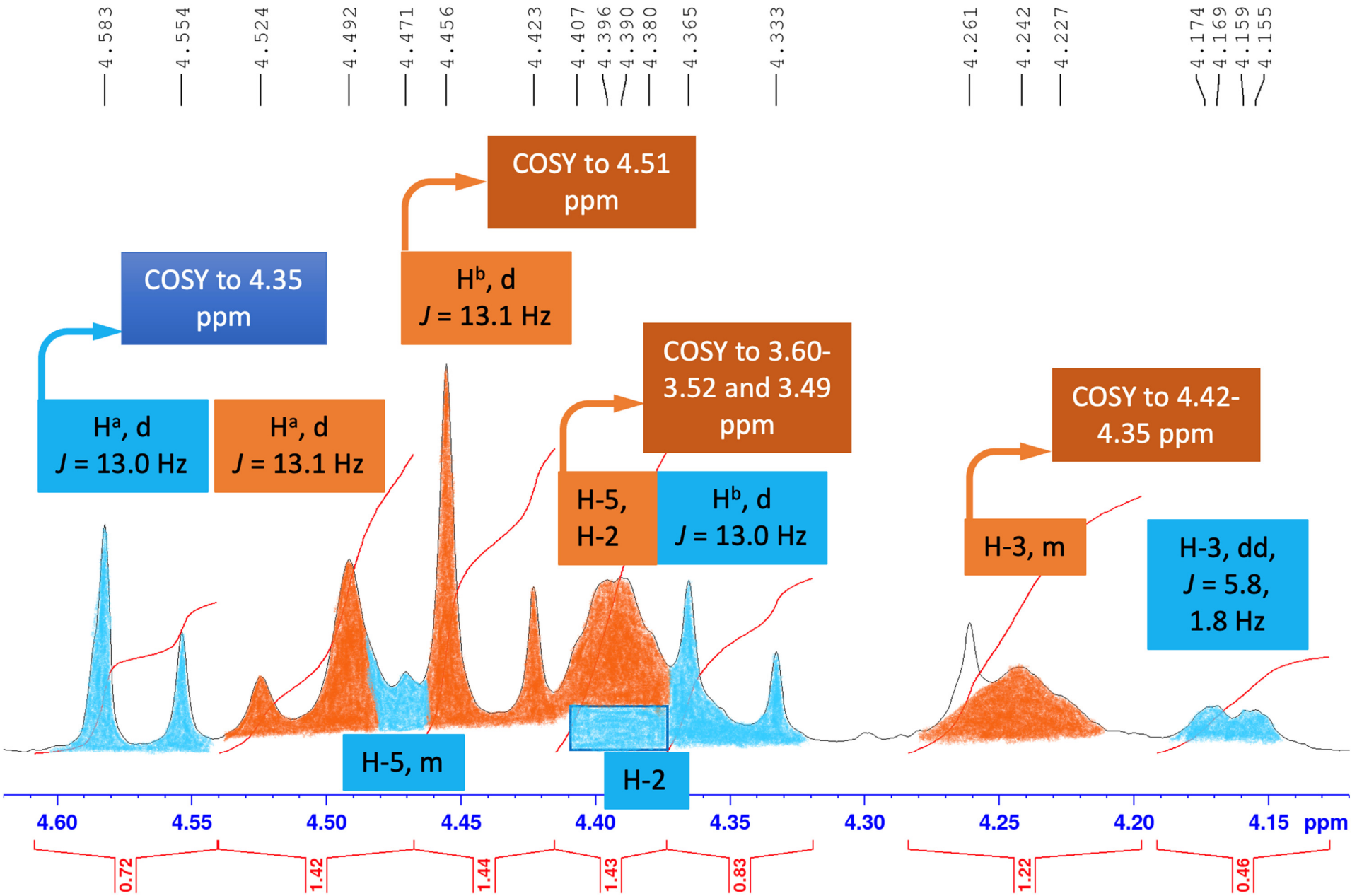
A



B


5.447
5.437
5.293
5.288
4.937
4.920
4.906
4.890
COSY to 4.42-4.35 ppm
H-1, d, J = 4.3 Hz
COSY to 4.42-4.35 ppm
H-1, d, J = 2.1 Hz
COSY to 4.49-4.44 and 4.16 ppm
COSY to 4.42-4.35 ppm and 4.27-4.20 ppm
H-4, app-d, J = 5.6 Hz
H-4, app-d, J = 5.6 Hz
5.45
5.40
5.35
5.30
5.25
5.20
5.15
5.10
5.05
5.00
4.95
ppm
1.00
0.43
2.10

C


4.583
4.554
4.524
4.492
4.471
4.456
4.423
4.407
4.396
4.390
4.380
4.365
4.333
4.261
4.242
4.227
4.174
4.169
4.159
4.155
COSY to 4.51 ppm
COSY to 4.35 ppm
Hb, d J = 13.1 Hz
COSY to 3.60-3.52 and 3.49 ppm
COSY to 4.42-4.35 ppm
Ha, d J = 13.0 Hz
Ha, d J = 13.1 Hz
H-5, H-2
Hb, d J = 13.0 Hz
H-3, m
H-3, dd, J = 5.8, 1.8 Hz
H-5, m
H-2
4.60
4.55
4.50
4.45
4.40
4.35
4.30
4.25
4.20
4.15 ppm
0.72
1.42
1.44
1.43
0.83
1.22
0.46

D

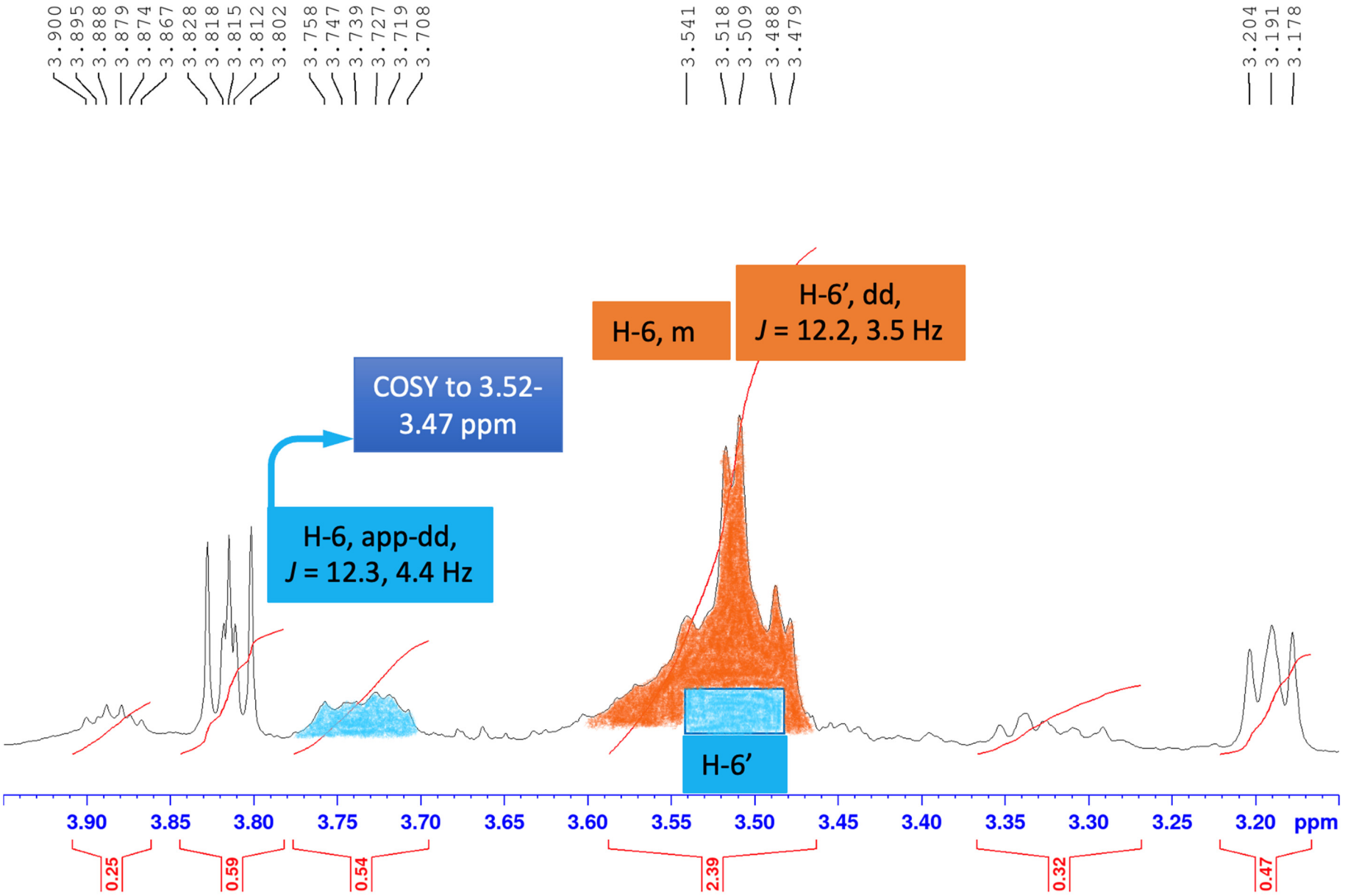
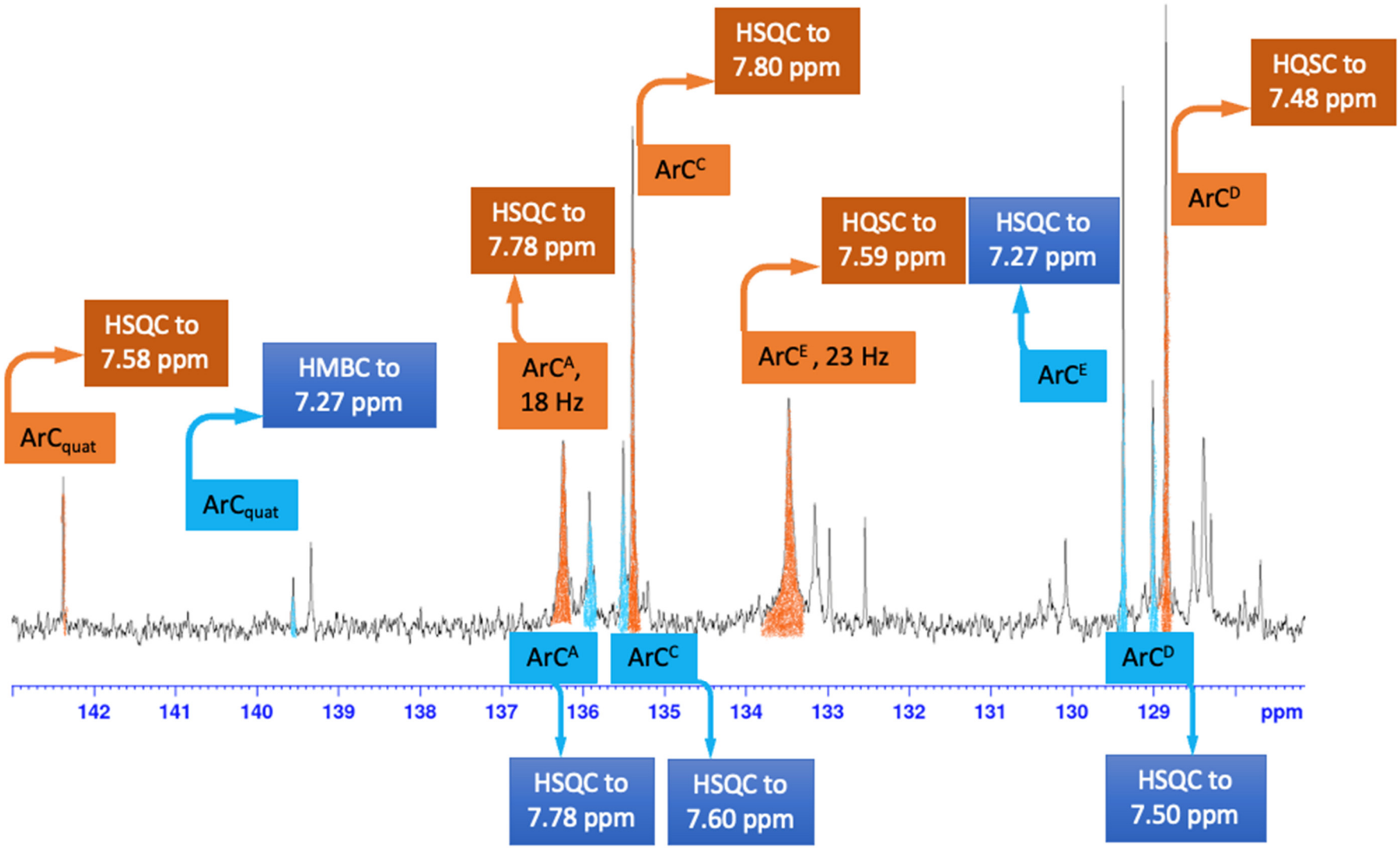
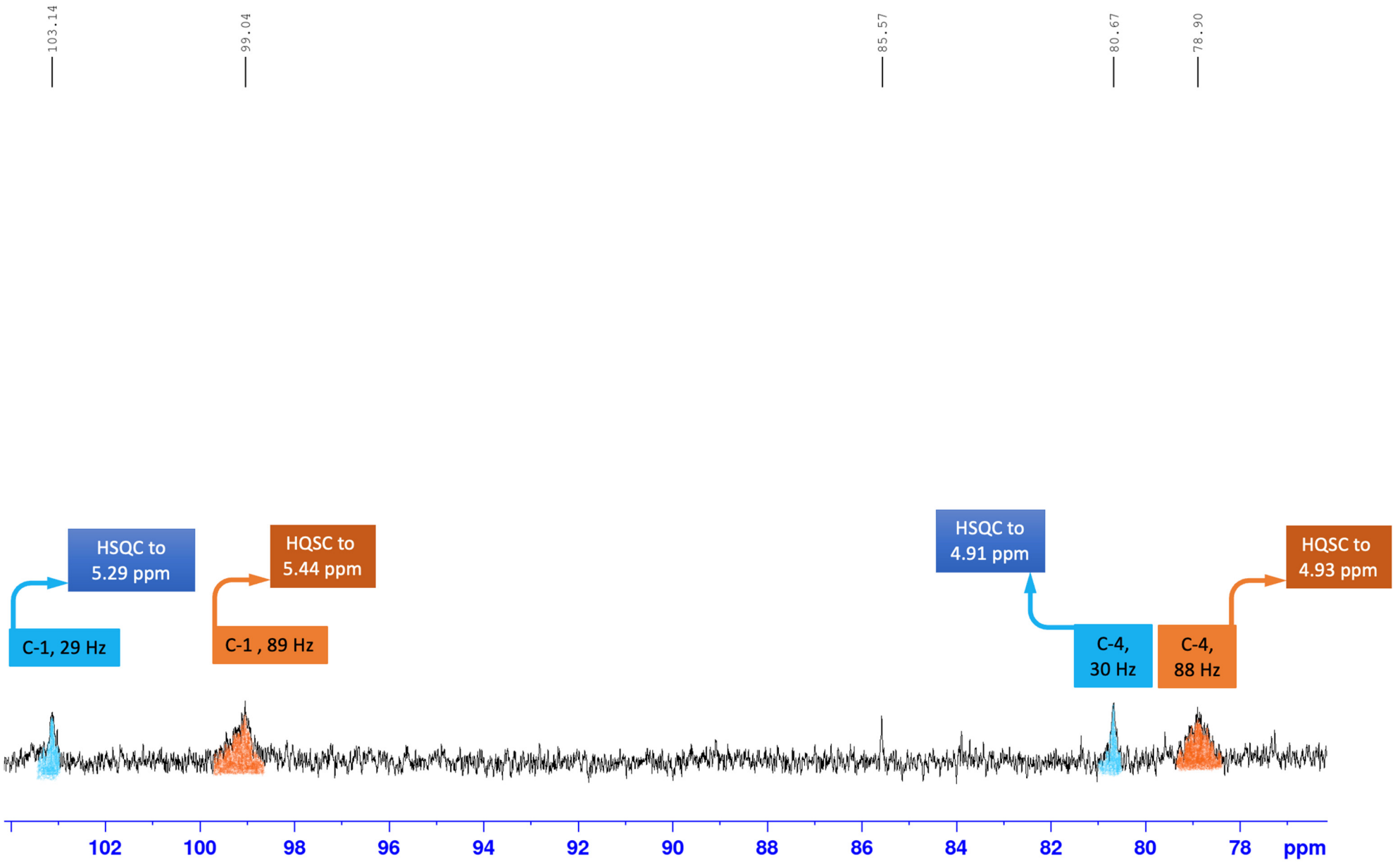

3.900
3.895
3.888
3.879
3.874
3.867
3.828
3.818
3.815
3.812
3.802
3.758
3.747
3.739
3.727
3.719
3.708
3.541
3.518
3.509
3.488
3.479
3.204
3.191
3.178
COSY to 3.52-3.47 ppm
H-6, app-dd, J = 12.3, 4.4 Hz
H-6, m
H-6', dd, J = 12.2, 3.5 Hz
H-6'
3.90
3.85
3.80
3.75
3.70
3.65
3.60
3.55
3.50
3.45
3.40
3.35
3.30
3.25
3.20
ppm
0.25
0.59
0.54
2.39
0.32
0.47

Figure S4. ^{13}C-NMR spectrum (100 MHz, D_2O) sections of intermediate **meta 3** with colour-coded signals, highlighting the furanose anomeric forms they belong to, with interpretation of the isolated signals and tentative interpretation of the overlapping ones. Namely, the orange designates the *α-fur* form and indigo designates the *β-fur* form. A) section 141.5 ppm to 127.2 ppm; B) section 104.0 ppm to 76.0 ppm; C) section 75.5 ppm to 56.7 ppm. Highlighted are also the principal HSQC and HMBC correlations to hydrogen atoms within the same spin systems.

A



B


103.14
99.04
85.57
80.67
78.90
HSQC to 5.29 ppm
HQSC to 5.44 ppm
HSQC to 4.91 ppm
HQSC to 4.93 ppm
C-1, 29 Hz
C-1 , 89 Hz
C-4, 30 Hz
C-4, 88 Hz
102
100
98
96
94
92
90
88
86
84
82
80
78
ppm

C

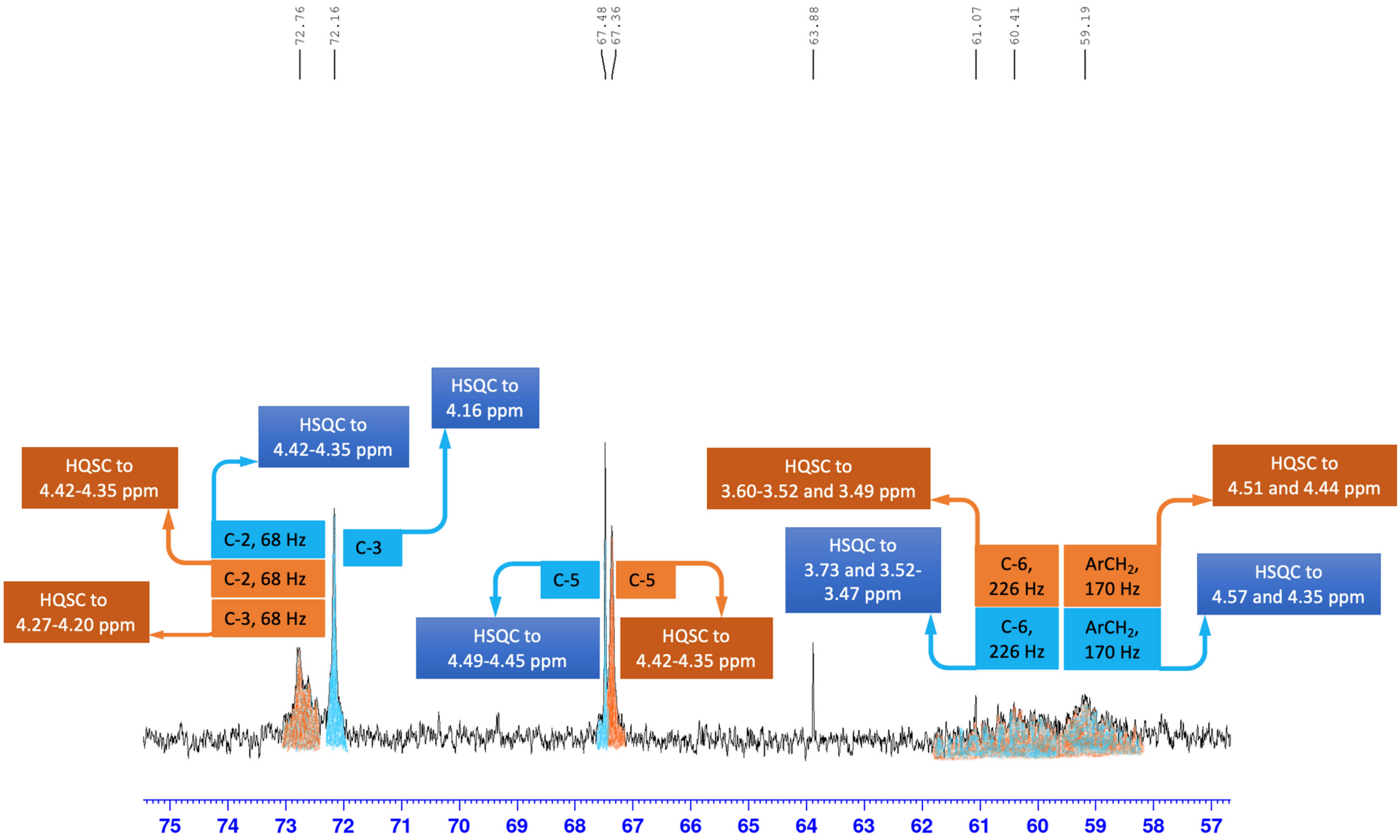
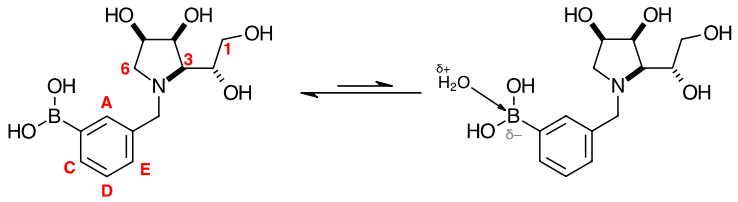

72.76
72.16
67.48
67.36
63.88
61.07
60.41
59.19
HSQC to 4.16 ppm
HSQC to 4.42-4.35 ppm
HQSC to 4.42-4.35 ppm
C-2, 68 Hz
C-3
C-2, 68 Hz
C-3, 68 Hz
HQSC to 4.27-4.20 ppm
C-5
C-5
HSQC to 4.49-4.45 ppm
HQSC to 4.42-4.35 ppm
HQSC to 3.60-3.52 and 3.49 ppm
HSQC to 3.73 and 3.52-3.47 ppm
C-6, 226 Hz
C-6, 226 Hz
ArCH2, 170 Hz
ArCH2, 170 Hz
HQSC to 4.51 and 4.44 ppm
HSQC to 4.57 and 4.35 ppm
75 74 73 72 71 70 69 68 67 66 65 64 63 62 61 60 59 58 57

Figure S5. ^{1}H- (400 MHz), ^{13}C-NMR (100 MHz), DEPT, ^{11}B-NMR (128 MHz), COSY and HSQC spectra of *N*-(3-methylphenyl boronic acid)-1,4-dideoxy-1,4-imino-L-gulitol **meta 4** in D_2O.

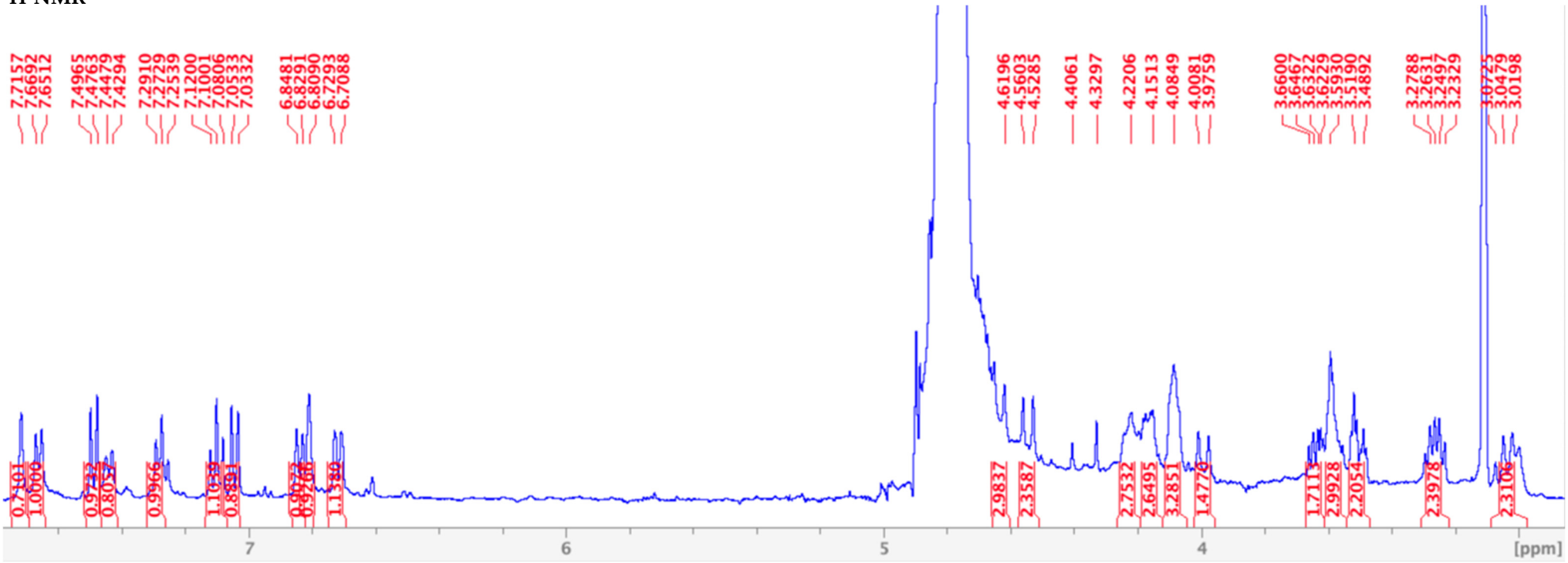


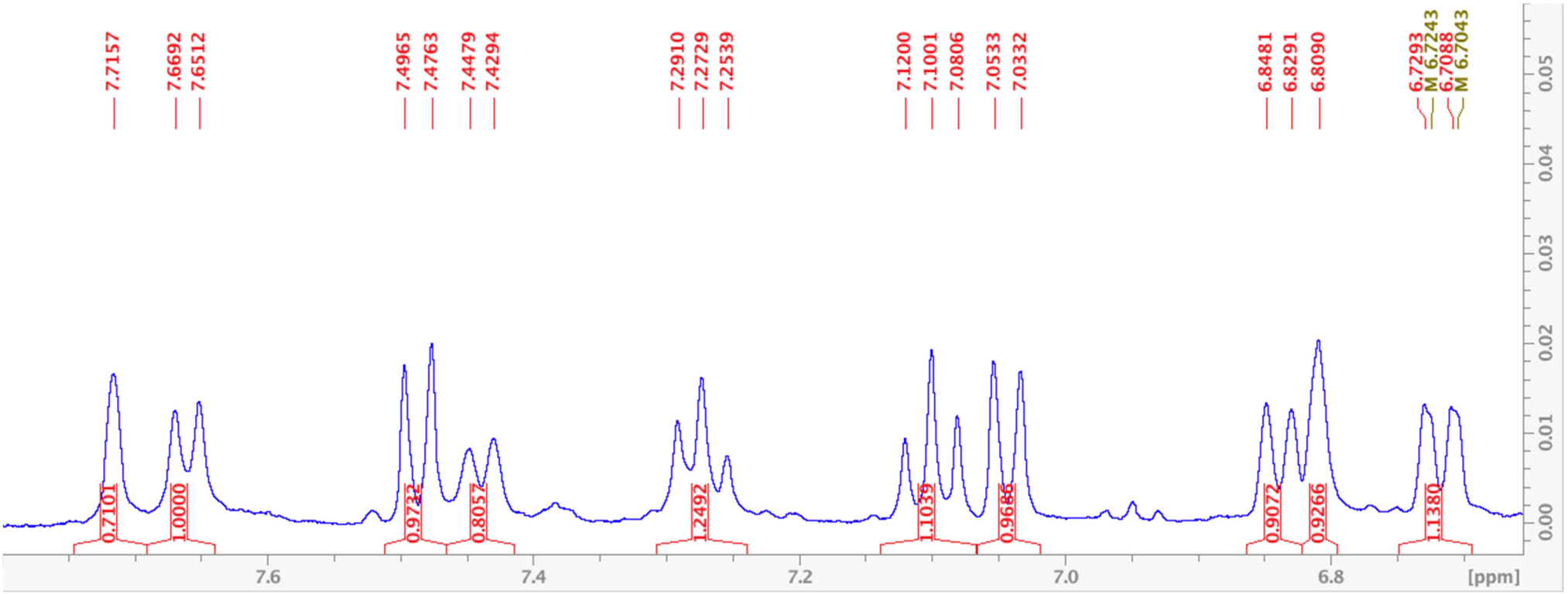
Boronic acid species. **δH**: 7.72 (1H, s, ArH^{A}), 7.66 (1H, d, $J_{HC,HD}$ 7.4 Hz, ArH^{C}), 7.43 (1H, d, $J_{HE,HD}$ 7.5 Hz, ArH^{E}), 7.27 (1H, app-t, $J_{HD,HC/HE}$ 7.3 Hz, ArH^{D}), 4.55 (1H, d, $J_{Ha,Hb}$ 12.9 Hz, ArC*H*^{a}H^{b}), 4.26-4.05 (m, H-2, H-3, H-5), 3.99 (1H, d, $J_{Hb,Ha}$ 12.9 Hz, ArCH^{a}*H*^{b}), 3.63-3.53 (m, H-6, H-4), 3.52 (1H, app-t, *J* 3.5 Hz, H-6'), 3.31-3.20 (m, H-1), 3.09-2.97 (m, H-1'); **δC**: 137.6 (ArC^{A}), 136.5 (ArC^{C}), 134.2 (ArC^{E}), 129.6 (ArC^{D}), 72.18 (C-3), 71.63 (C-2), 70.5 (C-4), 70.12 (C-5), 64.7 (C-6), 62.5 (ArCH$_2$,width 21 Hz), 54.3 (C-1, width 27 Hz). The two C$_{quat}$ are not discernible.

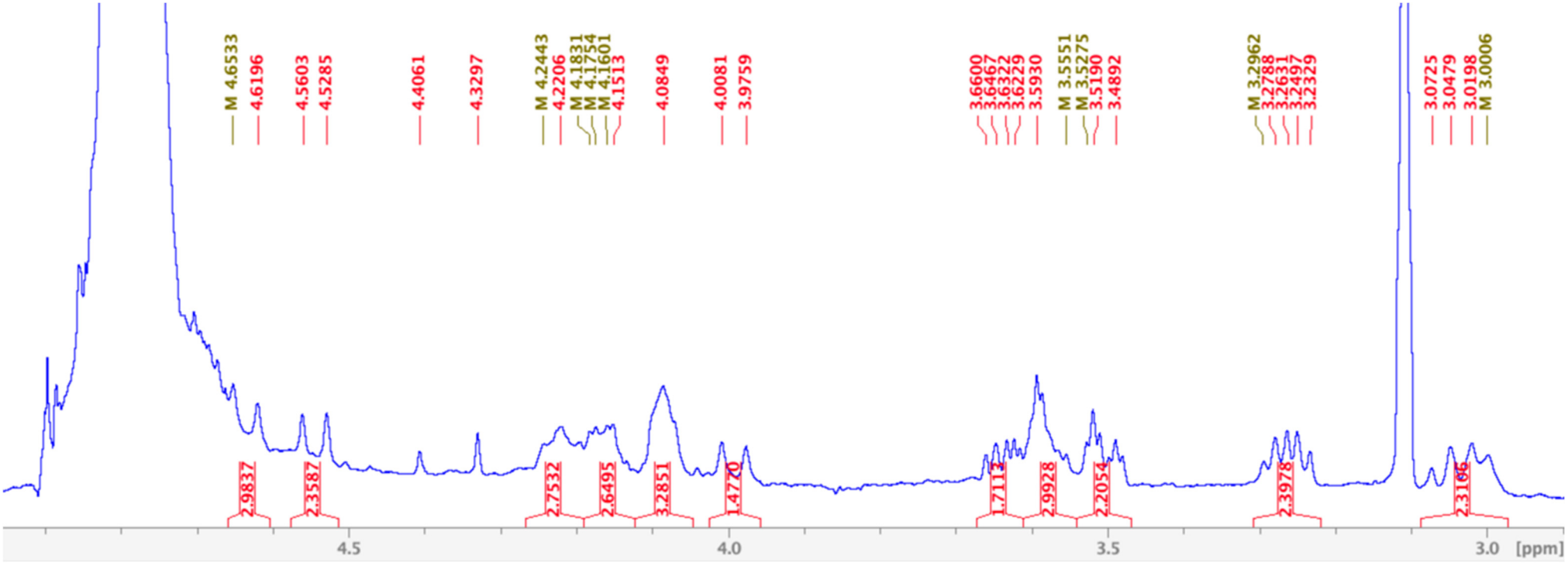
Boronate species. **δH**: 7.10 (1H, app-t, $J_{HD,HC/HE}$ 8.0 Hz, ArH^{D}), 6.83 (1H, d, $J_{HC,HD}$ 8.0 Hz, ArH^{C}), 6.81 (1H, s, H^{A}), 6.71 (1H, d, $J_{HE,HD}$ 8.1 Hz, ArH^{E}), 4.64 (1H, d, $J_{Ha,Hb}$ 12.9 Hz, ArC*H*^{a}H^{b}), 4.08 (1H, obscured, ArCH^{a}*H*^{b}), 3.67-3.62 (m, H-1), 3.63-3.53 (m, H-6, H-4), 3.49 (1H, app-t, *J* 3.5 Hz, H-6'), 3.09-2.97 (m, H-1'). **δC**: 131.5 (ArC^{D}), 123.3 (ArC^{E}), 118.8 (ArC^{A}), 118.1 (ArC^{C}), 72.17 (C-3), 71.62 (C-2), 70.5 (C-4), 70.10 (C-5), 64.6 (C-6), 62.7 (ArCH$_2$), 57.8 (C-1, width 17 Hz). The two C$_{quat}$ are not discernible.

δB: 27.8 (broad, integration: 1.7), 19.3 (sharp, integration: 1.0).

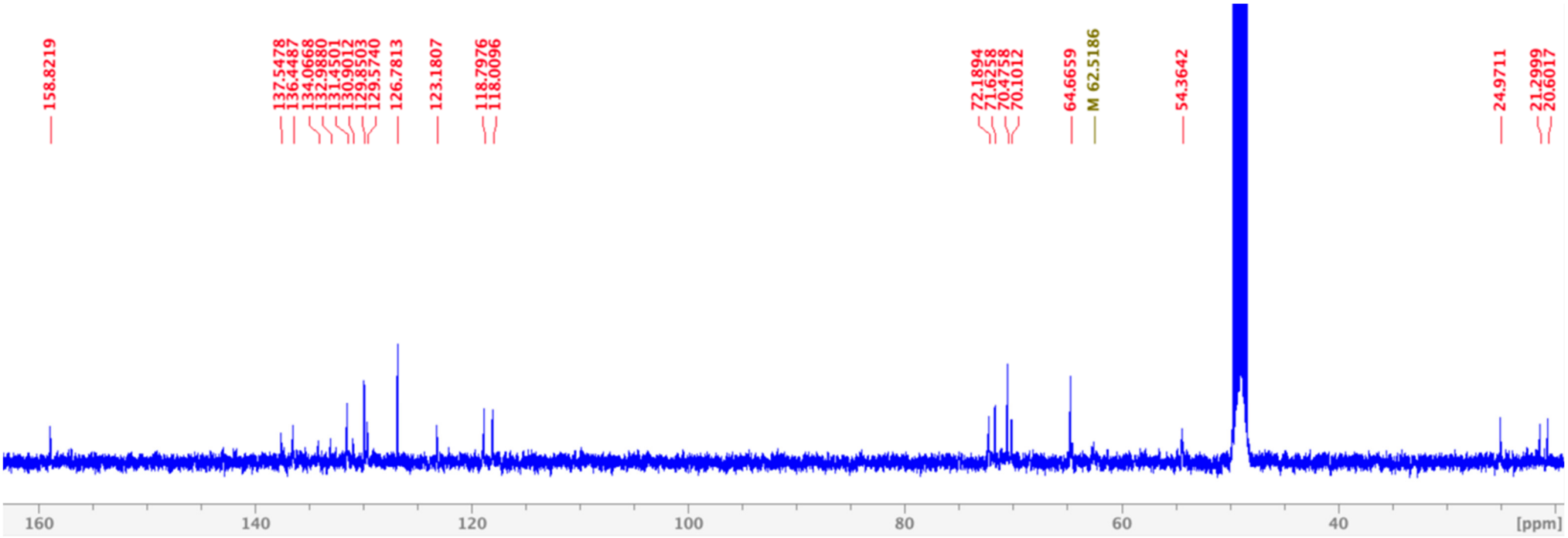
^{1}H-NMR

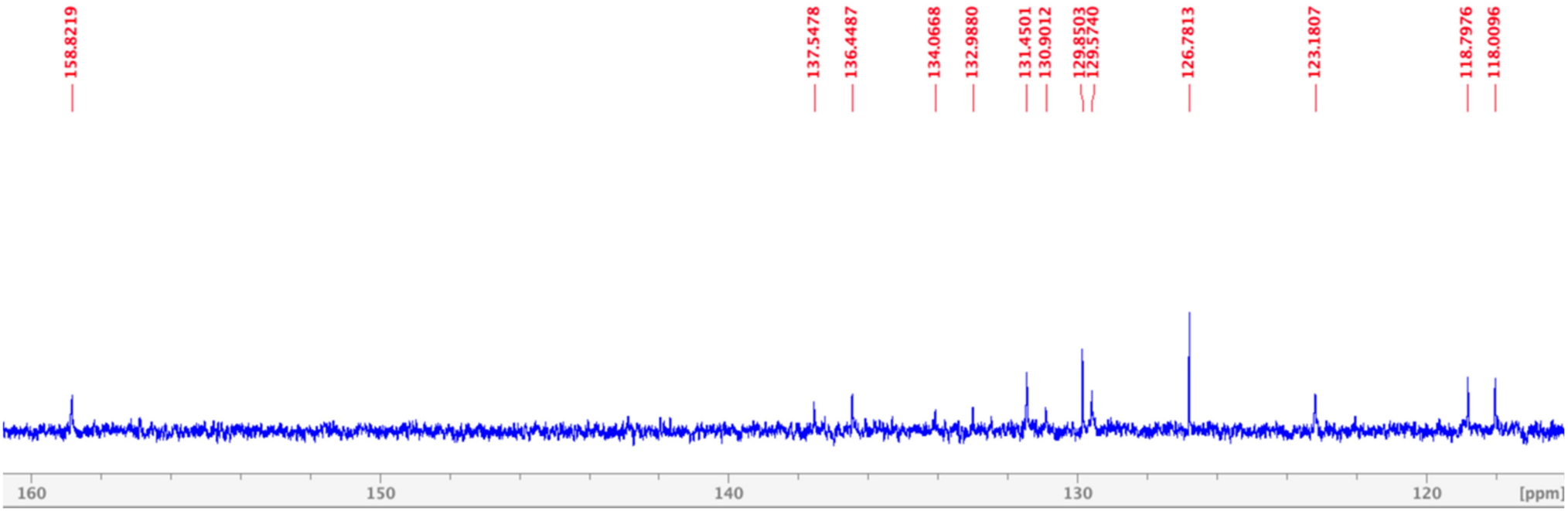

7.7157
7.6692
7.6512
7.4965
7.4763
7.4479
7.4294
7.2910
7.2729
7.2539
7.1200
7.1001
7.0806
7.0533
7.0332
6.8481
6.8291
6.8090
6.7293
6.7088
4.6196
4.5603
4.5285
4.4061
4.3297
4.2206
4.1513
4.0849
4.0081
3.9759
3.6600
3.6467
3.6322
3.6229
3.5930
3.5190
3.4892
3.2788
3.2631
3.2497
3.2329
3.0725
3.0479
3.0198
0.7101
1.0000
0.9966
1.1580
2.9837
2.3587
2.7532
2.6495
3.2851
1.4776
1.7113
2.9928
2.2054
2.3978
2.3106
7
6
5
4
[ppm]

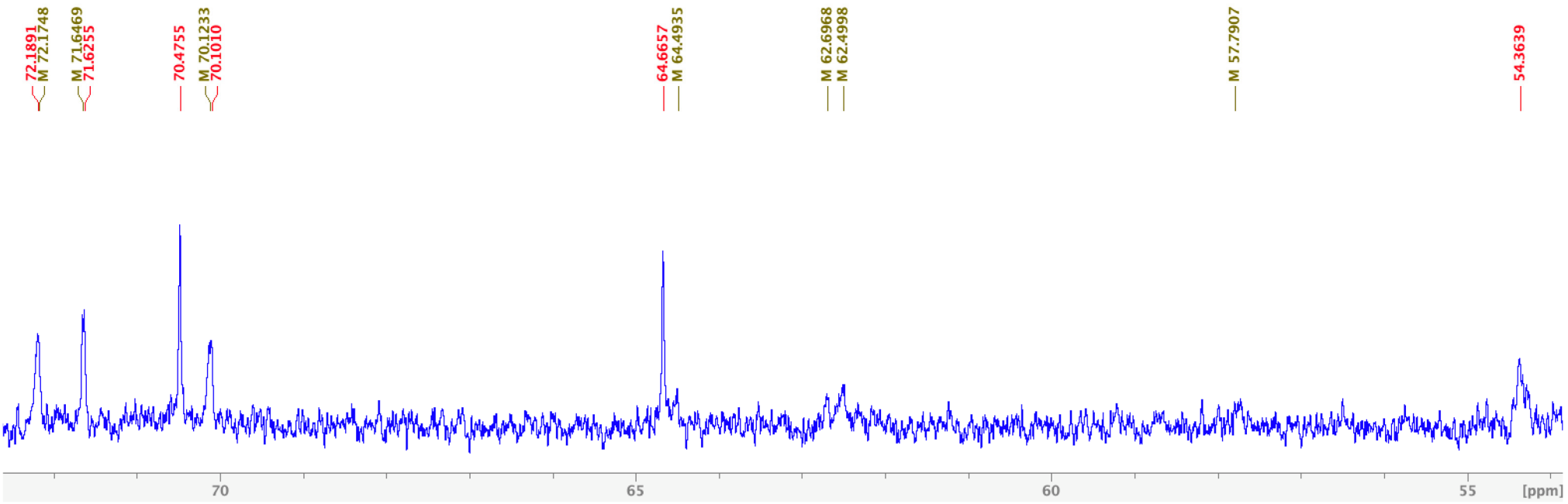

7.7157
7.6692
7.6512
7.4965
7.4763
7.4479
7.4294
7.2910
7.2729
7.2539
7.1200
7.1001
7.0806
7.0533
7.0332
6.8481
6.8291
6.8090
6.7293
M 6.7243
6.7088
M 6.7043
0.7101
1.0000
0.9732
0.8057
1.2492
1.1039
0.9686
0.9072
0.9266
1.1380
7.6
7.4
7.2
7.0
6.8
[ppm]
0.00
0.01
0.02
0.03
0.04
0.05


M 4.6533
4.6196
4.5603
4.5285
4.4061
4.3297
M 4.2443
4.2206
M 4.1831
M 4.1754
M 4.1601
4.1513
4.0849
4.0081
3.9759
3.6600
3.6467
3.6322
3.6229
3.5930
M 3.5551
M 3.5275
3.5190
3.4892
M 3.2962
3.2788
3.2631
3.2497
3.2329
3.0725
3.0479
3.0198
M 3.0006
2.9837
2.3587
2.7532
2.6495
3.2851
1.4720
1.7112
2.9928
2.2054
2.3978
2.3166
4.5
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3.5
3.0
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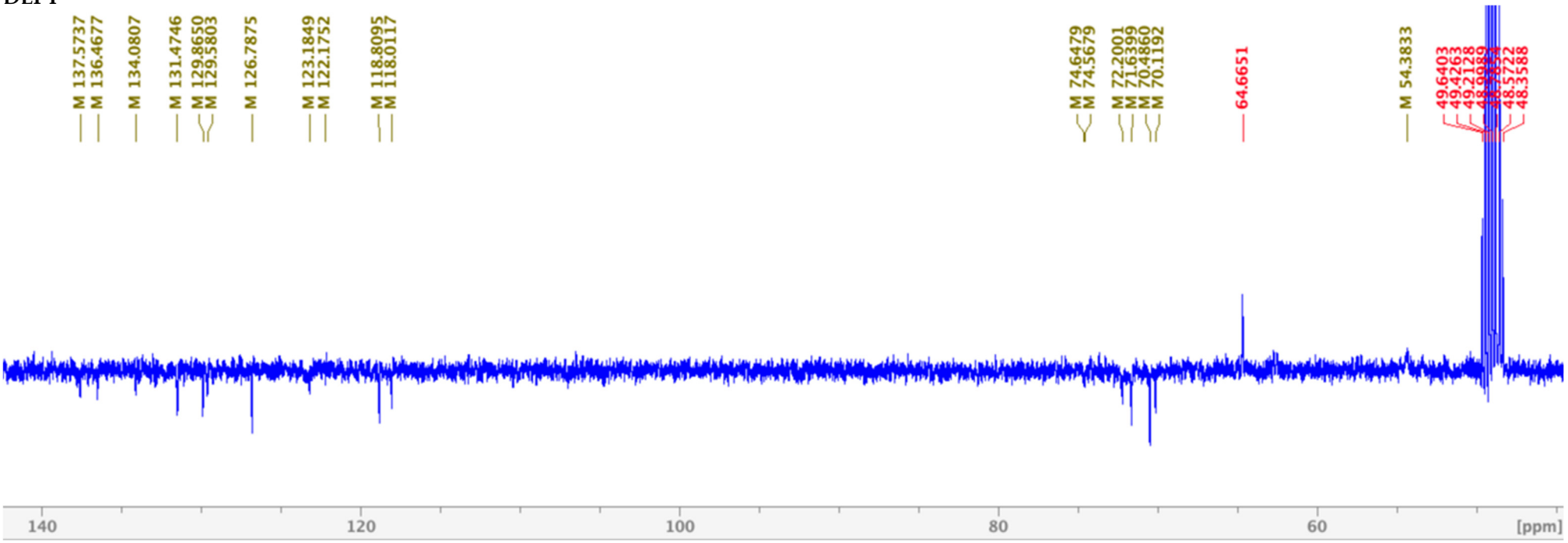
^{13}C-NMR


158.8219
137.5478
136.4487
134.0668
132.9880
131.4501
130.9012
129.8503
129.5740
126.7813
123.1807
118.7976
118.0096
72.1894
71.6258
70.4758
70.1012
64.6659
M 62.5186
54.3642
24.9711
21.2999
20.6017
160
140
120
100
80
60
40
[ppm]

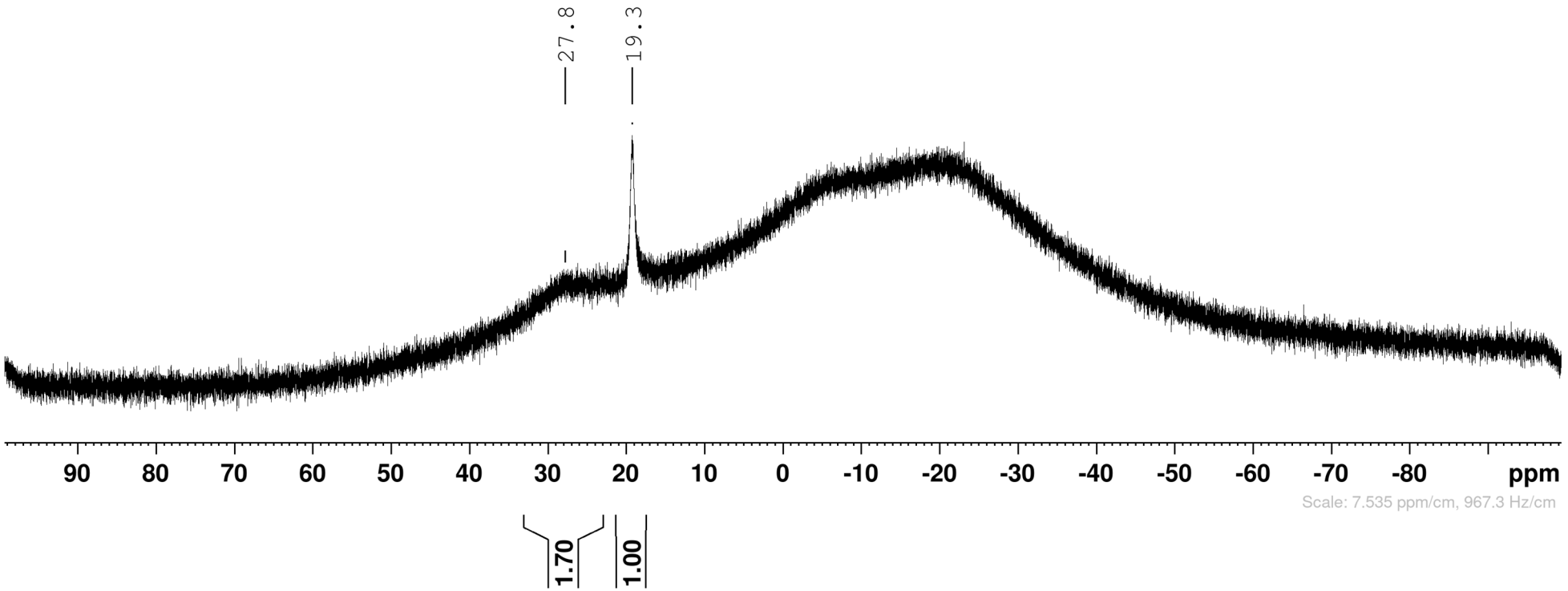

158.8219
137.5478
136.4487
134.0668
132.9880
131.4501
130.9012
129.8503
129.5740
126.7813
123.1807
118.7976
118.0096
160
150
140
130
120
[ppm]


72.1891
M 72.1748
M 71.6469
71.6255
70.4755
M 70.1233
70.1010
64.6657
M 64.4935
M 62.6968
M 62.4998
M 57.7907
54.3639
70
65
60
55
[ppm]

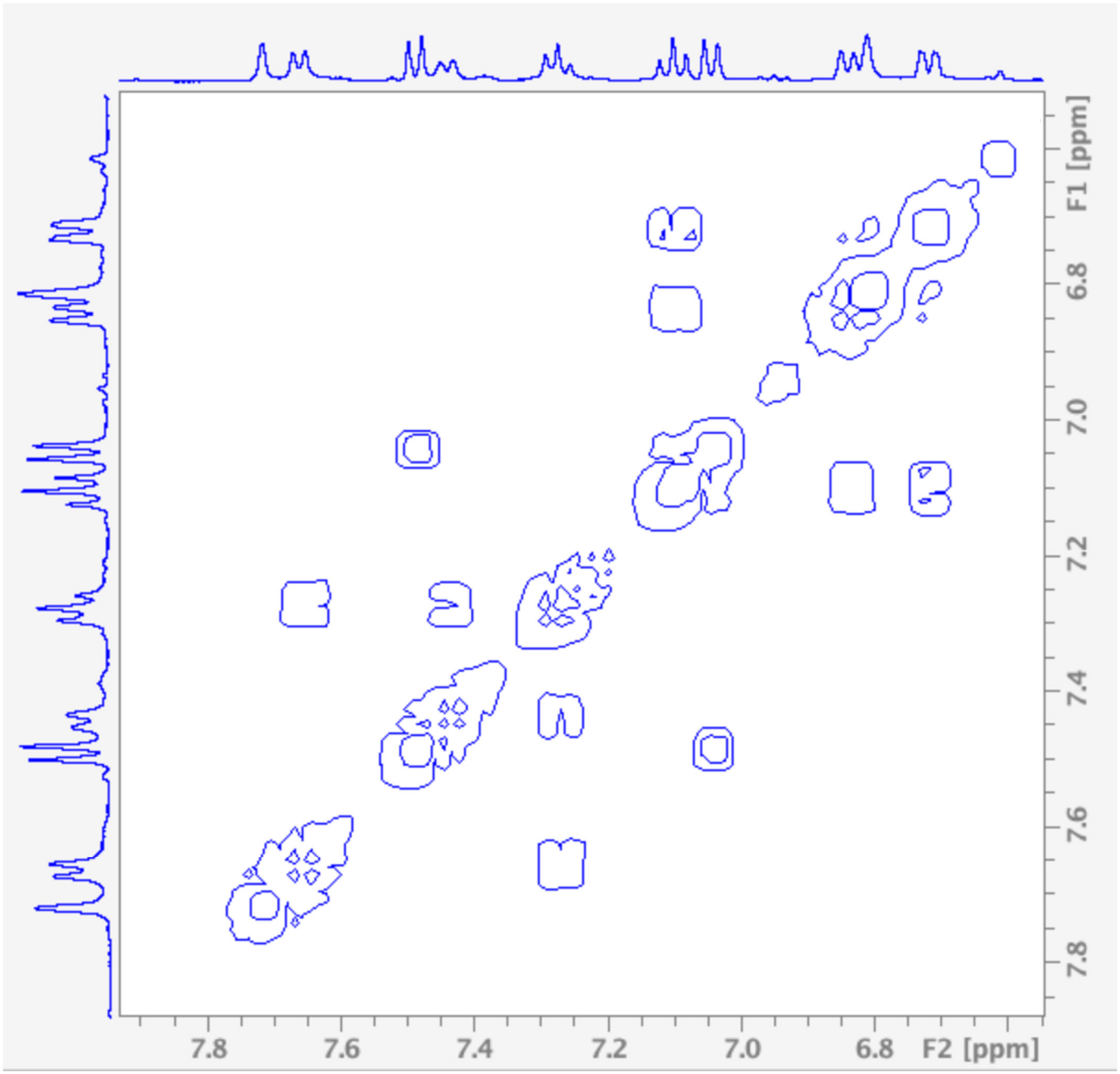
DEPT

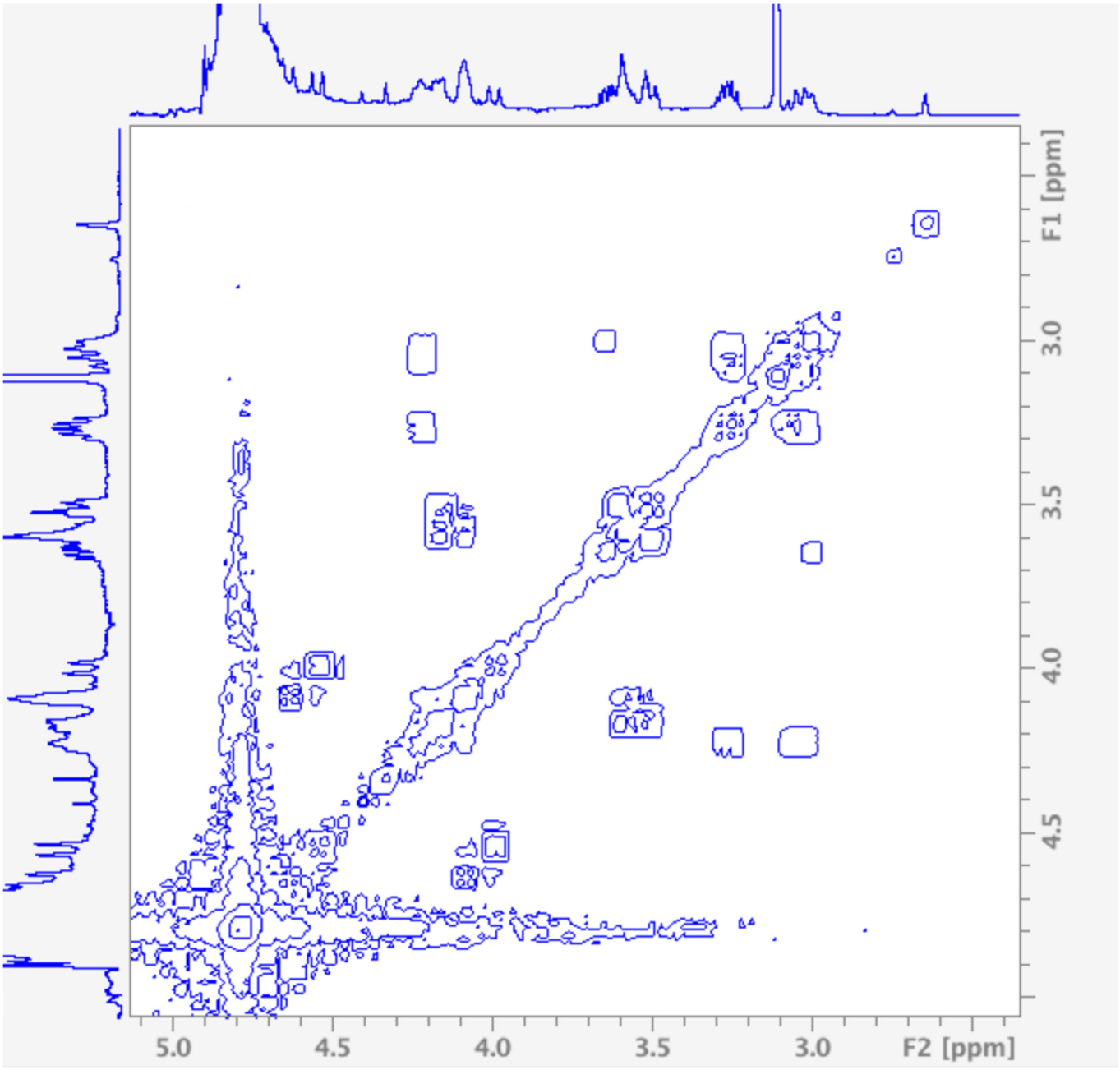

M 137.5737
M 136.4677
M 134.0807
M 131.4746
M 129.8650
M 129.5803
M 126.7875
M 123.1849
M 122.1752
M 118.8095
M 118.0117
M 74.6479
M 74.5679
M 72.2001
M 71.6399
M 70.4860
M 70.1192
64.6651
M 54.3833
49.6403
49.4263
49.2128
48.5722
48.3588
140
120
100
80
60
[ppm]

^{11}B-NMR

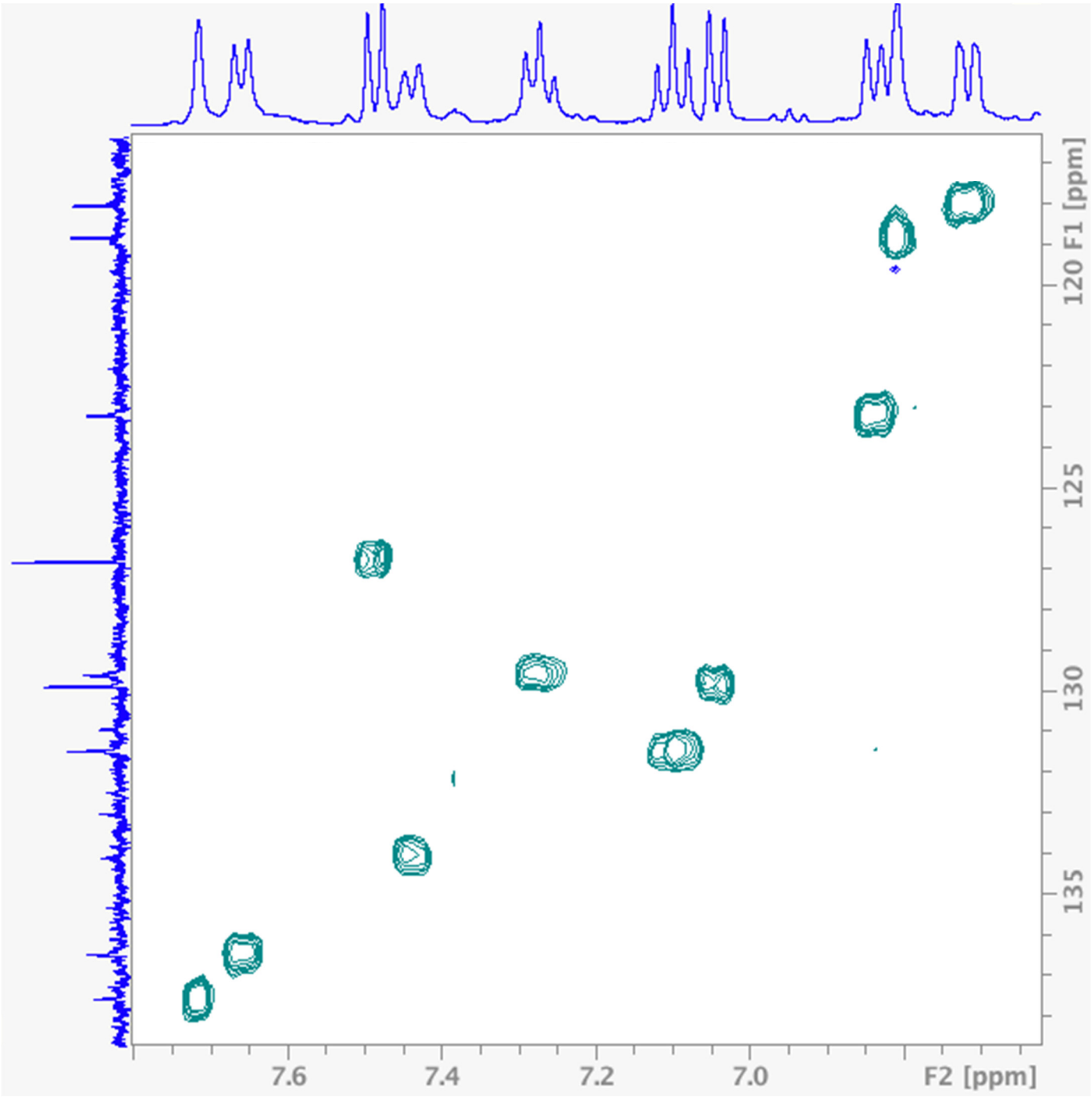

27.8
19.3
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
ppm
Scale: 7.535 ppm/cm, 967.3 Hz/cm
1.70
1.00

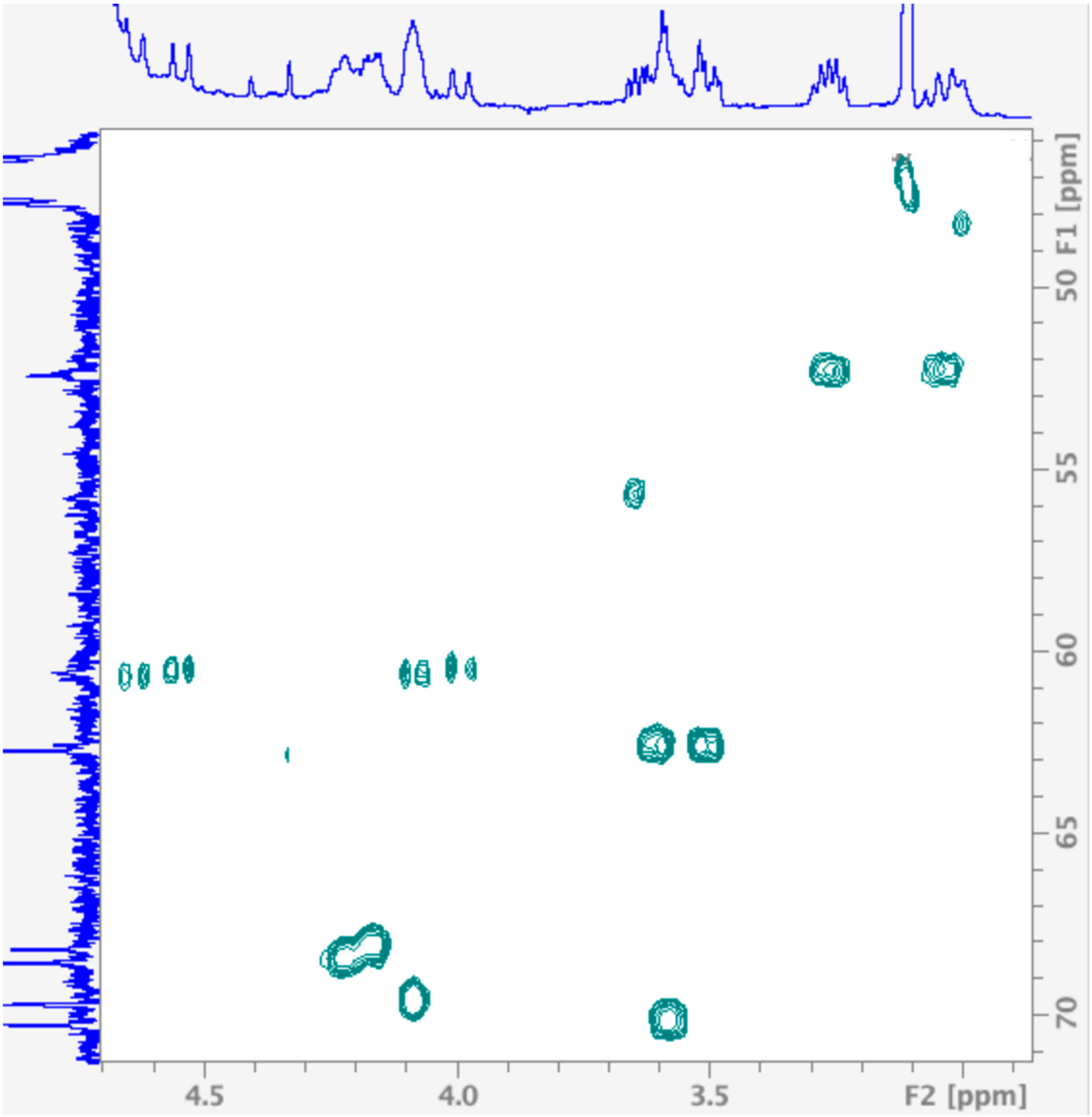
COSY




F1 [ppm]
3.0
3.5
4.0
4.5
5.0
4.5
4.0
3.5
3.0
F2 [ppm]

HSQC




50
55
60
65
70
F1 [ppm]
4.5
4.0
3.5
F2 [ppm]

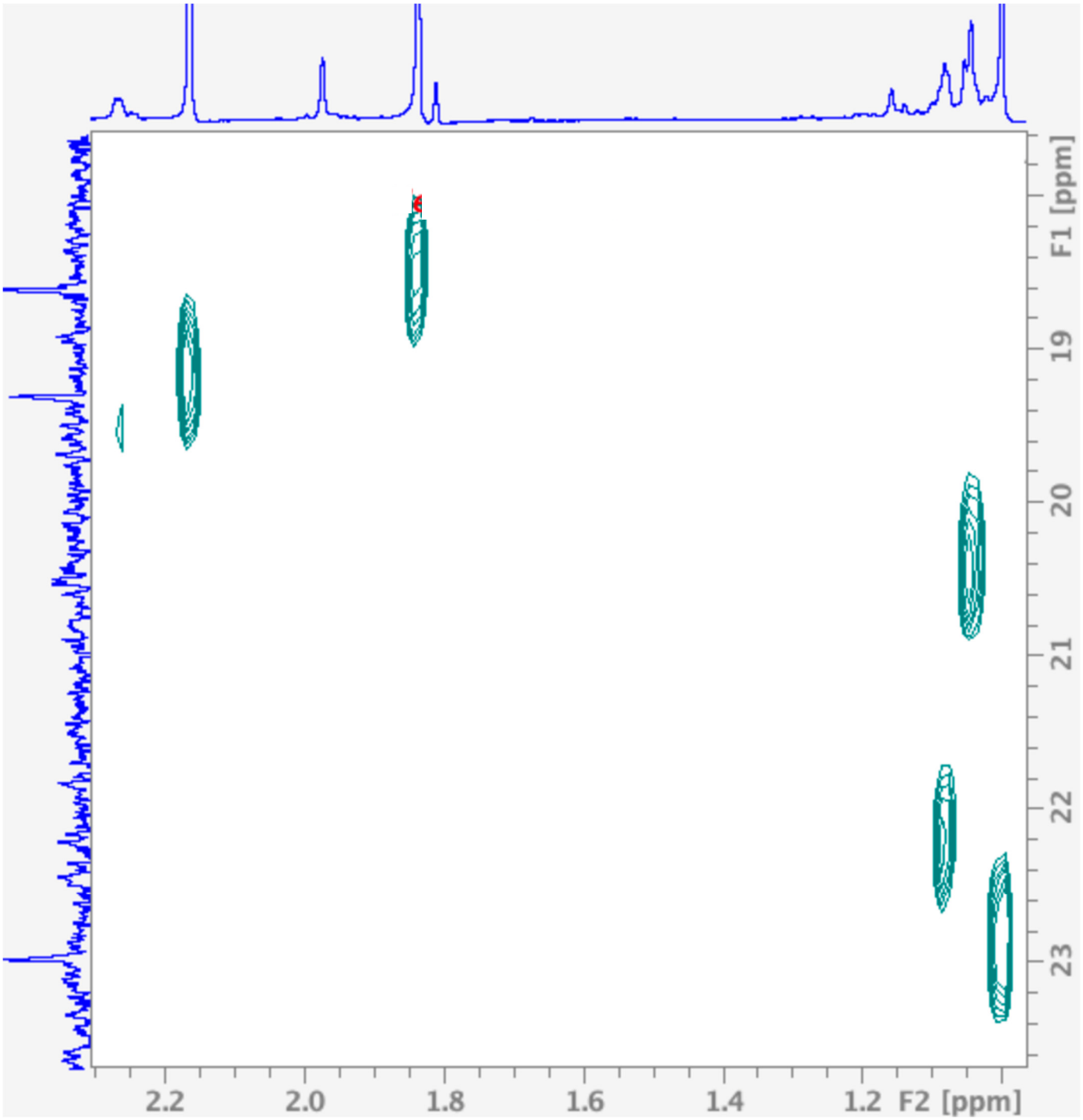
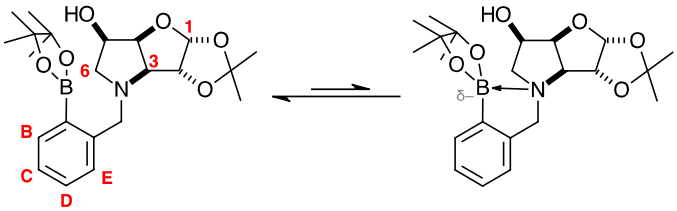
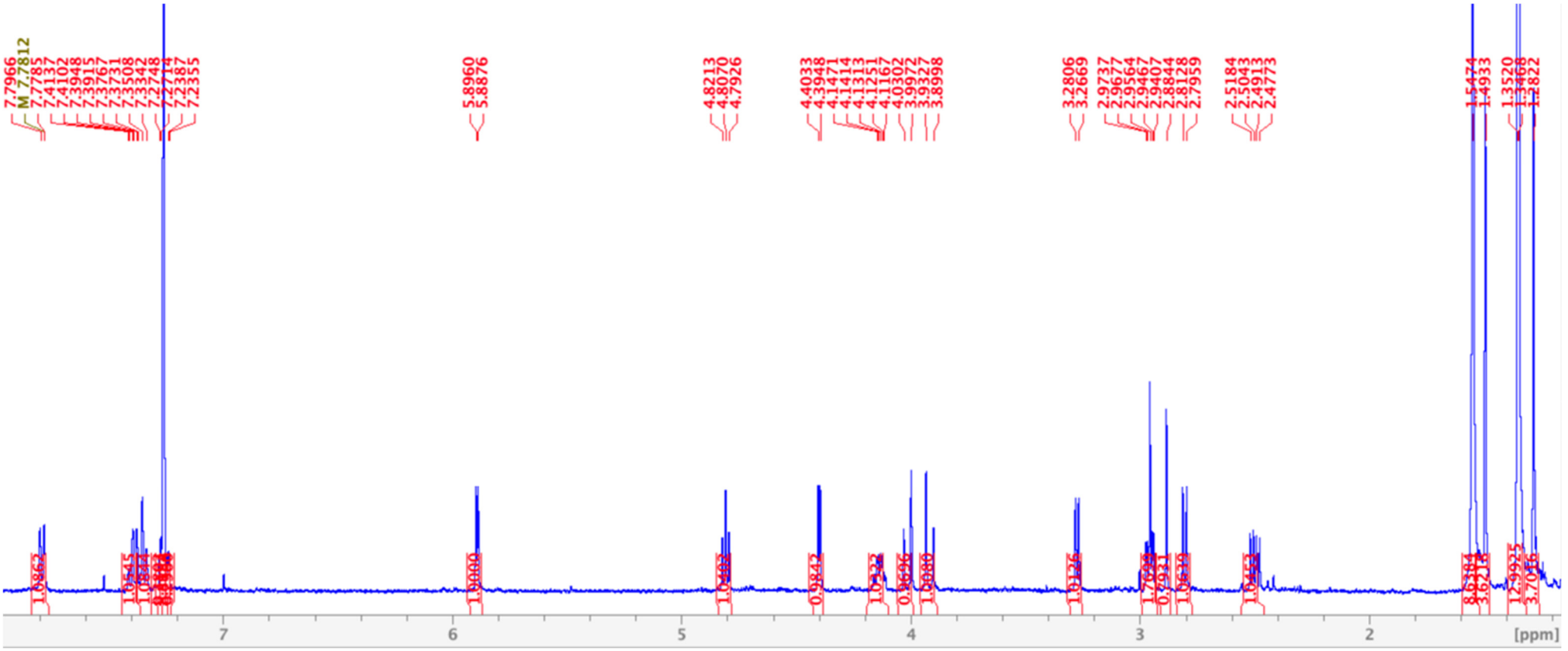

F1 [ppm]
19
20
21
22
23
2.2
2.0
1.8
1.6
1.4
1.2
F2 [ppm]

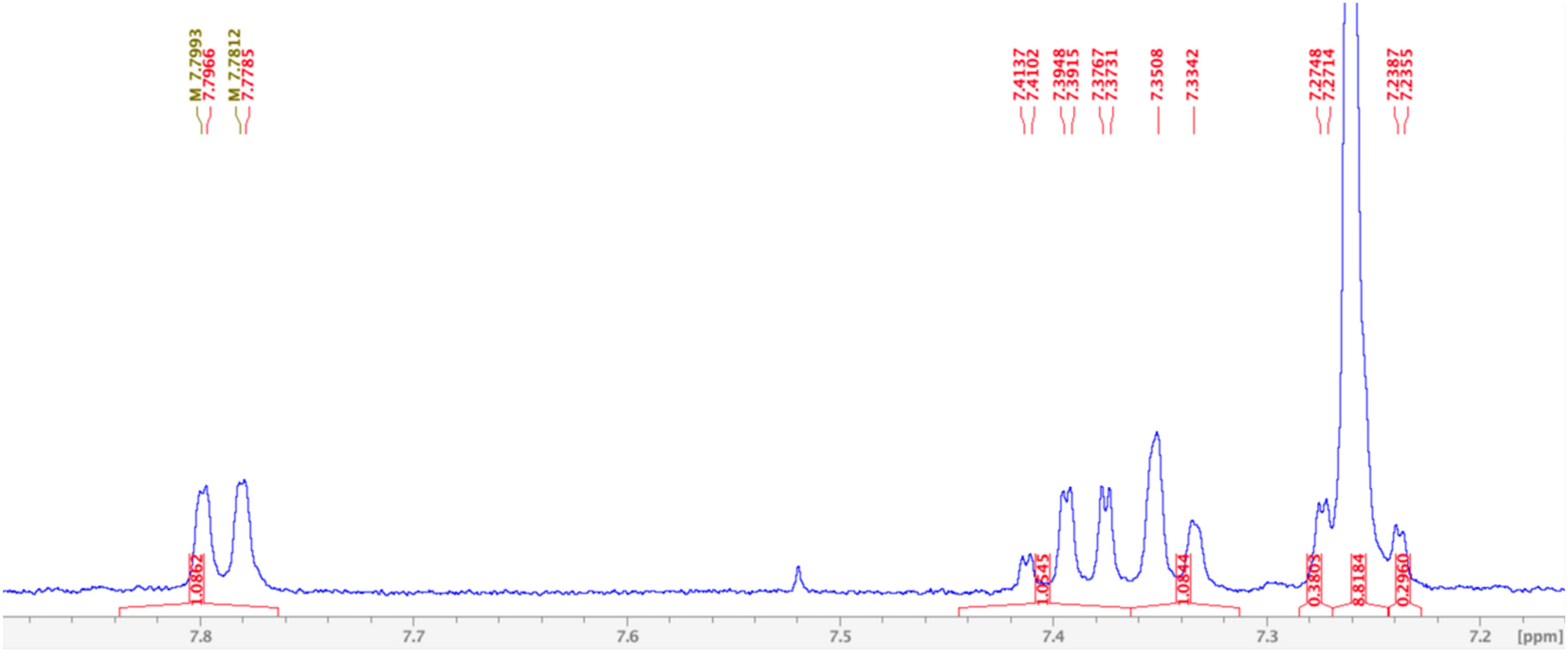
Figure S6. ^{1}H- (400 MHz), ^{13}C-NMR (100 MHz), DEPT, ^{11}B-NMR (128 MHz), COSY, HSQC and HMBC spectra of *N*-(2-methylphenyl boronic acid pinacol ester)-3,6-dideoxy-3,6-imino-1,2-*O*-isopropylidene-α-D-gulofuranose **ortho 2** in $CDCl_3$.

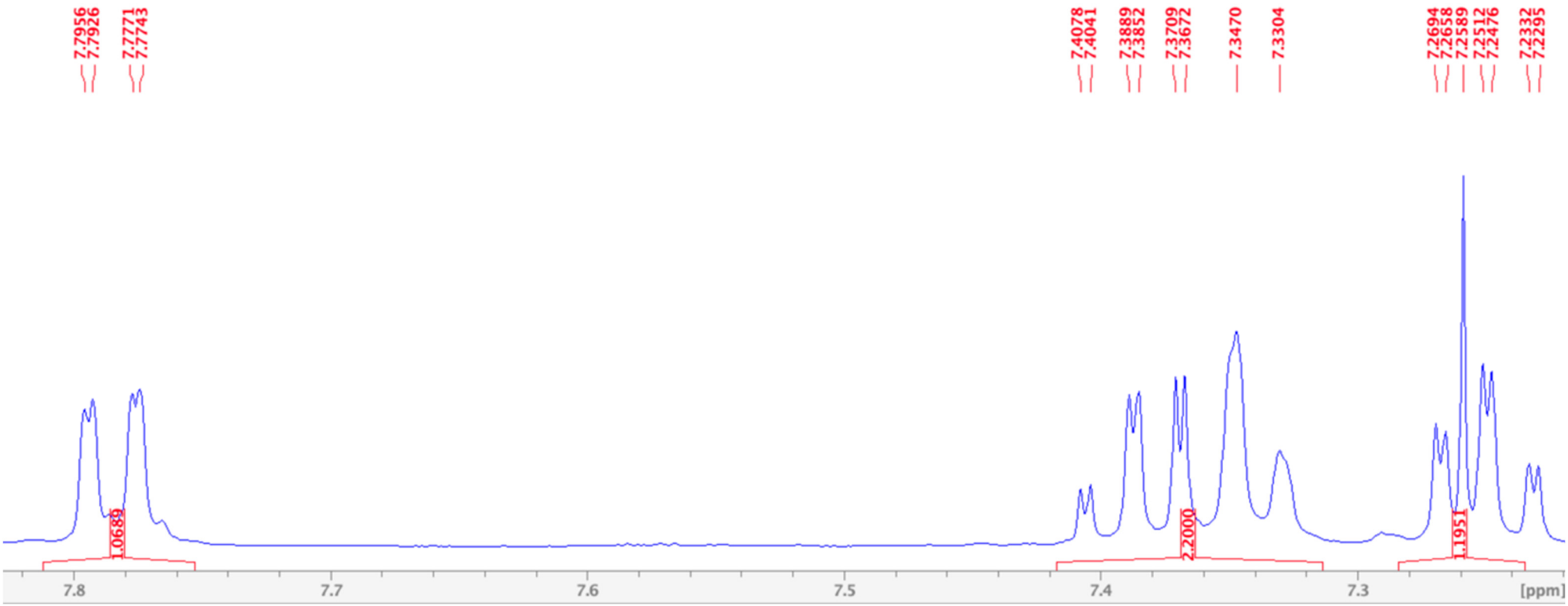


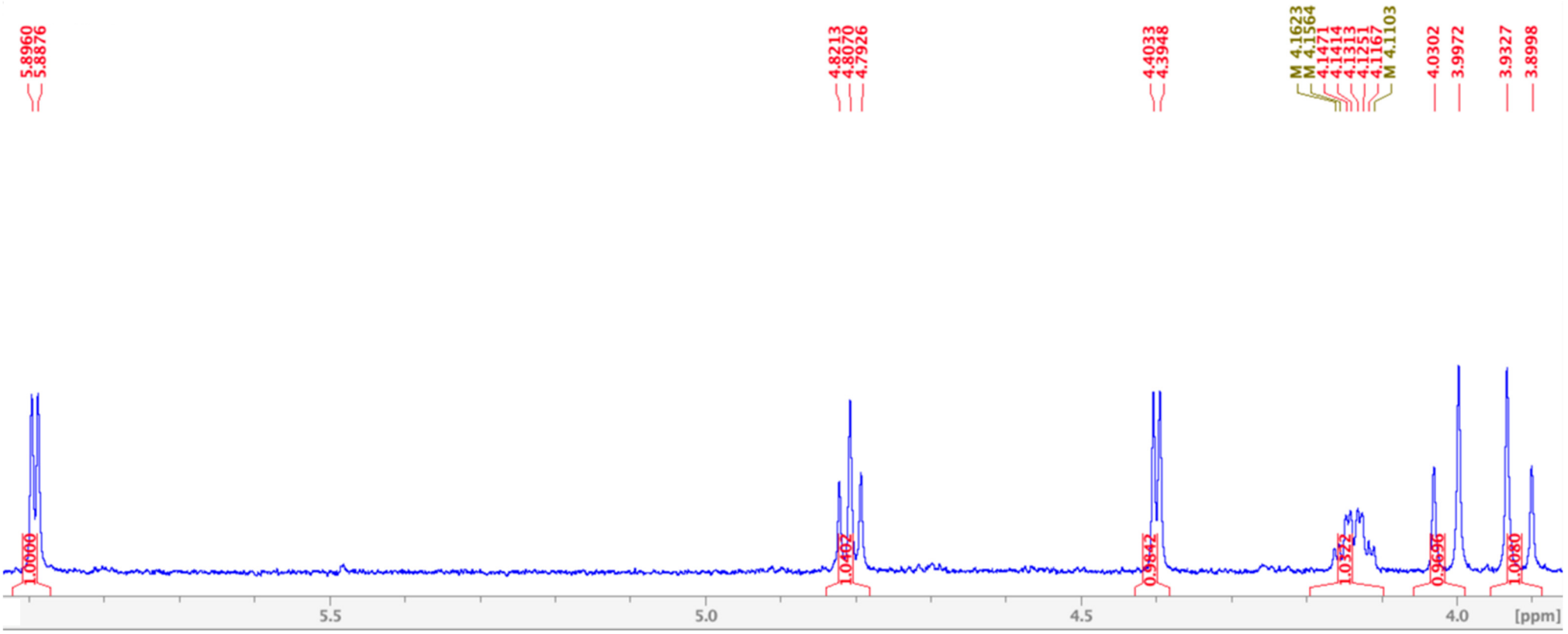
Boronic acid species. **δH**: 7.80 (1H, dd, $J_{HB,HC}$ 7.3 Hz, $J_{HB,HD}$ 1.1 Hz, ArH^{B}), 7.39 (1H, td, $J_{HD,HC/HE}$ 7.5 Hz, $J_{HD,HB}$ 1.4 Hz, ArH^{D}), 7.34 (1H, app-d, $J_{HE,HD}$ 7.6 Hz, ArH^{E}), 7.25 (1H, td partially obscured by $CDCl_3$ signal, $J_{HC,HB/HD}$ 7.2 Hz, $J_{HC,HE}$ 1.3 Hz, ArH^{C}), 5.89 (1H, d, $J_{H\text{-}1,H\text{-}2}$ 3.4 Hz, H-1), 4.81 (1H, app-t, $J_{H\text{-}4,H\text{-}3/H\text{-}5}$ 5.8 Hz, H-4), 4.40 (1H, d, $J_{H\text{-}2,H\text{-}1}$ 3.4 Hz, H-2), 4.14 (1H, app-dddd, $J_{H\text{-}5,H\text{-}4/H\text{-}6/OH}$ 6.0 Hz, $J_{H\text{-}5,H\text{-}6'}$ 2.4 Hz, H-5), 4.01 (1H, d, $J_{Ha,Hb}$ 13.2 Hz, C*H^{a}*H^{b}Ar), 3.91 (1H, d, $J_{Hb,Ha}$ 13.2 Hz, CH^{a}*H^{b}*Ar), 3.27 (1H, app-d, $J_{H\text{-}3,H\text{-}4}$ 5.5 Hz, H-3), 2.96 (1H, dd, $J_{H\text{-}6',H\text{-}6}$ 10.8 Hz, $J_{H\text{-}6',H\text{-}5}$ 2.4 Hz, H-6'), 2.80 (1H, d, $J_{OH,H\text{-}5}$ 6.8 Hz, OH), 2.50 (1H, dd, $J_{H\text{-}6,H\text{-}6'}$ 10.8 Hz, $J_{H\text{-}6,H\text{-}5}$ 5.6 Hz, H-6), 1.49, 1.28 (6H, 2 x s, 2 x CH_3, acetonide), 1.352, 1.347 (12H, 2 x s, pinacol 4 x CH_3); **δC**: 144.2 (ArC$_{quat}$), 136.1 (ArC^{B}), 130.9 (ArC^{D}), 129.4 (ArC^{E}), 126.7 (ArC^{C}), 112.6 (C$_{quat}$ acetonide), 107.5 (C-1), 83.9 (C-2), 83.8 (2 x C$_{quat}$ pinacol), 83.2 (C-4), 72.9 (C-3), 69.7 (C-5), 61.6 (C-6), 57.7 (CH_2Ar), 27.7, 27.0 (2 x CH_3, acetonide), 25.1, 25.0 (4 x CH_3 pinacol). ArC$_{quat}$-B not discernible.
δB: 31.0 (sharp, integration: 6.4), 22.3 (sharp, integration: 1.0).

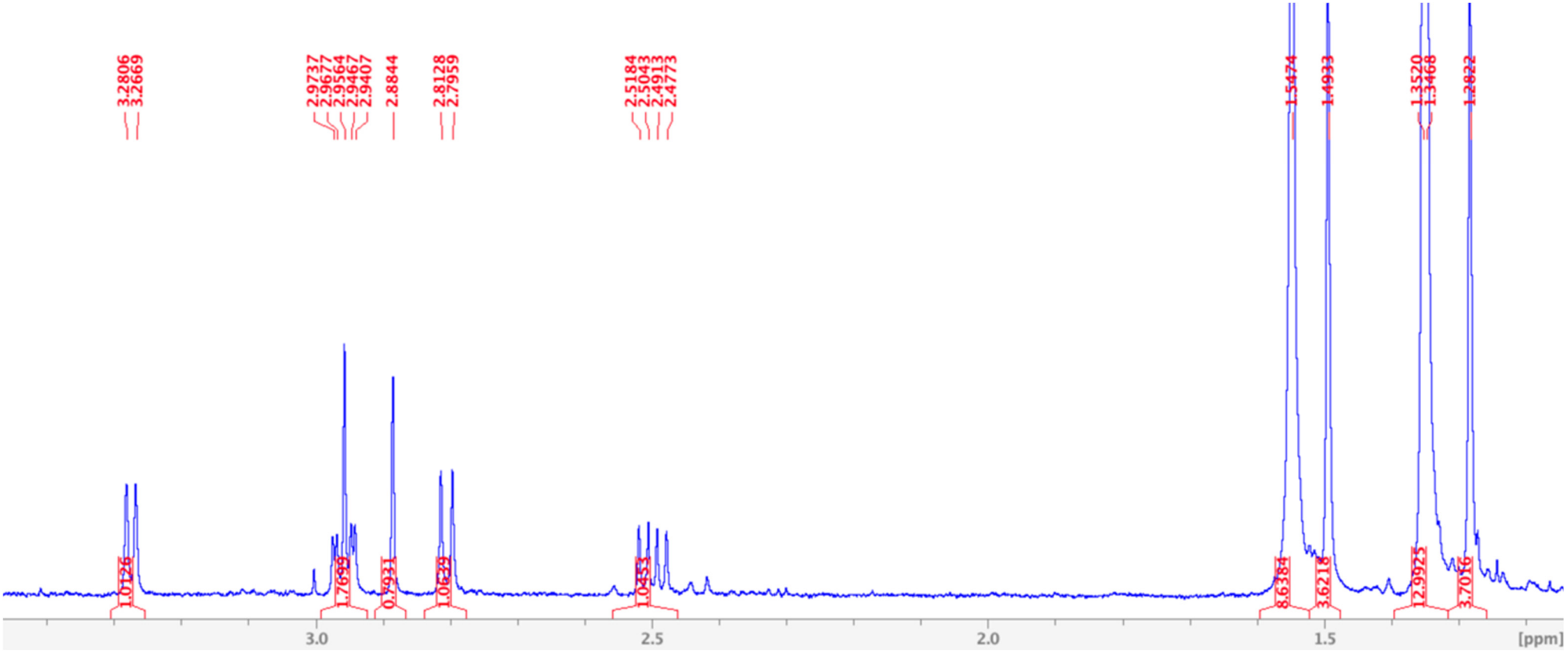
^{1}H-NMR


7.7966
M 7.7812
7.7785
7.4137
7.4102
7.3948
7.3915
7.3767
7.3731
7.3508
7.3342
7.3248
7.2714
7.2387
7.2355
5.8960
5.8876
4.8213
4.8070
4.7926
4.4033
4.3948
4.1471
4.1414
4.1313
4.1251
4.1167
4.0302
3.9972
3.9327
3.8998
3.2806
3.2669
2.9737
2.9677
2.9564
2.9467
2.9407
2.8844
2.8128
2.7959
2.5184
2.5043
2.4913
2.4773
1.5474
1.4933
1.3520
1.3468
1.2822
7
6
5
4
3
2
[ppm]

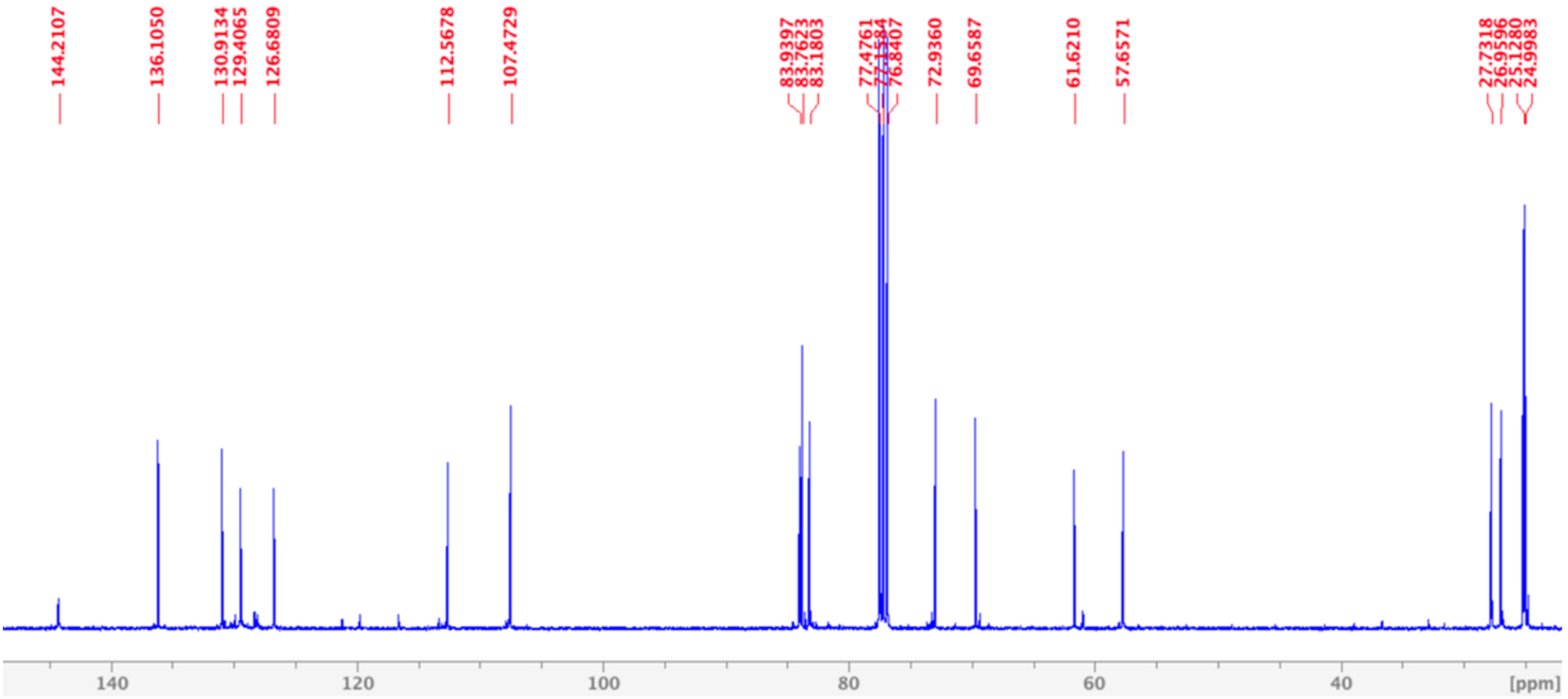

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M 7.7812
7.7785
7.4137
7.4102
7.3948
7.3915
7.3767
7.3731
7.3508
7.3342
7.2748
7.2714
7.2387
7.2355
1.0862
1.0545
1.0844
0.3805
8.8184
0.2960
7.8
7.7
7.6
7.5
7.4
7.3
7.2
[ppm]


7.7956
7.7926
7.7771
7.7743
7.4078
7.4041
7.3889
7.3852
7.3709
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7.2512
7.2476
7.2332
7.2295
1.0689
2.2000
1.1951
7.8
7.7
7.6
7.5
7.4
7.3
[ppm]

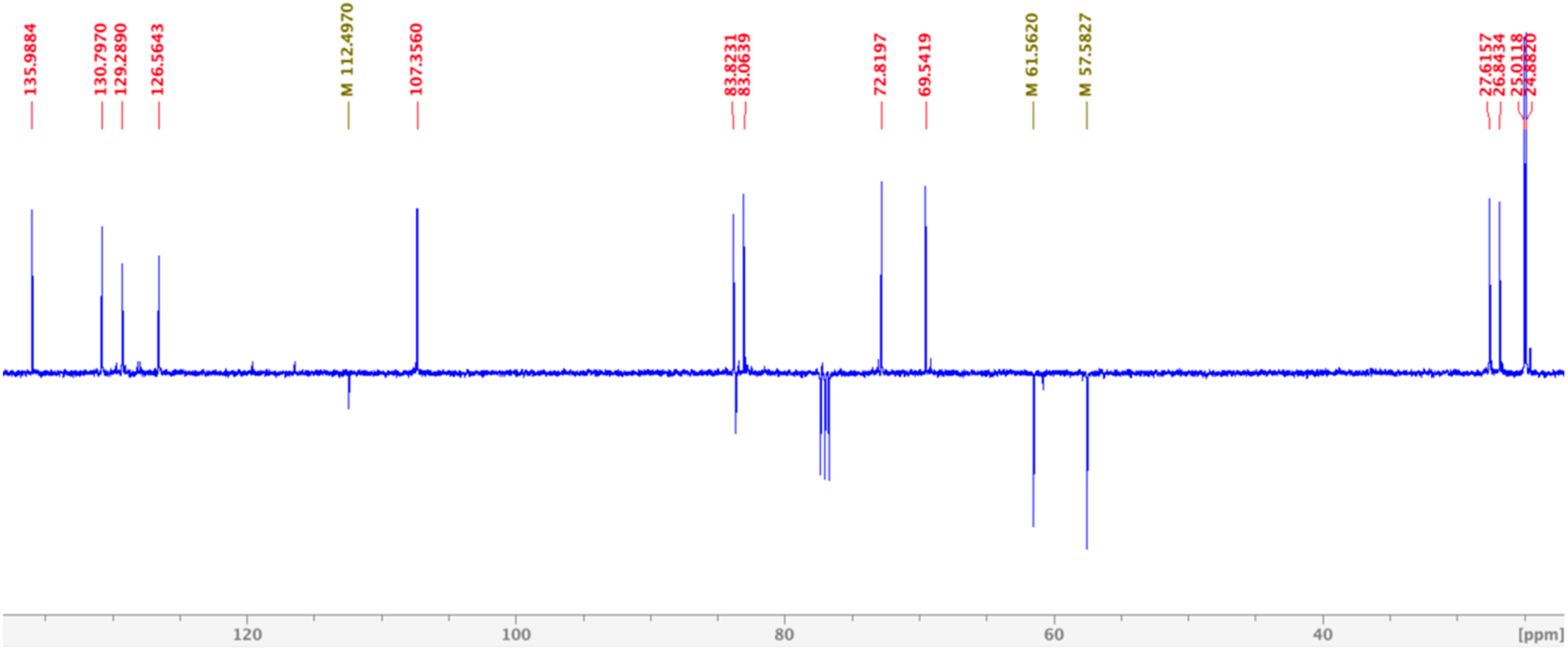

5.8960
5.8876
4.8213
4.8070
4.7926
4.4033
4.3948
M 4.1623
M 4.1564
4.1471
4.1414
4.1313
4.1251
4.1167
M 4.1103
4.0302
3.9972
3.9327
3.8998
1.0000
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0.9842
1.0322
0.9696
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4.5
4.0
[ppm]

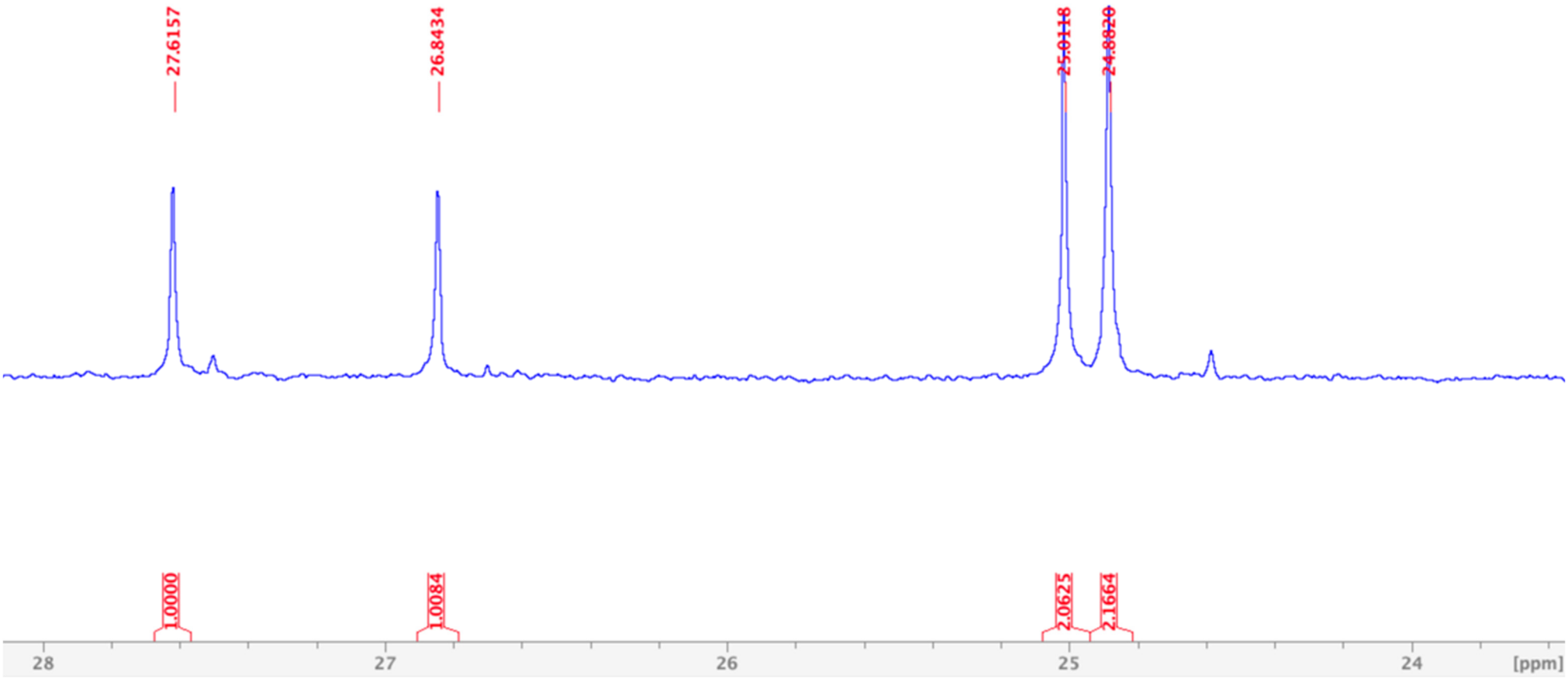

3.2806
3.2669
2.9737
2.9677
2.9564
2.9467
2.9407
2.8844
2.8128
2.7959
2.5184
2.5043
2.4913
2.4773
1.5474
1.4933
1.3520
1.3468
1.2822
1.0126
1.7699
0.7931
1.0629
1.0453
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3.0
2.5
2.0
1.5
[ppm]

^{13}C-NMR

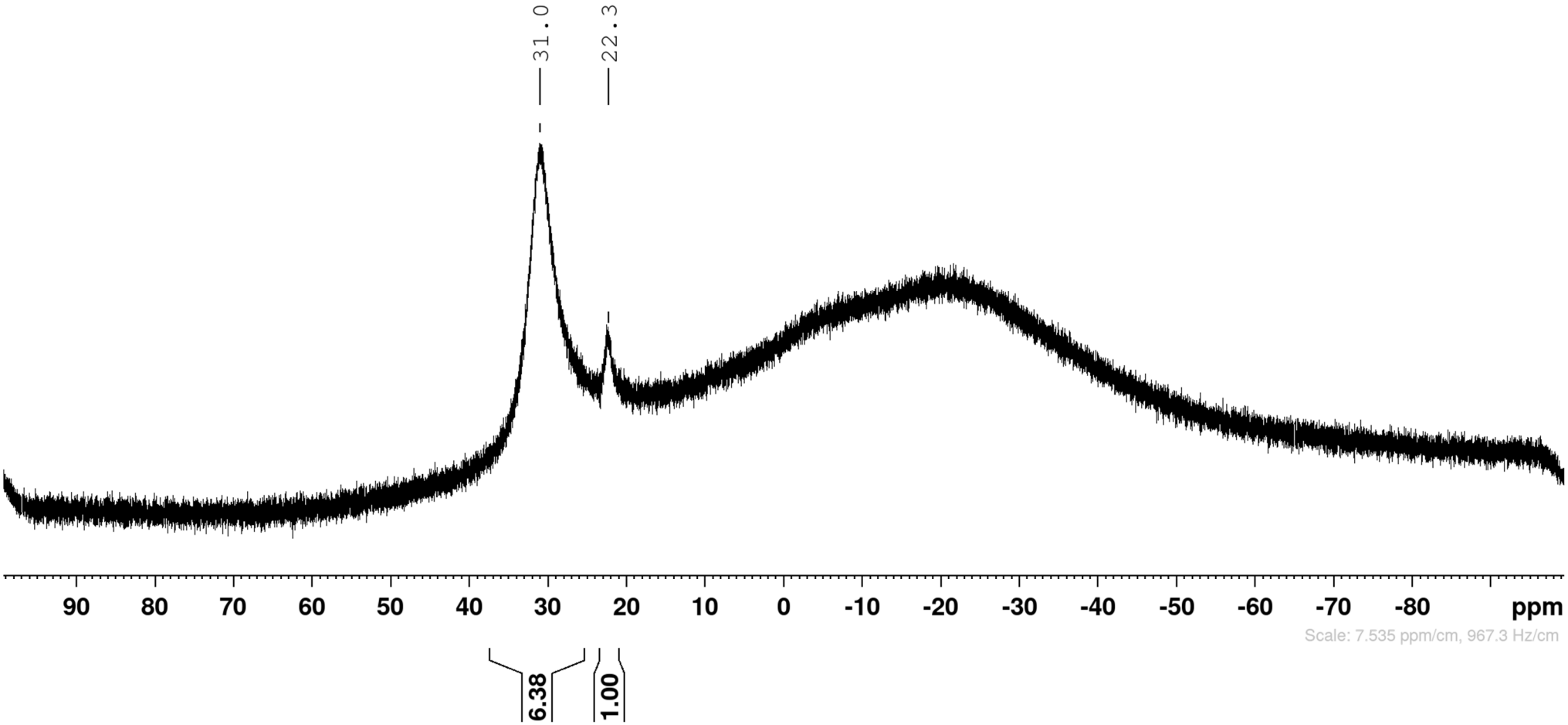

144.2107
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130.9134
129.4065
126.6809
112.5678
107.4729
83.9397
83.7623
83.1803
77.4761
77.1584
76.8407
72.9360
69.6587
61.6210
57.6571
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26.9596
25.1280
24.9983
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120
100
80
60
40
[ppm]

DEPT

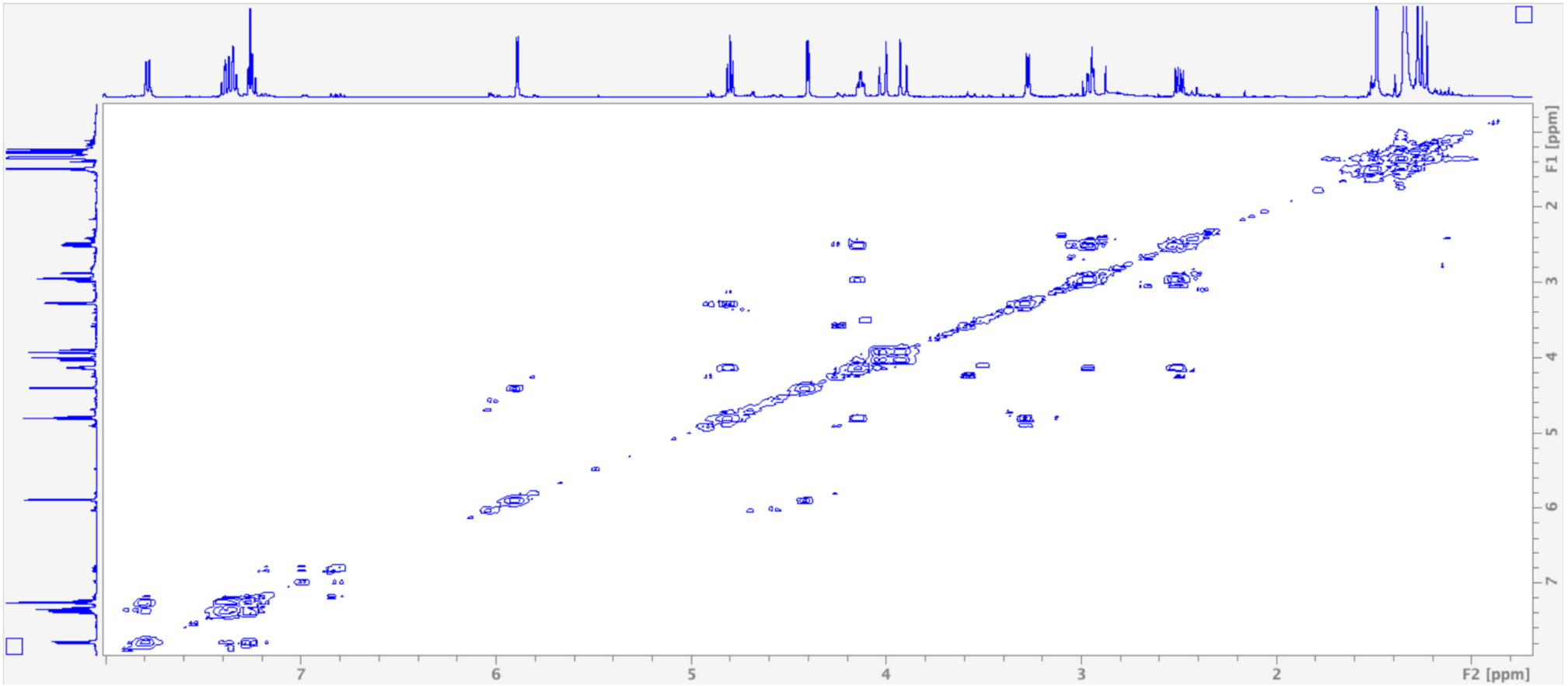

135.9884
130.7970
129.2890
126.5643
M 112.4970
107.3560
83.8231
83.0639
72.8197
69.5419
M 61.5620
M 57.5827
27.6157
26.8434
25.0118
24.8820
120
100
80
60
40
[ppm]


27.6157
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24.8020
1.0000
1.0084
2.0625
2.1664
28
27
26
25
24
[ppm]

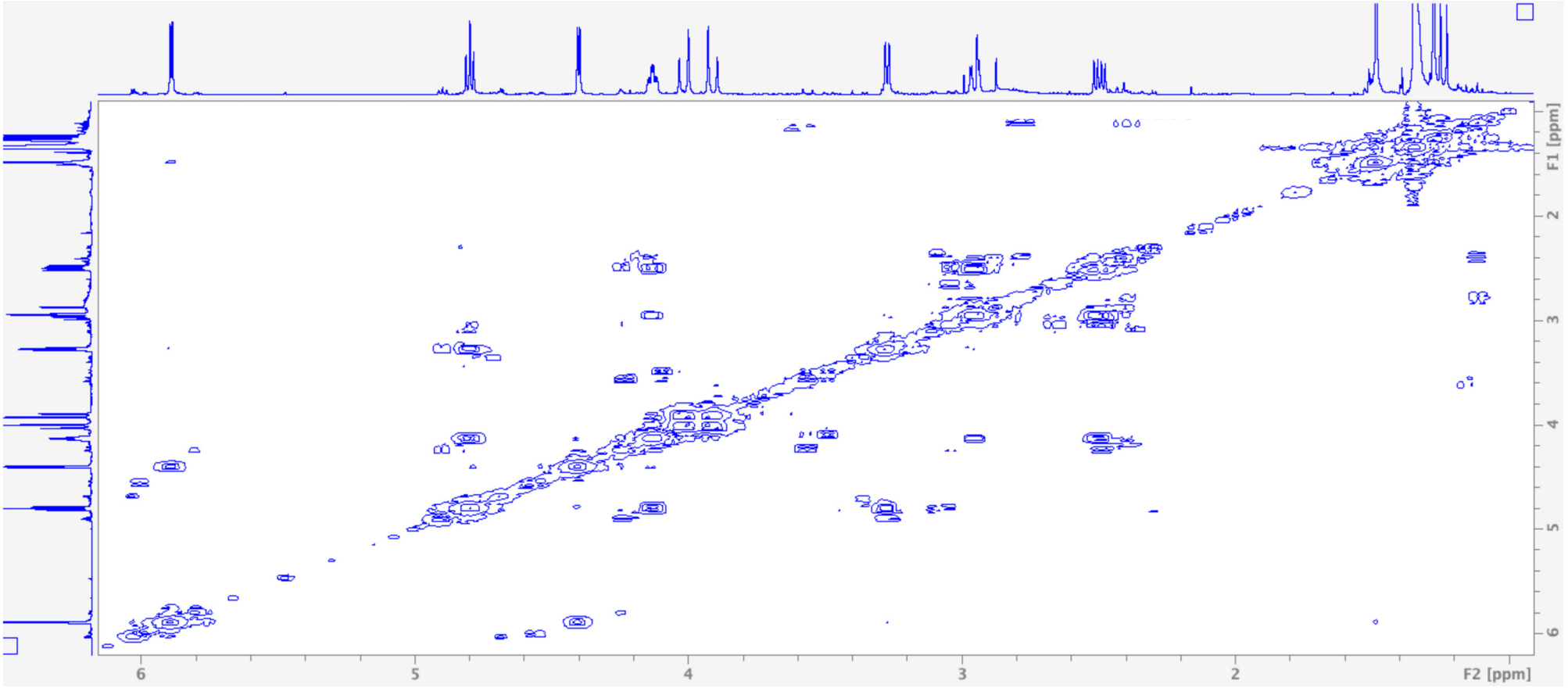
^{11}B-NMR

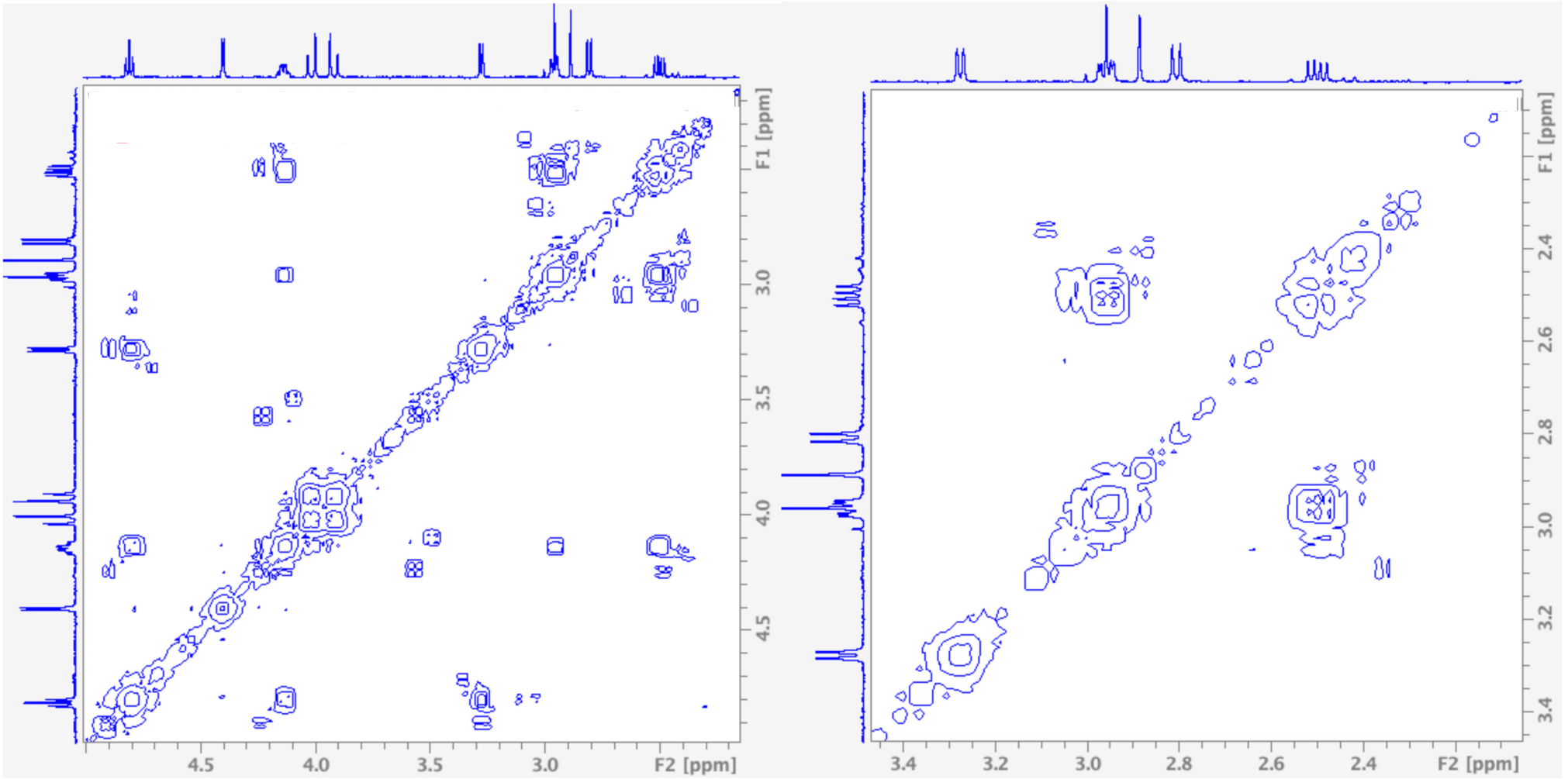

31.0
22.3
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
ppm
Scale: 7.535 ppm/cm, 967.3 Hz/cm
6.38
1.00

COSY

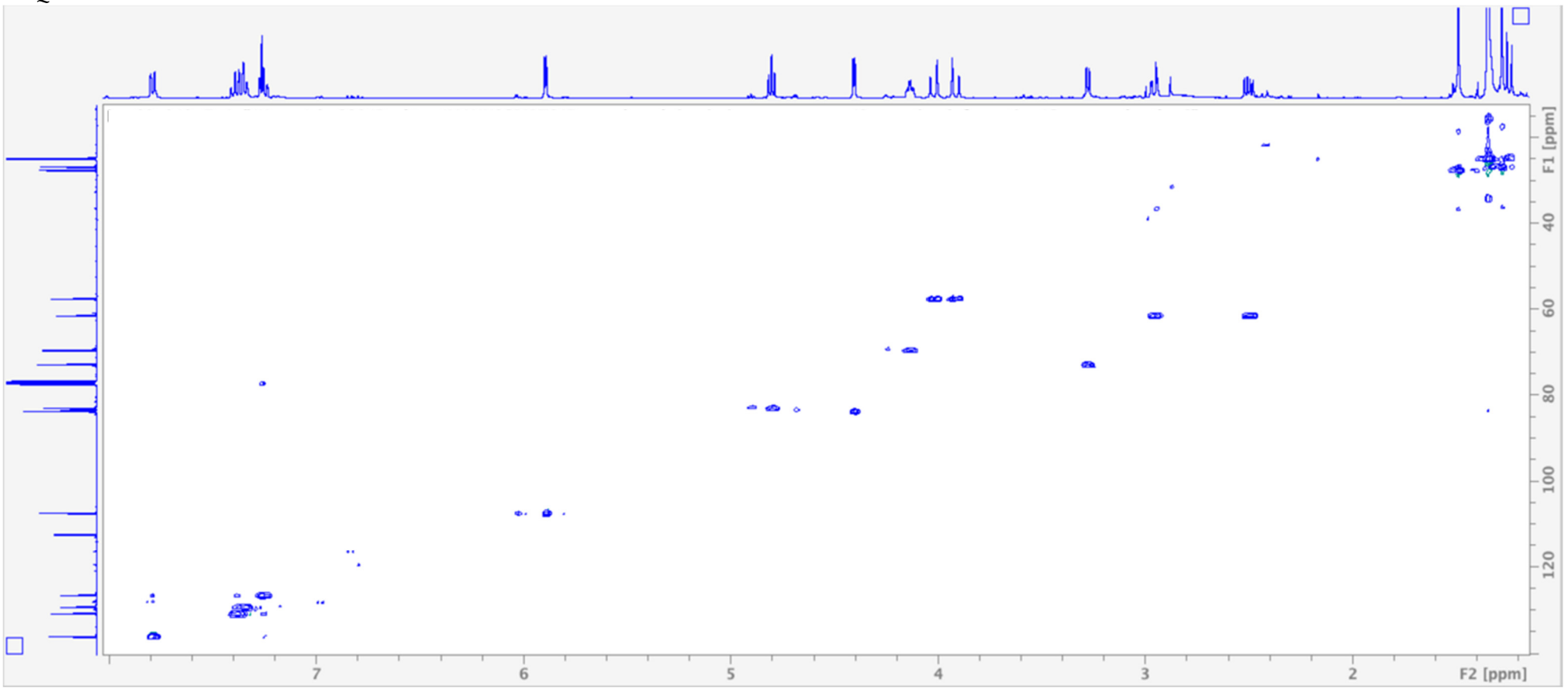


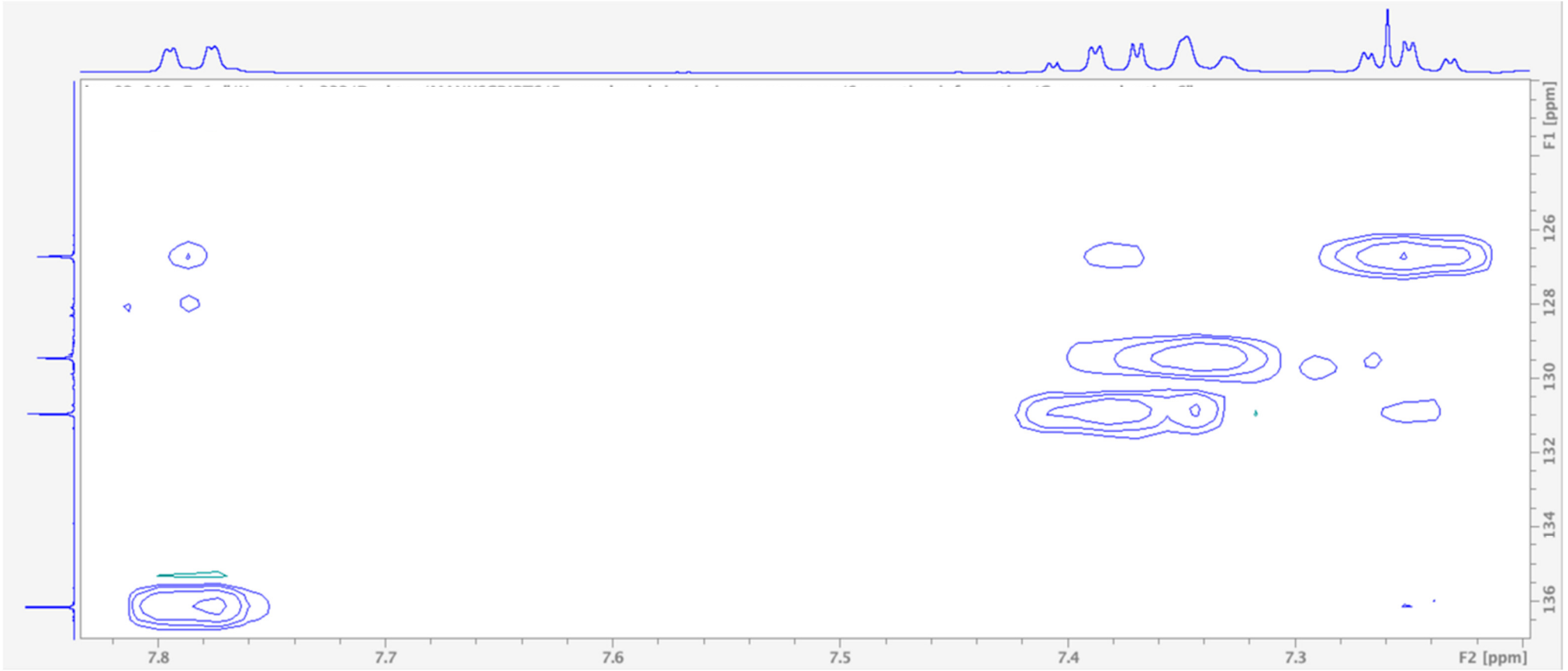

F1 [ppm]
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7.6
7.8
7.8
7.6
7.4
7.2
F2 [ppm]

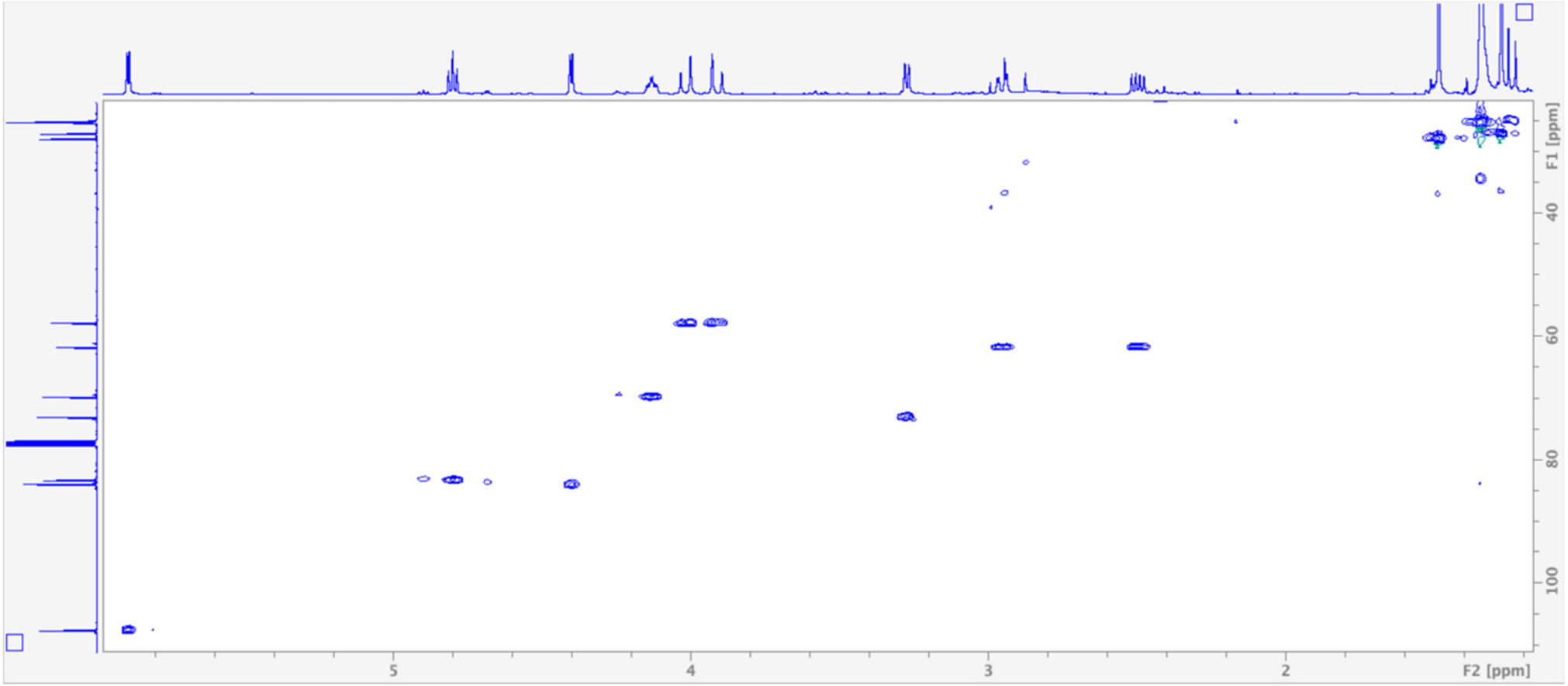

F1 [ppm]
2
3
4
5
6
6
5
4
3
2
F2 [ppm]


F1 [ppm]
3.0
3.5
4.0
4.5
4.5
4.0
3.5
3.0
F2 [ppm]
F1 [ppm]
2.4
2.6
2.8
3.0
3.2
3.4
3.4
3.2
3.0
2.8
2.6
2.4
F2 [ppm]

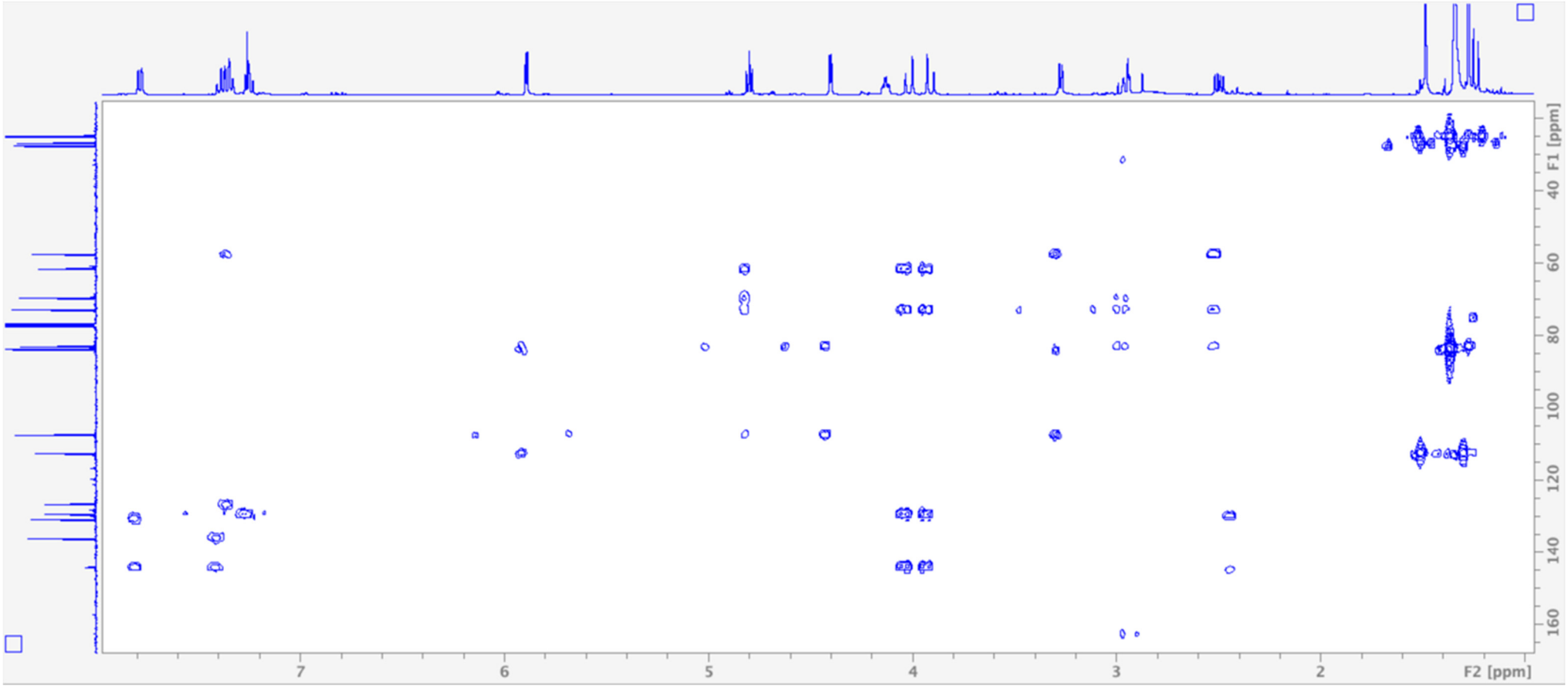
HSQC

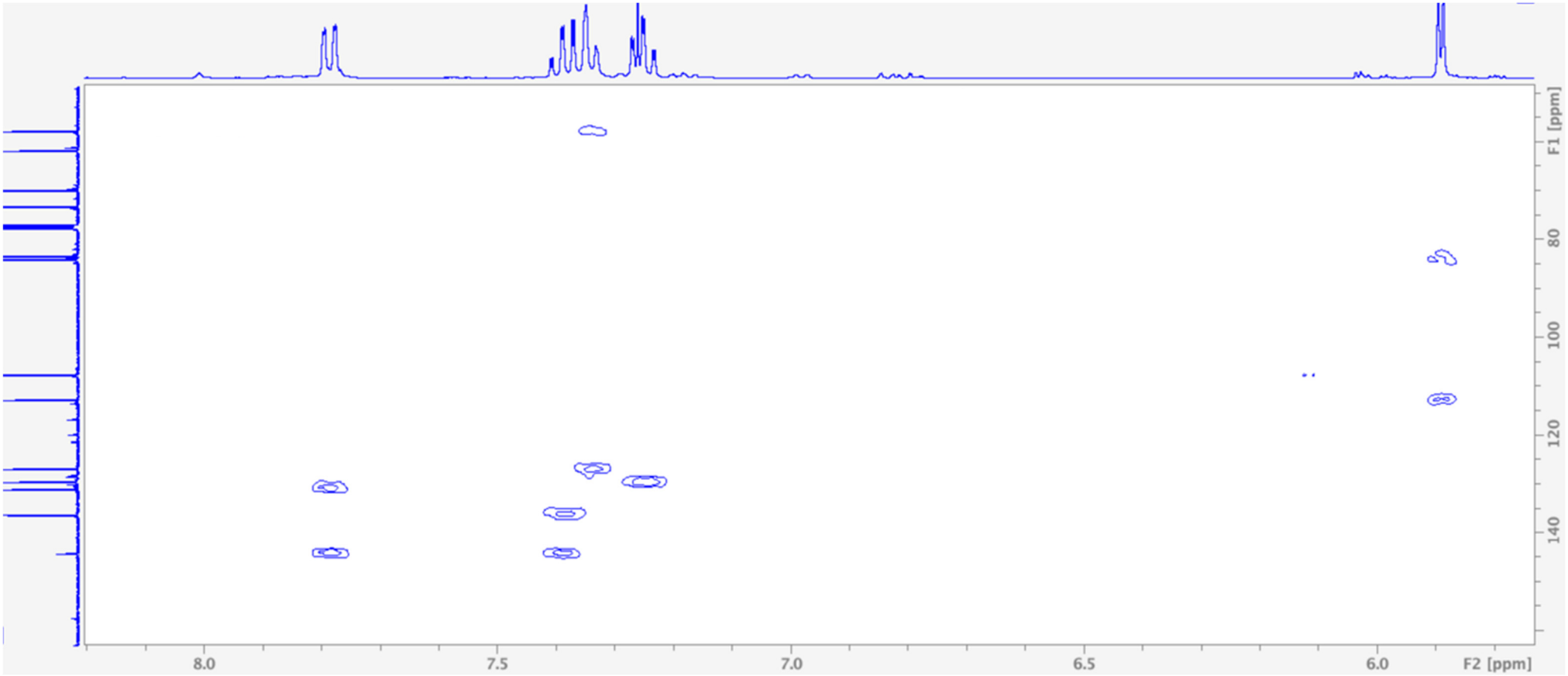


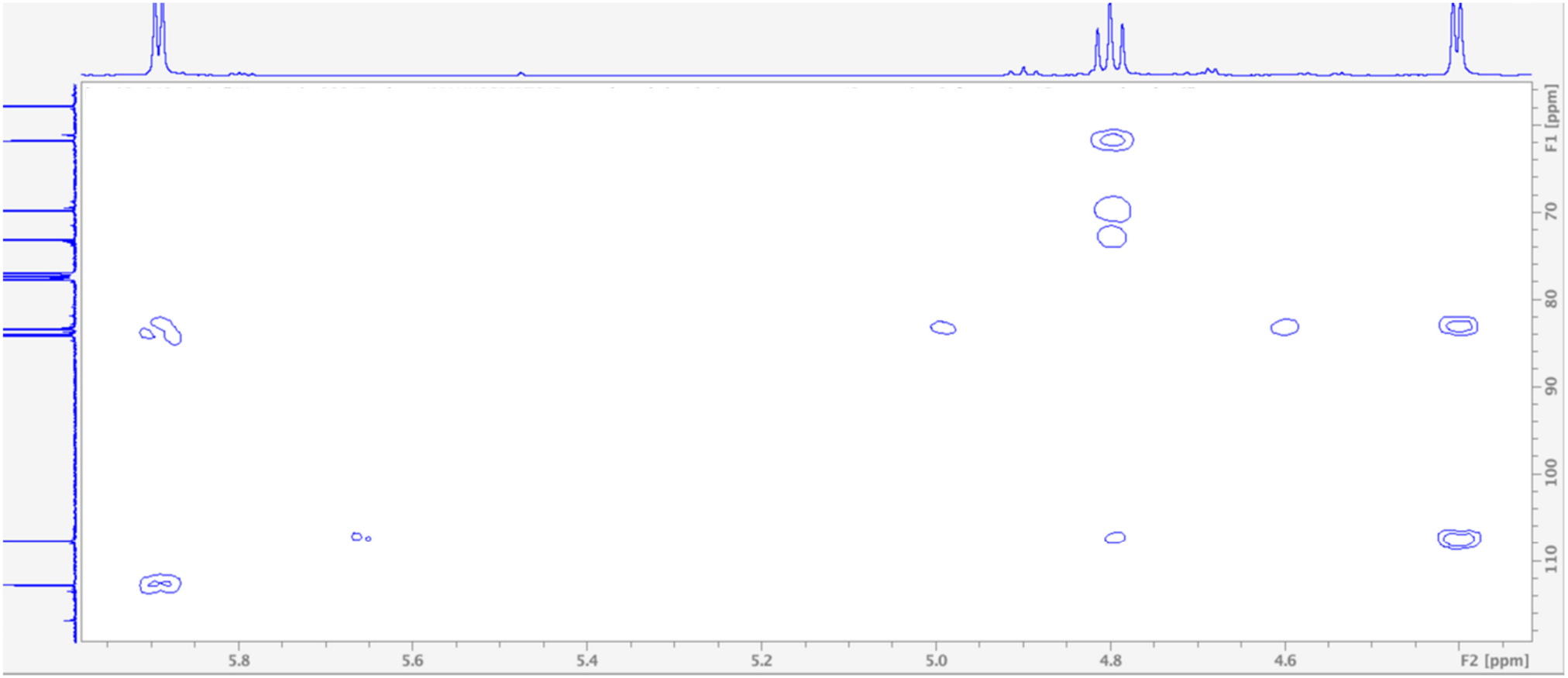

F1 [ppm]
126
128
130
132
134
136
7.8
7.7
7.6
7.5
7.4
7.3
F2 [ppm]


F1 [ppm]
40
60
80
100
5
4
3
2
F2 [ppm]

HMBC




F1 [ppm]
80
100
120
140
8.0
7.5
7.0
6.5
6.0
F2 [ppm]


F1 [ppm]
70
80
90
100
110
5.8
5.6
5.4
5.2
5.0
4.8
4.6
F2 [ppm]

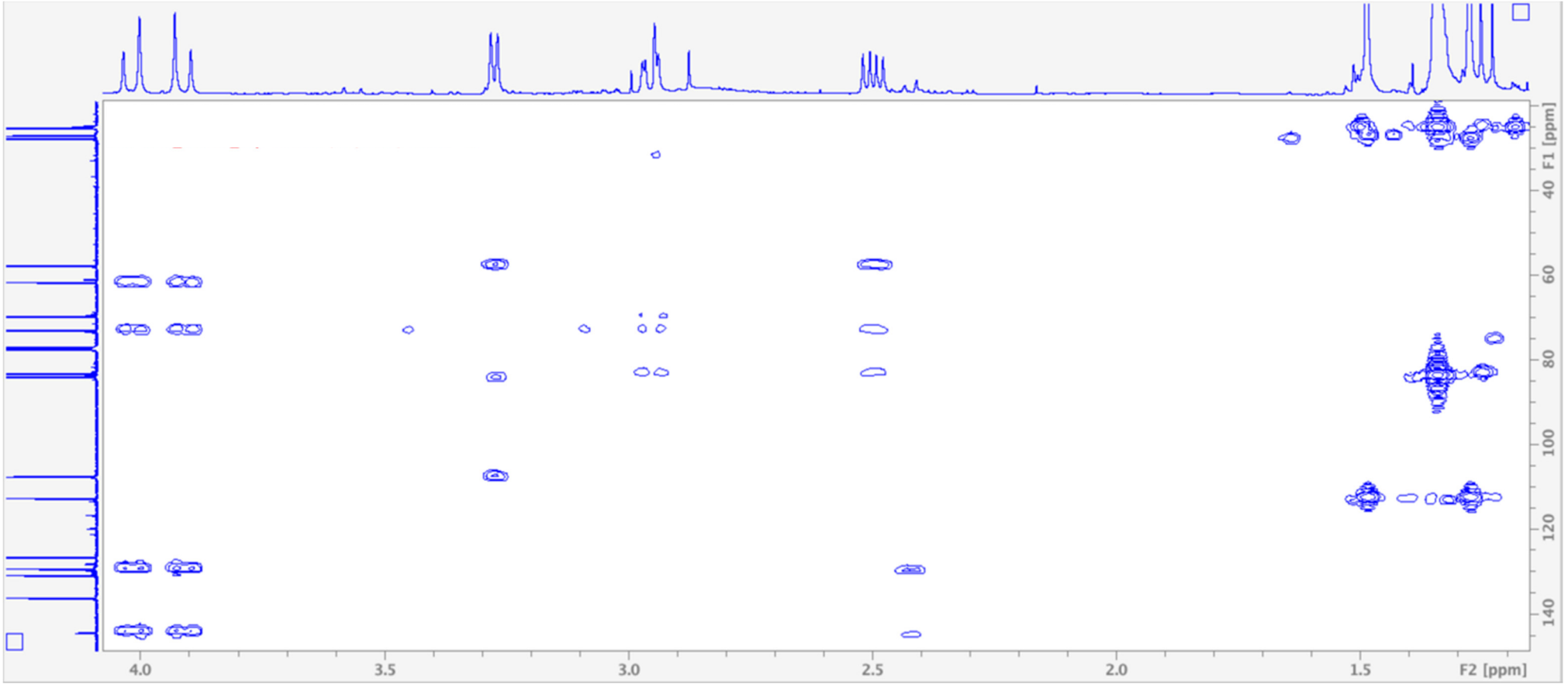
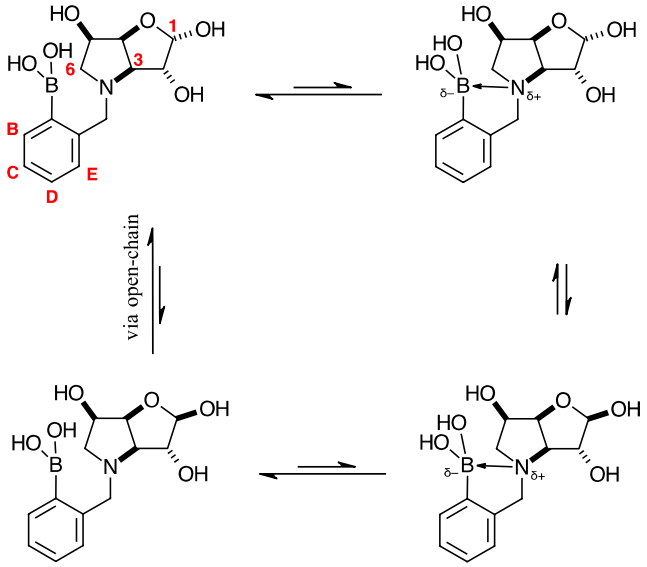

F1 [ppm]
40
60
80
100
120
140
4.0
3.5
3.0
2.5
2.0
1.5
F2 [ppm]

Figure S7. ^{1}H- (400 MHz), ^{13}C-NMR (100 MHz), DEPT, ^{11}B-NMR (128 MHz), COSY, HSQC and HMBC spectra of *N*-(2-methylphenyl boronic acid)-3,6-dideoxy-3,6-imino-D-gulofuranose **ortho 3** in D_2O.



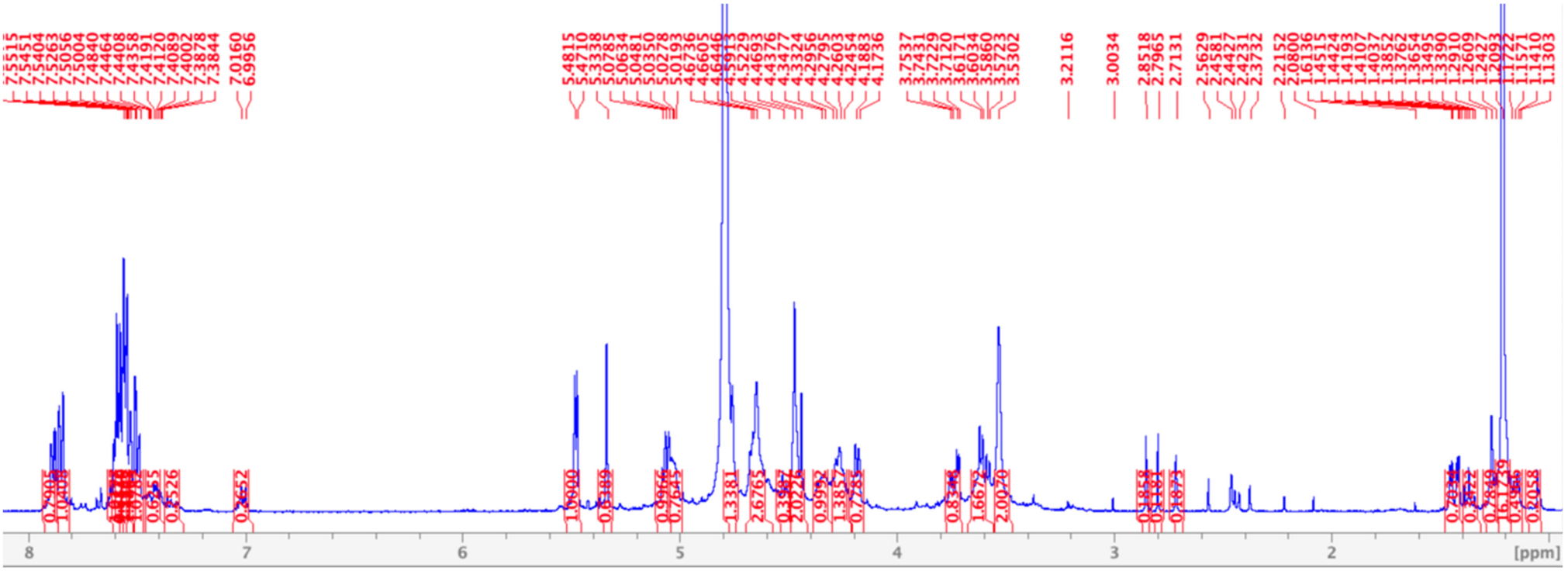
Ratio *α-fur* anomer : *β-fur* anomer, 1.0 : 0.7.

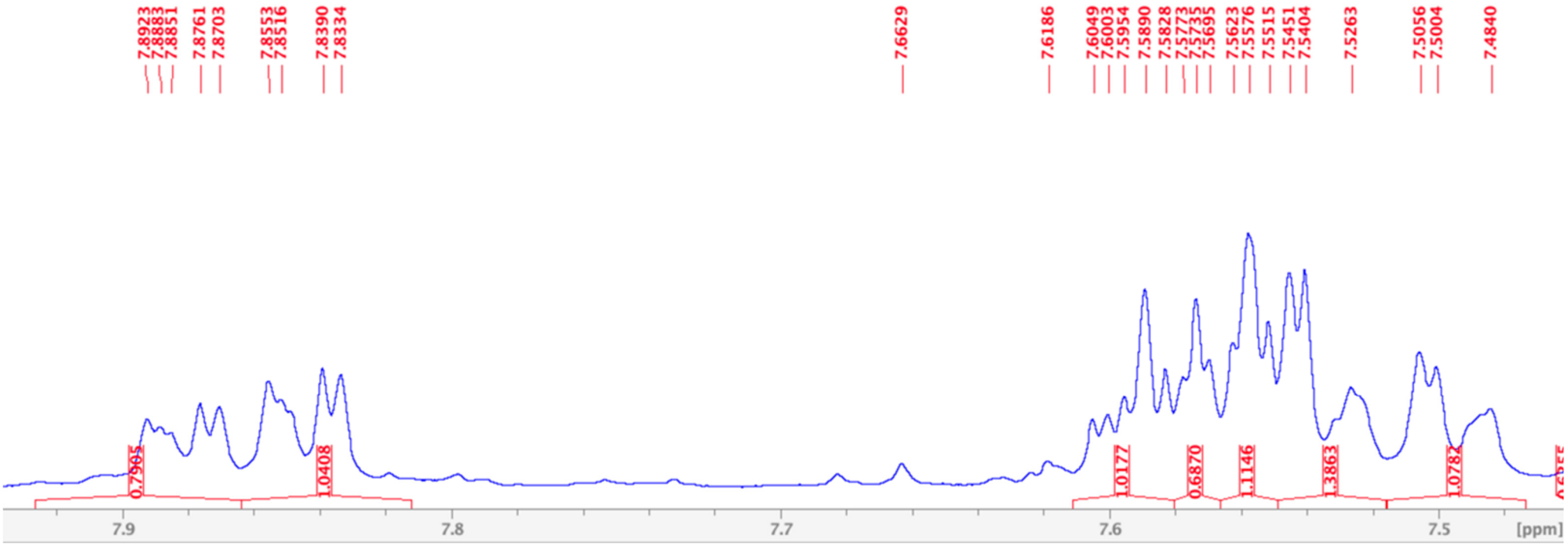
***α-fur* anomer**. **δH**: 7.85 (1H, app-dd, $J_{HA,HB}$ 6.6 Hz, $J_{HA,HC}$ 2.2 Hz, $J_{HA,HD}$ 1.5 Hz, ArH^{A}), 7.58-7.54 (2Hs, m, overlapping, ArH^{B} and ArH^{C}), 7.50 (1H, app-dd, $J_{HD,HC}$ 6.6 Hz, $J_{HD,HB}$ 2.1 Hz, $J_{HD,HA}$ 1.4 Hz, ArH^{D}), 5.48 (1H, d, $J_{H\text{-}1,H\text{-}2}$ 4.3 Hz, H-1), 5.06-4.99 (1H, m, partially obscured, H-4), 4.76 (1H, d, partially obscured by D_2O signal, ArC*H*^{a}H^{b}), 4.49-4.44 (1H, obscured, H-5), 4.45 (1H, d, $J_{Hb,Ha}$ 12.8 Hz, ArCH^{a}*H*^{b}), 4.39-4.28 (1H, m, H-2), 4.31-4.22 (1H, m, H-3), 3.55-3.51 (1Hs, app-s, H-6 and H-6′); **δC**: 135.7 ppm (ArC^{A}), 134.1 (ArC$_{quat}$), 132.1 (ArC^{D}), 131.3, 129.6 (ArC^{B} and ArC^{C}), 99.5 (C-1, broad signal, width 59 Hz), 79.4 (C-4, broad signal, width 65 Hz), 73.4 (C-2), 72.0 (C-3), 67.2 (C-5), 60.7 (ArCH$_2$, broad signal, width 32 Hz), 60.1 (C-6, broad signal, width 105 Hz); ArC$_{quat}$-B was not discernible.

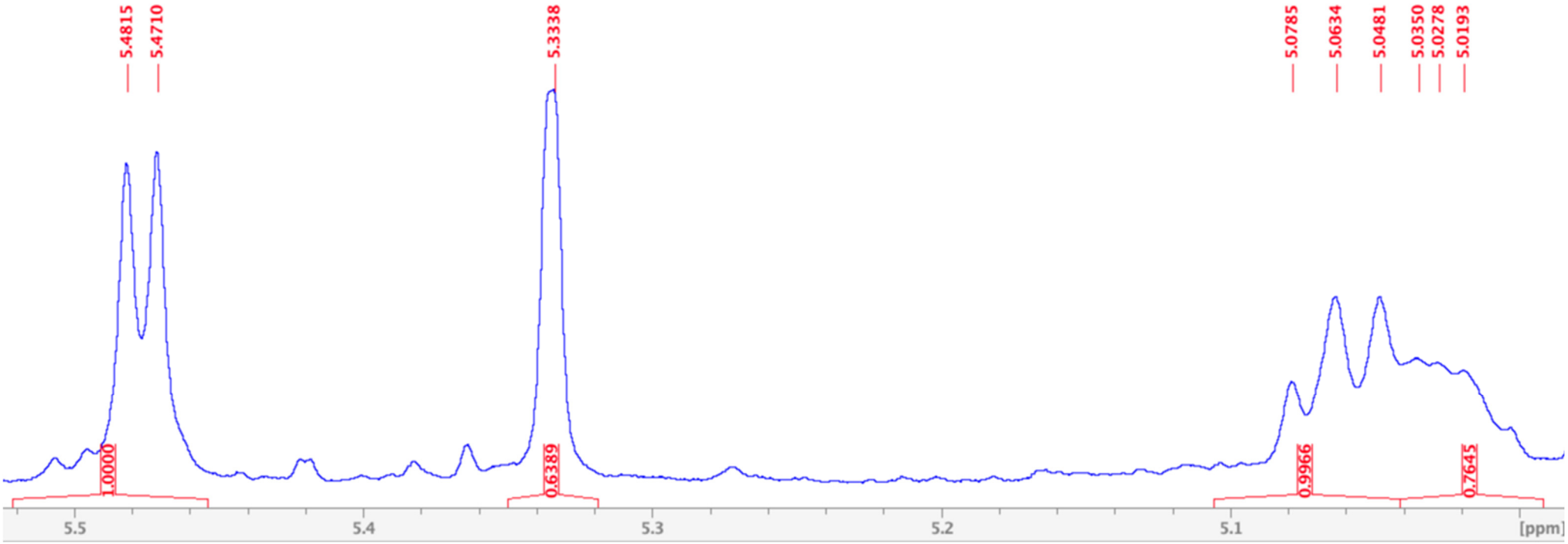
β-fur anomer. **δH**: 7.88 (1H, app-dd, $J_{HA,HB}$ 6.4 Hz, $J_{HA,HC}$ 2.4 Hz, $J_{HA,HD}$ 1.6 Hz, ArH^{A}), 7.61-7.56 (2Hs, m, obscured, ArH^{B} and ArH^{C}), 7.54 (1H, app-dd, $J_{HD,HC}$ 6.8 Hz, $J_{HD,HB/HA}$ 1.9 Hz, ArH^{D}), 5.33 (1H, s, H-1), 5.06 (1H, app-t, partially obscured, $J_{H\text{-}4,H\text{-}3}$ 6.1 Hz, H-4), 4.69-4.60 (3H, m, ArC*H*^{a}H^{b}, ArCH^{a}*H*^{b} and H-5), 4.18 (1H, d, *J* 6.4 Hz, H-3), 3.73 (1H, dd, $J_{H\text{-}6,H\text{-}6'}$ 12.4, $J_{H\text{-}6,H\text{-}5}$ 4.5 Hz, H-6), 3.65-3.58 (1H, obscured, H-2), 3.59 (1H, dd, $J_{H\text{-}6',H\text{-}6}$ 12.4, $J_{H\text{-}6',H\text{-}5}$ 5.6 Hz, H-6′); **δC**: 135.9 (ArC^{A}), 134.3 (ArC$_{quat}$), 132.2 (ArC^{D}), 131.5, 129.8 (ArC^{B} and ArC^{C}), 102.9 (C-1), 81.8 (C-4, slightly broadened, width 29 Hz), 75.6 (C-2), 73.4 (C-3, slightly broadened, width 27 Hz), 67.4 (C-5), 60.1 (C-6, broad, 105 Hz), 60.1 (ArCH$_2$, broad, width 105 Hz); ArC$_{quat}$-B was not discernible.

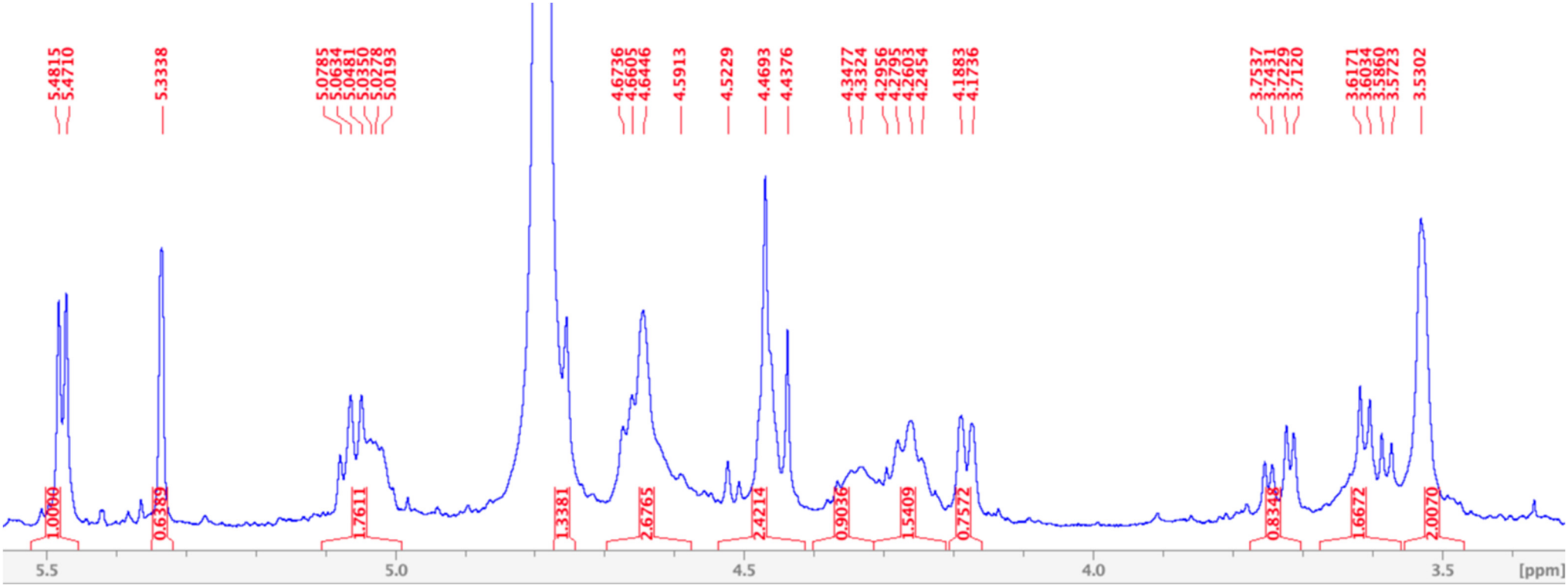
δB: 28.0 (broad, integration: 4.0), 19.3 (sharp, integration: 1.0).

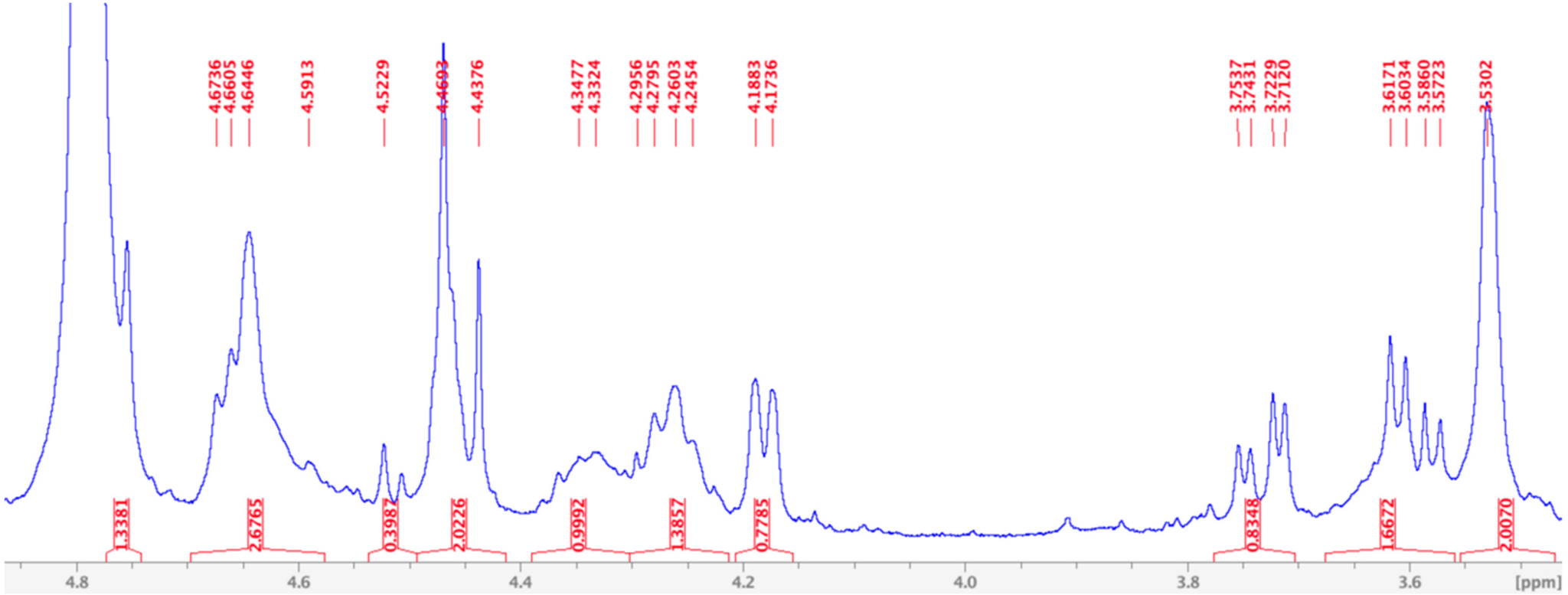
^{1}H-NMR



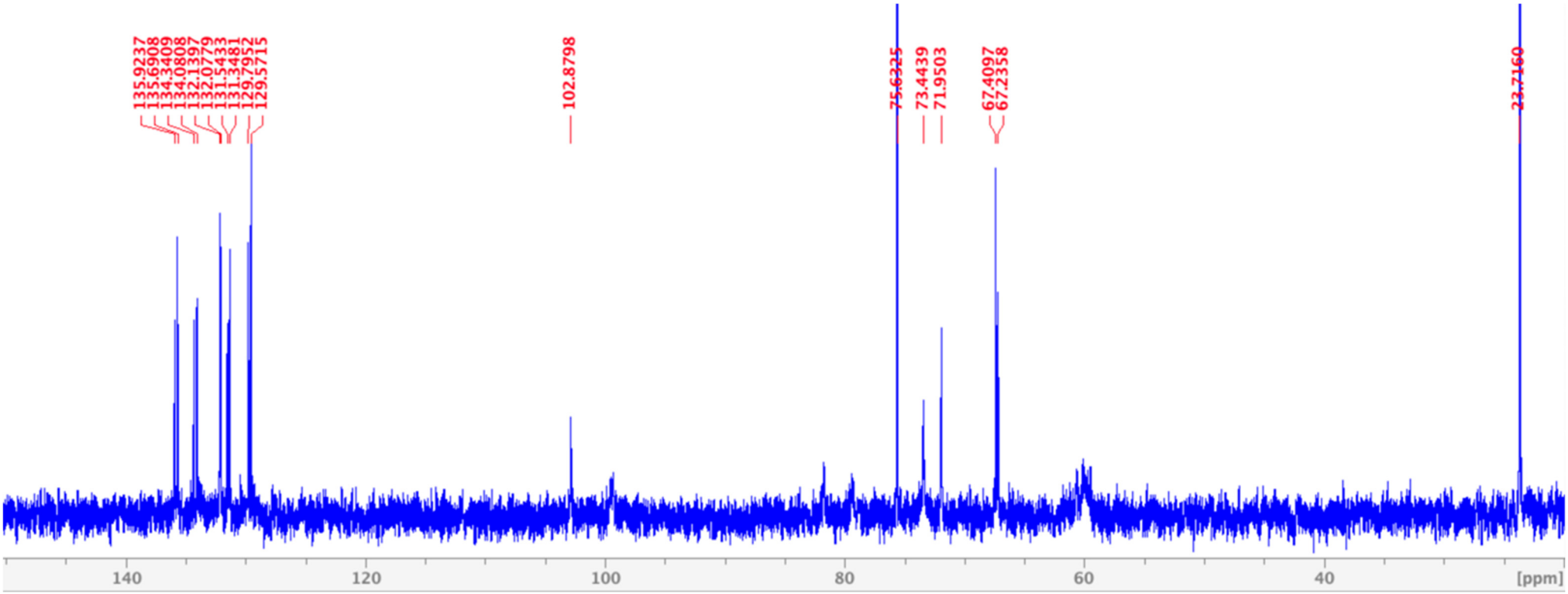

7.8923
7.8883
7.8851
7.8761
7.8703
7.8553
7.8516
7.8390
7.8334
7.6629
7.6186
7.6049
7.6003
7.5954
7.5890
7.5828
7.5773
7.5735
7.5695
7.5623
7.5576
7.5515
7.5451
7.5404
7.5263
7.5056
7.5004
7.4840
0.7905
1.0408
1.0177
0.6870
1.1146
1.3863
1.0782
7.9
7.8
7.7
7.6
7.5
[ppm]

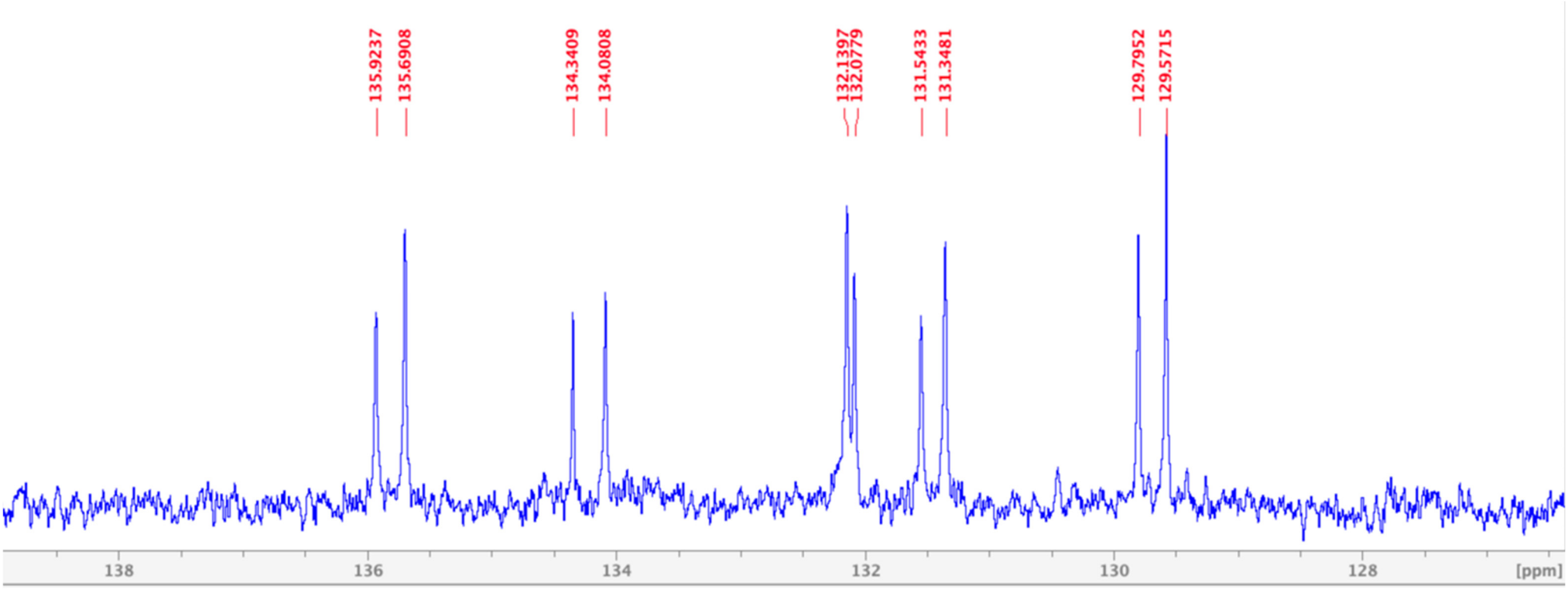

5.4815
5.4710
5.3338
5.0785
5.0634
5.0481
5.0350
5.0278
5.0193
1.0000
0.6389
0.9966
0.7645
5.5
5.4
5.3
5.2
5.1
[ppm]

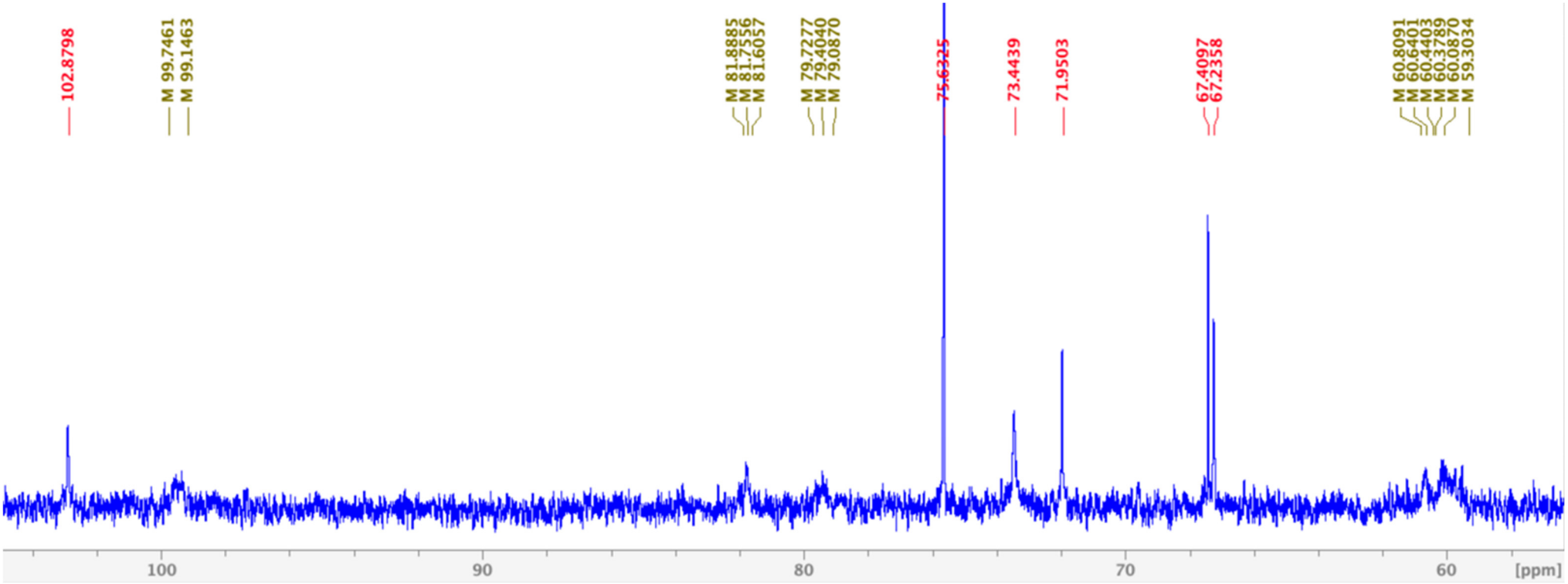

5.4815
5.4710
5.3338
5.0785
5.0634
5.0481
5.0350
5.0278
5.0193
4.6736
4.6605
4.6446
4.5913
4.5229
4.4693
4.4376
4.3477
4.3324
4.2956
4.2795
4.2603
4.2454
4.1883
4.1736
3.7537
3.7431
3.7229
3.7120
3.6171
3.6034
3.5860
3.5723
3.5302
1.0000
0.6389
1.7611
1.3381
2.6765
2.4214
0.9036
1.5409
0.7572
0.8348
1.6672
2.0070
5.5
5.0
4.5
4.0
3.5
[ppm]

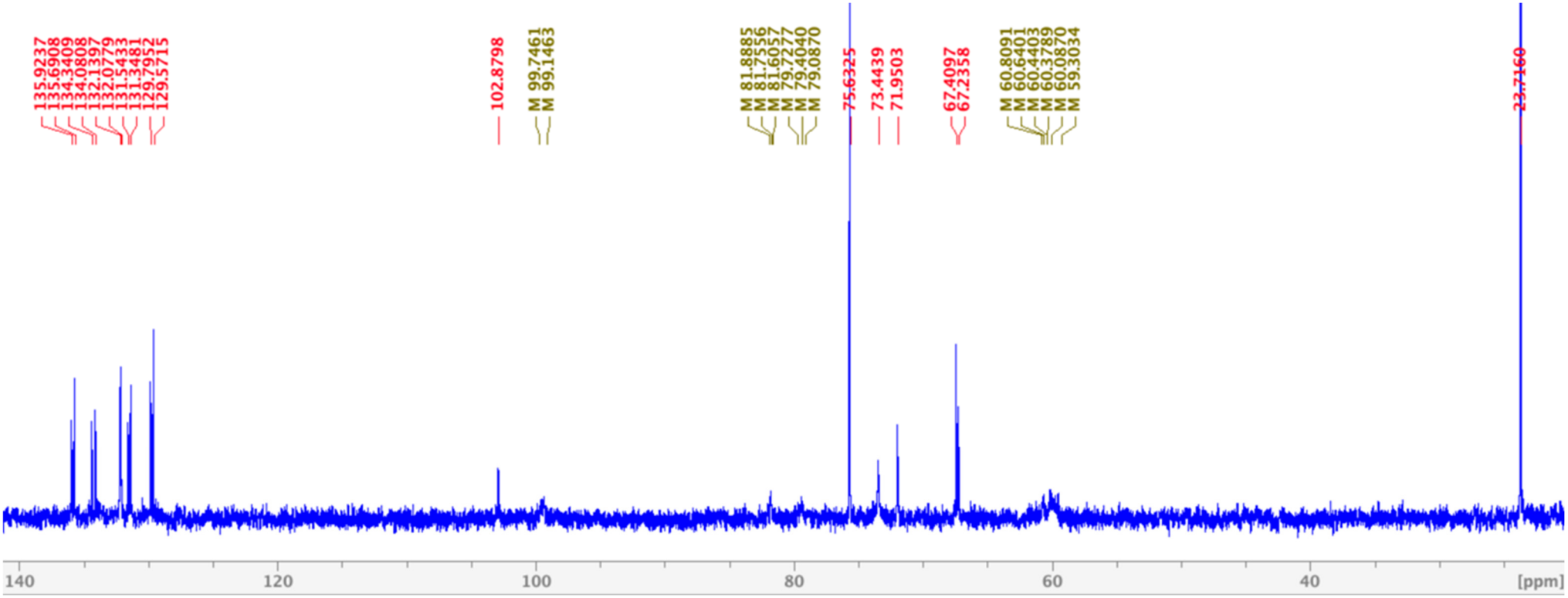

4.6736
4.6605
4.6446
4.5913
4.5229
4.4693
4.4376
4.3477
4.3324
4.2956
4.2795
4.2603
4.2454
4.1883
4.1736
3.7537
3.7431
3.7229
3.7120
3.6171
3.6034
3.5860
3.5723
3.5302
1.3381
2.6765
0.3987
2.0226
0.9992
1.3857
0.7785
0.8348
1.6672
2.0070
4.8
4.6
4.4
4.2
4.0
3.8
3.6
[ppm]

^{13}C-NMR

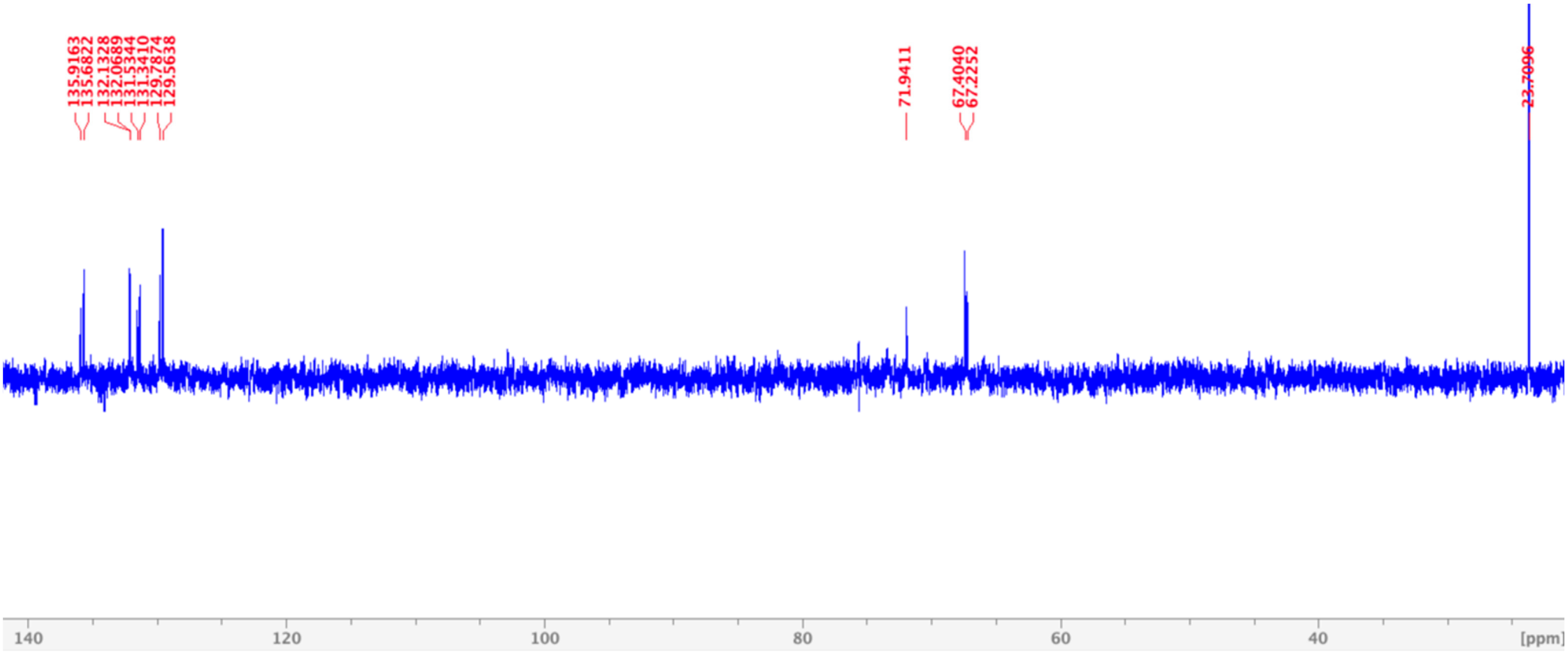

135.9237
135.6908
134.3409
134.0808
132.1397
132.0779
131.5433
131.3481
129.7952
129.5715
102.8798
75.6325
73.4439
71.9503
67.4097
67.2358
23.7160
140
120
100
80
60
40
[ppm]


135.9237
135.6908
134.3409
134.0808
132.1397
132.0779
131.5433
131.3481
129.7952
129.5715
138
136
134
132
130
128
[ppm]

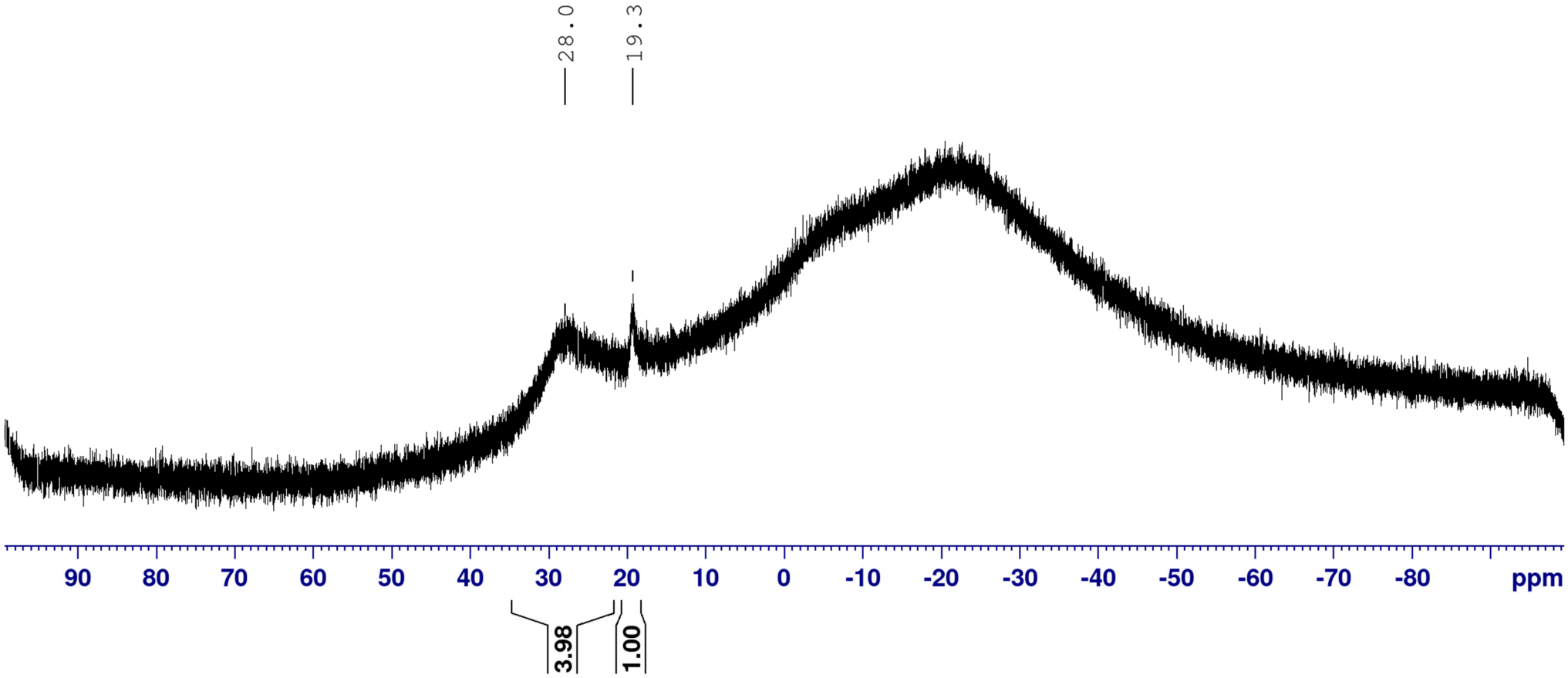

102.8798
M 99.7461
M 99.1463
M 81.8885
M 81.7556
M 81.6057
M 79.7277
M 79.4040
M 79.0870
75.6325
73.4439
71.9503
67.4097
67.2358
M 60.8091
M 60.6401
M 60.4403
M 60.3789
M 60.0870
M 59.3034
100
90
80
70
60
[ppm]


135.9237
135.6908
134.3409
134.0808
132.1397
132.0779
131.5433
131.3481
129.7952
129.5715
102.8798
M 99.7461
M 99.1463
M 81.8885
M 81.7556
M 81.6057
M 79.7277
M 79.4040
M 79.0870
75.6325
73.4439
71.9503
67.4097
67.2358
M 60.8091
M 60.6401
M 60.4403
M 60.3789
M 60.0870
M 59.3034
23.7160
140
120
100
80
60
40
[ppm]

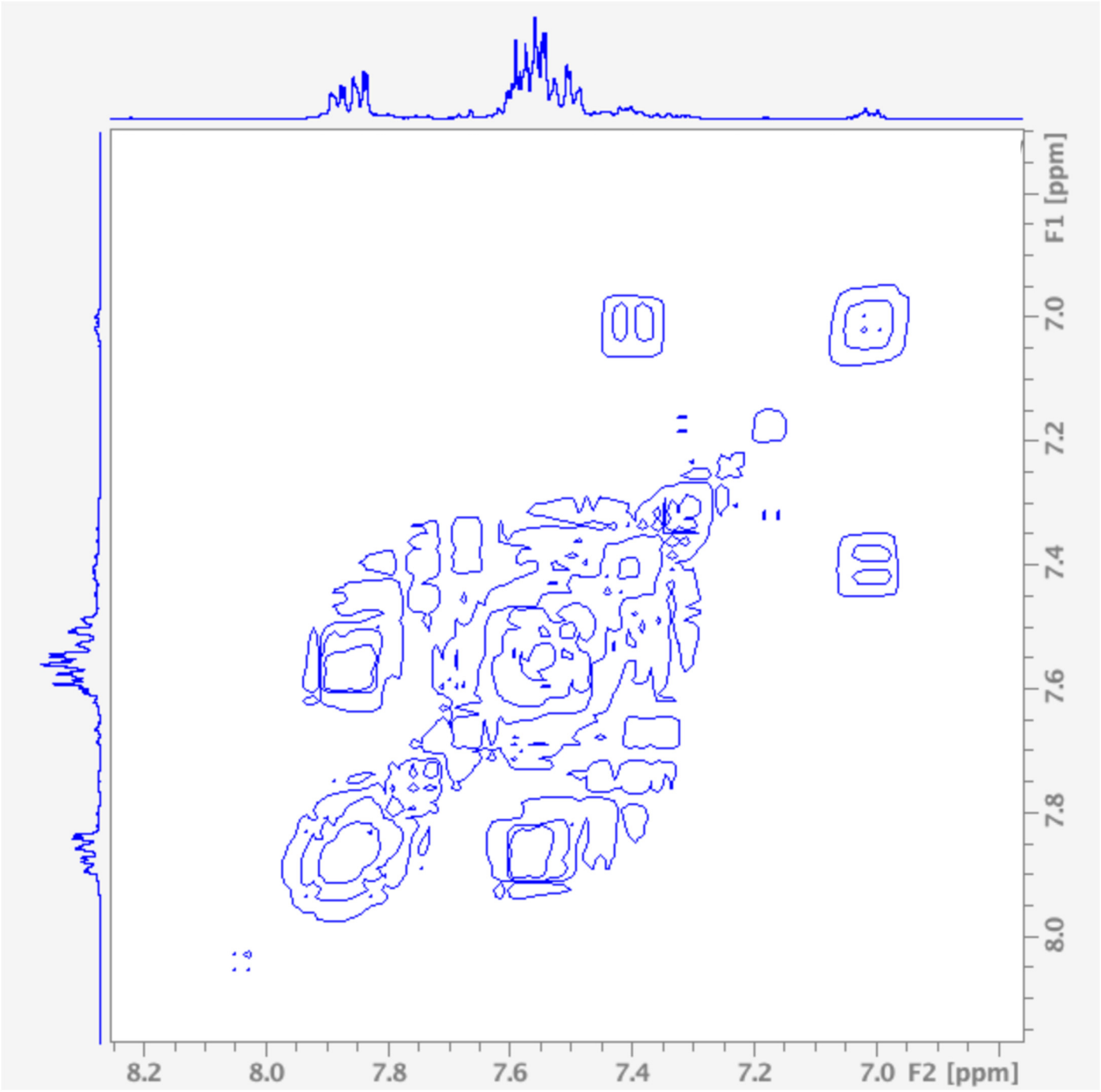
DEPT

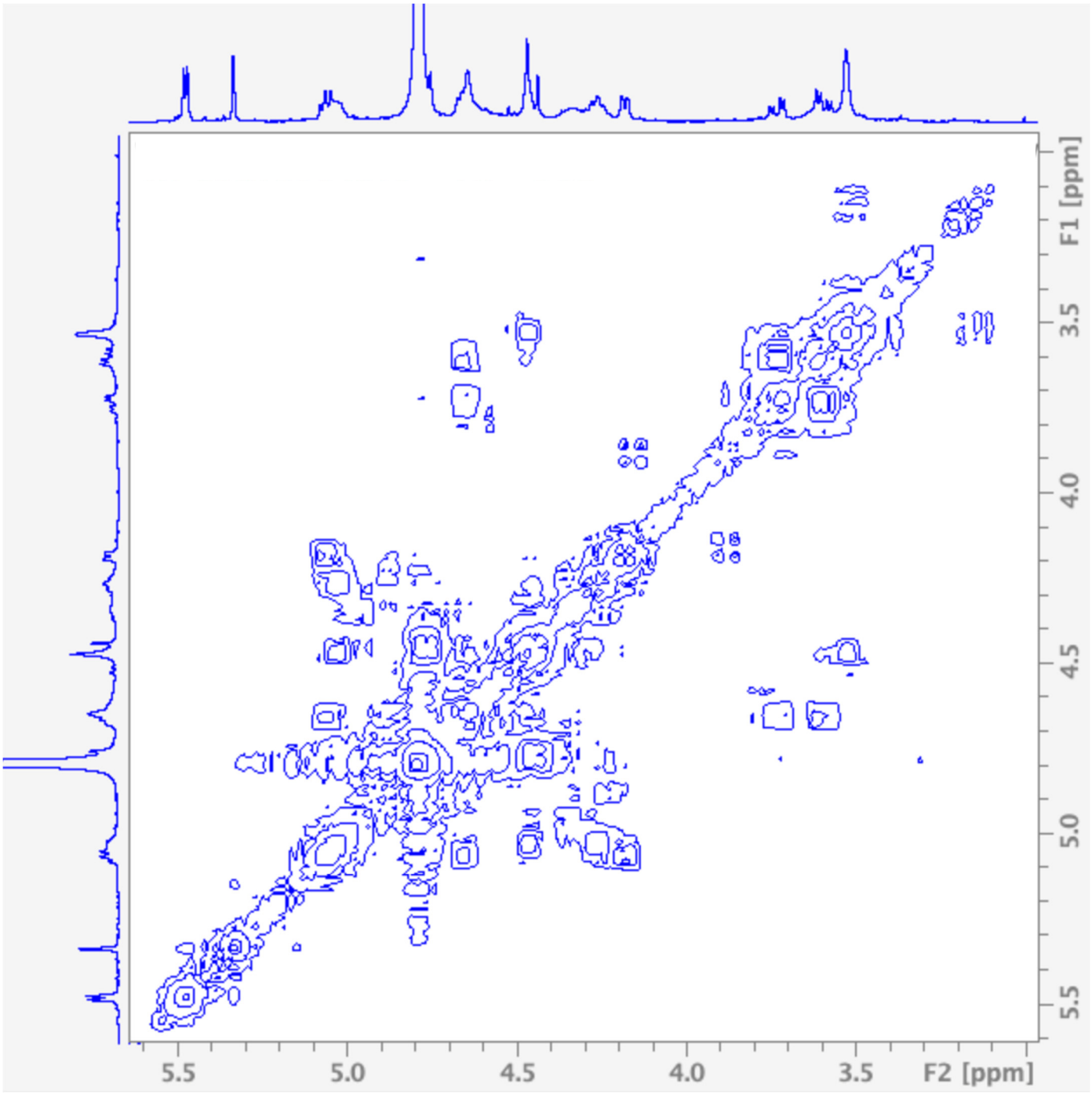

135.9163
135.6822
132.1328
132.0689
131.5344
131.3410
129.7874
129.5638
71.9411
67.4040
67.2252
23.7096
140
120
100
80
60
40
[ppm]

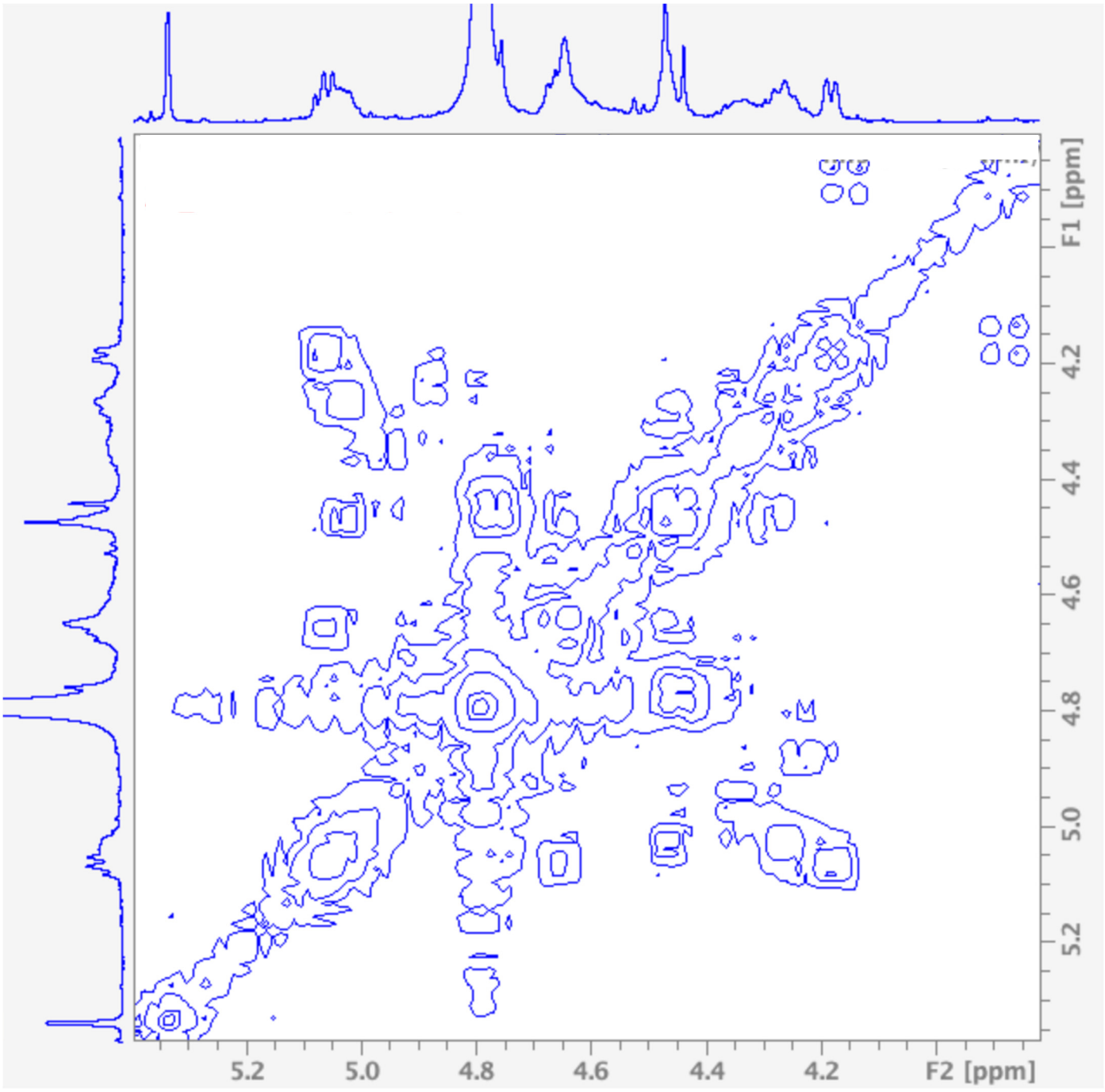
^{11}B-NMR


28.0
19.3
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
ppm
3.98
1.00

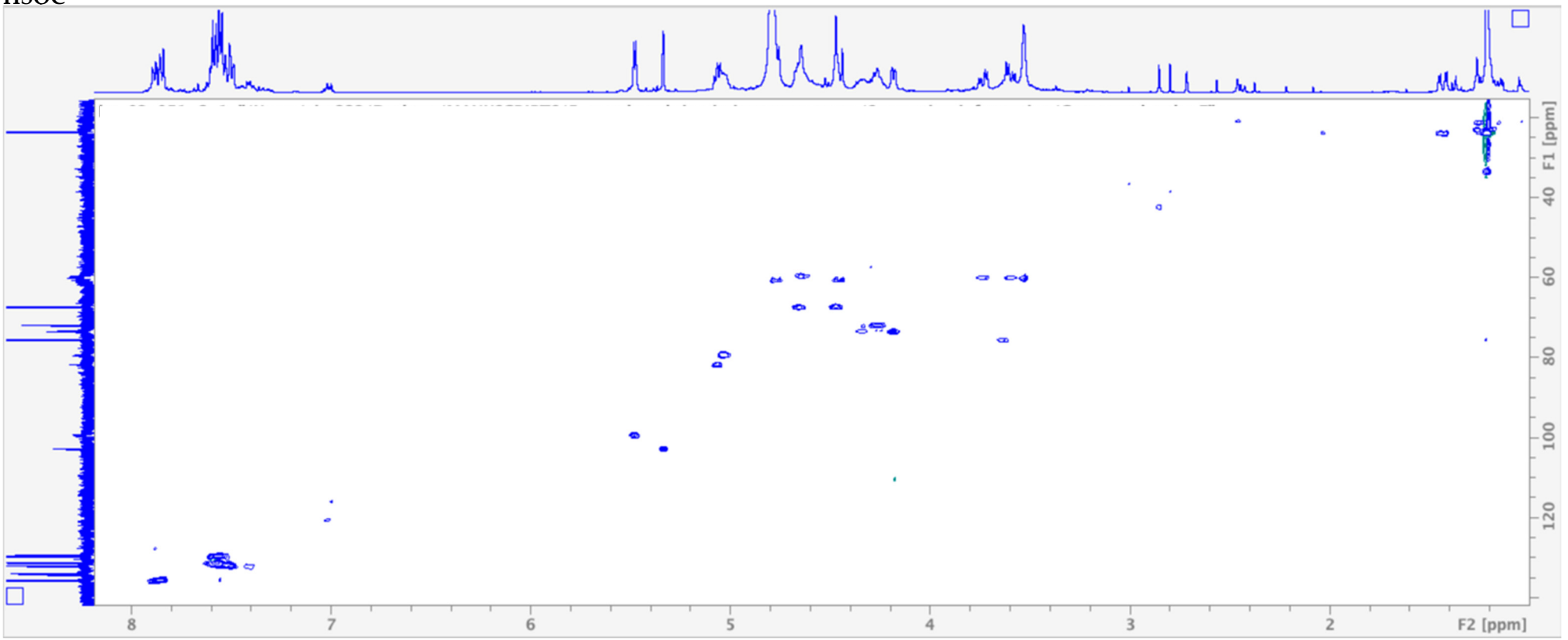
COSY

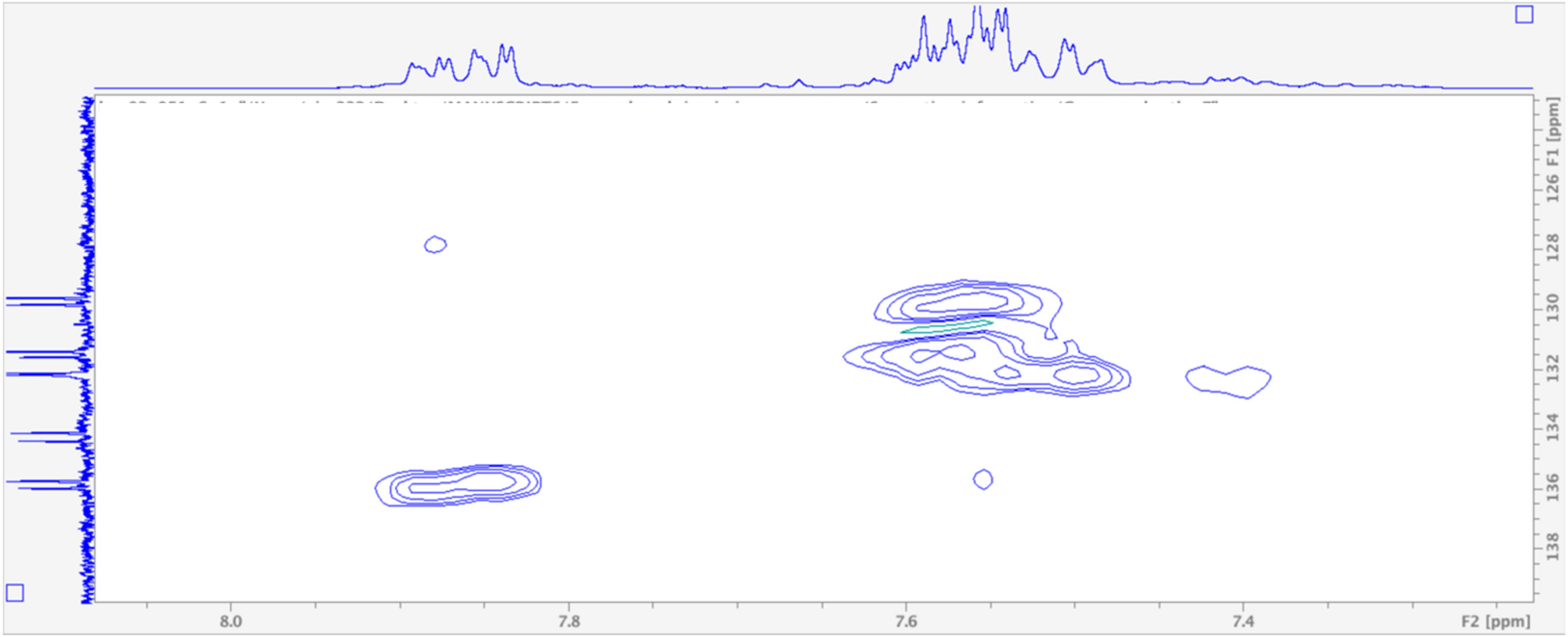


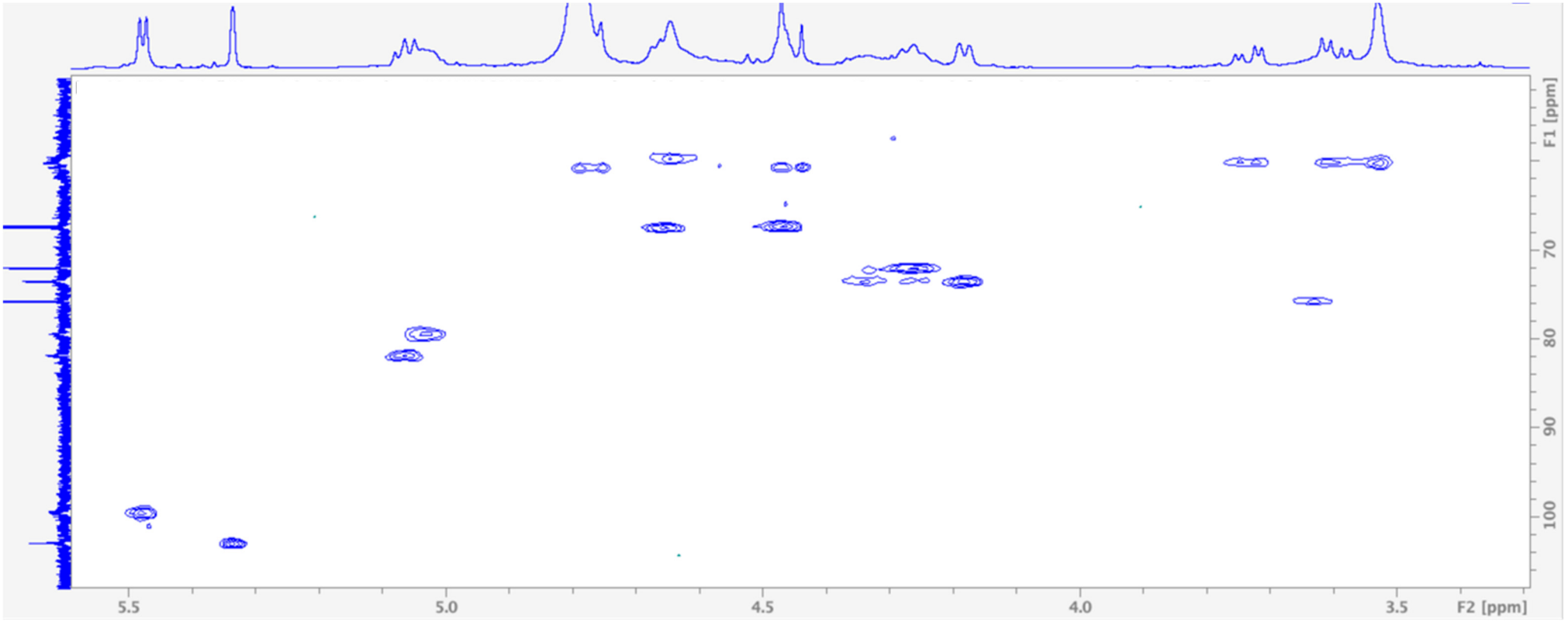

F1 [ppm]
3.5
4.0
4.5
5.0
5.5
5.5
5.0
4.5
4.0
3.5
F2 [ppm]

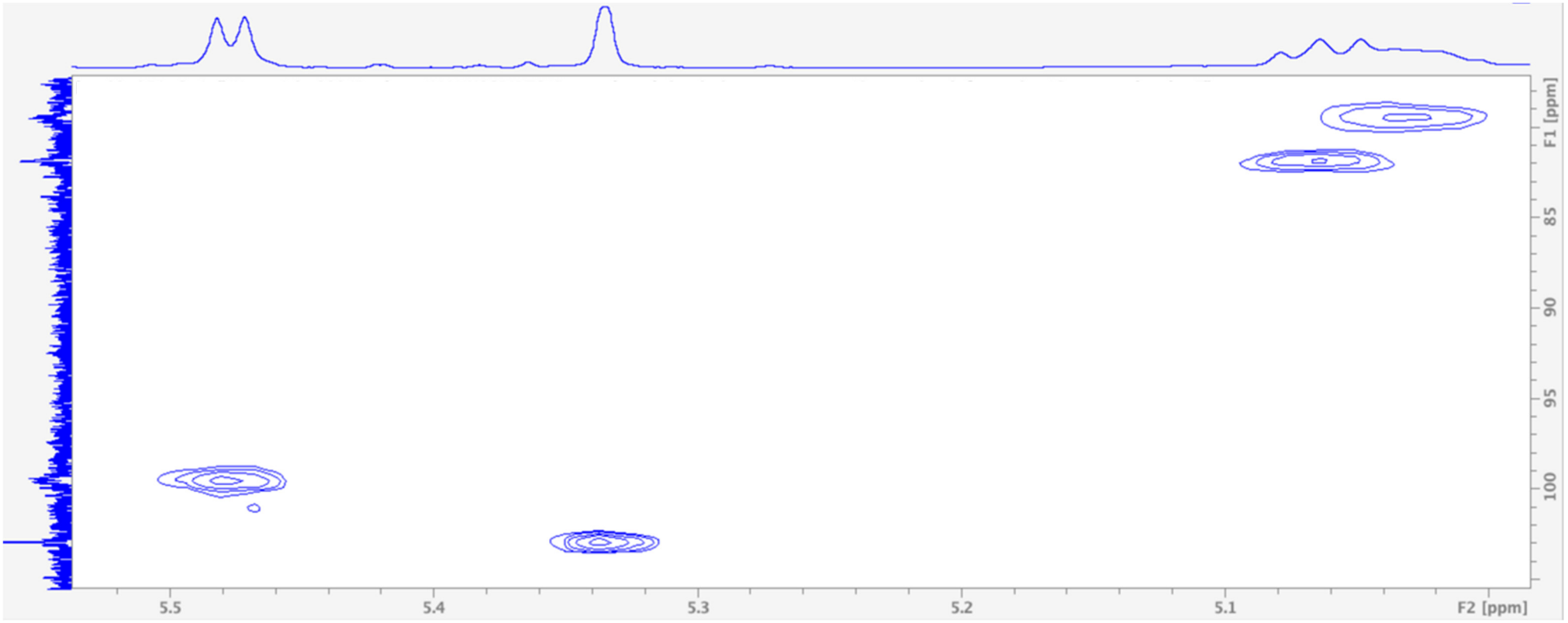

F1 [ppm]
5.2
5.0
4.8
4.6
4.4
4.2
F2 [ppm]

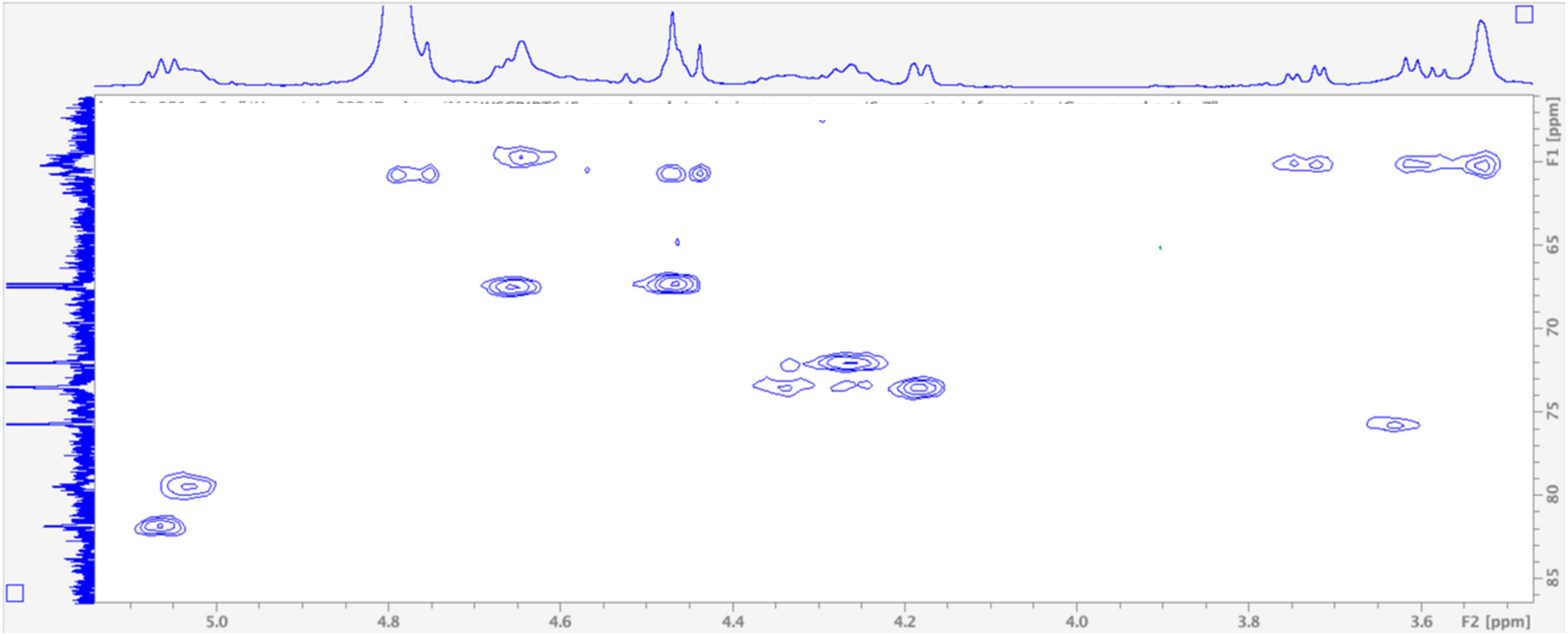
HSOC

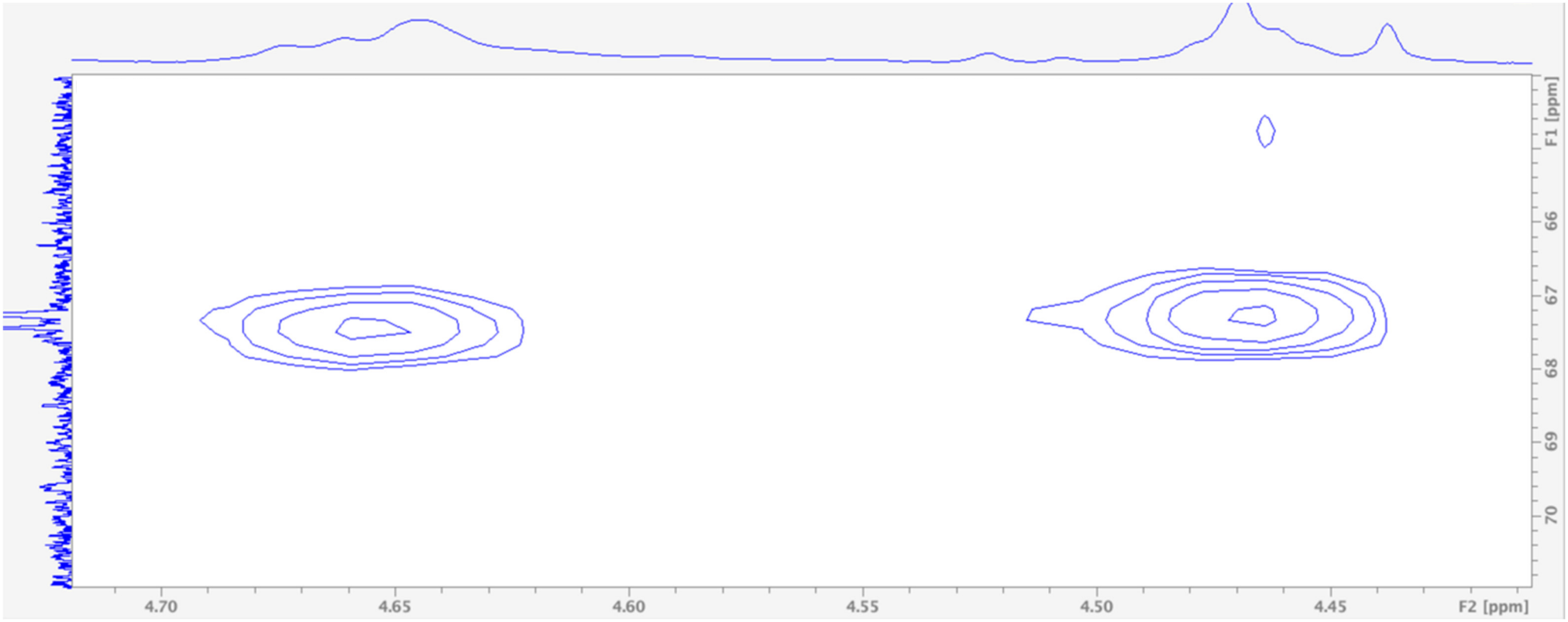



8.0
7.8
7.6
7.4
F2 [ppm]
126
128
130
132
134
136
138
F1 [ppm]

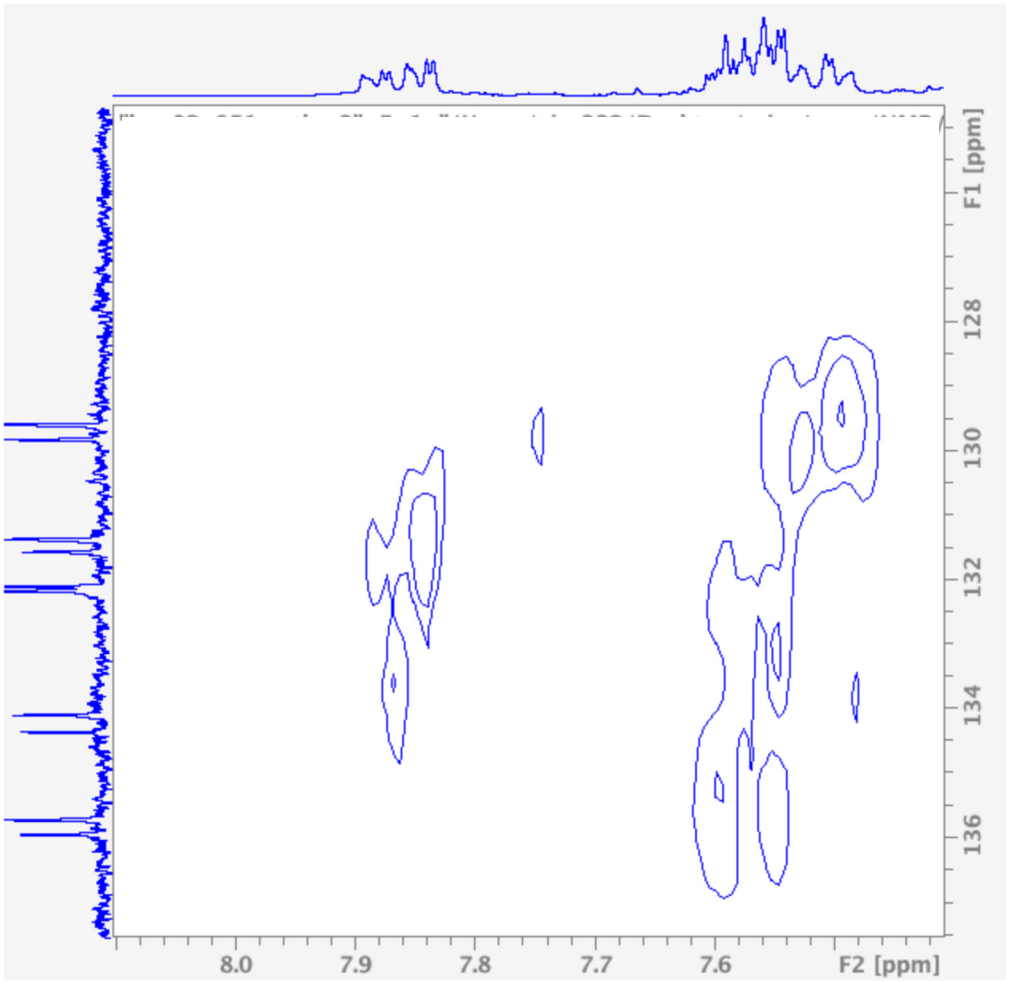

F1 [ppm]
70
80
90
100
5.5
5.0
4.5
4.0
3.5
F2 [ppm]

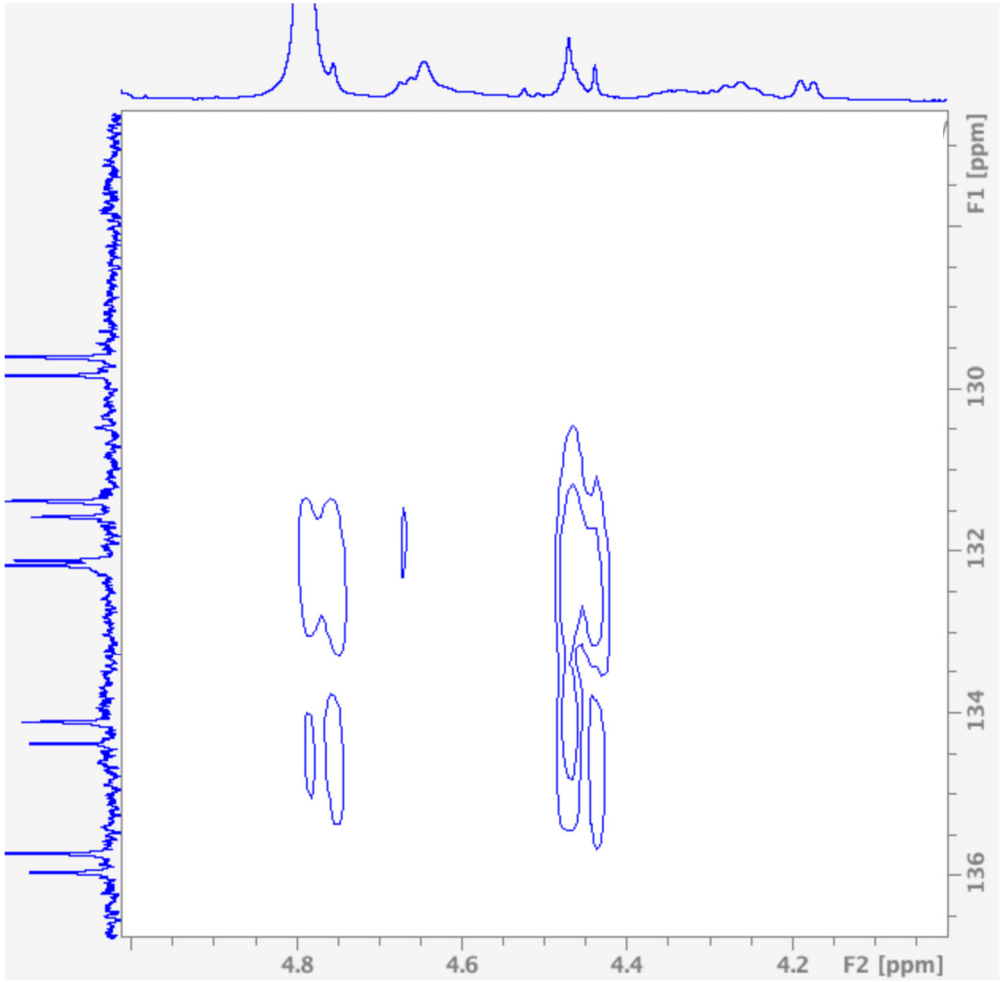

F1 [ppm]
85
90
95
100
5.5
5.4
5.3
5.2
5.1
F2 [ppm]


F1 [ppm]
65
70
75
80
85
5.0
4.8
4.6
4.4
4.2
4.0
3.8
3.6
F2 [ppm]


F1 [ppm]
66
67
68
69
70
4.70
4.65
4.60
4.55
4.50
4.45
F2 [ppm]

HMBC


F1 [ppm]
128
130
132
134
136
8.0
7.9
7.8
7.7
7.6
F2 [ppm]


F1 [ppm]
130
132
134
136
4.8
4.6
4.4
4.2
F2 [ppm]

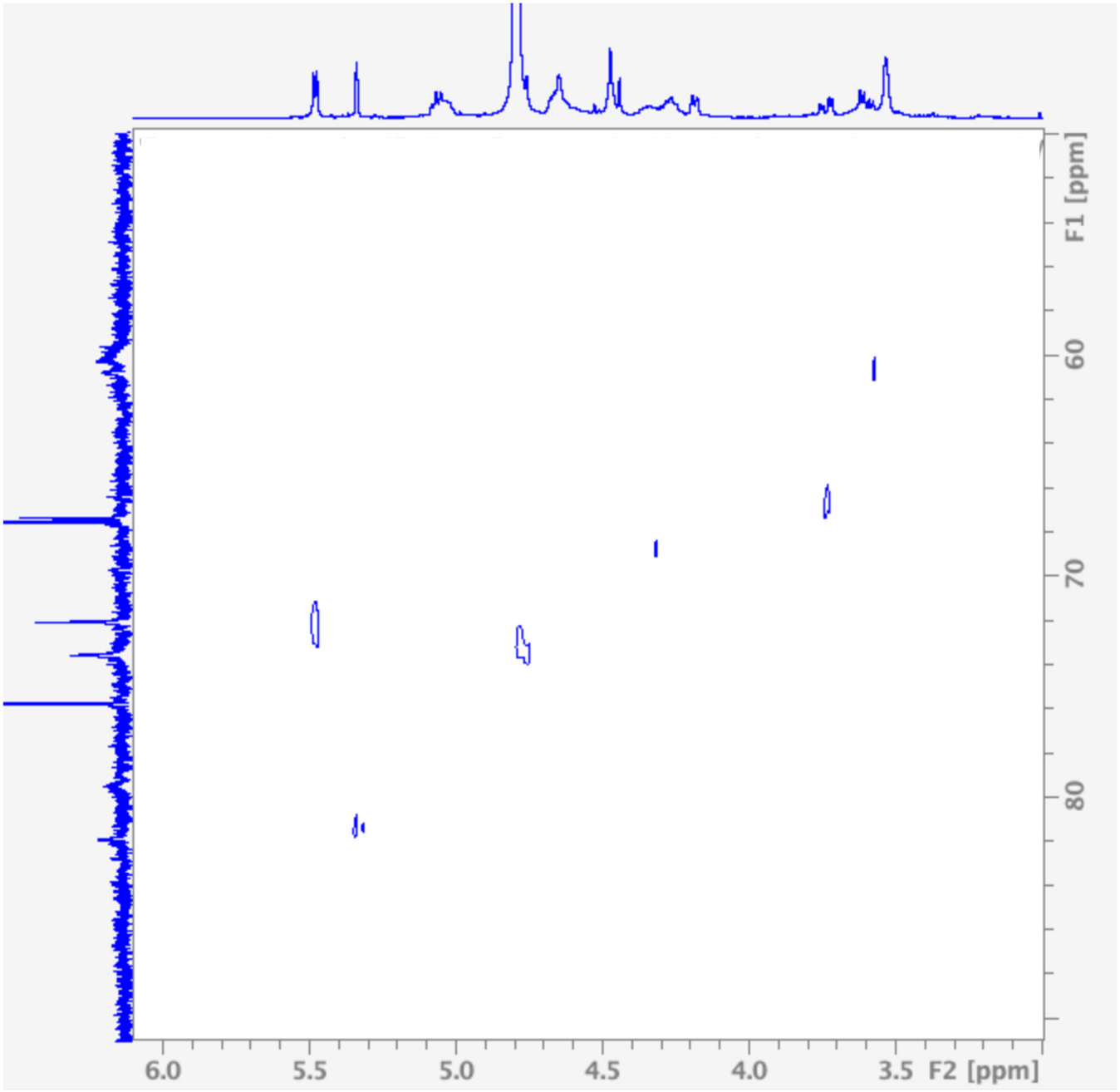
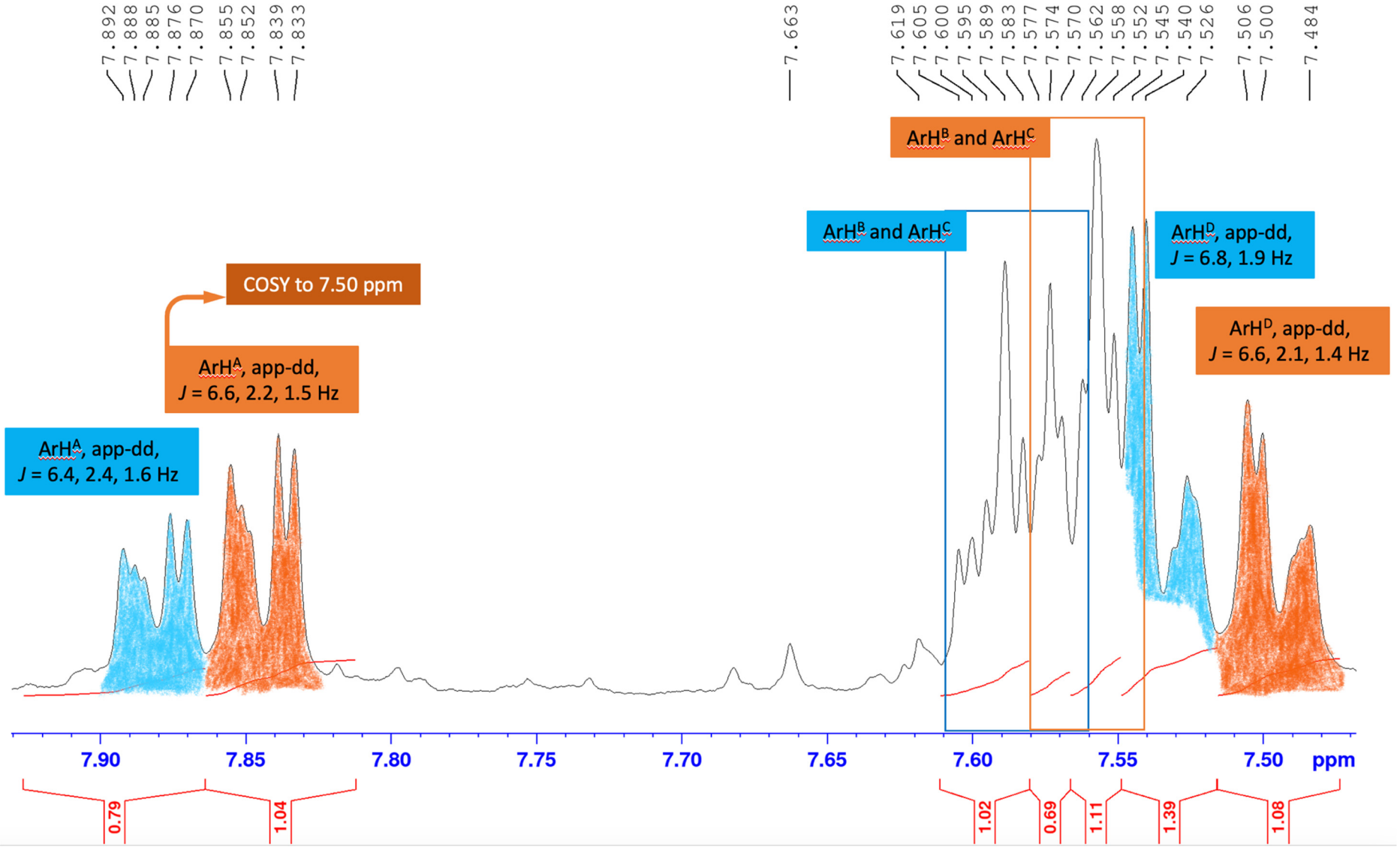
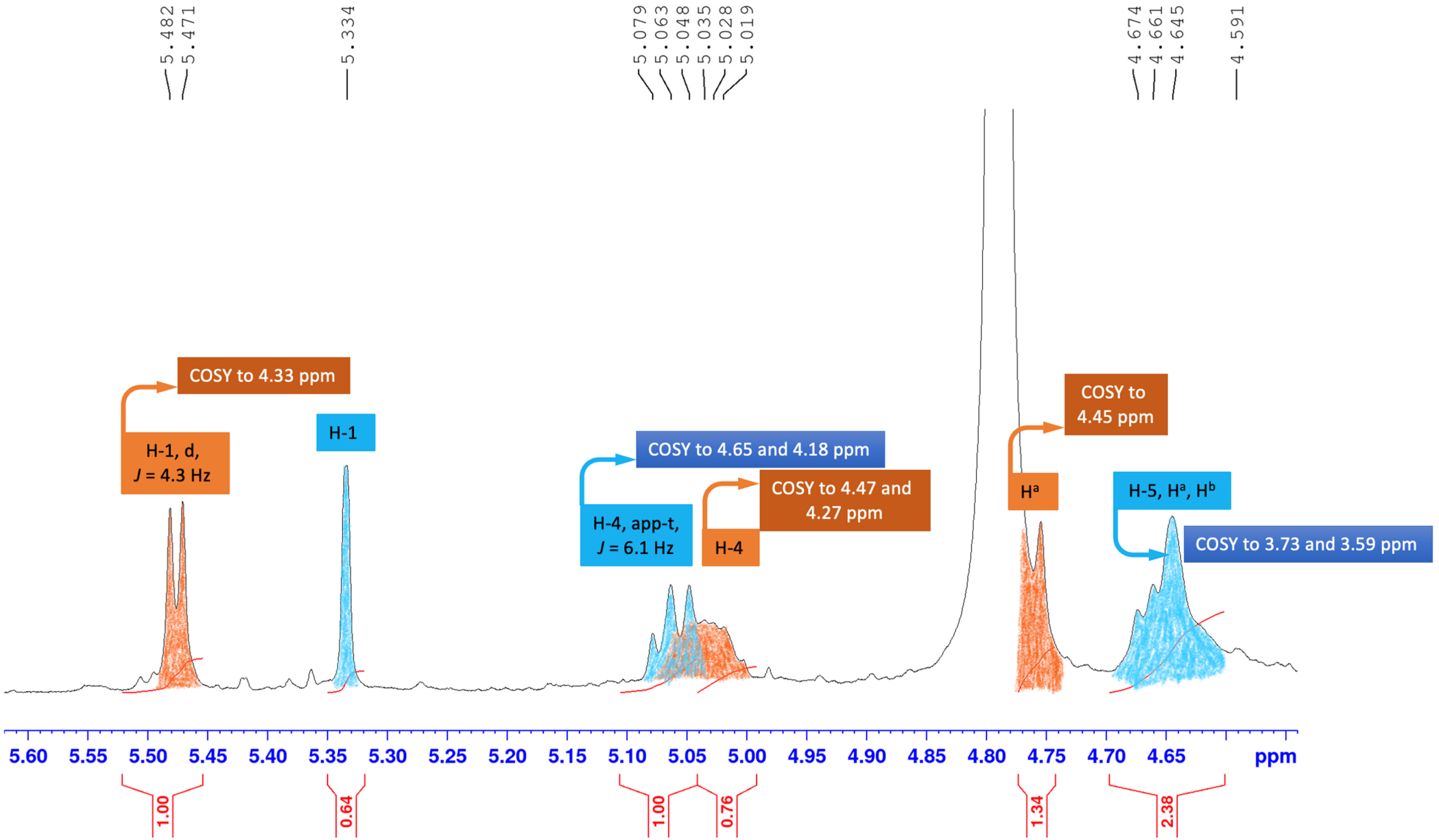

F1 [ppm]
60
70
80
6.0
5.5
5.0
4.5
4.0
3.5
F2 [ppm]

Figure S8. ^{1}H-NMR spectrum (400 MHz, D_2O) of intermediate **ortho 3** with colour-coded signals, highlighting the furanose anomeric forms they belong to, with interpretation of the isolated signals and tentative interpretation of the overlapping ones. Namely, the orange designates the *α-fur* form and indigo designates the *β-fur* form. A) section 7.93 ppm to 7.47 ppm; B) section 5.57 ppm to 4.54 ppm; C) section 4.54 ppm to 3.40 ppm. Highlighted are also the principal COSY correlations to hydrogen atoms within the same spin systems.

A



B


5.482
5.471
5.334
5.079
5.063
5.048
5.035
5.028
5.019
4.674
4.661
4.645
4.591
COSY to 4.33 ppm
H-1, d,
J = 4.3 Hz
H-1
COSY to 4.65 and 4.18 ppm
H-4, app-t,
J = 6.1 Hz
COSY to 4.47 and
4.27 ppm
H-4
COSY to
4.45 ppm
Ha
H-5, Ha, Hb
COSY to 3.73 and 3.59 ppm
5.60 5.55 5.50 5.45 5.40 5.35 5.30 5.25 5.20 5.15 5.10 5.05 5.00 4.95 4.90 4.85 4.80 4.75 4.70 4.65 ppm
1.00
0.64
1.00
0.76
1.34
2.38

C

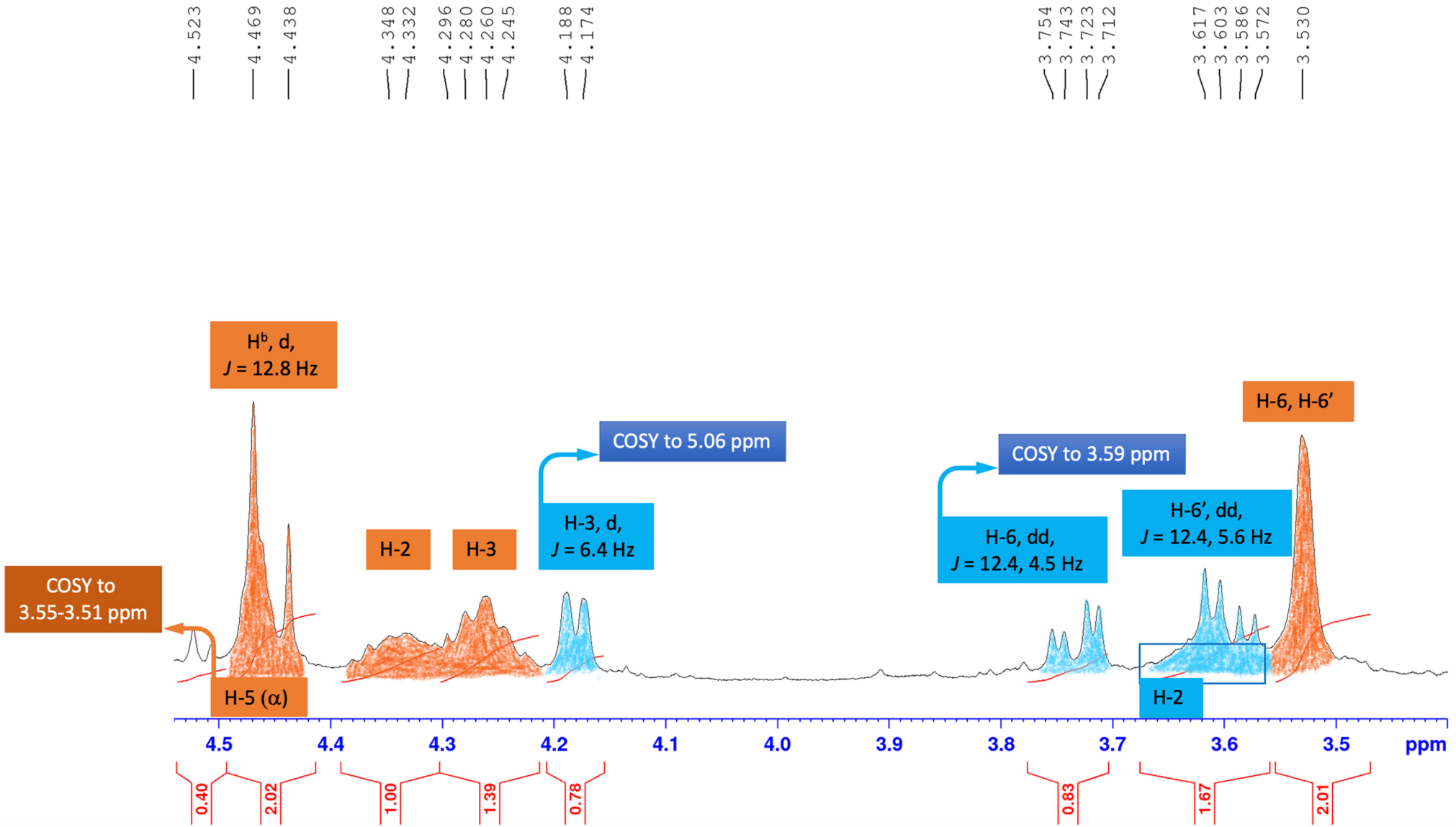
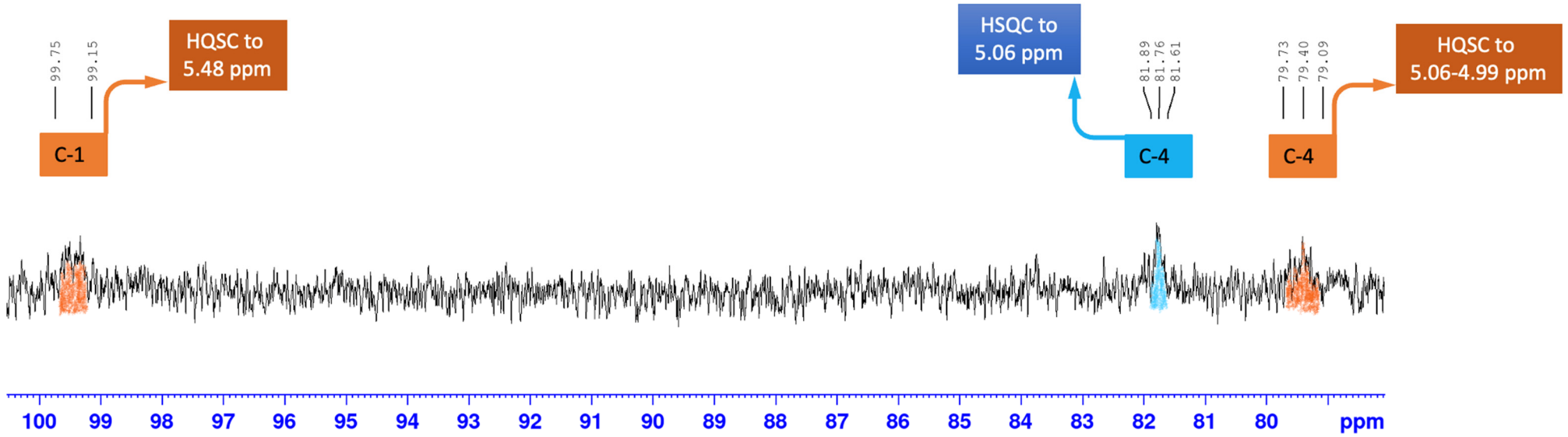

4.523
4.469
4.438
4.348
4.332
4.296
4.280
4.260
4.245
4.188
4.174
3.754
3.743
3.723
3.712
3.617
3.603
3.586
3.572
3.530
Hᵇ, d,
J = 12.8 Hz
COSY to 5.06 ppm
H-2
H-3
H-3, d,
J = 6.4 Hz
COSY to 3.59 ppm
H-6, H-6'
H-6, dd,
J = 12.4, 4.5 Hz
H-6', dd,
J = 12.4, 5.6 Hz
COSY to
3.55-3.51 ppm
H-5 (α)
H-2
4.5
4.4
4.3
4.2
4.1
4.0
3.9
3.8
3.7
3.6
3.5
ppm
0.40
2.02
1.00
1.39
0.78
0.83
1.67
2.01

Figure S9. ^{13}C-NMR spectrum (100 MHz, D_2O) sections of intermediate **ortho 3** with colour-coded signals, highlighting the furanose anomeric forms they belong to, with interpretation of the isolated signals and tentative interpretation of the overlapping ones. Namely, the orange designates the *α-fur* form and indigo designates the *β-fur* form. A) section 100.5 ppm to 79 ppm; B) section 105.0 ppm to 90.0 ppm; C) section 78.0 ppm to 58.0 ppm. Highlighted are also the principal HSQC correlations to hydrogen atoms within the same spin systems.

A



B

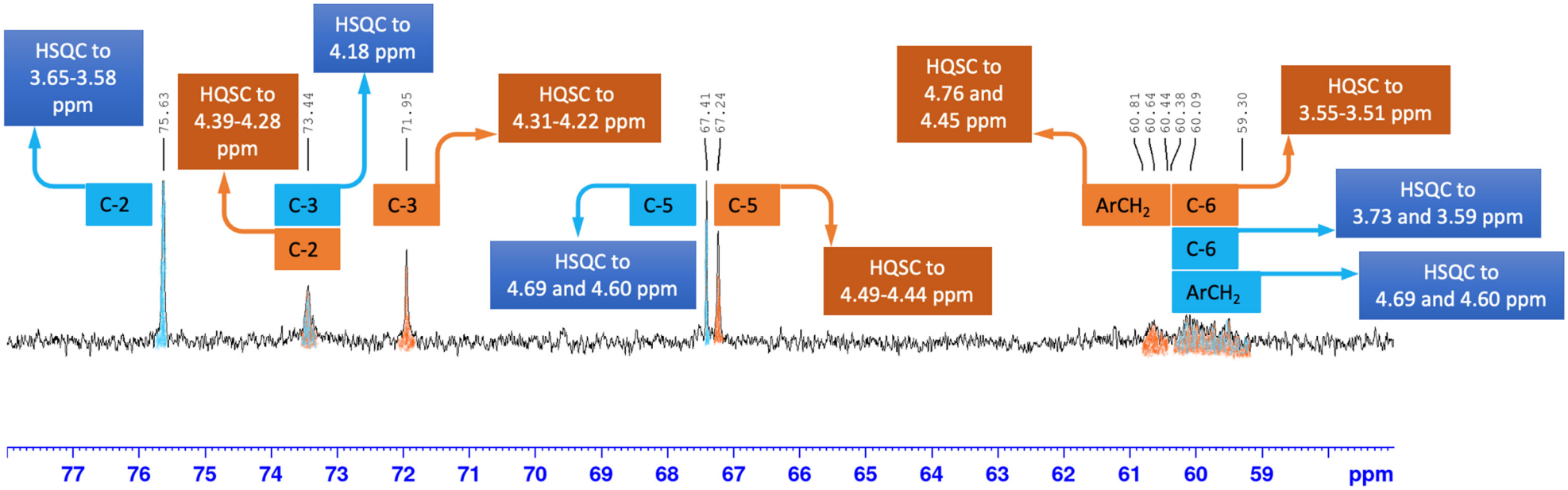
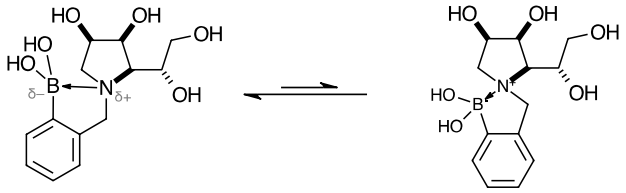

HSQC to 3.65-3.58 ppm
HQSC to 4.39-4.28 ppm
HSQC to 4.18 ppm
HQSC to 4.31-4.22 ppm
HQSC to 4.76 and 4.45 ppm
HQSC to 3.55-3.51 ppm
HSQC to 3.73 and 3.59 ppm
HSQC to 4.69 and 4.60 ppm
HQSC to 4.49-4.44 ppm
HSQC to 4.69 and 4.60 ppm
C-2
C-3
C-2
C-3
C-5
C-5
ArCH2
C-6
C-6
ArCH2
75.63
73.44
71.95
67.41
67.24
60.81
60.64
60.44
60.38
60.09
59.30
77 76 75 74 73 72 71 70 69 68 67 66 65 64 63 62 61 60 59 ppm

Figure S10. ^{1}H- (400 MHz), ^{13}C-NMR (100 MHz), DEPT, ^{11}B-NMR (128 MHz), COSY, HSQC and HMBC spectra of *N*-(2-methylphenyl boronic acid)-1,4-dideoxy-1,4-imino-L-gulitol **ortho 4** in D_2O.

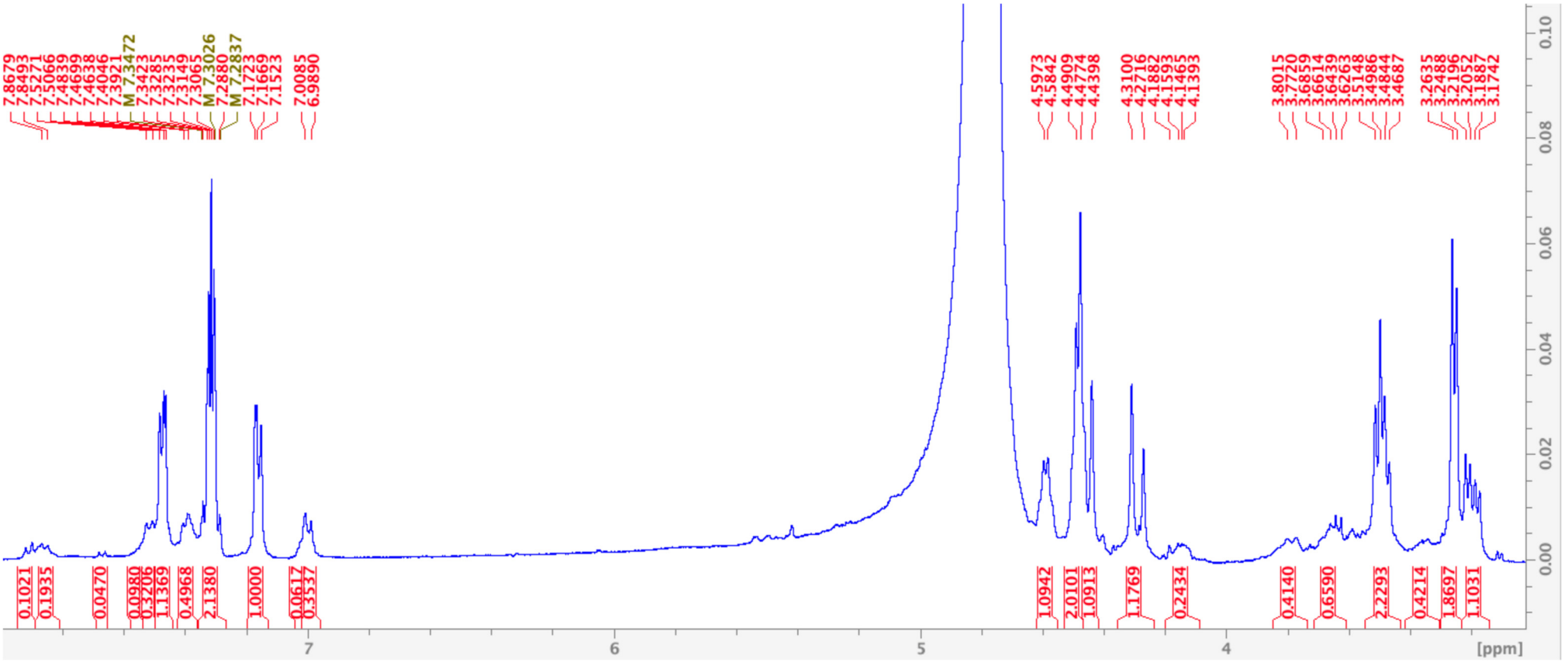


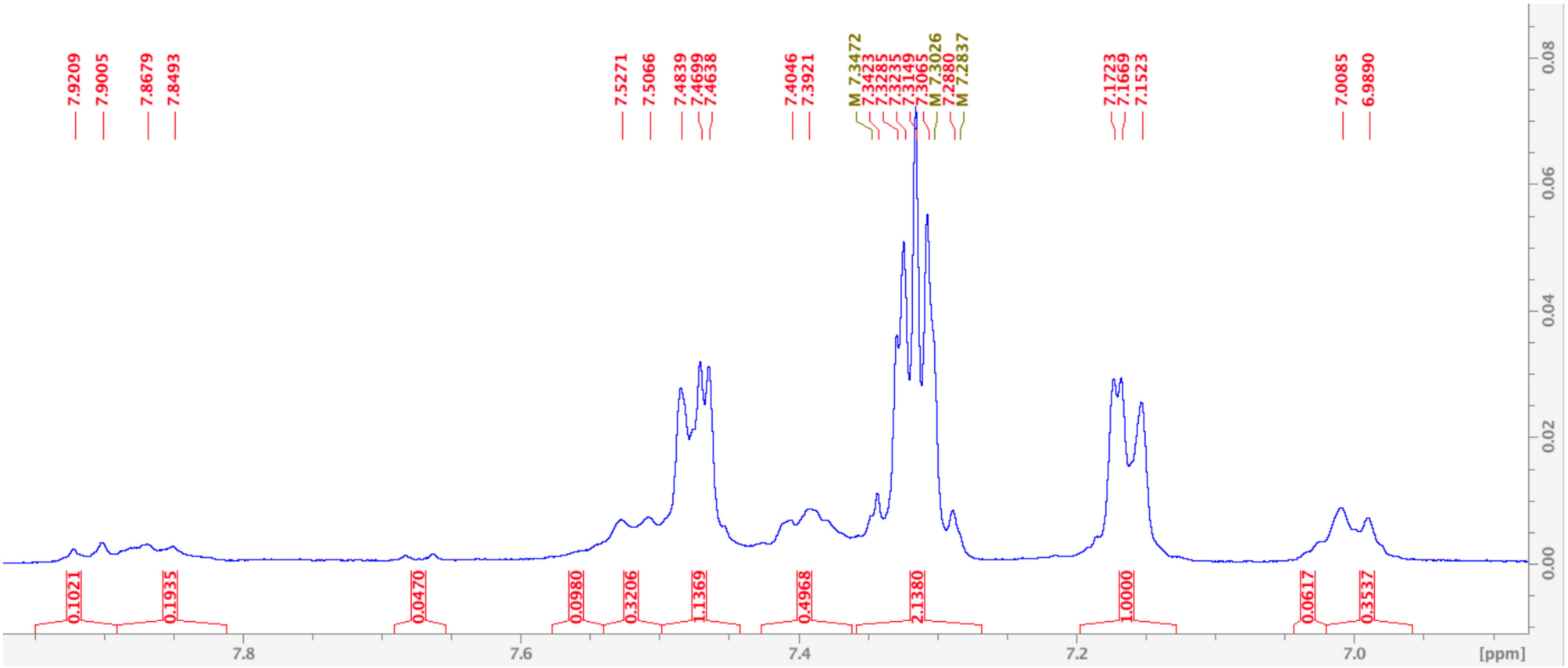
Boronate ammonium species (partially tetrahedral). **δH**: 7.48 (1H, app-dd, *J* 5.6, 2.4 Hz, ArH^{B}), 7.33 (1H, ddd, partially obscured, *J* 7.6, 5.6, 2.0 Hz, ArH^{C}), 7.31 (1H, ddd, partially obscured, *J* 7.4, 5.8, 2.1 Hz, ArH^{D}), 7.17 (1H, app-dd, *J* 5.8, 2.2Hz, ArH^{E}), 4.60 (1H, app-dd, *J* 10.9 Hz, $J_{H\text{-}2,H\text{-}1'}$ 5.8 Hz, H-2), 4.53-4.45 (2H, app-dd, partially obscured, *J* 10.7, 5.4 Hz, H-5 and H-3), 4.45 (1H, partially obscured, $J_{Ha,Hb}$ 15.2 Hz, ArC*H*^{a}H^{b}), 4.29 (1H, d, $J_{Hb,Ha}$ 15.2 Hz, ArCH^{a}*H*^{b}), 3.55-3.45 (2H, partially obscured dd and m, $J_{H\text{-}1',H\text{-}1}$ 12.3 Hz, $J_{H\text{-}1',H\text{-}2}$ 6.5 Hz, H-1′ and H-4), 3.25 (2H, app-d, $J_{H\text{-}6,H\text{-}6'=H\text{-}6',H\text{-}6}$ 5.8 Hz, H-6 and H-6′), 3.20 (1H, dd, $J_{H\text{-}1',H\text{-}1}$ 12.4 Hz, $J_{H\text{-}1',H\text{-}2}$ 5.8 Hz, H-1′); **δC**: 140.6 (ArC$_{quat}$), 129.4 (ArC^{B}), 128.2 (ArC^{C}), 127.6 (ArC^{D}), 122.7 (ArC^{E}), 72.9 (C-3), 72.7 (C-4), 71.6 (C-2), 70.4 (C-5), 67.3 (ArCH$_2$), 63.8 (C-6), 57.4 (C-1); ArC-B not discernible.

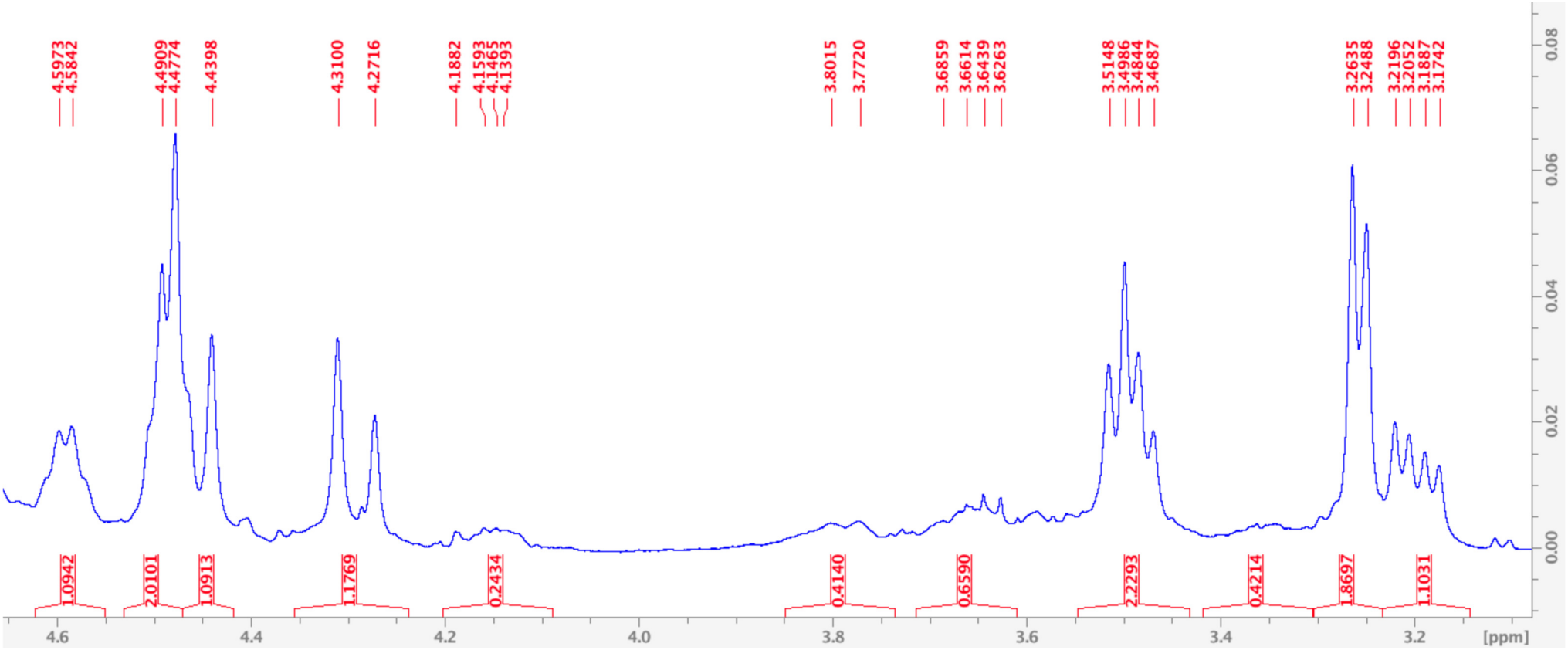
Boronate ammonium species (tetrahedral). **δH**: 7.88 (1H, app-dt, *J* 13.0, 8.4, 8.4 Hz, ArH), 7.52 (1H, app-d, partially obscured, *J* 8.3 Hz, ArH), 7.40 (1H, dd, J 7.2, 1.8 Hz, ArH), 7.05-6.95 (1H, m, ArH), 4.50-4.39 (1H, obscured), 4.20-4.10 (1H, m), 3.85-3.75 (1H, m, H-6), 3.72-3.62 (2H, m, H and H-6′), 3.40-3.30 (1H, m), 1.45-1.28 (2H, m, H-1 and H-1′). **δC**: 132.2, 130.3, 127.9, 115.9, 72.3 (ArCH$_2$, width 52 Hz), 68.1, 62.9 (C-6), 55.2, 9.9 (C-1).

δB: 28.3 (broad, integration: 1.1), 19.4 (sharp, integration: 6.1), 12.3 (sharp and merging with 11.0 ppm signal, integration: 1.2), 11.0 (sharp and merging with 12.3 ppm signal, integration: 1.0).

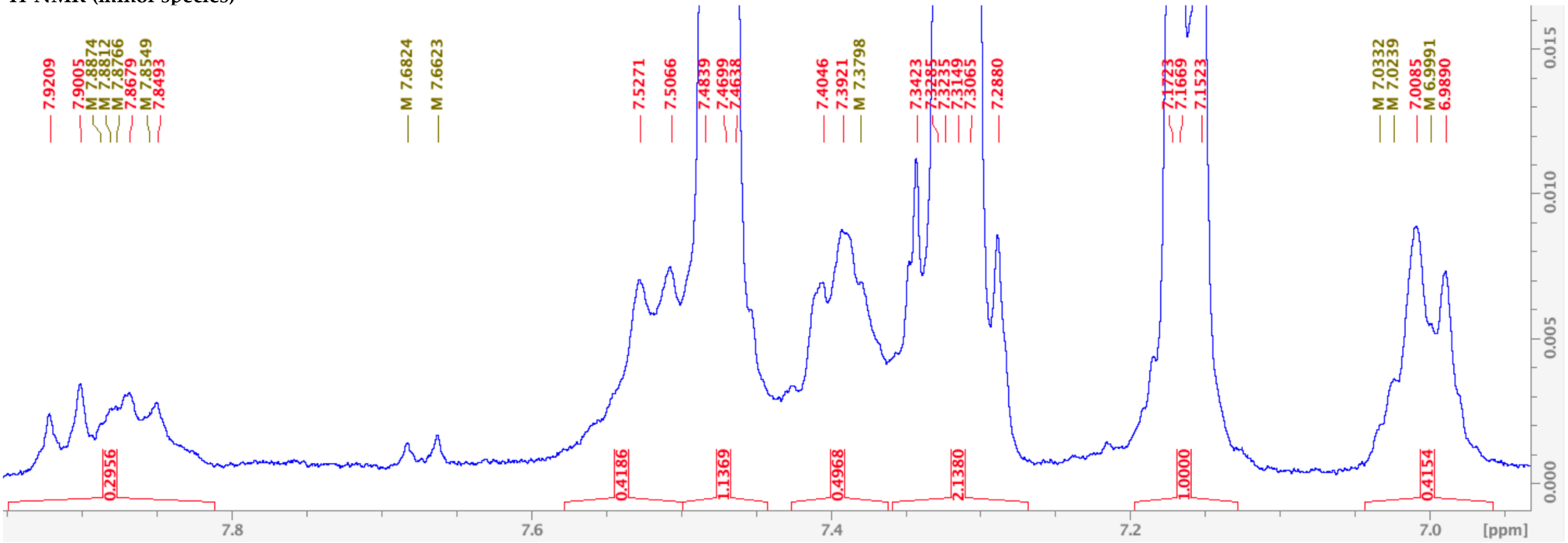
^{1}H-NMR

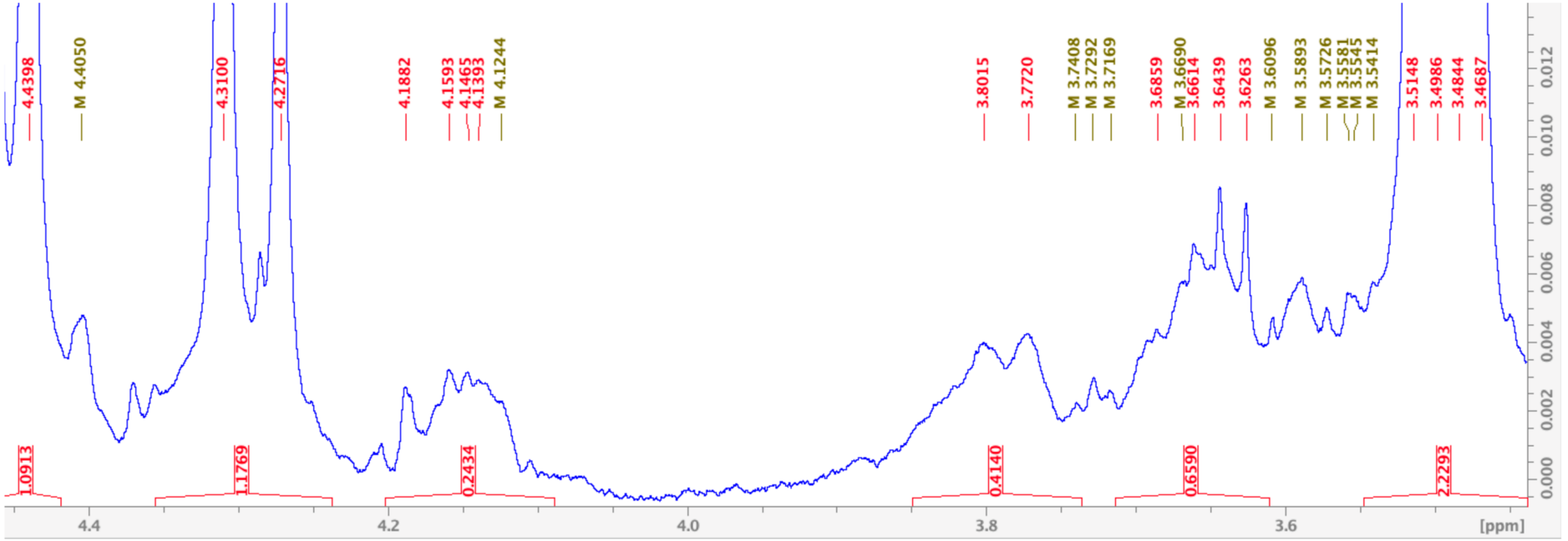

7.8679
7.8493
7.5271
7.5066
7.4839
7.4699
7.4638
7.4046
7.3921
M 7.3472
7.3423
7.3285
7.3235
7.3149
7.3065
M 7.3026
7.2880
M 7.2837
7.1723
7.1669
7.1523
7.0085
6.9890
4.5973
4.5842
4.4909
4.4774
4.4398
4.3100
4.2716
4.1882
4.1593
4.1465
4.1393
3.8015
3.7720
3.6859
3.6614
3.6439
3.6263
3.5148
3.4986
3.4844
3.4687
3.2635
3.2488
3.2196
3.2052
3.1887
3.1742
0.1021
0.1935
0.0470
0.0980
0.3206
1.1369
0.4968
2.1380
1.0000
0.0617
0.3537
1.0942
2.0101
1.0913
1.1769
0.2434
0.4140
0.6590
2.2293
0.4214
1.8697
1.1031
0.10
0.08
0.06
0.04
0.02
0.00
7
6
5
4
[ppm]

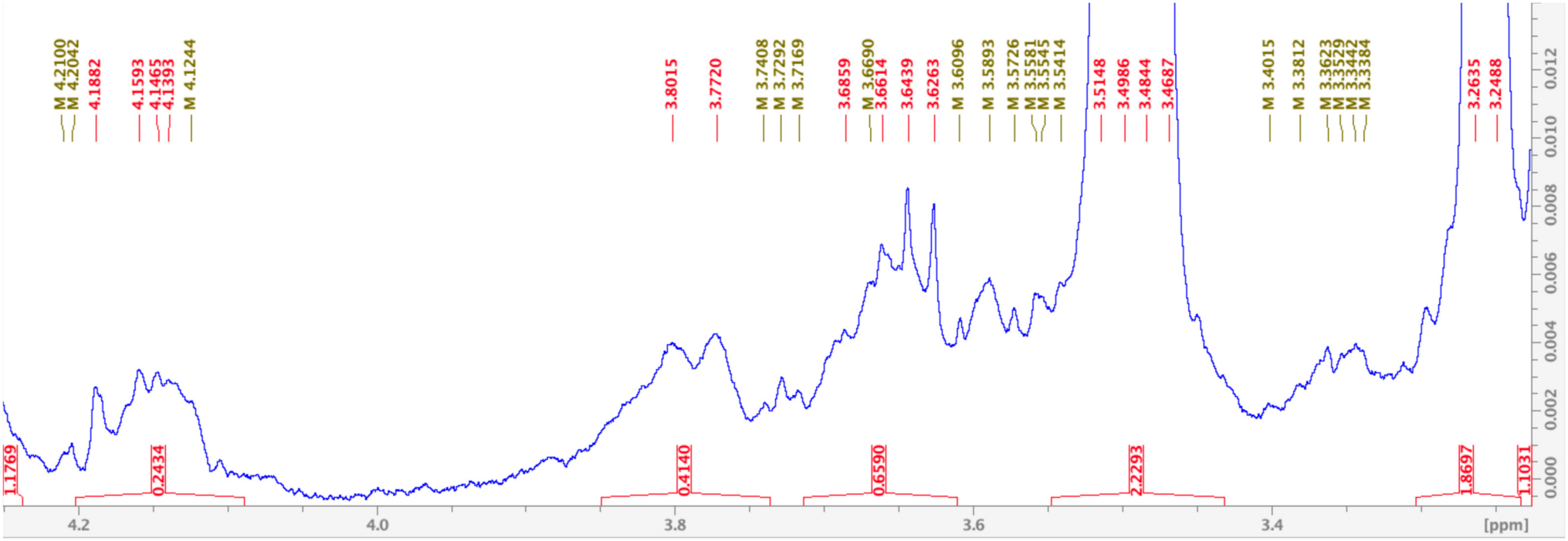

7.9209
7.9005
7.8679
7.8493
7.5271
7.5066
7.4839
7.4699
7.4638
7.4046
7.3921
M 7.3472
7.3423
7.3285
7.3235
7.3149
7.3065
M 7.3026
7.2880
M 7.2837
7.1723
7.1669
7.1523
7.0085
6.9890
0.1021
0.1935
0.0470
0.0980
0.3206
1.1369
0.4968
2.1380
1.0000
0.0617
0.3537
7.8
7.6
7.4
7.2
7.0
[ppm]
0.08
0.06
0.04
0.02
0.00

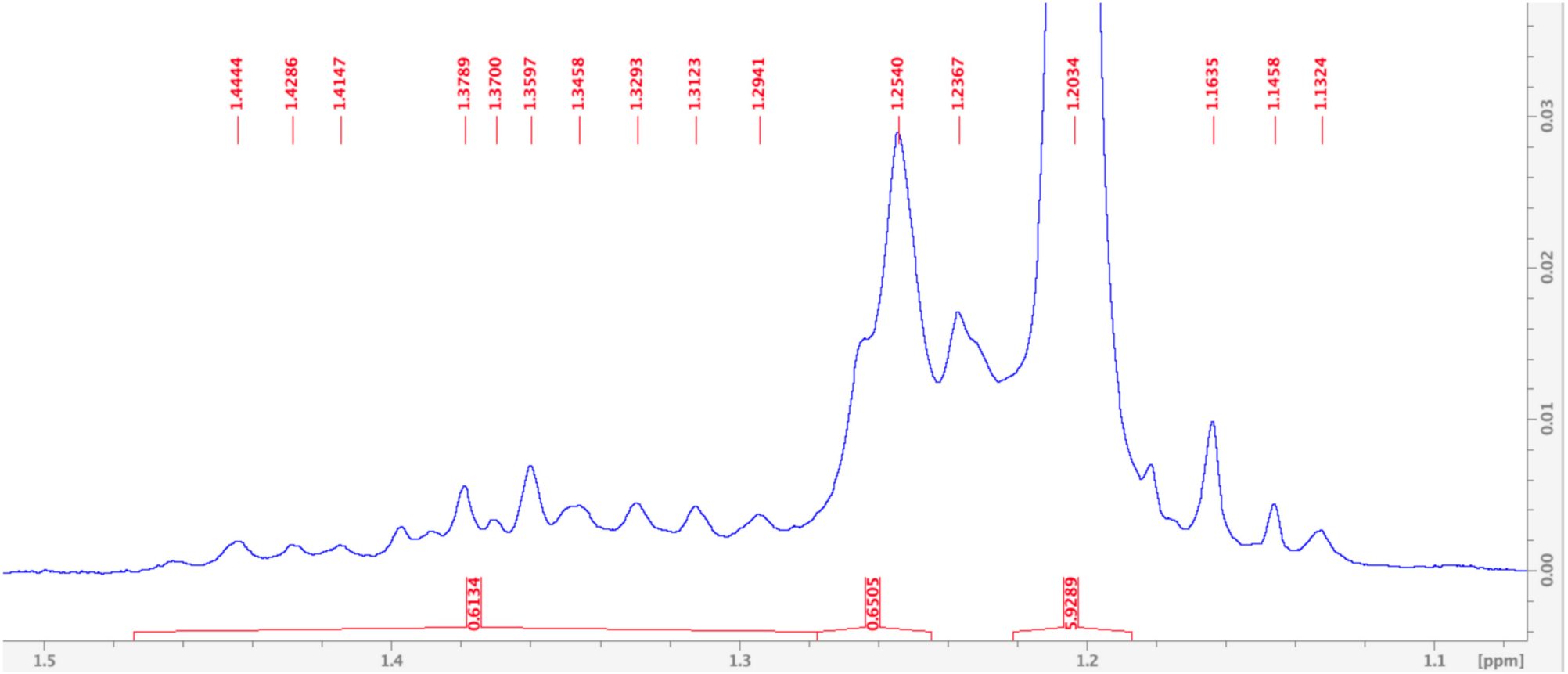

4.5973
4.5842
4.4909
4.4774
4.4398
4.3100
4.2716
4.1882
4.1593
4.1465
4.1393
3.8015
3.7720
3.6859
3.6614
3.6439
3.6263
3.5148
3.4986
3.4844
3.4687
3.2635
3.2488
3.2196
3.2052
3.1887
3.1742
1.0942
2.0101
1.0913
1.1769
0.2434
0.4140
0.6590
2.2293
0.4214
1.8697
1.1031
0.08
0.06
0.04
0.02
0.00
4.6
4.4
4.2
4.0
3.8
3.6
3.4
3.2
[ppm]

^{1}H-NMR (minor species)

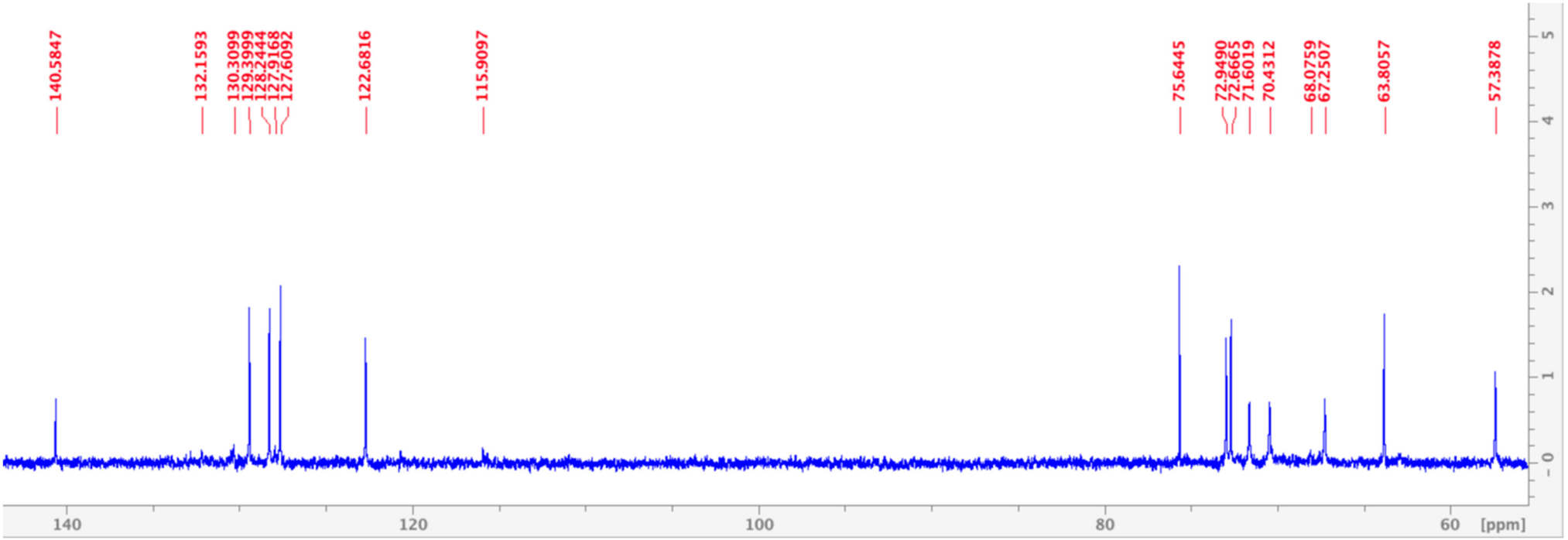


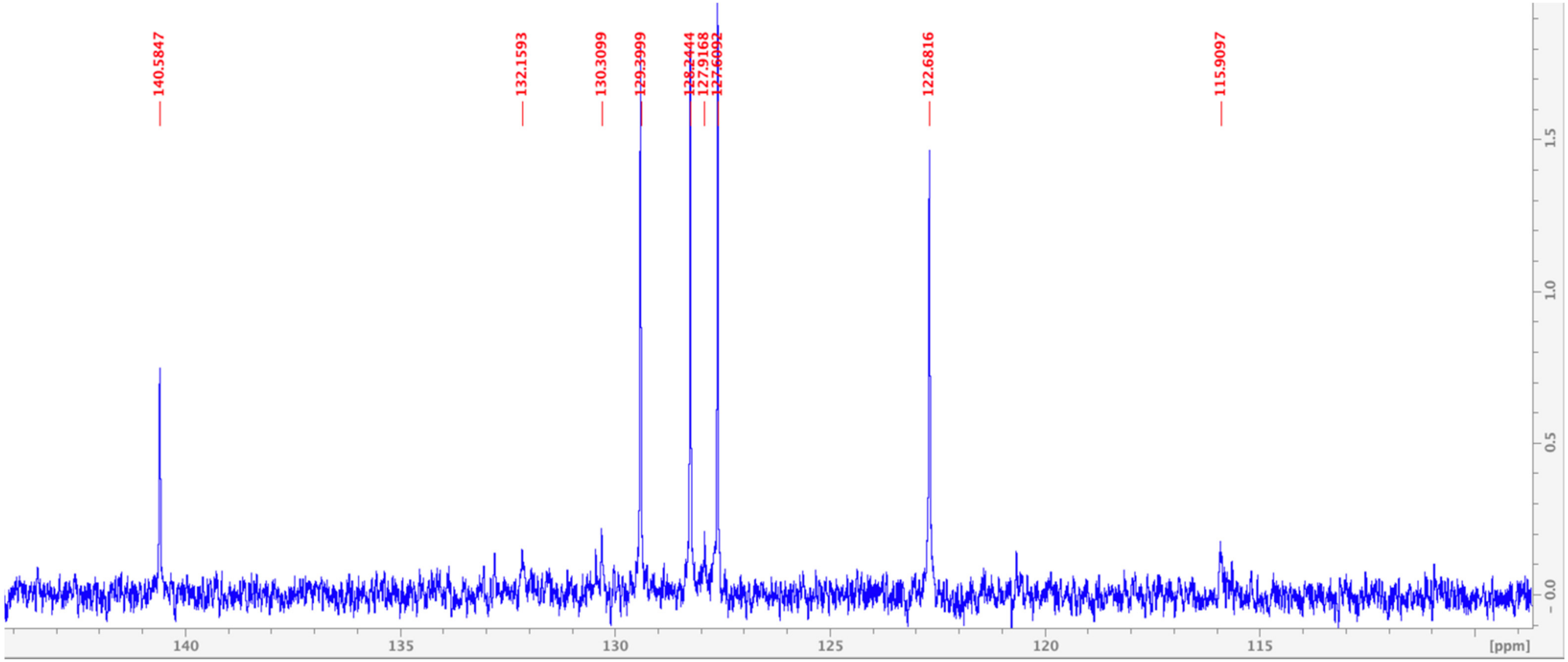

4.4398
M 4.4050
4.3100
4.2716
4.1882
4.1593
4.1465
4.1393
M 4.1244
3.8015
3.7720
M 3.7408
M 3.7292
M 3.7169
3.6859
M 3.6690
3.6614
3.6439
3.6263
M 3.6096
M 3.5893
M 3.5726
M 3.5581
M 3.5545
M 3.5414
3.5148
3.4986
3.4844
3.4687
1.0913
1.1769
0.2434
0.4140
0.6590
2.2293
0.012
0.010
0.008
0.006
0.004
0.002
0.000
4.4
4.2
4.0
3.8
3.6
[ppm]

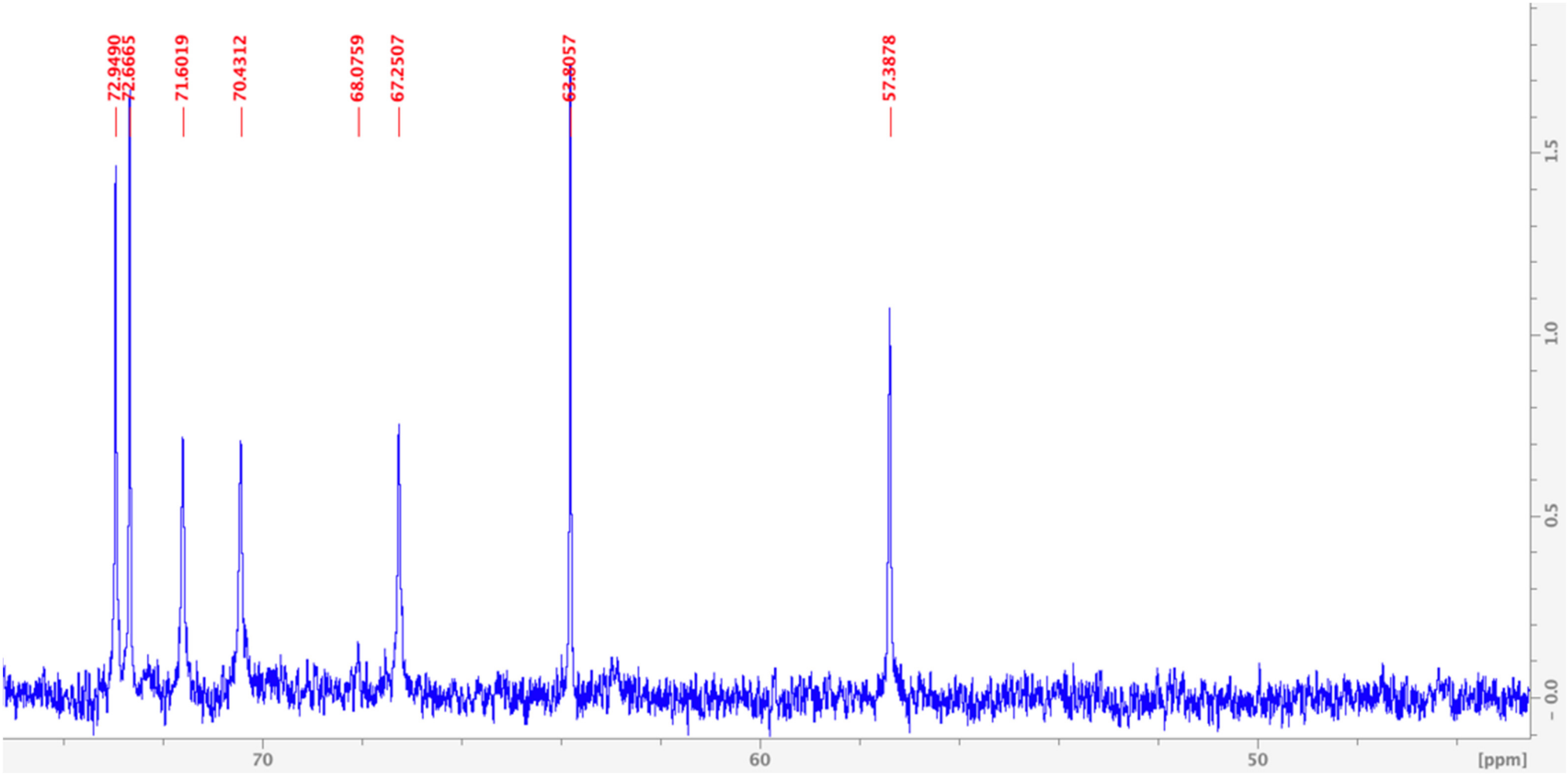

M 4.2100
M 4.2042
4.1882
4.1593
4.1465
4.1393
M 4.1244
3.8015
3.7720
M 3.7408
M 3.7292
M 3.7169
3.6859
M 3.6690
3.6614
3.6439
3.6263
M 3.6096
M 3.5893
M 3.5726
M 3.5581
M 3.5545
M 3.5414
3.5148
3.4986
3.4844
3.4687
M 3.4015
M 3.3812
M 3.3623
M 3.3529
M 3.3442
M 3.3384
3.2635
3.2488
1.1769
0.2434
0.4140
0.6590
2.2293
1.8697
1.1031
4.2
4.0
3.8
3.6
3.4
[ppm]
0.000
0.002
0.004
0.006
0.008
0.010
0.012


1.4444
1.4286
1.4147
1.3789
1.3700
1.3597
1.3458
1.3293
1.3123
1.2941
1.2540
1.2367
1.2034
1.1635
1.1458
1.1324
0.6134
0.6505
5.9289
1.5
1.4
1.3
1.2
1.1
[ppm]
0.00
0.01
0.02
0.03

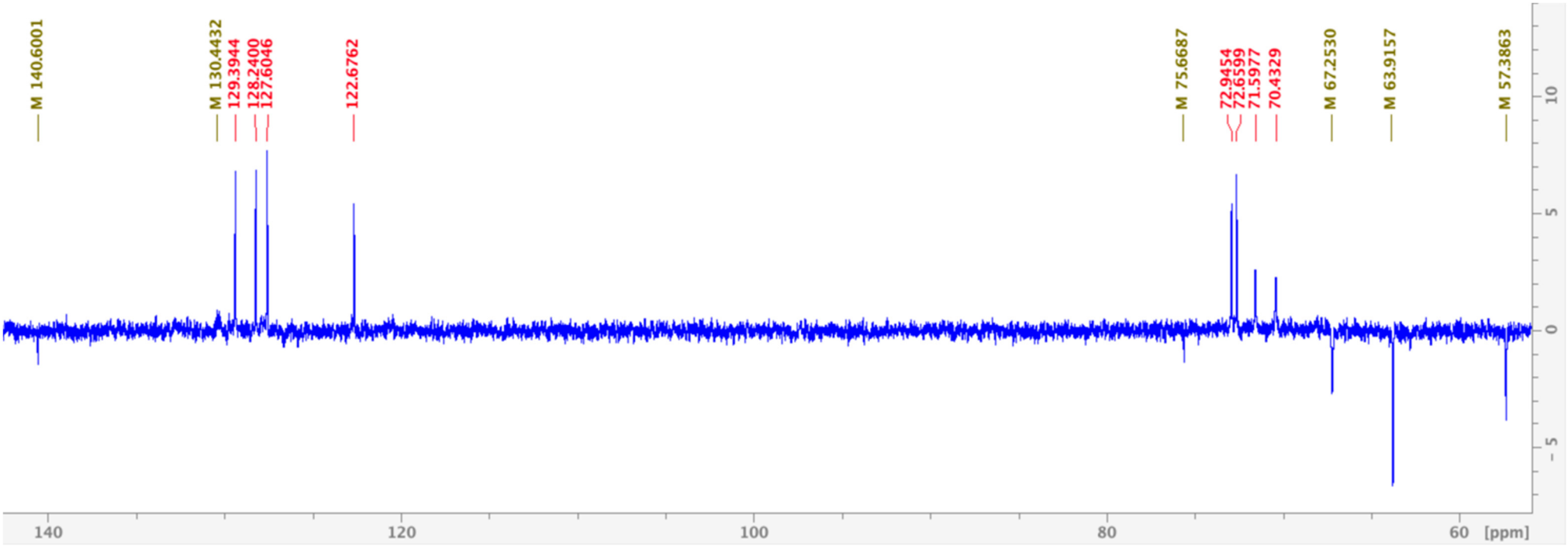
^{13}C-NMR


140.5847
132.1593
130.3099
129.3999
128.2444
127.9168
127.6092
122.6816
115.9097
75.6445
72.9490
72.6665
71.6019
70.4312
68.0759
67.2507
63.8057
57.3878
140
120
100
80
60
[ppm]
0
1
2
3
4
5

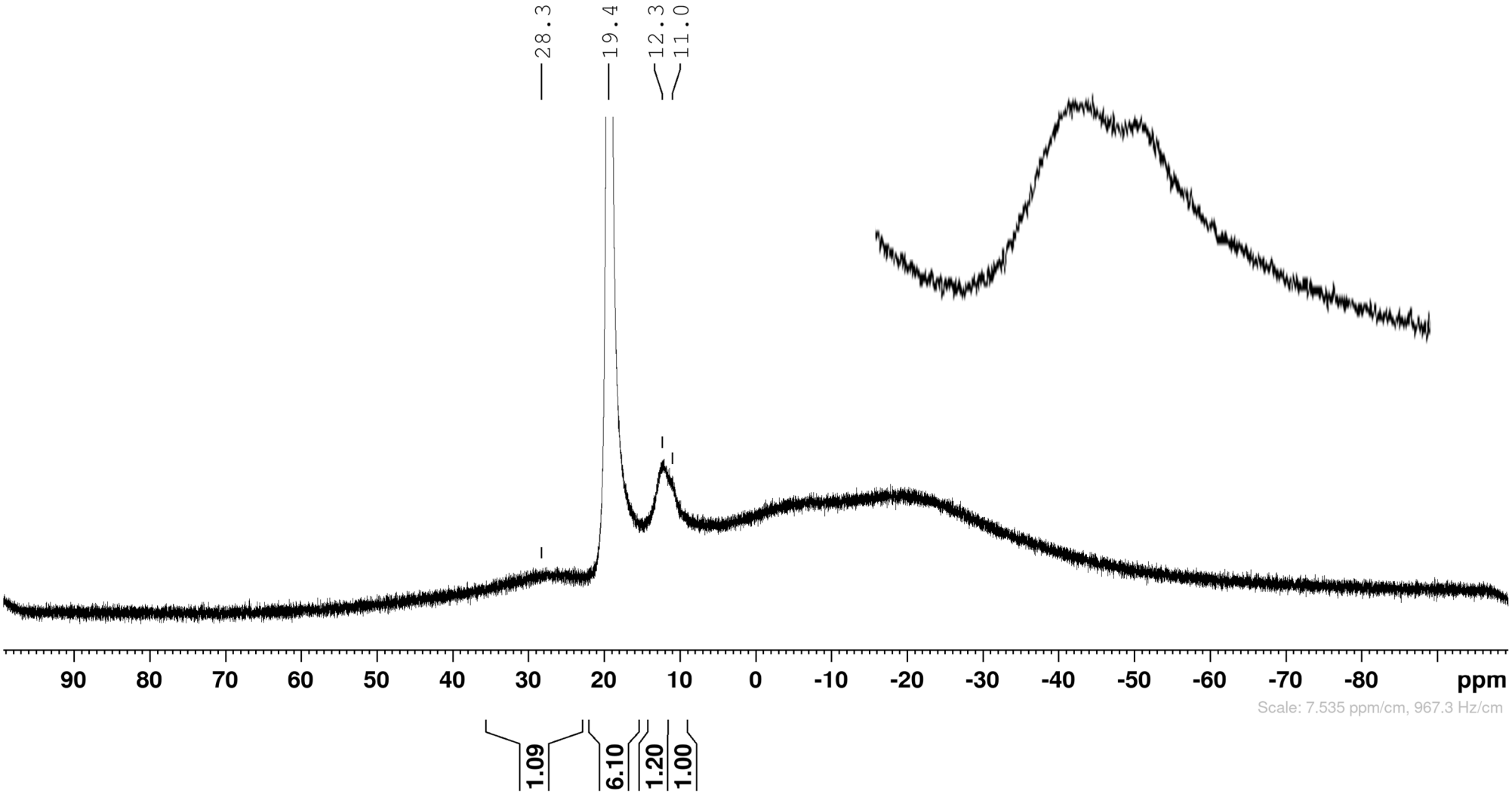

140.5847
132.1593
130.3099
129.3999
128.2444
127.9168
127.6092
122.6816
115.9097
1.5
1.0
0.5
0.0
140
135
130
125
120
115
[ppm]


72.9490
72.6665
71.6019
70.4312
68.0759
67.2507
63.8057
57.3878
1.5
1.0
0.5
0.0
70
60
50
[ppm]

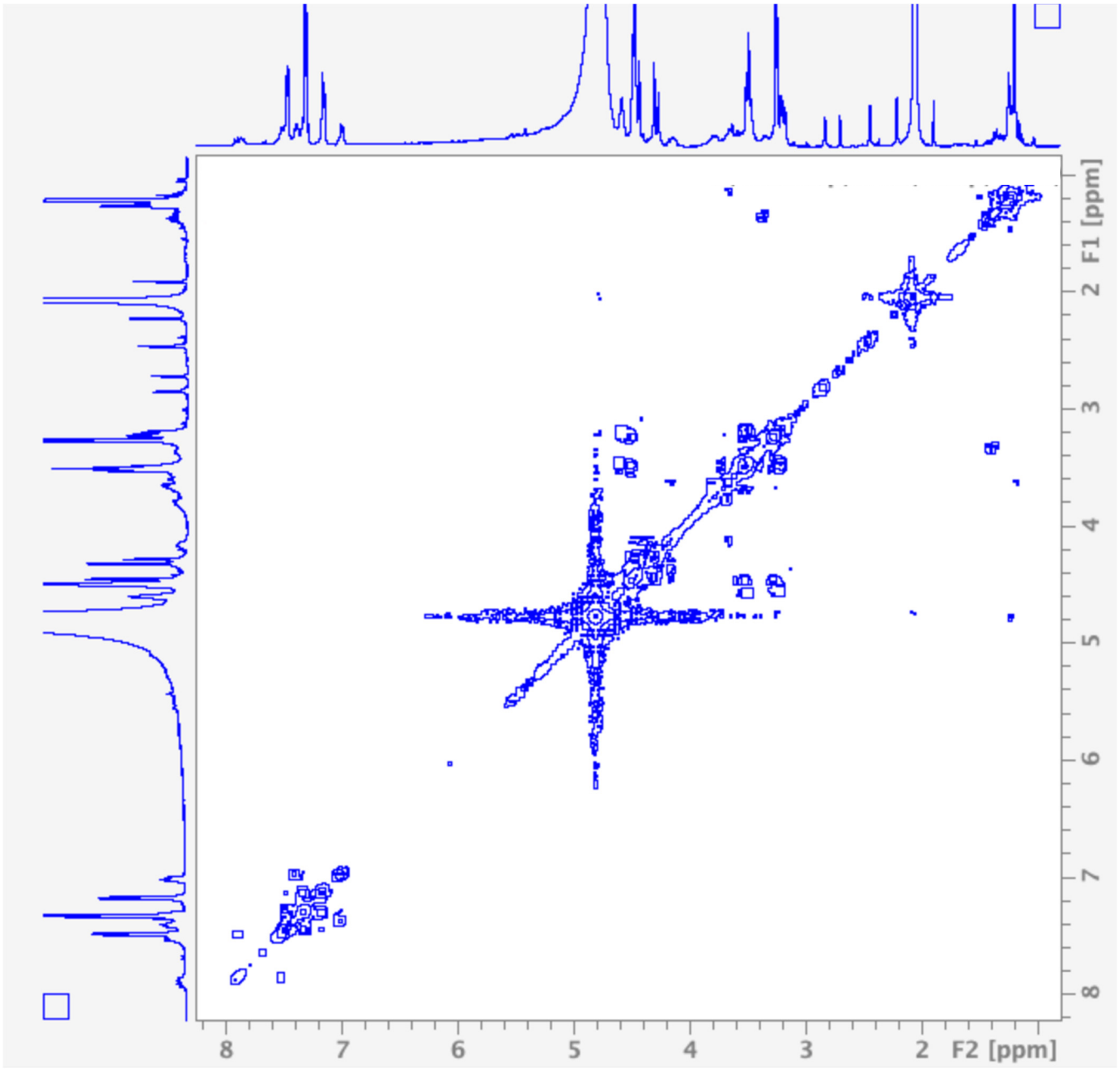
DEPT

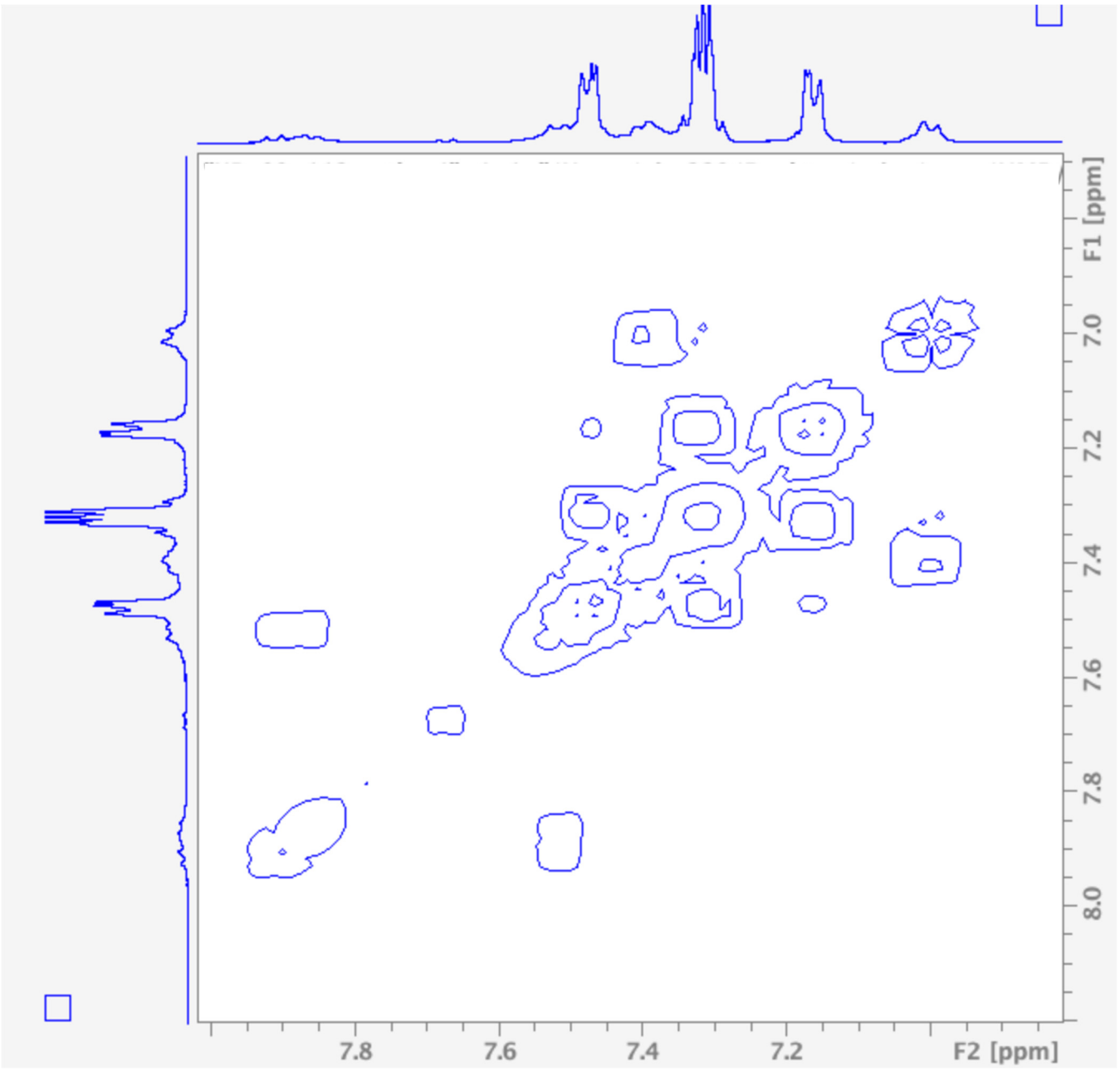

M 140.6001
M 130.4432
129.3944
128.2400
127.6046
122.6762
M 75.6687
72.9454
72.6599
71.5977
70.4329
M 67.2530
M 63.9157
M 57.3863
10
5
0
- 5
140
120
100
80
60
[ppm]

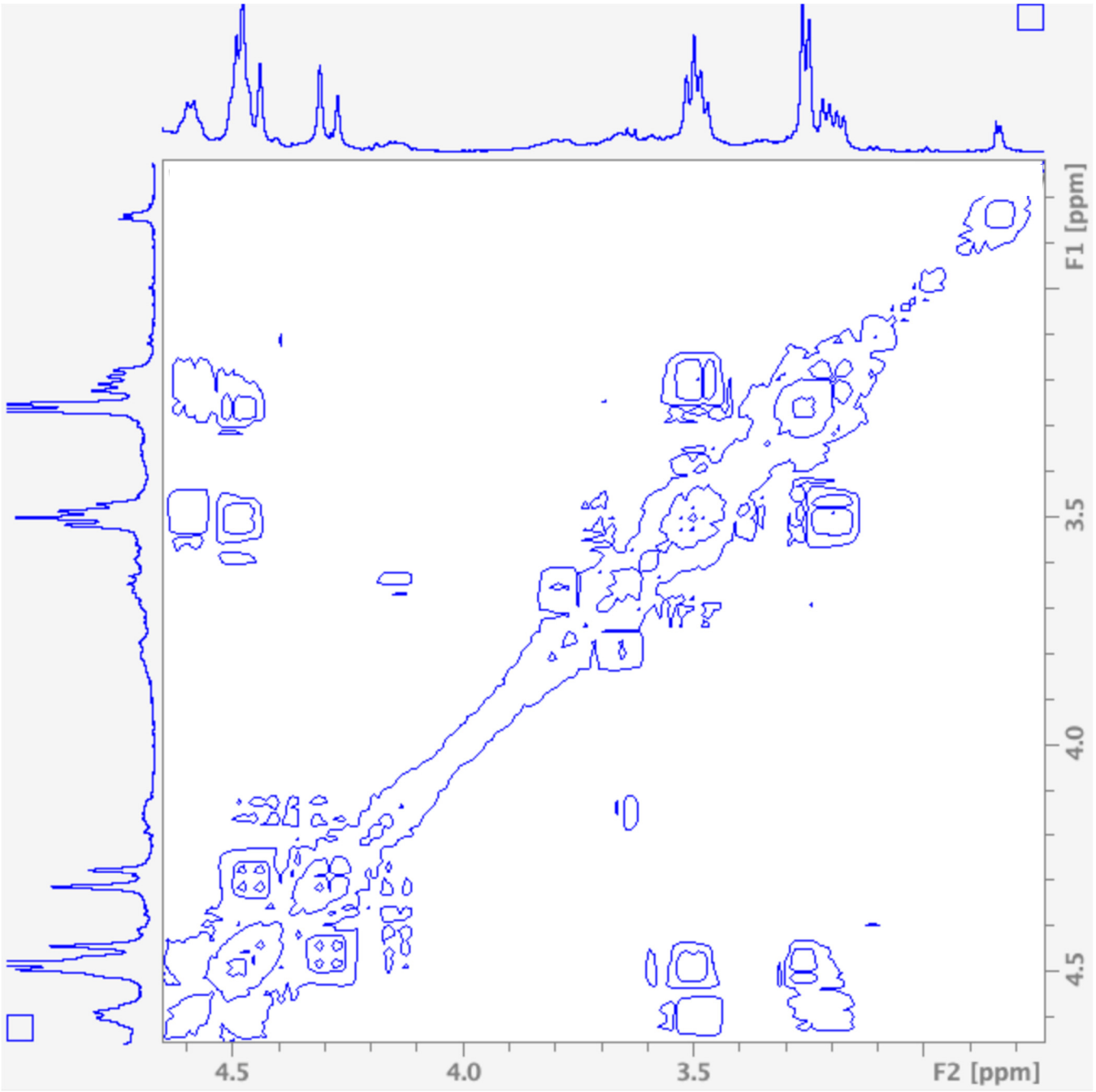
^{11}B-NMR

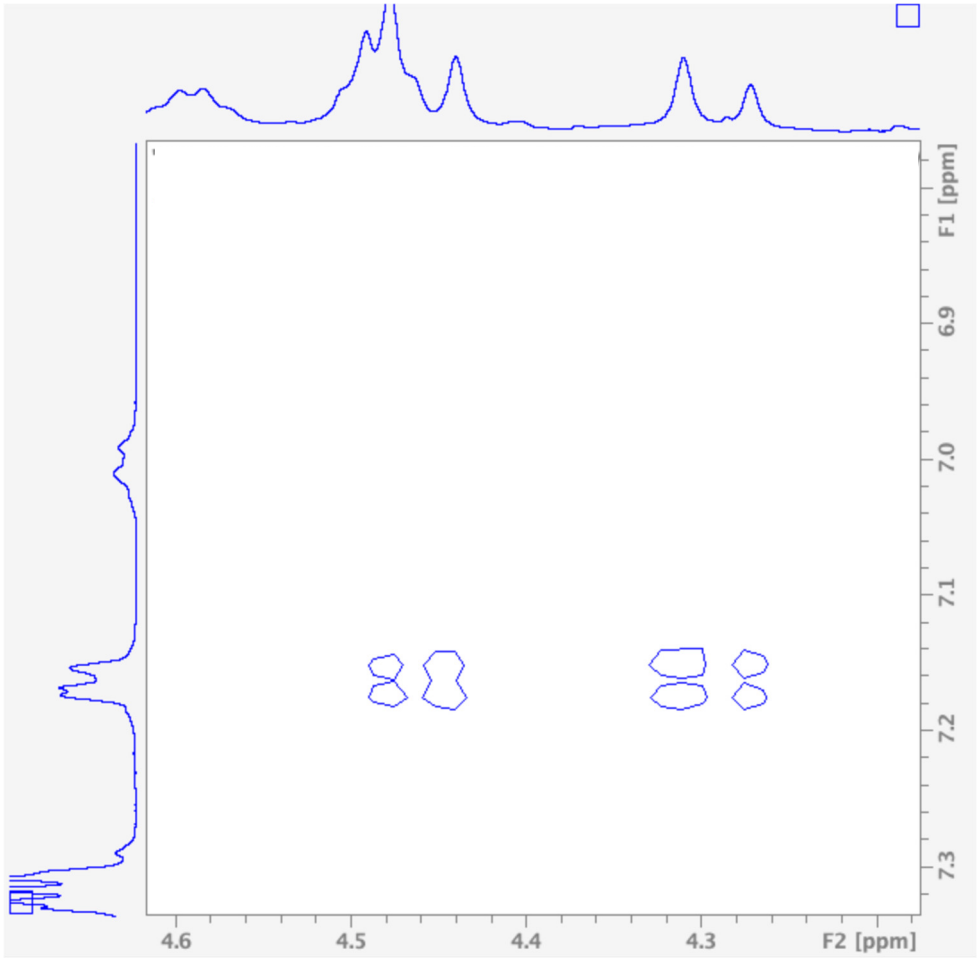

28.3
19.4
12.3
11.0
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 ppm
Scale: 7.535 ppm/cm, 967.3 Hz/cm
1.09
6.10
1.20
1.00

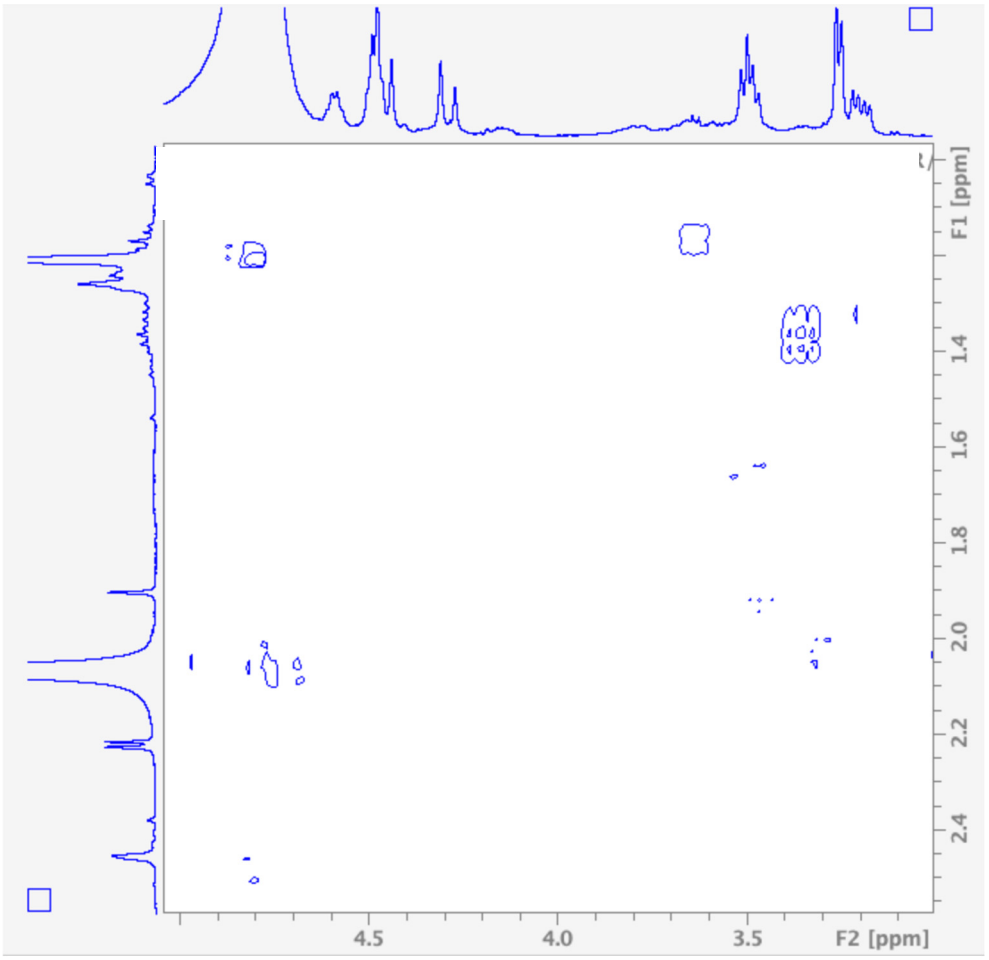
COSY



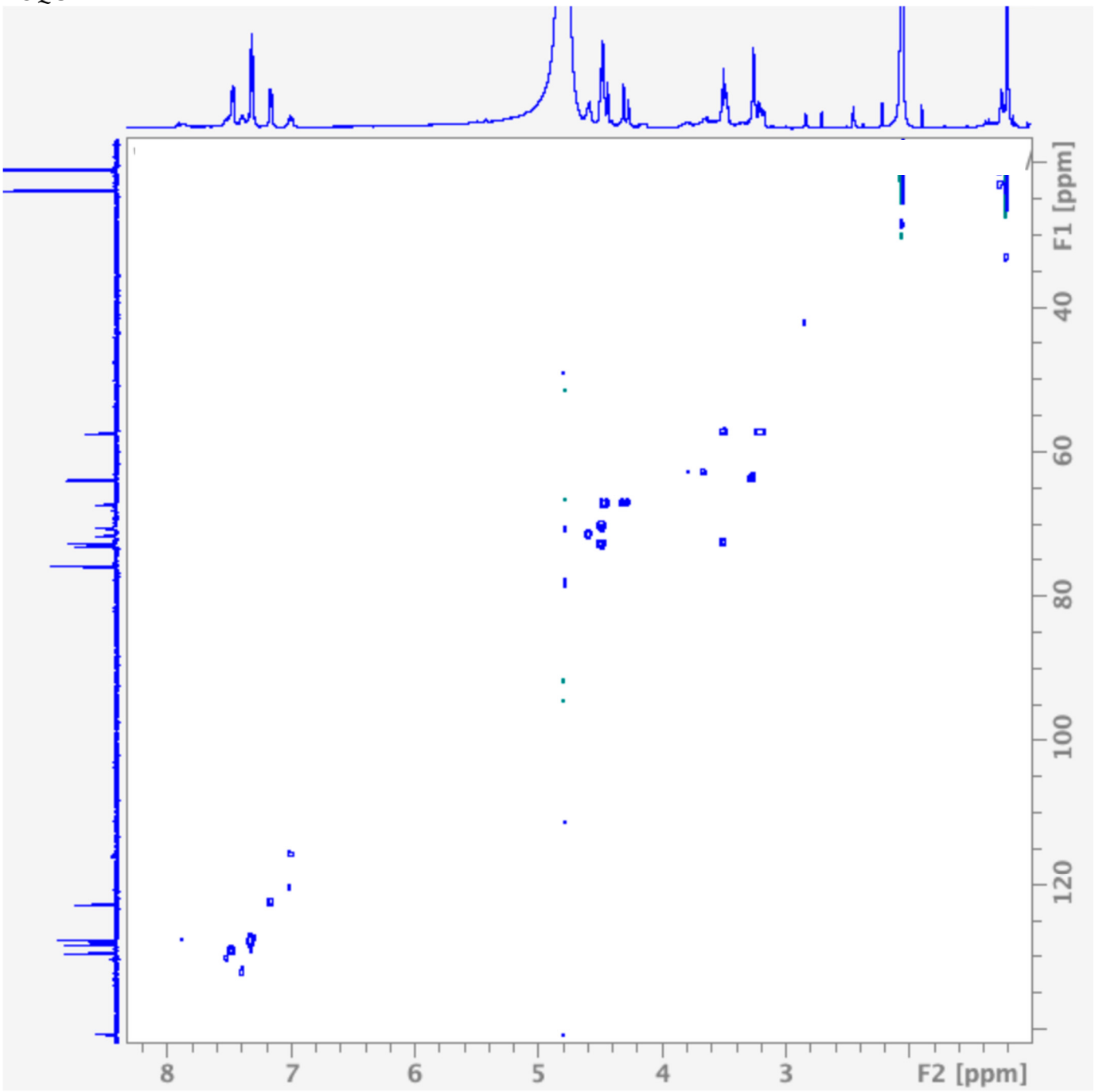

F1 [ppm]
7.0
7.2
7.4
7.6
7.8
8.0
7.8
7.6
7.4
7.2
F2 [ppm]

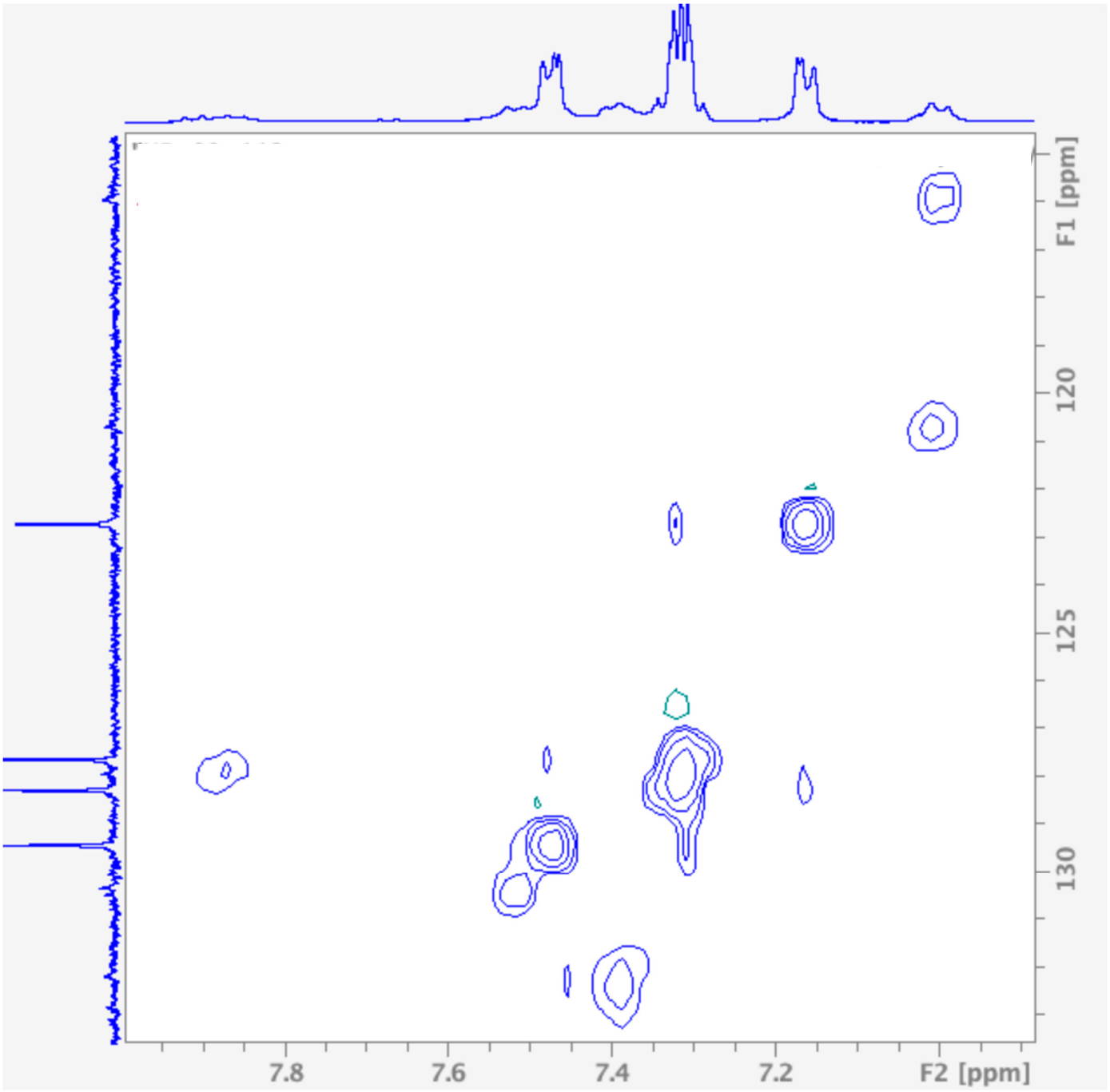

F1 [ppm]
3.5
4.0
4.5
4.5
4.0
3.5
F2 [ppm]

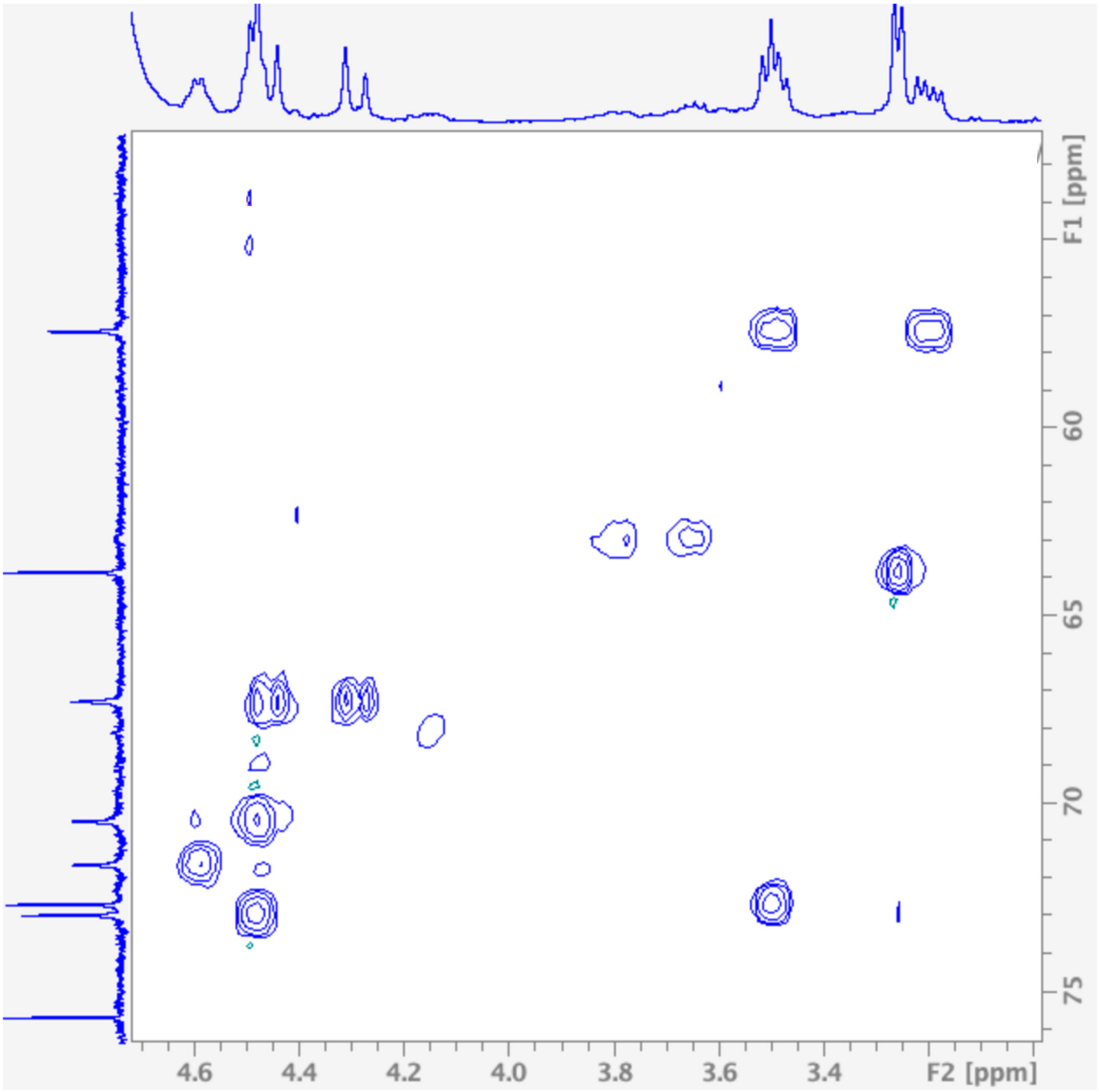

F1 [ppm]
6.9
7.0
7.1
7.2
7.3
4.6
4.5
4.4
4.3
F2 [ppm]

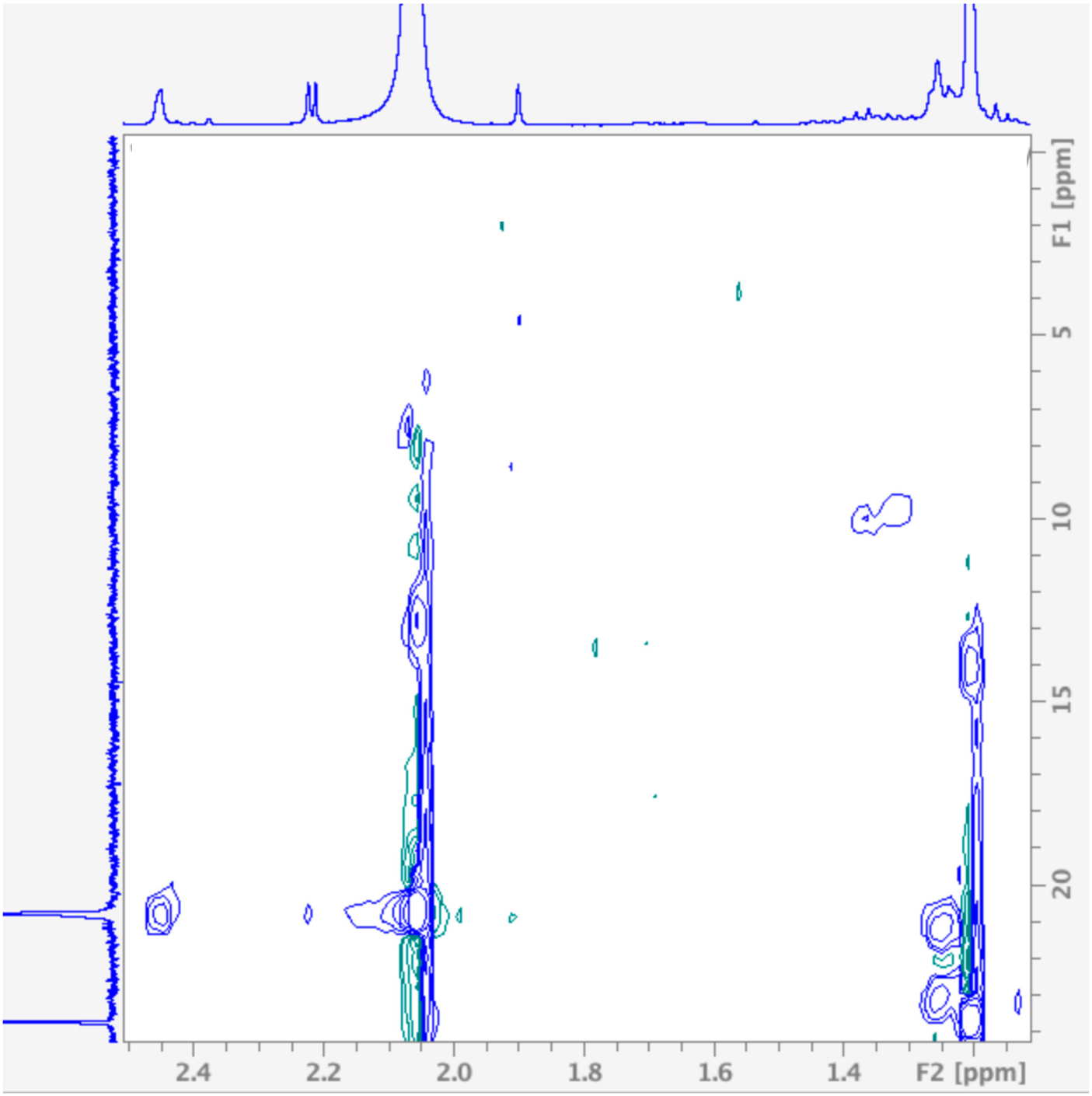

F1 [ppm]
1.4
1.6
1.8
2.0
2.2
2.4
4.5
4.0
3.5
F2 [ppm]

HSQC

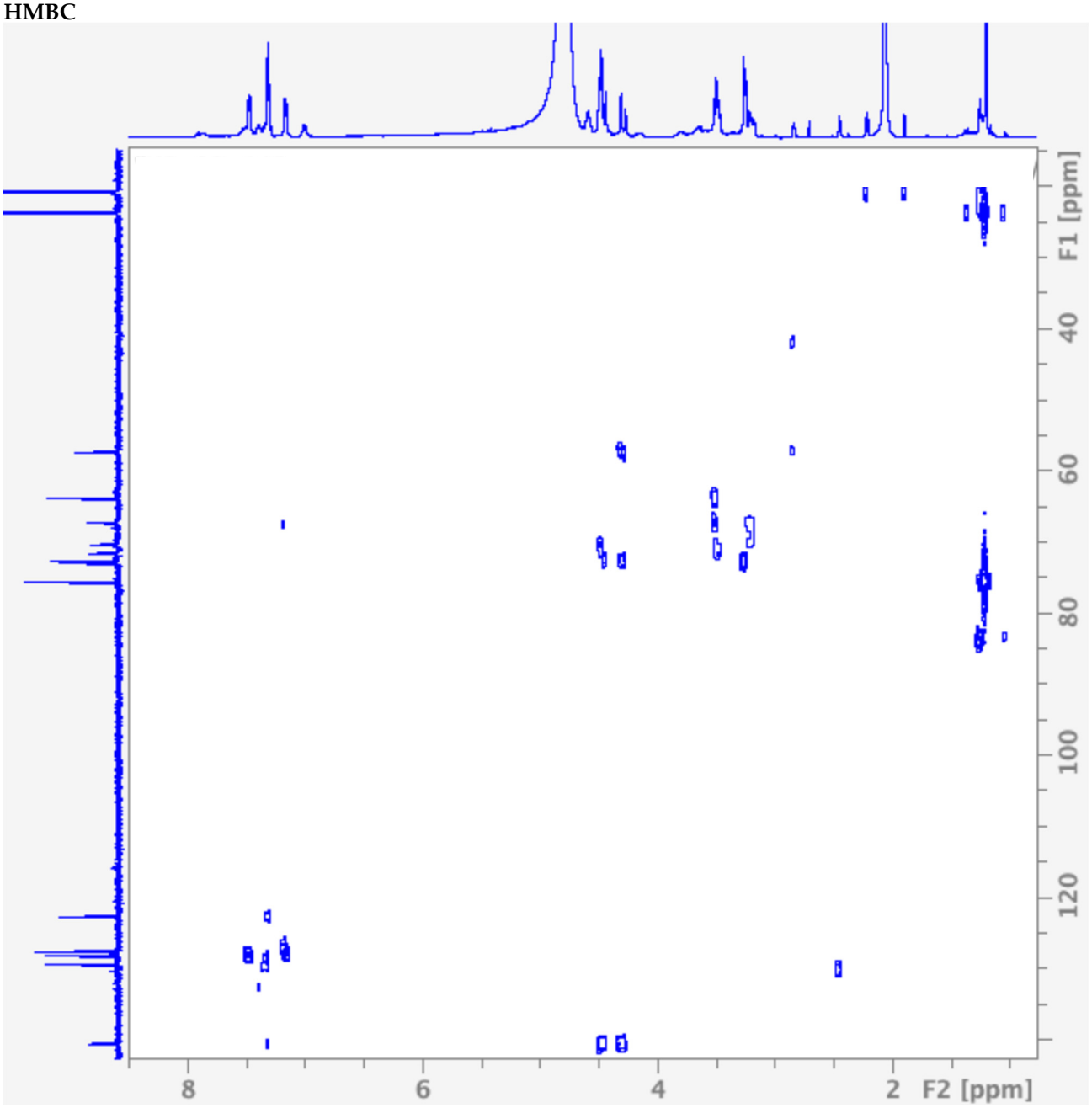


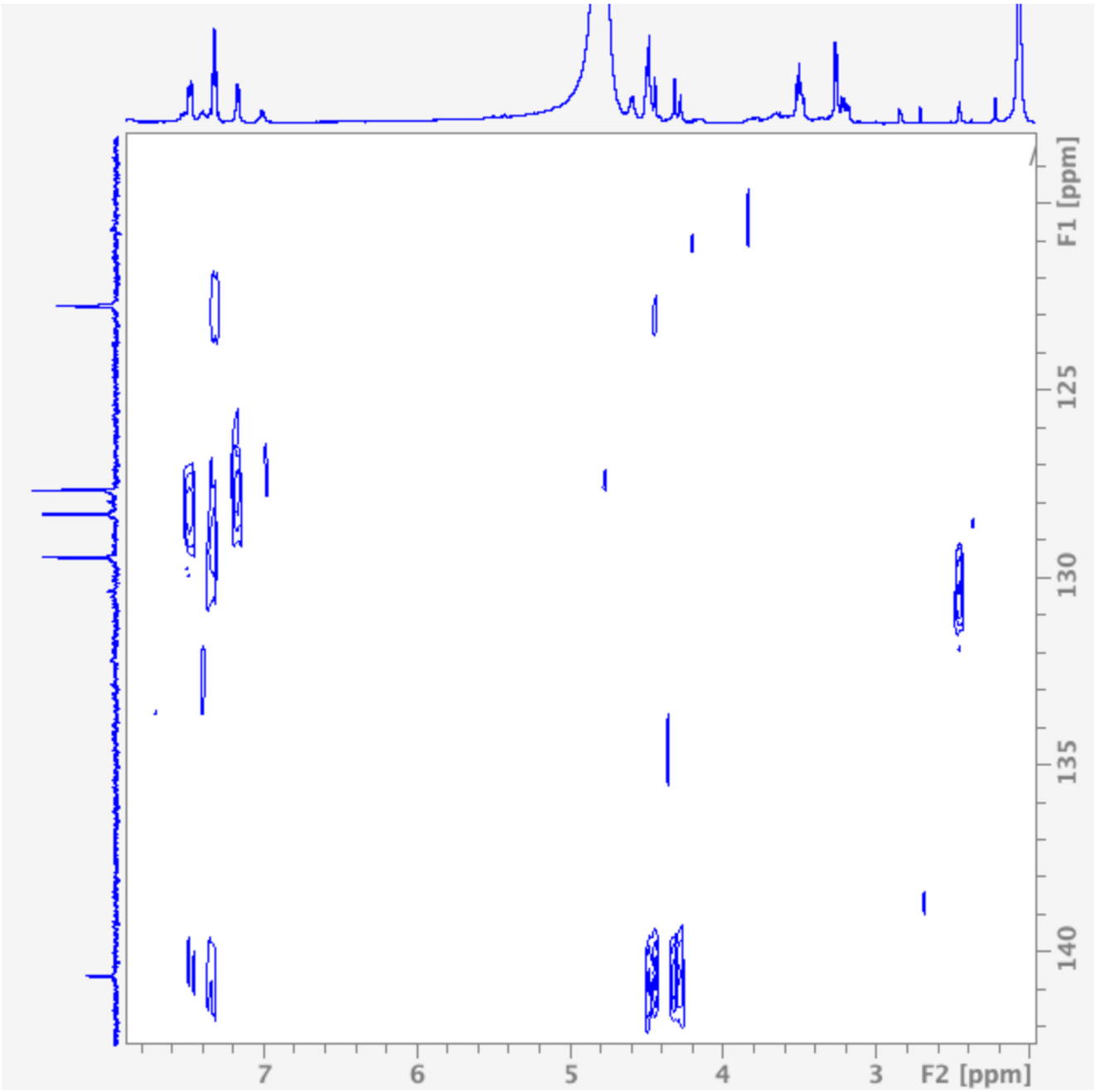

F1 [ppm]
120
125
130
7.8
7.6
7.4
7.2
F2 [ppm]

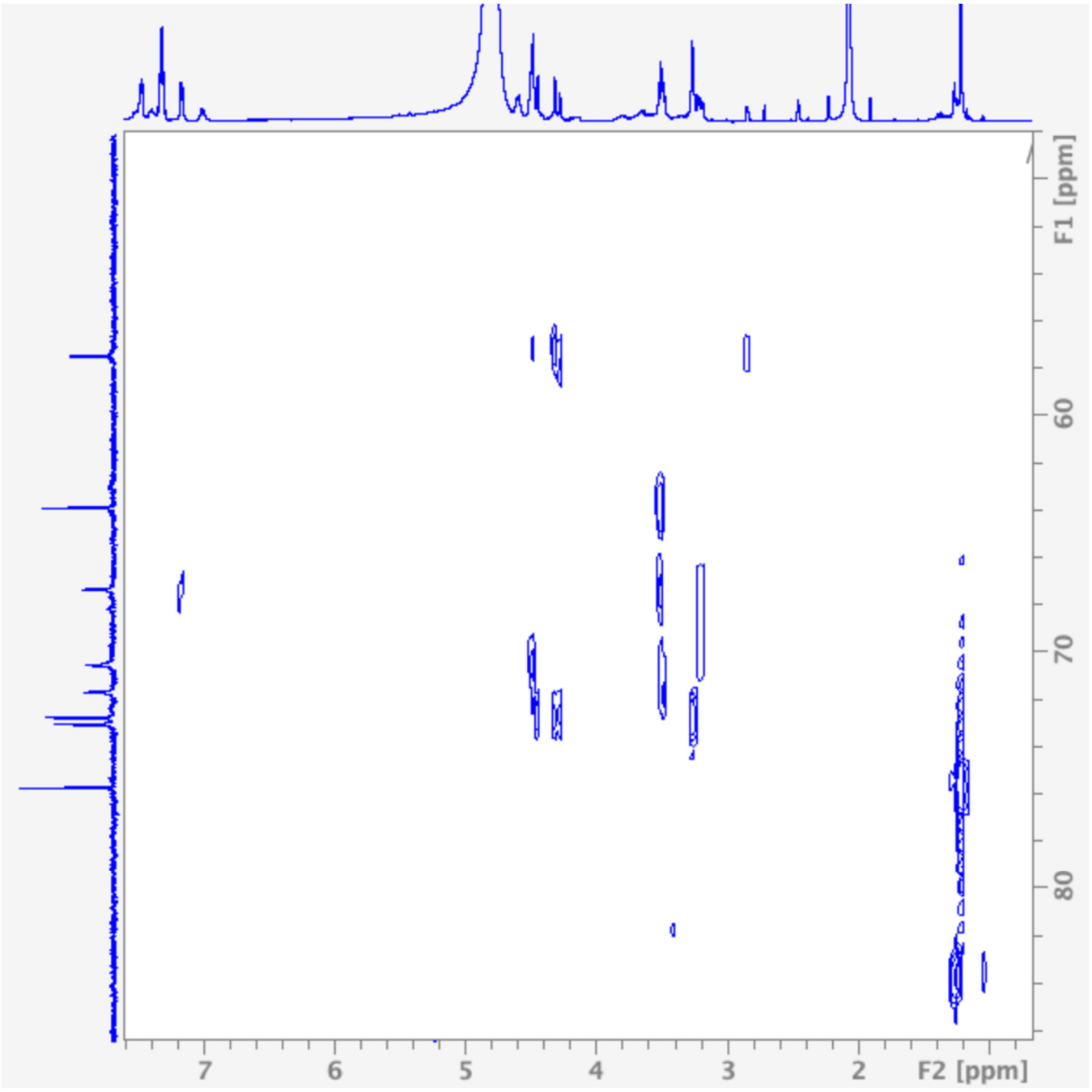

F1 [ppm]
60
65
70
75
4.6
4.4
4.2
4.0
3.8
3.6
3.4
F2 [ppm]

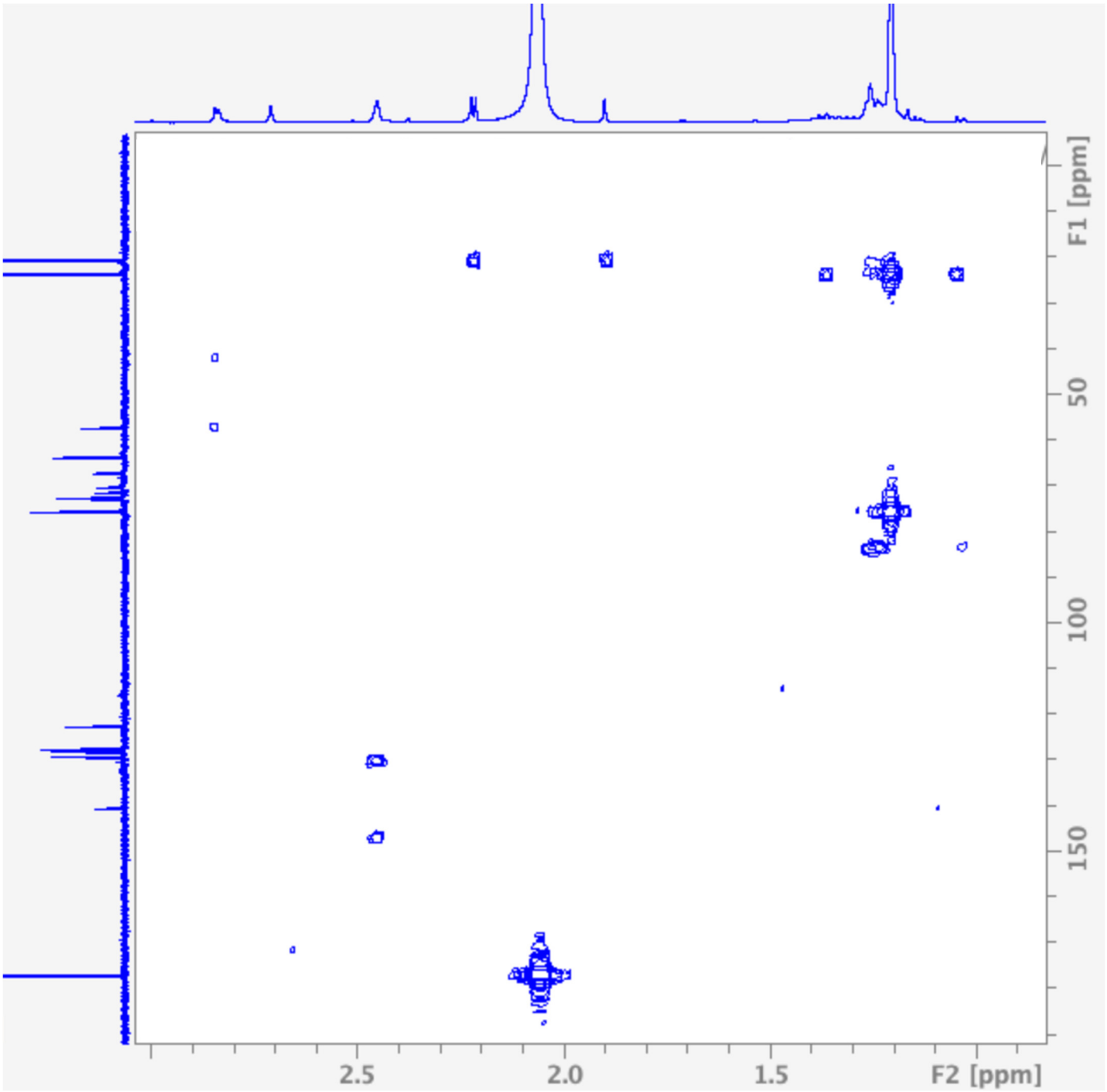

F1 [ppm]
5
10
15
20
2.4
2.2
2.0
1.8
1.6
1.4
F2 [ppm]

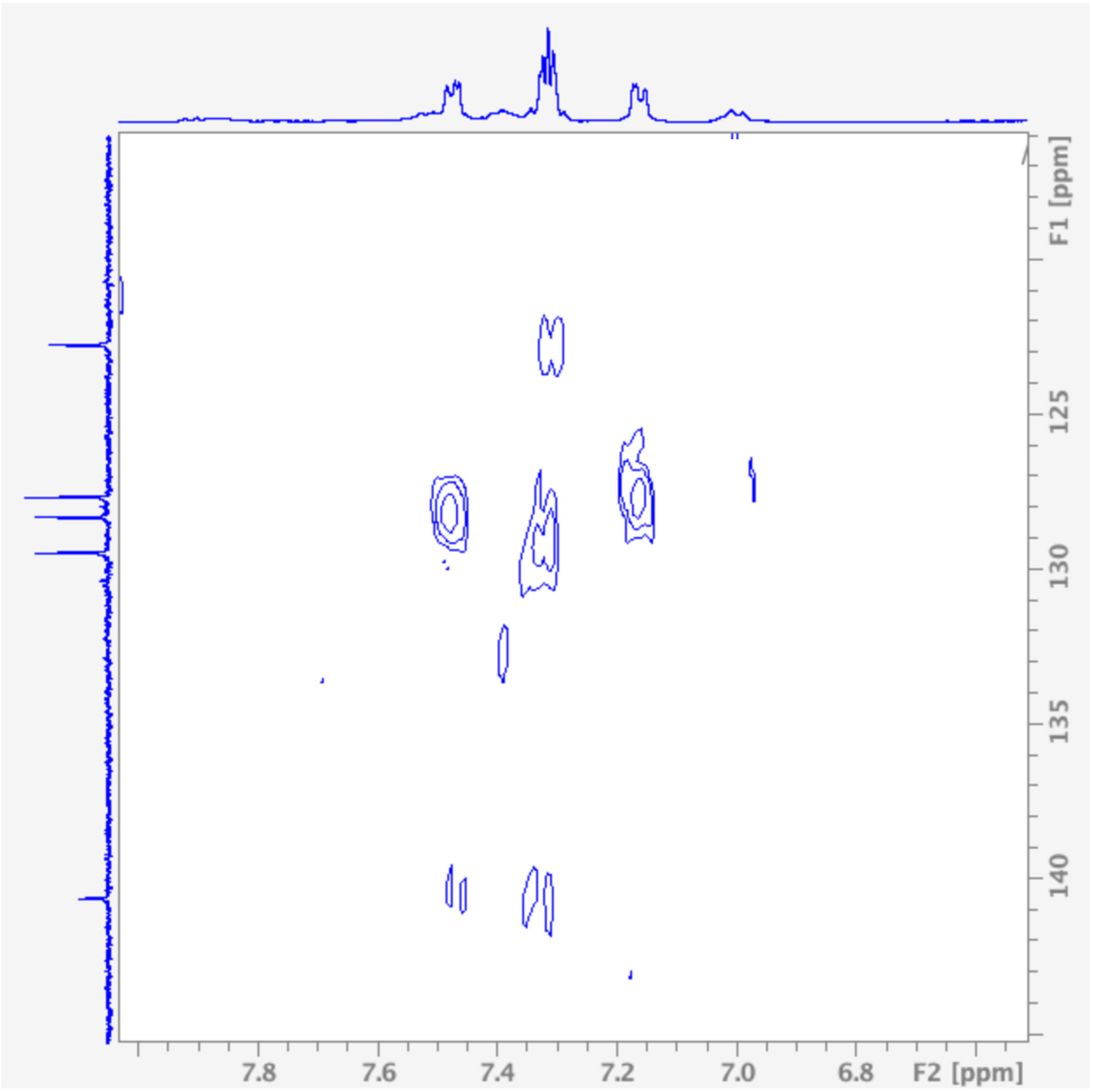
HMBC




F1 [ppm]
125
130
135
140
7
6
5
4
3
F2 [ppm]


F1 [ppm]
60
70
80
7
6
5
4
3
2
F2 [ppm]


F1 [ppm]
50
100
150
2.5
2.0
1.5
F2 [ppm]


F1 [ppm]
125
130
135
140
7.8
7.6
7.4
7.2
7.0
6.8
F2 [ppm]

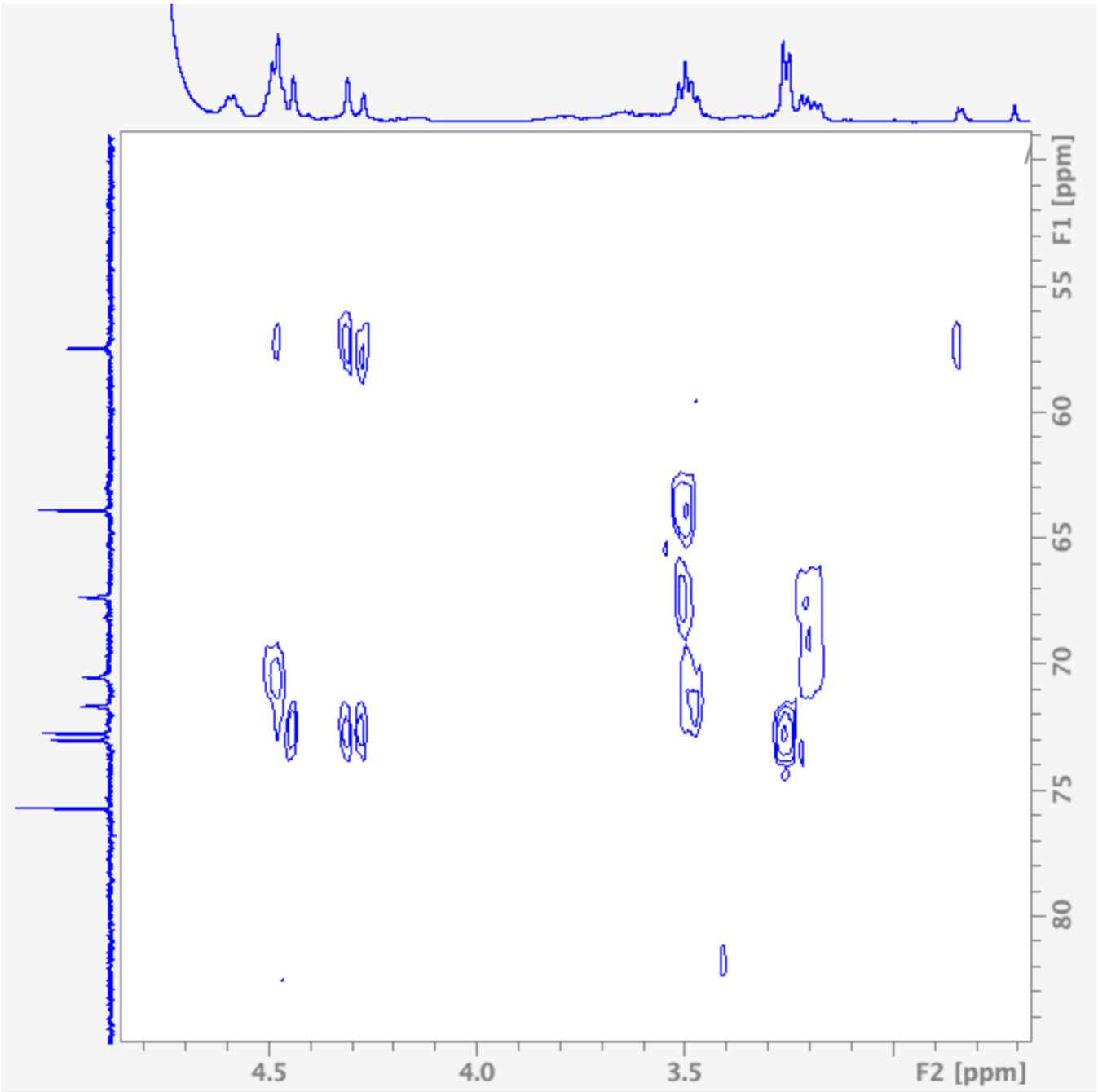
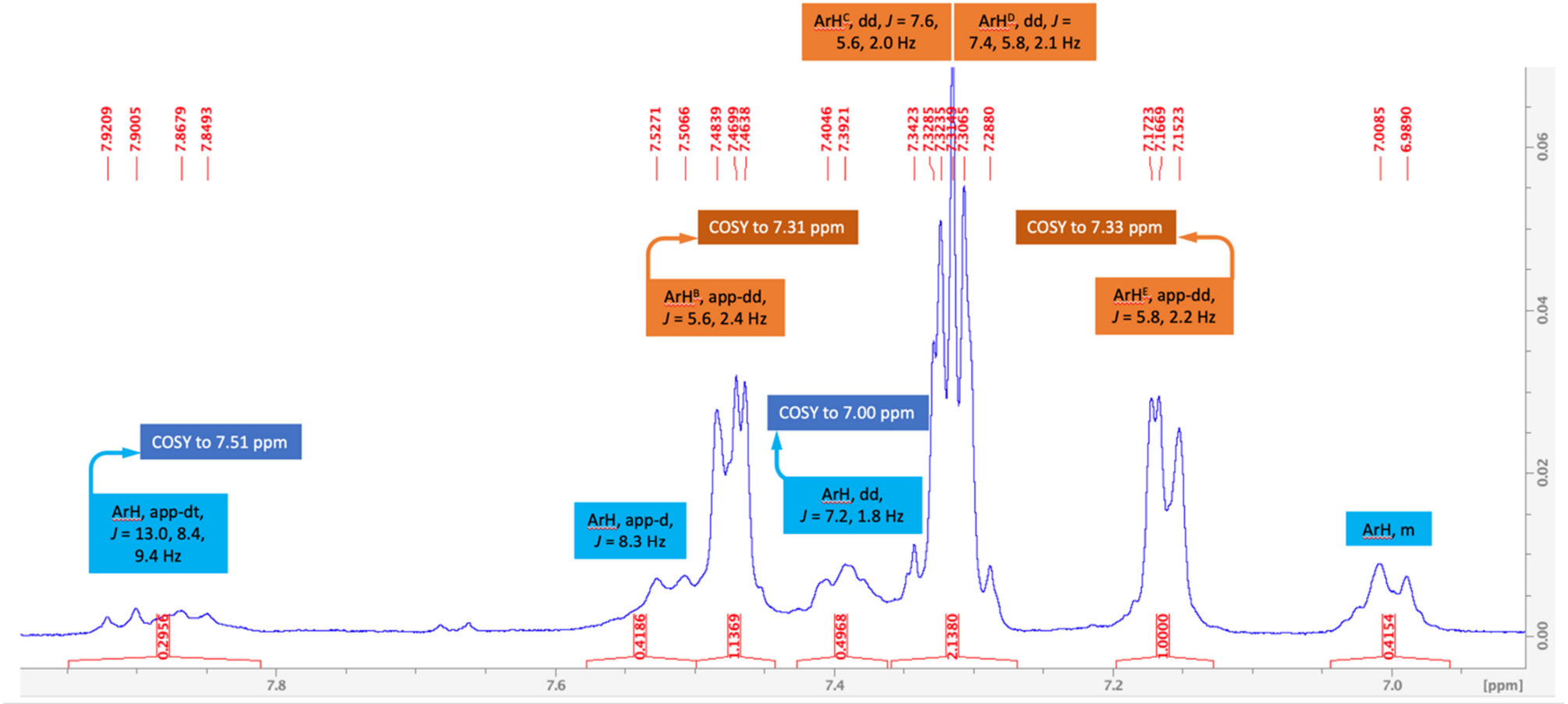
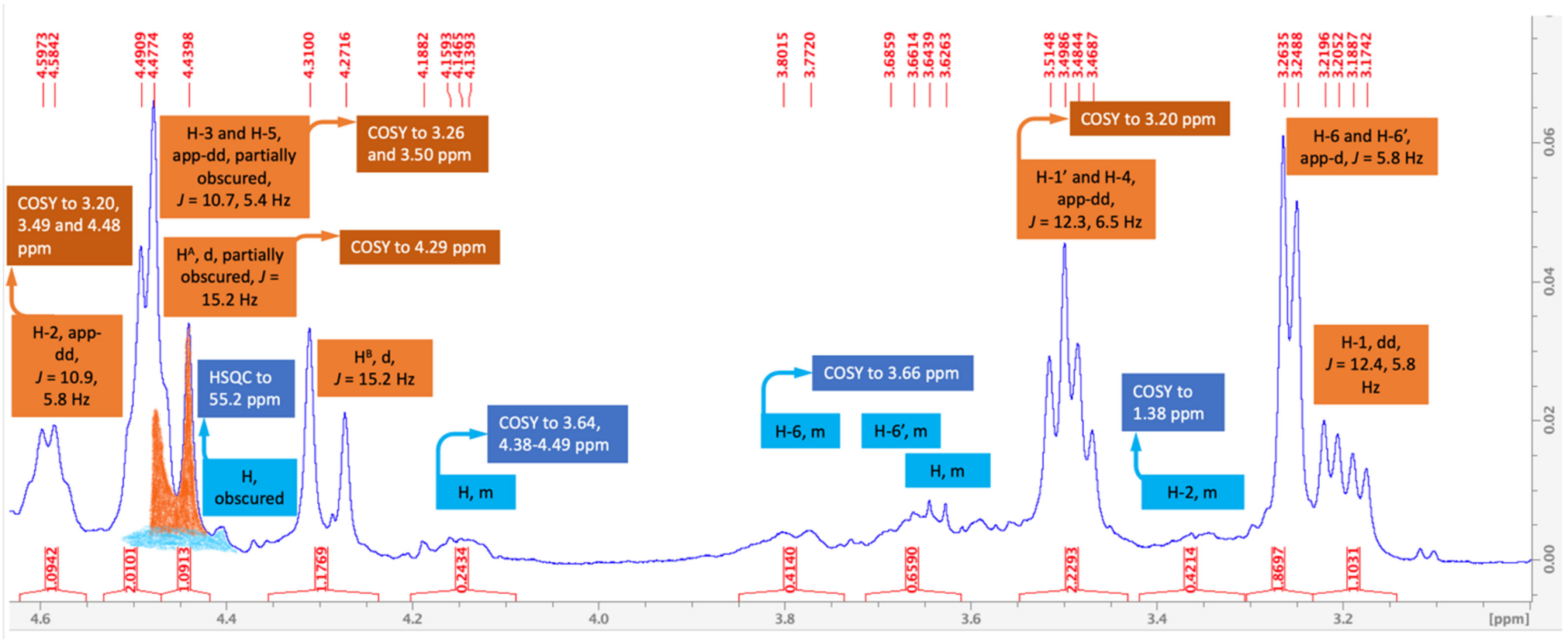

F1 [ppm]
55
60
65
70
75
80
4.5
4.0
3.5
F2 [ppm]

Figure S11. ^{1}H-NMR spectrum (400 MHz, D_2O) of final compound **ortho 4** with colour-coded signals, highlighting the boronic acid and boronate forms they belong to, with interpretation of the isolated signals and tentative interpretation of the overlapping ones. Namely, the orange designates the boronic acid form and indigo designates the boronate form. A) section 8.00 ppm to 6.90 ppm; B) section 4.65 ppm to 3.00 ppm; C) section 1.80 ppm to 1.10 ppm. Highlighted are also the principal COSY correlations to hydrogen atoms within the same spin systems.

A



B


4.5973
4.5842
4.4909
4.4774
4.4398
4.3100
4.2716
4.1882
4.1593
4.1465
4.1393
3.8015
3.7720
3.6859
3.6614
3.6439
3.6263
3.5148
3.4986
3.4844
3.4687
3.2635
3.2488
3.2196
3.2052
3.1887
3.1742
COSY to 3.20, 3.49 and 4.48 ppm
H-2, app-dd, J = 10.9, 5.8 Hz
H-3 and H-5, app-dd, partially obscured, J = 10.7, 5.4 Hz
COSY to 3.26 and 3.50 ppm
HA, d, partially obscured, J = 15.2 Hz
COSY to 4.29 ppm
HSQC to 55.2 ppm
H, obscured
HB, d, J = 15.2 Hz
COSY to 3.64, 4.38-4.49 ppm
H, m
COSY to 3.66 ppm
H-6, m
H-6', m
H, m
COSY to 3.20 ppm
H-1' and H-4, app-dd, J = 12.3, 6.5 Hz
COSY to 1.38 ppm
H-2, m
H-6 and H-6', app-d, J = 5.8 Hz
H-1, dd, J = 12.4, 5.8 Hz
1.0942
2.0101
1.0913
1.1769
0.2434
0.4140
0.6590
2.2293
0.4214
1.8697
1.1031
4.6
4.4
4.2
4.0
3.8
3.6
3.4
3.2
[ppm]
0.00
0.02
0.04
0.06

C

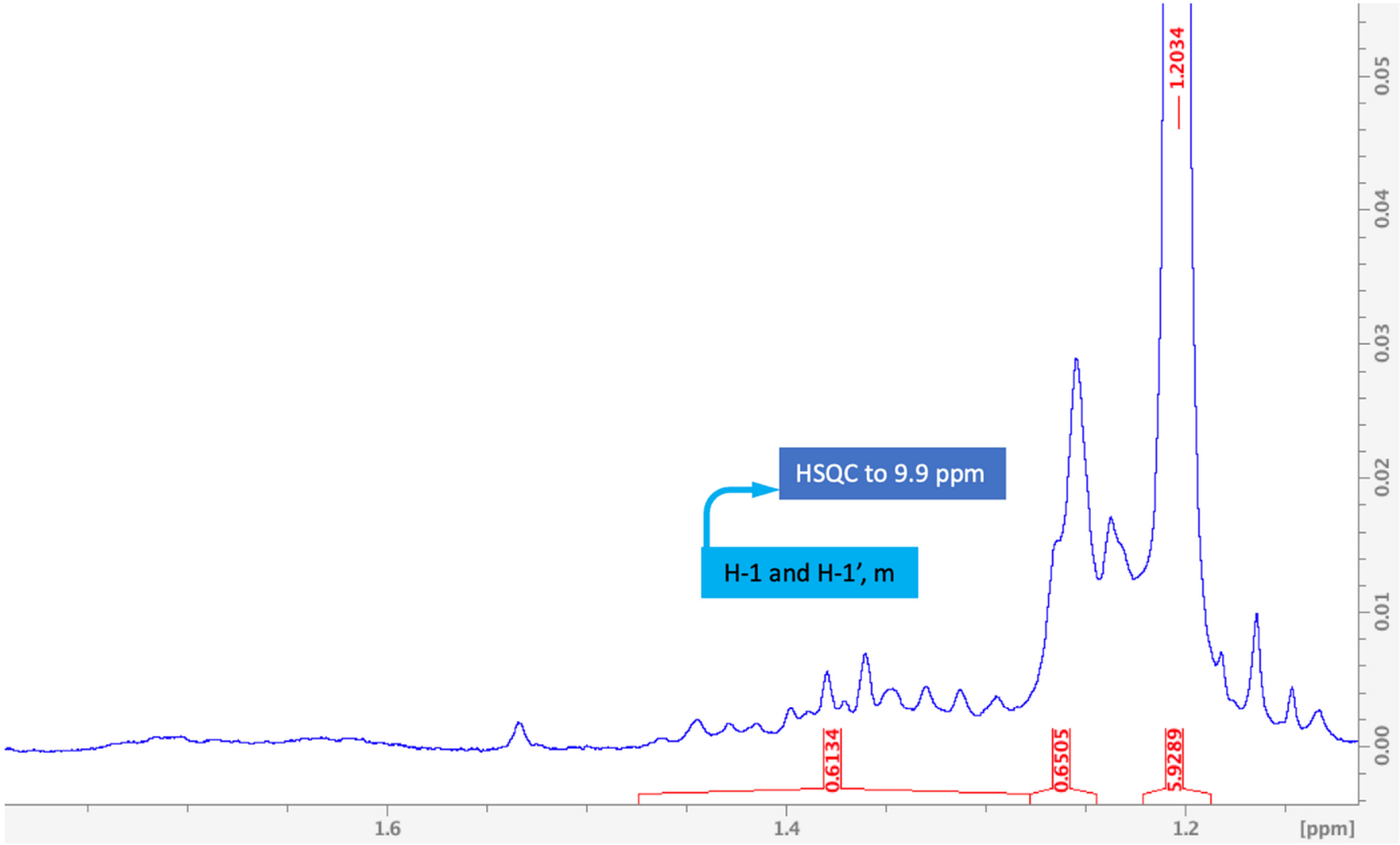
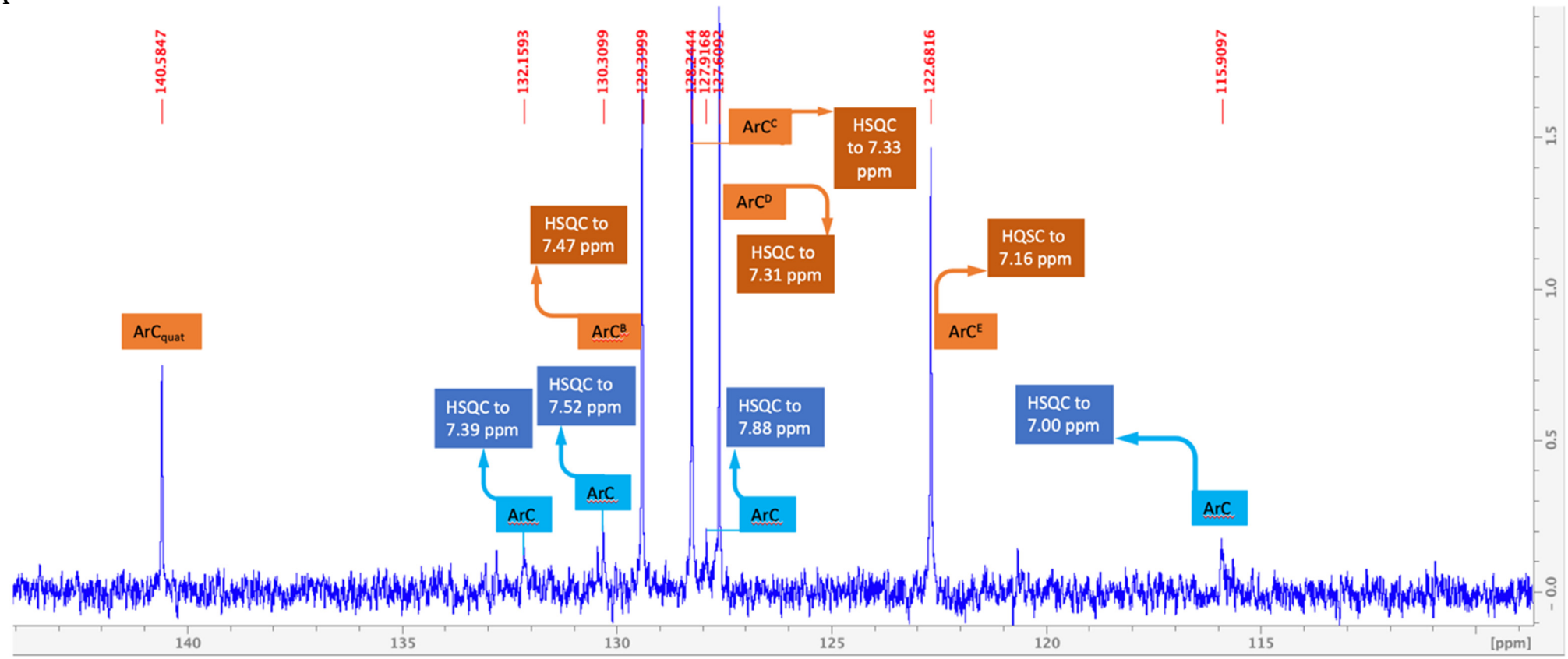

HSQC to 9.9 ppm
H-1 and H-1', m
1.2034
0.6134
0.6505
5.9289
1.6
1.4
1.2
[ppm]
0.00
0.01
0.02
0.03
0.04
0.05

Figure S12. ^{13}C-NMR spectrum (100 MHz, D_2O) sections of final compound **ortho 4** with colour-coded signals, highlighting the boronic acid and boronate forms they belong to, with interpretation of the isolated signals and tentative interpretation of the overlapping ones. Namely, the orange designates the boronic acid form and indigo designates the boronate form. A) section 144 ppm to 109 ppm; B) section 76 ppm to 44 ppm.

A



B

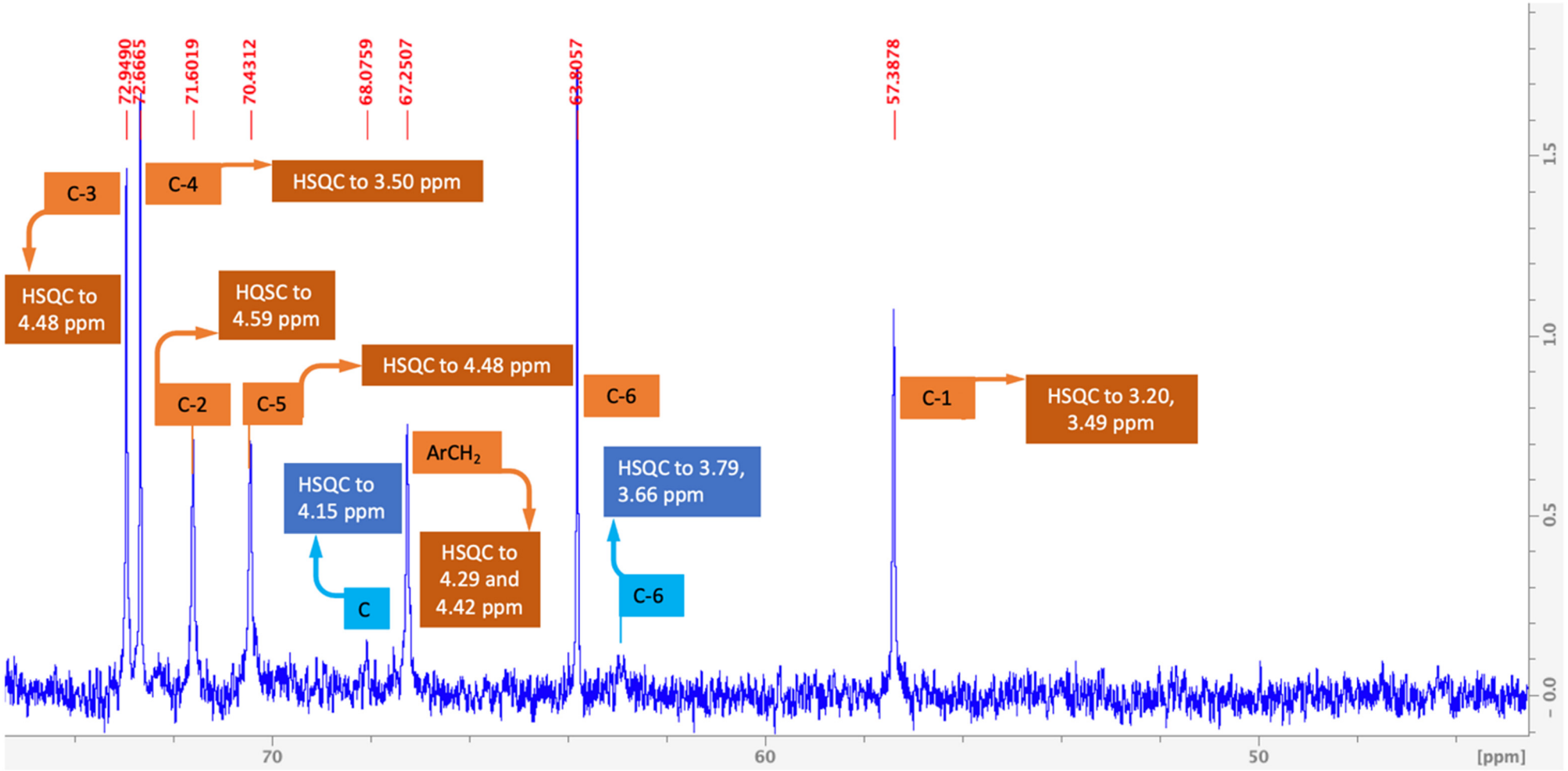
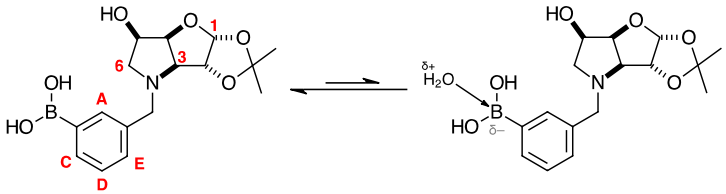

72.9490
72.6665
71.6019
70.4312
68.0759
67.2507
63.8057
57.3878
C-3
HSQC to 4.48 ppm
C-4
HSQC to 3.50 ppm
HQSC to 4.59 ppm
C-2
C-5
HSQC to 4.48 ppm
C-6
C-1
HSQC to 3.20, 3.49 ppm
ArCH2
HSQC to 4.15 ppm
HSQC to 3.79, 3.66 ppm
HSQC to 4.29 and 4.42 ppm
C
C-6
1.5
1.0
0.5
0.0
70
60
50
[ppm]

Figure S13. ^{1}H- (400 MHz), ^{13}C-NMR (100 MHz), ^{11}B-NMR (128 MHz), COSY and HSQC spectra of *N*-(3-methylphenyl boronic acid)-3,6-dideoxy-3,6-imino-1,2-*O*-isopropylidene-*α*-D-gulofuranose **meta 5** in MeOD.

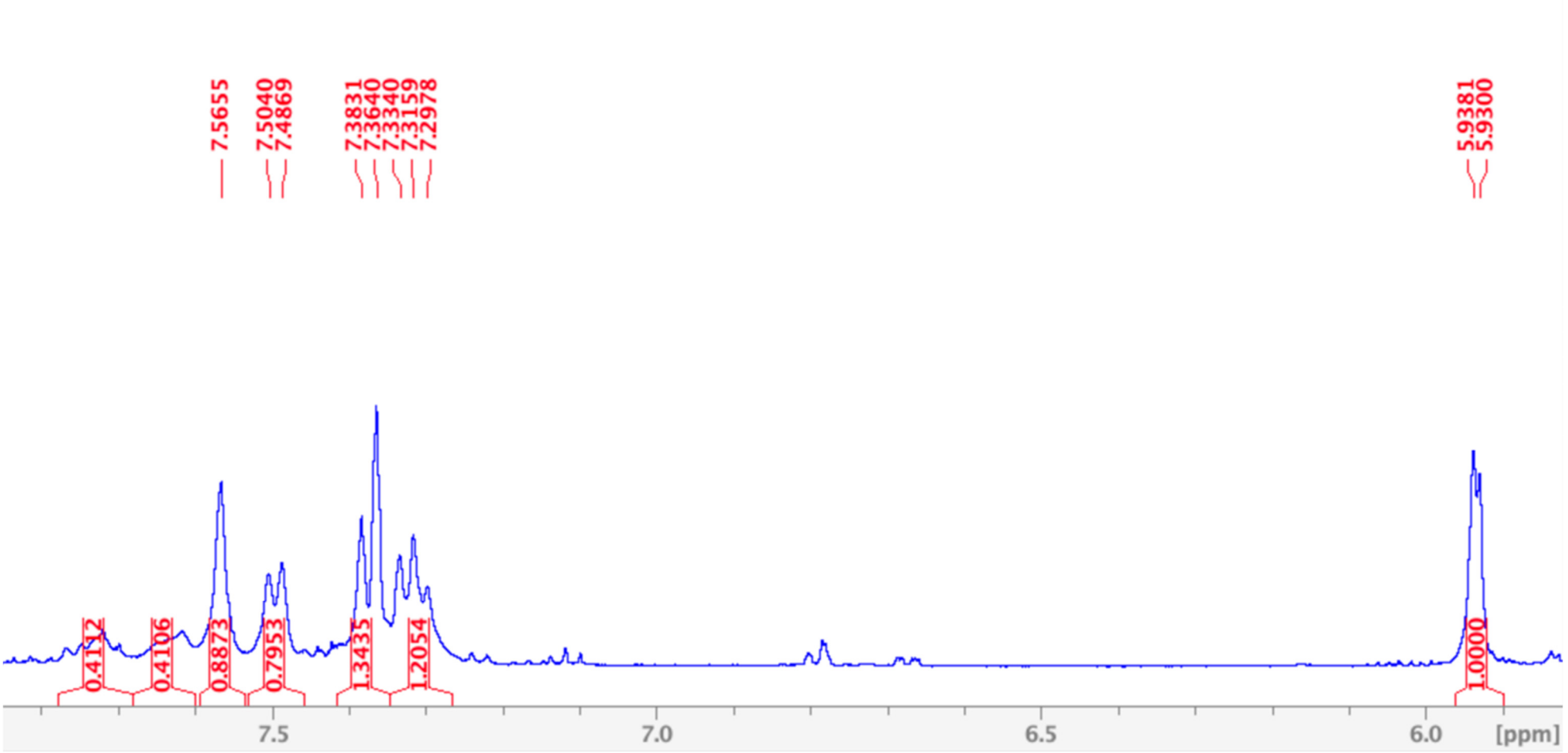


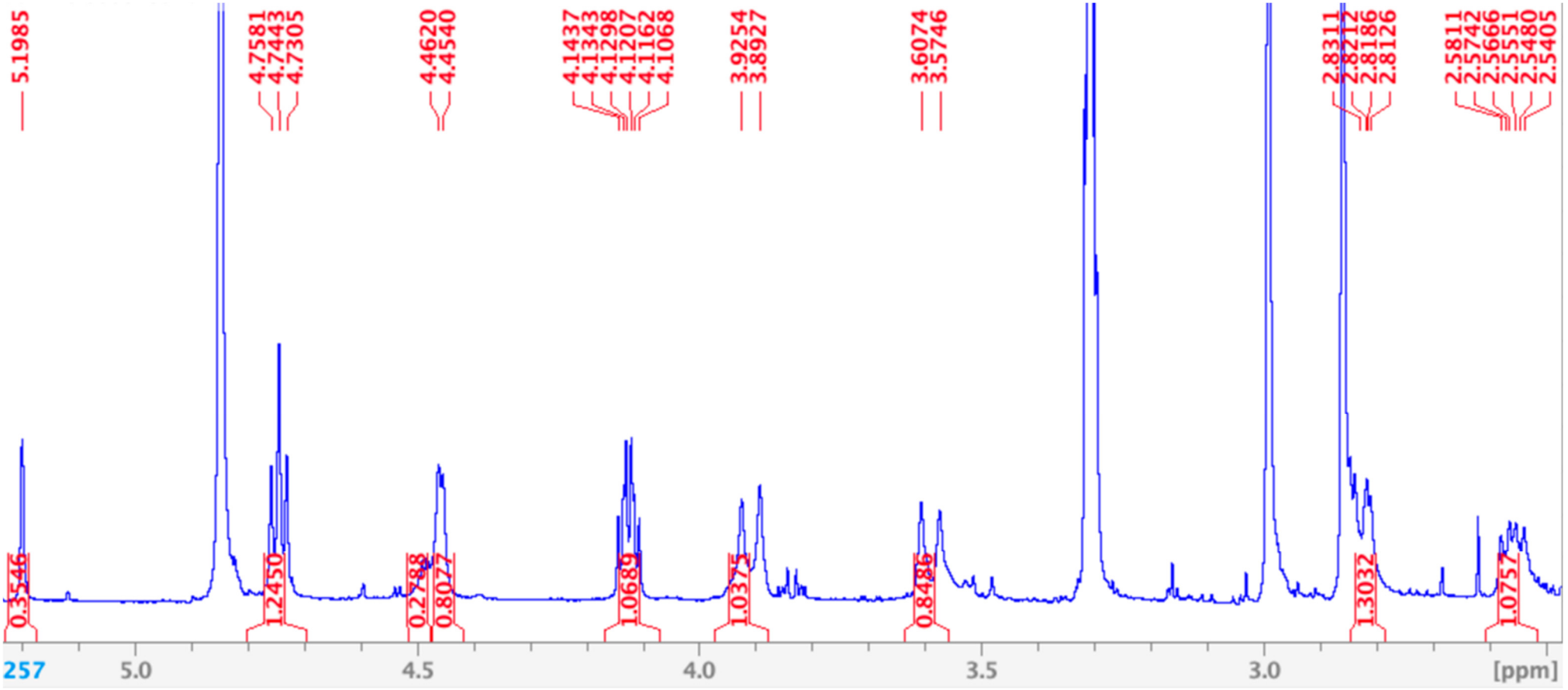
Boronic acid species. **δH**: 7.57 (1H, d, $J_{HA,HE}$ 1.5 Hz, ArH^{A}), 7.49 (1H, d, $J_{HC,HD}$ 7.2 Hz, ArH^{C}), 7.37 (1H, dt, $J_{HE,HD}$ 7.6 Hz, $J_{HE,HA}$ 1.5 Hz, ArH^{E}), 7.32 (1H, app-t, $J_{HD,HC\,=\,HD,HE}$ 7.3 Hz, ArH^{D}), 5.93 (1H, d, $J_{H\text{-}1,H\text{-}2}$ 3.3 Hz, H-1), 4.74 (1H, app-t, $J_{H\text{-}4,H\text{-}3\,=\,H\text{-}4,H\text{-}5}$ 5.5 Hz, H-4), 4.46 (1H, d, $J_{H\text{-}2,H\text{-}1}$ 3.3, H-2), 4.13 (1H, ddd, $J_{H\text{-}5,H\text{-}4}$ 5.6 Hz, $J_{H\text{-}5,H\text{-}6}$ 5.5 Hz, $J_{H\text{-}5,H\text{-}6'}$ 3.7 Hz, H-5), 3.91 (1H, d, $J_{Ha,Hb}$ 13.2 Hz, ArC*H*^{a}H^{b}), 3.59 (1H, d, $J_{Hb,Ha}$ 13.2 Hz, ArCH^{a}*H*^{b}), 3.30 (1H, d, partially obscured, $J_{H\text{-}3,H\text{-}4}$ 5.7 Hz, H-3), 2.83 (1H, dd, $J_{H\text{-}6',H\text{-}6}$ 10.6 Hz, $J_{H\text{-}6',H\text{-}5}$ 3.5 Hz, H-6'), 2.53 (1H, dd, $J_{H\text{-}6,H\text{-}6'}$ 10.3 Hz, $J_{H\text{-}6,H\text{-}5}$ 5.7 Hz, H-6), 1.44 (3H, s, CH_3 acetonide), 1.27 (3H, s, CH_3 acetonide); **δC**: 138.5 (ArC$_{quat}$), 135.2 (ArC^{A}), 133.4 (ArC^{C}), 131.4 (ArC^{E}), 128.7 (ArC^{D}), 113.4 (C$_{quat}$ acetonide), 108.8 (C-1), 85.8 (C-2), 84.8 (C-4), 73.4 (C-3), 71.2 (C-5), 60.3 (C-6), 59.9 (CH_2Ar), 27.8, 27.0 (2 x CH_3, acetonide); ArC^{B}-B was not discernible.

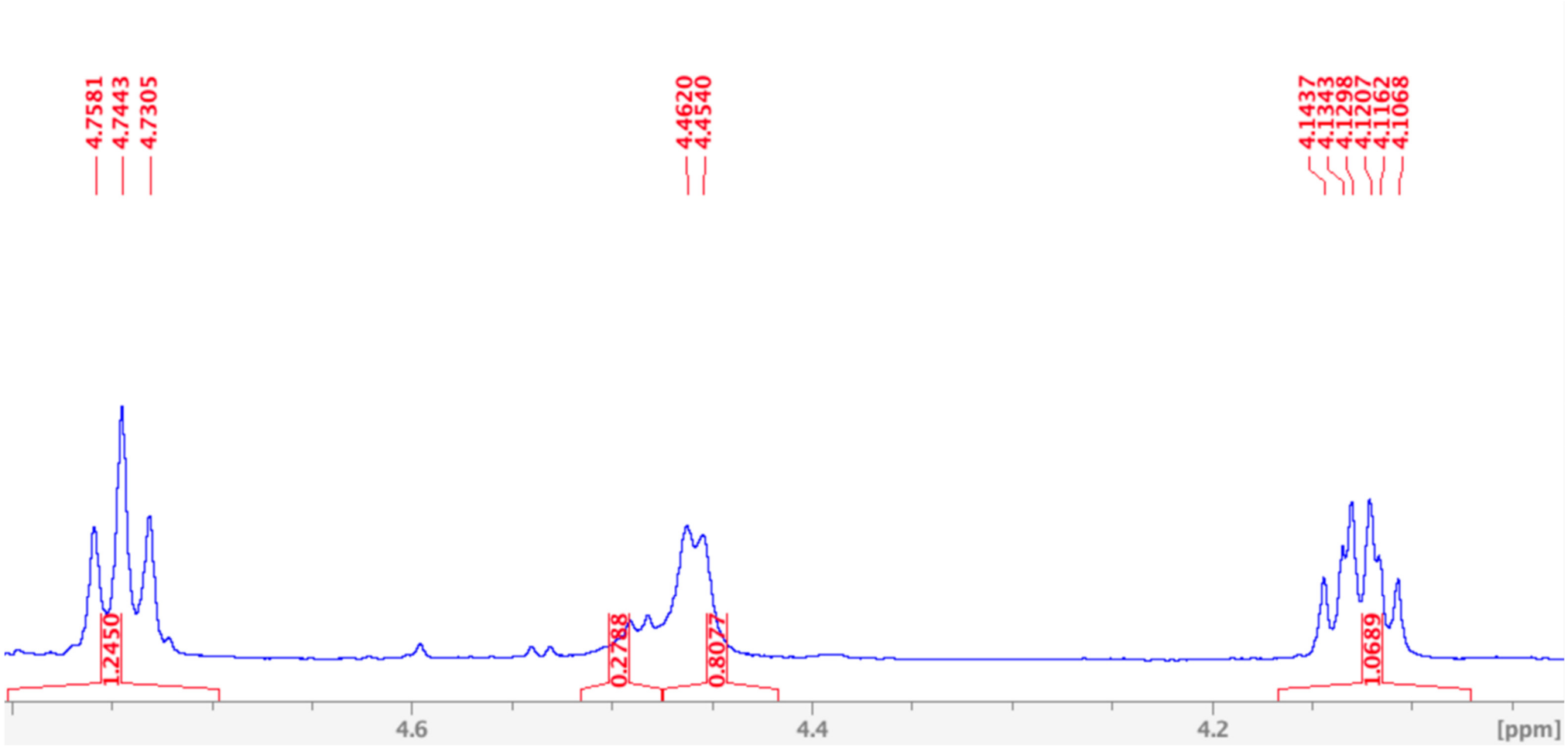
Boronate species (not all signals are visible). **δH**: 7.12 (1H, td, $J_{HC,HD}$ 7.6 Hz, $J_{HC,HE}$ 0.8 Hz, ArH^{C}), 6.79 (1H, dd, $J_{HE,HD}$ 7.6 Hz, $J_{HE,HA}$ 1.5 Hz, ArH^{E}), 7.32 (1H, ddd, $J_{HD,HE}$ 8.2 Hz, $J_{HD,HC}$ 2.4 Hz, $J_{HD,HA}$ 0.9 Hz, ArH^{D}), 5.83 (1H, d, $J_{H\text{-}1,H\text{-}2}$ 3.3 Hz, H-1), 4.54 (1H, d, $J_{H\text{-}4,H\text{-}3}$ 3.5 Hz, H-4), 4.49 (1H, d, $J_{H\text{-}2,H\text{-}1}$ 3.4, H-2), 3.84, 3.83 (2 x 1H, 2 x d, *J* 12.7 Hz, ArCH^{a}H^{b}), 1.61 (1H, dd, $J_{H\text{-}6,H\text{-}6'}$ 13.0 Hz, $J_{H\text{-}6,H\text{-}5}$ 6.6 Hz, H-6), 1.59 (1H, dd, $J_{H\text{-}6',H\text{-}6}$ 12.8 Hz, $J_{H\text{-}6',H\text{-}5}$ 6.6 Hz, H-6'), 1.44 (3H, s, partially obscured, CH_3 acetonide), 1.32 (3H, s, CH_3 acetonide); **δC**: not discernible.

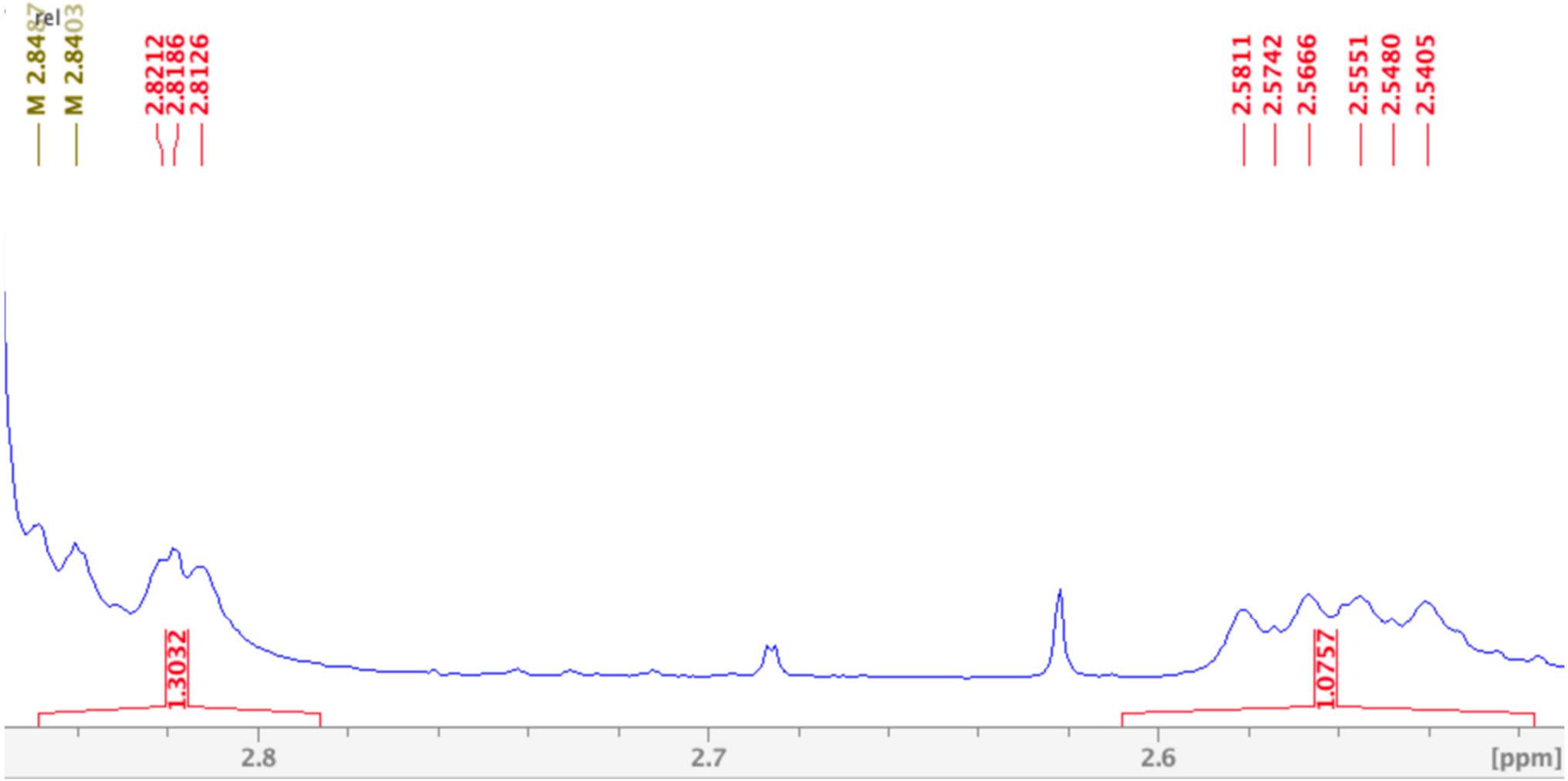
δB: 28.6 (sharp, integration: 8.6), 18.6 (sharp, integration: 1.0).

^{1}H-NMR

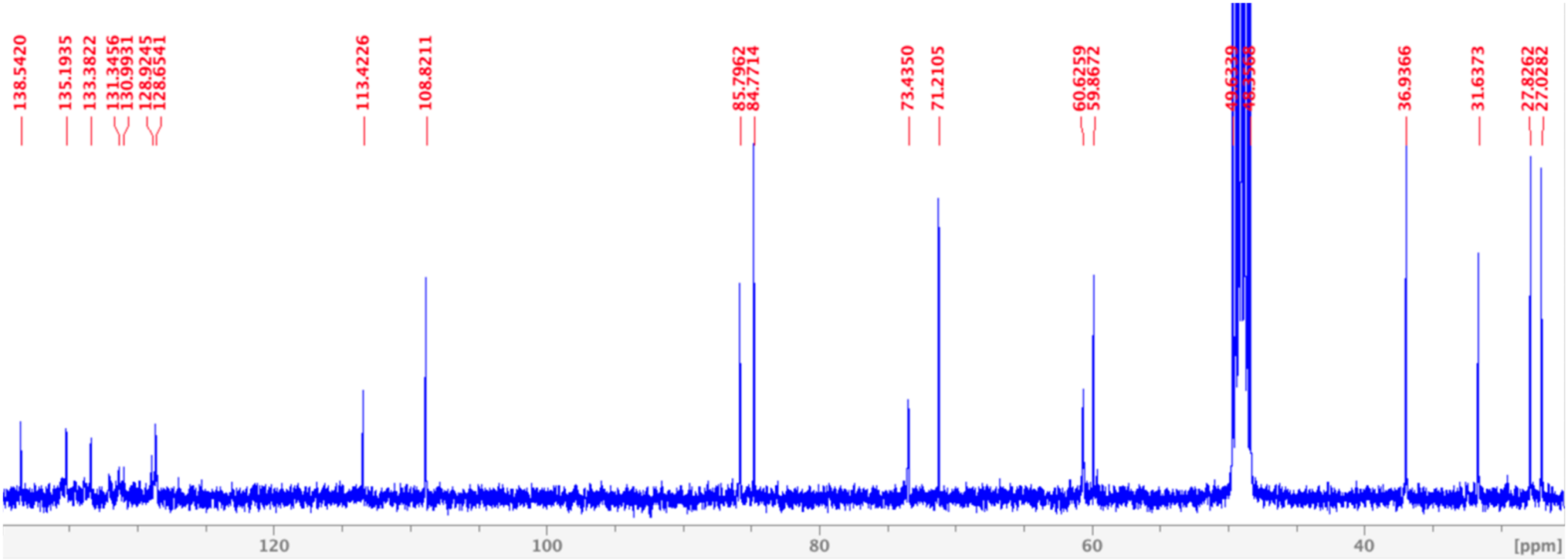

7.5655
7.5040
7.4869
7.3831
7.3640
7.3340
7.3159
7.2978
5.9381
5.9300
0.4112
0.4106
0.8873
0.7953
1.3435
1.2054
1.0000
7.5
7.0
6.5
6.0
[ppm]

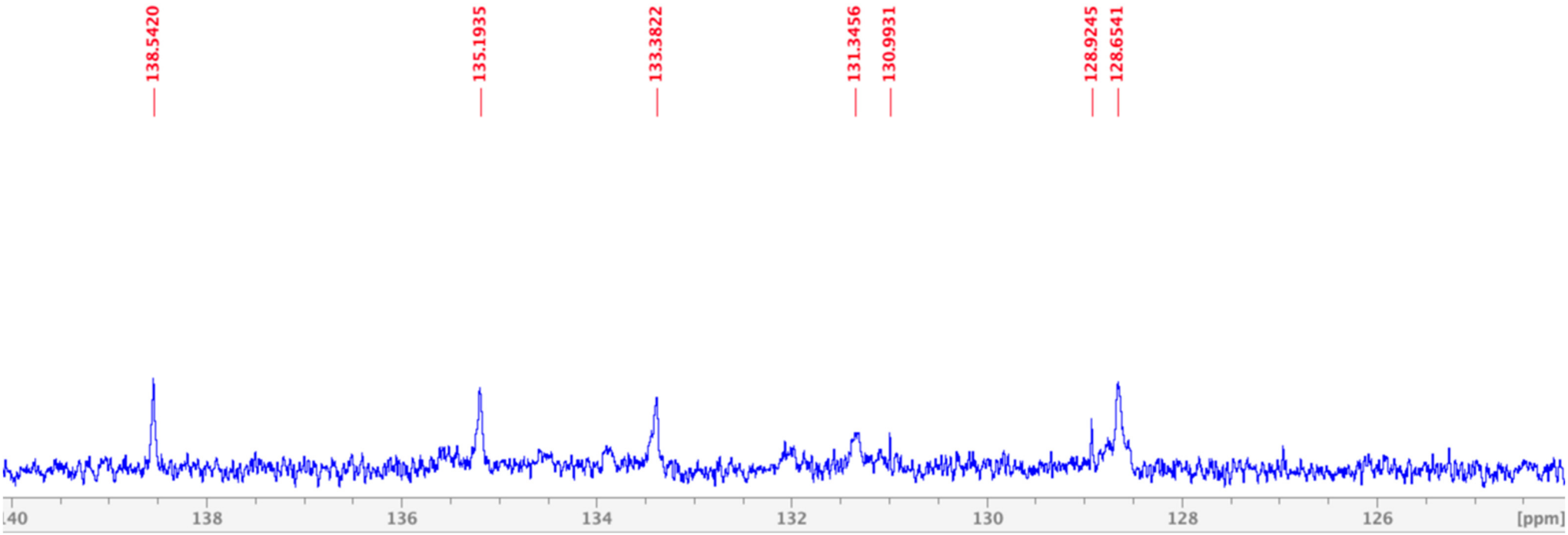

5.1985
4.7581
4.7443
4.7305
4.4620
4.4540
4.1437
4.1343
4.1298
4.1207
4.1162
4.1068
3.9254
3.8927
3.6074
3.5746
2.8311
2.8212
2.8186
2.8126
2.5811
2.5742
2.5666
2.5551
2.5480
2.5405
0.3546
1.2450
0.2788
0.8077
1.0689
1.0375
0.8486
1.3032
1.0757
257
5.0
4.5
4.0
3.5
3.0
[ppm]


4.7581
4.7443
4.7305
4.4620
4.4540
4.1437
4.1343
4.1298
4.1207
4.1162
4.1068
1.2450
0.2788
0.8077
1.0689
4.6
4.4
4.2
[ppm]

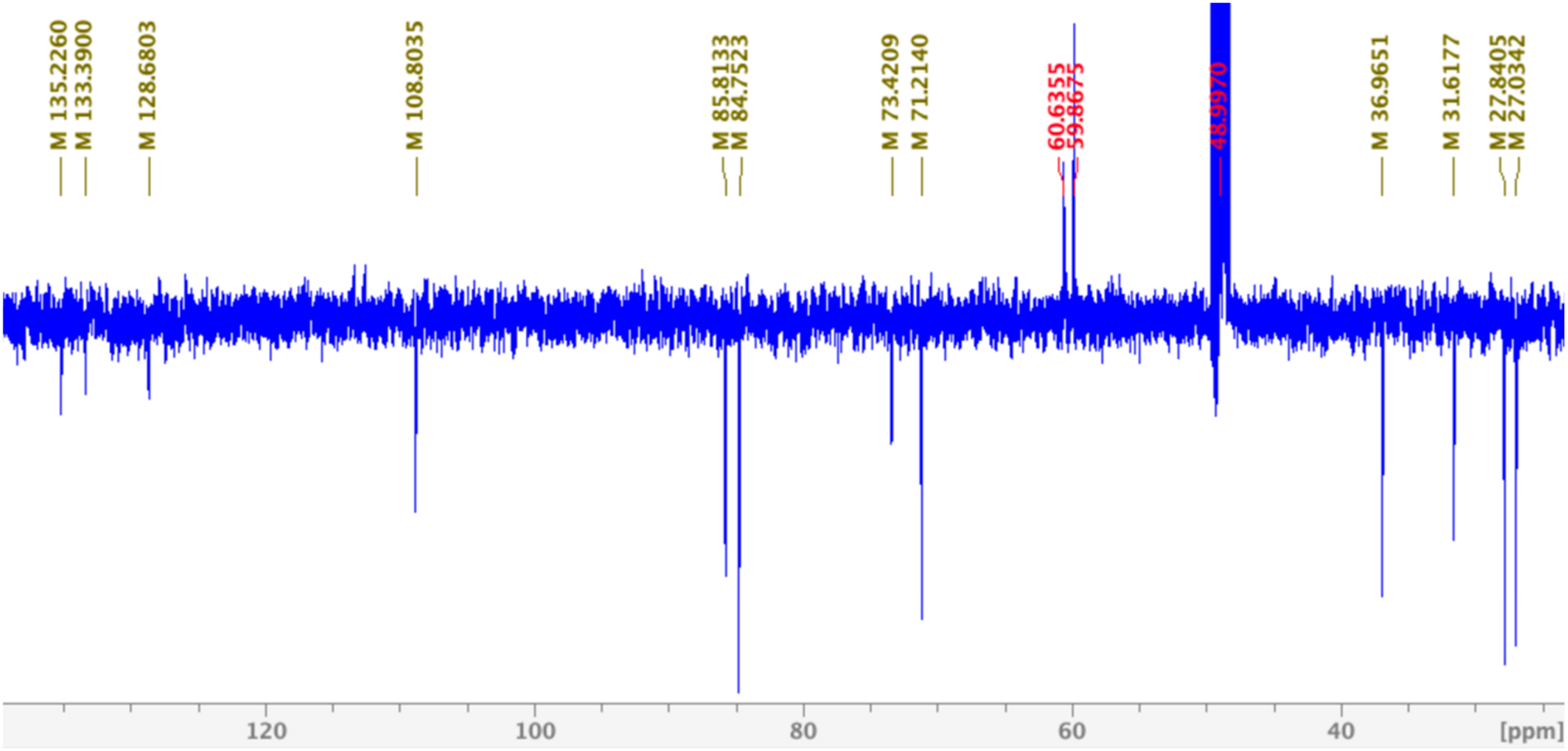

rel
M 2.84487
M 2.84403
2.8212
2.8186
2.8126
2.5811
2.5742
2.5666
2.5551
2.5480
2.5405
1.3032
1.0757
2.8
2.7
2.6
[ppm]

^{13}C-NMR

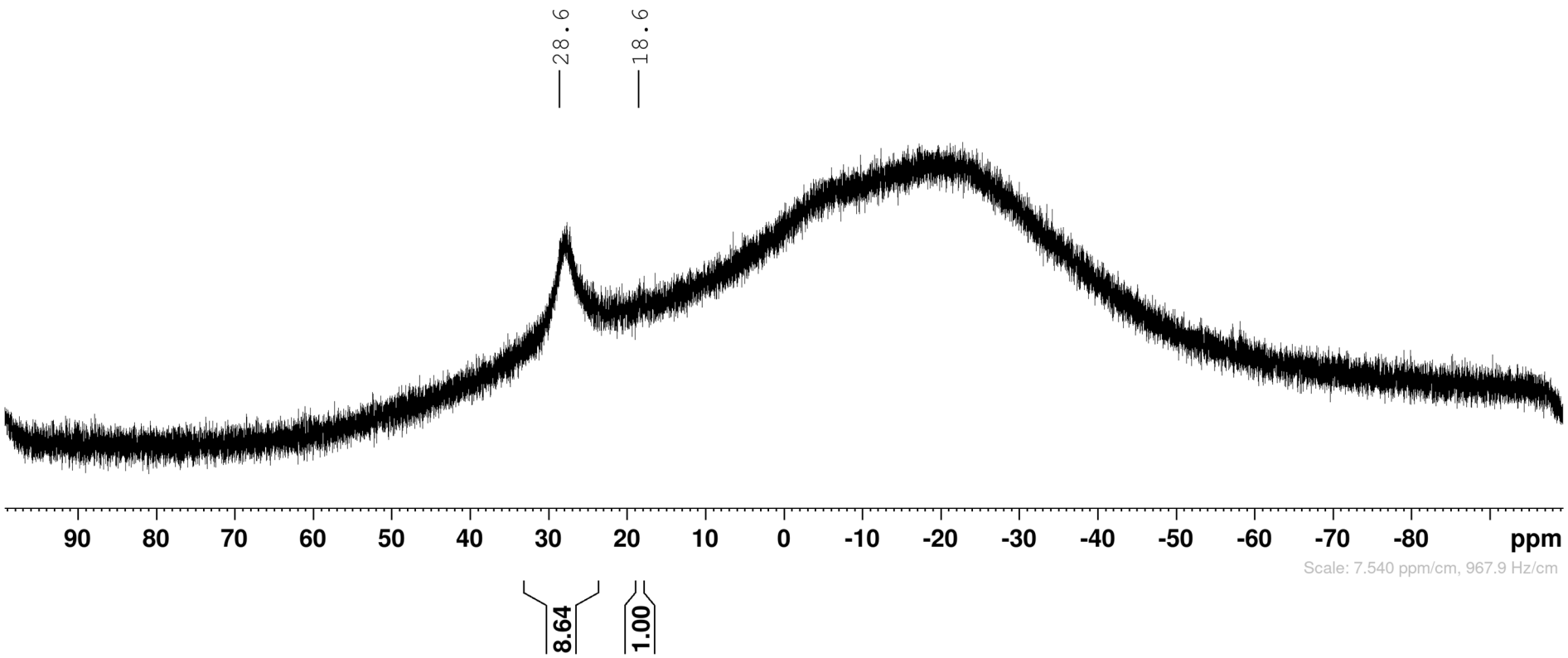

138.5420
135.1935
133.3822
131.3456
130.9931
128.9245
128.6541
113.4226
108.8211
85.7962
84.7714
73.4350
71.2105
60.6259
59.8672
49.6229
48.1568
36.9366
31.6373
27.8262
27.0282
120
100
80
60
40
[ppm]


138.5420
135.1935
133.3822
131.3456
130.9931
128.9245
128.6541
40
138
136
134
132
130
128
126
[ppm]

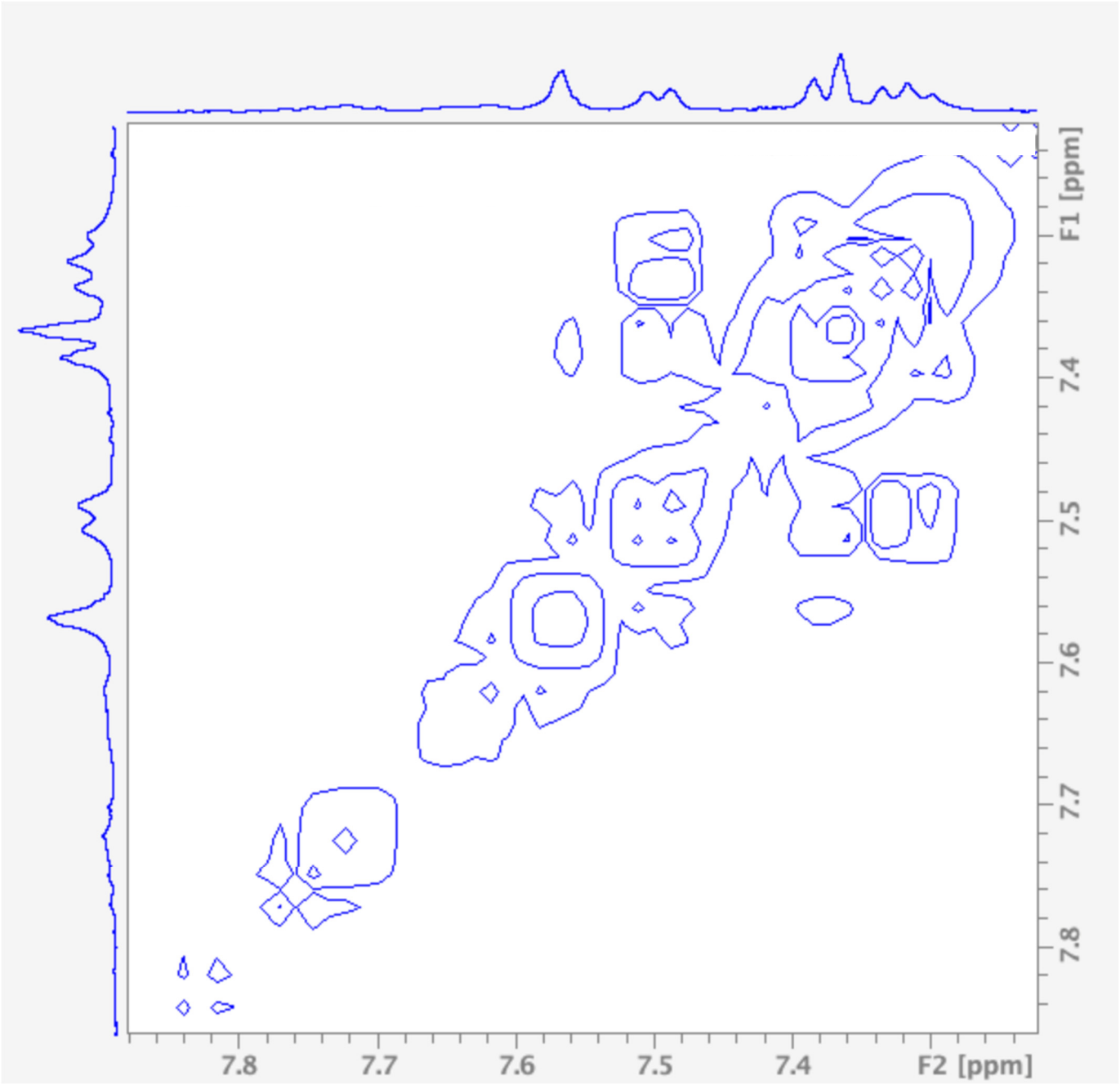
DEPT

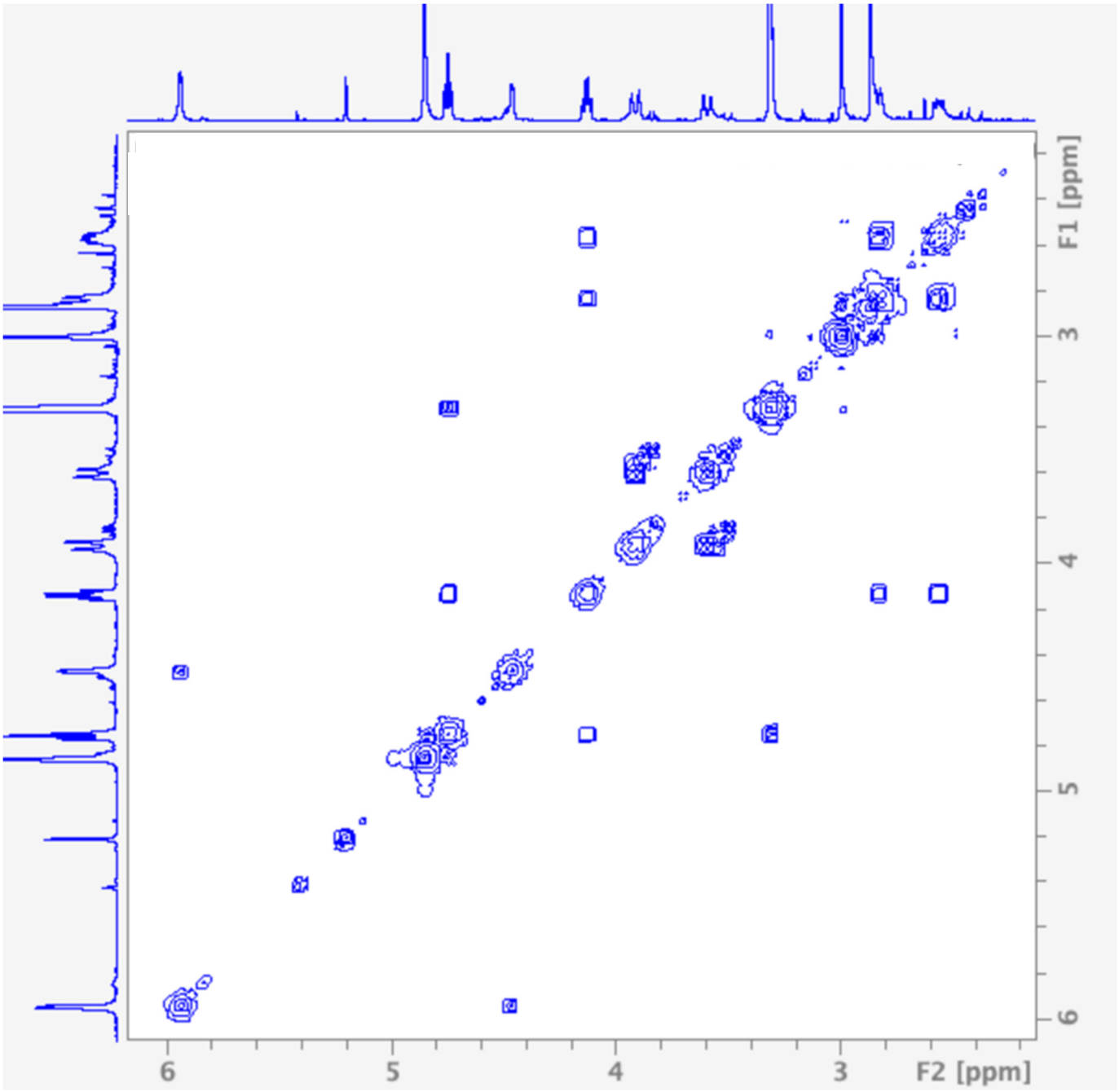

M 135.2260
M 133.3900
M 128.6803
M 108.8035
M 85.8133
M 84.7523
M 73.4209
M 71.2140
60.6355
59.8675
48.9970
M 36.9651
M 31.6177
M 27.8405
M 27.0342
120
100
80
60
40
[ppm]

^{11}B-NMR


28.6
18.6
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
ppm
Scale: 7.540 ppm/cm, 967.9 Hz/cm
8.64
1.00

COSY




F1 [ppm]
3
4
5
6
6
5
4
3
F2 [ppm]

HSQC

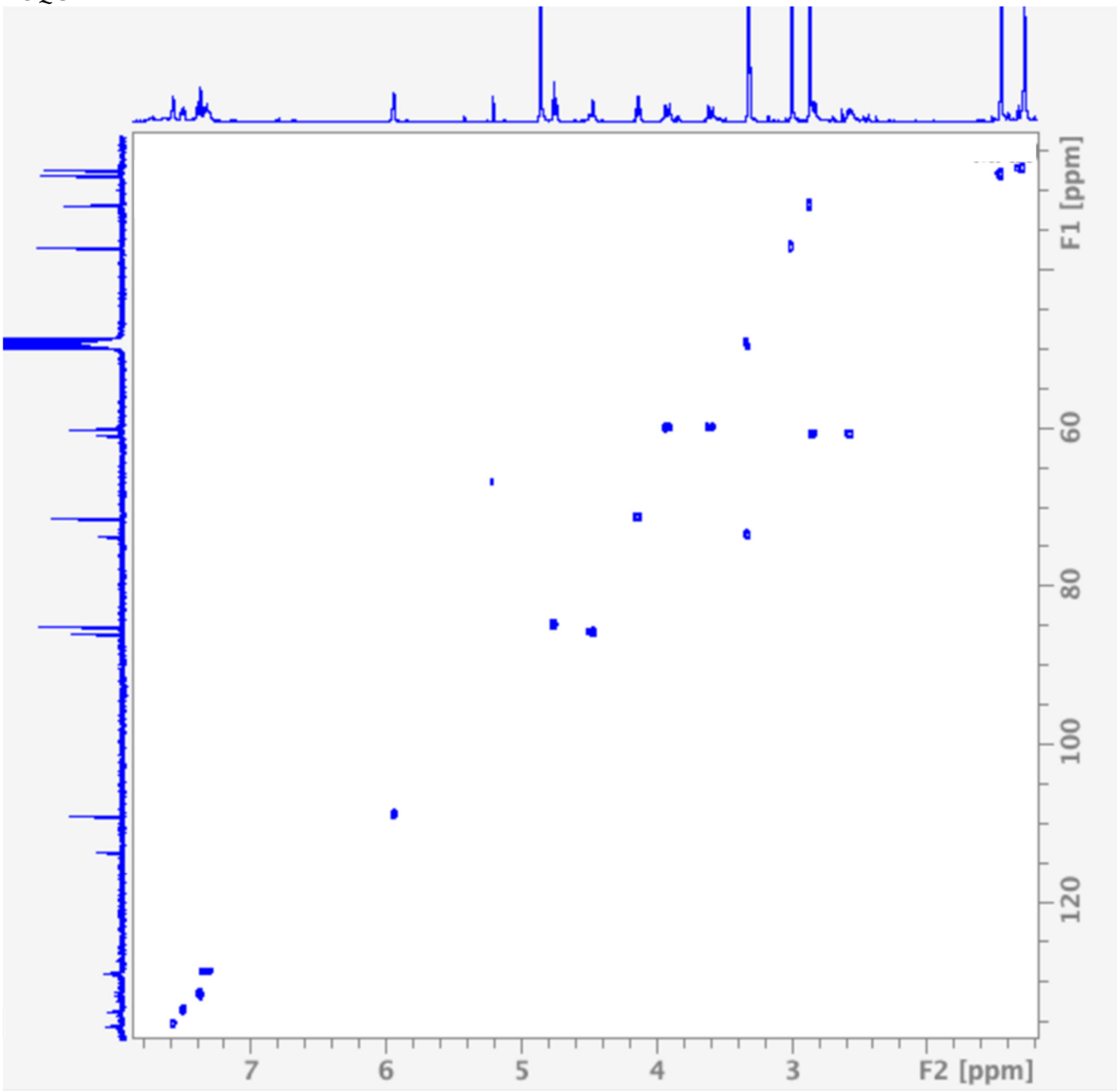
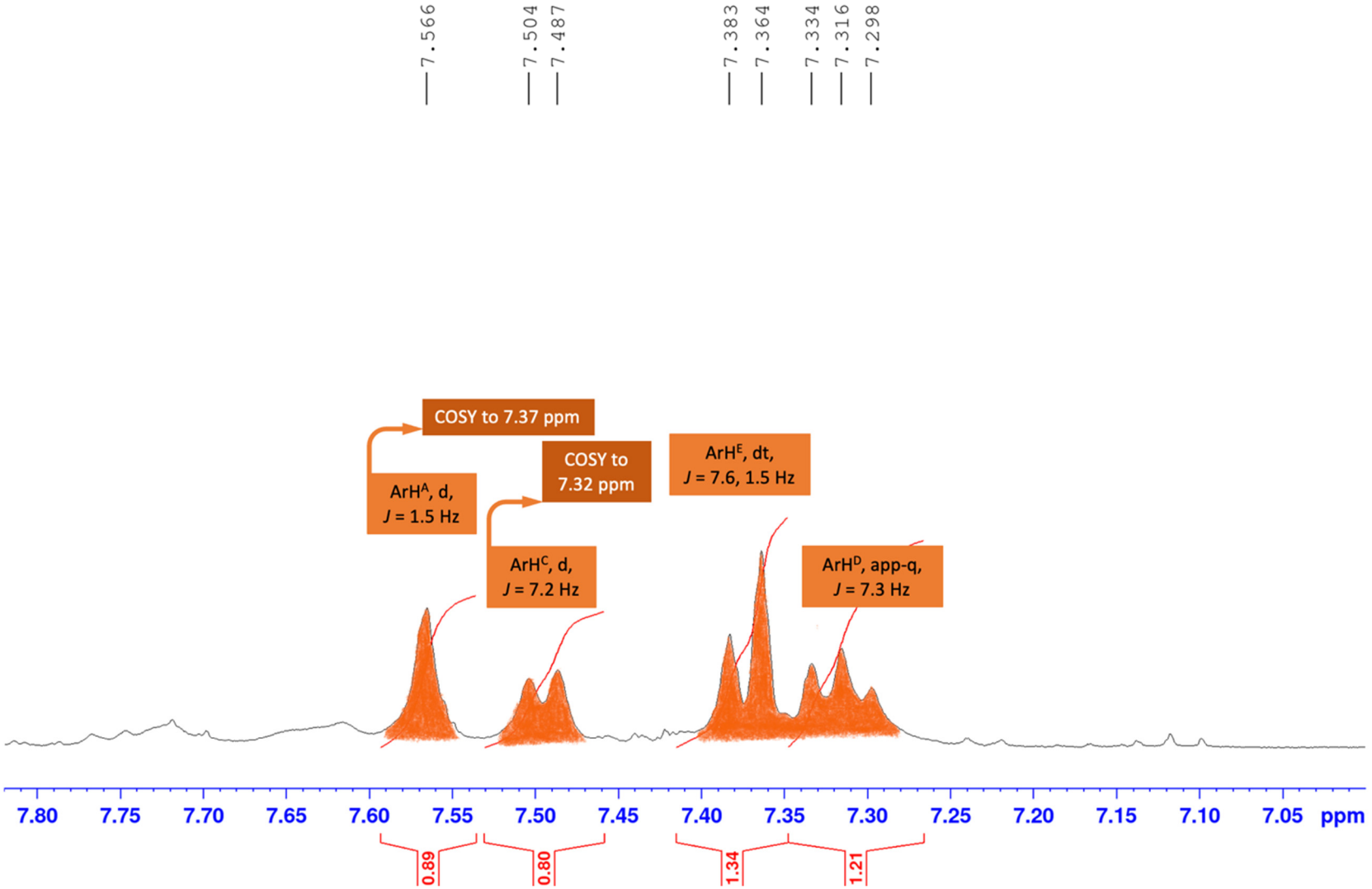
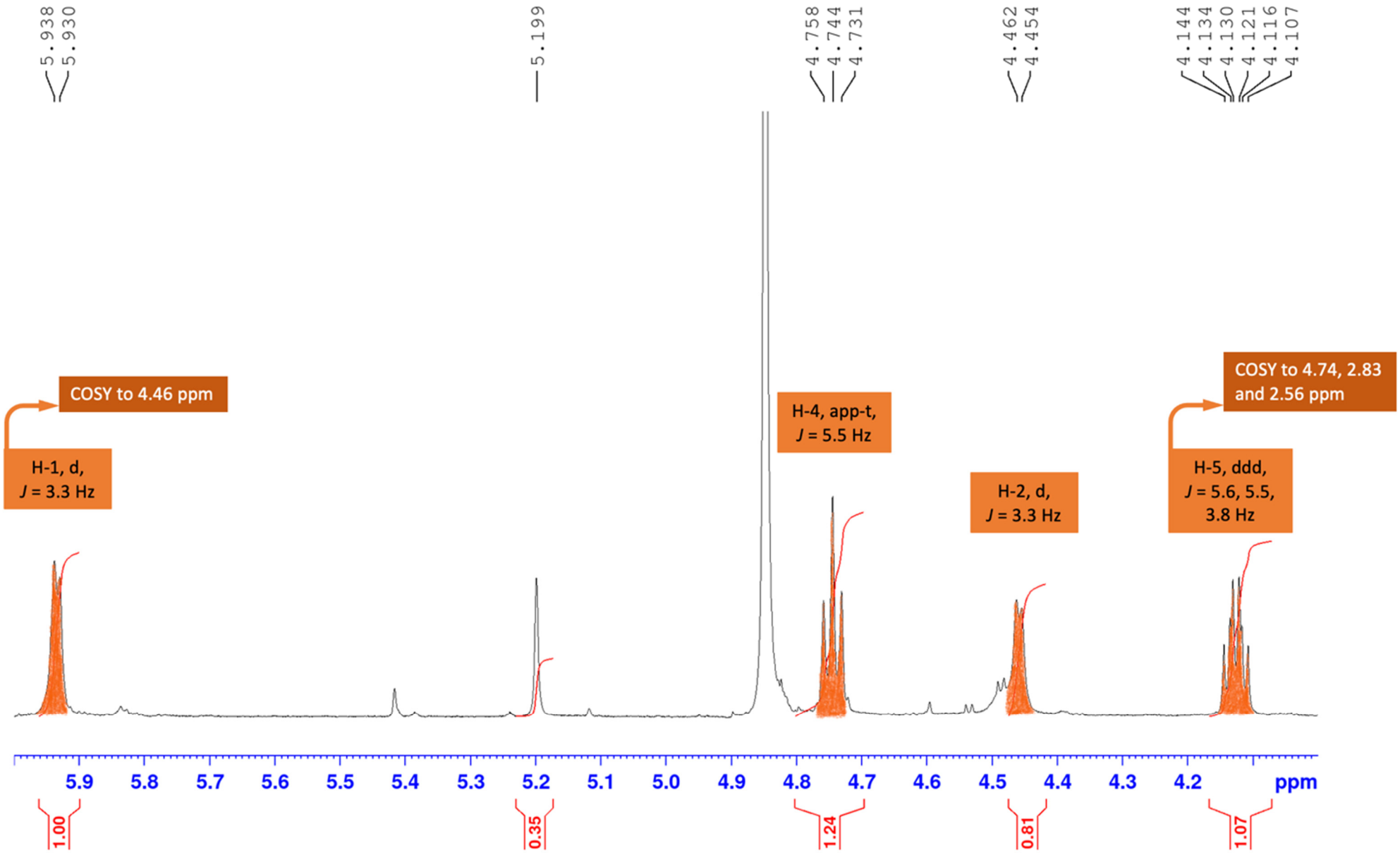


Figure S14. [1]H-NMR spectrum (400 MHz, MeOD) of compound **meta 5** with colour-coded signals, highlighting the boronic acid form they belong to, with interpretation of the isolated signals and tentative interpretation of the overlapping ones. Namely, the orange designates the boronic acid form. A) section 7.82 ppm to 7.00 ppm; B) section 6.00 ppm to 4.00 ppm; C) section 4.00 ppm to 2.50 ppm; D) section 1.50 ppm to 1.00 ppm. Highlighted are also the principal COSY correlations to hydrogen atoms within the same spin systems.

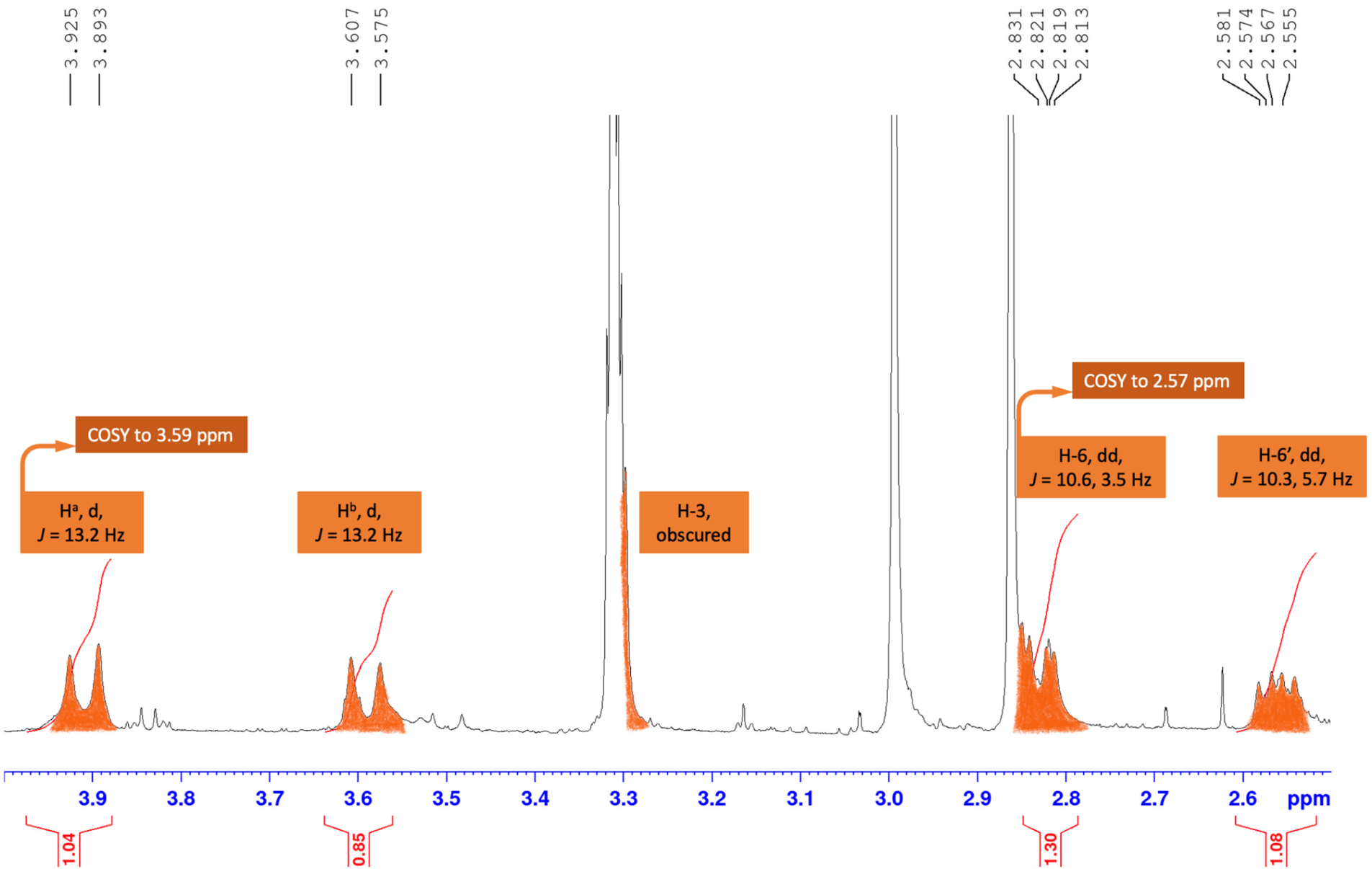
A



B


5.938
5.930
5.199
4.758
4.744
4.731
4.462
4.454
4.144
4.134
4.130
4.121
4.116
4.107
COSY to 4.46 ppm
H-1, d,
J = 3.3 Hz
H-4, app-t,
J = 5.5 Hz
H-2, d,
J = 3.3 Hz
COSY to 4.74, 2.83
and 2.56 ppm
H-5, ddd,
J = 5.6, 5.5,
3.8 Hz
5.9
5.8
5.7
5.6
5.5
5.4
5.3
5.2
5.1
5.0
4.9
4.8
4.7
4.6
4.5
4.4
4.3
4.2
ppm
1.00
0.35
1.24
0.81
1.07

C


3.925
3.893
3.607
3.575
2.831
2.821
2.819
2.813
2.581
2.574
2.567
2.555
COSY to 3.59 ppm
Hᵃ, d,
J = 13.2 Hz
Hᵇ, d,
J = 13.2 Hz
H-3,
obscured
COSY to 2.57 ppm
H-6, dd,
J = 10.6, 3.5 Hz
H-6', dd,
J = 10.3, 5.7 Hz
3.9
3.8
3.7
3.6
3.5
3.4
3.3
3.2
3.1
3.0
2.9
2.8
2.7
2.6
ppm
1.04
0.85
1.30
1.08

D

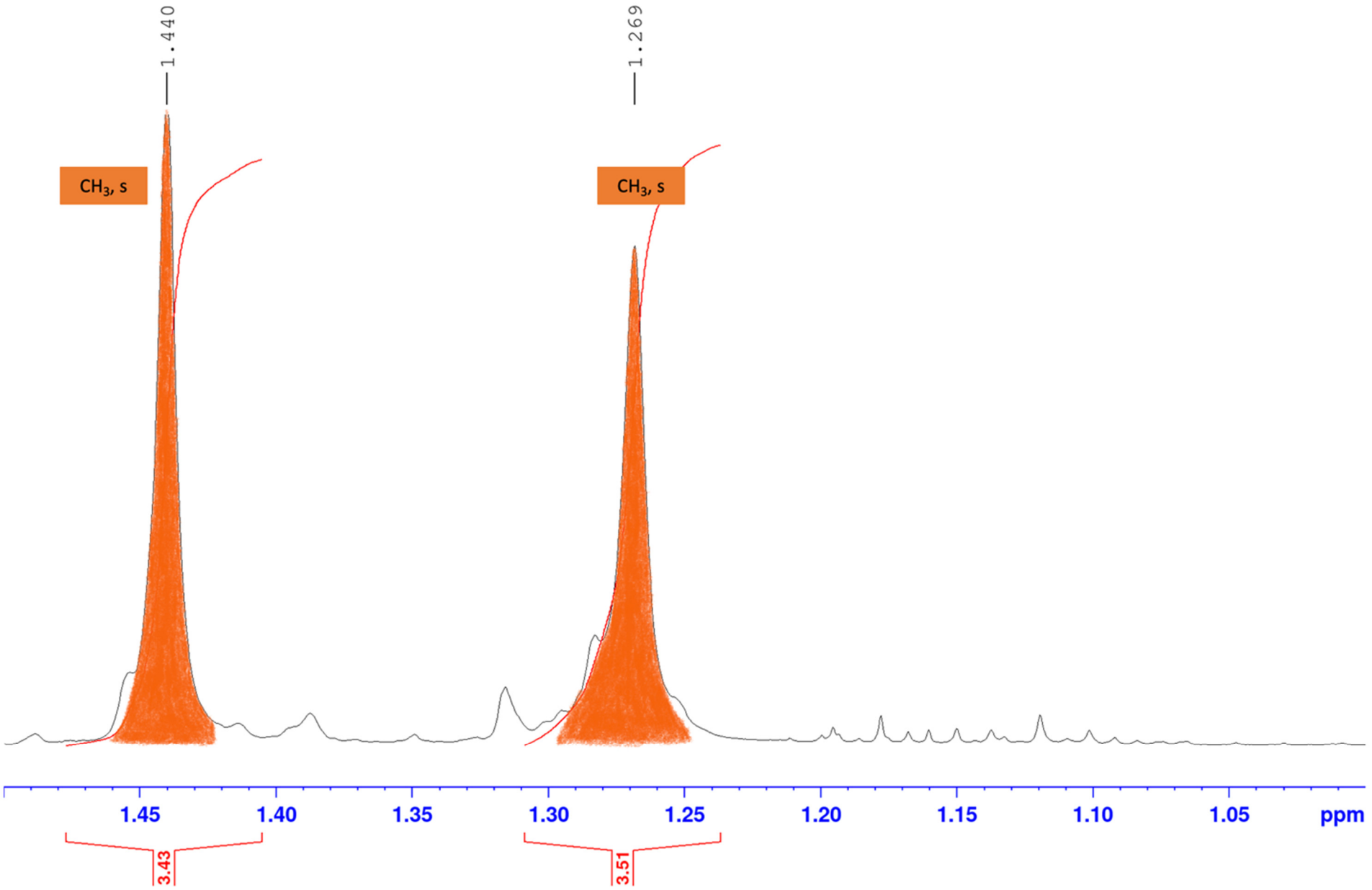
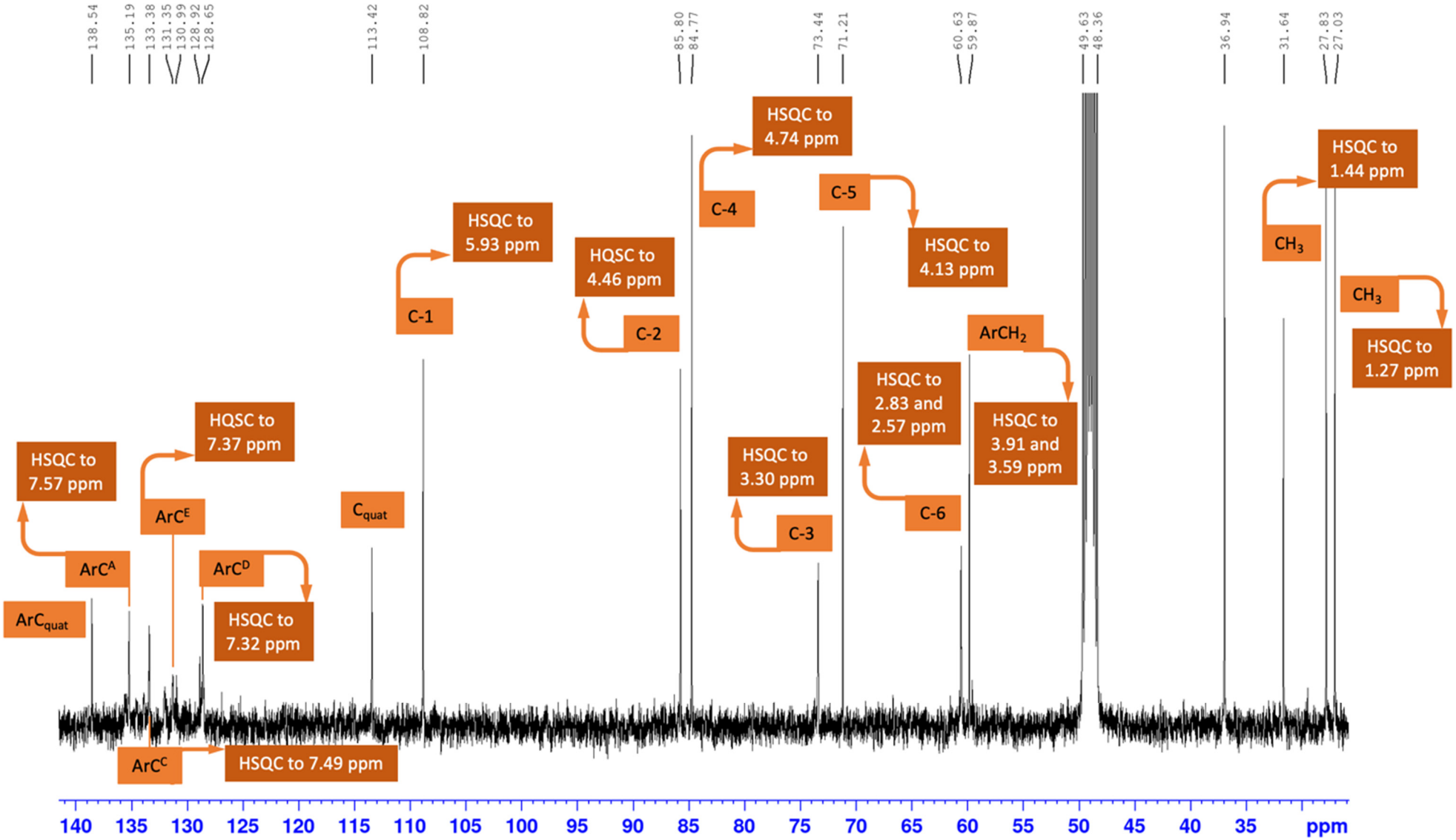

1.440
1.269
CH3, s
CH3, s
1.45
1.40
1.35
1.30
1.25
1.20
1.15
1.10
1.05
ppm
3.43
3.51

Figure S15. ^{13}C-NMR spectrum (100 MHz, MeOD) of compound **meta 5** with colour-coded signals, highlighting the boronic acid form they belong to, with interpretation of the isolated signals and tentative interpretation of the overlapping ones. Namely, the orange designates the boronic acid form. A) section 141 ppm to 25 ppm; B) section 140 ppm to 120 ppm.

A



B

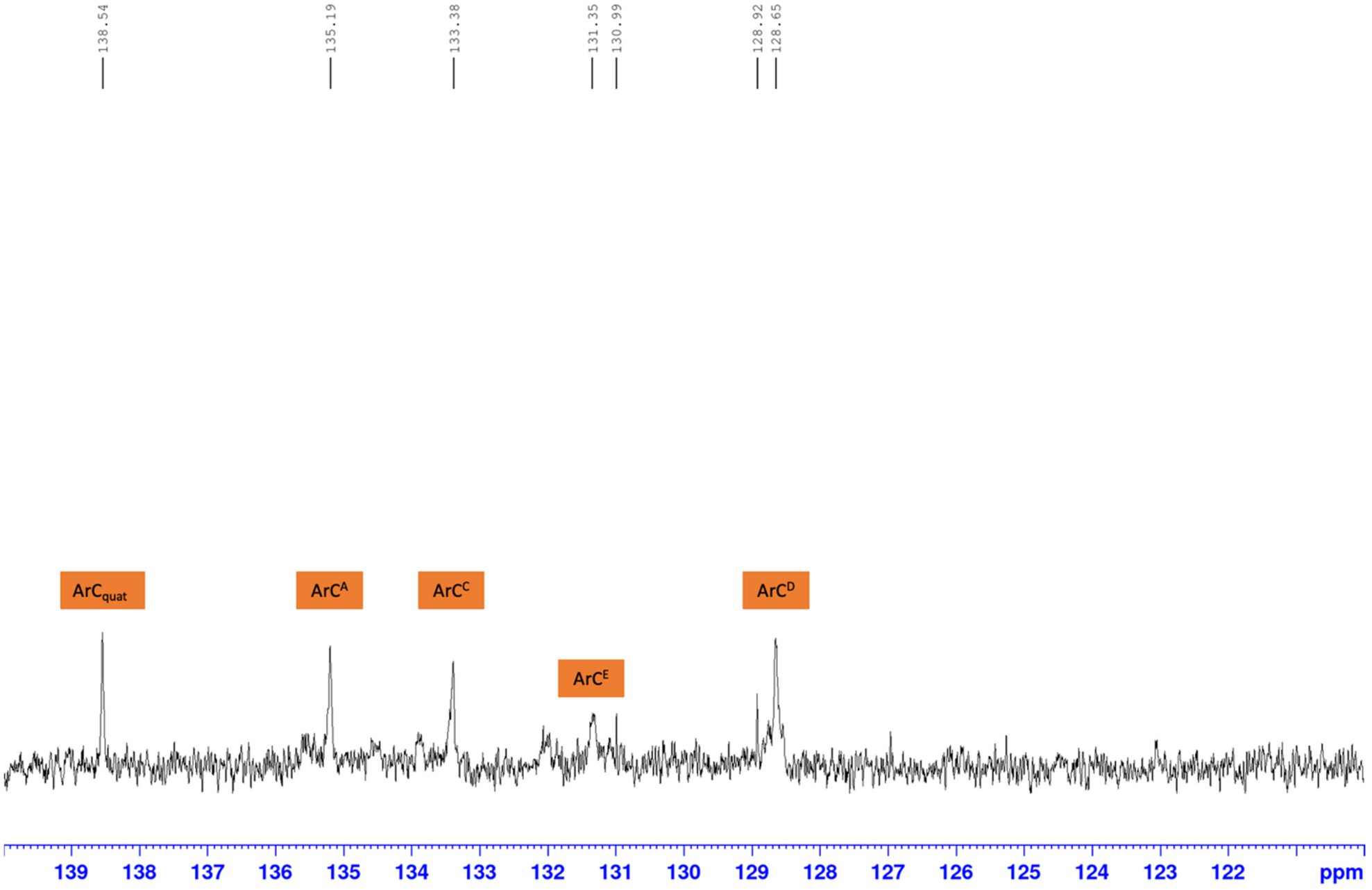
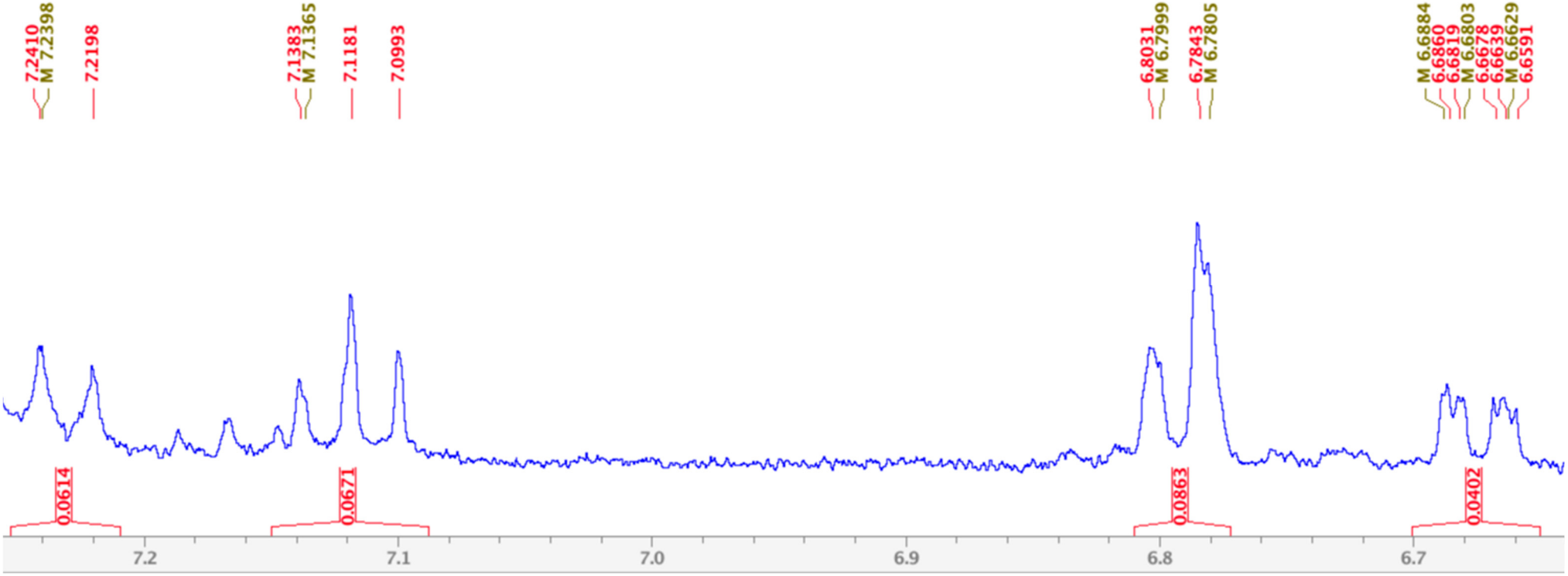
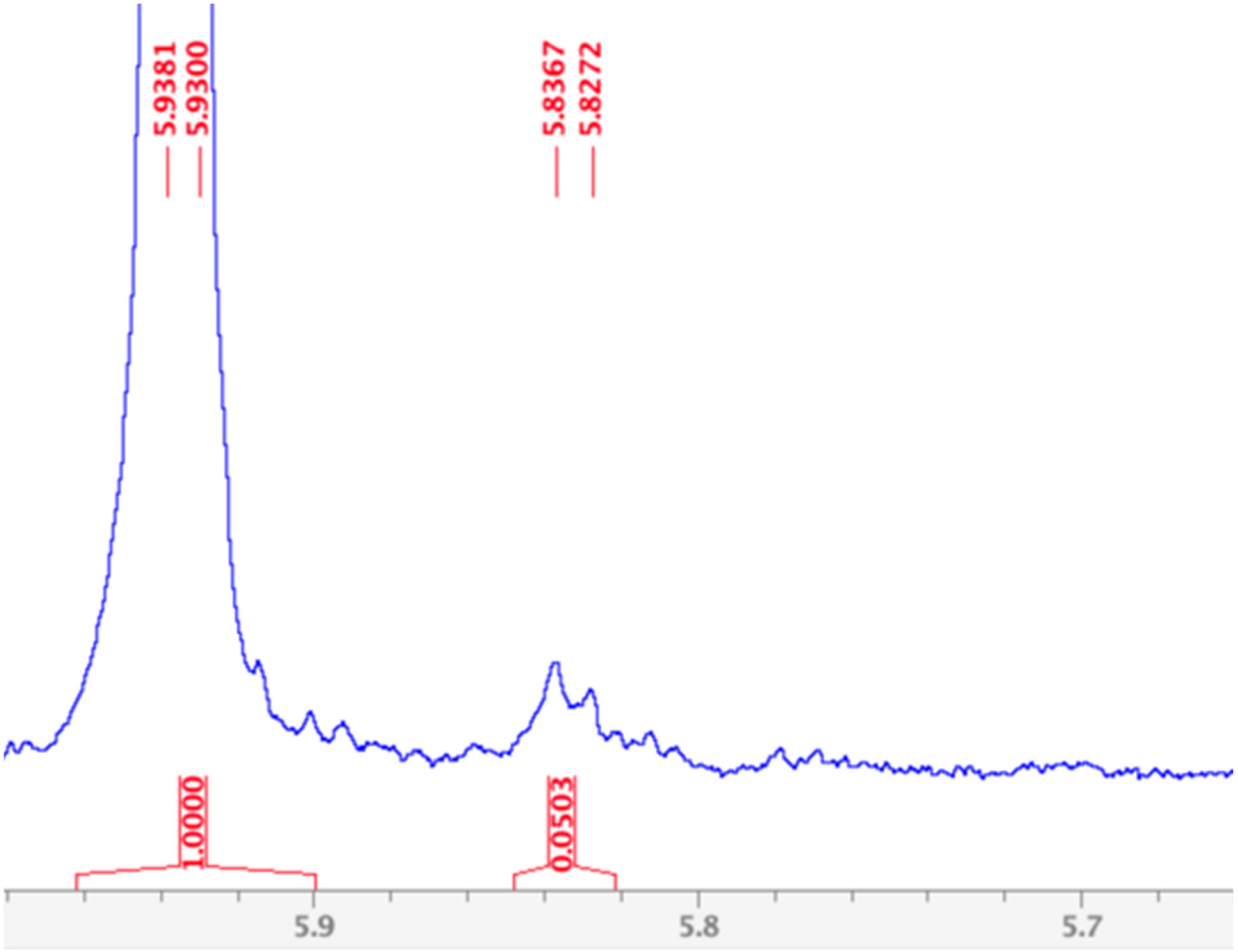
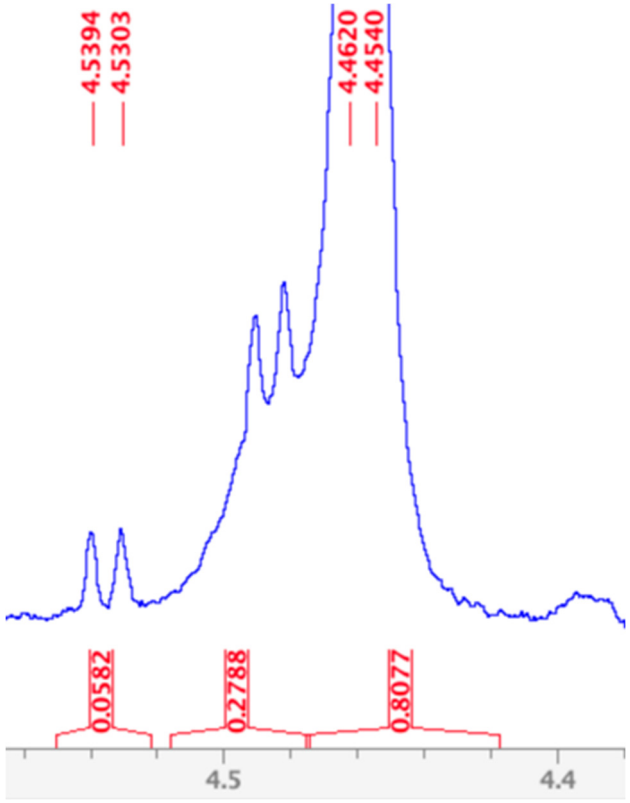

138.54
135.19
133.38
131.35
130.99
128.92
128.65
ArC$_{quat}$
ArC^{A}
ArC^{C}
ArC^{E}
ArC^{D}
139
138
137
136
135
134
133
132
131
130
129
128
127
126
125
124
123
122
ppm

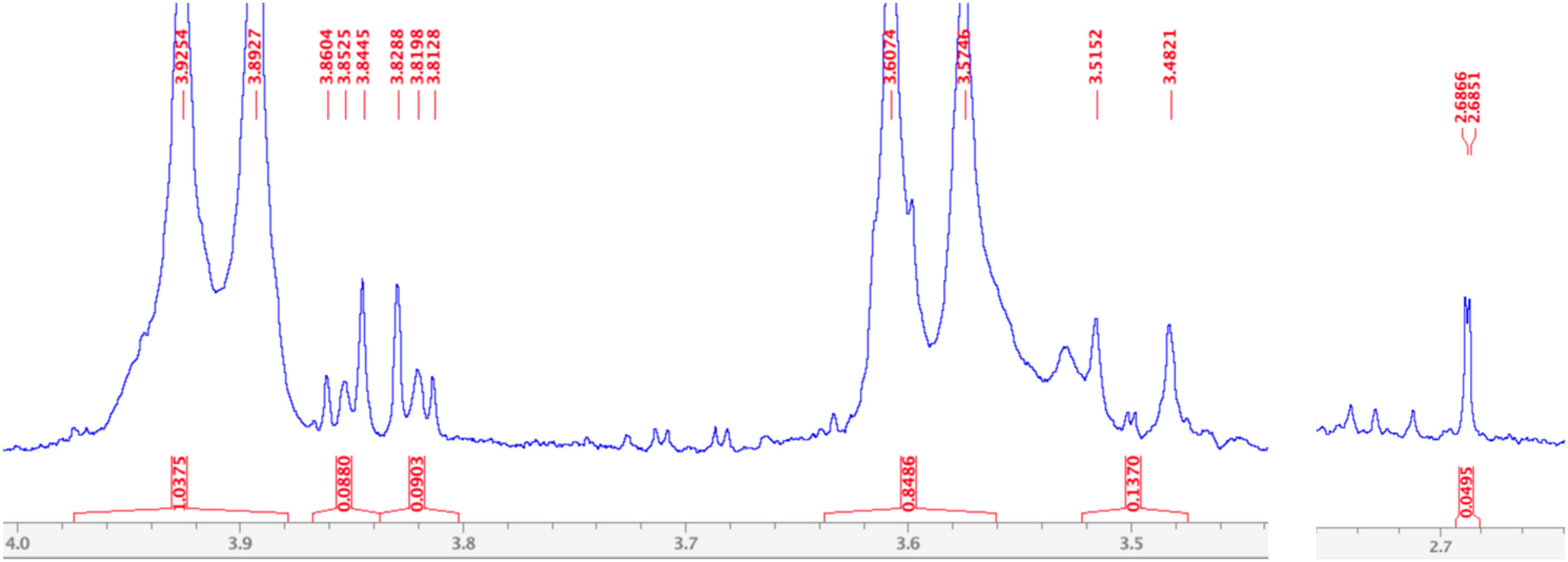
Figure S16. ^{1}H- (400 MHz) and COSY spectra of the minor species for *N*-(3-methylphenyl boronic acid)-3,6-dideoxy-3,6-imino-1,2-*O*-isopropylidene-*α*-D-gulofuranose **meta 5** in MeOD.

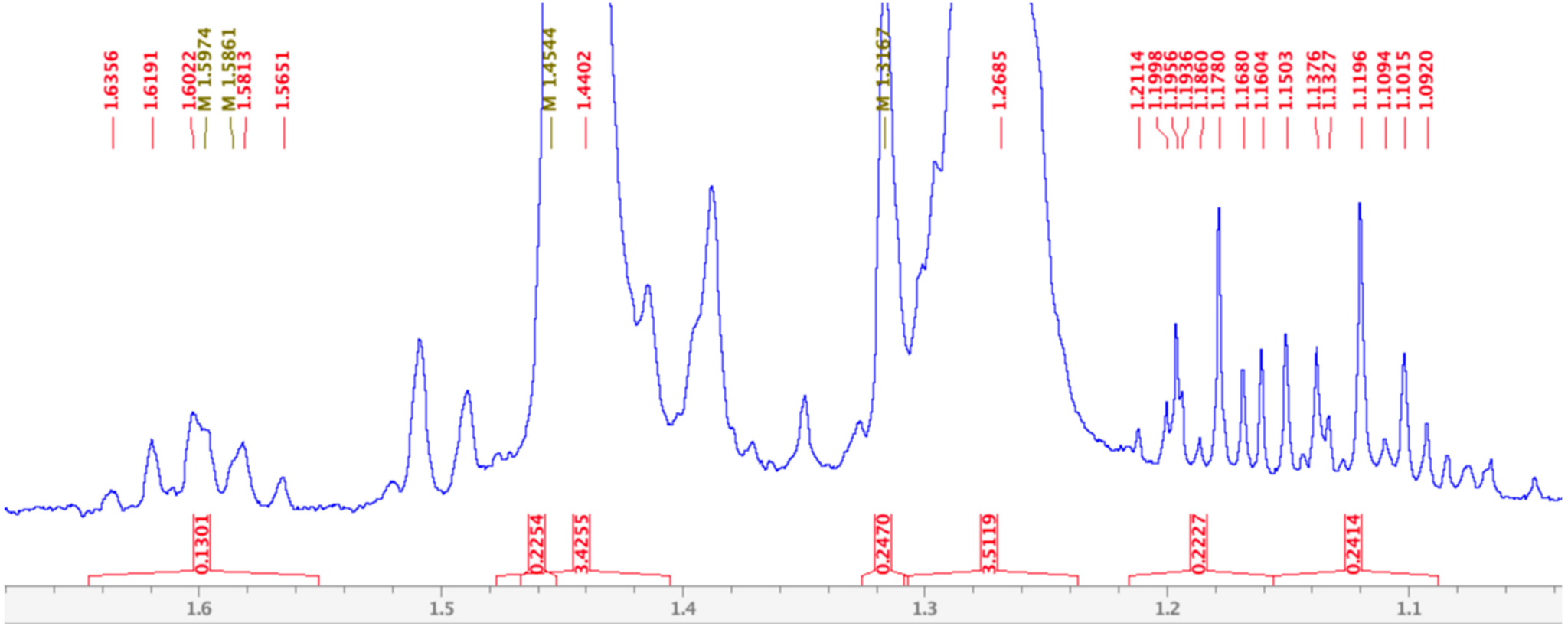
^{1}H-NMR



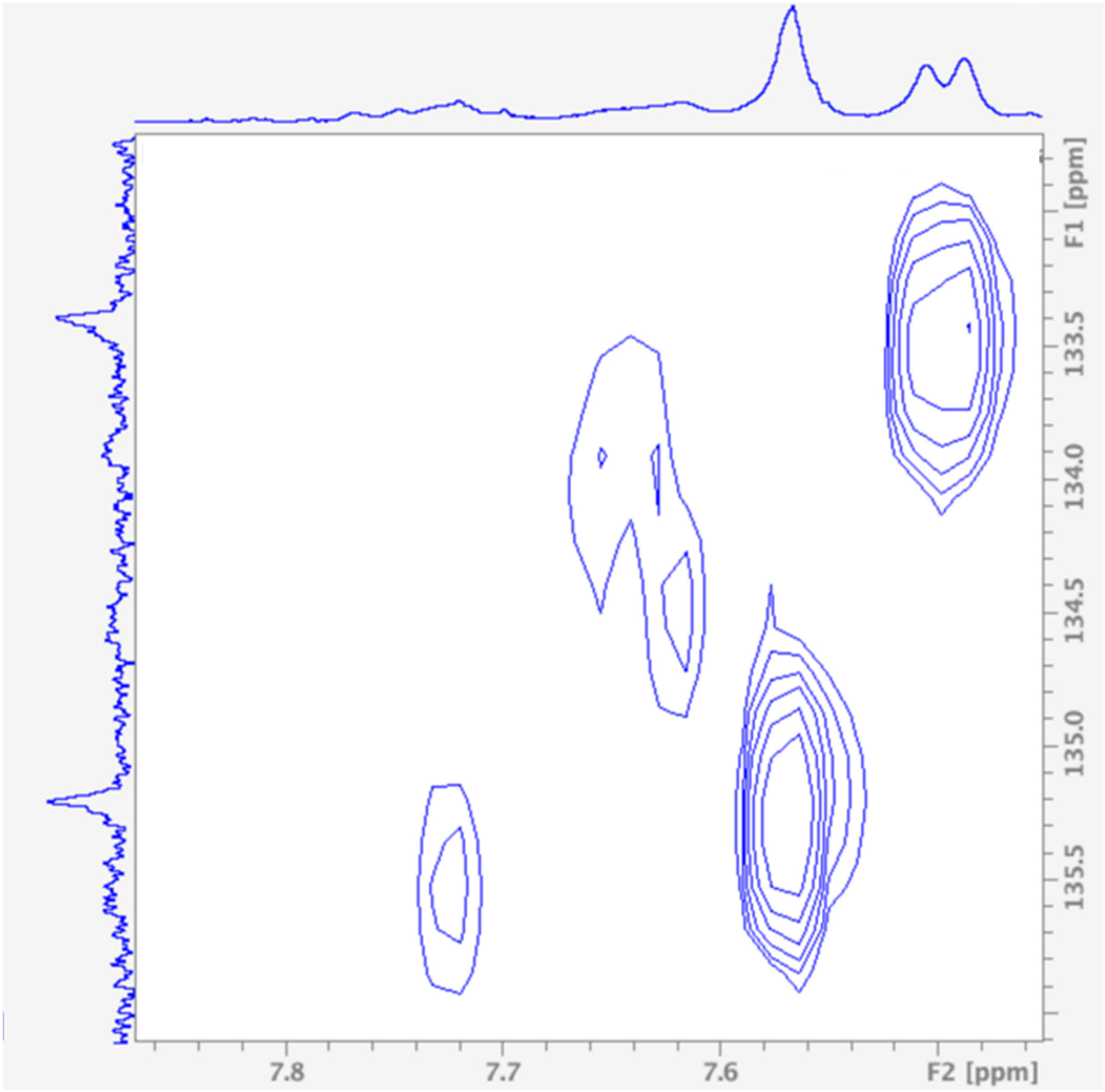

5.9381
5.9300
5.8367
5.8272
1.0000
0.0503
5.9
5.8
5.7

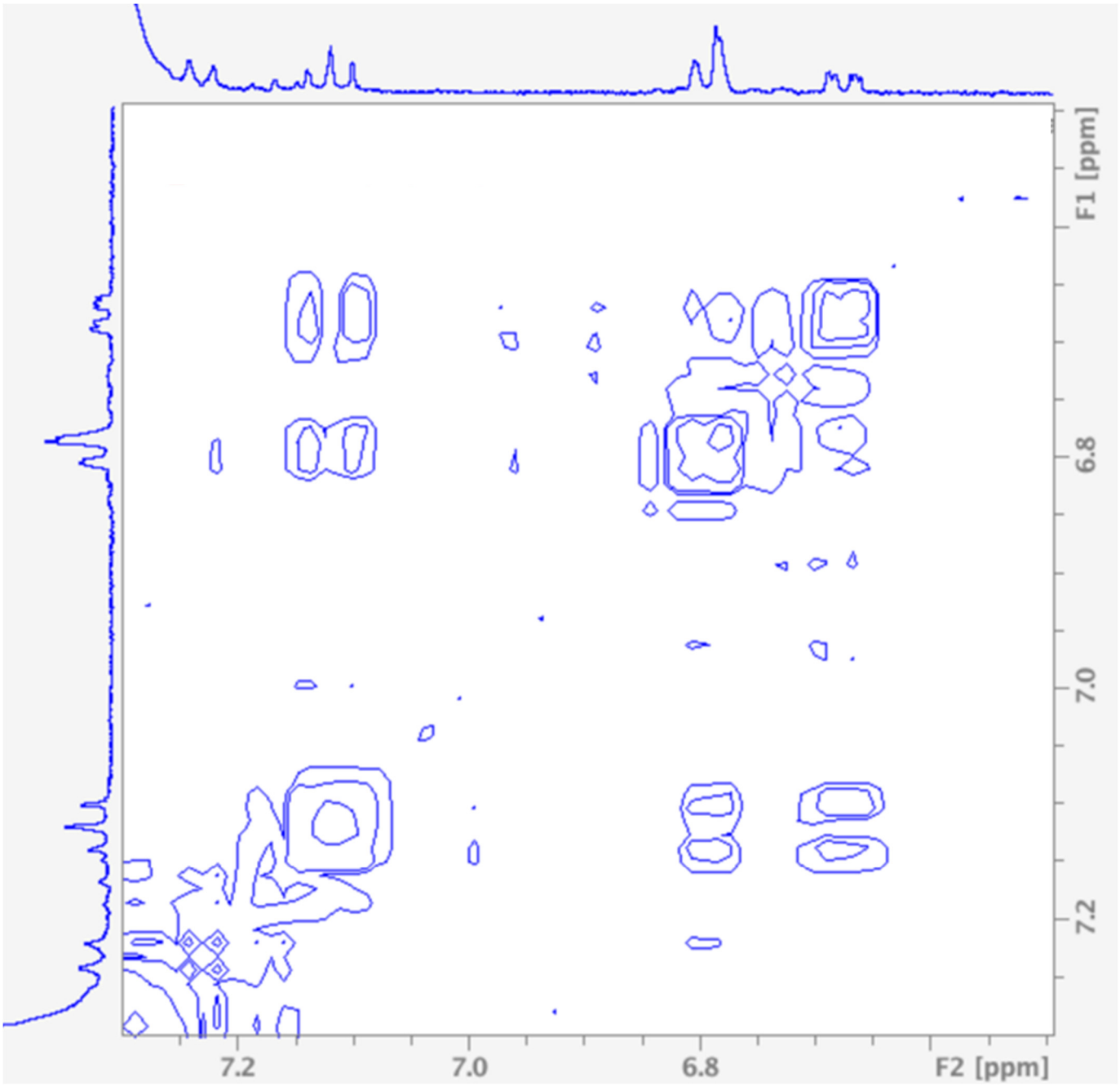

4.5394
4.5303
4.4620
4.4540
0.0582
0.2788
0.8077
4.5
4.4

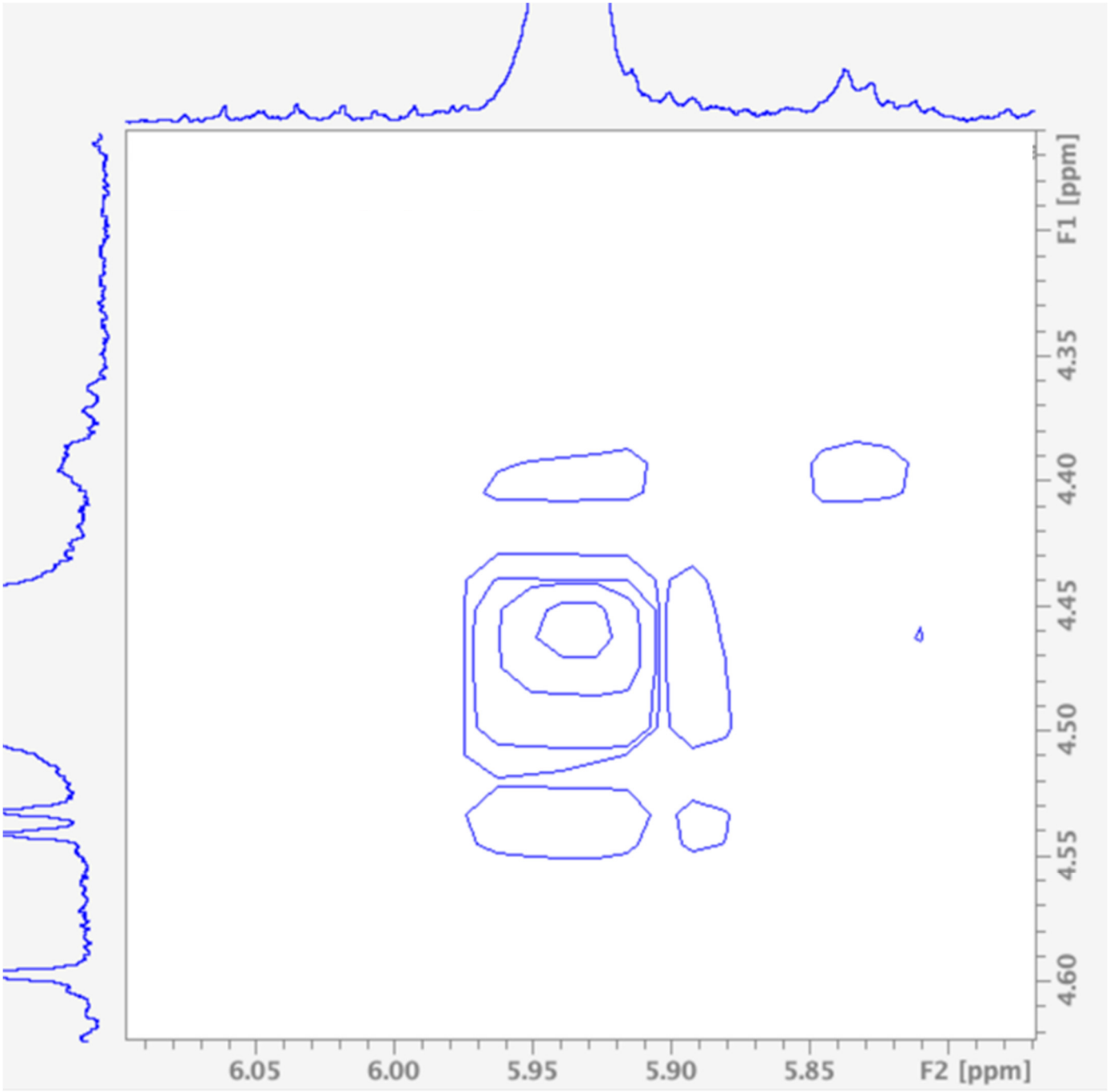

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3.8927
3.8604
3.8525
3.8445
3.8288
3.8198
3.8128
3.6074
3.5746
3.5152
3.4821
2.6866
2.6851
1.0375
0.0880
0.0903
0.8486
0.1370
0.0495
4.0
3.9
3.8
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3.6
3.5
2.7

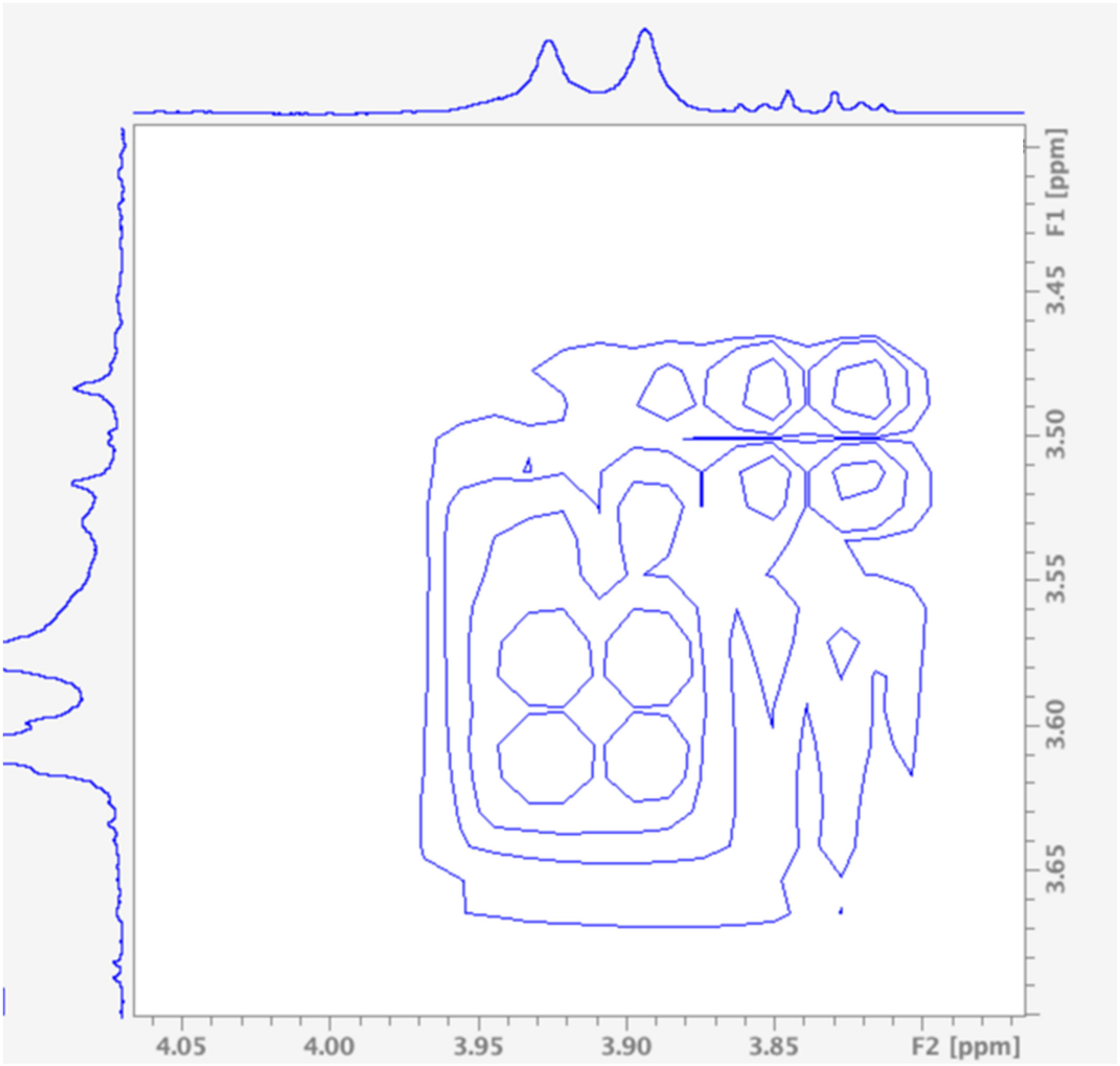

1.6356
1.6191
1.6022
M 1.5974
M 1.5861
1.5813
1.5651
M 1.4544
1.4402
M 1.3167
1.2685
1.2114
1.1998
1.1956
1.1936
1.1860
1.1780
1.1680
1.1604
1.1503
1.1376
1.1327
1.1196
1.1094
1.1015
1.0920
0.1301
0.2254
3.4255
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3.5119
0.2227
0.2414
1.6
1.5
1.4
1.3
1.2
1.1

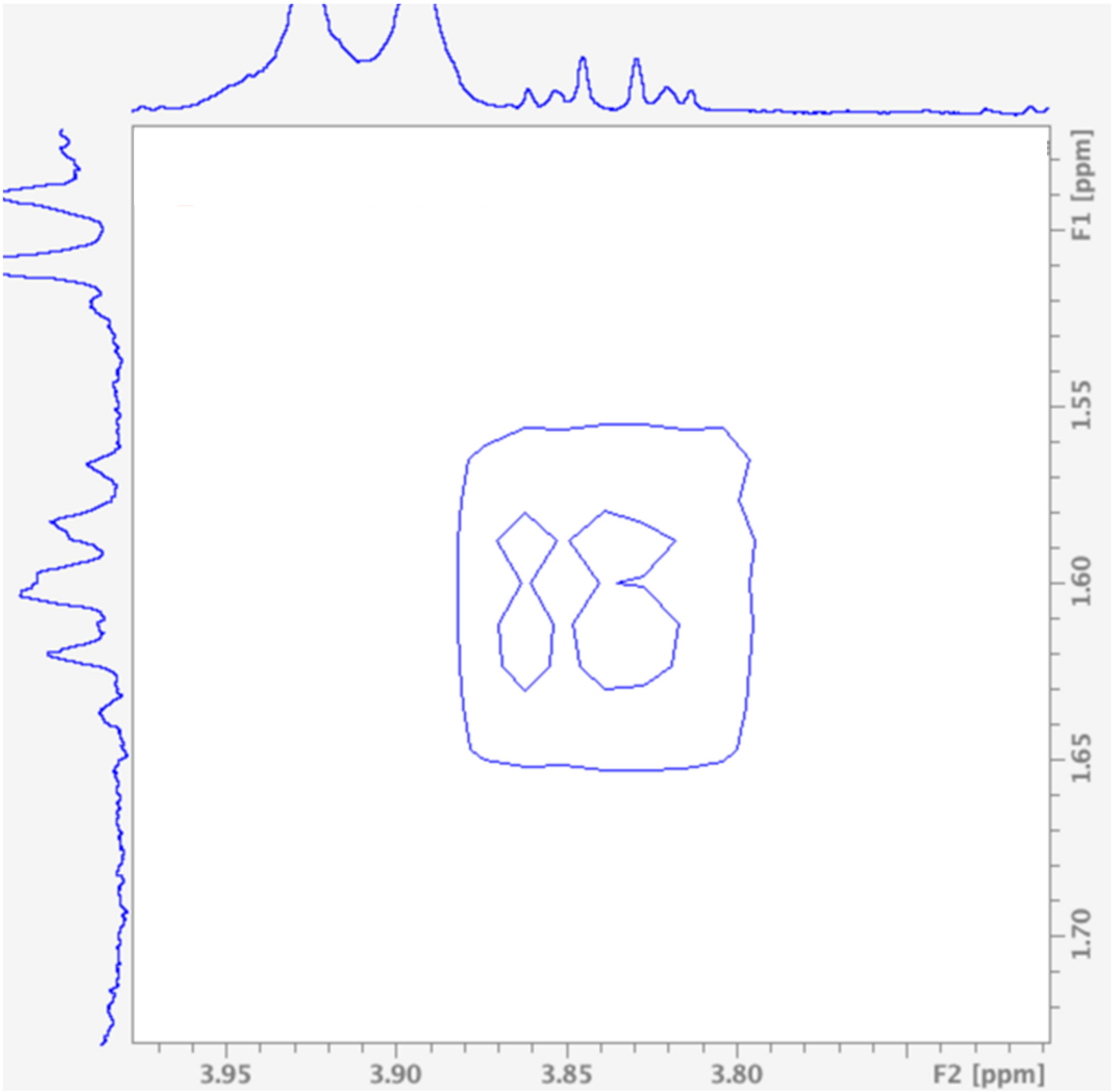
COSY




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7.2
7.0
6.8
F2 [ppm]


F1 [ppm]
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4.40
4.45
4.50
4.55
4.60
6.05
6.00
5.95
5.90
5.85
F2 [ppm]


F1 [ppm]
3.45
3.50
3.55
3.60
3.65
4.05
4.00
3.95
3.90
3.85
F2 [ppm]


F1 [ppm]
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1.60
1.65
1.70
3.95
3.90
3.85
3.80
F2 [ppm]

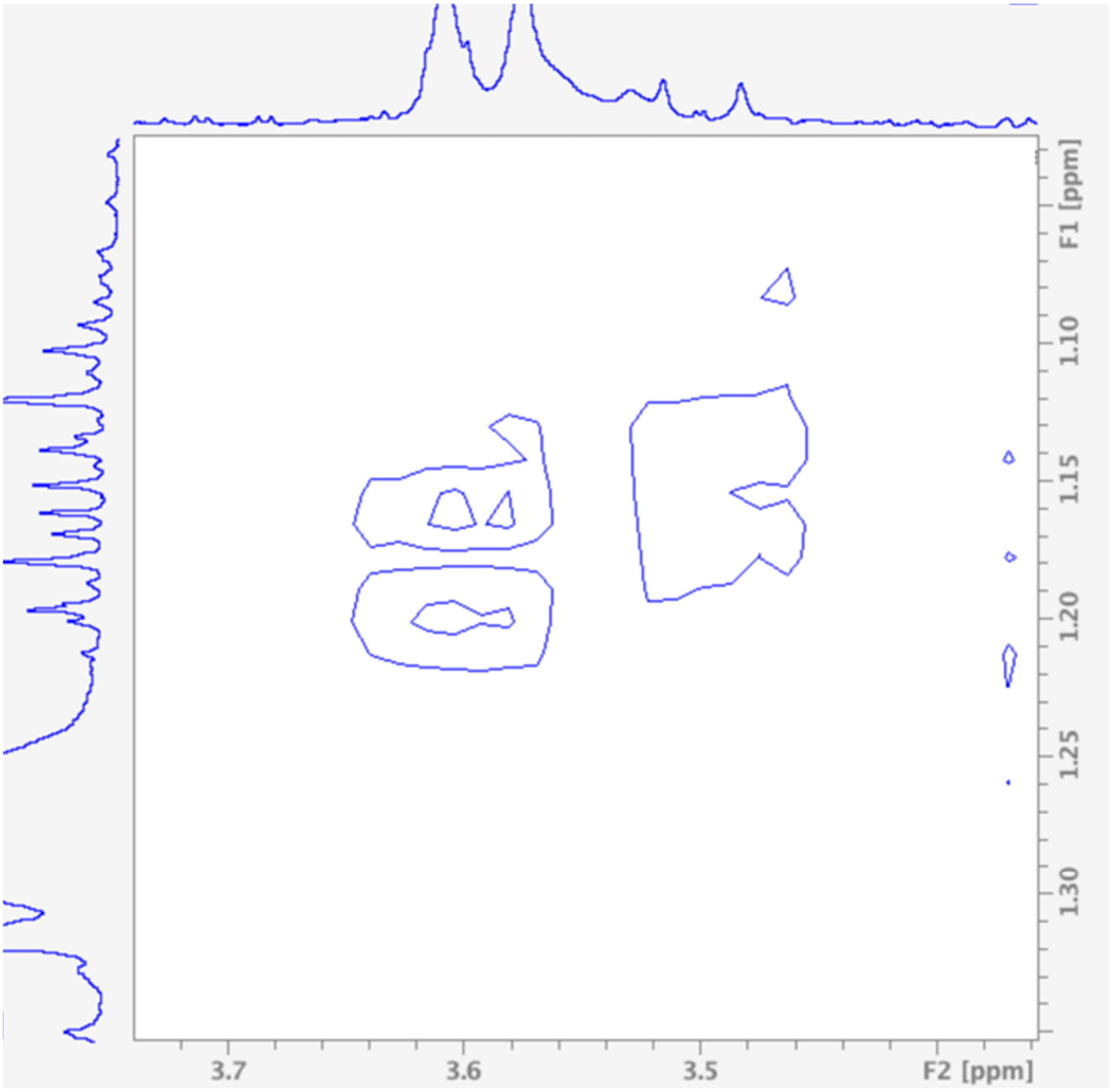
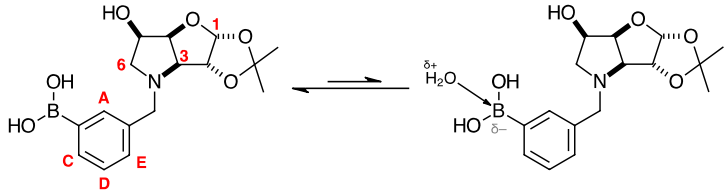
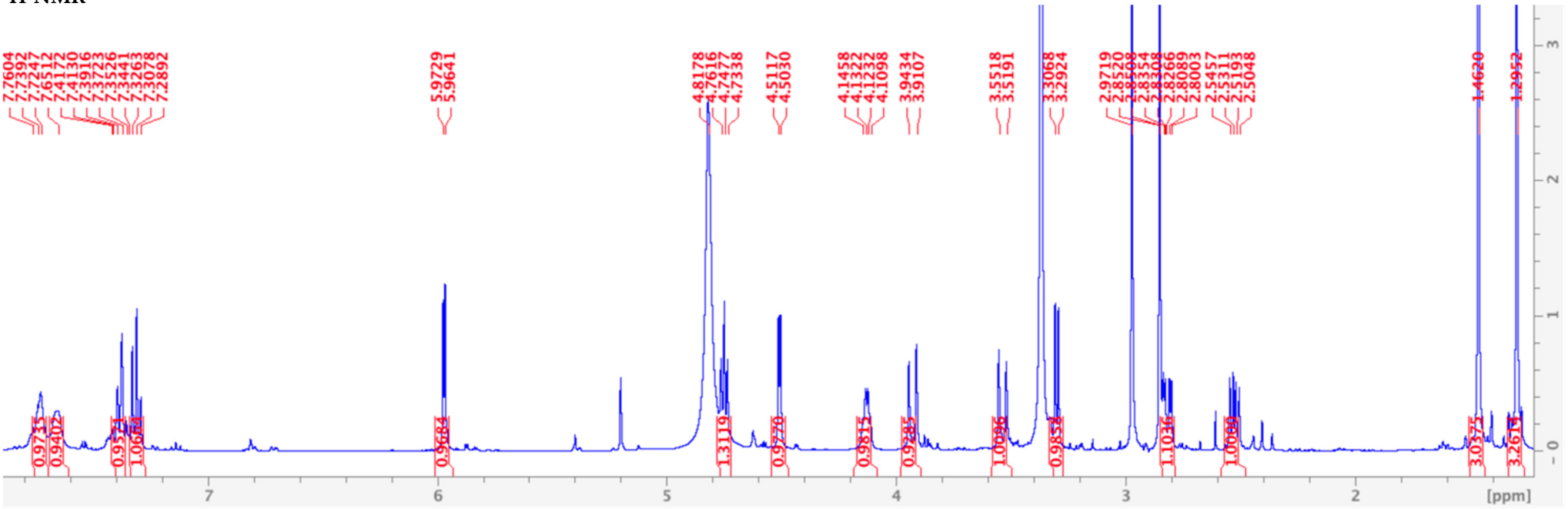

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1.20
1.25
1.30
3.7
3.6
3.5
F2 [ppm]

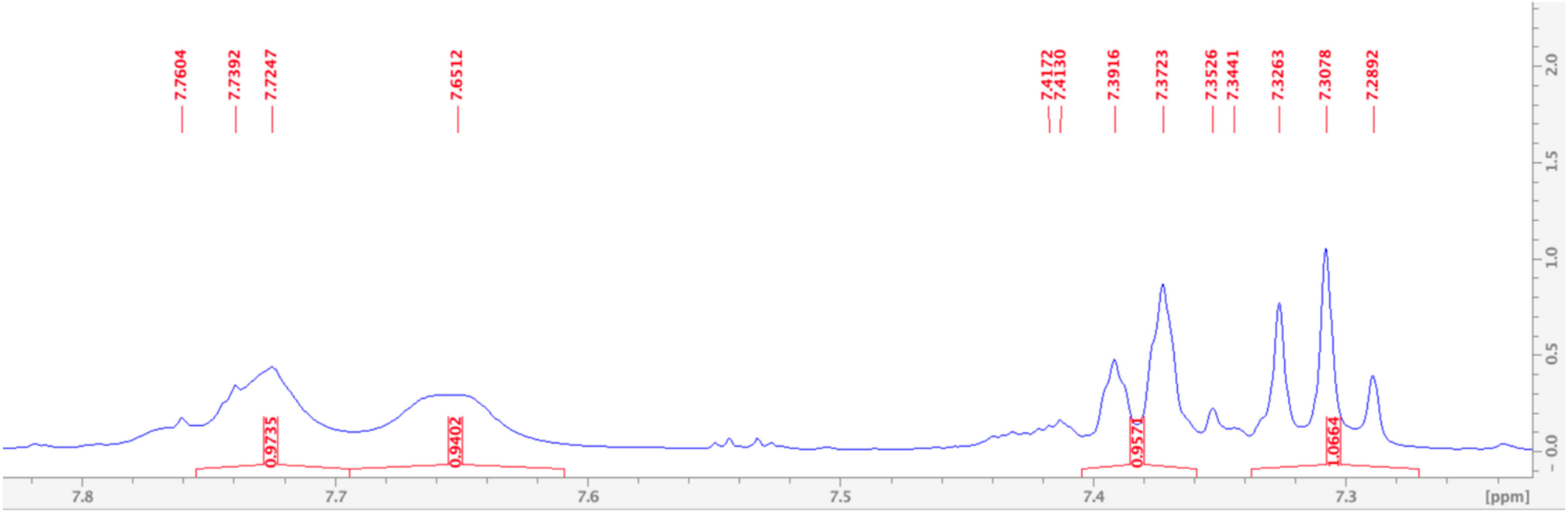
Figure S17. ^{1}H- (400 MHz), ^{13}C-NMR (100 MHz), DEPT, HSQC and HMBC spectra of *N*-(3-methylphenyl boronic acid)-3,6-dideoxy-3,6-imino-1,2-*O*-isopropylidene-*α*-D-gulofuranose **meta 5** in MeOD.

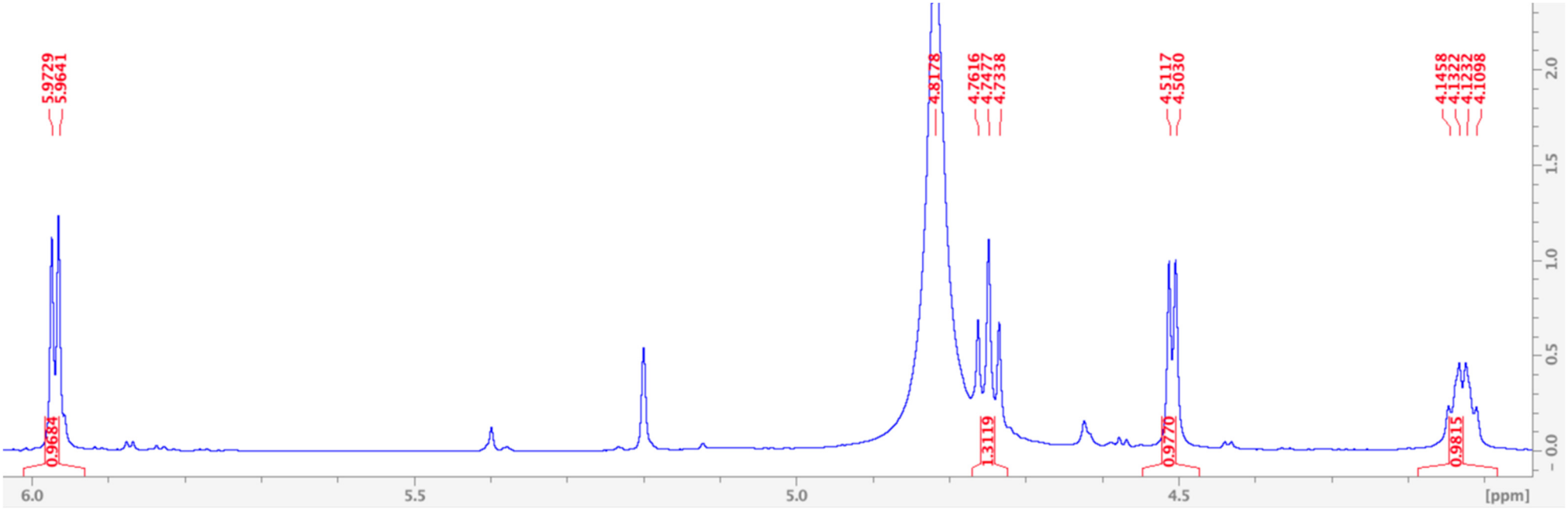
Figure S13. ^{1}H- (400 MHz), ^{13}C-NMR (100 MHz), ^{11}B-NMR (128 MHz), COSY and HSQC spectra of *N*-(3-methylphenyl boronic acid)-3,6-dideoxy-3,6-imino-1,2-*O*-isopropylidene-*α*-D-gulofuranose **meta 5** in MeOD.

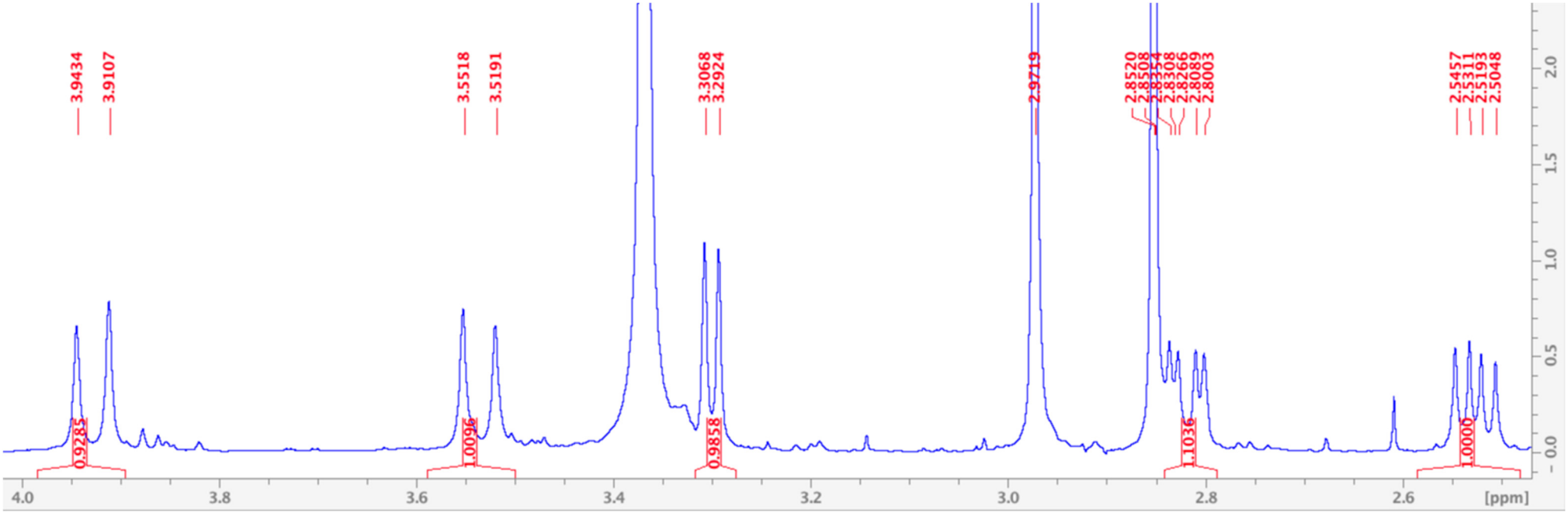


^{1}H-NMR

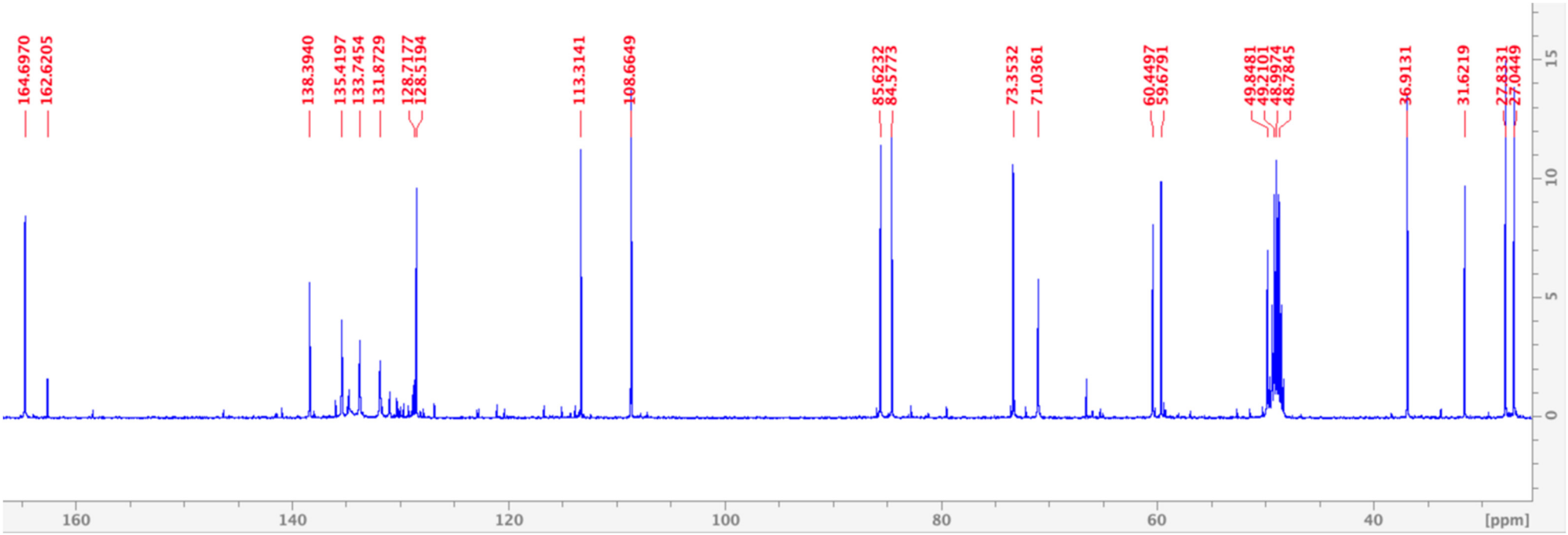


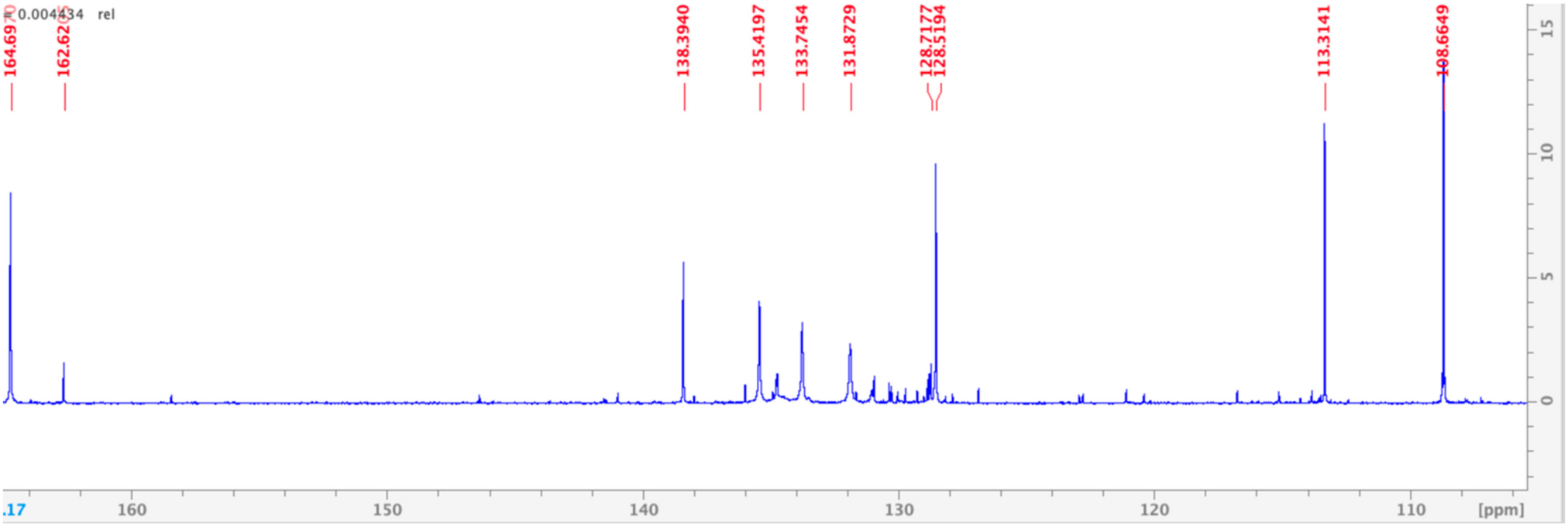

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7.7247
7.6512
7.4172
7.4130
7.3916
7.3723
7.3526
7.3441
7.3263
7.3078
7.2892
0.9735
0.9402
0.9574
1.0664
7.8
7.7
7.6
7.5
7.4
7.3
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1.5
1.0
0.5
0.0

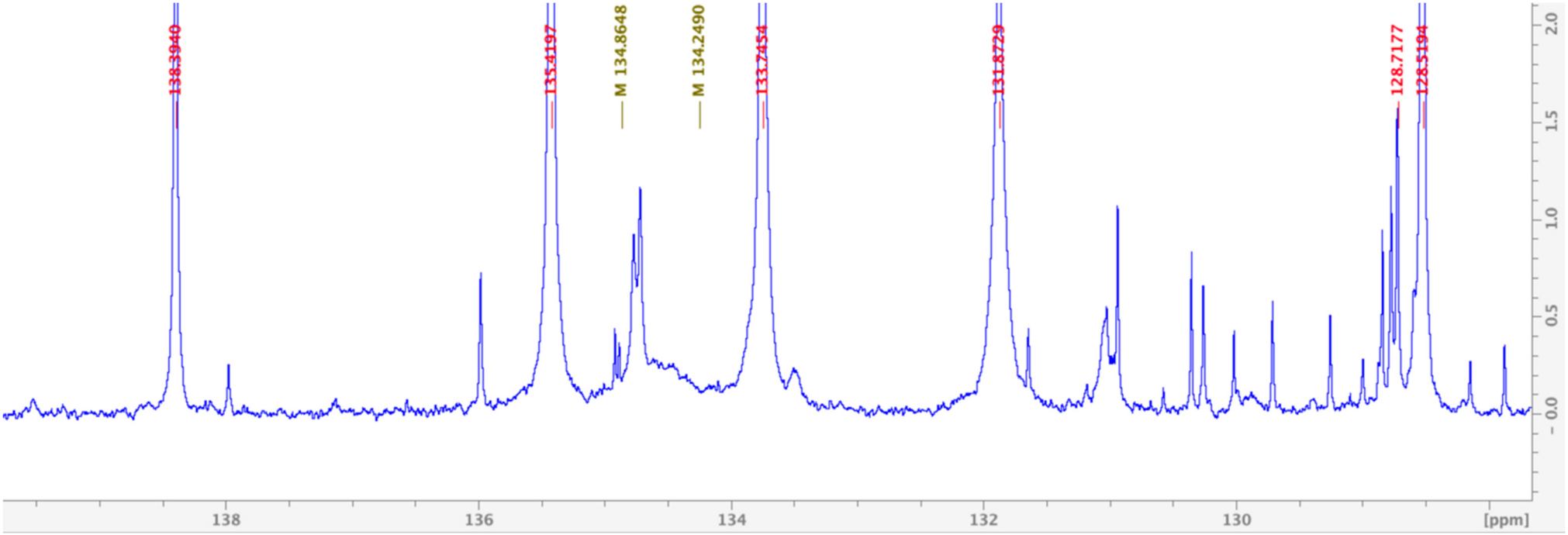

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5.9641
4.8178
4.7616
4.7477
4.7338
4.5117
4.5030
4.1458
4.1322
4.1232
4.1098
0.9684
1.3119
0.9770
0.9815
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5.5
5.0
4.5
[ppm]
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1.5
1.0
0.5
0.0

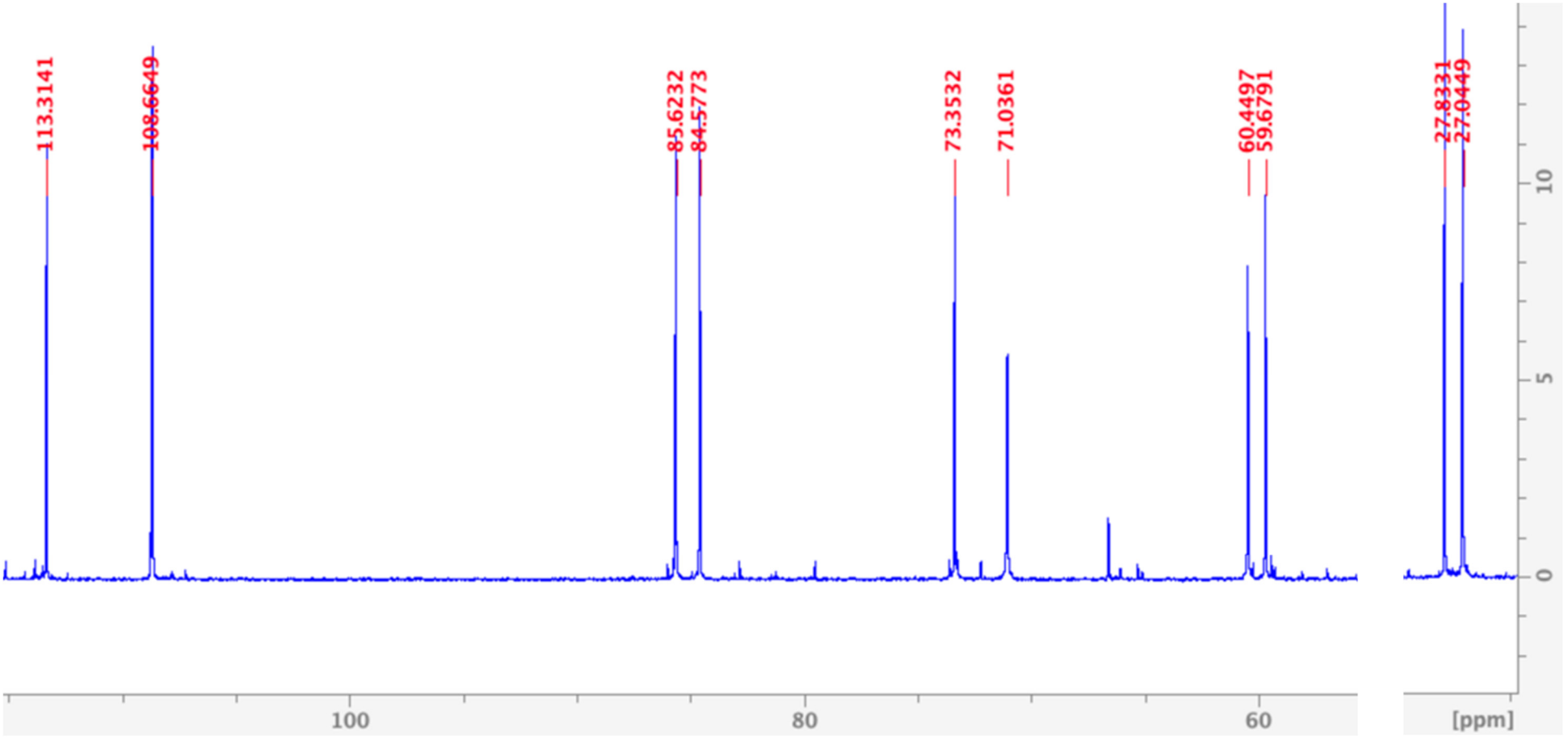

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3.9107
3.5518
3.5191
3.3068
3.2924
2.9719
2.8520
2.8508
2.8354
2.8308
2.8266
2.8089
2.8003
2.5457
2.5311
2.5193
2.5048
0.9285
1.0096
0.9858
1.1036
1.0000
4.0
3.8
3.6
3.4
3.2
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2.8
2.6
[ppm]
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1.0
0.5
0.0

^{13}C-NMR

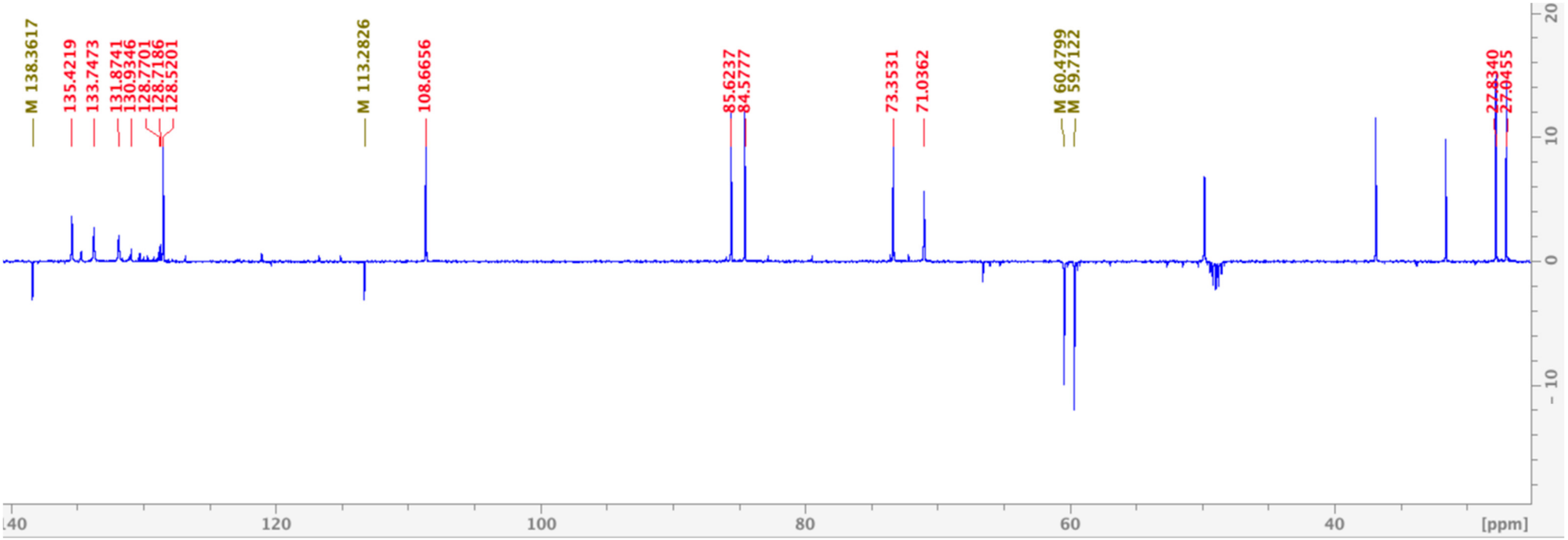

164.6970
162.6205
138.3940
135.4197
133.7454
131.8729
128.7177
128.5194
113.3141
108.6649
85.6232
84.5773
73.3532
71.0361
60.4497
59.6791
49.8481
49.2101
48.9974
48.7845
36.9131
31.6219
27.8331
27.0449
160
140
120
100
80
60
40
[ppm]
0
5
10
15


0.004434 rel
164.6970
162.6243
138.3940
135.4197
133.7454
131.8729
128.7177
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113.3141
108.6649
15
10
5
0
160
150
140
130
120
110
[ppm]

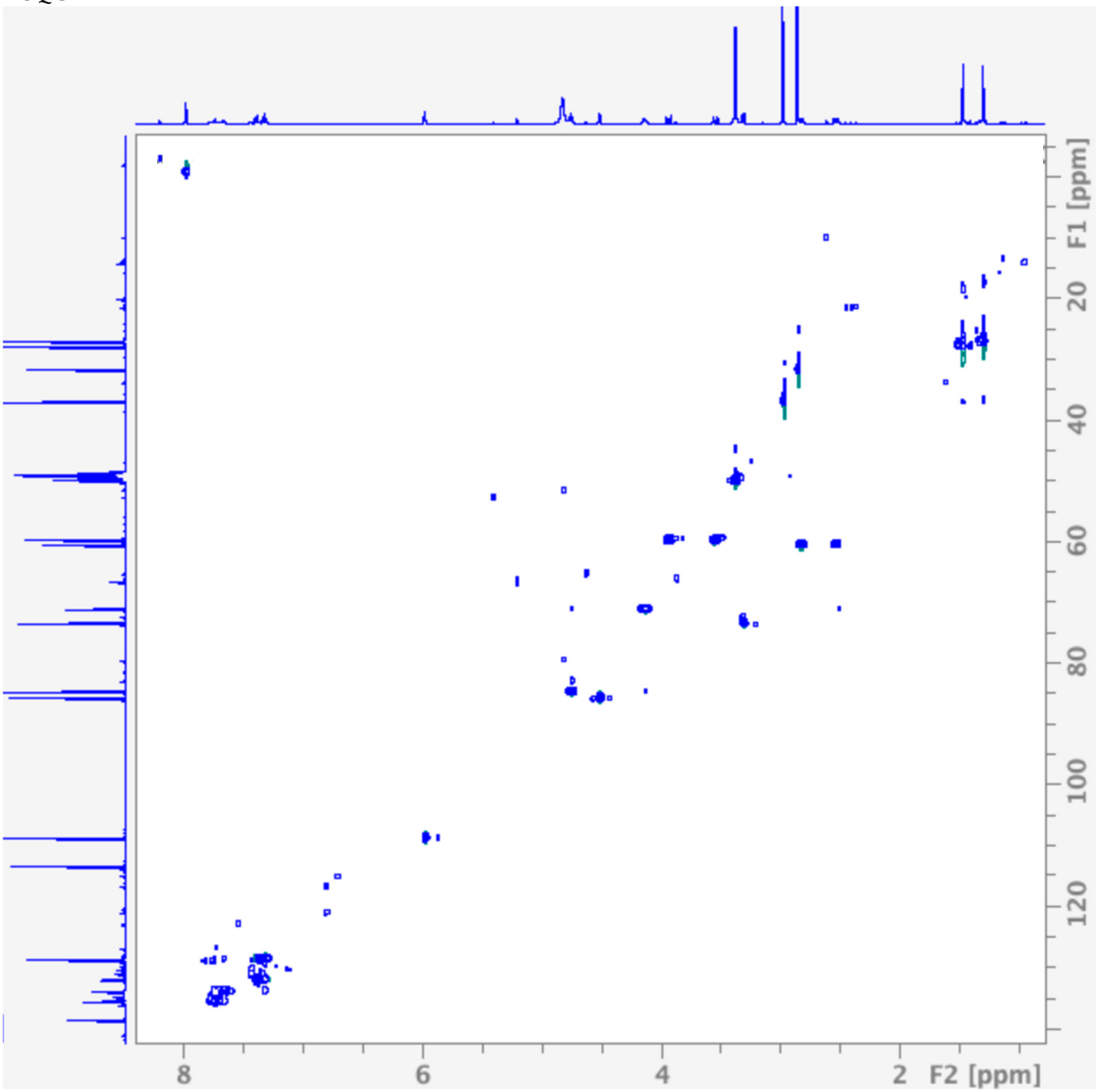

138.3940
135.4197
M 134.8648
M 134.2490
133.7454
131.8729
128.7177
128.5194
2.0
1.5
1.0
0.5
- 0.0
138
136
134
132
130
[ppm]

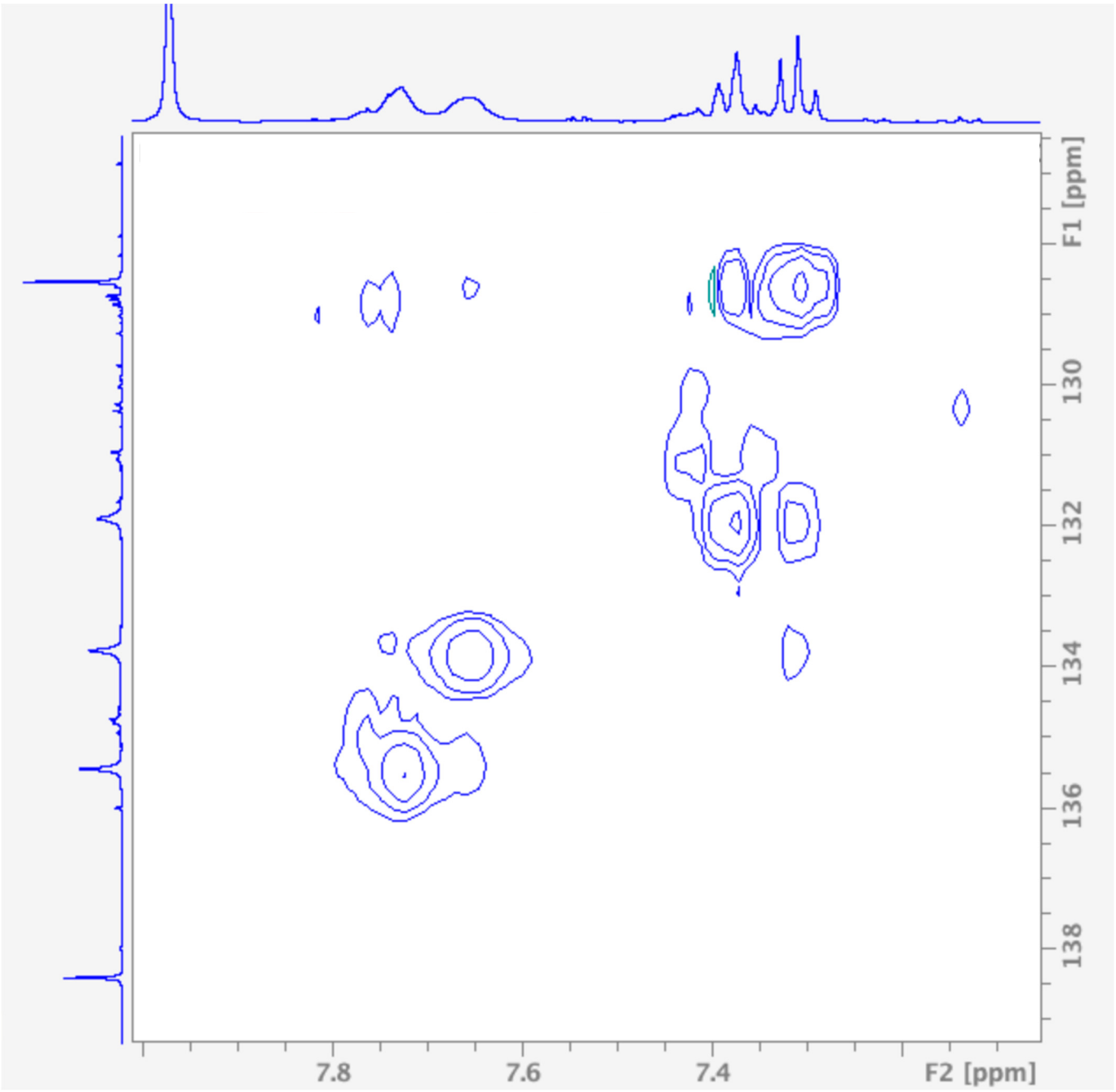

113.3141
108.6649
85.6232
84.5773
73.3532
71.0361
60.4497
59.6791
27.8331
27.0449
100
80
60
[ppm]
0
5
10

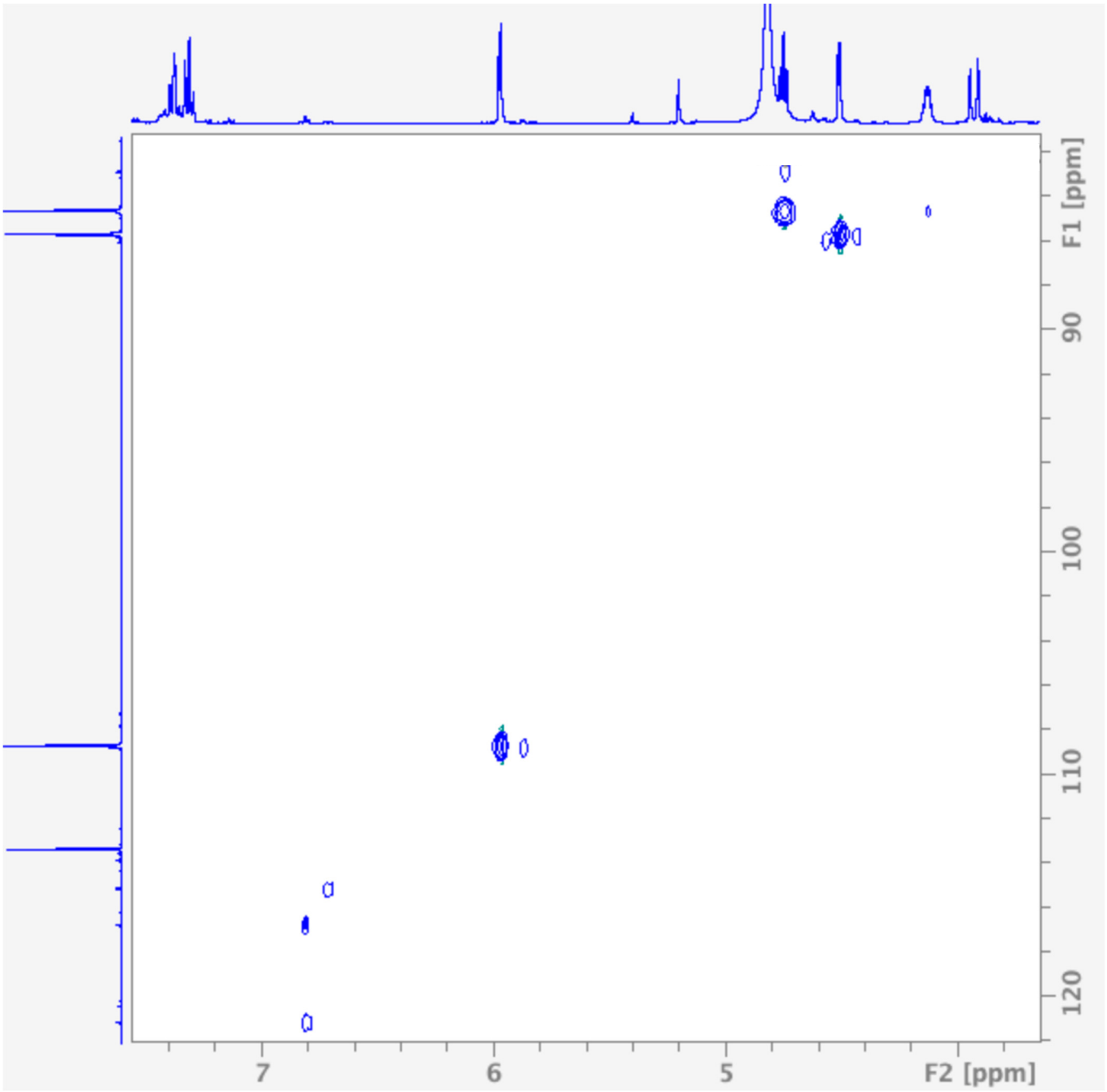
DEPT

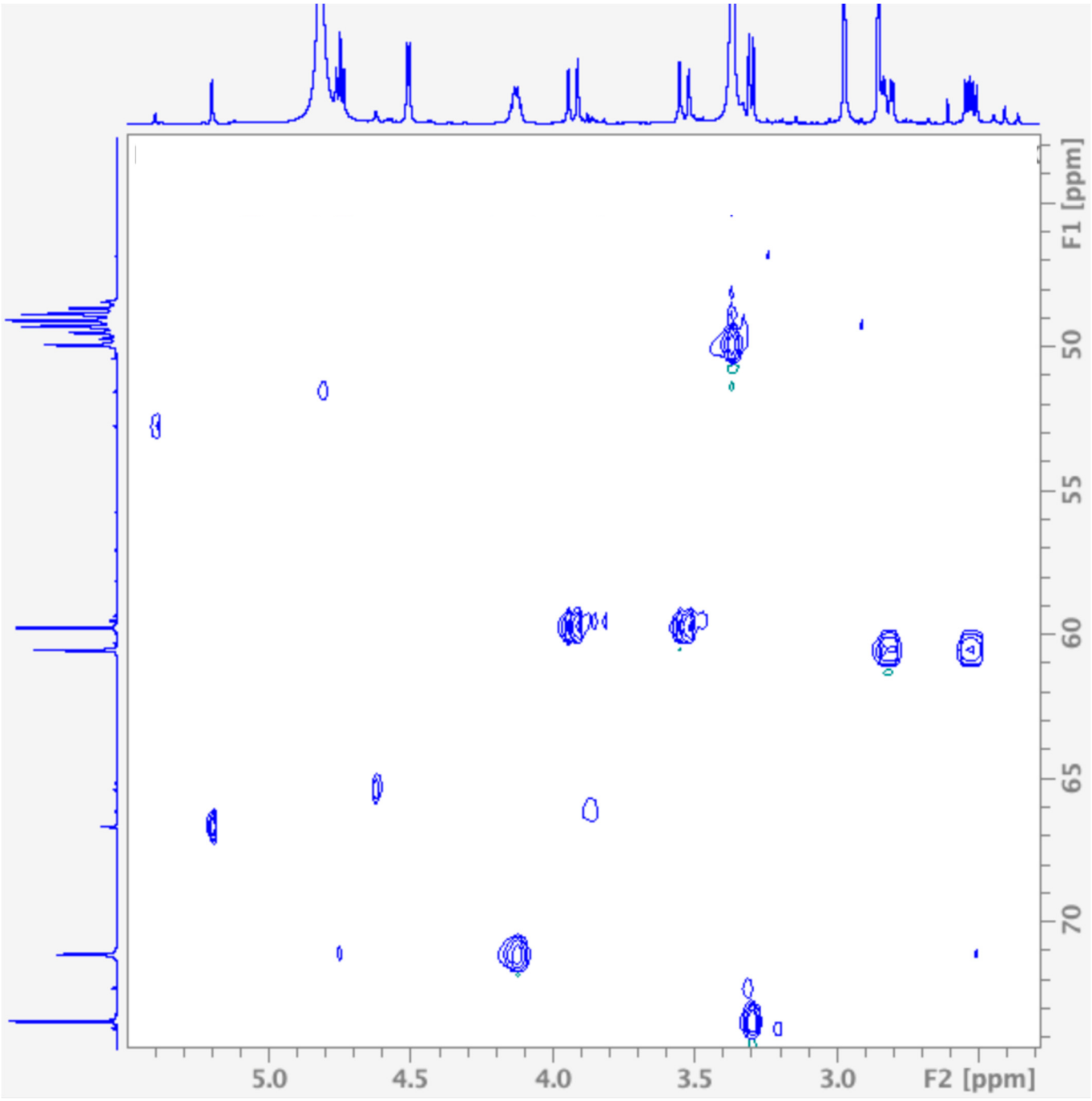

M 138.3617
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133.7473
131.8741
130.9346
128.7701
128.7186
128.5201
M 113.2826
108.6656
85.6237
84.5777
73.3531
71.0362
M 60.4799
M 59.7122
27.8340
27.0455
20
10
0
- 10
40
120
100
80
60
40
[ppm]

HSQC

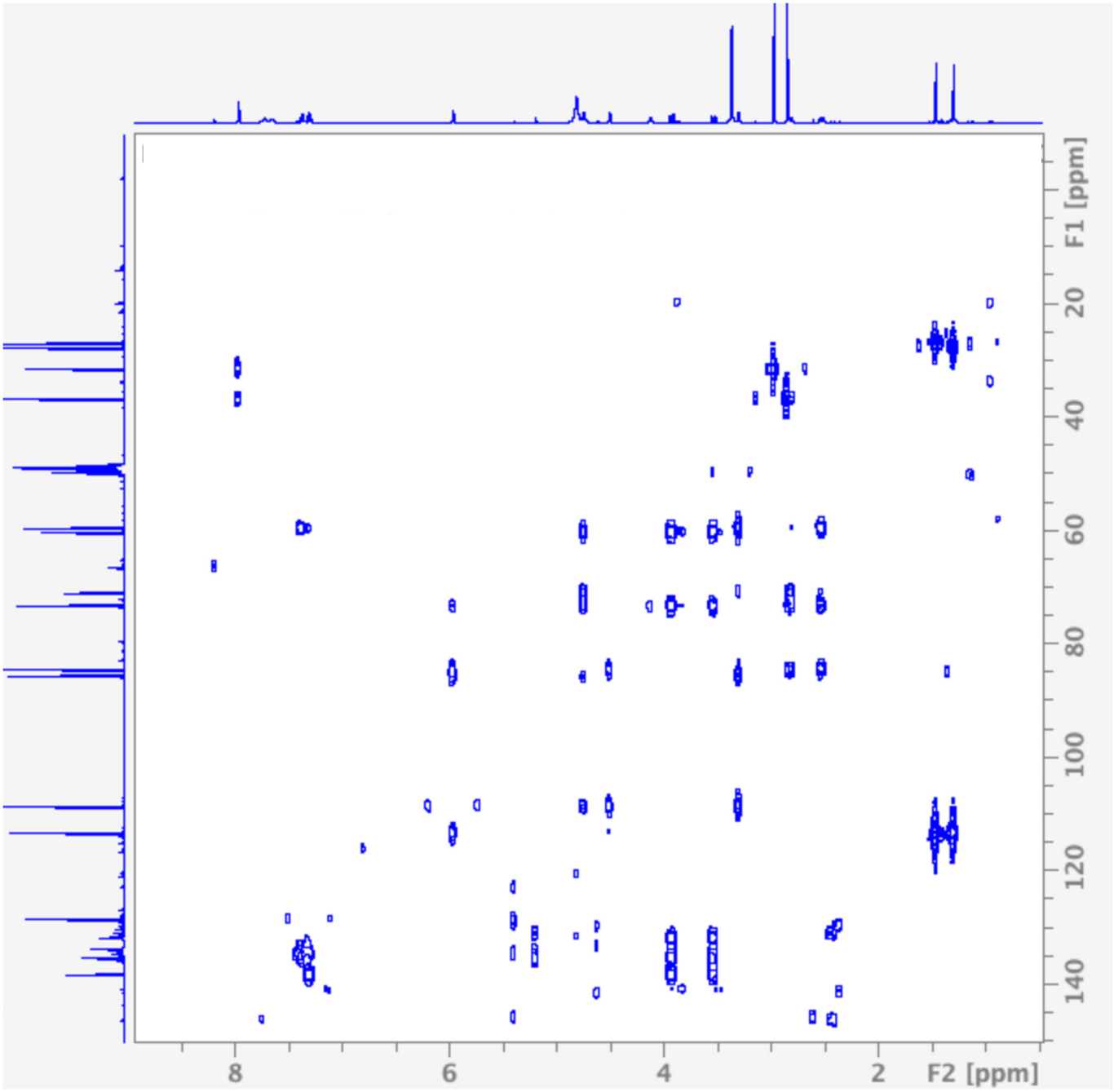


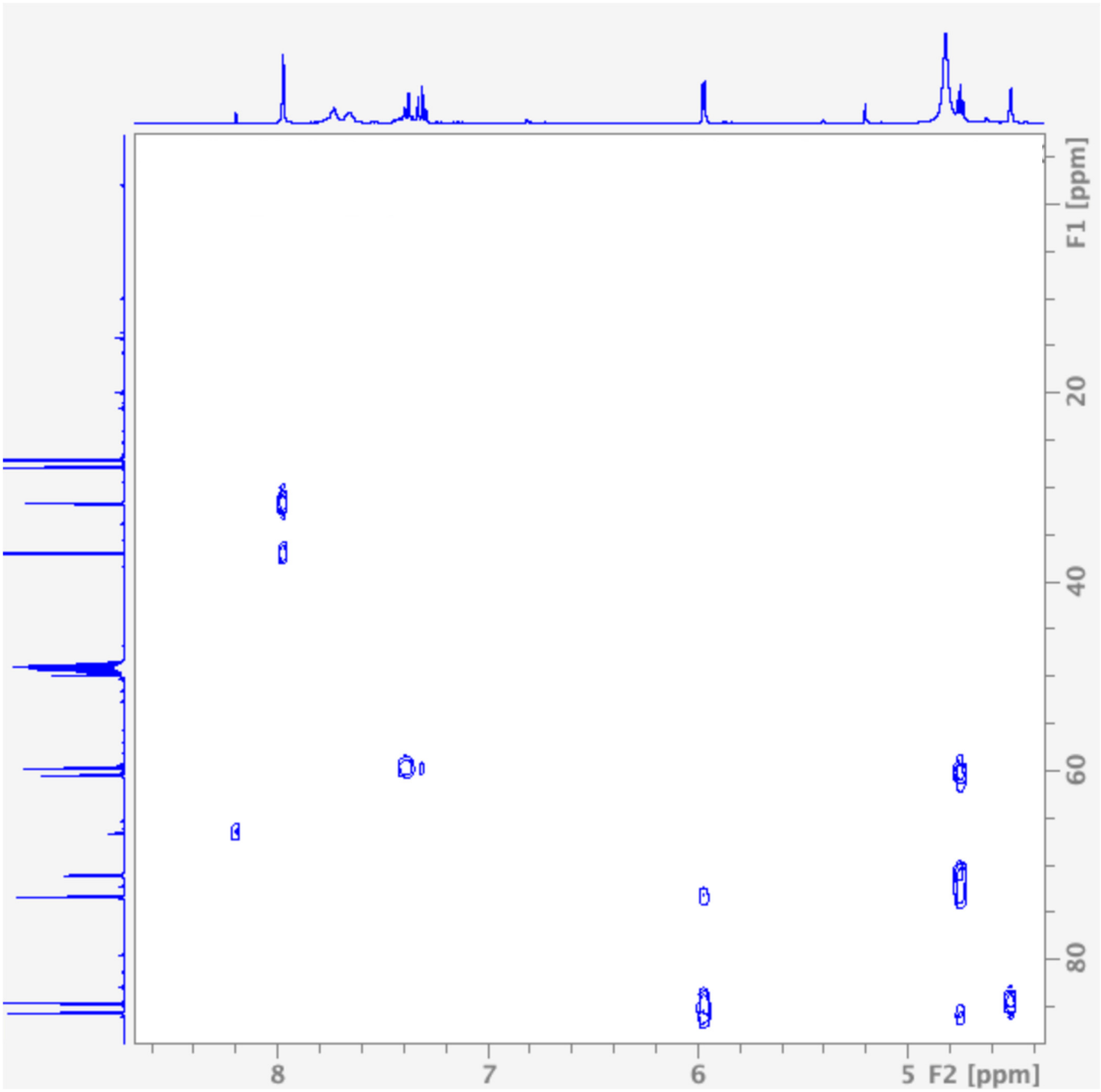

F1 [ppm]
130
132
134
136
138
7.8
7.6
7.4
F2 [ppm]

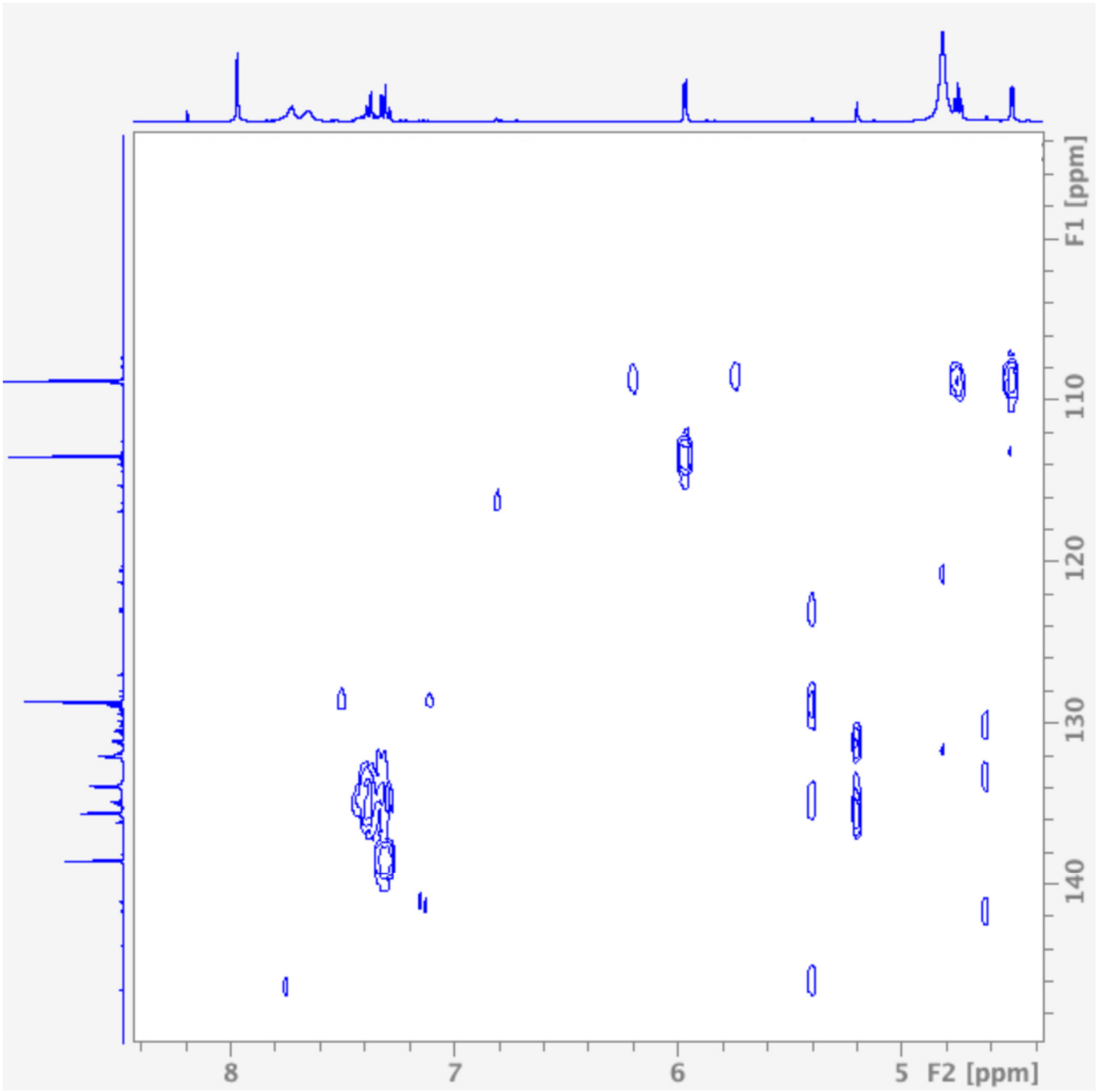

F1 [ppm]
90
100
110
120
7
6
5
F2 [ppm]

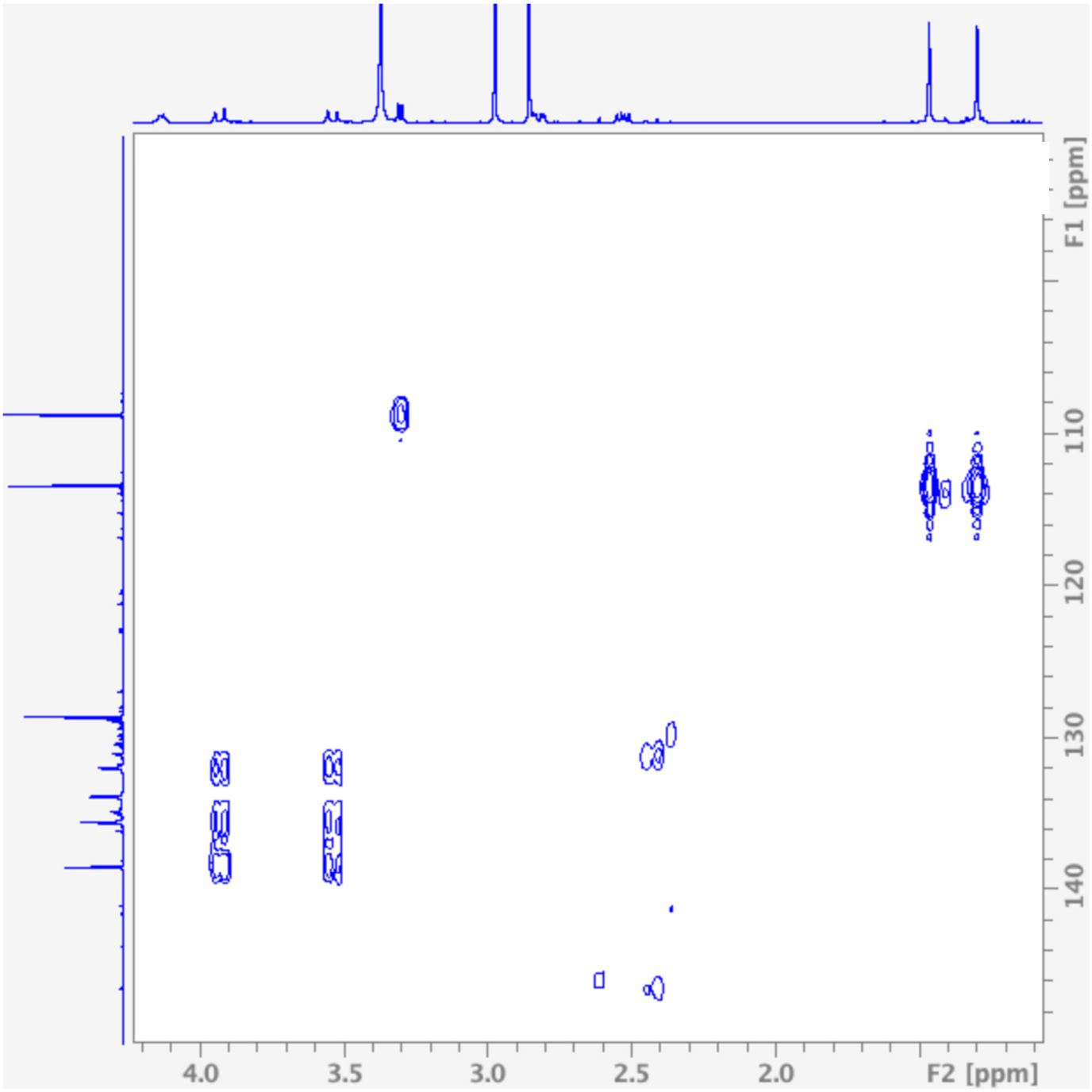

F1 [ppm]
50
55
60
65
70
5.0
4.5
4.0
3.5
3.0
F2 [ppm]

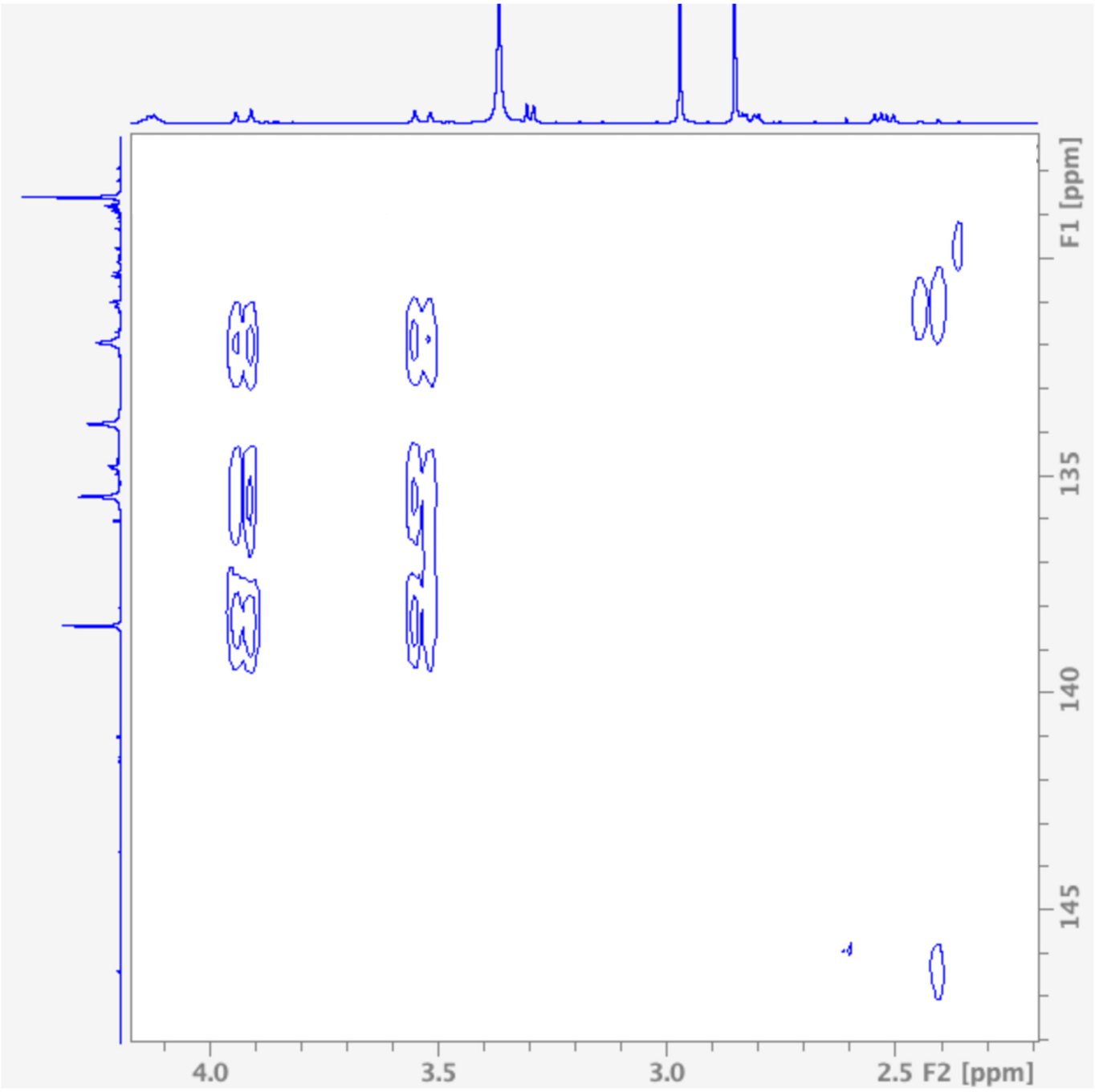
HMBC




F1 [ppm]
20
40
60
80
8
7
6
5
F2 [ppm]


F1 [ppm]
110
120
130
140
8
7
6
5
F2 [ppm]


F1 [ppm]
110
120
130
140
4.0
3.5
3.0
2.5
2.0
F2 [ppm]


F1 [ppm]
135
140
145
4.0
3.5
3.0
2.5 F2 [ppm]

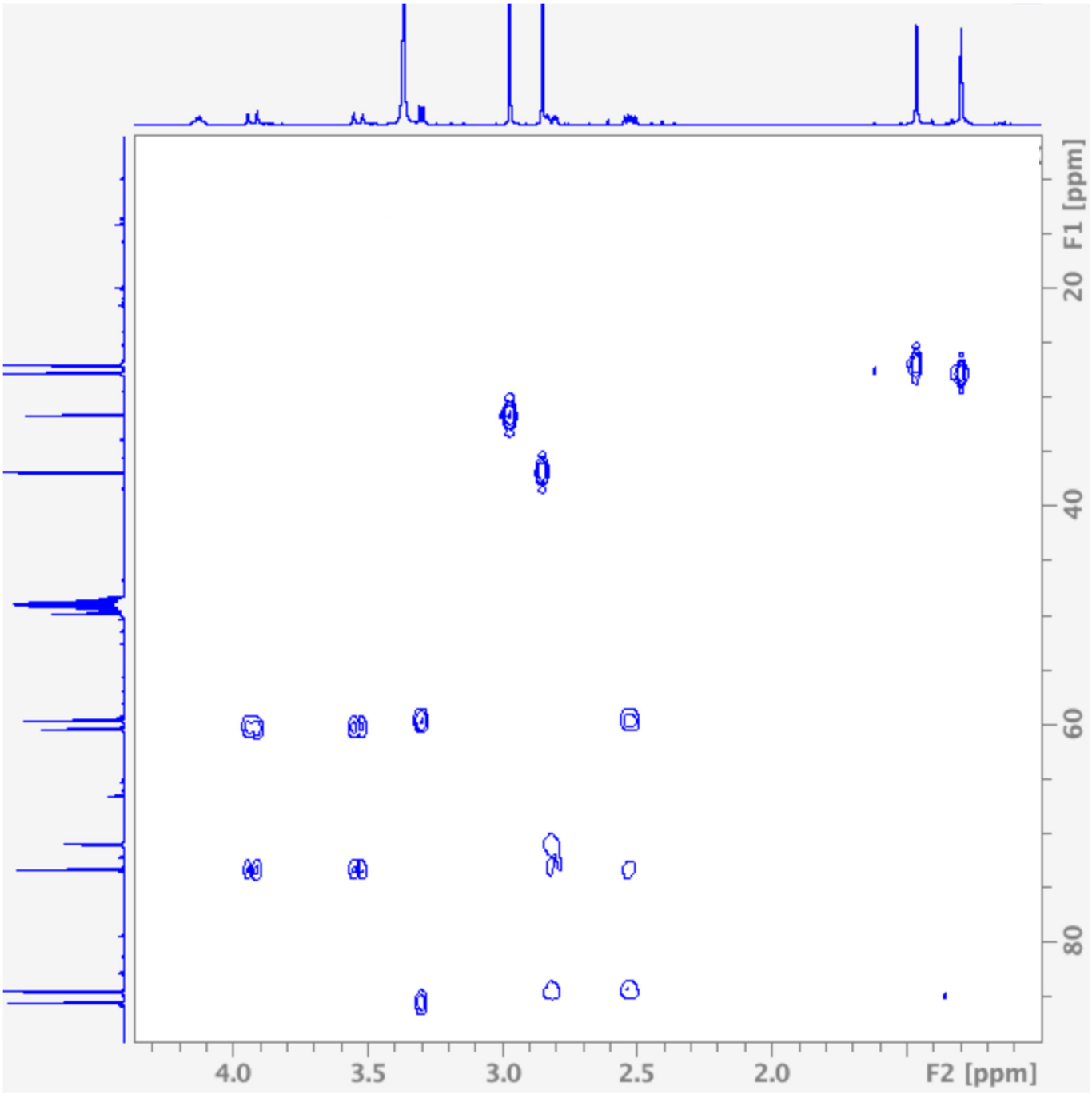
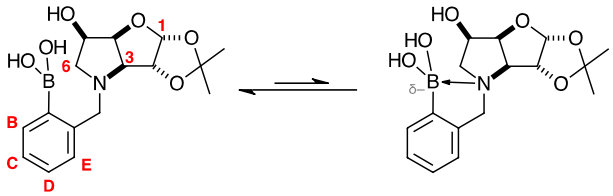

F1 [ppm]
20
40
60
80
4.0
3.5
3.0
2.5
2.0
F2 [ppm]

Figure S18. ^{1}H- (400 MHz, D_2O), ^{13}C-NMR (100 MHz), DEPT, ^{11}B-NMR (128 MHz), COSY and HSQC spectra of *N*-(2-methylphenyl boronic acid)-3,6-dideoxy-3,6-imino-1,2-*O*-isopropylidene-α-D-gulofuranose **ortho 5** in D_2O.



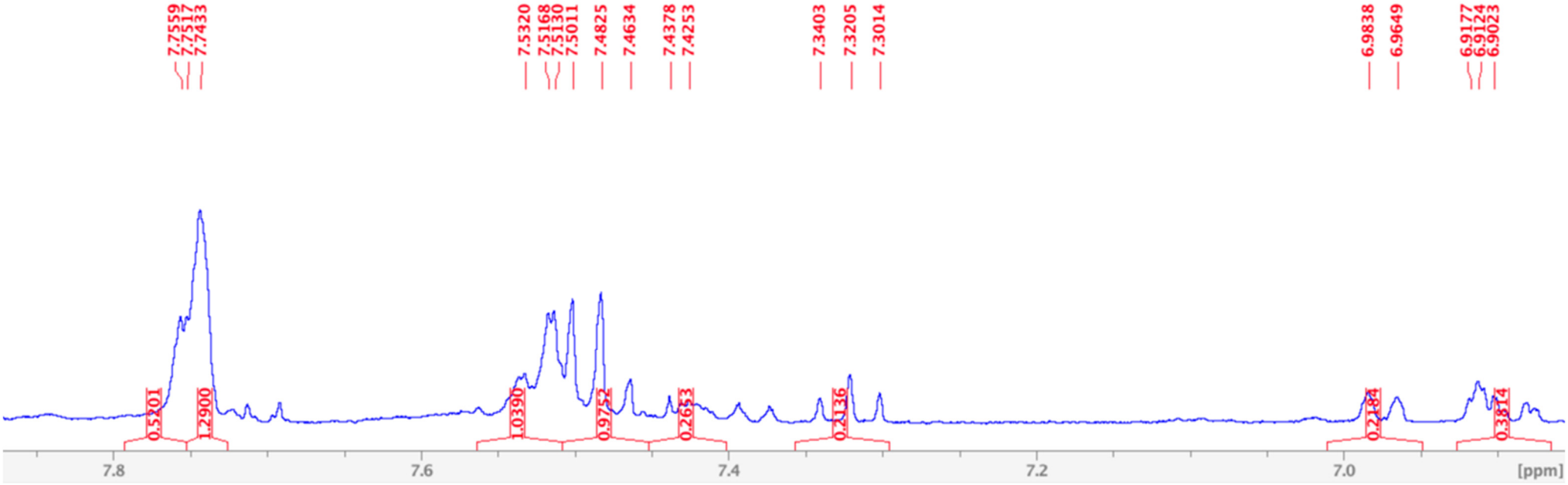
Ratio boronic acid species : boronate species, ~4.2 : 1.0 ratio.

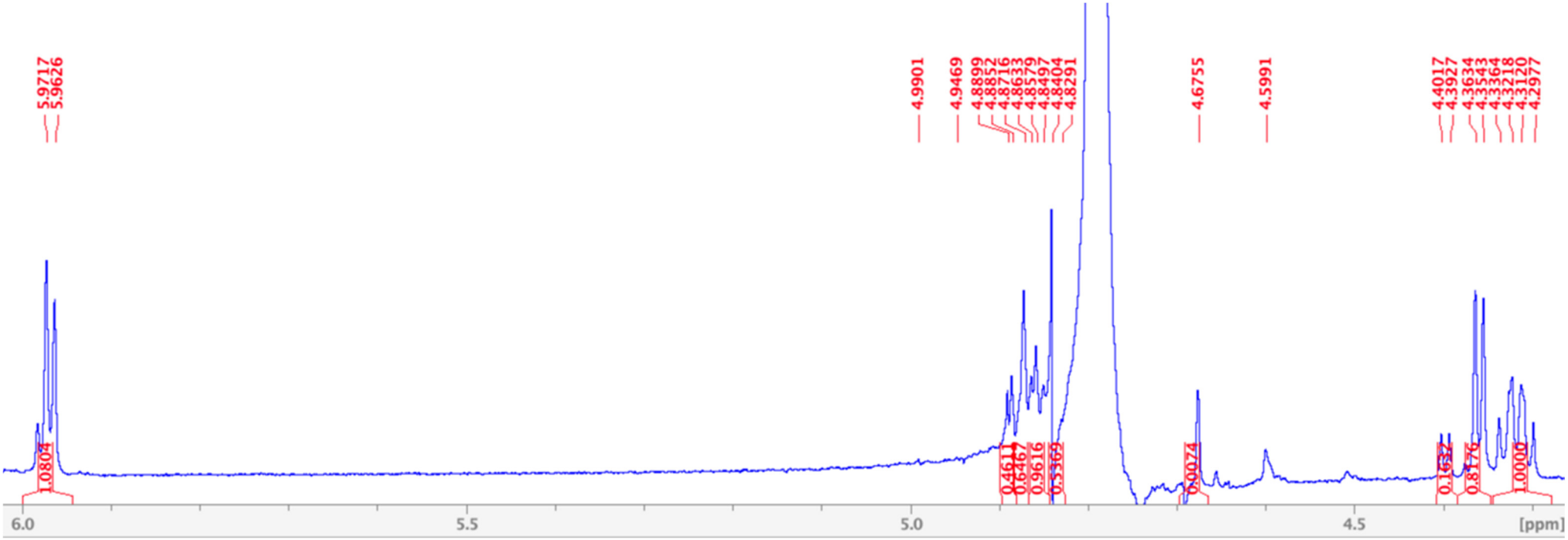
Boronic acid species. **δH:** 7.75 (1H, $J_{HB,HD}$ 1.6 Hz, partially obscured, ArH^{B}), 7.77-7.73 (1H, m, partially obscured, ArH^{E}), 7.53 (1H, app-dd or partially obscured td, $J_{HD,HC(/HE)}$ 7.6, $J_{HD,HB}$ 1.6 Hz, ArH^{D})), 7.48 (1H, app-t, $J_{HC,HB/HD}$ 7.6 Hz, ArH^{C}), 5.96 (1H, d, $J_{H\text{-}1,H\text{-}2}$ 3.7 Hz, H-1), 4.88 (1H, d, $J_{Ha,Hb}$ 5.5 Hz, ArC*H*^{a}H^{b}), 4.89-4.83 (1H, m, partially obscured, H-4), 4.36 (1H, d, $J_{H\text{-}2,H\text{-}1}$ 3.7 Hz, H-2) 4.31 (1H, ddd, $J_{H\text{-}5,H\text{-}6'}$ 6.0, $J_{H\text{-}5,H\text{-}6}$ 4.3 Hz, *J* 4.3 Hz, H-5), 3.83 (1H, d, *J* 2.1 Hz, H-3), 3.48 (1H, d, $J_{Hb,Ha}$ 5.7 Hz, ArCH^{a}*H*^{b}), 2.93, (1H, dd, $J_{H\text{-}6,H\text{-}6'}$ 11.3 Hz, $J_{H\text{-}6,H\text{-}5}$ 4.2 Hz, H-6), 2.83 (1H, dd, $J_{H\text{-}6',H\text{-}6}$ 11.4, $J_{H\text{-}6',H\text{-}5}$ 6.0 Hz, H-6'), 1.49 (3H, s, CH_3), 1.27 (3H, s, CH_3); **δc:** 135.9 (ArC$_{quat}$), 135.0 (ArC^{E}), 133.1 (ArC^{B}), 132.4 (ArC^{D}), 128.3 (ArC^{C}), 113.0 (C$_{quat}$), 106.71 (C-1), 83.9 (C-2), 83.4 (C-4), 71.1 (ArCH$_2$), 69.1 (C-5), 58.8 (C-6), 58.6 (C-3), 26.1, 25.3 (2 x CH_3).

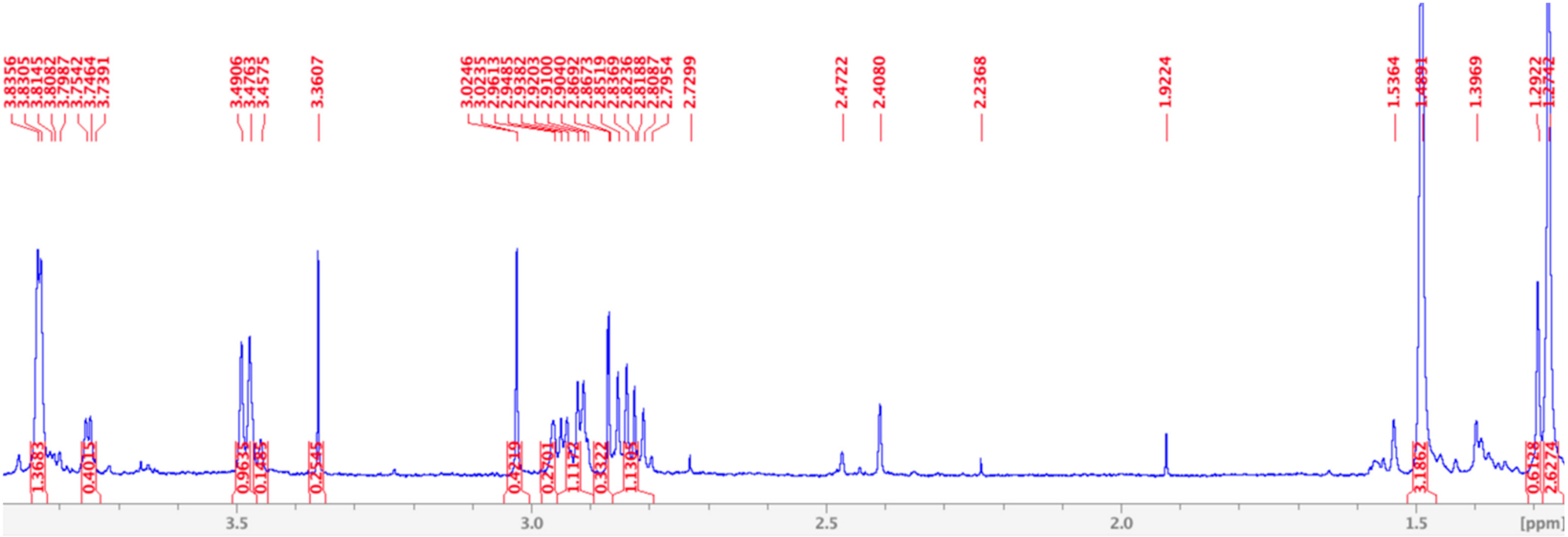
Boronate species. **δH:** 7.32 (1H, t, $J_{HD,HE\,=\,HD,HC}$ 7.8 Hz, ArH^{D}), 6.97 (1H, dt, $J_{HE,HD}$ 7.6 Hz, $J_{HE,HC\,=\,HE,HB}$ 1.1 Hz, ArH^{E}), 6.92-6.90 (1H, m, ArH^{B}), 6.89 (1H, ddd, $J_{HC,HD}$ 8.0 Hz, $J_{HC,HB}$ 2.5 Hz, $J_{HC,HE}$ 0.9 Hz, ArH^{C}), 5.98 (1H, d, partially obscured, H-1), 4.85-4.80 (1H, obscured, ArC*H*^{a}H^{b}), 4.40 (1H, d, $J_{H\text{-}2,H\text{-}1}$ 3.7 Hz, H-2), 3.75 (1H, app-d, *J* 3.2 Hz, H-3), 3.46 (1H, d, partially obscured, $J_{Hb,Ha}$ 5.9 Hz, H^{a}*H*^{b}), 2.96 (1H, app-s, partially obscured, H-5), 2.95-2.90 (1H, obscured, H-6'), 2.84-2.79 (1H, obscured, H-6), 1.49 (3H, s, CH_3 obscured), 1.29 (3H, s, CH_3); **δc:** 130.1 (ArC^{D}), 122.0 (ArC^{E}), 116.6 (ArC^{B}), 106.69 (C-1), 71.2 (ArCH$_2$), 58.6 (C-6), 58.4 (C-3), 26.1, 25.2 (2 x CH_3). Other C atoms were not discernible.

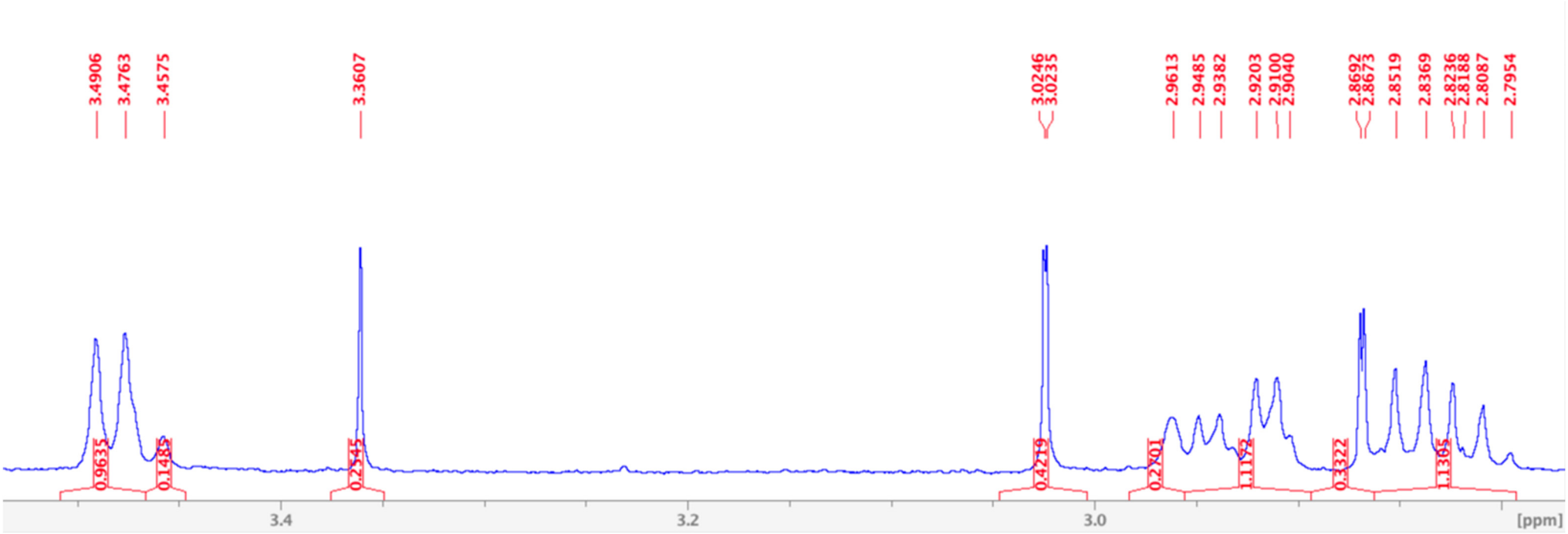
δB: 29.5 (broad, integration: 4.5), 19.3 (sharp, integration: 1.0).

^{1}H-NMR

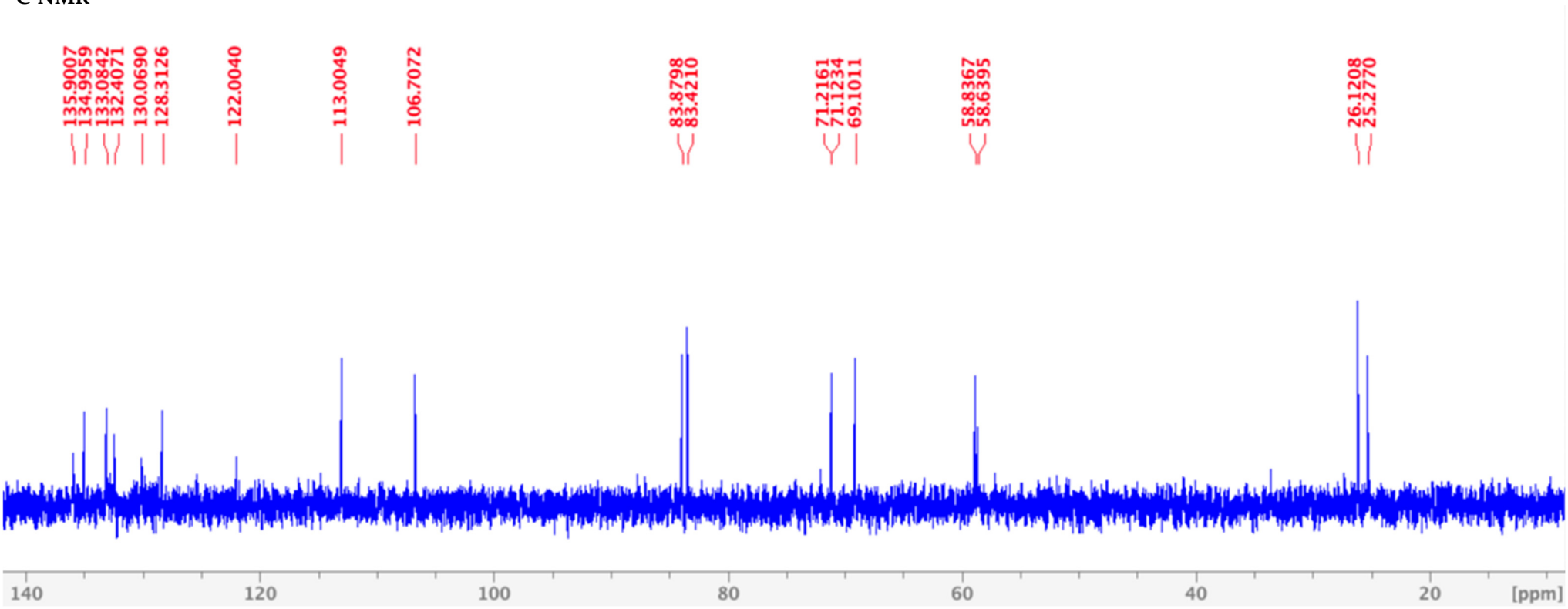

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7.5011
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7.4253
7.3403
7.3205
7.3014
6.9838
6.9649
6.9177
6.9124
6.9023
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0.2184
0.3814
7.8
7.6
7.4
7.2
7.0
[ppm]


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5.9626
4.9901
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4.6755
4.5991
4.4017
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4.3634
4.3543
4.3364
4.3218
4.3120
4.2977
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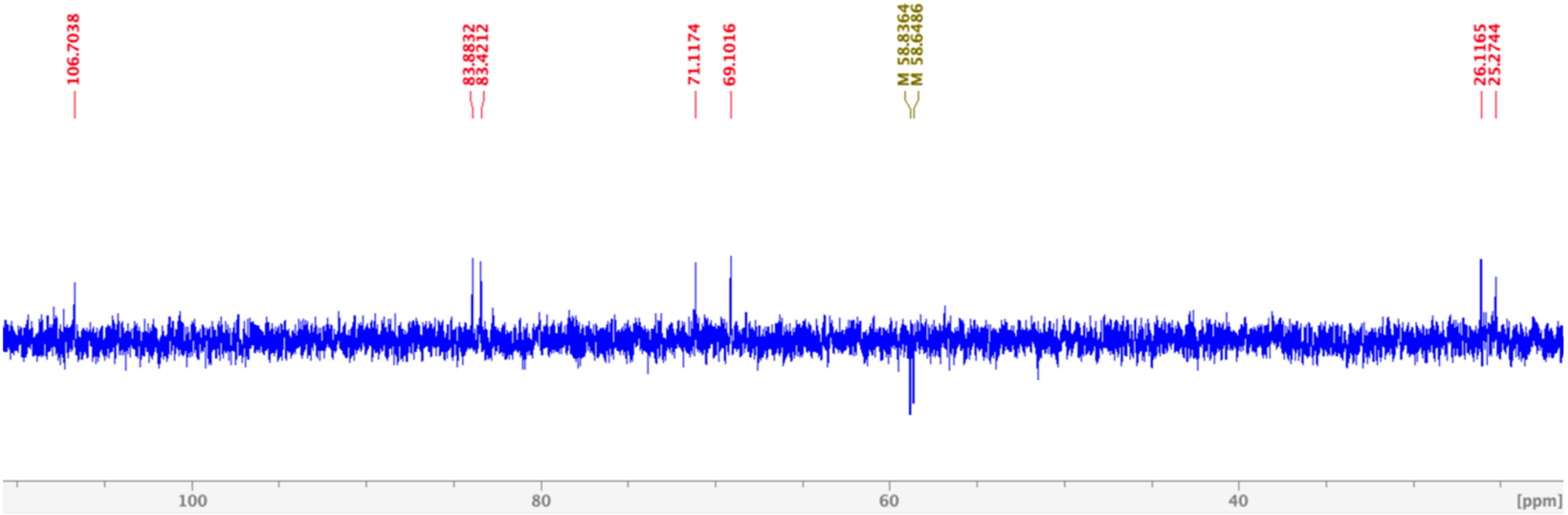

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3.0246
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2.9100
2.9040
2.8692
2.8673
2.8519
2.8369
2.8236
2.8188
2.8087
2.7954
2.7299
2.4722
2.4080
2.2368
1.9224
1.5364
1.4891
1.3969
1.2922
1.2742
1.3683
0.4015
0.9631
0.1485
0.2545
0.4219
0.2701
1.1172
0.3322
1.1363
3.1862
0.6138
2.6274
3.5
3.0
2.5
2.0
1.5
[ppm]


3.4906
3.4763
3.4575
3.3607
3.0246
3.0235
2.9613
2.9485
2.9382
2.9203
2.9100
2.9040
2.8692
2.8673
2.8519
2.8369
2.8236
2.8188
2.8087
2.7954
0.9635
0.1485
0.2545
0.4219
0.2701
1.1173
0.3322
1.1385
3.4
3.2
3.0
[ppm]

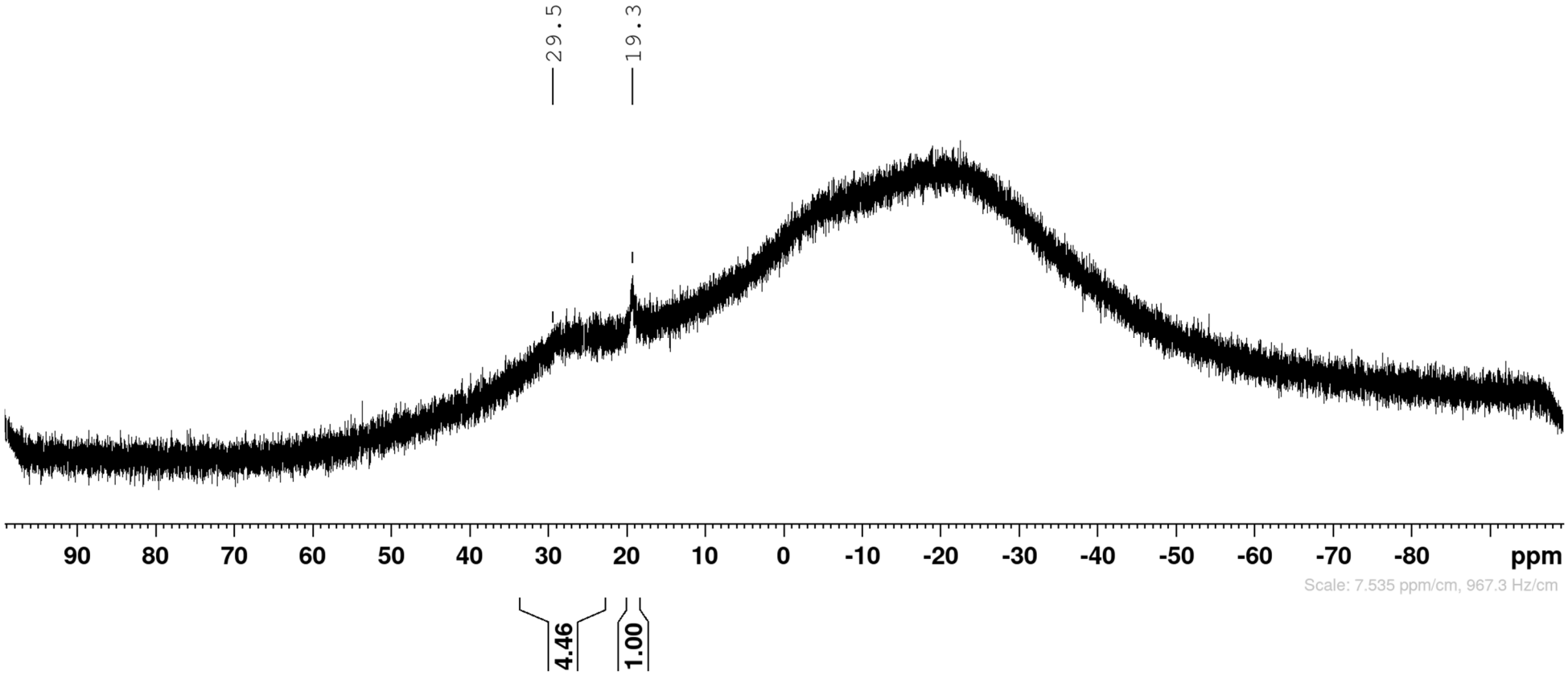
^{13}C-NMR


135.9007
134.9959
133.0842
132.4071
130.0690
128.3126
122.0040
113.0049
106.7072
83.8798
83.4210
71.2161
71.1234
69.1011
58.8367
58.6395
26.1208
25.2770
140
120
100
80
60
40
20
[ppm]

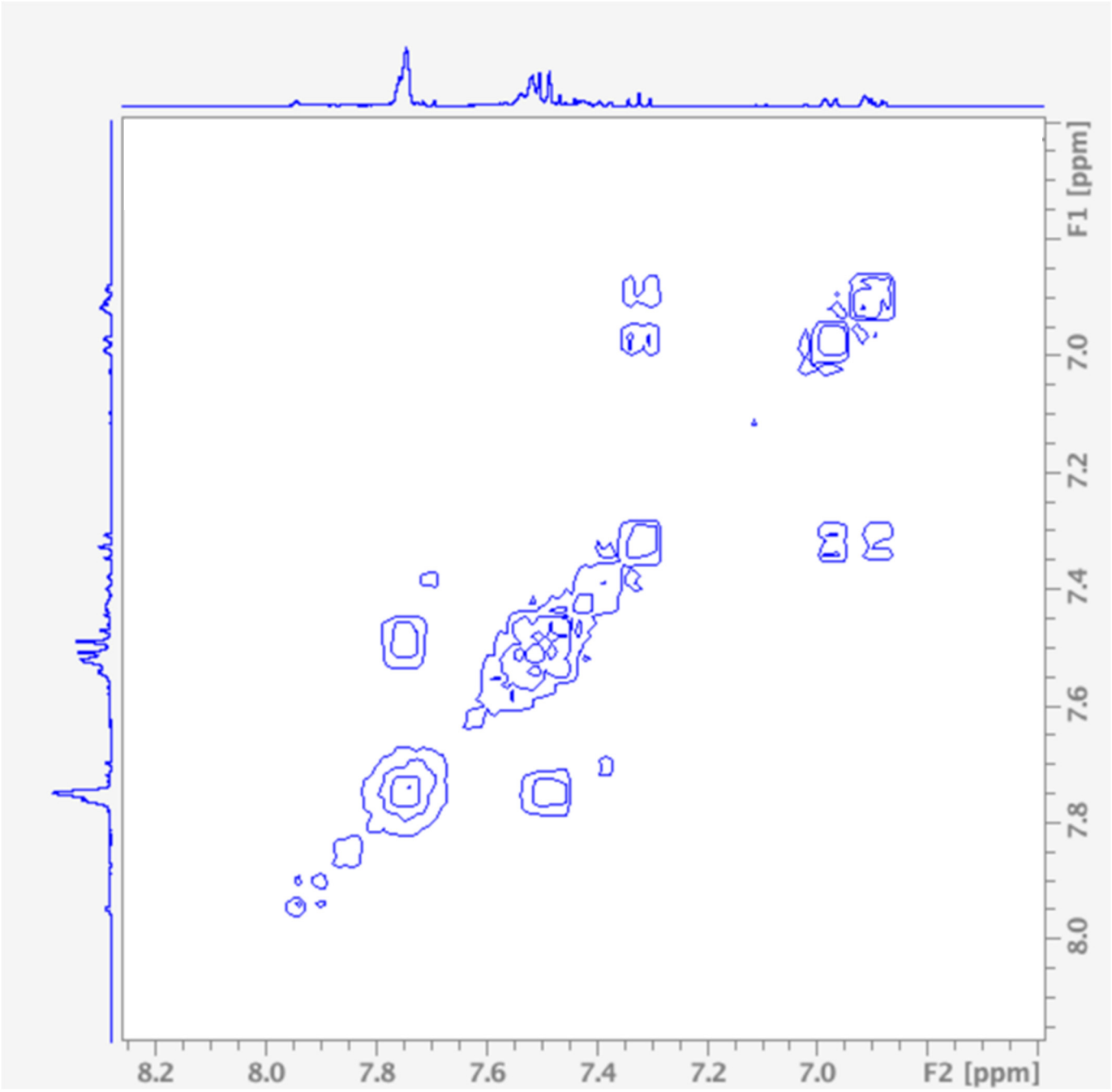
DEPT

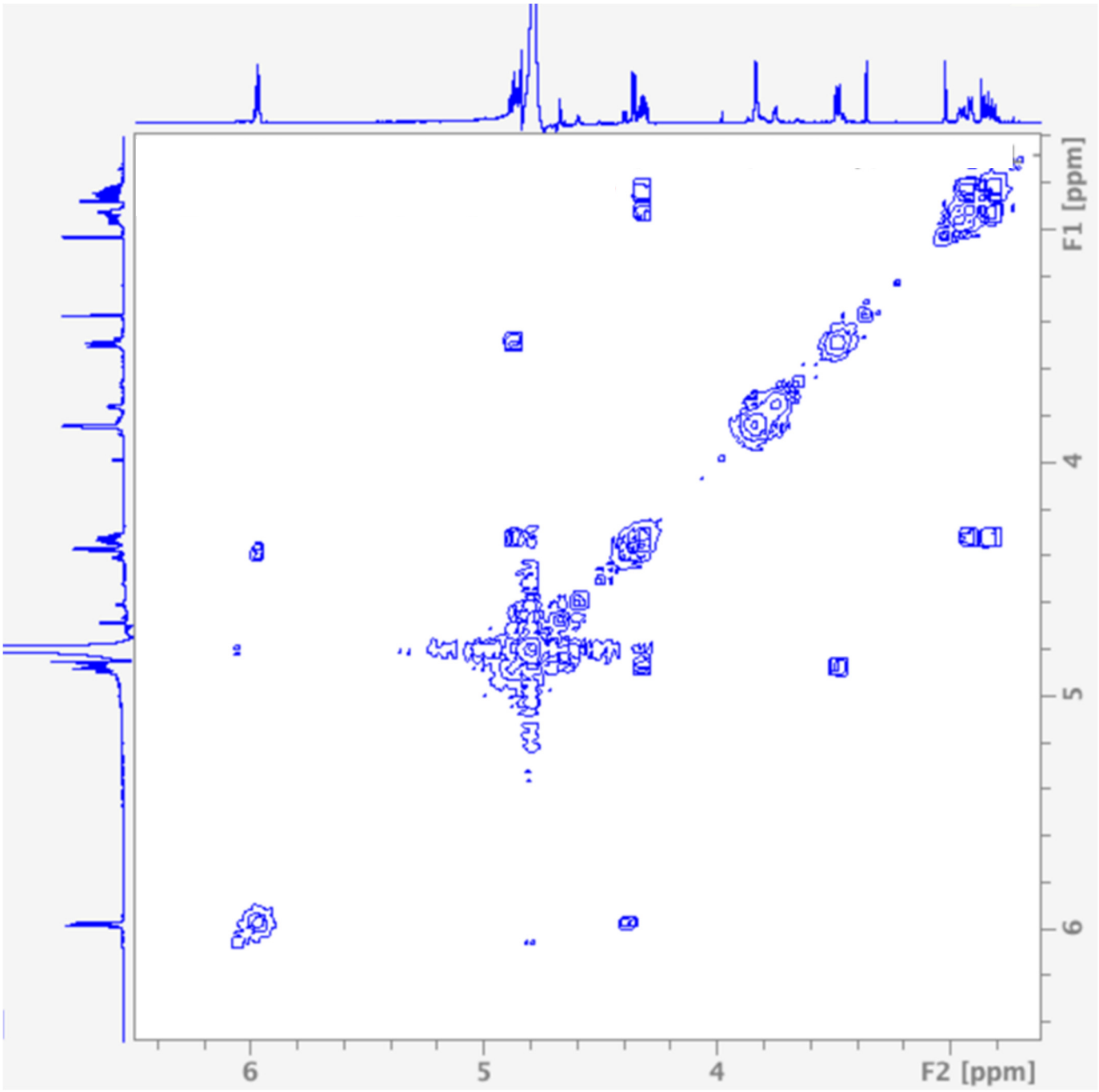

106.7038
83.8832
83.4212
71.1174
69.1016
M 58.8364
M 58.6486
26.1165
25.2744
100
80
60
40
[ppm]

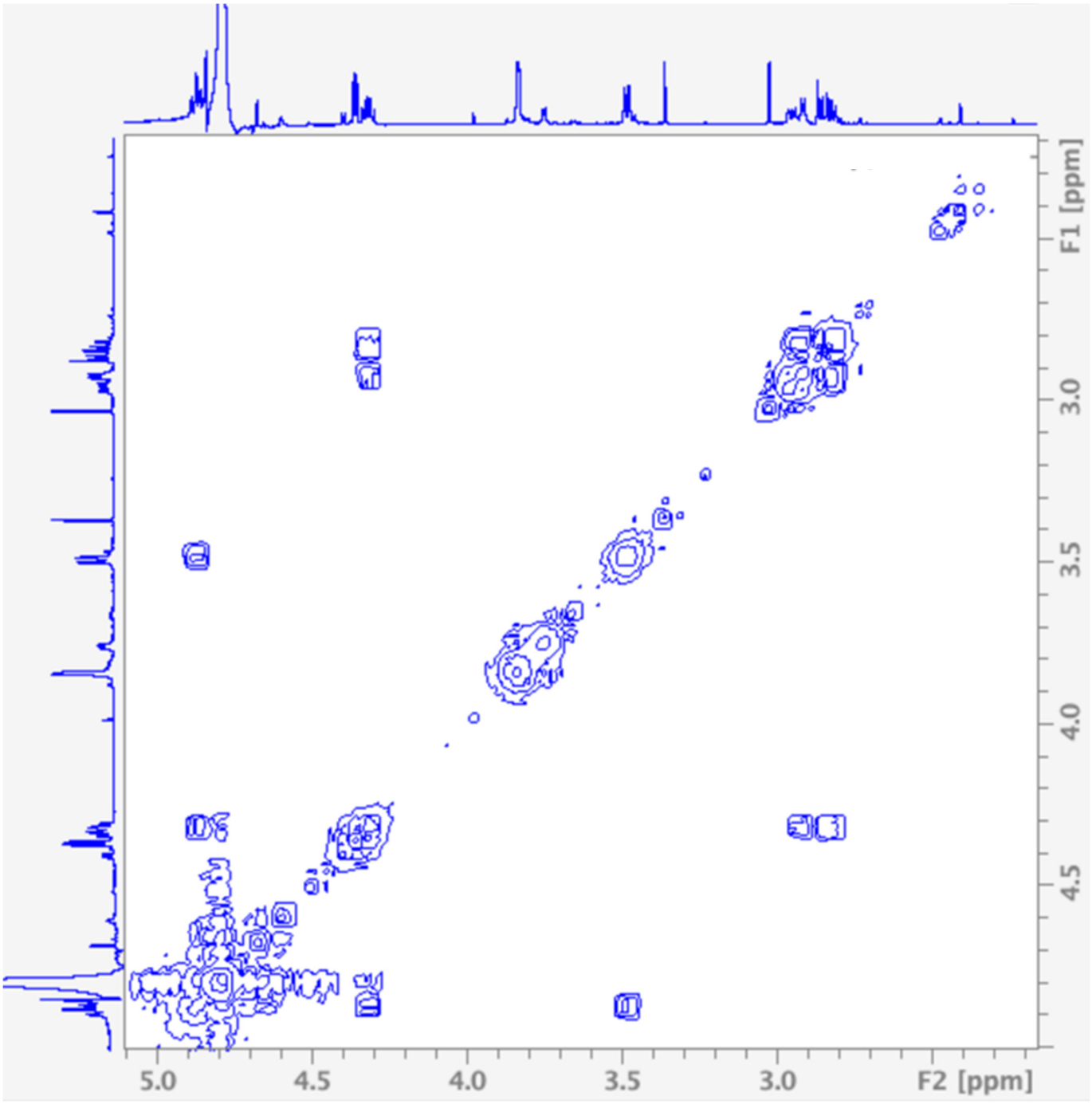
^{11}B-NMR


29.5
19.3
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
ppm
Scale: 7.535 ppm/cm, 967.3 Hz/cm
4.46
1.00

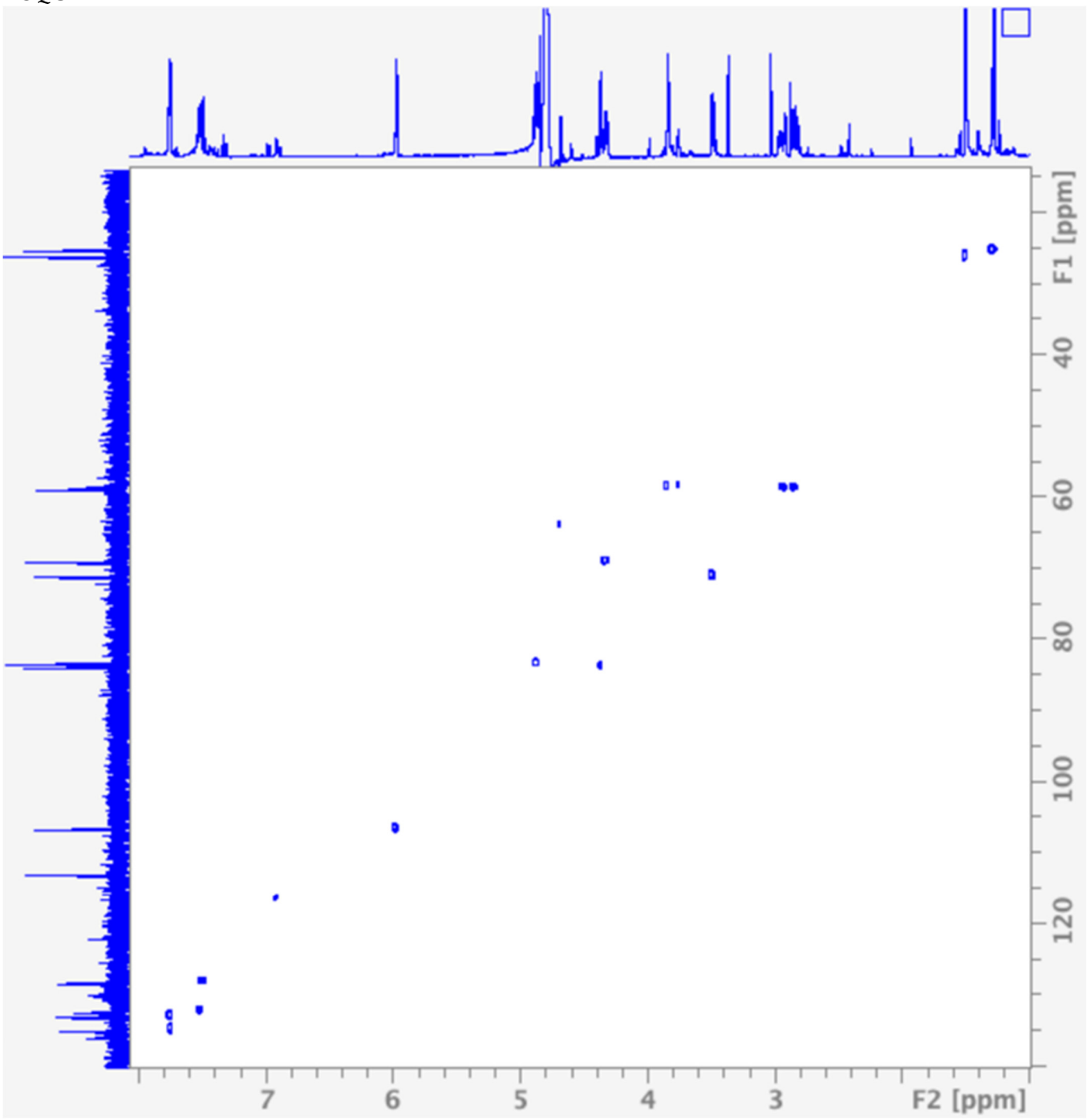
COSY

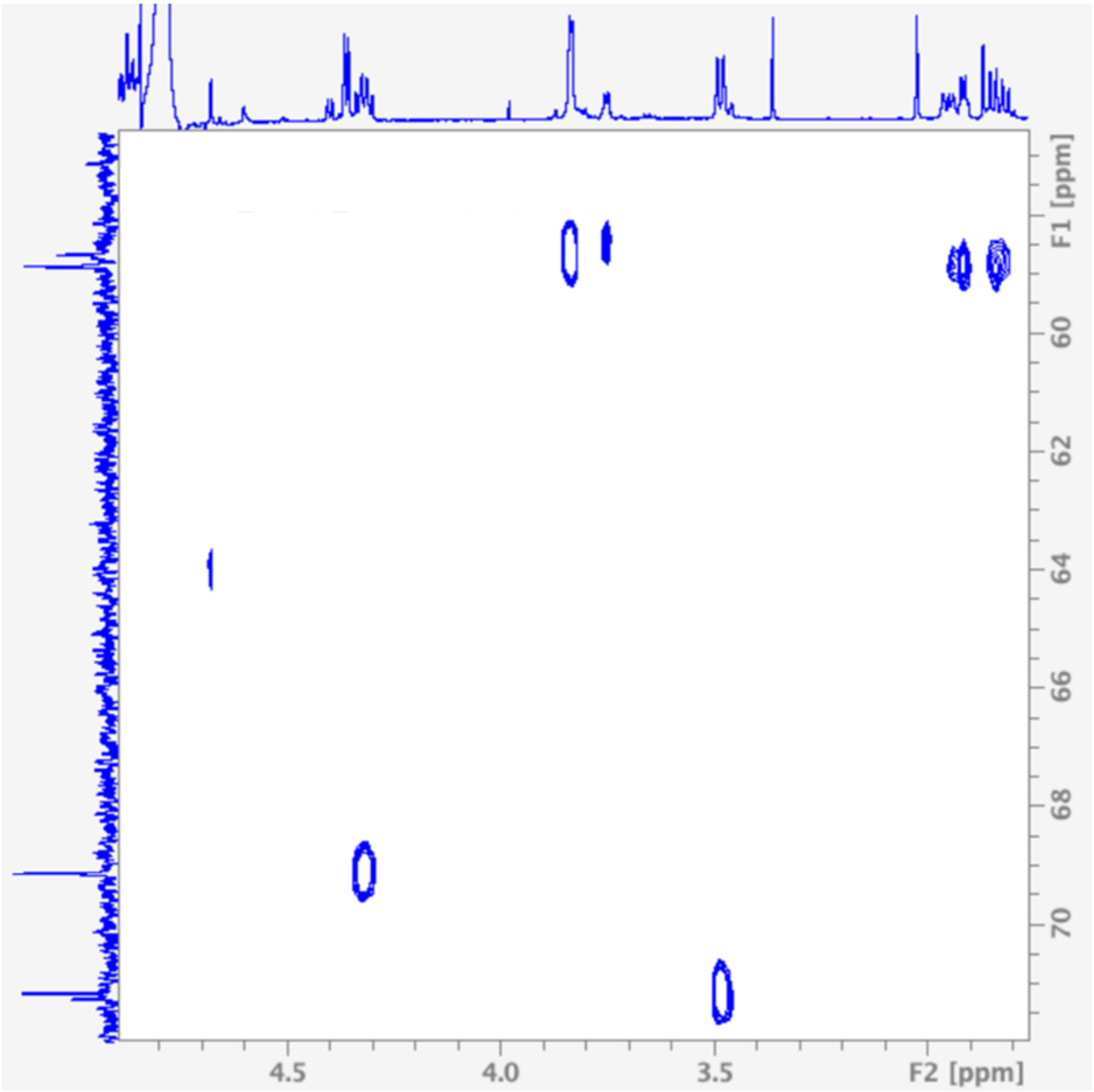


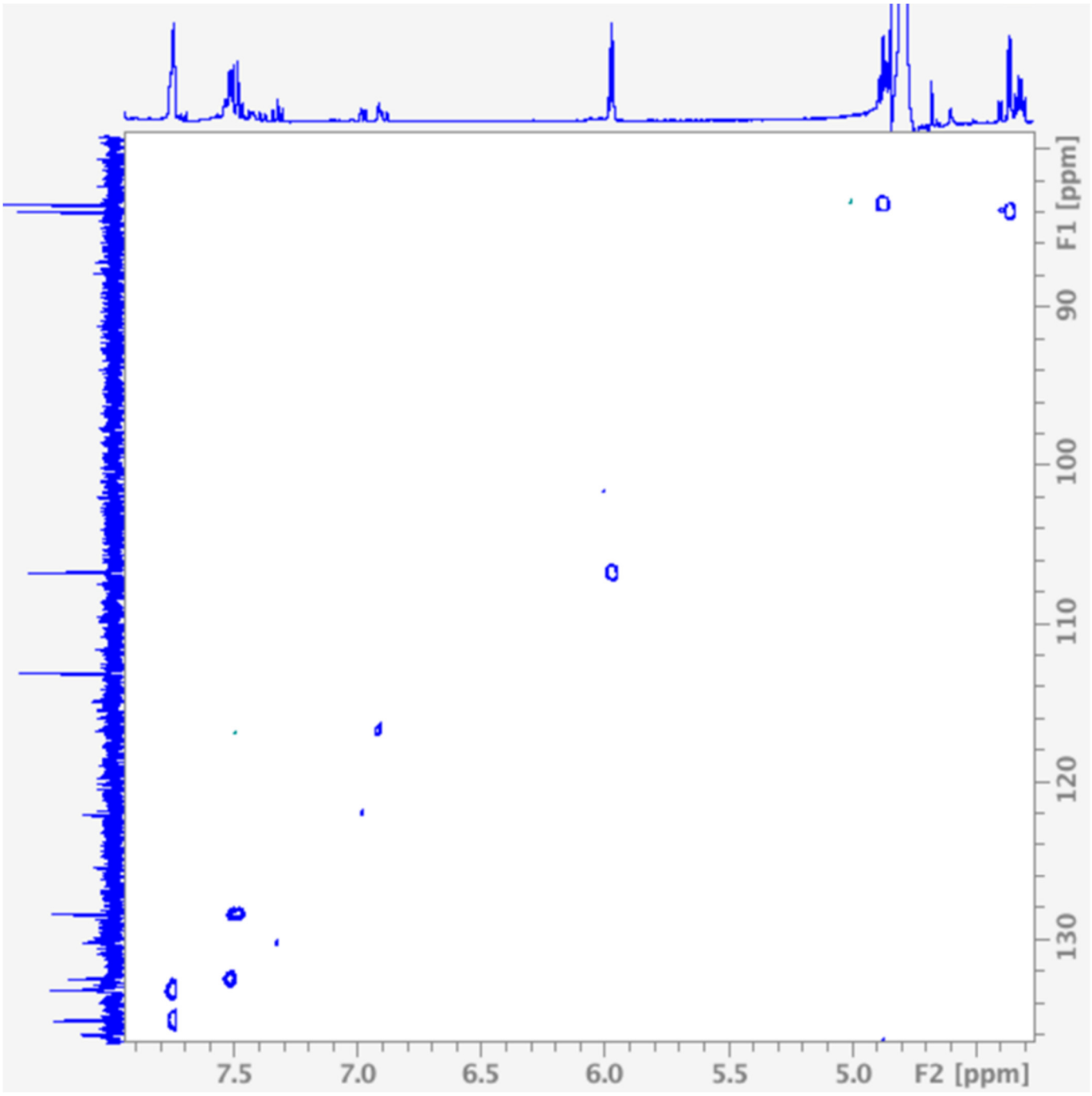

F1 [ppm]
4
5
6
6
5
4
F2 [ppm]


F1 [ppm]
3.0
3.5
4.0
4.5
5.0
4.5
4.0
3.5
3.0
F2 [ppm]

HSQC




F1 [ppm]
60
62
64
66
68
70
4.5
4.0
3.5
F2 [ppm]


F1 [ppm]
90
100
110
120
130
7.5
7.0
6.5
6.0
5.5
5.0
F2 [ppm]

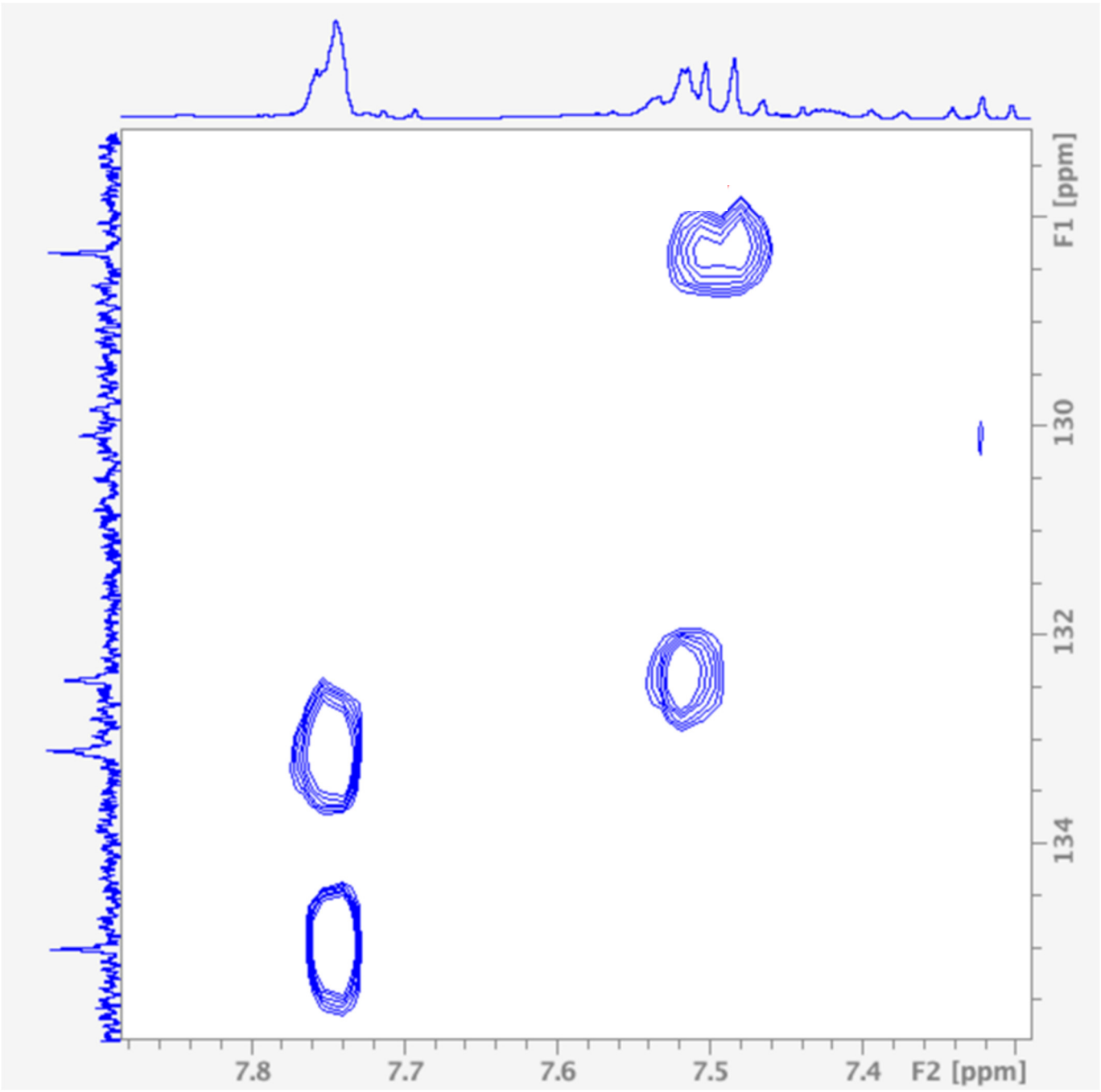
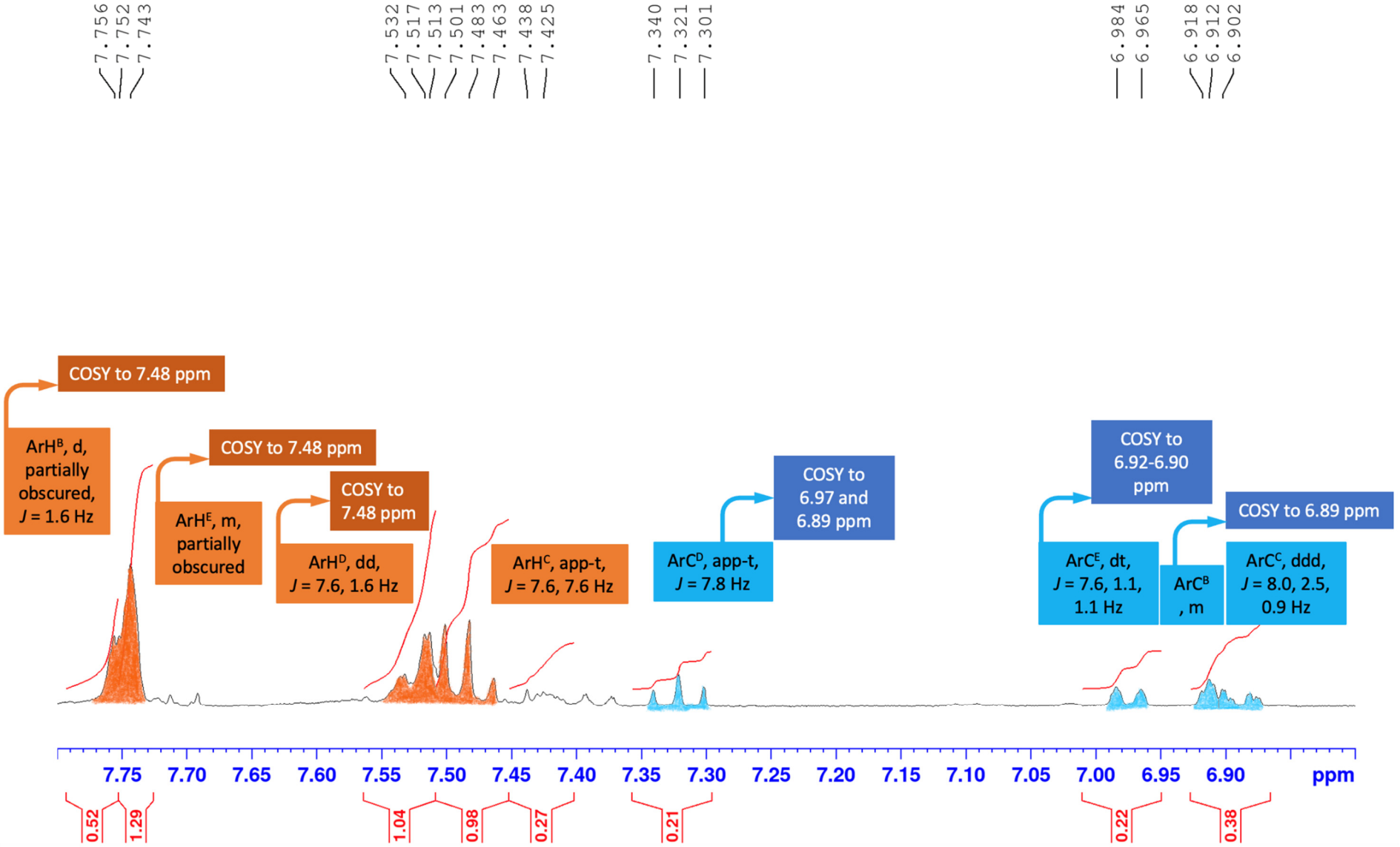
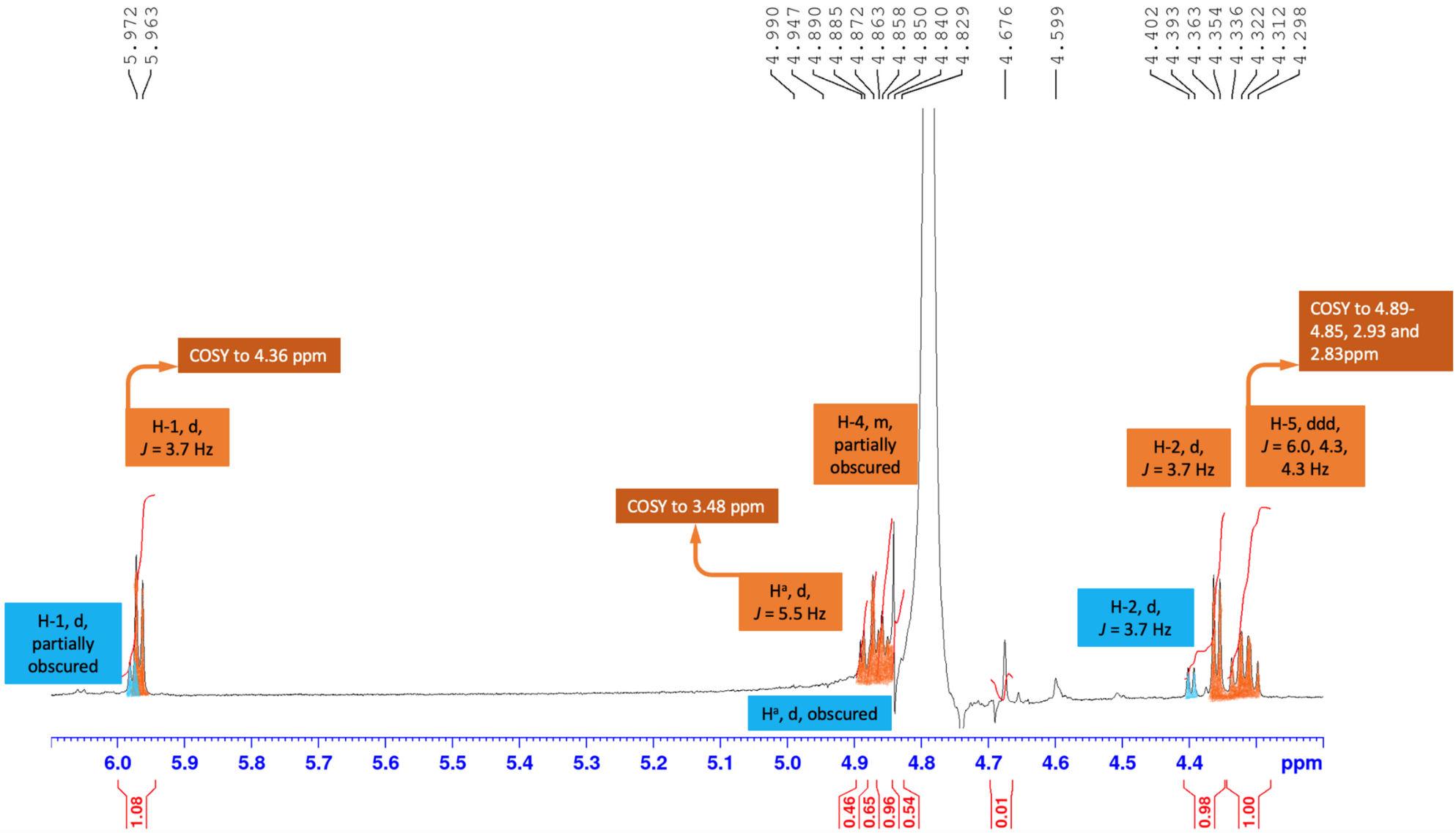

F1 [ppm]
130
132
134
7.8
7.7
7.6
7.5
7.4
F2 [ppm]

Figure S19. ^{1}H-NMR spectrum (400 MHz, D_2O) of compound **ortho 5** with colour-coded signals, highlighting the boronic acid and boronate forms they belong to, with interpretation of the isolated signals and tentative interpretation of the overlapping ones. Namely, the orange designates the boronic acid form and indigo designates the boronate form. A) section 7.80 ppm to 6.80 ppm; B) section 6.10 ppm to 4.20 ppm; C) section 3.90 ppm to 2.70 ppm; D) section 1.60 ppm to 1.25 ppm. Highlighted are also the principal COSY correlations to hydrogen atoms within the same spin systems.

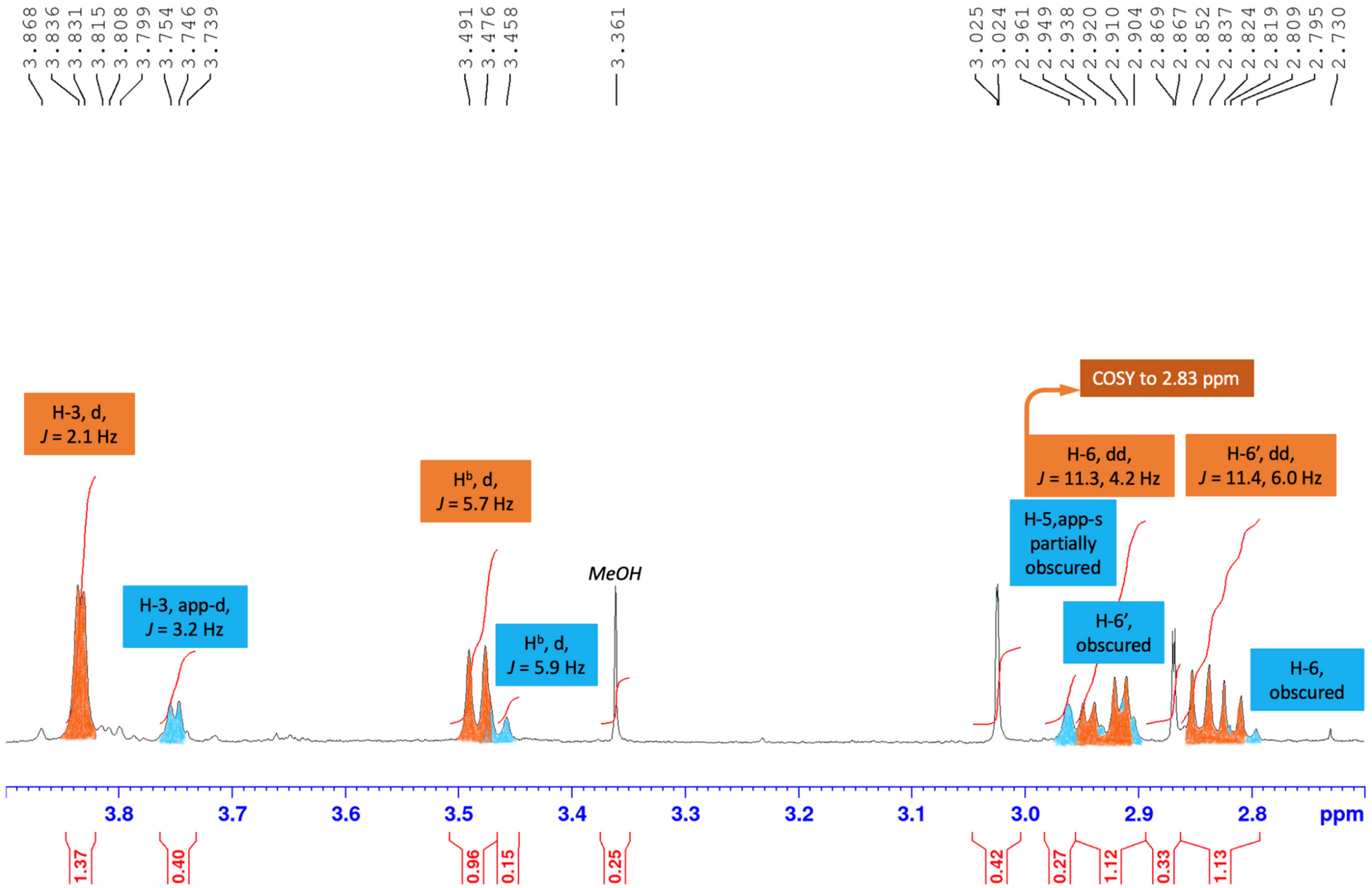


A

B


5.972
5.963
4.990
4.947
4.890
4.885
4.872
4.863
4.858
4.850
4.840
4.829
4.676
4.599
4.402
4.393
4.363
4.354
4.336
4.322
4.312
4.298
COSY to 4.36 ppm
H-1, d,
J = 3.7 Hz
H-1, d,
partially
obscured
COSY to 3.48 ppm
Hᵃ, d,
J = 5.5 Hz
Hᵃ, d, obscured
H-4, m,
partially
obscured
H-2, d,
J = 3.7 Hz
H-2, d,
J = 3.7 Hz
COSY to 4.89-
4.85, 2.93 and
2.83ppm
H-5, ddd,
J = 6.0, 4.3,
4.3 Hz
6.0
5.9
5.8
5.7
5.6
5.5
5.4
5.3
5.2
5.1
5.0
4.9
4.8
4.7
4.6
4.5
4.4
ppm
1.08
0.46
0.65
0.96
0.54
0.01
0.98
1.00

C


3.868
3.836
3.831
3.815
3.808
3.799
3.754
3.746
3.739
3.491
3.476
3.458
3.361
3.025
3.024
2.961
2.949
2.938
2.920
2.910
2.904
2.869
2.867
2.852
2.837
2.824
2.819
2.809
2.795
2.730
H-3, d, J = 2.1 Hz
H-3, app-d, J = 3.2 Hz
Hb, d, J = 5.7 Hz
Hb, d, J = 5.9 Hz
MeOH
COSY to 2.83 ppm
H-6, dd, J = 11.3, 4.2 Hz
H-6', dd, J = 11.4, 6.0 Hz
H-5,app-s partially obscured
H-6', obscured
H-6, obscured
3.8
3.7
3.6
3.5
3.4
3.3
3.2
3.1
3.0
2.9
2.8
ppm
1.37
0.40
0.96
0.15
0.25
0.42
0.27
1.12
0.33
1.13

D

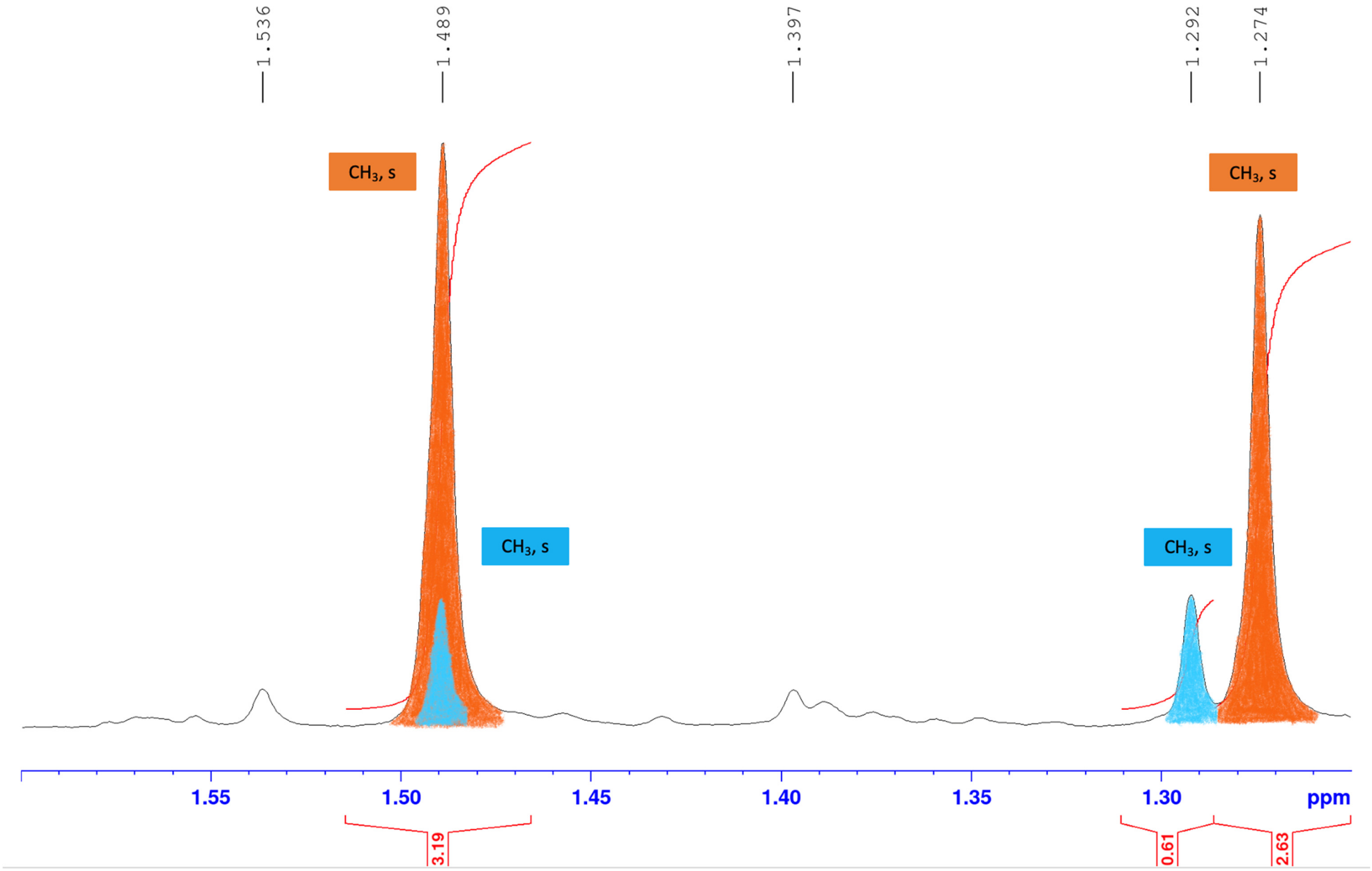
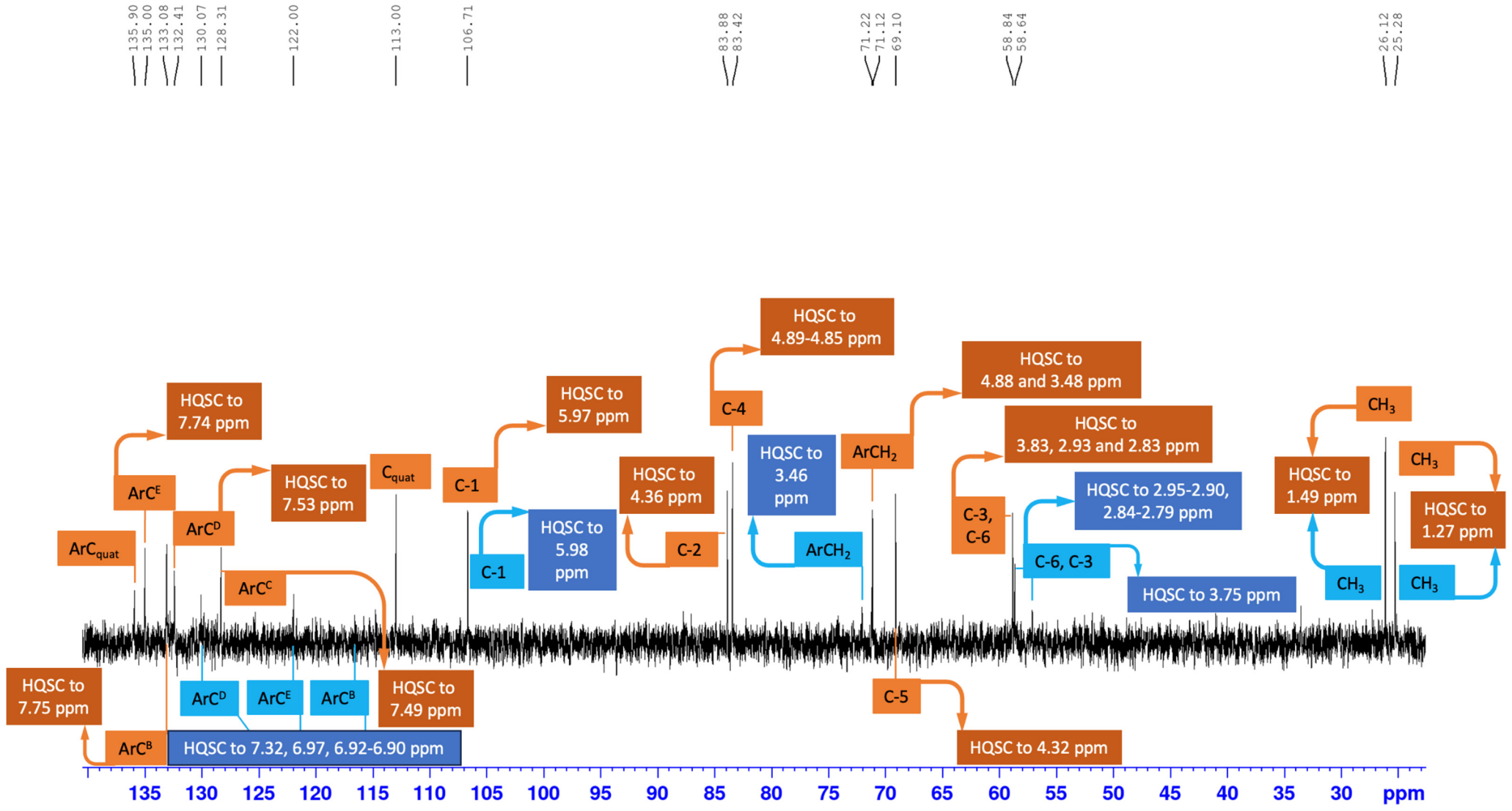

1.536
1.489
1.397
1.292
1.274
CH3, s
CH3, s
CH3, s
CH3, s
1.55
1.50
1.45
1.40
1.35
1.30
ppm
3.19
0.61
2.63

Figure S20. ^{13}C-NMR spectrum (100 MHz, D_2O) of compound **ortho 5** with colour-coded signals, highlighting the boronic acid and boronate forms they belong to, with interpretation of the isolated signals and tentative interpretation of the overlapping ones. Namely, the orange designates the boronic acid form and indigo designates the boronate form. A) section 140 ppm to 23 ppm.

A



NMR Experimental details

	para 6	para 7	para 8
^{1}H	F2 - Acquisition Parameters Date_ 20171025 Time 15.39 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zg TD 48076 SOLVENT CDCl3 NS 8 DS 2 SWH 6002.401 Hz FIDRES 0.124852 Hz AQ 4.0047307 sec RG 23.44 DW 83.300 usec DE 16.70 usec TE 298.2 K D1 5.00000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 400.1320007 MHz NUC1 1H P1 15.00 usec PLW1 12.50300026 W F2 - Processing parameters SI 32768 SF 400.1300096 MHz WDW EM SSB 0 LB 0.30 Hz GB 0 PC 1.00	F2 - Acquisition Parameters Date_ 20180209 Time 10.59 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zg30 TD 65536 SOLVENT D2O NS 16 DS 2 SWH 8012.820 Hz FIDRES 0.122266 Hz AQ 4.0894465 sec RG 86.04 DW 62.400 usec DE 6.50 usec TE 298.2 K D1 1.00000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 400.1324710 MHz NUC1 1H P1 15.00 usec PLW1 12.50300026 W F2 - Processing parameters SI 65536 SF 400.1299656 MHz WDW EM SSB 0 LB 0.30 Hz GB 0 PC 1.00	F2 - Acquisition Parameters Date_ 20180221 Time 11.07 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zg30 TD 65536 SOLVENT D2O NS 16 DS 2 SWH 8012.820 Hz FIDRES 0.122266 Hz AQ 4.0894465 sec RG 33.39 DW 62.400 usec DE 6.50 usec TE 298.2 K D1 1.00000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 400.1324710 MHz NUC1 1H P1 15.00 usec PLW1 12.50300026 W F2 - Processing parameters SI 65536 SF 400.1300000 MHz WDW EM SSB 0 LB 0.30 Hz GB 0 PC 1.00
^{13}C	F2 - Acquisition Parameters Date_ 20171027 Time 21.00 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zgpg30 TD 65536 SOLVENT CDCl3 NS 4000 DS 4 SWH 22058.824 Hz FIDRES 0.336591 Hz AQ 1.4854827 sec RG 192.58 DW 22.667 usec DE 6.50 usec TE 298.2 K D1 2.00000000 sec D11 0.03000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 100.6223253 MHz NUC1 13C P1 9.80 usec PLW1 60.95399857 W ======== CHANNEL f2 ======== SFO2 400.1316005 MHz NUC2 1H CPDPRG[2 waltz16 PCPD2 90.00 usec PLW2 12.50300026 W PLW12 0.34731001 W PLW13 0.28132001 W F2 - Processing parameters SI 65536 SF 100.6127589 MHz WDW EM SSB 0 LB 1.00 Hz GB 0 PC 1.40	F2 - Acquisition Parameters Date_ 20180213 Time 23.03 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zgpg30 TD 65536 SOLVENT D2O NS 3000 DS 4 SWH 22058.824 Hz FIDRES 0.336591 Hz AQ 1.4854827 sec RG 192.58 DW 22.667 usec DE 6.50 usec TE 298.2 K D1 2.00000000 sec D11 0.03000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 100.6223253 MHz NUC1 13C P1 9.80 usec PLW1 60.95399857 W ======== CHANNEL f2 ======== SFO2 400.1316005 MHz NUC2 1H CPDPRG[2 waltz16 PCPD2 90.00 usec PLW2 12.50300026 W PLW12 0.34731001 W PLW13 0.28132001 W F2 - Processing parameters SI 65536 SF 100.6127690 MHz WDW EM SSB 0 LB 1.00 Hz GB 0 PC 1.40	F2 - Acquisition Parameters Date_ 20180221 Time 20.00 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zgpg30 TD 65536 SOLVENT D2O NS 3000 DS 4 SWH 22058.824 Hz FIDRES 0.336591 Hz AQ 1.4854827 sec RG 192.58 DW 22.667 usec DE 6.50 usec TE 298.1 K D1 2.00000000 sec D11 0.03000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 100.6223253 MHz NUC1 13C P1 9.80 usec PLW1 60.95399857 W ======== CHANNEL f2 ======== SFO2 400.1316005 MHz NUC2 1H CPDPRG[2 waltz16 PCPD2 90.00 usec PLW2 12.50300026 W PLW12 0.34731001 W PLW13 0.28132001 W F2 - Processing parameters SI 65536 SF 100.6127690 MHz WDW EM SSB 0 LB 1.00 Hz GB 0 PC 1.40

DEPT

```
F2 - Acquisition Parameters
Date_           20171025
Time               15.56
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG        deptqgpsp
TD                 65536
SOLVENT            CDC13
NS                   256
DS                     4
SWH            22058.824 Hz
FIDRES          0.336591 Hz
AQ             1.4854827 sec
RG                192.58
DW                22.667 usec
DE                 6.50 usec
TE                298.2 K
CNST2         145.0000000
CNST12          1.5000000
D1           2.00000000 sec
D2           0.00344828 sec
D12          0.00002000 sec
D16          0.00020000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6223258 MHz
NUC1                 13C
P1                  9.80 usec
P13              2000.00 usec
PLW0                   0 W
PLW1         60.95399857 W
SPNAM[5]     Crp60comp.4
SPOAL5             0.500
SPOFFS5                0 Hz
SPW5          8.94419956 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
P0                 22.50 usec
P3                 15.00 usec
P4                 30.00 usec
PCPD2              90.00 usec
PLW2         12.50300026 W
PLW12         0.34731001 W

====== GRADIENT CHANNEL =====
GPNAM[1]       SMSQ10.32
GPNAM[2]       SMSQ10.32
GPNAM[3]       SMSQ10.32
GPZ1               31.00 %
GPZ2               31.00 %
GPZ3               31.00 %
P16              1000.00 usec

F2 - Processing parameters
SI                 65536
SF           100.6127584 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

```
F2 - Acquisition Parameters
Date_           20180209
Time               15.41
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG        deptqgpsp
TD                 65536
SOLVENT              D2O
NS                   256
DS                     4
SWH            22058.824 Hz
FIDRES          0.336591 Hz
AQ             1.4854827 sec
RG                192.58
DW                22.667 usec
DE                 6.50 usec
TE                298.2 K
CNST2         145.0000000
CNST12          1.5000000
D1           2.00000000 sec
D2           0.00344828 sec
D12          0.00002000 sec
D16          0.00020000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6223258 MHz
NUC1                 13C
P1                  9.80 usec
P13              2000.00 usec
PLW0                   0 W
PLW1         60.95399857 W
SPNAM[5]     Crp60comp.4
SPOAL5             0.500
SPOFFS5                0 Hz
SPW5          8.94419956 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
P0                 22.50 usec
P3                 15.00 usec
P4                 30.00 usec
PCPD2              90.00 usec
PLW2         12.50300026 W
PLW12         0.34731001 W

====== GRADIENT CHANNEL =====
GPNAM[1]       SMSQ10.32
GPNAM[2]       SMSQ10.32
GPNAM[3]       SMSQ10.32
GPZ1               31.00 %
GPZ2               31.00 %
GPZ3               31.00 %
P16              1000.00 usec

F2 - Processing parameters
SI                 65536
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

```
F2 - Acquisition Parameters
Date_           20180221
Time               16.18
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG        deptqgpsp
TD                 65536
SOLVENT              D2O
NS                   256
DS                     4
SWH            22058.824 Hz
FIDRES          0.336591 Hz
AQ             1.4854827 sec
RG                192.58
DW                22.667 usec
DE                 6.50 usec
TE                298.1 K
CNST2         145.0000000
CNST12          1.5000000
D1           2.00000000 sec
D2           0.00344828 sec
D12          0.00002000 sec
D16          0.00020000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6223258 MHz
NUC1                 13C
P1                  9.80 usec
P13              2000.00 usec
PLW0                   0 W
PLW1         60.95399857 W
SPNAM[5]     Crp60comp.4
SPOAL5             0.500
SPOFFS5                0 Hz
SPW5          8.94419956 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
P0                 22.50 usec
P3                 15.00 usec
P4                 30.00 usec
PCPD2              90.00 usec
PLW2         12.50300026 W
PLW12         0.34731001 W

====== GRADIENT CHANNEL =====
GPNAM[1]       SMSQ10.32
GPNAM[2]       SMSQ10.32
GPNAM[3]       SMSQ10.32
GPZ1               31.00 %
GPZ2               31.00 %
GPZ3               31.00 %
P16              1000.00 usec

F2 - Processing parameters
SI                 65536
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

^{11}B	F2 - Acquisition Parameters Date_ 20171026 Time 5.49 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zg TD 65536 SOLVENT CDCl3 NS 128 DS 4 SWH 25510.203 Hz FIDRES 0.389255 Hz AQ 1.2845056 sec RG 192.58 DW 19.600 usec DE 6.50 usec TE 298.1 K D1 1.00000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 128.3776052 MHz NUC1 11B P1 17.05 usec PLW1 11.69499969 W F2 - Processing parameters SI 32768 SF 128.3776052 MHz WDW no SSB 0 LB 0 Hz GB 0 PC 1.40	F2 - Acquisition Parameters Date_ 20180212 Time 5.50 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zg TD 65536 SOLVENT D2O NS 128 DS 4 SWH 25510.203 Hz FIDRES 0.389255 Hz AQ 1.2845056 sec RG 192.58 DW 19.600 usec DE 6.50 usec TE 298.1 K D1 1.00000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 128.3776052 MHz NUC1 11B P1 17.05 usec PLW1 11.69499969 W F2 - Processing parameters SI 32768 SF 128.3776052 MHz WDW no SSB 0 LB 0 Hz GB 0 PC 1.40	F2 - Acquisition Parameters Date_ 20180221 Time 20.24 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zg TD 65536 SOLVENT D2O NS 128 DS 4 SWH 25510.203 Hz FIDRES 0.389255 Hz AQ 1.2845056 sec RG 192.58 DW 19.600 usec DE 6.50 usec TE 298.1 K D1 1.00000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 128.3776052 MHz NUC1 11B P1 17.05 usec PLW1 11.69499969 W F2 - Processing parameters SI 32768 SF 128.3776052 MHz WDW no SSB 0 LB 0 Hz GB 0 PC 1.40

COSY

```
F2 - Acquisition Parameters
Date_           20171025
Time               15.58
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG         cosygpqf
TD                 2048
SOLVENT            CDCl3
NS                    1
DS                    8
SWH             4807.692 Hz
FIDRES          2.347506 Hz
AQ             0.2129920 sec
RG                86.04
DW               104.000 usec
DE                 6.50 usec
TE                298.0 K
D0            0.00000300 sec
D1            1.48689198 sec
D13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00020800 sec

======== CHANNEL f1 ========
SFO1         400.1322007 MHz
NUC1                1H
P0                15.00 usec
P1                15.00 usec
PLW1         12.50300026 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPZ1              10.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  128
SFO1            400.1322 MHz
FIDRES         75.120193 Hz
SW                12.015 ppm
FnMODE              QF

F2 - Processing parameters
SI                 1024
SF           400.1300631 MHz
WDW                SINE
SSB                   0
LB                    0 Hz
GB                    0
PC                 1.40

F1 - Processing parameters
SI                 1024
MC2                  QF
SF           400.1300630 MHz
WDW                SINE
SSB                   0
LB                    0 Hz
GB                    0
```

```
F2 - Acquisition Parameters
Date_           20180209
Time               15.48
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG         cosygpqf
TD                 2048
SOLVENT             D2O
NS                    1
DS                    8
SWH             4807.692 Hz
FIDRES          2.347506 Hz
AQ             0.2129920 sec
RG                192.58
DW               104.000 usec
DE                 6.50 usec
TE                298.1 K
D0            0.00000300 sec
D1            1.48689198 sec
D13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00020800 sec

======== CHANNEL f1 ========
SFO1         400.1322007 MHz
NUC1                1H
P0                15.00 usec
P1                15.00 usec
PLW1         12.50300026 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPZ1              10.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  128
SFO1            400.1322 MHz
FIDRES         75.120193 Hz
SW                12.015 ppm
FnMODE              QF

F2 - Processing parameters
SI                 1024
SF           400.1299556 MHz
WDW                SINE
SSB                   0
LB                    0 Hz
GB                    0
PC                 1.40

F1 - Processing parameters
SI                 1024
MC2                  QF
SF           400.1299552 MHz
WDW                SINE
SSB                   0
LB                    0 Hz
GB                    0
```

```
F2 - Acquisition Parameters
Date_           20180221
Time               16.25
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG         cosygpqf
TD                 2048
SOLVENT             D2O
NS                    1
DS                    8
SWH             4807.692 Hz
FIDRES          2.347506 Hz
AQ             0.2129920 sec
RG                192.58
DW               104.000 usec
DE                 6.50 usec
TE                298.1 K
D0            0.00000300 sec
D1            1.48689198 sec
D13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00020800 sec

======== CHANNEL f1 ========
SFO1         400.1322007 MHz
NUC1                1H
P0                15.00 usec
P1                15.00 usec
PLW1         12.50300026 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPZ1              10.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  128
SFO1            400.1322 MHz
FIDRES         75.120193 Hz
SW                12.015 ppm
FnMODE              QF

F2 - Processing parameters
SI                 1024
SF           400.1300000 MHz
WDW                SINE
SSB                   0
LB                    0 Hz
GB                    0
PC                 1.40

F1 - Processing parameters
SI                 1024
MC2                  QF
SF           400.1300000 MHz
WDW                SINE
SSB                   0
LB                    0 Hz
GB                    0
```

HSQC

```
F2 - Acquisition Parameters
Date_           20171025
Time               16.28
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG  hsqcetgpsisp2.2
TD                 2048
SOLVENT            CDCl3
NS                    2
DS                   16
SWH             5341.880 Hz
FIDRES          2.608340 Hz
AQ             0.1916928 sec
RG                192.58
DW                93.600 usec
DE                 6.50 usec
TE                298.3 K
CNST2        145.0000000
CNST17        -0.5000000
D0             0.00000300 sec
D1             1.50000000 sec
D4             0.00172414 sec
D11            0.03000000 sec
D16            0.00020000 sec
D24            0.00086207 sec
IN0            0.00003000 sec

======== CHANNEL f1 ========
SFO1         400.1324057 MHz
NUC1                1H
P1                15.00 usec
P2                30.00 usec
P28             1000.00 usec
PLW1         12.50300026 W

======== CHANNEL f2 ========
SFO2         100.6202713 MHz
NUC2                13C
CPDPRG[2 bi_p5m4sp_4sp.2
P3                 9.80 usec
P14              500.00 usec
P24             2000.00 usec
P63             1500.00 usec
PLW0                  0 W
PLW2         60.95399857 W
PLW12         0.91469002 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3             0.500
SPOFFS3                0 Hz
SPW3          8.94419956 W
SPNAM[7]    Crp60comp.4
SPOAL7             0.500
SPOFFS7                0 Hz
SPW7          8.94419956 W
SPNAM[14 Crp32,1.5,20.2
SPOAL14            0.500
SPOFFS14               0 Hz
SPW14         3.81620002 W
SPNAM[31 Crp32,1.5,20.2
SPOAL31            0.500
SPOFFS31               0 Hz
SPW31         0.95405000 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPZ1               80.00 %
GPZ2               20.10 %
GPZ3               11.00 %
GPZ4               -5.00 %
P16              1000.00 usec
P19               600.00 usec

F1 - Acquisition parameters
TD                   256
SFO1           100.6203 MHz
FIDRES         130.208328 Hz
SW               165.639 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                  1024
SF           400.1300624 MHz
WDW                QSINE
SSB                    2
LB                     0 Hz
GB                     0
PC                  1.40

F1 - Processing parameters
SI                  1024
MC2         echo-antiecho
SF           100.6127604 MHz
WDW                QSINE
SSB                    2
LB                     0 Hz
GB                     0
```

```
F2 - Acquisition Parameters
Date_           20180213
Time               23.06
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG  hsqcetgpsisp2.2
TD                 2048
SOLVENT             D2O
NS                    2
DS                   16
SWH             5341.880 Hz
FIDRES          2.608340 Hz
AQ             0.1916928 sec
RG                192.58
DW                93.600 usec
DE                 6.50 usec
TE                298.0 K
CNST2        145.0000000
CNST17        -0.5000000
D0             0.00000300 sec
D1             1.50000000 sec
D4             0.00172414 sec
D11            0.03000000 sec
D16            0.00020000 sec
D24            0.00086207 sec
IN0            0.00003000 sec

======== CHANNEL f1 ========
SFO1         400.1324057 MHz
NUC1                1H
P1                15.00 usec
P2                30.00 usec
P28             1000.00 usec
PLW1         12.50300026 W

======== CHANNEL f2 ========
SFO2         100.6202713 MHz
NUC2                13C
CPDPRG[2 bi_p5m4sp_4sp.2
P3                 9.80 usec
P14              500.00 usec
P24             2000.00 usec
P63             1500.00 usec
PLW0                  0 W
PLW2         60.95399857 W
PLW12         0.91469002 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3             0.500
SPOFFS3                0 Hz
SPW3          8.94419956 W
SPNAM[7]    Crp60comp.4
SPOAL7             0.500
SPOFFS7                0 Hz
SPW7          8.94419956 W
SPNAM[14 Crp32,1.5,20.2
SPOAL14            0.500
SPOFFS14               0 Hz
SPW14         3.81620002 W
SPNAM[31 Crp32,1.5,20.2
SPOAL31            0.500
SPOFFS31               0 Hz
SPW31         0.95405000 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPZ1               80.00 %
GPZ2               20.10 %
GPZ3               11.00 %
GPZ4               -5.00 %
P16              1000.00 usec
P19               600.00 usec

F1 - Acquisition parameters
TD                   256
SFO1           100.6203 MHz
FIDRES         130.208328 Hz
SW               165.639 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                  1024
SF           400.1299619 MHz
WDW                QSINE
SSB                    2
LB                     0 Hz
GB                     0
PC                  1.40

F1 - Processing parameters
SI                  1024
MC2         echo-antiecho
SF           100.6127690 MHz
WDW                QSINE
SSB                    2
LB                     0 Hz
GB                     0
```

```
F2 - Acquisition Parameters
Date_           20180221
Time               20.03
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG  hsqcetgpsisp2.2
TD                 2048
SOLVENT             D2O
NS                    2
DS                   16
SWH             5341.880 Hz
FIDRES          2.608340 Hz
AQ             0.1916928 sec
RG                192.58
DW                93.600 usec
DE                 6.50 usec
TE                298.0 K
CNST2        145.0000000
CNST17        -0.5000000
D0             0.00000300 sec
D1             1.50000000 sec
D4             0.00172414 sec
D11            0.03000000 sec
D16            0.00020000 sec
D24            0.00086207 sec
IN0            0.00003000 sec

======== CHANNEL f1 ========
SFO1         400.1324057 MHz
NUC1                1H
P1                15.00 usec
P2                30.00 usec
P28             1000.00 usec
PLW1         12.50300026 W

======== CHANNEL f2 ========
SFO2         100.6202713 MHz
NUC2                13C
CPDPRG[2 bi_p5m4sp_4sp.2
P3                 9.80 usec
P14              500.00 usec
P24             2000.00 usec
P63             1500.00 usec
PLW0                  0 W
PLW2         60.95399857 W
PLW12         0.91469002 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3             0.500
SPOFFS3                0 Hz
SPW3          8.94419956 W
SPNAM[7]    Crp60comp.4
SPOAL7             0.500
SPOFFS7                0 Hz
SPW7          8.94419956 W
SPNAM[14 Crp32,1.5,20.2
SPOAL14            0.500
SPOFFS14               0 Hz
SPW14         3.81620002 W
SPNAM[31 Crp32,1.5,20.2
SPOAL31            0.500
SPOFFS31               0 Hz
SPW31         0.95405000 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPZ1               80.00 %
GPZ2               20.10 %
GPZ3               11.00 %
GPZ4               -5.00 %
P16              1000.00 usec
P19               600.00 usec

F1 - Acquisition parameters
TD                   256
SFO1           100.6203 MHz
FIDRES         130.208328 Hz
SW               165.639 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                  1024
SF           400.1300000 MHz
WDW                QSINE
SSB                    2
LB                     0 Hz
GB                     0
PC                  1.40

F1 - Processing parameters
SI                  1024
MC2         echo-antiecho
SF           100.6127690 MHz
WDW                QSINE
SSB                    2
LB                     0 Hz
GB                     0
```

HMBC

```
F2 - Acquisition Parameters
Date_           20171025
Time              16.11
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG    hmbcgpl2ndqf
TD                2048
SOLVENT           CDCl3
NS                   4
DS                  16
SWH           4807.692 Hz
FIDRES        2.347506 Hz
AQ           0.2129920 sec
RG              192.58
DW             104.000 usec
DE               6.50 usec
TE              298.2 K
CNST6       125.0000000
CNST7       165.0000000
CNST13        7.5000000
D0          0.00000300 sec
D1          1.50000000 sec
D6          0.06666667 sec
D16         0.00020000 sec
IN0         0.00002240 sec

======== CHANNEL f1 ========
SFO1        400.1322007 MHz
NUC1               1H
P1                15.00 usec
P2                30.00 usec
PLW1        12.50300026 W

======== CHANNEL f2 ========
SFO2        100.6228119 MHz
NUC2               13C
P3                 9.80 usec
PLW2        60.95399857 W

====== GRADIENT CHANNEL =====
GPNAM[1]     SMSQ10.100
GPNAM[2]     SMSQ10.100
GPNAM[3]     SMSQ10.100
GPNAM[4]     SMSQ10.100
GPNAM[5]     SMSQ10.100
GPNAM[6]     SMSQ10.100
GPZ1              50.00 %
GPZ2              30.00 %
GPZ3              40.10 %
GPZ4              15.00 %
GPZ5             -10.00 %
GPZ6              -5.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                 128
SFO1          100.6228 MHz
FIDRES       348.772308 Hz
SW              221.833 ppm
FnMODE              QF

F2 - Processing parameters
SI                2048
SF          400.1300628 MHz
WDW               SINE
SSB                  0
LB                   0 Hz
GB                   0
PC                1.40

F1 - Processing parameters
SI                1024
MC2                 QF
SF          100.6127463 MHz
WDW               SINE
SSB                  0
LB                   0 Hz
GB                   0
```

```
F2 - Acquisition Parameters
Date_           20180209
Time              15.54
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG    hmbcgpl2ndqf
TD                2048
SOLVENT            D2O
NS                   4
DS                  16
SWH           4807.692 Hz
FIDRES        2.347506 Hz
AQ           0.2129920 sec
RG              192.58
DW             104.000 usec
DE               6.50 usec
TE              298.1 K
CNST6       125.0000000
CNST7       165.0000000
CNST13        7.5000000
D0          0.00000300 sec
D1          1.50000000 sec
D6          0.06666667 sec
D16         0.00020000 sec
IN0         0.00002240 sec

======== CHANNEL f1 ========
SFO1        400.1322007 MHz
NUC1               1H
P1                15.00 usec
P2                30.00 usec
PLW1        12.50300026 W

======== CHANNEL f2 ========
SFO2        100.6228119 MHz
NUC2               13C
P3                 9.80 usec
PLW2        60.95399857 W

====== GRADIENT CHANNEL =====
GPNAM[1]     SMSQ10.100
GPNAM[2]     SMSQ10.100
GPNAM[3]     SMSQ10.100
GPNAM[4]     SMSQ10.100
GPNAM[5]     SMSQ10.100
GPNAM[6]     SMSQ10.100
GPZ1              50.00 %
GPZ2              30.00 %
GPZ3              40.10 %
GPZ4              15.00 %
GPZ5             -10.00 %
GPZ6              -5.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                 128
SFO1          100.6228 MHz
FIDRES       348.772308 Hz
SW              221.833 ppm
FnMODE              QF

F2 - Processing parameters
SI                2048
SF          400.1299573 MHz
WDW               SINE
SSB                  0
LB                   0 Hz
GB                   0
PC                1.40

F1 - Processing parameters
SI                1024
MC2                 QF
SF          100.6127310 MHz
WDW               SINE
SSB                  0
LB                   0 Hz
GB                   0
```

```
F2 - Acquisition Parameters
Date_           20180221
Time              16.31
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG    hmbcgpl2ndqf
TD                2048
SOLVENT            D2O
NS                   4
DS                  16
SWH           4807.692 Hz
FIDRES        2.347506 Hz
AQ           0.2129920 sec
RG              192.58
DW             104.000 usec
DE               6.50 usec
TE              298.1 K
CNST6       125.0000000
CNST7       165.0000000
CNST13        7.5000000
D0          0.00000300 sec
D1          1.50000000 sec
D6          0.06666667 sec
D16         0.00020000 sec
IN0         0.00002240 sec

======== CHANNEL f1 ========
SFO1        400.1322007 MHz
NUC1               1H
P1                15.00 usec
P2                30.00 usec
PLW1        12.50300026 W

======== CHANNEL f2 ========
SFO2        100.6228119 MHz
NUC2               13C
P3                 9.80 usec
PLW2        60.95399857 W

====== GRADIENT CHANNEL =====
GPNAM[1]     SMSQ10.100
GPNAM[2]     SMSQ10.100
GPNAM[3]     SMSQ10.100
GPNAM[4]     SMSQ10.100
GPNAM[5]     SMSQ10.100
GPNAM[6]     SMSQ10.100
GPZ1              50.00 %
GPZ2              30.00 %
GPZ3              40.10 %
GPZ4              15.00 %
GPZ5             -10.00 %
GPZ6              -5.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                 128
SFO1          100.6228 MHz
FIDRES       348.772308 Hz
SW              221.833 ppm
FnMODE              QF

F2 - Processing parameters
SI                2048
SF          400.1299618 MHz
WDW               SINE
SSB                  0
LB                   0 Hz
GB                   0
PC                1.40

F1 - Processing parameters
SI                1024
MC2                 QF
SF          100.6127690 MHz
WDW               SINE
SSB                  0
LB                   0 Hz
GB                   0
```

	meta 2	meta 3
^{1}H	F2 - Acquisition Parameters Date_ 20180405 Time 14.40 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zg30 TD 65536 SOLVENT CDCl3 NS 16 DS 2 SWH 8012.820 Hz FIDRES 0.122266 Hz AQ 4.0894465 sec RG 33.39 DW 62.400 usec DE 6.50 usec TE 298.2 K D1 1.00000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 400.1324710 MHz NUC1 1H P1 15.00 usec PLW1 12.50300026 W F2 - Processing parameters SI 65536 SF 400.1300095 MHz WDW EM SSB 0 LB 0.30 Hz GB 0 PC 1.00	F2 - Acquisition Parameters Date_ 20180410 Time 5.47 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zg TD 48076 SOLVENT D2O NS 8 DS 2 SWH 6002.401 Hz FIDRES 0.124852 Hz AQ 4.0047307 sec RG 17.75 DW 83.300 usec DE 16.70 usec TE 298.1 K D1 5.00000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 400.1320007 MHz NUC1 1H P1 15.00 usec PLW1 12.50300026 W F2 - Processing parameters SI 32768 SF 400.1299714 MHz WDW EM SSB 0 LB 0.30 Hz GB 0 PC 1.00

DEPT	F2 - Acquisition Parameters Date_ 20180405 Time 15.05 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG deptqgpsp TD 65536 SOLVENT CDCl3 NS 256 DS 4 SWH 22058.824 Hz FIDRES 0.336591 Hz AQ 1.4854827 sec RG 192.58 DW 22.667 usec DE 6.50 usec TE 298.1 K CNST2 145.0000000 CNST12 1.5000000 D1 2.00000000 sec D2 0.00344828 sec D12 0.00002000 sec D16 0.00020000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 100.6223258 MHz NUC1 13C P1 9.80 usec P13 2000.00 usec PLW0 0 W PLW1 60.95399857 W SPNAM[5] Crp60comp.4 SPOAL5 0.500 SPOFFS5 0 Hz SPW5 8.94419956 W ======== CHANNEL f2 ======== SFO2 400.1316005 MHz NUC2 1H CPDPRG[2 waltz16 P0 22.50 usec P3 15.00 usec P4 30.00 usec PCPD2 90.00 usec PLW2 12.50300026 W PLW12 0.34731001 W ====== GRADIENT CHANNEL ===== GPNAM[1] SMSQ10.32 GPNAM[2] SMSQ10.32 GPNAM[3] SMSQ10.32 GPZ1 31.00 % GPZ2 31.00 % GPZ3 31.00 % P16 1000.00 usec F2 - Processing parameters SI 65536 SF 100.6127625 MHz WDW EM SSB 0 LB 1.00 Hz GB 0 PC 1.40	F2 - Acquisition Parameters Date_ 20180410 Time 6.04 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG deptqgpsp TD 65536 SOLVENT D2O NS 256 DS 4 SWH 22058.824 Hz FIDRES 0.336591 Hz AQ 1.4854827 sec RG 192.58 DW 22.667 usec DE 6.50 usec TE 298.2 K CNST2 145.0000000 CNST12 1.5000000 D1 2.00000000 sec D2 0.00344828 sec D12 0.00002000 sec D16 0.00020000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 100.6223258 MHz NUC1 13C P1 9.80 usec P13 2000.00 usec PLW0 0 W PLW1 60.95399857 W SPNAM[5] Crp60comp.4 SPOAL5 0.500 SPOFFS5 0 Hz SPW5 8.94419956 W ======== CHANNEL f2 ======== SFO2 400.1316005 MHz NUC2 1H CPDPRG[2 waltz16 P0 22.50 usec P3 15.00 usec P4 30.00 usec PCPD2 90.00 usec PLW2 12.50300026 W PLW12 0.34731001 W ====== GRADIENT CHANNEL ===== GPNAM[1] SMSQ10.32 GPNAM[2] SMSQ10.32 GPNAM[3] SMSQ10.32 GPZ1 31.00 % GPZ2 31.00 % GPZ3 31.00 % P16 1000.00 usec F2 - Processing parameters SI 65536 SF 100.6127690 MHz WDW EM SSB 0 LB 1.00 Hz GB 0 PC 1.40

^{13}C	F2 - Acquisition Parameters Date_ 20180408 Time 7.42 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zgpg30 TD 65536 SOLVENT CDCl3 NS 3000 DS 4 SWH 22058.824 Hz FIDRES 0.336591 Hz AQ 1.4854827 sec RG 192.58 DW 22.667 usec DE 6.50 usec TE 298.1 K D1 2.00000000 sec D11 0.03000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 100.6223253 MHz NUC1 13C P1 9.80 usec PLW1 60.95399857 W ======== CHANNEL f2 ======== SFO2 400.1316005 MHz NUC2 1H CPDPRG[2 waltz16 PCPD2 90.00 usec PLW2 12.50300026 W PLW12 0.34731001 W PLW13 0.28132001 W F2 - Processing parameters SI 65536 SF 100.6127636 MHz WDW EM SSB 0 LB 1.00 Hz GB 0 PC 1.40	F2 - Acquisition Parameters Date_ 20180411 Time 0.04 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zgpg30 TD 65536 SOLVENT D2O NS 3000 DS 4 SWH 22058.824 Hz FIDRES 0.336591 Hz AQ 1.4854827 sec RG 192.58 DW 22.667 usec DE 6.50 usec TE 298.2 K D1 2.00000000 sec D11 0.03000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 100.6223253 MHz NUC1 13C P1 9.80 usec PLW1 60.95399857 W ======== CHANNEL f2 ======== SFO2 400.1316005 MHz NUC2 1H CPDPRG[2 waltz16 PCPD2 90.00 usec PLW2 12.50300026 W PLW12 0.34731001 W PLW13 0.28132001 W F2 - Processing parameters SI 65536 SF 100.6127690 MHz WDW EM SSB 0 LB 1.00 Hz GB 0 PC 1.40
^{11}B	F2 - Acquisition Parameters Date_ 20180406 Time 7.57 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zg TD 65536 SOLVENT CDCl3 NS 128 DS 4 SWH 25510.203 Hz FIDRES 0.389255 Hz AQ 1.2845056 sec RG 192.58 DW 19.600 usec DE 6.50 usec TE 298.2 K D1 1.00000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 128.3776052 MHz NUC1 11B P1 17.05 usec PLW1 11.69499969 W F2 - Processing parameters SI 32768 SF 128.3776052 MHz WDW no SSB 0 LB 0 Hz GB 0 PC 1.40	F2 - Acquisition Parameters Date_ 20180409 Time 14.58 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zg TD 65536 SOLVENT D2O NS 128 DS 4 SWH 25510.203 Hz FIDRES 0.389255 Hz AQ 1.2845056 sec RG 192.58 DW 19.600 usec DE 6.50 usec TE 298.2 K D1 1.00000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 128.3776052 MHz NUC1 11B P1 17.05 usec PLW1 11.69499969 W F2 - Processing parameters SI 32768 SF 128.3776052 MHz WDW no SSB 0 LB 0 Hz GB 0 PC 1.40

COSY

```
F2 - Acquisition Parameters
Date_           20180405
Time               15.07
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG         cosygpqf
TD                 2048
SOLVENT            CDCl3
NS                    1
DS                    8
SWH             4807.692 Hz
FIDRES          2.347506 Hz
AQ             0.2129920 sec
RG                56.54
DW               104.000 usec
DE                 6.50 usec
TE                298.0 K
D0            0.00000300 sec
D1            1.48689198 sec
D13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00020800 sec

======== CHANNEL f1 ========
SFO1         400.1322007 MHz
NUC1                1H
P0                15.00 usec
P1                15.00 usec
PLW1         12.50300026 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPZ1              10.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  128
SFO1            400.1322 MHz
FIDRES         75.120193 Hz
SW                12.015 ppm
FnMODE              QF

F2 - Processing parameters
SI                 1024
SF           400.1300078 MHz
WDW                SINE
SSB                   0
LB                    0 Hz
GB                    0
PC                 1.40

F1 - Processing parameters
SI                 1024
MC2                  QF
SF           400.1300092 MHz
WDW                SINE
SSB                   0
LB                    0 Hz
GB                    0
```

```
F2 - Acquisition Parameters
Date_           20180410
Time               6.07
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG         cosygpqf
TD                 2048
SOLVENT             D2O
NS                    1
DS                    8
SWH             4807.692 Hz
FIDRES          2.347506 Hz
AQ             0.2129920 sec
RG                56.54
DW               104.000 usec
DE                 6.50 usec
TE                298.0 K
D0            0.00000300 sec
D1            1.48689198 sec
D13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00020800 sec

======== CHANNEL f1 ========
SFO1         400.1322007 MHz
NUC1                1H
P0                15.00 usec
P1                15.00 usec
PLW1         12.50300026 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPZ1              10.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  128
SFO1            400.1322 MHz
FIDRES         75.120193 Hz
SW                12.015 ppm
FnMODE              QF

F2 - Processing parameters
SI                 1024
SF           400.1299700 MHz
WDW                SINE
SSB                   0
LB                    0 Hz
GB                    0
PC                 1.40

F1 - Processing parameters
SI                 1024
MC2                  QF
SF           400.1299710 MHz
WDW                SINE
SSB                   0
LB                    0 Hz
GB                    0
```

HSQC

```
F2 - Acquisition Parameters
Date_           20180408
Time                7.45
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG  hsqcetgpsisp2.2
TD                  2048
SOLVENT            CDC13
NS                     2
DS                    16
SWH             5341.880 Hz
FIDRES          2.608340 Hz
AQ             0.1916928 sec
RG                192.58
DW                93.600 usec
DE                 6.50 usec
TE                298.0 K
CNST2        145.0000000
CNST17        -0.5000000
D0            0.00000300 sec
D1            1.50000000 sec
D4            0.00172414 sec
D11           0.03000000 sec
D16           0.00020000 sec
D24           0.00086207 sec
IN0           0.00003000 sec

======== CHANNEL f1 ========
SFO1         400.1324057 MHz
NUC1                  1H
P1                15.00 usec
P2                30.00 usec
P28             1000.00 usec
PLW1         12.50300026 W

======== CHANNEL f2 ========
SFO2         100.6202713 MHz
NUC2                 13C
CPDPRG[2 bi_p5m4sp_4sp.2
P3                 9.80 usec
P14              500.00 usec
P24             2000.00 usec
P63             1500.00 usec
PLW0                  0 W
PLW2         60.95399857 W
PLW12         0.91469002 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3              0.500
SPOFFS3               0 Hz
SPW3          8.94419956 W
SPNAM[7]     Crp60comp.4
SPOAL7              0.500
SPOFFS7               0 Hz
SPW7          8.94419956 W
SPNAM[14 Crp32,1.5,20.2
SPOAL14             0.500
SPOFFS14              0 Hz
SPW14         3.81620002 W
SPNAM[31 Crp32,1.5,20.2
SPOAL31             0.500
SPOFFS31              0 Hz
SPW31         0.95405000 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPZ1                80.00 %
GPZ2                20.10 %
GPZ3                11.00 %
GPZ4                -5.00 %
P16               1000.00 usec
P19                600.00 usec

F1 - Acquisition parameters
TD                   256
SFO1            100.6203 MHz
FIDRES        130.208328 Hz
SW               165.639 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                  1024
SF           400.1300069 MHz
WDW                 QSINE
SSB                     2
LB                      0 Hz
GB                      0
PC                   1.40

F1 - Processing parameters
SI                  1024
MC2        echo-antiecho
SF            100.6127690 MHz
WDW                 QSINE
SSB                     2
LB                      0 Hz
GB                      0
```

```
F2 - Acquisition Parameters
Date_           20180411
Time                0.06
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG  hsqcetgpsisp2.2
TD                  2048
SOLVENT              D2O
NS                     2
DS                    16
SWH             5341.880 Hz
FIDRES          2.608340 Hz
AQ             0.1916928 sec
RG                192.58
DW                93.600 usec
DE                 6.50 usec
TE                298.0 K
CNST2        145.0000000
CNST17        -0.5000000
D0            0.00000300 sec
D1            1.50000000 sec
D4            0.00172414 sec
D11           0.03000000 sec
D16           0.00020000 sec
D24           0.00086207 sec
IN0           0.00003000 sec

======== CHANNEL f1 ========
SFO1         400.1324057 MHz
NUC1                  1H
P1                15.00 usec
P2                30.00 usec
P28             1000.00 usec
PLW1         12.50300026 W

======== CHANNEL f2 ========
SFO2         100.6202713 MHz
NUC2                 13C
CPDPRG[2 bi_p5m4sp_4sp.2
P3                 9.80 usec
P14              500.00 usec
P24             2000.00 usec
P63             1500.00 usec
PLW0                  0 W
PLW2         60.95399857 W
PLW12         0.91469002 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3              0.500
SPOFFS3               0 Hz
SPW3          8.94419956 W
SPNAM[7]     Crp60comp.4
SPOAL7              0.500
SPOFFS7               0 Hz
SPW7          8.94419956 W
SPNAM[14 Crp32,1.5,20.2
SPOAL14             0.500
SPOFFS14              0 Hz
SPW14         3.81620002 W
SPNAM[31 Crp32,1.5,20.2
SPOAL31             0.500
SPOFFS31              0 Hz
SPW31         0.95405000 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPZ1                80.00 %
GPZ2                20.10 %
GPZ3                11.00 %
GPZ4                -5.00 %
P16               1000.00 usec
P19                600.00 usec

F1 - Acquisition parameters
TD                   256
SFO1            100.6203 MHz
FIDRES        130.208328 Hz
SW               165.639 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                  1024
SF           400.1299725 MHz
WDW                 QSINE
SSB                     2
LB                      0 Hz
GB                      0
PC                   1.40

F1 - Processing parameters
SI                  1024
MC2        echo-antiecho
SF            100.6127690 MHz
WDW                 QSINE
SSB                     2
LB                      0 Hz
GB                      0
```

HMBC

```
F2 - Acquisition Parameters
Date_          20180405
Time              15.20
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG    hmbcgpl2ndqf
TD                2048
SOLVENT           CDC13
NS                   4
DS                  16
SWH            4807.692 Hz
FIDRES         2.347506 Hz
AQ            0.2129920 sec
RG               192.58
DW              104.000 usec
DE                6.50 usec
TE                298.2 K
CNST6       125.0000000
CNST7       165.0000000
CNST13        7.5000000
D0          0.00000300 sec
D1          1.50000000 sec
D6          0.06666667 sec
D16         0.00020000 sec
IN0         0.00002240 sec

======== CHANNEL f1 ========
SFO1        400.1322007 MHz
NUC1                1H
P1                15.00 usec
P2                30.00 usec
PLW1        12.50300026 W

======== CHANNEL f2 ========
SFO2        100.6228119 MHz
NUC2               13C
P3                 9.80 usec
PLW2        60.95399857 W

====== GRADIENT CHANNEL =====
GPNAM[1]     SMSQ10.100
GPNAM[2]     SMSQ10.100
GPNAM[3]     SMSQ10.100
GPNAM[4]     SMSQ10.100
GPNAM[5]     SMSQ10.100
GPNAM[6]     SMSQ10.100
GPZ1              50.00 %
GPZ2              30.00 %
GPZ3              40.10 %
GPZ4              15.00 %
GPZ5             -10.00 %
GPZ6              -5.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                 128
SFO1          100.6228 MHz
FIDRES       348.772308 Hz
SW              221.833 ppm
FnMODE               QF

F2 - Processing parameters
SI                2048
SF          400.1300104 MHz
WDW               SINE
SSB                  0
LB                   0 Hz
GB                   0
PC                1.40

F1 - Processing parameters
SI                1024
MC2                 QF
SF          100.6127690 MHz
WDW               SINE
SSB                  0
LB                   0 Hz
GB                   0
```

```
F2 - Acquisition Parameters
Date_          20180410
Time              6.12
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG    hmbcgpl2ndqf
TD                2048
SOLVENT            D2O
NS                   4
DS                  16
SWH            4807.692 Hz
FIDRES         2.347506 Hz
AQ            0.2129920 sec
RG               192.58
DW              104.000 usec
DE                6.50 usec
TE                298.1 K
CNST6       125.0000000
CNST7       165.0000000
CNST13        7.5000000
D0          0.00000300 sec
D1          1.50000000 sec
D6          0.06666667 sec
D16         0.00020000 sec
IN0         0.00002240 sec

======== CHANNEL f1 ========
SFO1        400.1322007 MHz
NUC1                1H
P1                15.00 usec
P2                30.00 usec
PLW1        12.50300026 W

======== CHANNEL f2 ========
SFO2        100.6228119 MHz
NUC2               13C
P3                 9.80 usec
PLW2        60.95399857 W

====== GRADIENT CHANNEL =====
GPNAM[1]     SMSQ10.100
GPNAM[2]     SMSQ10.100
GPNAM[3]     SMSQ10.100
GPNAM[4]     SMSQ10.100
GPNAM[5]     SMSQ10.100
GPNAM[6]     SMSQ10.100
GPZ1              50.00 %
GPZ2              30.00 %
GPZ3              40.10 %
GPZ4              15.00 %
GPZ5             -10.00 %
GPZ6              -5.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                 128
SFO1          100.6228 MHz
FIDRES       348.772308 Hz
SW              221.833 ppm
FnMODE               QF

F2 - Processing parameters
SI                2048
SF          400.1299701 MHz
WDW               SINE
SSB                  0
LB                   0 Hz
GB                   0
PC                1.40

F1 - Processing parameters
SI                1024
MC2                 QF
SF          100.6127690 MHz
WDW               SINE
SSB                  0
LB                   0 Hz
GB                   0
```

	meta 4	meta 5
^{1}H	F2 - Acquisition Parameters Date_ 20180507 Time 16.31 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zg30 TD 65536 SOLVENT MeOD NS 16 DS 2 SWH 8012.820 Hz FIDRES 0.122266 Hz AQ 4.0894465 sec RG 20.83 DW 62.400 usec DE 6.50 usec TE 298.1 K D1 1.00000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 400.1324710 MHz NUC1 1H P1 15.00 usec PLW1 12.50300026 W F2 - Processing parameters SI 65536 SF 400.1300859 MHz WDW EM SSB 0 LB 0.30 Hz GB 0 PC 1.00	F2 - Acquisition Parameters Date_ 20171006 Time 11.40 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zg TD 48076 SOLVENT MeOD NS 8 DS 2 SWH 6002.401 Hz FIDRES 0.124852 Hz AQ 4.0047307 sec RG 107 DW 83.300 usec DE 16.70 usec TE 298.2 K D1 5.00000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 400.1320007 MHz NUC1 1H P1 15.00 usec PLW1 12.50300026 W F2 - Processing parameters SI 32768 SF 400.1300076 MHz WDW EM SSB 0 LB 0.30 Hz GB 0 PC 1.00

^{13}C	F2 - Acquisition Parameters Date_ 20180510 Time 7.11 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zgpg30 TD 65536 SOLVENT MeOD NS 3000 DS 4 SWH 22058.824 Hz FIDRES 0.336591 Hz AQ 1.4854827 sec RG 42.64 DW 22.667 usec DE 6.50 usec TE 298.2 K D1 2.00000000 sec D11 0.03000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 100.6223253 MHz NUC1 13C P1 9.80 usec PLW1 60.95399857 W ======== CHANNEL f2 ======== SFO2 400.1316005 MHz NUC2 1H CPDPRG[2 waltz16 PCPD2 90.00 usec PLW2 12.50300026 W PLW12 0.34731001 W PLW13 0.28132001 W F2 - Processing parameters SI 65536 SF 100.6126421 MHz WDW EM SSB 0 LB 1.00 Hz GB 0 PC 1.40	F2 - Acquisition Parameters Date_ 20171007 Time 5.10 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zgpg30 TD 65536 SOLVENT MeOD NS 4000 DS 4 SWH 22058.824 Hz FIDRES 0.336591 Hz AQ 1.4854827 sec RG 192.58 DW 22.667 usec DE 6.50 usec TE 298.2 K D1 2.00000000 sec D11 0.03000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 100.6223253 MHz NUC1 13C P1 9.80 usec PLW1 60.95399857 W ======== CHANNEL f2 ======== SFO2 400.1316005 MHz NUC2 1H CPDPRG[2 waltz16 PCPD2 90.00 usec PLW2 12.50300026 W PLW12 0.34731001 W PLW13 0.28132001 W F2 - Processing parameters SI 65536 SF 100.6126280 MHz WDW EM SSB 0 LB 1.00 Hz GB 0 PC 1.40

DEPT	F2 - Acquisition Parameters Date_ 20180509 Time 15.44 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG deptqgpsp TD 65536 SOLVENT MeOD NS 256 DS 4 SWH 22058.824 Hz FIDRES 0.336591 Hz AQ 1.4854827 sec RG 192.58 DW 22.667 usec DE 6.50 usec TE 298.2 K CNST2 145.0000000 CNST12 1.5000000 D1 2.00000000 sec D2 0.00344828 sec D12 0.00002000 sec D16 0.00020000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 100.6223258 MHz NUC1 13C P1 9.80 usec P13 2000.00 usec PLW0 0 W PLW1 60.95399857 W SPNAM[5] Crp60comp.4 SPOAL5 0.500 SPOFFS5 0 Hz SPW5 8.94419956 W ======== CHANNEL f2 ======== SFO2 400.1316005 MHz NUC2 1H CPDPRG[2 waltz16 P0 22.50 usec P3 15.00 usec P4 30.00 usec PCPD2 90.00 usec PLW2 12.50300026 W PLW12 0.34731001 W ====== GRADIENT CHANNEL ===== GPNAM[1] SMSQ10.32 GPNAM[2] SMSQ10.32 GPNAM[3] SMSQ10.32 GPZ1 31.00 % GPZ2 31.00 % GPZ3 31.00 % P16 1000.00 usec F2 - Processing parameters SI 65536 SF 100.6126418 MHz WDW EM SSB 0 LB 1.00 Hz GB 0 PC 1.40	F2 - Acquisition Parameters Date_ 20171006 Time 12.19 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG deptqgpsp TD 65536 SOLVENT MeOD NS 256 DS 4 SWH 22058.824 Hz FIDRES 0.336591 Hz AQ 1.4854827 sec RG 192.58 DW 22.667 usec DE 6.50 usec TE 298.2 K CNST2 145.0000000 CNST12 1.5000000 D1 2.00000000 sec D2 0.00344828 sec D12 0.00002000 sec D16 0.00020000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 100.6223258 MHz NUC1 13C P1 9.80 usec P13 2000.00 usec PLW0 0 W PLW1 60.95399857 W SPNAM[5] Crp60comp.4 SPOAL5 0.500 SPOFFS5 0 Hz SPW5 8.94419956 W ======== CHANNEL f2 ======== SFO2 400.1316005 MHz NUC2 1H CPDPRG[2 waltz16 P0 22.50 usec P3 15.00 usec P4 30.00 usec PCPD2 90.00 usec PLW2 12.50300026 W PLW12 0.34731001 W ====== GRADIENT CHANNEL ===== GPNAM[1] SMSQ10.32 GPNAM[2] SMSQ10.32 GPNAM[3] SMSQ10.32 GPZ1 31.00 % GPZ2 31.00 % GPZ3 31.00 % P16 1000.00 usec F2 - Processing parameters SI 65536 SF 100.6126275 MHz WDW EM SSB 0 LB 1.00 Hz GB 0 PC 1.40

^{11}B	F2 - Acquisition Parameters Date_ 20180507 Time 16.37 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zg TD 65536 SOLVENT MeOD NS 128 DS 4 SWH 25510.203 Hz FIDRES 0.389255 Hz AQ 1.2845056 sec RG 192.58 DW 19.600 usec DE 6.50 usec TE 298.2 K D1 1.00000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 128.3776052 MHz NUC1 11B P1 17.05 usec PLW1 11.69499969 W F2 - Processing parameters SI 32768 SF 128.3776052 MHz WDW no SSB 0 LB 0 Hz GB 0 PC 1.40	F2 - Acquisition Parameters Date_ 20171007 Time 5.17 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zg TD 65536 SOLVENT MeOD NS 128 DS 4 SWH 25510.203 Hz FIDRES 0.389255 Hz AQ 1.2845056 sec RG 192.58 DW 19.600 usec DE 6.50 usec TE 298.1 K D1 1.00000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 128.3776052 MHz NUC1 11B P1 17.05 usec PLW1 11.69499969 W F2 - Processing parameters SI 32768 SF 128.3776052 MHz WDW no SSB 0 LB 0 Hz GB 0 PC 1.40

COSY	F2 - Acquisition Parameters Date_ 20180509 Time 15.46 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG cosygpqf TD 2048 SOLVENT MeOD NS 1 DS 8 SWH 4807.692 Hz FIDRES 2.347506 Hz AQ 0.2129920 sec RG 58.62 DW 104.000 usec DE 6.50 usec TE 298.0 K D0 0.00000300 sec D1 1.48689198 sec D13 0.00000400 sec D16 0.00020000 sec IN0 0.00020800 sec ======== CHANNEL f1 ======== SFO1 400.1322007 MHz NUC1 1H P0 15.00 usec P1 15.00 usec PLW1 12.50300026 W ====== GRADIENT CHANNEL ===== GPNAM[1] SMSQ10.100 GPZ1 10.00 % P16 1000.00 usec F1 - Acquisition parameters TD 128 SFO1 400.1322 MHz FIDRES 75.120193 Hz SW 12.015 ppm FnMODE QF F2 - Processing parameters SI 1024 SF 400.1300901 MHz WDW SINE SSB 0 LB 0 Hz GB 0 PC 1.40 F1 - Processing parameters SI 1024 MC2 QF SF 400.1300897 MHz WDW SINE SSB 0 LB 0 Hz GB 0	F2 - Acquisition Parameters Date_ 20171006 Time 12.21 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG cosygpqf TD 2048 SOLVENT MeOD NS 1 DS 8 SWH 4807.692 Hz FIDRES 2.347506 Hz AQ 0.2129920 sec RG 192.58 DW 104.000 usec DE 6.50 usec TE 298.0 K D0 0.00000300 sec D1 1.48689198 sec D13 0.00000400 sec D16 0.00020000 sec IN0 0.00020800 sec ======== CHANNEL f1 ======== SFO1 400.1322007 MHz NUC1 1H P0 15.00 usec P1 15.00 usec PLW1 12.50300026 W ====== GRADIENT CHANNEL ===== GPNAM[1] SMSQ10.100 GPZ1 10.00 % P16 1000.00 usec F1 - Acquisition parameters TD 128 SFO1 400.1322 MHz FIDRES 75.120193 Hz SW 12.015 ppm FnMODE QF F2 - Processing parameters SI 1024 SF 400.1300073 MHz WDW SINE SSB 0 LB 0 Hz GB 0 PC 1.40 F1 - Processing parameters SI 1024 MC2 QF SF 400.1300067 MHz WDW SINE SSB 0 LB 0 Hz GB 0

HSQC

```
F2 - Acquisition Parameters
Date_            20180510
Time                7.14
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG  hsqcetgpsisp2.2
TD                  2048
SOLVENT             MeOD
NS                     2
DS                    16
SWH             5341.880 Hz
FIDRES          2.608340 Hz
AQ             0.1916928 sec
RG                192.58
DW                93.600 usec
DE                 6.50 usec
TE                298.0 K
CNST2        145.0000000
CNST17        -0.5000000
D0             0.00000300 sec
D1             1.50000000 sec
D4             0.00172414 sec
D11            0.03000000 sec
D16            0.00020000 sec
D24            0.00086207 sec
IN0            0.00003000 sec

======== CHANNEL f1 ========
SFO1         400.1324057 MHz
NUC1                   1H
P1                  15.00 usec
P2                  30.00 usec
P28               1000.00 usec
PLW1         12.50300026 W

======== CHANNEL f2 ========
SFO2         100.6202713 MHz
NUC2                  13C
CPDPRG[2 bi_p5m4sp_4sp.2
P3                   9.80 usec
P14                500.00 usec
P24               2000.00 usec
P63               1500.00 usec
PLW0                    0 W
PLW2         60.95399857 W
PLW12         0.91469002 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3                0.500
SPOFFS3                 0 Hz
SPW3          8.94419956 W
SPNAM[7]     Crp60comp.4
SPOAL7                0.500
SPOFFS7                 0 Hz
SPW7          8.94419956 W
SPNAM[14 Crp32,1.5,20.2
SPOAL14               0.500
SPOFFS14                0 Hz
SPW14         3.81620002 W
SPNAM[31 Crp32,1.5,20.2
SPOAL31               0.500
SPOFFS31                0 Hz
SPW31         0.95405000 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPZ1                80.00 %
GPZ2                20.10 %
GPZ3                11.00 %
GPZ4                -5.00 %
P16               1000.00 usec
P19                600.00 usec

F1 - Acquisition parameters
TD                    256
SFO1            100.6203 MHz
FIDRES        130.208328 Hz
SW              165.639 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                   1024
SF           400.1300871 MHz
WDW                 QSINE
SSB                     2
LB                      0 Hz
GB                      0
PC                   1.40

F1 - Processing parameters
SI                   1024
MC2        echo-antiecho
SF           100.6126499 MHz
WDW                 QSINE
SSB                     2
LB                      0 Hz
GB                      0
```

```
F2 - Acquisition Parameters
Date_            20171006
Time                12.43
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG  hsqcetgpsisp2.2
TD                  2048
SOLVENT             MeOD
NS                     2
DS                    16
SWH             5341.880 Hz
FIDRES          2.608340 Hz
AQ             0.1916928 sec
RG                192.58
DW                93.600 usec
DE                 6.50 usec
TE                298.3 K
CNST2        145.0000000
CNST17        -0.5000000
D0             0.00000300 sec
D1             1.50000000 sec
D4             0.00172414 sec
D11            0.03000000 sec
D16            0.00020000 sec
D24            0.00086207 sec
IN0            0.00003000 sec

======== CHANNEL f1 ========
SFO1         400.1324057 MHz
NUC1                   1H
P1                  15.00 usec
P2                  30.00 usec
P28               1000.00 usec
PLW1         12.50300026 W

======== CHANNEL f2 ========
SFO2         100.6202713 MHz
NUC2                  13C
CPDPRG[2 bi_p5m4sp_4sp.2
P3                   9.80 usec
P14                500.00 usec
P24               2000.00 usec
P63               1500.00 usec
PLW0                    0 W
PLW2         60.95399857 W
PLW12         0.91469002 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3                0.500
SPOFFS3                 0 Hz
SPW3          8.94419956 W
SPNAM[7]     Crp60comp.4
SPOAL7                0.500
SPOFFS7                 0 Hz
SPW7          8.94419956 W
SPNAM[14 Crp32,1.5,20.2
SPOAL14               0.500
SPOFFS14                0 Hz
SPW14         3.81620002 W
SPNAM[31 Crp32,1.5,20.2
SPOAL31               0.500
SPOFFS31                0 Hz
SPW31         0.95405000 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPZ1                80.00 %
GPZ2                20.10 %
GPZ3                11.00 %
GPZ4                -5.00 %
P16               1000.00 usec
P19                600.00 usec

F1 - Acquisition parameters
TD                    256
SFO1            100.6203 MHz
FIDRES        130.208328 Hz
SW              165.639 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                   1024
SF           400.1300052 MHz
WDW                 QSINE
SSB                     2
LB                      0 Hz
GB                      0
PC                   1.40

F1 - Processing parameters
SI                   1024
MC2        echo-antiecho
SF           100.6126258 MHz
WDW                 QSINE
SSB                     2
LB                      0 Hz
GB                      0
```

HMBC	F2 - Acquisition Parameters Date_ 20180509 Time 15.52 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG hmbcgpl2ndqf TD 2048 SOLVENT MeOD NS 4 DS 16 SWH 4807.692 Hz FIDRES 2.347506 Hz AQ 0.2129920 sec RG 192.58 DW 104.000 usec DE 6.50 usec TE 298.1 K CNST6 125.0000000 CNST7 165.0000000 CNST13 7.5000000 D0 0.00000300 sec D1 1.50000000 sec D6 0.06666667 sec D16 0.00020000 sec IN0 0.00002240 sec ======== CHANNEL f1 ======== SFO1 400.1322007 MHz NUC1 1H P1 15.00 usec P2 30.00 usec PLW1 12.50300026 W ======== CHANNEL f2 ======== SFO2 100.6228119 MHz NUC2 13C P3 9.80 usec PLW2 60.95399857 W ====== GRADIENT CHANNEL ===== GPNAM[1] SMSQ10.100 GPNAM[2] SMSQ10.100 GPNAM[3] SMSQ10.100 GPNAM[4] SMSQ10.100 GPNAM[5] SMSQ10.100 GPNAM[6] SMSQ10.100 GPZ1 50.00 % GPZ2 30.00 % GPZ3 40.10 % GPZ4 15.00 % GPZ5 -10.00 % GPZ6 -5.00 % P16 1000.00 usec F1 - Acquisition parameters TD 128 SFO1 100.6228 MHz FIDRES 348.772308 Hz SW 221.833 ppm FnMODE QF F2 - Processing parameters SI 2048 SF 400.1300877 MHz WDW SINE SSB 0 LB 0 Hz GB 0 PC 1.40 F1 - Processing parameters SI 1024 MC2 QF SF 100.6141285 MHz WDW SINE SSB 0 LB 0 Hz GB 0	F2 - Acquisition Parameters Date_ 20171006 Time 12.26 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG hmbcgpl2ndqf TD 2048 SOLVENT MeOD NS 4 DS 16 SWH 4807.692 Hz FIDRES 2.347506 Hz AQ 0.2129920 sec RG 192.58 DW 104.000 usec DE 6.50 usec TE 298.1 K CNST6 125.0000000 CNST7 165.0000000 CNST13 7.5000000 D0 0.00000300 sec D1 1.50000000 sec D6 0.06666667 sec D16 0.00020000 sec IN0 0.00002240 sec ======== CHANNEL f1 ======== SFO1 400.1322007 MHz NUC1 1H P1 15.00 usec P2 30.00 usec PLW1 12.50300026 W ======== CHANNEL f2 ======== SFO2 100.6228119 MHz NUC2 13C P3 9.80 usec PLW2 60.95399857 W ====== GRADIENT CHANNEL ===== GPNAM[1] SMSQ10.100 GPNAM[2] SMSQ10.100 GPNAM[3] SMSQ10.100 GPNAM[4] SMSQ10.100 GPNAM[5] SMSQ10.100 GPNAM[6] SMSQ10.100 GPZ1 50.00 % GPZ2 30.00 % GPZ3 40.10 % GPZ4 15.00 % GPZ5 -10.00 % GPZ6 -5.00 % P16 1000.00 usec F1 - Acquisition parameters TD 128 SFO1 100.6228 MHz FIDRES 348.772308 Hz SW 221.833 ppm FnMODE QF F2 - Processing parameters SI 2048 SF 400.1300000 MHz WDW SINE SSB 0 LB 0 Hz GB 0 PC 1.40 F1 - Processing parameters SI 1024 MC2 QF SF 100.6127690 MHz WDW SINE SSB 0 LB 0 Hz GB 0

	ortho 2	ortho 3
^{1}H	F2 - Acquisition Parameters Date_ 20171004 Time 15.36 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zg TD 48076 SOLVENT CDCl3 NS 8 DS 2 SWH 6002.401 Hz FIDRES 0.124852 Hz AQ 4.0047307 sec RG 46.86 DW 83.300 usec DE 16.70 usec TE 298.1 K D1 5.00000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 400.1320007 MHz NUC1 1H P1 15.00 usec PLW1 12.50300026 W F2 - Processing parameters SI 32768 SF 400.1300100 MHz WDW EM SSB 0 LB 0.30 Hz GB 0 PC 1.00	F2 - Acquisition Parameters Date_ 20171009 Time 12.05 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zg30 TD 65536 SOLVENT D2O NS 16 DS 2 SWH 8012.820 Hz FIDRES 0.122266 Hz AQ 4.0894465 sec RG 119.51 DW 62.400 usec DE 6.50 usec TE 298.2 K D1 1.00000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 400.1324710 MHz NUC1 1H P1 15.00 usec PLW1 12.50300026 W F2 - Processing parameters SI 65536 SF 400.1299661 MHz WDW EM SSB 0 LB 0.30 Hz GB 0 PC 1.00

^{13}C	F2 - Acquisition Parameters Date_ 20171004 Time 21.00 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zgpg30 TD 65536 SOLVENT CDCl3 NS 4000 DS 4 SWH 22058.824 Hz FIDRES 0.336591 Hz AQ 1.4854827 sec RG 192.58 DW 22.667 usec DE 6.50 usec TE 298.2 K D1 2.00000000 sec D11 0.03000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 100.6223253 MHz NUC1 13C P1 9.80 usec PLW1 60.95399857 W ======== CHANNEL f2 ======== SFO2 400.1316005 MHz NUC2 1H CPDPRG[2 waltz16 PCPD2 90.00 usec PLW2 12.50300026 W PLW12 0.34731001 W PLW13 0.28132001 W F2 - Processing parameters SI 65536 SF 100.6127573 MHz WDW EM SSB 0 LB 1.00 Hz GB 0 PC 1.40	F2 - Acquisition Parameters Date_ 20171012 Time 7.14 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zgpg30 TD 65536 SOLVENT D2O NS 4000 DS 4 SWH 22058.824 Hz FIDRES 0.336591 Hz AQ 1.4854827 sec RG 192.58 DW 22.667 usec DE 6.50 usec TE 298.2 K D1 2.00000000 sec D11 0.03000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 100.6223253 MHz NUC1 13C P1 9.80 usec PLW1 60.95399857 W ======== CHANNEL f2 ======== SFO2 400.1316005 MHz NUC2 1H CPDPRG[2 waltz16 PCPD2 90.00 usec PLW2 12.50300026 W PLW12 0.34731001 W PLW13 0.28132001 W F2 - Processing parameters SI 65536 SF 100.6127690 MHz WDW EM SSB 0 LB 1.00 Hz GB 0 PC 1.40

DEPT

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F2 - Acquisition Parameters
Date_           20171004
Time               15.53
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG        deptqgpsp
TD                 65536
SOLVENT            CDCl3
NS                   256
DS                     4
SWH            22058.824 Hz
FIDRES          0.336591 Hz
AQ             1.4854827 sec
RG                192.58
DW                22.667 usec
DE                 6.50 usec
TE                298.2 K
CNST2        145.0000000
CNST12         1.5000000
D1            2.00000000 sec
D2            0.00344828 sec
D12           0.00002000 sec
D16           0.00020000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6223258 MHz
NUC1                13C
P1                 9.80 usec
P13             2000.00 usec
PLW0                  0 W
PLW1         60.95399857 W
SPNAM[5]     Crp60comp.4
SPOAL5            0.500
SPOFFS5               0 Hz
SPW5          8.94419956 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
P0                22.50 usec
P3                15.00 usec
P4                30.00 usec
PCPD2             90.00 usec
PLW2         12.50300026 W
PLW12         0.34731001 W

====== GRADIENT CHANNEL =====
GPNAM[1]       SMSQ10.32
GPNAM[2]       SMSQ10.32
GPNAM[3]       SMSQ10.32
GPZ1              31.00 %
GPZ2              31.00 %
GPZ3              31.00 %
P16             1000.00 usec

F2 - Processing parameters
SI                 65536
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

```
F2 - Acquisition Parameters
Date_           20171010
Time               14.59
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG        deptqgpsp
TD                 65536
SOLVENT              D2O
NS                   256
DS                     4
SWH            22058.824 Hz
FIDRES          0.336591 Hz
AQ             1.4854827 sec
RG                192.58
DW                22.667 usec
DE                 6.50 usec
TE                298.2 K
CNST2        145.0000000
CNST12         1.5000000
D1            2.00000000 sec
D2            0.00344828 sec
D12           0.00002000 sec
D16           0.00020000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6223258 MHz
NUC1                13C
P1                 9.80 usec
P13             2000.00 usec
PLW0                  0 W
PLW1         60.95399857 W
SPNAM[5]     Crp60comp.4
SPOAL5            0.500
SPOFFS5               0 Hz
SPW5          8.94419956 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
P0                22.50 usec
P3                15.00 usec
P4                30.00 usec
PCPD2             90.00 usec
PLW2         12.50300026 W
PLW12         0.34731001 W

====== GRADIENT CHANNEL =====
GPNAM[1]       SMSQ10.32
GPNAM[2]       SMSQ10.32
GPNAM[3]       SMSQ10.32
GPZ1              31.00 %
GPZ2              31.00 %
GPZ3              31.00 %
P16             1000.00 usec

F2 - Processing parameters
SI                 65536
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

^{11}B	F2 - Acquisition Parameters Date_ 20171026 Time 12.25 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zg TD 65536 SOLVENT CDCl3 NS 128 DS 4 SWH 25510.203 Hz FIDRES 0.389255 Hz AQ 1.2845056 sec RG 192.58 DW 19.600 usec DE 6.50 usec TE 298.2 K D1 1.00000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 128.3776052 MHz NUC1 11B P1 17.05 usec PLW1 11.69499969 W F2 - Processing parameters SI 32768 SF 128.3776052 MHz WDW no SSB 0 LB 0 Hz GB 0 PC 1.40	F2 - Acquisition Parameters Date_ 20171011 Time 6.57 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zg TD 65536 SOLVENT D2O NS 128 DS 4 SWH 25510.203 Hz FIDRES 0.389255 Hz AQ 1.2845056 sec RG 192.58 DW 19.600 usec DE 6.50 usec TE 298.1 K D1 1.00000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 128.3776052 MHz NUC1 11B P1 17.05 usec PLW1 11.69499969 W F2 - Processing parameters SI 32768 SF 128.3776052 MHz WDW no SSB 0 LB 0 Hz GB 0 PC 1.40

COSY	F2 - Acquisition Parameters Date_ 20171004 Time 15.55 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG cosygpqf TD 2048 SOLVENT CDCl3 NS 1 DS 8 SWH 4807.692 Hz FIDRES 2.347506 Hz AQ 0.2129920 sec RG 107 DW 104.000 usec DE 6.50 usec TE 298.0 K D0 0.00000300 sec D1 1.48689198 sec D13 0.00000400 sec D16 0.00020000 sec IN0 0.00020800 sec ======== CHANNEL f1 ======== SFO1 400.1322007 MHz NUC1 1H P0 15.00 usec P1 15.00 usec PLW1 12.50300026 W ====== GRADIENT CHANNEL ===== GPNAM[1] SMSQ10.100 GPZ1 10.00 % P16 1000.00 usec F1 - Acquisition parameters TD 128 SFO1 400.1322 MHz FIDRES 75.120193 Hz SW 12.015 ppm FnMODE QF F2 - Processing parameters SI 1024 SF 400.1300093 MHz WDW SINE SSB 0 LB 0 Hz GB 0 PC 1.40 F1 - Processing parameters SI 1024 MC2 QF SF 400.1300094 MHz WDW SINE SSB 0 LB 0 Hz GB 0	F2 - Acquisition Parameters Date_ 20171010 Time 15.01 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG cosygpqf TD 2048 SOLVENT D2O NS 1 DS 8 SWH 4807.692 Hz FIDRES 2.347506 Hz AQ 0.2129920 sec RG 192.58 DW 104.000 usec DE 6.50 usec TE 298.0 K D0 0.00000300 sec D1 1.48689198 sec D13 0.00000400 sec D16 0.00020000 sec IN0 0.00020800 sec ======== CHANNEL f1 ======== SFO1 400.1322007 MHz NUC1 1H P0 15.00 usec P1 15.00 usec PLW1 12.50300026 W ====== GRADIENT CHANNEL ===== GPNAM[1] SMSQ10.100 GPZ1 10.00 % P16 1000.00 usec F1 - Acquisition parameters TD 128 SFO1 400.1322 MHz FIDRES 75.120193 Hz SW 12.015 ppm FnMODE QF F2 - Processing parameters SI 1024 SF 400.1299653 MHz WDW SINE SSB 0 LB 0 Hz GB 0 PC 1.40 F1 - Processing parameters SI 1024 MC2 QF SF 400.1299652 MHz WDW SINE SSB 0 LB 0 Hz GB 0

HSQC

```
F2 - Acquisition Parameters
Date_           20171004
Time              16.18
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG  hsqcetgpsisp2.2
TD                2048
SOLVENT           CDCl3
NS                   2
DS                  16
SWH            5341.880 Hz
FIDRES         2.608340 Hz
AQ            0.1916928 sec
RG               192.58
DW               93.600 usec
DE                6.50 usec
TE               298.3 K
CNST2       145.0000000
CNST17       -0.5000000
D0           0.00000300 sec
D1           1.50000000 sec
D4           0.00172414 sec
D11          0.03000000 sec
D16          0.00020000 sec
D24          0.00086207 sec
IN0          0.00003000 sec

======== CHANNEL f1 ========
SFO1         400.1324057 MHz
NUC1                1H
P1                15.00 usec
P2                30.00 usec
P28             1000.00 usec
PLW1         12.50300026 W

======== CHANNEL f2 ========
SFO2         100.6202713 MHz
NUC2                13C
CPDPRG[2 bi_p5m4sp_4sp.2
P3                 9.80 usec
P14              500.00 usec
P24             2000.00 usec
P63             1500.00 usec
PLW0                  0 W
PLW2         60.95399857 W
PLW12         0.91469002 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3             0.500
SPOFFS3                0 Hz
SPW3          8.94419956 W
SPNAM[7]    Crp60comp.4
SPOAL7             0.500
SPOFFS7                0 Hz
SPW7          8.94419956 W
SPNAM[14 Crp32,1.5,20.2
SPOAL14            0.500
SPOFFS14               0 Hz
SPW14         3.81620002 W
SPNAM[31 Crp32,1.5,20.2
SPOAL31            0.500
SPOFFS31               0 Hz
SPW31         0.95405000 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPZ1               80.00 %
GPZ2               20.10 %
GPZ3               11.00 %
GPZ4               -5.00 %
P16              1000.00 usec
P19               600.00 usec

F1 - Acquisition parameters
TD                   256
SFO1           100.6203 MHz
FIDRES       130.208328 Hz
SW              165.639 ppm
FnMODE    Echo-Antiecho

F2 - Processing parameters
SI                  1024
SF           400.1300099 MHz
WDW                QSINE
SSB                    2
LB                     0 Hz
GB                     0
PC                  1.40

F1 - Processing parameters
SI                  1024
MC2        echo-antiecho
SF           100.6127604 MHz
WDW                QSINE
SSB                    2
LB                     0 Hz
GB                     0
```

```
F2 - Acquisition Parameters
Date_           20171010
Time              15.41
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG  hsqcetgpsisp2.2
TD                2048
SOLVENT             D2O
NS                   2
DS                  16
SWH            5341.880 Hz
FIDRES         2.608340 Hz
AQ            0.1916928 sec
RG               192.58
DW               93.600 usec
DE                6.50 usec
TE               298.2 K
CNST2       145.0000000
CNST17       -0.5000000
D0           0.00000300 sec
D1           1.50000000 sec
D4           0.00172414 sec
D11          0.03000000 sec
D16          0.00020000 sec
D24          0.00086207 sec
IN0          0.00003000 sec

======== CHANNEL f1 ========
SFO1         400.1324057 MHz
NUC1                1H
P1                15.00 usec
P2                30.00 usec
P28             1000.00 usec
PLW1         12.50300026 W

======== CHANNEL f2 ========
SFO2         100.6202713 MHz
NUC2                13C
CPDPRG[2 bi_p5m4sp_4sp.2
P3                 9.80 usec
P14              500.00 usec
P24             2000.00 usec
P63             1500.00 usec
PLW0                  0 W
PLW2         60.95399857 W
PLW12         0.91469002 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3             0.500
SPOFFS3                0 Hz
SPW3          8.94419956 W
SPNAM[7]    Crp60comp.4
SPOAL7             0.500
SPOFFS7                0 Hz
SPW7          8.94419956 W
SPNAM[14 Crp32,1.5,20.2
SPOAL14            0.500
SPOFFS14               0 Hz
SPW14         3.81620002 W
SPNAM[31 Crp32,1.5,20.2
SPOAL31            0.500
SPOFFS31               0 Hz
SPW31         0.95405000 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPZ1               80.00 %
GPZ2               20.10 %
GPZ3               11.00 %
GPZ4               -5.00 %
P16              1000.00 usec
P19               600.00 usec

F1 - Acquisition parameters
TD                   256
SFO1           100.6203 MHz
FIDRES       130.208328 Hz
SW              165.639 ppm
FnMODE    Echo-Antiecho

F2 - Processing parameters
SI                  1024
SF           400.1299674 MHz
WDW                QSINE
SSB                    2
LB                     0 Hz
GB                     0
PC                  1.40

F1 - Processing parameters
SI                  1024
MC2        echo-antiecho
SF           100.6127649 MHz
WDW                QSINE
SSB                    2
LB                     0 Hz
GB                     0
```

HMBC

```
F2 - Acquisition Parameters
Date_           20171004
Time               16.01
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG    hmbcgpl2ndqf
TD                 2048
SOLVENT            CDC13
NS                    4
DS                   16
SWH             4807.692 Hz
FIDRES          2.347506 Hz
AQ             0.2129920 sec
RG                192.58
DW               104.000 usec
DE                 6.50 usec
TE                298.1 K
CNST6        125.0000000
CNST7        165.0000000
CNST13         7.5000000
D0            0.00000300 sec
D1            1.50000000 sec
D6            0.06666667 sec
D16           0.00020000 sec
IN0           0.00002240 sec

======== CHANNEL f1 ========
SFO1         400.1322007 MHz
NUC1                 1H
P1                15.00 usec
P2                30.00 usec
PLW1         12.50300026 W

======== CHANNEL f2 ========
SFO2         100.6228119 MHz
NUC2                13C
P3                 9.80 usec
PLW2         60.95399857 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPNAM[5]      SMSQ10.100
GPNAM[6]      SMSQ10.100
GPZ1               50.00 %
GPZ2               30.00 %
GPZ3               40.10 %
GPZ4               15.00 %
GPZ5              -10.00 %
GPZ6               -5.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  128
SFO1            100.6228 MHz
FIDRES        348.772308 Hz
SW               221.833 ppm
FnMODE              QF

F2 - Processing parameters
SI                 2048
SF           400.1300104 MHz
WDW                SINE
SSB                   0
LB                    0 Hz
GB                    0
PC                 1.40

F1 - Processing parameters
SI                 1024
MC2                  QF
SF           100.6127690 MHz
WDW                SINE
SSB                   0
LB                    0 Hz
GB                    0
```

```
F2 - Acquisition Parameters
Date_           20171010
Time               15.06
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG    hmbcgpl2ndqf
TD                 2048
SOLVENT             D2O
NS                    4
DS                   16
SWH             4807.692 Hz
FIDRES          2.347506 Hz
AQ             0.2129920 sec
RG                192.58
DW               104.000 usec
DE                 6.50 usec
TE                298.1 K
CNST6        125.0000000
CNST7        165.0000000
CNST13         7.5000000
D0            0.00000300 sec
D1            1.50000000 sec
D6            0.06666667 sec
D16           0.00020000 sec
IN0           0.00002240 sec

======== CHANNEL f1 ========
SFO1         400.1322007 MHz
NUC1                 1H
P1                15.00 usec
P2                30.00 usec
PLW1         12.50300026 W

======== CHANNEL f2 ========
SFO2         100.6228119 MHz
NUC2                13C
P3                 9.80 usec
PLW2         60.95399857 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPNAM[5]      SMSQ10.100
GPNAM[6]      SMSQ10.100
GPZ1               50.00 %
GPZ2               30.00 %
GPZ3               40.10 %
GPZ4               15.00 %
GPZ5              -10.00 %
GPZ6               -5.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  128
SFO1            100.6228 MHz
FIDRES        348.772308 Hz
SW               221.833 ppm
FnMODE              QF

F2 - Processing parameters
SI                 2048
SF           400.1299655 MHz
WDW                SINE
SSB                   0
LB                    0 Hz
GB                    0
PC                 1.40

F1 - Processing parameters
SI                 1024
MC2                  QF
SF           100.6127690 MHz
WDW                SINE
SSB                   0
LB                    0 Hz
GB                    0
```

	ortho 4	ortho 5
^{1}H	F2 - Acquisition Parameters Date_ 20180308 Time 15.49 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zg30 TD 65536 SOLVENT D2O NS 16 DS 2 SWH 8012.820 Hz FIDRES 0.122266 Hz AQ 4.0894465 sec RG 23.44 DW 62.400 usec DE 6.50 usec TE 298.2 K D1 1.00000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 400.1324710 MHz NUC1 1H P1 15.00 usec PLW1 12.50300026 W F2 - Processing parameters SI 65536 SF 400.1299640 MHz WDW EM SSB 0 LB 0.30 Hz GB 0 PC 1.00	F2 - Acquisition Parameters Date_ 20171012 Time 11.33 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zg TD 48076 SOLVENT D2O NS 8 DS 2 SWH 6002.401 Hz FIDRES 0.124852 Hz AQ 4.0047307 sec RG 132.62 DW 83.300 usec DE 16.70 usec TE 298.2 K D1 5.00000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 400.1320007 MHz NUC1 1H P1 15.00 usec PLW1 12.50300026 W F2 - Processing parameters SI 32768 SF 400.1299643 MHz WDW EM SSB 0 LB 0.30 Hz GB 0 PC 1.00

^{13}C	F2 - Acquisition Parameters Date_ 20180312 Time 0.07 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zgpg30 TD 65536 SOLVENT D2O NS 3000 DS 4 SWH 22058.824 Hz FIDRES 0.336591 Hz AQ 1.4854827 sec RG 192.58 DW 22.667 usec DE 6.50 usec TE 298.2 K D1 2.00000000 sec D11 0.03000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 100.6223253 MHz NUC1 13C P1 9.80 usec PLW1 60.95399857 W ======== CHANNEL f2 ======== SFO2 400.1316005 MHz NUC2 1H CPDPRG[2 waltz16 PCPD2 90.00 usec PLW2 12.50300026 W PLW12 0.34731001 W PLW13 0.28132001 W F2 - Processing parameters SI 65536 SF 100.6127690 MHz WDW EM SSB 0 LB 1.00 Hz GB 0 PC 1.40	F2 - Acquisition Parameters Date_ 20171014 Time 22.26 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zgpg30 TD 65536 SOLVENT D2O NS 4000 DS 4 SWH 22058.824 Hz FIDRES 0.336591 Hz AQ 1.4854827 sec RG 192.58 DW 22.667 usec DE 6.50 usec TE 298.2 K D1 2.00000000 sec D11 0.03000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 100.6223253 MHz NUC1 13C P1 9.80 usec PLW1 60.95399857 W ======== CHANNEL f2 ======== SFO2 400.1316005 MHz NUC2 1H CPDPRG[2 waltz16 PCPD2 90.00 usec PLW2 12.50300026 W PLW12 0.34731001 W PLW13 0.28132001 W F2 - Processing parameters SI 65536 SF 100.6127690 MHz WDW EM SSB 0 LB 1.00 Hz GB 0 PC 1.40

DEPT	F2 - Acquisition Parameters Date_ 20180308 Time 17.16 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG deptqgpsp TD 65536 SOLVENT D2O NS 256 DS 4 SWH 22058.824 Hz FIDRES 0.336591 Hz AQ 1.4854827 sec RG 192.58 DW 22.667 usec DE 6.50 usec TE 298.2 K CNST2 145.0000000 CNST12 1.5000000 D1 2.00000000 sec D2 0.00344828 sec D12 0.00002000 sec D16 0.00020000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 100.6223258 MHz NUC1 13C P1 9.80 usec P13 2000.00 usec PLW0 0 W PLW1 60.95399857 W SPNAM[5] Crp60comp.4 SPOAL5 0.500 SPOFFS5 0 Hz SPW5 8.94419956 W ======== CHANNEL f2 ======== SFO2 400.1316005 MHz NUC2 1H CPDPRG[2 waltz16 P0 22.50 usec P3 15.00 usec P4 30.00 usec PCPD2 90.00 usec PLW2 12.50300026 W PLW12 0.34731001 W ====== GRADIENT CHANNEL ===== GPNAM[1] SMSQ10.32 GPNAM[2] SMSQ10.32 GPNAM[3] SMSQ10.32 GPZ1 31.00 % GPZ2 31.00 % GPZ3 31.00 % P16 1000.00 usec F2 - Processing parameters SI 65536 SF 100.6127690 MHz WDW EM SSB 0 LB 1.00 Hz GB 0 PC 1.40	F2 - Acquisition Parameters Date_ 20171012 Time 11.51 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG deptqgpsp TD 65536 SOLVENT D2O NS 256 DS 4 SWH 22058.824 Hz FIDRES 0.336591 Hz AQ 1.4854827 sec RG 192.58 DW 22.667 usec DE 6.50 usec TE 298.2 K CNST2 145.0000000 CNST12 1.5000000 D1 2.00000000 sec D2 0.00344828 sec D12 0.00002000 sec D16 0.00020000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 100.6223258 MHz NUC1 13C P1 9.80 usec P13 2000.00 usec PLW0 0 W PLW1 60.95399857 W SPNAM[5] Crp60comp.4 SPOAL5 0.500 SPOFFS5 0 Hz SPW5 8.94419956 W ======== CHANNEL f2 ======== SFO2 400.1316005 MHz NUC2 1H CPDPRG[2 waltz16 P0 22.50 usec P3 15.00 usec P4 30.00 usec PCPD2 90.00 usec PLW2 12.50300026 W PLW12 0.34731001 W ====== GRADIENT CHANNEL ===== GPNAM[1] SMSQ10.32 GPNAM[2] SMSQ10.32 GPNAM[3] SMSQ10.32 GPZ1 31.00 % GPZ2 31.00 % GPZ3 31.00 % P16 1000.00 usec F2 - Processing parameters SI 65536 SF 100.6127690 MHz WDW EM SSB 0 LB 1.00 Hz GB 0 PC 1.40

^{11}B	F2 - Acquisition Parameters Date_ 20180312 Time 0.31 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zg TD 65536 SOLVENT D2O NS 128 DS 4 SWH 25510.203 Hz FIDRES 0.389255 Hz AQ 1.2845056 sec RG 192.58 DW 19.600 usec DE 6.50 usec TE 298.1 K D1 1.00000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 128.3776052 MHz NUC1 11B P1 17.05 usec PLW1 11.69499969 W F2 - Processing parameters SI 32768 SF 128.3776052 MHz WDW no SSB 0 LB 0 Hz GB 0 PC 1.40	F2 - Acquisition Parameters Date_ 20171013 Time 7.17 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG zg TD 65536 SOLVENT D2O NS 128 DS 4 SWH 25510.203 Hz FIDRES 0.389255 Hz AQ 1.2845056 sec RG 192.58 DW 19.600 usec DE 6.50 usec TE 298.1 K D1 1.00000000 sec TD0 1 ======== CHANNEL f1 ======== SFO1 128.3776052 MHz NUC1 11B P1 17.05 usec PLW1 11.69499969 W F2 - Processing parameters SI 32768 SF 128.3776052 MHz WDW no SSB 0 LB 0 Hz GB 0 PC 1.40

COSY	F2 - Acquisition Parameters Date_ 20180309 Time 9.05 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG cosygpqf TD 2048 SOLVENT D2O NS 1 DS 8 SWH 4807.692 Hz FIDRES 2.347506 Hz AQ 0.2129920 sec RG 86.04 DW 104.000 usec DE 6.50 usec TE 298.1 K D0 0.00000300 sec D1 1.48689198 sec D13 0.00000400 sec D16 0.00020000 sec IN0 0.00020800 sec ======== CHANNEL f1 ======== SFO1 400.1322007 MHz NUC1 1H P0 15.00 usec P1 15.00 usec PLW1 12.50300026 W ====== GRADIENT CHANNEL ===== GPNAM[1] SMSQ10.100 GPZ1 10.00 % P16 1000.00 usec F1 - Acquisition parameters TD 128 SFO1 400.1322 MHz FIDRES 75.120193 Hz SW 12.015 ppm FnMODE QF F2 - Processing parameters SI 1024 SF 400.1299639 MHz WDW SINE SSB 0 LB 0 Hz GB 0 PC 1.40 F1 - Processing parameters SI 1024 MC2 QF SF 400.1299637 MHz WDW SINE SSB 0 LB 0 Hz GB 0	F2 - Acquisition Parameters Date_ 20171012 Time 11.53 INSTRUM spect PROBHD 5 mm PABBO BB- PULPROG cosygpqf TD 2048 SOLVENT D2O NS 1 DS 8 SWH 4807.692 Hz FIDRES 2.347506 Hz AQ 0.2129920 sec RG 192.58 DW 104.000 usec DE 6.50 usec TE 298.0 K D0 0.00000300 sec D1 1.48689198 sec D13 0.00000400 sec D16 0.00020000 sec IN0 0.00020800 sec ======== CHANNEL f1 ======== SFO1 400.1322007 MHz NUC1 1H P0 15.00 usec P1 15.00 usec PLW1 12.50300026 W ====== GRADIENT CHANNEL ===== GPNAM[1] SMSQ10.100 GPZ1 10.00 % P16 1000.00 usec F1 - Acquisition parameters TD 128 SFO1 400.1322 MHz FIDRES 75.120193 Hz SW 12.015 ppm FnMODE QF F2 - Processing parameters SI 1024 SF 400.1299635 MHz WDW SINE SSB 0 LB 0 Hz GB 0 PC 1.40 F1 - Processing parameters SI 1024 MC2 QF SF 400.1299638 MHz WDW SINE SSB 0 LB 0 Hz GB 0

HSQC

```
F2 - Acquisition Parameters
Date_          20180312
Time               0.10
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG  hsqcetgpsisp2.2
TD                 2048
SOLVENT             D2O
NS                    2
DS                   16
SWH            5341.880 Hz
FIDRES         2.608340 Hz
AQ            0.1916928 sec
RG               192.58
DW                93.600 usec
DE                 6.50 usec
TE                298.0 K
CNST2       145.0000000
CNST17       -0.5000000
D0            0.00000300 sec
D1            1.50000000 sec
D4            0.00172414 sec
D11           0.03000000 sec
D16           0.00020000 sec
D24           0.00086207 sec
IN0           0.00003000 sec

======== CHANNEL f1 ========
SFO1        400.1324057 MHz
NUC1                1H
P1                15.00 usec
P2                30.00 usec
P28             1000.00 usec
PLW1        12.50300026 W

======== CHANNEL f2 ========
SFO2        100.6202713 MHz
NUC2                13C
CPDPRG[2 bi_p5m4sp_4sp.2
P3                 9.80 usec
P14              500.00 usec
P24             2000.00 usec
P63             1500.00 usec
PLW0                  0 W
PLW2         60.95399857 W
PLW12         0.91469002 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3             0.500
SPOFFS3                0 Hz
SPW3          8.94419956 W
SPNAM[7]    Crp60comp.4
SPOAL7             0.500
SPOFFS7                0 Hz
SPW7          8.94419956 W
SPNAM[14 Crp32,1.5,20.2
SPOAL14            0.500
SPOFFS14               0 Hz
SPW14         3.81620002 W
SPNAM[31 Crp32,1.5,20.2
SPOAL31            0.500
SPOFFS31               0 Hz
SPW31         0.95405000 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPZ1              80.00 %
GPZ2              20.10 %
GPZ3              11.00 %
GPZ4              -5.00 %
P16             1000.00 usec
P19              600.00 usec

F1 - Acquisition parameters
TD                   256
SFO1           100.6203 MHz
FIDRES        130.208328 Hz
SW             165.639 ppm
FnMODE    Echo-Antiecho

F2 - Processing parameters
SI                  1024
SF           400.1300000 MHz
WDW                QSINE
SSB                    2
LB                     0 Hz
GB                     0
PC                  1.40

F1 - Processing parameters
SI                  1024
MC2       echo-antiecho
SF           100.6127690 MHz
WDW                QSINE
SSB                    2
LB                     0 Hz
GB                     0
```

```
F2 - Acquisition Parameters
Date_          20171012
Time              12.22
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG  hsqcetgpsisp2.2
TD                 2048
SOLVENT             D2O
NS                    2
DS                   16
SWH            5341.880 Hz
FIDRES         2.608340 Hz
AQ            0.1916928 sec
RG               192.58
DW                93.600 usec
DE                 6.50 usec
TE                298.3 K
CNST2       145.0000000
CNST17       -0.5000000
D0            0.00000300 sec
D1            1.50000000 sec
D4            0.00172414 sec
D11           0.03000000 sec
D16           0.00020000 sec
D24           0.00086207 sec
IN0           0.00003000 sec

======== CHANNEL f1 ========
SFO1        400.1324057 MHz
NUC1                1H
P1                15.00 usec
P2                30.00 usec
P28             1000.00 usec
PLW1        12.50300026 W

======== CHANNEL f2 ========
SFO2        100.6202713 MHz
NUC2                13C
CPDPRG[2 bi_p5m4sp_4sp.2
P3                 9.80 usec
P14              500.00 usec
P24             2000.00 usec
P63             1500.00 usec
PLW0                  0 W
PLW2         60.95399857 W
PLW12         0.91469002 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3             0.500
SPOFFS3                0 Hz
SPW3          8.94419956 W
SPNAM[7]    Crp60comp.4
SPOAL7             0.500
SPOFFS7                0 Hz
SPW7          8.94419956 W
SPNAM[14 Crp32,1.5,20.2
SPOAL14            0.500
SPOFFS14               0 Hz
SPW14         3.81620002 W
SPNAM[31 Crp32,1.5,20.2
SPOAL31            0.500
SPOFFS31               0 Hz
SPW31         0.95405000 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPZ1              80.00 %
GPZ2              20.10 %
GPZ3              11.00 %
GPZ4              -5.00 %
P16             1000.00 usec
P19              600.00 usec

F1 - Acquisition parameters
TD                   256
SFO1           100.6203 MHz
FIDRES        130.208328 Hz
SW             165.639 ppm
FnMODE    Echo-Antiecho

F2 - Processing parameters
SI                  1024
SF           400.1299657 MHz
WDW                QSINE
SSB                    2
LB                     0 Hz
GB                     0
PC                  1.40

F1 - Processing parameters
SI                  1024
MC2       echo-antiecho
SF           100.6127690 MHz
WDW                QSINE
SSB                    2
LB                     0 Hz
GB                     0
```

HMBC

```
F2 - Acquisition Parameters
Date_           20180309
Time                9.11
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG    hmbcgpl2ndqf
TD                 2048
SOLVENT             D2O
NS                    4
DS                   16
SWH            4807.692 Hz
FIDRES         2.347506 Hz
AQ            0.2129920 sec
RG               192.58
DW              104.000 usec
DE                6.50 usec
TE               298.1 K
CNST6        125.0000000
CNST7        165.0000000
CNST13         7.5000000
D0            0.00000300 sec
D1            1.50000000 sec
D6            0.06666667 sec
D16           0.00020000 sec
IN0           0.00002240 sec

======== CHANNEL f1 ========
SFO1         400.1322007 MHz
NUC1                1H
P1                15.00 usec
P2                30.00 usec
PLW1         12.50300026 W

======== CHANNEL f2 ========
SFO2         100.6228119 MHz
NUC2                13C
P3                 9.80 usec
PLW2         60.95399857 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPNAM[5]      SMSQ10.100
GPNAM[6]      SMSQ10.100
GPZ1              50.00 %
GPZ2              30.00 %
GPZ3              40.10 %
GPZ4              15.00 %
GPZ5             -10.00 %
GPZ6              -5.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  128
SFO1          100.6228 MHz
FIDRES        348.772308 Hz
SW              221.833 ppm
FnMODE              QF

F2 - Processing parameters
SI                 2048
SF          400.1300000 MHz
WDW                SINE
SSB                   0
LB                    0 Hz
GB                    0
PC                 1.40

F1 - Processing parameters
SI                 1024
MC2                  QF
SF          100.6127690 MHz
WDW                SINE
SSB                   0
LB                    0 Hz
GB                    0
```

```
F2 - Acquisition Parameters
Date_           20171012
Time               11.58
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG    hmbcgpl2ndqf
TD                 2048
SOLVENT             D2O
NS                    4
DS                   16
SWH            4807.692 Hz
FIDRES         2.347506 Hz
AQ            0.2129920 sec
RG               192.58
DW              104.000 usec
DE                6.50 usec
TE               298.1 K
CNST6        125.0000000
CNST7        165.0000000
CNST13         7.5000000
D0            0.00000300 sec
D1            1.50000000 sec
D6            0.06666667 sec
D16           0.00020000 sec
IN0           0.00002240 sec

======== CHANNEL f1 ========
SFO1         400.1322007 MHz
NUC1                1H
P1                15.00 usec
P2                30.00 usec
PLW1         12.50300026 W

======== CHANNEL f2 ========
SFO2         100.6228119 MHz
NUC2                13C
P3                 9.80 usec
PLW2         60.95399857 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPNAM[5]      SMSQ10.100
GPNAM[6]      SMSQ10.100
GPZ1              50.00 %
GPZ2              30.00 %
GPZ3              40.10 %
GPZ4              15.00 %
GPZ5             -10.00 %
GPZ6              -5.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  128
SFO1          100.6228 MHz
FIDRES        348.772308 Hz
SW              221.833 ppm
FnMODE              QF

F2 - Processing parameters
SI                 2048
SF          400.1300000 MHz
WDW                SINE
SSB                   0
LB                    0 Hz
GB                    0
PC                 1.40

F1 - Processing parameters
SI                 1024
MC2                  QF
SF          100.6127690 MHz
WDW                SINE
SSB                   0
LB                    0 Hz
GB                    0
```

References

1. Campkin, D.M.; Shimadate, Y.; Bartholomew, B.; Bernhardt, P.V.; Nash, R.J.; Sakoff, J.A.; Kato, A.; Simone, M. Borylated 2,3,4,5-Tetrachlorophthalimide and Their 2,3,4,5-Tetrachlorobenzamide Analogues: Synthesis, Their Glycosidase Inhibition and Anticancer Properties in View to Boron Neutron Capture Therapy. *Molecules* **2022**, *27*, 3447-3475, doi:https://doi.org/10.3390/molecules27113447.
2. Boron Molecular, https://www.boronmolecular.com. A fine chemicals manufacturer.
3. Legge, W.J.; Shimadate, Y.; Sakoff, J.; Houston, T.A.; Kato, A.; Bernhardt, P.V.; Simone, M. Borylated methyl cinnamates: Green synthesis, characterization, crystallographic analysis and biological activities – in glycosidase inhibition and in cancer cells lines. *Beilstein Arch.* **2021**, 20214, doi:https://doi.org/10.3762/bxiv.2021.4.v1.
4. Simone, M. Diastereoselective Synthesis of the Borylated D-Galactose Monosaccharide 3-Boronic-3-Deoxy-D-Galactose and Biological Evaluation in Glycosidase Inhibition and in Cancer for Boron Neutron Capture Therapy (BNCT). *Molecules* **2023**, *28*, 4321-4337, doi:https://doi.org/10.3390/molecules28114321.
5. Simone, M. Borylated Monosaccharide 3-Boronic-3-deoxy-D-galactose: Detailed NMR Spectroscopic Characterisation, and Method for Spectroscopic Analysis of Anomeric and Boron Equilibria. *Internat. J. Mol. Sci.* **2024**, *25*, 12396, doi:https://doi.org/10.3390/ijms252212396.