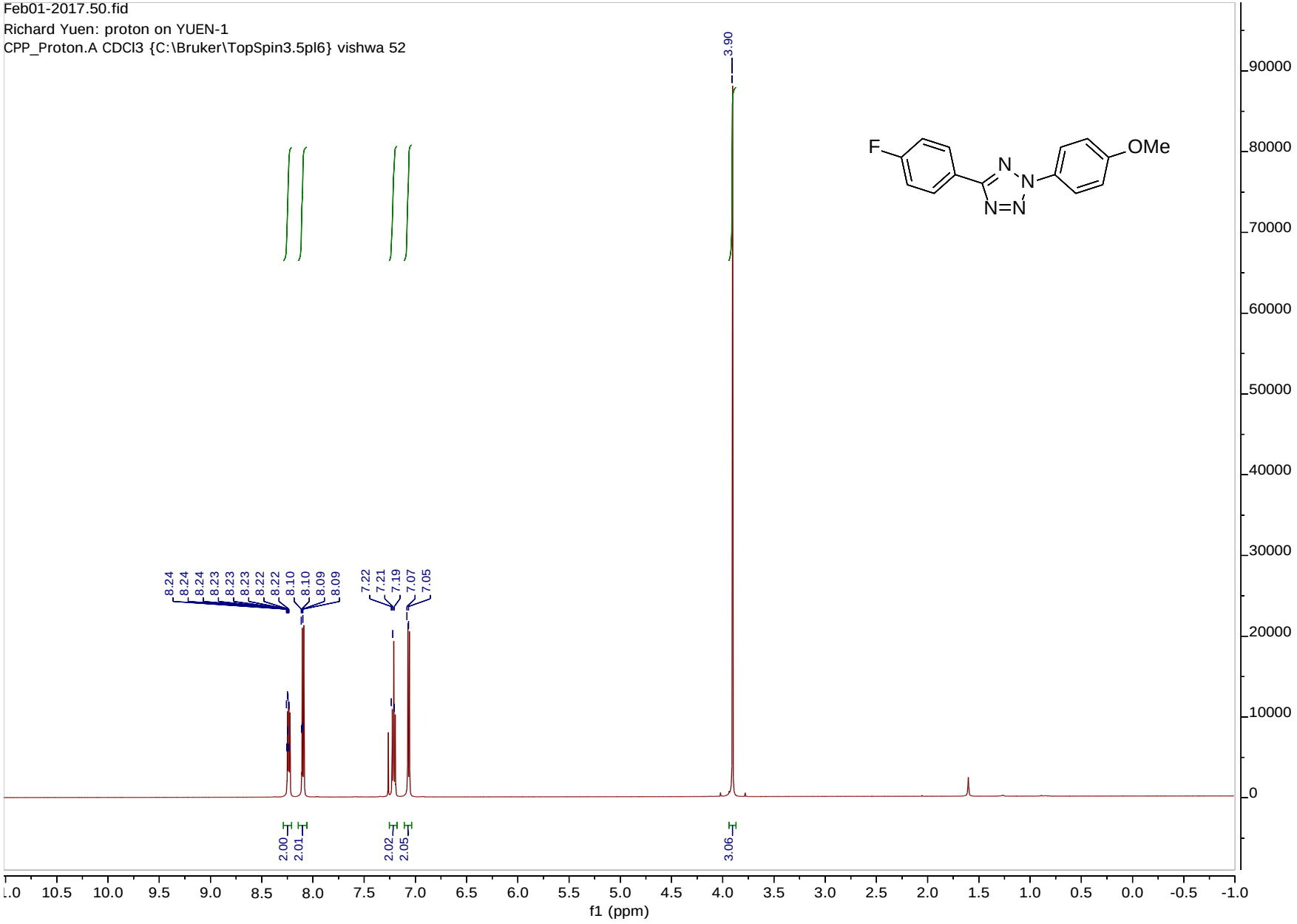
Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

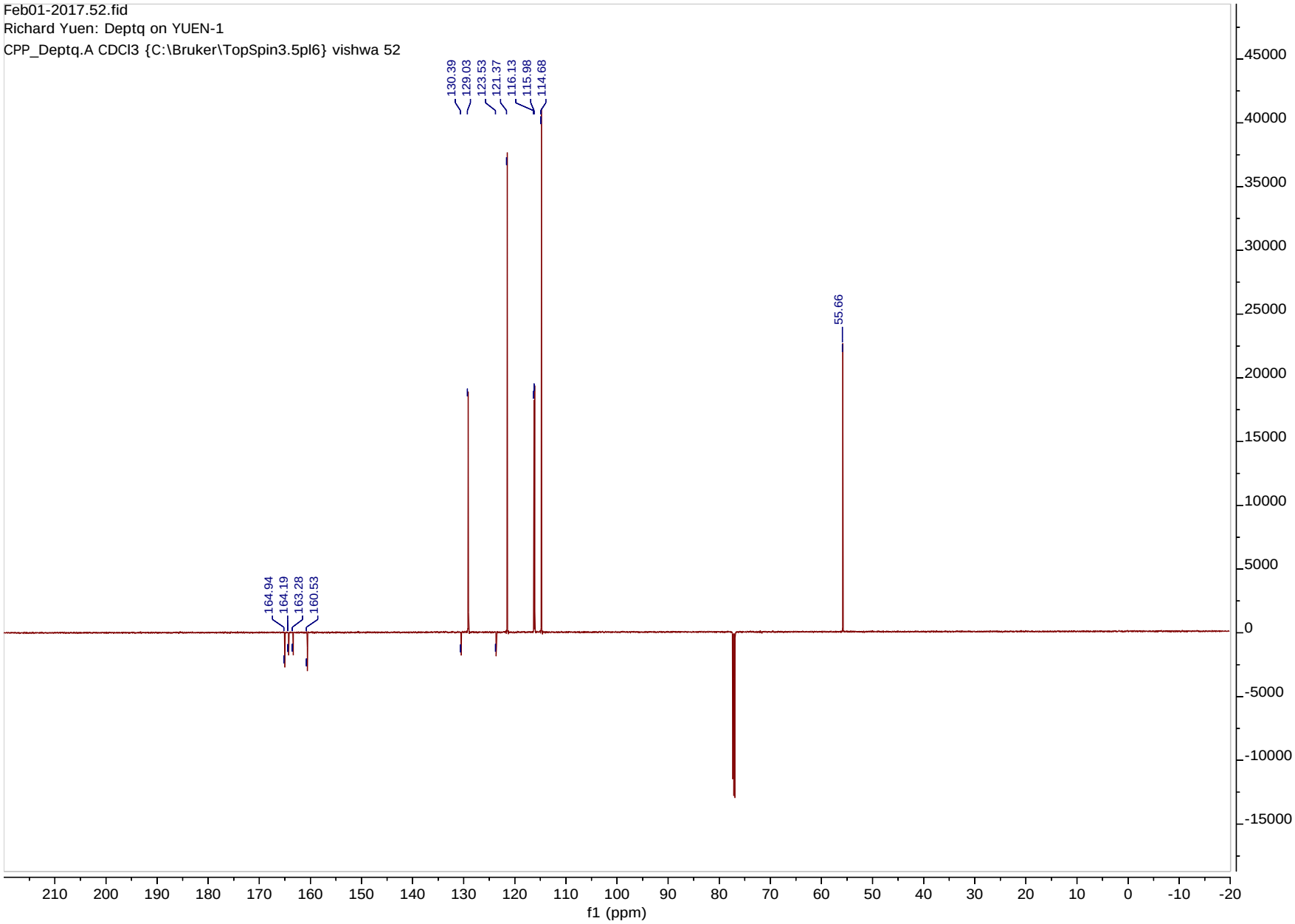
Design, synthesis, and evaluation of positron emission tomography/fluorescence dual imaging probes for targeting facilitated glucose transporter 1 (GLUT1)

Richard Yuen, Michael Wagner, Susan Richter, Jennifer Dufour, Melinda Wuest, Frederick G. West, Frank Wuest

Supplementary Information

NMR Data


Feb01-2017.50.fid
Richard Yuen: proton on YUEN-1
CPP_Proton.A CDCl3 {C:\Bruker\TopSpin3.5pl6} vishwa 52
3.90
F
N
N
N=N
OMe
8.24
8.24
8.24
8.23
8.23
8.23
8.22
8.22
8.10
8.10
8.09
8.09
8.09
7.22
7.21
7.19
7.07
7.05
2.00
2.01
2.02
2.05
3.06
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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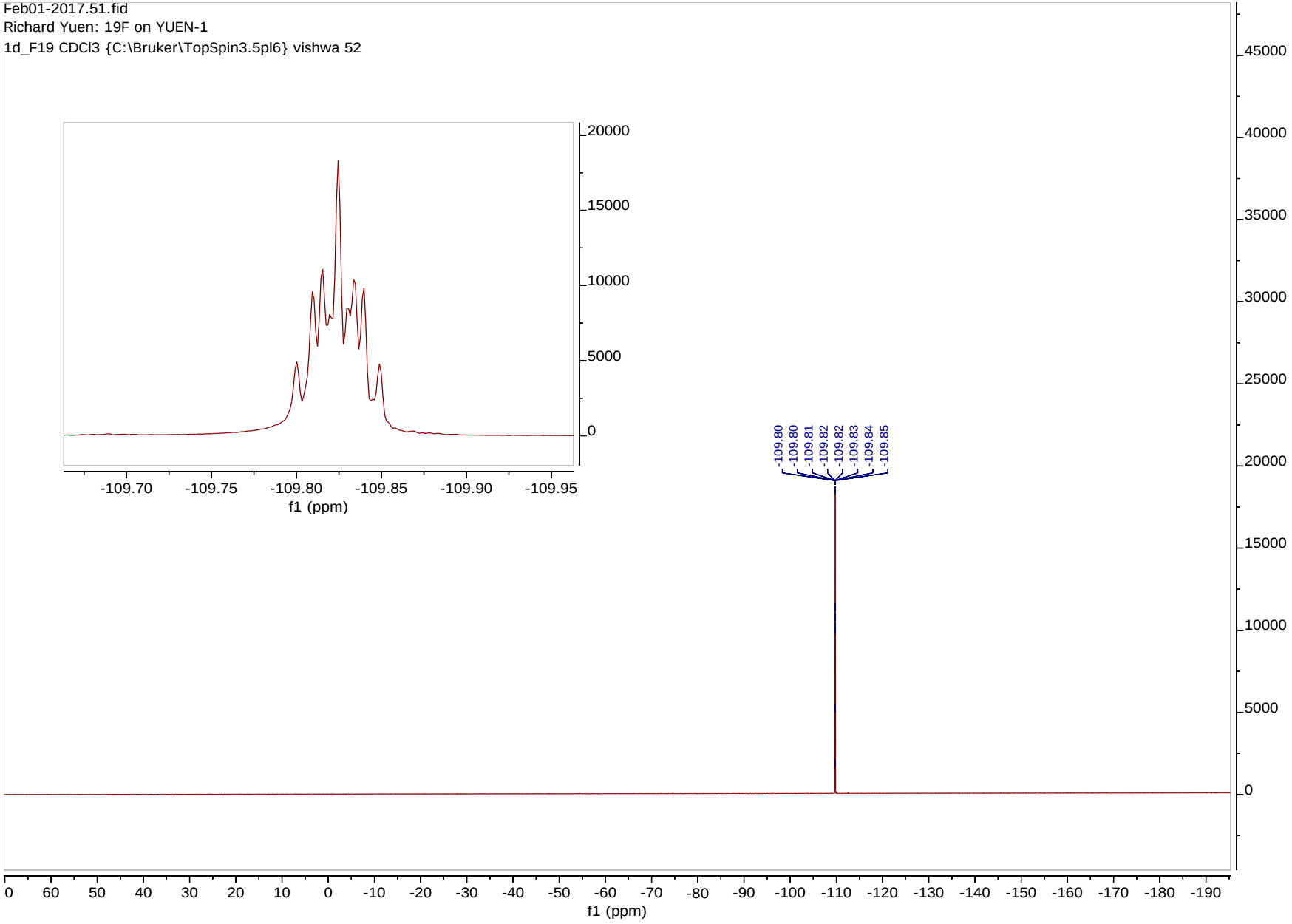
Richard Yuen: Deptq on YUEN-1
CPP_Deptq.A CDCl3 {C:\Bruker\TopSpin3.5pl6} vishwa 52
130.39
129.03
123.53
121.37
116.13
115.98
114.68
55.66
164.94
164.19
163.28
160.53
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
-10000
-15000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)


Feb01-2017.51.fid
Richard Yuen: 19F on YUEN-1
1d_F19 CDCl3 {C:\Bruker\TopSpin3.5pl6} vishwa 52
20000
15000
10000
5000
0
-109.70
-109.75
-109.80
-109.85
-109.90
-109.95
f1 (ppm)
-109.80
-109.80
-109.81
-109.82
-109.82
-109.83
-109.84
-109.85
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
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-160
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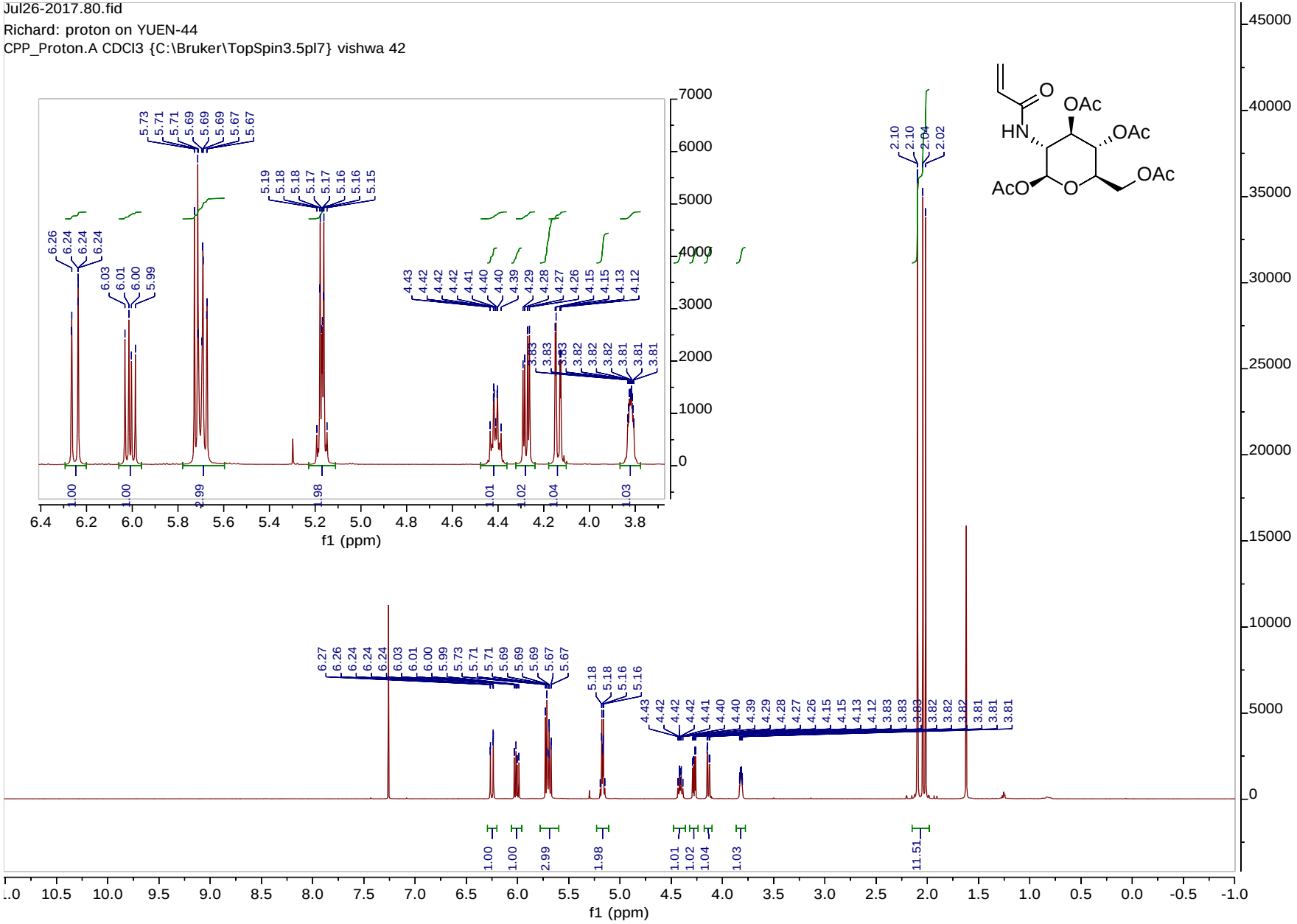
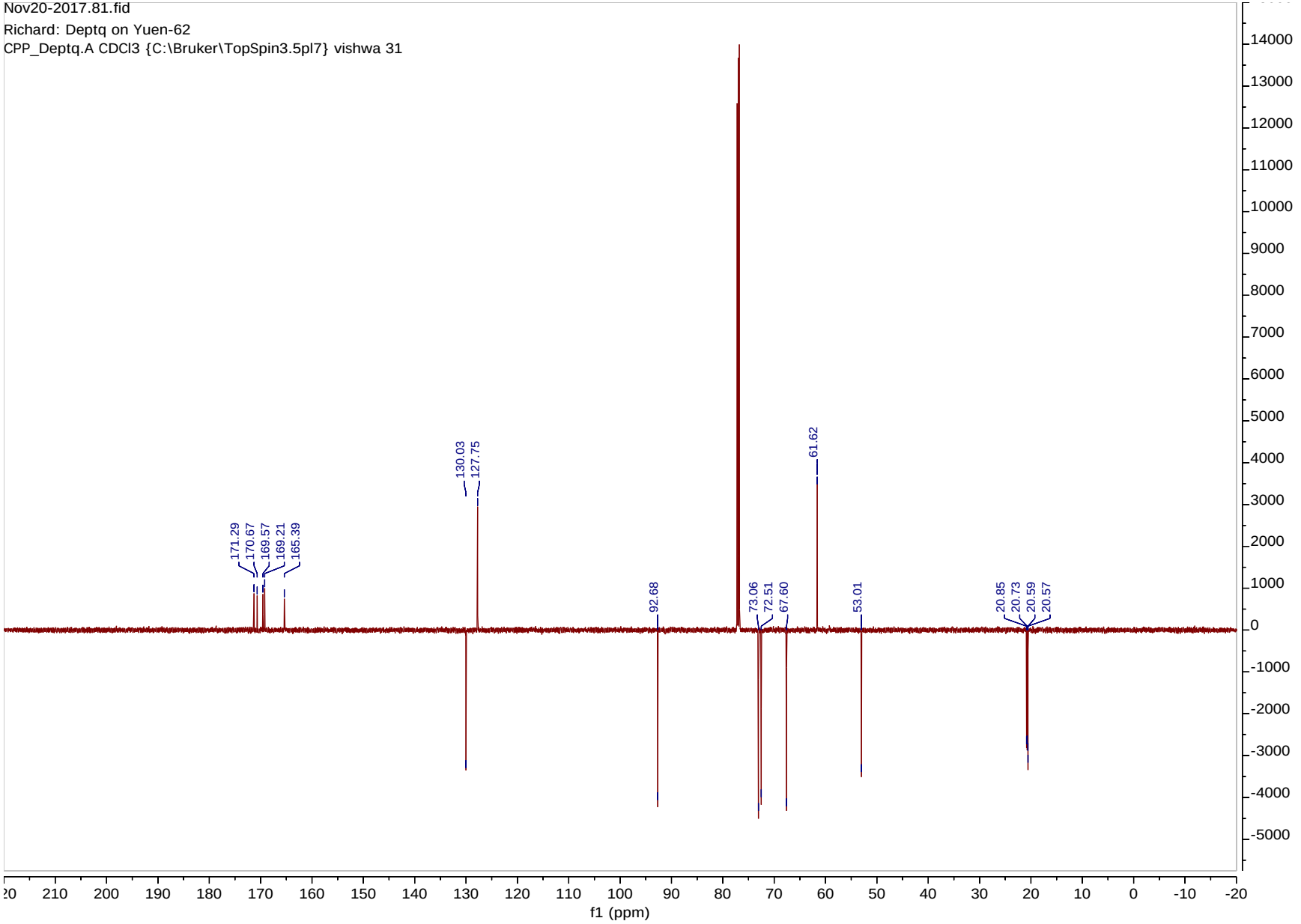
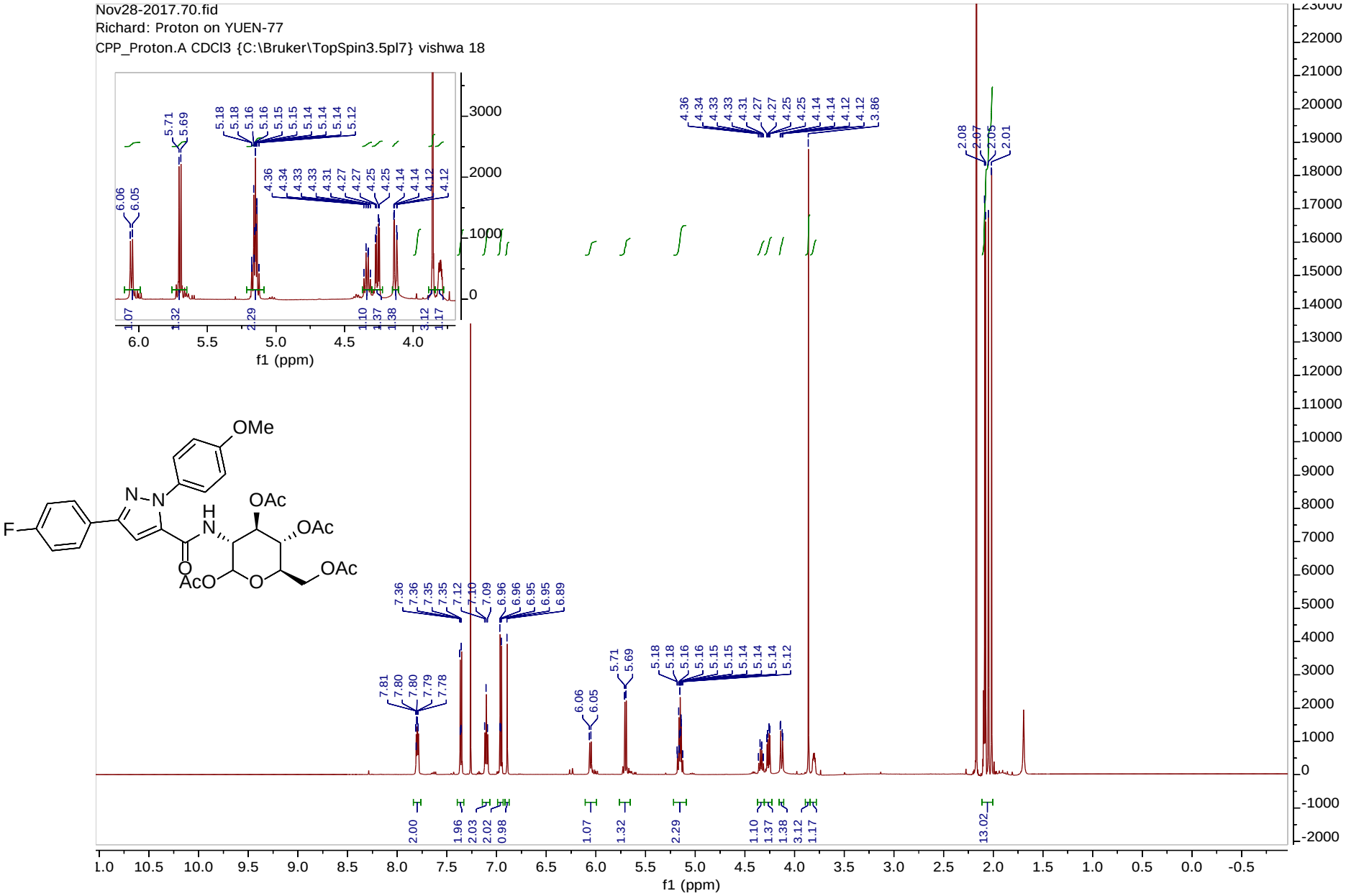
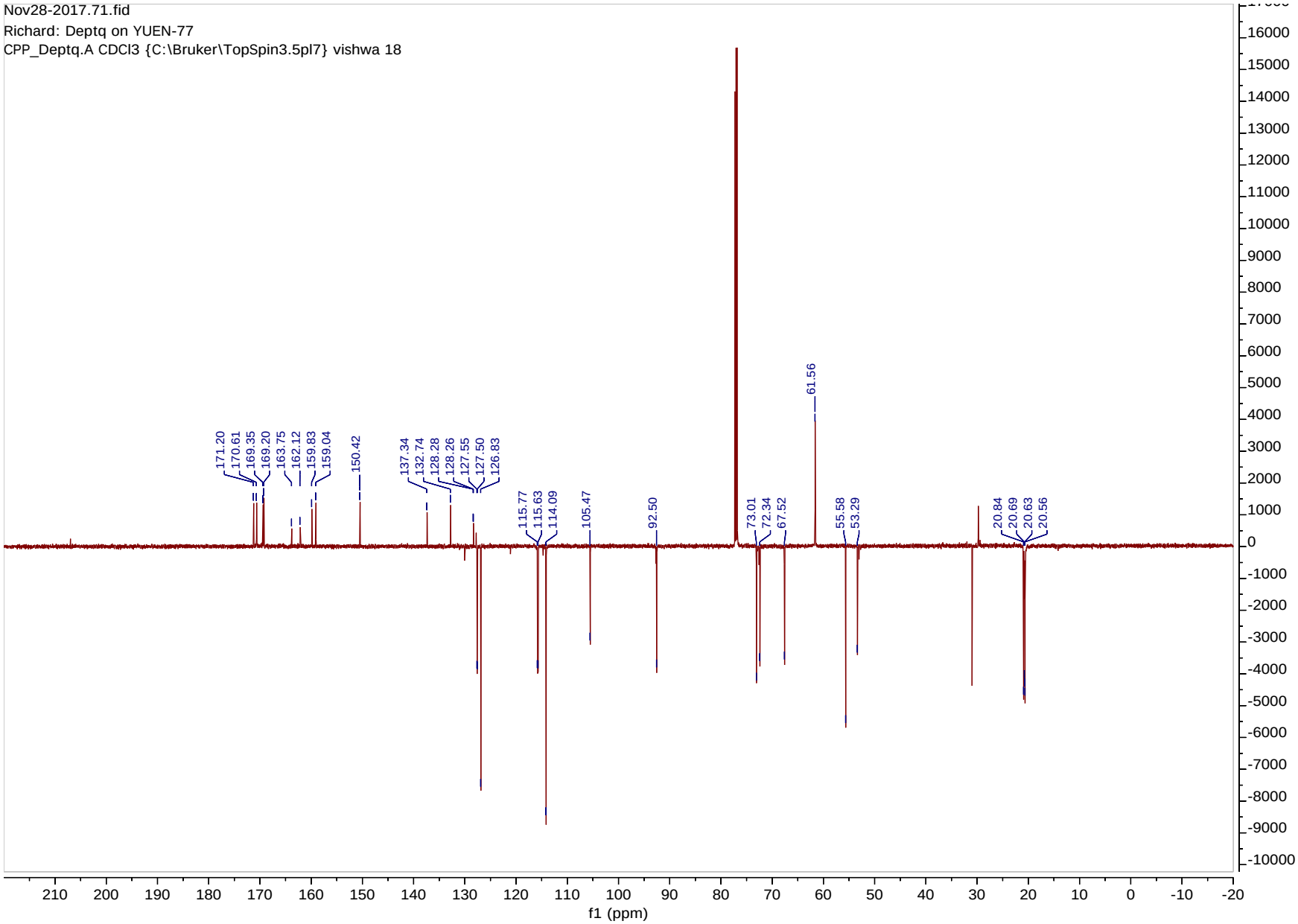
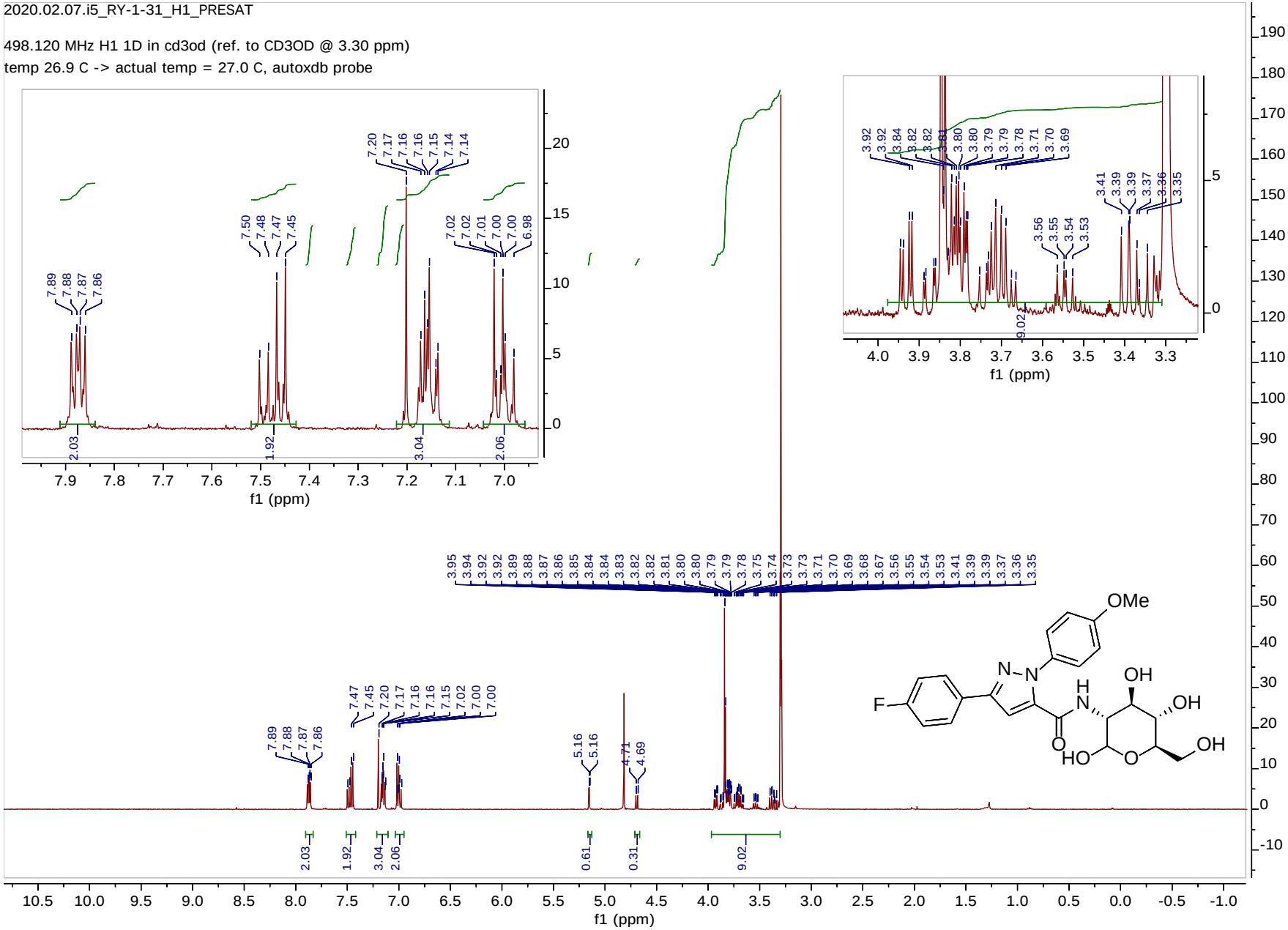
-180
-190
f1 (ppm)

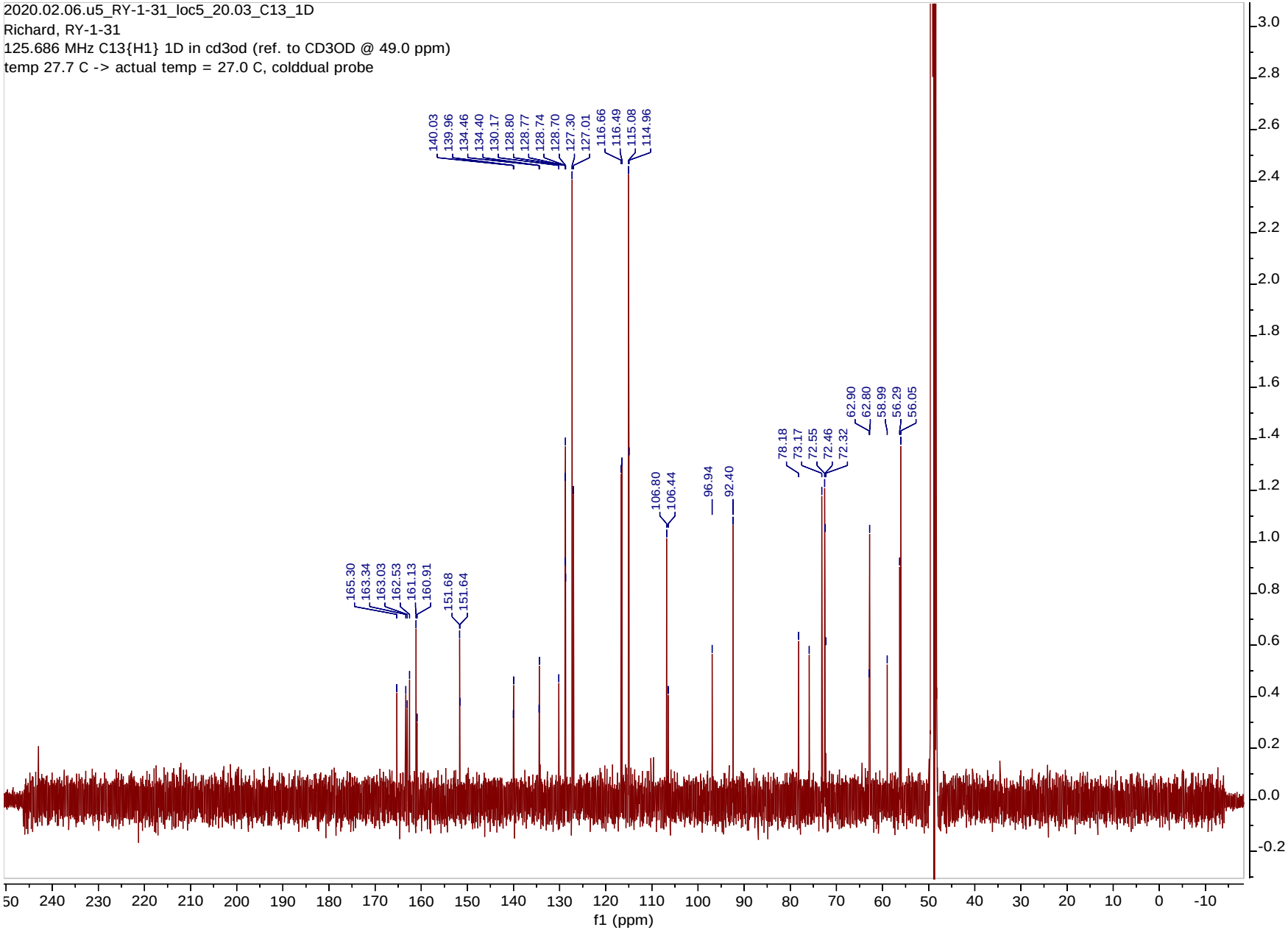

Jul26-2017.80.fid
Richard: proton on YUEN-44
CPP_Proton.A CDCl3 {C:\Bruker\TopSpin3.5pl7} vishwa 42
f1 (ppm)
f1 (ppm)
HN
O
OAc
OAc
OAc
AcO
O

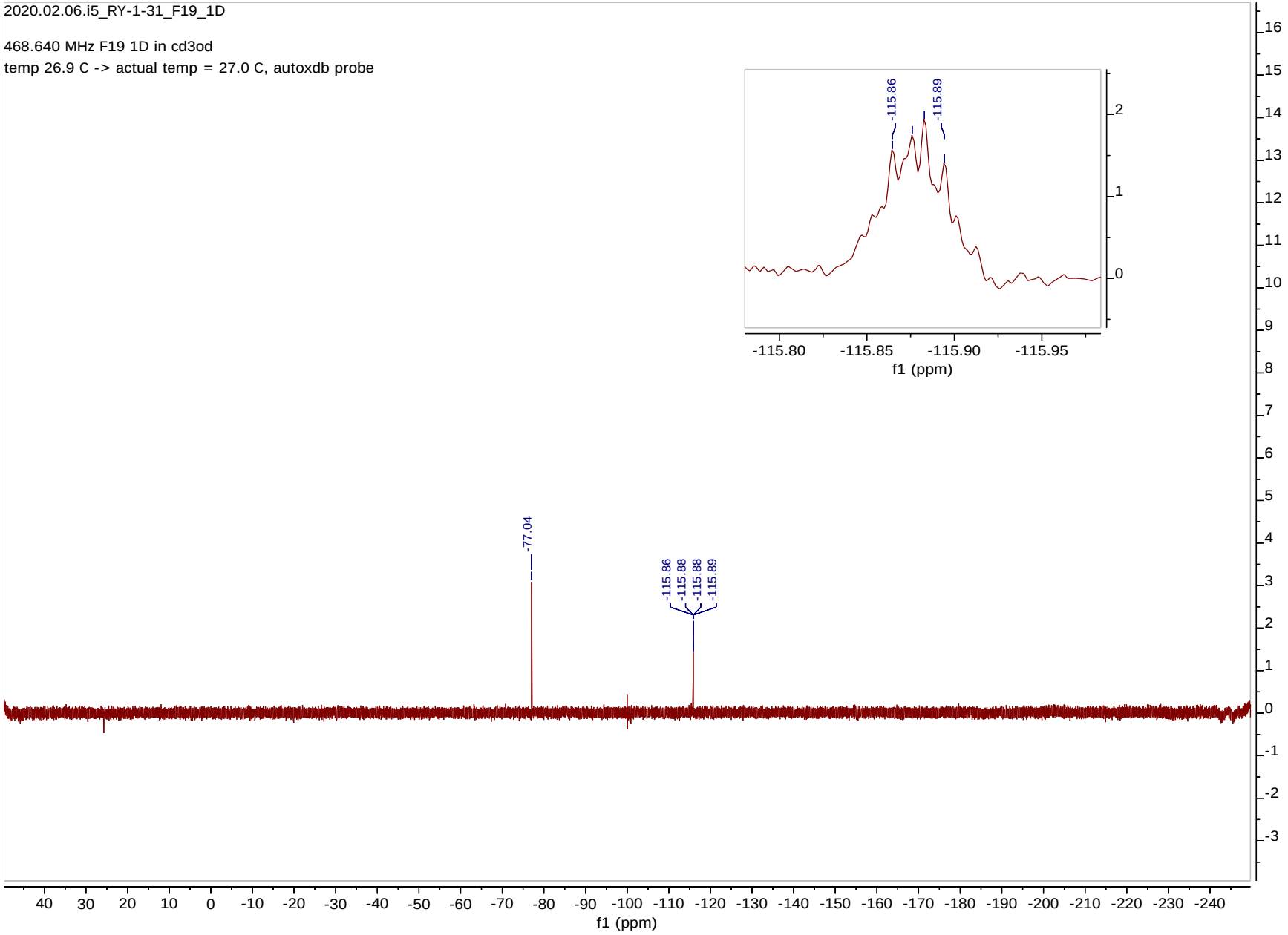

Nov20-2017.81.fid
Richard: Deptq on Yuen-62
CPP_Deptq.A CDCl3 {C:\Bruker\TopSpin3.5pl7} vishwa 31
171.29
170.67
169.57
169.21
165.39
130.03
127.75
92.68
73.06
72.51
67.60
61.62
53.01
20.85
20.73
20.59
20.57
f1 (ppm)

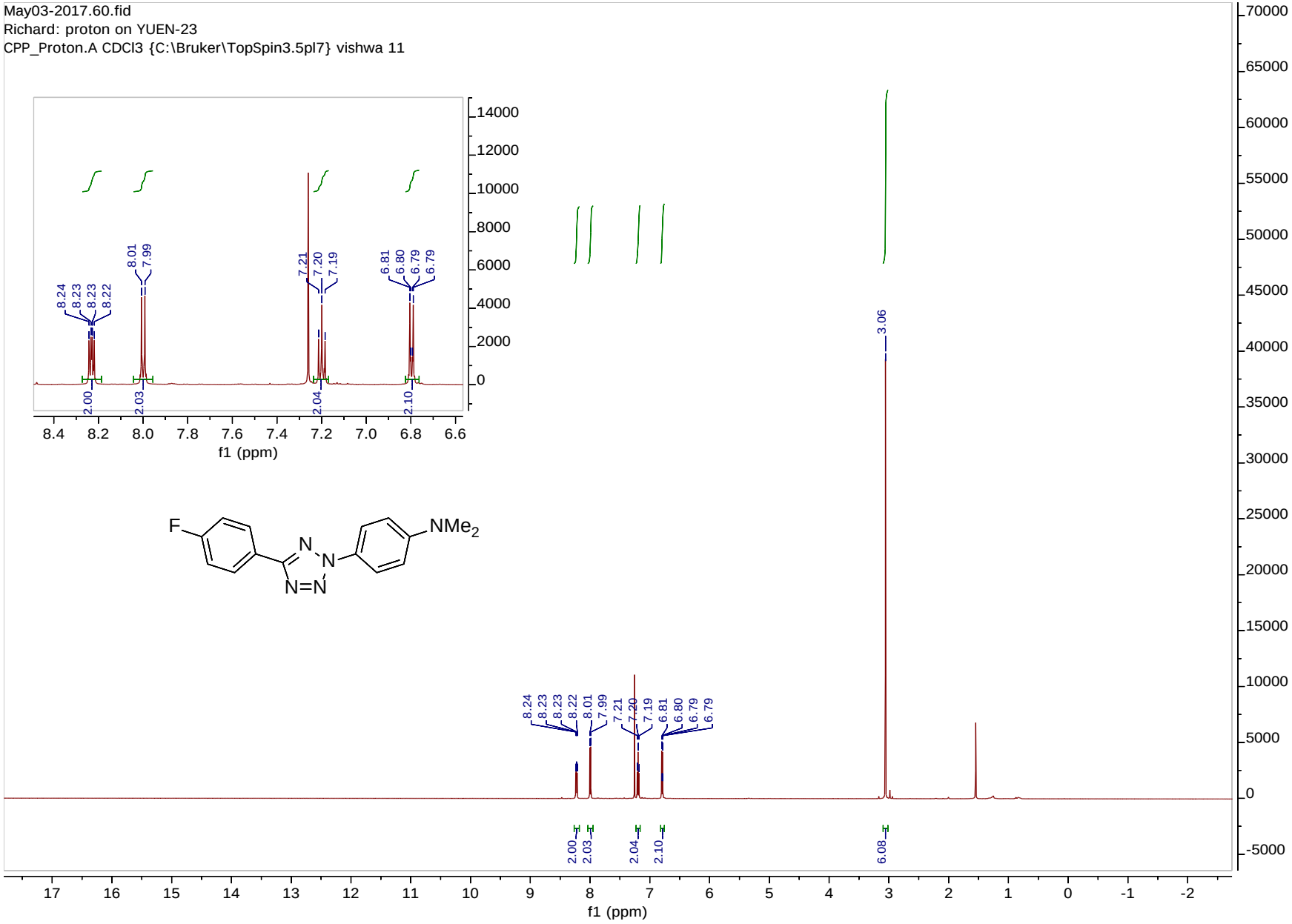

Nov28-2017.70.fid
Richard: Proton on YUEN-77
CPP_Proton.A CDCl3 {C:\Bruker\TopSpin3.5pl7} vishwa 18
f1 (ppm)
OMe
OAc
OAc
OAc
AcO
F
N
N
H
O
O

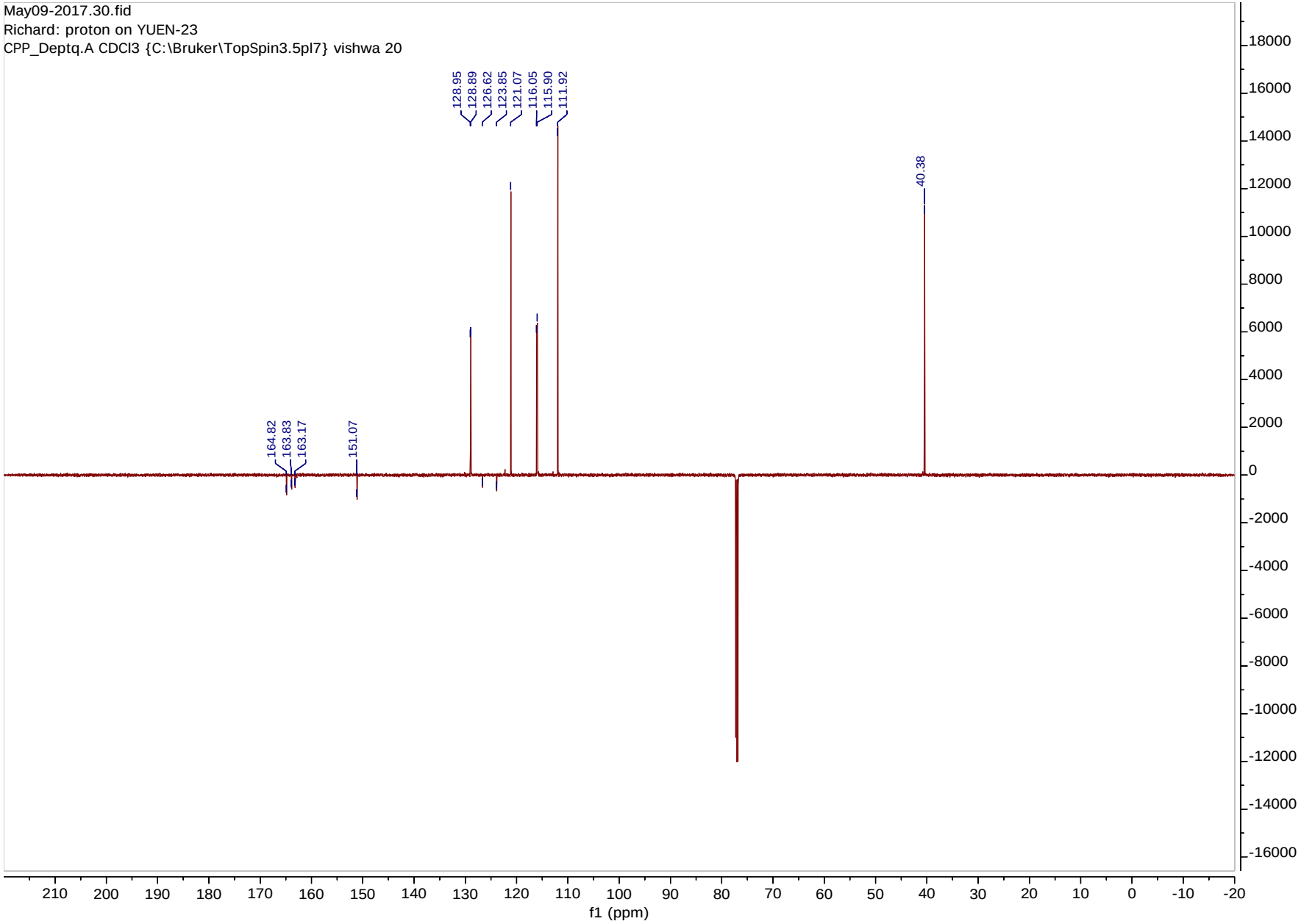

Nov28-2017.71.fid
Richard: Deptq on YUEN-77
CPP_Deptq.A CDCl3 {C:\Bruker\TopSpin3.5pl7} vishwa 18
171.20
170.61
169.35
169.20
163.75
162.12
159.83
159.04
150.42
137.34
132.74
128.28
128.26
127.55
127.50
126.83
115.77
115.63
114.09
105.47
92.50
73.01
72.34
67.52
61.56
55.58
53.29
20.84
20.69
20.63
20.56
f1 (ppm)

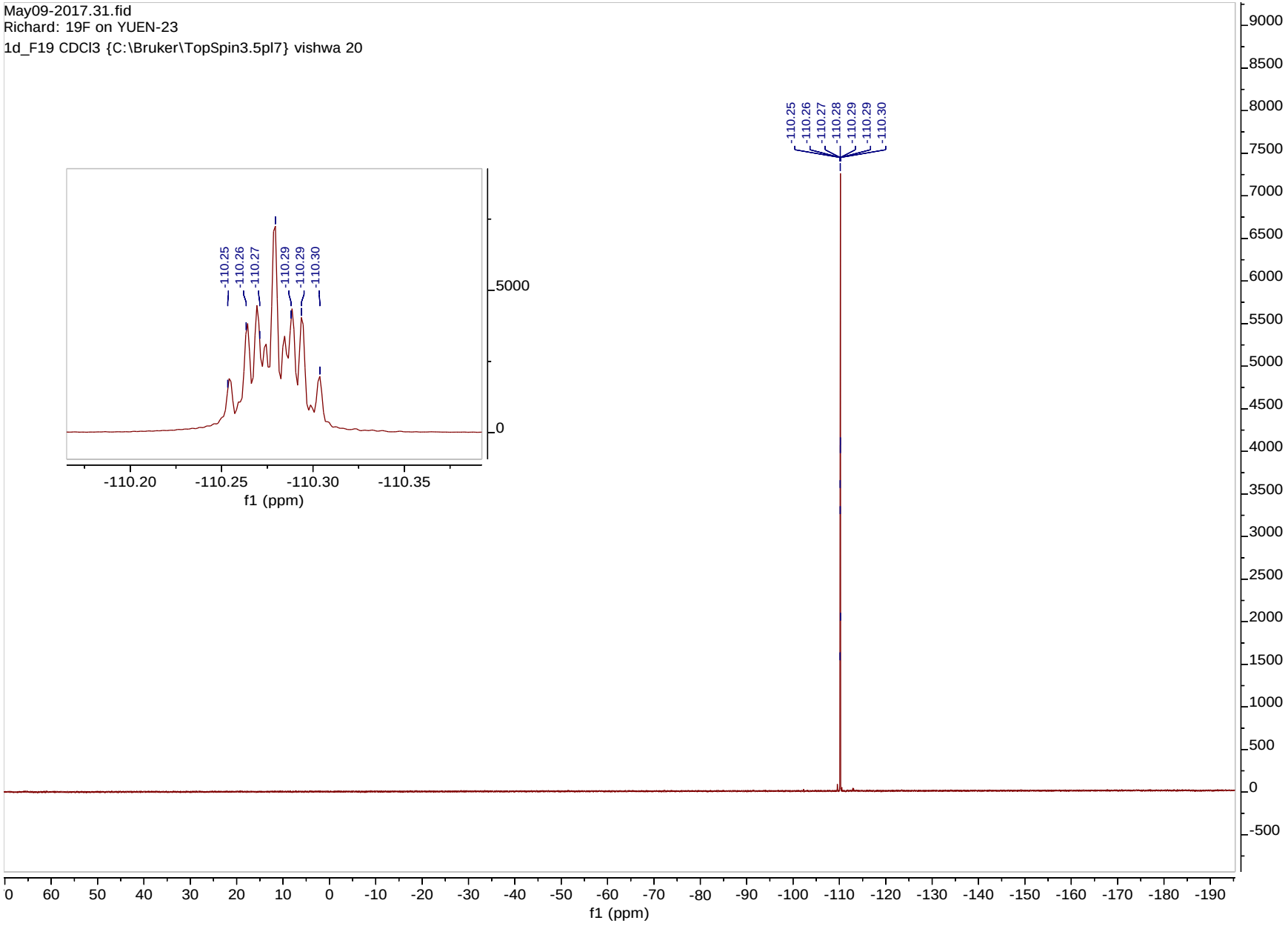

2020.02.07.i5_RY-1-31_H1_PRESAT
498.120 MHz H1 1D in cd3od (ref. to CD3OD @ 3.30 ppm)
temp 26.9 C -> actual temp = 27.0 C, autoxdb probe
f1 (ppm)
OMe
OH
OH
OH
F
N
H
O
HO

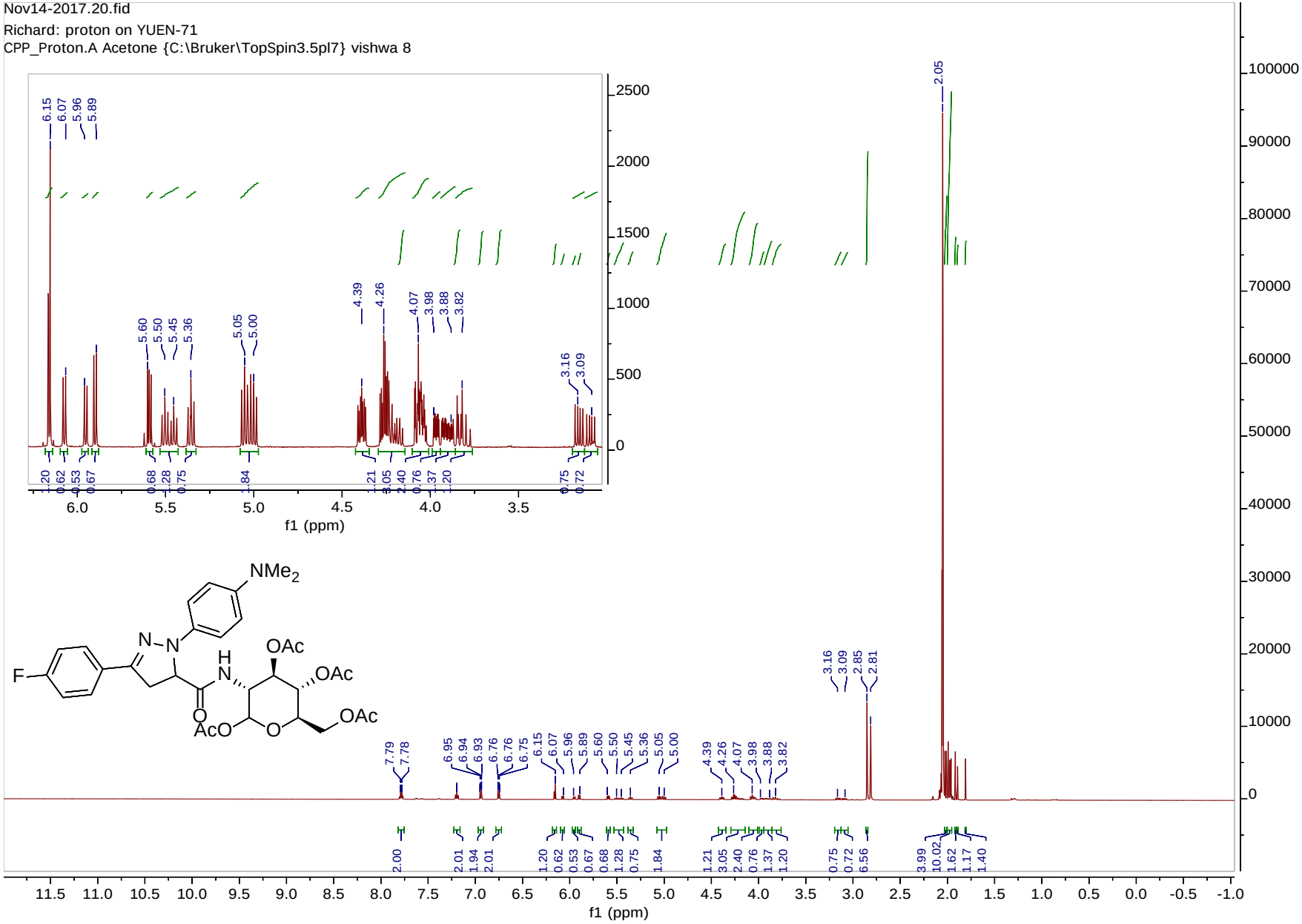

2020.02.06.u5_RY-1-31_loc5_20.03_C13_1D
Richard, RY-1-31
125.686 MHz C13{H1} 1D in cd3od (ref. to CD3OD @ 49.0 ppm)
temp 27.7 C -> actual temp = 27.0 C, colddual probe
140.03
139.96
134.46
134.40
130.17
128.80
128.77
128.74
128.70
127.30
127.01
116.66
116.49
115.08
114.96
165.30
163.34
163.03
162.53
161.13
160.91
151.68
151.64
106.80
106.44
96.94
92.40
78.18
73.17
72.55
72.46
72.32
62.90
62.80
58.99
56.29
56.05
f1 (ppm)

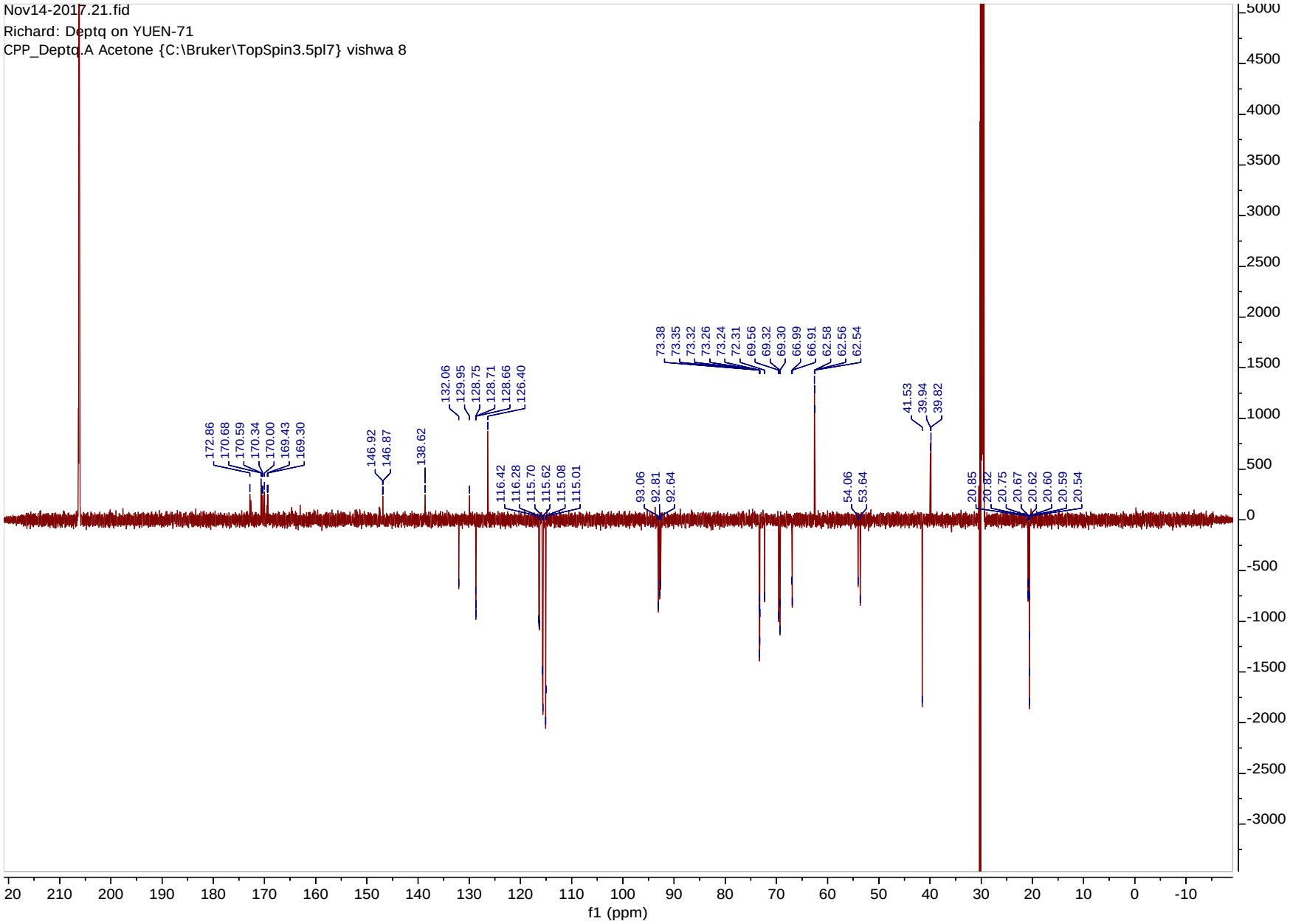

2020.02.06.i5_RY-1-31_F19_1D
468.640 MHz F19 1D in cd3od
temp 26.9 C -> actual temp = 27.0 C, autoxdb probe
-115.86
-115.89
-115.80
-115.85
-115.90
-115.95
f1 (ppm)
-77.04
-115.86
-115.88
-115.88
-115.89
f1 (ppm)

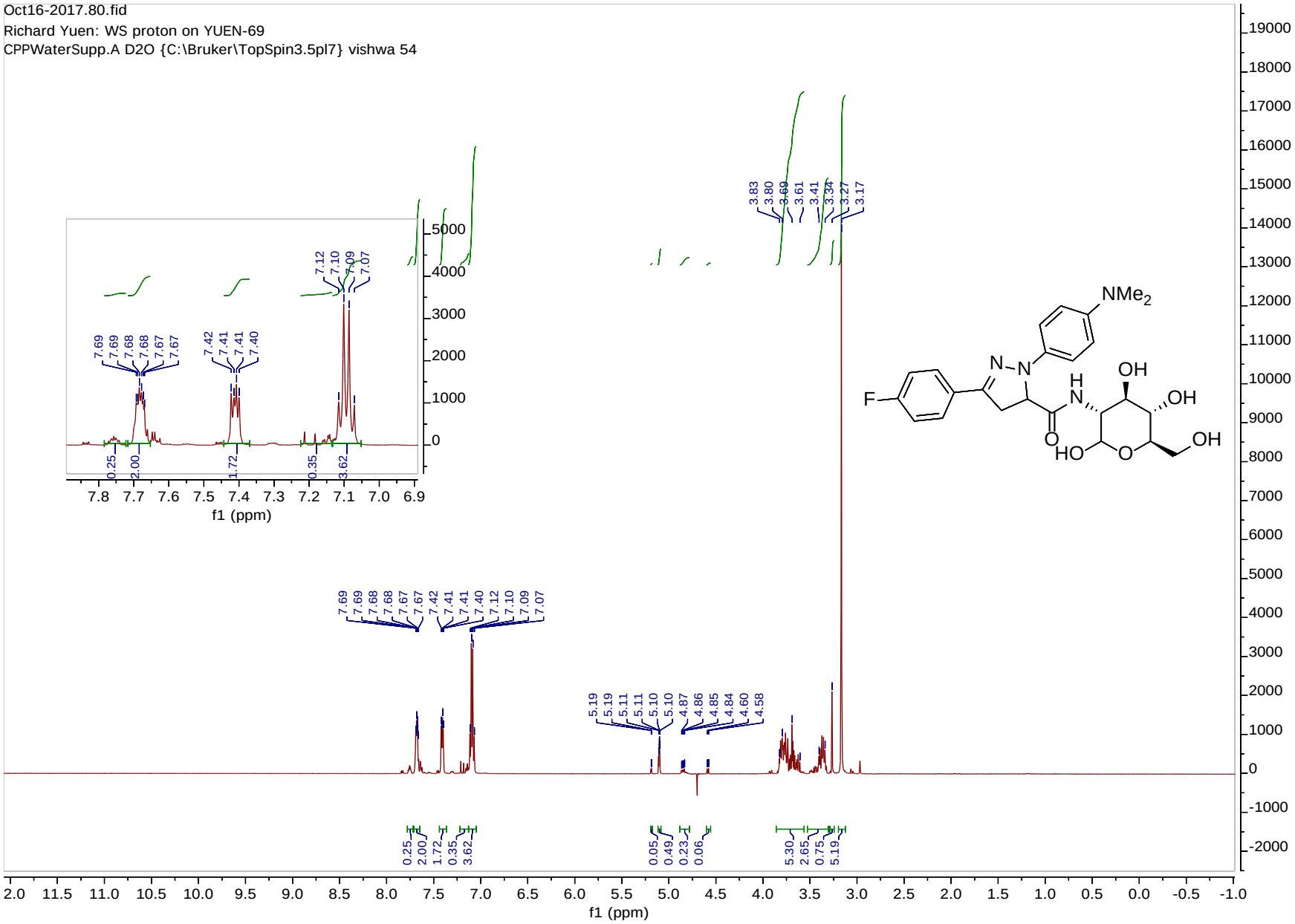

May03-2017.60.fid
Richard: proton on YUEN-23
CPP_Proton.A CDCl3 {C:\Bruker\TopSpin3.5pl7} vishwa 11
8.24
8.23
8.23
8.22
8.01
7.99
7.21
7.20
7.19
6.81
6.80
6.79
6.79
3.06
2.00
2.03
2.04
2.10
6.08
f1 (ppm)
F
N
N
N=N
NMe2

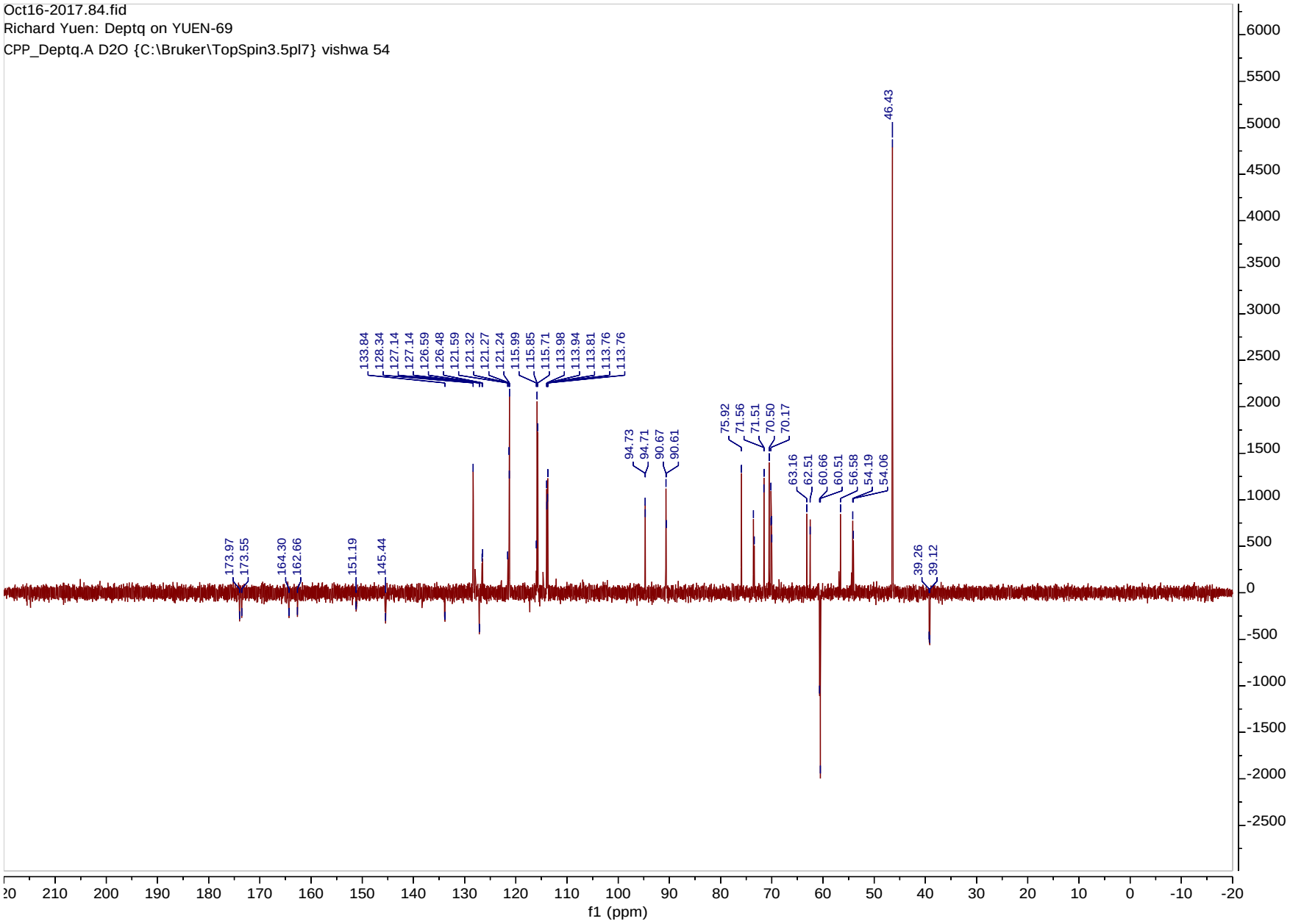

May09-2017.30.fid
Richard: proton on YUEN-23
CPP_Deptq.A CDCl3 {C:\Bruker\TopSpin3.5pl7} vishwa 20
128.95
128.89
126.62
123.85
121.07
116.05
115.90
111.92
40.38
164.82
163.83
163.17
151.07
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
-4000
-6000
-8000
-10000
-12000
-14000
-16000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)

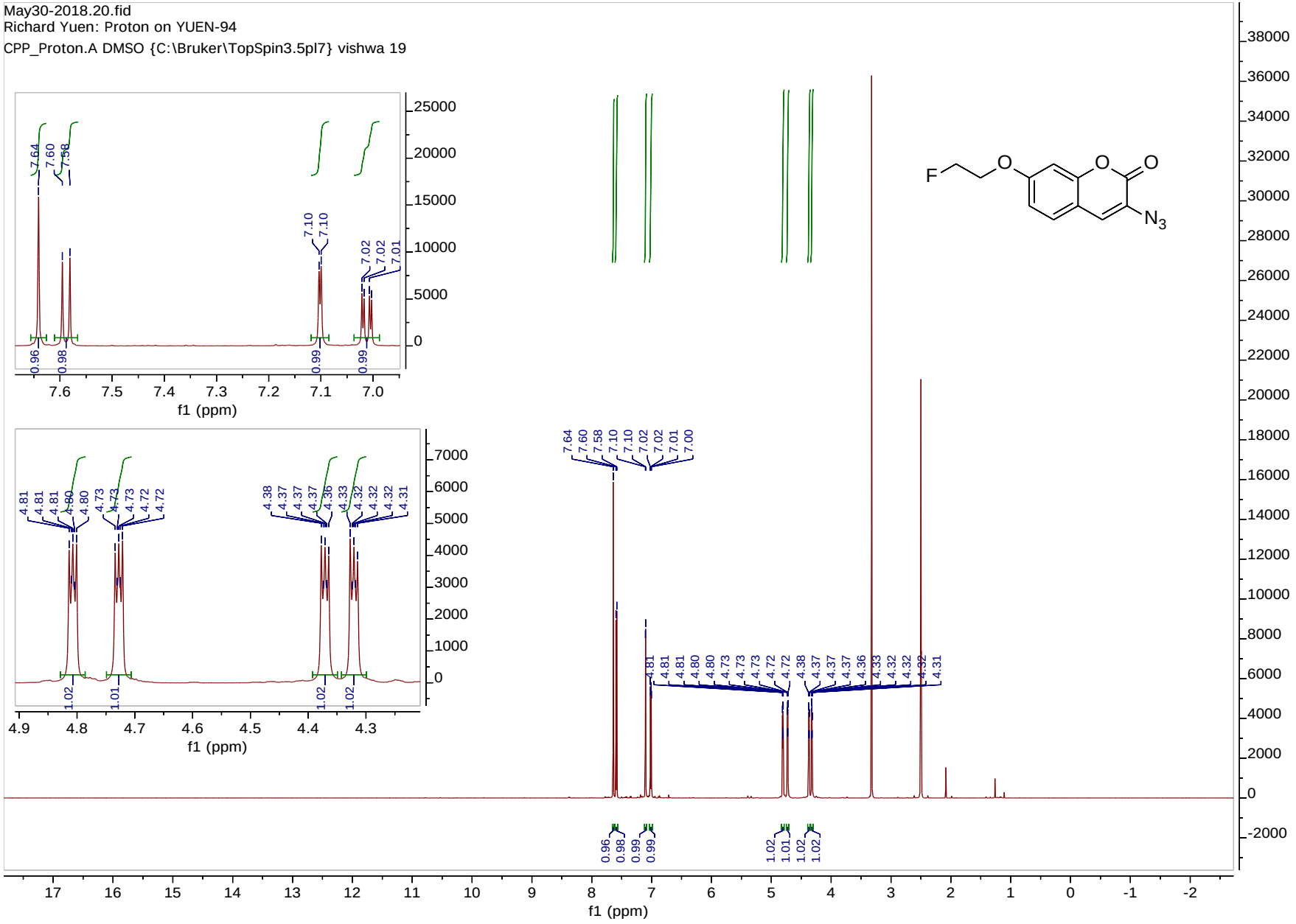

May09-2017.31.fid
Richard: 19F on YUEN-23
1d_F19 CDCl3 {C:\Bruker\TopSpin3.5pl7} vishwa 20
-110.25
-110.26
-110.27
-110.28
-110.29
-110.29
-110.30
5000
0
-110.20
-110.25
-110.30
-110.35
f1 (ppm)
9000
8500
8000
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

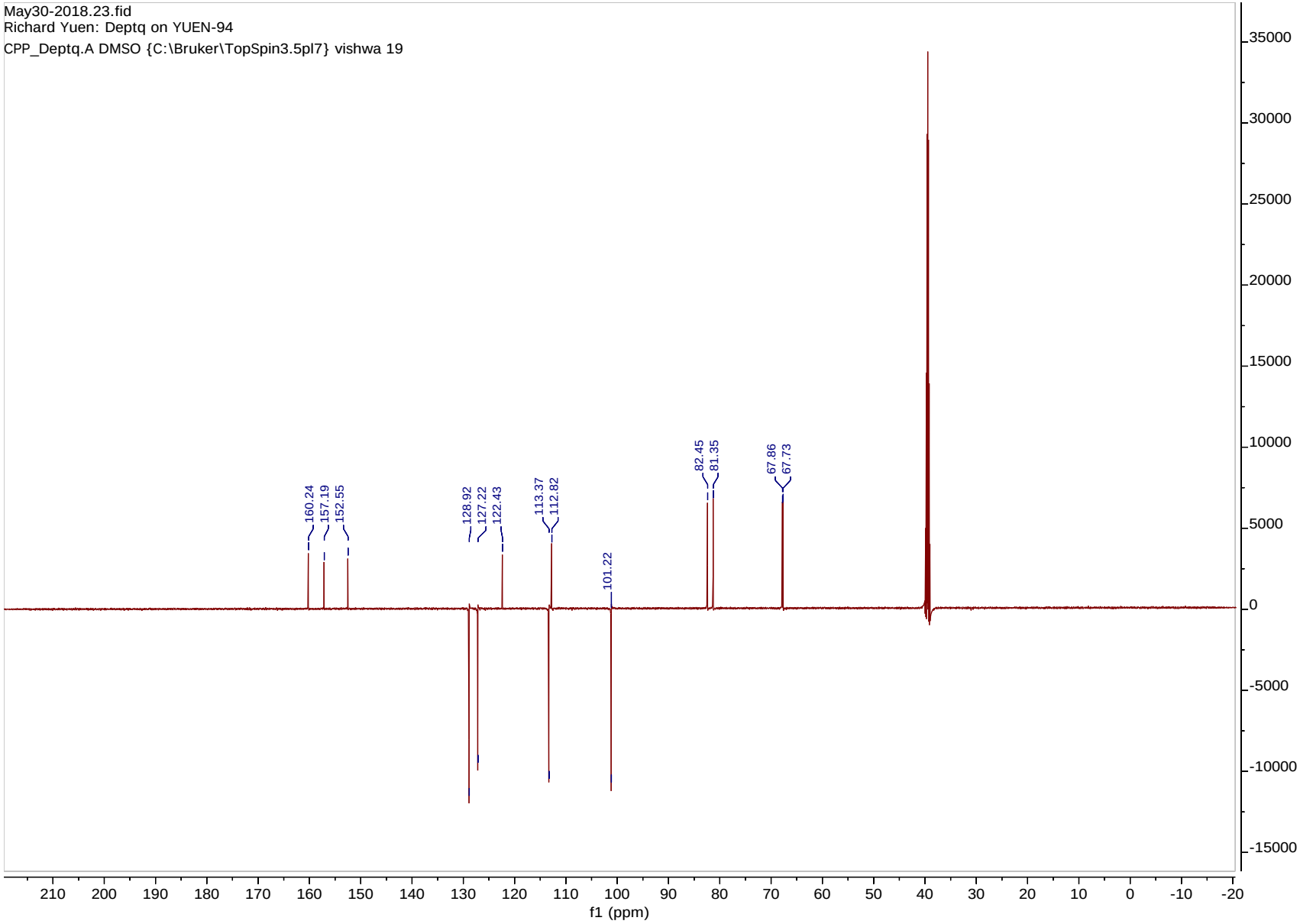

Nov14-2017.20.fid
Richard: proton on YUEN-71
CPP_Proton.A Acetone {C:\Bruker\TopSpin3.5pl7} vishwa 8
f1 (ppm)
NMe2
F
N
N
H
N
OAc
OAc
OAc
O
AcO
O


Nov14-2017.21.fid
Richard: Deptq on YUEN-71
CPP_Deptq.A Acetone {C:\Bruker\TopSpin3.5pl7} vishwa 8
172.86
170.68
170.59
170.34
170.00
169.43
169.30
146.92
146.87
138.62
132.06
129.95
128.75
128.71
128.66
126.40
116.42
116.28
115.70
115.62
115.08
115.01
93.06
92.81
92.64
73.38
73.35
73.32
73.26
73.24
72.31
69.56
69.32
69.30
66.99
66.91
62.58
62.56
62.54
54.06
53.64
41.53
39.94
39.82
20.85
20.82
20.75
20.67
20.62
20.60
20.59
20.54
f1 (ppm)


Oct16-2017.80.fid
Richard Yuen: WS proton on YUEN-69
CPPWaterSupp.A D2O {C:\Bruker\TopSpin3.5pl7} vishwa 54
f1 (ppm)
NMe2
F
N
N
H
N
OH
OH
OH
HO
O
O


Oct16-2017.84.fid
Richard Yuen: Deptq on YUEN-69
CPP_Deptq.A D2O {C:\Bruker\TopSpin3.5pl7} vishwa 54
46.43
133.84
128.34
127.14
127.14
126.59
126.48
121.59
121.32
121.27
121.24
115.99
115.85
115.71
113.98
113.94
113.81
113.76
113.76
94.73
94.71
90.67
90.61
75.92
71.56
71.51
70.50
70.17
63.16
62.51
60.66
60.51
56.58
54.19
54.06
173.97
173.55
164.30
162.66
151.19
145.44
39.26
39.12
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
-1000
-1500
-2000
-2500
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)


May30-2018.20.fid
Richard Yuen: Proton on YUEN-94
CPP_Proton.A DMSO {C:\Bruker\TopSpin3.5pl7} vishwa 19
f1 (ppm)
F
O
O
O
N3


May30-2018.23.fid
Richard Yuen: Deptq on YUEN-94
CPP_Deptq.A DMSO {C:\Bruker\TopSpin3.5pl7} vishwa 19
160.24
157.19
152.55
128.92
127.22
122.43
113.37
112.82
101.22
82.45
81.35
67.86
67.73
35000
30000
25000
20000
15000
10000
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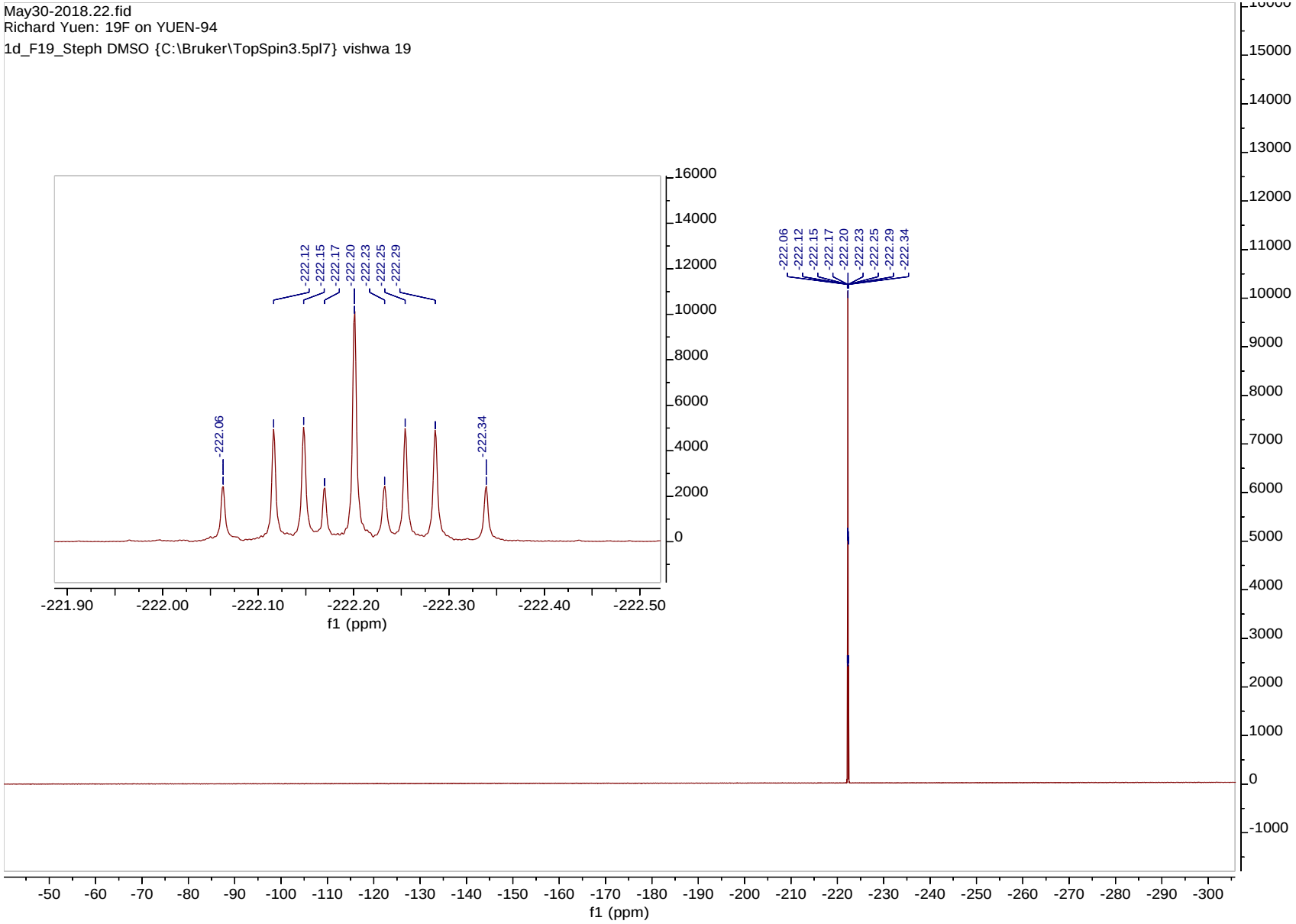
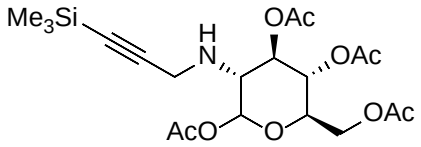
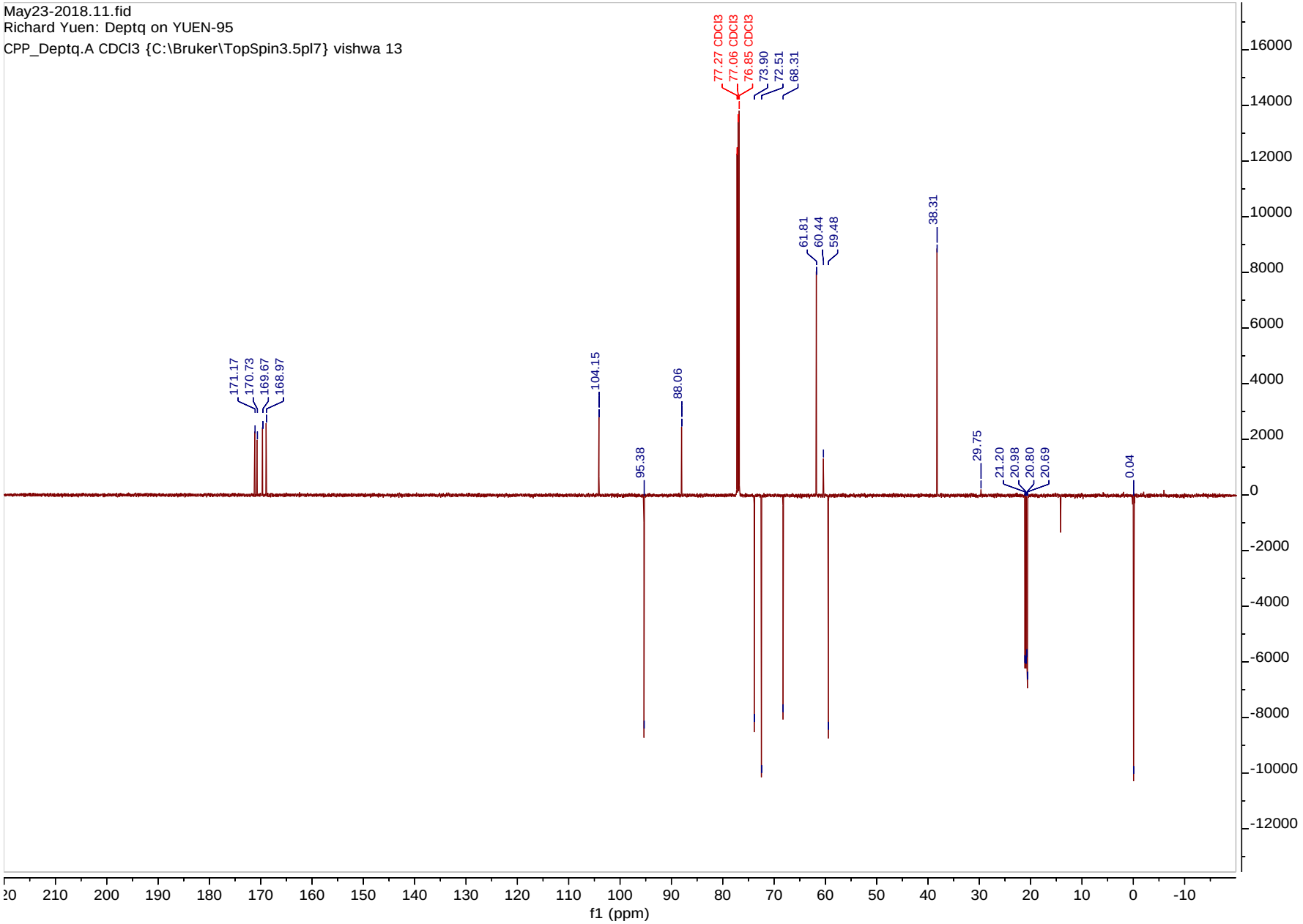
0
-5000
-10000
-15000
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

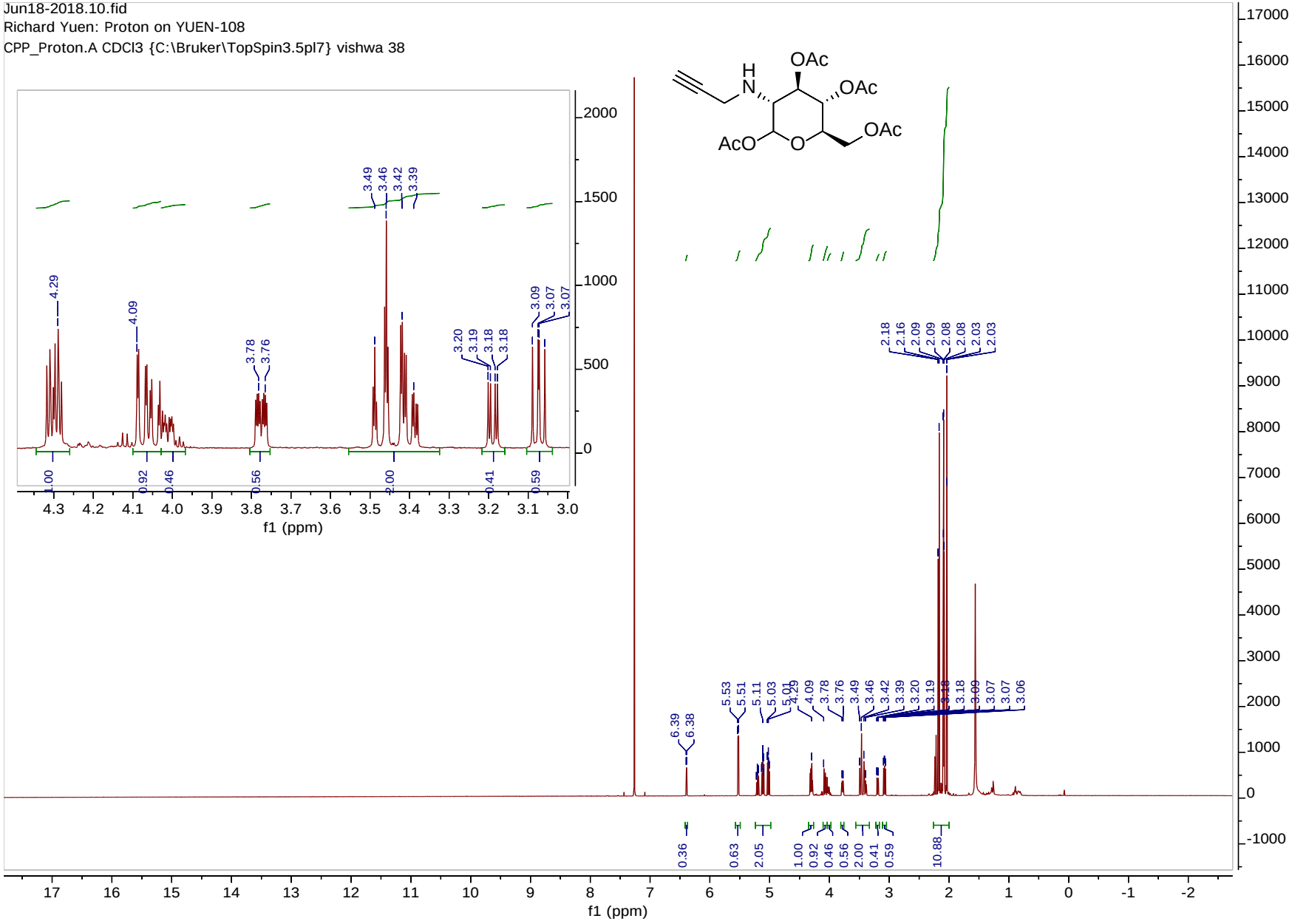

May30-2018.22.fid
Richard Yuen: 19F on YUEN-94
1d_F19_Steph DMSO {C:\Bruker\TopSpin3.5pl7} vishwa 19
-222.06
-222.12
-222.15
-222.17
-222.20
-222.23
-222.25
-222.29
-222.34
f1 (ppm)

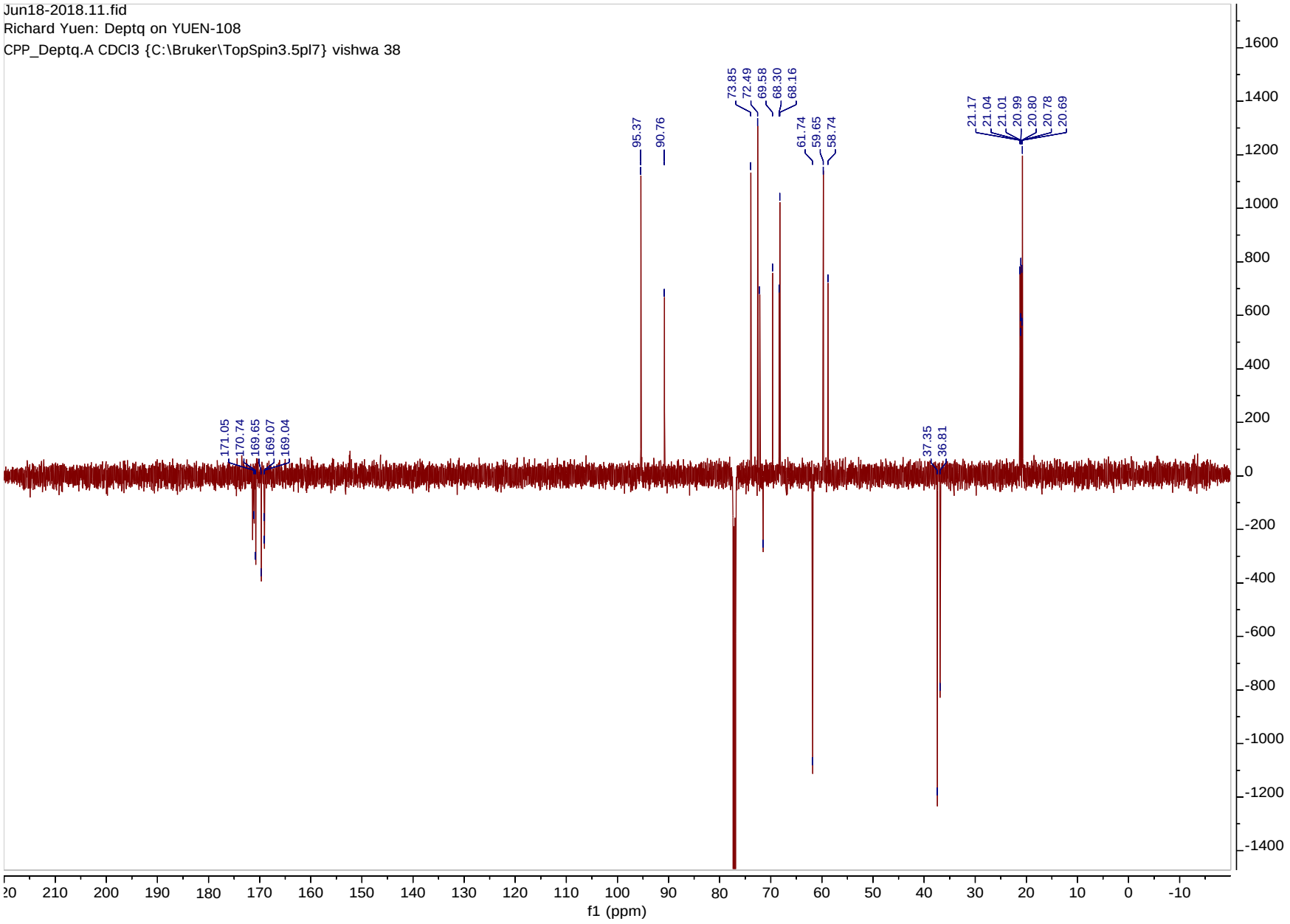


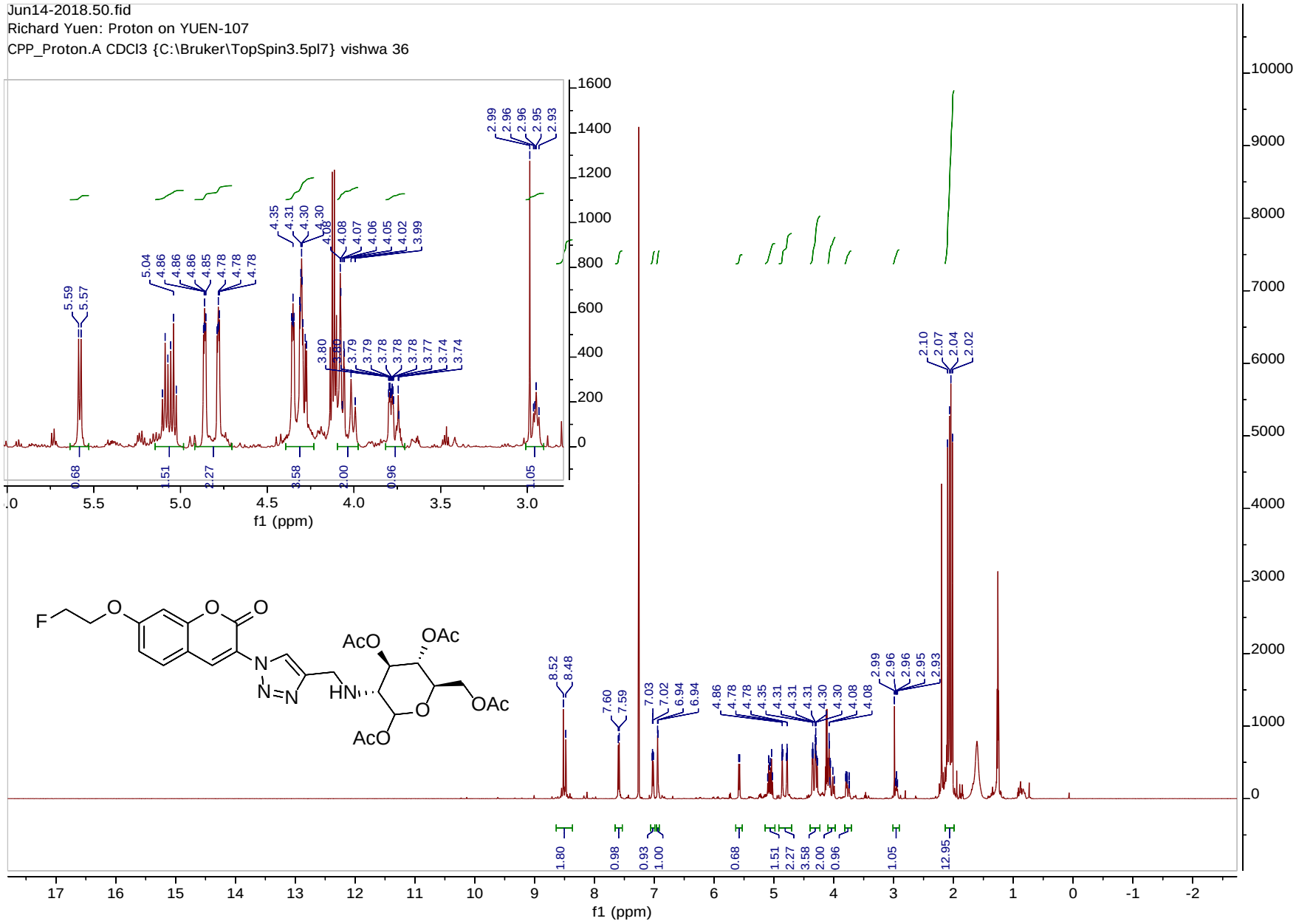
May23-2018.10.fid
Richard Yuen: Proton on YUEN-95
CPP_Proton.A CDCl3 {C:\Bruker\TopSpin3.5pl7} vishwa 13

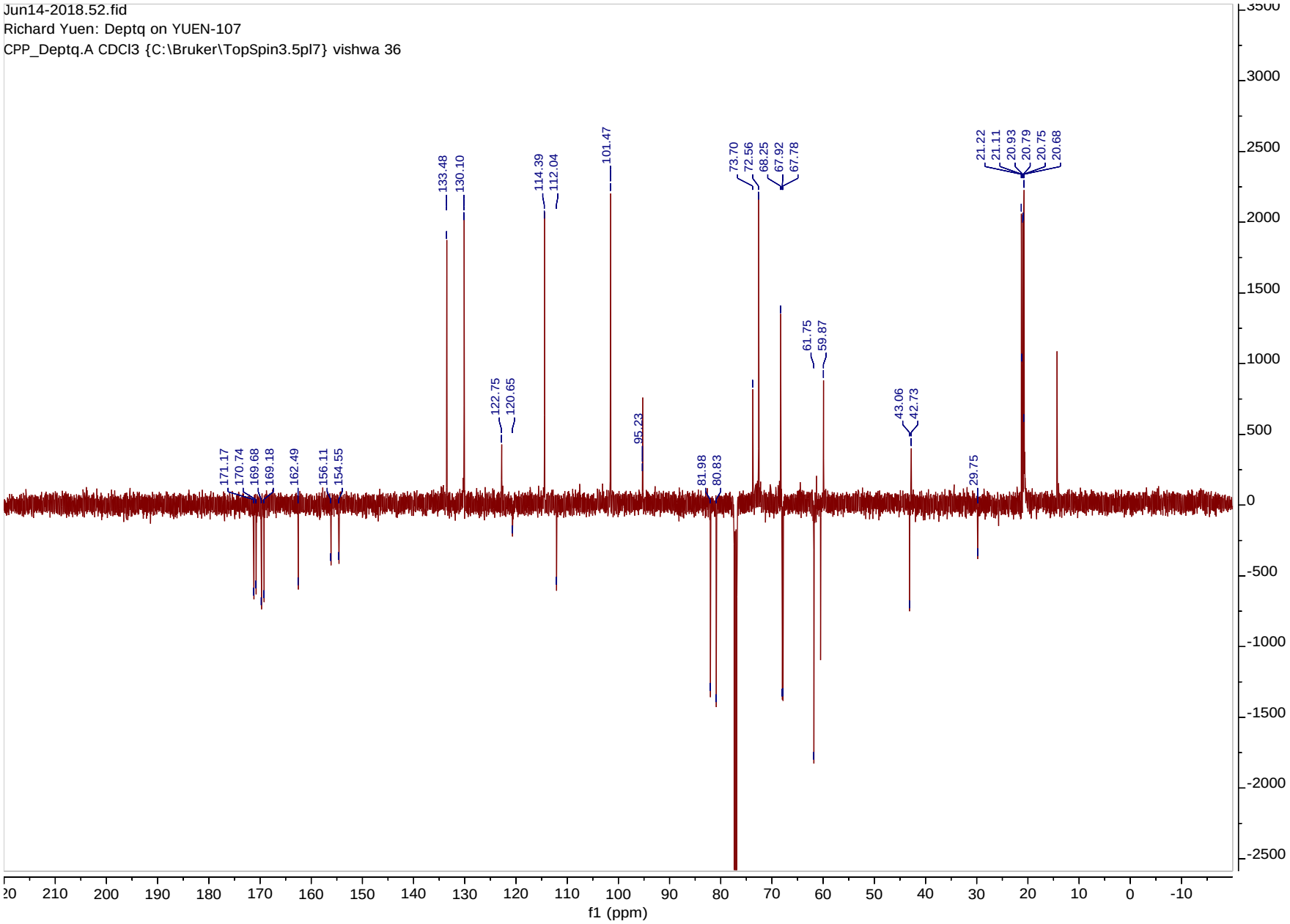
1,3,4,6-tetra-O-acetyl-N-(3-trimethylsilyl)propargyl-β-D-glucosamine

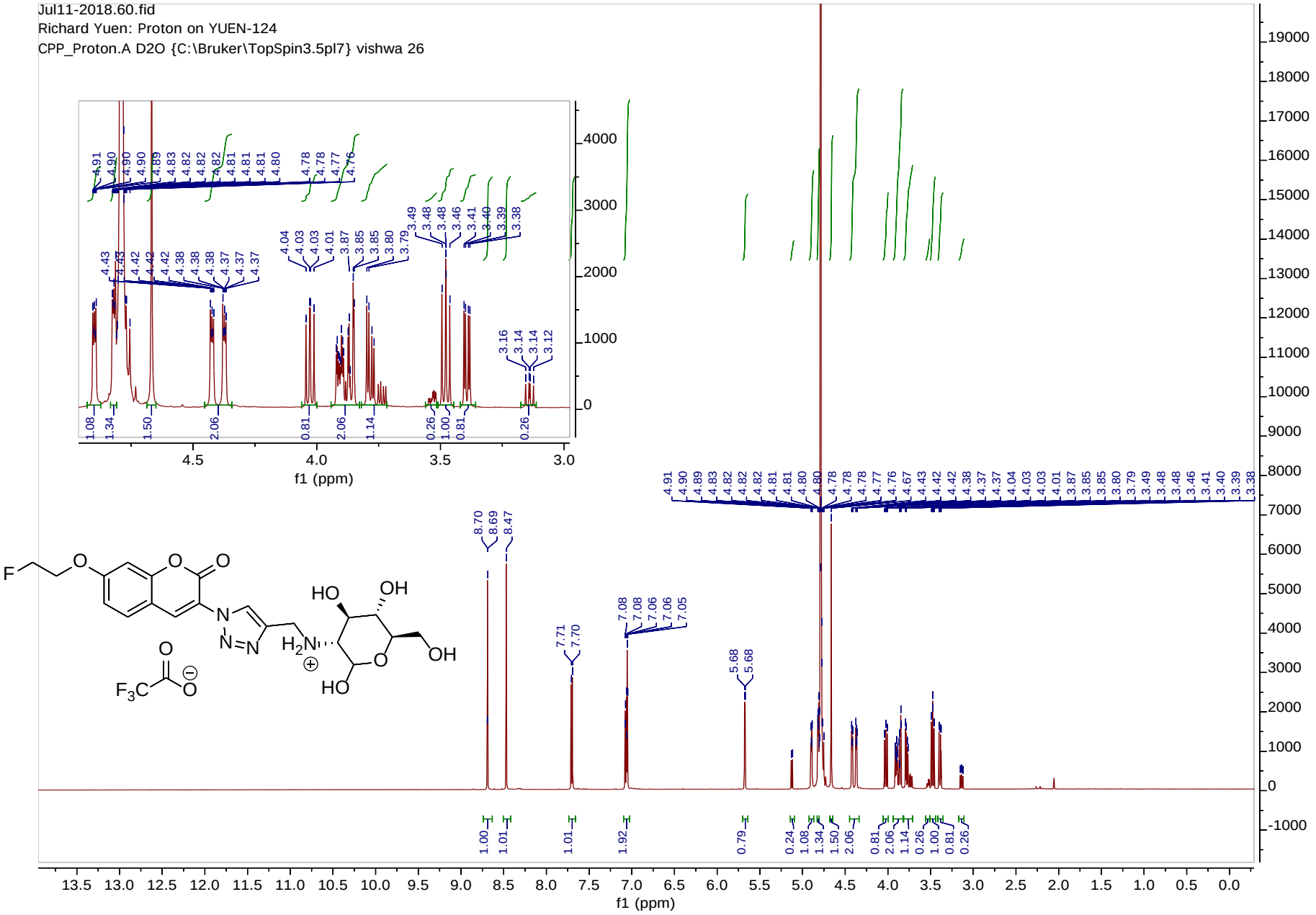

May23-2018.11.fid
Richard Yuen: Deptq on YUEN-95
CPP_Deptq.A CDCl3 {C:\Bruker\TopSpin3.5pl7} vishwa 13
77.27 CDCl3
77.06 CDCl3
76.85 CDCl3
73.90
72.51
68.31
171.17
170.73
169.67
168.97
104.15
95.38
88.06
61.81
60.44
59.48
38.31
29.75
21.20
20.98
20.80
20.69
0.04
f1 (ppm)

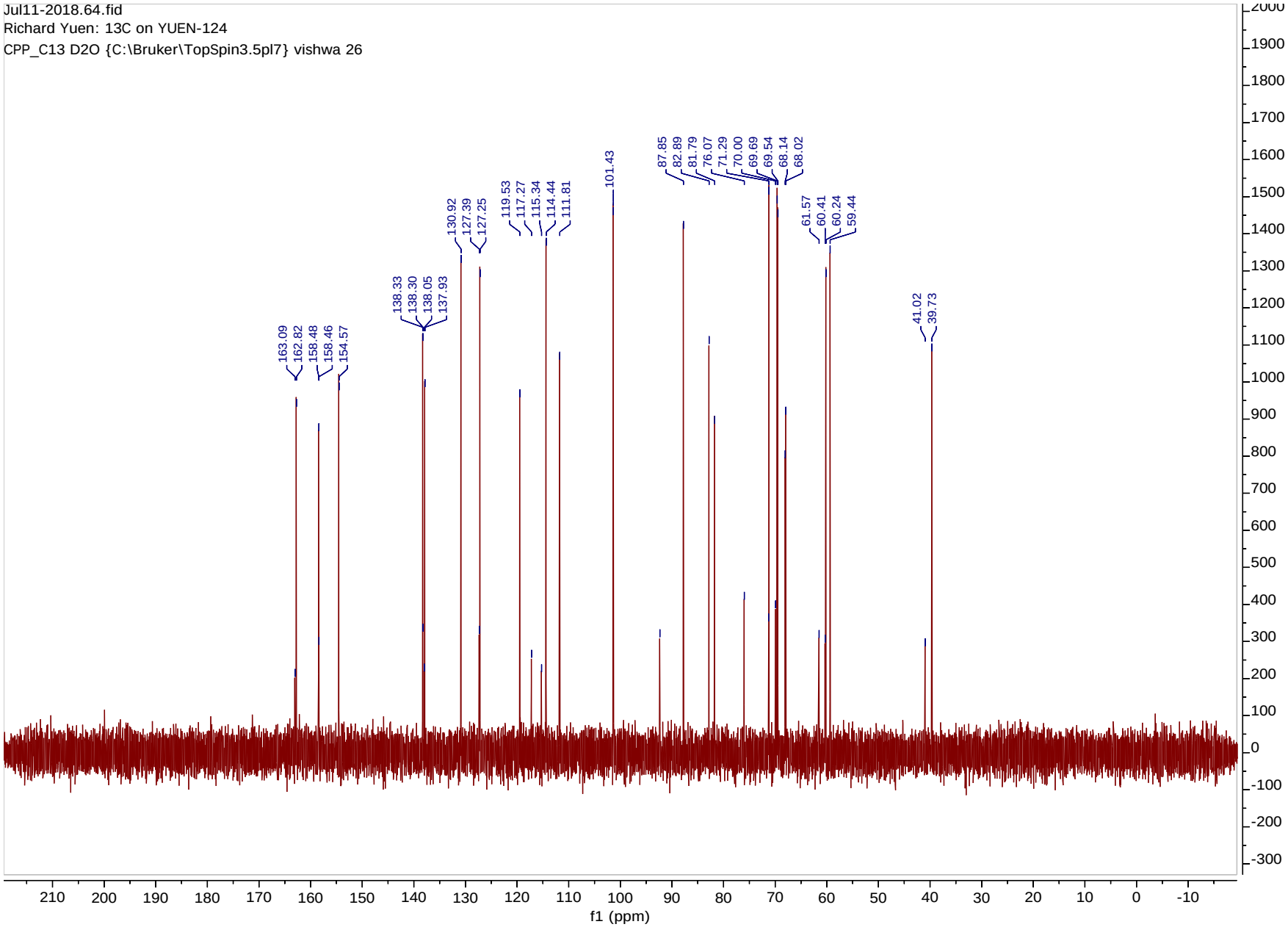

Jun18-2018.10.fid
Richard Yuen: Proton on YUEN-108
CPP_Proton.A CDCl3 {C:\Bruker\TopSpin3.5pl7} vishwa 38
OAc
OAc
OAc
AcO
O
H
N
f1 (ppm)
f1 (ppm)

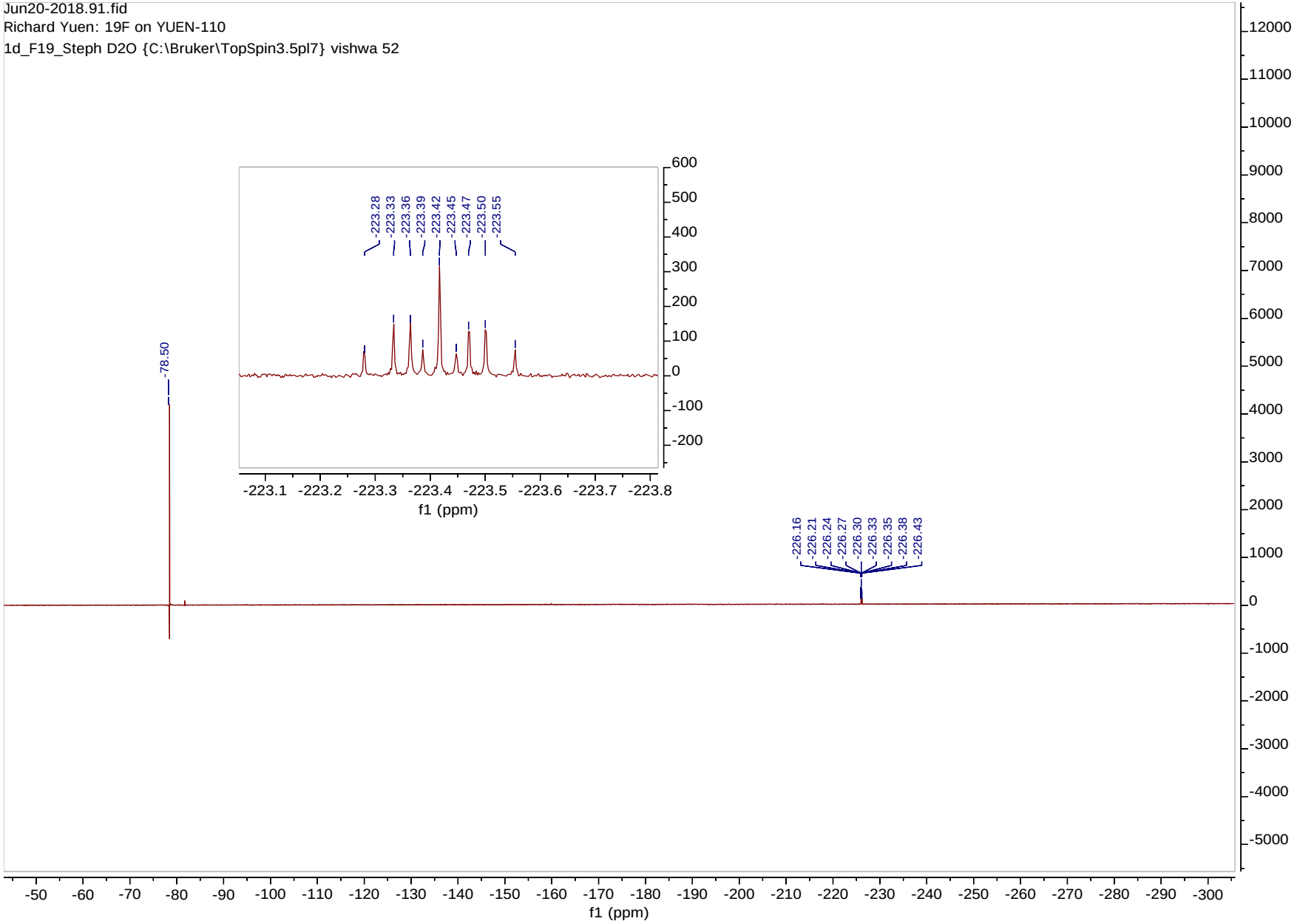

Jun18-2018.11.fid
Richard Yuen: Deptq on YUEN-108
CPP_Deptq.A CDCl3 {C:\Bruker\TopSpin3.5pl7} vishwa 38
171.05
170.74
169.65
169.07
169.04
95.37
90.76
73.85
72.49
69.58
68.30
68.16
61.74
59.65
58.74
37.35
36.81
21.17
21.04
21.01
20.99
20.80
20.78
20.69
f1 (ppm)

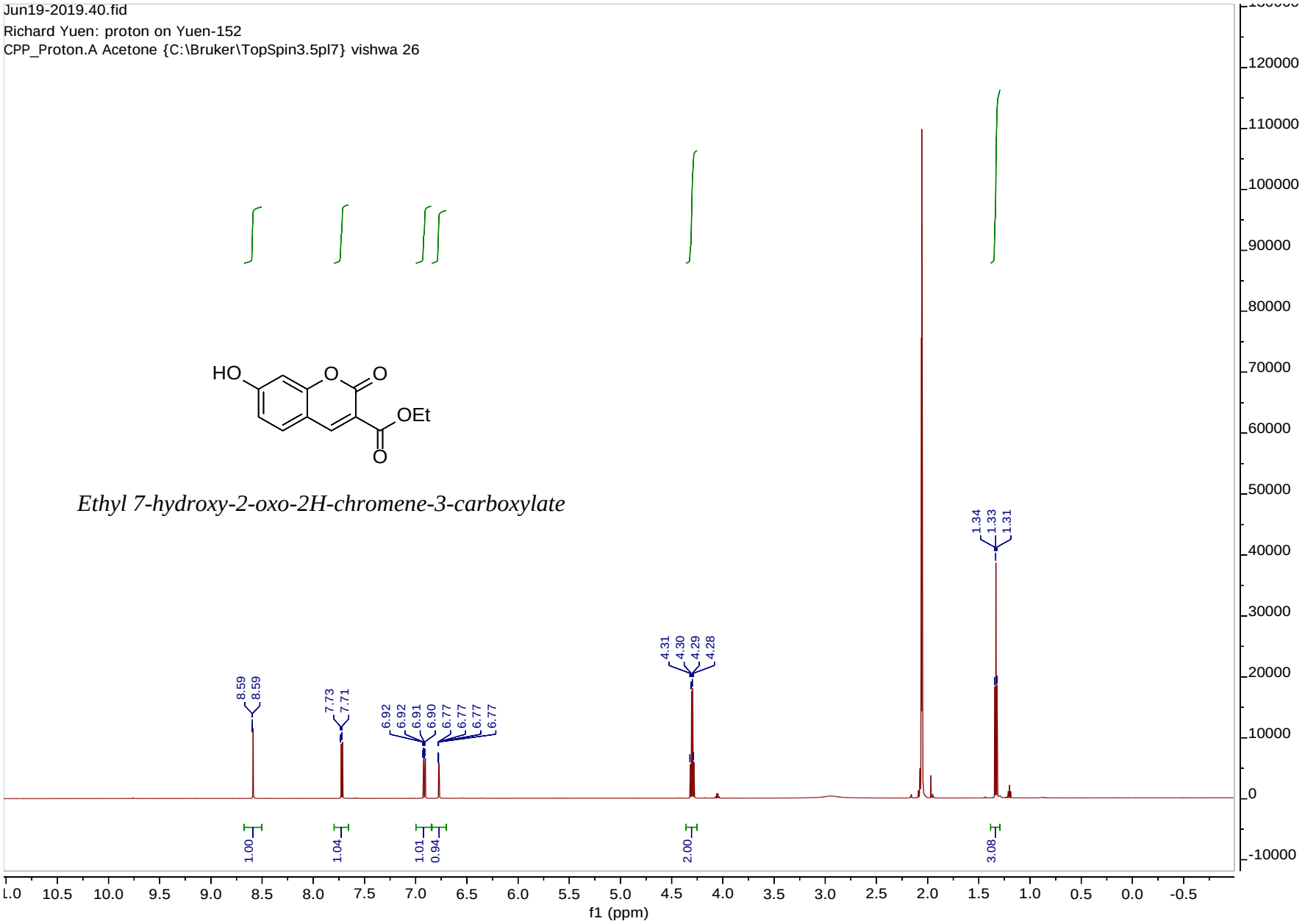

Jun14-2018.50.fid
Richard Yuen: Proton on YUEN-107
CPP_Proton.A CDCl3 {C:\Bruker\TopSpin3.5pl7} vishwa 36
f1 (ppm)
f1 (ppm)
F
O
O
O
N
N=N
HN
AcO
OAc
AcO
OAc


Jun14-2018.52.fid
Richard Yuen: Deptq on YUEN-107
CPP_Deptq.A CDCl3 {C:\Bruker\TopSpin3.5pl7} vishwa 36
171.17
170.74
169.68
169.18
162.49
156.11
154.55
133.48
130.10
122.75
120.65
114.39
112.04
101.47
95.23
81.98
80.83
73.70
72.56
68.25
67.92
67.78
61.75
59.87
43.06
42.73
29.75
21.22
21.11
20.93
20.79
20.75
20.68
f1 (ppm)

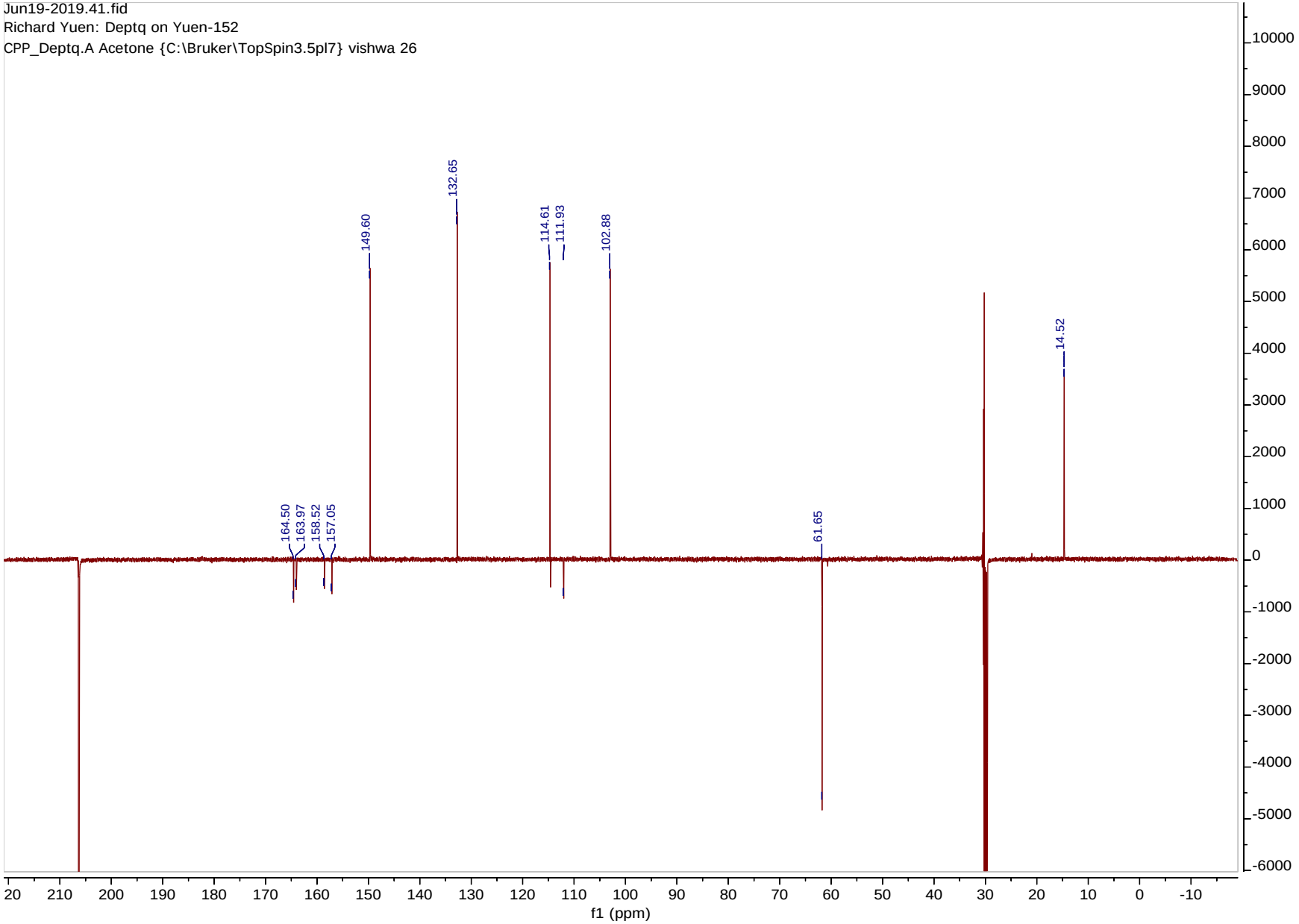

Jul11-2018.60.fid
Richard Yuen: Proton on YUEN-124
CPP_Proton.A D2O {C:\Bruker\TopSpin3.5pl7} vishwa 26
f1 (ppm)
f1 (ppm)

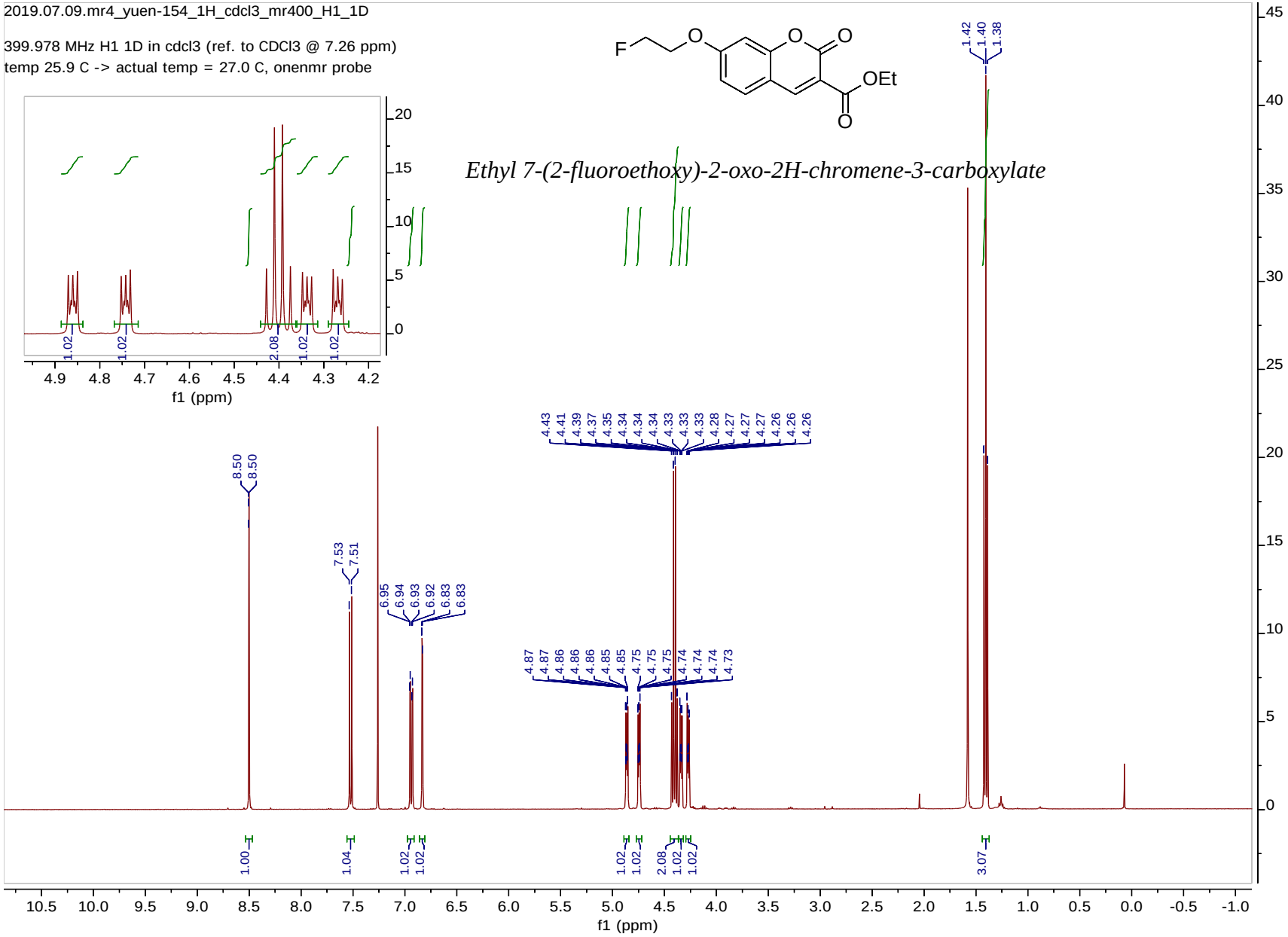

Jul11-2018.64.fid
Richard Yuen: 13C on YUEN-124
CPP_C13 D2O {C:\Bruker\TopSpin3.5pl7} vishwa 26
163.09
162.82
158.48
158.46
154.57
138.33
138.30
138.05
137.93
130.92
127.39
127.25
119.53
117.27
115.34
114.44
111.81
101.43
87.85
82.89
81.79
76.07
71.29
70.00
69.69
69.54
68.14
68.02
61.57
60.41
60.24
59.44
41.02
39.73
f1 (ppm)

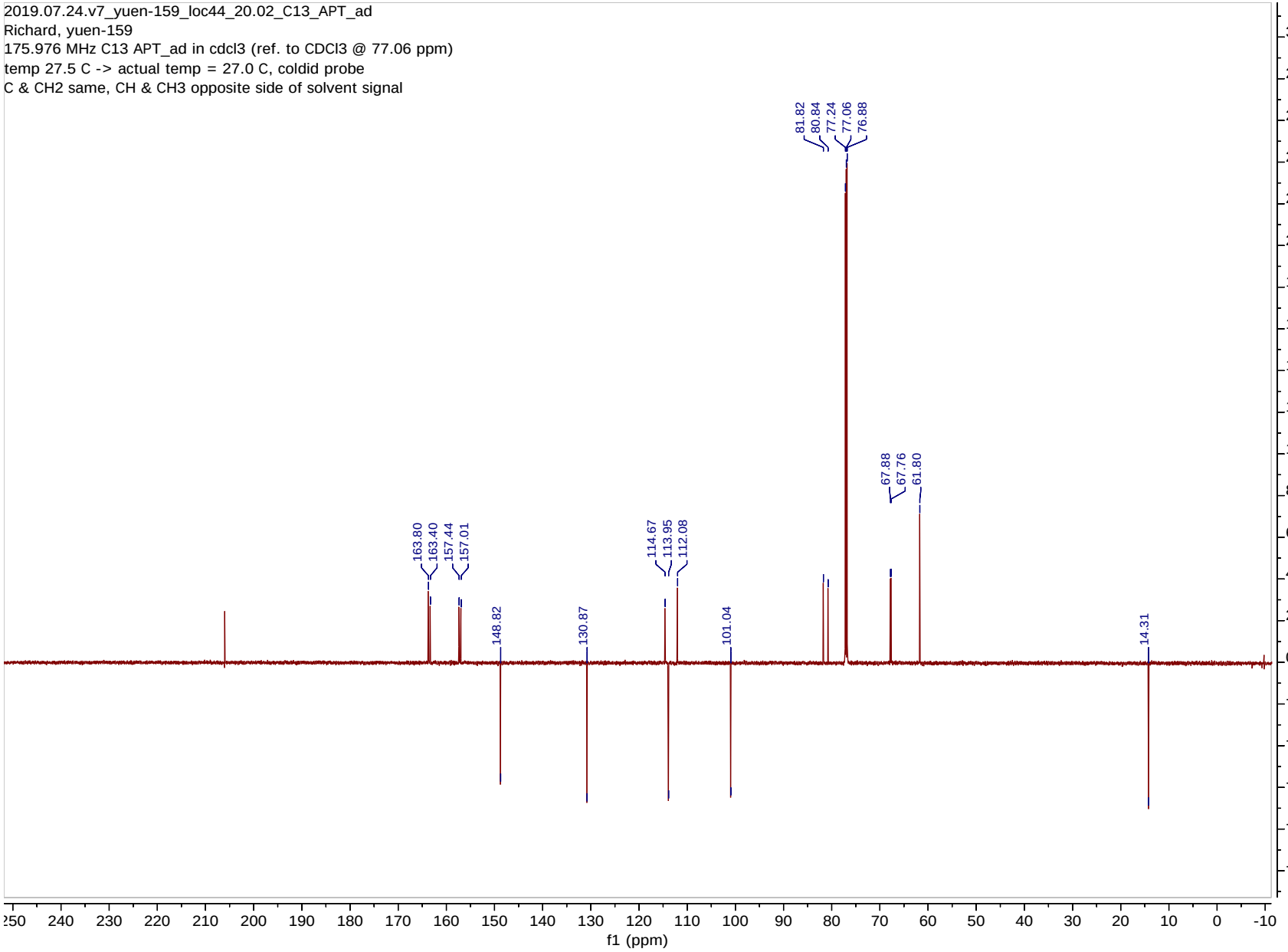

Jun20-2018.91.fid
Richard Yuen: 19F on YUEN-110
1d_F19_Steph D2O {C:\Bruker\TopSpin3.5pl7} vishwa 52
-78.50
-223.28
-223.33
-223.36
-223.39
-223.42
-223.45
-223.47
-223.50
-223.55
-226.16
-226.21
-226.24
-226.27
-226.30
-226.33
-226.35
-226.38
-226.43
f1 (ppm)
f1 (ppm)

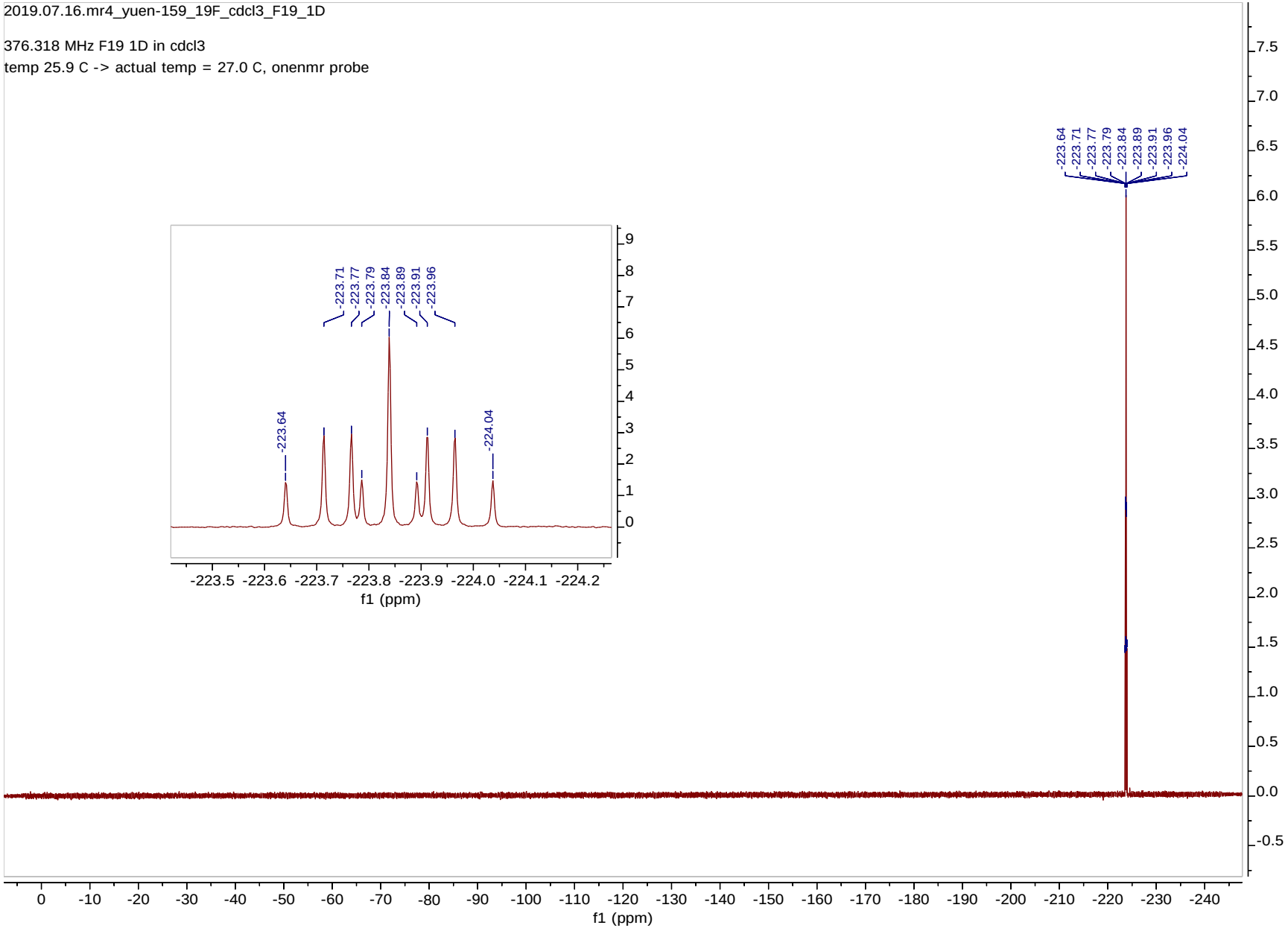


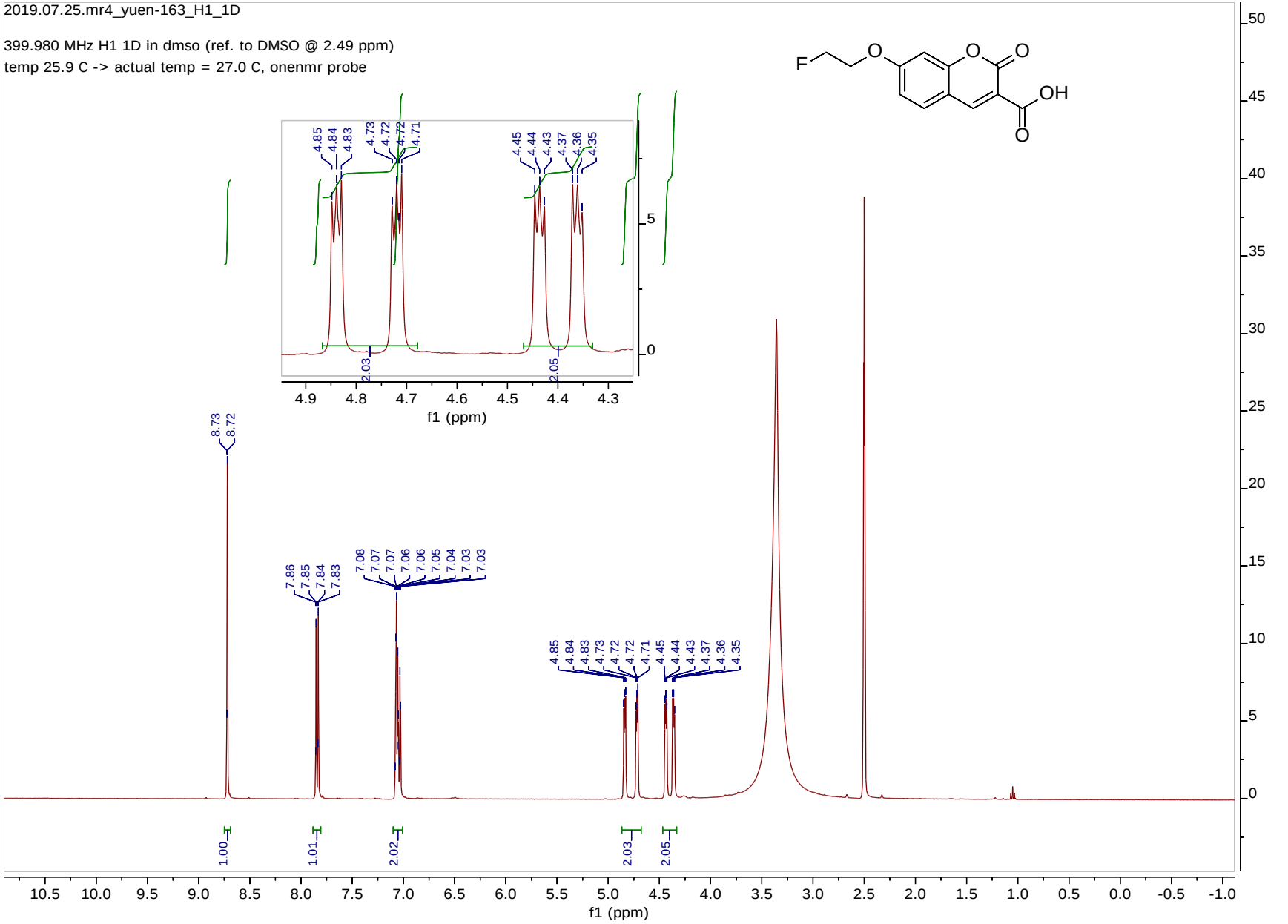
Ethyl 7-hydroxy-2-oxo-2H-chromene-3-carboxylate

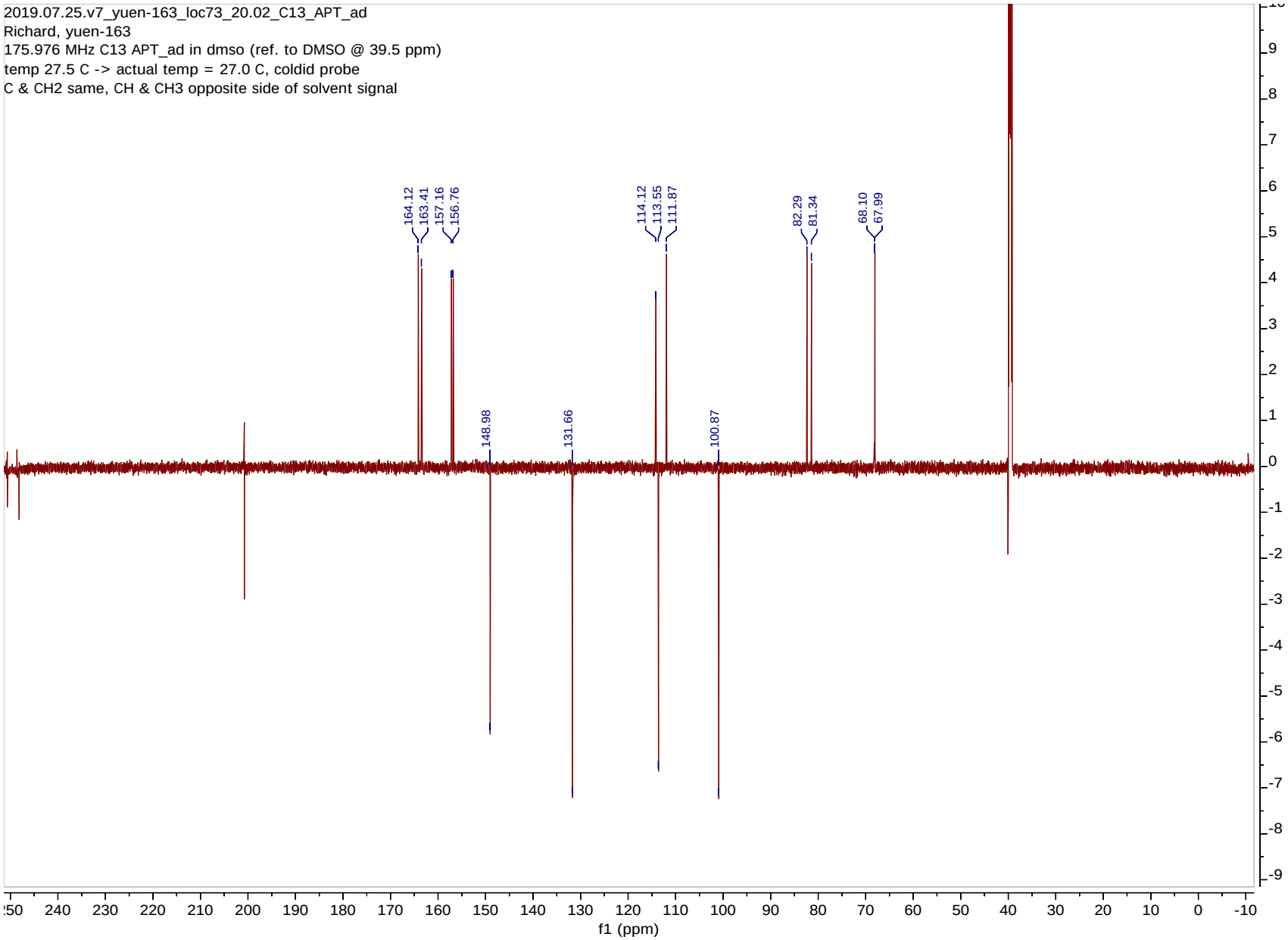

Jun19-2019.41.fid
Richard Yuen: Deptq on Yuen-152
CPP_Deptq.A Acetone {C:\Bruker\TopSpin3.5pl7} vishwa 26
164.50
163.97
158.52
157.05
149.60
132.65
114.61
111.93
102.88
61.65
14.52
f1 (ppm)

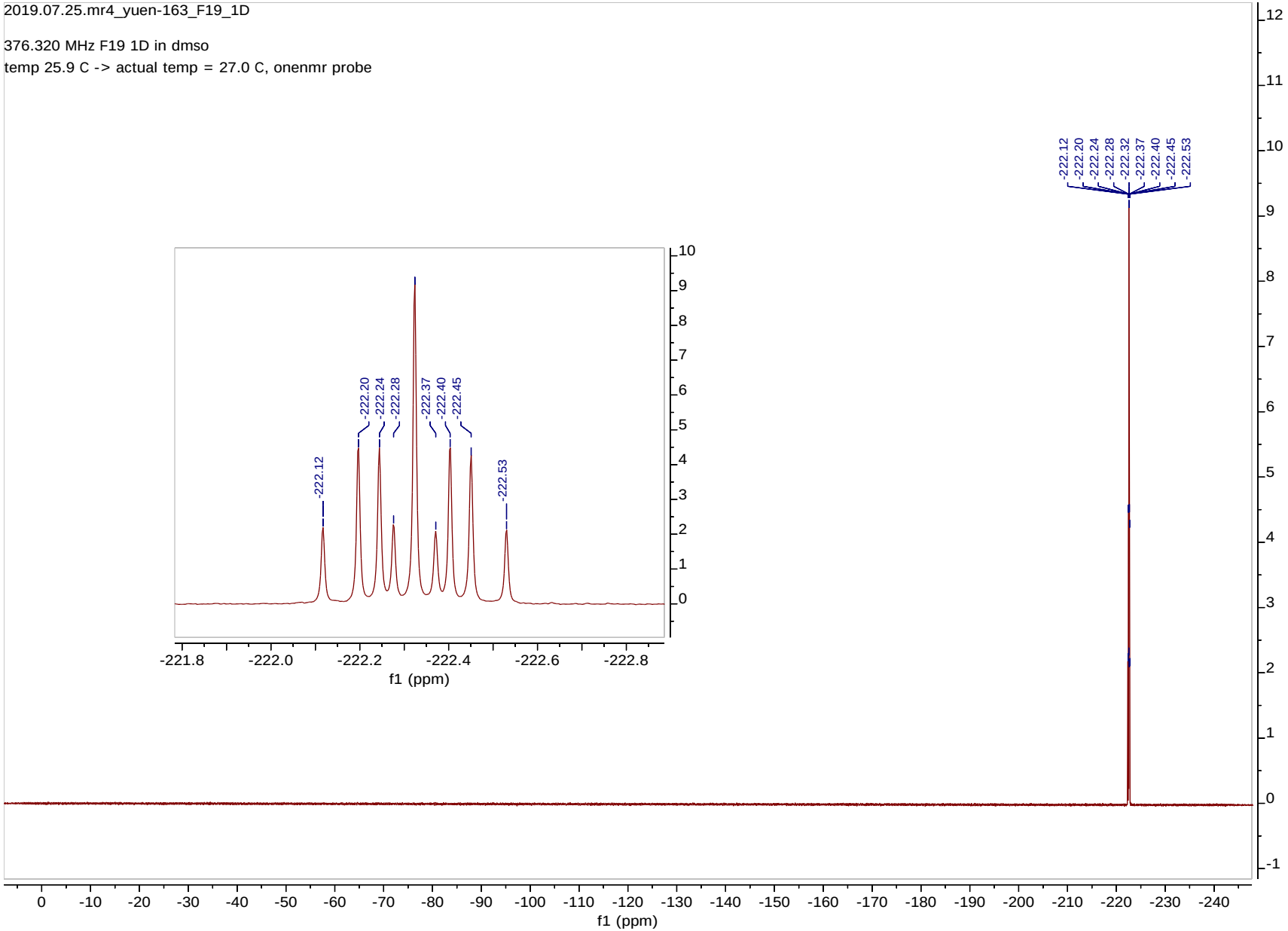

2019.07.09.mr4_yuen-154_1H_cdcl3_mr400_H1_1D
399.978 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 25.9 C -> actual temp = 27.0 C, onenmr probe
F
O
O
O
O
OEt
O
Ethyl 7-(2-fluoroethoxy)-2-oxo-2H-chromene-3-carboxylate
f1 (ppm)
f1 (ppm)

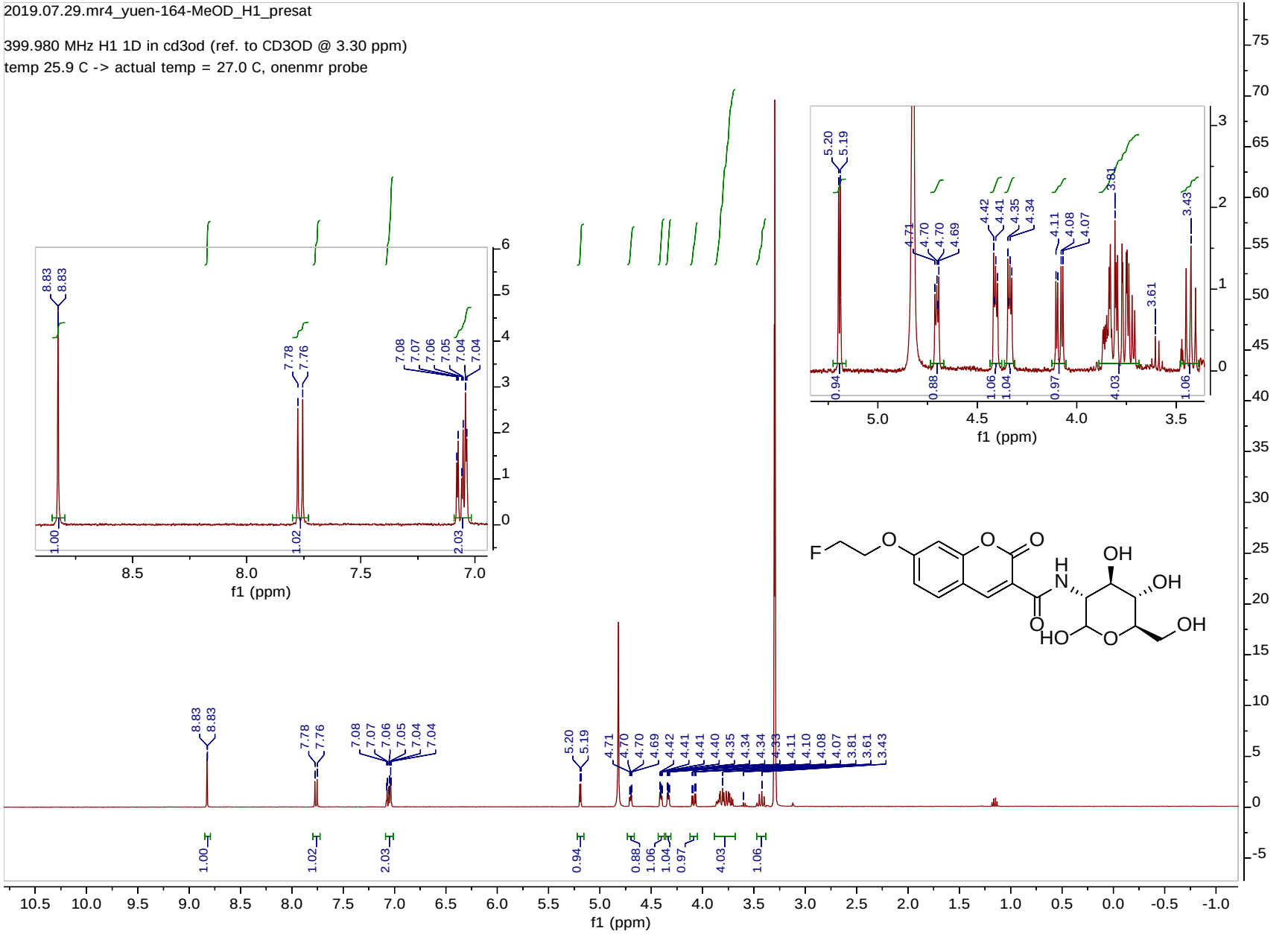

2019.07.24.v7_yuen-159_loc44_20.02_C13_APT_ad
Richard, yuen-159
175.976 MHz C13 APT_ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.5 C -> actual temp = 27.0 C, coldid probe
C & CH2 same, CH & CH3 opposite side of solvent signal
81.82
80.84
77.24
77.06
76.88
67.88
67.76
61.80
163.80
163.40
157.44
157.01
148.82
130.87
114.67
113.95
112.08
101.04
14.31
f1 (ppm)

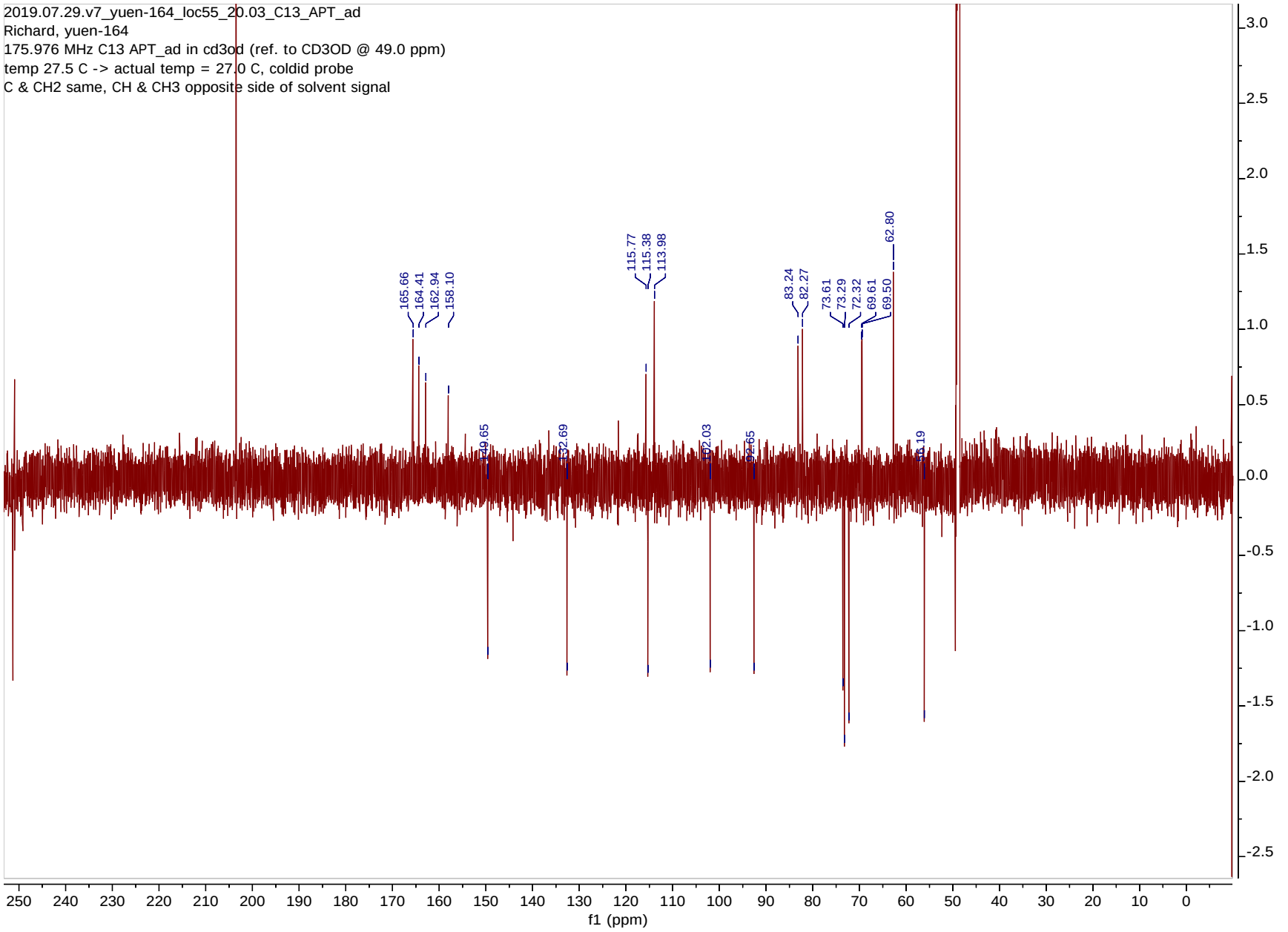

2019.07.16.mr4_yuen-159_19F_cdcl3_F19_1D
376.318 MHz F19 1D in cdcl3
temp 25.9 C -> actual temp = 27.0 C, onenmr probe
-223.64
-223.71
-223.77
-223.79
-223.84
-223.89
-223.91
-223.96
-224.04
f1 (ppm)

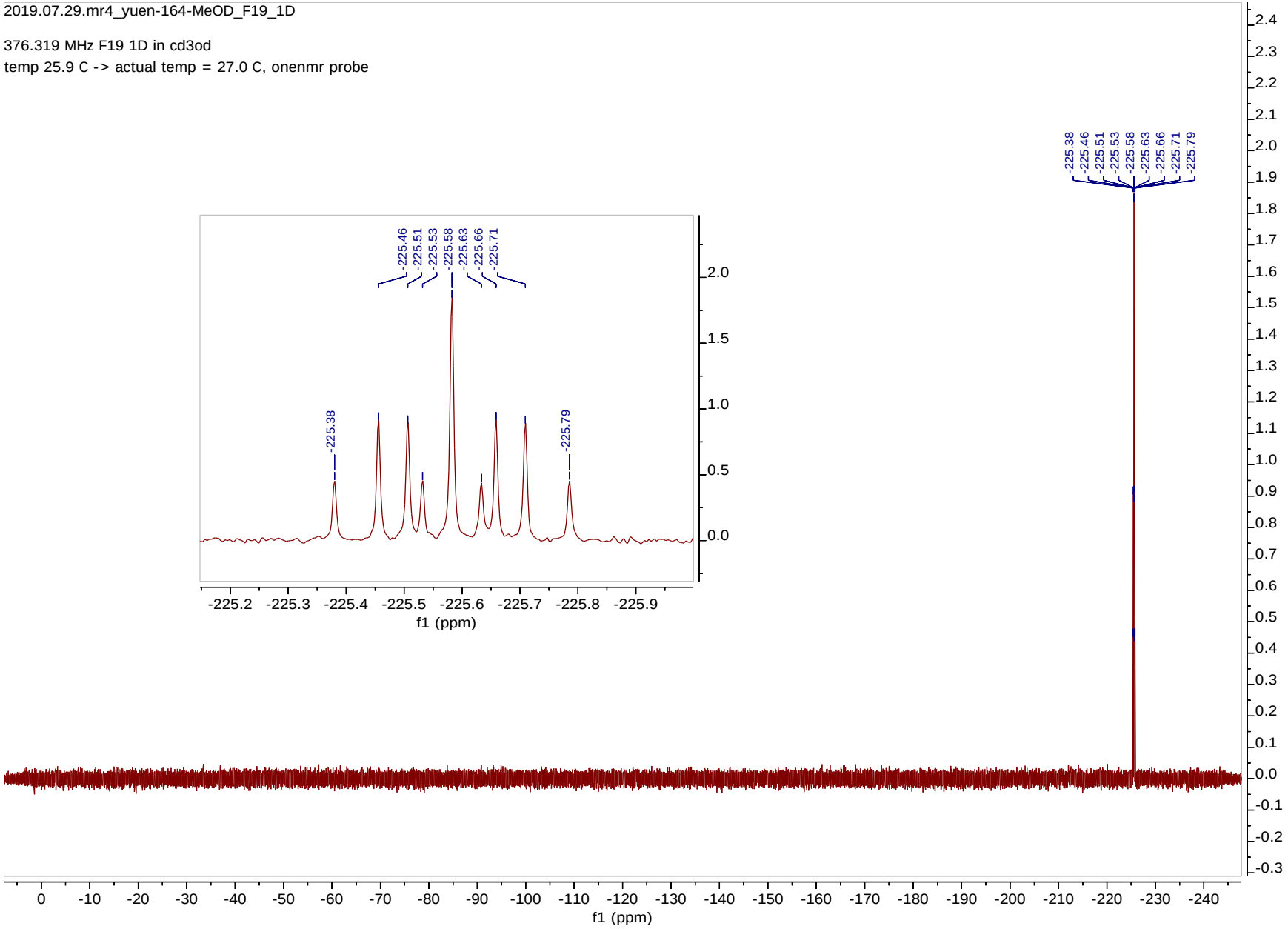

2019.07.25.mr4_yuen-163_H1_1D
399.980 MHz H1 1D in dmso (ref. to DMSO @ 2.49 ppm)
temp 25.9 C -> actual temp = 27.0 C, onenmr probe
F
O
O
O
OH
O
f1 (ppm)

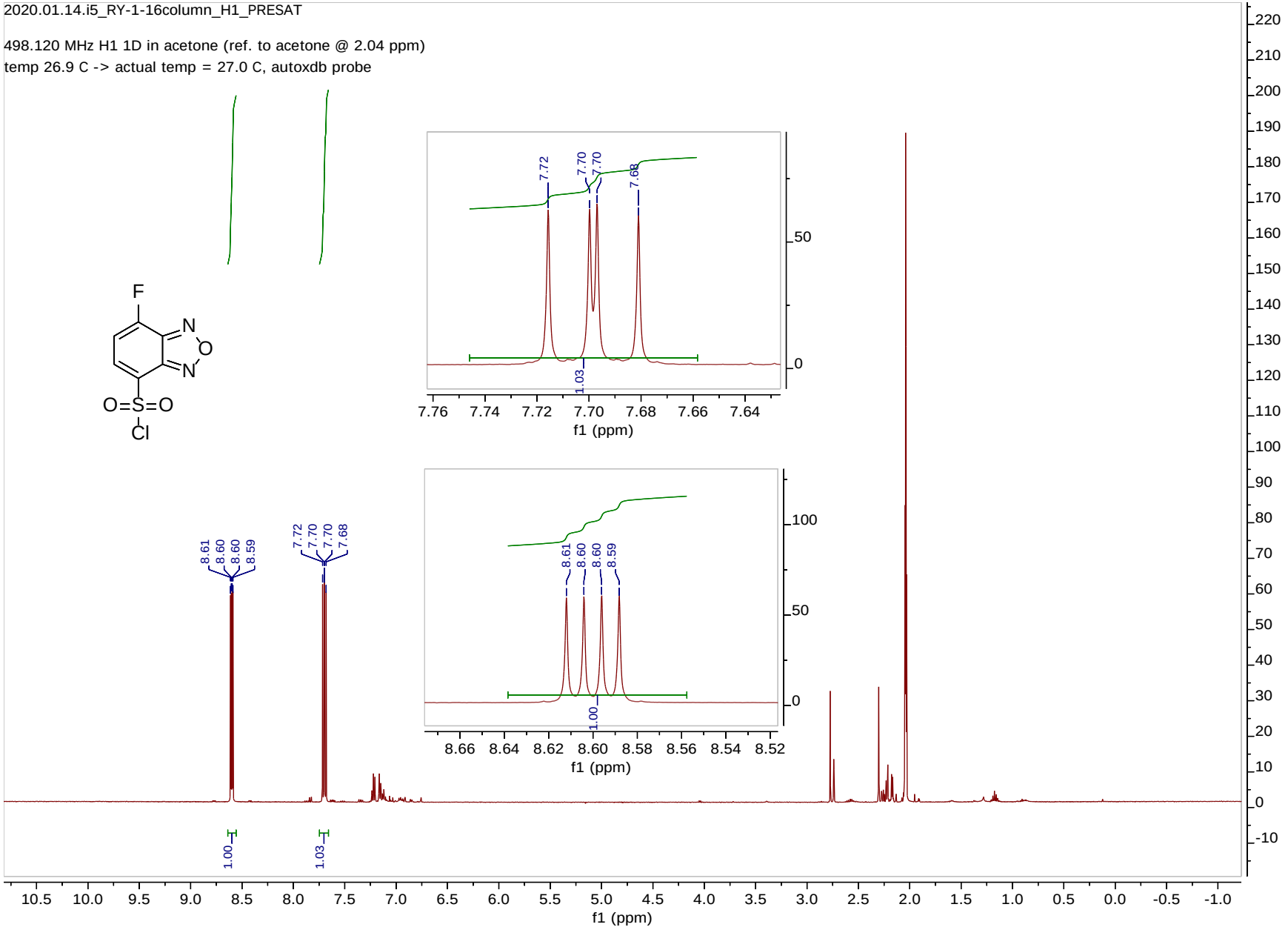

2019.07.25.v7_yuen-163_loc73_20.02_C13_APT_ad
Richard, yuen-163
175.976 MHz C13 APT_ad in dmso (ref. to DMSO @ 39.5 ppm)
temp 27.5 C -> actual temp = 27.0 C, coldid probe
C & CH2 same, CH & CH3 opposite side of solvent signal
164.12
163.41
157.16
156.76
148.98
131.66
114.12
113.55
111.87
100.87
82.29
81.34
68.10
67.99
f1 (ppm)

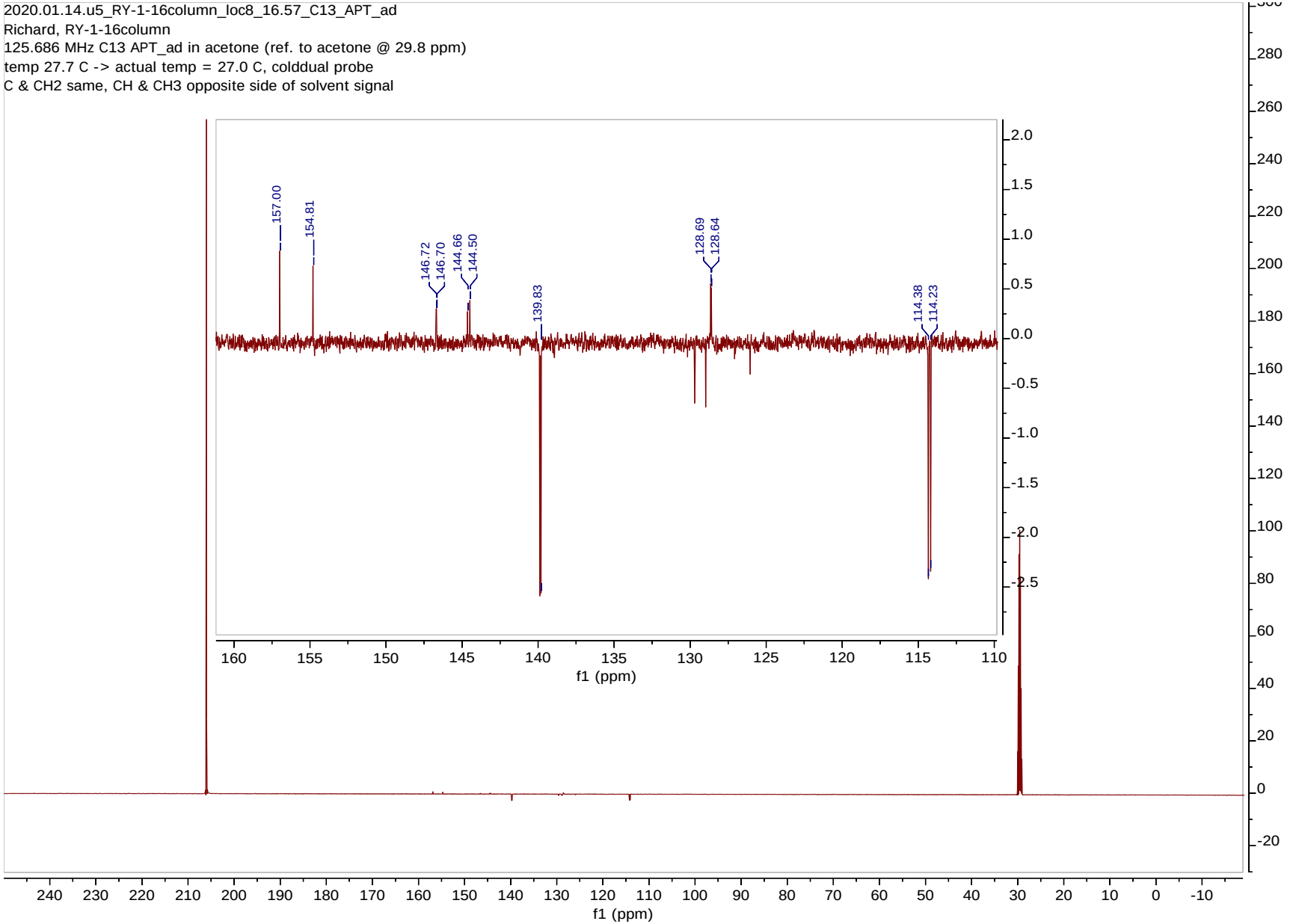

2019.07.25.mr4_yuen-163_F19_1D
376.320 MHz F19 1D in dmso
temp 25.9 C -> actual temp = 27.0 C, onenmr probe
-222.12
-222.20
-222.24
-222.28
-222.32
-222.37
-222.40
-222.45
-222.53
f1 (ppm)

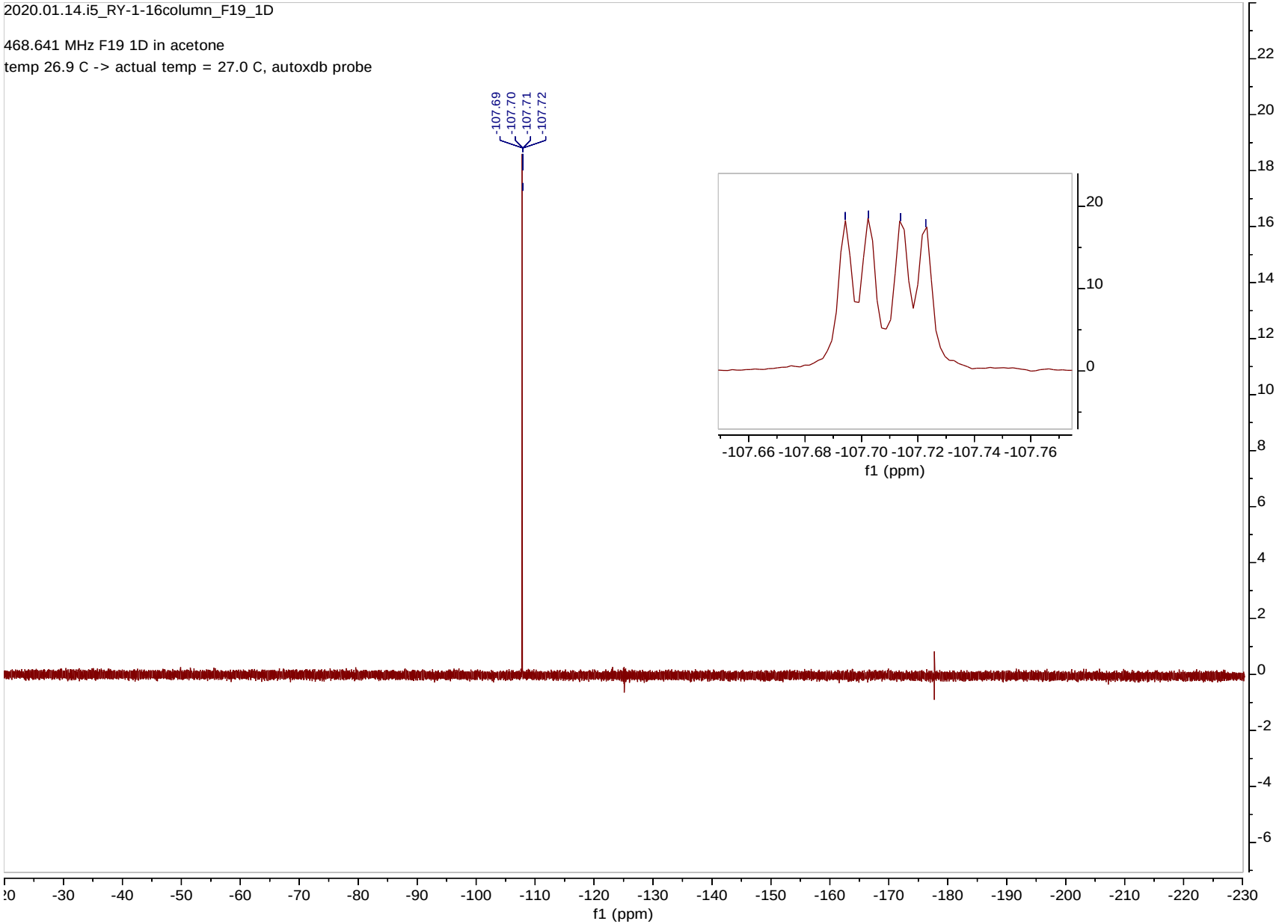

2019.07.29.mr4_yuen-164-MeOD_H1_presat
399.980 MHz H1 1D in cd3od (ref. to CD3OD @ 3.30 ppm)
temp 25.9 C -> actual temp = 27.0 C, onenmr probe
f1 (ppm)
F
O
O
O
H
N
OH
OH
OH
HO
O

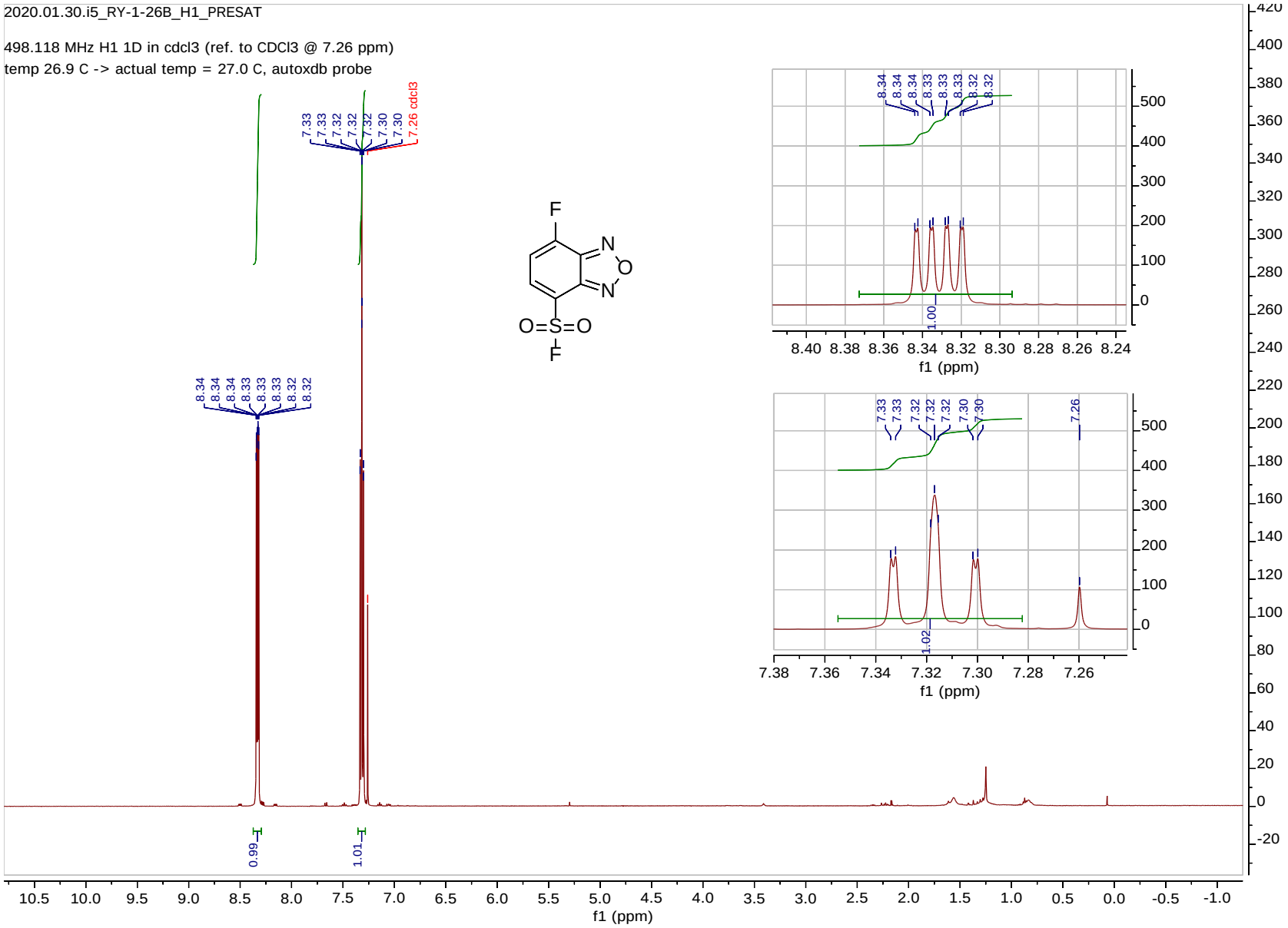

2019.07.29.v7_yuen-164_loc55_20.03_C13_APT_ad
Richard, yuen-164
175.976 MHz C13 APT_ad in cd3od (ref. to CD3OD @ 49.0 ppm)
temp 27.5 C -> actual temp = 27.0 C, coldid probe
C & CH2 same, CH & CH3 opposite side of solvent signal
165.66
164.41
162.94
158.10
149.65
132.69
115.77
115.38
113.98
92.03
82.65
83.24
82.27
73.61
73.29
72.32
69.61
69.50
62.80
55.19
f1 (ppm)

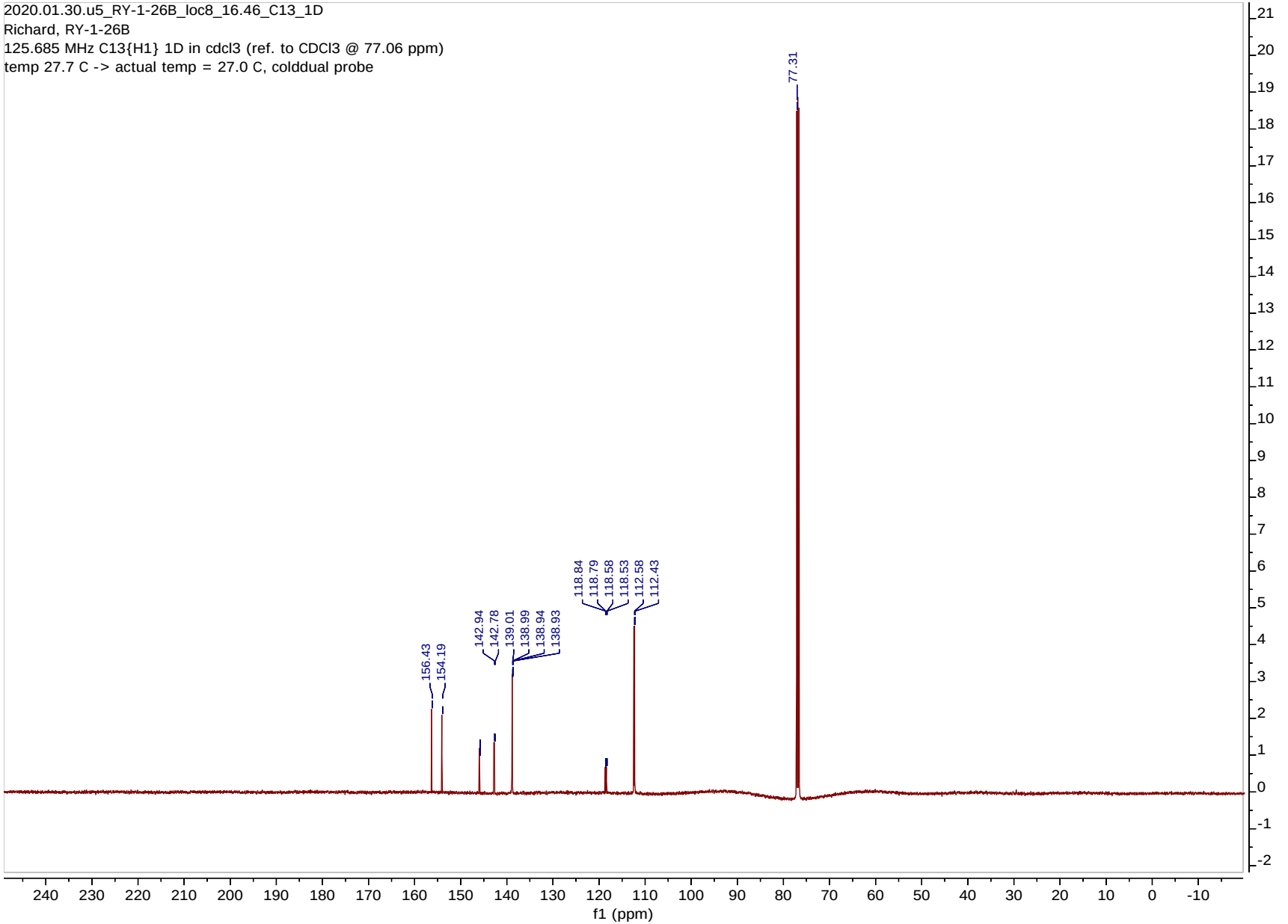

2019.07.29.mr4_yuen-164-MeOD_F19_1D
376.319 MHz F19 1D in cd3od
temp 25.9 C -> actual temp = 27.0 C, onenmr probe
-225.38
-225.46
-225.51
-225.53
-225.58
-225.63
-225.66
-225.71
-225.79
f1 (ppm)

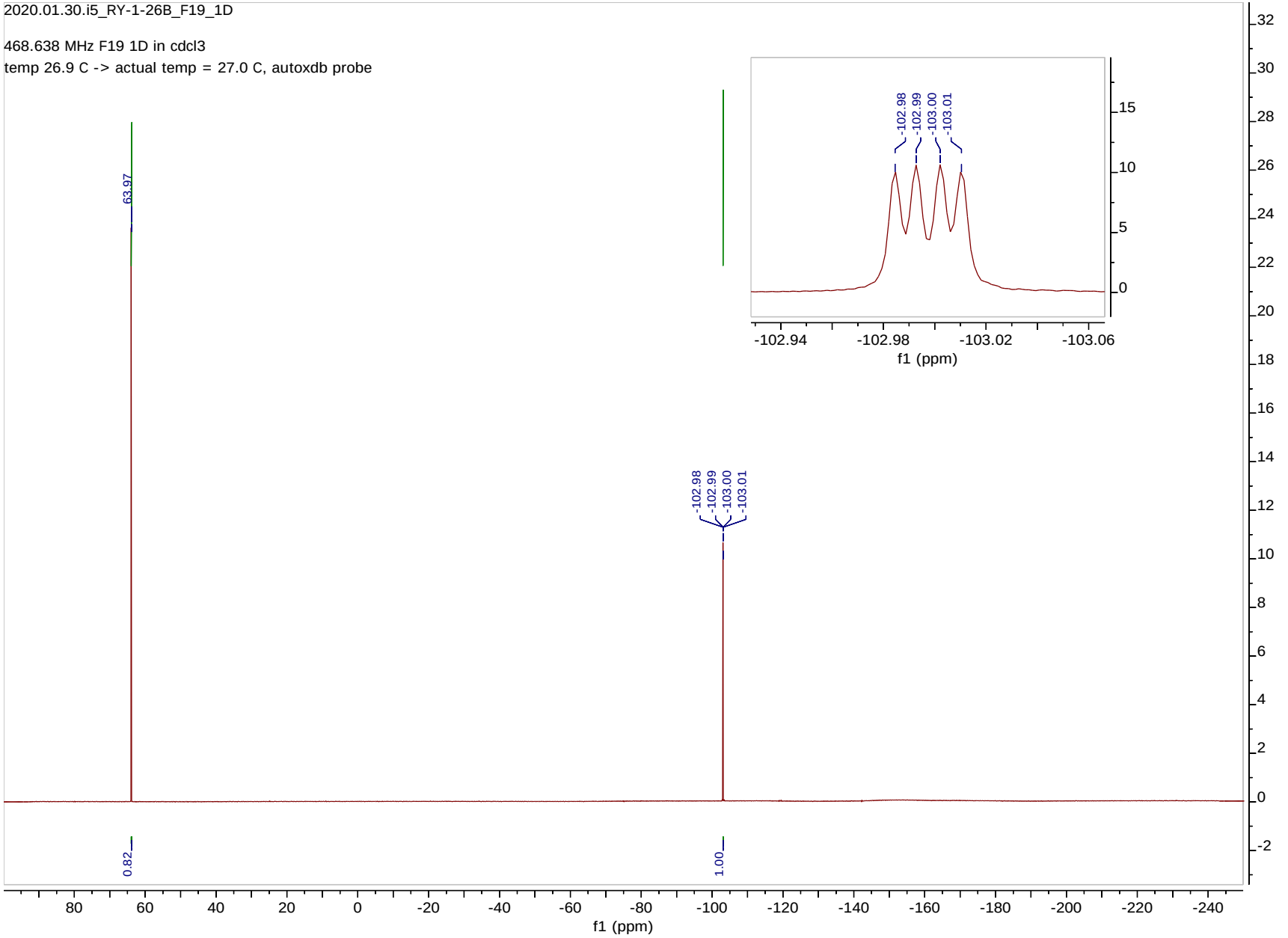

2020.01.14.i5_RY-1-16column_H1_PRESAT
498.120 MHz H1 1D in acetone (ref. to acetone @ 2.04 ppm)
temp 26.9 C -> actual temp = 27.0 C, autoxdb probe
F
N
O
N
O=S=O
Cl
7.72
7.70
7.70
7.68
1.03
7.76 7.74 7.72 7.70 7.68 7.66 7.64
f1 (ppm)
8.61
8.60
8.60
8.59
1.00
8.66 8.64 8.62 8.60 8.58 8.56 8.54 8.52
f1 (ppm)
1.00
1.03
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

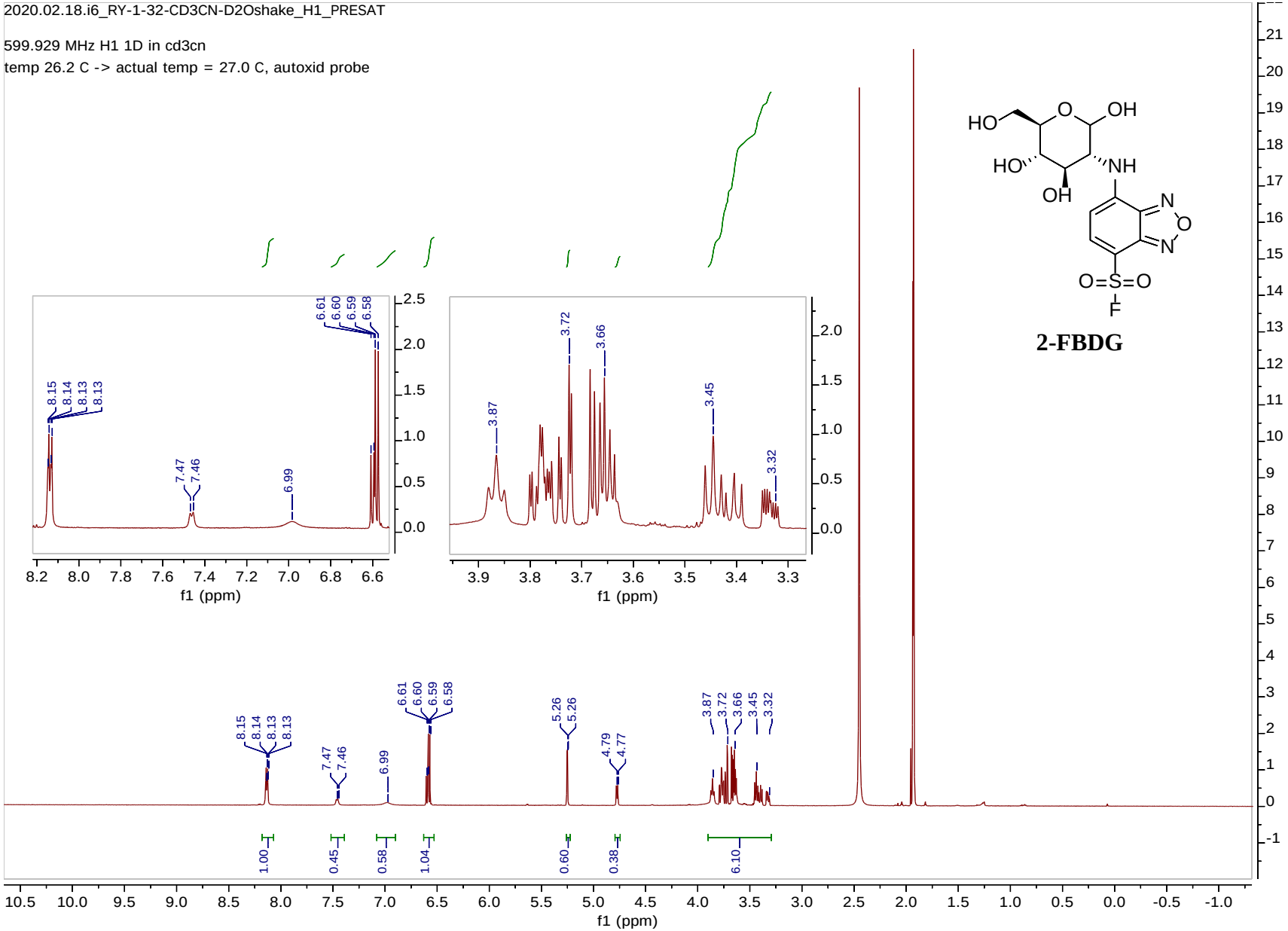

2020.01.14.u5_RY-1-16column_loc8_16.57_C13_APT_ad
Richard, RY-1-16column
125.686 MHz C13 APT_ad in acetone (ref. to acetone @ 29.8 ppm)
temp 27.7 C -> actual temp = 27.0 C, colddual probe
C & CH2 same, CH & CH3 opposite side of solvent signal
157.00
154.81
146.72
146.70
144.66
144.50
139.83
128.69
128.64
114.38
114.23
f1 (ppm)
f1 (ppm)

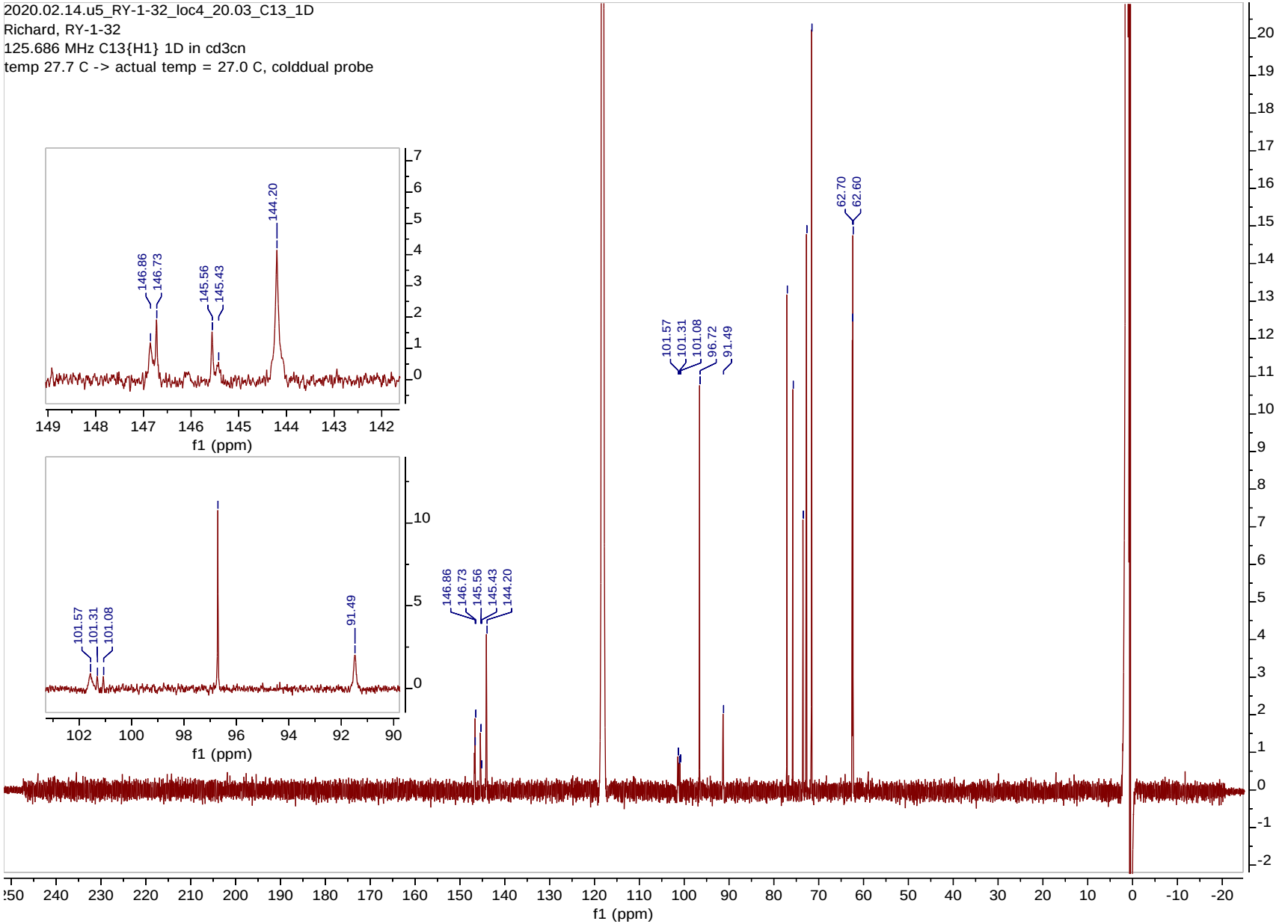

2020.01.14.i5_RY-1-16column_F19_1D
468.641 MHz F19 1D in acetone
temp 26.9 C -> actual temp = 27.0 C, autoxdb probe
-107.69
-107.70
-107.71
-107.72
-107.66 -107.68 -107.70 -107.72 -107.74 -107.76
f1 (ppm)
f1 (ppm)

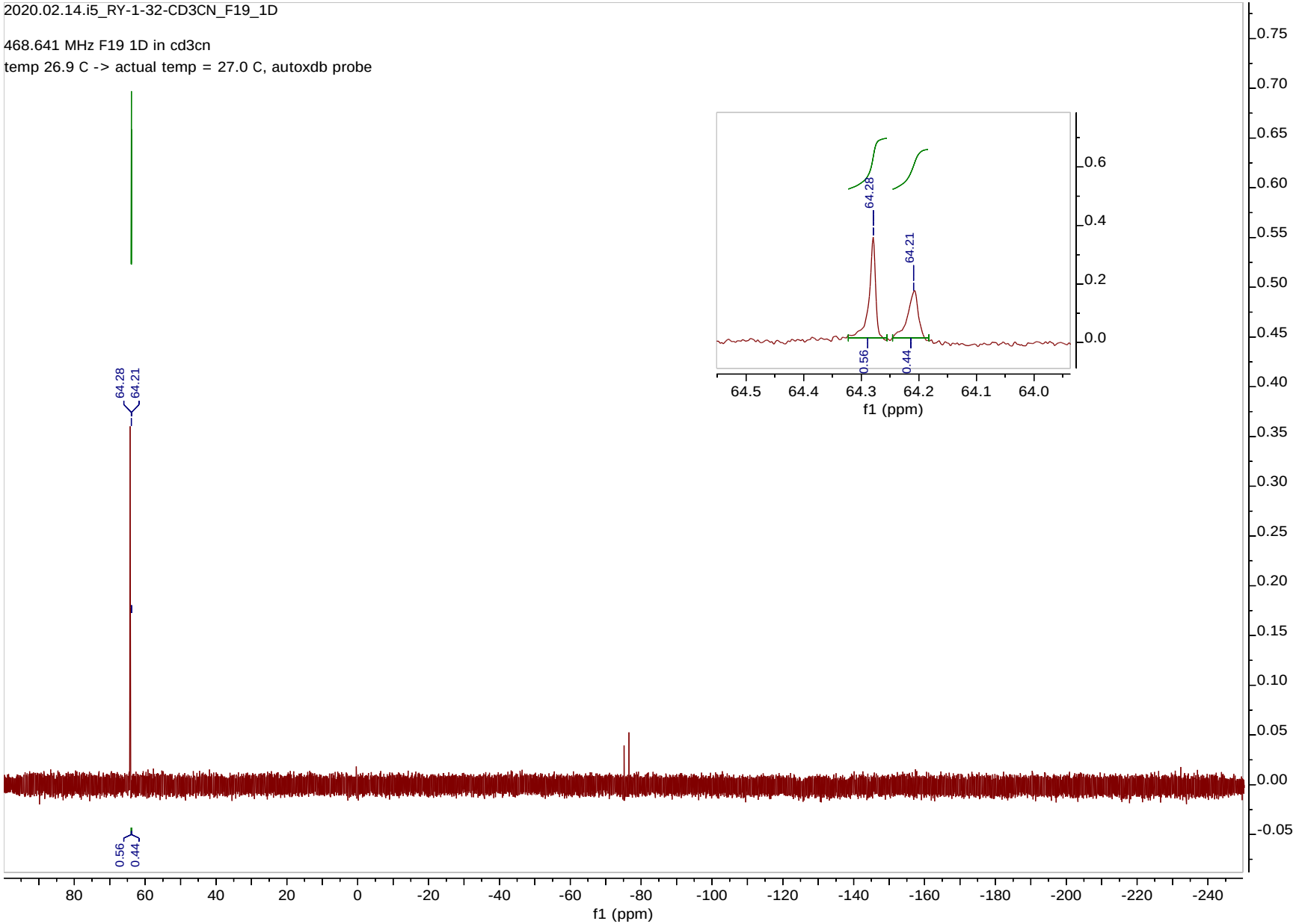

2020.01.30.i5_RY-1-26B_H1_PRESAT
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 26.9 C -> actual temp = 27.0 C, autoxdb probe
7.33
7.33
7.32
7.32
7.32
7.30
7.30
7.26 cdcl3
8.34
8.34
8.34
8.33
8.33
8.33
8.32
8.32
F
N
O
N
O=S=O
F
0.99
1.01
1.00
1.02
f1 (ppm)


2020.01.30.u5_RY-1-26B_loc8_16.46_C13_1D
Richard, RY-1-26B
125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.7 C -> actual temp = 27.0 C, colddual probe
77.31
156.43
154.19
142.94
142.78
139.01
138.99
138.94
138.93
118.84
118.79
118.58
118.53
112.58
112.43
f1 (ppm)

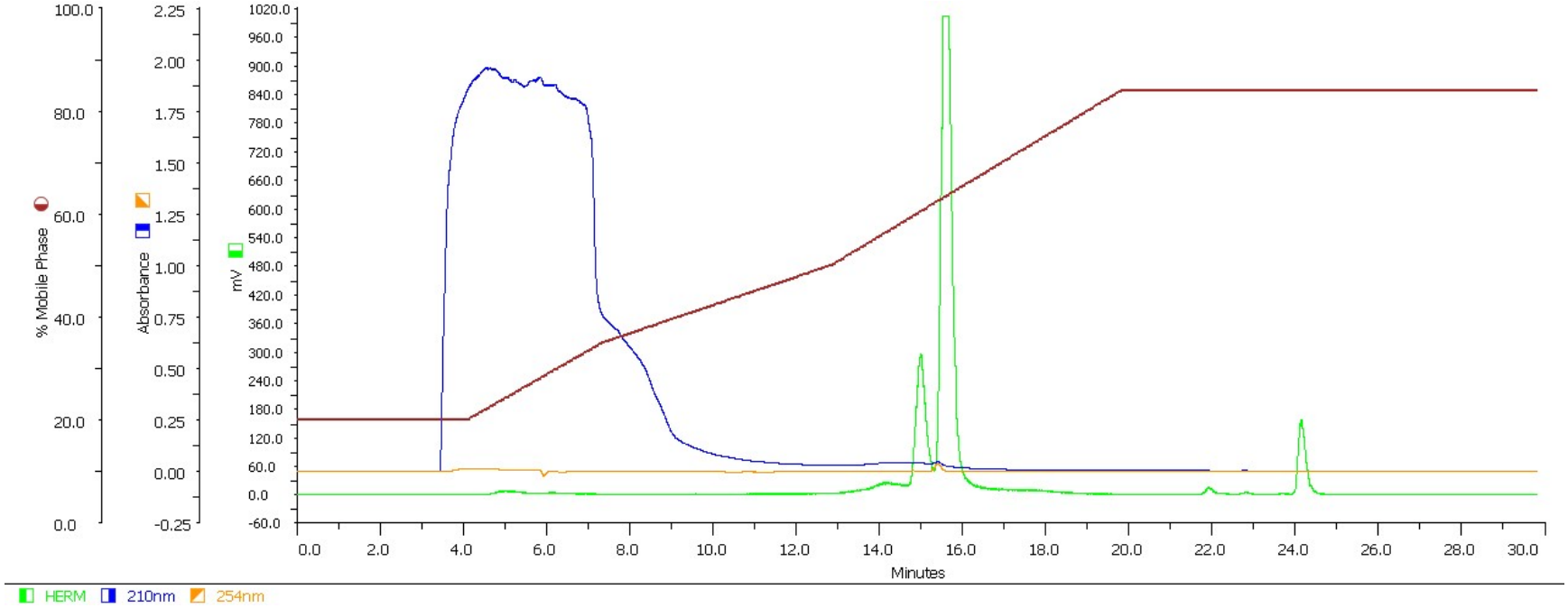

2020.01.30.i5_RY-1-26B_F19_1D
468.638 MHz F19 1D in cdcl3
temp 26.9 C -> actual temp = 27.0 C, autoxdb probe
63.97
-102.98
-102.99
-103.00
-103.01
0.82
1.00
f1 (ppm)
-102.94
-102.98
-103.02
-103.06


2020.02.18.i6_RY-1-32-CD3CN-D2Oshake_H1_PRESAT
599.929 MHz H1 1D in cd3cn
temp 26.2 C -> actual temp = 27.0 C, autoxid probe
2-FBDG
f1 (ppm)

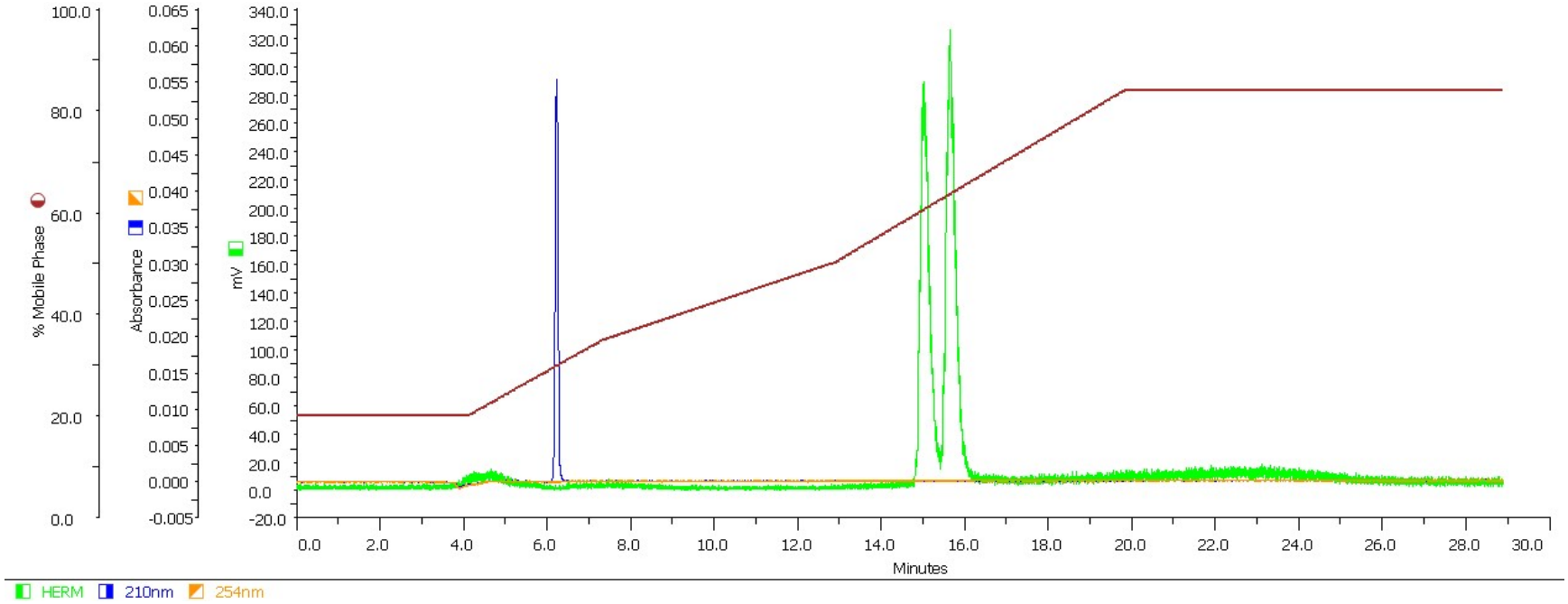

2020.02.14.u5_RY-1-32_loc4_20.03_C13_1D
Richard, RY-1-32
125.686 MHz C13{H1} 1D in cd3cn
temp 27.7 C -> actual temp = 27.0 C, colddual probe
146.86
146.73
145.56
145.43
144.20
101.57
101.31
101.08
96.72
91.49
62.70
62.60
f1 (ppm)


2020.02.14.i5_RY-1-32-CD3CN_F19_1D
468.641 MHz F19 1D in cd3cn
temp 26.9 C -> actual temp = 27.0 C, autoxdb probe
64.28
64.21
0.56
0.44
f1 (ppm)

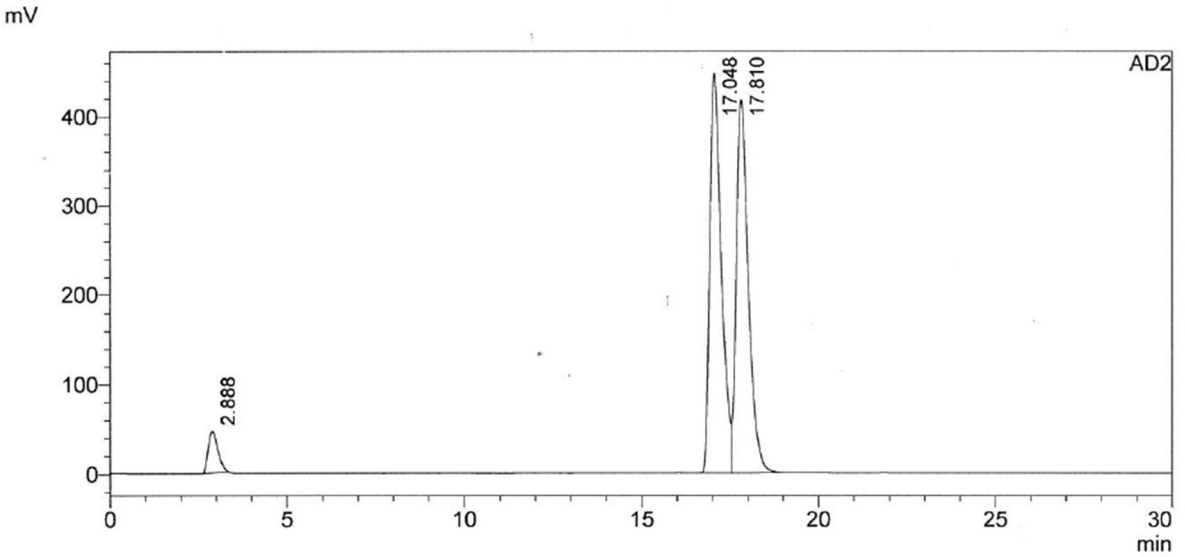
HPLC Traces for 2-[^{18}F]FBDG

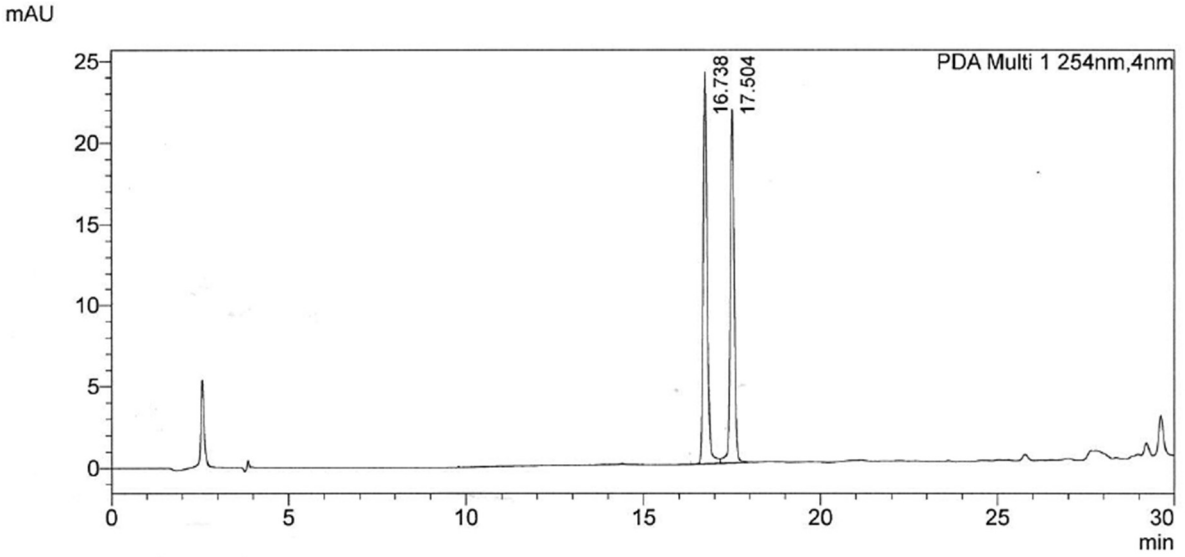


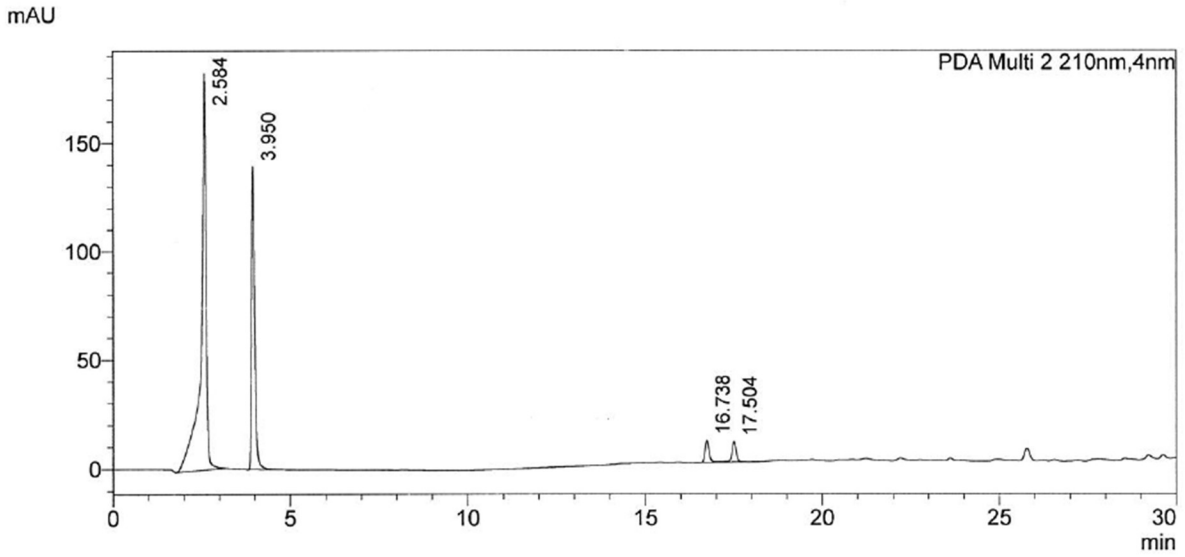
HPLC trace for the purification of the crude 2-[^{18}F]FBDG (mixture of α/β anomers). Unreacted [^{18}F]FBDF has t_R = 24.1 min.



HPLC trace for the purified 2-[^{18}F]FBDG product (t_R = 15.0 and 15.7 min)


mV
AD2
17.048
17.810
2.888
400
300
200
100
0
0
5
10
15
20
25
30
min


mAU
PDA Multi 1 254nm,4nm
16.738
17.504
25
20
15
10
5
0
0
5
10
15
20
25
30
min



<Peak Table>

AD2

Peak#	Ret. Time	Height	Area	Area%	Name
1	2.888	46356	883035	4.221	
2	17.048	446600	10206543	48.793	
3	17.810	416662	9828429	46.985	
Total		909618	20918008	100.000	

PDA Ch1 254nm

Peak#	Ret. Time	Height	Area	Area%	Name
1	16.738	23994	193736	52.455	
2	17.504	21651	175599	47.545	
Total		45645	369335	100.000	

PDA Ch2 210nm

Peak#	Ret. Time	Height	Area	Area%	Name
1	2.584	181550	1887909	64.259	
2	3.950	139087	884042	30.090	
3	16.738	10178	85773	2.919	
4	17.504	9357	80252	2.732	
Total		340172	2937976	100.000	

Quality control HPLC run of 2-[^{18}F]FBDG on a Phenomenex Luna 10u C18(2) 100A, 250 × 4.6 mm column