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

Regioselective Routes to Orthogonally-Substituted Aromatic MIDA boronates.

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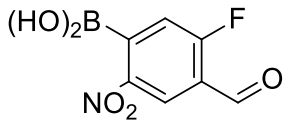
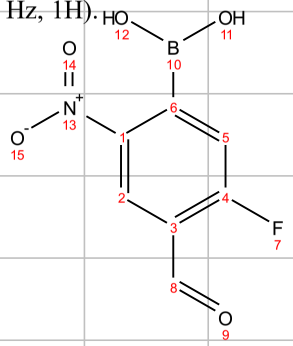
5-Fluoro-4-formyl-2-nitrophenyl boronic acid 2a



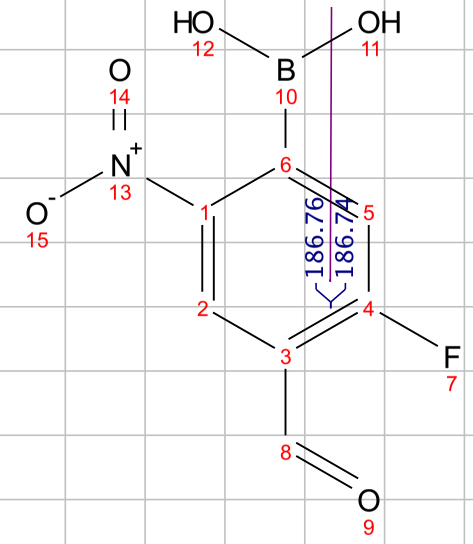
PROTON_01
AC102N007

^{1}H NMR (500 MHz, dmso) δ 10.20 (s, 1H), 8.54 (d, J = 5.6 Hz, 1H), 8.46 (s, 2H), 7.61 (d, J = 9.8 Hz, 1H).



CARBON_01

^{13}C NMR (126 MHz, dmso) δ 186.75 (d, *J* = 2.1 Hz), 165.25 (d, *J* = 268.4 Hz), 146.29 (d, *J* = 1.8 Hz), 145.06 (s), 124.73 (d, *J* = 2.2 Hz), 123.73 (d, *J* = 11.1 Hz), 120.68 (d, *J* = 21.4 Hz).



186.76
186.74
166.31
164.18
146.29
146.28
145.06
124.73
124.72
123.77
123.69
120.77
120.60
39.52 Dimethyl Sulfoxide-d6

8 (d)
186.75

4 (d)
165.25

6 (s)
145.06

1 (d)
146.29

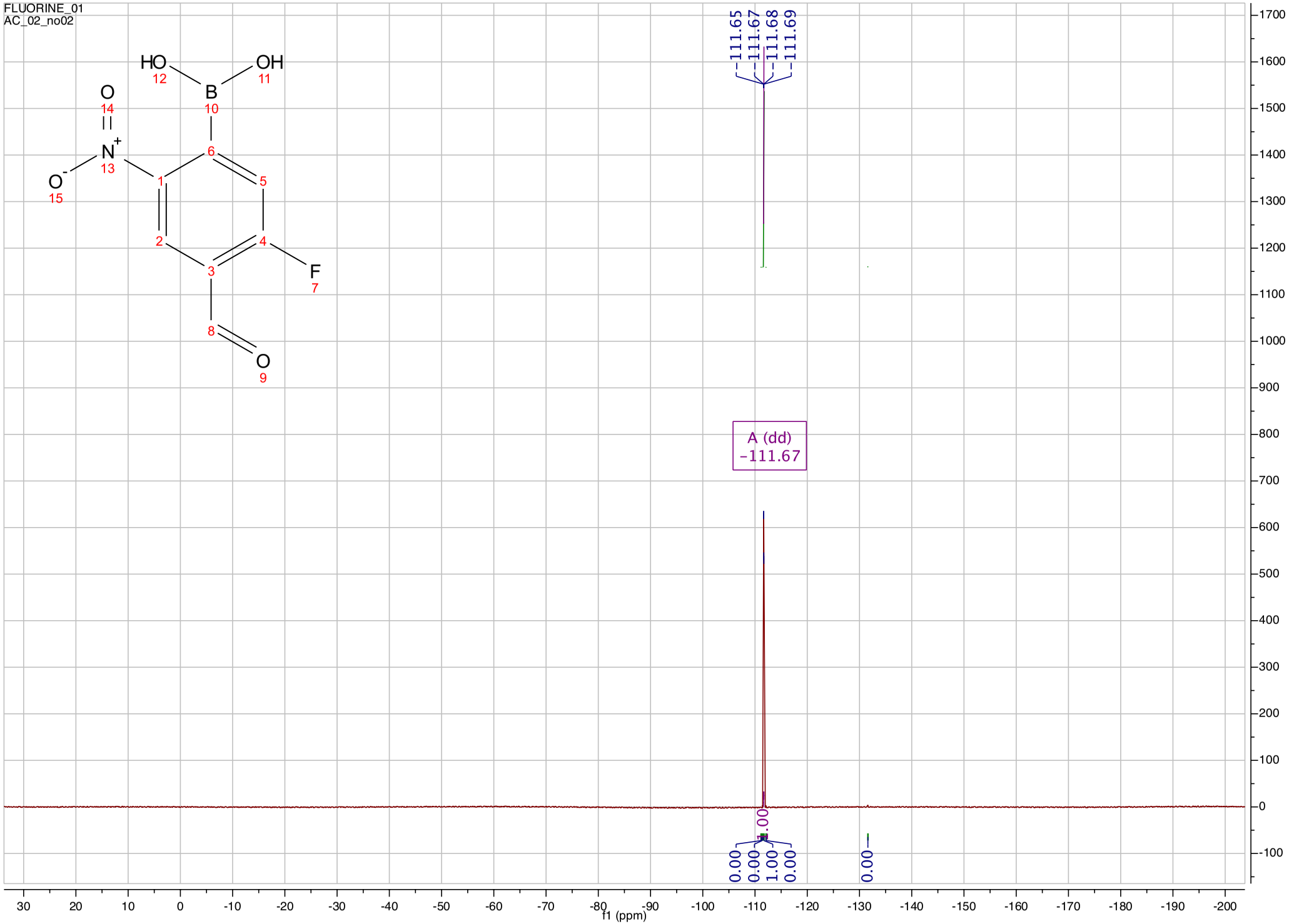
3 (d)
123.73

2 (d)
124.73

5 (d)
120.68

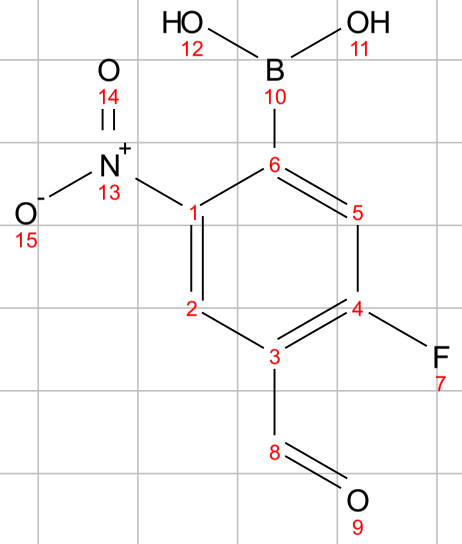
1.00
0.31
0.36
0.34
1.02
0.42
1.03

f1 (ppm)


FLUORINE_01
AC_02_no02
HO
12
OH
11
B
10
O
14
N+
13
O-
15
1
2
3
4
5
6
F
7
8
O
9
111.65
111.67
111.68
111.69
A (dd)
-111.67
1.00
0.00
0.00
1.00
0.00
0.00
f1 (ppm)

s2pul_01

^{11}B NMR (128 MHz, dmso) δ 27.77 (s).



A (s)
27.77

1.00

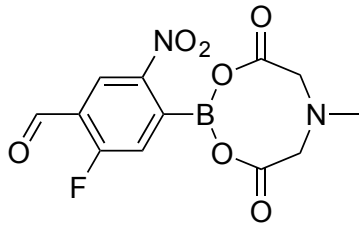
f1 (ppm)

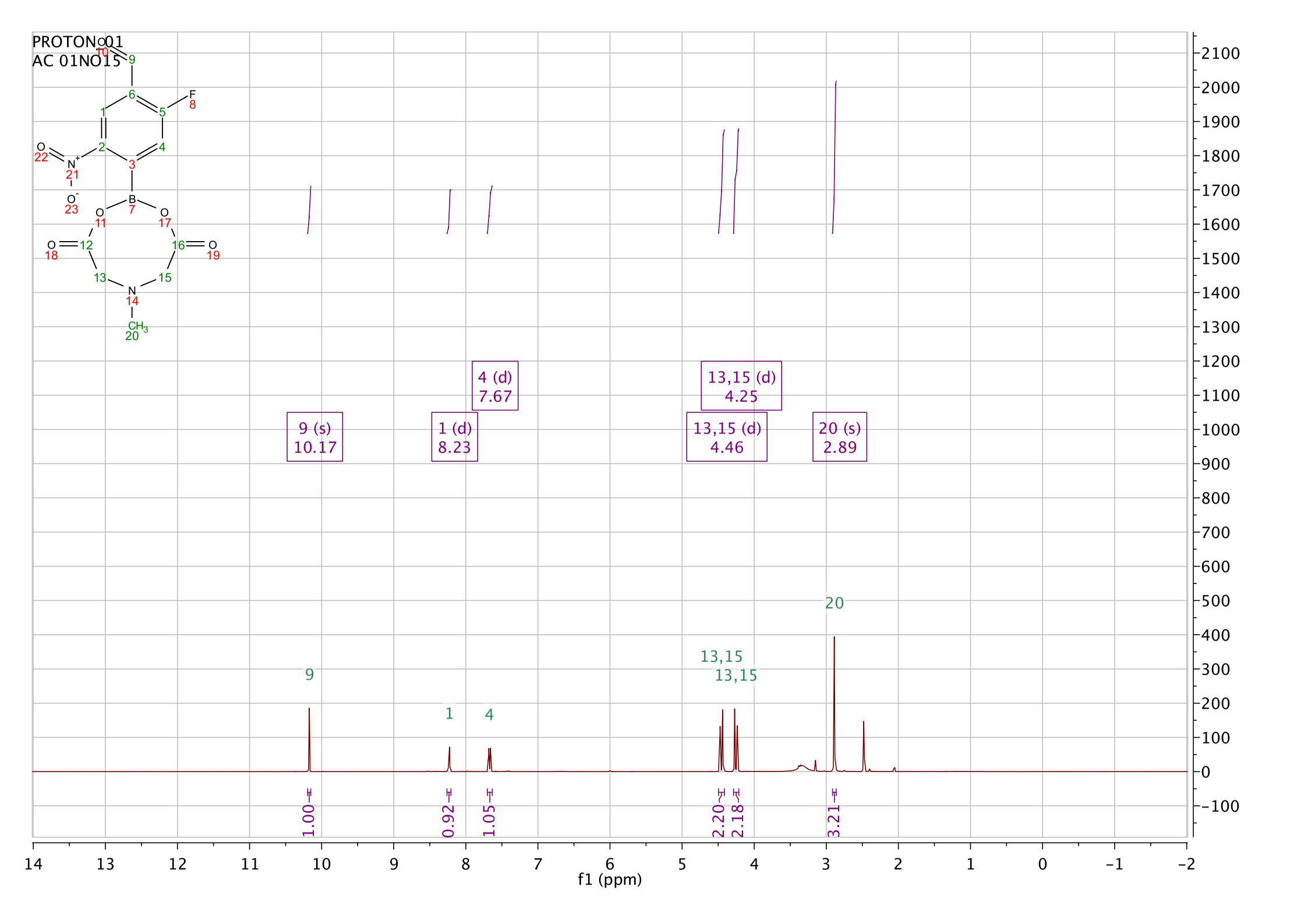
Isotope:	Min. .. Max.
12 C	0....80
1 H	0....120
16 O	0....12
14 N	0....12
10 B	0....2
19 F	0....5
Tolerance Window:	+- 5.00 ppm
Db/Ring Equiv:	-3.. 100
Fits:	100

N-Rule: Do not use
Charge: 1

Mass	Theoretical Mass	Delta [ppm]	RDB	Composition
238.0430	238.0430	-0.0	8.5	$C_3 N_{11} {}^{10}B_1 F_2$
	238.0430	-0.1	3.0	$C_4 H_6 O_5 N_4 {}^{10}B_1 F_2$
	238.0430	0.1	-0.5	$C_1 H_9 O_8 N_5 F_1$
	238.0430	0.2	5.0	$H_3 O_3 N_{12} F_1$
	238.0431	-0.3	6.5	$C_7 H_3 O_2 N_3 {}^{10}B_2 F_3$
	238.0429	0.6	3.0	$C_2 H_2 O_2 N_6 {}^{10}B_2 F_4$
	238.0432	-0.7	8.5	$C_5 H_4 O_3 N_9$
	238.0432	-0.7	3.0	$C_6 H_{10} O_8 N_2$
	238.0428	0.7	5.0	$C_6 H_6 O_7 N_2 {}^{10}B_2$
	238.0428	0.8	10.5	$C_5 O_2 N_9 {}^{10}B_2$
	238.0432	-0.9	1.0	$C_2 H_6 O_3 N_6 F_4$
	238.0432	-0.9	12.0	$C_8 H_1 N_8 {}^{10}B_1 F_1$
	238.0432	-0.9	6.5	$C_9 H_7 O_5 N_1 {}^{10}B_1 F_1$
	238.0432	-1.0	4.5	$C_5 H_3 N_5 {}^{10}B_1 F_5$
	238.0433	-1.1	10.0	$C_{12} H_4 O_2 {}^{10}B_2 F_2$
	238.0426	1.6	1.5	$C_1 H_5 O_7 N_5 {}^{10}B_2 F_1$
	238.0434	-1.7	4.5	$C_7 H_7 O_3 N_3 F_3$
	238.0434	-1.7	15.5	$C_{13} H_2 N_5 {}^{10}B_1$
	238.0434	-1.9	8.0	$C_{10} H_4 N_2 {}^{10}B_1 F_4$
	238.0425	2.3	12.0	$C_{15} H_7 O_2 F_1$
	238.0436	-2.5	8.0	$C_{12} H_8 O_3 F_2$
	238.0423	3.1	8.5	$C_{10} H_6 O_2 N_3 F_2$
	238.0421	3.7	14.0	$C_{15} H_3 O_1 {}^{10}B_2 F_1$
	238.0439	-3.9	-2.5	$H_{10} O_{11} N_3 {}^{10}B_1$
	238.0421	3.9	10.5	$C_{12} H_6 O_4 N_1 {}^{10}B_1$
	238.0421	4.0	5.0	$C_5 H_5 O_2 N_6 F_3$
	238.0440	-4.0	6.5	$C_2 H_1 O_3 N_9 {}^{10}B_2 F_1$
	238.0440	-4.1	1.0	$C_3 H_7 O_8 N_2 {}^{10}B_2 F_1$
	238.0419	4.5	10.5	$C_{10} H_2 O_1 N_3 {}^{10}B_2 F_2$
	238.0419	4.6	-0.5	$C_4 H_7 O_4 N_1 {}^{10}B_1 F_5$
	238.0419	4.7	7.0	$C_7 H_5 O_4 N_4 {}^{10}B_1 F_1$

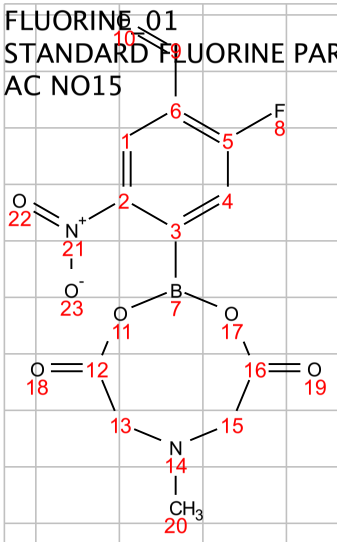
5-Fluoro-4-formyl-2-nitrophenyl MIDA boronate 2b




PROTON_01
AC 01NO15
9 (s)
10.17
1 (d)
8.23
4 (d)
7.67
13,15 (d)
4.46
13,15 (d)
4.25
20 (s)
2.89
9
1
4
13,15
13,15
20
1.00
0.92
1.05
2.20
2.18
3.21
f1 (ppm)


CARBON_01
AC 01NO15
12,16 (s)
169.23
2 (d)
151.38
1 (d)
125.63
9 (d)
186.98
5 (d)
163.38
4 (d)
124.95
6 (d)
125.23
13,15 (s)
64.76
20 (s)
49.91
13,15
12,16
20
9
5
2
6
4
1
2.77
2.32
1.00
0.95
2.77
1.10
2.61
5.68
3.10
f1 (ppm)

FLUORINE_01
STANDARD FLUORINE PARAMETERS
AC NO15



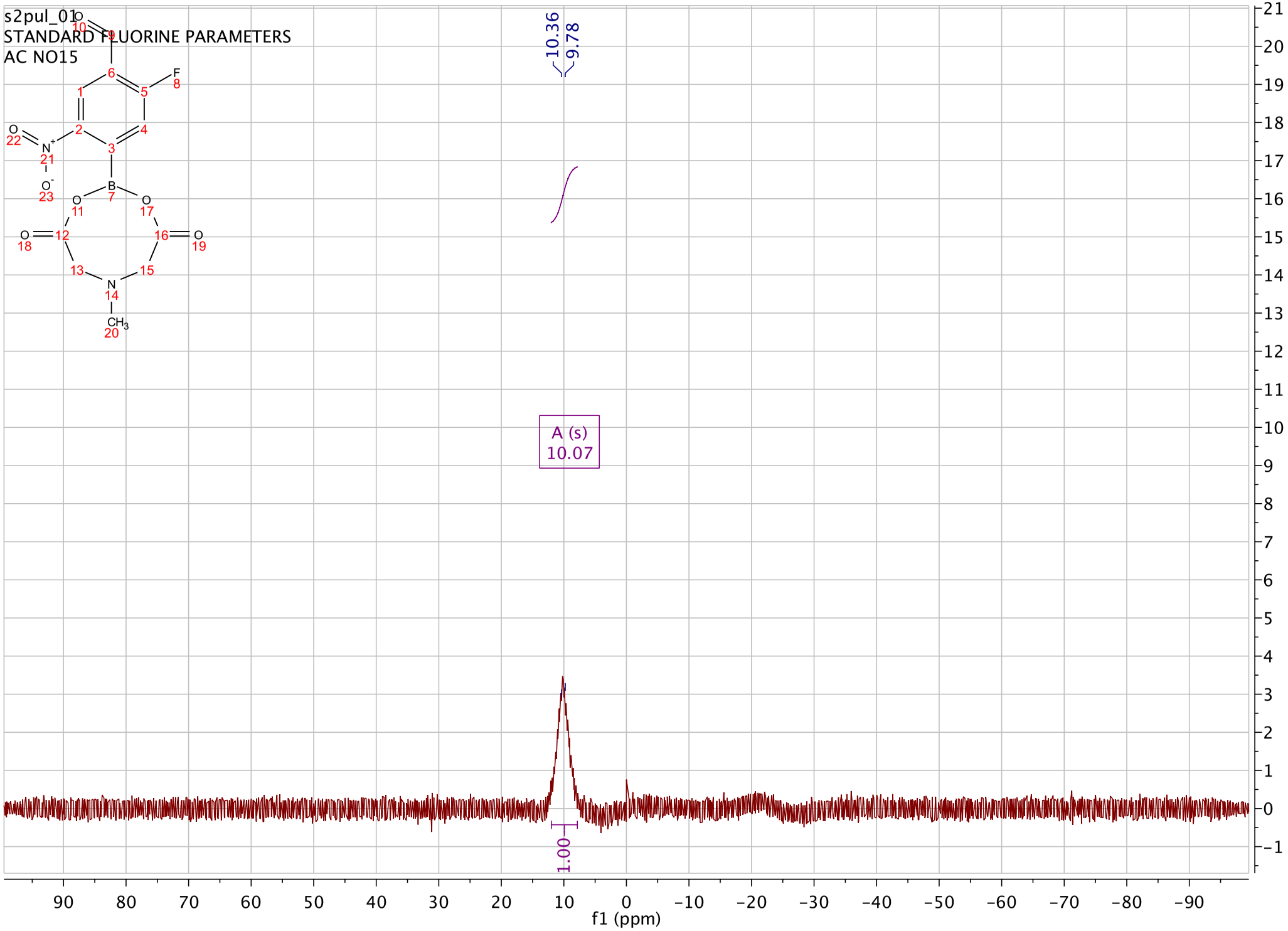
-114.36
-114.37
-114.39
-114.40

A (dd)
-114.38

1.00

2200 2100 2000 1900 1800 1700 1600 1500 1400 1300 1200 1100 1000 900 800 700 600 500 400 300 200 100 0 -100 -200

30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)

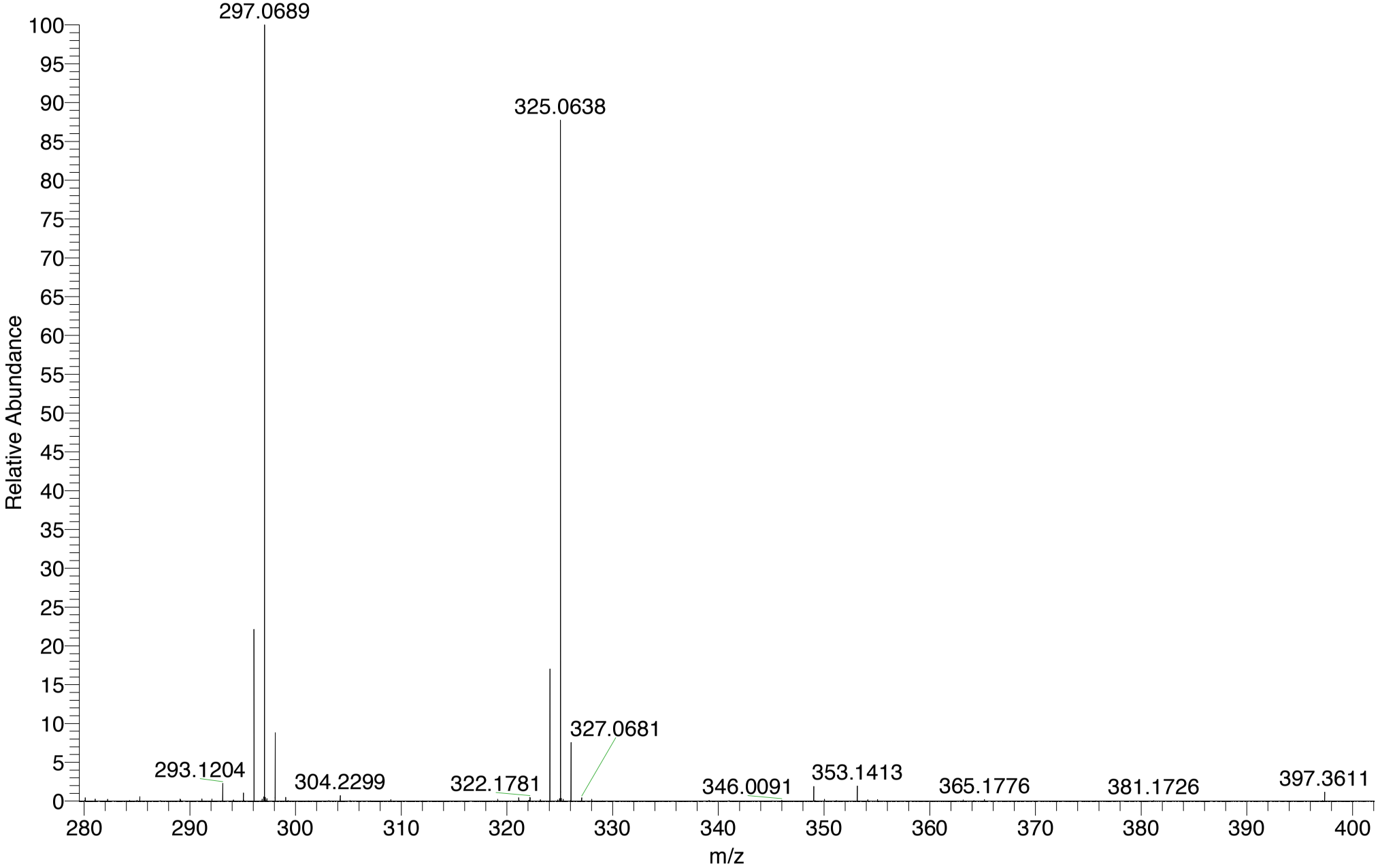

s2pul_01
STANDARD FLUORINE PARAMETERS
AC NO15
10.36
9.78
A (s)
10.07
1.00
f1 (ppm)

NO15 MW=324?
ASAP (MeCN)

EPSRC National Facility Swansea
LTQ Orbitrap XL

Adam
09/09/2015 03:11:44 PM

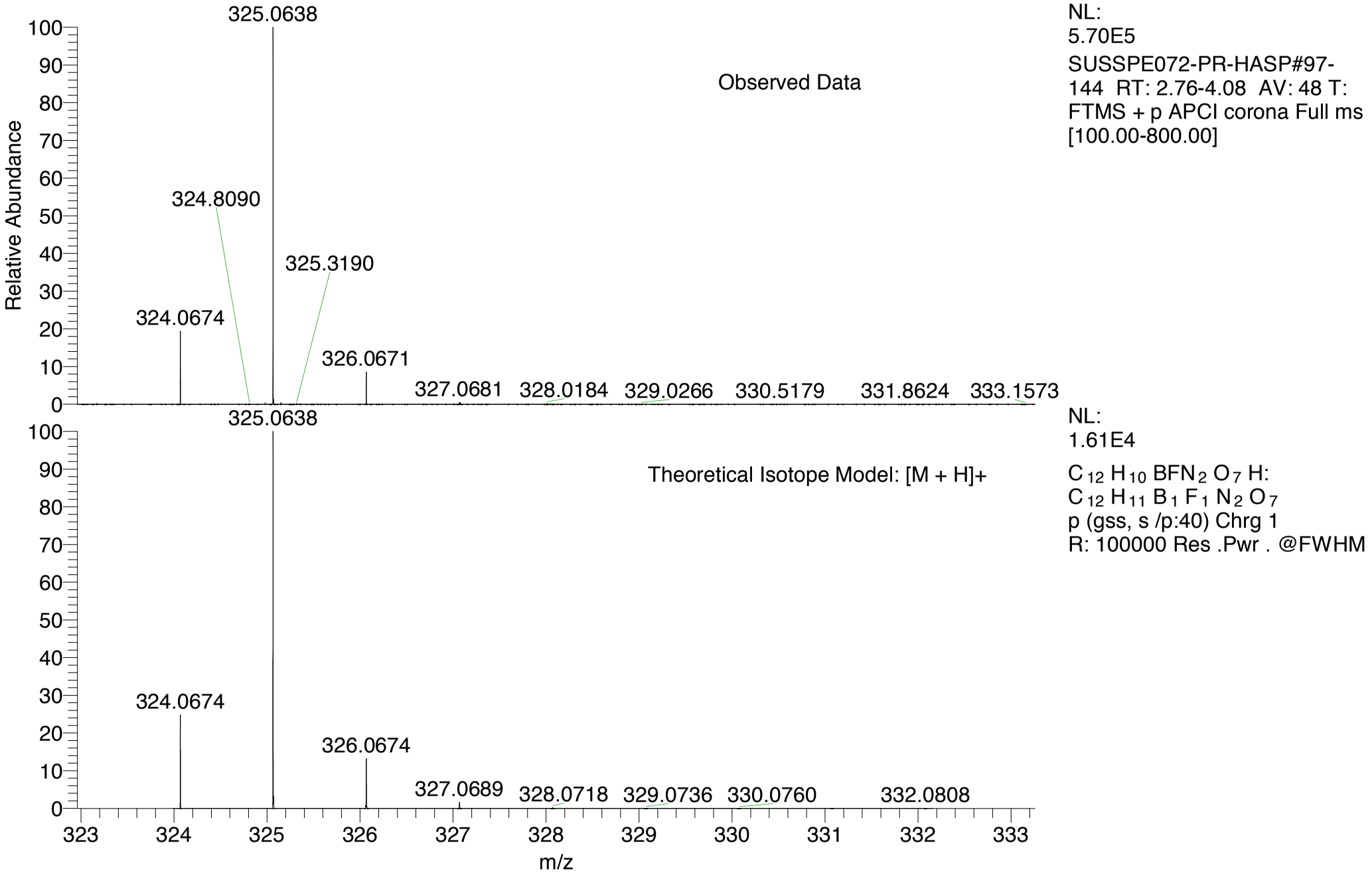
SUSSPE072-PR-HASP #97-144 RT: 2.76-4.08 AV: 48 NL: 6.50E5
T: FTMS + p APCI corona Full ms [100.00-800.00]



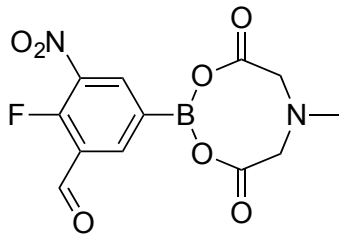
NO15 MW=324?
ASAP (MeCN)

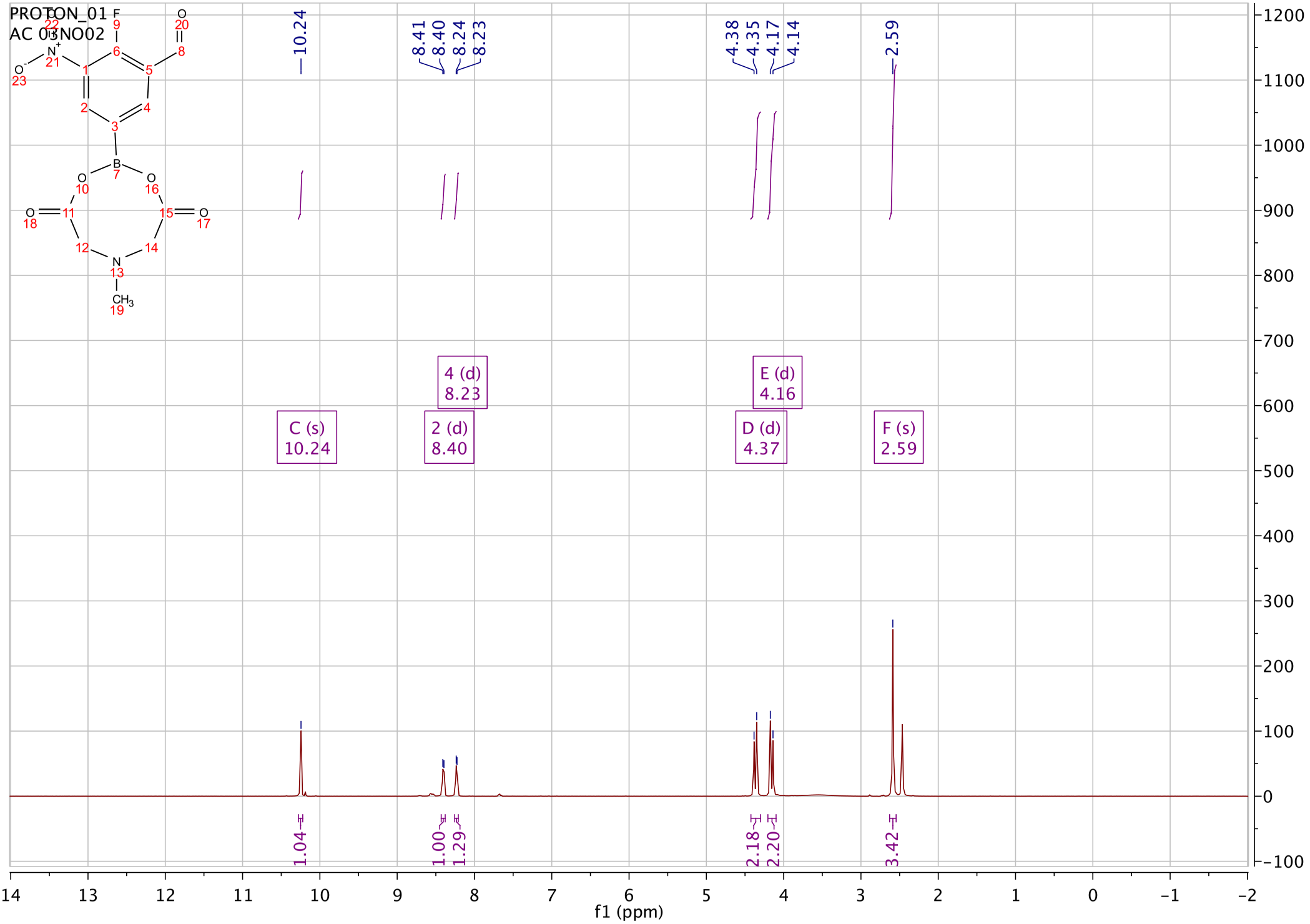
EPSRC National Facility Swansea
LTQ Orbitrap XL

Adam
09/09/2015 03:11:44 PM



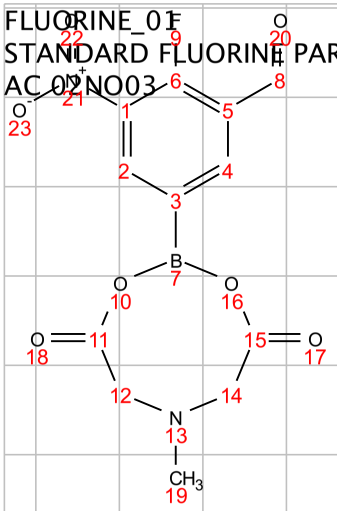
4-Fluoro-3-formyl-5-nitrophenyl MIDA boronate 2c




PROTON_01
AC 03NO02
10.24
8.41
8.40
8.24
8.23
4.38
4.35
4.17
4.14
2.59
C (s)
10.24
2 (d)
8.40
4 (d)
8.23
D (d)
4.37
E (d)
4.16
F (s)
2.59
1.04
1.00
1.29
2.18
2.20
3.42
f1 (ppm)


CARBON_01
AC 03NO02
187.43
187.39
169.52
157.95
155.76
139.69
139.67
137.76
137.71
135.69
125.53
125.48
62.93
48.48
6 (d)
156.86
1 (d)
137.73
8 (d)
187.41
11,15 (s)
169.52
4 (d)
139.68
5 (d)
125.51
2 (s)
135.69
12,14 (s)
62.93
19 (s)
48.48
1.00
0.81
0.36
0.93
0.36
0.95
0.44
2.08
1.03
f1 (ppm)

FLUORINE_01
STANDARD FLUORINE PARAMETERS
AC_02NO03

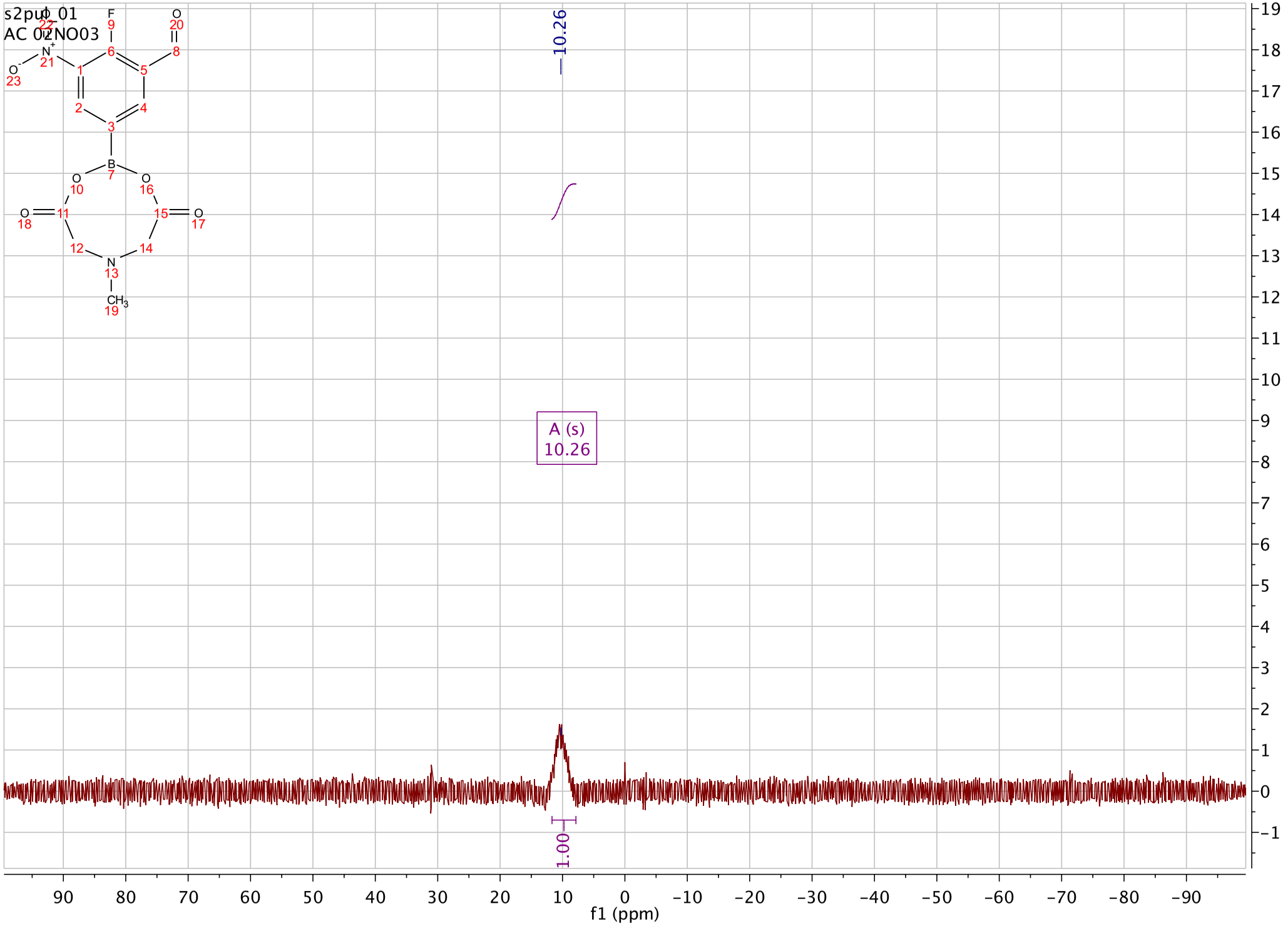


-128.36

A (s)
-128.36

1.00

f1 (ppm)

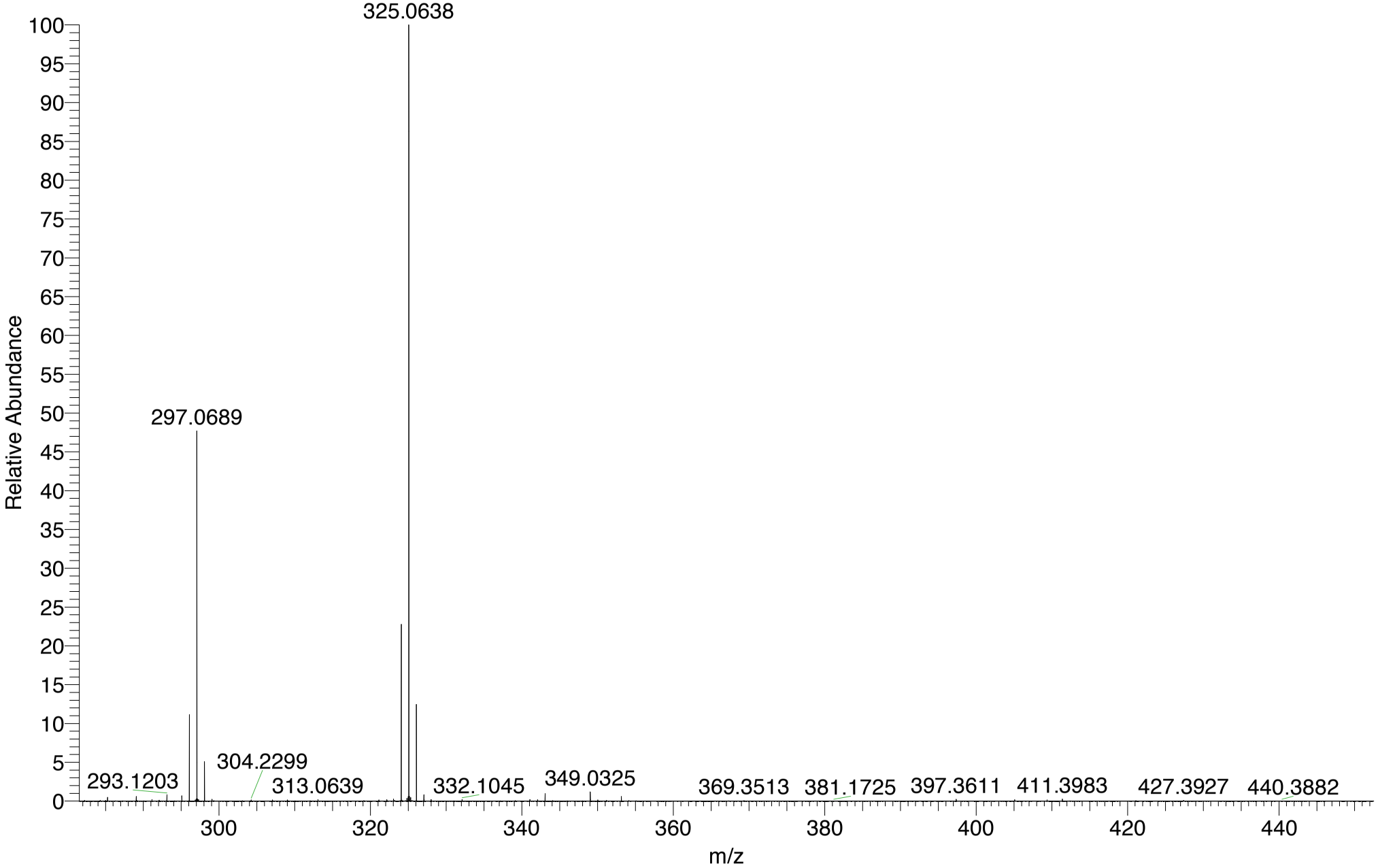

s2pul 01
AC 02NO03
10.26
A (s)
10.26
1.00
f1 (ppm)

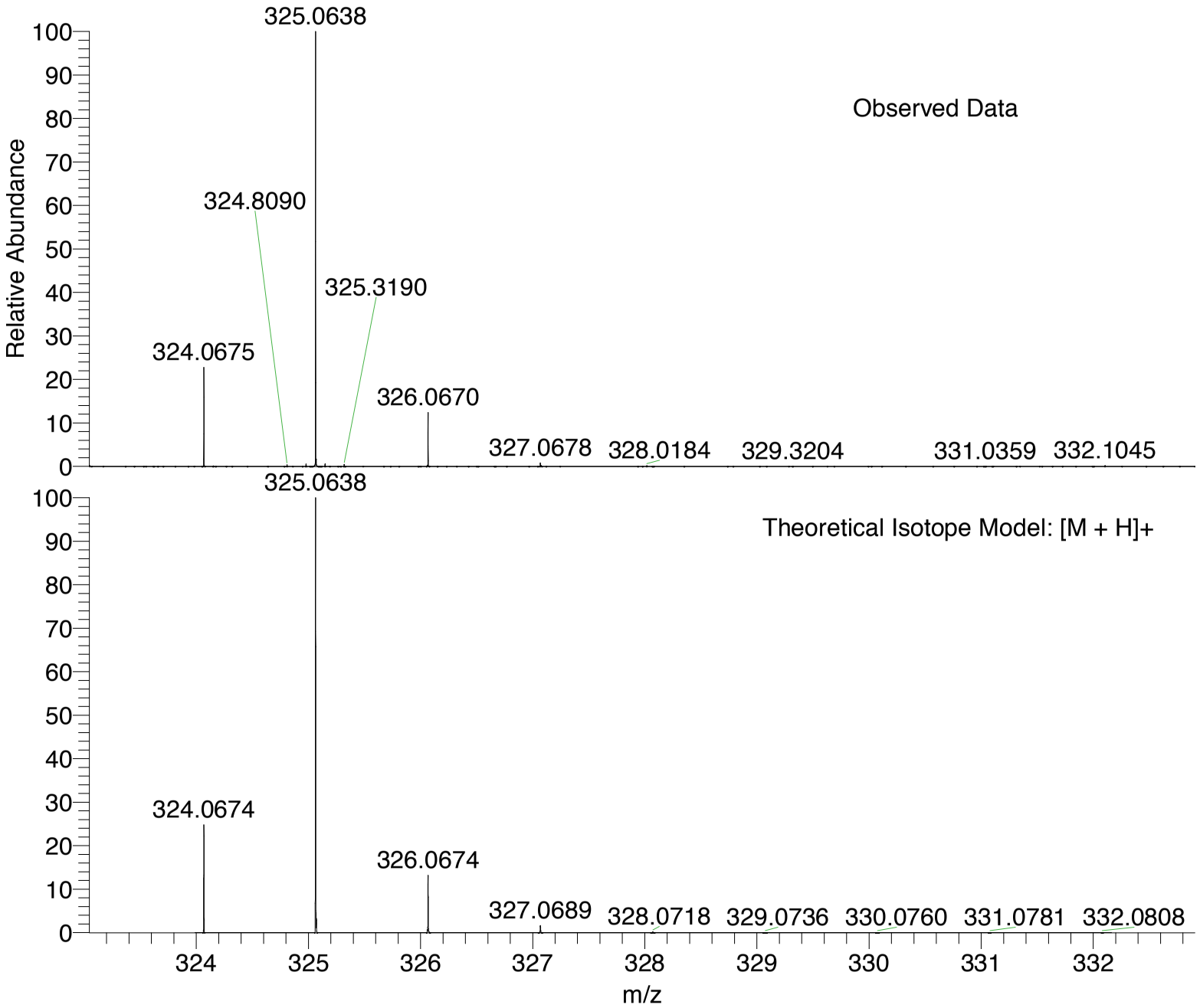
02NO03 MW=324?
ASAP (MeCN)

EPSRC National Facility Swansea
LTQ Orbitrap XL

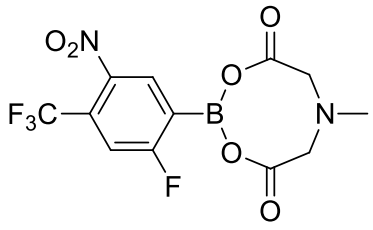
Adam
09/09/2015 03:30:00 PM

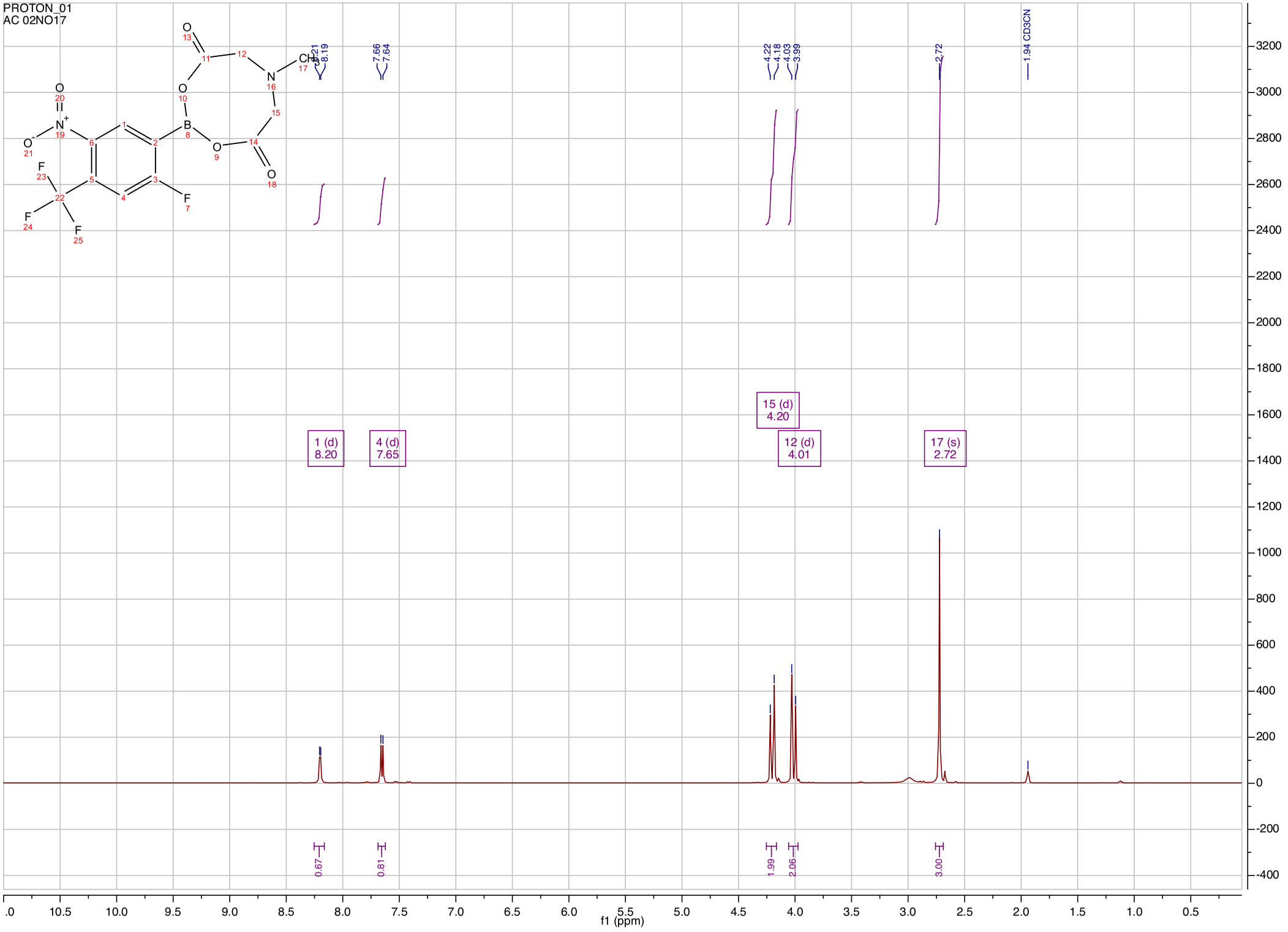
SUSSPE074-PR-HASP #45-59 RT: 1.24-1.64 AV: 15 NL: 3.11E6
T: FTMS + p APCI corona Full ms [100.00-800.00]

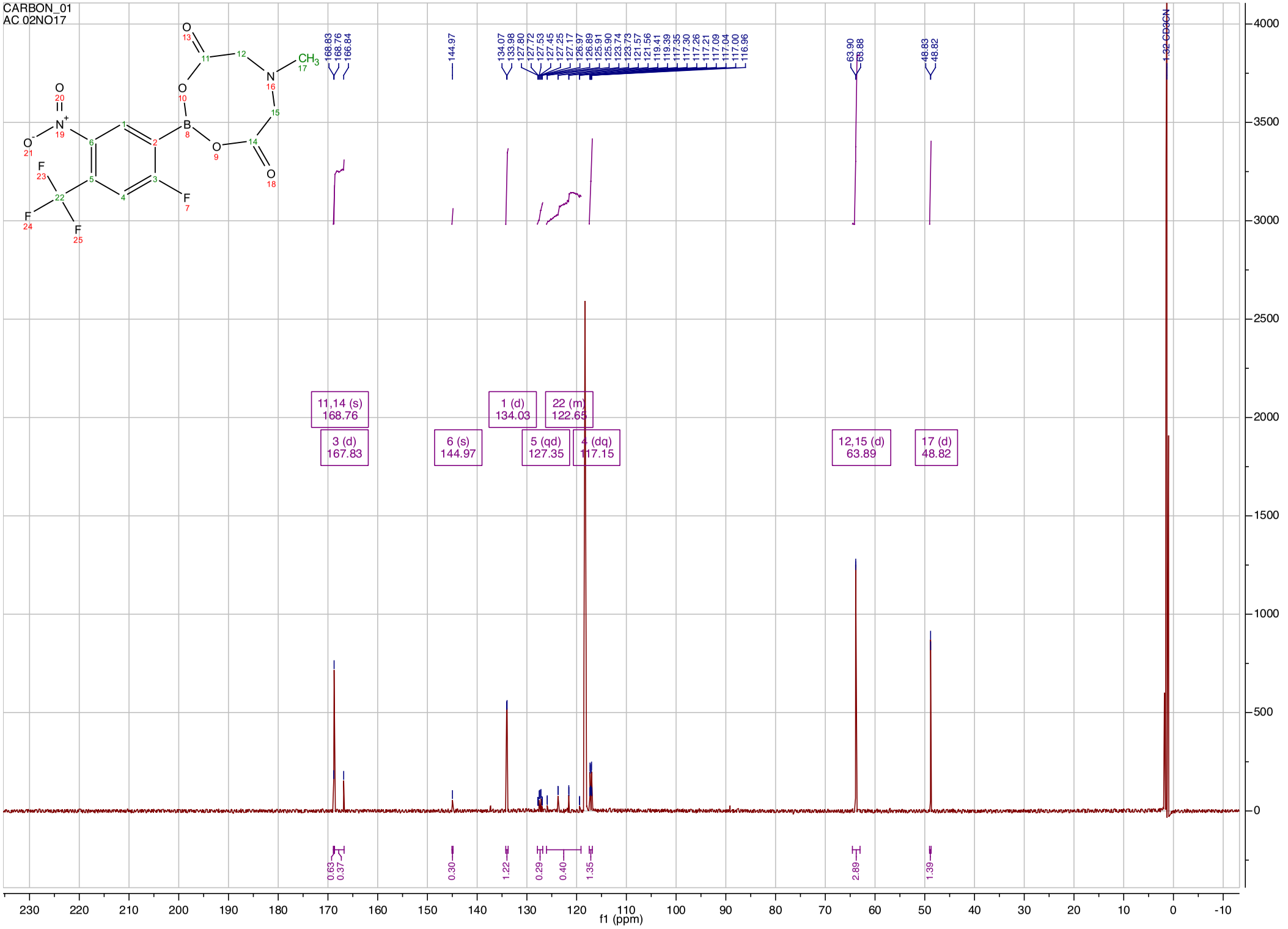


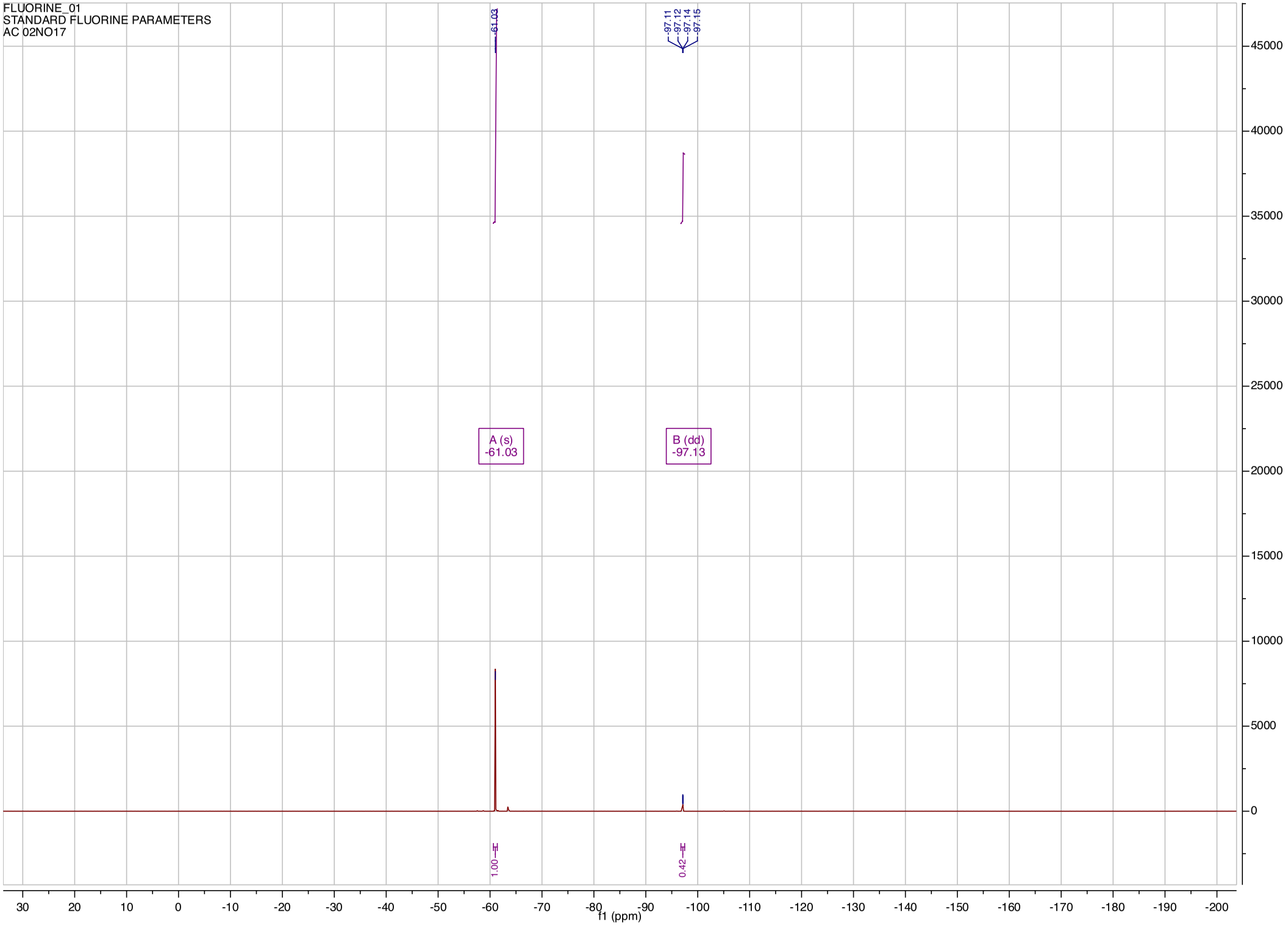

325.0638
324.8090
325.3190
324.0675
326.0670
327.0678
328.0184
329.3204
331.0359
332.1045
Observed Data
NL:
3.11E6
SUSSPE074-PR-HASP#45-59
RT: 1.24-1.64 AV: 15 T: FTMS
+ p APCI corona Full ms
[100.00-800.00]
325.0638
324.0674
326.0674
327.0689
328.0718
329.0736
330.0760
331.0781
332.0808
Theoretical Isotope Model: [M + H]+
NL:
1.61E4
C12 H10 BFN2 O7 H:
C12 H11 B1 F1 N2 O7
p (gss, s /p:40) Chrg 1
R: 100000 Res .Pwr . @FWHM
Relative Abundance
m/z
0
10
20
30
40
50
60
70
80
90
100
324
325
326
327
328
329
330
331
332

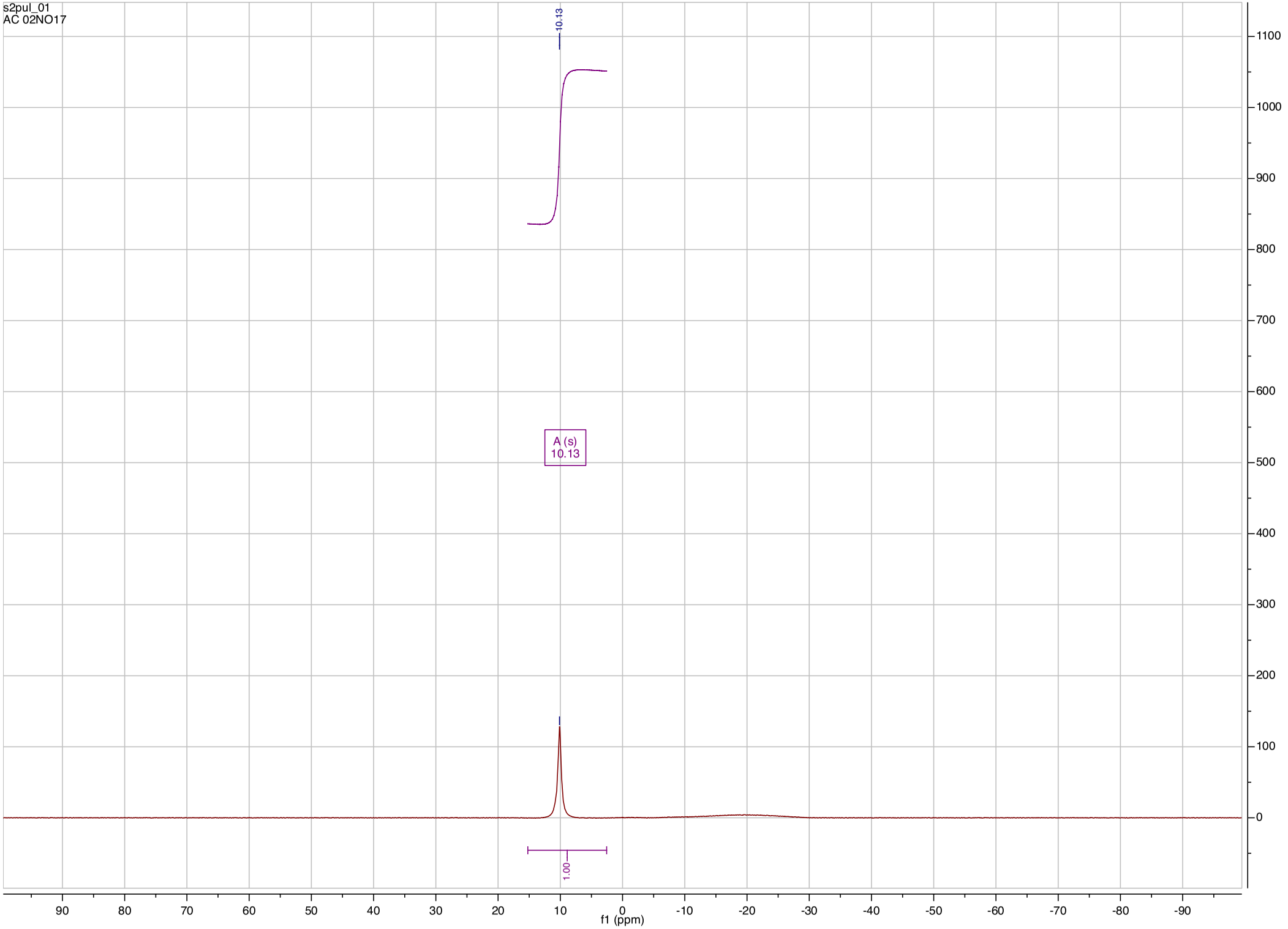
2-Fluoro-5-nitro-4-(trifluoromethyl)phenyl MIDA boronate 2d




PROTON_01
AC 02NO17
8.21
8.19
7.66
7.64
4.22
4.18
4.03
3.99
2.72
1.94 CD3CN
1 (d)
8.20
4 (d)
7.65
15 (d)
4.20
12 (d)
4.01
17 (s)
2.72
0.67
0.81
1.99
2.06
3.00
f1 (ppm)


CARBON_01
AC 02NO17
168.83
168.76
166.84
144.97
134.07
133.98
127.80
127.72
127.53
127.45
127.25
127.17
126.97
126.89
125.91
125.90
123.74
123.73
121.57
121.56
119.41
119.39
117.35
117.30
117.26
117.21
117.09
117.04
117.00
116.96
63.90
63.88
48.83
48.82
1.32 CD3CN
11,14 (s)
168.76
1 (d)
134.03
22 (m)
122.65
3 (d)
167.83
6 (s)
144.97
5 (qd)
127.35
4 (dq)
117.15
12,15 (d)
63.89
17 (d)
48.82
0.63
0.37
0.30
1.22
0.29
0.40
1.35
2.89
1.39
f1 (ppm)


FLUORINE_01
STANDARD FLUORINE PARAMETERS
AC 02NO17
-61.03
-97.11
-97.12
-97.14
-97.15
A (s)
-61.03
B (dd)
-97.13
1.00
0.42
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)

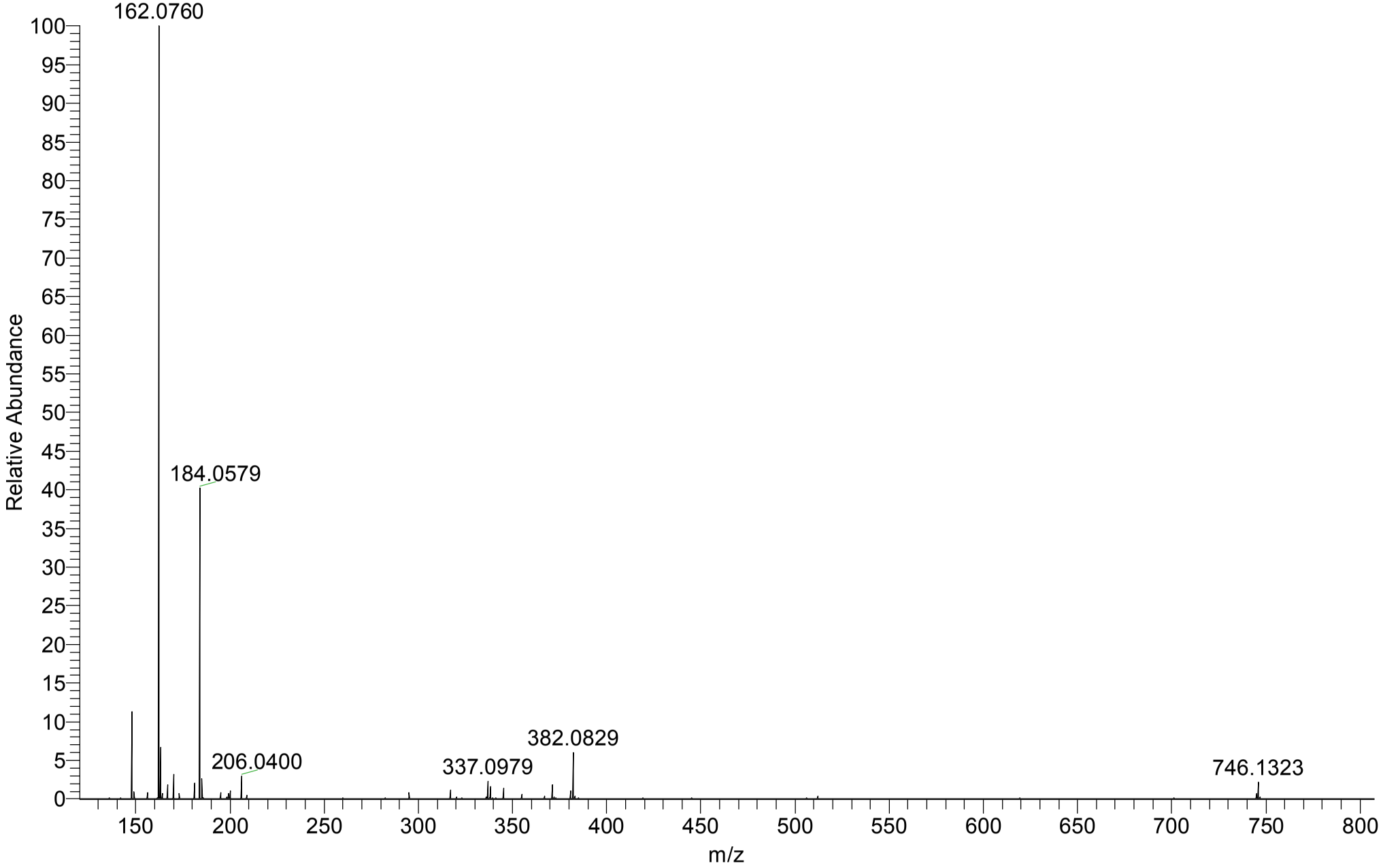

s2pul_01
AC 02NO17
10.13
A (s)
10.13
1.00
1100
1000
900
800
700
600
500
400
300
200
100
0
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
f1 (ppm)

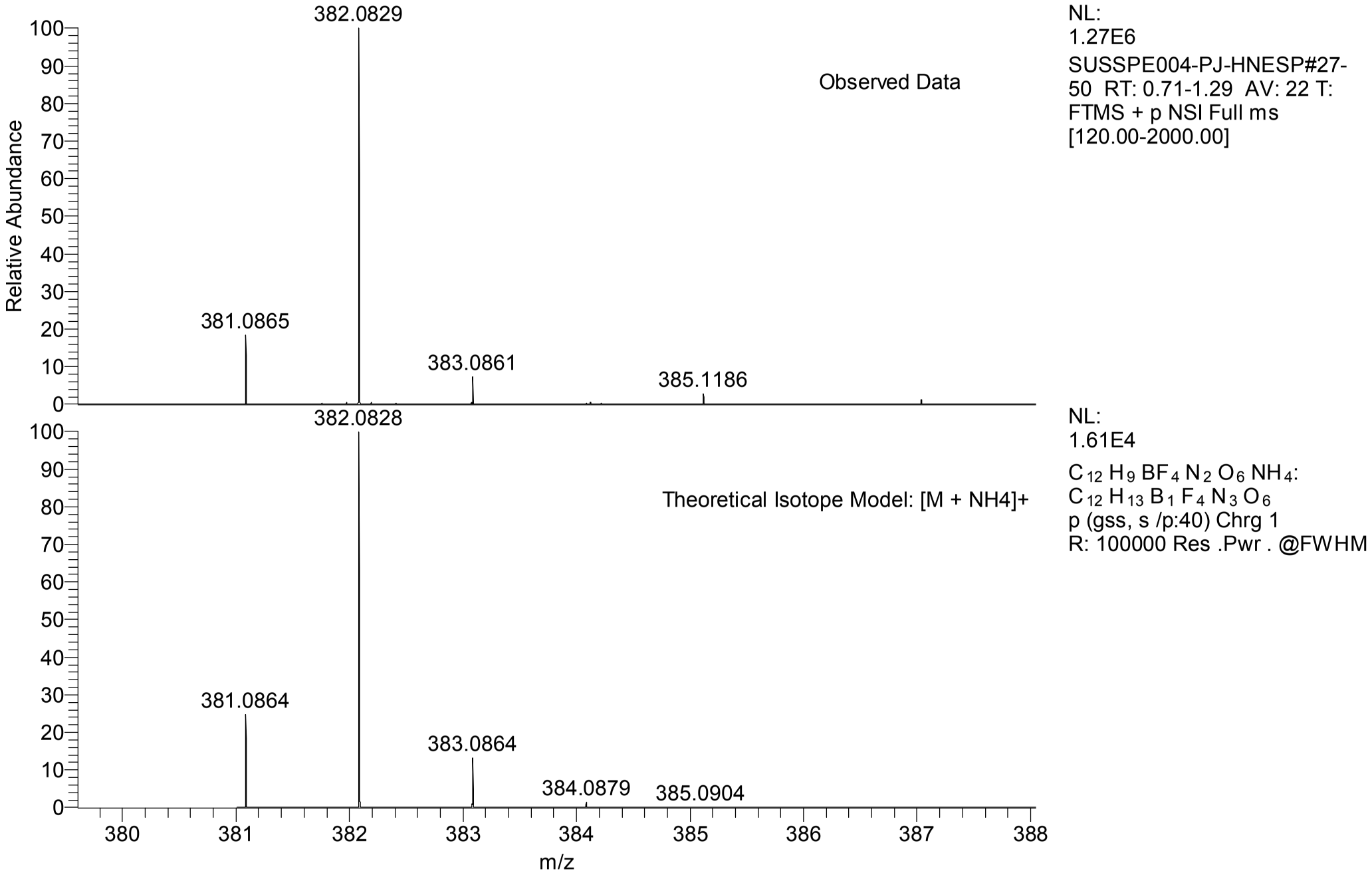
AC_02NO17 MW=364?
(MeOH)/MeOH + NH4OAc

EPSRC National Centre Swansea
LTQ Orbitrap XL

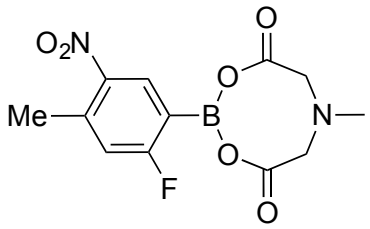
Adam Close
12/02/2013 12:48:47

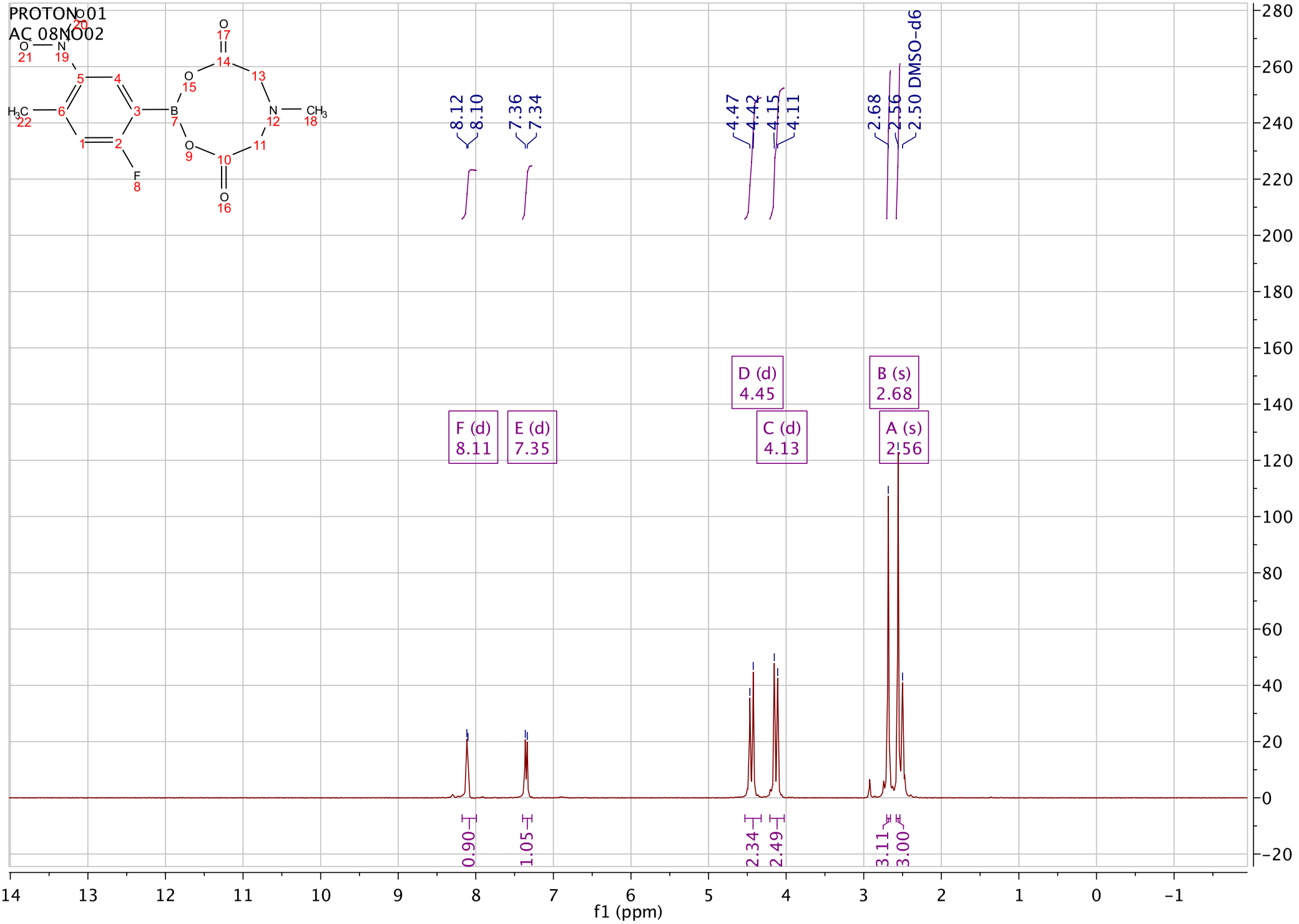
SUSSPE004-PJ-HNESP #27-50 RT: 0.71-1.29 AV: 22 SM: 7G NL: 2.09E7
T: FTMS + p NSI Full ms [120.00-2000.00]

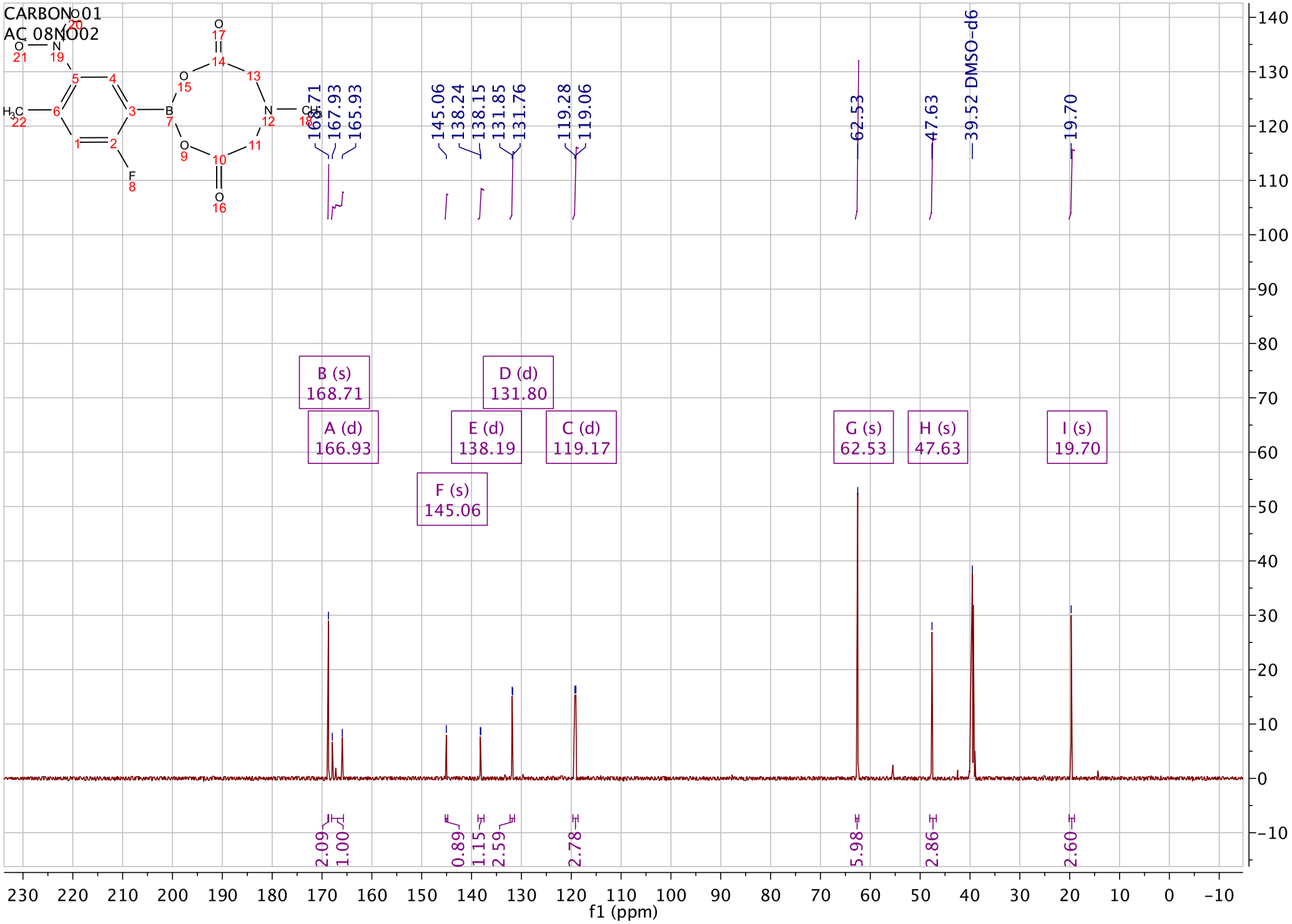


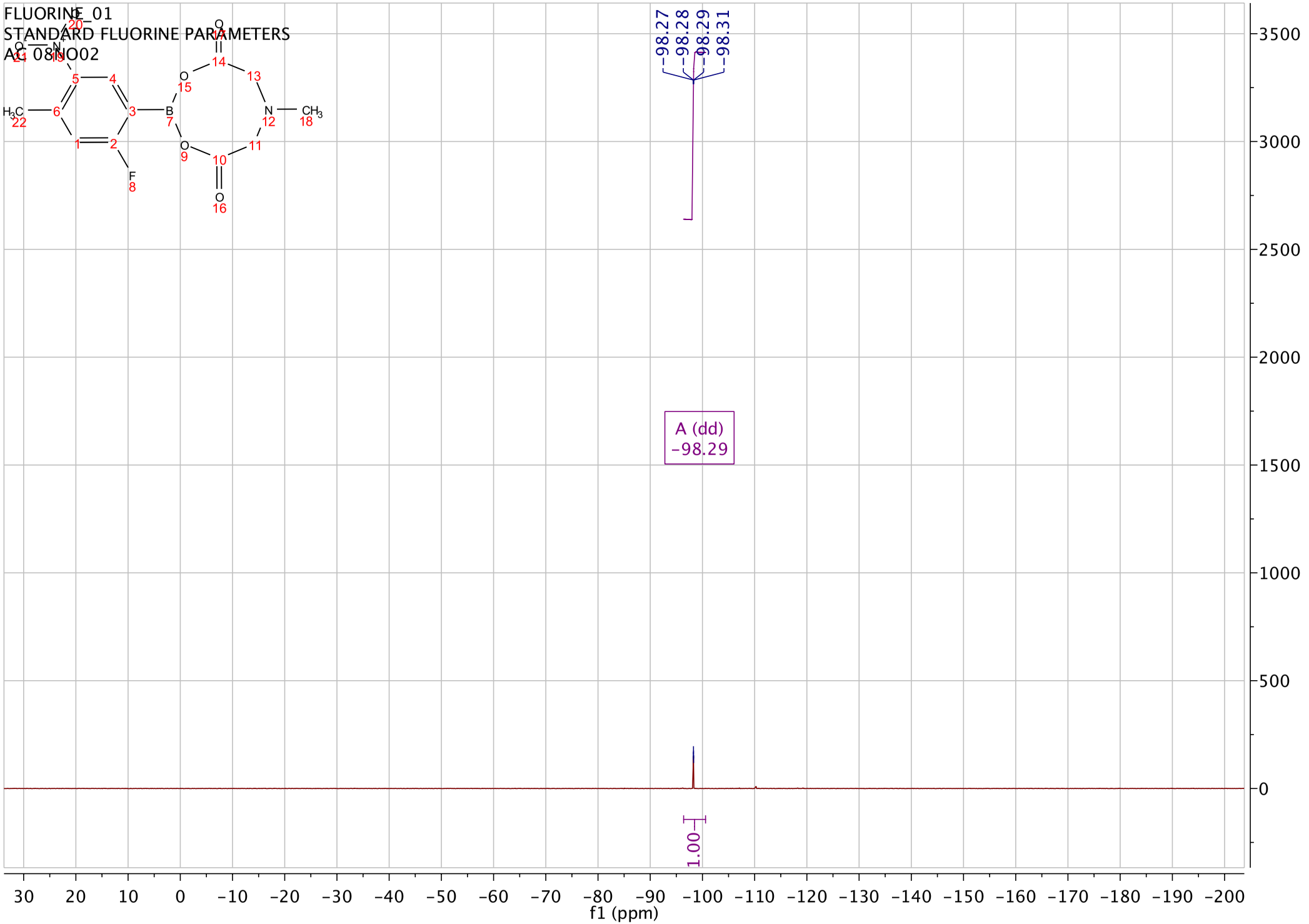

382.0829
381.0865
383.0861
385.1186
Observed Data
NL:
1.27E6
SUSSPE004-PJ-HNESP#27-50 RT: 0.71-1.29 AV: 22 T: FTMS + p NSI Full ms [120.00-2000.00]
382.0828
381.0864
383.0864
384.0879
385.0904
Theoretical Isotope Model: [M + NH4]+
NL:
1.61E4
C12 H9 BF4 N2 O6 NH4:
C12 H13 B1 F4 N3 O6
p (gss, s /p:40) Chrg 1
R: 100000 Res .Pwr . @FWHM
Relative Abundance
m/z
100
90
80
70
60
50
40
30
20
10
0
380
381
382
383
384
385
386
387
388

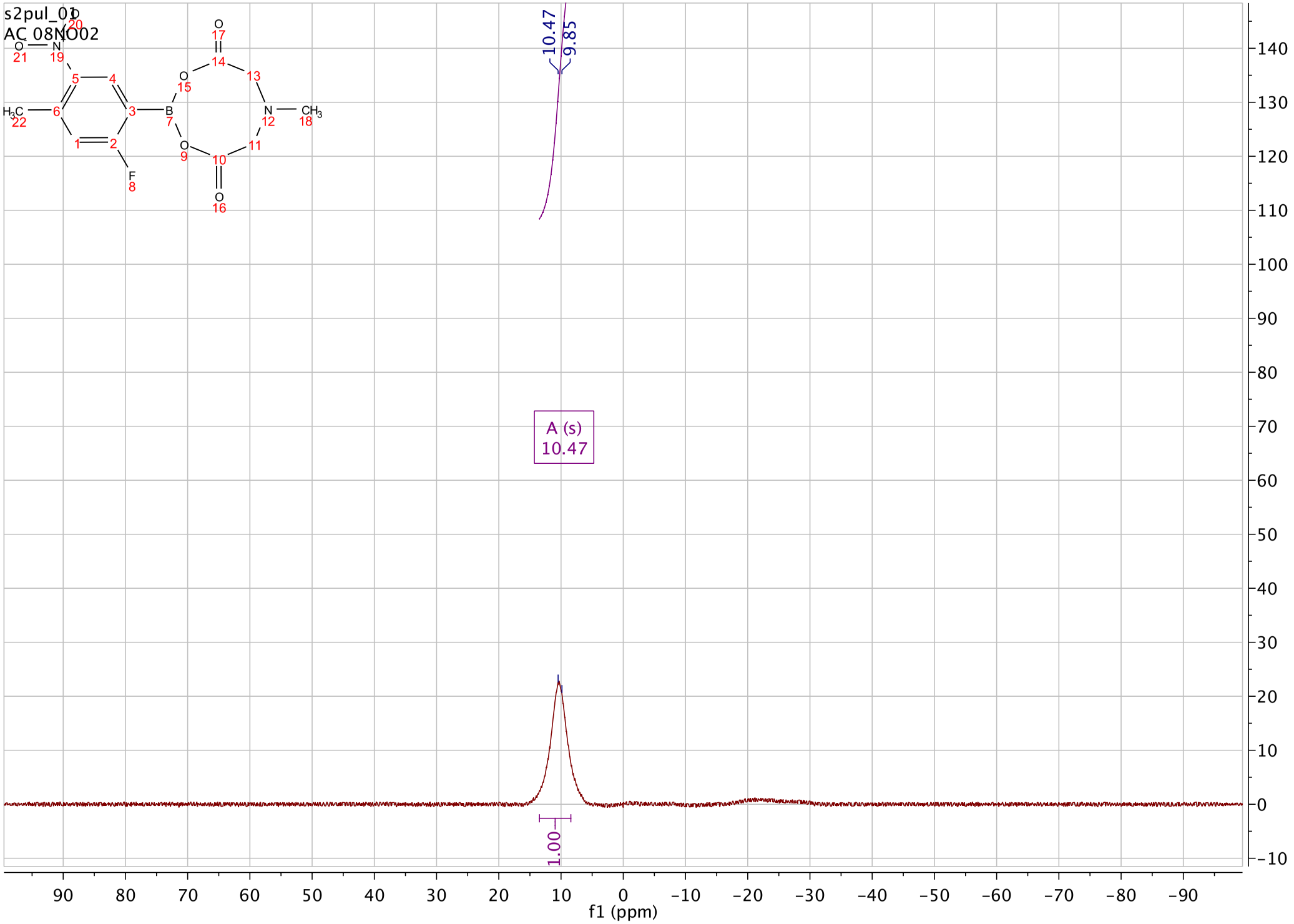
2-Fluoro-4-methyl-5-nitrophenyl MIDA boronate 2e




PROTON-01
AC-08NO02
8.12
8.10
7.36
7.34
4.47
4.42
4.15
4.11
2.68
2.56
2.50 DMSO-d6
F (d)
8.11
E (d)
7.35
D (d)
4.45
C (d)
4.13
B (s)
2.68
A (s)
2.56
0.90
1.05
2.34
2.49
3.11
3.00
f1 (ppm)


CARBON 01
AC 08NO02
168.71
167.93
165.93
145.06
138.24
138.15
131.85
131.76
119.28
119.06
62.53
47.63
39.52 DMSO-d6
19.70
B (s)
168.71
A (d)
166.93
F (s)
145.06
E (d)
138.19
D (d)
131.80
C (d)
119.17
G (s)
62.53
H (s)
47.63
I (s)
19.70
2.09
1.00
0.89
1.15
2.59
2.78
5.98
2.86
2.60
f1 (ppm)


FLUORINE_01
STANDARD FLUORINE PARAMETERS
AG 08NO02
-98.27
-98.28
-98.29
-98.31
A (dd)
-98.29
1.00
f1 (ppm)

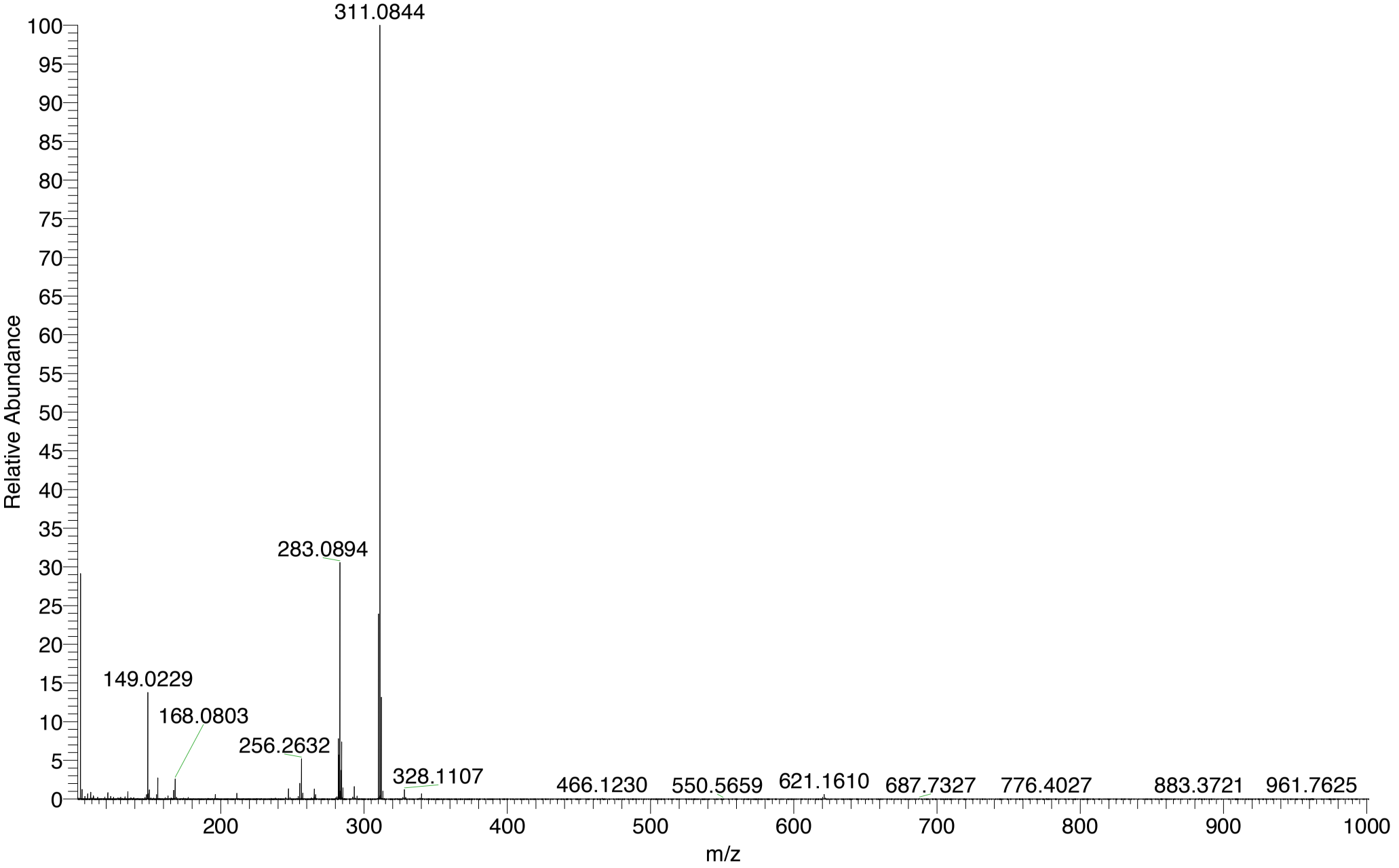

s2pul_01
AC 08NO02
10.47
9.85
A (s)
10.47
1.00
f1 (ppm)

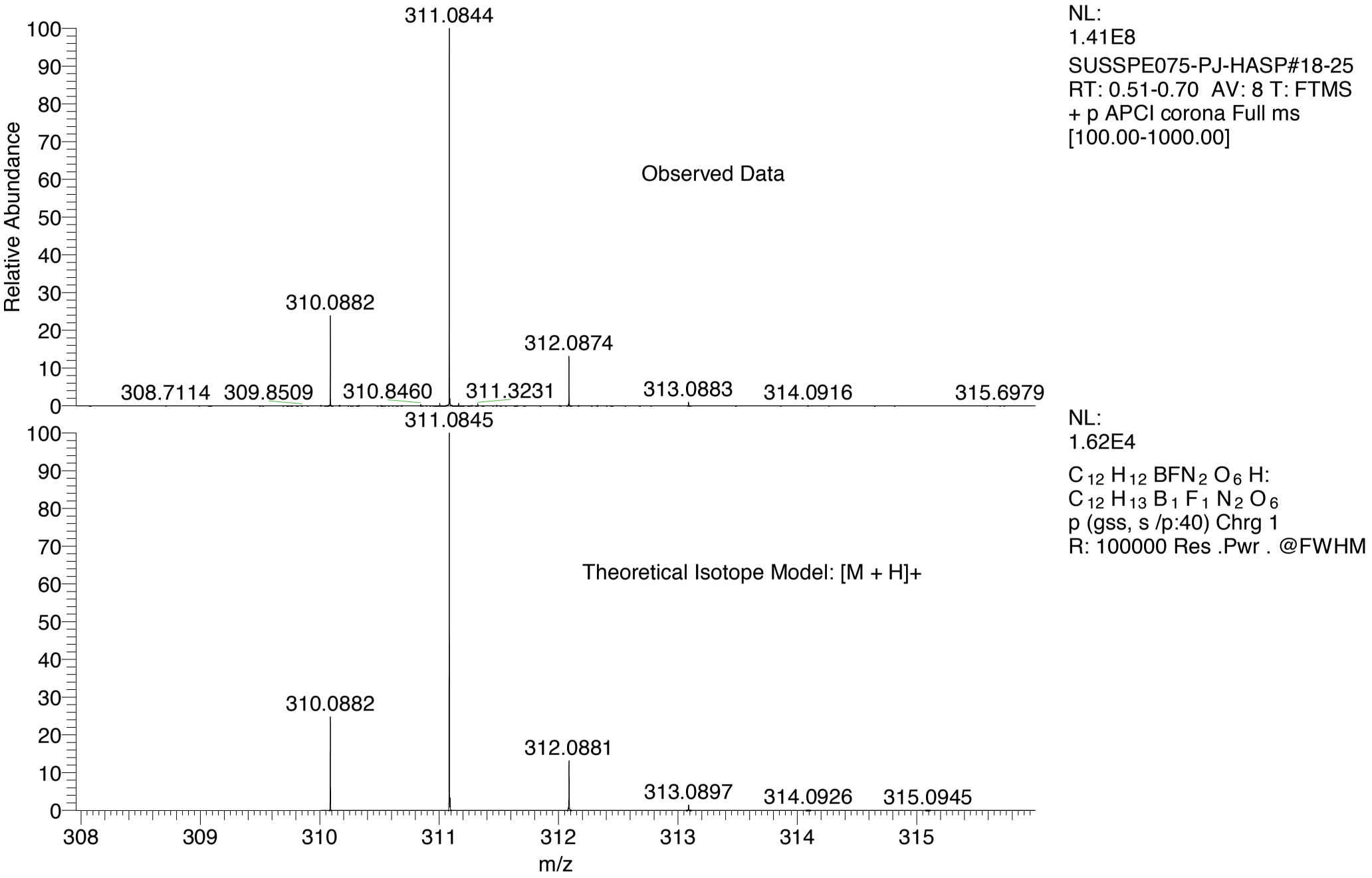
08NO02 MW=310?
ASAP (SOLID)

EPSRC National Facility Swansea
LTQ Orbitrap XL

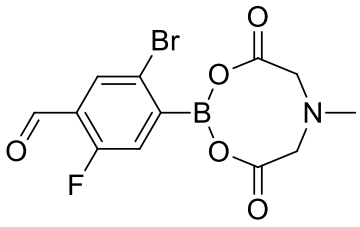
Adam Close
17/09/2015 01:52:03 PM

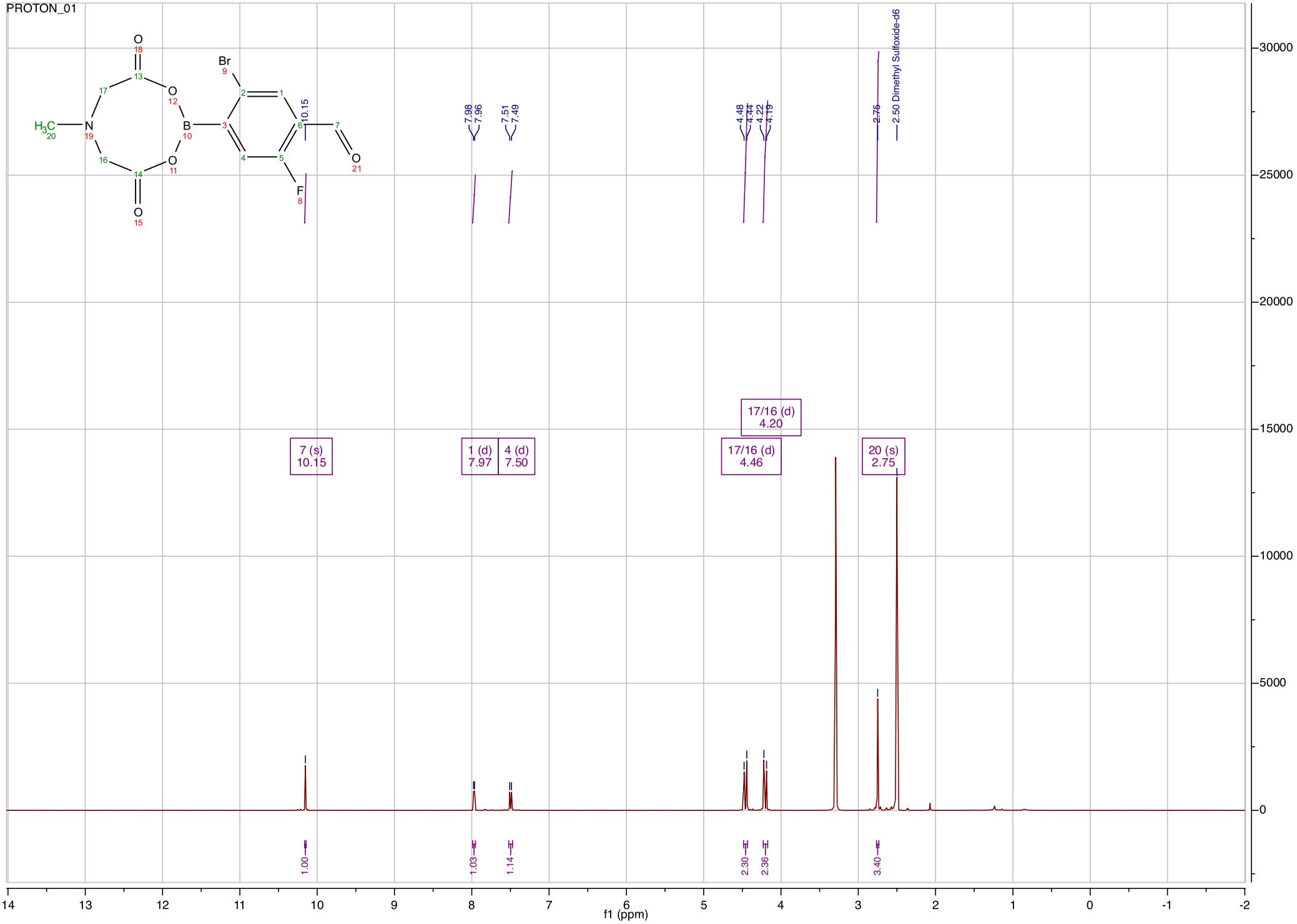
SUSSPE075-PJ-HASP #18-25 RT: 0.51-0.70 AV: 8 NL: 1.41E8
T: FTMS + p APCI corona Full ms [100.00-1000.00]




311.0844
310.0882
312.0874
308.7114
309.8509
310.8460
311.3231
313.0883
314.0916
315.6979
Observed Data
NL:
1.41E8
SUSSPE075-PJ-HASP#18-25
RT: 0.51-0.70 AV: 8 T: FTMS
+ p APCI corona Full ms
[100.00-1000.00]
311.0845
310.0882
312.0881
313.0897
314.0926
315.0945
Theoretical Isotope Model: [M + H]+
NL:
1.62E4
C12 H12 BFN2 O6 H:
C12 H13 B1 F1 N2 O6
p (gss, s /p:40) Chrg 1
R: 100000 Res .Pwr . @FWHM
Relative Abundance
m/z

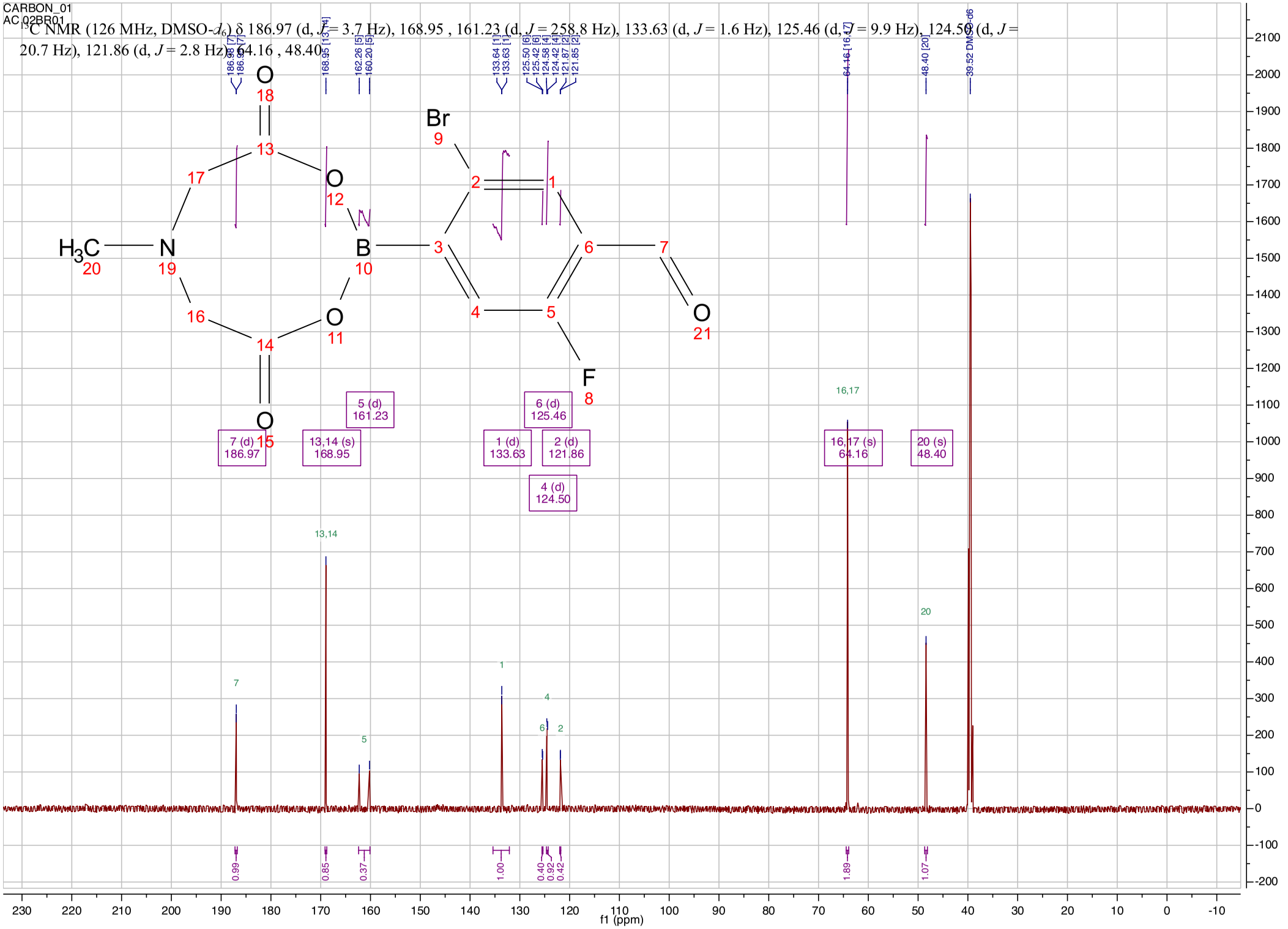
2-Bromo-5-fluoro-4-formylphenyl MIDA boronate 3b



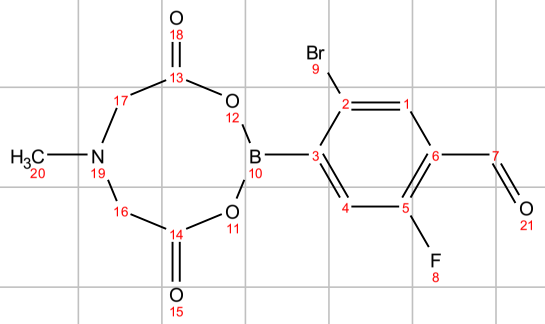

PROTON_01
7 (s)
10.15
1 (d)
7.97
4 (d)
7.50
17/16 (d)
4.20
17/16 (d)
4.46
20 (s)
2.75
2.50 Dimethyl Sulfoxide-d6
f1 (ppm)

CARBON_01
AC_02BR01

^{13}C NMR (126 MHz, DMSO-d_6) δ 186.97 (d, J = 3.7 Hz), 168.95 , 161.23 (d, J = 258.8 Hz), 133.63 (d, J = 1.6 Hz), 125.46 (d, J = 9.9 Hz), 124.50 (d, J = 20.7 Hz), 121.86 (d, J = 2.8 Hz), 64.16 , 48.40 .



FLUORINE_01
STANDARD FLUORINE PARAMETERS

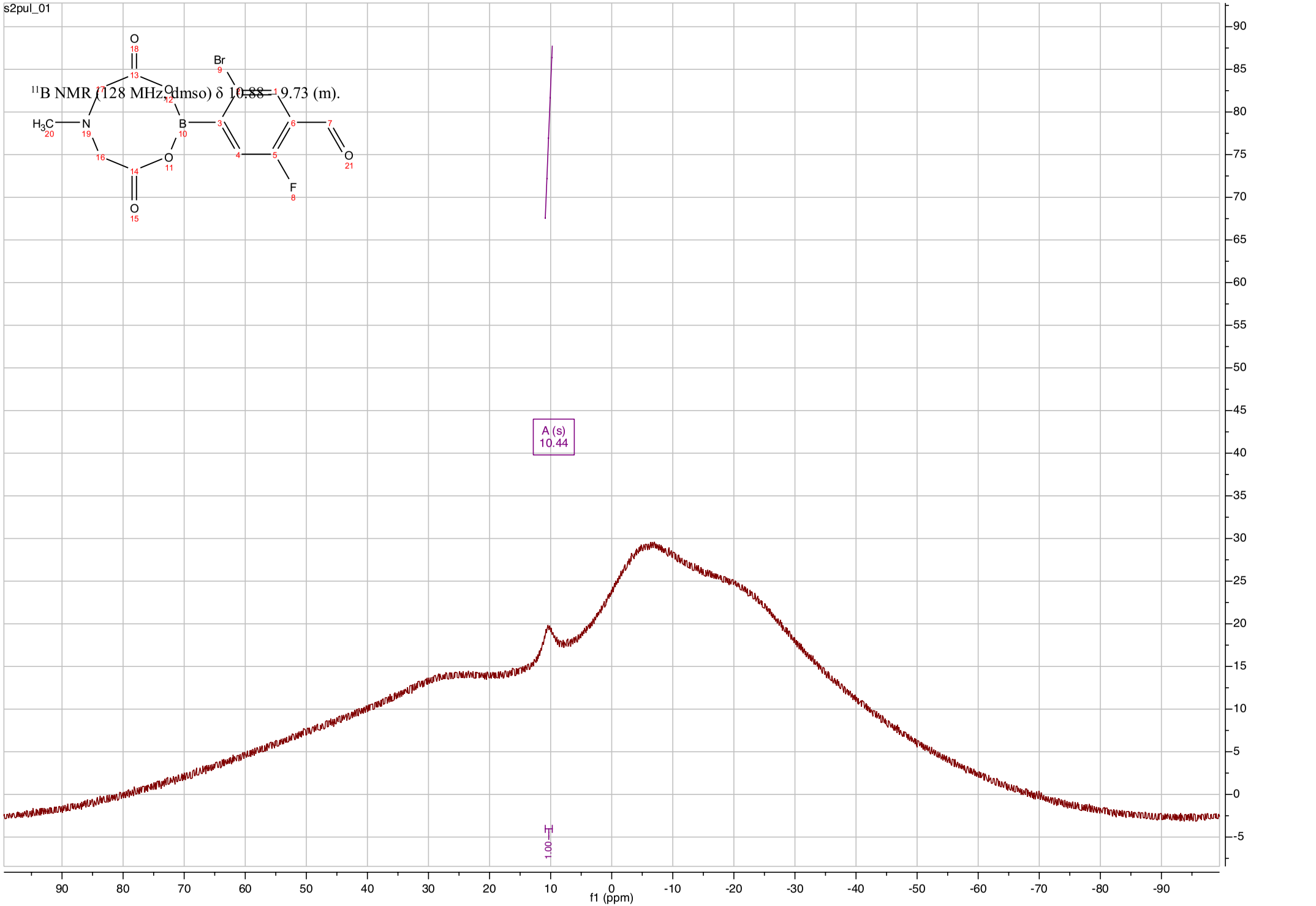


-123.34
-123.35
-123.37
-123.38

A (dd)
-123.36

1.00

f1 (ppm)



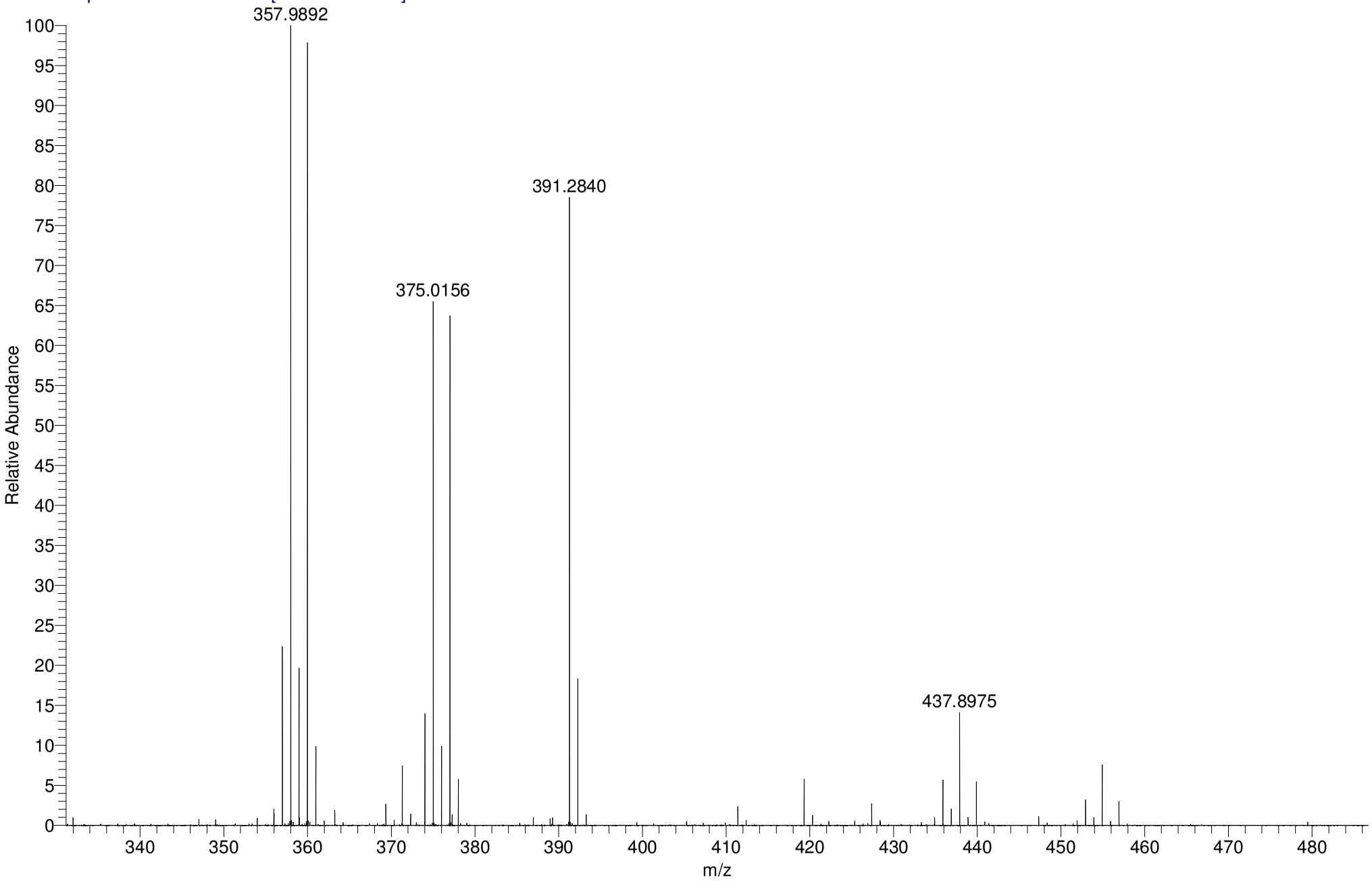
^{11}B NMR (128 MHz, dmso) δ 10.88 – 9.73 (m).

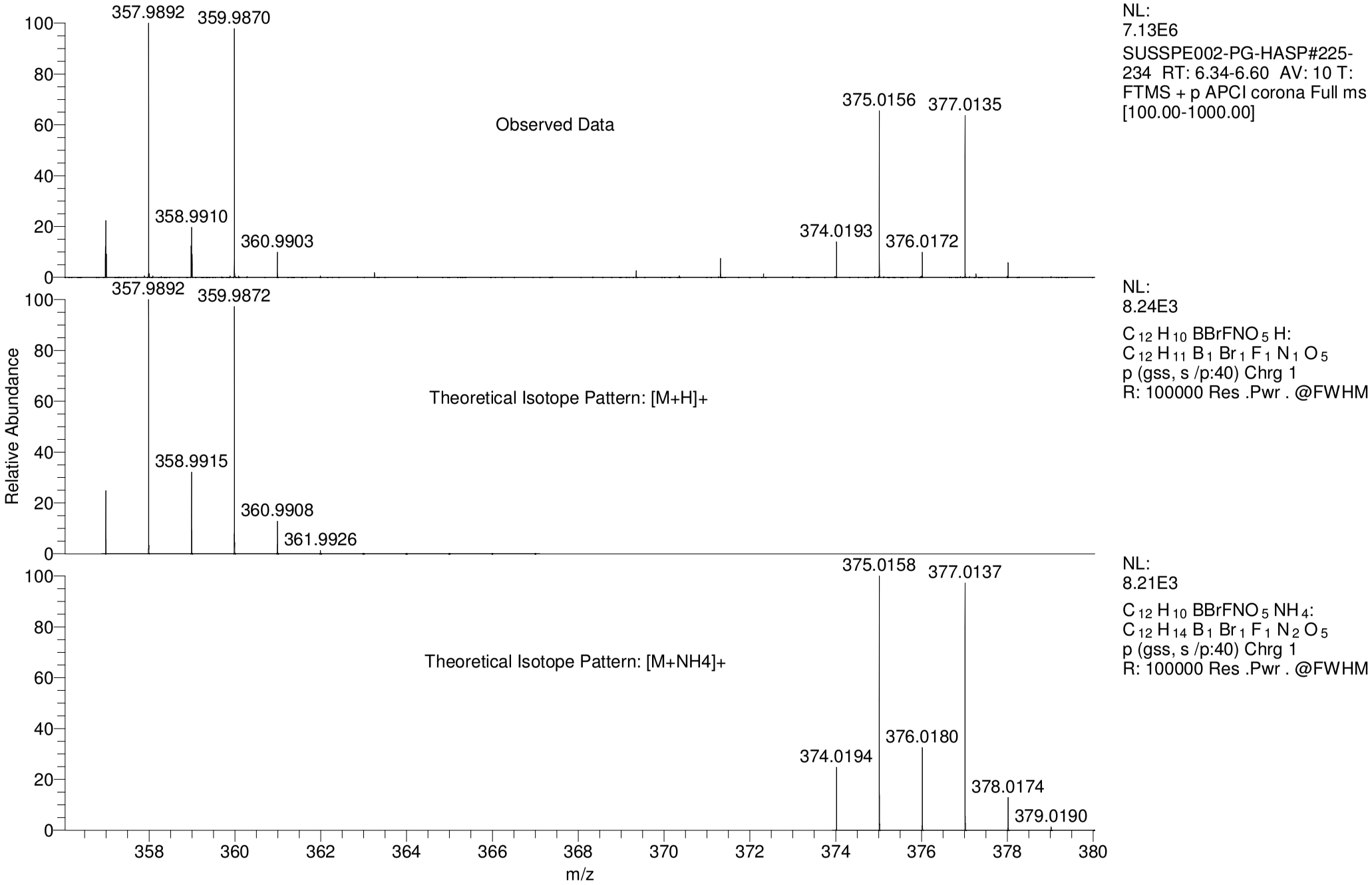
127348 MW=357?
ASAP(SOLID)

EPSRC National Centre Swansea
LTQ Orbitrap XL

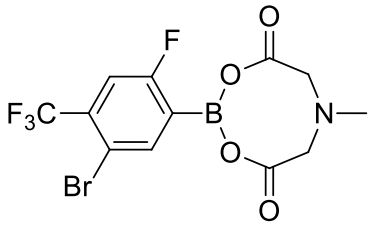
Dr Spencer
03/01/2013 12:29:16

SUSSPE002-PG-HASP #225-234 RT: 6.34-6.60 AV: 10 SM: 7G NL: 7.13E6
T: FTMS + p APCI corona Full ms [100.00-1000.00]



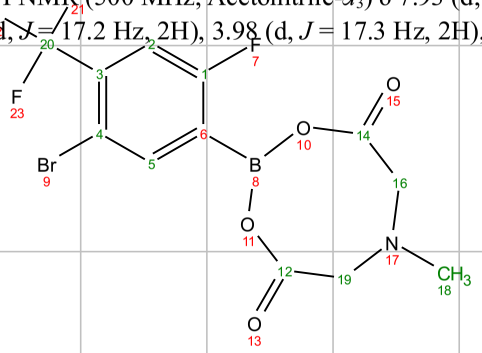

Observed Data
357.9892
359.9870
358.9910
360.9903
375.0156
377.0135
374.0193
376.0172
NL:
7.13E6
SUSSPE002-PG-HASP#225-234 RT: 6.34-6.60 AV: 10 T: FTMS + p APCI corona Full ms [100.00-1000.00]
Theoretical Isotope Pattern: [M+H]+
357.9892
359.9872
358.9915
360.9908
361.9926
NL:
8.24E3
C12 H10 BBrFNO5 H:
C12 H11 B1 Br1 F1 N1 O5
p (gss, s /p:40) Chrg 1
R: 100000 Res .Pwr . @FWHM
Theoretical Isotope Pattern: [M+NH4]+
375.0158
377.0137
374.0194
376.0180
378.0174
379.0190
NL:
8.21E3
C12 H10 BBrFNO5 NH4:
C12 H14 B1 Br1 F1 N2 O5
p (gss, s /p:40) Chrg 1
R: 100000 Res .Pwr . @FWHM
Relative Abundance
m/z
0
20
40
60
80
100
358
360
362
364
366
368
370
372
374
376
378
380

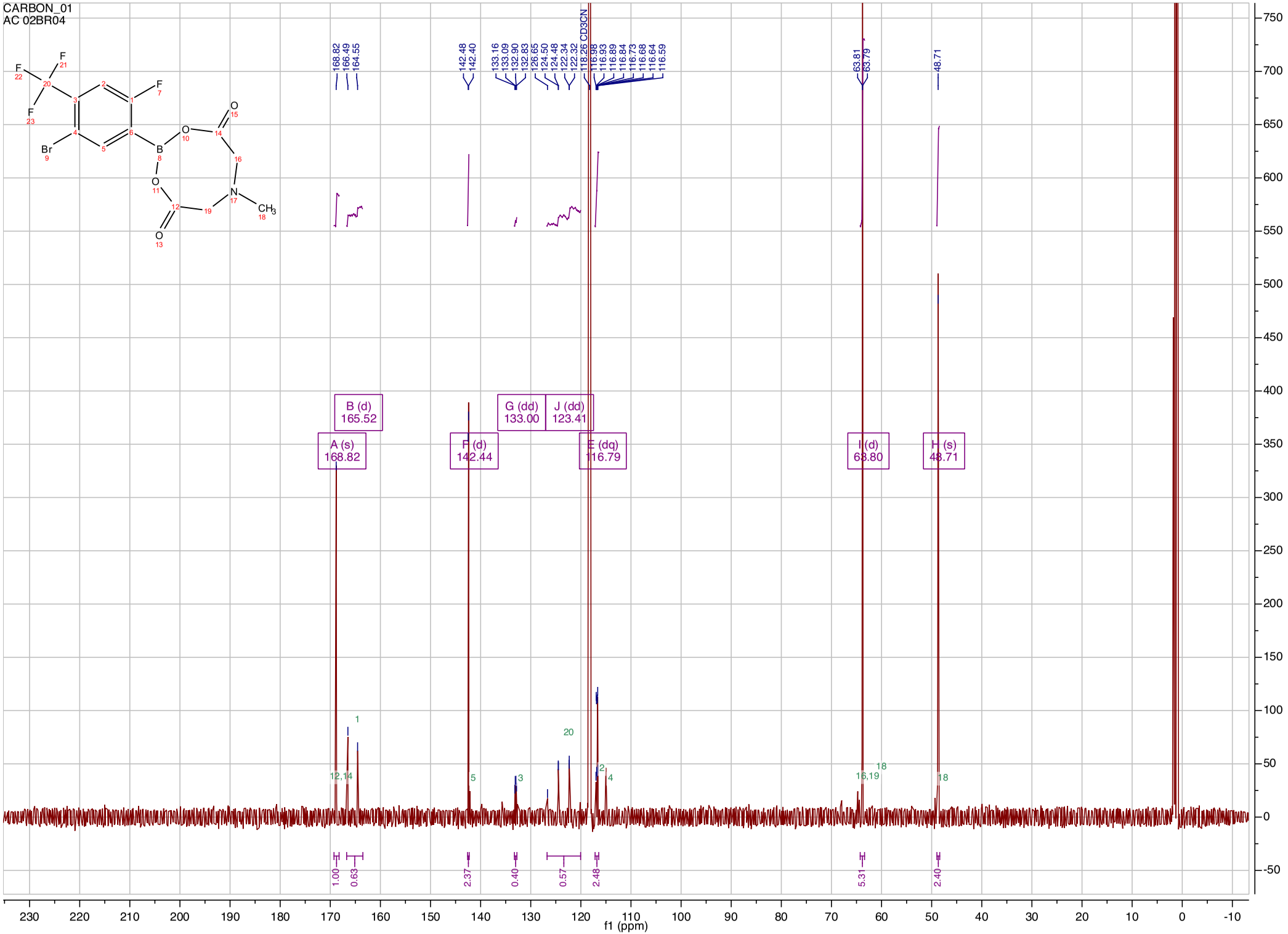
5-Bromo-2-fluoro-4-(trifluoromethyl)phenyl MIDA boronate 3d

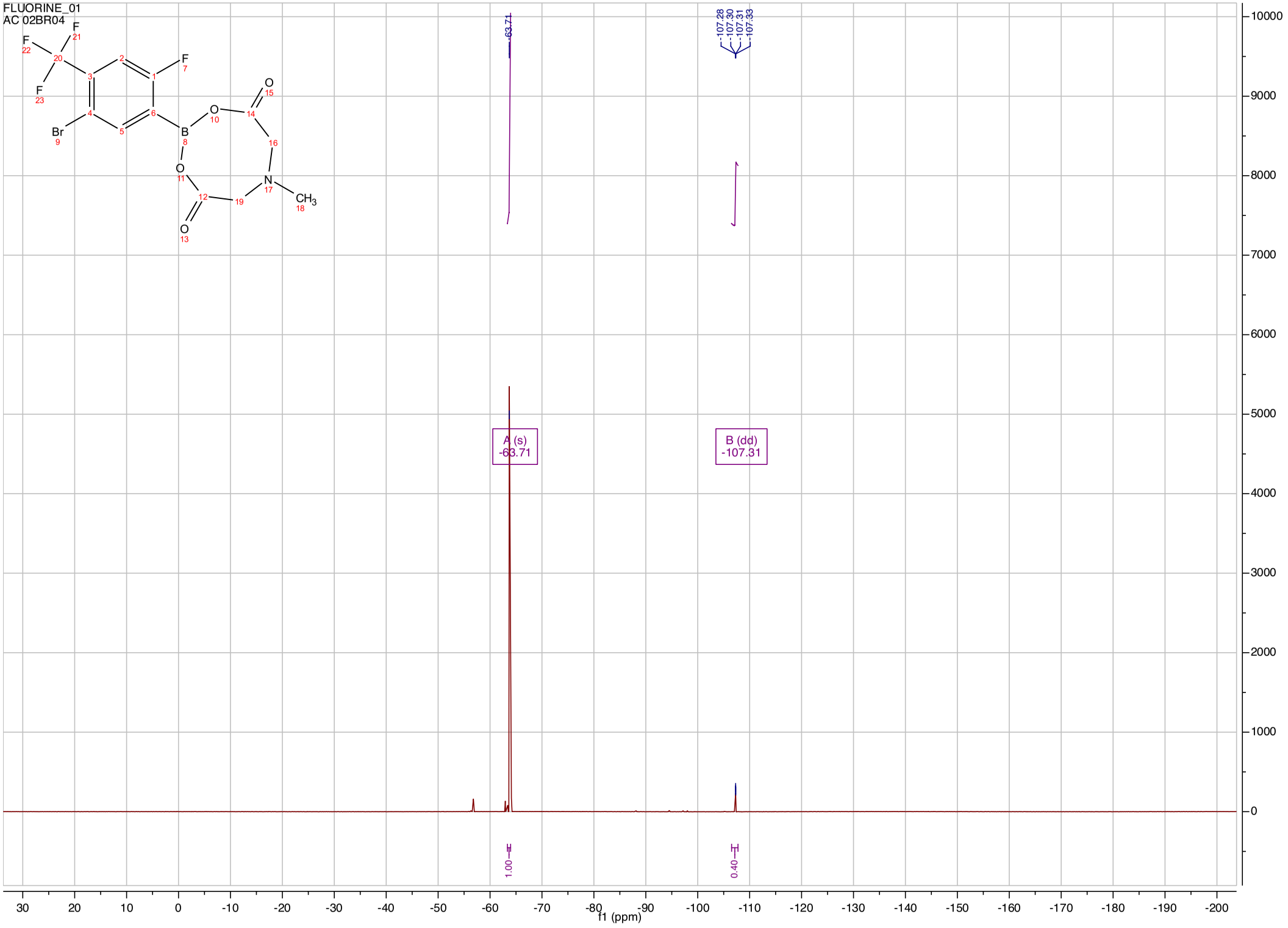


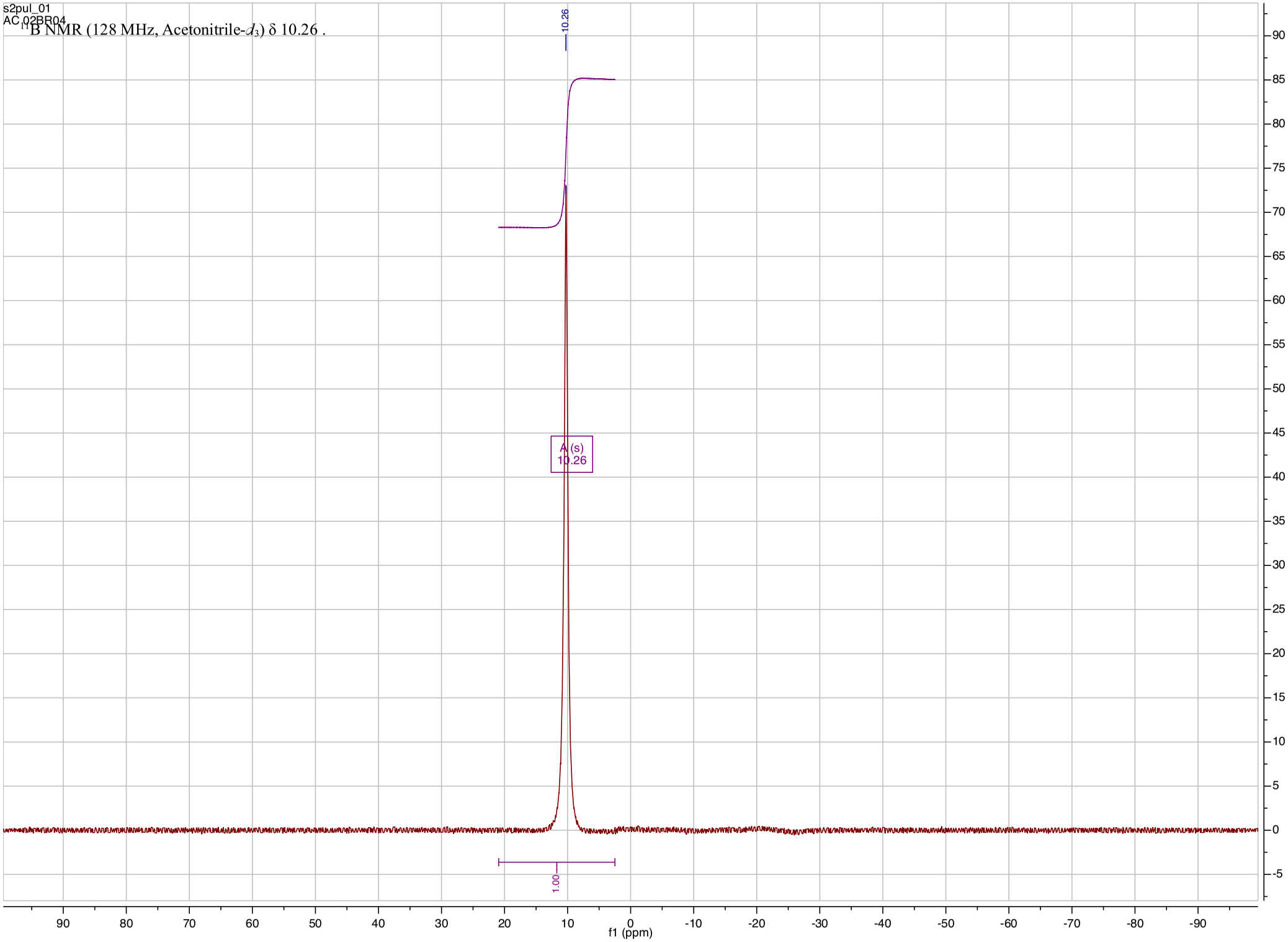
PROTON_01
AC_02BR04

^{1}H NMR (500 MHz, Acetonitrile-d_3) δ 7.93 (d, J = 5.7 Hz, 1H), 7.51 (d, J = 9.8 Hz, 1H), 4.16 (d, J = 17.2 Hz, 2H), 3.98 (d, J = 17.3 Hz, 2H), 2.70 (s, 3H).




CARBON_01
AC 02BR04
168.82
166.49
164.55
142.48
142.40
133.16
133.09
132.90
132.83
126.65
124.50
124.48
122.34
122.32
118.26 CD3CN
116.98
116.93
116.89
116.84
116.73
116.68
116.64
116.59
63.81
63.79
48.71
B (d) 165.52
A (s) 168.82
F (d) 142.44
G (dd) 133.00
J (dd) 123.41
C (dq) 116.79
I (d) 63.80
H (s) 48.71
1.00
0.63
2.37
0.40
0.57
2.48
5.31
2.40
f1 (ppm)


FLUORINE_01
AC 02BR04
-63.71
-107.28
-107.30
-107.31
-107.33
A (s)
-63.71
B (dd)
-107.31
1.00
0.40
f1 (ppm)

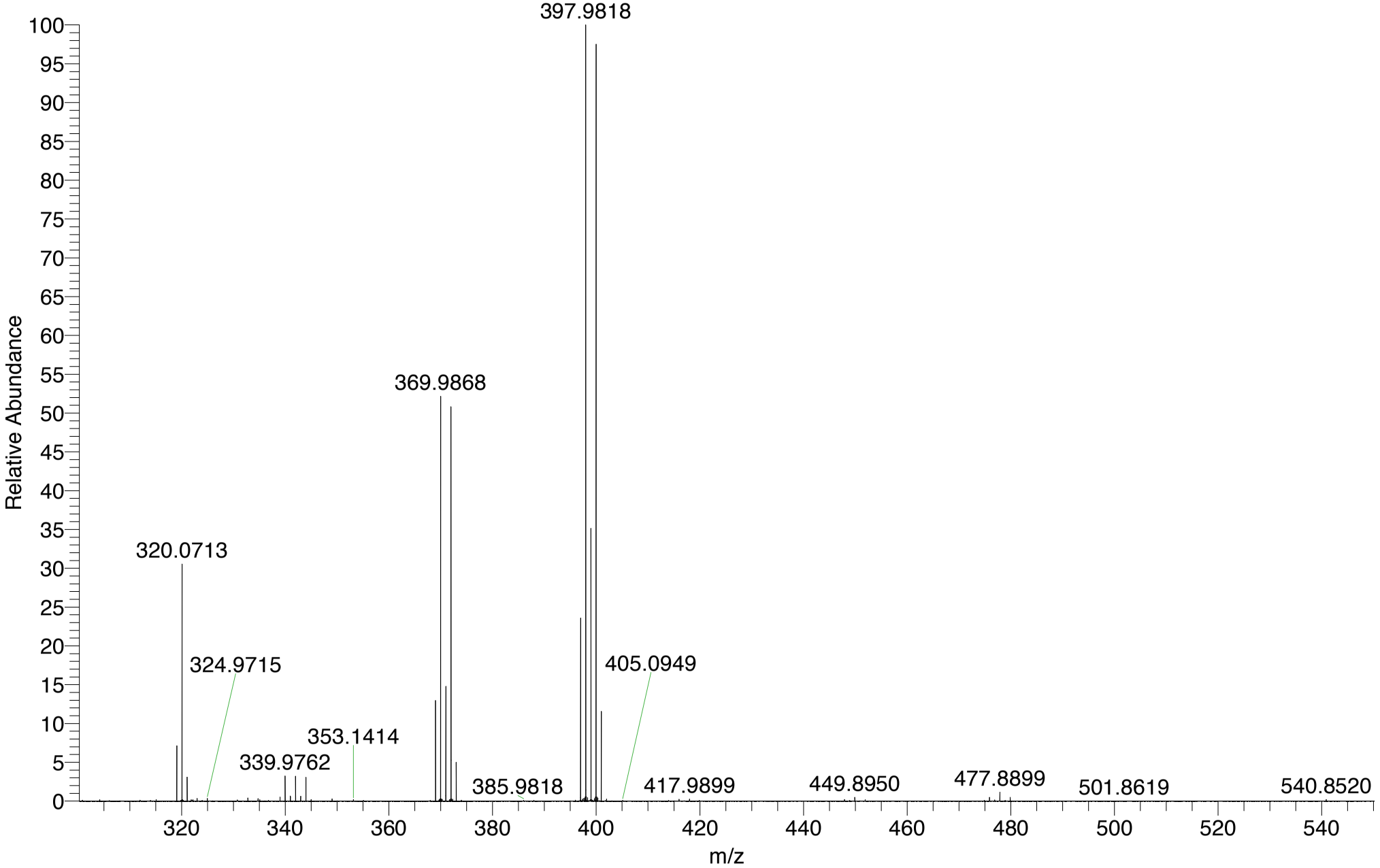

s2pul_01
AC_02BR04
B NMR (128 MHz, Acetonitrile-d_3) δ 10.26 .
10.26
A (s)
10.26
1.00
f1 (ppm)

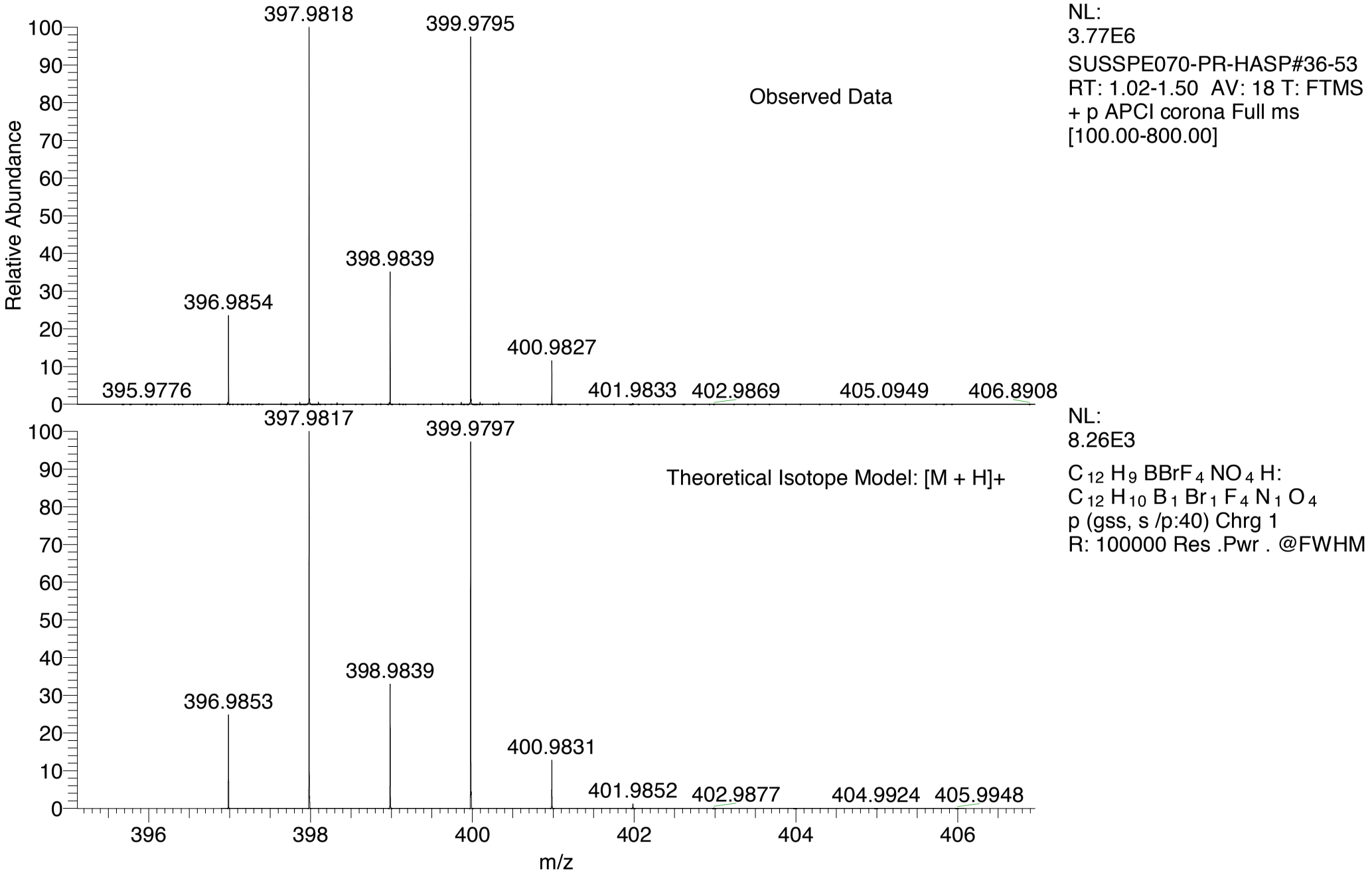
07BR MW=398?
ASAP (MeCN)

EPSRC National Facility Swansea
LTQ Orbitrap XL

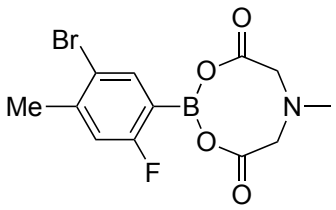
Adam
09/09/2015 03:00:58 PM

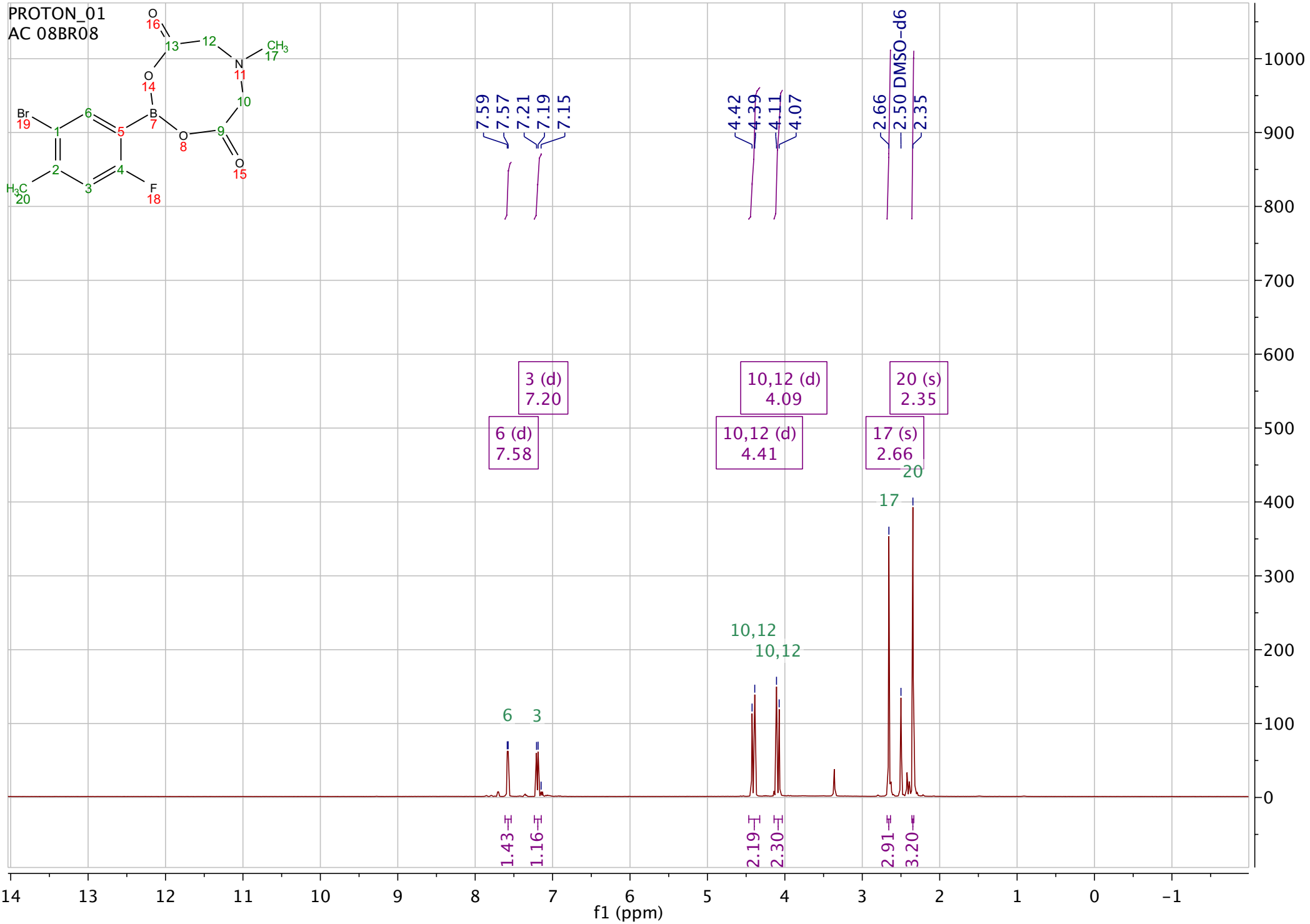
SUSSPE070-PR-HASP #36-53 RT: 1.02-1.50 AV: 18 NL: 3.77E6
T: FTMS + p APCI corona Full ms [100.00-800.00]

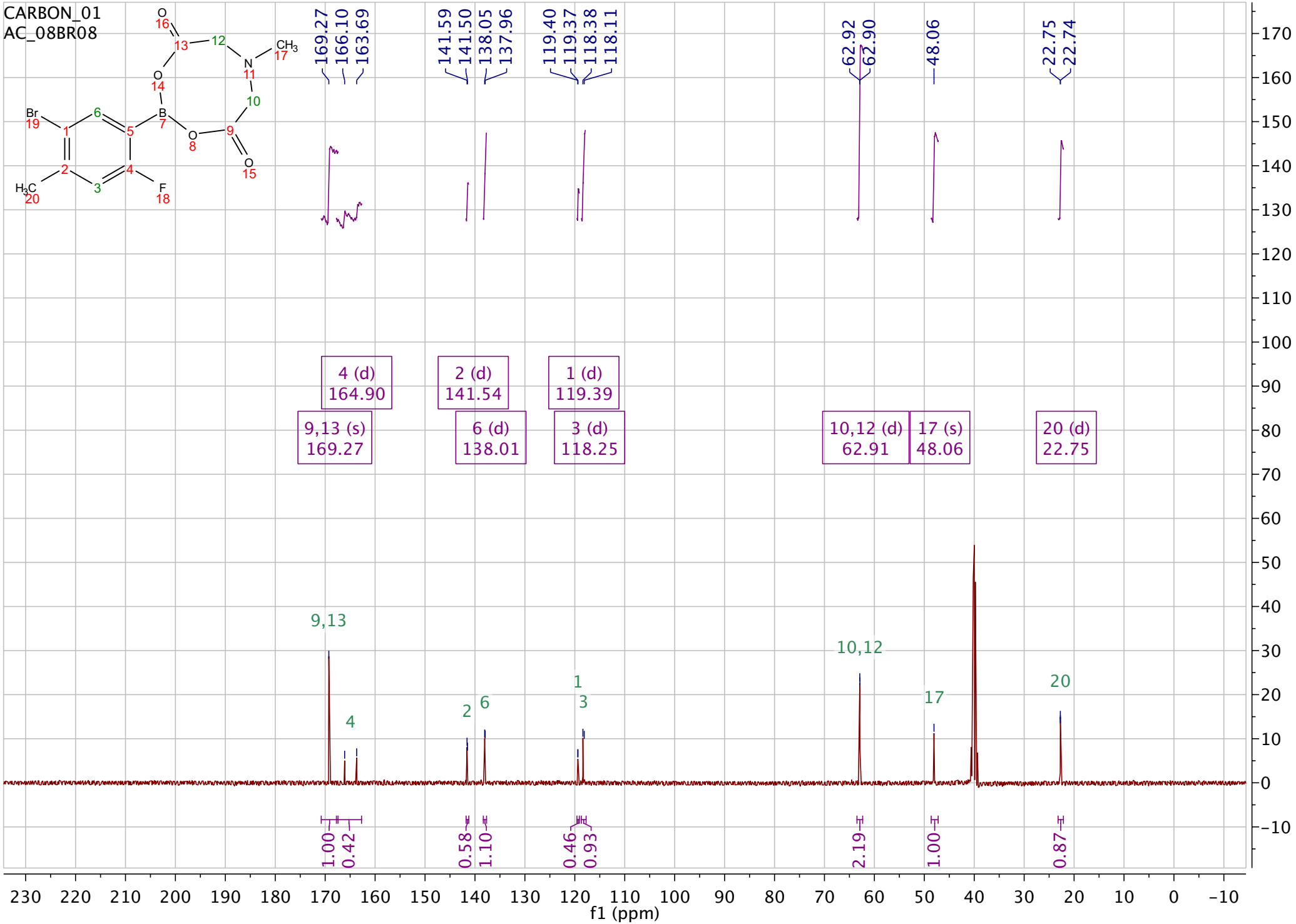



Relative Abundance
m/z
Observed Data
397.9818
399.9795
398.9839
396.9854
400.9827
395.9776
401.9833
402.9869
405.9049
406.8908
NL:
3.77E6
SUSSPE070-PR-HASP#36-53
RT: 1.02-1.50 AV: 18 T: FTMS
+ p APCI corona Full ms
[100.00-800.00]
Theoretical Isotope Model: [M + H]+
397.9817
399.9797
398.9839
396.9853
400.9831
401.9852
402.9877
404.9924
405.9948
NL:
8.26E3
C12 H9 BBrF4 NO4 H:
C12 H10 B1 Br1 F4 N1 O4
p (gss, s /p:40) Chrg 1
R: 100000 Res .Pwr . @FWHM

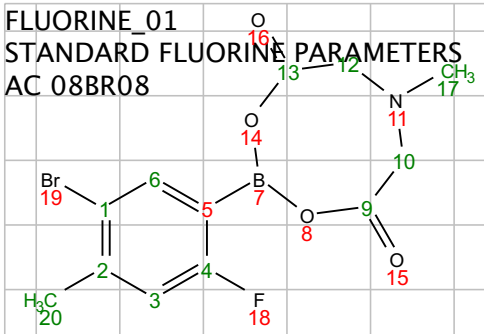
5-Bromo-2-fluoro-4-methylphenyl MIDA boronate 3e

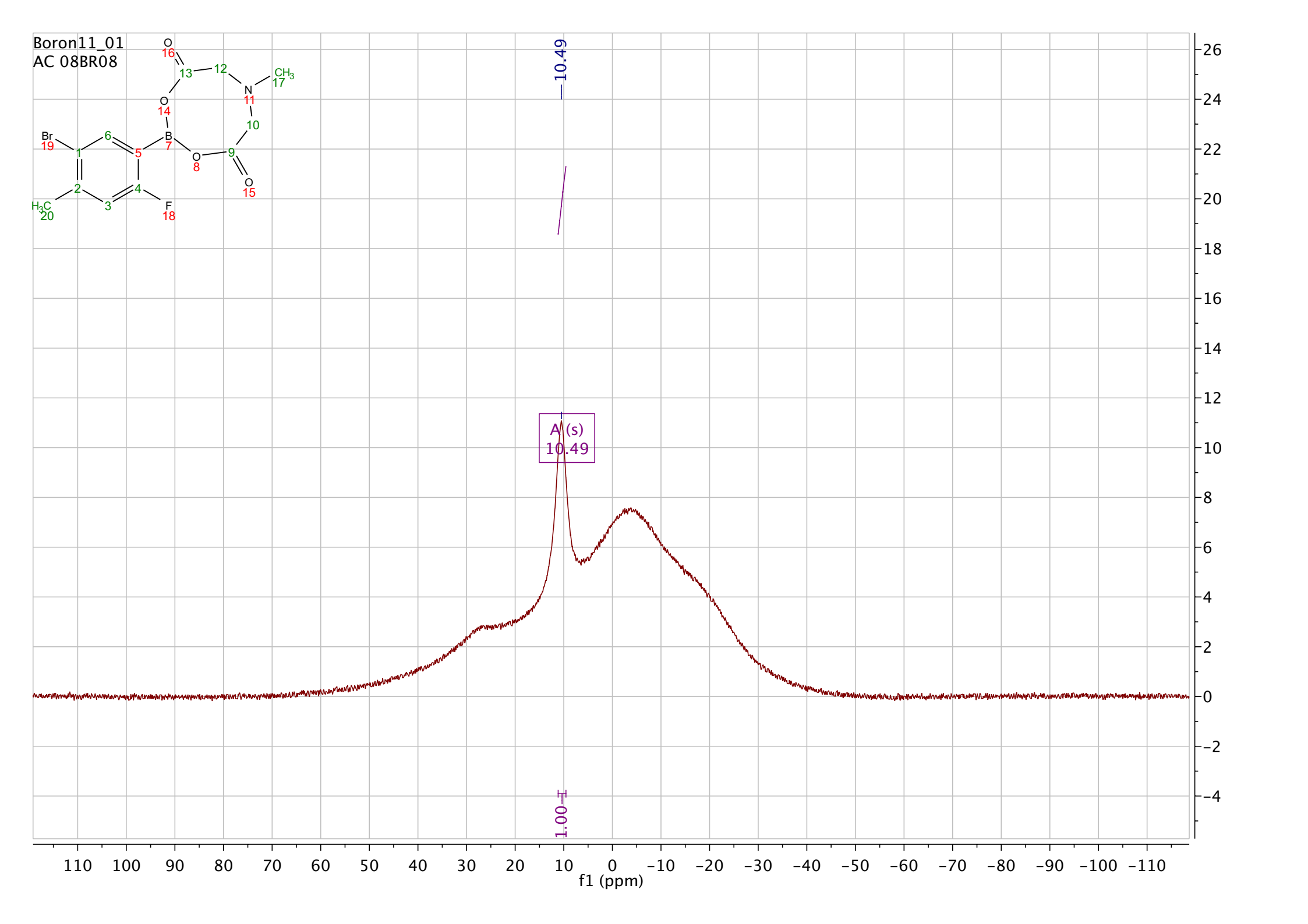



PROTON_01
AC 08BR08
7.59
7.57
7.21
7.19
7.15
4.42
4.39
4.11
4.07
2.66
2.50 DMSO-d6
2.35
3 (d)
7.20
6 (d)
7.58
10,12 (d)
4.09
10,12 (d)
4.41
20 (s)
2.35
17 (s)
2.66
20
17
10,12
10,12
6
3
1.43
1.16
2.19
2.30
2.91
3.20
f1 (ppm)


CARBON_01
AC_08BR08
169.27
166.10
163.69
141.59
141.50
138.05
137.96
119.40
119.37
118.38
118.11
62.92
62.90
48.06
22.75
22.74
4 (d)
164.90
2 (d)
141.54
1 (d)
119.39
9,13 (s)
169.27
6 (d)
138.01
3 (d)
118.25
10,12 (d)
62.91
17 (s)
48.06
20 (d)
22.75
9,13
4
2
6
1
3
10,12
17
20
1.00
0.42
0.58
1.10
0.46
0.93
2.19
1.00
0.87
f1 (ppm)

FLUORINE_01
STANDARD FLUORINE PARAMETERS
AC 08BR08



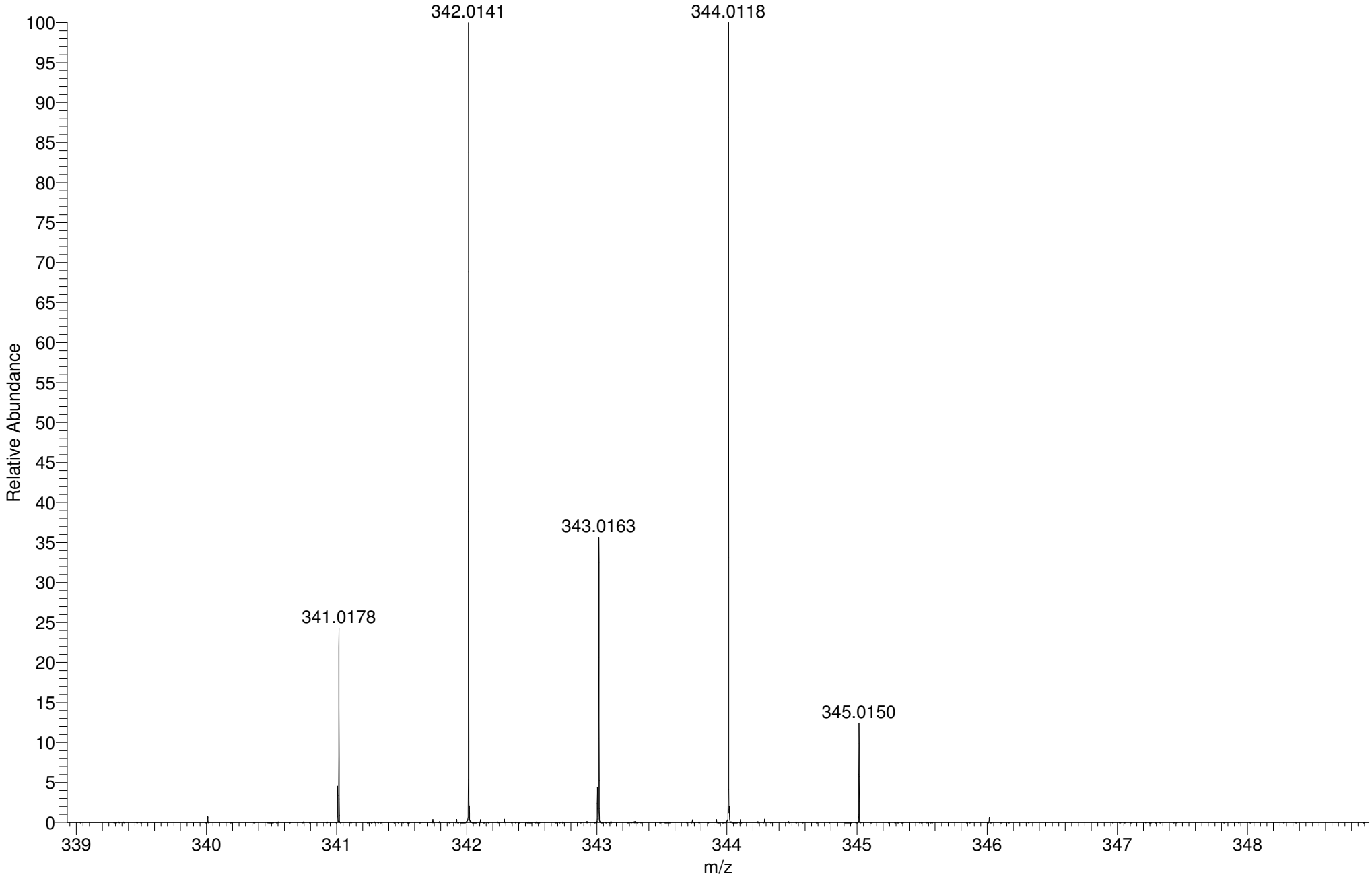

Boron11_01
AC 08BR08
10.49
A (s)
10.49
1.00
f1 (ppm)

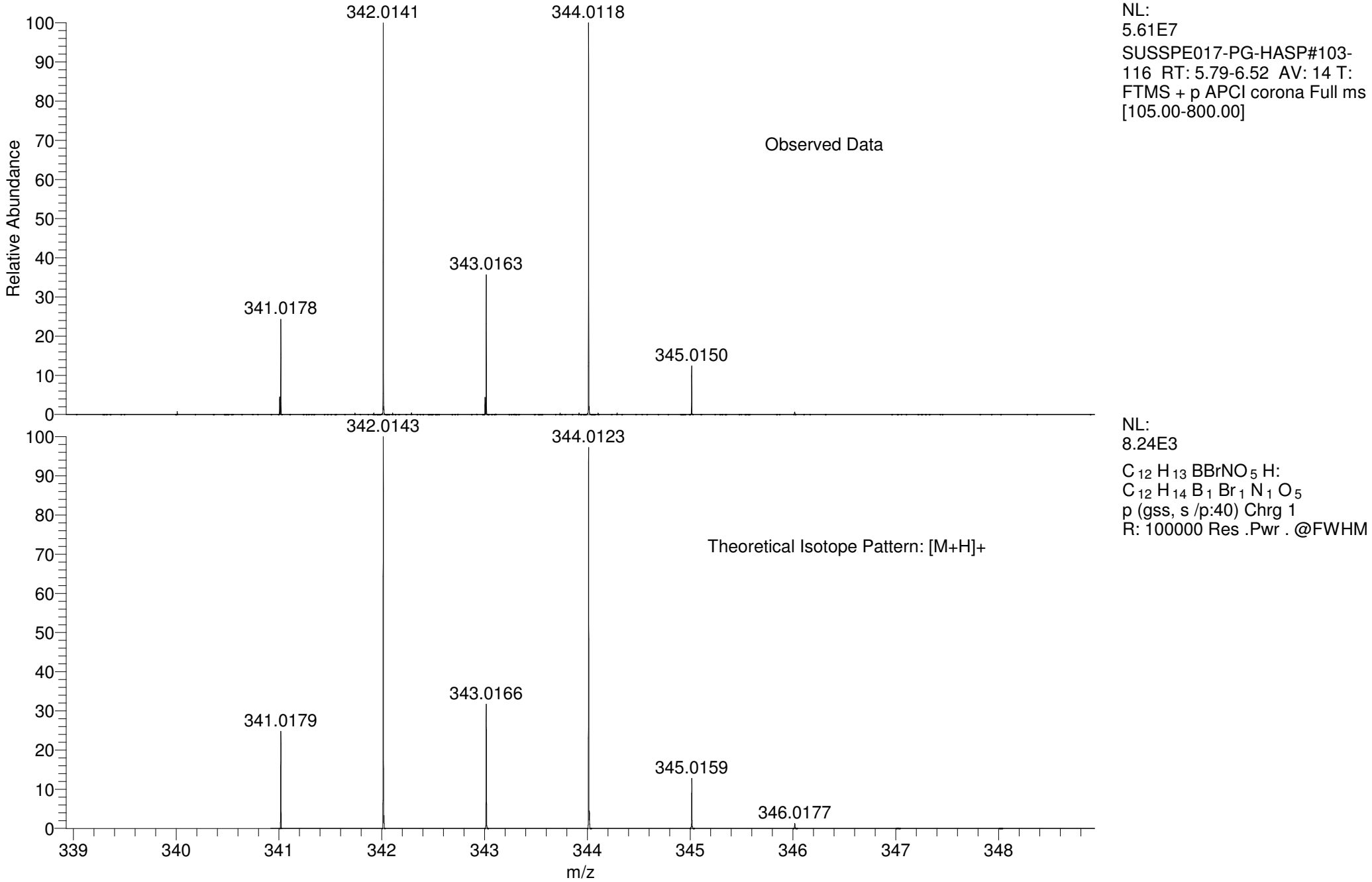
04BR06 MW=342?
ASAP(SOLID)

EPSRC National Facility Swansea
LTQ Orbitrap XL

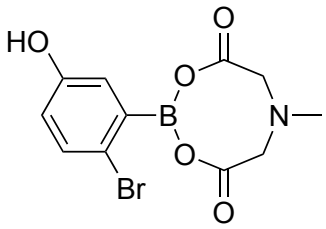
Close
23/07/2013 10:02:55

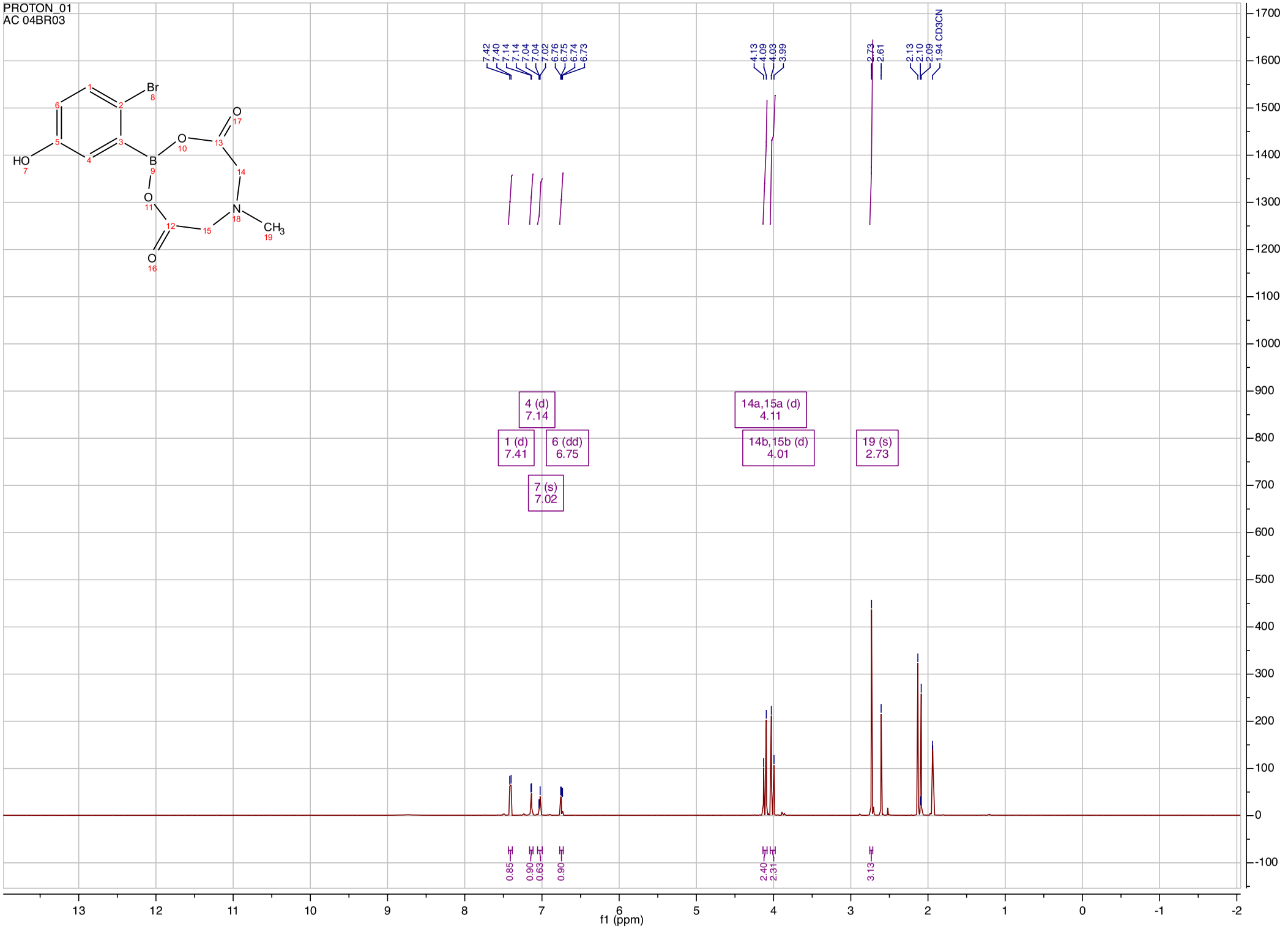
SUSSPE017-PG-HASP #103-116 RT: 5.79-6.52 AV: 14 SM: 7G NL: 5.61E7
T: FTMS + p APCI corona Full ms [105.00-800.00]

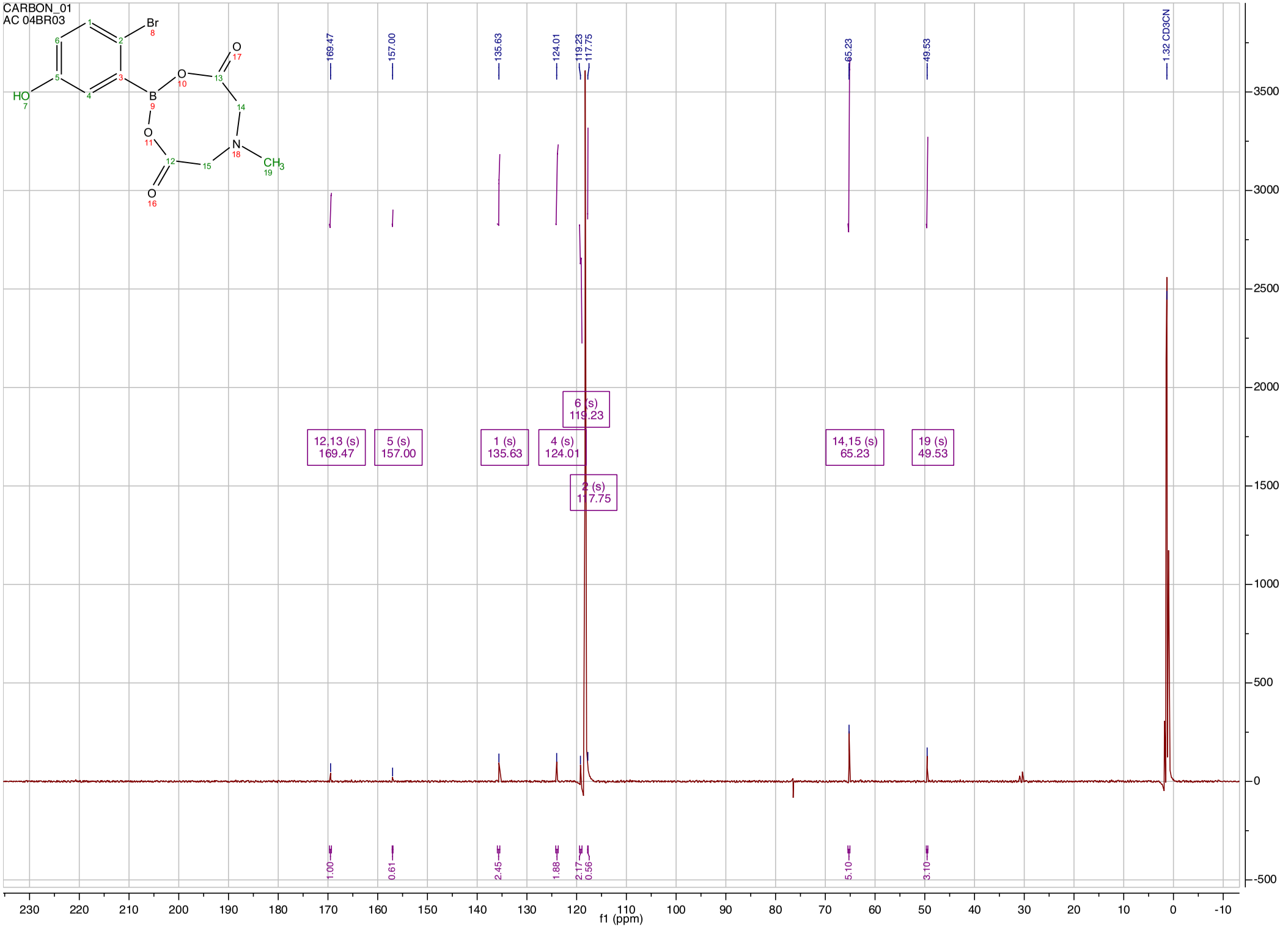



342.0141
344.0118
343.0163
341.0178
345.0150
Observed Data
NL:
5.61E7
SUSSPE017-PG-HASP#103-116 RT: 5.79-6.52 AV: 14 T: FTMS + p APCI corona Full ms [105.00-800.00]
342.0143
344.0123
343.0166
341.0179
345.0159
346.0177
Theoretical Isotope Pattern: [M+H]+
NL:
8.24E3
C 12 H 13 BBrNO 5 H:
C 12 H 14 B 1 Br 1 N 1 O 5
p (gss, s /p:40) Chrg 1
R: 100000 Res .Pwr . @FWHM
Relative Abundance
m/z
100
90
80
70
60
50
40
30
20
10
0
339
340
341
342
343
344
345
346
347
348

2-Bromo-5-hydroxyphenyl MIDA boronate 3g




PROTON_01
AC 04BR03
7.42
7.40
7.14
7.14
7.04
7.04
7.02
6.76
6.75
6.74
6.73
4.13
4.09
4.03
3.99
2.73
2.61
2.13
2.10
2.09
1.94 CD3CN
4 (d)
7.14
1 (d)
7.41
6 (dd)
6.75
7 (s)
7.02
14a,15a (d)
4.11
14b,15b (d)
4.01
19 (s)
2.73
0.85
0.90
0.83
0.90
2.40
2.31
3.13
f1 (ppm)

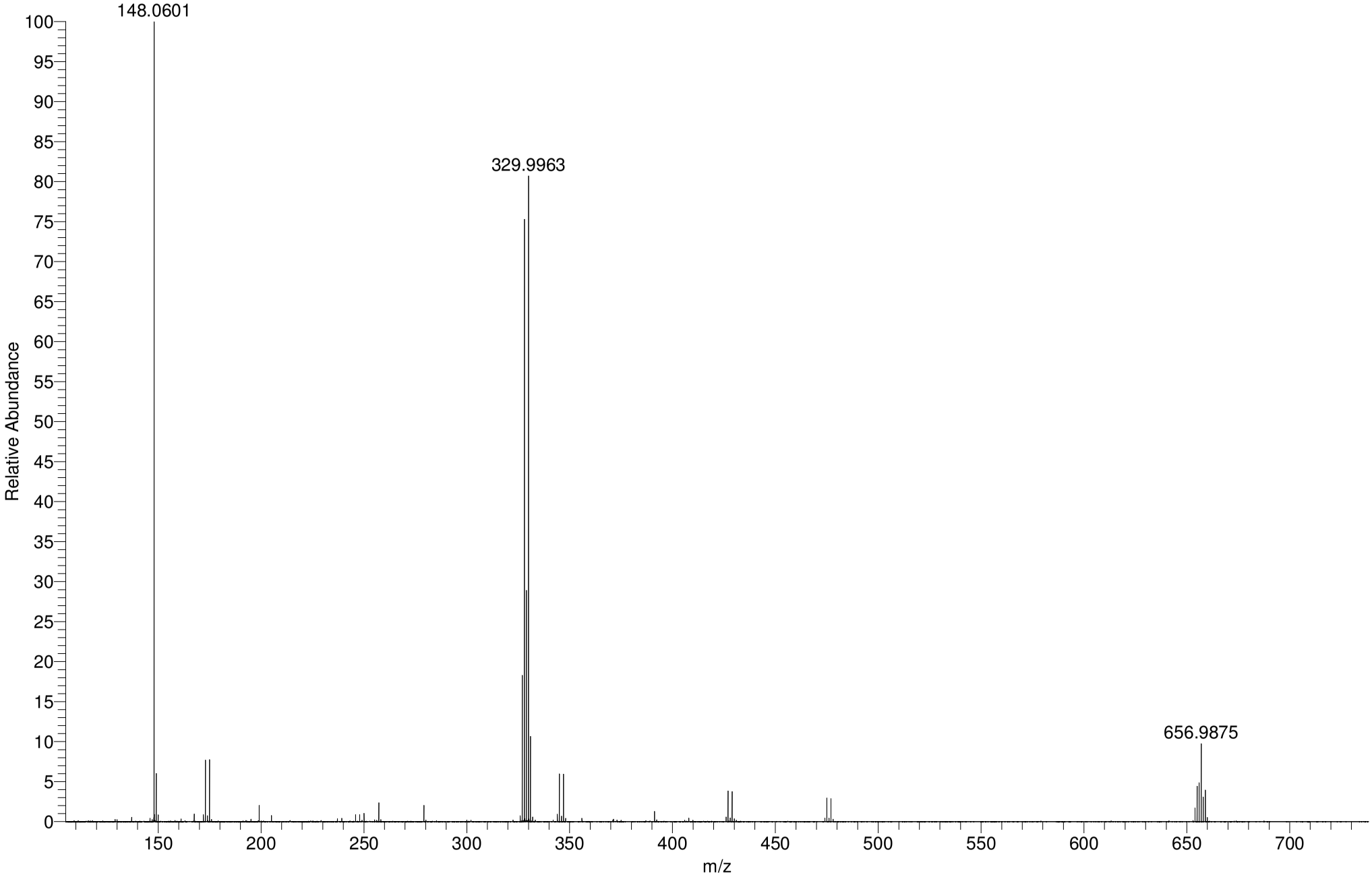

CARBON_01
AC 04BR03
Br
8
HO
7
O
17
O
10
13
B
9
14
O
11
N
18
CH3
19
12
15
O
16
169.47
157.00
135.63
124.01
119.23
117.75
65.23
49.53
1.32 CD3CN
6 (s)
119.23
12,13 (s)
169.47
5 (s)
157.00
1 (s)
135.63
4 (s)
124.01
2 (s)
117.75
14,15 (s)
65.23
19 (s)
49.53
1.00
0.61
2.45
1.88
2.17
0.56
5.10
3.10
3500
3000
2500
2000
1500
1000
500
0
-500
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

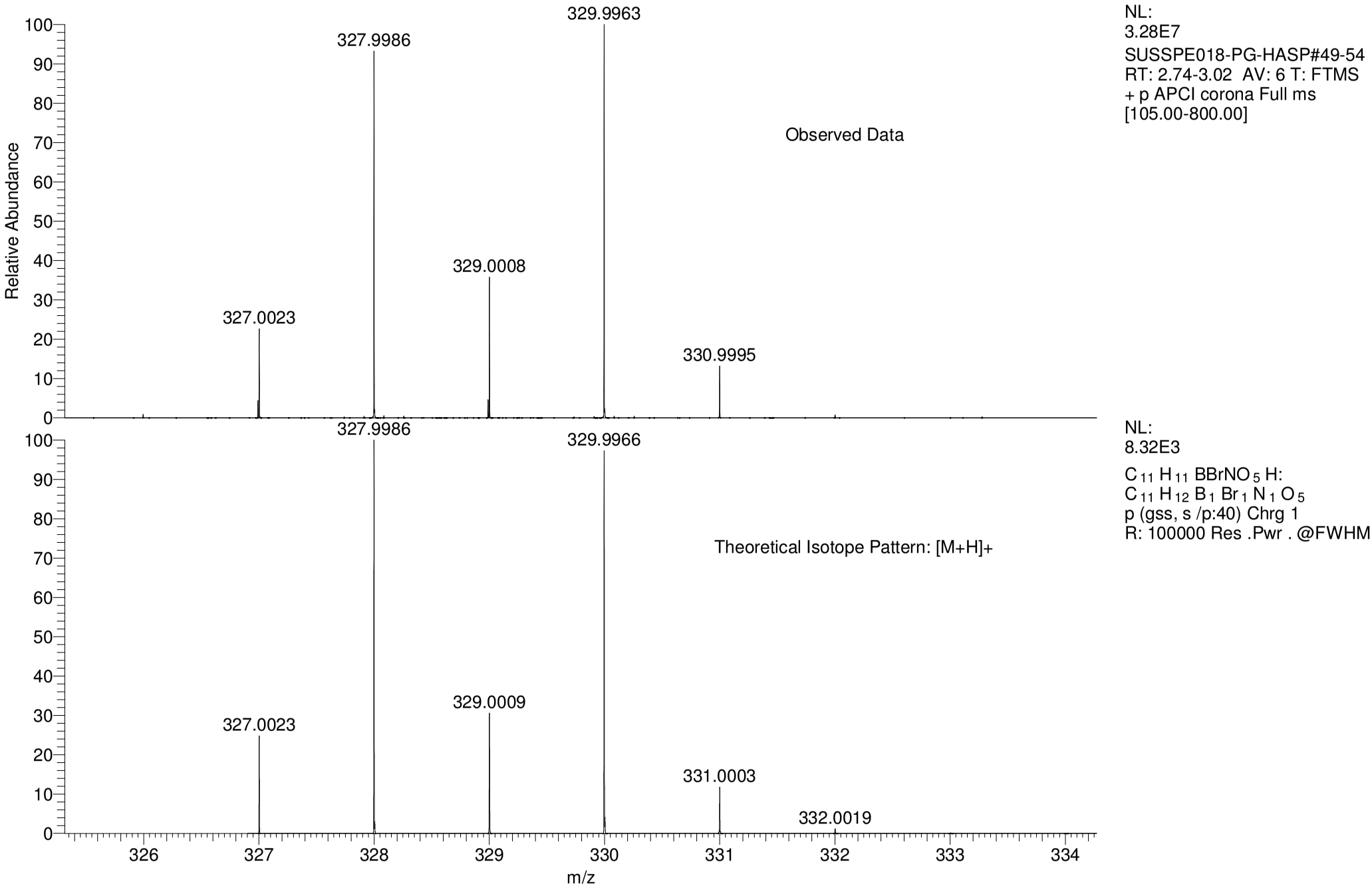
04BR03 MW=328?
ASAP(SOLID)

EPSRC National Facility Swansea
LTQ Orbitrap XL

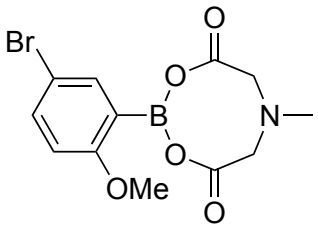
Close
23/07/2013 10:16:37

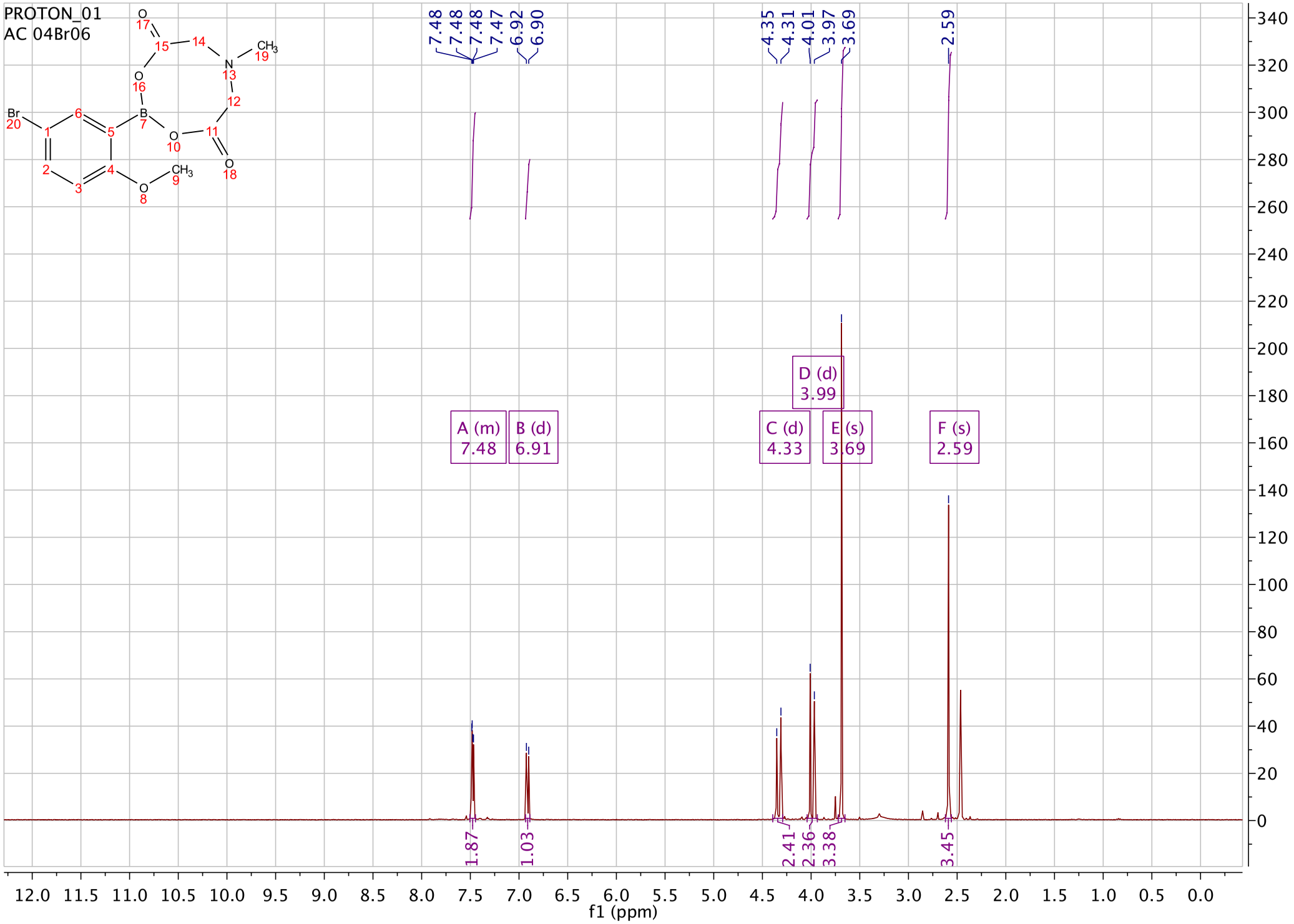
SUSSPE018-PG-HASP #49-54 RT: 2.74-3.02 AV: 6 SM: 7G NL: 4.06E7
T: FTMS + p APCI corona Full ms [105.00-800.00]

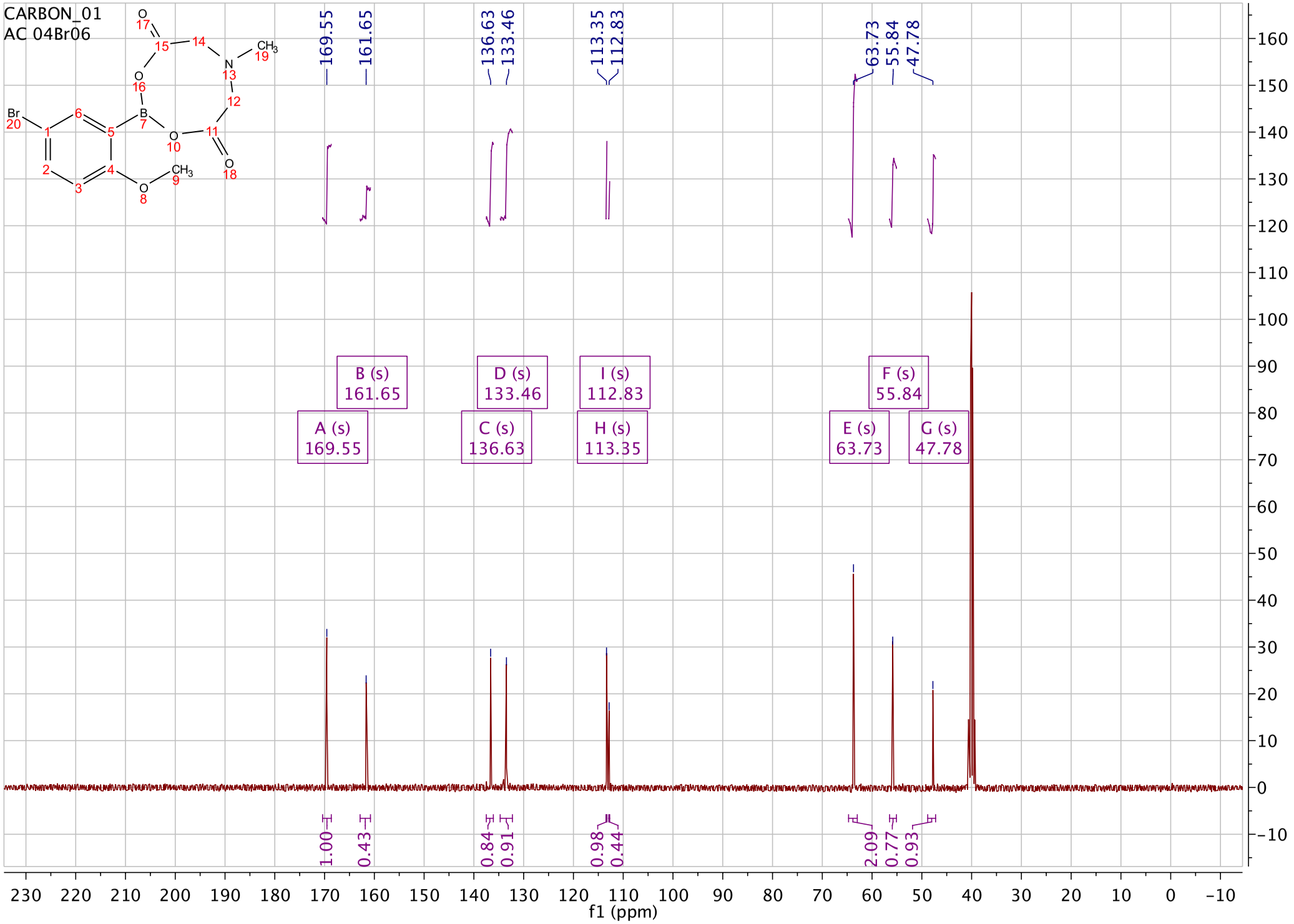


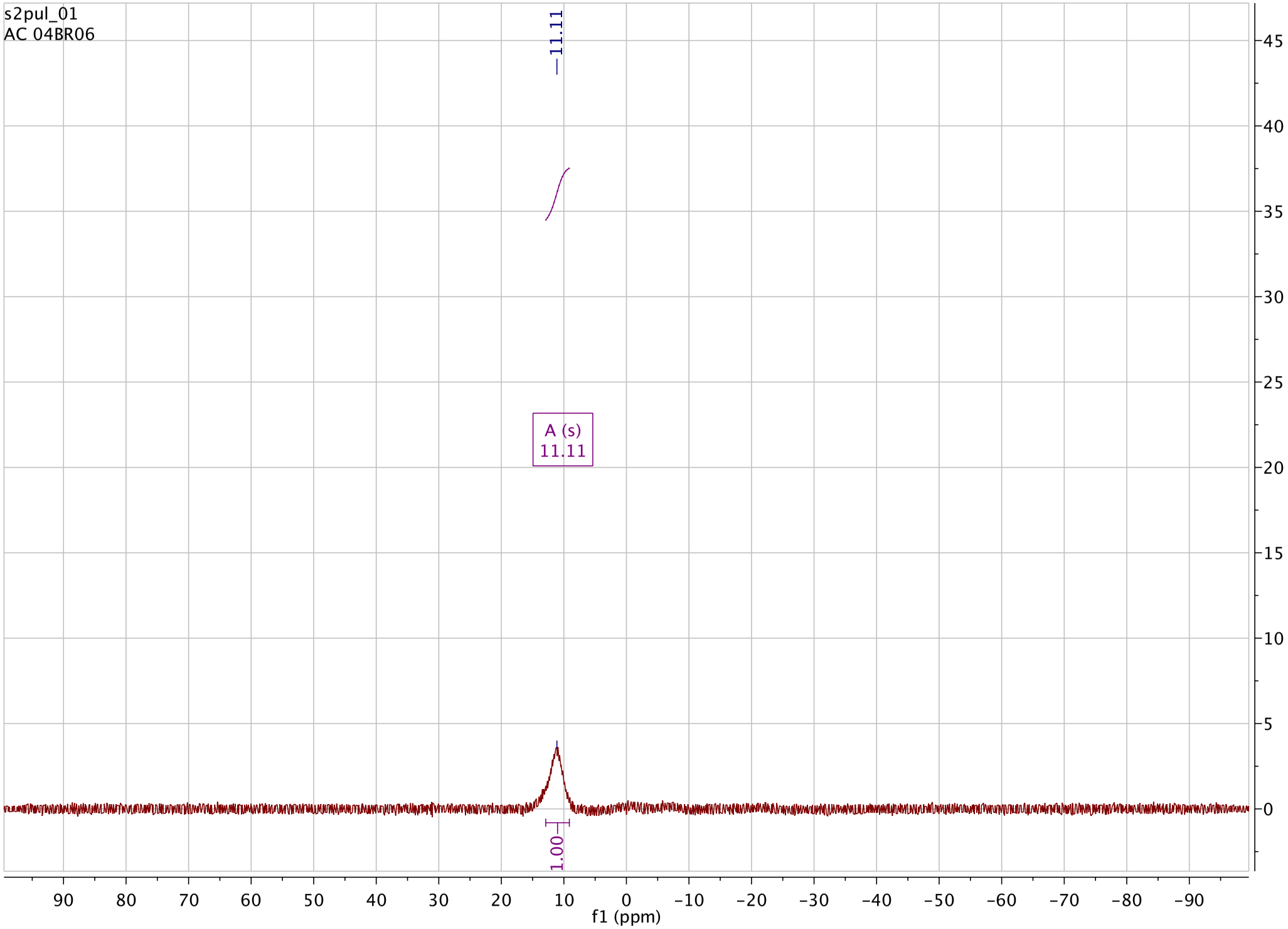

329.9963
327.9986
329.0008
327.0023
330.9995
Observed Data
NL:
3.28E7
SUSSPE018-PG-HASP#49-54
RT: 2.74-3.02 AV: 6 T: FTMS
+ p APCI corona Full ms
[105.00-800.00]
Relative Abundance
327.9986
329.9966
329.0009
327.0023
331.0003
332.0019
Theoretical Isotope Pattern: [M+H]+
NL:
8.32E3
C11 H11 BBrNO5 H:
C11 H12 B1 Br1 N1 O5
p (gss, s /p:40) Chrg 1
R: 100000 Res .Pwr . @FWHM
m/z

5-Bromo-2-methoxyphenyl MIDA boronate 3h

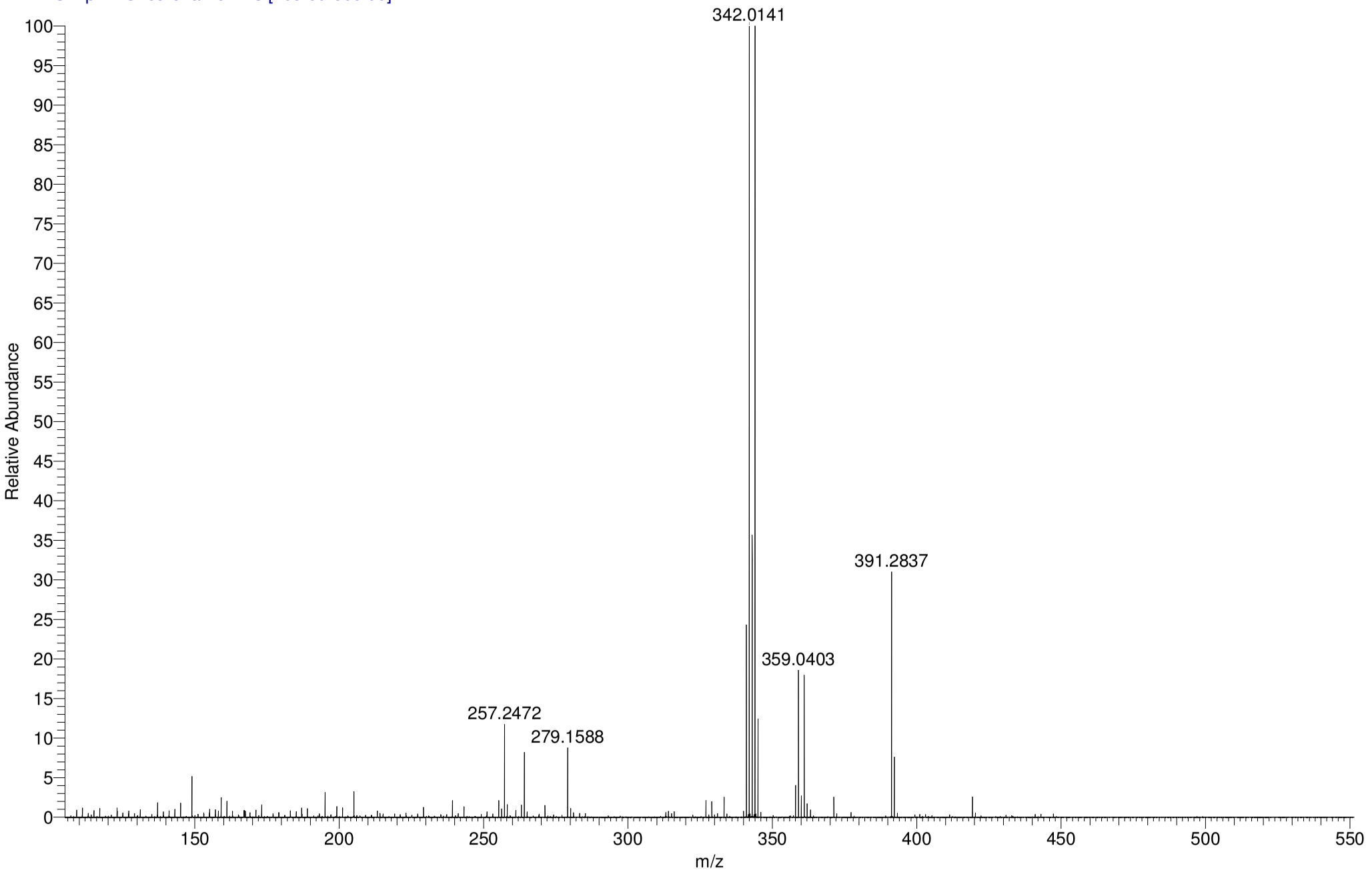


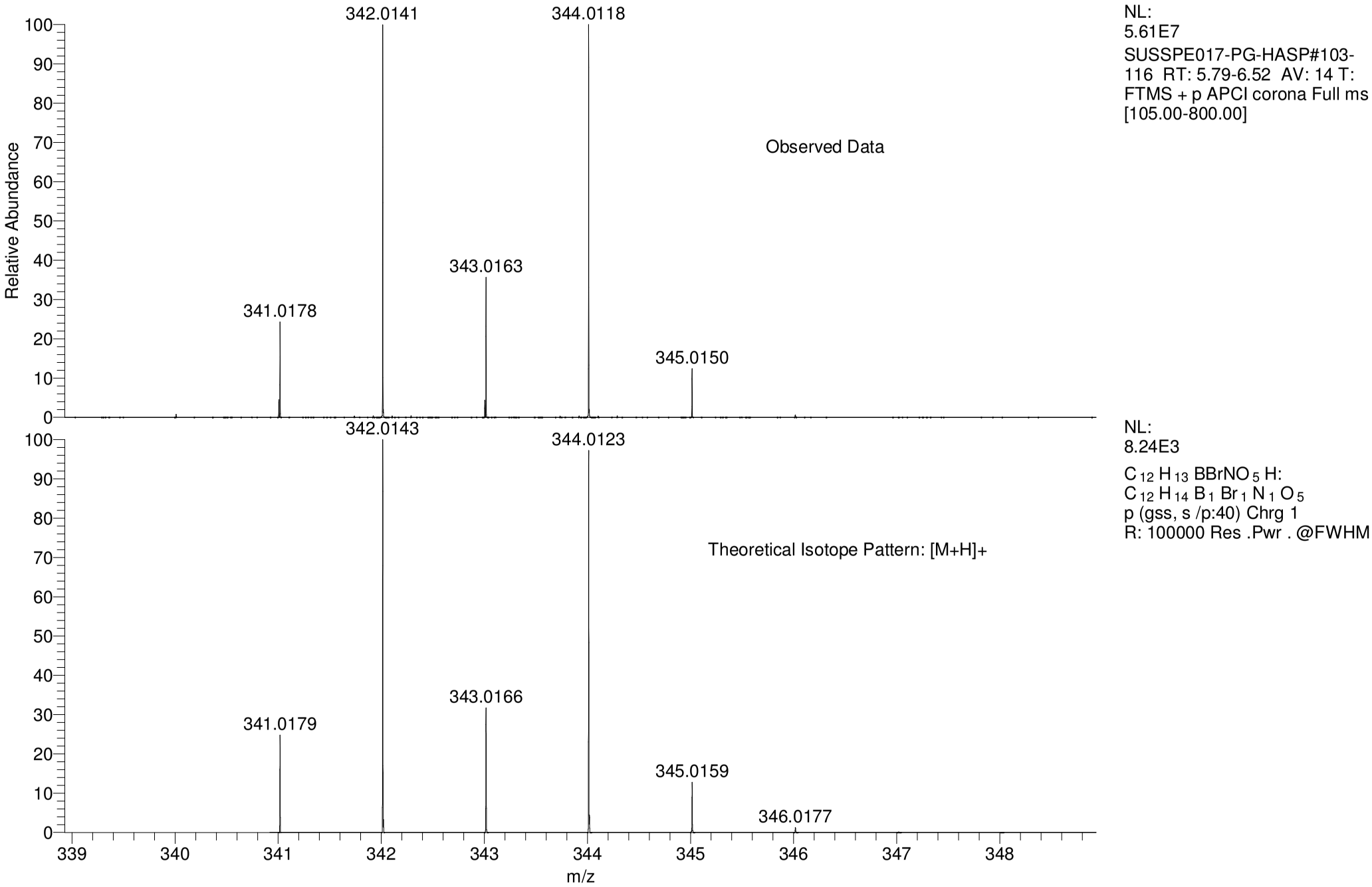

PROTON_01
AC 04Br06
7.48
7.48
7.48
7.47
6.92
6.90
4.35
4.31
4.01
3.97
3.69
2.59
A (m)
7.48
B (d)
6.91
D (d)
3.99
C (d)
4.33
E (s)
3.69
F (s)
2.59
1.87
1.03
2.41
2.36
3.38
3.45
f1 (ppm)


CARBON_01
AC 04Br06
169.55
161.65
136.63
133.46
113.35
112.83
63.73
55.84
47.78
B (s) 161.65
D (s) 133.46
I (s) 112.83
F (s) 55.84
A (s) 169.55
C (s) 136.63
H (s) 113.35
E (s) 63.73
G (s) 47.78
1.00
0.43
0.84
0.91
0.98
0.44
2.09
0.77
0.93
f1 (ppm)

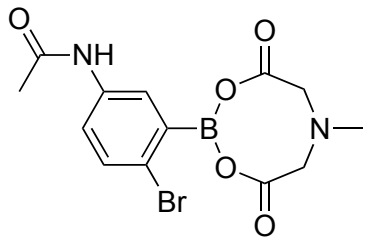

s2pul_01
AC 04BR06
11.11
A (s)
11.11
1.00
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
f1 (ppm)
45
40
35
30
25
20
15
10
5
0

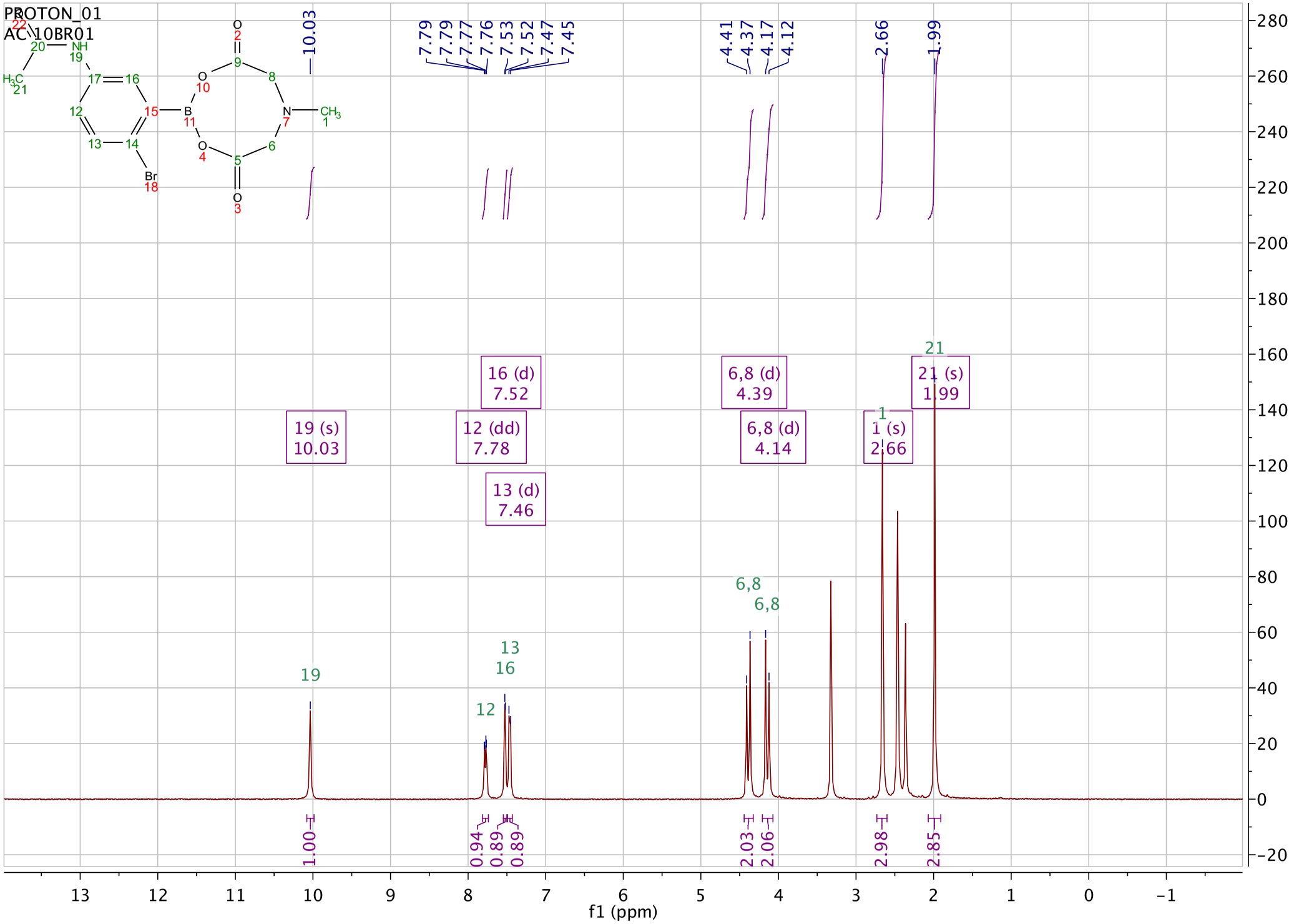
SUSSPE017-PG-HASP #103-116 RT: 5.79-6.52 AV: 14 SM: 7G NL: 5.61E7
T: FTMS + p APCI corona Full ms [105.00-800.00]

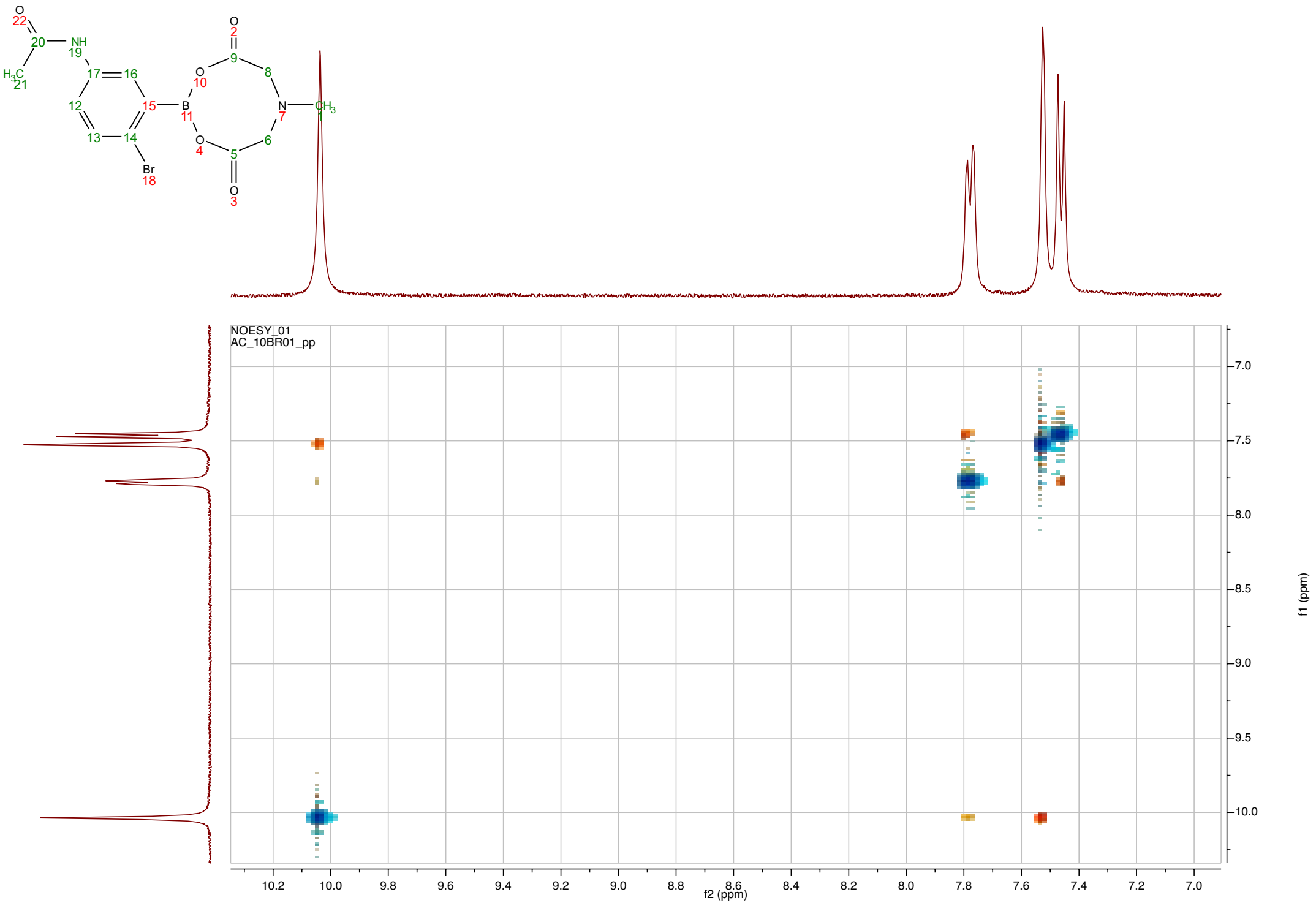


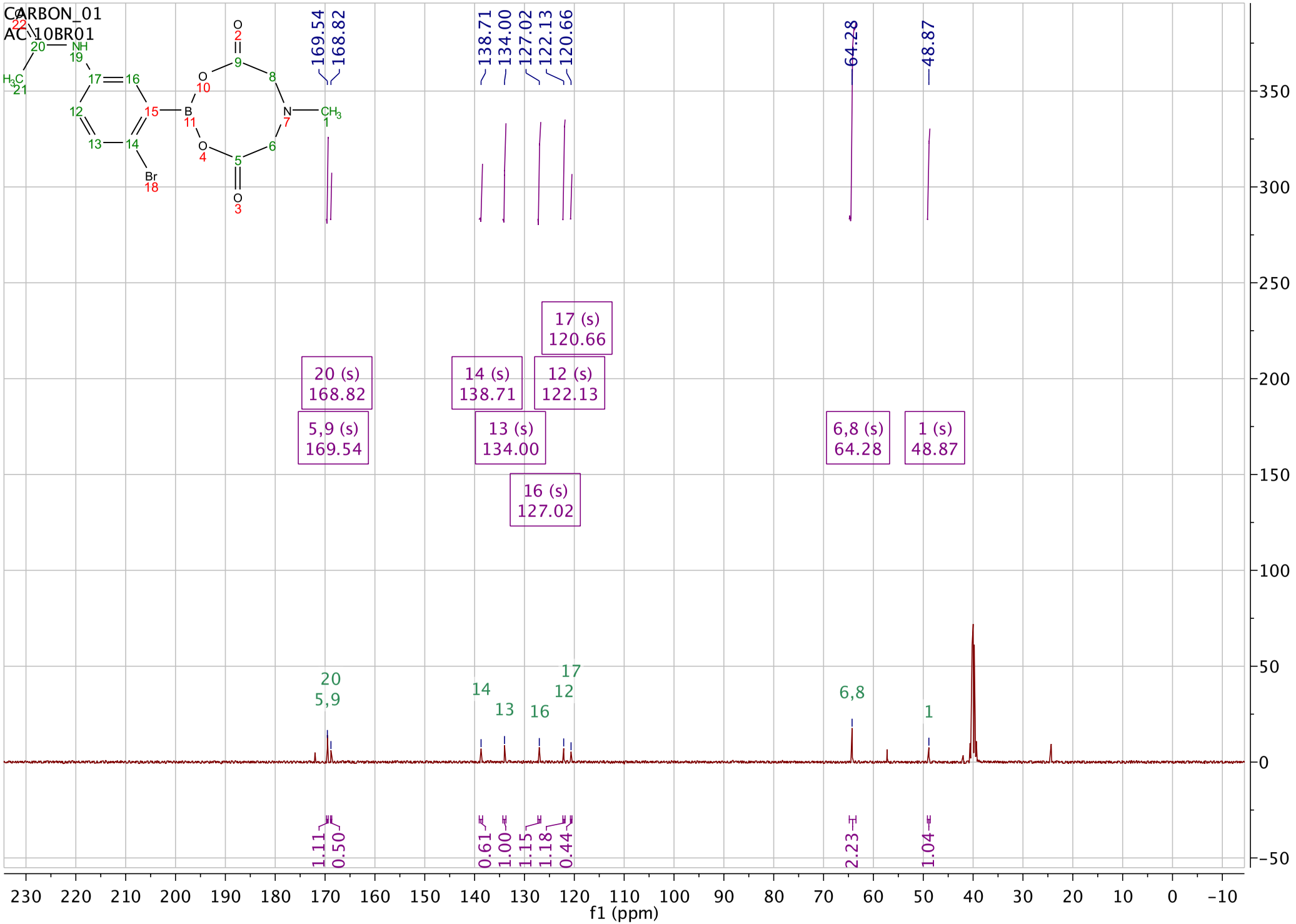

342.0141
344.0118
343.0163
341.0178
345.0150
Observed Data
NL:
5.61E7
SUSSPE017-PG-HASP#103-116 RT: 5.79-6.52 AV: 14 T: FTMS + p APCI corona Full ms [105.00-800.00]
342.0143
344.0123
343.0166
341.0179
345.0159
346.0177
Theoretical Isotope Pattern: [M+H]+
NL:
8.24E3
C12 H13 BBrNO5 H:
C12 H14 B1 Br1 N1 O5
p (gss, s /p:40) Chrg 1
R: 100000 Res .Pwr . @FWHM
Relative Abundance
m/z
0
10
20
30
40
50
60
70
80
90
100
339
340
341
342
343
344
345
346
347
348

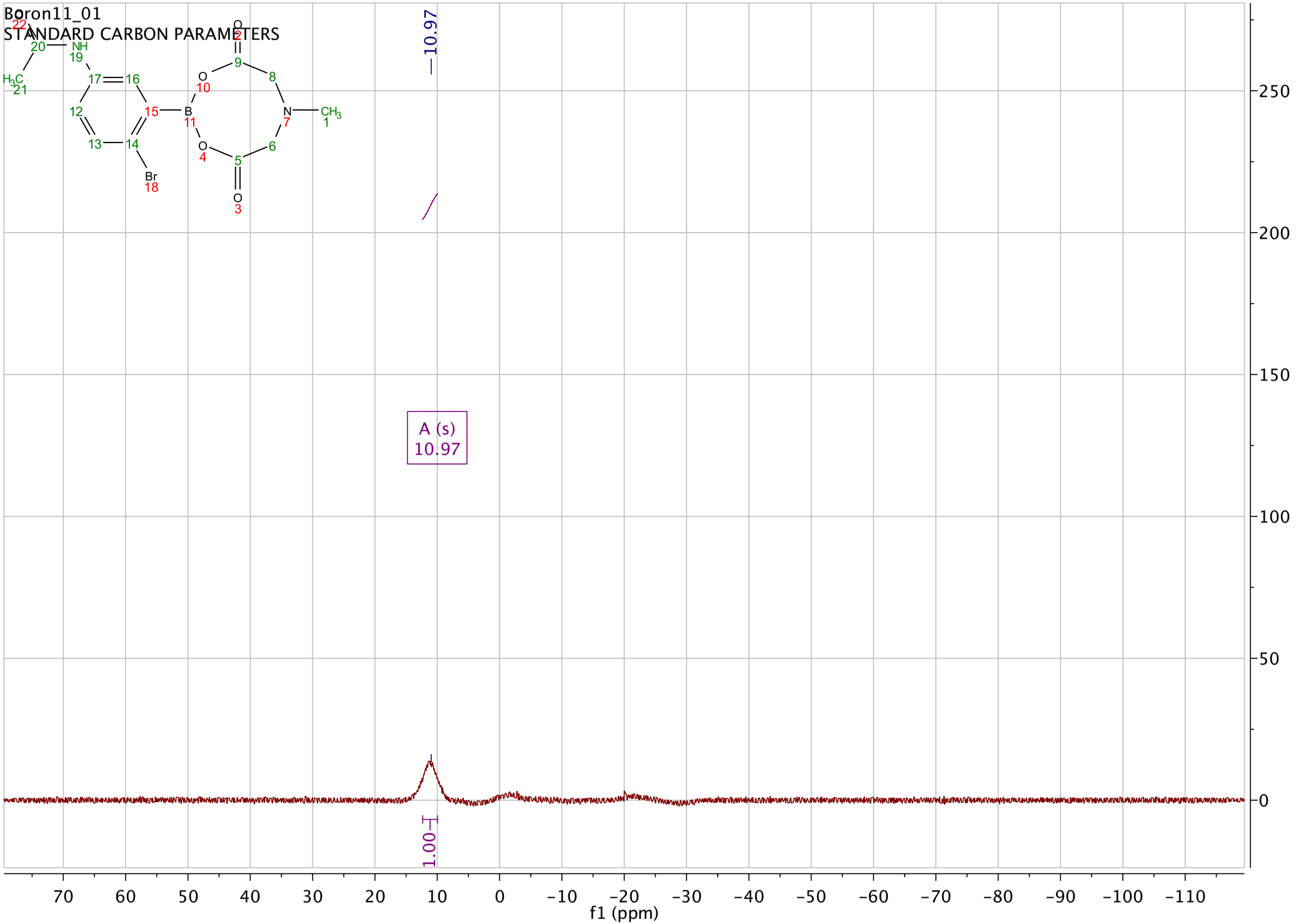
5-Acetamido-2-bromophenyl MIDA boronate 3j




PROTON_01
AC\10BR01
10.03
7.79
7.79
7.77
7.76
7.53
7.52
7.47
7.45
4.41
4.37
4.17
4.12
2.66
1.99
19 (s)
10.03
16 (d)
7.52
12 (dd)
7.78
13 (d)
7.46
6,8 (d)
4.39
6,8 (d)
4.14
1 (s)
2.66
21 (s)
1.99
19
12
13
16
6,8
6,8
1
21
1.00
0.94
0.89
0.89
2.03
2.06
2.98
2.85
f1 (ppm)


NOESY_01
AC_10BR01_pp
f2 (ppm)
f1 (ppm)
10.2
10.0
9.8
9.6
9.4
9.2
9.0
8.8
8.6
8.4
8.2
8.0
7.8
7.6
7.4
7.2
7.0
7.0
7.5
8.0
8.5
9.0
9.5
10.0


CARBON_01
AC_10BR01
169.54
168.82
138.71
134.00
127.02
122.13
120.66
64.28
48.87
20 (s)
168.82
5,9 (s)
169.54
14 (s)
138.71
13 (s)
134.00
16 (s)
127.02
17 (s)
120.66
12 (s)
122.13
6,8 (s)
64.28
1 (s)
48.87
20
5,9
14
13
16
17
12
6,8
1
1.11
0.50
0.61
1.00
1.15
1.18
0.44
2.23
1.04
f1 (ppm)

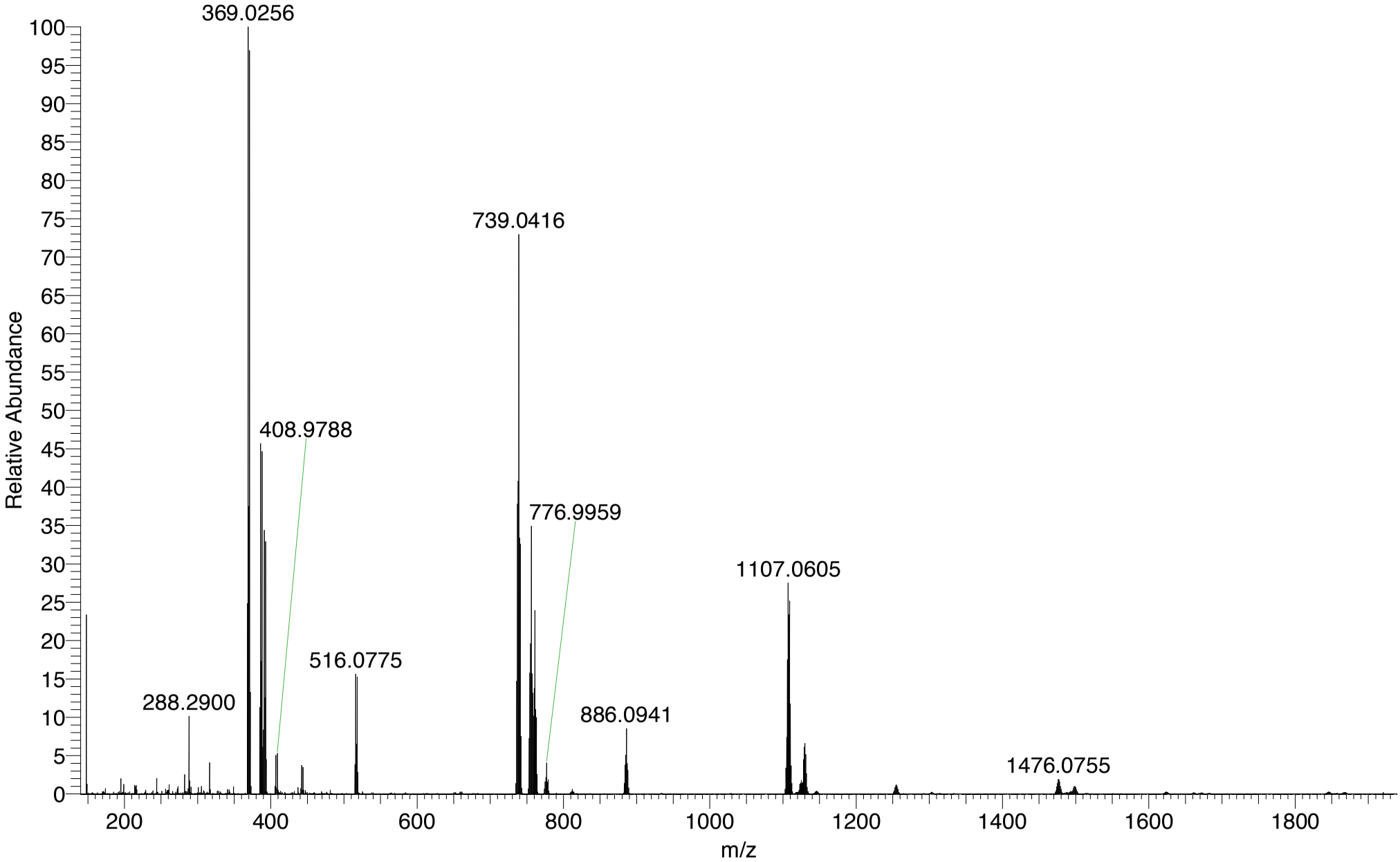

Boron11_01
STANDARD CARBON PARAMETERS
10.97
A (s)
10.97
1.00
250
200
150
100
50
0
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
f1 (ppm)

10BR01 MW=368?
(MeCN)/MeCN
C13H14BBrN2O5

EPSRC National Facility Swansea
LTQ Orbitrap XL

Adam Close
07/09/2015 12:22:24

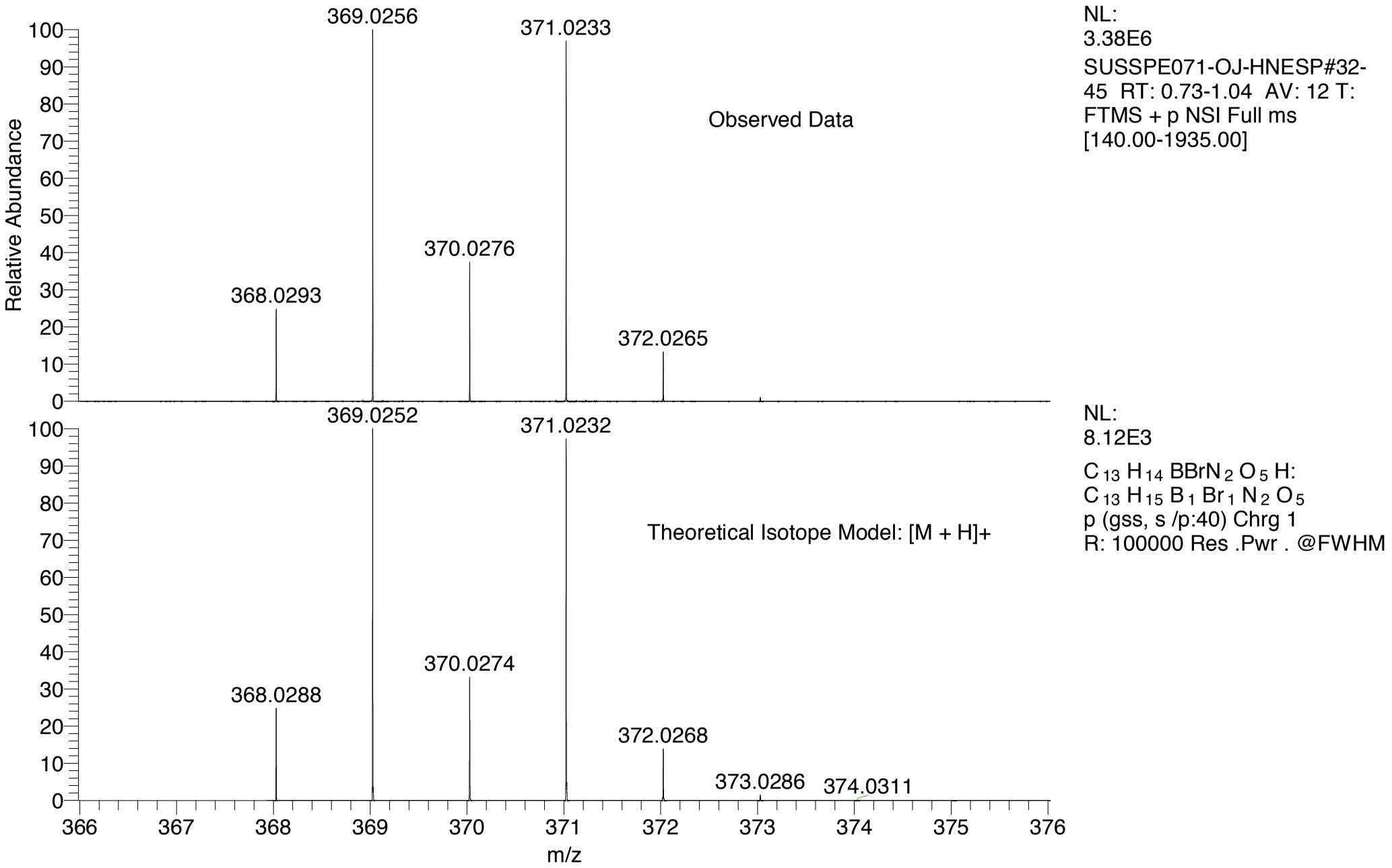
SUSSPE071-OJ-HNESP #32-45 RT: 0.73-1.04 AV: 12 SM: 7G NL: 3.38E6
T: FTMS + p NSI Full ms [140.00-1935.00]



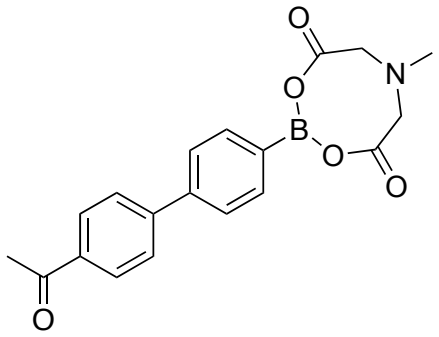
10BR01 MW=368?
(MeCN)/MeCN
C13H14BBrN2O5

EPSRC National Facility Swansea
LTQ Orbitrap XL

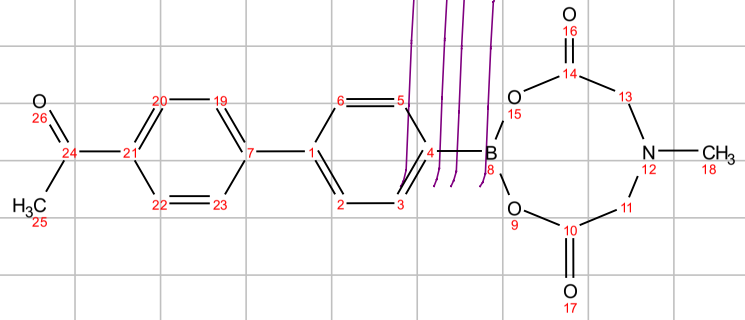
Adam Close
07/09/2015 12:22:24



4'-Acetyl-[1,1'-biphenyl]-4-yl MIDA boronate 4a



140616.270.fid
Name Adam Close
M No kzmr981
Notebook Ref EN07373-58
proton128.az DMSO /opt/topspin2.1 chem 35



8.05
8.03
7.86
7.84
7.76
7.74
7.59
7.57

4.39
4.34
4.17
4.13

2.61
2.57
2.50 DMSO-d6

F (d)
7.75

H (d)
8.04

E (d)
7.58

G (d)
7.85

D (d)
4.37

C (d)
4.15

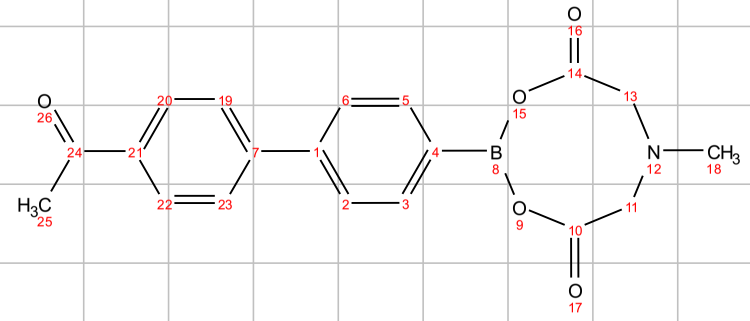
B (s)
2.61

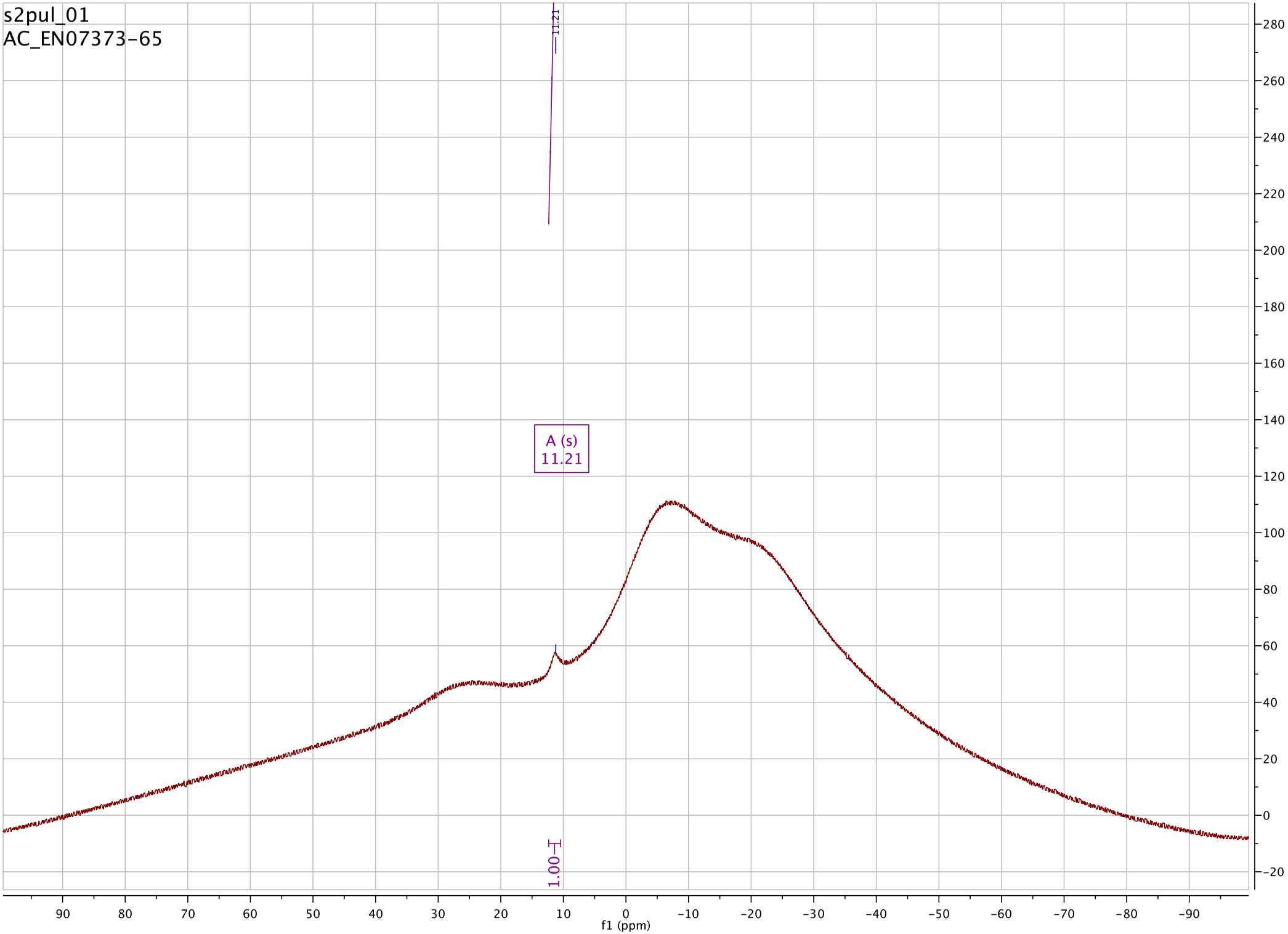
A (s)
2.57

2.00
2.02
1.98
1.94
1.96
1.98
2.82
2.95

f1 (ppm)

140616.271.fid
Name Adam Close
M No kzmr981
Notebook Ref EN07373-58
carbon.az DMSO /opt/topspin2.1 chem 35




s2pul_01
AC_EN07373-65
11.21
A (s)
11.21
1.00
f1 (ppm)

AstraZeneca

Mass Spectrometry Analysis Report

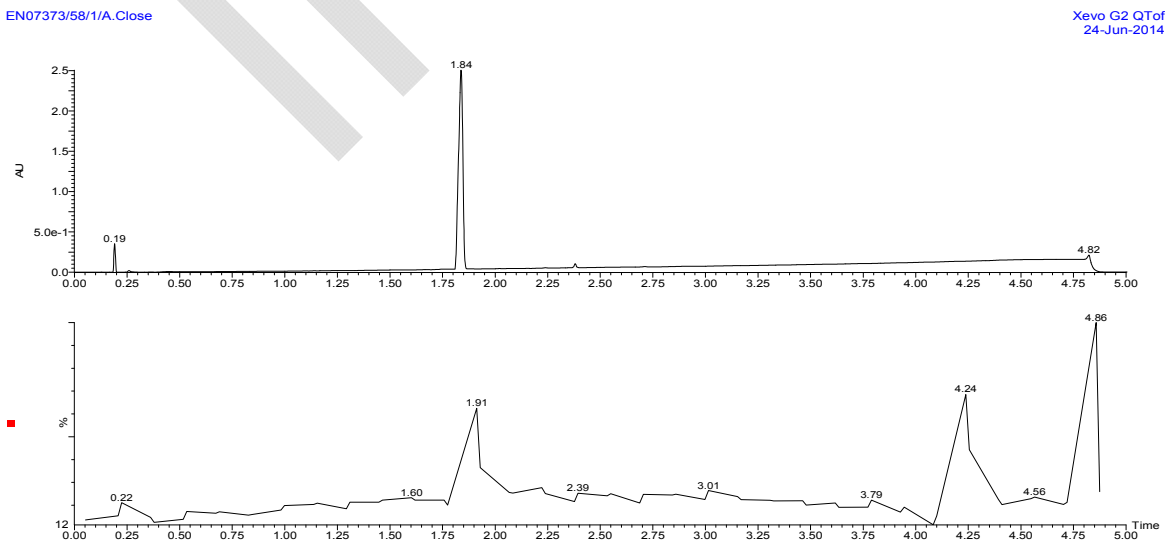
Summary:

The peak seen at RT: 1.84 mins m/z 350 is the major product. The peak has accurate mass and generated empirical formula that are consistent with the required product structure.

Raw data File: EN07373_58_1

ELN Reference:	EN07373_58_1	Date:	24th June 2014
Chemist	A.Close	Analyst:	P.Davey
Ion Type:	ESI -ve	Empirical Formula:	C19H18BNO5
Analysis Method:	Acid5min	Purity	99%
Instrument:	Waters Xevo G2 Qtof	Retention Time (mins):	1.84
Instrument method:	Open Access neg	Accurate mass (obs):	350.1193
Column:	Acquity 50 x 2.1 C18 BEH 1.7um	Accurate mass (Calc):	350.1200
Column temp:	40C	Error (ppm):	2.0

Chromatogram



AstraZeneca
Health Connects Us All

AstraZeneca

Mass Spectrometry Analysis Report

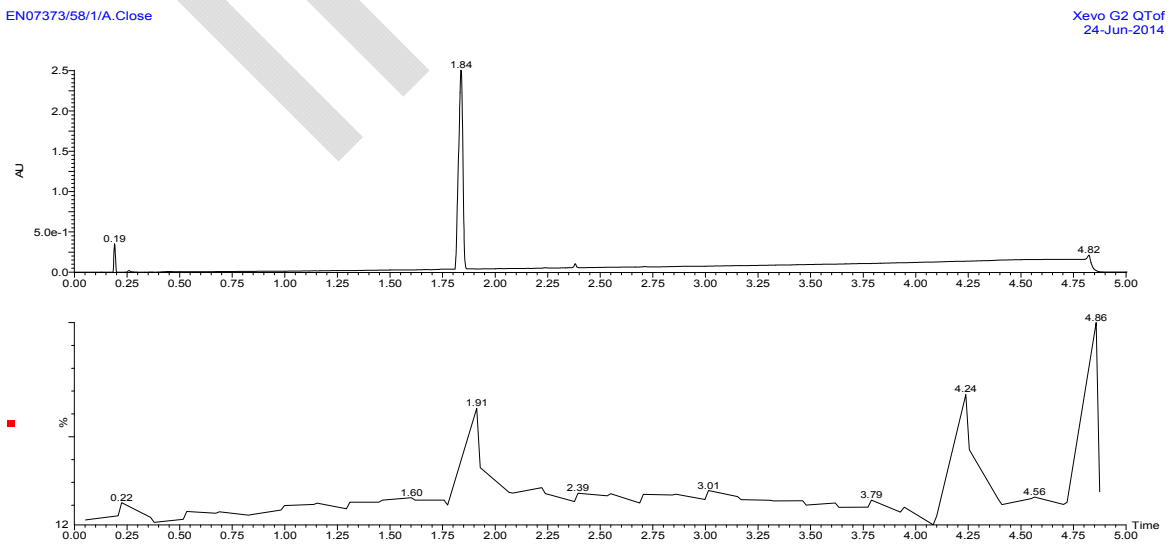
Summary:

The peak seen at RT: 1.84 mins m/z 350 is the major product. The peak has accurate mass and generated empirical formula that are consistent with the required product structure.

Raw data File: EN07373_58_1

ELN Reference:	EN07373_58_1	Date:	24th June 2014
Chemist	A.Close	Analyst:	P.Davey
Ion Type:	ESI -ve	Empirical Formula:	C19H18BNO5
Analysis Method:	Acid5min	Purity	99%
Instrument:	Waters Xevo G2 Qtof	Retention Time (mins):	1.84
Instrument method:	Open Access neg	Accurate mass (obs):	350.1193
Column:	Acquity 50 x 2.1 C18 BEH 1.7um	Accurate mass (Calc):	350.1200
Column temp:	40C	Error (ppm):	2.0

Chromatogram



AstraZeneca
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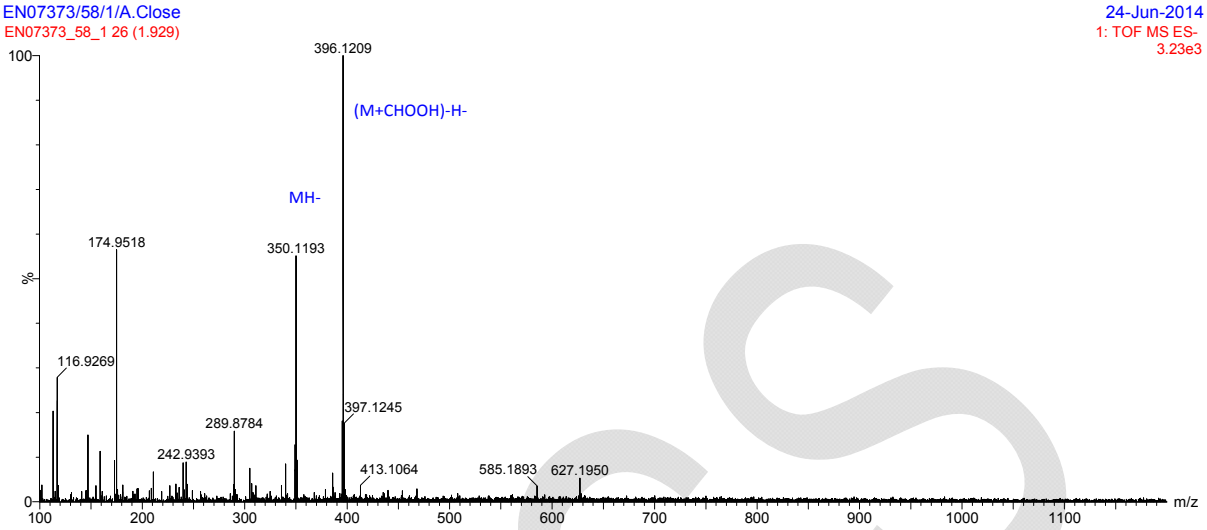
Spectra

MS



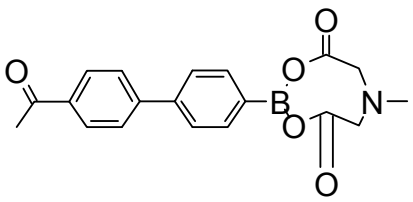
Elemental Composition Report

Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

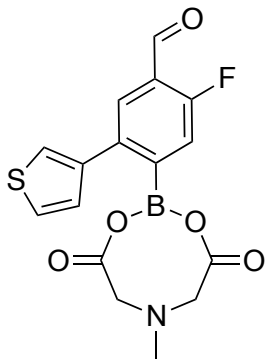
Monoisotopic Mass, Even Electron Ions
29 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-19 H: 0-50 11B: 0-1 N: 0-1 O: 0-5

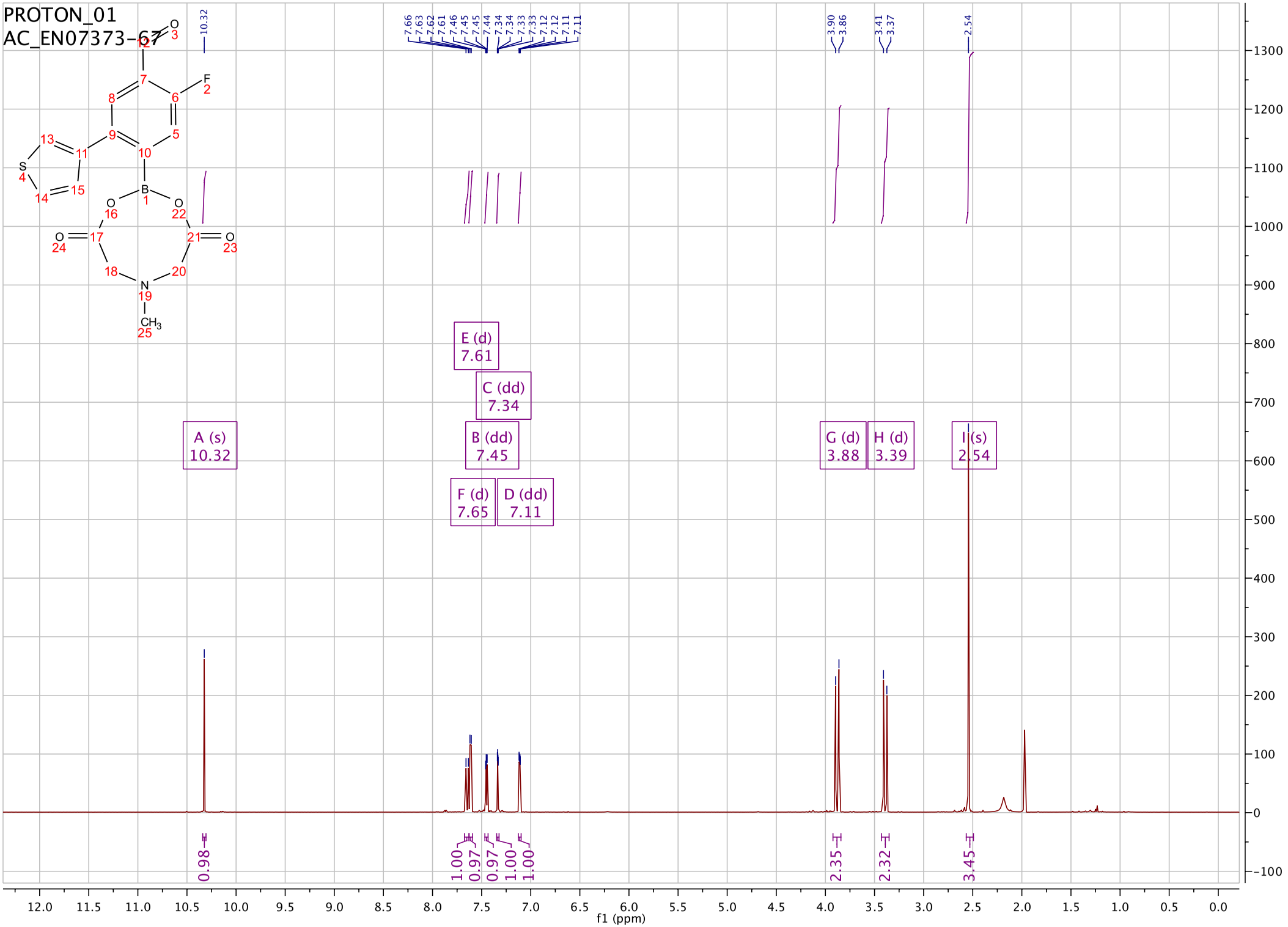
Minimum:				-1.5	
Maximum:		5.0	50.0	50.0	
Mass	Calc. Mass	mDa	PPM	DBE	Formula
350.1193	350.1200	-0.7	-2.0	12.5	C19 H17 11B N O5

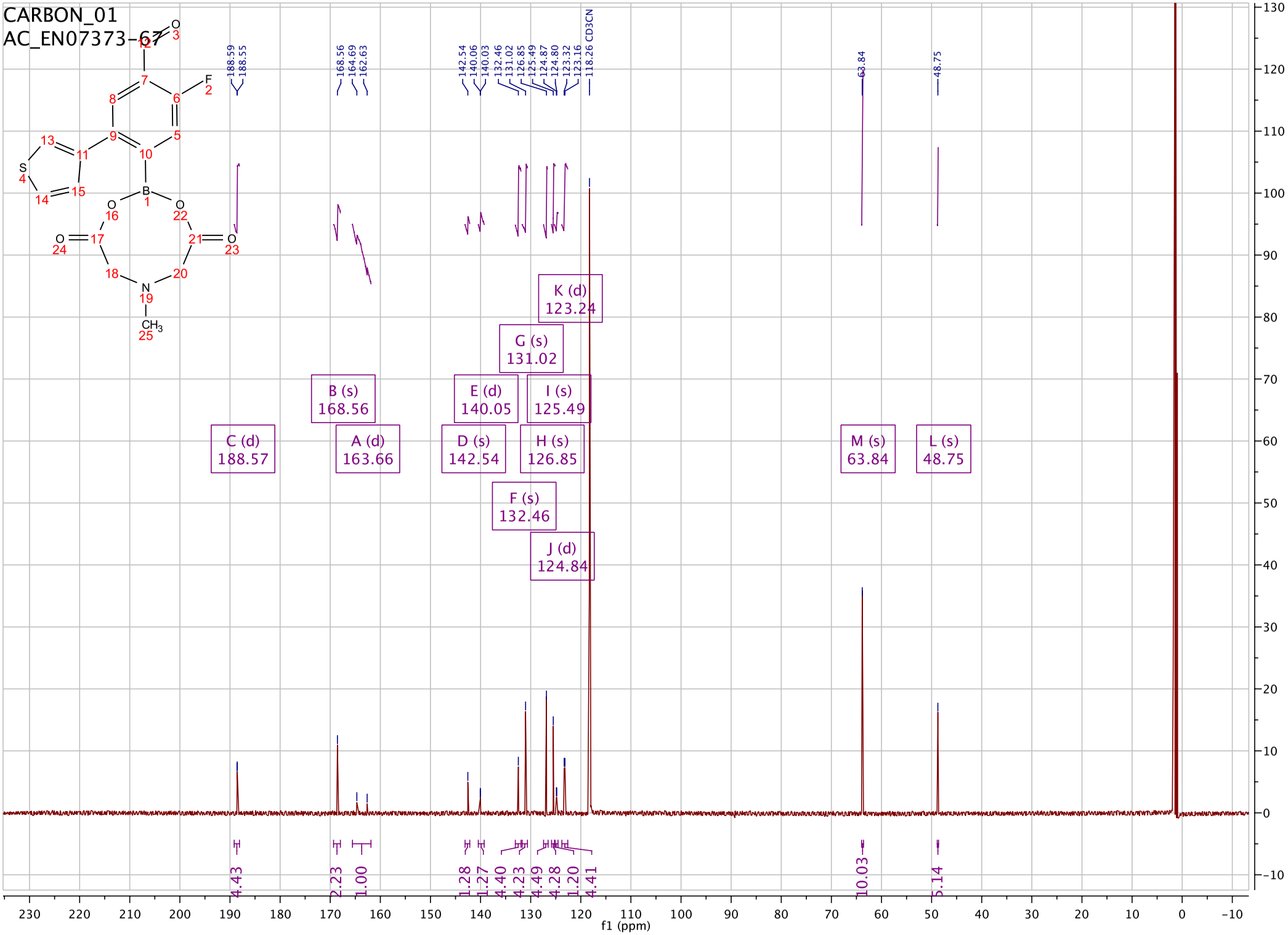
Structure



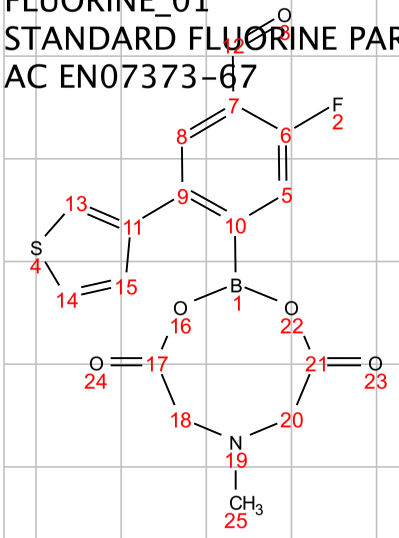
5-Fluoro-4-formyl-2-(thiophen-3-yl)phenyl MIDA boronate 4b




PROTON_01
AC_EN07373-67
10.32
7.66
7.63
7.62
7.61
7.46
7.45
7.45
7.44
7.34
7.34
7.33
7.33
7.12
7.12
7.11
7.11
3.90
3.86
3.41
3.37
2.54
A (s)
10.32
E (d)
7.61
C (dd)
7.34
B (dd)
7.45
F (d)
7.65
D (dd)
7.11
G (d)
3.88
H (d)
3.39
I (s)
2.54
0.98
1.00
0.97
0.97
1.00
1.00
2.35
2.32
3.45
f1 (ppm)


CARBON_01
AC_EN07373-67
188.59
188.55
168.56
164.69
162.63
142.54
140.06
140.03
132.46
131.02
126.85
125.49
124.87
124.80
123.32
123.16
118.26 CD3CN
63.84
48.75
K (d)
123.24
G (s)
131.02
B (s)
168.56
E (d)
140.05
I (s)
125.49
C (d)
188.57
A (d)
163.66
D (s)
142.54
H (s)
126.85
M (s)
63.84
L (s)
48.75
F (s)
132.46
J (d)
124.84
4.43
2.23
1.00
1.28
1.27
4.40
4.23
4.49
4.28
1.20
4.41
10.03
5.14
f1 (ppm)

FLUORINE_01
STANDARD FLUORINE PARAMETERS
AC EN07373-67



-125.59
-125.60
-125.62
-125.64

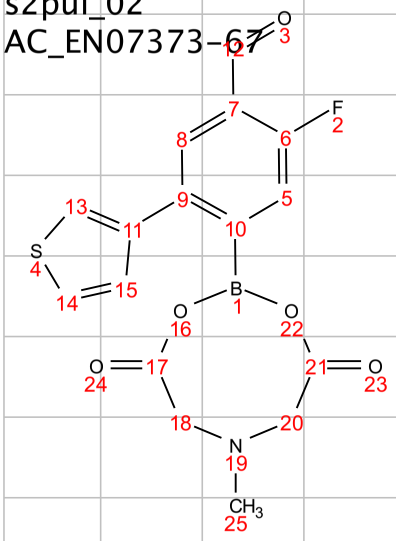
A (dd)
-125.61

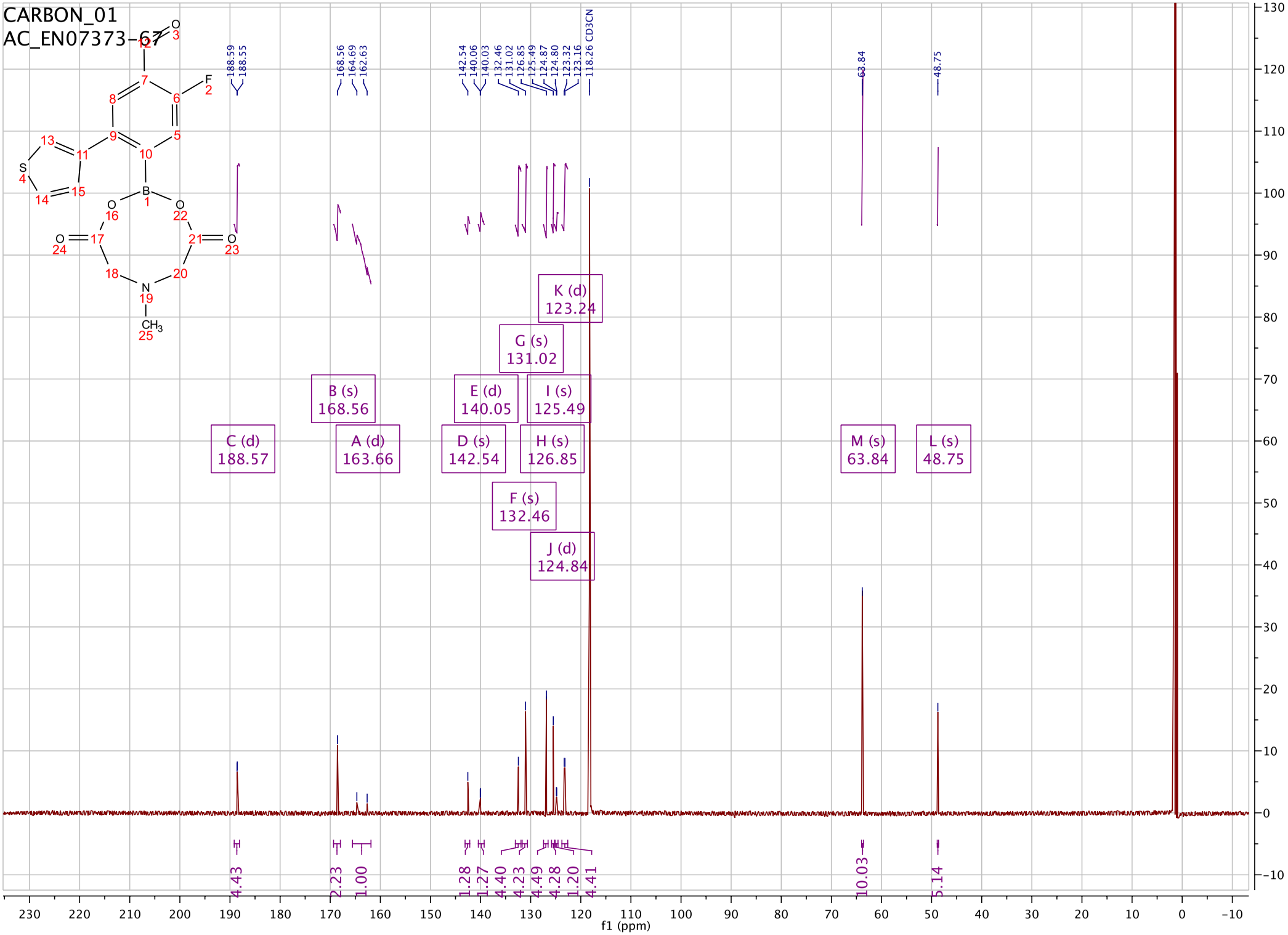
1.00

600
550
500
450
400
350
300
250
200
150
100
50
0
-50

30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)

s2pul_02
AC_EN07373-67



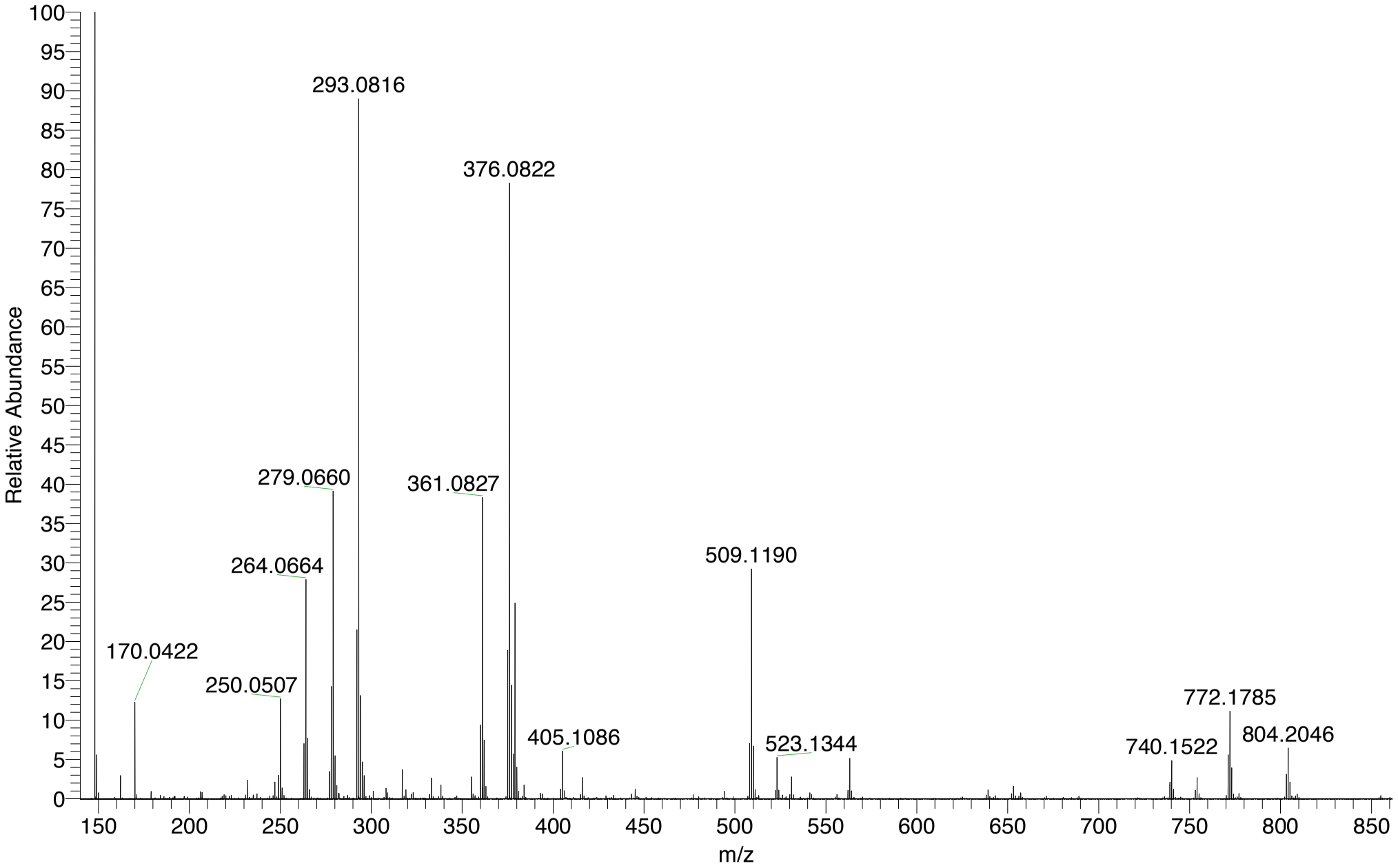

CARBON_01
AC_EN07373-67
188.59
188.55
168.56
164.69
162.63
142.54
140.06
140.03
132.46
131.02
126.85
125.49
124.87
124.80
123.32
123.16
118.26 CD3CN
63.84
48.75
K (d)
123.24
G (s)
131.02
B (s)
168.56
E (d)
140.05
I (s)
125.49
C (d)
188.57
A (d)
163.66
D (s)
142.54
H (s)
126.85
M (s)
63.84
L (s)
48.75
F (s)
132.46
J (d)
124.84
4.43
2.23
1.00
1.28
1.27
4.40
4.23
4.49
4.28
1.20
4.41
10.03
5.14
f1 (ppm)

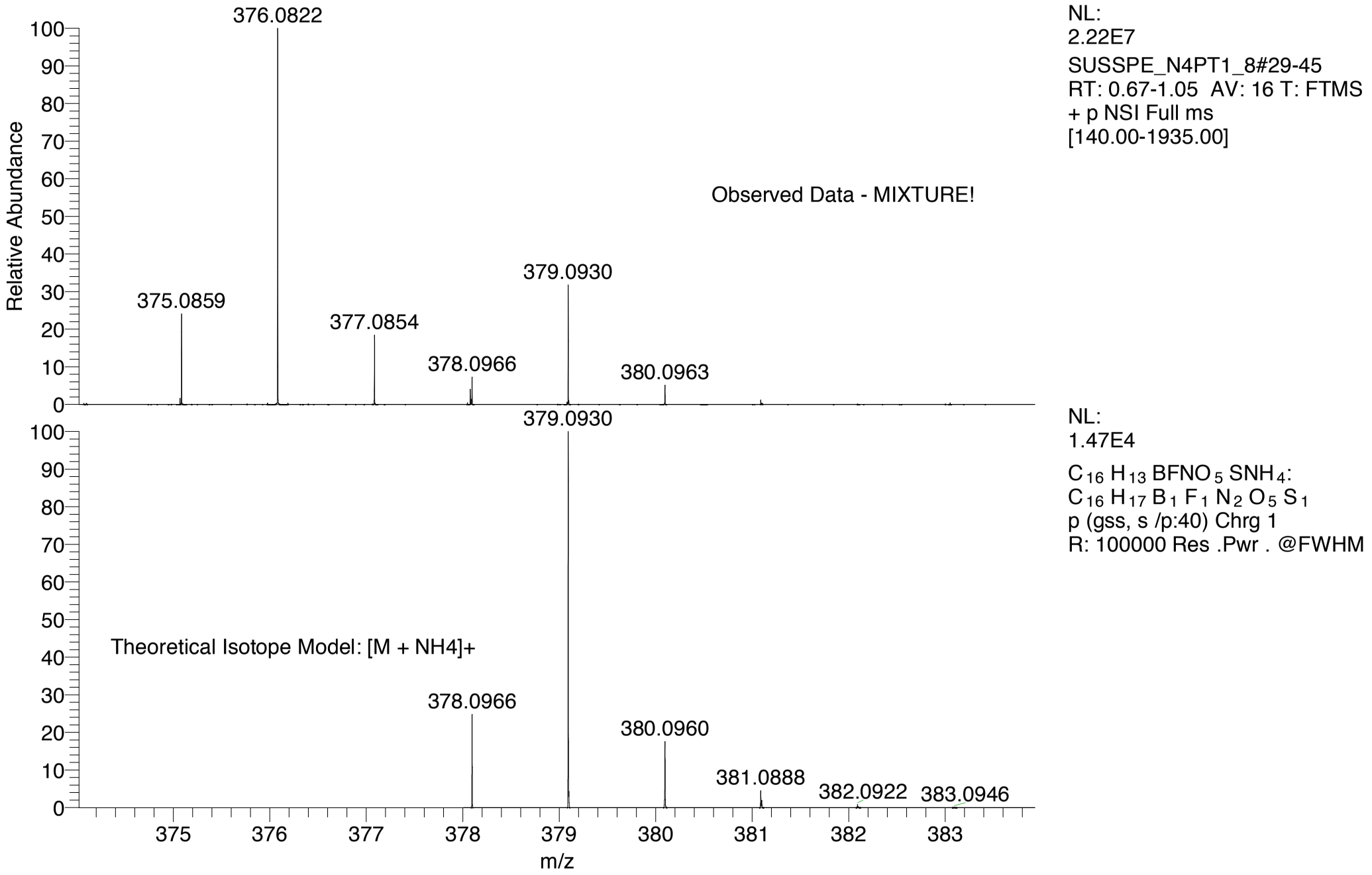
Sample Reference: EN07373-67
Username: SUSSPE

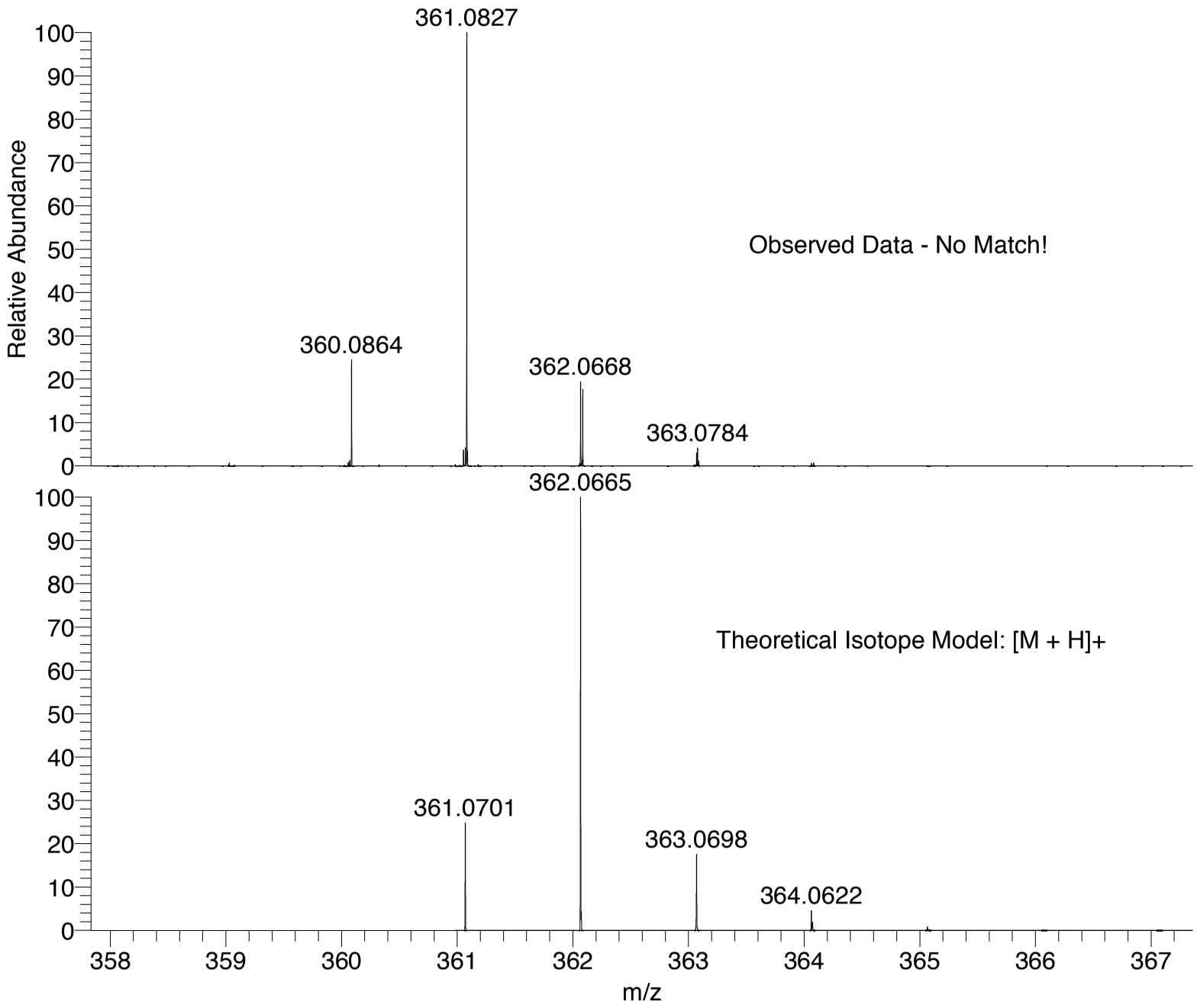
EPRSC National Facility Swansea
LTQ Orbitrap XL

C16H13BFNO5S
21/01/2016 07:27:36 AM

SUSSPE_N4PT1_8 #29-45 RT: 0.67-1.05 AV: 16 NL: 2.83E7
T: FTMS + p NSI Full ms [140.00-1935.00]



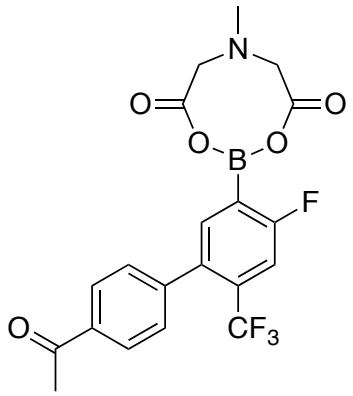

376.0822
375.0859
377.0854
378.0966
379.0930
380.0963
Observed Data - MIXTURE!
NL:
2.22E7
SUSSPE_N4PT1_8#29-45
RT: 0.67-1.05 AV: 16 T: FTMS
+ p NSI Full ms
[140.00-1935.00]
379.0930
378.0966
380.0960
381.0888
382.0922
383.0946
Theoretical Isotope Model: [M + NH4]+
NL:
1.47E4
C16 H13 BFNO5 SNH4:
C16 H17 B1 F1 N2 O5 S1
p (gss, s /p:40) Chrg 1
R: 100000 Res .Pwr . @FWHM
Relative Abundance
m/z
0
10
20
30
40
50
60
70
80
90
100
375
376
377
378
379
380
381
382
383



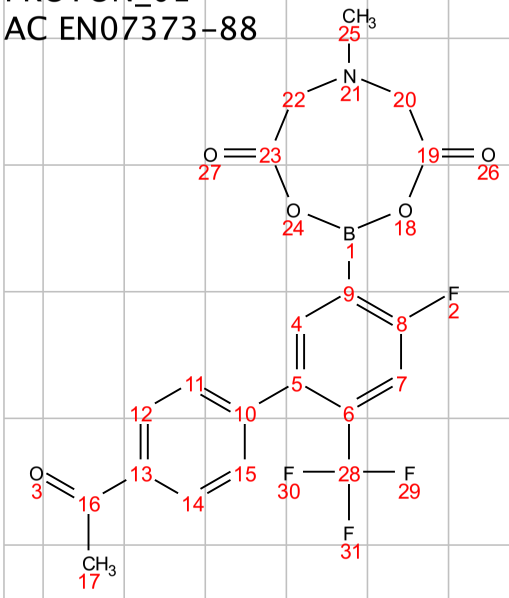
NL:
1.09E7
SUSSPE_N4PT1_8#29-45
RT: 0.67-1.05 AV: 16 T: FTMS
+ p NSI Full ms
[140.00-1935.00]

NL:
1.48E4
$C_{16}H_{13}BFNO_5$ SH:
$C_{16}H_{14}B_1F_1N_1O_5S_1$
p (gss, s /p:40) Chrg 1
R: 100000 Res .Pwr . @FWHM

4'-Acetyl-4-fluoro-6-(trifluoromethyl)-[1,1'-biphenyl]-3-yl MIDA boronate 4c



PROTON_01
AC EN07373-88



8.06 8.06 8.04 8.04 7.58 7.58 7.56 7.56 7.52 7.50

4.21 4.18 4.03 4.03 4.00

2.75 2.64 2.64

E (m) 7.57

D (m) 8.05

F (m) 7.51

C (d) 4.20

B (d) 4.02

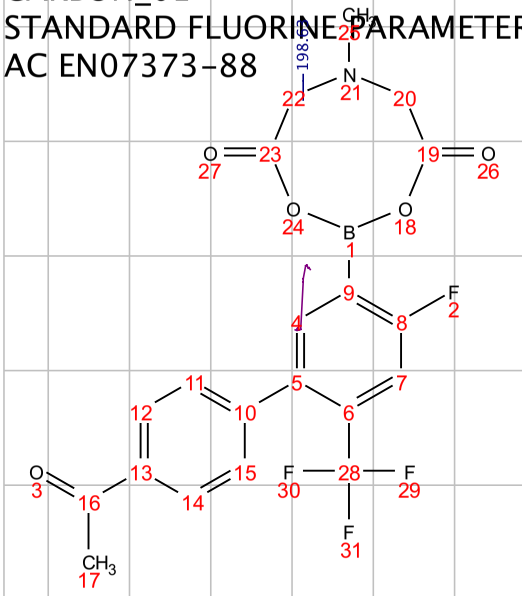
25 (s) 2.75

17 (s) 2.64

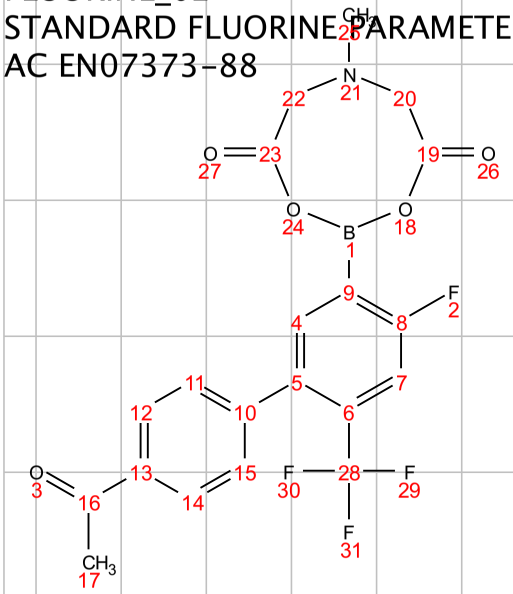
1.56 1.95 1.87 2.11 2.14 2.55 3.16

f1 (ppm)

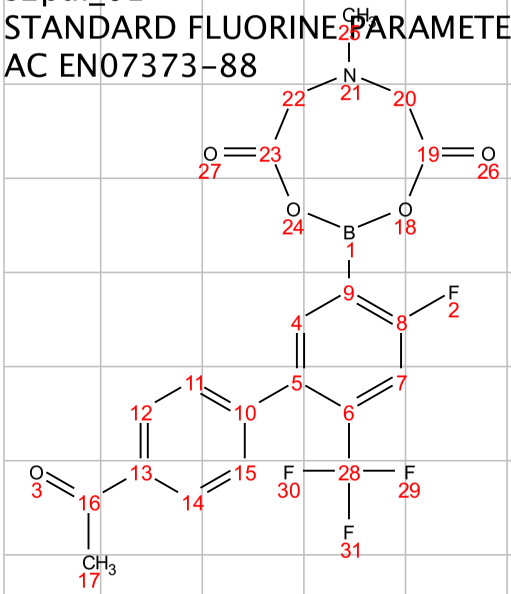
CARBON_01
STANDARD FLUORINE PARAMETERS
AC EN07373-88



FLUORINE_02
STANDARD FLUORINE PARAMETERS
AC EN07373-88



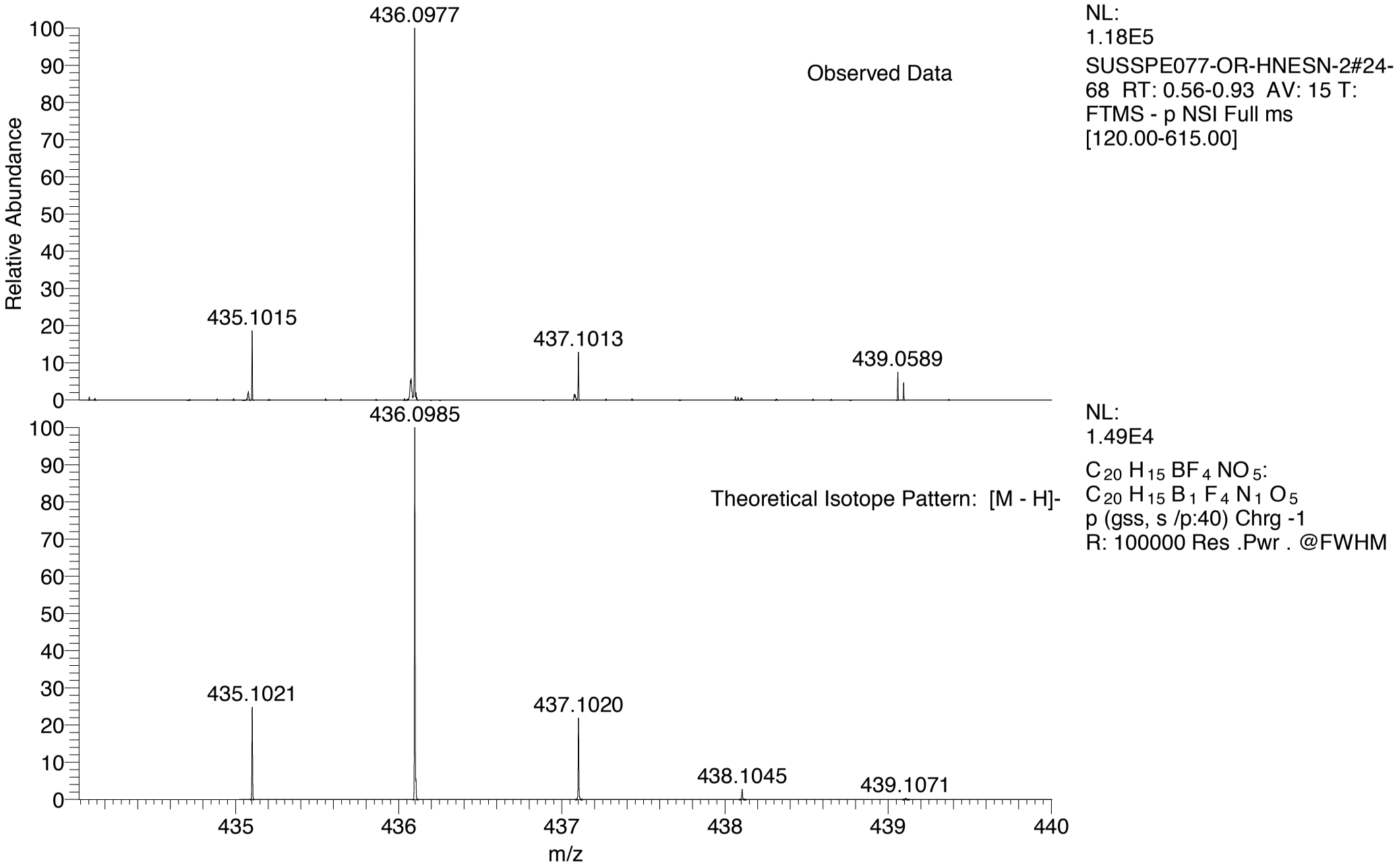
s2pul_01
STANDARD FLUORINE PARAMETERS
AC EN07373-88



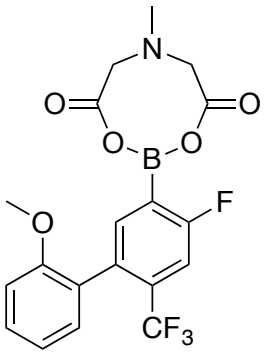
EN07373-87 MW=437?
C20H16BF4NO5
(MeOH)/MeOH+DEA

EPSRC National Facility Swansea
LTQ Orbitrap XL

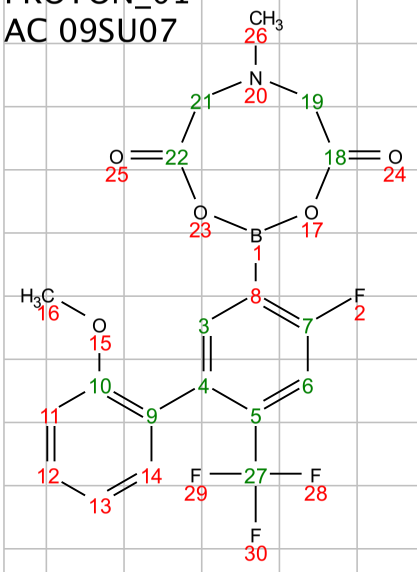
Adam Close
12/11/2015 09:46:31



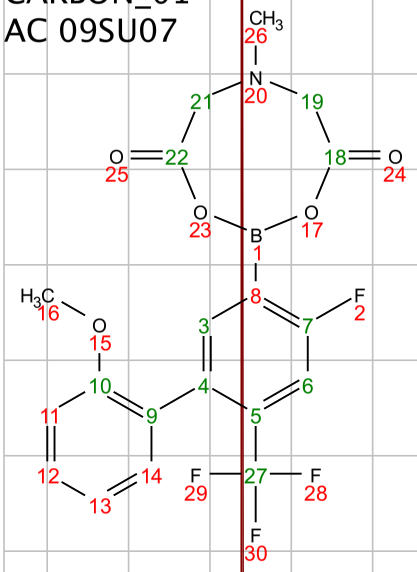
4-Fluoro-2'-methoxy-6-(trifluoromethyl)-[1,1'-biphenyl]-3-yl MIDA boronate 4d



PROTON_01
AC 09SU07



CARBON_01
AC 09SU07



4 (s)
128.67

9 (s)
134.39

18,22 (s)
168.66

C (s)
130.50

A (s)
111.75

G (s)
55.83

7 (d)
165.66

3 (d)
140.51

27 (dd)
124.46

6 (dq)
113.65

F (d)
63.75

H (s)
48.50

10 (s)
157.99

D (s)
131.53

B (s)
120.66

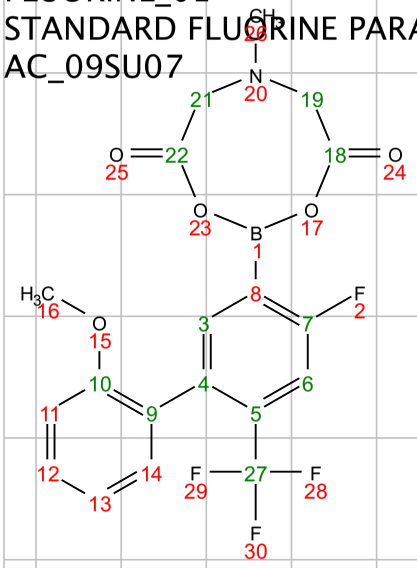
5 (dd)
132.42

18,22
7
10
3
9
5
4
27
6

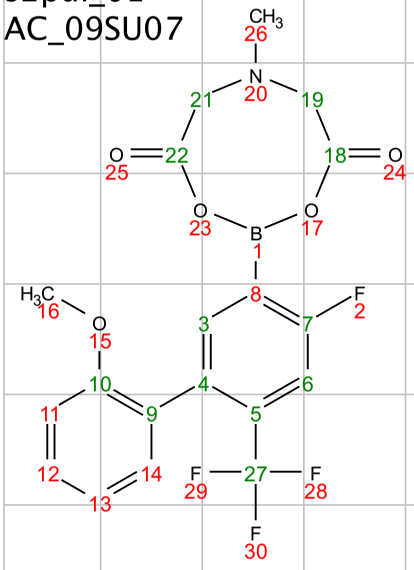
2.08
1.00
1.00
3.75
1.02
0.68
3.78
3.41
0.74
0.59
3.09
3.75
3.57
8.42
3.09
4.18

f1 (ppm)

FLUORINE_01
STANDARD FLUORINE PARAMETERS
AC_09SU07



s2pul_01
AC_09SU07



10.77

A (s)
10.77

1.00

f1 (ppm)

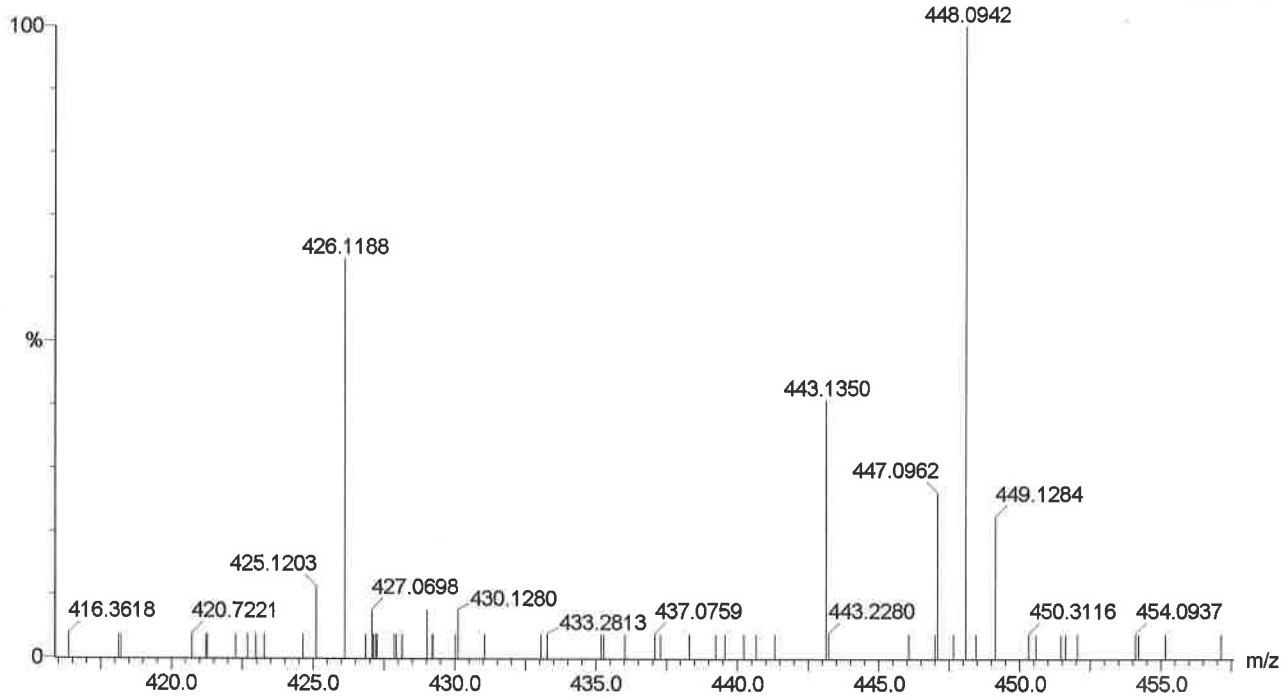
Single Mass Analysis

Tolerance = 100.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
6 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 19-19 H: 0-50 11B: 0-1 N: 1-1 O: 5-5 F: 4-4 Na: 0-2

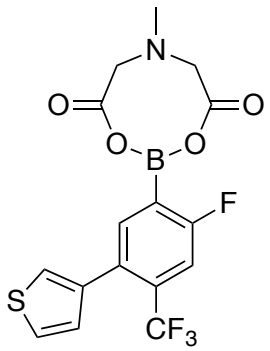
09SU07
ADAM4483 281 (3.787)

1: TOF MS ES+
2.73e+001



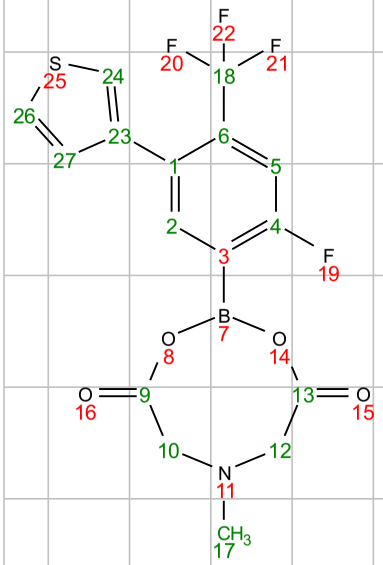
Minimum:				-1.5		
Maximum:		5.0	100.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
448.0942	448.0955	-1.3	-2.9	10.5	0.7	C19 H16 11B N O5 F4 Na

2-Fluoro-5-(thiophen-3-yl)-4-(trifluoromethyl)phenyl MIDA boronate 4e




PROTON_01
AC_EN07373-87
7.61 [2]
7.59 [2]
7.52 [5]
7.50 [5]
7.49 [27]
7.48 [27]
7.48 [27]
7.47 [27]
7.41 [24]
7.40 [24]
7.18 [26]
7.17 [26]
4.19 [10,12]
4.14 [10,12]
4.00 [10,12]
3.96 [10,12]
2.71 [17]
1.94 CD3CN
26 (d)
7.18
27 (dd)
7.48
5 (d)
7.51
2 (d)
7.60
24 (d)
7.41
10,12 (d)
4.16
10,12 (d)
3.98
17 (s)
2.71
17
10,12
10,12
24
27
5
2
26
0.83
1.00
1.05
0.90
0.87
1.91
1.95
2.76
f1 (ppm)

CARBON_02
AC_EN07373-87



168.99 [9,13]
166.93 [4]
164.52 [4]
140.38 [2]
140.29 [2]
139.59 [23]
132.88 [1]
132.87 [1]
132.09 [6]
132.00 [6]
131.78 [6]
131.69 [6]
131.47 [6]
131.39 [6]
131.17 [6]
131.08 [6]
130.09 [26]
130.07 [26]
130.05
126.52 [27]
125.90 [18]
125.87 [18]
125.34 [24]
125.32 [24]
125.30 [24]
125.28 [24]
123.18 [18]
123.15 [18]
114.62 [5]
114.57 [5]
114.51 [5]
114.45 [5]
114.33 [5]
114.27 [5]
114.21 [5]
114.16 [5]
63.80 [10,12]
63.77 [10,12]
48.72 [17]
48.70 [17]
-1.32 CDCN

6 (qd)
131.58

18 (dd)
124.53

9,13 (s)
168.99

23 (s)
139.59

5 (dq)
114.39

4 (d)
165.72

2 (d)
140.33

27 (s)
126.52

10,12 (d)
63.79

17 (d)
48.71

26 (d)
130.08

24 (d)
125.31

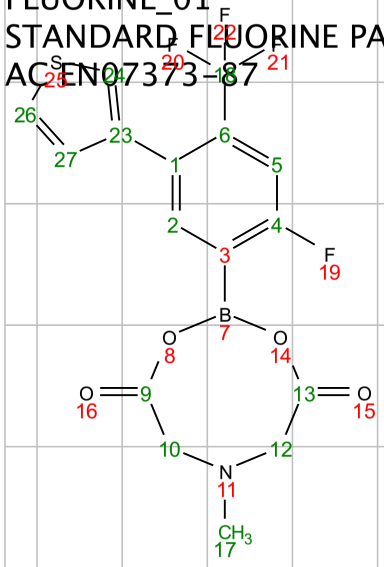
1 (m)
132.87

9,13
4
23
2
6
26
1
27
24
18
5
10,12
17

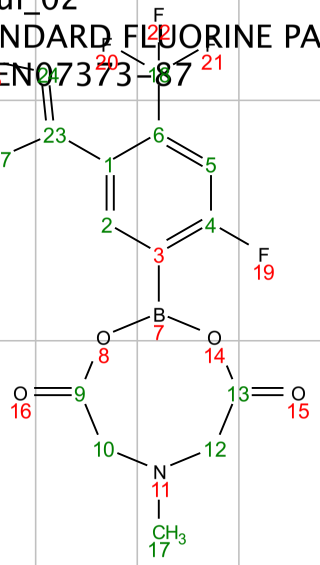
1.99
1.00
2.67
0.82
0.77
0.84
1.75
2.39
1.91
0.78
2.93
5.42
2.70

f1 (ppm)

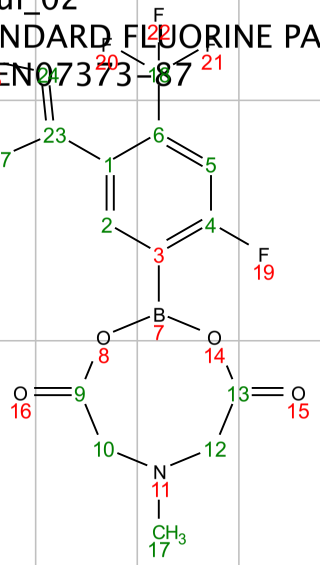
FLUORINE_01
STANDARD FLUORINE PARAMETERS
ACEN07373-87



s2pul_02
STANDARD FLUORINE PARAMETERS
ACEN07373-87



s2pul_02
STANDARD FLUORINE PARAMETERS
ACEN07373-87



Elemental Composition Report

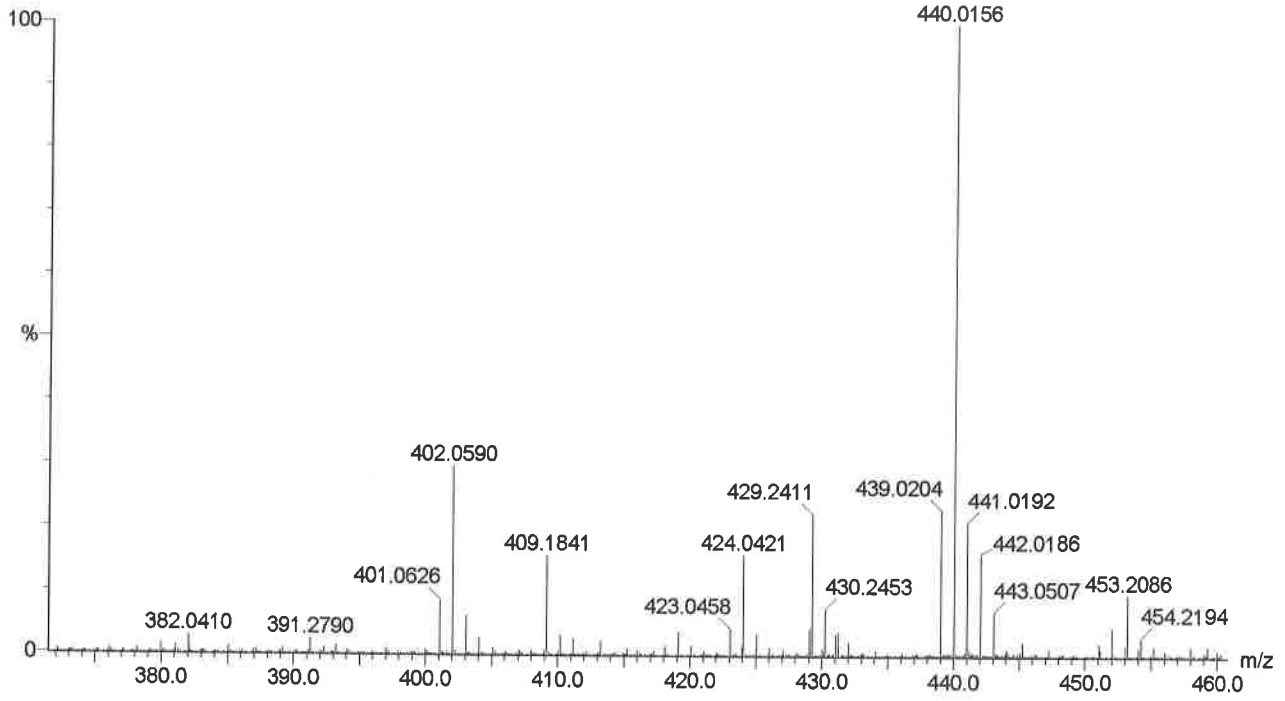
Single Mass Analysis

Tolerance = 100.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
1 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 16-16 H: 13-13 11B: 0-1 N: 1-1 O: 4-4 F: 4-4 S: 1-1

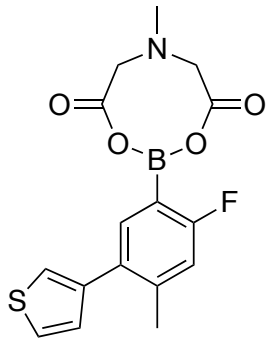
E1V07373-87
ADAM4465 149 (2.013)

1: TOF MS ES+
2.25e+003

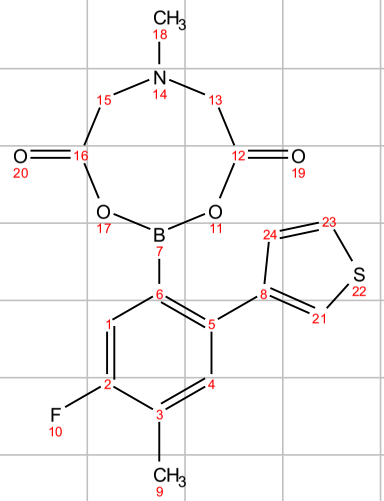


Minimum:				-1.5		
Maximum:		5.0	100.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
402.0590	402.0594	-0.4	-1.0	9.5	1.0	C16 H13 11B N O4 F4 S

2-Fluoro-4-methyl-5-(thiophen-3-yl)phenyl MIDA boronate 4f



140616.280.fid
Name Adam Close
M No kzmr981
Notebook Ref EN07373-64
proton.az DMSO /opt/topspin2.1 chem 36



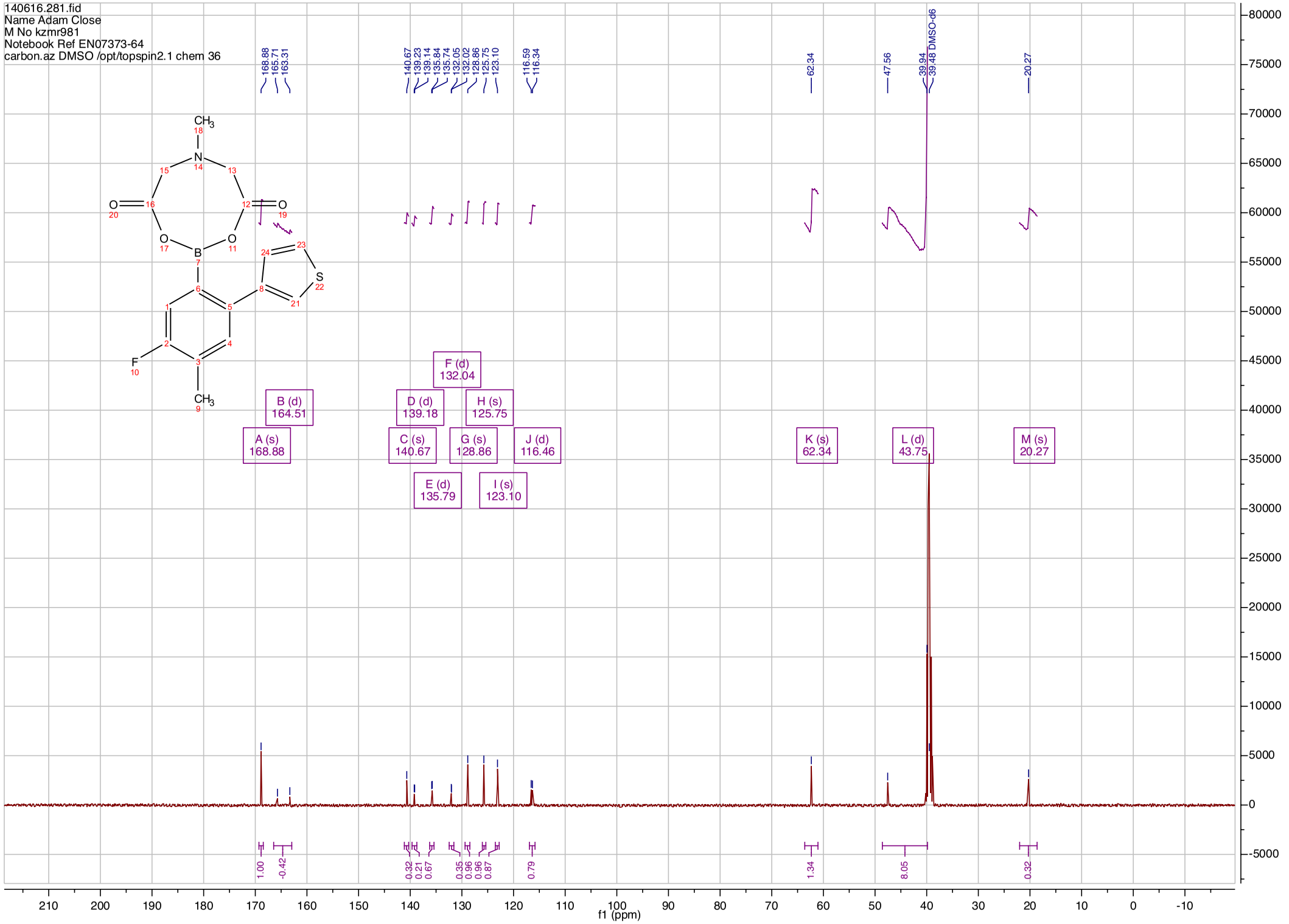
7.63 7.62 7.62 7.61 7.52 7.52 7.51 7.39 7.37 7.25 7.23 7.23 7.09 7.06

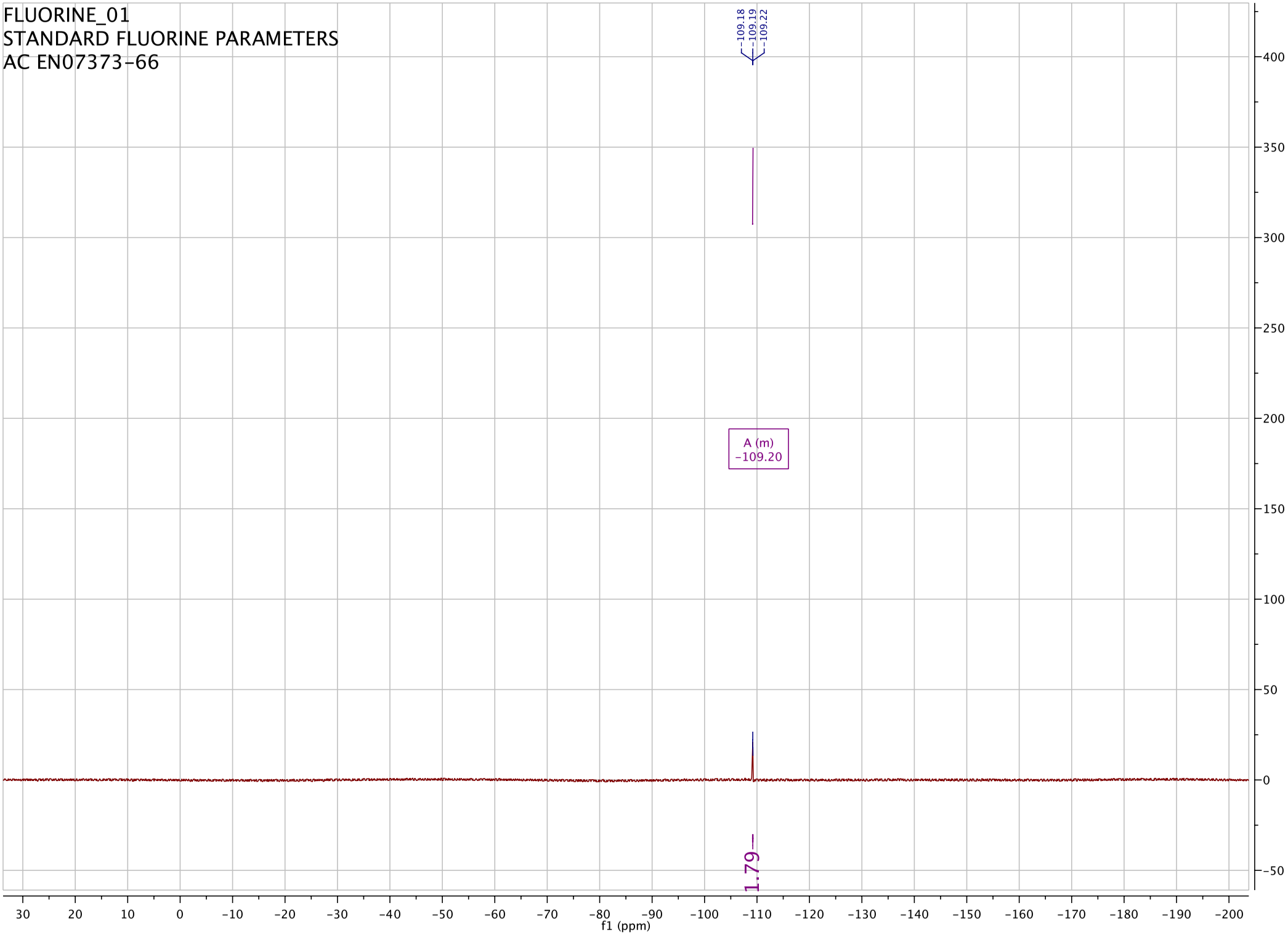
4.43 4.38 4.11 4.07

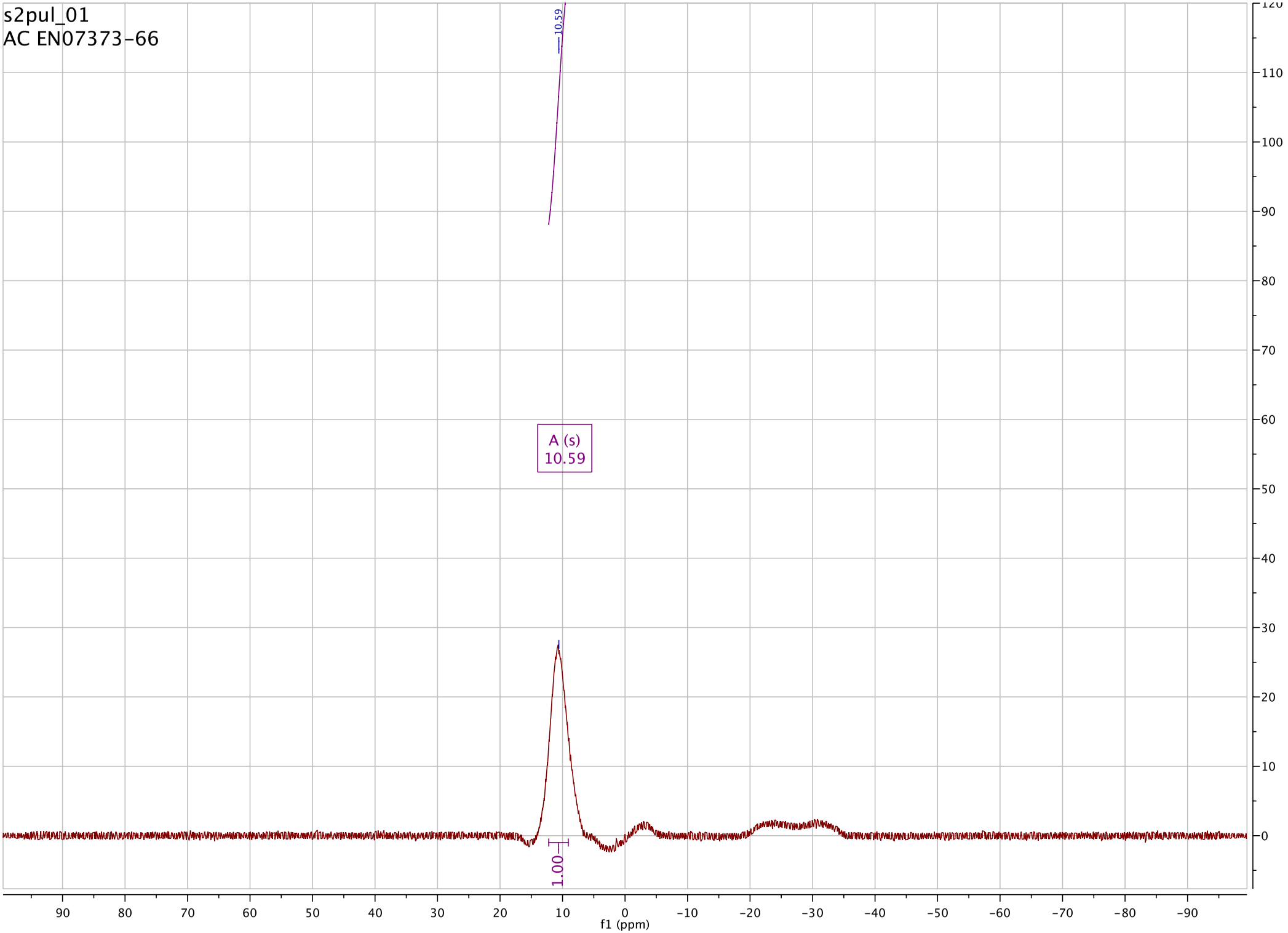
E (dd) 7.62
B (d) 7.24
1 (d) 7.07
4 (d) 7.38
D (m) 7.52
C (d) 4.09
A (d) 4.40

1.03 0.99 1.00 1.02 1.00
2.21 2.30

f1 (ppm)


140616.281.fid
Name Adam Close
M No kzmr981
Notebook Ref EN07373-64
carbon.az DMSO /opt/topspin2.1 chem 36
168.88
165.71
163.31
140.67
139.23
139.14
135.84
135.74
132.05
132.02
128.86
125.75
123.10
116.59
116.34
62.34
47.56
39.94
39.48 DMSO-d6
20.27
A (s) 168.88
B (d) 164.51
C (s) 140.67
D (d) 139.18
E (d) 135.79
F (d) 132.04
G (s) 128.86
H (s) 125.75
I (s) 123.10
J (d) 116.46
K (s) 62.34
L (d) 43.75
M (s) 20.27
1.00
-0.42
0.32
0.21
0.67
0.35
0.96
0.96
0.87
0.79
1.34
8.05
0.32
f1 (ppm)


FLUORINE_01
STANDARD FLUORINE PARAMETERS
AC EN07373-66
-109.18
-109.19
-109.22
A (m)
-109.20
1.79
400
350
300
250
200
150
100
50
0
-50
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)


s2pul_01
AC EN07373-66
10.59
A (s)
10.59
1.00
f1 (ppm)

AstraZeneca

Mass Spectrometry Analysis Report

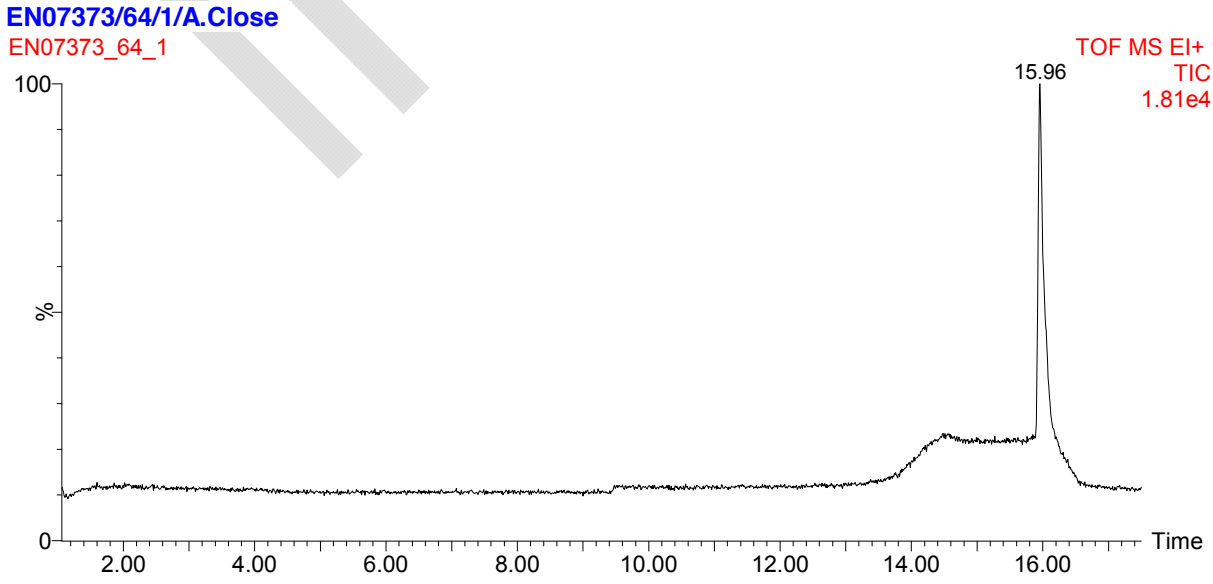
Summary:

The peak seen at RT 15.96 m/z 347 is consistent with being the required product. Accurate mass and fragments are consistent with required structure.

Raw data File: EN07373_64_1

ELN Reference:	EN07373_64_1	Date:	18th June 2014
Chemist:	A.Close	Analyst:	P.Davey
Ion Type:	EI +ve	Empirical Formula:	C16H15FNBO4S
Analysis Method:	Accurate mass GCMS	Purity	100% (MS TIC)
Instrument:	Waters GCT/HP6890	Retention Time (mins):	15.96
Instrument method:	EI & GCEI	Accurate mass (obs):	347.0792
Column:	VF-5ms, 25m, 0.32mm i.d. 0.52um	Accurate mass (Calc):	347.0799
Column temp:	Ramped	Error (ppm):	2.0

Chromatogram



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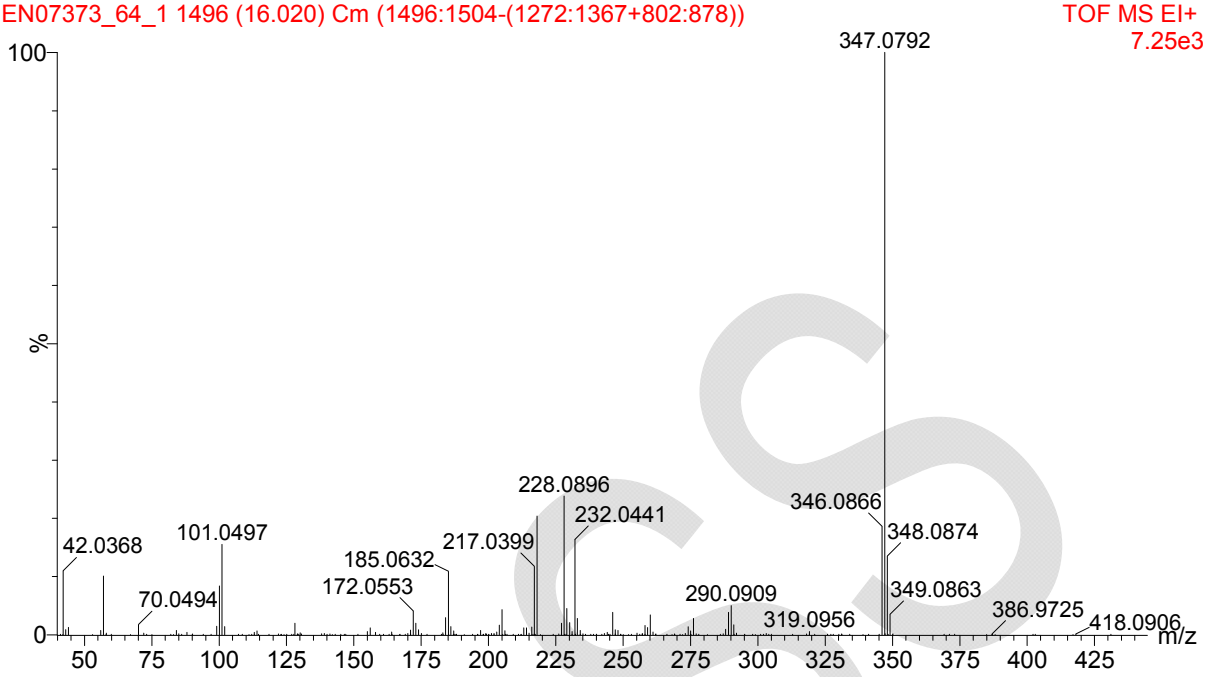
Spectra

EN07373/64/1/A.Close

EN07373_64_1 1496 (16.020) Cm (1496:1504-(1272:1367+802:878)) TOF MS EI+ 7.25e3



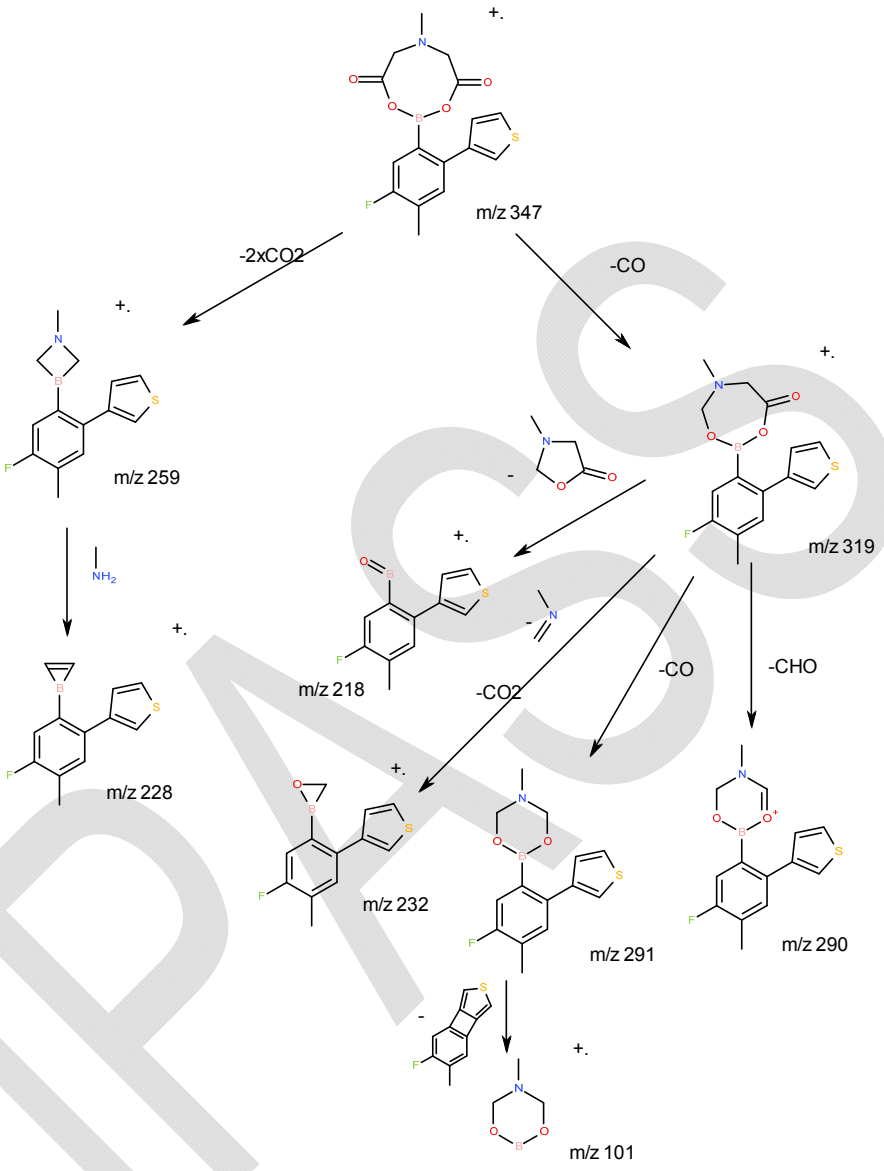
Elemental Composition Report

Single Mass Analysis
Tolerance = 30.0 PPM / DBE: min = -1.5, max = 30.0
Element prediction: Off

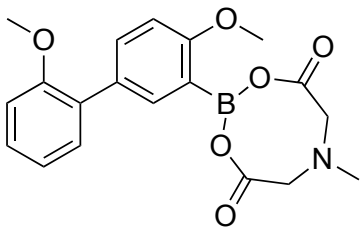
Monoisotopic Mass, Odd and Even Electron Ions
73 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-16 H: 0-16 11B: 0-1 N: 0-1 O: 0-4 F: 0-1 S: 0-1

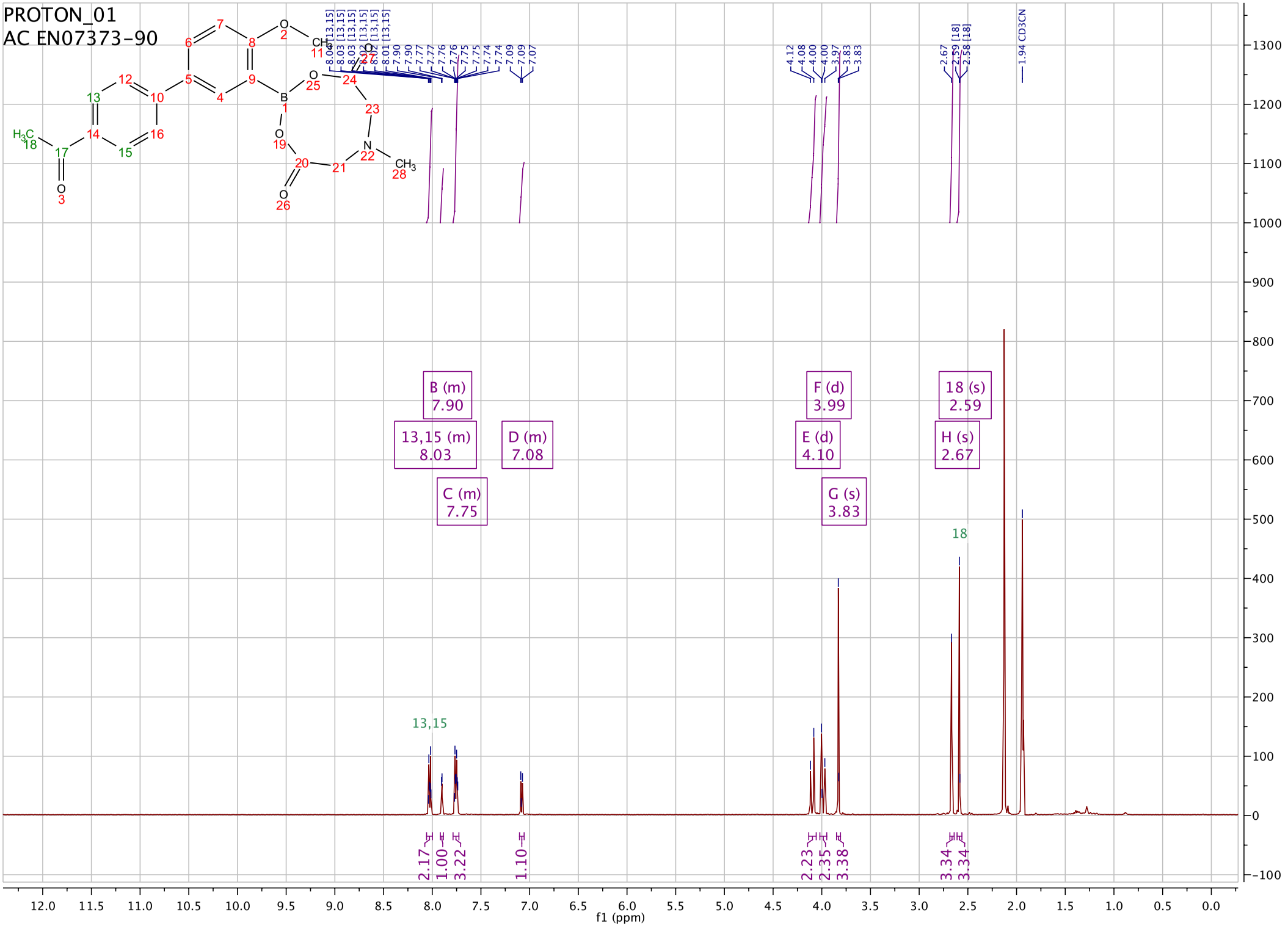
Minimum:				-1.5		
Maximum:		10.0		30.0		30.0
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
347.0792	347.0799	-0.7	-2.0	10.0	177.6	C16 H15 11B N O4 F S

Fragmentation

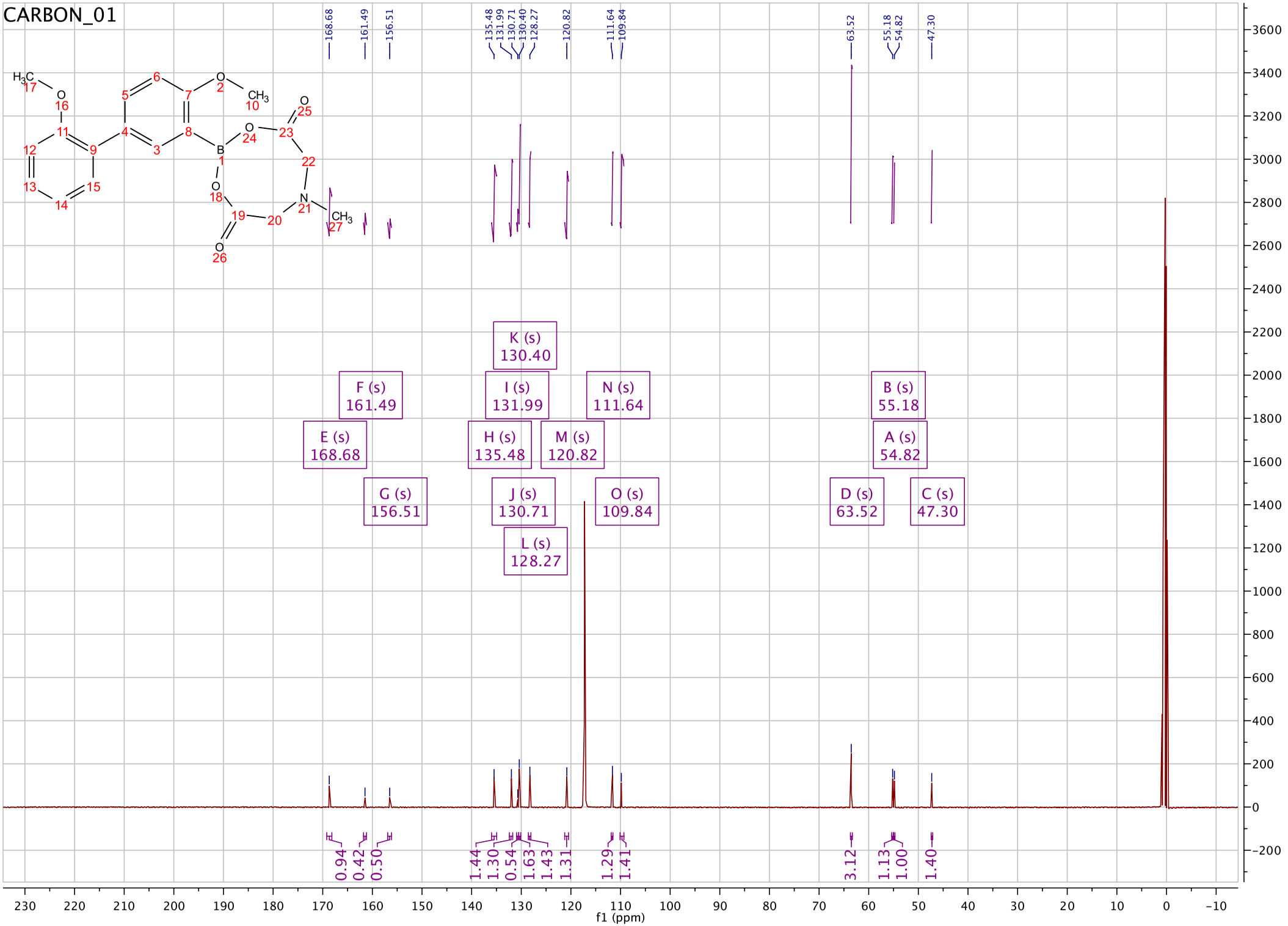


2',4-Dimethoxy-[1,1'-biphenyl]-3-yl MIDA boronate 4g

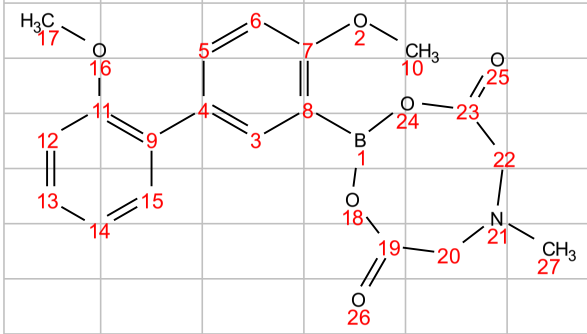



PROTON_01
AC EN07373-90
B (m)
7.90
13,15 (m)
8.03
C (m)
7.75
D (m)
7.08
F (d)
3.99
E (d)
4.10
G (s)
3.83
18 (s)
2.59
H (s)
2.67
13,15
18
2.17
1.00
3.22
1.10
2.23
2.35
3.38
3.34
3.34
f1 (ppm)


PROTON_01
AC_08SU04PPDRY
7.66
7.59
7.58
7.57
7.34
7.33
7.32
7.09
7.07
7.05
7.04
7.02
7.01
4.12
4.09
4.01
3.98
3.83
3.81
2.68
B (s)
7.66
A (m)
7.58
D (m)
7.05
C (m)
7.33
H (s)
3.81
F (d)
3.99
E (d)
4.10
G (s)
3.83
I (s)
2.68
0.88
0.90
1.95
3.01
2.51
2.20
3.45
3.28
3.32
f1 (ppm)


CARBON_01
168.68
161.49
156.51
135.48
131.99
130.71
130.40
128.27
120.82
111.64
109.84
63.52
55.18
54.82
47.30
K (s) 130.40
F (s) 161.49
I (s) 131.99
N (s) 111.64
B (s) 55.18
E (s) 168.68
H (s) 135.48
M (s) 120.82
A (s) 54.82
G (s) 156.51
J (s) 130.71
O (s) 109.84
D (s) 63.52
C (s) 47.30
L (s) 128.27
0.94
0.42
0.50
1.44
1.30
0.54
1.63
1.43
1.31
1.29
1.41
3.12
1.13
1.00
1.40
f1 (ppm)

s2pul_04
AC_EN07373-90

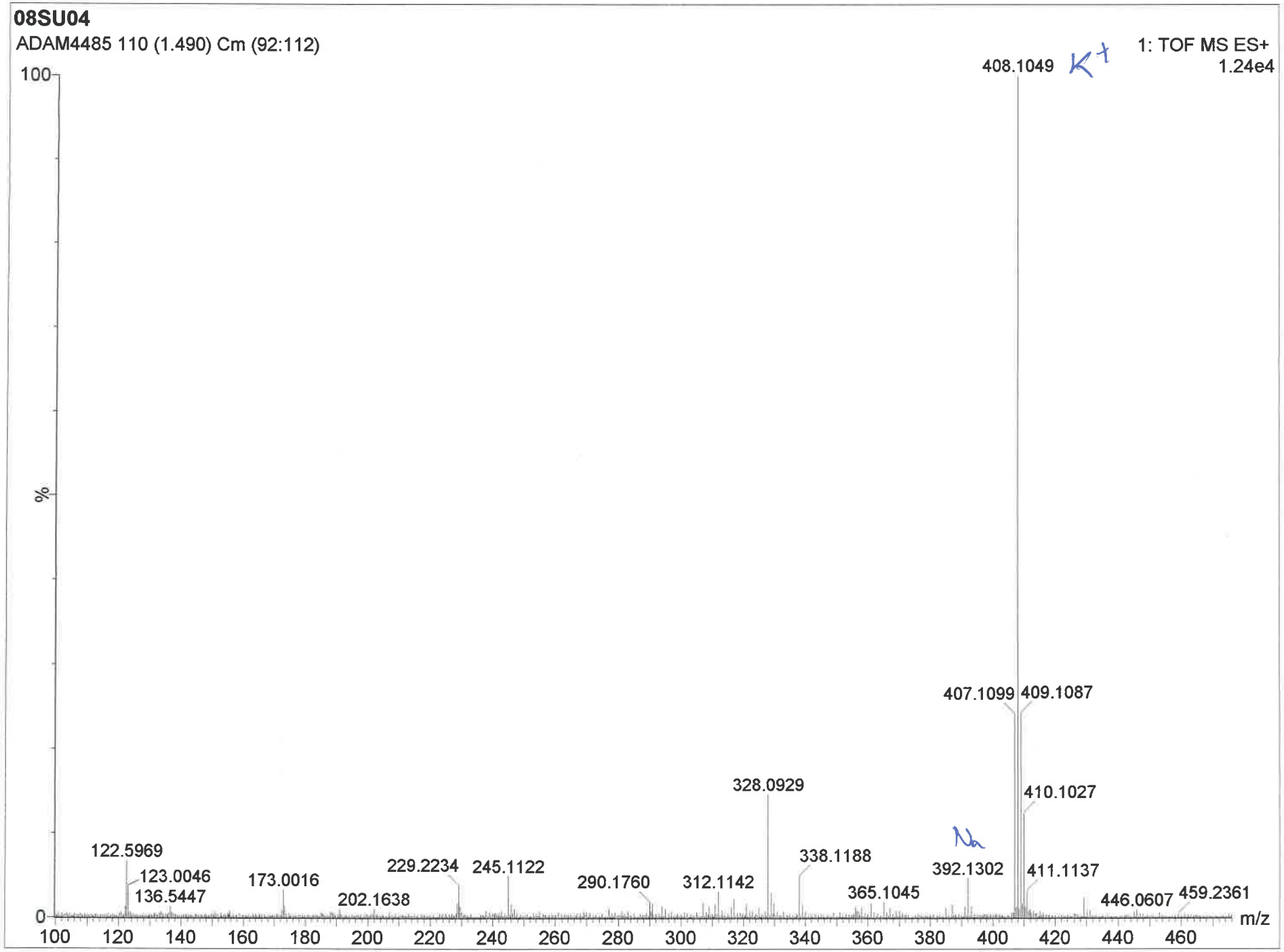


11.95

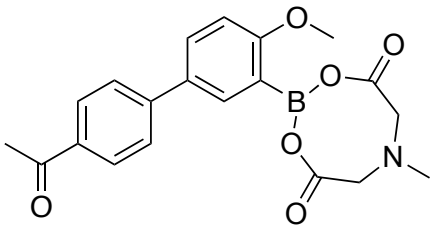
A (s)
11.95

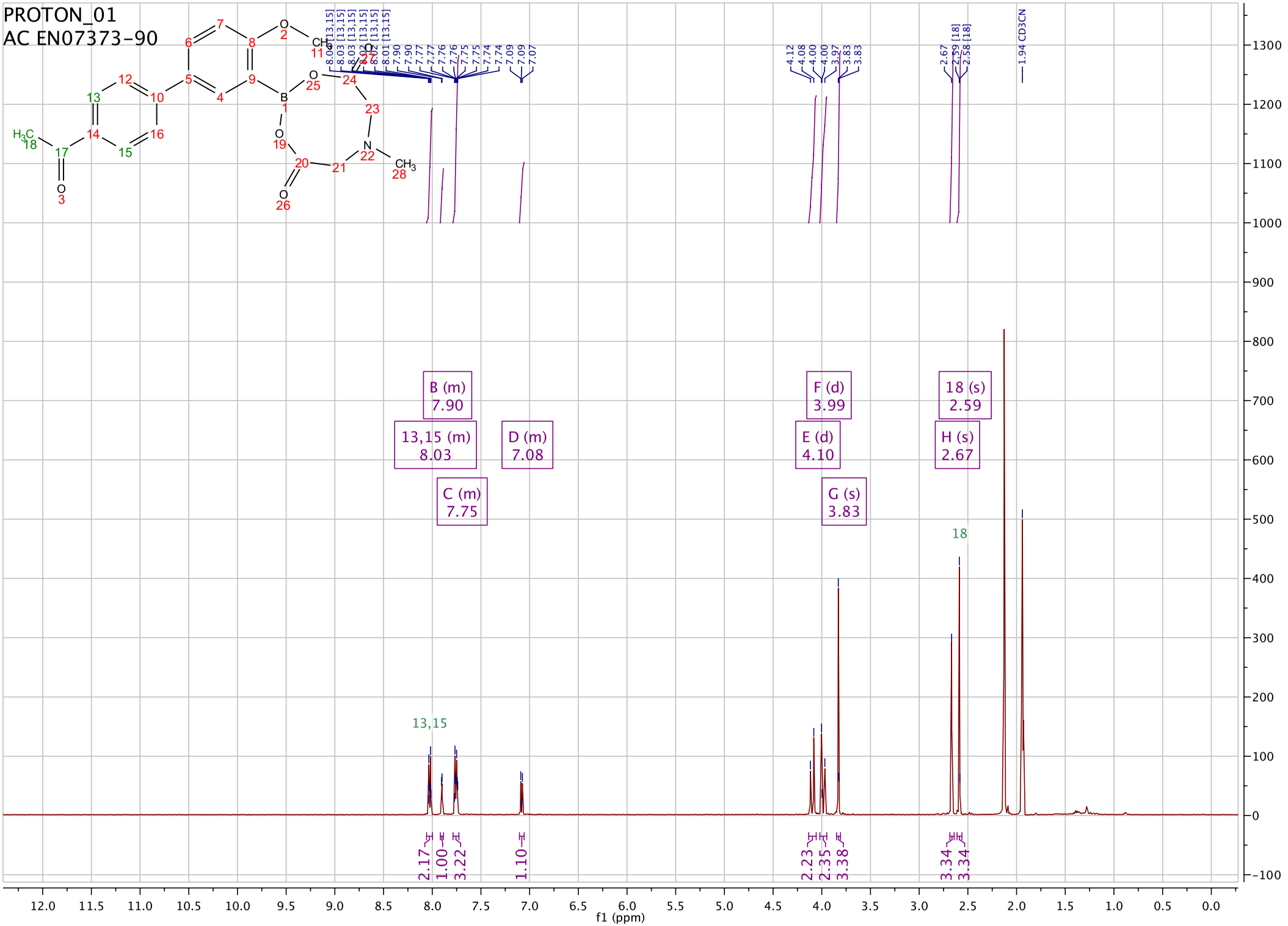
1.00

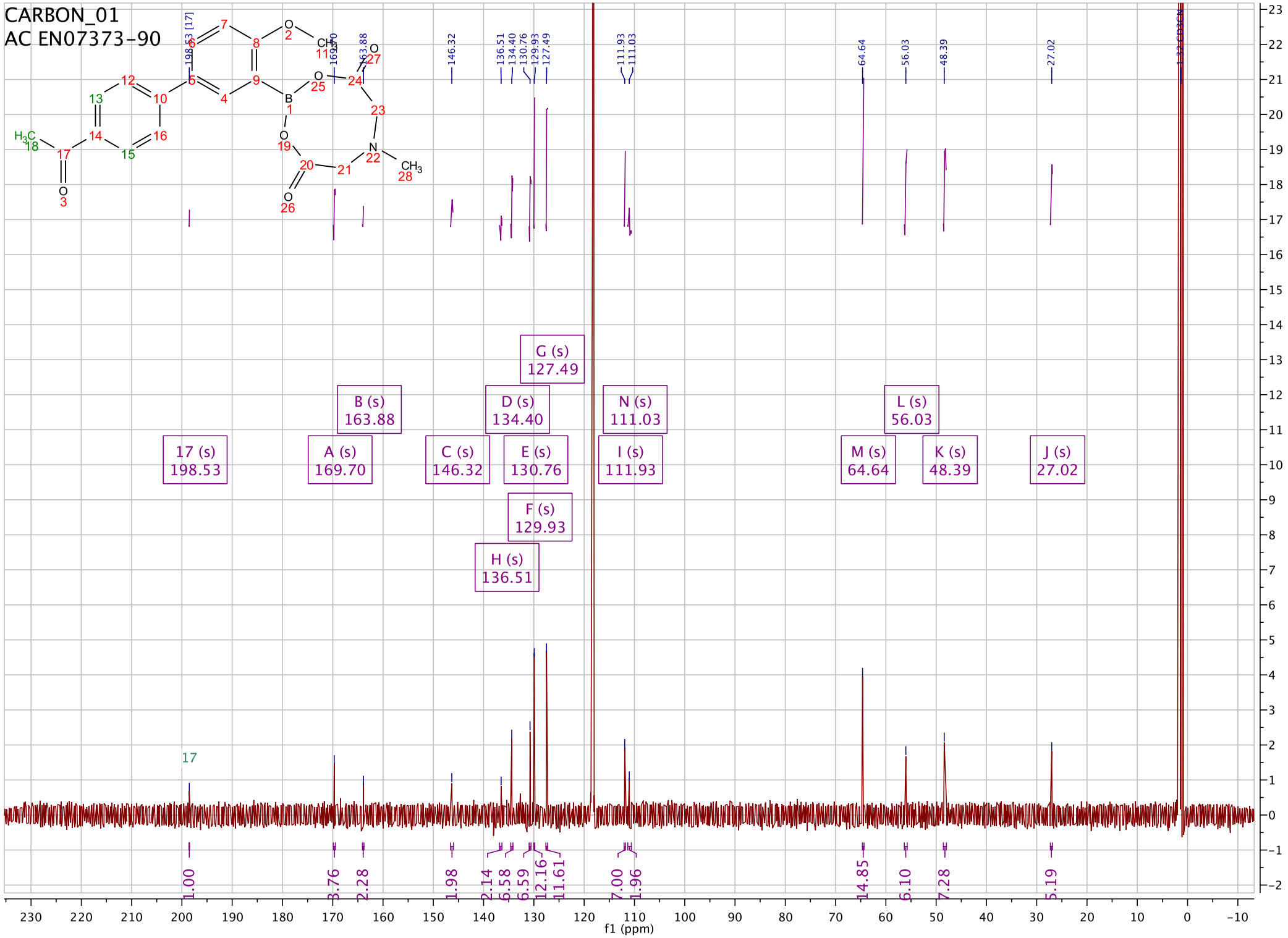
f1 (ppm)

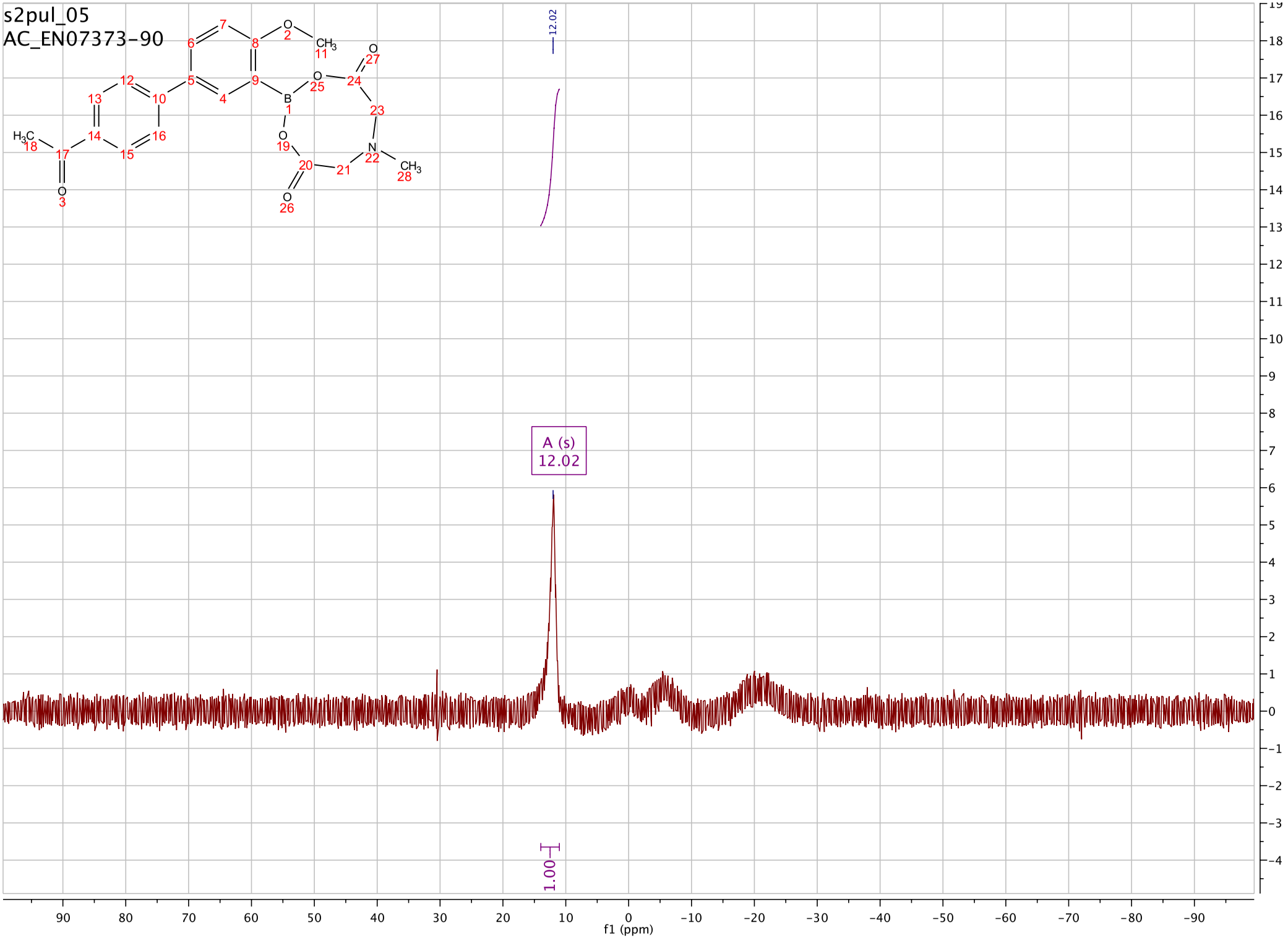

08SU04
ADAM4485 110 (1.490) Cm (92:112)
1: TOF MS ES+
1.24e4
408.1049
K+
100
%
0
407.1099
409.1087
328.0929
410.1027
Na
122.5969
123.0046
136.5447
173.0016
202.1638
229.2234
245.1122
290.1760
312.1142
338.1188
365.1045
392.1302
411.1137
446.0607
459.2361
m/z
100
120
140
160
180
200
220
240
260
280
300
320
340
360
380
400
420
440
460

4'-Acetyl-4-methoxy-[1,1'-biphenyl]-3-yl MIDA boronate 4h




PROTON_01
AC EN07373-90
B (m)
7.90
13,15 (m)
8.03
C (m)
7.75
D (m)
7.08
F (d)
3.99
E (d)
4.10
G (s)
3.83
18 (s)
2.59
H (s)
2.67
13,15
18
2.17
1.00
3.22
1.10
2.23
2.35
3.38
3.34
3.34
f1 (ppm)


CARBON_01
AC EN07373-90
198.53 [17]
169.70
163.88
146.32
136.51
134.40
130.76
129.93
127.49
111.93
111.03
64.64
56.03
48.39
27.02
1.33 CH3CN
G (s)
127.49
B (s)
163.88
D (s)
134.40
N (s)
111.03
L (s)
56.03
17 (s)
198.53
A (s)
169.70
C (s)
146.32
E (s)
130.76
I (s)
111.93
M (s)
64.64
K (s)
48.39
J (s)
27.02
F (s)
129.93
H (s)
136.51
17
1.00
3.76
2.28
1.98
2.14
6.58
6.59
12.16
11.61
7.00
1.96
14.85
6.10
7.28
5.19
f1 (ppm)


s2pul_05
AC_EN07373-90
12.02
A (s)
12.02
1.00
f1 (ppm)

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Mass Spectrometry Analysis Report

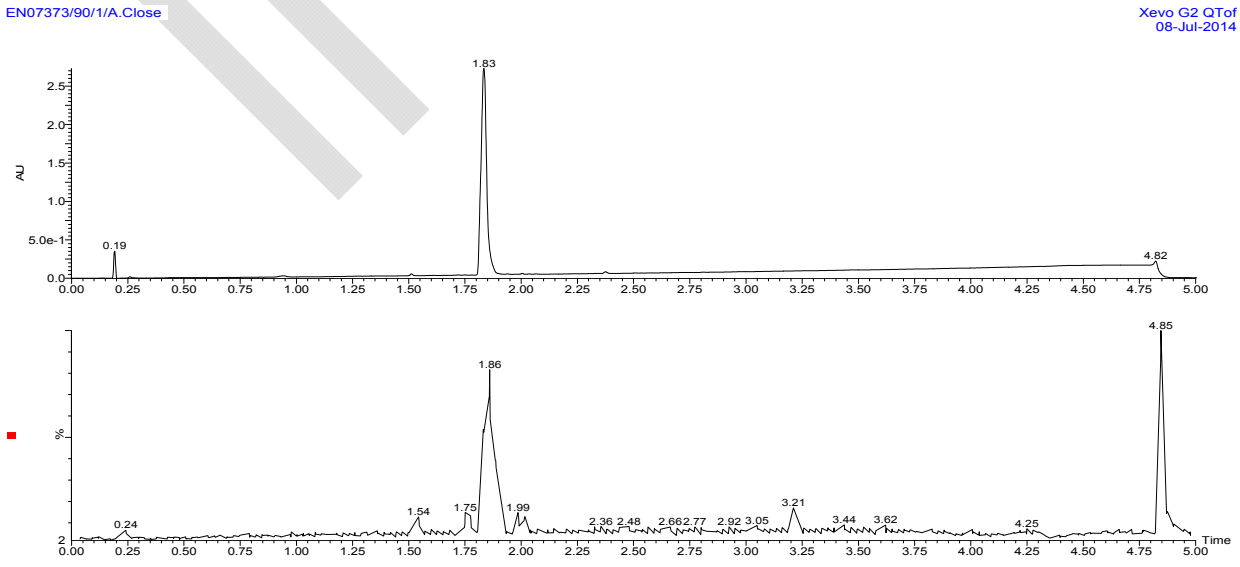
Summary:

The peak seen at RT: 1.88 mins m/z 382 is the required product. Accurate mass, fragments and generated empirical formula are consistent with the required structure. Unfortunately MSMS data was only obtained on the 2M+H+ ion, but some fragments seen were consistent with the parent ion.

Raw data File: EN07373_90_1

ELN Reference:	EN07373_90_1	Date:	8th July 2014
Chemist	A.Close	Analyst:	P.Davey
Ion Type:	ESI +ve	Empirical Formula:	C20H20BNO6
Analysis Method:	Acid5min	Purity	99%
Instrument:	Waters Xevo G2 Qtof	Retention Time (mins):	1.88
Instrument method:	Open Access	Accurate mass (obs):	382.1451
Column:	Acquity 50 x 2.1 C18 BEH 1.7um	Accurate mass (Calc):	382.1462
Column temp:	40C	Error (ppm):	2.9

Chromatogram



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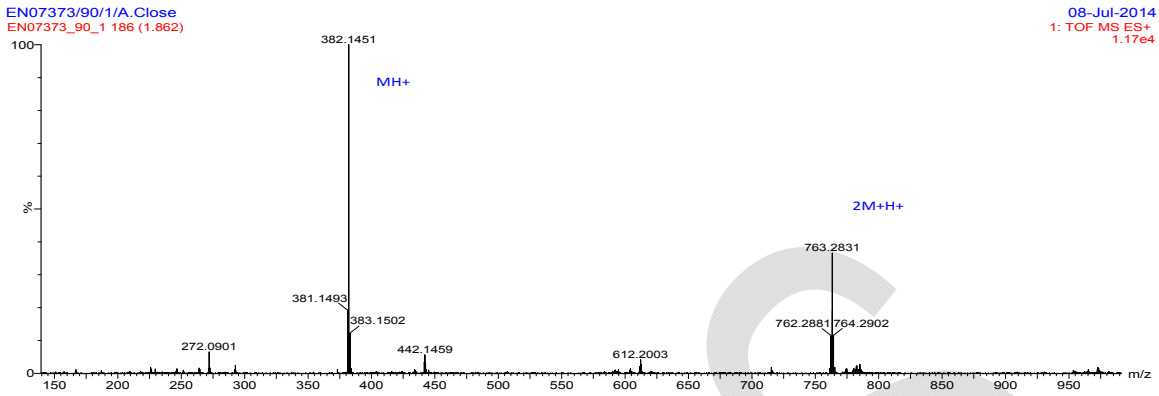
MS



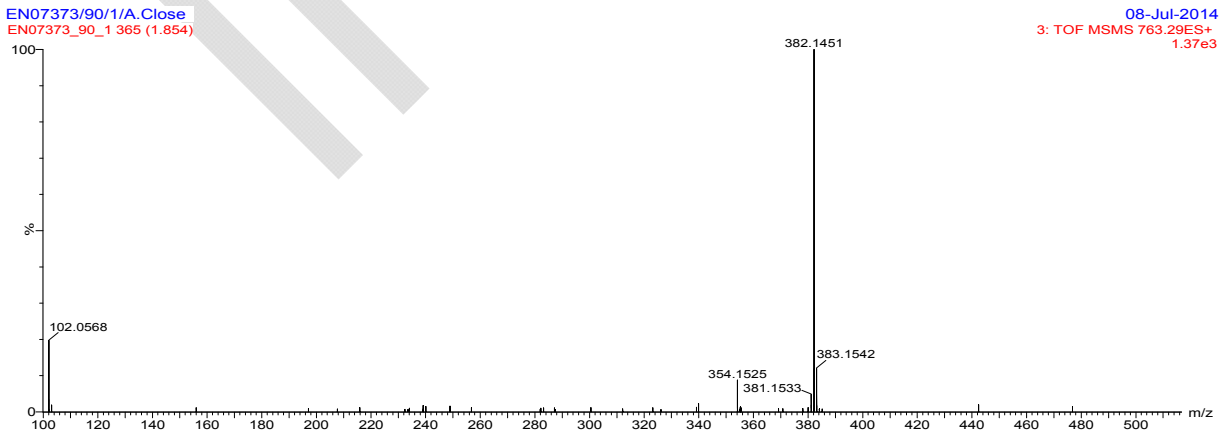
Elemental Composition Report

Single Mass Analysis
Tolerance = 3.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

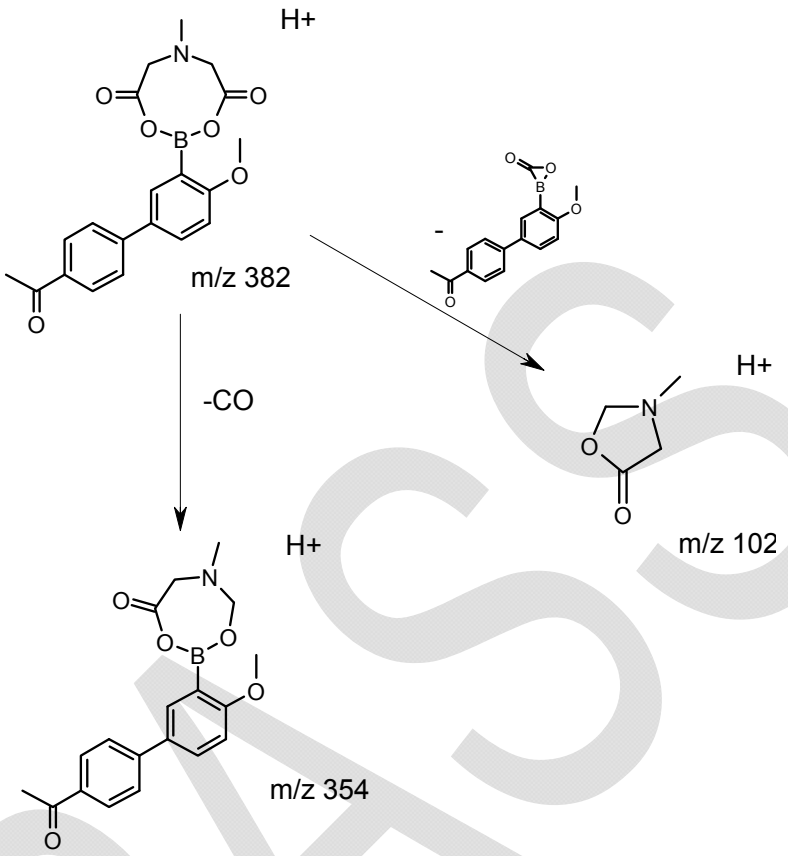
Monoisotopic Mass, Even Electron Ions
43 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-20 H: 0-22 B: 0-1 N: 0-1 O: 0-6 Na: 0-1

Minimum:				-1.5	
Maximum:		5.0	3.0	50.0	
Mass	Calc. Mass	mDa	PPM	DBE	Formula
382.1451	382.1462	-1.1	-2.9	11.5	C20 H21 B N O6

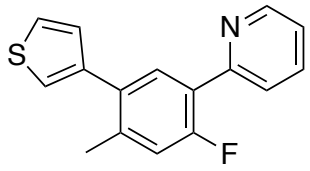
MSMS (of (2M+H)+ ion)

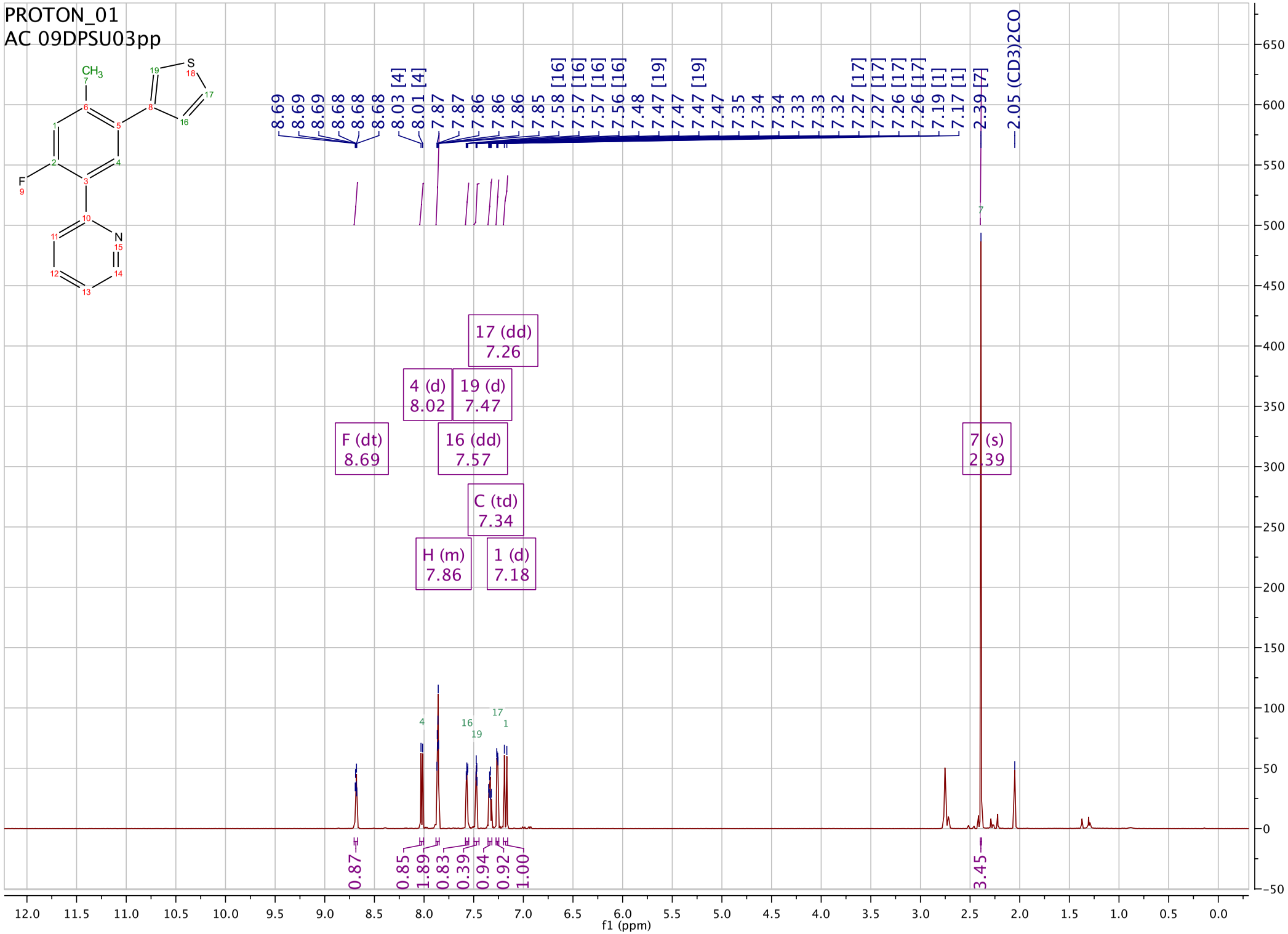


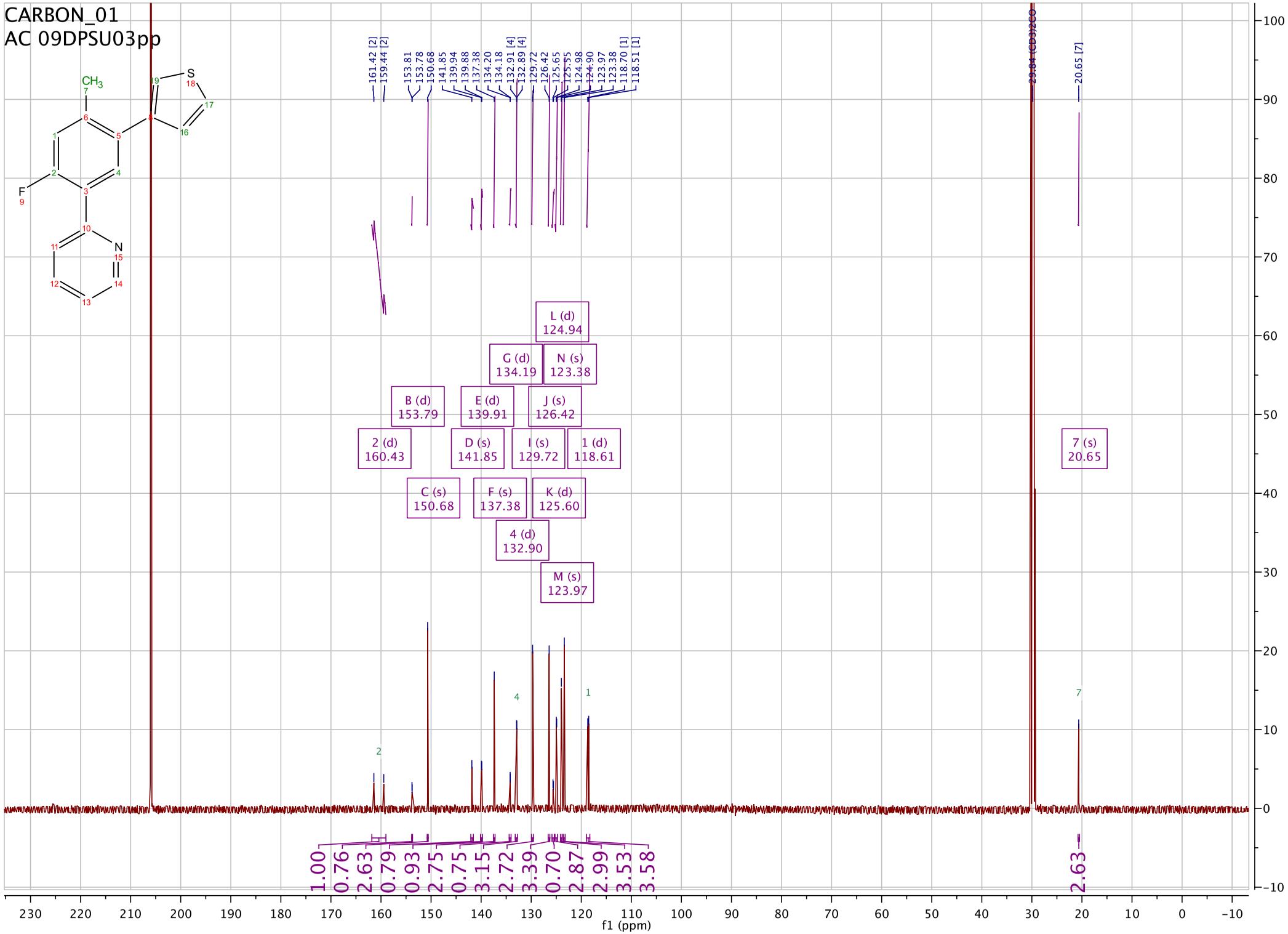
Fragmentation

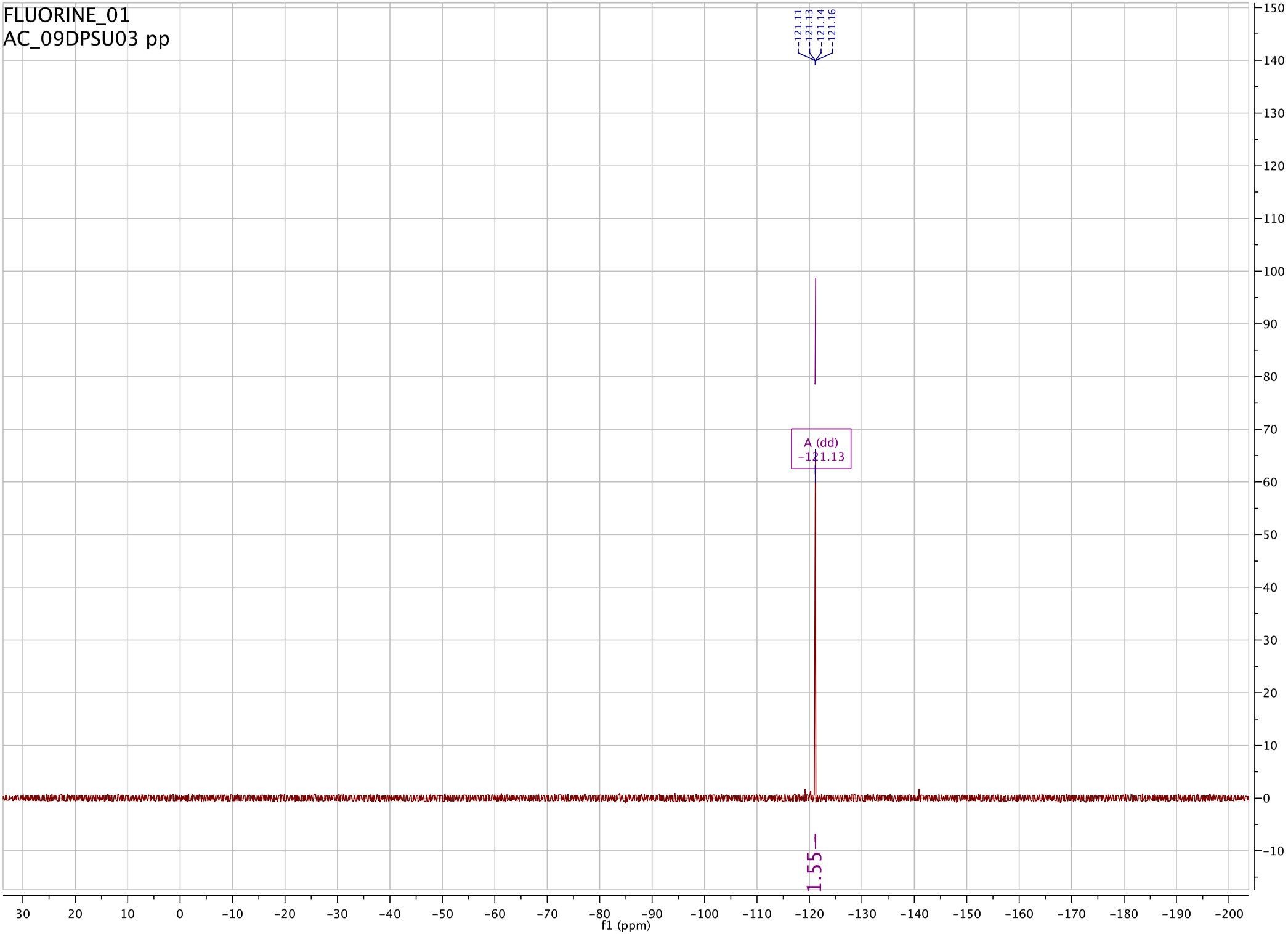


2-(2-Fluoro-4-methyl-5-(thiophen-3-yl)phenyl)pyridine 5b




PROTON_01
AC 09DPSU03pp
CH3
S
F
N
8.69 8.69 8.69 8.68 8.68 8.68 8.03 [4] 8.01 [4] 7.87 7.87 7.86 7.86 7.86 7.85 7.58 [16] 7.57 [16] 7.57 [16] 7.56 [16] 7.48 7.47 [19] 7.47 7.47 [19] 7.47 7.35 7.34 7.34 7.33 7.33 7.32 7.27 [17] 7.27 [17] 7.26 [17] 7.26 [17] 7.19 [1] 7.17 [1] 2.39 [7] 2.05 (CD3)2CO
17 (dd) 7.26
4 (d) 8.02
19 (d) 7.47
F (dt) 8.69
16 (dd) 7.57
C (td) 7.34
H (m) 7.86
1 (d) 7.18
7 (s) 2.39
0.87 0.85 1.89 0.83 0.39 0.94 0.92 1.00 3.45
f1 (ppm)


CARBON_01
AC 09DPSU03pp
CH3
S
F
N
161.42 [2]
159.44 [2]
153.81
153.78
150.68
141.85
139.94
139.88
137.38
134.20
134.18
132.91 [4]
132.89 [4]
129.72
126.42
125.65
125.55
124.98
124.90
123.97
123.38
118.70 [1]
118.51 [1]
20.65 [7]
L (d) 124.94
G (d) 134.19
N (s) 123.38
B (d) 153.79
E (d) 139.91
J (s) 126.42
2 (d) 160.43
D (s) 141.85
I (s) 129.72
1 (d) 118.61
7 (s) 20.65
C (s) 150.68
F (s) 137.38
K (d) 125.60
4 (d) 132.90
M (s) 123.97
1.00
0.76
2.63
0.79
0.93
2.75
0.75
3.15
2.72
3.39
0.70
2.87
2.99
3.53
3.58
2.63
f1 (ppm)


FLUORINE_01
AC_09DPSU03 pp
-121.11
-121.13
-121.14
-121.16
A (dd)
-121.13
1.55
f1 (ppm)

Elemental Composition Report

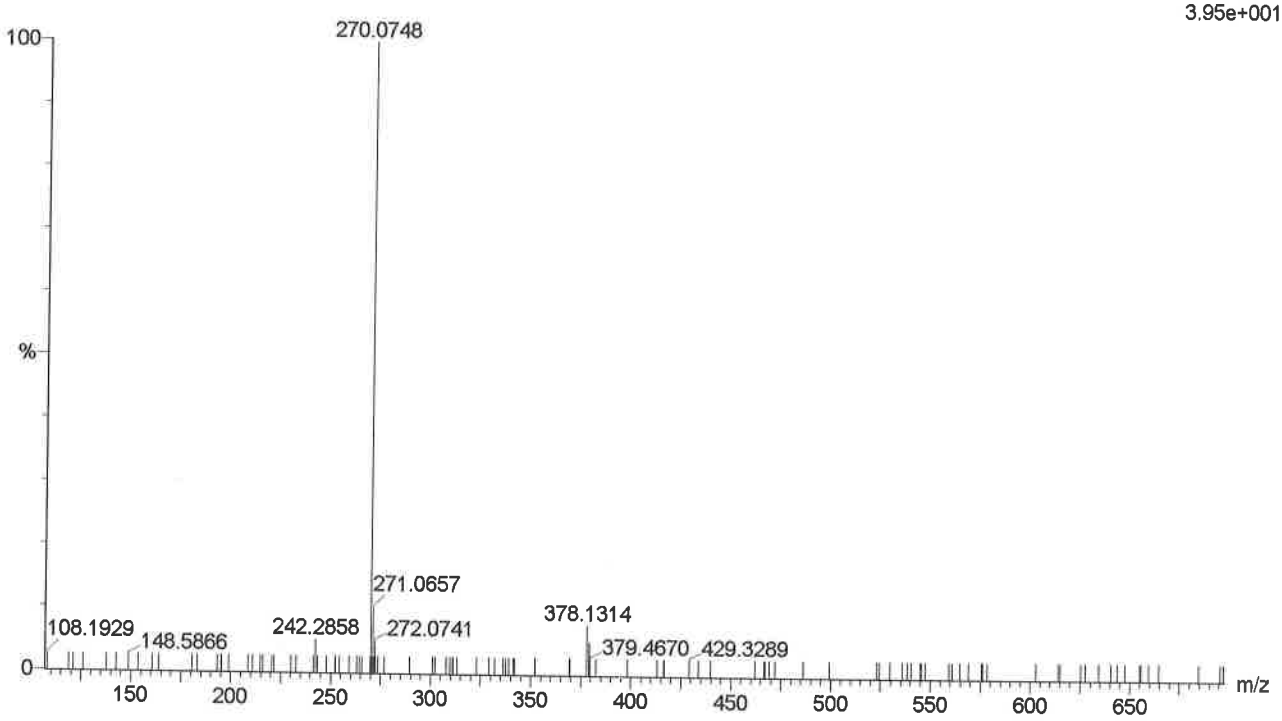
Single Mass Analysis

Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
3 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 16-16 H: 0-200 N: 1-1 F: 1-1 Na: 0-2 S: 1-1

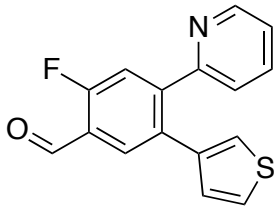
no sample code
adam4452a 102 (1.382)

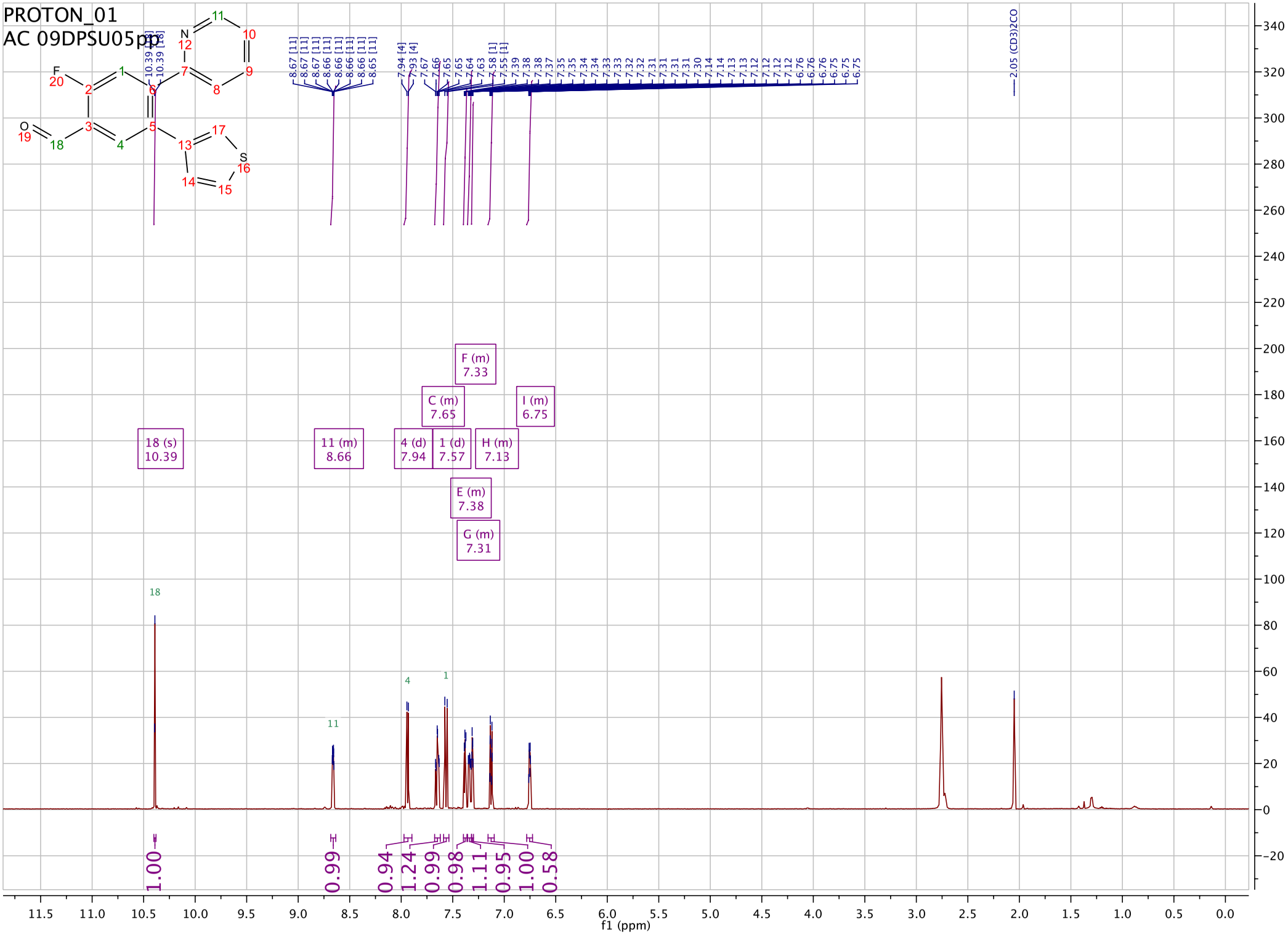
1: TOF MS ES+
3.95e+001

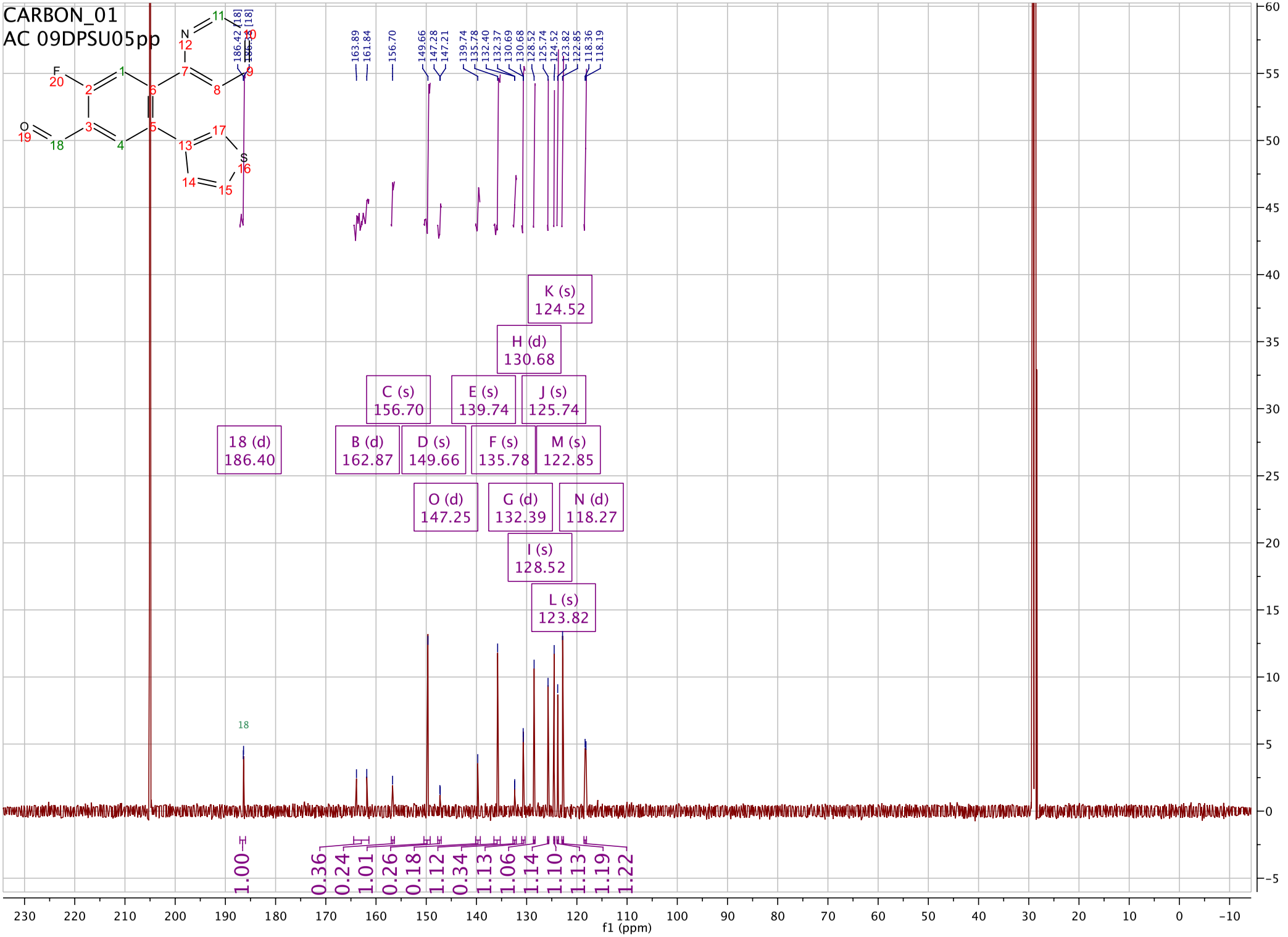


Minimum:				-1.5		
Maximum:		5.0	1000.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
270.0748	270.0753	-0.5	-1.9	10.5	1.1	C16 H13 N F S

2-Fluoro-4-(pyridin-2-yl)-5-(thiophen-3-yl)benzaldehyde 5b




PROTON_01
AC 09DPSU05pp
F (m) 7.33
C (m) 7.65
I (m) 6.75
18 (s) 10.39
11 (m) 8.66
4 (d) 7.94
1 (d) 7.57
H (m) 7.13
E (m) 7.38
G (m) 7.31
2.05 (CD3)2CO
f1 (ppm)


CARBON_01
AC 09DPSU05pp
163.89
161.84
156.70
149.66
147.28
147.21
139.74
135.78
132.40
132.37
130.69
130.68
128.52
125.74
124.52
123.82
122.85
118.36
118.19
186.42 [18]
186.38 [18]
K (s)
124.52
H (d)
130.68
C (s)
156.70
E (s)
139.74
J (s)
125.74
18 (d)
186.40
B (d)
162.87
D (s)
149.66
F (s)
135.78
M (s)
122.85
O (d)
147.25
G (d)
132.39
N (d)
118.27
I (s)
128.52
L (s)
123.82
18
1.00
0.36
0.24
1.01
0.26
0.18
1.12
0.34
1.13
1.06
1.14
1.10
1.13
1.19
1.22
f1 (ppm)


FLUORINE_01
STANDARD FLUORINE PARAMETERS
AC_09DPSU05_pp
-120.13
-120.15
-120.16
-120.18
A (dd)
-120.15
1.00
f1 (ppm)

Elemental Composition Report

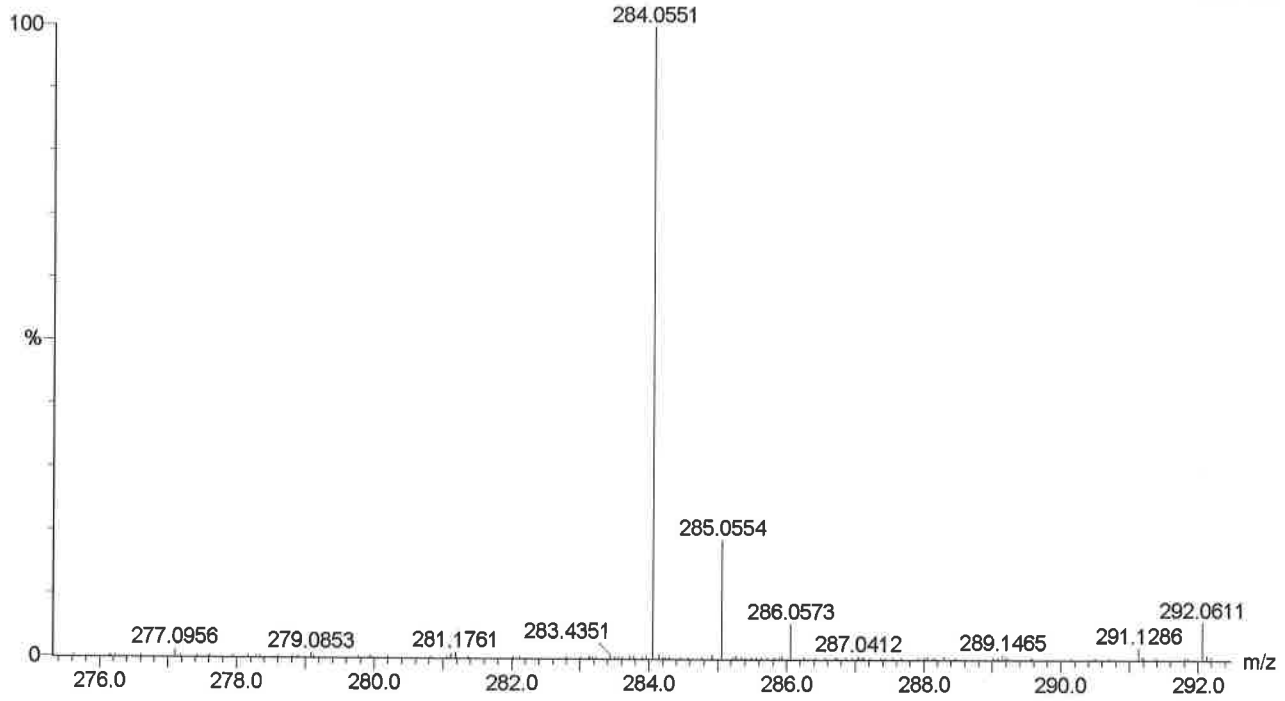
Single Mass Analysis

Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
3 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 16-16 H: 0-200 N: 1-1 O: 1-1 F: 1-1 Na: 0-2 S: 1-1

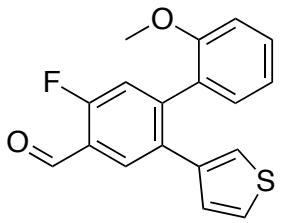
09dpsu05
adam4453 76 (1.027)

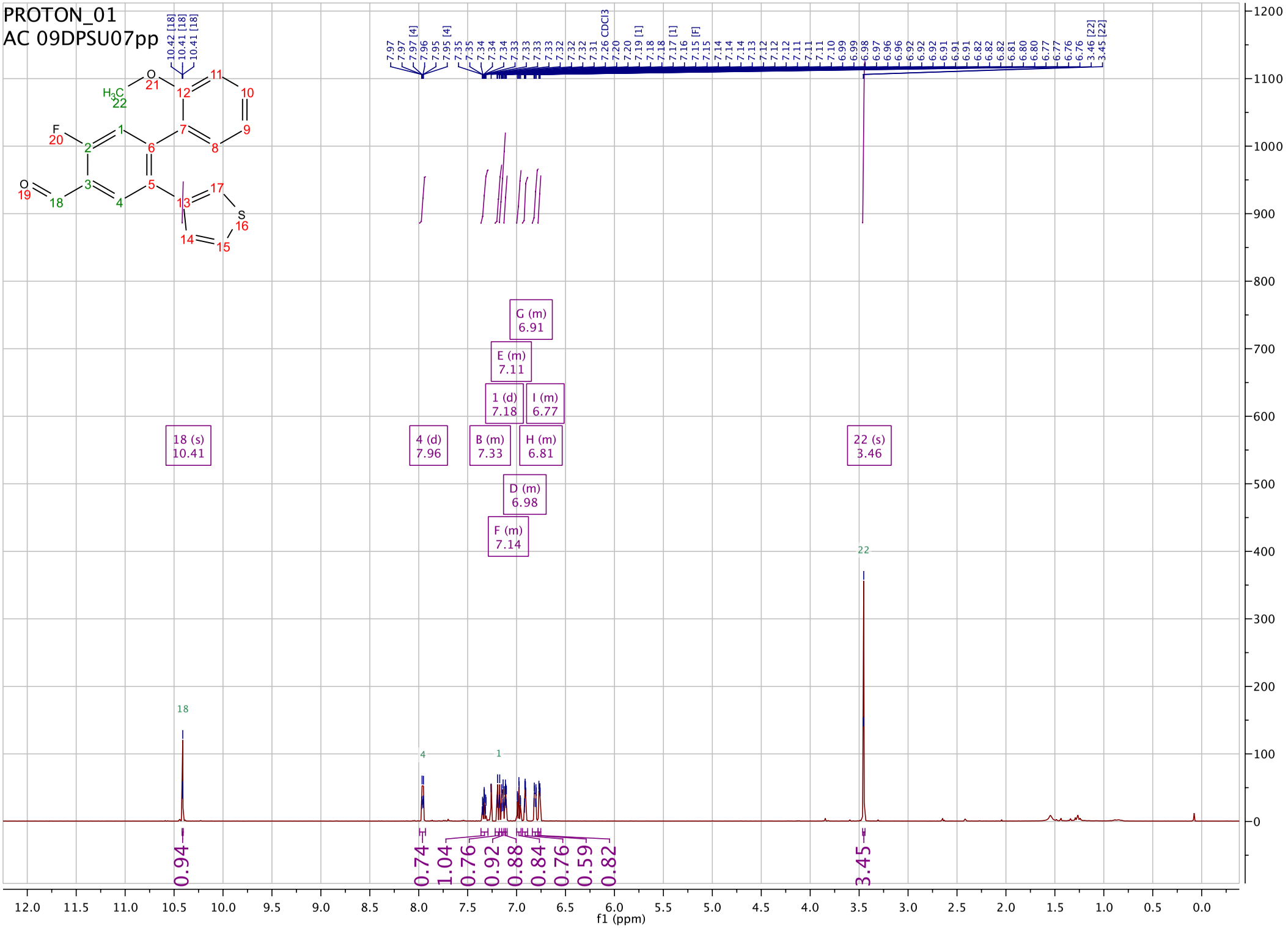
1: TOF MS ES+
4.72e+002



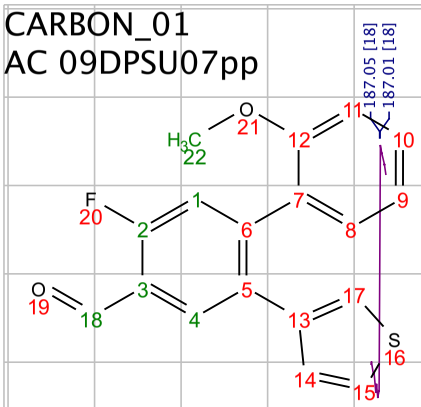
Minimum:				-1.5		
Maximum:		5.0	1000.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
284.0551	284.0545	0.6	2.1	11.5	0.5	C16 H11 N O F S

5-Fluoro-2'-methoxy-2-(thiophen-3-yl)-[1,1'-biphenyl]-4-carbaldehyde 5d




PROTON_01
AC 09DPSU07pp
10.42 [18]
10.41 [18]
10.41 [18]
7.97
7.97 [4]
7.96
7.95
7.95 [4]
7.35
7.35
7.34
7.34
7.34
7.33
7.33
7.33
7.32
7.32
7.32
7.31
7.26 CDCl3
7.20
7.20
7.19 [1]
7.18
7.18
7.17 [1]
7.16
7.15 [F]
7.15
7.14
7.14
7.14
7.13
7.12
7.12
7.12
7.11
7.11
7.11
7.10
6.99
6.99
6.98
6.97
6.96
6.96
6.92
6.92
6.92
6.91
6.91
6.91
6.82
6.82
6.82
6.81
6.80
6.80
6.77
6.76
6.76
3.46 [22]
3.45 [22]
G (m) 6.91
E (m) 7.11
1 (d) 7.18
I (m) 6.77
18 (s) 10.41
4 (d) 7.96
B (m) 7.33
H (m) 6.81
22 (s) 3.46
D (m) 6.98
F (m) 7.14
22
18
4
1
0.94
0.74
1.04
0.76
0.92
0.88
0.84
0.76
0.59
0.82
3.45
f1 (ppm)

CARBON_01
AC 09DPSU07pp



187.05 [18]
187.01 [18]
164.47 [2]
162.41 [2]
156.32
146.33
146.26
140.42
133.68
133.66
130.80
130.10
129.31 [4]
129.29 [4]
128.89
128.21
124.70
123.16 [3]
123.10 [3]
122.75
120.89
119.14 [1]
118.98 [1]
111.33
77.16 CDCl3
55.34 [22]

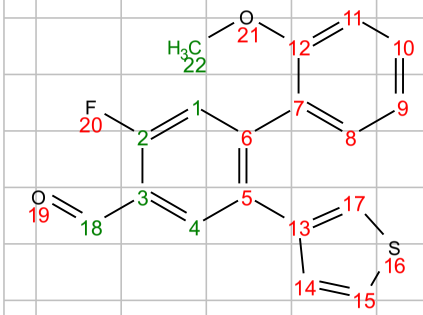
P (s) 130.80
4 (d) 129.30
L (s) 128.21
3 (d) 123.13
C (s) 156.32
E (s) 140.42
H (s) 120.89
18 (d) 187.03
2 (d) 163.44
A (d) 146.30
F (d) 133.67
1 (d) 119.06
22 (s) 55.34
I (s) 122.75
Q (s) 111.33
K (s) 124.70
M (s) 128.89
O (s) 130.10

18
2
4
3
1
22

1.00 H
0.27 H
0.26 H
0.29
0.25 H
0.25 H
1.08 H
0.98 H
1.06 H
0.28 H
0.96 H
0.98 H
0.28 H
1.07 H
0.96 H
1.07 H
1.09
1.03 H

f1 (ppm)

FLUORINE_02
AC_09DPSU07 pp



-125.14
-125.15
-125.17
-125.18

A (dd)
-125.16

1.00

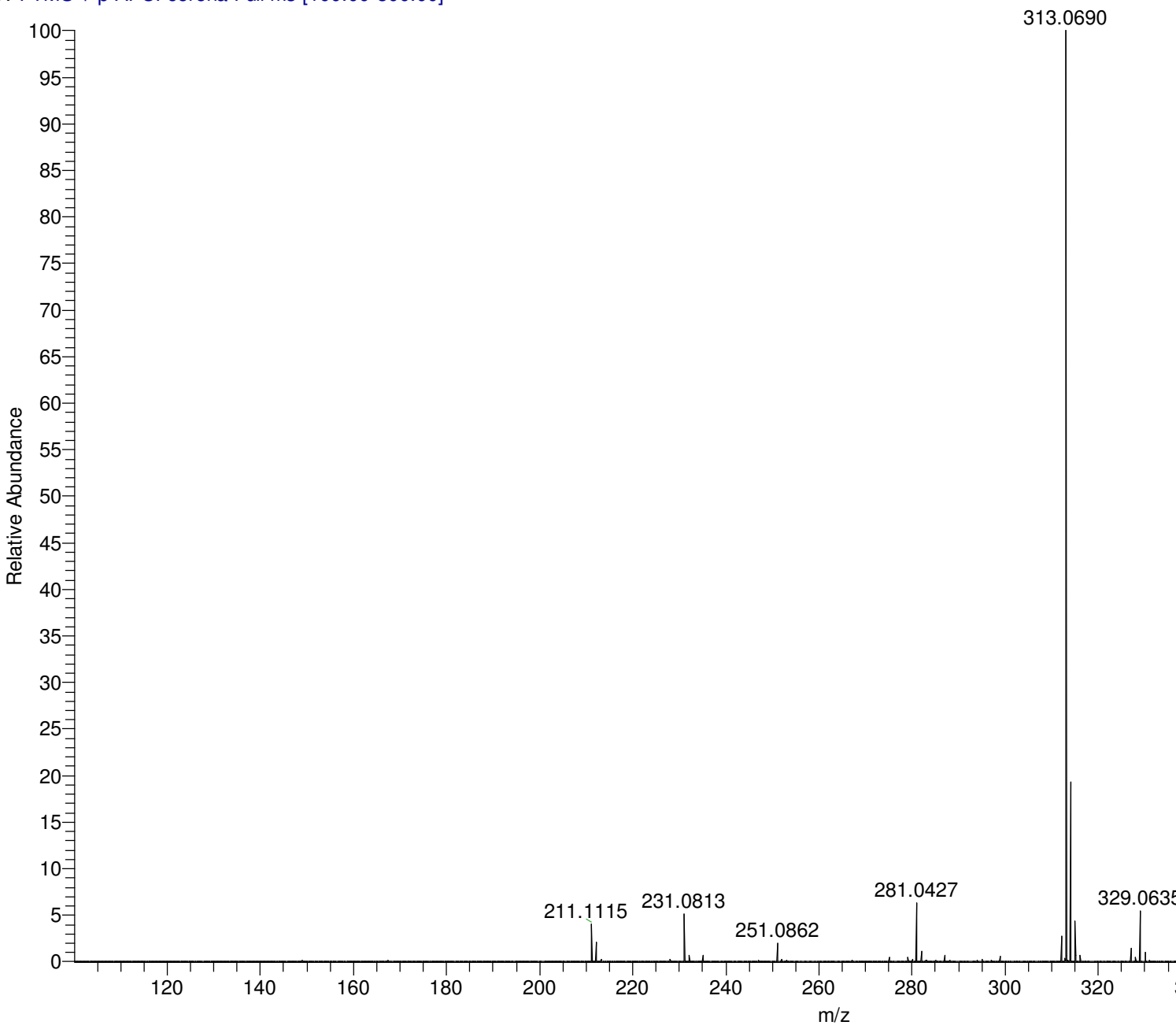
f1 (ppm)

AC09SU07 MW=312?
ASAP(SOLID)

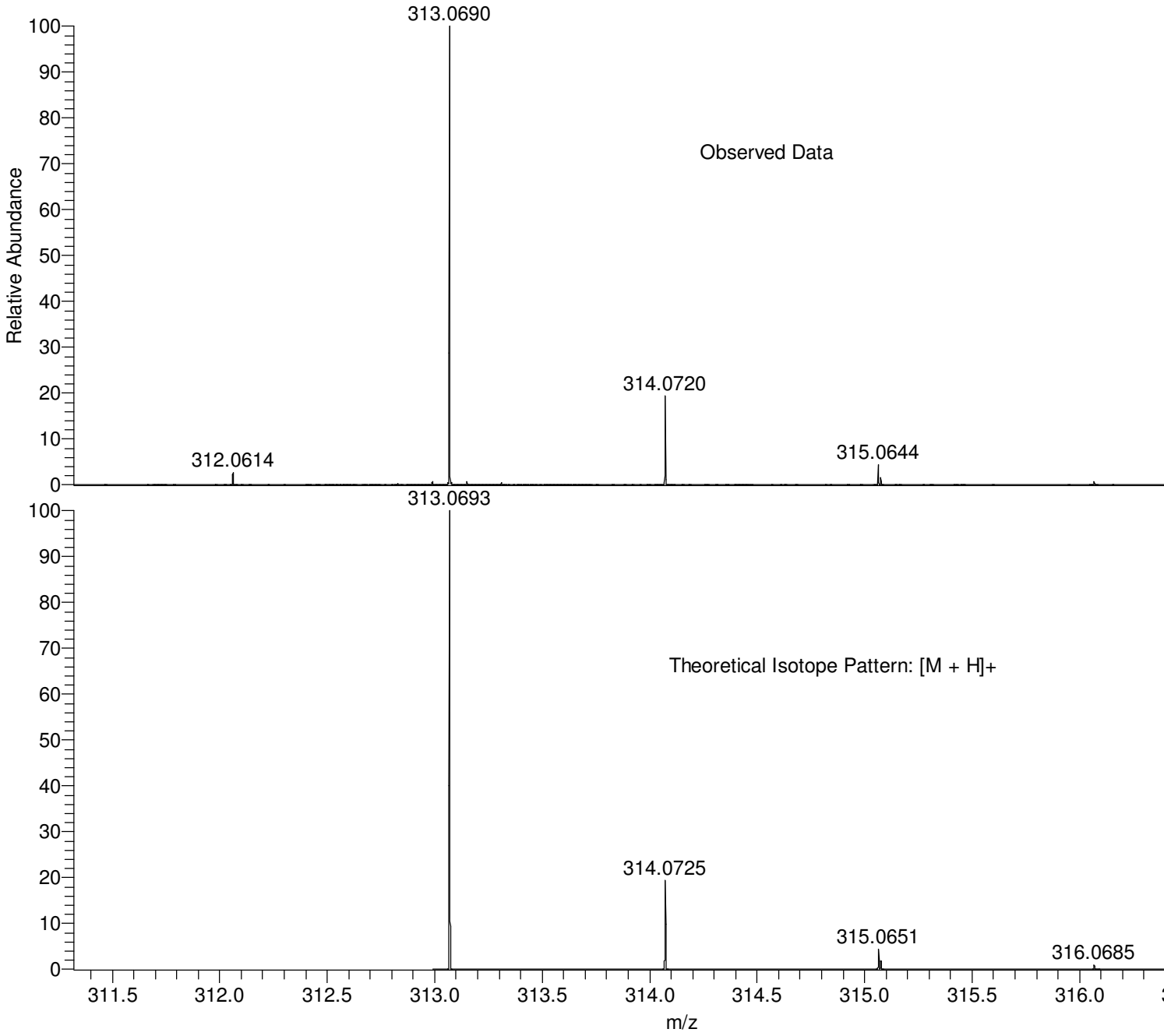
EPSRC National Centre Swansea
LTQ Orbitrap XL

Dr Sp
03/11/

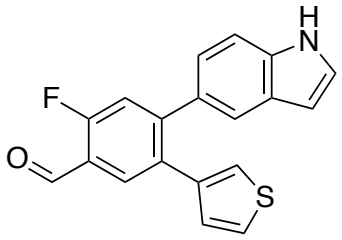
SUSSPE037-PG-HASP #17-23 RT: 0.47-0.64 AV: 7 SM: 7G NL: 3.32E8
T: FTMS + p APCI corona Full ms [100.00-800.00]

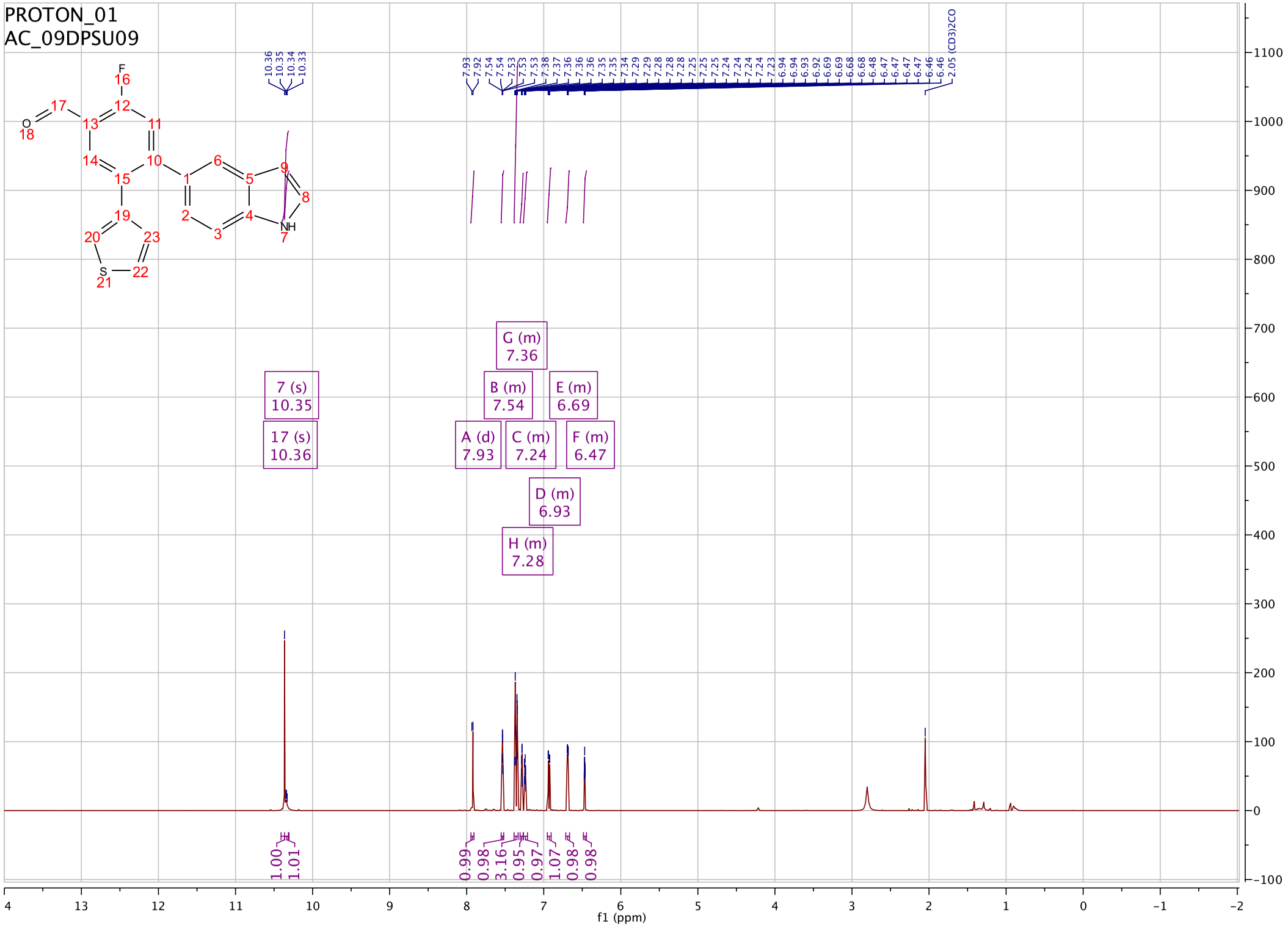


SM: 7G

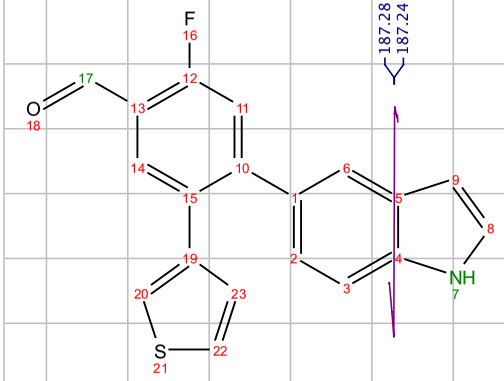

313.0690
Observed Data
314.0720
312.0614
315.0644
313.0693
Theoretical Isotope Pattern: [M + H]+
314.0725
315.0651
316.0685
Relative Abundance
m/z
100
90
80
70
60
50
40
30
20
10
0
311.5
312.0
312.5
313.0
313.5
314.0
314.5
315.0
315.5
316.0

2-Fluoro-4-(1*H*-indol-5-yl)-5-(thiophen-3-yl)benzaldehyde 5e




PROTON_01
AC_09DPSU09
F
16
17
12
O
18
13
11
14
10
6
15
1
5
9
8
19
2
4
NH
20
23
3
7
S
22
21
10.36
10.35
10.34
10.33
7.93
7.92
7.54
7.54
7.53
7.53
7.53
7.38
7.37
7.36
7.36
7.36
7.35
7.35
7.34
7.29
7.29
7.28
7.28
7.28
7.25
7.25
7.25
7.24
7.24
7.24
7.24
7.23
6.94
6.94
6.93
6.92
6.69
6.69
6.68
6.68
6.48
6.47
6.47
6.47
6.46
6.46
2.05 (CD3)2CO
G (m)
7.36
B (m)
7.54
E (m)
6.69
7 (s)
10.35
A (d)
7.93
C (m)
7.24
F (m)
6.47
17 (s)
10.36
D (m)
6.93
H (m)
7.28
1.00
1.01
0.99
0.98
3.16
0.95
0.97
1.07
0.98
0.98
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
4
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)

CARBON_01
AC_09DPSU09

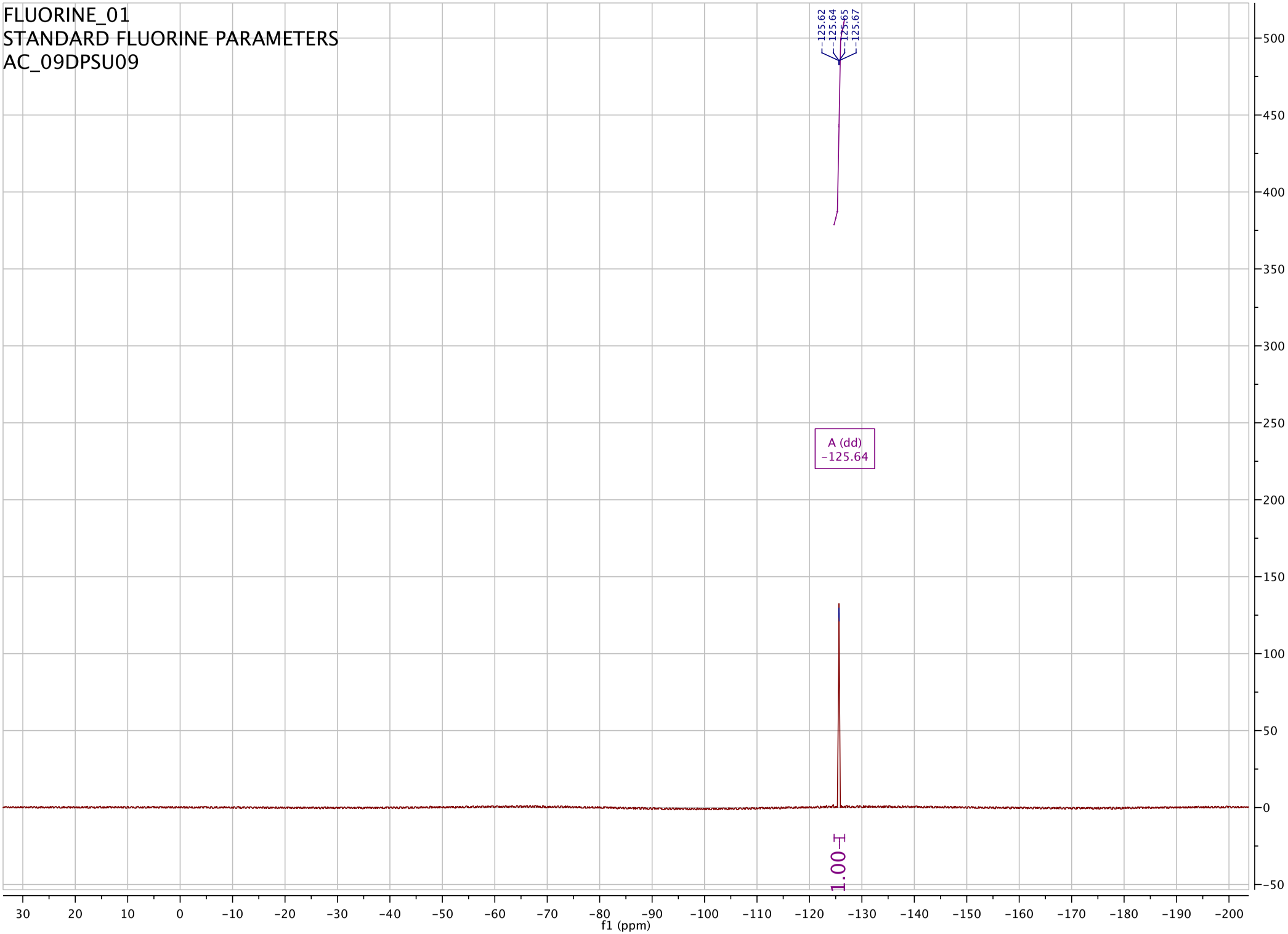


187.28
187.24
164.82
162.77
151.57
151.49
141.46
136.95
133.41
133.38
131.51
131.35
131.33
129.64
129.15
126.66
125.96
124.42
123.59
123.43
123.36
122.05
119.47
119.31
111.97
103.05
29.84 (CD3)2CO

P (s) 131.51
M (s) 129.15
J (s) 125.96
S (s) 141.46
H (d) 123.39
R (s) 136.95
G (s) 123.59
D (s) 111.97
A (d) 187.26
B (d) 163.80
T (d) 151.53
O (d) 131.34
E (d) 119.39
C (s) 103.05
Q (d) 133.40
F (s) 122.05
I (s) 124.42
K (s) 126.66
N (s) 129.64

1.00 0.27 0.29 0.26 0.22 0.25 0.32 1.04 0.95 0.21 0.85 0.97 0.88 0.93 0.29 0.95 0.98 0.83 0.80

f1 (ppm)

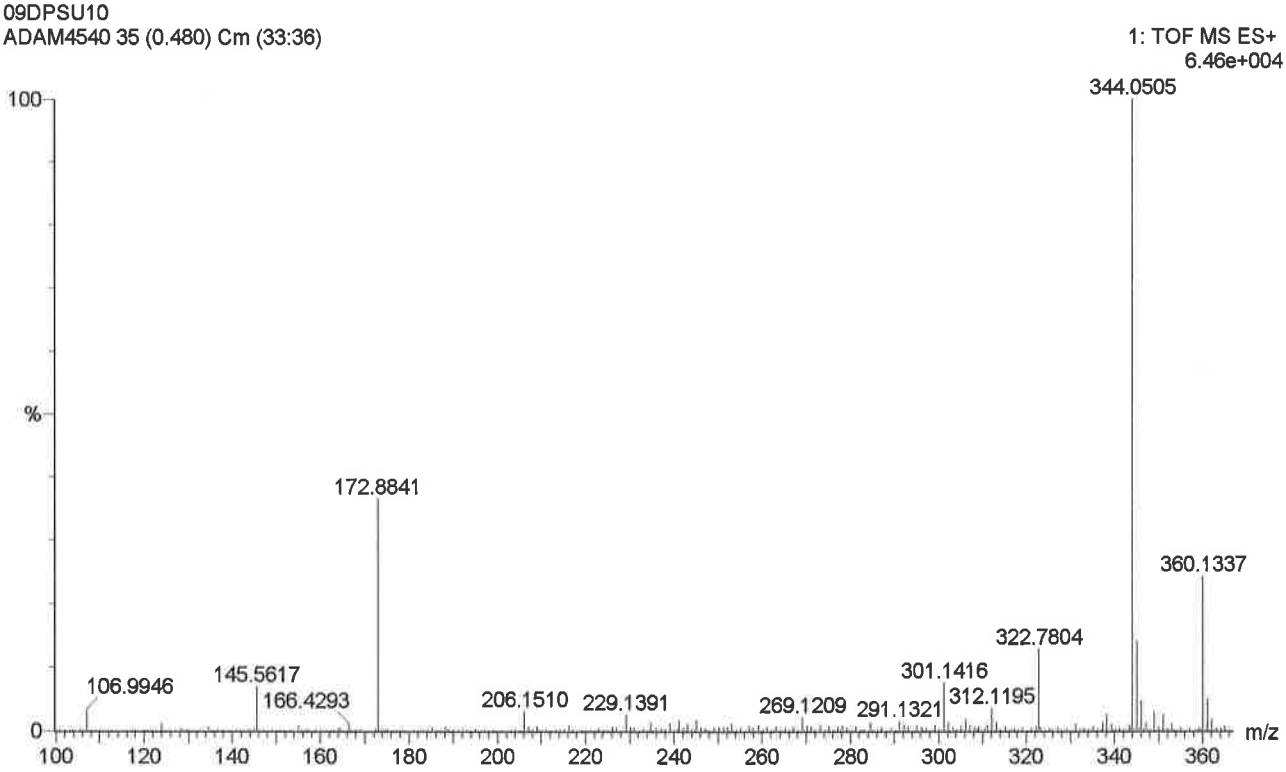

FLUORINE_01
STANDARD FLUORINE PARAMETERS
AC_09DPSU09
-125.62
-125.64
-125.65
-125.67
A (dd)
-125.64
1.00
f1 (ppm)

Single Mass Analysis

Tolerance = 100.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
3 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 19-19 H: 0-50 N: 1-1 O: 1-1 F: 1-1 Na: 0-2 S: 1-1

09DPSU10
ADAM4540 35 (0.480) Cm (33:36)



Minimum:				-1.5		
Maximum:		5.0	100.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
344.0505	344.0521	-1.6	-4.7	13.5	1361.3	C19 H12 N O F Na S