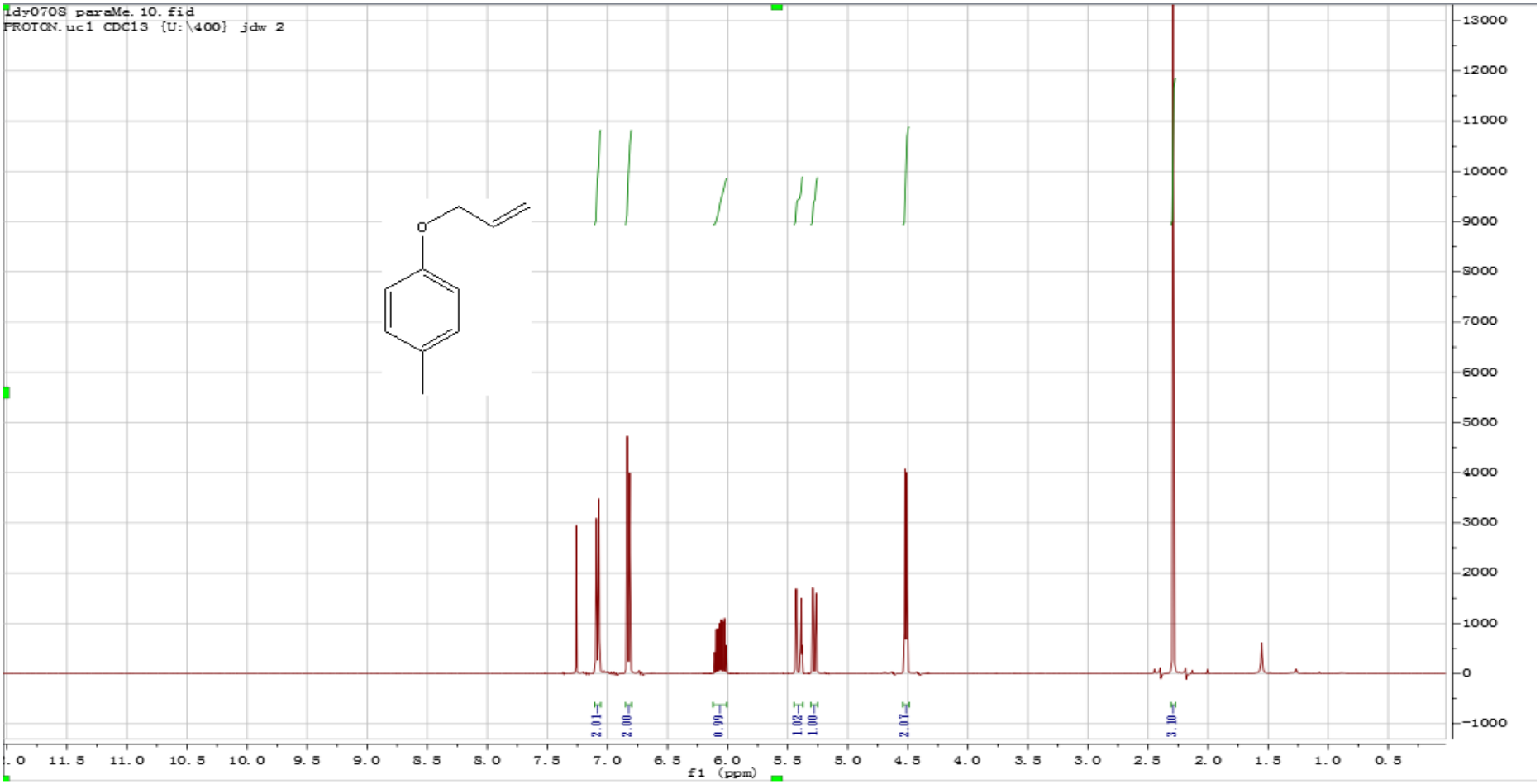
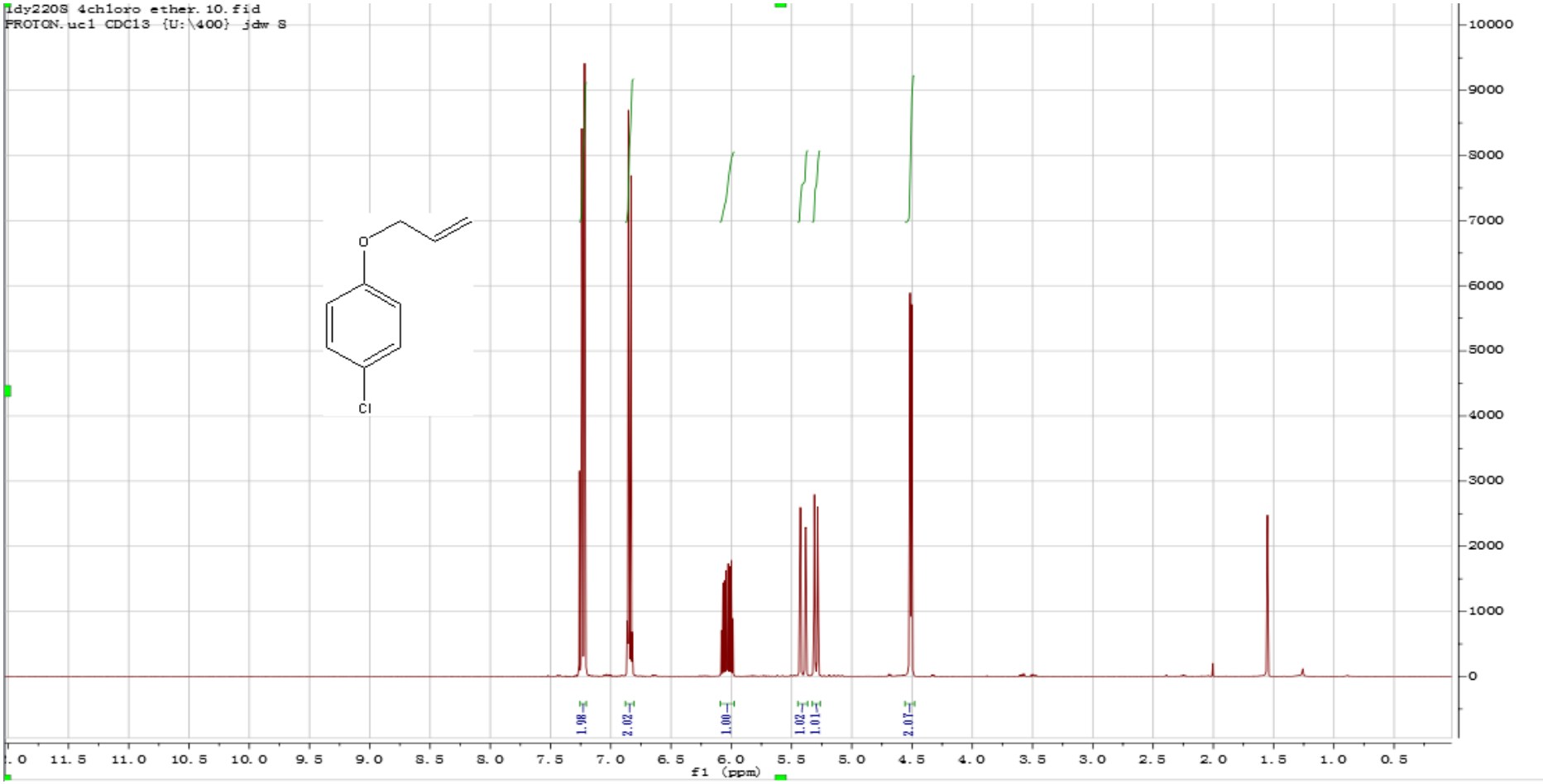
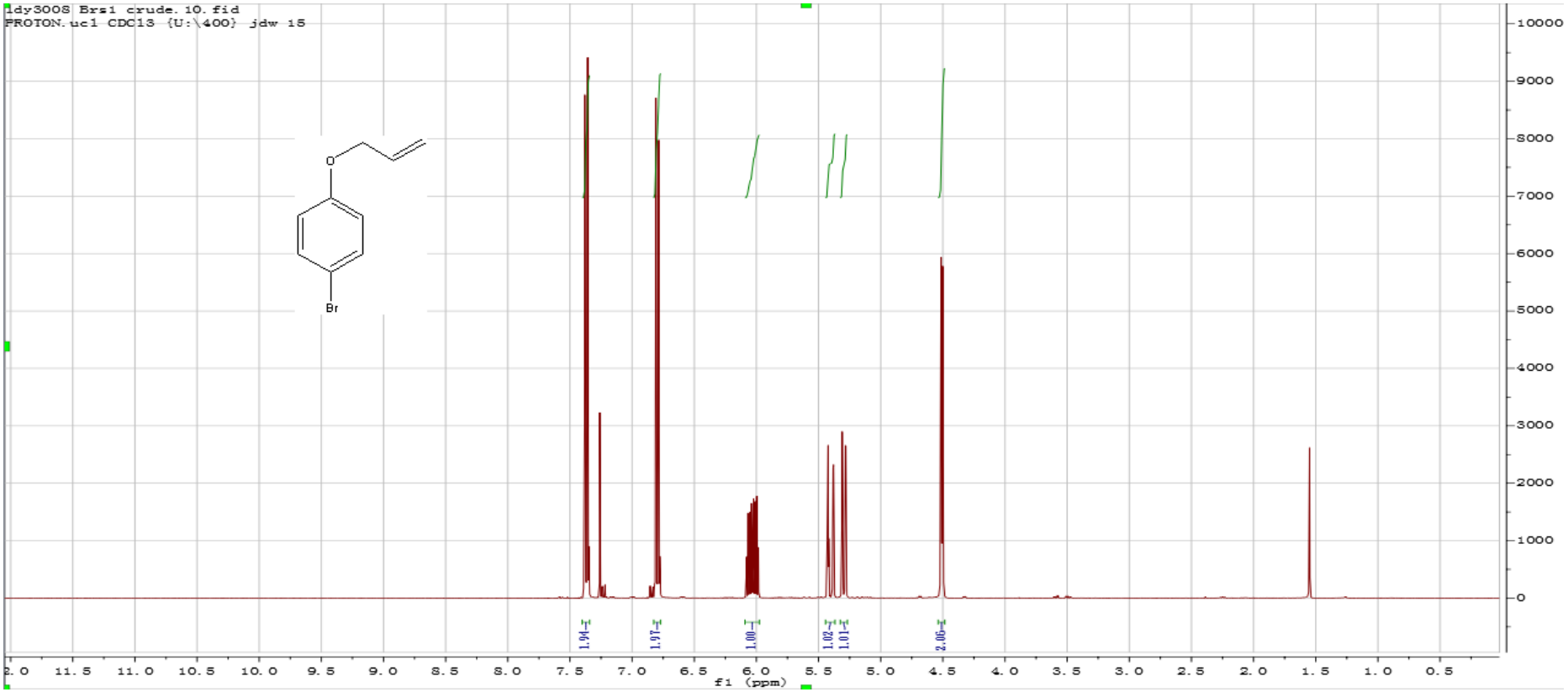
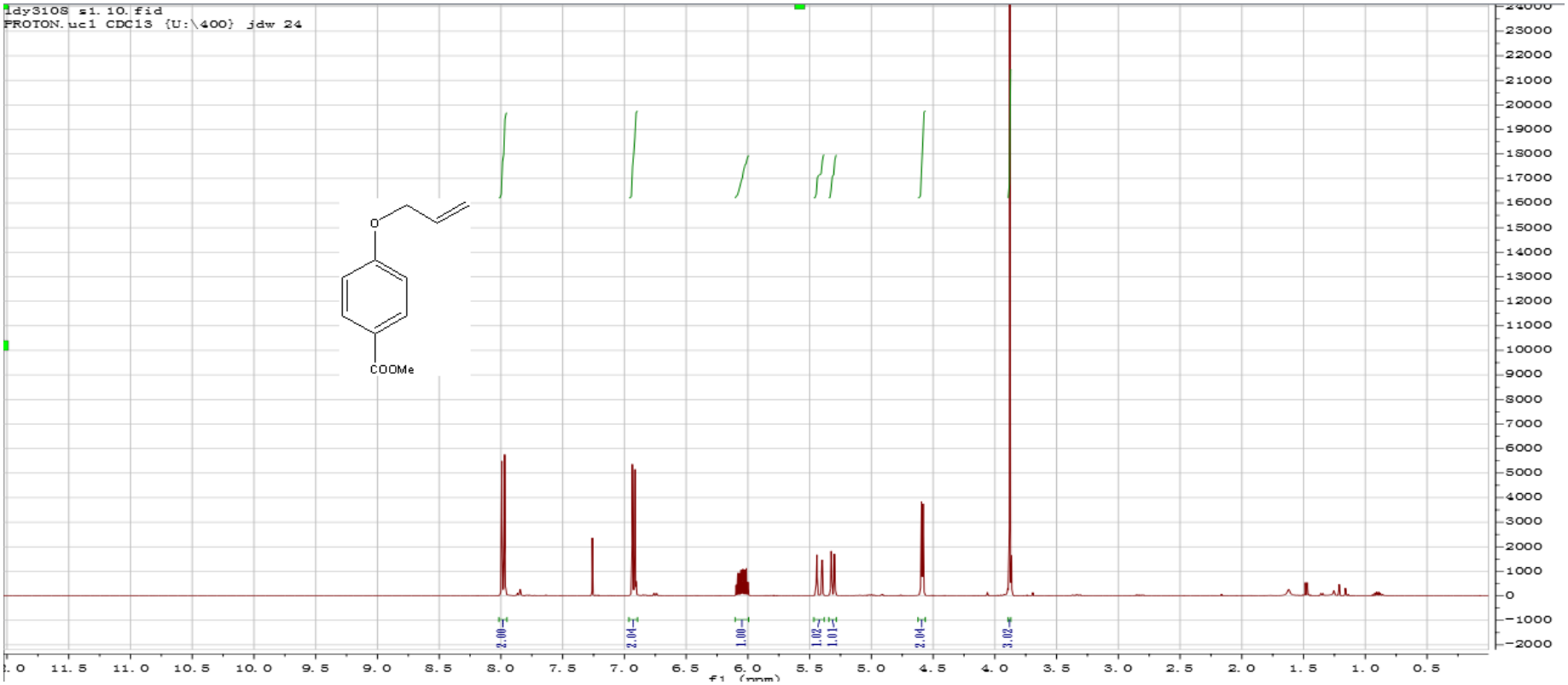
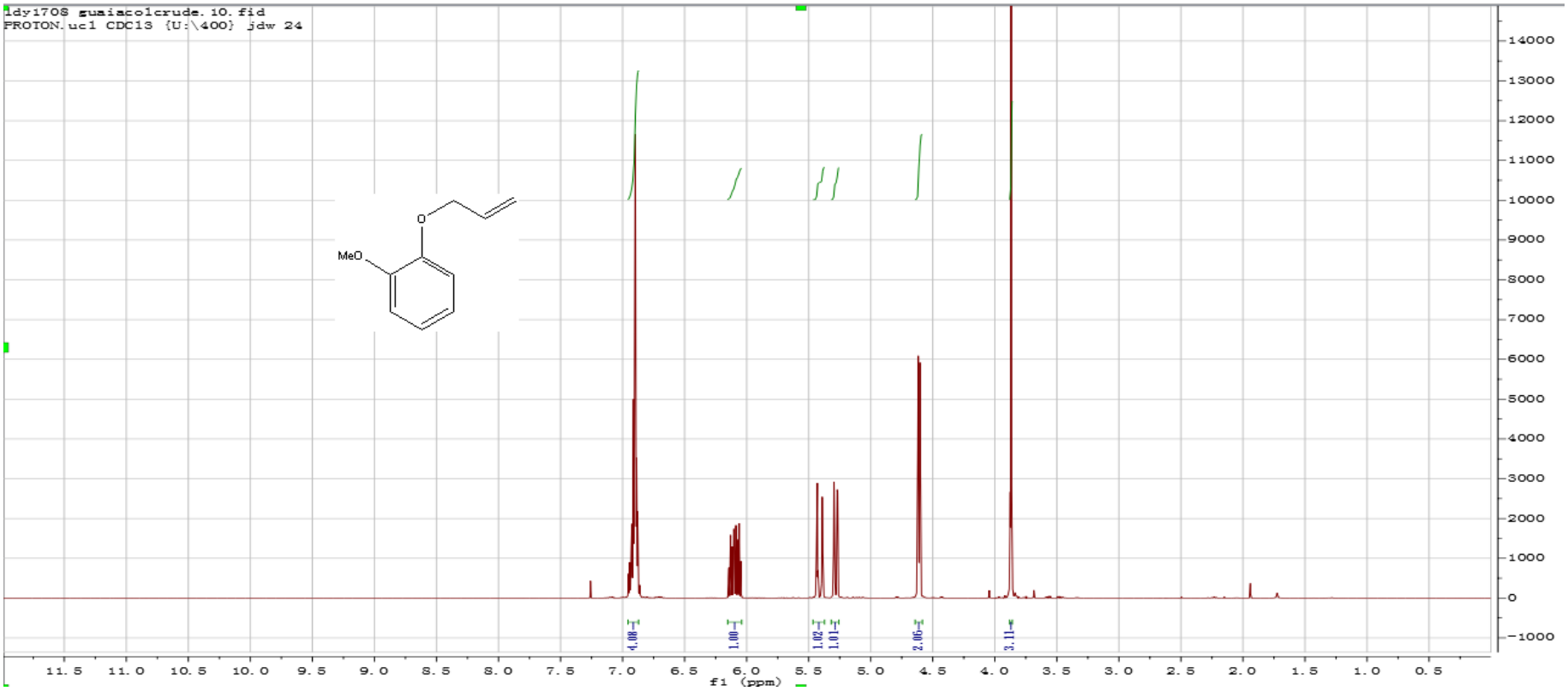
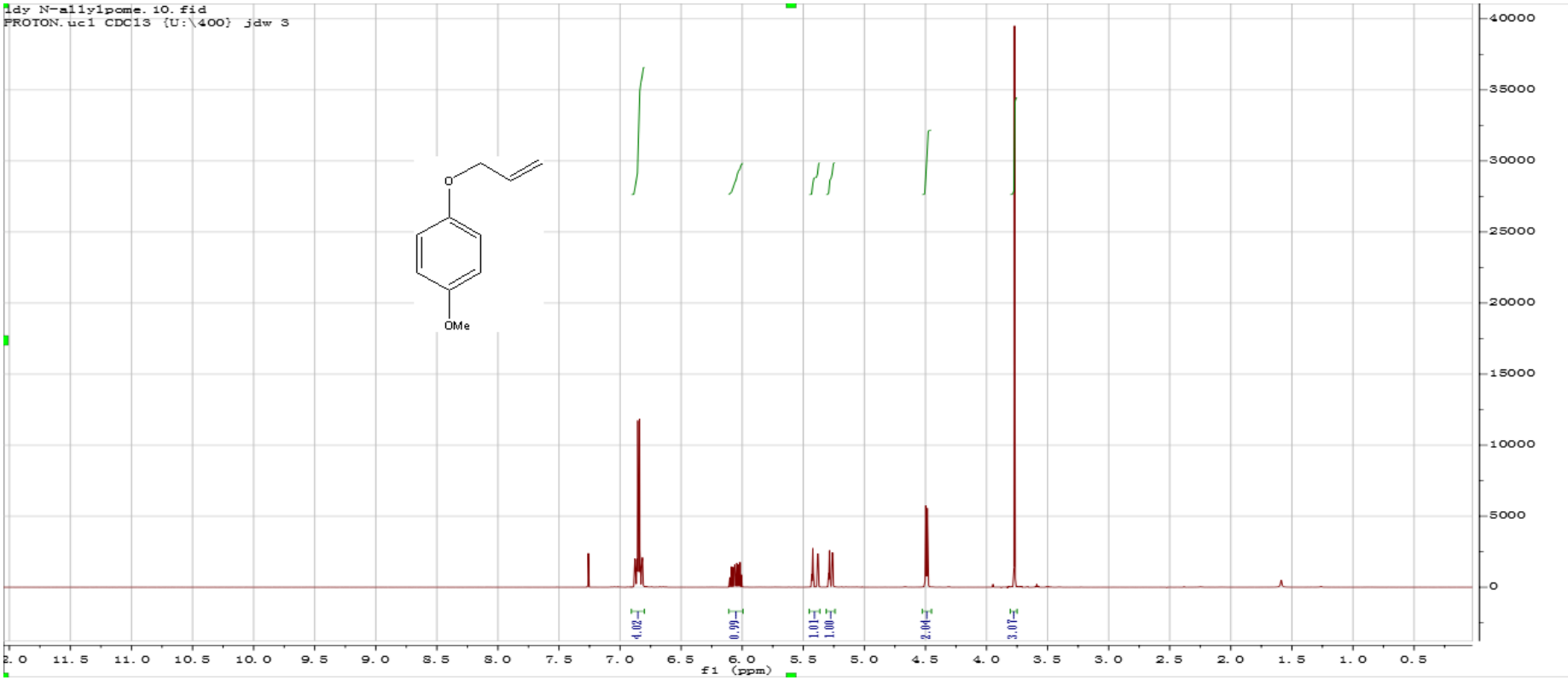

ldy0708 paraMe.10.fid
PROTON.uc1 CDCl3 {U:\400} jdw 2
O
2.01
2.00
0.99
1.02
1.00
2.07
3.10
f1 (ppm)

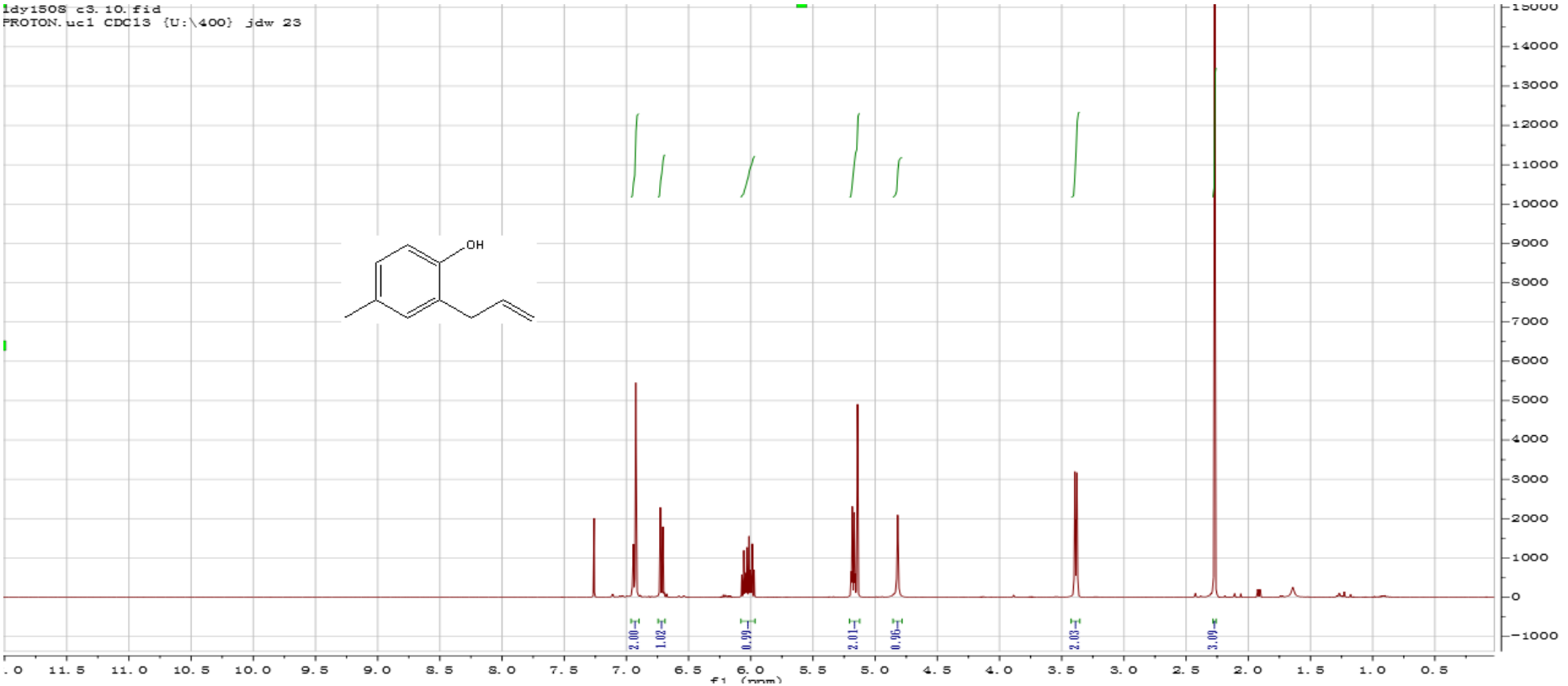

ldy220S 4chloro ether.10.fid
PROTON.uc1 CDCl3 {U:\400} jdw S
O
Cl
1.98
2.02
1.00
1.02
1.01
2.07
f1 (ppm)

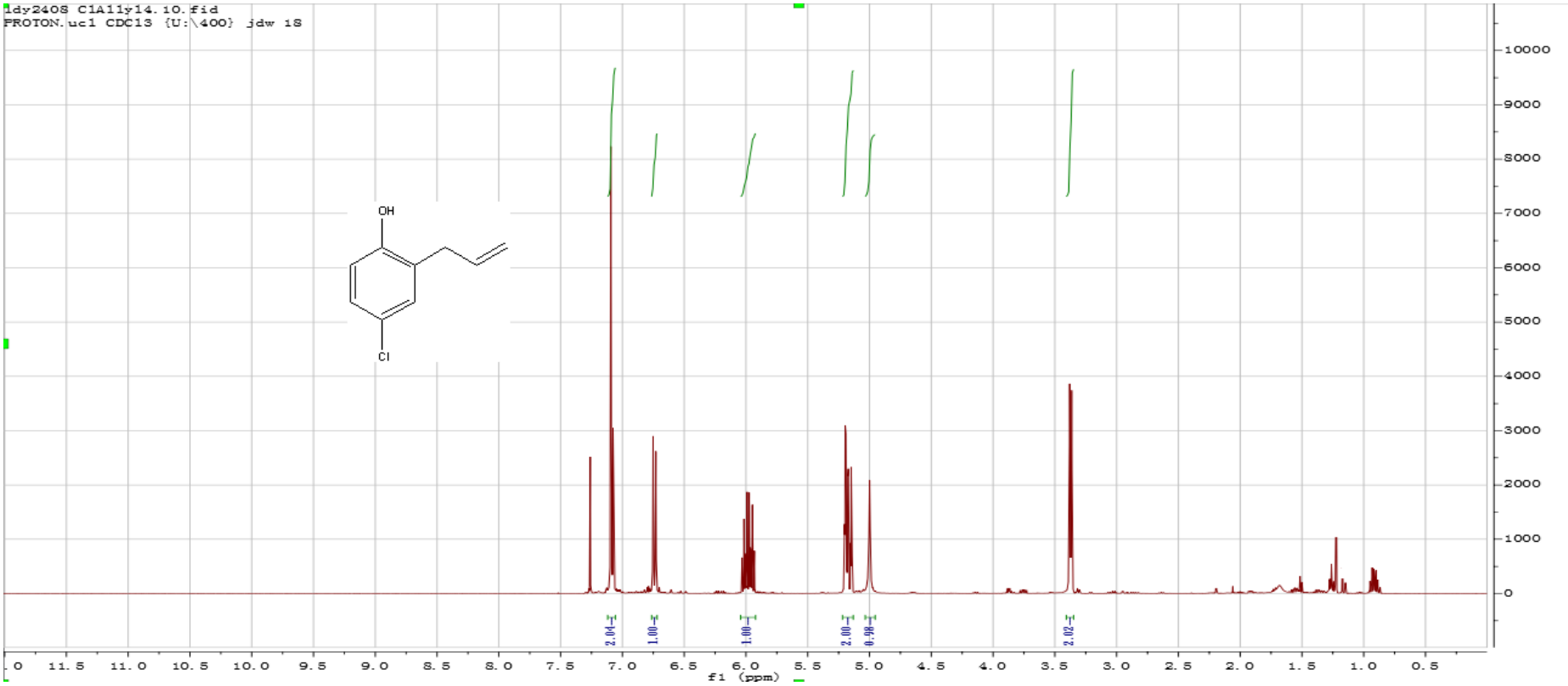

1dy3008 Br=1 crude. 10. fid
PROTON.uc1 CDCl3 {U:\400} jdw 15
O
Br
1.94
1.97
1.00
1.02
1.01
2.06
f1 (ppm)

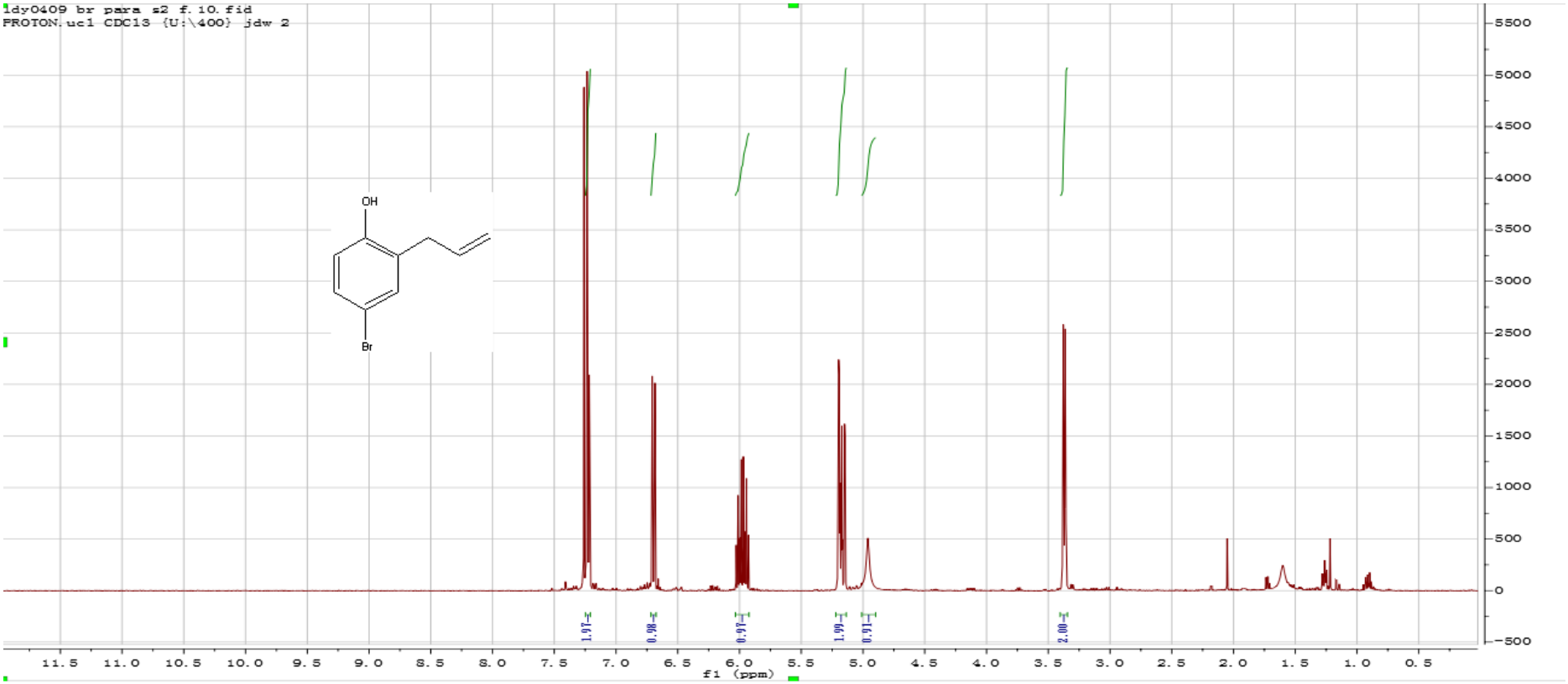

ldy3108 s1.10.fid
PROTON.uc1 CDCl3 {U:\400} jdw 24
O
COOMe
2.00
2.04
1.00
1.02
1.01
2.04
3.02
f1 (ppm)

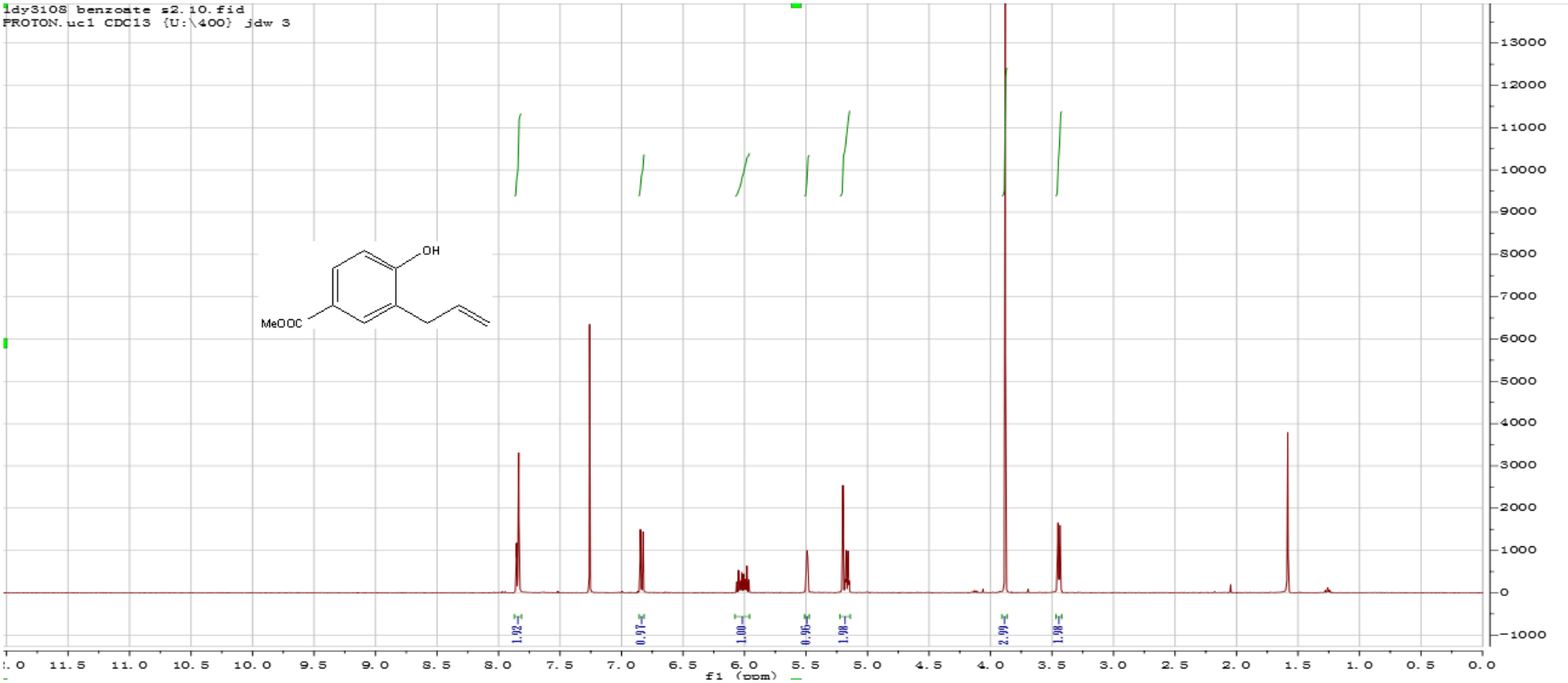

ldyi708 guaiacolcrude.10.fid
PROTON.uc1 CDCl3 {U:\400} jdw 24
MeO
O
4.00
1.00
1.02
1.01
2.06
3.11
f1 (ppm)

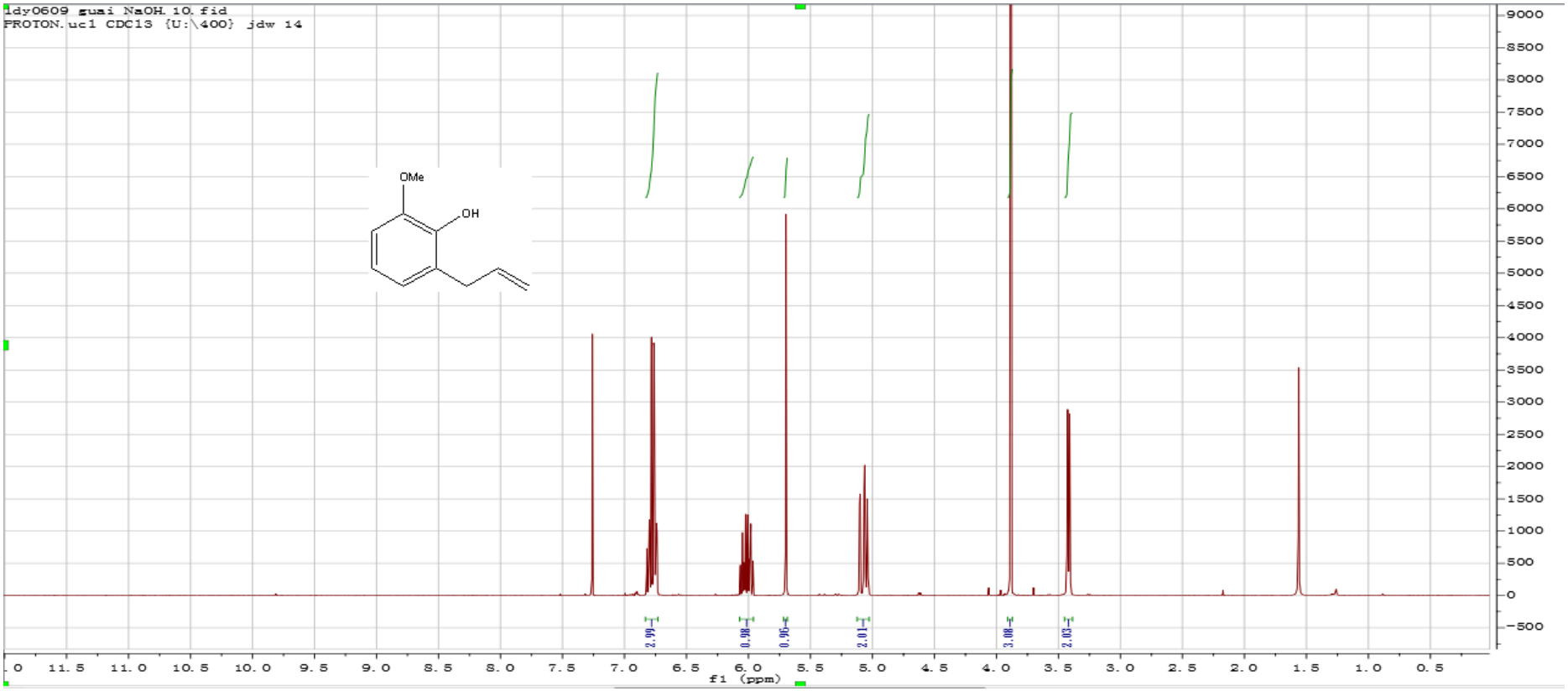

ldy N-allylpome.10.fid
PROTON.ucl CDCl3 {U:\400} jdw 3
O
OMe
4.02
0.99
1.01
1.00
2.04
3.01
f1 (ppm)

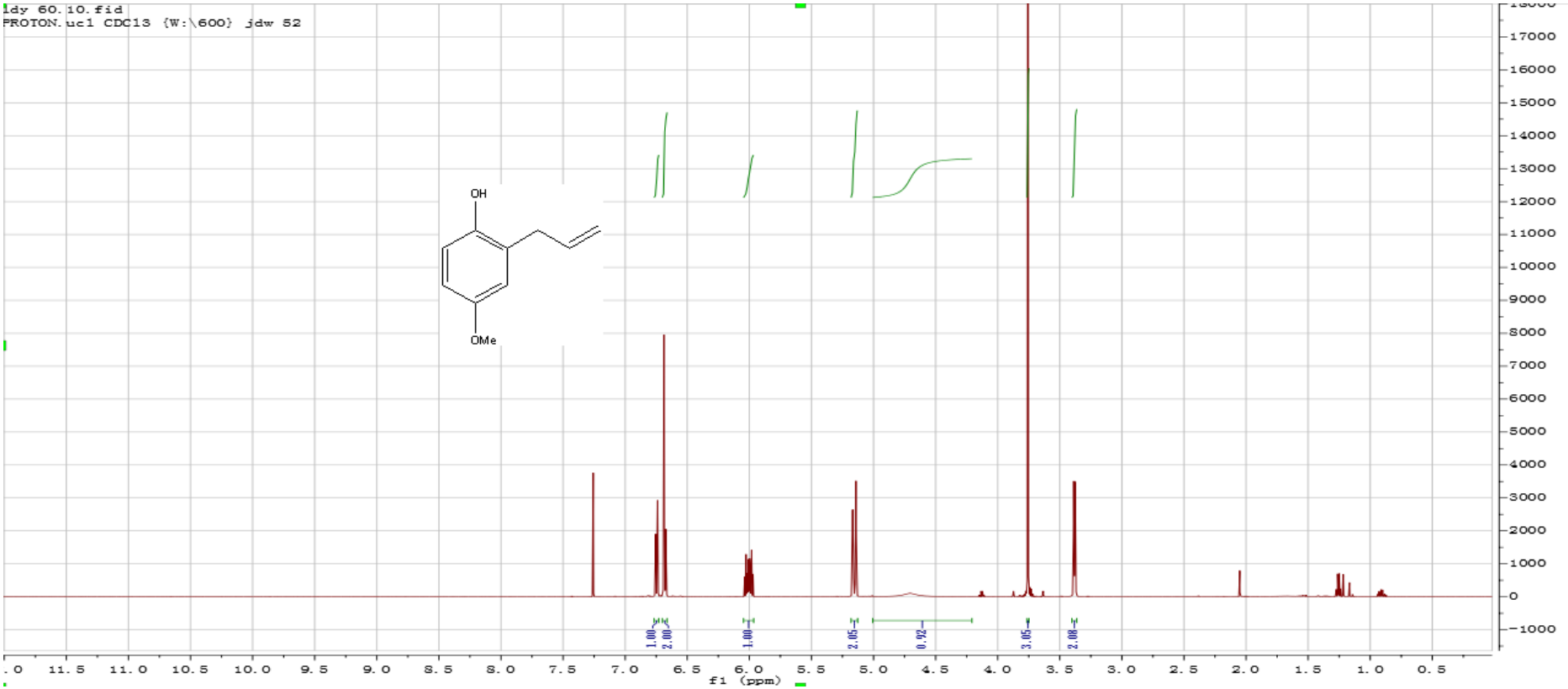

ldy1508 c3.10.fid
PROTON.ucl CDCl3 {U:\400} jdw 23
OH
2.00
1.02
0.99
2.01
0.96
2.03
3.09
f1 (ppm)

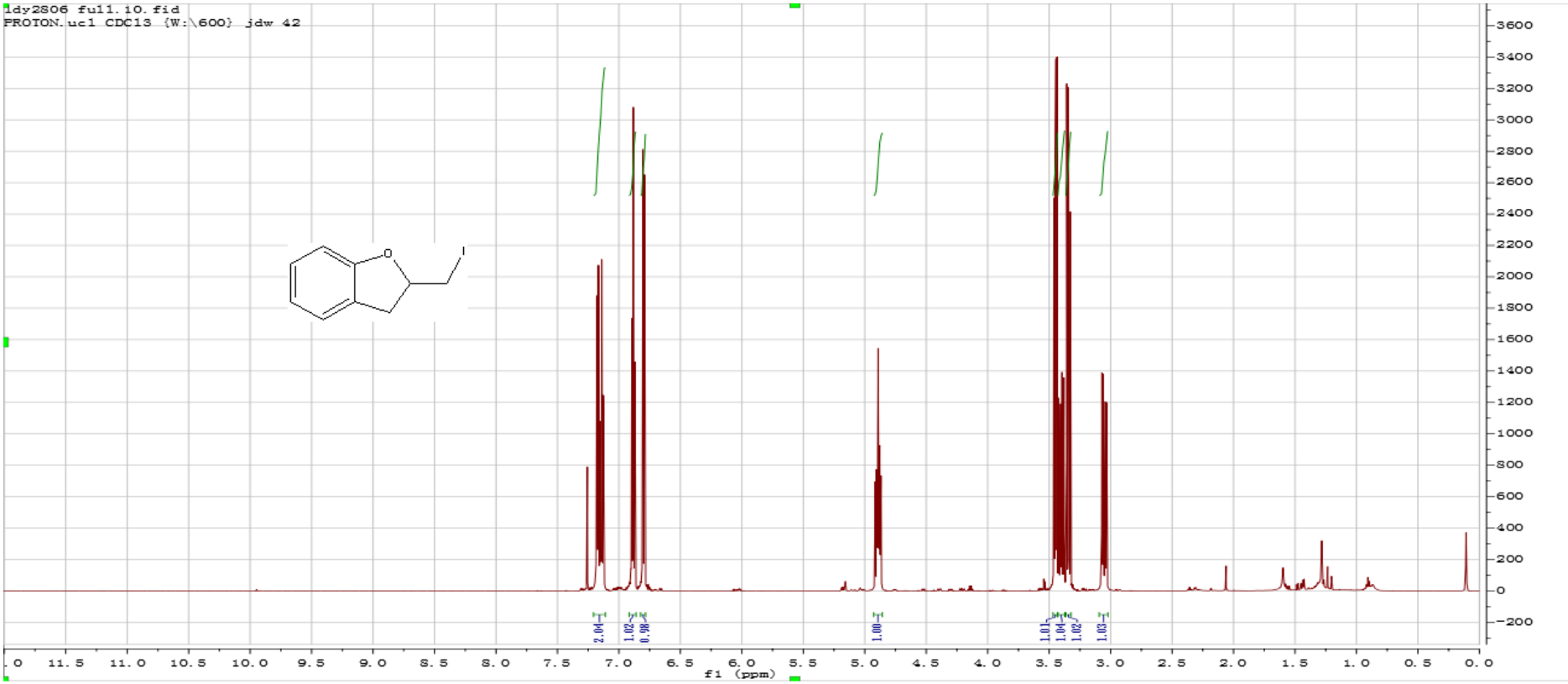

ldy2408 C1A11y14.10.fid
PROTON.uc1 CDC13 {U:\400} jdw 18
OH
Cl
2.04
1.00
1.00
2.00
0.98
2.02
f1 (ppm)

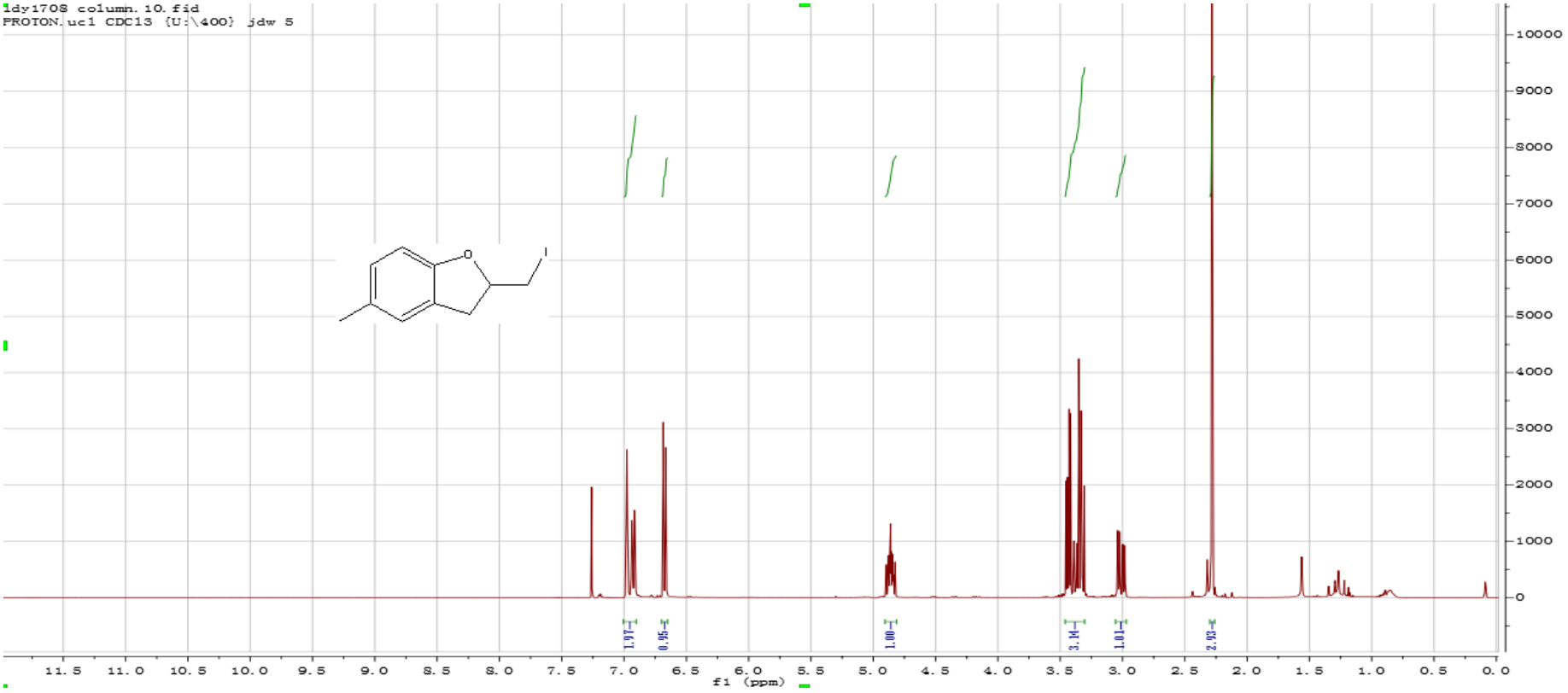

1dy0409 br para s2 f.10.fid
PROTON.uc1 CDCl3 {U:\400} jdw 2
OH
Br
1.97
0.98
0.97
1.99
0.91
2.00
f1 (ppm)

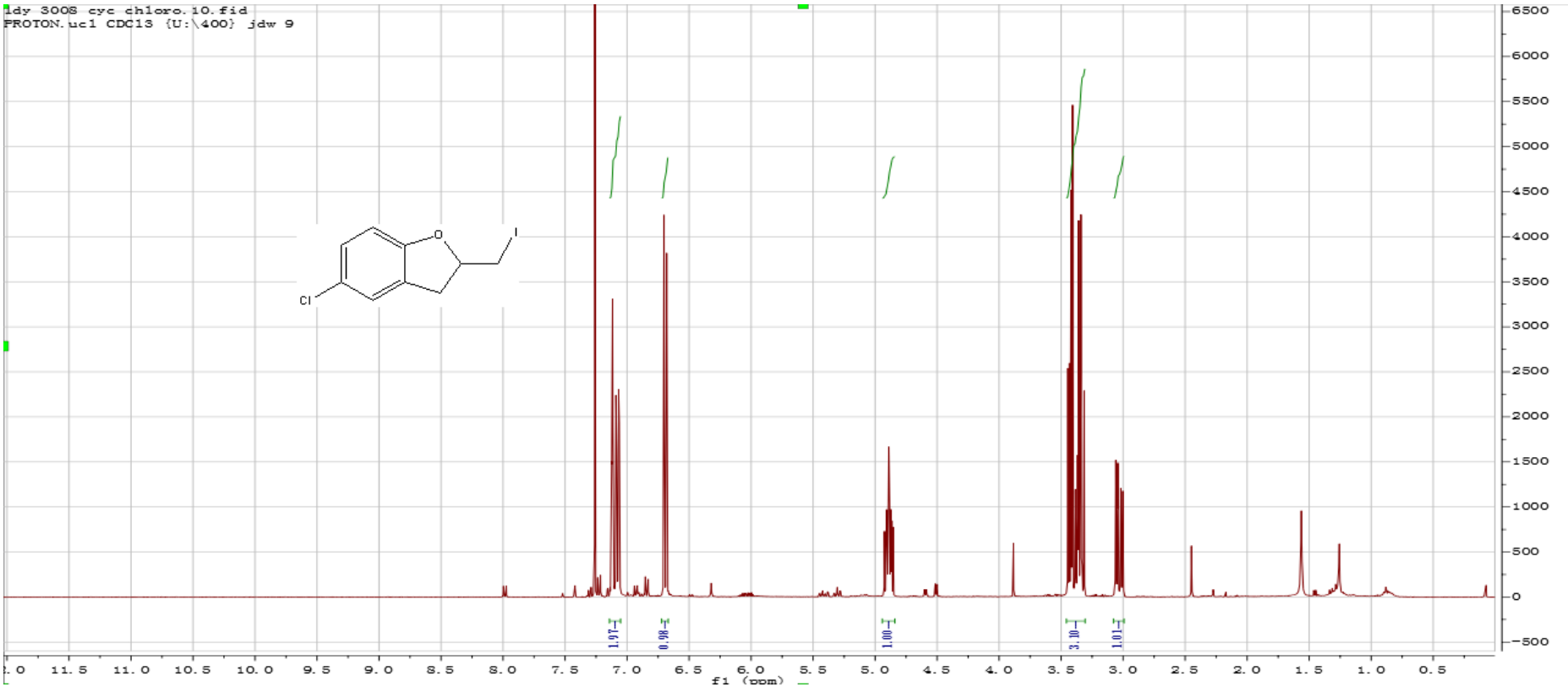

1dy3108 benzoate s2.10.fid
PROTON.uc1 CDCl3 {U:\400} jdw 3
OH
MeOOC
1.92
0.97
1.00
0.96
1.98
2.99
1.98
f1 (ppm)

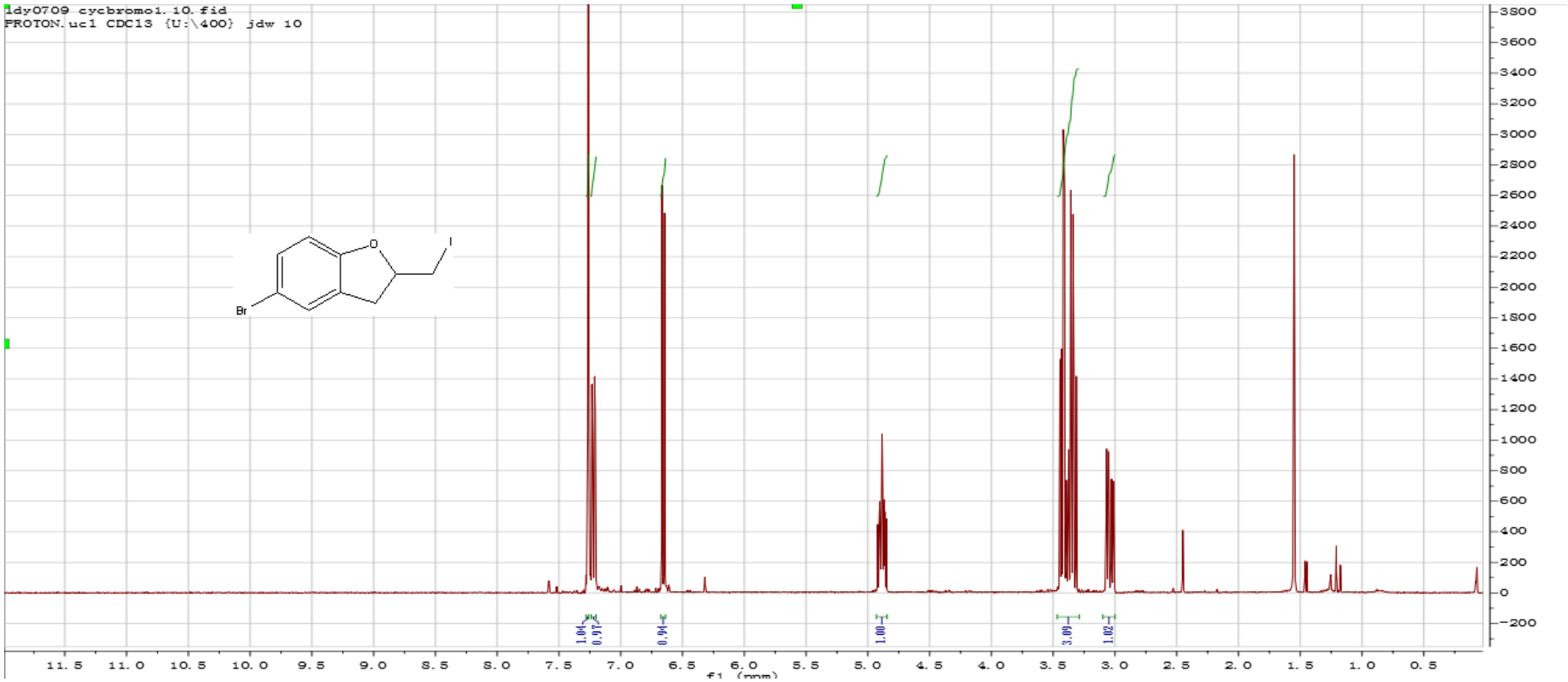

ldy0609 guai NaOH 10.fid
PROTON.uc1 CDC13 {U:\400} jdw 14
OMe
OH
2.99
0.98
0.96
2.01
3.08
2.03
f1 (ppm)


jdy 60.10.fid
PROTON.uc1 CDCl3 {W:\600} jdw 52
OH
OMe
1.00
2.00
1.00
2.05
0.92
3.05
2.08
f1 (ppm)

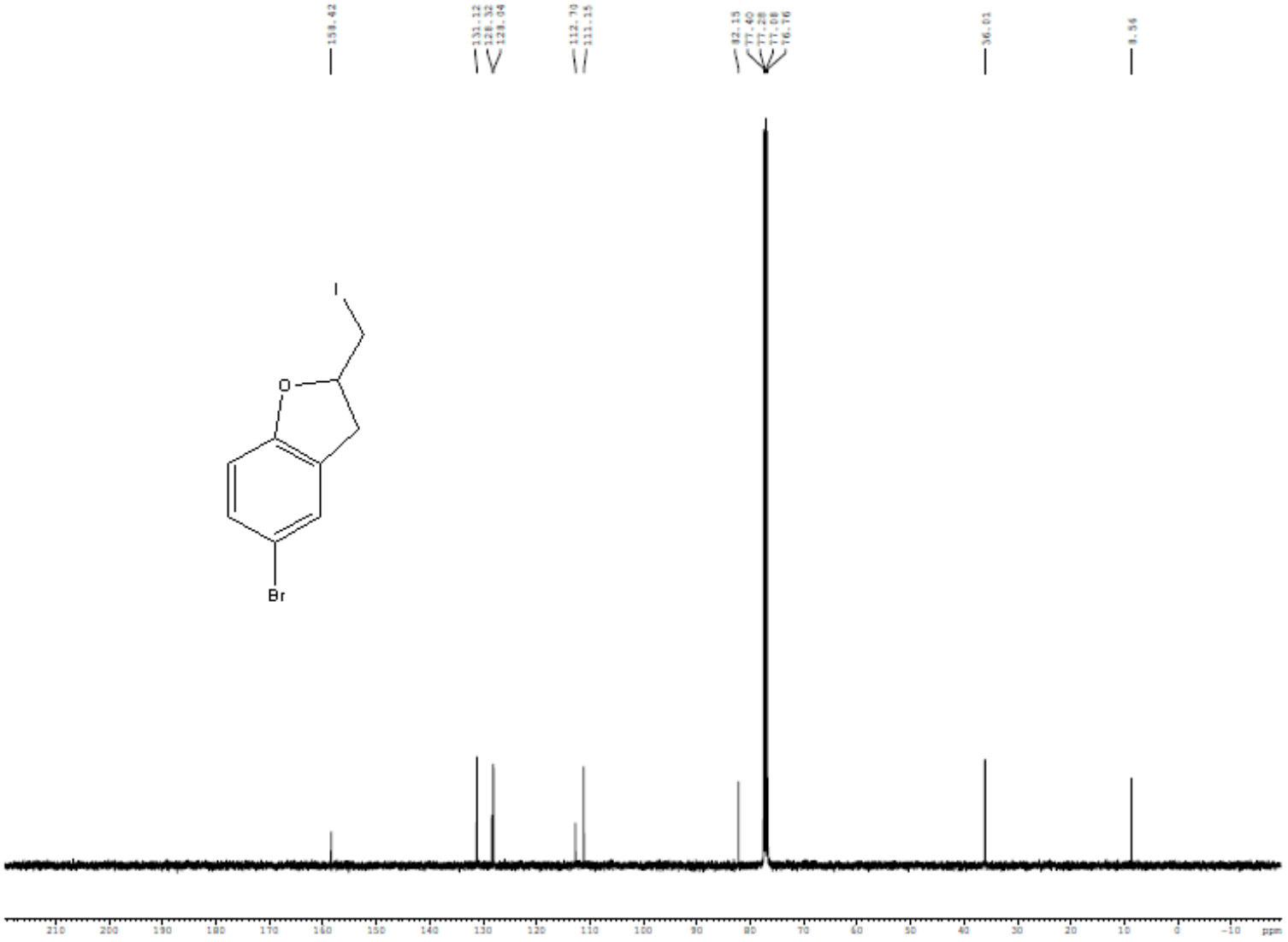

ldy2806 full.10.fid
PROTON.uc1 CDC13 {W:\600} jdw 42
O
I
2.04
1.02
0.98
1.00
1.01
1.04
1.02
1.03
f1 (ppm)


ldy170S column. 10. fid
PROTON. uc1 CDC13 {U:\400} jdw 5
O
I
1.97
0.95
1.00
3.14
1.01
2.93
f1 (ppm)

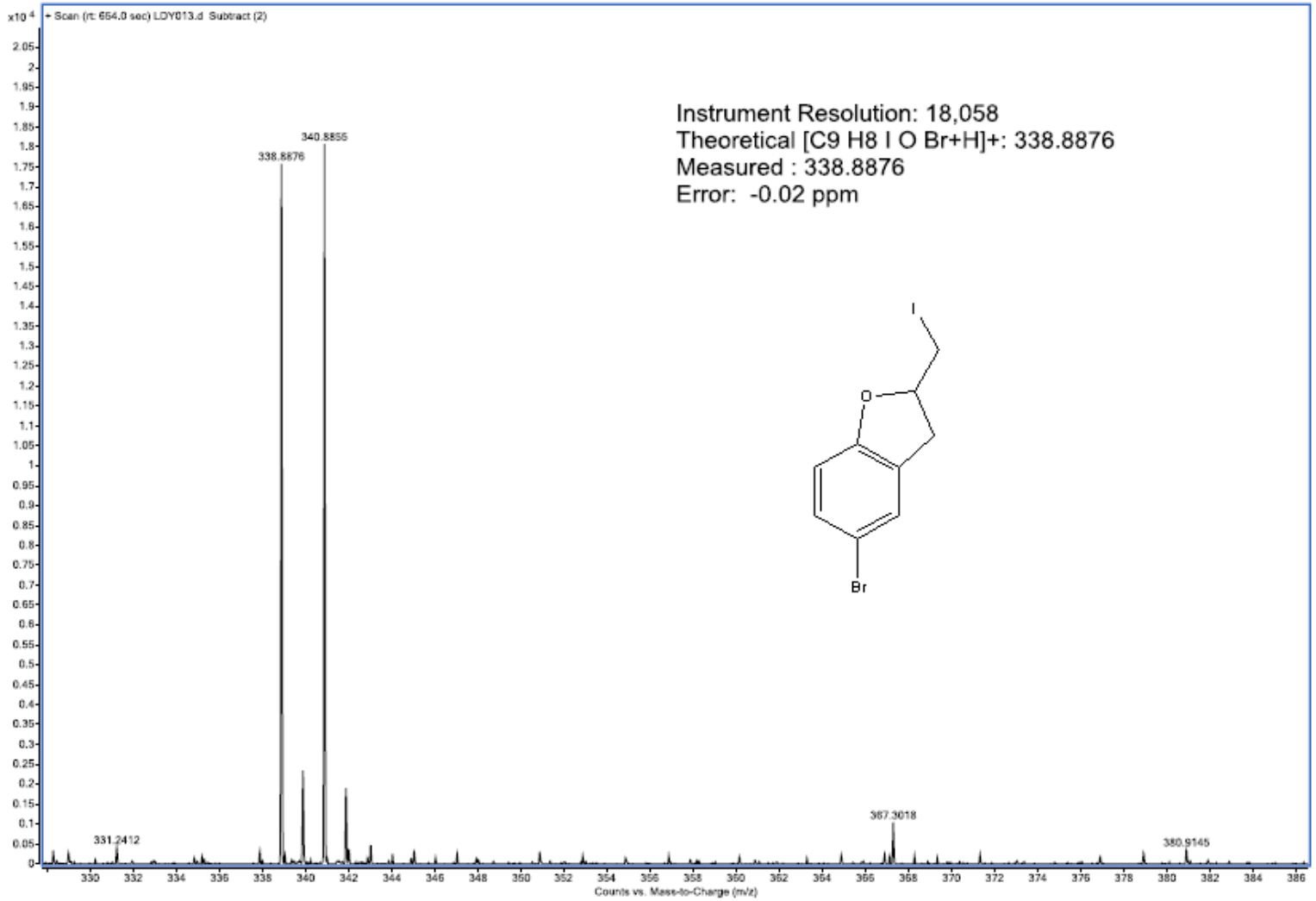

1dy 3008 cyc chloro.10.fid
PROTON.uc1 CDCl3 {U:\400} jdw 9
O
I
Cl
1.97
0.98
1.00
3.10
1.01
f1 (ppm)

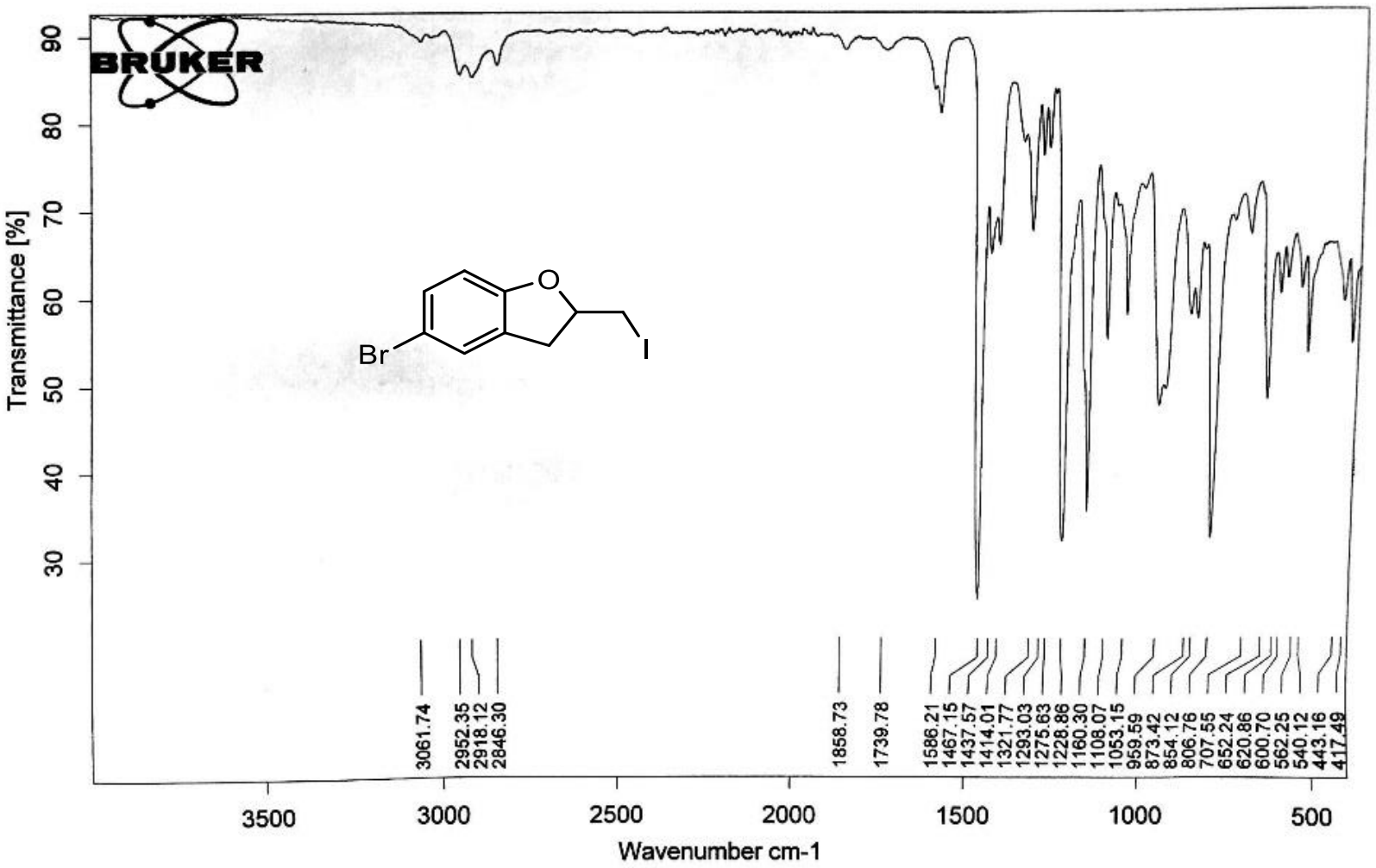

ldy0709 cycbromo1.10.fid
PROTON.uc1 CDC13 {U:\400} jdw 10
O
I
Br
1.04
0.97
0.94
1.00
3.09
1.02
f1 (ppm)

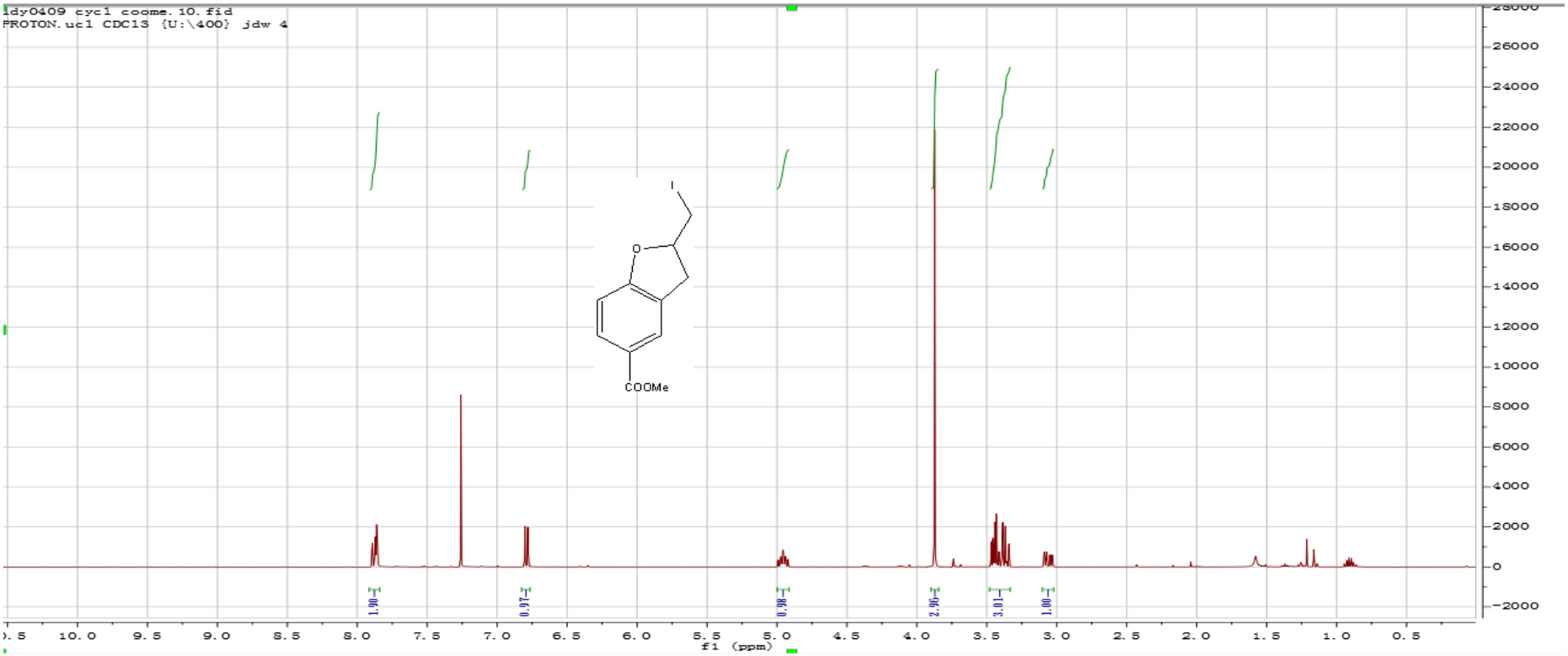
C13CPD.ucl CDCl3 (U:\400) jdw 10



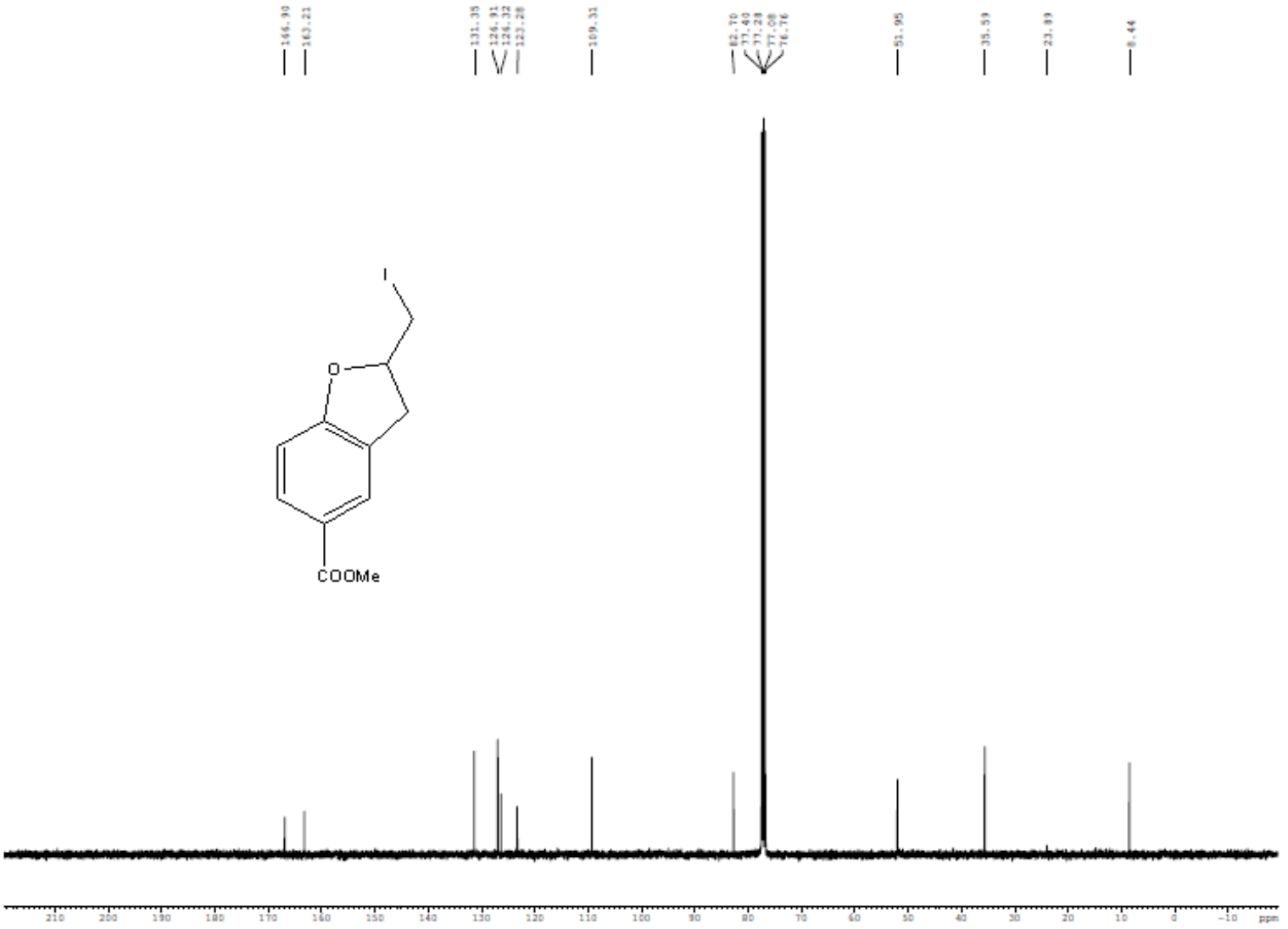

```
Current Data Parameters
NAME      ldy0709 cychromo
EXPNO              10
PROCNO              1

F2 - Acquisition Parameter
Date_           20180907
Time              17.35
INSTRUM           av400
PROBHD   5 mm QNP 1H/15
PULPROG          zgpg30
TD               52882
SOLVENT          CDCl3
NS                1200
DS                   0
SWH          24038.461 H
FIDRES        0.454568 H
AQ            1.0999455 s
RG                 256
DW               20.800 u
DE                6.50 u
TE              297.8 K
D1           2.00000000 s
D11          0.03000000 s
TD0                  75

======== CHANNEL f1 ======
SFO1        100.6228298 M
NUC1               13C
P1                9.00 u
PLW1         65.29000092 W

======== CHANNEL f2 ======
SFO2        400.1316005 M
NUC2               1H
CPDPRG[2         waltz16
PCPD2             90.00 u
PLW2         13.81499958 W
PLW12         0.28823999 W
PLW13         0.23346999 W

F2 - Processing parameter
SI                65536
SF          100.6127626 M
WDW                  EM
SSB       0
LB                1.00 H
GB        0
PC                1.40
```


Scan (rt: 654.0 sec) LDY013.d Subtract (2)
x10 4
Instrument Resolution: 18,058
Theoretical [C9 H8 I O Br+H]+: 338.8876
Measured : 338.8876
Error: -0.02 ppm
338.8876
340.8855
331.2412
367.3018
380.9145
I
O
Br
Counts vs. Mass-to-Charge (m/z)

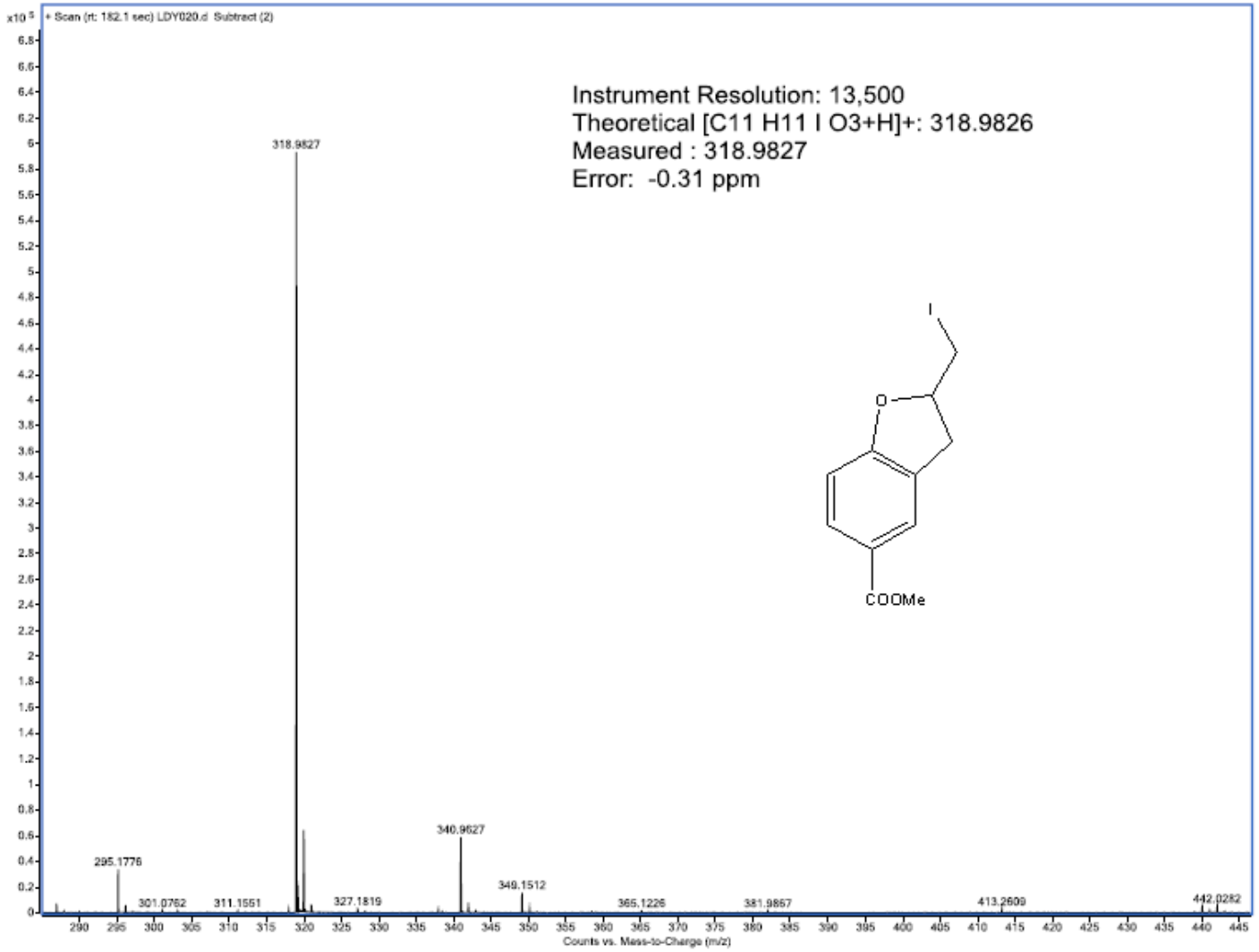

BRUKER
Transmittance [%]
90
80
70
60
50
40
30
Br
O
I
3061.74
2952.35
2918.12
2846.30
1858.73
1739.78
1586.21
1467.15
1437.57
1414.01
1321.77
1293.03
1275.63
1228.86
1160.30
1108.07
1053.15
959.59
873.42
854.12
806.76
707.55
652.24
620.86
600.70
562.25
540.12
443.16
417.49
3500
3000
2500
2000
1500
1000
500
Wavenumber cm-1

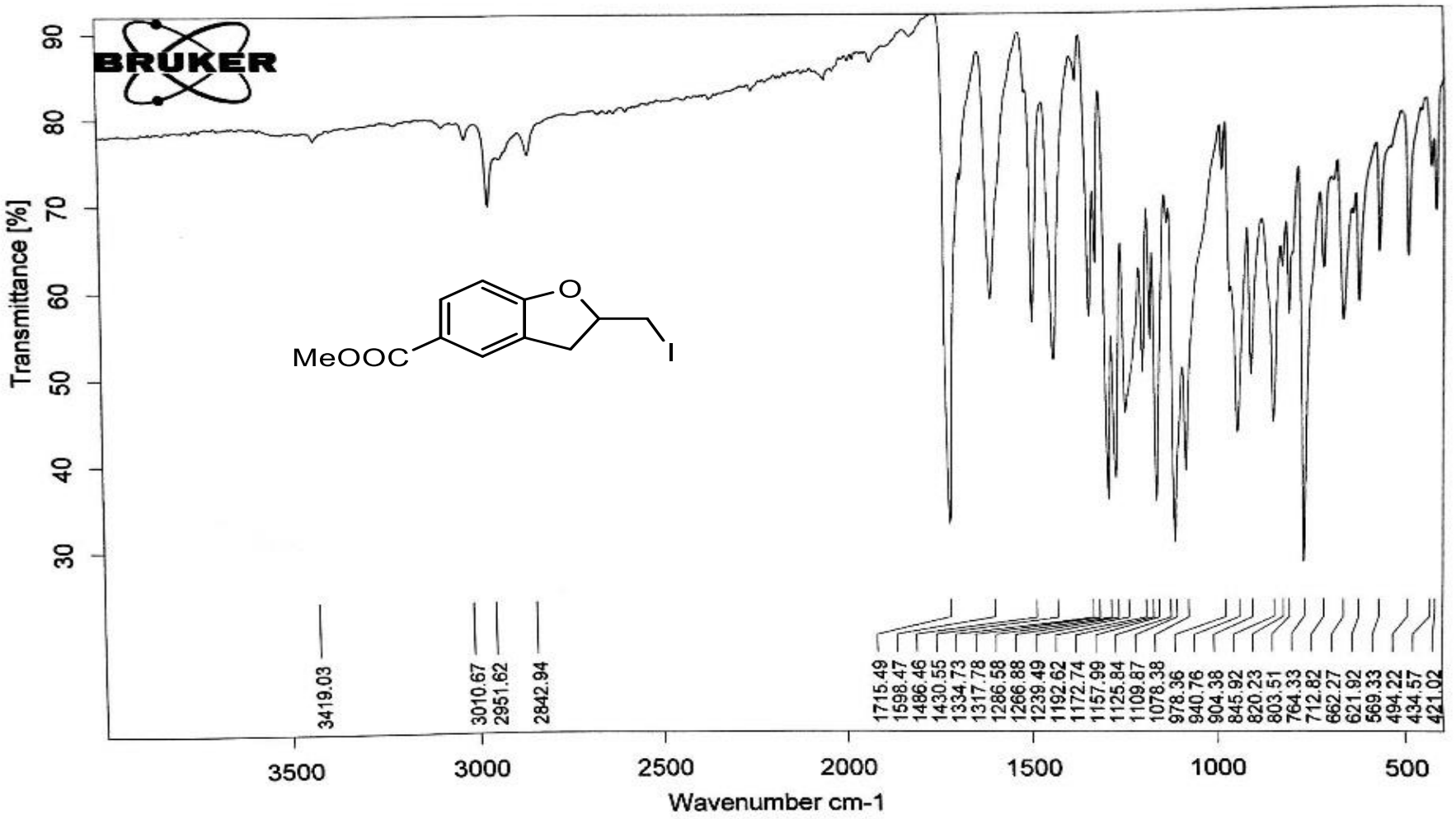

jdy0409 cyc1 coome. 10. fid
PROTON. uc1 CDC13 {U:\400} jdw 4
O
I
COOMe
1.90
0.97
0.98
2.96
3.01
1.00
f1 (ppm)

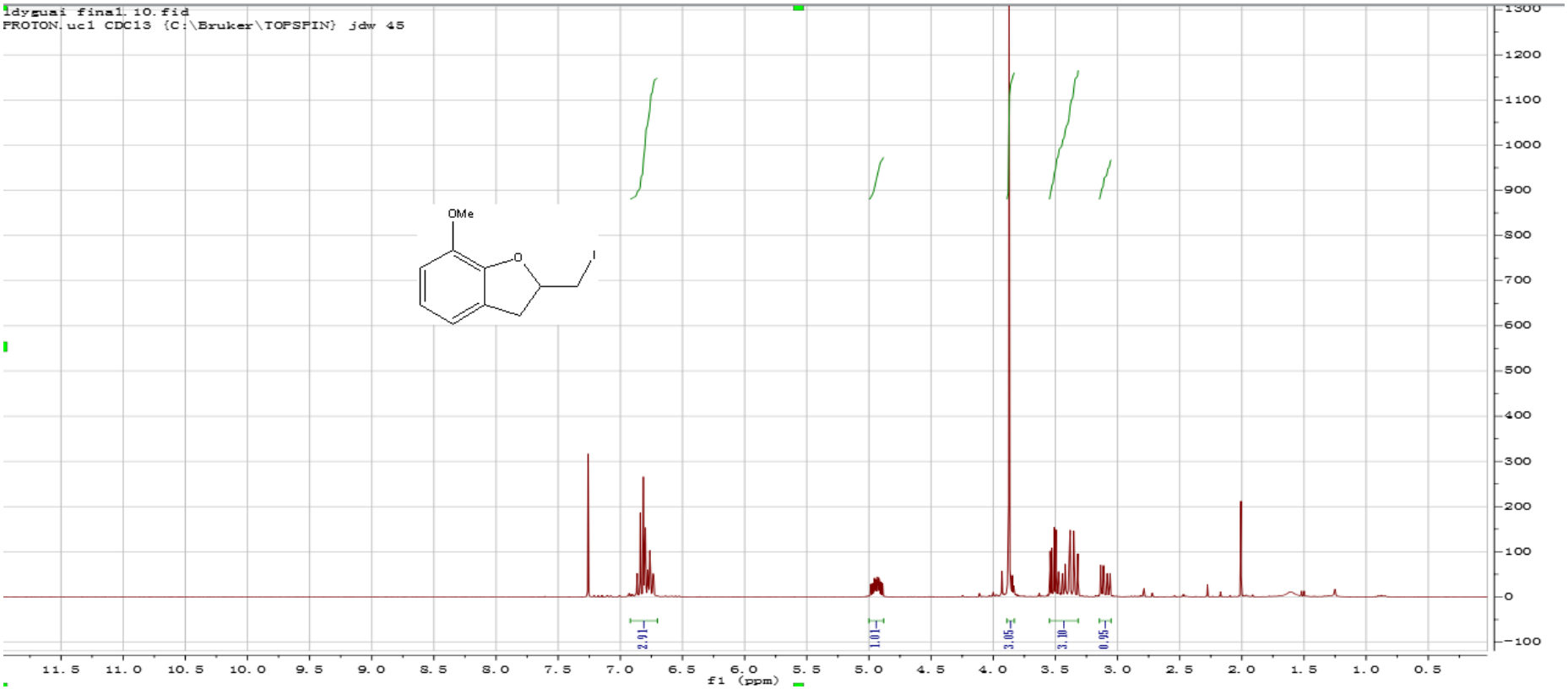
C13CPD.ucl CDCl3 (U:\400) jdw 4



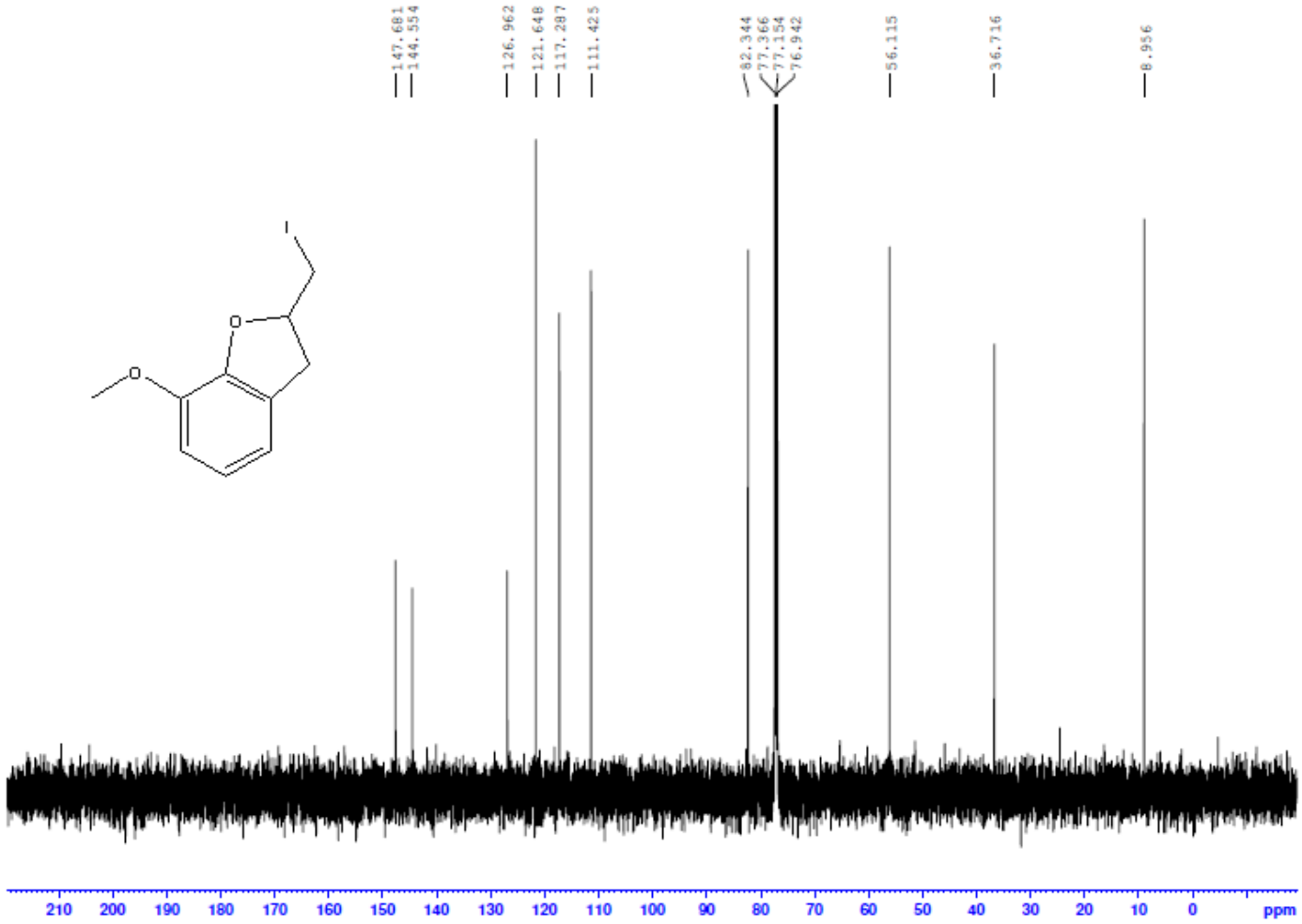

```
Current Data Parameters
NAME      1dy cycl ccome c
EXPNO              10
PROCNO              1

F2 - Acquisition Parameter
Date_          20180904
Time             21.02
INSTRUM          av400
PROBHD   5 mm QNP 1H/15
PULPROG          zgpg30
TD              52882
SOLVENT          CDCl3
NS               1200
DS                  0
SWH          24038.461 H
FIDRES        0.454568 H
AQ            1.0999455 s
RG                256
DW             20.800 u
DE              8.38 u
TE             298.2 K
D1          2.00000000 s
D11         0.03000000 s
TD0                 75

======== CHANNEL f1 ======
SFO1       100.6228298 M
NUC1             13C
P1              9.00 u
PLW1       65.29000092 W

======== CHANNEL f2 ======
SFO2       400.1316005 M
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 u
PLW2       13.81499958 W
PLW12       0.28823999 W
PLW13       0.23348999 W

F2 - Processing parameter
SI                65536
SF         100.6127626 M
WDW                EM
SSB         0
LB                1.00 H
GB          0
PC                1.40
```


+ Scan (rt: 182.1 sec) LDY020.d Subtract (2)
Instrument Resolution: 13,500
Theoretical [C11 H11 I O3+H]+: 318.9826
Measured : 318.9827
Error: -0.31 ppm
318.9827
340.9627
295.1776
349.1512
301.0762
311.1551
327.1819
365.1226
381.9867
413.2609
442.0282
I
O
COOMe
Counts vs. Mass-to-Charge (m/z)

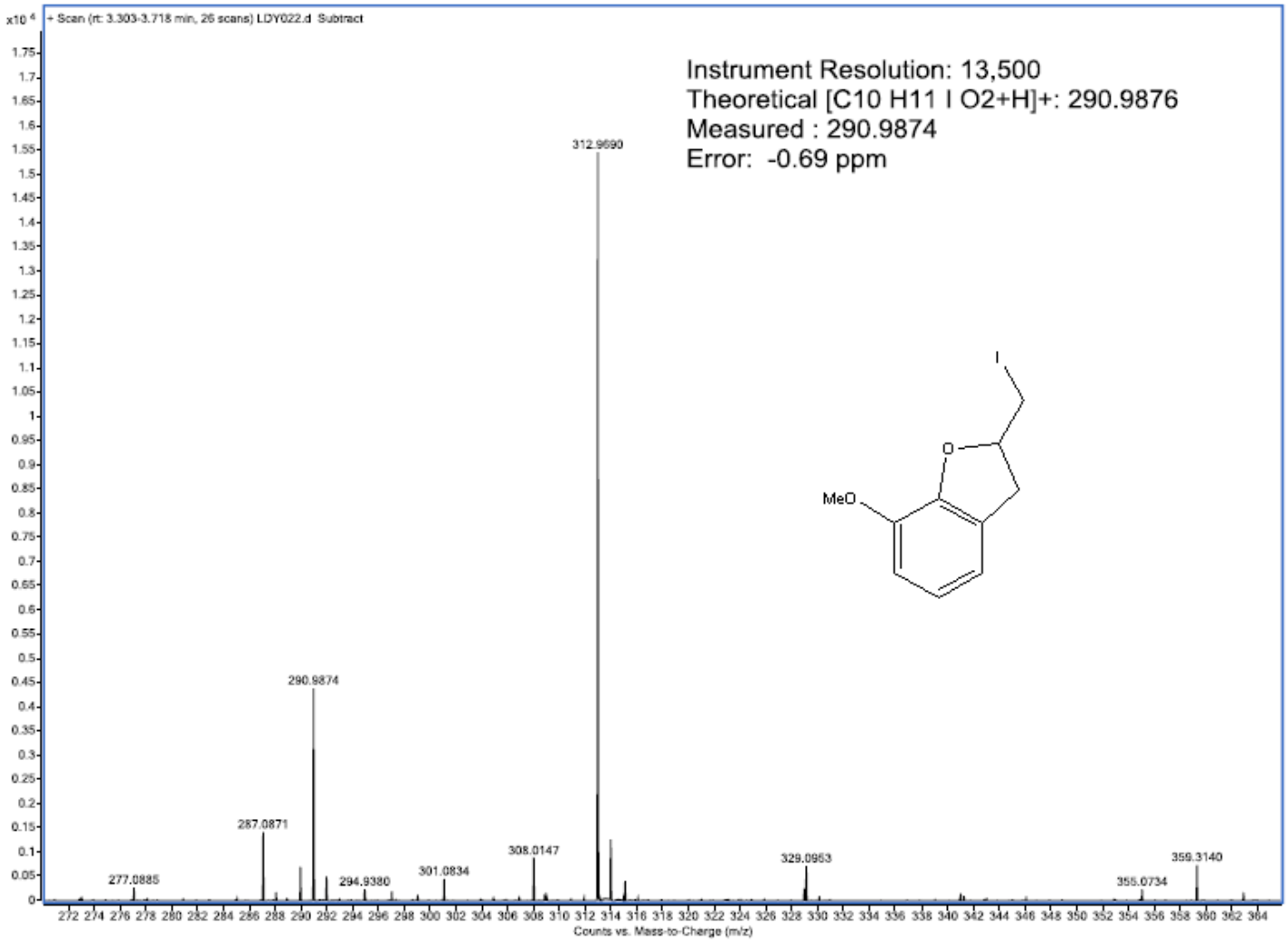

BRUKER
Transmittance [%]
90
80
70
60
50
40
30
MeOOC
O
I
3419.03
3010.67
2951.62
2842.94
1715.49
1598.47
1486.46
1430.55
1334.73
1317.78
1286.58
1266.88
1239.49
1192.62
1172.74
1157.99
1125.84
1109.87
1078.38
978.36
940.76
904.38
845.92
820.23
803.51
764.33
712.82
662.27
621.92
569.33
494.22
434.57
421.02
3500
3000
2500
2000
1500
1000
500
Wavenumber cm-1

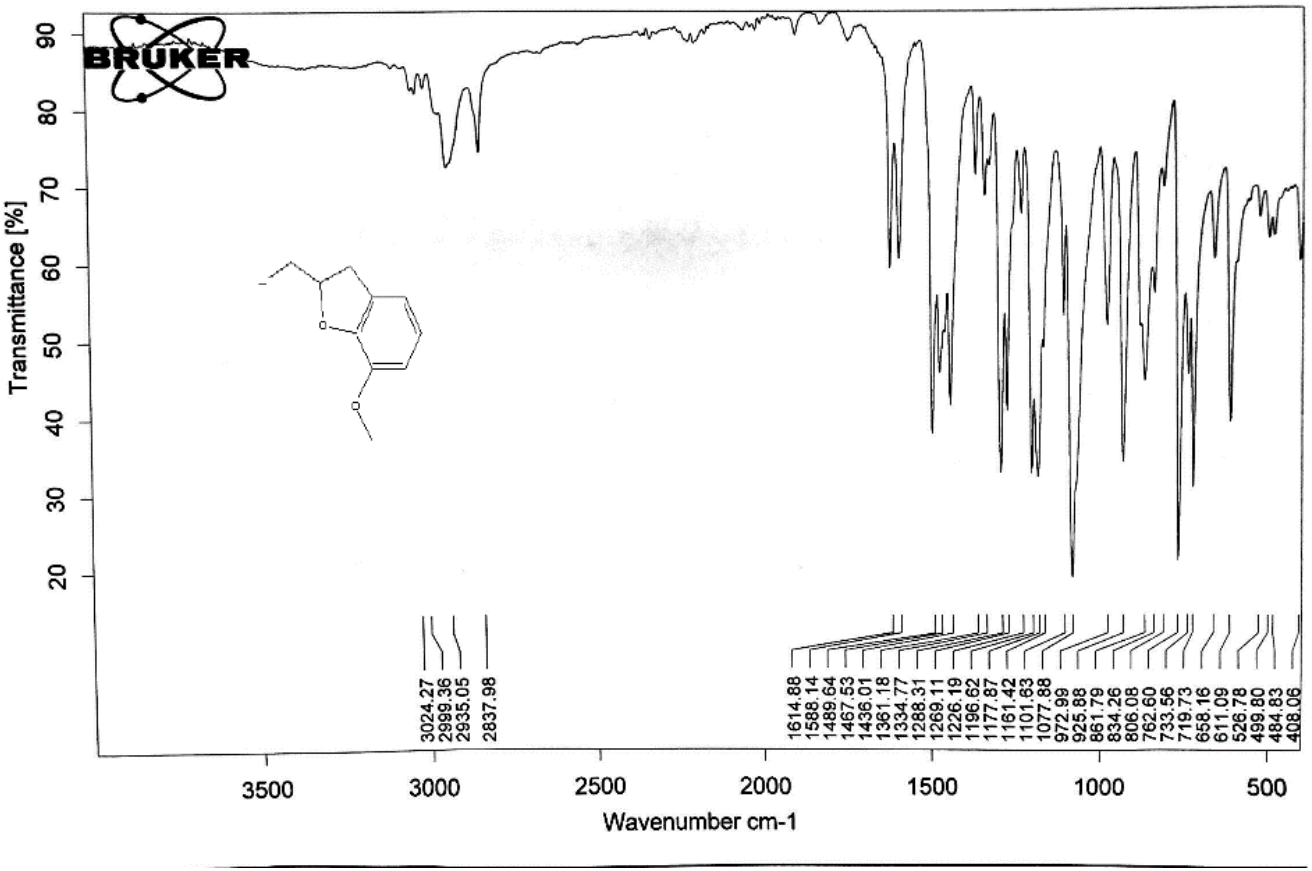

idyguai final 10.fid
PROTON uc1 CDCl3 {C:\Bruker\TOPSPIN} jdw 45
OMe
O
I
2.91
1.01
3.05
3.10
0.95
f1 (ppm)

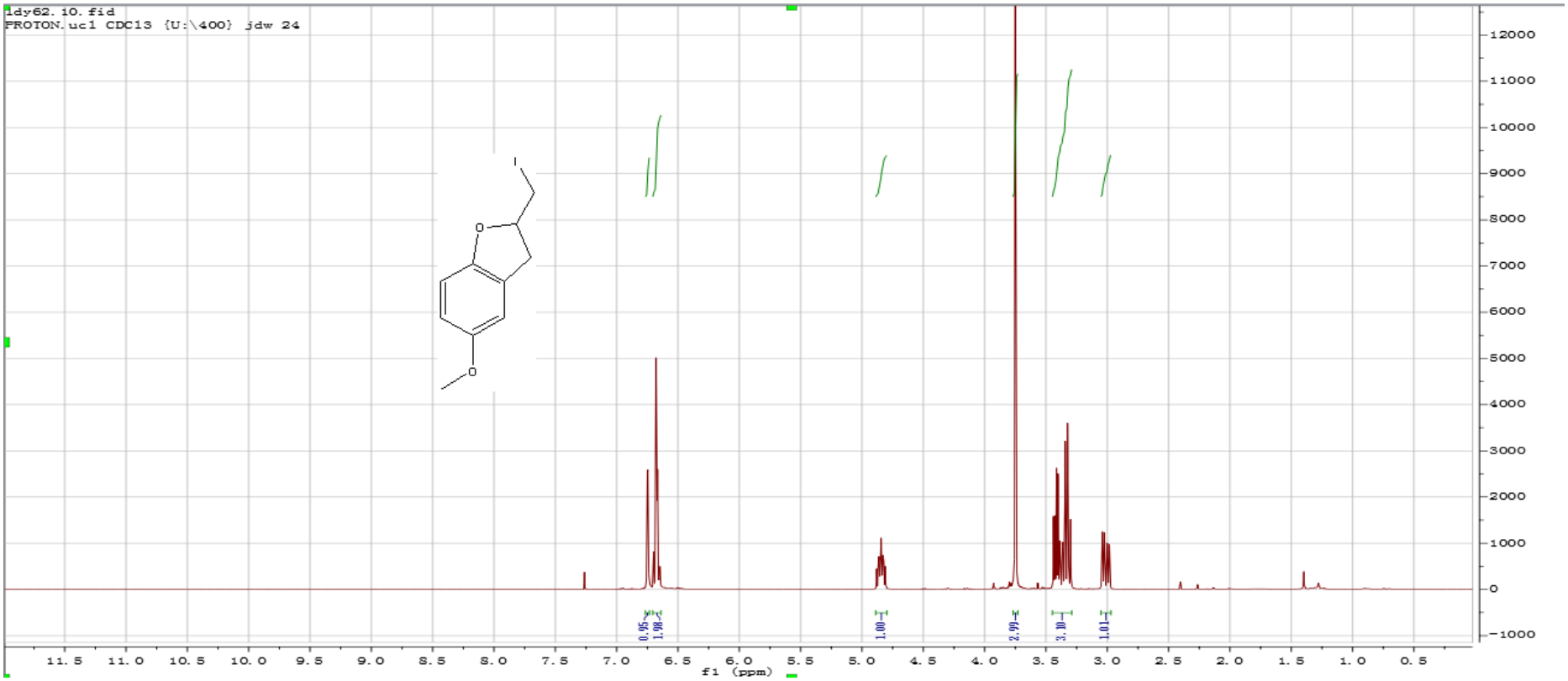
C13CPD.ucl CDCl3 {W:\600} jdw 58



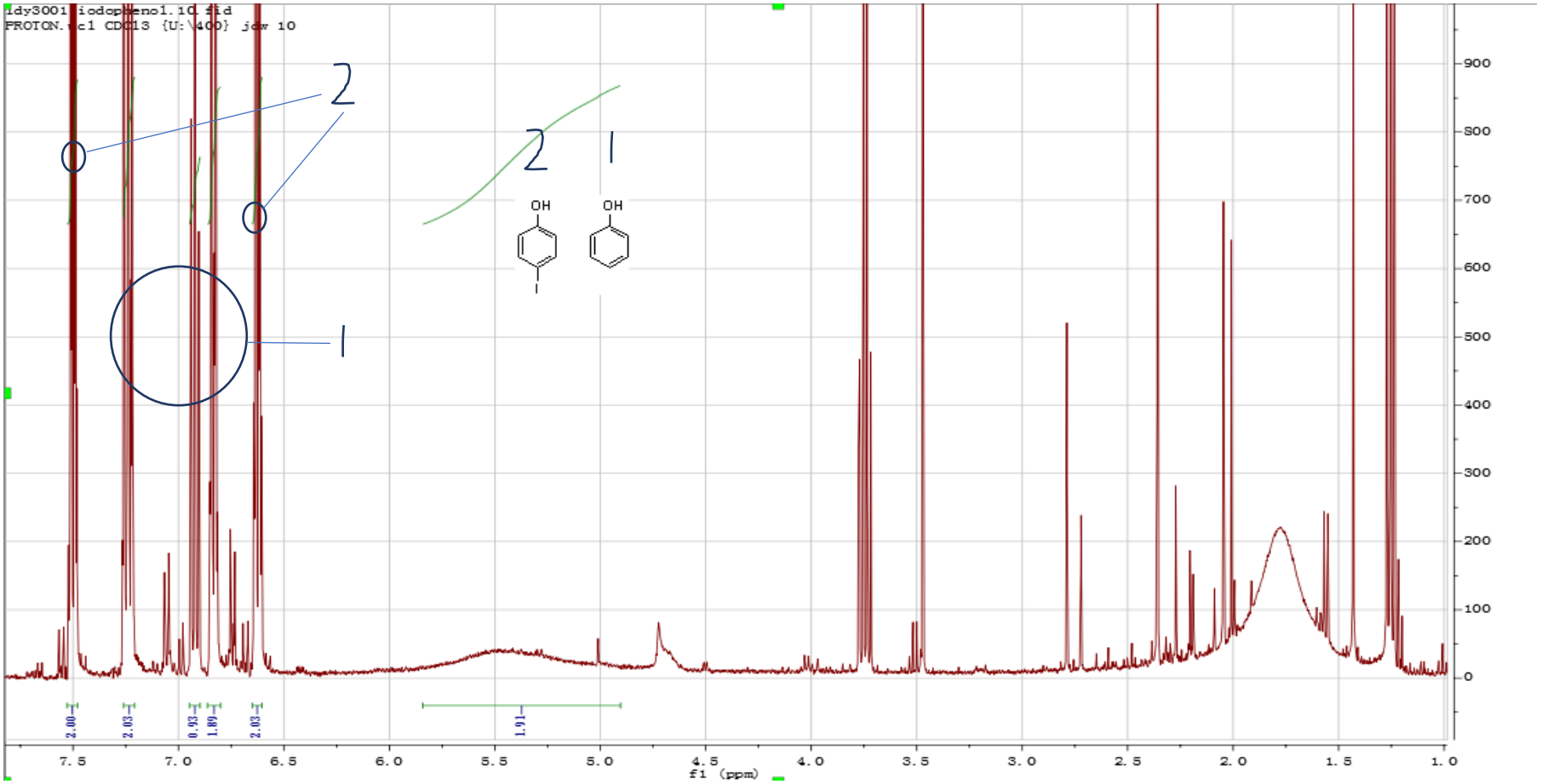

```
Current Data Parameters
NAME          1dy qual cyc carb
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameter
Date_              20180913
Time                  21.26
INSTRUM                AV600
PROBHD    5 mm TXI 1H/D-
PULPROG              zgpg30
TD                    65340
SOLVENT               CDCl3
NS                     1024
DS                        4
SWH                36057.691 Hz
FIDRES              0.551847 Hz
AQ                  0.9060480 sec
RG                       1030
DW                    13.867 usec
DE                      6.05 usec
TE                    298.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1           150.9178976 MHz
NUC1                     13C
P1                     12.80 usec
PLW1          260.00000000 W

======== CHANNEL f2 ========
SFO2           600.1324005 MHz
NUC2                     1H
CPDPRG[2 bi_waltz65_128
PCPD2                  70.00 usec
PLW2          14.50000000 W
PLW12          0.18939000 W
PLW13          0.09280000 W

F2 - Processing parameters
SI                       32768
SF             150.9027906 MHz
WDW                         EM
SSB           0
LB                     1.00 Hz
GB            0
PC                     1.40
```


+ Scan (rt: 3.303-3.718 min, 26 scans) LDY022.d Subtract
Instrument Resolution: 13,500
Theoretical [C10 H11 I O2+H]+: 290.9876
Measured : 290.9874
Error: -0.69 ppm
312.9690
290.9874
287.0871
277.0885
294.9380
301.0834
308.0147
329.0963
355.0734
359.3140
I
O
MeO
Counts vs. Mass-to-Charge (m/z)


BRUKER
Transmittance [%]
90
80
70
60
50
40
30
20
3024.27
2999.36
2935.05
2837.98
1614.88
1588.14
1489.64
1467.53
1436.01
1361.18
1334.77
1288.31
1269.11
1226.19
1196.62
1177.87
1161.42
1101.63
1077.88
972.99
925.88
861.79
834.26
806.08
762.60
733.56
719.73
658.16
611.09
526.78
499.80
484.83
408.06
3500
3000
2500
2000
1500
1000
500
Wavenumber cm-1

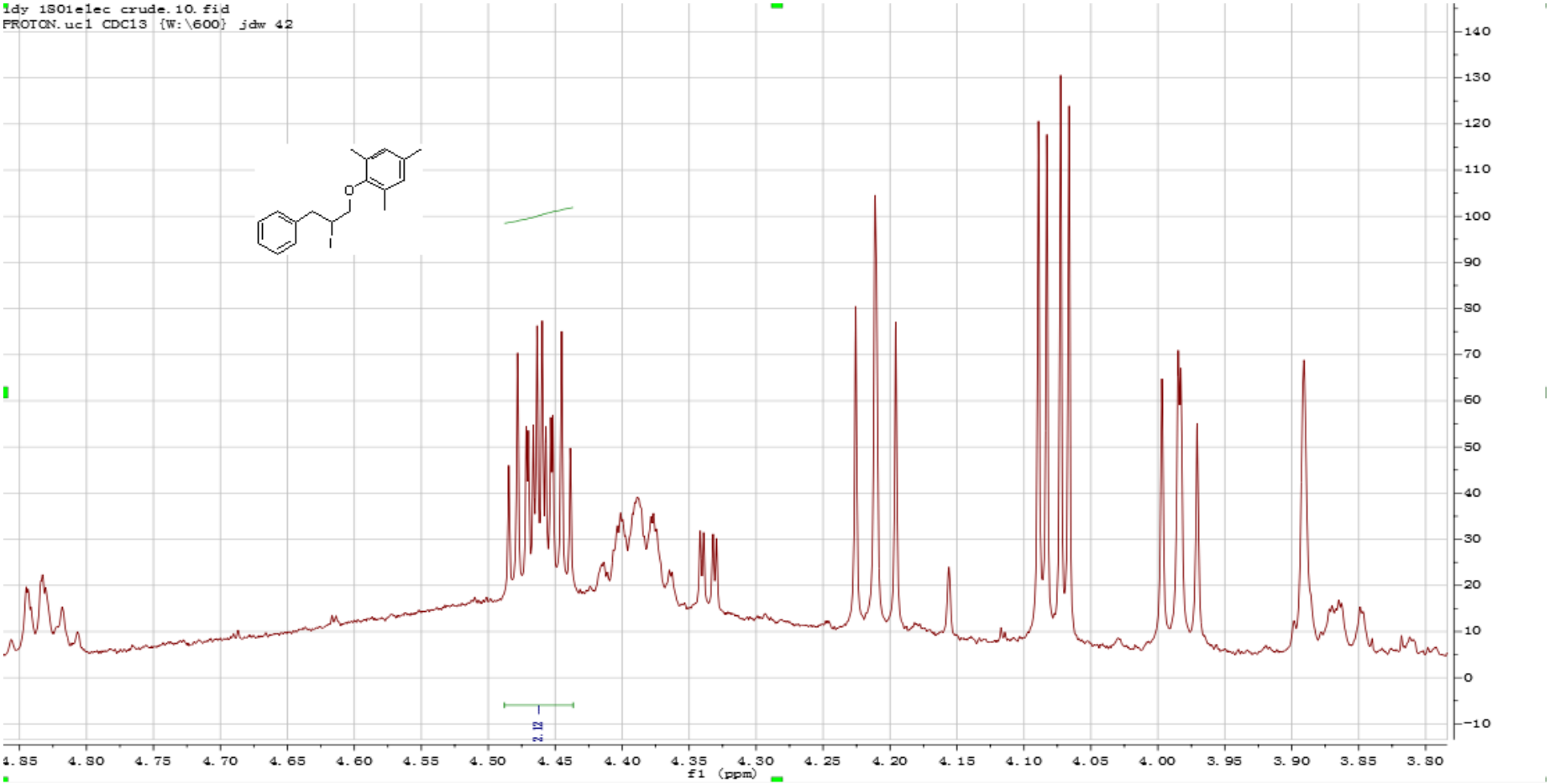

ldy62.10.fid
PROTON.uc1 CDC13 {U:\400} jdw 24
I
O
O
0.95
1.98
1.00
2.99
3.10
1.01
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

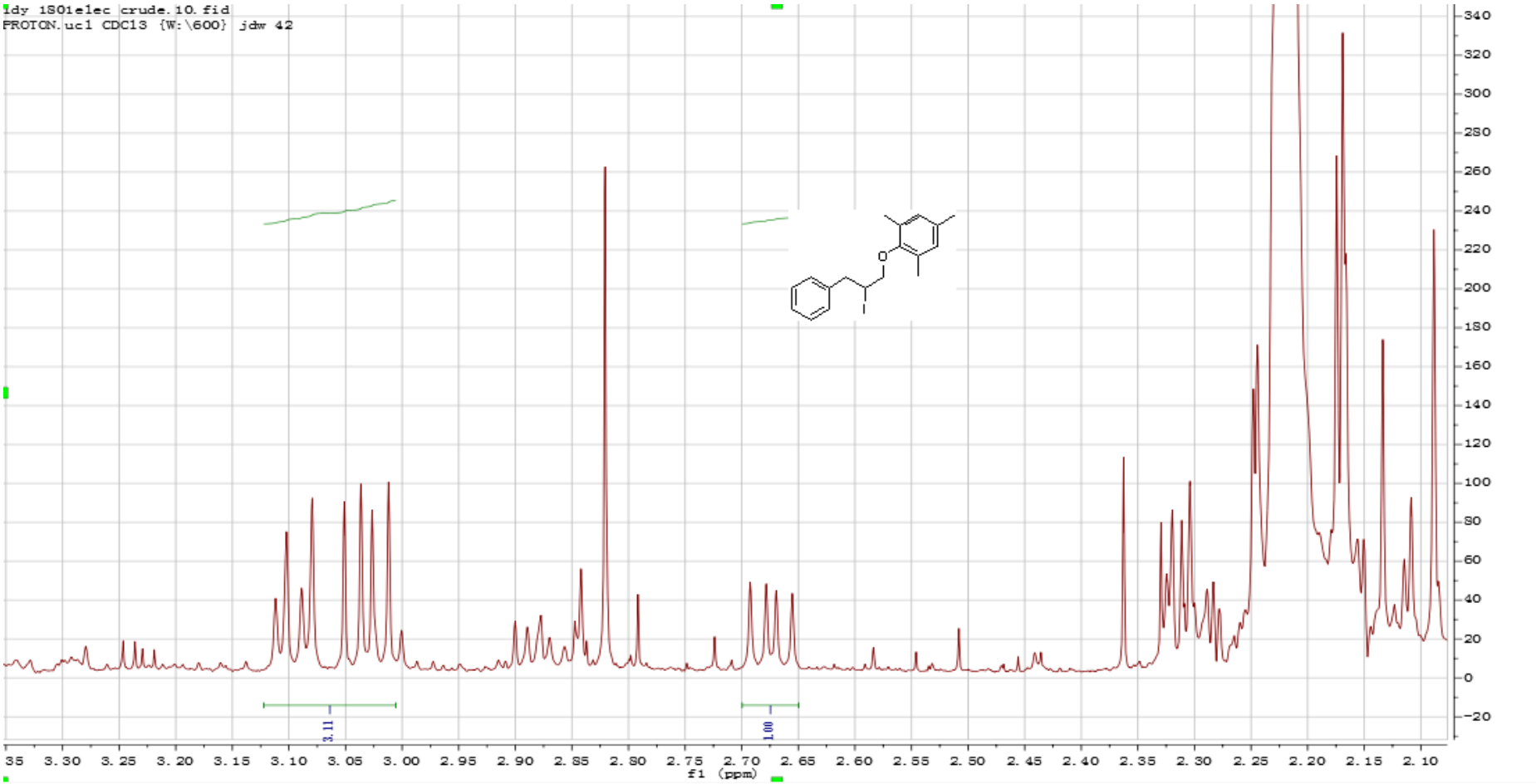
Crude NMR showing a mixture of phenol (product) and 4-iodophenol (Starting material)



For the aromatic part of this NMR spectrum, the middle three peaks pertaining to phenol (1), the left and right peaks belong to 4-iodophenol (2)

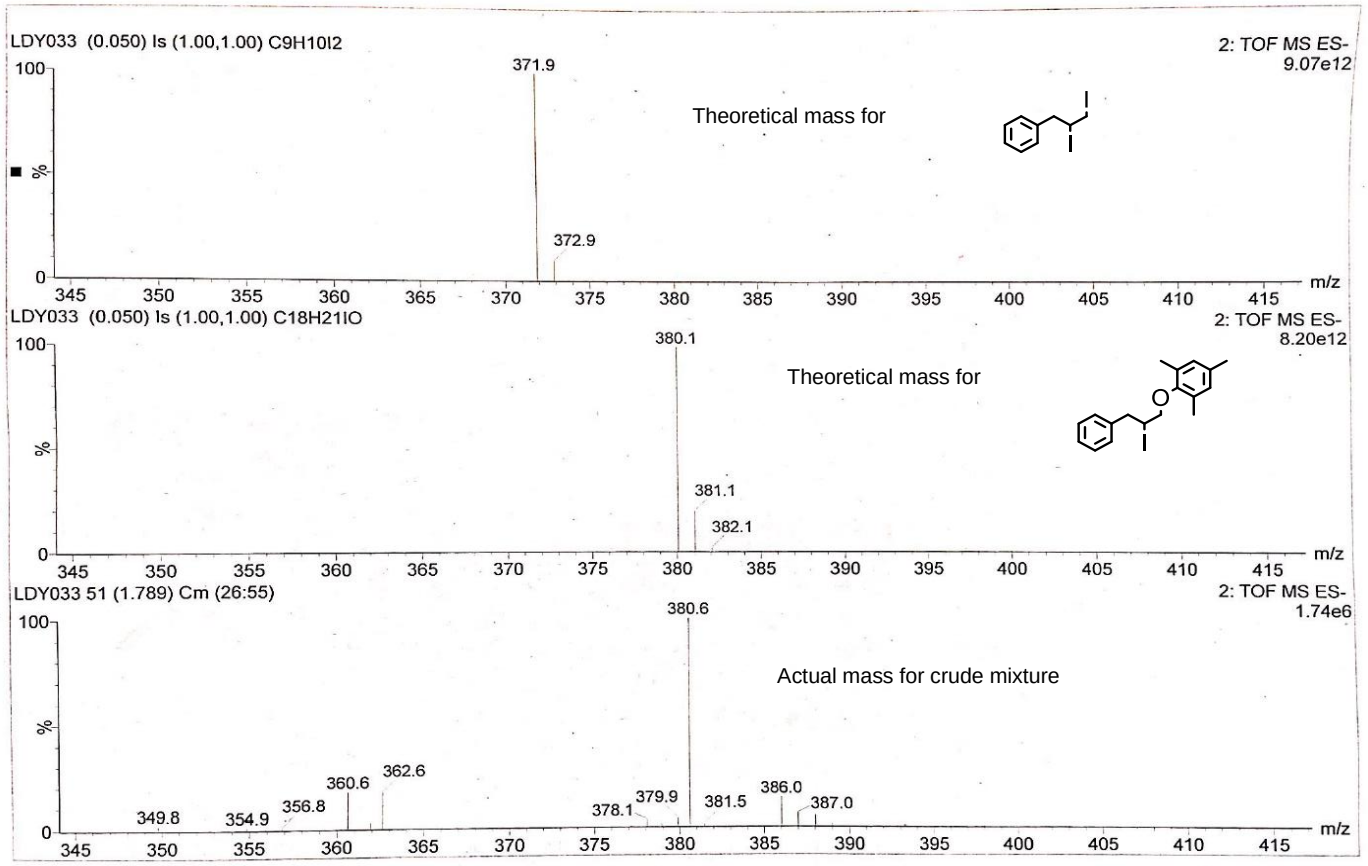
Crude ^{1}H NMR showing the characteristic peaks of the bottom product for Scheme 13:

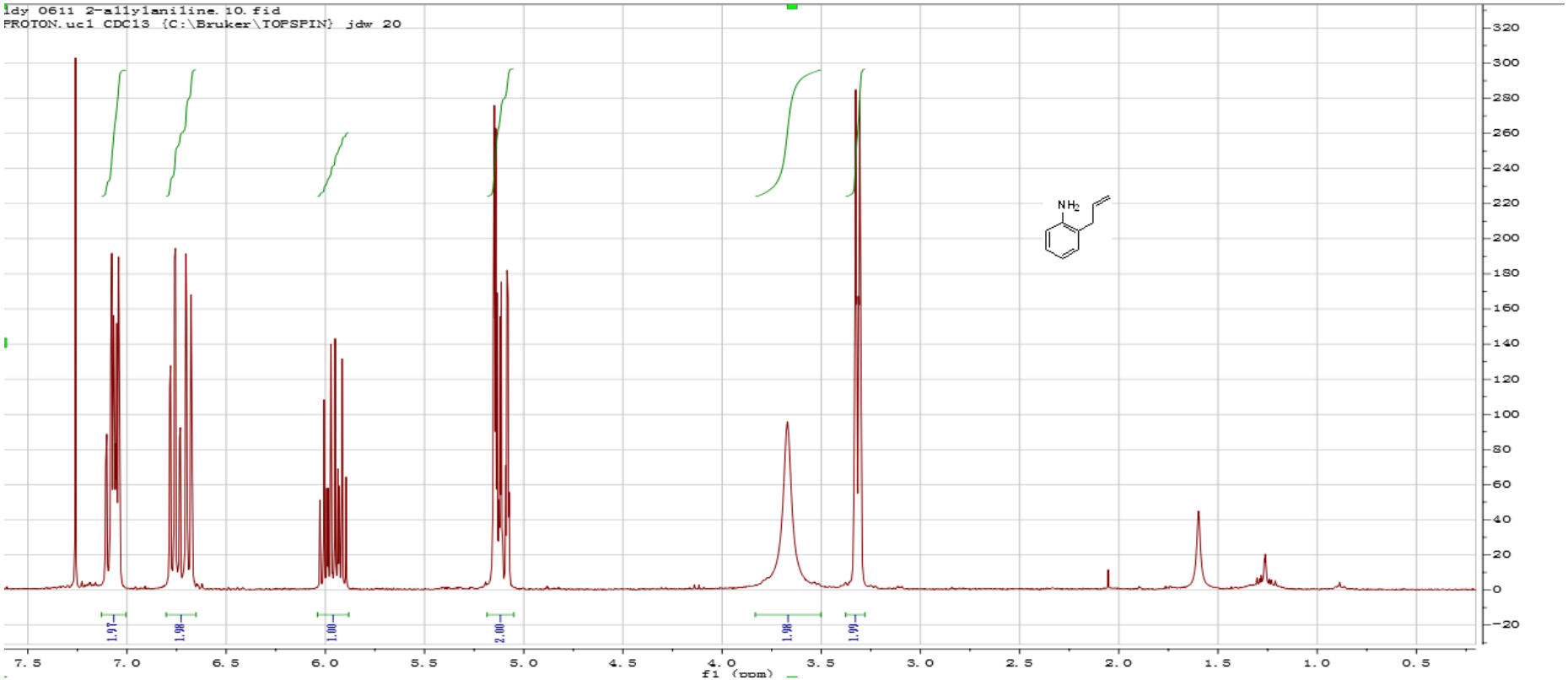




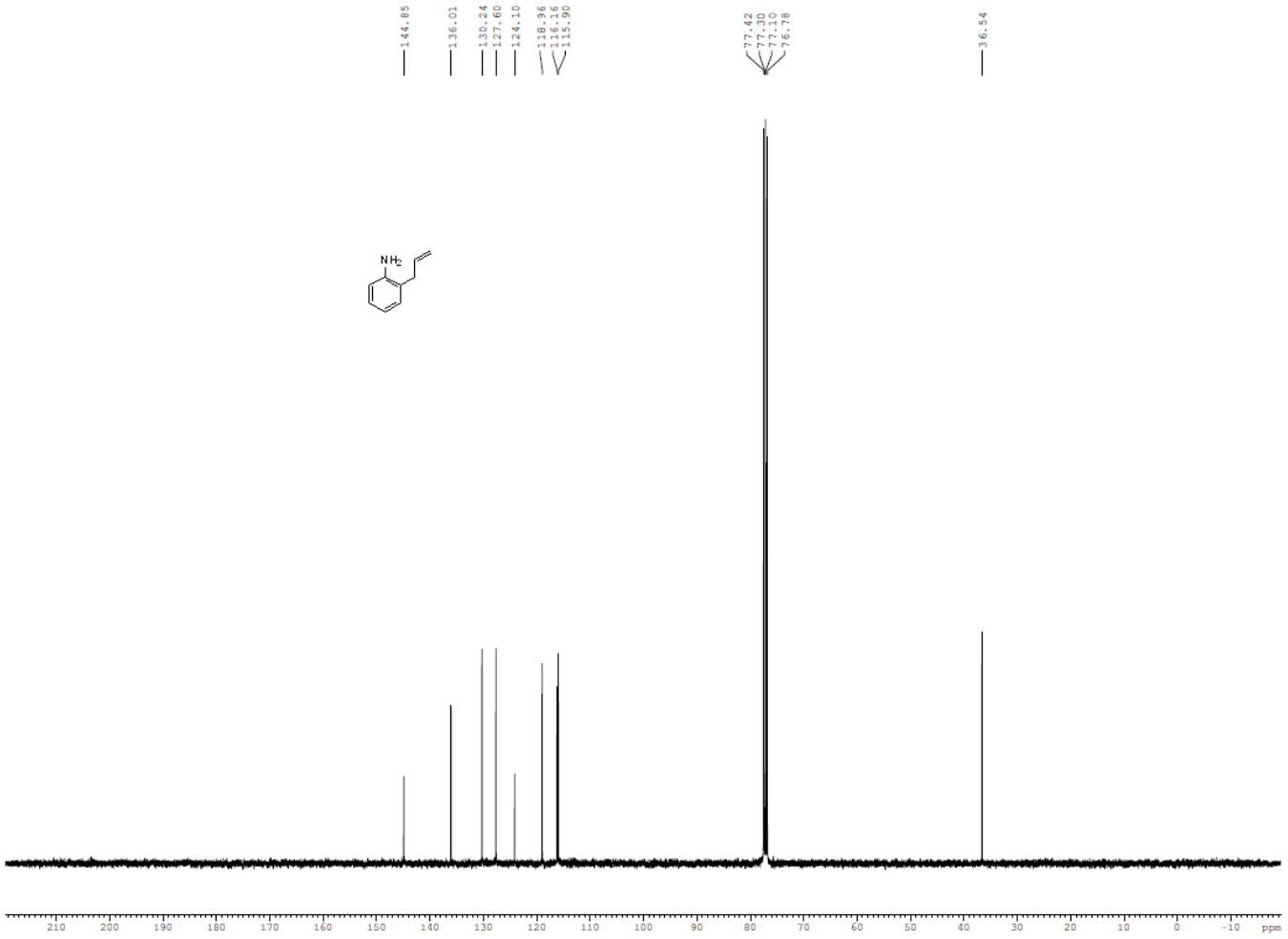
The 4.45 ppm peak has combined with OH, hence a little bit larger in integration

Mass spec below proved the existence of Scheme 13 bottom product:

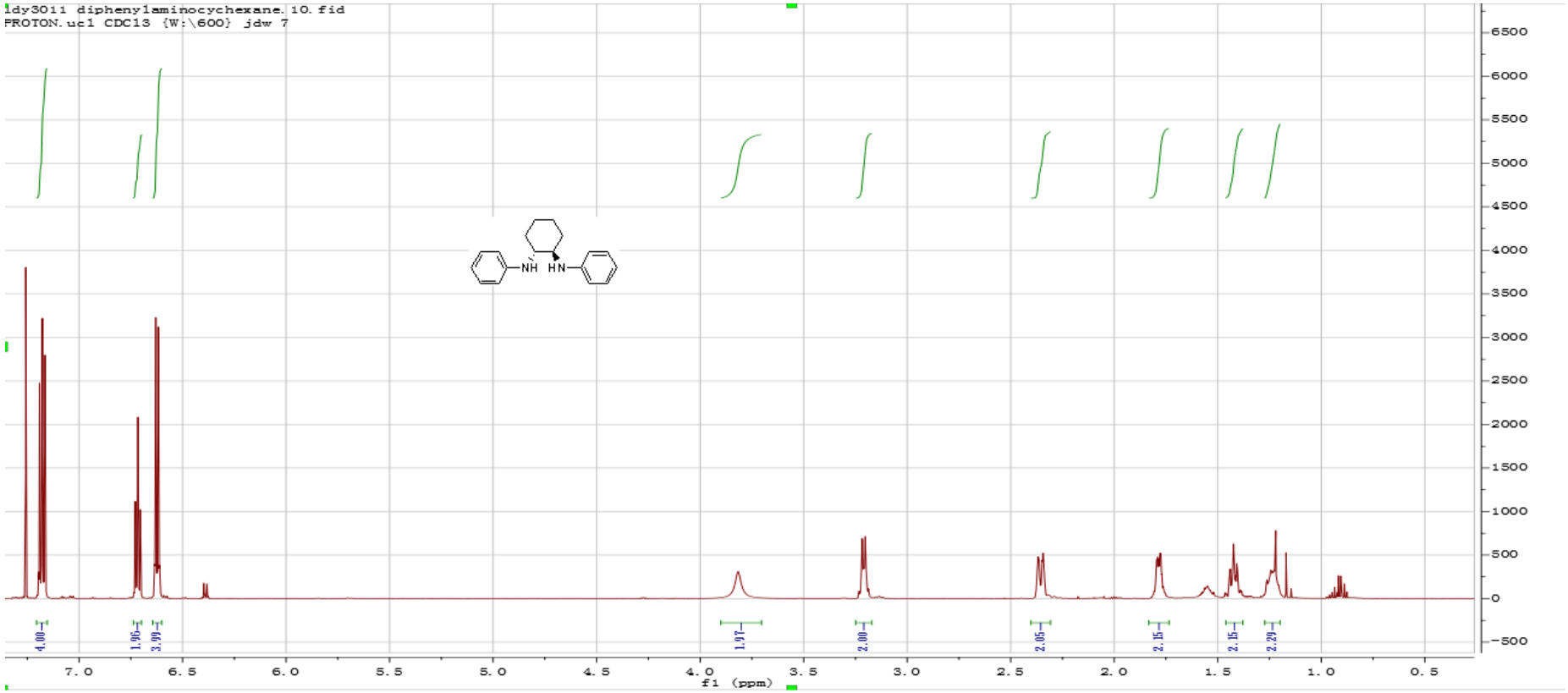



ldy 0611 2-allylaniline.10.fid
PROTON.uc1 CDCl3 {C:\Bruker\TOPSPIN} jdw 20
NH2
1.97
1.98
1.00
2.00
1.98
1.99
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)
320
300
280
260
240
220
200
180
160
140
120
100
80
60
40
20
0
-20

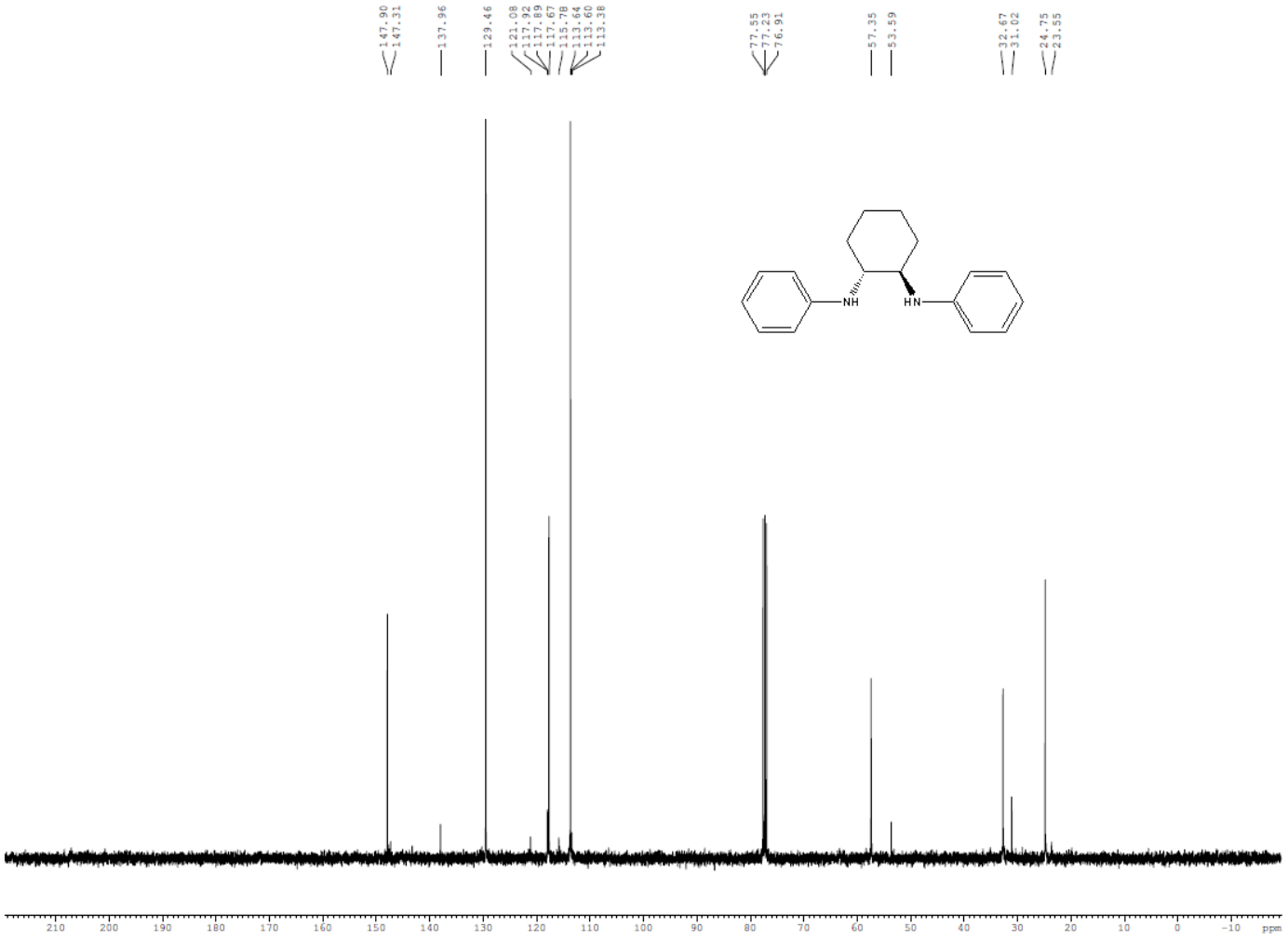
C13CPD.ucl CDCl3 {U:\400} jdw 3



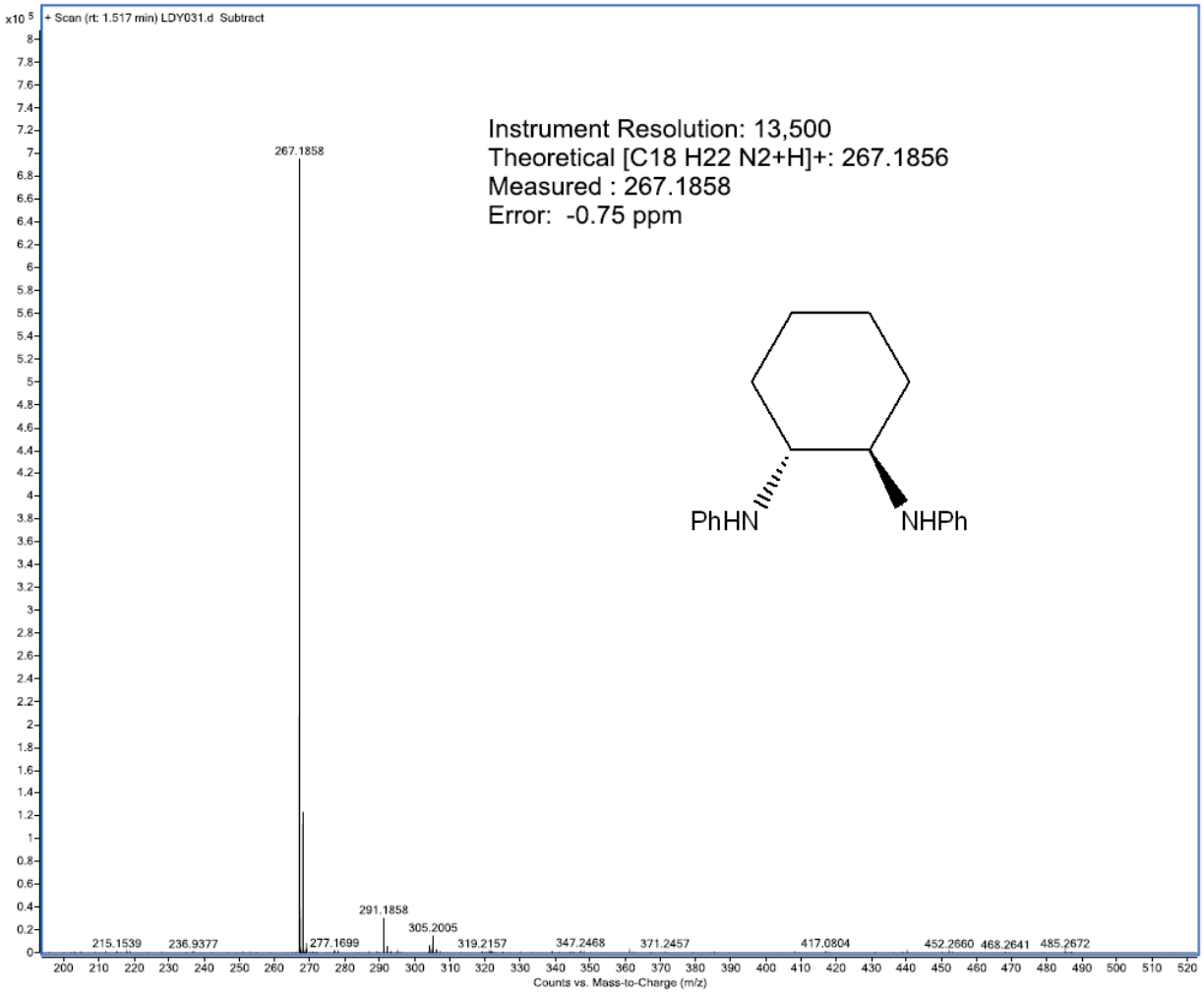

```
Current Data Parameters
NAME      ldy 2-allylanili
EXPNO                   11
PROCNO                   1

F2 - Acquisition Parameter
Date_           20181109
Time                8.23
INSTRUM             av400
PROBHD   5 mm QNP 1H/15
PULPROG          zgpg30
TD                52882
SOLVENT           CDCl3
NS                 1200
DS                    0
SWH           24038.461 H
FIDRES         0.454568 H
AQ             1.0999455 s
RG                  256
DW                20.800 u
DE                 8.38 u
TE                298.2 K
D1            2.00000000 s
D11           0.03000000 s
TD0                  75

======= CHANNEL f1 =====
SFO1         100.6228298 M
NUC1                13C
P1                 9.00 u
PLW1         65.29000092 W

======= CHANNEL f2 =====
SFO2         400.1316005 M
NUC2                 1H
CPDPRG[2          waltz16
PCPD2              90.00 u
PLW2         13.81499958 W
PLW12         0.28823999 W
PLW13         0.23346999 W

F2 - Processing parameter
SI                  65536
SF           100.6127626 M
WDW                    EM
SSB       0
LB                 1.00 H
GB        0
PC                 1.40
```


ldy3011 diphenylaminocychexane.10.fid
PROTON.uc1 CDC13 {W:\600} jdw 7
NH HN
4.00
1.96
3.99
1.97
2.00
2.05
2.15
2.15
2.29
f1 (ppm)

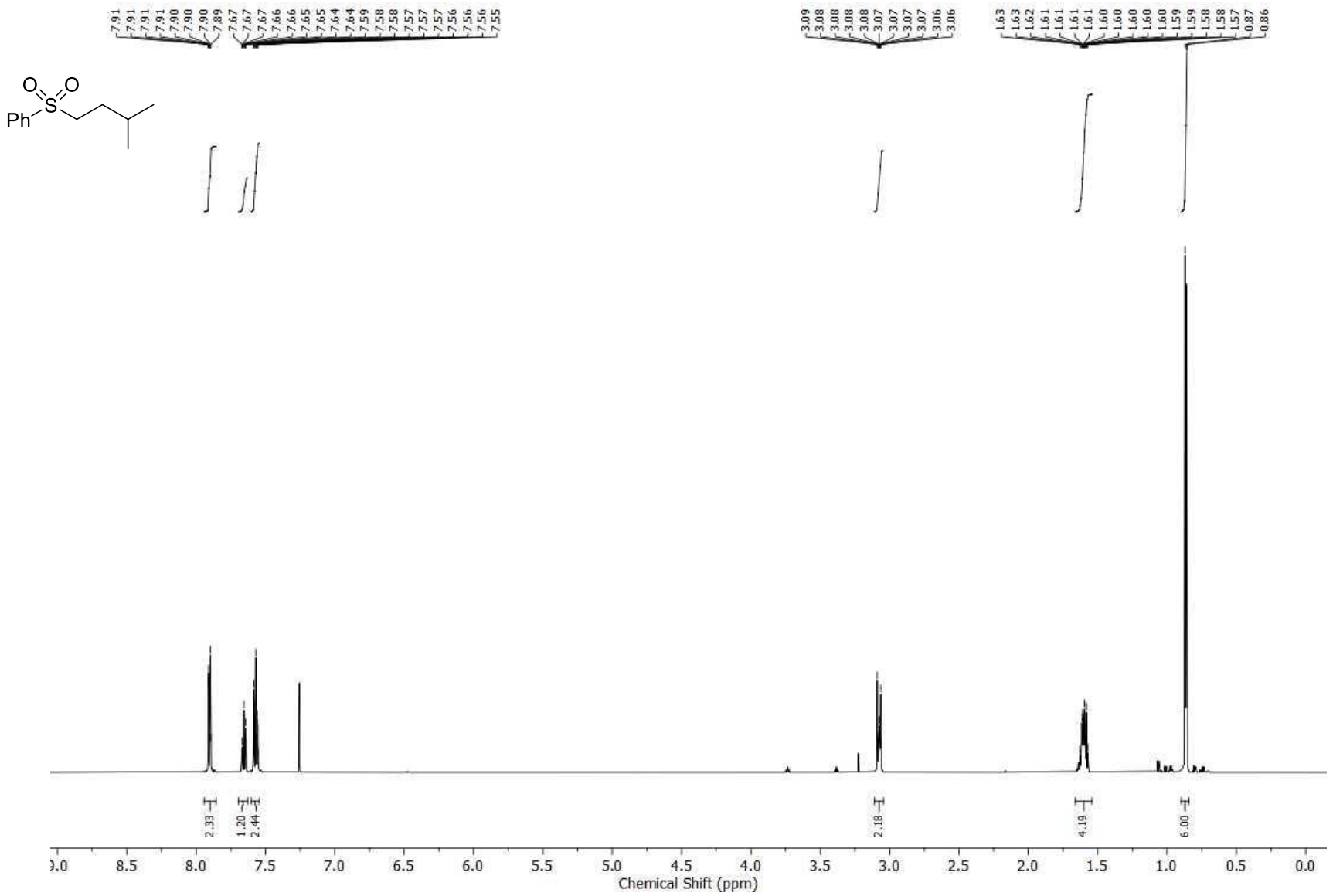
C13CPD.ucl CDCl3 {U:\400} jdw 16

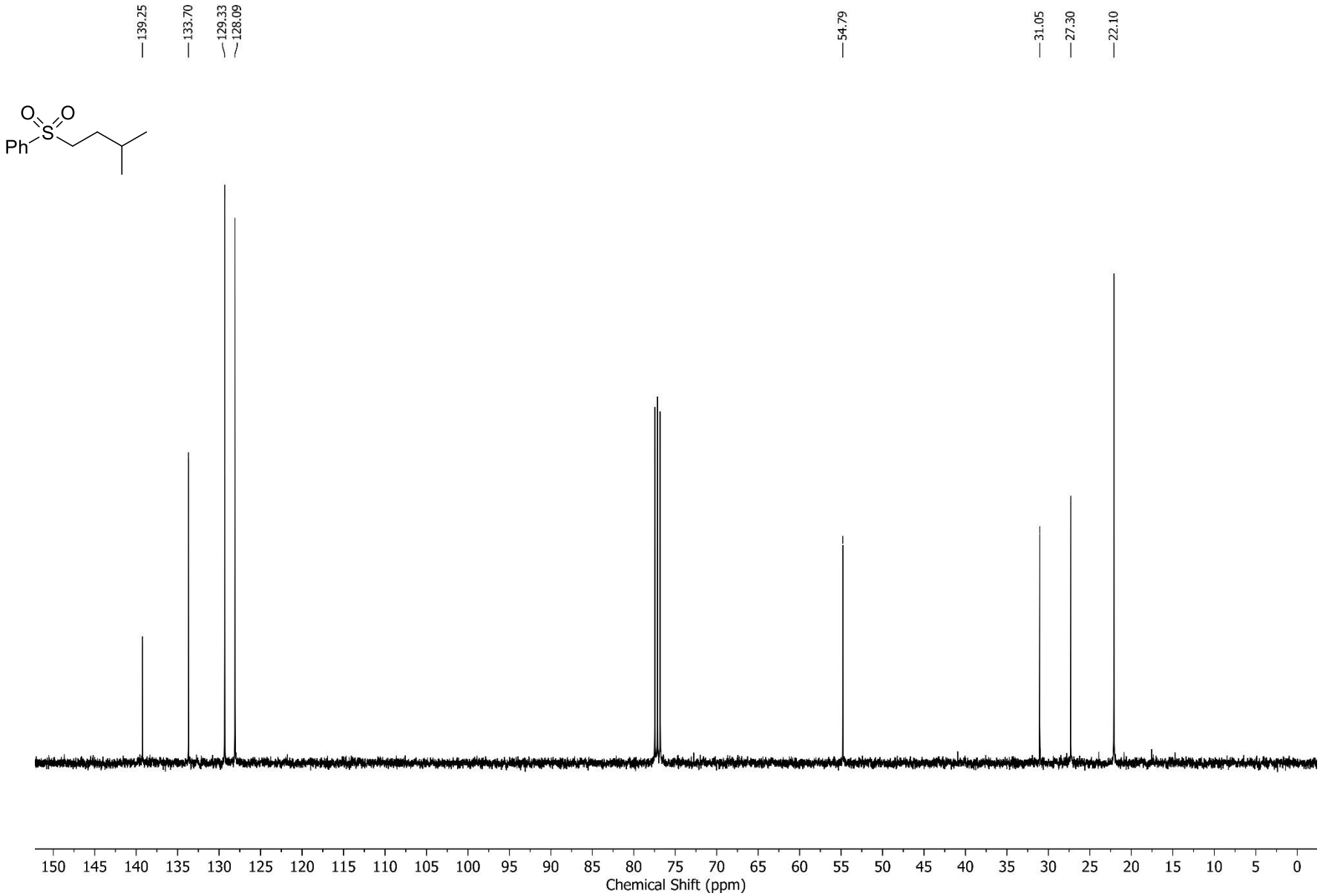


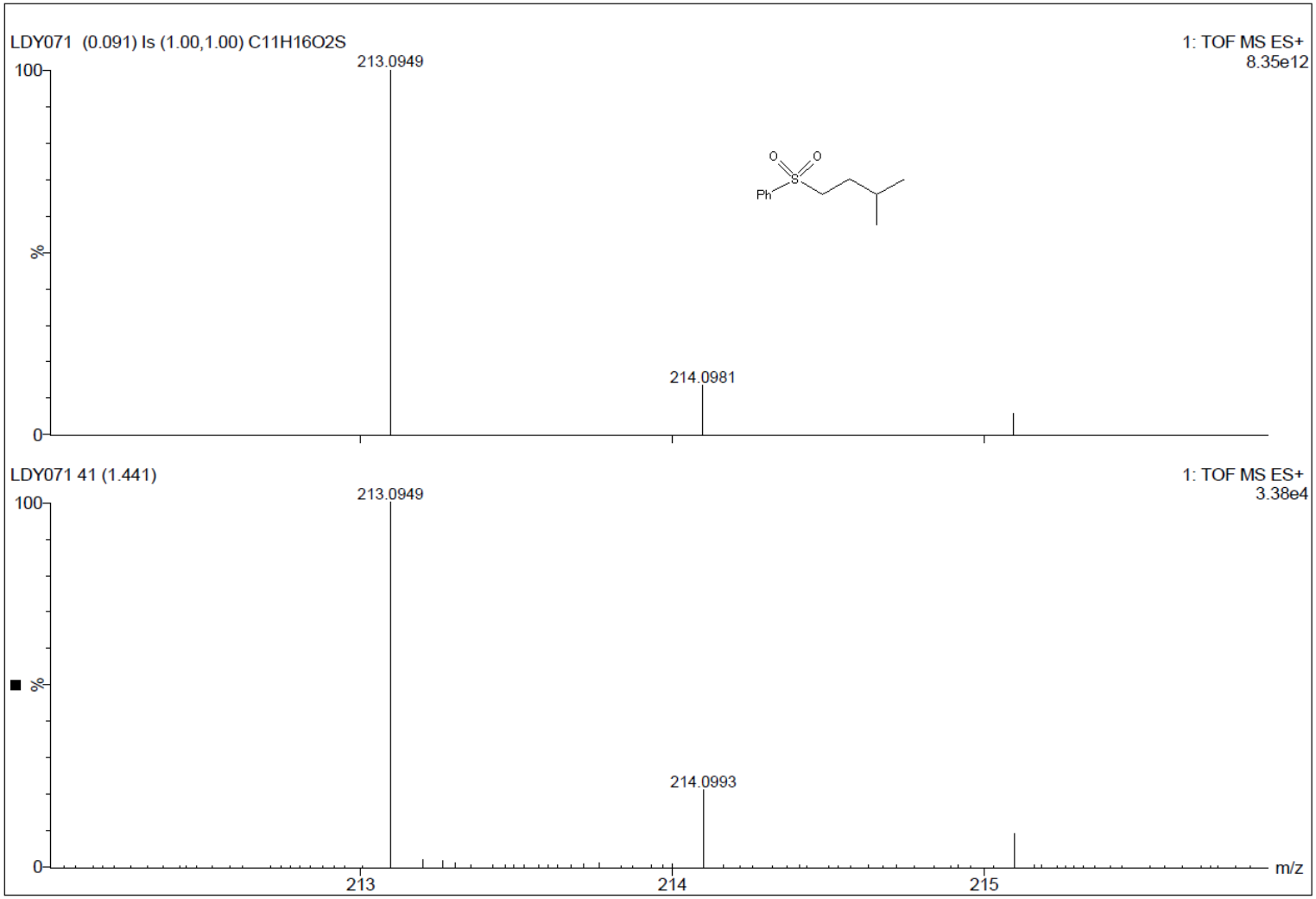

```
Current Data Parameters
NAME      ldy diami carbon
EXPNO                   11
PROCNO                   1

F2 - Acquisition Parameters
Date_              20190620
Time                 21.08
INSTRUM              av400
PROBHD   5 mm QNP 1H/15
PULPROG            zgpg30
TD                   52882
SOLVENT              CDCl3
NS                      64
DS                       0
SWH              24038.461 H
FIDRES           0.454568 H
AQ               1.0999455 s
RG                     256
DW                  20.800 u
DE                   6.38 u
TE                 298.0 K
D1               2.00000000 s
D11              0.03000000 s
TD0                     64

======== CHANNEL f1 ======
SFO1            100.6228298 M
NUC1                    13C
P1                     9.00 u
PLW1           65.29000092 W

======== CHANNEL f2 ======
SFO2            400.1316005 M
NUC2                    1H
CPDPRG[2            waltz16
PCPD2                 90.00 u
PLW2           13.81499958 W
PLW12           0.28823999 W
PLW13           0.23346999 W

F2 - Processing parameters
SI                      65536
SF              100.6127626 M
WDW                        EM
SSB       0
LB                     1.00 H
GB        0
PC                     1.40
```

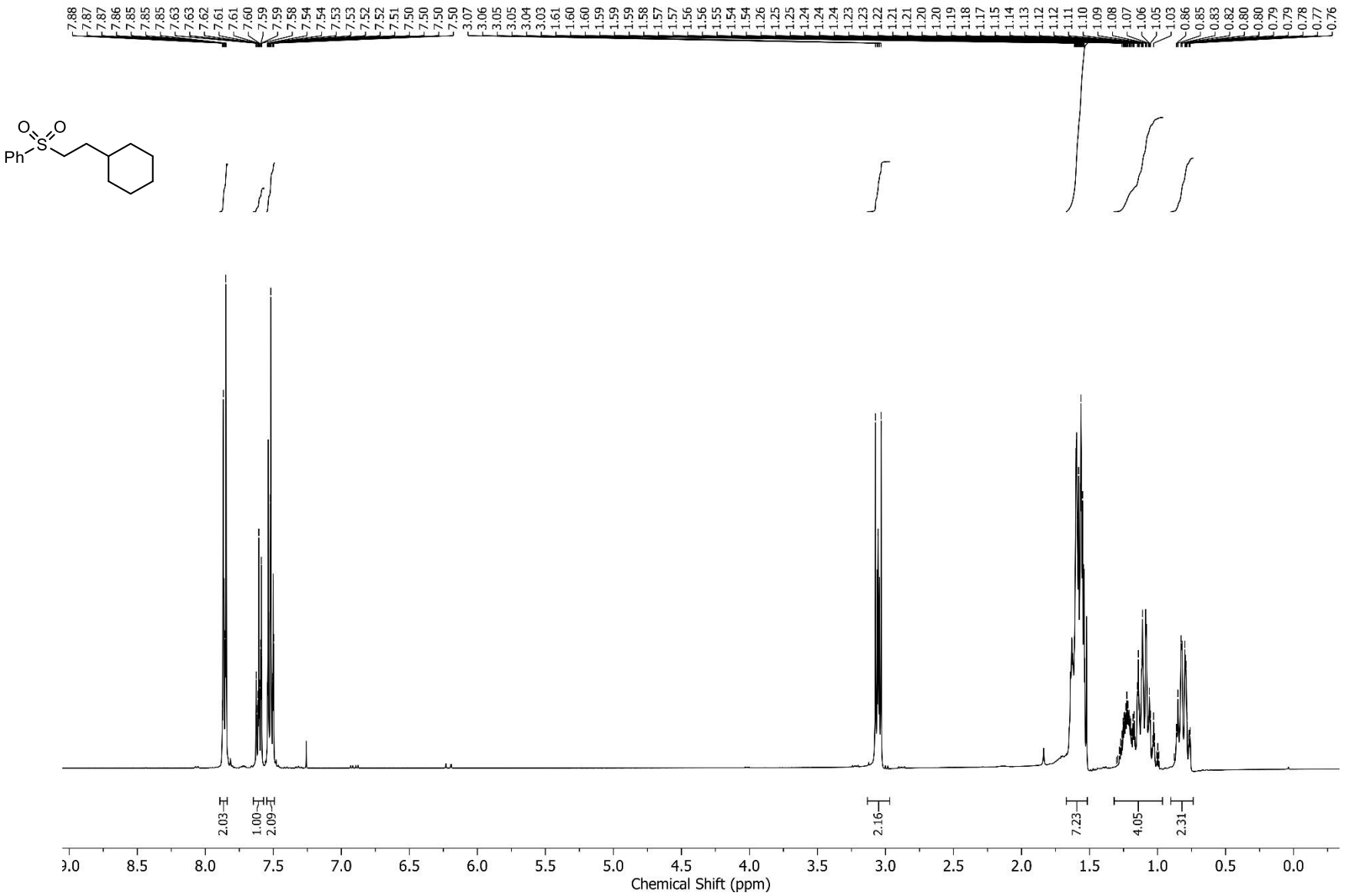


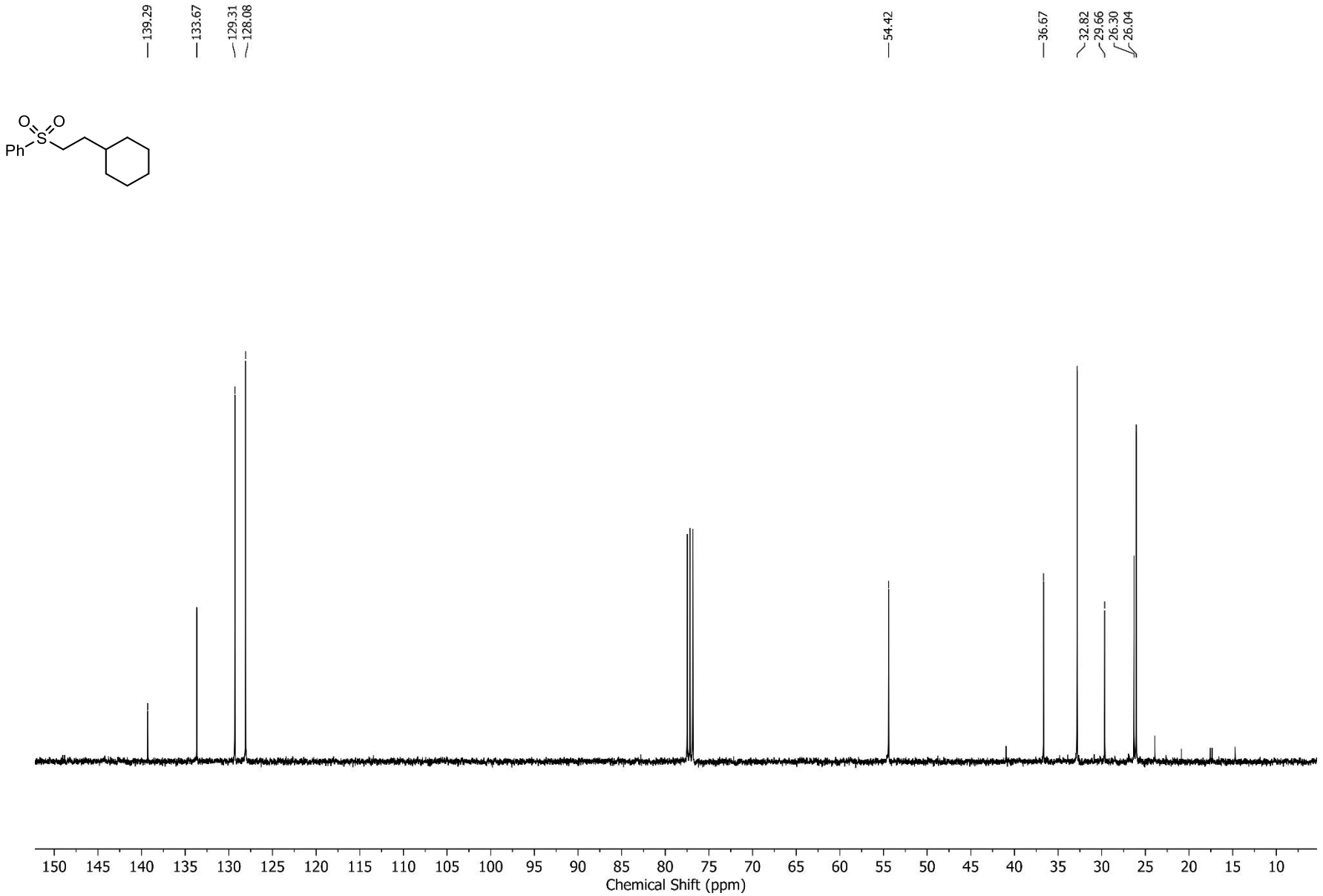
Agilent LC system connected to Agilent 6510 Q TOF mass spectrometer

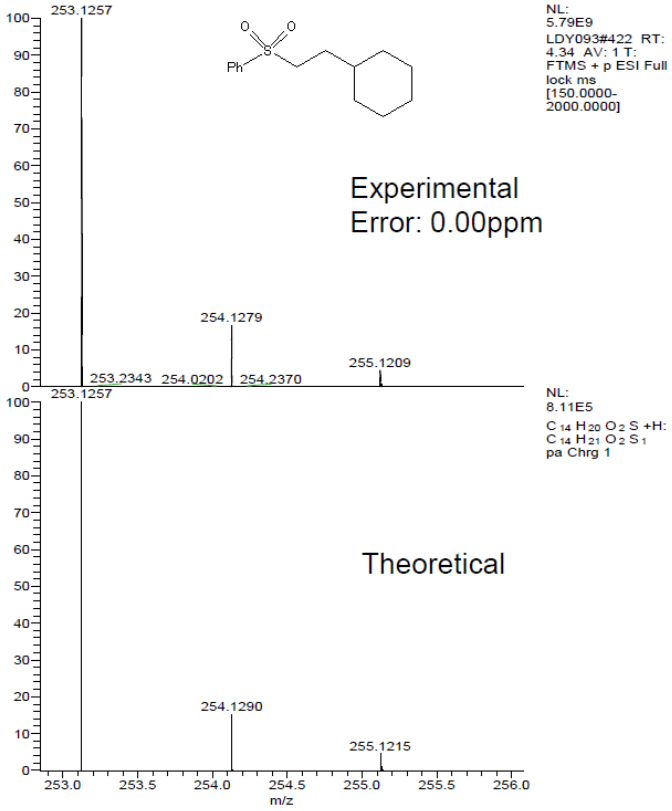

7.91 7.91 7.91 7.91 7.90 7.90 7.90 7.89 7.67 7.67 7.67 7.66 7.66 7.65 7.65 7.64 7.64 7.59 7.58 7.58 7.57 7.57 7.57 7.56 7.56 7.55
3.09 3.08 3.08 3.08 3.08 3.07 3.07 3.07 3.07 3.06 3.06
1.63 1.63 1.62 1.61 1.61 1.61 1.61 1.60 1.60 1.60 1.60 1.60 1.59 1.59 1.58 1.58 1.57 0.87 0.86
O
O
S
Ph
2.33
1.20
2.44
2.18
4.19
6.00
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
Chemical Shift (ppm)

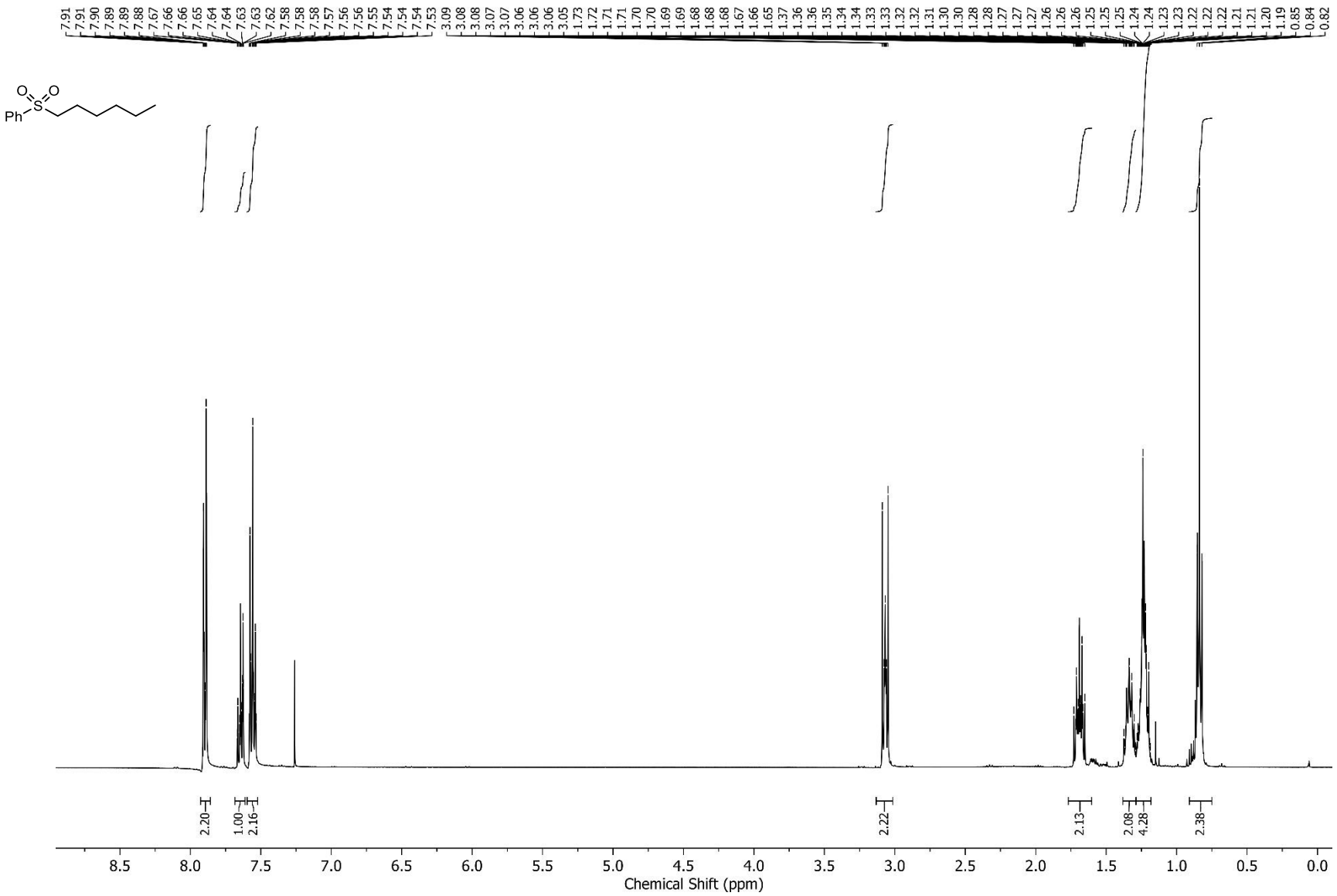

139.25
133.70
129.33
128.09
54.79
31.05
27.30
22.10
O
O
S
Ph
150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
Chemical Shift (ppm)

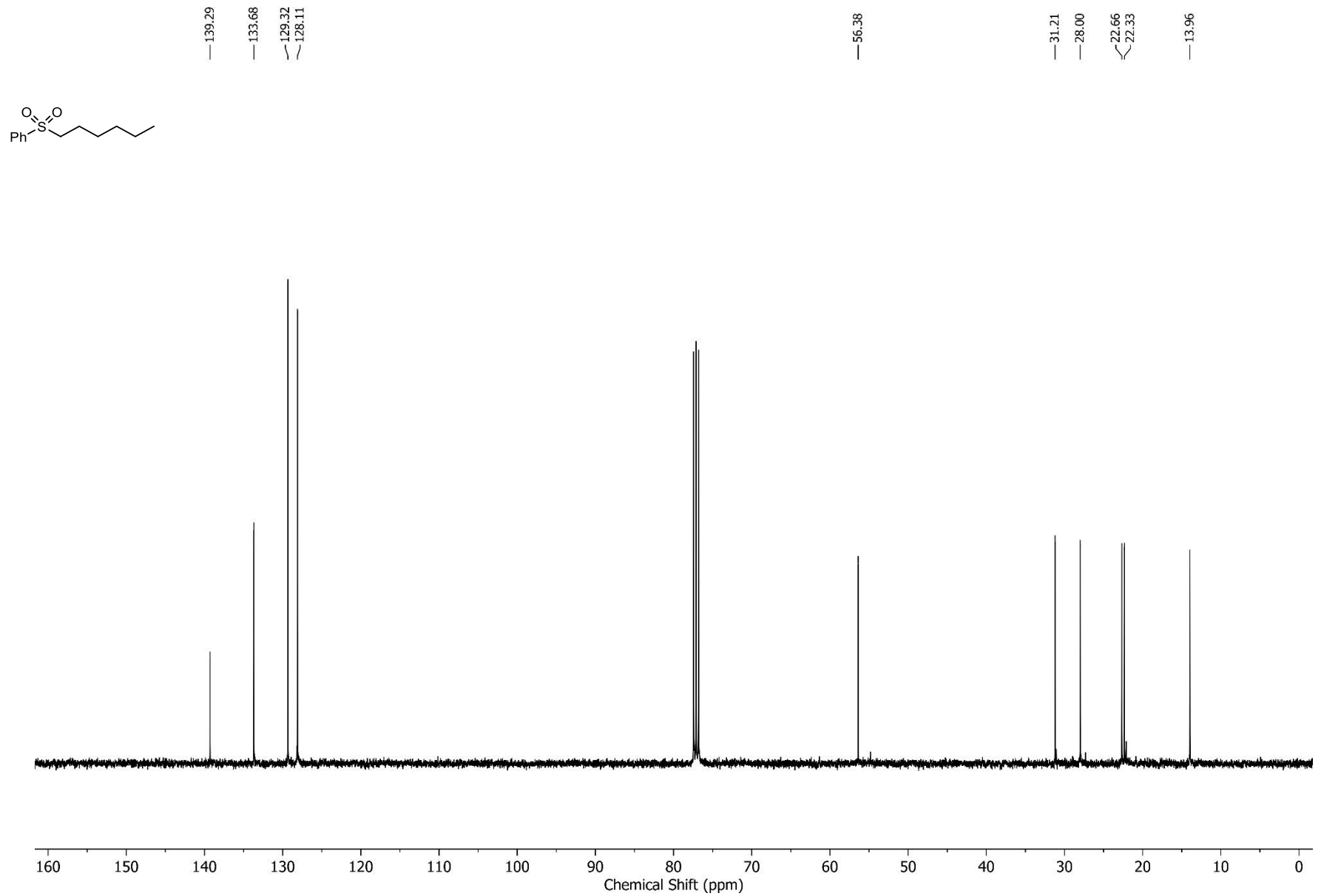

LDY071 (0.091) Is (1.00,1.00) C11H16O2S
1: TOF MS ES+
8.35e12
213.0949
100
%
0
214.0981
O
O
S
Ph
LDY071 41 (1.441)
1: TOF MS ES+
3.38e4
213.0949
100
%
0
214.0993
213
214
215
m/z

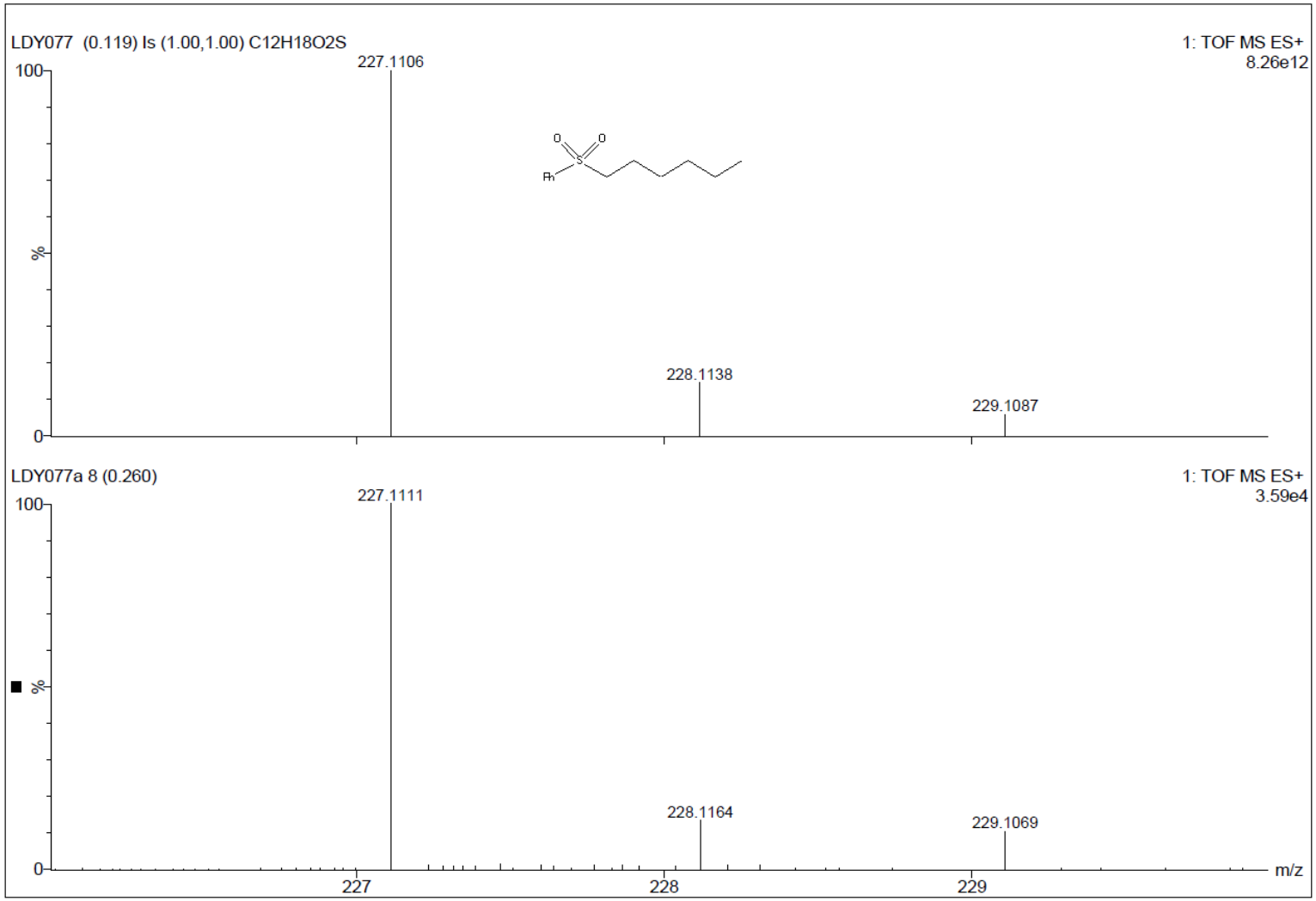

7.88 7.87 7.87 7.86 7.85 7.85 7.85 7.63 7.63 7.62 7.61 7.61 7.60 7.59 7.59 7.58 7.54 7.54 7.53 7.53 7.52 7.52 7.51 7.50 7.50 7.50 7.50
3.07 3.06 3.05 3.05 3.04 3.03 1.61 1.60 1.60 1.59 1.59 1.59 1.58 1.57 1.57 1.56 1.56 1.55 1.54 1.54 1.26 1.25 1.25 1.24 1.24 1.24 1.23 1.23 1.22 1.21 1.21 1.20 1.20 1.19 1.18 1.17 1.15 1.14 1.13 1.12 1.12 1.11 1.10 1.09 1.08 1.07 1.06 1.05 1.03 0.86 0.85 0.83 0.82 0.80 0.80 0.79 0.79 0.78 0.77 0.76
O
S
O
Ph
2.03
1.00
2.09
2.16
7.23
4.05
2.31
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
Chemical Shift (ppm)

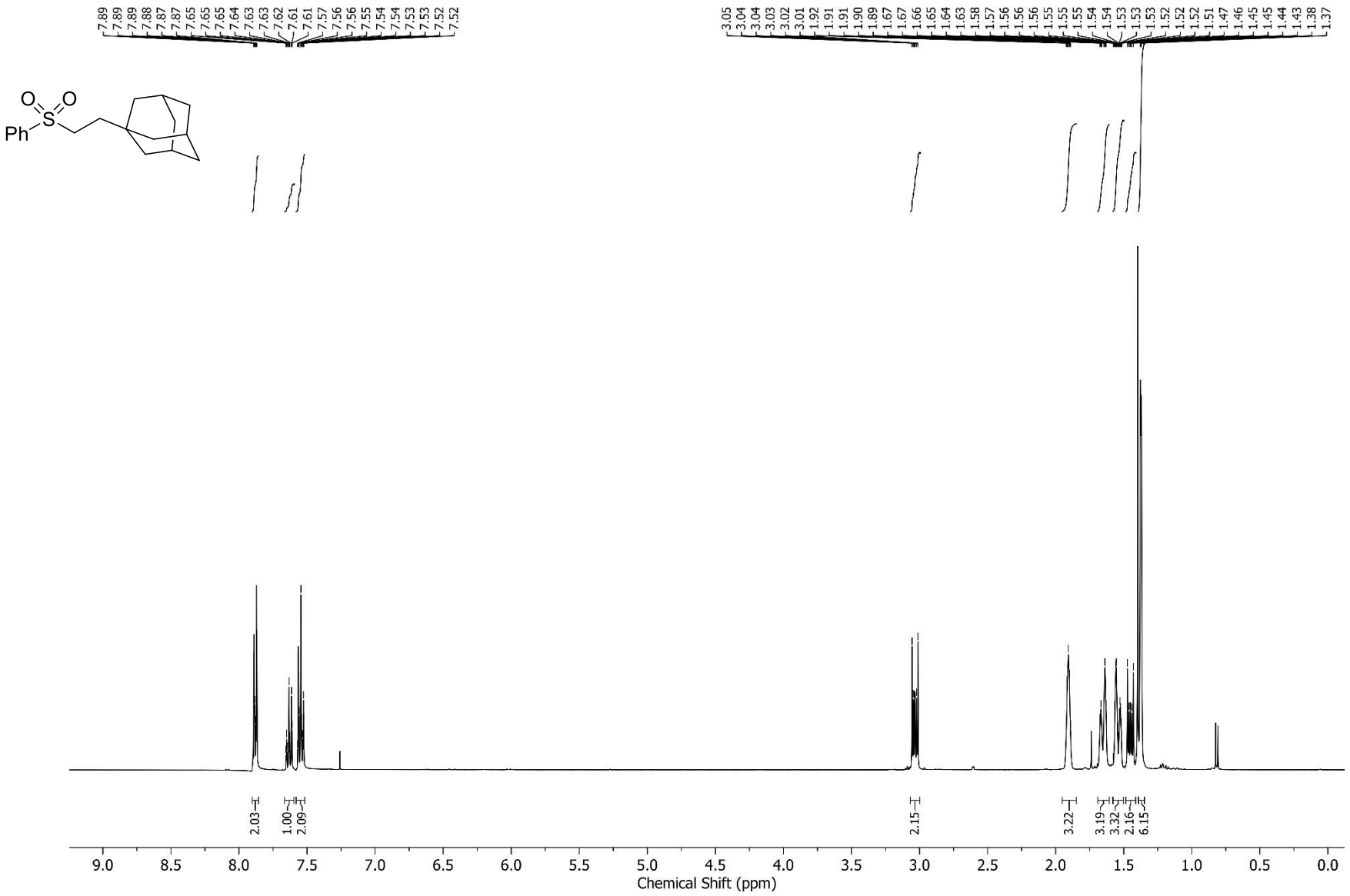

139.29
133.67
129.31
128.08
54.42
36.67
32.82
29.66
26.30
26.04
O
O
S
Ph
150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10
Chemical Shift (ppm)

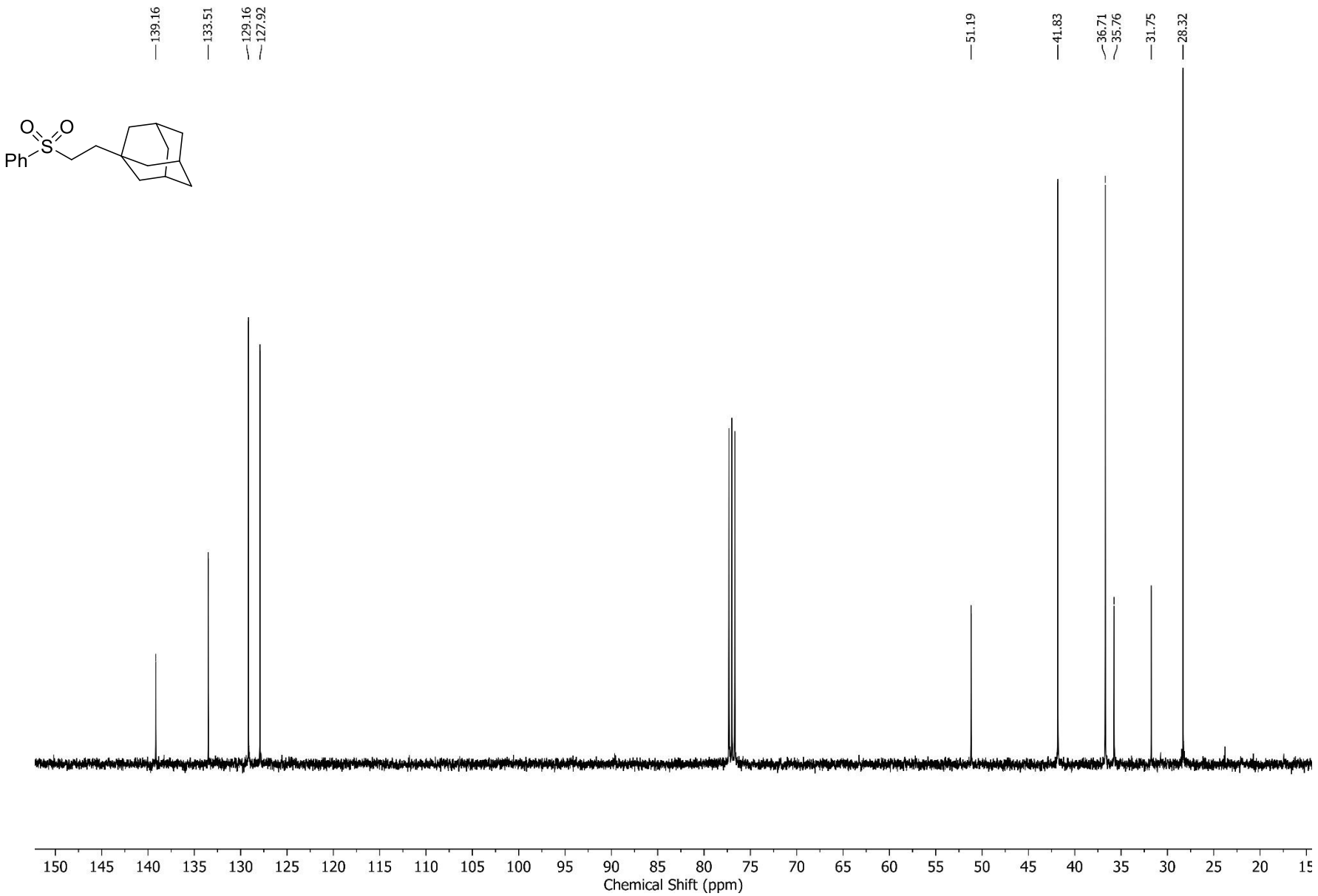

253.1257
NL:
5.79E9
LDY093#422 RT:
4.34 AV: 1 T:
FTMS + p ESI Full
lock ms
[150.0000-
2000.0000]
Ph
Experimental
Error: 0.00ppm
254.1279
255.1209
253.2343
254.0202
254.2370
253.1257
NL:
8.11E5
C 14 H 20 O 2 S +H:
C 14 H 21 O 2 S 1
pa Chrg 1
Theoretical
254.1290
255.1215
m/z

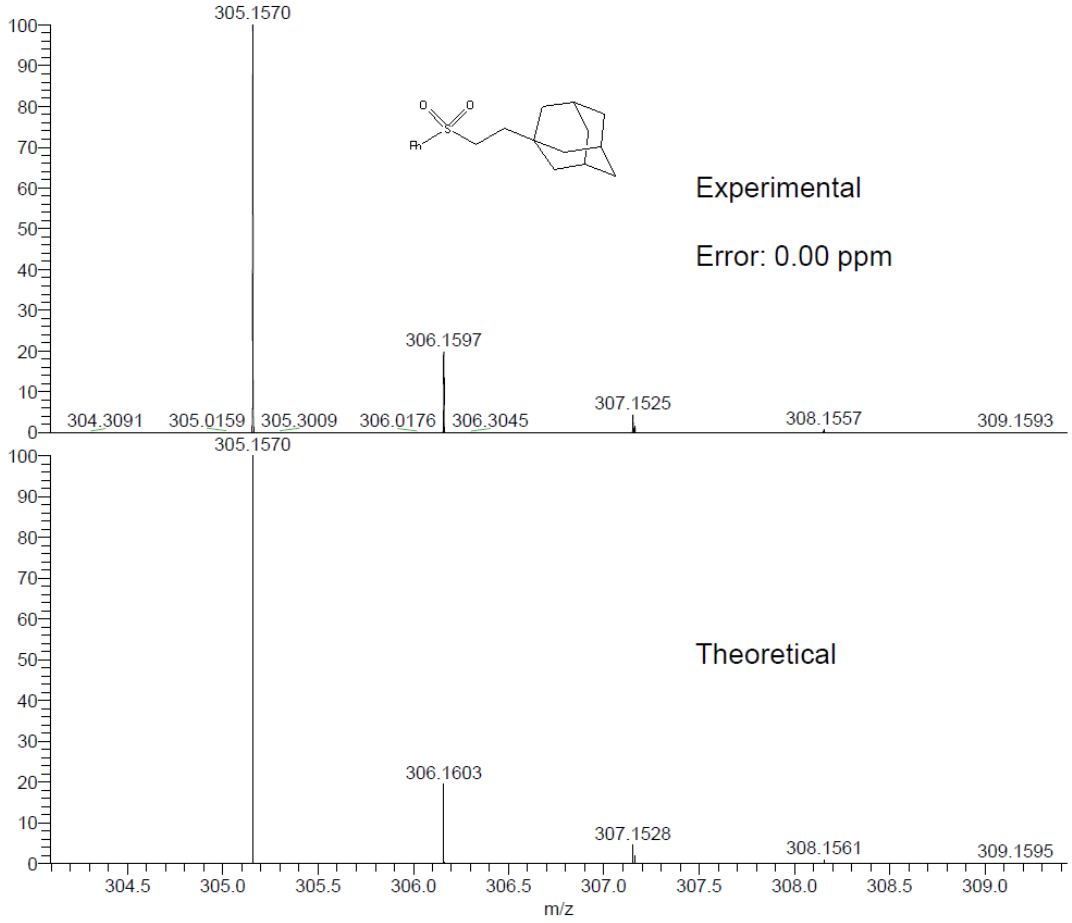

7.91 7.91 7.90 7.89 7.89 7.88 7.67 7.66 7.66 7.65 7.64 7.64 7.63 7.63 7.62 7.58 7.58 7.58 7.57 7.56 7.56 7.55 7.54 7.54 7.54 7.53
3.09 3.08 3.08 3.07 3.07 3.06 3.06 3.06 3.05 1.73 1.72 1.71 1.71 1.70 1.70 1.69 1.69 1.68 1.68 1.68 1.67 1.66 1.65 1.37 1.36 1.36 1.35 1.34 1.34 1.33 1.33 1.32 1.32 1.31 1.30 1.30 1.28 1.28 1.27 1.27 1.27 1.26 1.26 1.26 1.25 1.25 1.25 1.24 1.24 1.23 1.23 1.22 1.22 1.22 1.21 1.21 1.20 1.19 0.85 0.84 0.82
O
O
S
Ph
2.20
1.00
2.16
2.22
2.13
2.08
4.28
2.38
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
Chemical Shift (ppm)

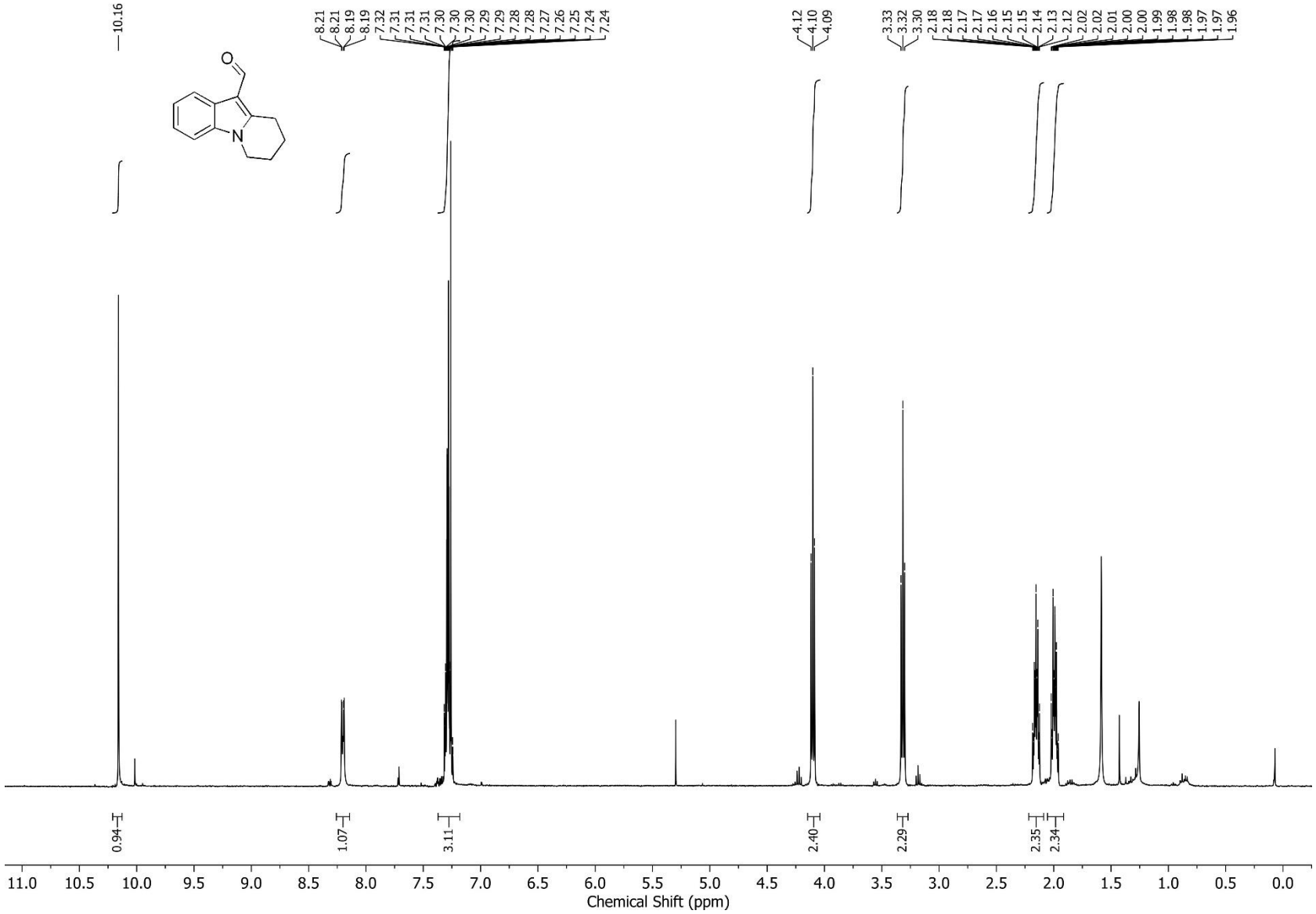

139.29
133.68
129.32
128.11
56.38
31.21
28.00
22.66
22.33
13.96
O
S
O
Ph
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)

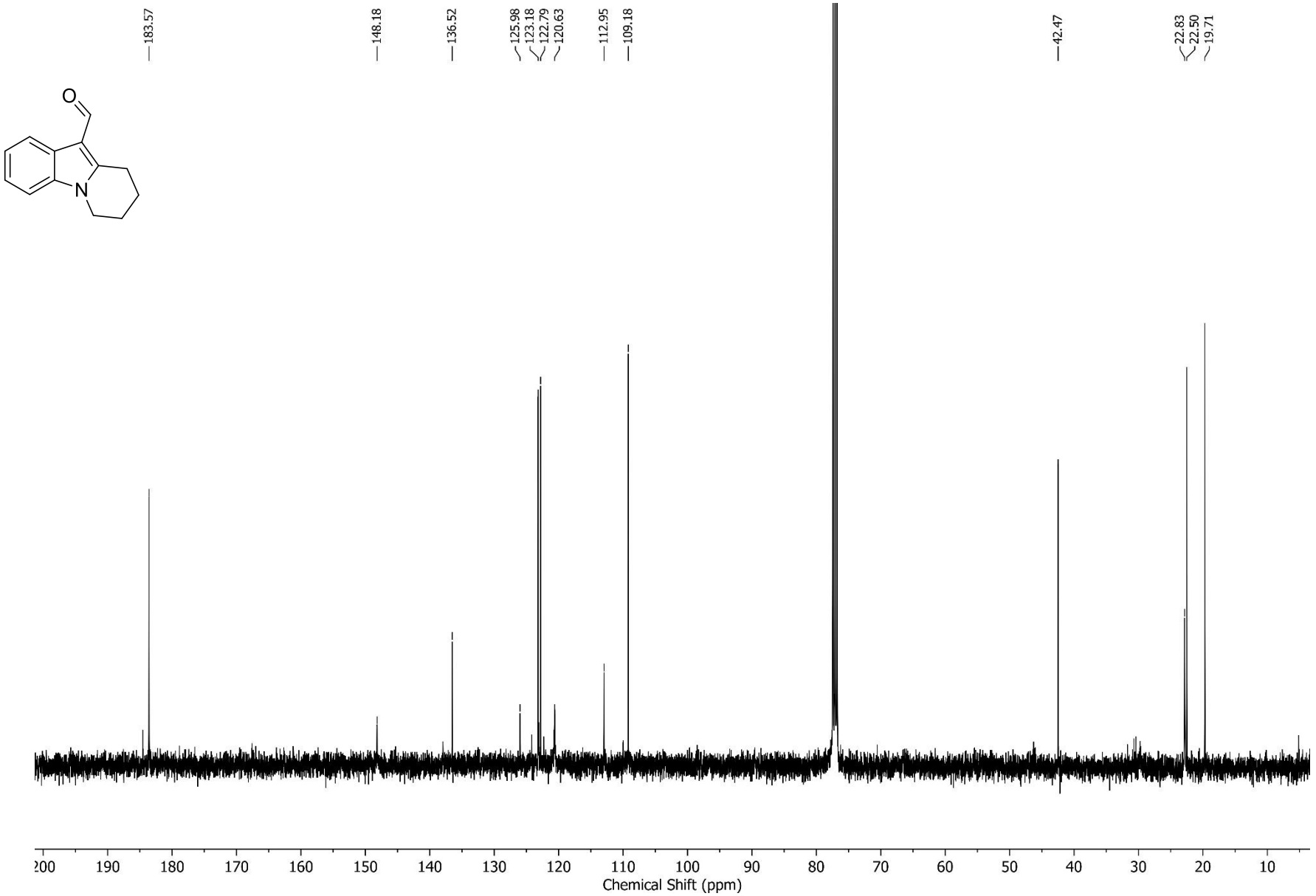

LDY077 (0.119) Is (1.00,1.00) C12H18O2S
1: TOF MS ES+
8.26e12
100
%
0
227.1106
228.1138
229.1087
LDY077a 8 (0.260)
1: TOF MS ES+
3.59e4
100
%
0
227.1111
228.1164
229.1069
227
228
229
m/z

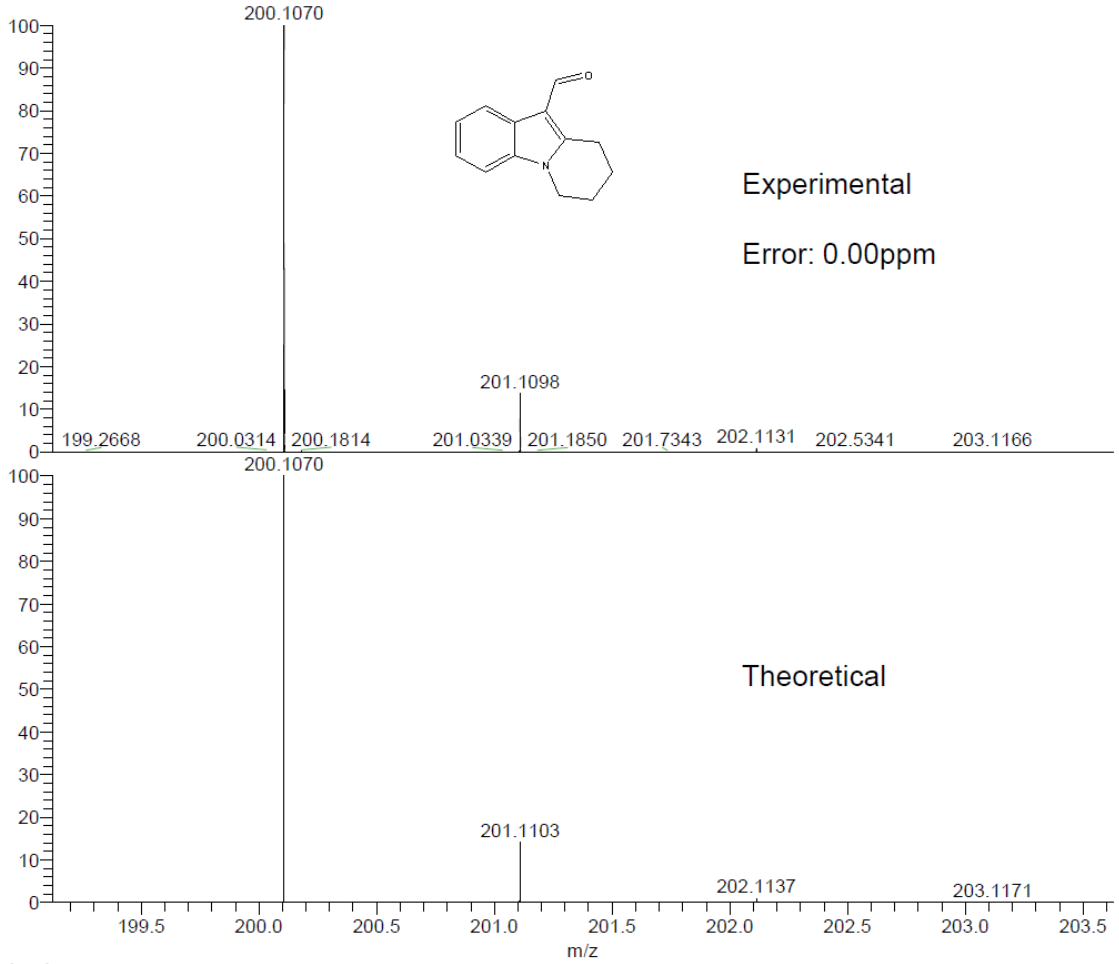

7.89 7.89 7.89 7.88 7.87 7.87 7.65 7.65 7.64 7.63 7.63 7.62 7.61 7.61 7.57 7.56 7.56 7.55 7.54 7.54 7.53 7.52 7.52
3.05 3.04 3.04 3.03 3.02 3.01 1.92 1.91 1.91 1.90 1.89 1.67 1.67 1.66 1.65 1.64 1.63 1.58 1.57 1.56 1.56 1.56 1.55 1.55 1.55 1.54 1.54 1.53 1.53 1.53 1.52 1.52 1.52 1.51 1.47 1.46 1.45 1.45 1.44 1.43 1.38 1.37
O
O
S
Ph
2.03
1.00
2.09
2.15
3.22
3.19
3.32
2.16
6.15
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
Chemical Shift (ppm)

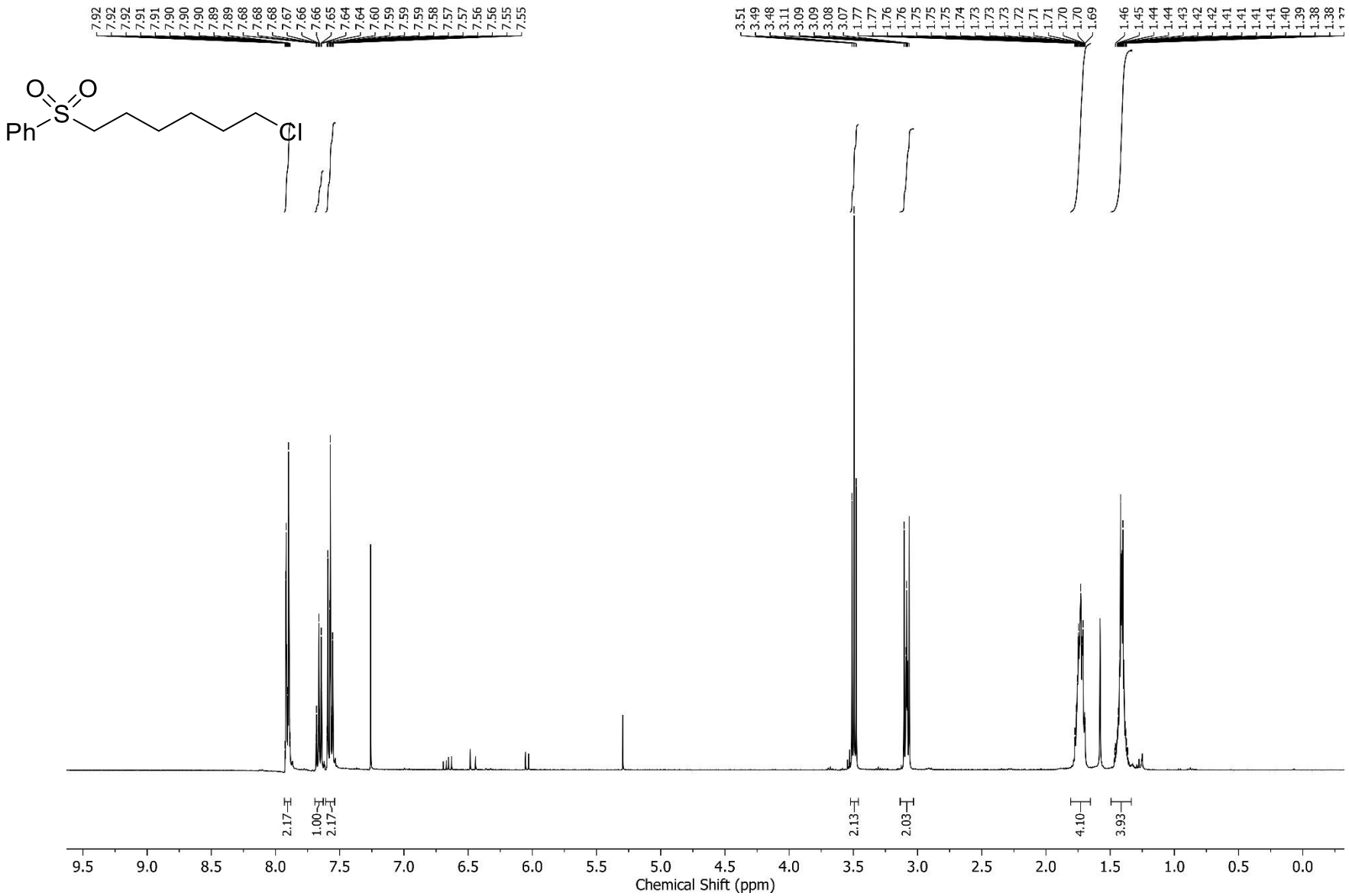

139.16
133.51
129.16
127.92
51.19
41.83
36.71
35.76
31.75
28.32
O
O
S
Ph
150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15
Chemical Shift (ppm)

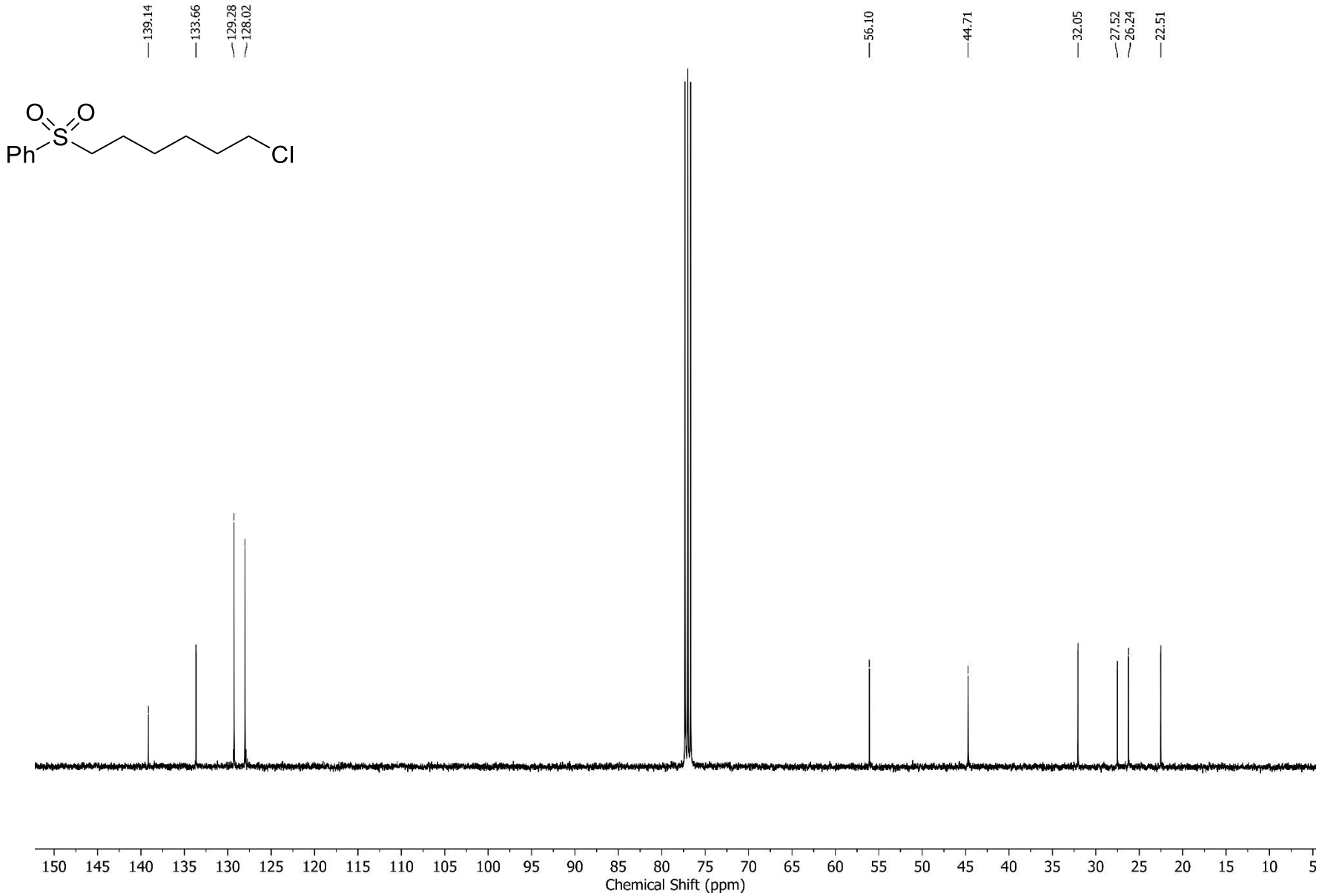

305.1570
NL:
6.94E8
LDY100#466 RT:
4.77 AV: 1 T:
FTMS + p ESI Full
lock ms
[150.0000-
2000.0000]
Experimental
Error: 0.00 ppm
306.1597
304.3091
305.0159
305.3009
306.0176
306.3045
307.1525
308.1557
309.1593
305.1570
NL:
7.76E5
C 18 H 24 O 2 S +H:
C 18 H 25 O 2 S 1
pa Chrg 1
Theoretical
306.1603
307.1528
308.1561
309.1595
304.5
305.0
305.5
306.0
306.5
307.0
307.5
308.0
308.5
309.0
m/z

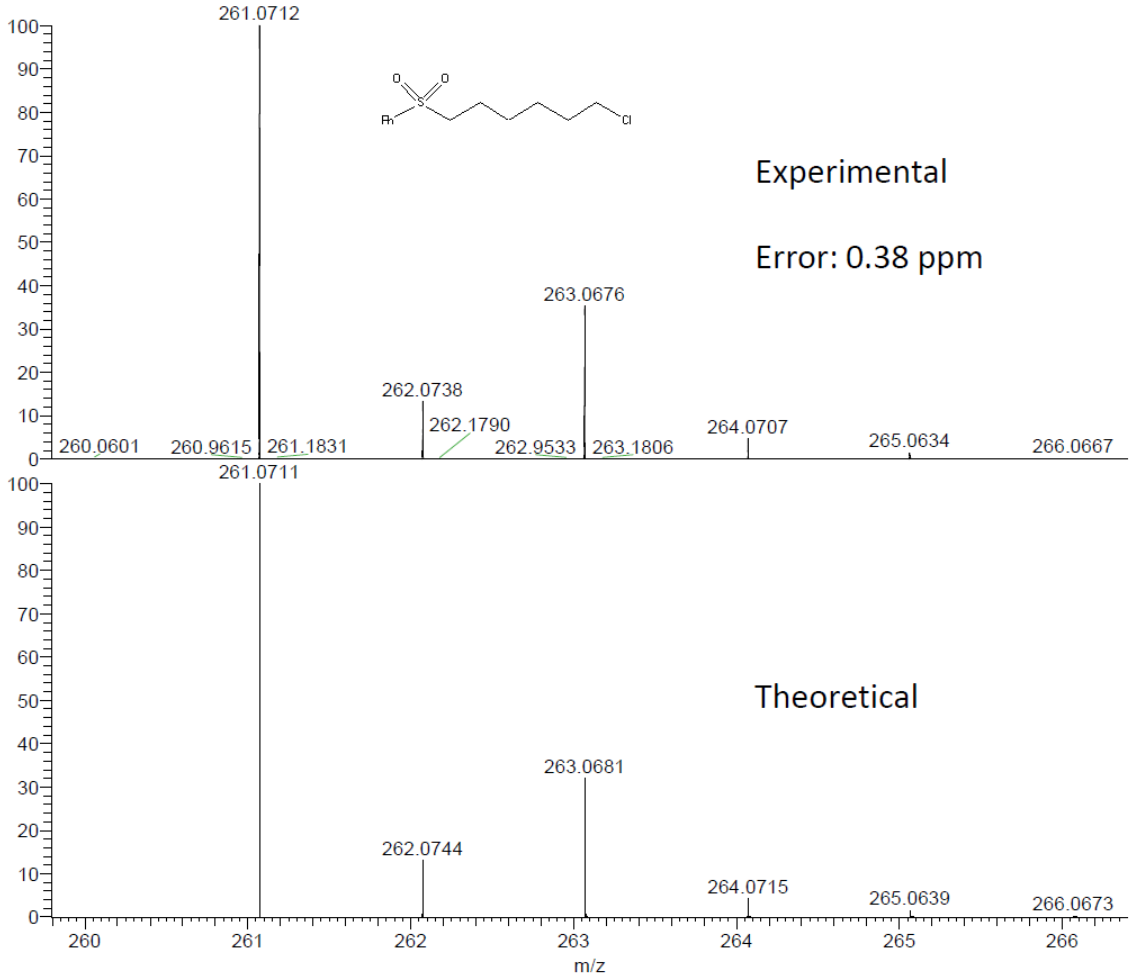

10.16
8.21
8.21
8.19
8.19
7.32
7.31
7.31
7.31
7.30
7.30
7.30
7.29
7.29
7.28
7.28
7.27
7.26
7.25
7.24
7.24
4.12
4.10
4.09
3.33
3.32
3.30
2.18
2.18
2.17
2.17
2.16
2.15
2.15
2.14
2.13
2.12
2.02
2.02
2.01
2.00
2.00
1.99
1.98
1.98
1.97
1.97
1.96
O
N
0.94
1.07
3.11
2.40
2.29
2.35
2.34
11.0
10.5
10.0
9.5
9.0
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
Chemical Shift (ppm)

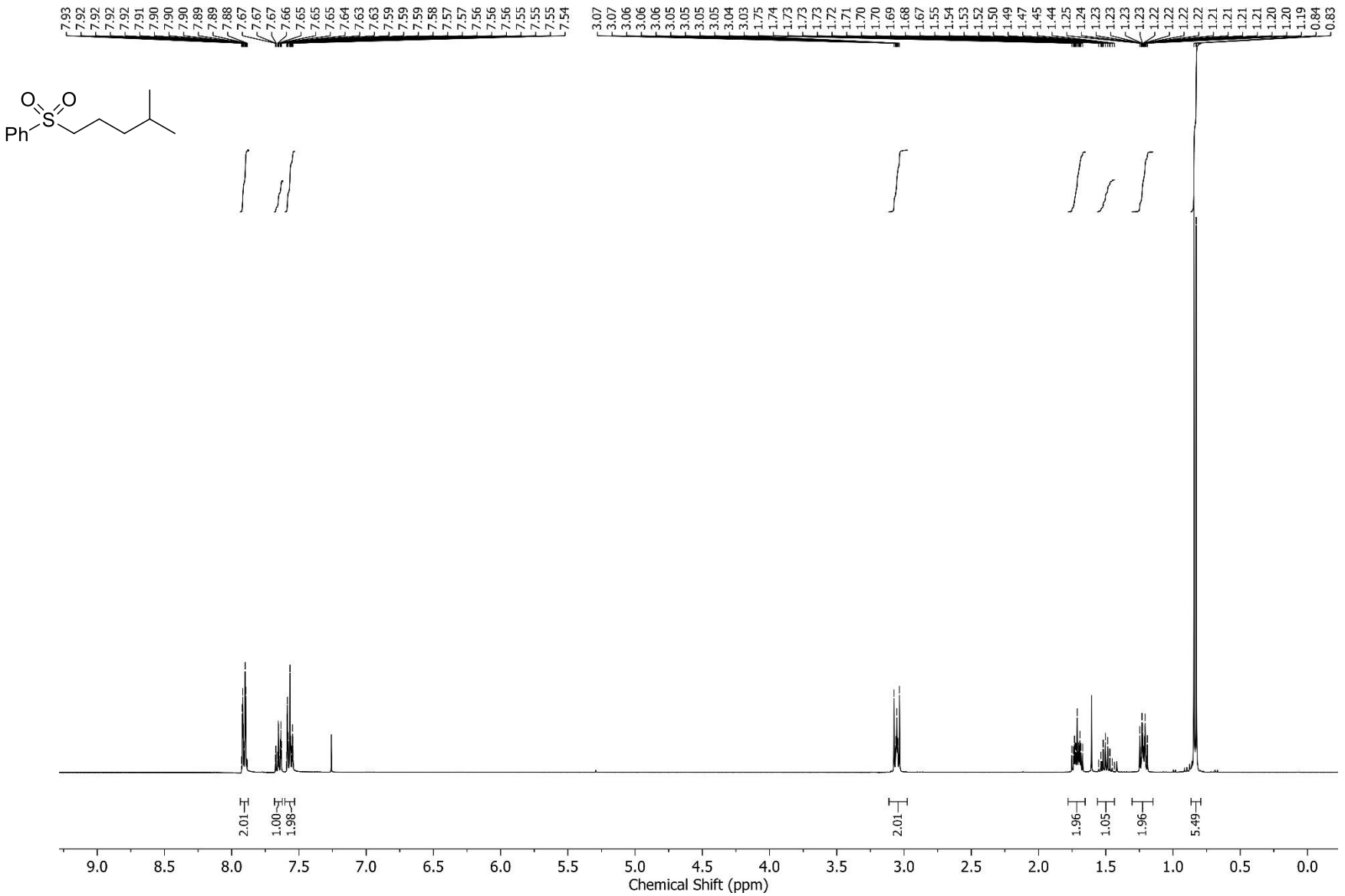

183.57
148.18
136.52
125.98
123.18
122.79
120.63
112.95
109.18
42.47
22.83
22.50
19.71
O
N
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
Chemical Shift (ppm)

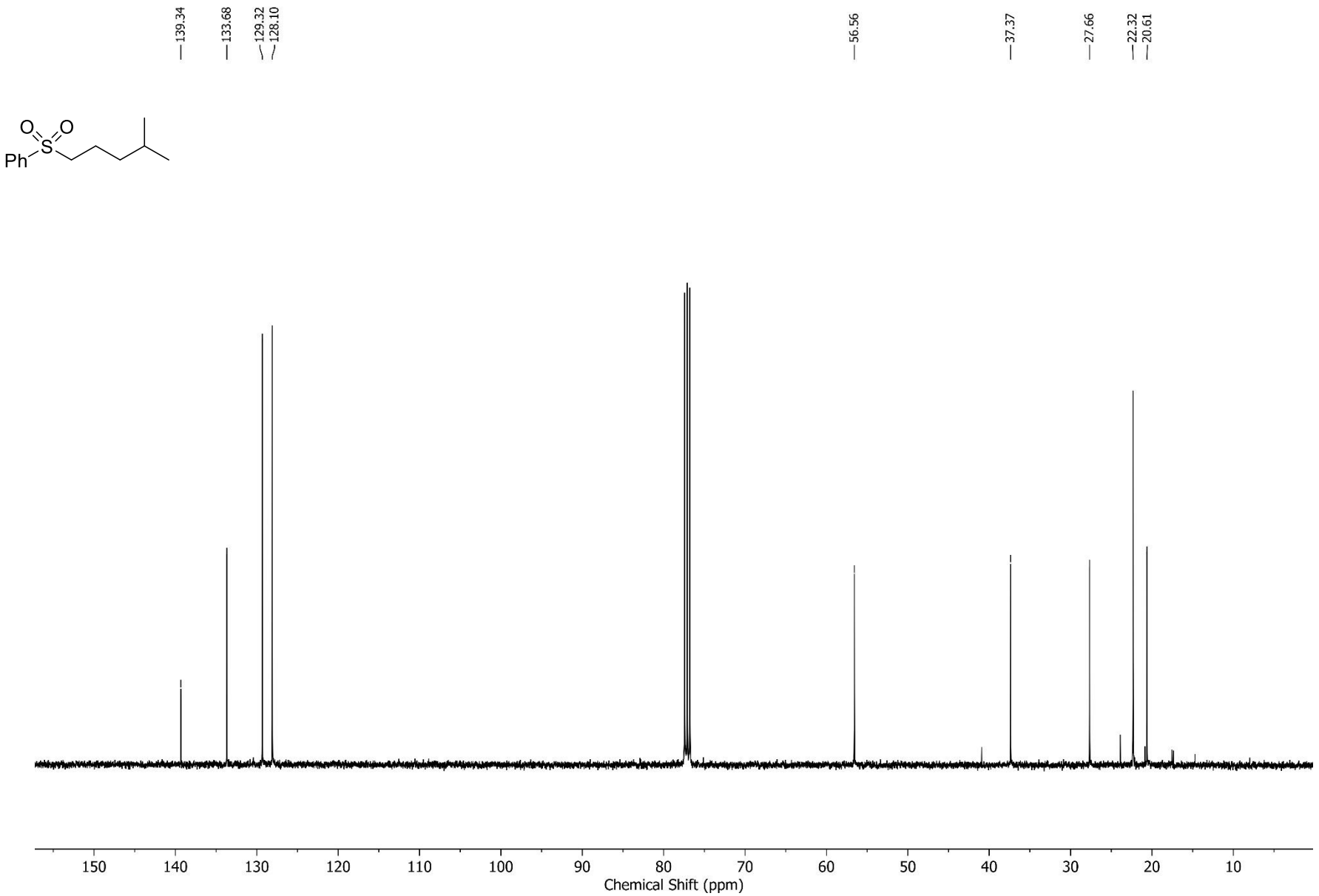

200.1070
NL:
3.48E9
LDY116_#385 RT:
3.77 AV: 1 T: FTMS
+ p ESI Full lock ms
[100.0000-
1500.0000]
Experimental
Error: 0.00ppm
201.1098
199.2668
200.0314
200.1814
201.0339
201.1850
201.7343
202.1131
202.5341
203.1166
200.1070
NL:
8.63E5
C 13 H 13 NO +H:
C 13 H 14 N 1 O 1
pa Chrg 1
Theoretical
201.1103
202.1137
203.1171
m/z

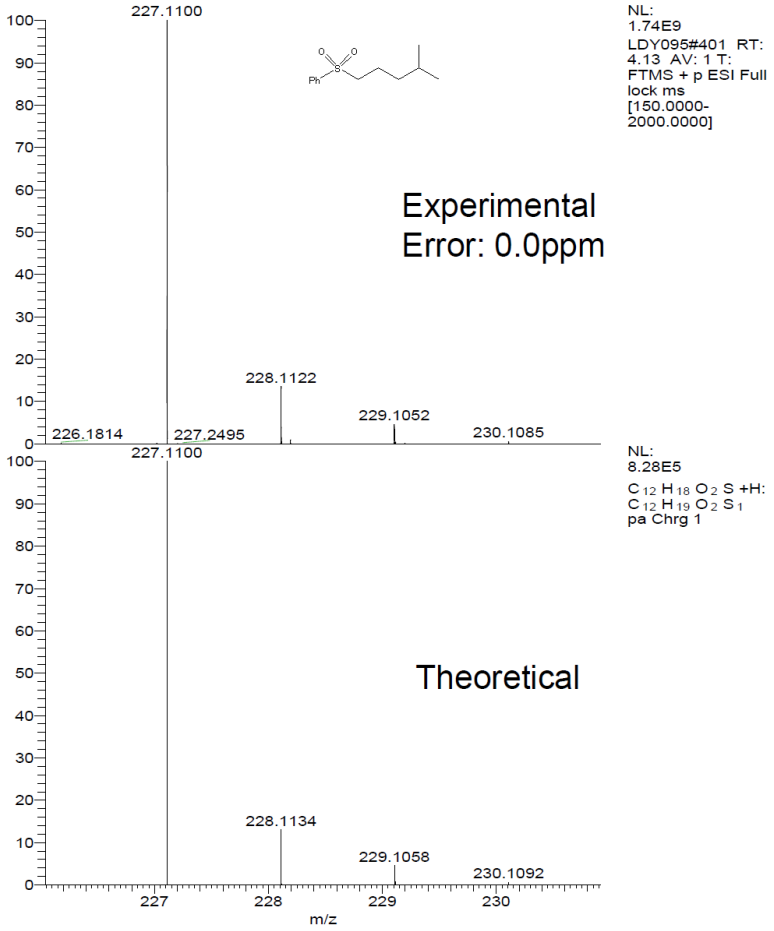

O
O
S
Ph
Cl
7.92 7.92 7.92 7.91 7.91 7.90 7.90 7.90 7.89 7.89 7.68 7.68 7.68 7.67 7.66 7.66 7.65 7.64 7.64 7.60 7.59 7.59 7.59 7.58 7.57 7.57 7.56 7.56 7.55 7.55
3.51 3.49 3.48 3.11 3.09 3.09 3.08 3.07 1.77 1.77 1.76 1.76 1.75 1.75 1.75 1.74 1.73 1.73 1.73 1.72 1.71 1.71 1.70 1.70 1.69
1.46 1.45 1.44 1.44 1.43 1.42 1.42 1.41 1.41 1.41 1.41 1.40 1.39 1.38 1.38
2.17
1.00
2.17
2.13
2.03
4.10
3.93
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
Chemical Shift (ppm)

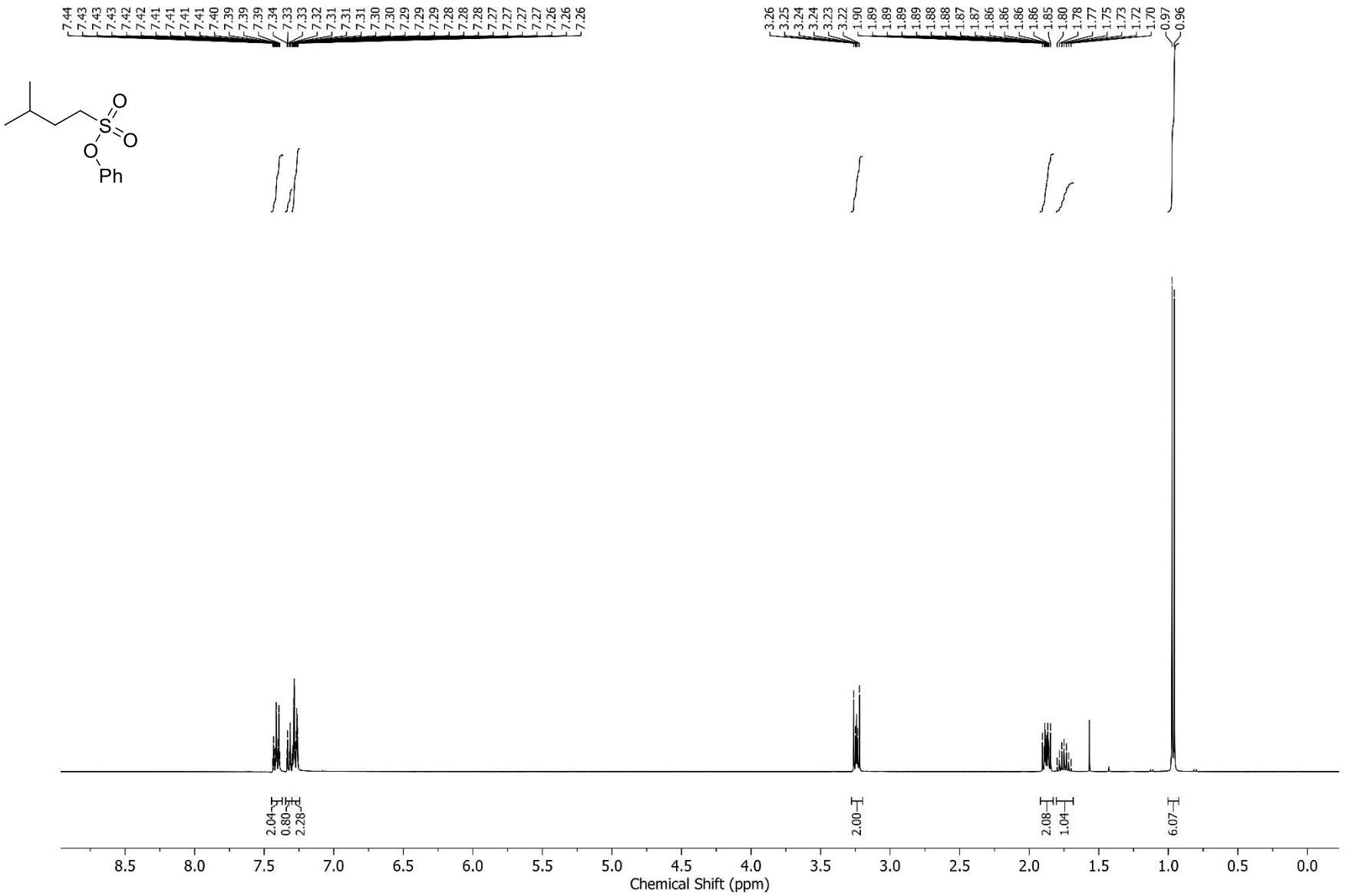

139.14
133.66
129.28
128.02
56.10
44.71
32.05
27.52
26.24
22.51
O
O
S
Ph
Cl
150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5
Chemical Shift (ppm)

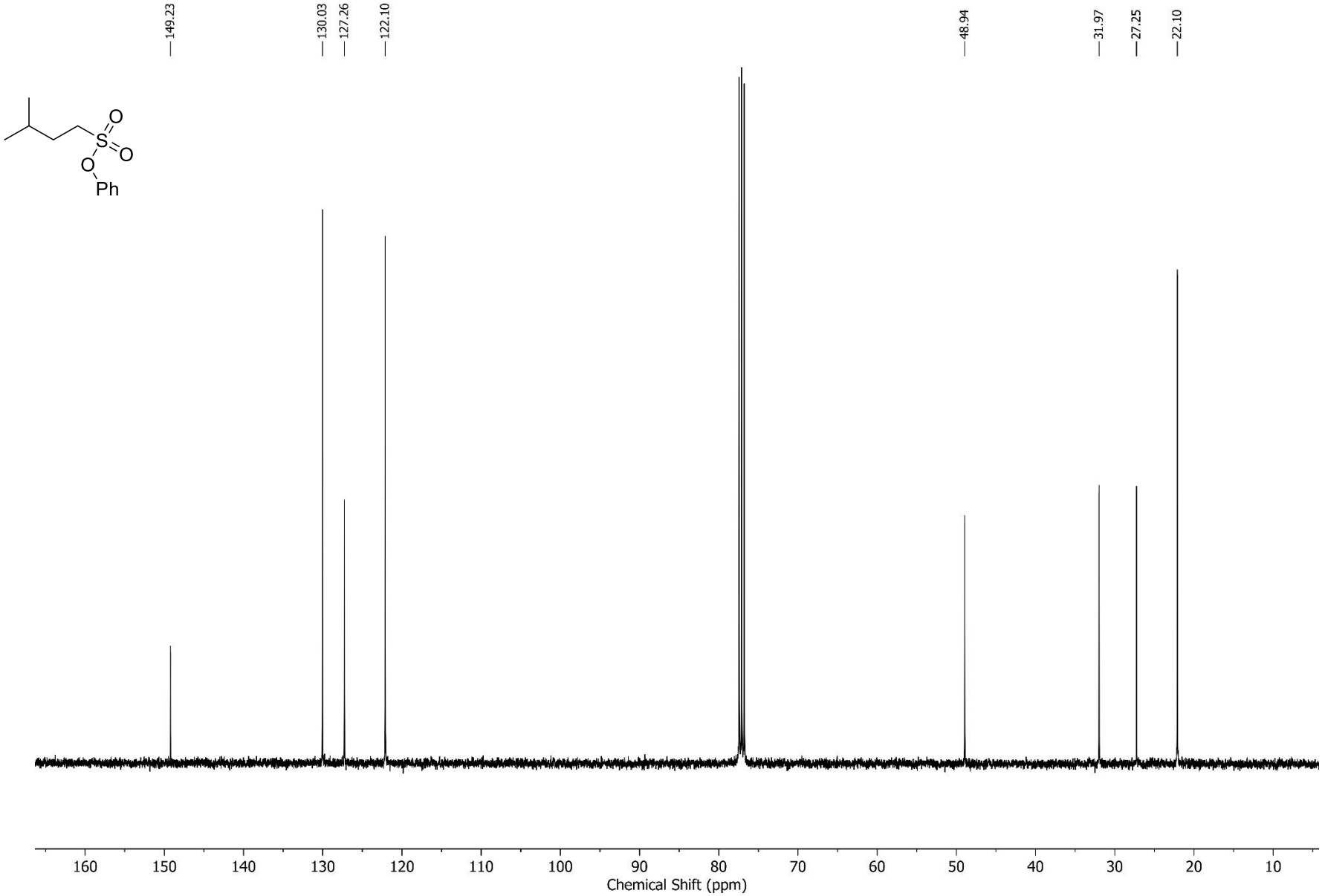

261.0712
263.0676
262.0738
262.1790
260.0601
260.9615
261.1831
262.9533
263.1806
264.0707
265.0634
266.0667
NL:
4.57E8
LDY115#402 RT:
4.07 AV: 1 T: FTMS
+ p ESI Full lock ms
[150.0000-
2000.0000]
Experimental
Error: 0.38 ppm
261.0711
263.0681
262.0744
264.0715
265.0639
266.0673
NL:
6.28E5
C 12 H 17 ClO 2 S +H:
C 12 H 18 Cl 1 O 2 S 1
pa Chrg 1
Theoretical
m/z

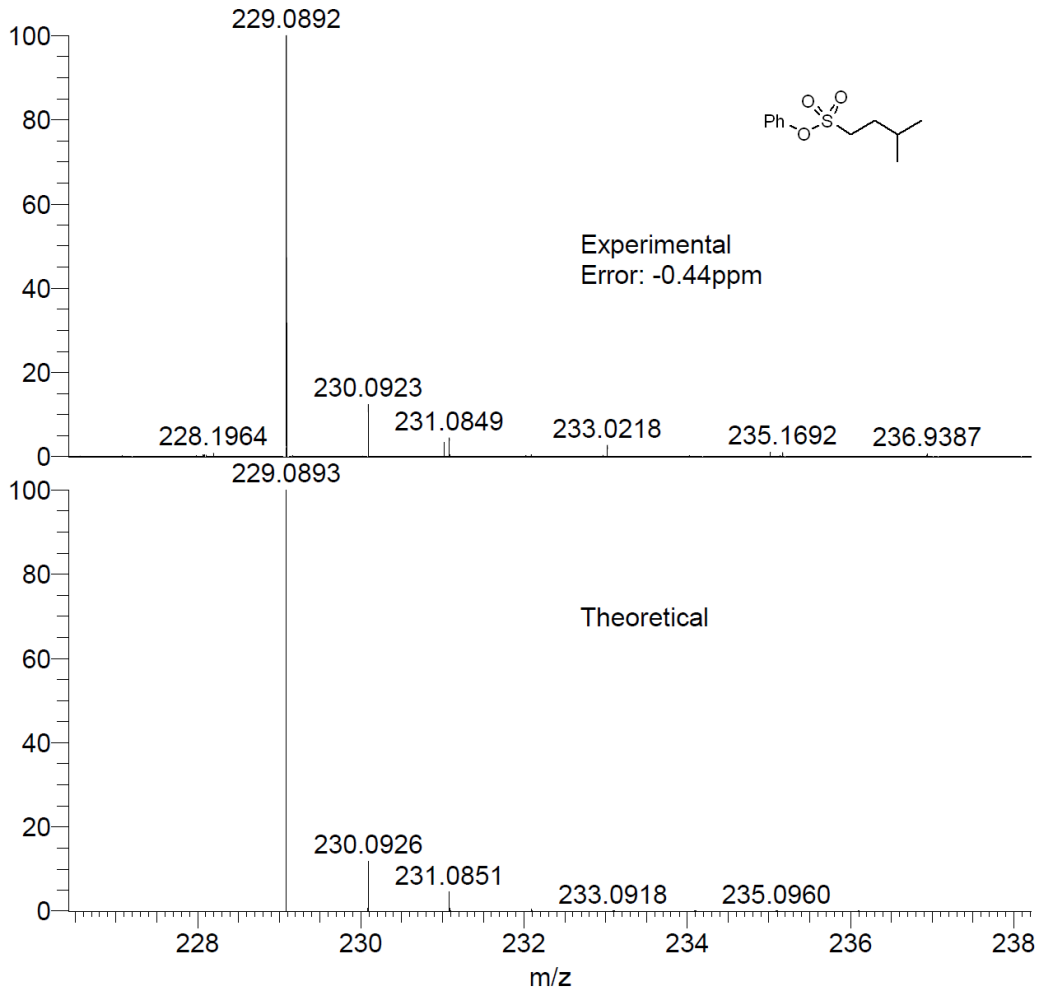

7.93 7.92 7.92 7.92 7.91 7.90 7.90 7.89 7.89 7.88 7.67 7.67 7.66 7.65 7.65 7.64 7.63 7.63 7.59 7.59 7.59 7.58 7.57 7.57 7.56 7.56 7.56 7.55 7.55 7.54
3.07 3.07 3.06 3.06 3.05 3.05 3.05 3.04 3.03 1.75 1.74 1.73 1.73 1.72 1.71 1.70 1.70 1.69 1.68 1.67 1.55 1.54 1.53 1.52 1.50 1.49 1.47 1.45 1.44 1.25 1.24 1.23 1.23 1.23 1.22 1.22 1.22 1.22 1.21 1.21 1.21 1.21 1.20 1.20 1.19 0.84 0.83
O
O
S
Ph
2.01
1.00
1.98
2.01
1.96
1.05
1.96
5.49
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
Chemical Shift (ppm)


139.34
133.68
129.32
128.10
56.56
37.37
27.66
22.32
20.61
O
O
S
Ph
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
Chemical Shift (ppm)


227.1100
NL:
1.74E9
LDY095#401 RT:
4.13 AV: 1 T:
FTMS + p ESI Full
lock ms
[150.0000-
2000.0000]
Experimental
Error: 0.0ppm
228.1122
229.1052
226.1814
227.2495
230.1085
227.1100
NL:
8.28E5
C 12 H 18 O 2 S +H:
C 12 H 19 O 2 S 1
pa Chrg 1
Theoretical
228.1134
229.1058
230.1092
227
228
229
230
m/z

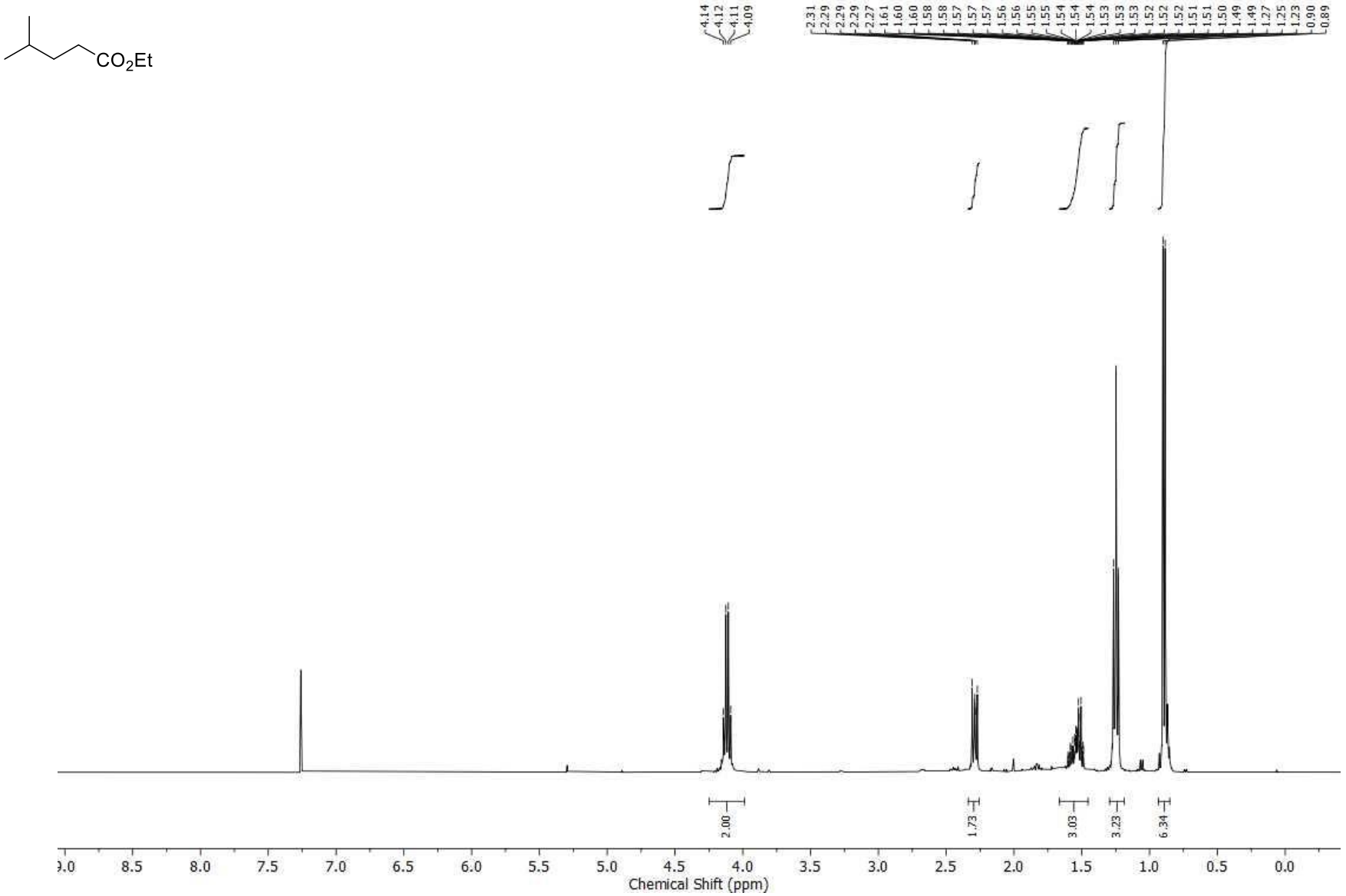

7.44 7.43 7.43 7.43 7.42 7.42 7.41 7.41 7.41 7.41 7.40 7.39 7.39 7.39 7.34 7.33 7.33 7.32 7.31 7.31 7.31 7.30 7.30 7.29 7.29 7.29 7.28 7.28 7.28 7.27 7.27 7.27 7.27 7.26 7.26 7.26
3.26 3.25 3.24 3.24 3.23 3.22 1.90 1.89 1.89 1.89 1.89 1.89 1.88 1.88 1.87 1.87 1.86 1.86 1.86 1.86 1.85 1.80 1.78 1.77 1.75 1.73 1.72 1.70 0.97 0.96
O
S
O
O
Ph
2.04
0.80
2.28
2.00
2.08
1.04
6.07
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
Chemical Shift (ppm)

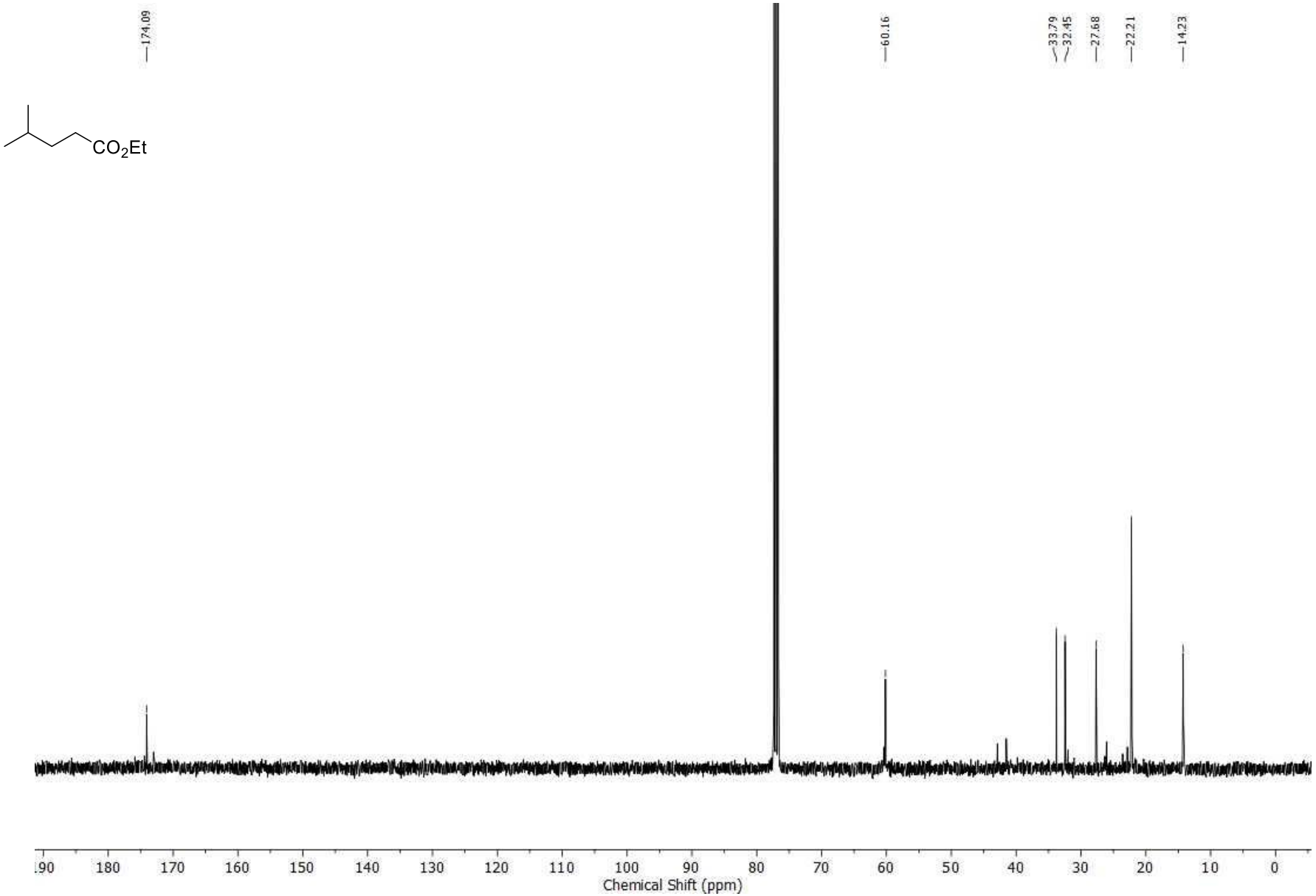

149.23
130.03
127.26
122.10
48.94
31.97
27.25
22.10
O
S
O
O
Ph
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
Chemical Shift (ppm)

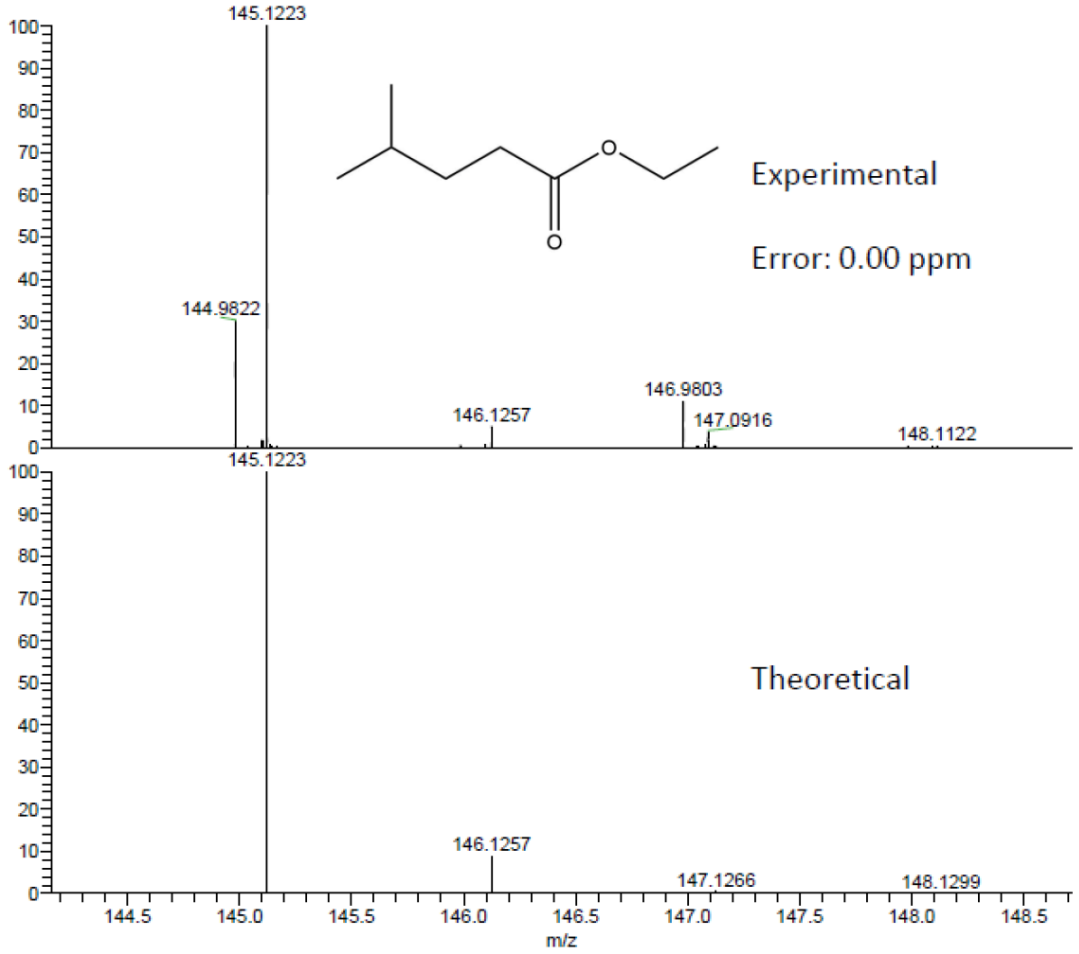


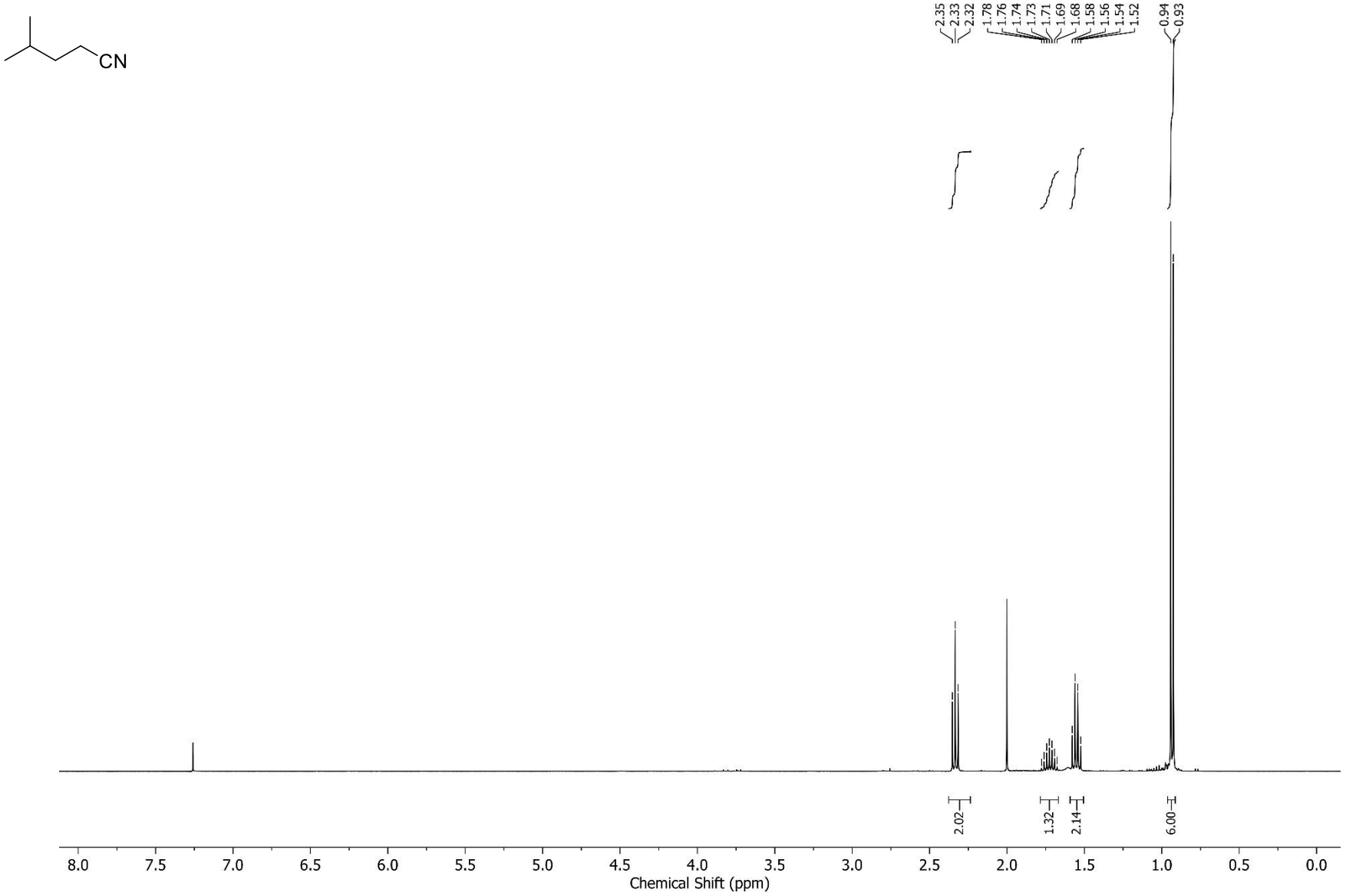
NL:
1.16E8
LDY128#391 RT: 4.19 AV: 1 T: FTMS + p ESI Full lock ms [150.0000-2000.0000]

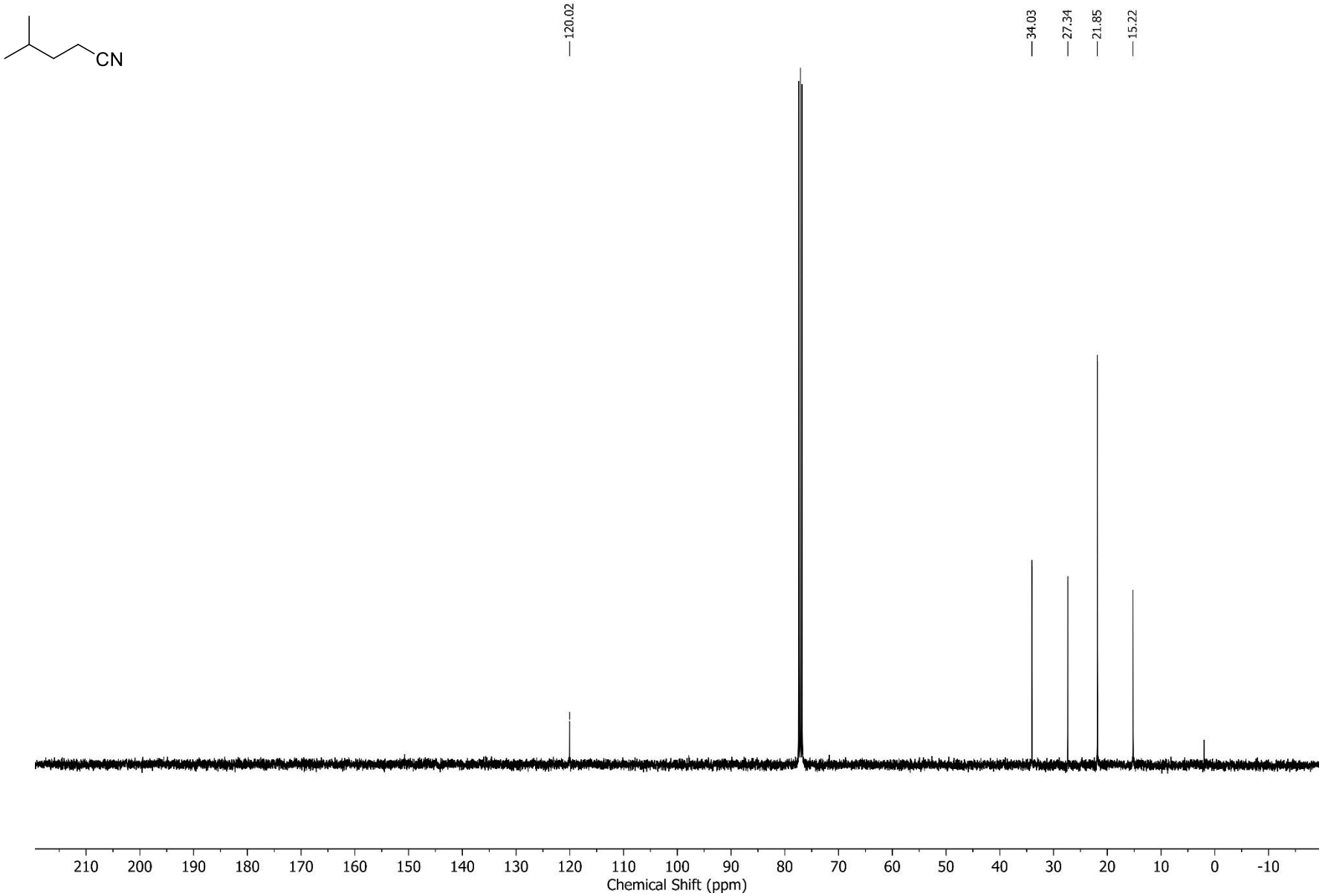
NL:
8.36E5
$C_{11}H_{16}O_3S$ +H:
$C_{11}H_{17}O_3S_1$
pa Chrg 1

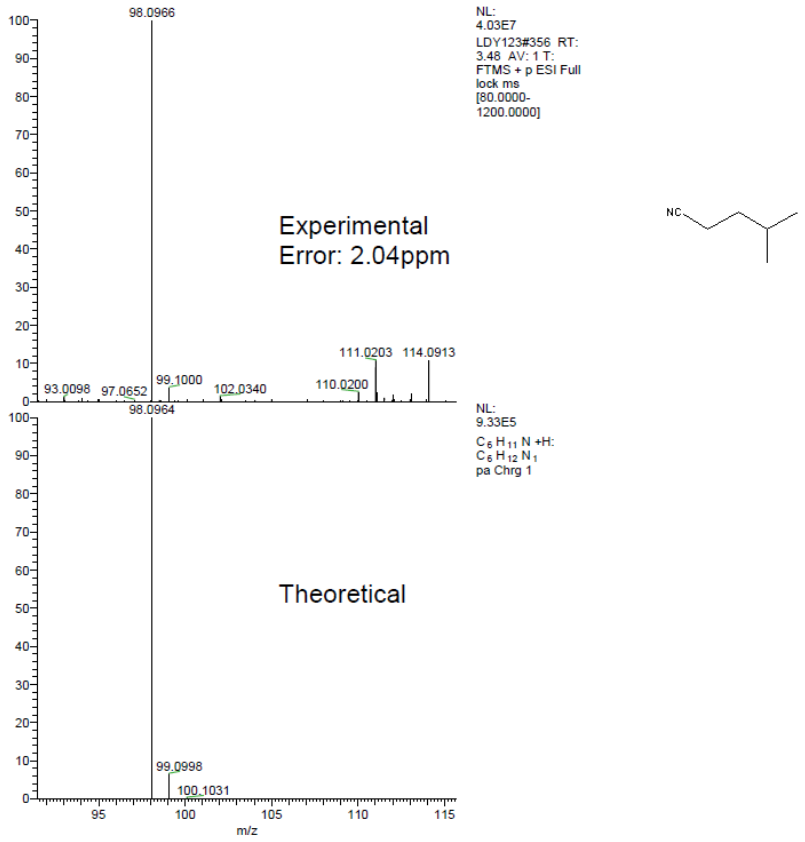

CO2Et
4.14
4.12
4.11
4.09
2.31
2.29
2.29
2.29
2.27
1.61
1.60
1.60
1.58
1.58
1.57
1.57
1.57
1.56
1.56
1.55
1.55
1.54
1.54
1.54
1.53
1.53
1.53
1.52
1.52
1.52
1.51
1.51
1.50
1.49
1.49
1.27
1.25
1.23
0.90
0.89
2.00
1.73
3.03
3.23
6.34
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
Chemical Shift (ppm)


174.09
60.16
33.79
32.45
27.68
22.21
14.23
CO2Et
Chemical Shift (ppm)

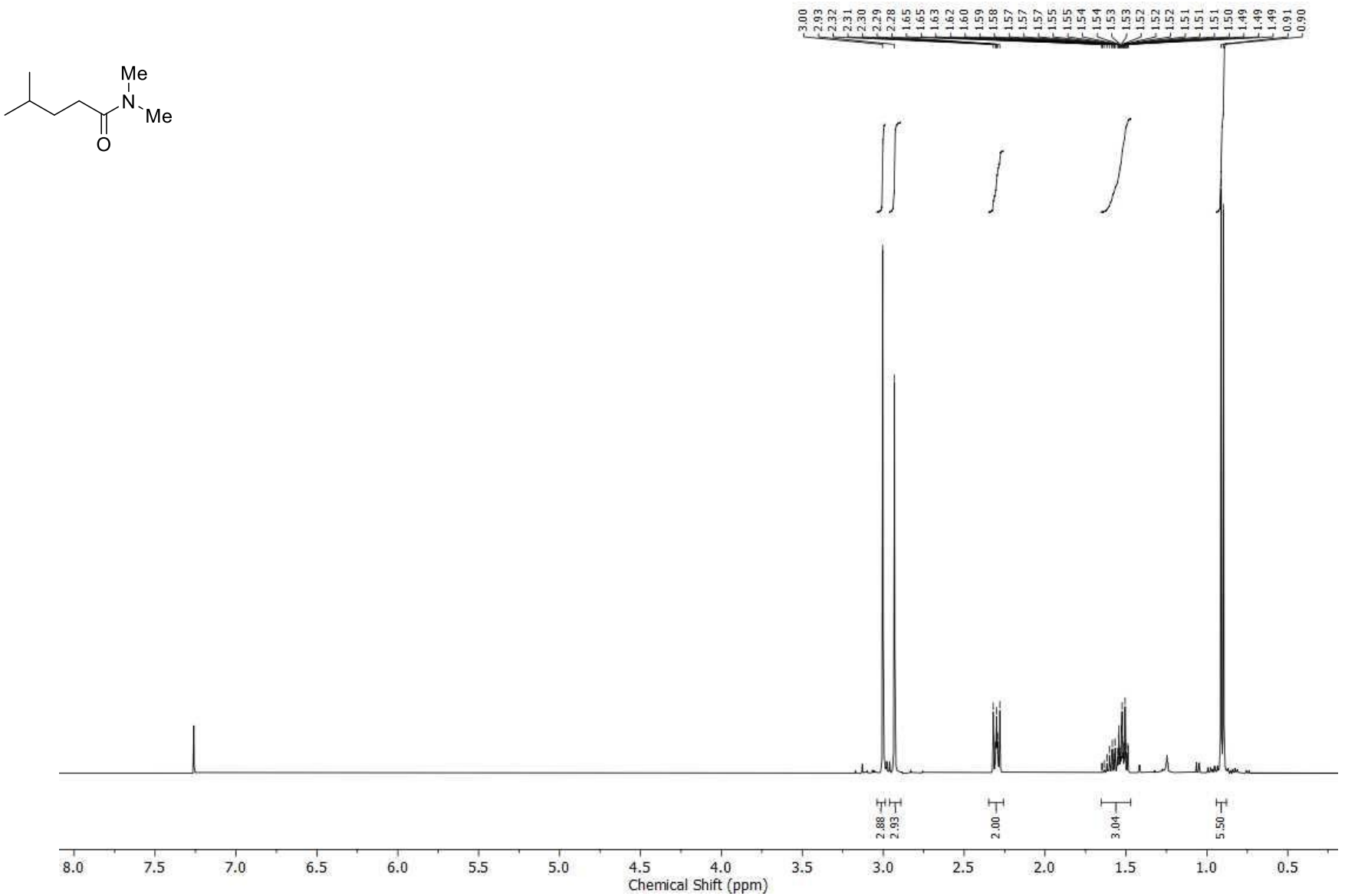

145.1223
144.9822
146.1257
146.9803
147.0916
148.1122
Experimental
Error: 0.00 ppm
NL:
7.86E6
RJS018_201912121257
09#419 RT: 4.19 AV: 1
T: FTMS + p ESI Full
lock ms
[100.0000-1500.0000]
145.1223
146.1257
147.1266
148.1299
Theoretical
NL:
9.11E5
C8 O2 H16 +H:
C8 O2 H17
pa Chrg 1
m/z

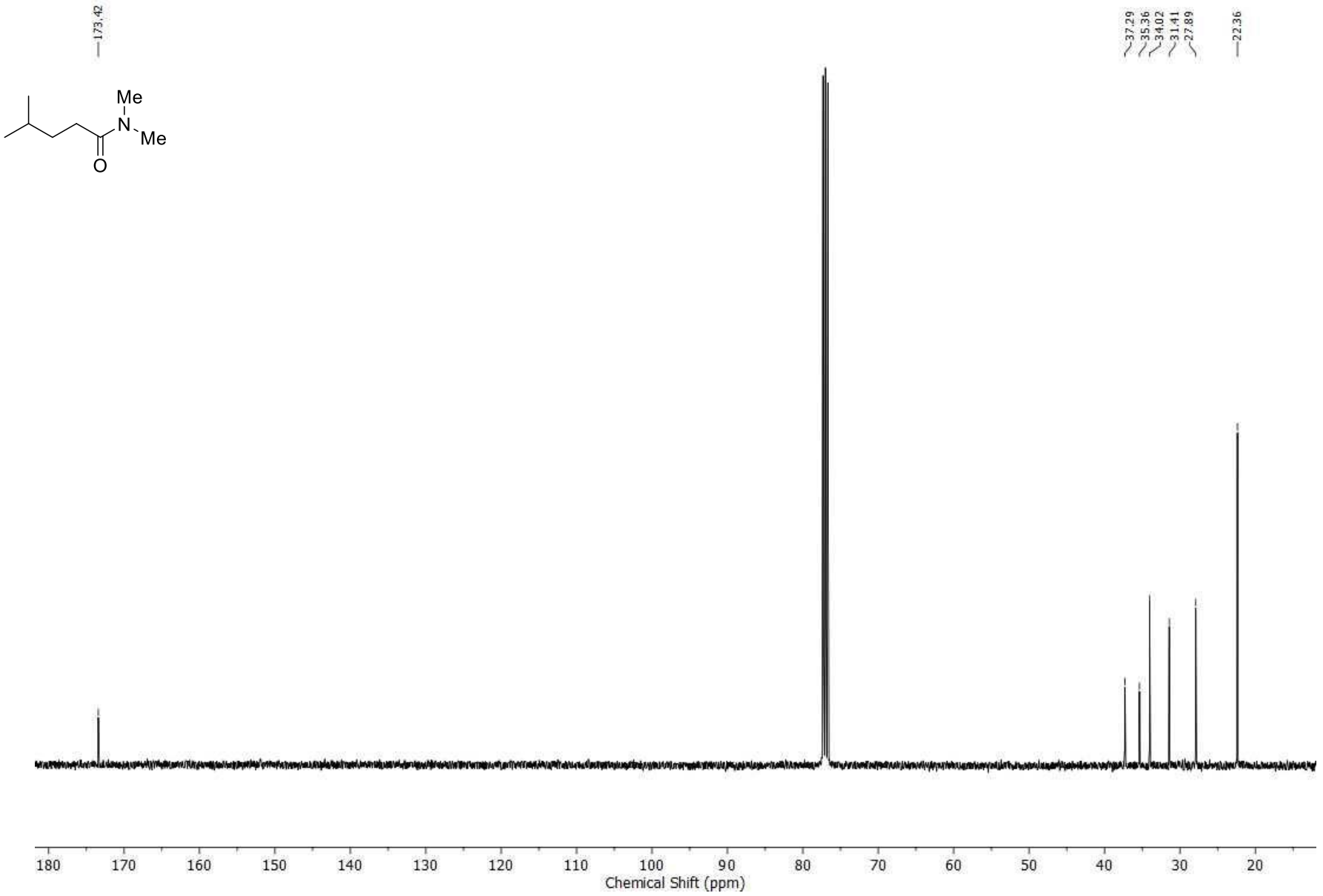

CN
2.35
2.33
2.32
1.78
1.76
1.74
1.73
1.71
1.69
1.68
1.58
1.56
1.54
1.52
0.94
0.93
2.02
1.32
2.14
6.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
Chemical Shift (ppm)

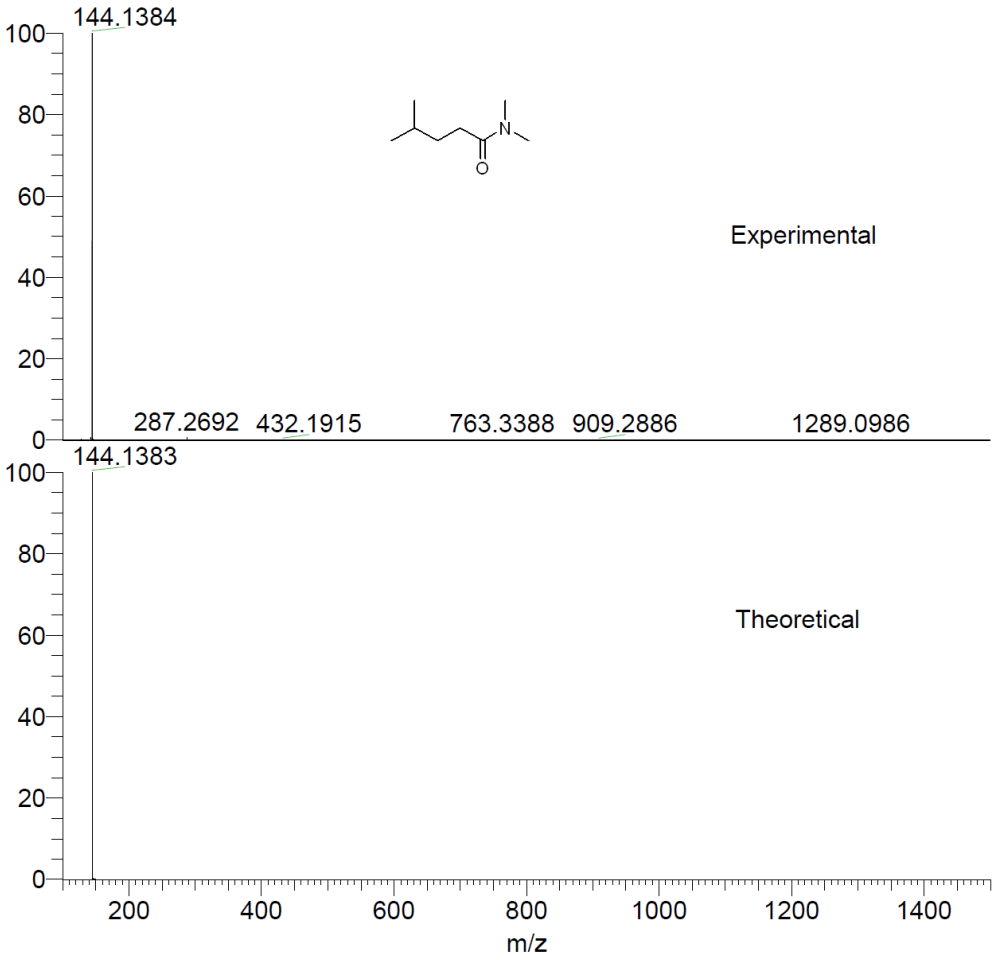

CN
120.02
34.03
27.34
21.85
15.22
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
Chemical Shift (ppm)


98.0966
NL:
4.03E7
LDY123#356 RT:
3.48 AV: 1 T:
FTMS + p ESI Full
lock ms
[80.0000-
1200.0000]
Experimental
Error: 2.04ppm
111.0203
114.0913
93.0098
97.0652
99.1000
102.0340
110.0200
98.0964
NL:
9.33E5
C6 H11 N +H:
C6 H12 N1
pa Chrg 1
Theoretical
99.0998
100.1031
m/z

NC

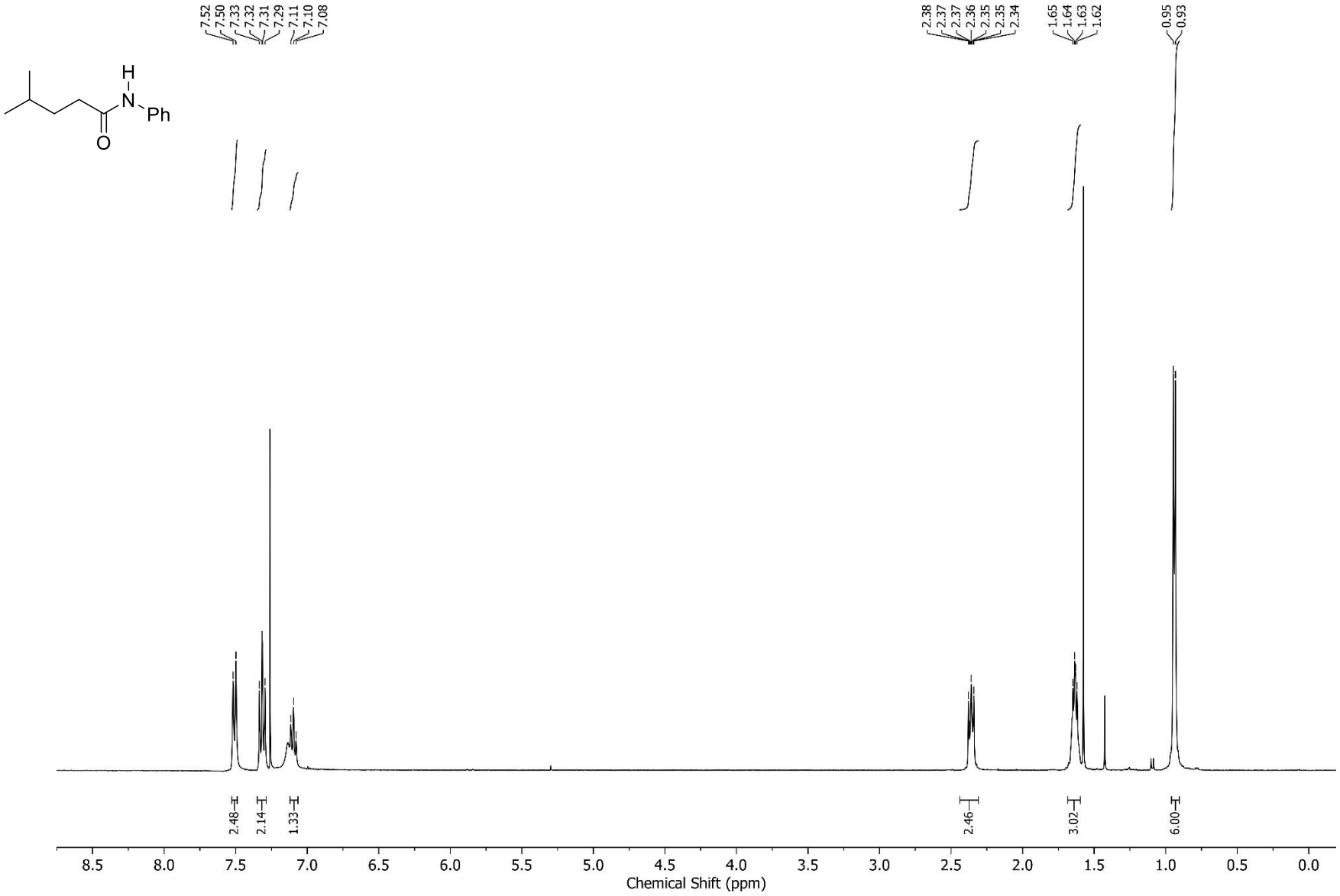

Me
N
Me
O
3.00
2.93
2.32
2.31
2.30
2.29
2.28
1.65
1.65
1.63
1.62
1.60
1.59
1.58
1.57
1.57
1.57
1.55
1.55
1.54
1.54
1.53
1.53
1.52
1.52
1.52
1.51
1.51
1.51
1.50
1.49
1.49
1.49
0.91
0.90
2.88
2.93
2.00
3.04
5.50
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
Chemical Shift (ppm)

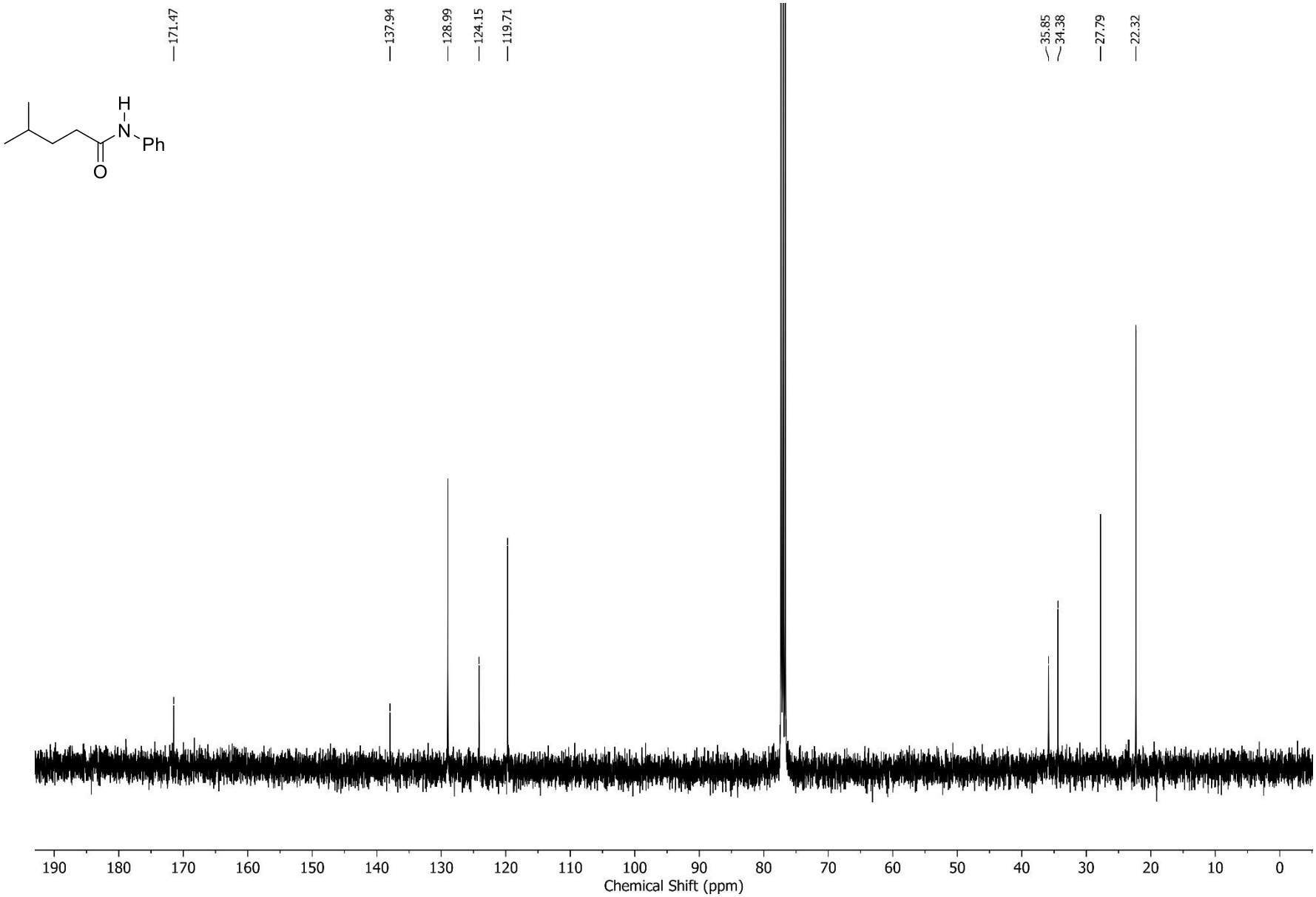

173.42
37.29
35.36
34.02
31.41
27.89
22.36
Me
N
Me
O
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
Chemical Shift (ppm)

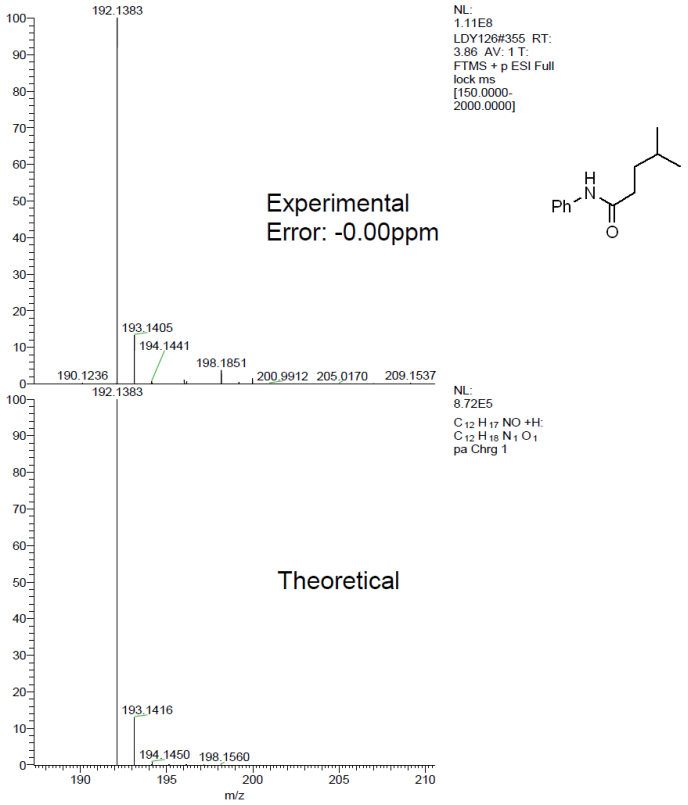


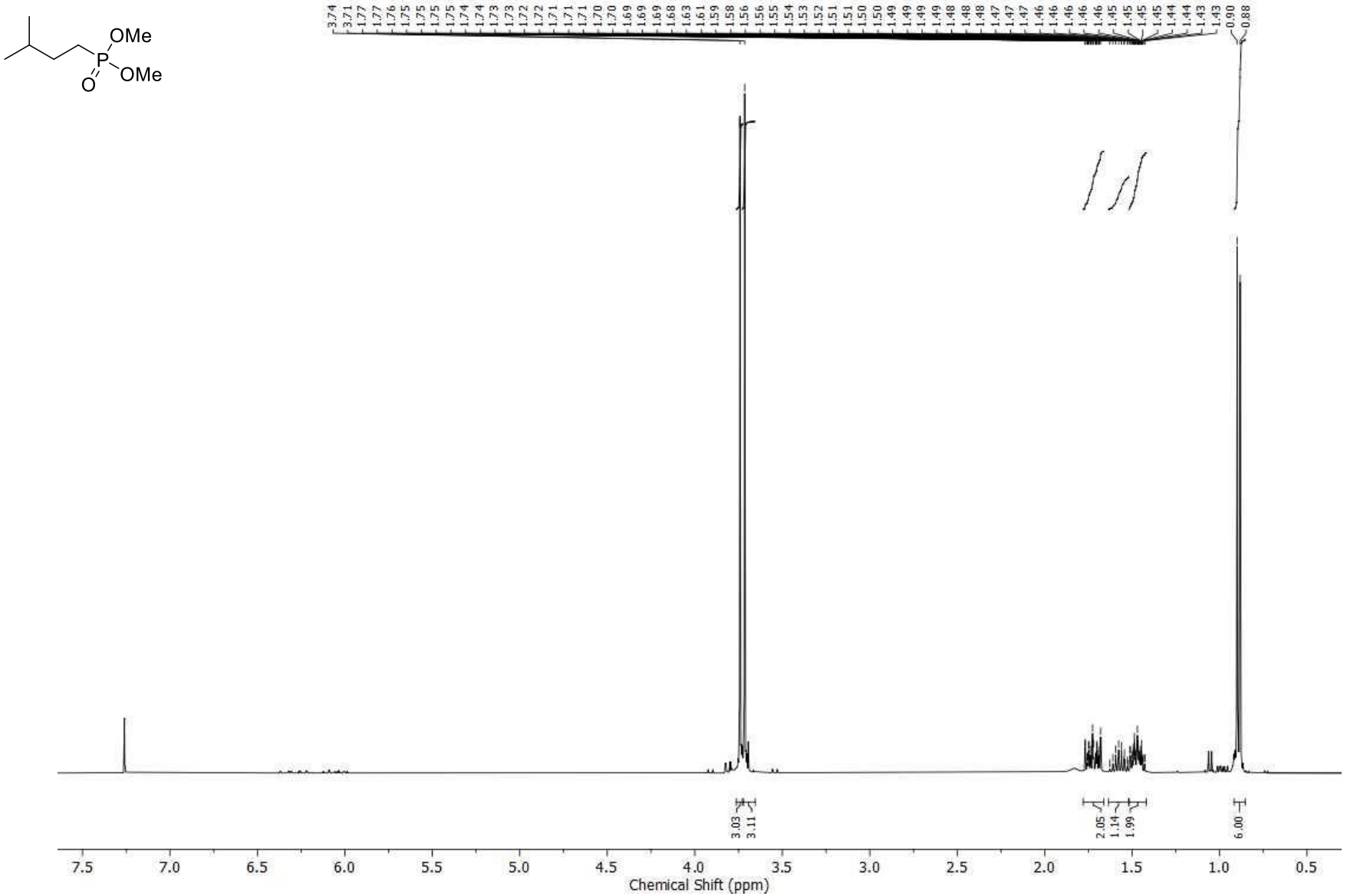
NL:
8.76E9
LDY129A#293 RT: 2.87 AV: 1 T: FTMS + p ESI Full lock ms [100.0000-1500.0000]

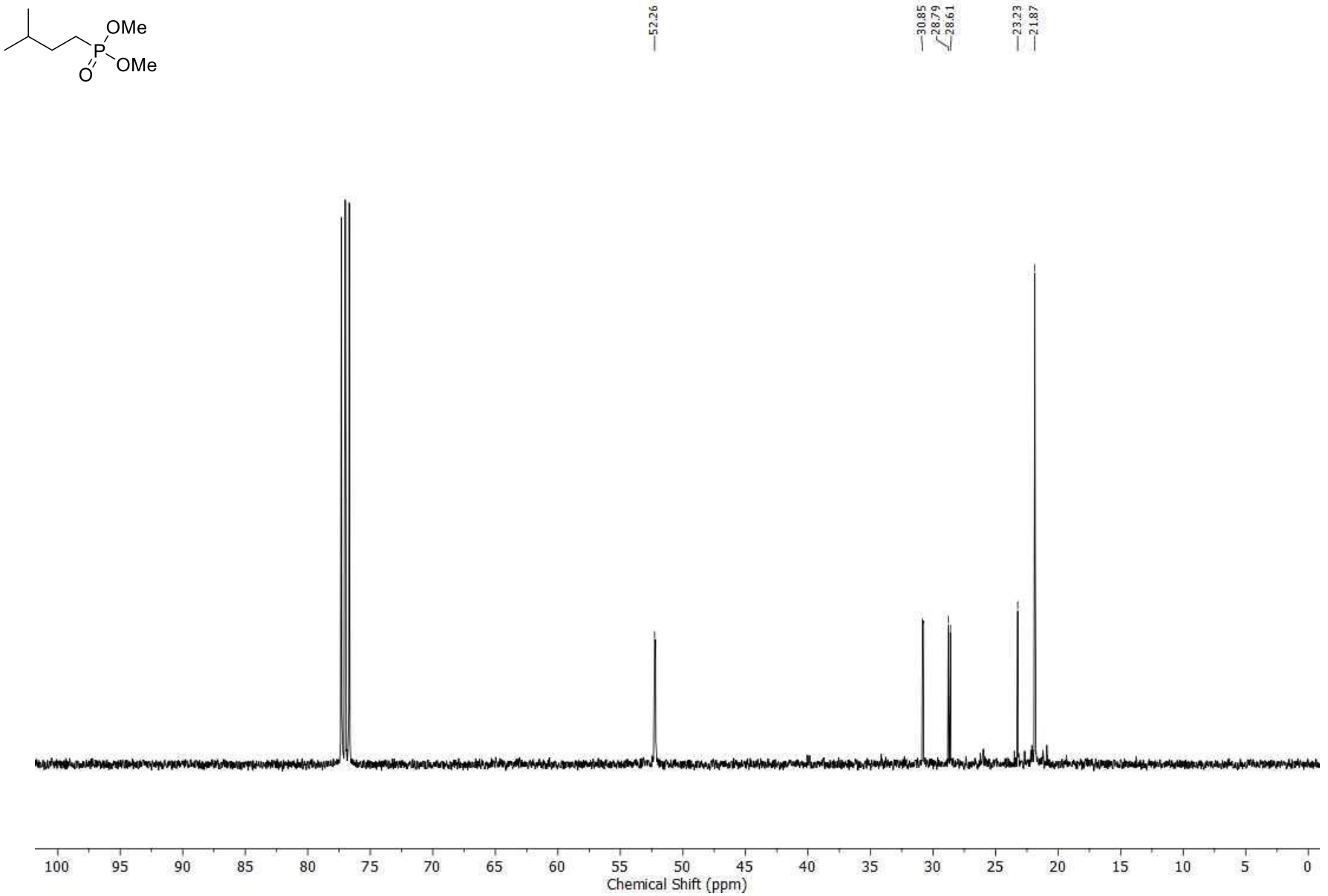
NL:
9.10E5
$C_8H_{17}NO$ +H:
$C_8H_{18}N_1O_1$
pa Chrg 1

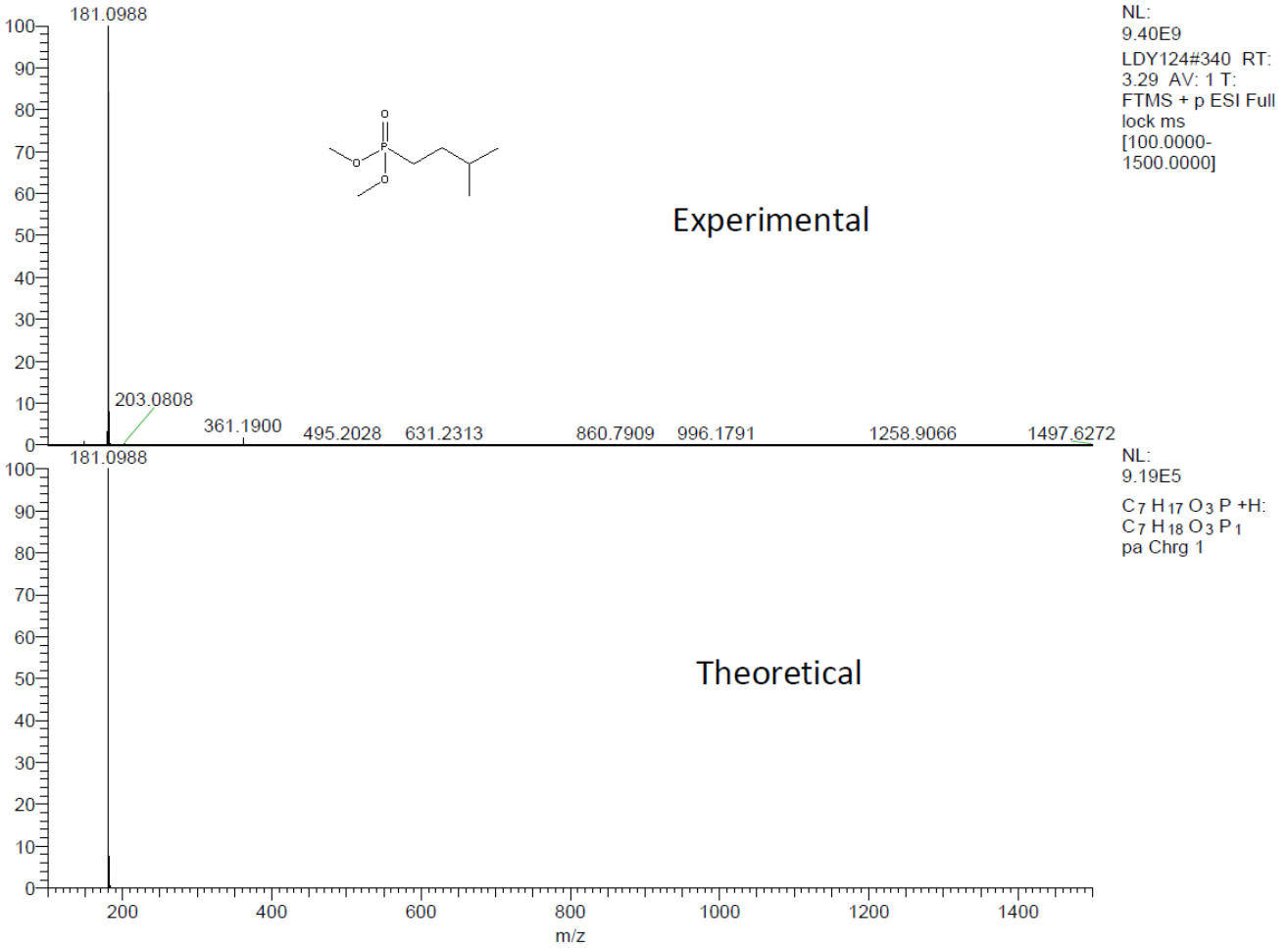

7.52
7.50
7.33
7.32
7.31
7.29
7.11
7.10
7.08
2.38
2.37
2.37
2.36
2.35
2.35
2.34
1.65
1.64
1.63
1.62
0.95
0.93
H
N
Ph
O
2.48
2.14
1.33
2.46
3.02
6.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
Chemical Shift (ppm)

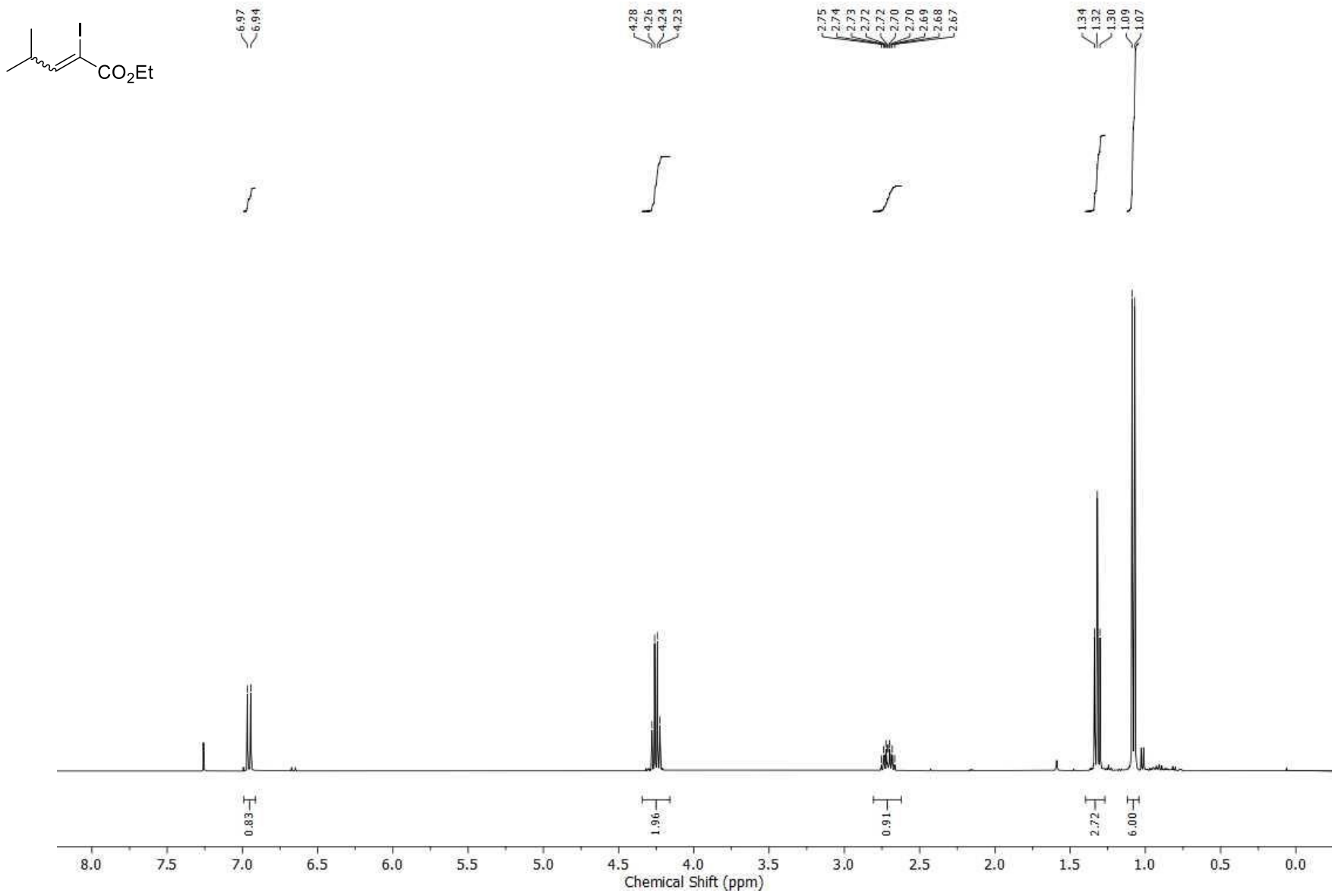

171.47
137.94
128.99
124.15
119.71
35.85
34.38
27.79
22.32
H
N
Ph
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)

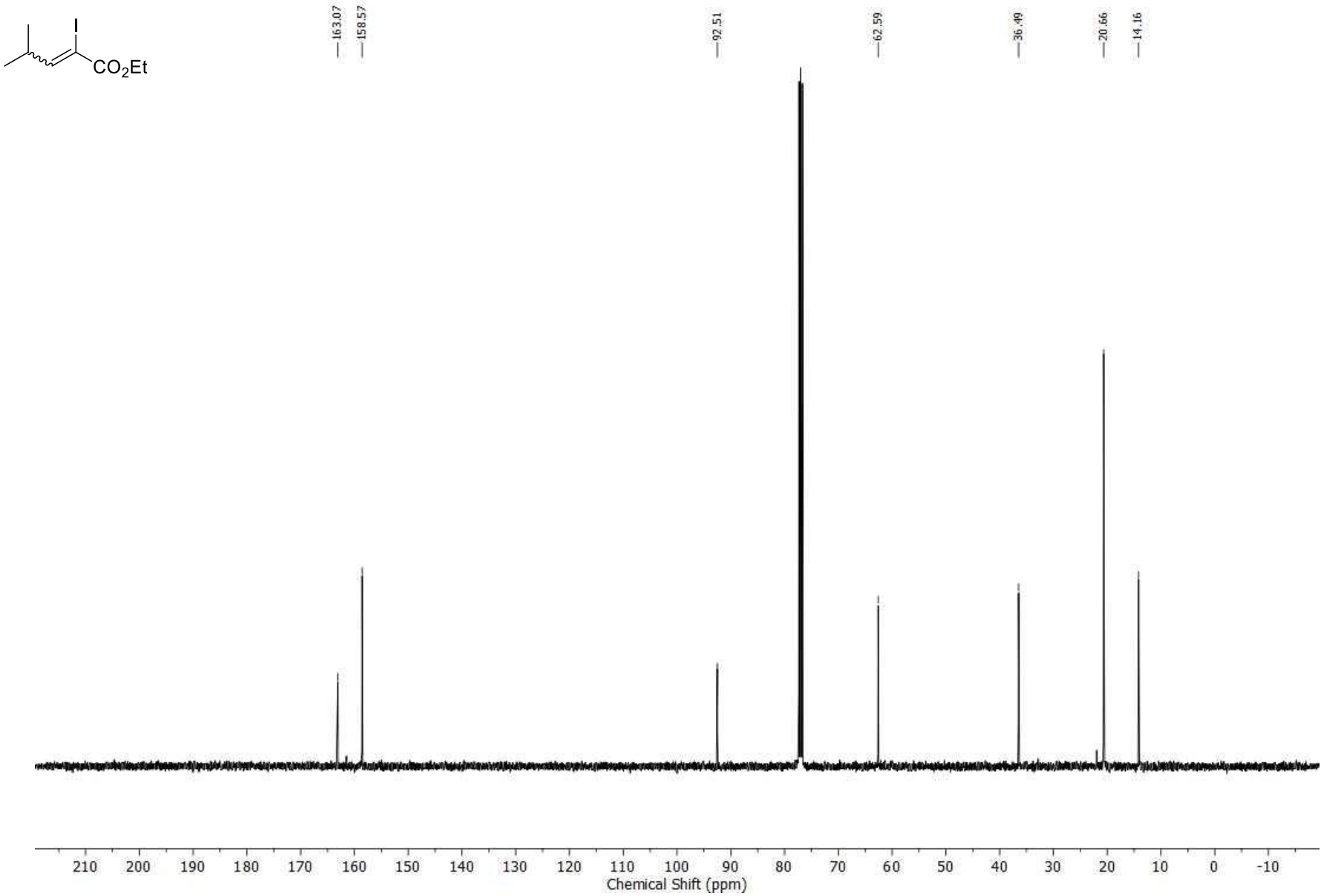

192.1383
NL:
1.11E8
LDY126#355 RT:
3.86 AV: 1 T:
FTMS + p ESI Full
lock ms
[150.0000-
2000.0000]
Experimental
Error: -0.00ppm
193.1405
194.1441
198.1851
190.1236
200.9912
205.0170
209.1537
192.1383
NL:
8.72E5
C12 H17 NO +H:
C12 H18 N1 O1
pa Chrg 1
Theoretical
193.1416
194.1450
198.1560
m/z

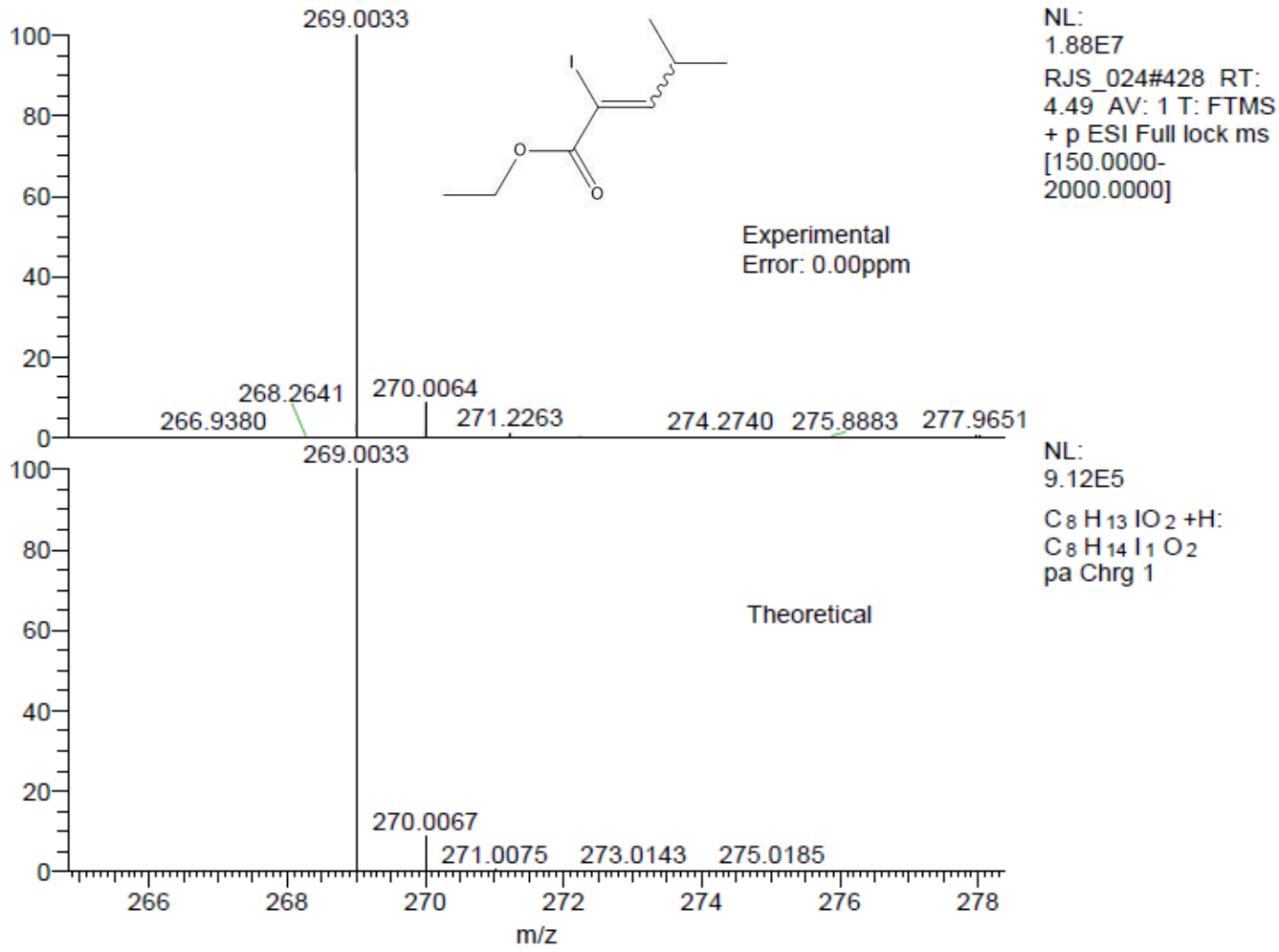

OMe
P
O
OMe
3.74
3.71
1.77
1.76
1.75
1.74
1.73
1.72
1.71
1.70
1.69
1.68
1.63
1.61
1.59
1.58
1.56
1.55
1.54
1.53
1.52
1.51
1.50
1.49
1.48
1.47
1.46
1.45
1.44
1.43
0.90
0.88
3.03
3.11
2.05
1.14
1.99
6.00
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
Chemical Shift (ppm)

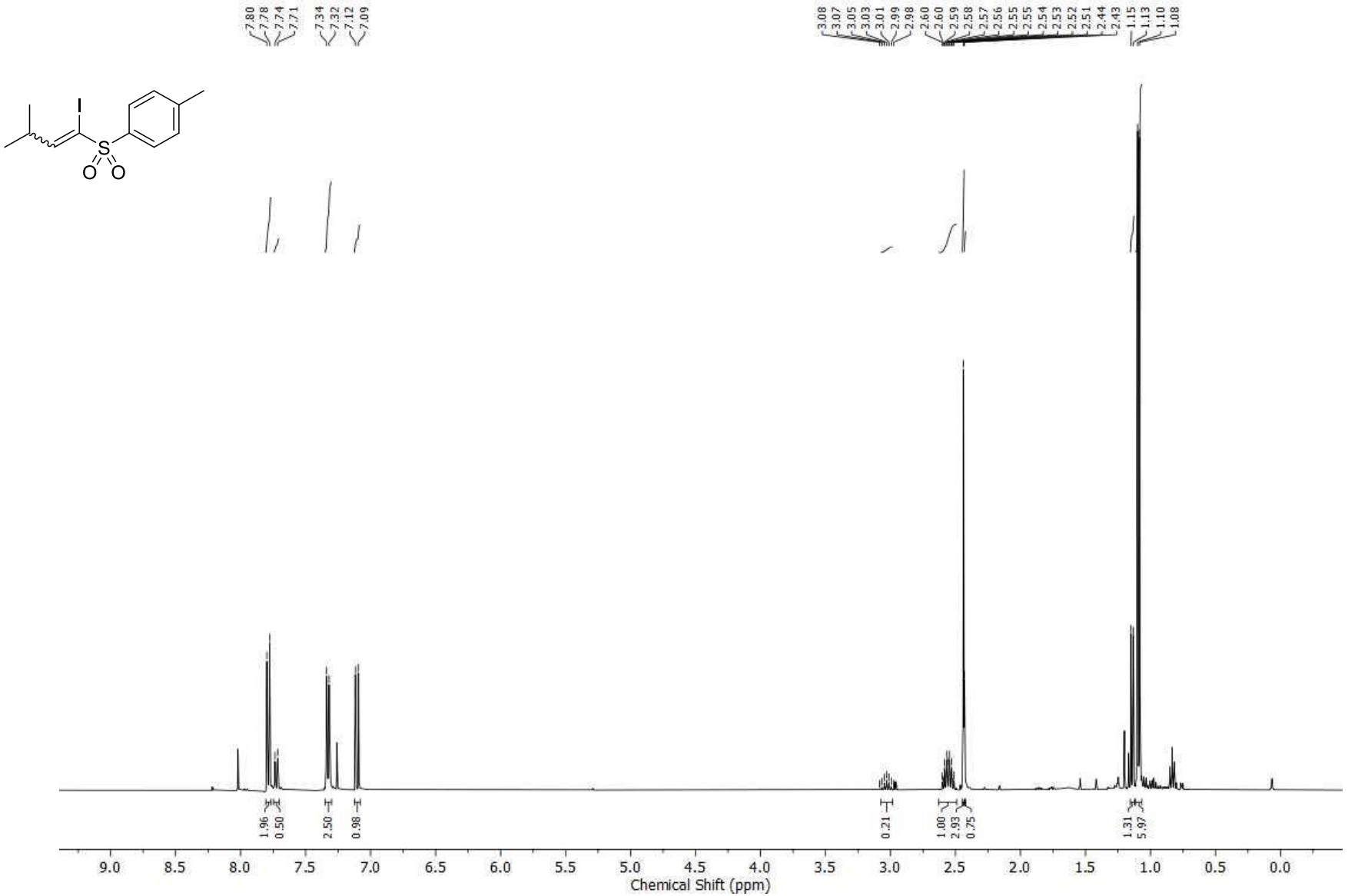

OMe
P
O
OMe
52.26
30.85
28.79
28.61
23.23
21.87
100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
Chemical Shift (ppm)

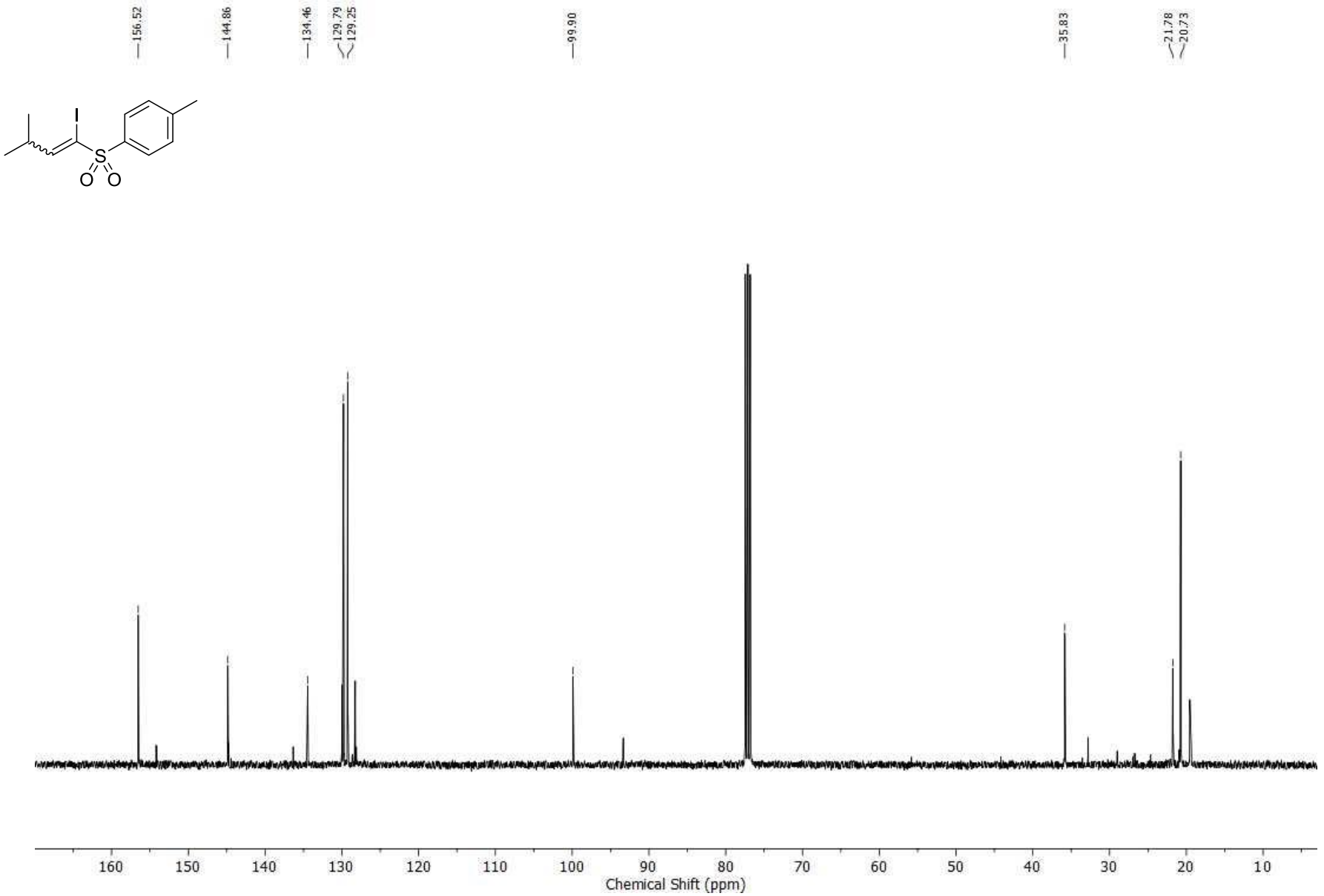

181.0988
NL:
9.40E9
LDY124#340 RT:
3.29 AV: 1 T:
FTMS + p ESI Full
lock ms
[100.0000-
1500.0000]
203.0808
361.1900
495.2028
631.2313
860.7909
996.1791
1258.9066
1497.6272
Experimental
181.0988
NL:
9.19E5
C7 H17 O3 P +H:
C7 H18 O3 P1
pa Chrg 1
Theoretical
m/z


I
CO2Et
6.97
6.94
4.28
4.26
4.24
4.23
2.75
2.74
2.73
2.72
2.72
2.70
2.70
2.69
2.68
2.67
1.34
1.32
1.30
1.09
1.07
0.83
1.96
0.91
2.72
6.00
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
Chemical Shift (ppm)

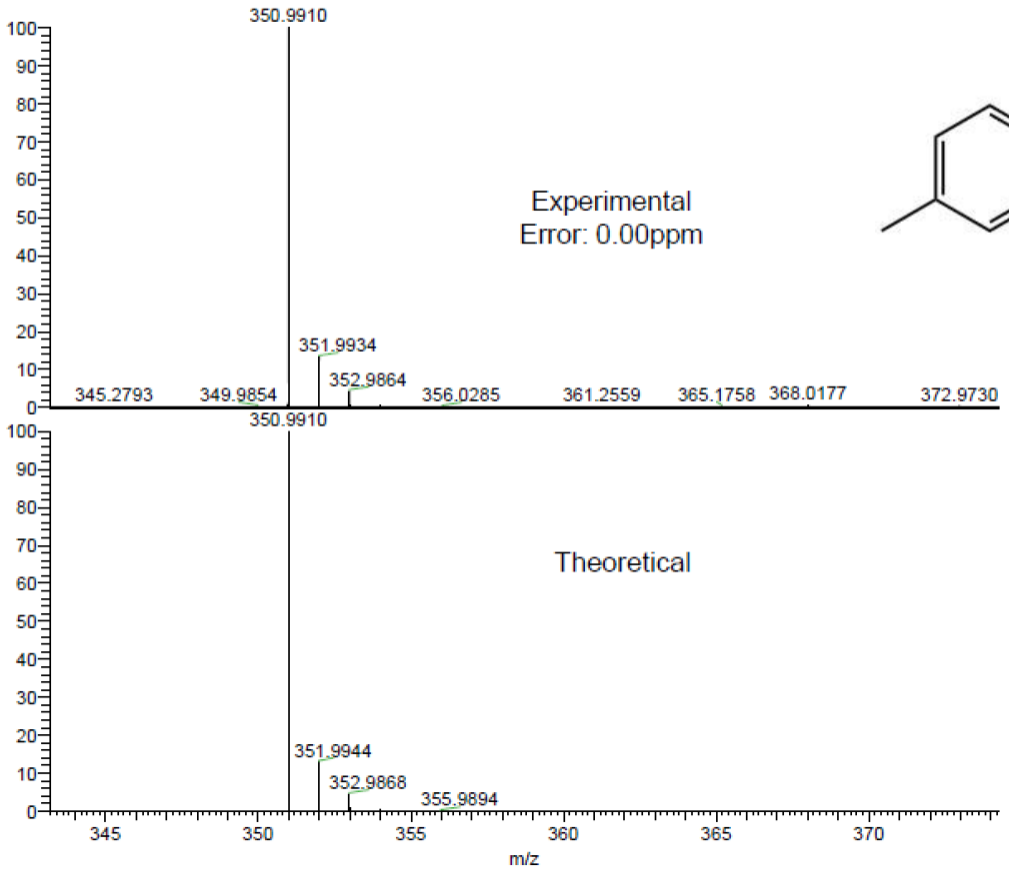

I
CO2Et
163.07
158.57
92.51
62.59
36.49
20.66
14.16
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
Chemical Shift (ppm)

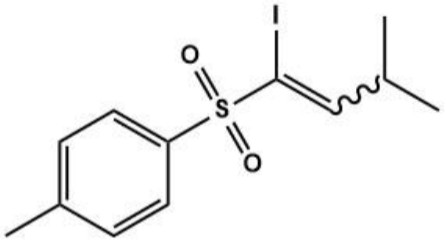

269.0033
NL:
1.88E7
RJS_024#428 RT: 4.49 AV: 1 T: FTMS + p ESI Full lock ms [150.0000-2000.0000]
Experimental
Error: 0.00ppm
268.2641
270.0064
266.9380
271.2263
274.2740
275.8883
277.9651
269.0033
NL:
9.12E5
C8 H13 IO2 +H:
C8 H14 I1 O2
pa Chrg 1
Theoretical
270.0067
271.0075
273.0143
275.0185
100
80
60
40
20
0
266
268
270
272
274
276
278
m/z


7.80
7.78
7.74
7.71
7.34
7.32
7.12
7.09
3.08
3.07
3.05
3.03
3.01
2.99
2.98
2.60
2.60
2.59
2.58
2.57
2.56
2.55
2.55
2.54
2.53
2.52
2.51
2.44
2.43
1.15
1.13
1.10
1.08
I
S
O
O
1.96
0.50
2.50
0.98
0.21
1.00
2.93
0.75
1.31
5.97
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
Chemical Shift (ppm)


156.52
144.86
134.46
129.79
129.25
99.90
35.83
21.78
20.73
I
S
O
O
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
Chemical Shift (ppm)

UCL Chemistry Mass Spectrometry





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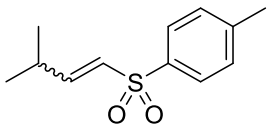
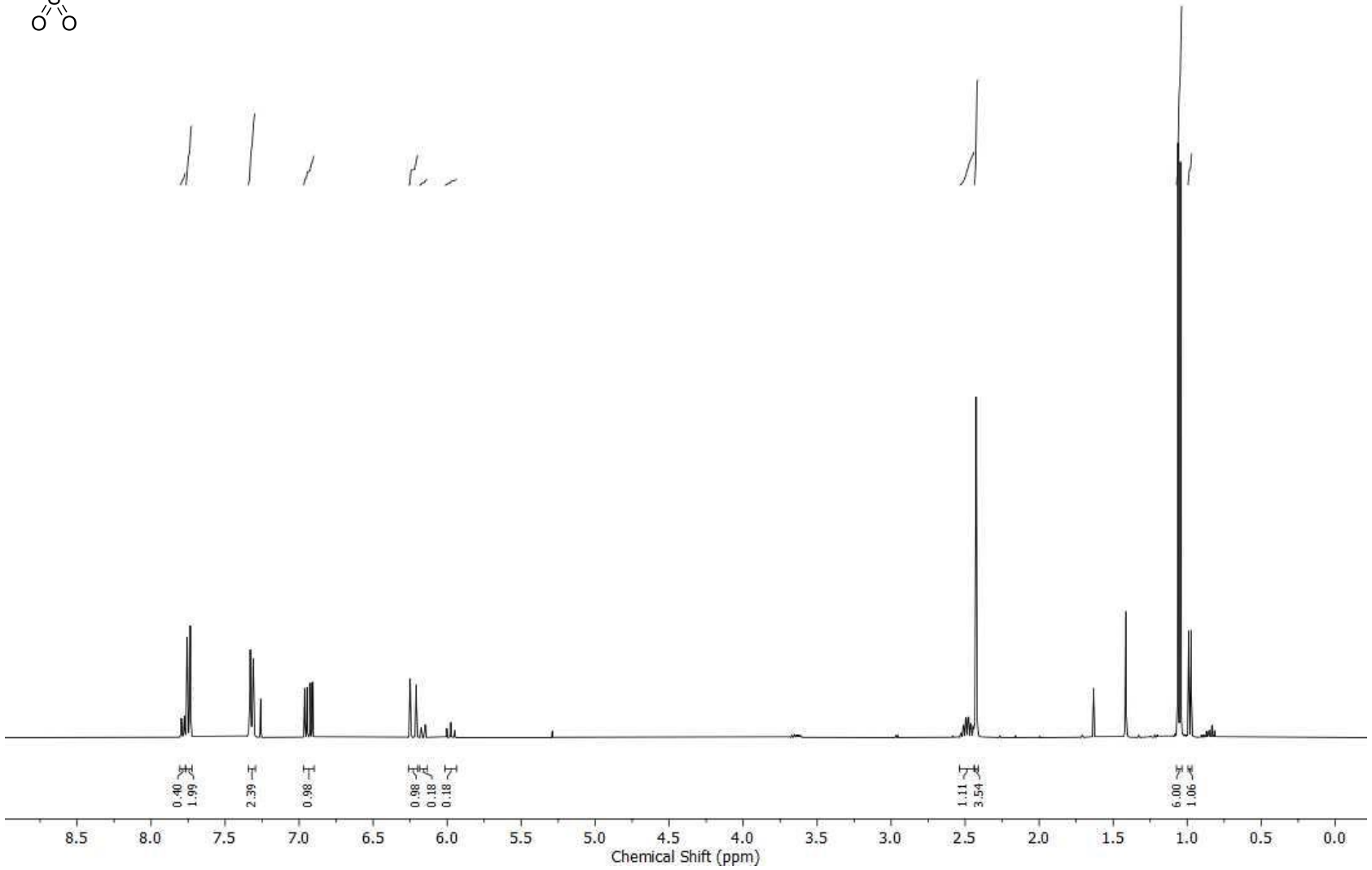
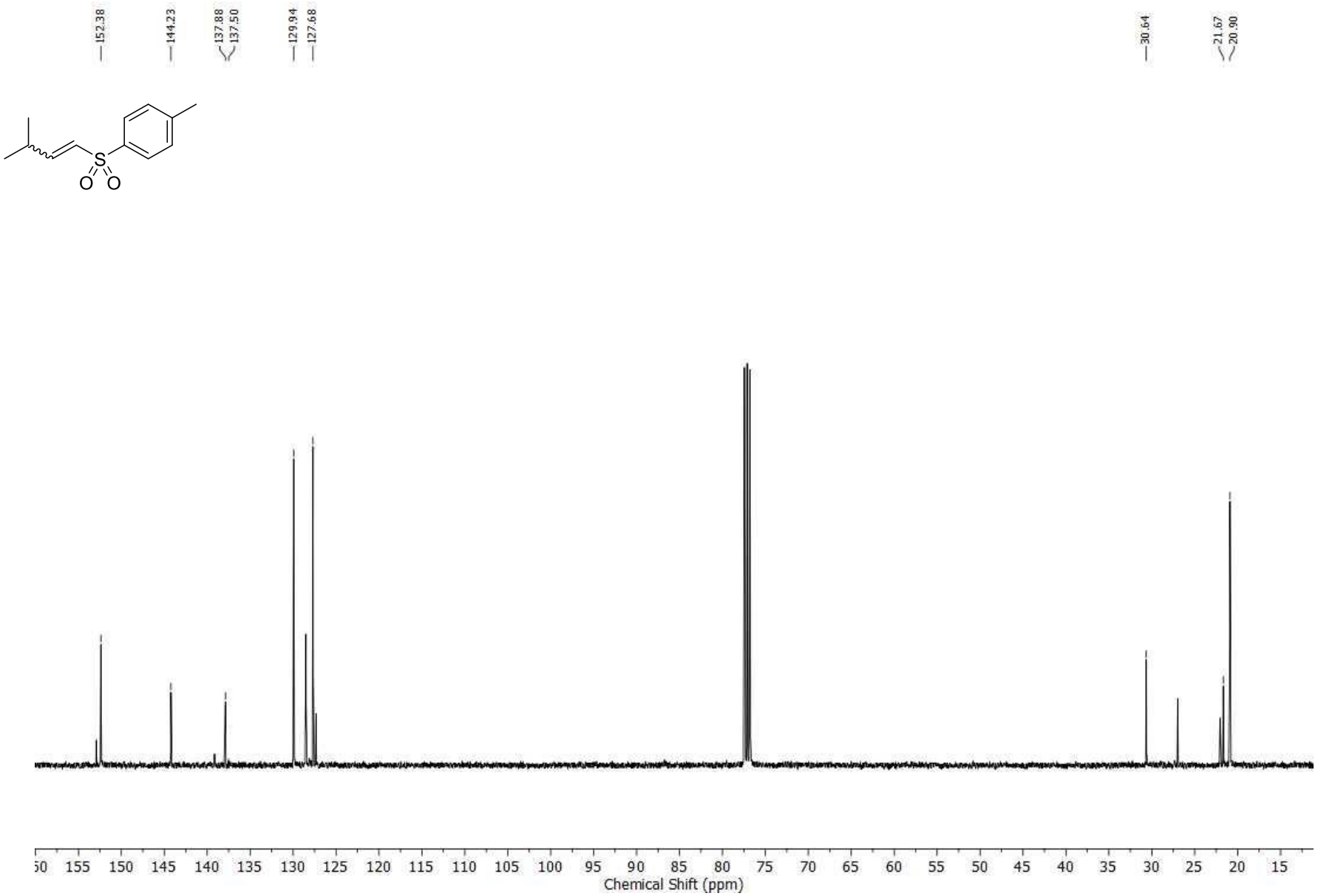
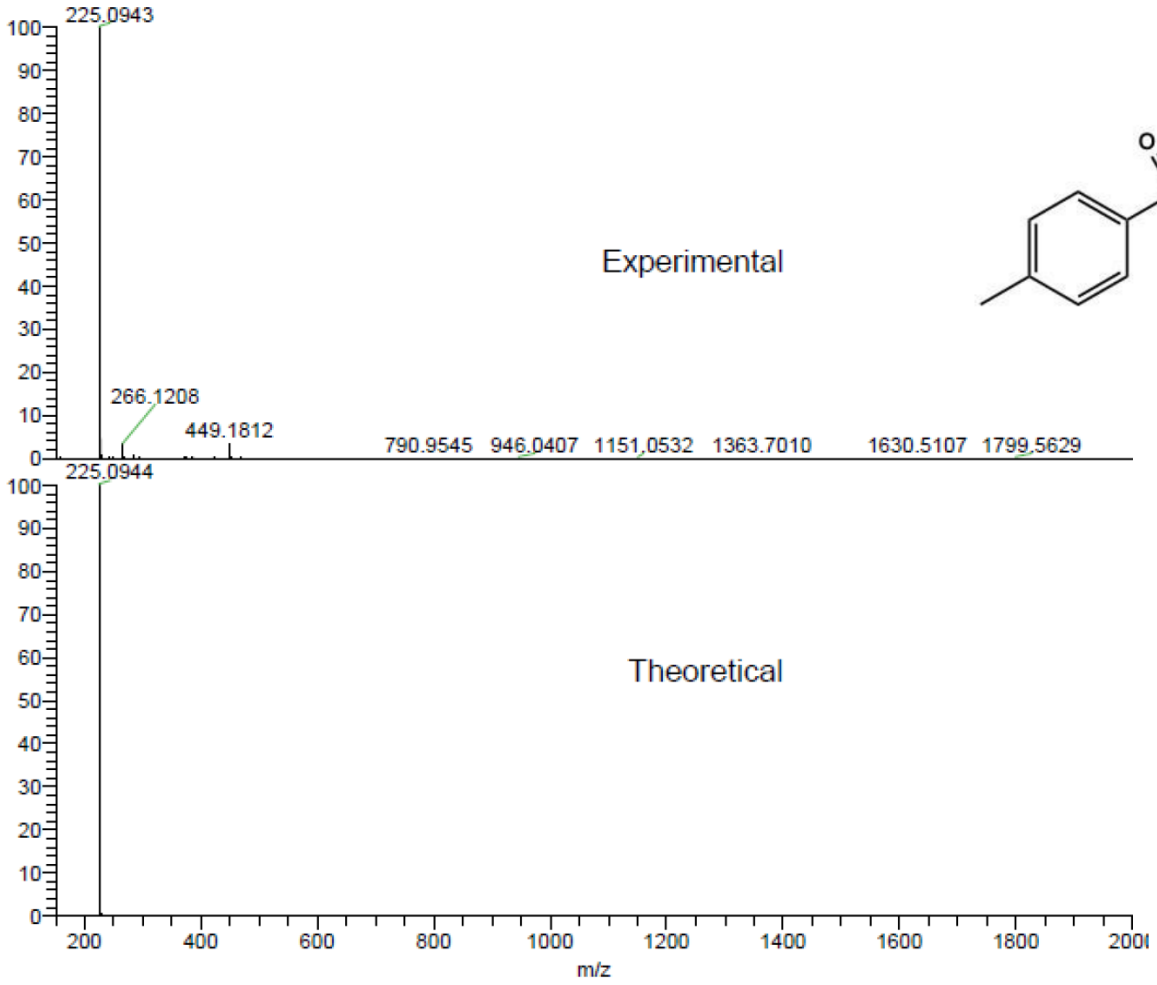
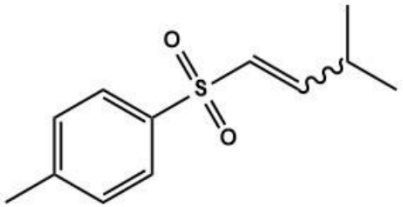

S
O
O

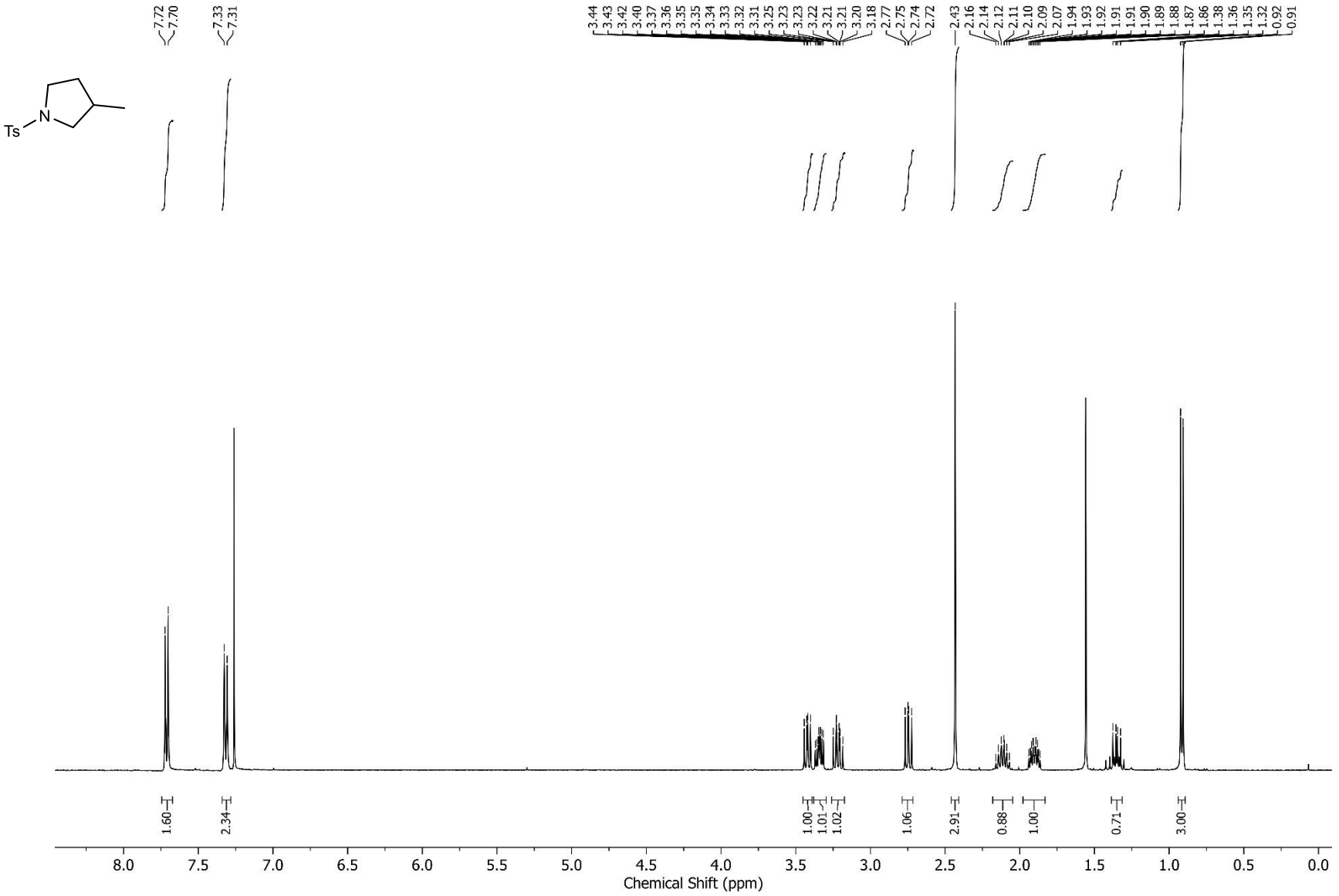

0.40
1.99
2.39
0.98
0.98
0.18
0.18
1.11
3.54
6.00
1.06
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
Chemical Shift (ppm)

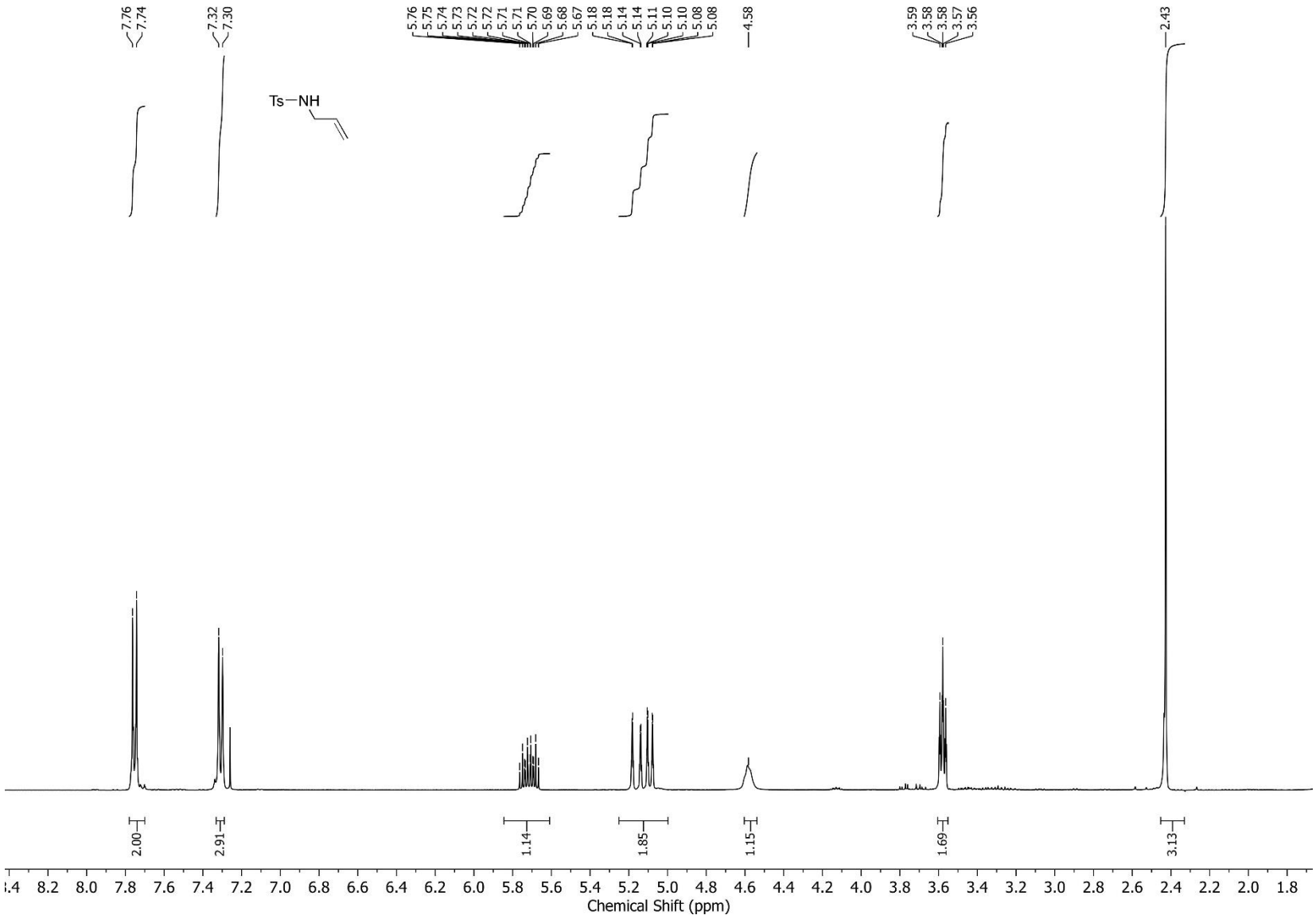

152.38
144.23
137.88
137.50
129.94
127.68
30.64
21.67
20.90
O
S
O
155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15
Chemical Shift (ppm)

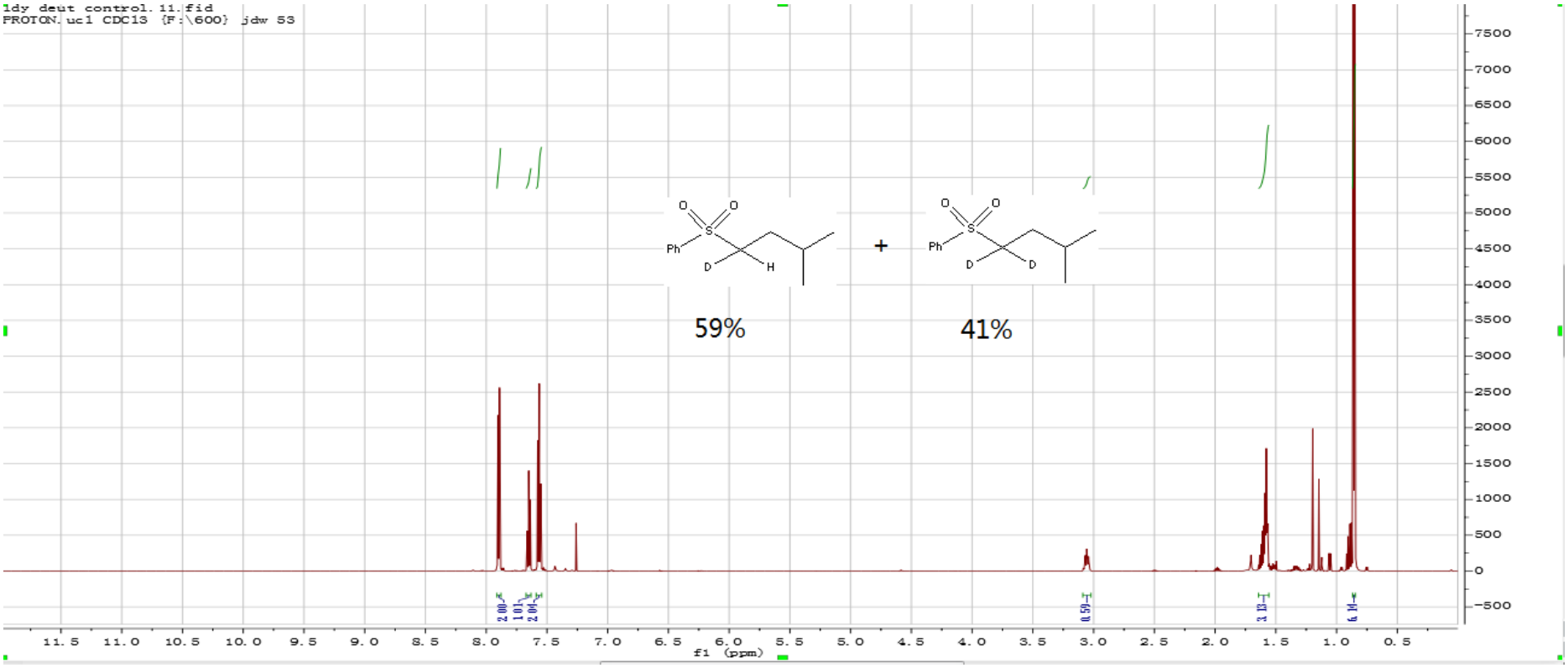
UCL Chemistry Mass Spectrometry

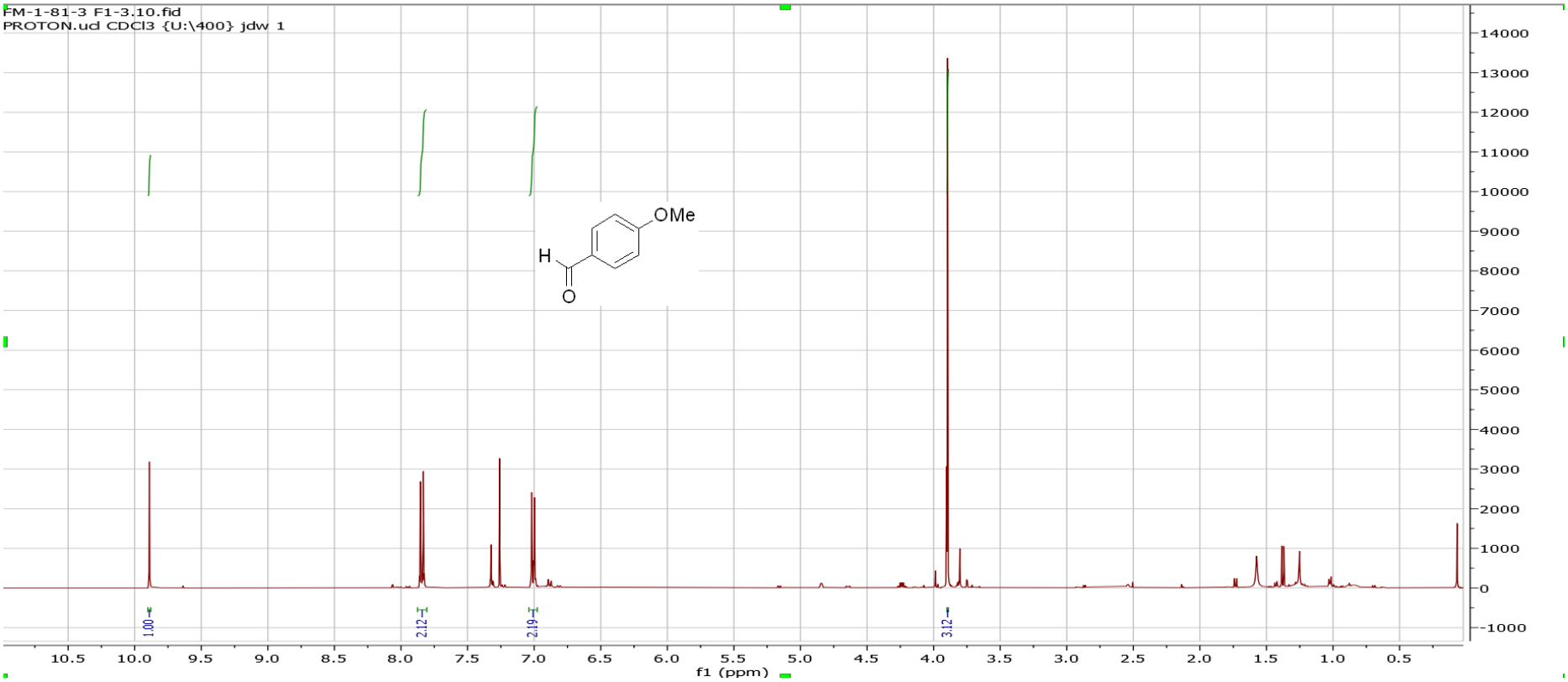





Ts
N
7.72
7.70
7.33
7.31
3.44
3.43
3.42
3.40
3.37
3.36
3.35
3.35
3.34
3.33
3.32
3.31
3.25
3.23
3.23
3.22
3.21
3.21
3.20
3.18
2.77
2.75
2.74
2.72
2.43
2.16
2.14
2.12
2.11
2.10
2.09
2.07
1.94
1.93
1.92
1.91
1.91
1.90
1.89
1.88
1.87
1.86
1.38
1.36
1.35
1.32
0.92
0.91
1.60
2.34
1.00
1.01
1.02
1.06
2.91
0.88
1.00
0.71
3.00
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
Chemical Shift (ppm)


7.76
7.74
7.32
7.30
5.76
5.75
5.74
5.73
5.72
5.72
5.71
5.71
5.70
5.69
5.68
5.67
5.18
5.18
5.14
5.14
5.11
5.10
5.10
5.08
5.08
4.58
3.59
3.58
3.58
3.57
3.56
2.43
Ts—NH
2.00
2.91
1.14
1.85
1.15
1.69
3.13
Chemical Shift (ppm)


1dy deut control.11.fid
PROTON.ucl CDCl3 (F:\600) jdw 53
59%
41%
f1 (ppm)


FM-1-81-3 F1-3.10.fid
PROTON.ud CDCl3 {U:\400} jdw 1
OMe
H
O
1.00
2.12
2.19
3.12
f1 (ppm)