

Supporting Information For

**Total Synthesis of (+)-Alopecuridine, (+)-Sieboldine A, and
(-)-Lycojapodine A**

Xiao-Ming Zhang, Hui Shao, Yong-Qiang Tu*, Fu-Min Zhang, and Shao-Hua Wang*

*State Key Laboratory of Applied Organic Chemistry, College of Chemistry and Chemical
Engineering, Lanzhou University, Gansu 730000, P. R. China*

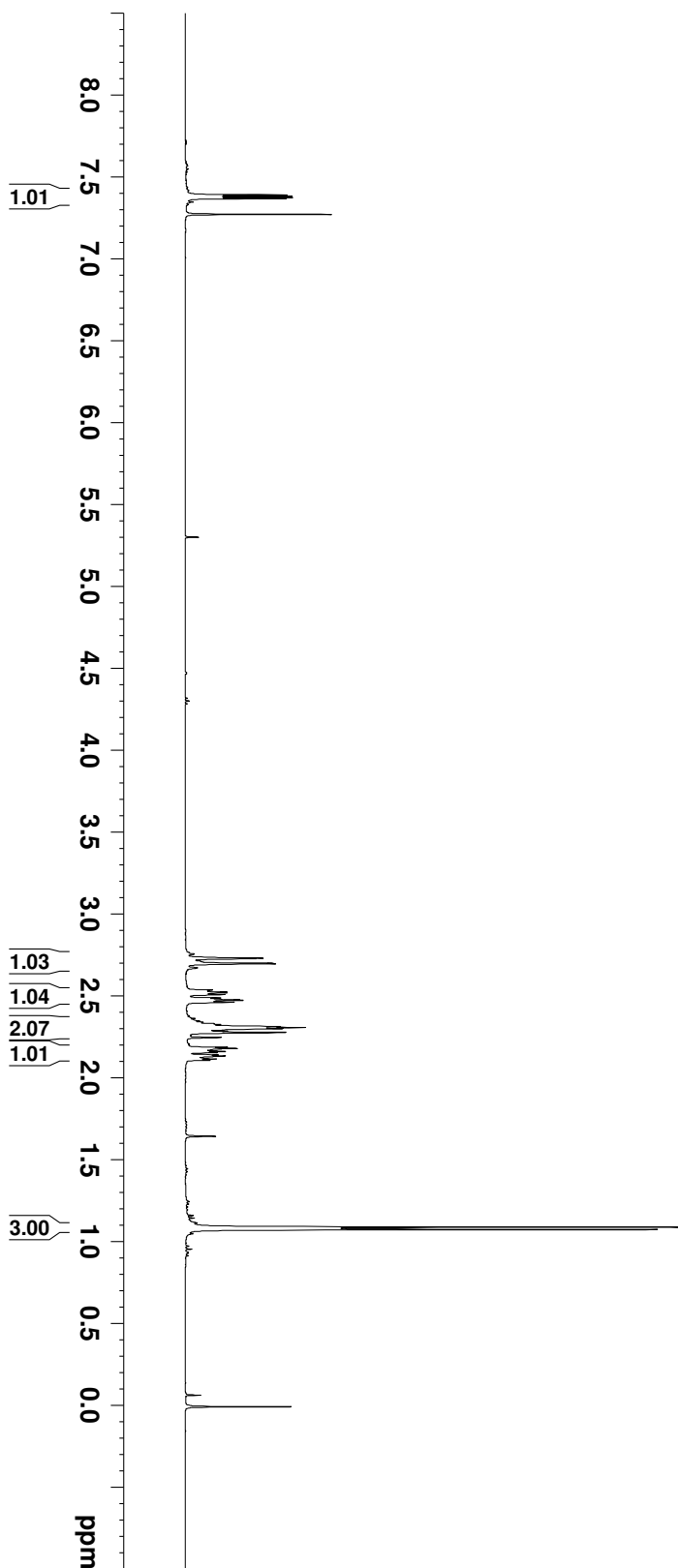
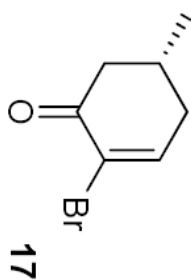
(E-mail: tuyq@lzu.edu.cn, wangshh@lzu.edu.cn)

Contents:

1. Copies of ^1H and ^{13}C spectra of new compounds-----S2-S60
2. Copies of HPLC spectra of compound **9** -----S61-S62

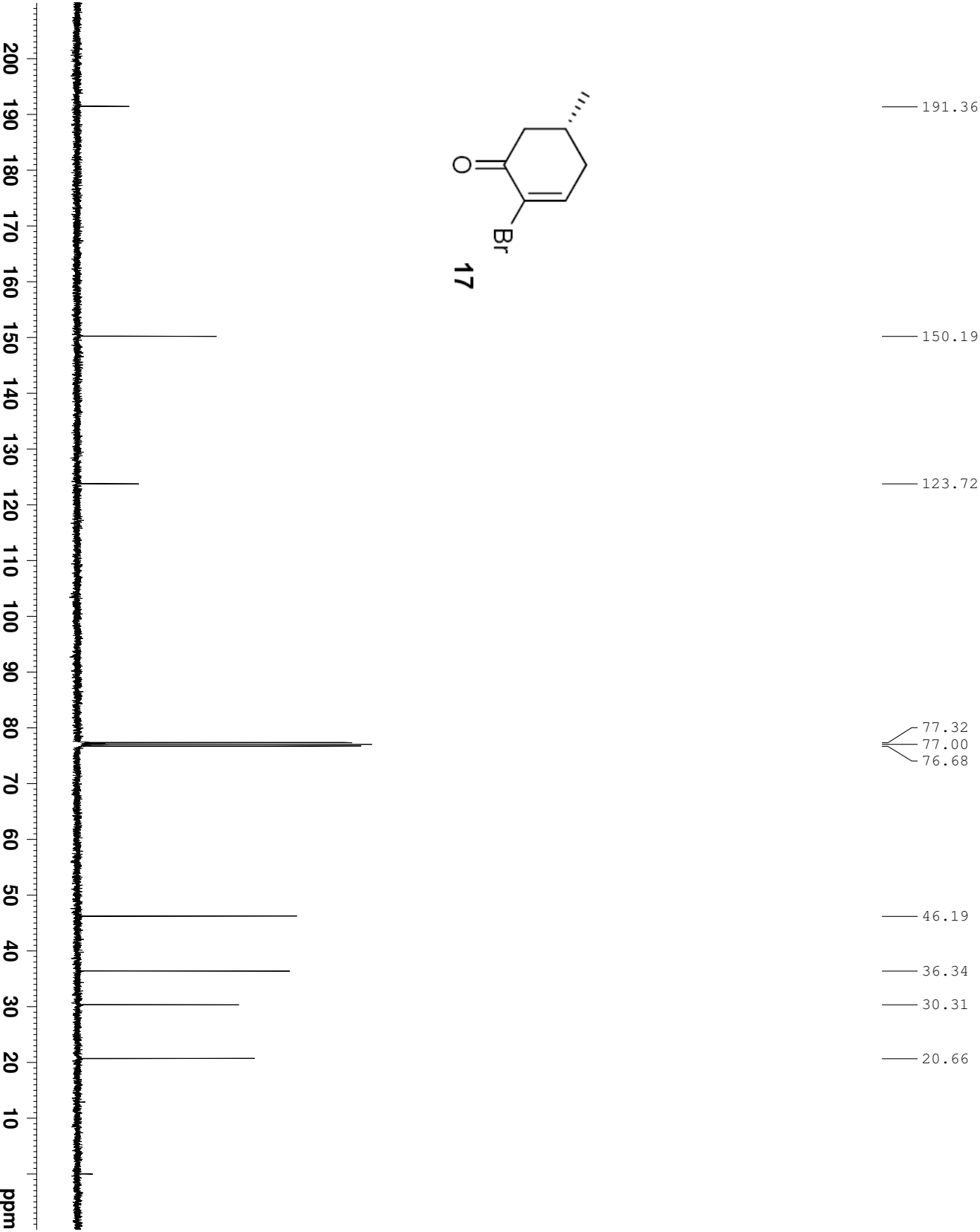
7.389
7.382
7.374
7.367
7.270

2.754
2.729
2.725
2.709
2.698
2.695
2.670
2.535
2.531
2.525
2.520
2.510
2.489
2.478
2.475
2.470
2.464
2.341
2.333
2.327
2.315
2.309
2.303
2.300
2.292
2.285
2.279
2.278
2.248
2.188
2.181
2.162
2.155
2.142
2.139
2.135
2.116
2.109
1.090
1.075
-0.007



NAME zxm-A-1
EXPNO 10
PROCNO 1
Date_ 20110413
Time 8.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 128
DW 60.800 usec
DE 6.50 usec
TE 294.6 K
D1 1.00000000 sec
TD0 1

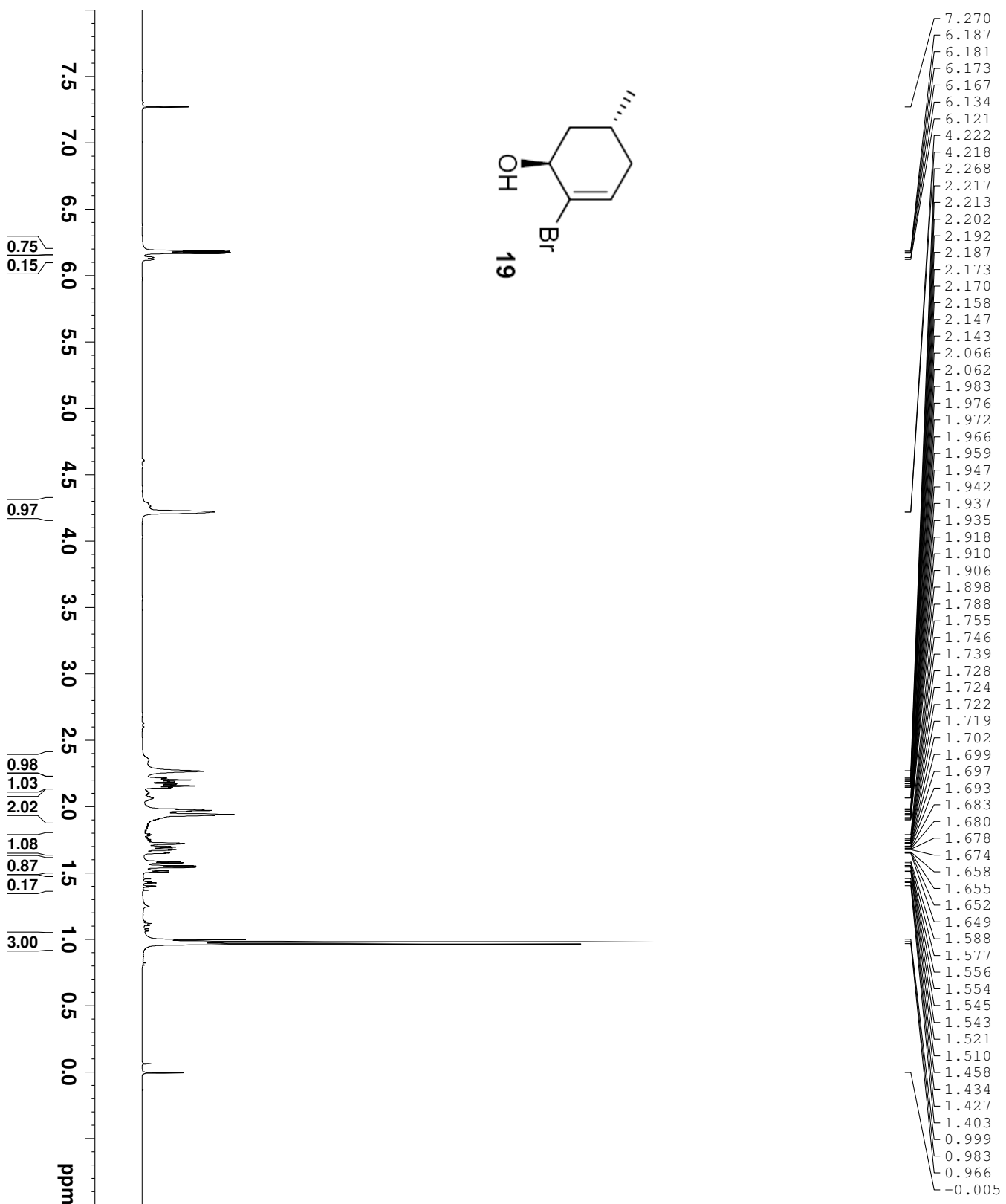
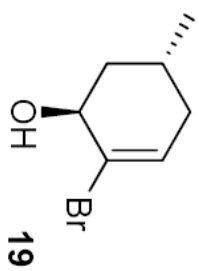
===== CHANNEL f1 =====
NUC1 1H
P1 14.70 usec
PL1 -1.00 dB
PL1W 13.75590801 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300011 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



```
NAME          zxm-A-1
EXPNO         11
PROCNO        1
Date_         20110413
Time_         8.38
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            100
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            57
DM            20.800 usec
DE            6.50 usec
TE            296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            9.70 usec
PL1           -2.00 dB
PL1W          56.13311005 W
SFO1          100.6228298 MHz

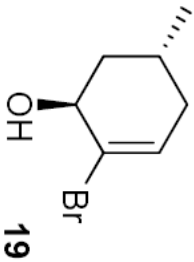
===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -2.10 dB
PL12          13.90 dB
PL13          13.90 dB
PL1W          17.72104263 W
PL2W          0.44513249 W
PL13W         0.44513249 W
SFO2          400.1316003 MHz
SI            32768
SF           100.6127720 MHz
SR            EM
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
```



```

NAME          zxm-sh-93
EXPNO         10
PROCNO        1
Date_         20110411
Time          9.41
INSTRUM       spect
PROBHD        5 mm PABBO BP-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            90.5
DW            60.800 usec
DE            6.50 usec
TE            290.0 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.70 usec
PL1          -1.00 dB
PL1W          13.75590801 W
SFO1          400.1324710 MHz
SI            32768
SF            400.1300013 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```



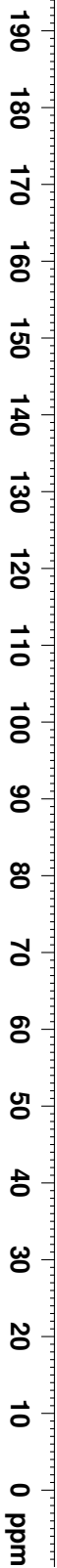
19

132.42
131.35
127.42
124.66

77.32
77.00
76.68
70.58
70.16

40.71
39.86
36.13
35.98

27.92
22.59
21.25
20.88



```

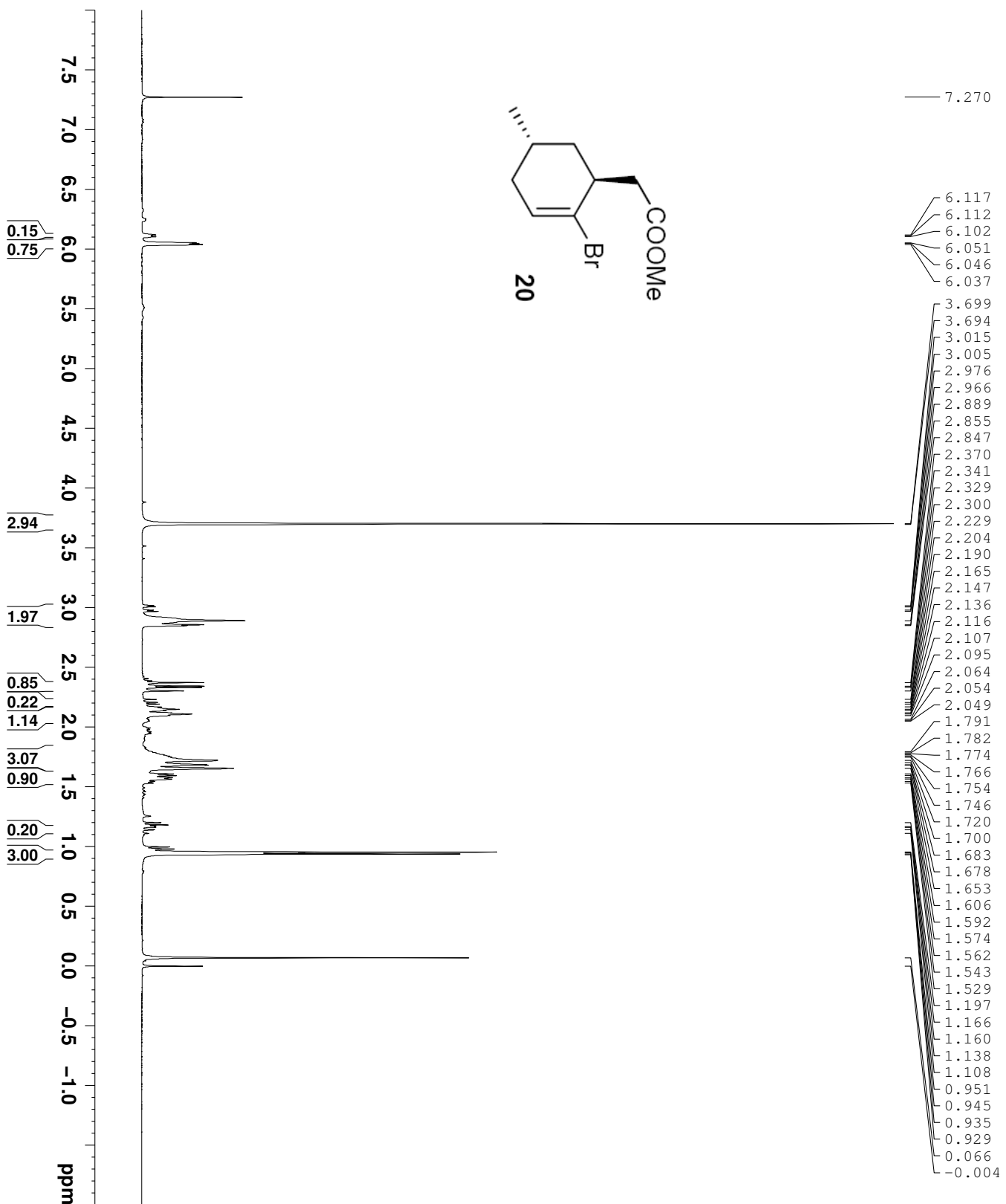
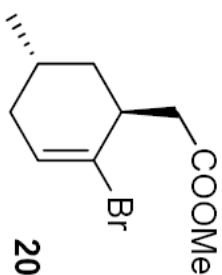
NAME          zxm-sh-93
EXPNO         11
PROCNO        1
Date_         20110411
Time_         9.49
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            100
DS            4
SWH           24038.461 Hz
FIDRES       0.366798 Hz
AQ           1.3631988 sec
RG            64
DM           20.800 usec
DE           6.50 usec
TE           291.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0          1
  
```

```

===== CHANNEL f1 =====
NUC1          13C
P1            9.70 usec
PL1          -2.00 dB
PL1W         56.13311005 W
SFO1         100.6228298 MHz
  
```

```

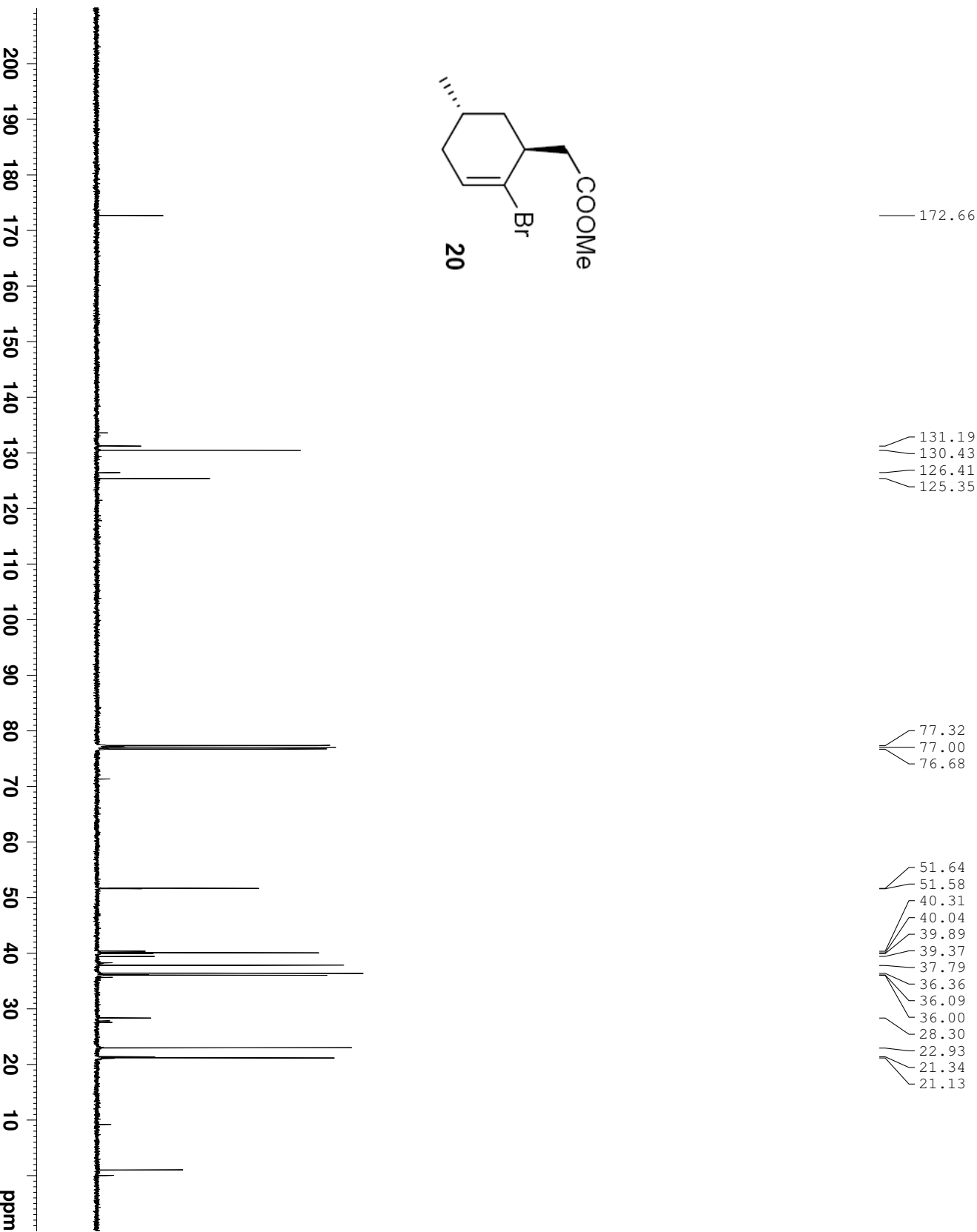
===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2          -2.10 dB
PL12         13.90 dB
PL13         13.90 dB
PL2W         17.72104263 W
PL12W        0.44513249 W
PL13W        0.44513249 W
SFO2         400.1316003 MHz
SI           32768
SF           100.6127734 MHz
WDW           EM
SSB           0
LB           1.00 Hz
GB           0
PC           1.40
  
```



```

NAME          zxm-A3-2
EXPNO         10
PROCNO        1
Date_         20110815
Time         12.47
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            71.8
DW            60.800 usec
DE            6.50 usec
TE            296.0 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.70 usec
PL1          -1.00 dB
PL1W         13.75590801 W
SFO1         400.1324710 MHz
SI           32768
SF           400.1300011 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           1.00
  
```

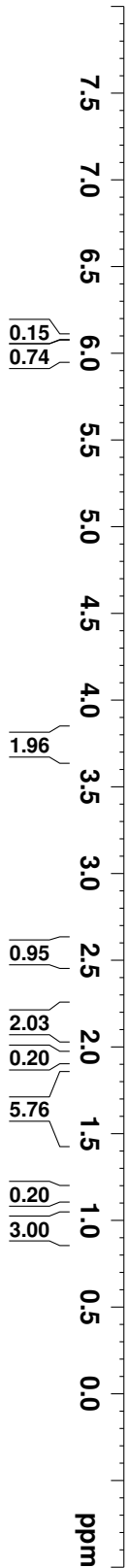
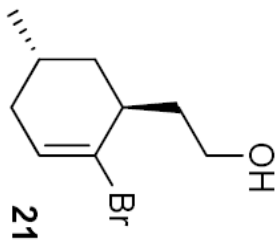
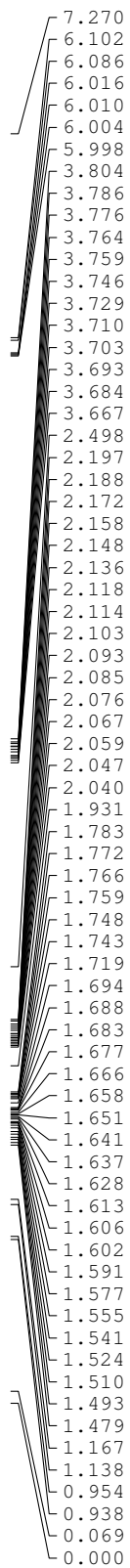


```

NAME          zxm-A3-2
EXPNO         11
PROCNO        1
Date_         20110815
Time          13.12
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            400
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            57
DM            20.800 usec
DE            6.50 usec
TE            297.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            9.70 usec
PL1           -2.00 dB
PL1W          56.13311005 W
SFO1          100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -2.10 dB
PL12          13.90 dB
PL13          13.90 dB
PL2W          17.72104263 W
PL12W         0.44513249 W
PL13W         0.44513249 W
SFO2          400.1316003 MHz
SI            32768
SF            100.6127700 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```



```

NAME                zxm-1151
EXPNO                10
PROCNO               1
Date_                20110421
Time                 0.08
INSTRUM              spect
PROBHD               5 mm PABBO BB-
PULPROG              zg30
TD                   65536
FIDRES               0.125483 Hz
AQ                   3.9846387 sec
RG                   90.5
DE                   60.800 usec
TE                   296.4 K
D1                   1.00000000 sec
TD0                  1

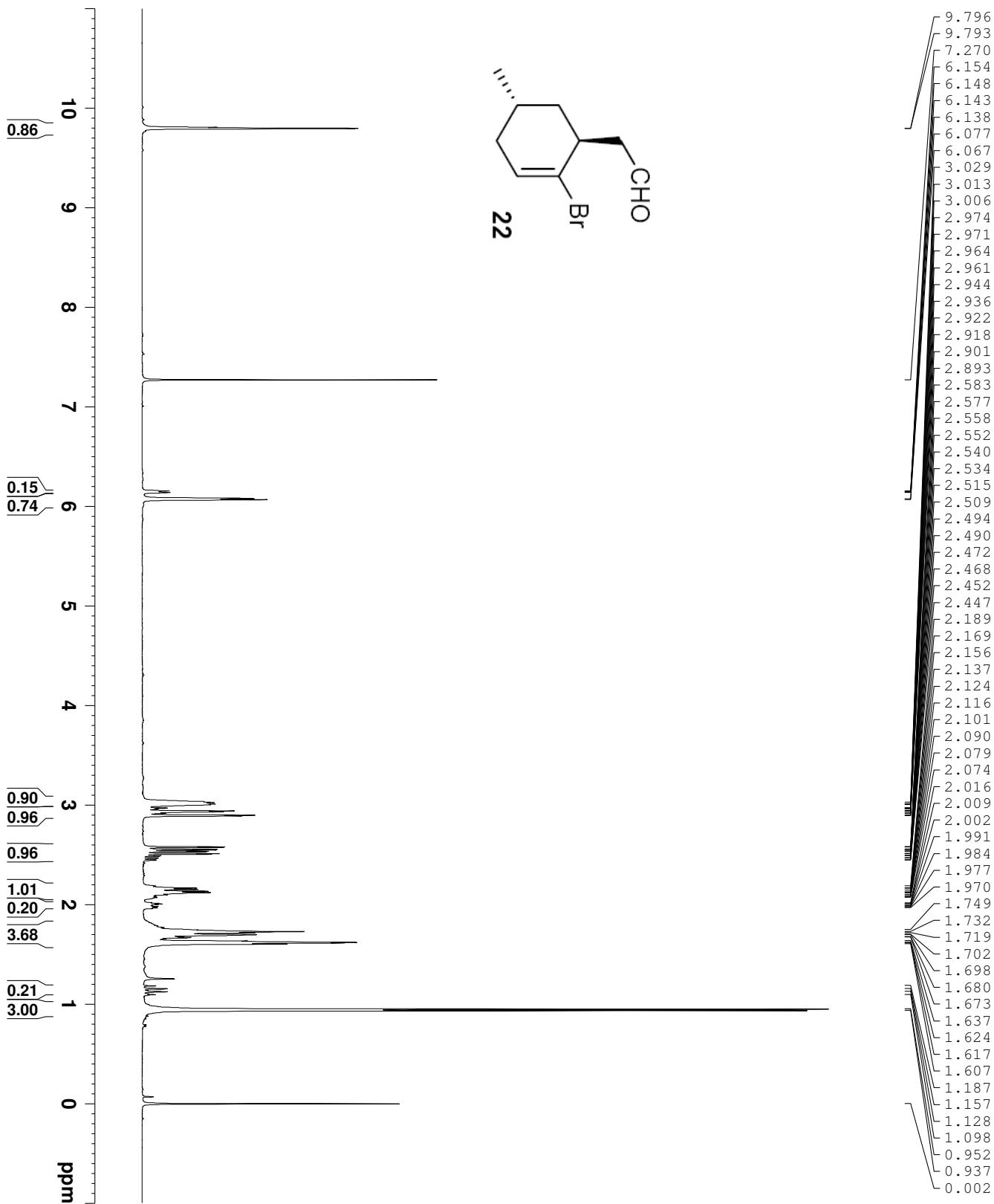
===== CHANNEL f1 =====
NUC1                 1H
P1                   14.70 usec
PL1                  -1.00 dB
PL1W                 13.7559801 W
SFO1                 400.1324710 MHz
SI                   32768
SF                   400.1300010 MHz
WDW                  EM
SSB                  0
LB                   0.30 Hz
GB                   0
PC                   1.00
  
```



NAME zxm-1151
EXPNO 11
PROCNO 1
Date_ 20110421
Time_ 0.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 170
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 645
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.70 usec
PL1 -2.00 dB
PL1W 56.13311005 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.10 dB
PL12 13.90 dB
PL13 13.90 dB
PL1W 17.72104263 W
PL2W 0.44513249 W
PL3W 0.44513249 W
SFO2 400.1316003 MHz
SI 32768
SF 100.6127707 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



```

NAME                zxm-1152
EXPNO                10
PROCNO               1
Date_                20110422
Time_                9.18
INSTRUM              spect
PROBHD               5 mm PABBO BB-
PULPROG              zg30
TD                   65536
FIDRES               0.125483 Hz
AQ                   3.9846387 sec
RG                   114
DW                   60.800 usec
DE                   6.50 usec
TE                   294.3 K
D1                   1.00000000 sec
TD0                  1

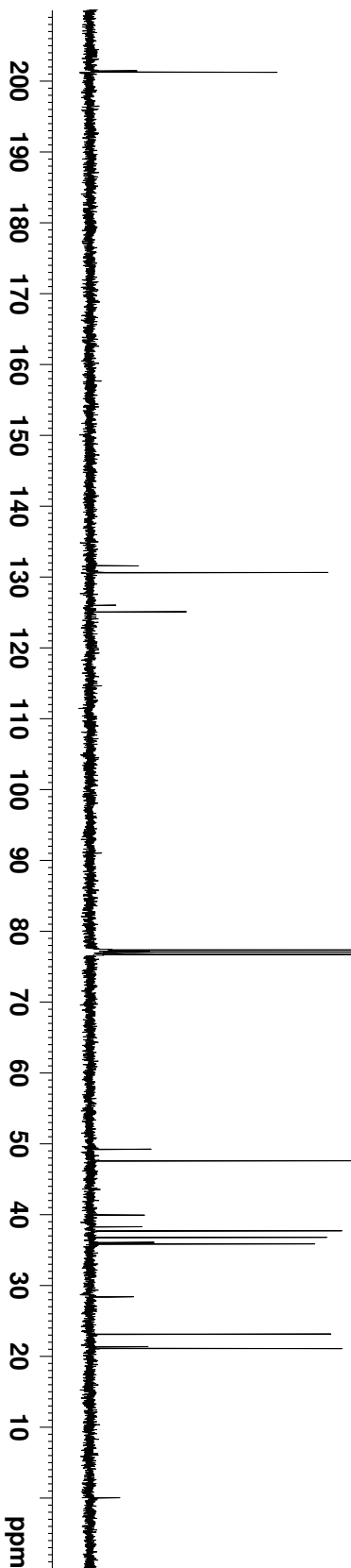
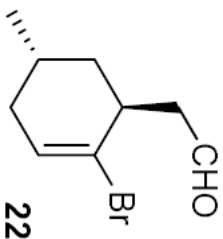
===== CHANNEL f1 =====
NUC1                 1H
P1                   14.70 usec
PL1                  -1.00 dB
PL1W                 13.75590801 W
SFO1                 400.1324710 MHz
SI                   32768
SF                   400.1300013 MHz
WDW                  EM
SSB                  0
LB                   0.30 Hz
GB                   0
PC                   1.00
  
```

201.34
201.16

131.55
130.59
125.98
125.06

77.32
77.00
76.68

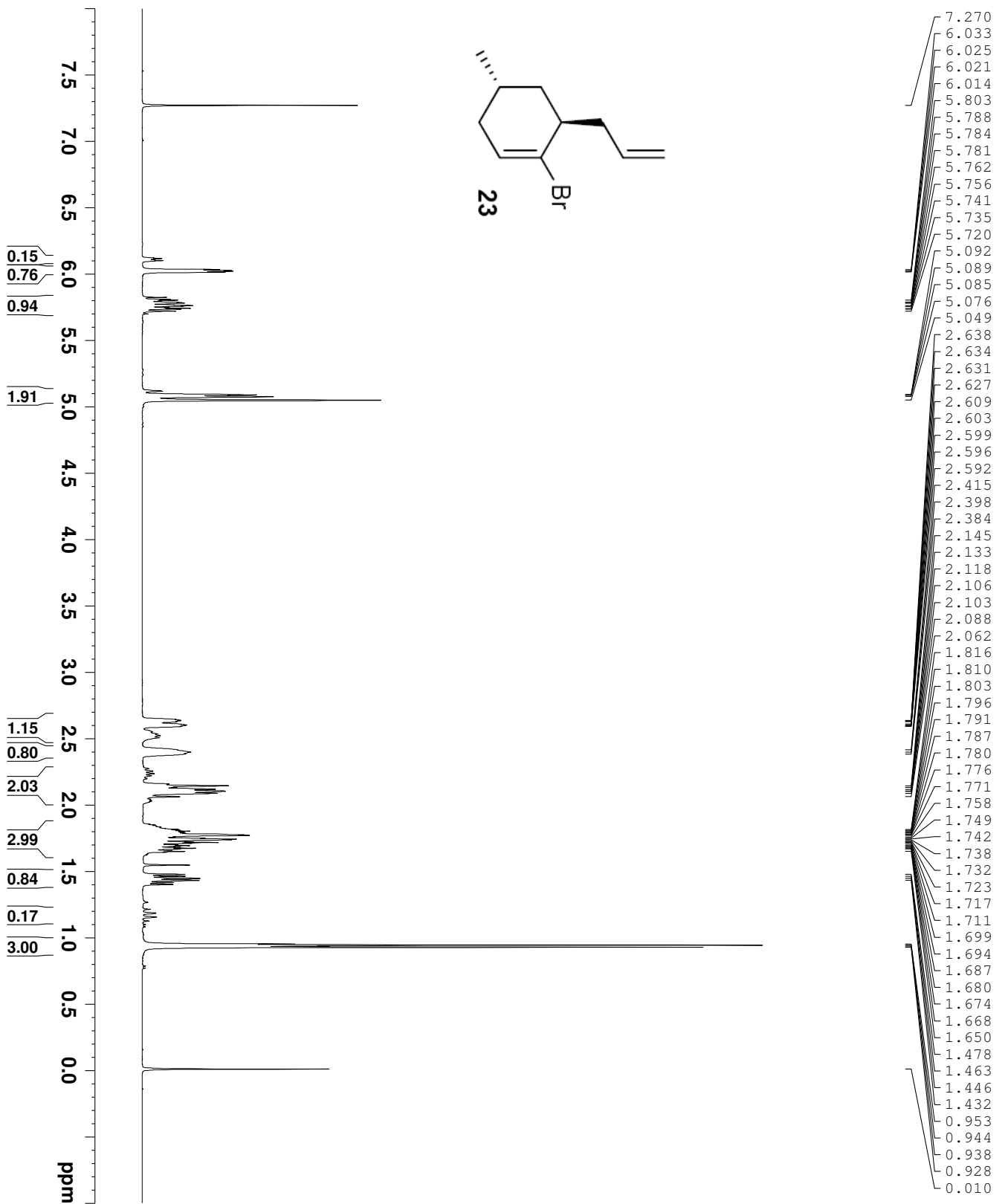
49.18
47.54
39.89
38.23
37.66
36.74
36.02
35.82
28.33
23.08
21.30
21.04



NAME zxm-1152
EXPNO 11
PROCNO 1
Date_ 20110422
Time 9.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 57
DM 20.800 usec
DE 6.50 usec
TE 295.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.70 usec
PL1 -2.00 dB
PL1W 56.13311005 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.10 dB
PL12 13.90 dB
PL13 13.90 dB
PL2W 17.72104263 W
PL12W 0.44513249 W
PL13W 0.44513249 W
SFO2 400.1316003 MHz
SI 32768
SF 100.6127700 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



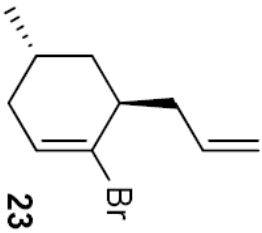
```

NAME          zxm-sh-133
EXPNO         10
PROCNO        1
Date_         20110615
Time          15.48
INSTRUM       spect
PROBHD        5 mm PABBO BP-
PULPROG       zg30
TD            65536
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            144
DW            60.800 usec
DE            6.50 usec
TE            294.7 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            12.00 usec
PL1           -3.00 dB
PL1W          22.90425682 W
SFO1          400.1324710 MHz
SI            32768
SF            400.1300008 MHz
WDW            EM
SSB            0
LB            0.30 Hz
GB            0
PC            1.00

```

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



136.74
135.50
130.85
129.40
128.30
127.22
117.00
116.56

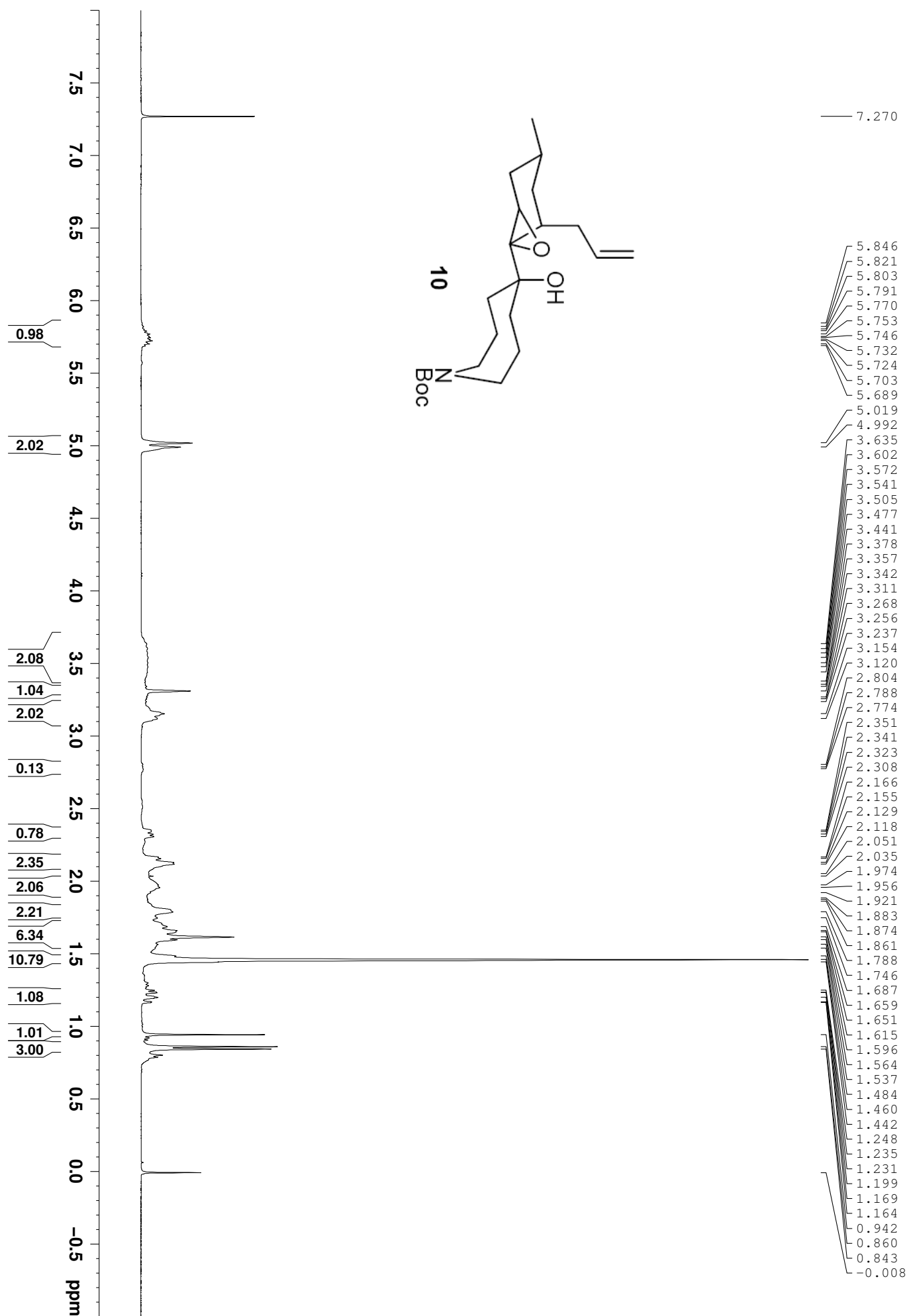
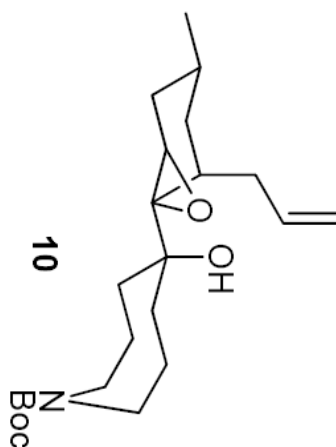
77.32
77.00
76.68

42.65
42.18
39.16
38.87
37.44
36.28
36.08
35.22
28.43
22.91
21.51
21.23

NAME zxm-sh-133
EXPNO 11
PROCNO 1
Date_ 20110615
Time 16.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 530
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DM 20.800 usec
DE 6.50 usec
TE 295.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.40 usec
PL1 -2.00 dB
SFO1 57.32743073 W
100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.50 dB
PL13 15.50 dB
PL2W 18.19349861 W
PL12W 0.32353121 W
PL13W 0.32353121 W
SFO2 400.1316003 MHz
SI 32768
SF 100.6127692 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

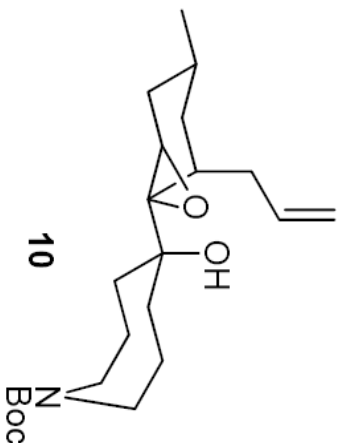


155.91
155.63

137.16

116.28
115.81

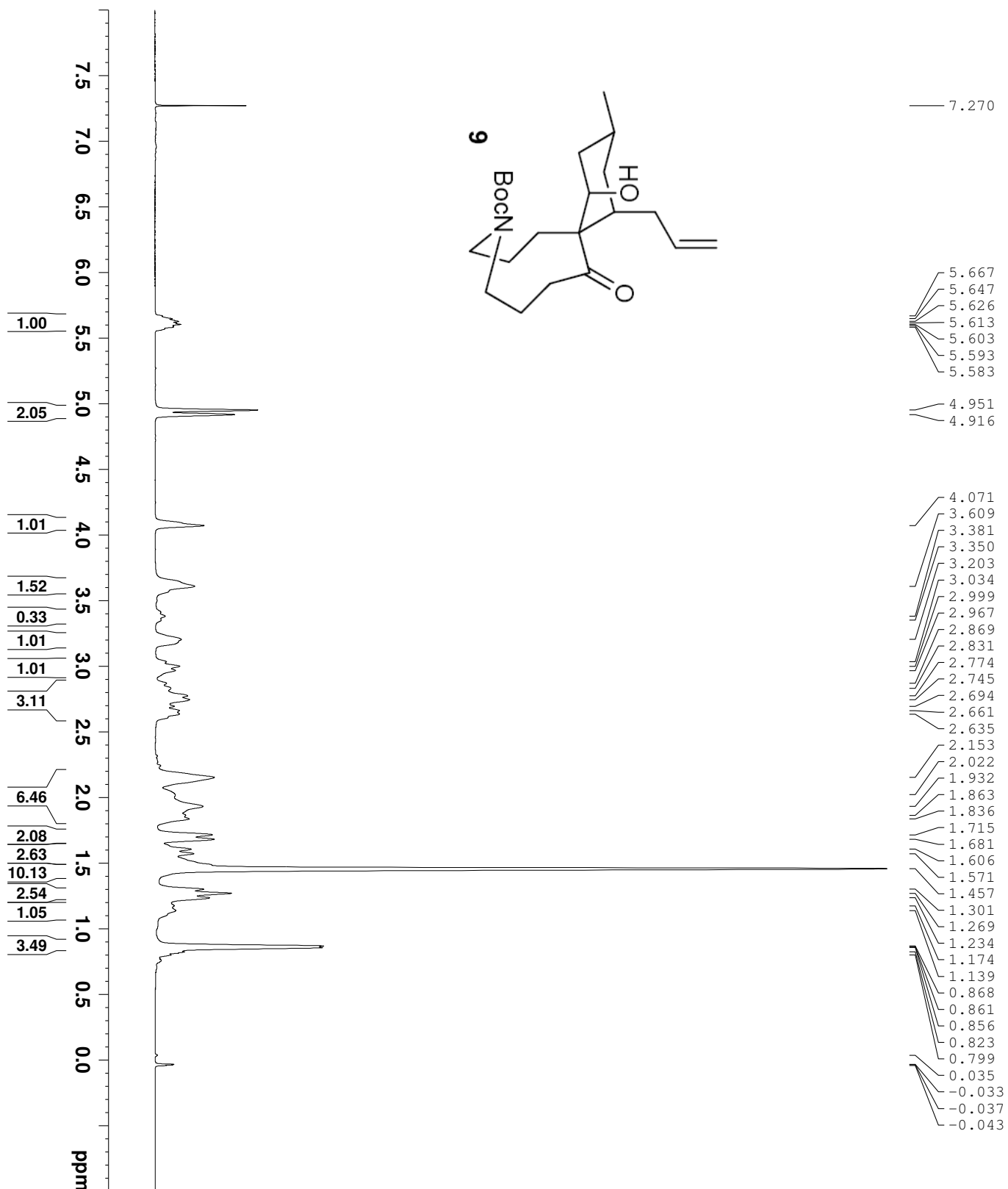
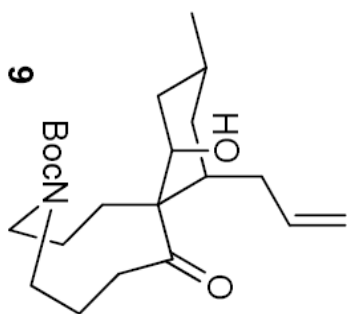
79.18
79.10
77.32
77.00
76.68
75.27
73.37
67.69
58.29
56.94
56.74
46.99
46.89
46.68
46.28
39.21
37.85
35.38
34.98
34.15
33.86
33.72
33.57
33.12
32.94
32.59
32.38
31.85
28.52
28.49
27.91
25.80
21.76
21.54
20.18



NAME zxm-sh-135-2
EXPNO 20
PROCNO 1
Date_ 20110621
Time 11.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1100
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 64
DM 20.800 usec
DE 6.50 usec
TE 296.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.70 usec
PL1 -2.00 dB
PL1W 56.13311005 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.10 dB
PL12 13.90 dB
PL13 13.90 dB
PL2W 17.72104263 W
PL13W 0.44513249 W
SFO2 400.1316003 MHz
SI 32768
SF 100.6127703 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



```

NAME          zxm-sh-131
EXPNO         10
PROCNO        1
Date_         20110611
Time          23.45
INSTRUM       spect
PROBHD        5 mm PABBO BP-
PULPROG       zg30
TD            2930
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            32
DE            60.800 usec
TE            294.8 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.70 usec
PL1          -1.00 dB
PL1W         13.7559801 W
SFO1         400.1324710 MHz
SI           32768
SF           400.1300006 MHz
WDW           EM
SSB           0
LB           0.30 Hz
GB           0
PC           1.00
  
```

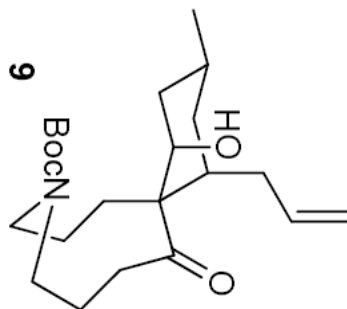
219.82

157.03
156.07

138.19

115.71

79.71
79.58
77.37
77.05
76.73
68.67
59.56
59.17
48.70
47.59
45.23
43.78
40.12
39.89
36.41
36.36
32.06
31.51
31.28
29.48
28.48
23.03
22.06
21.76
21.13
20.15

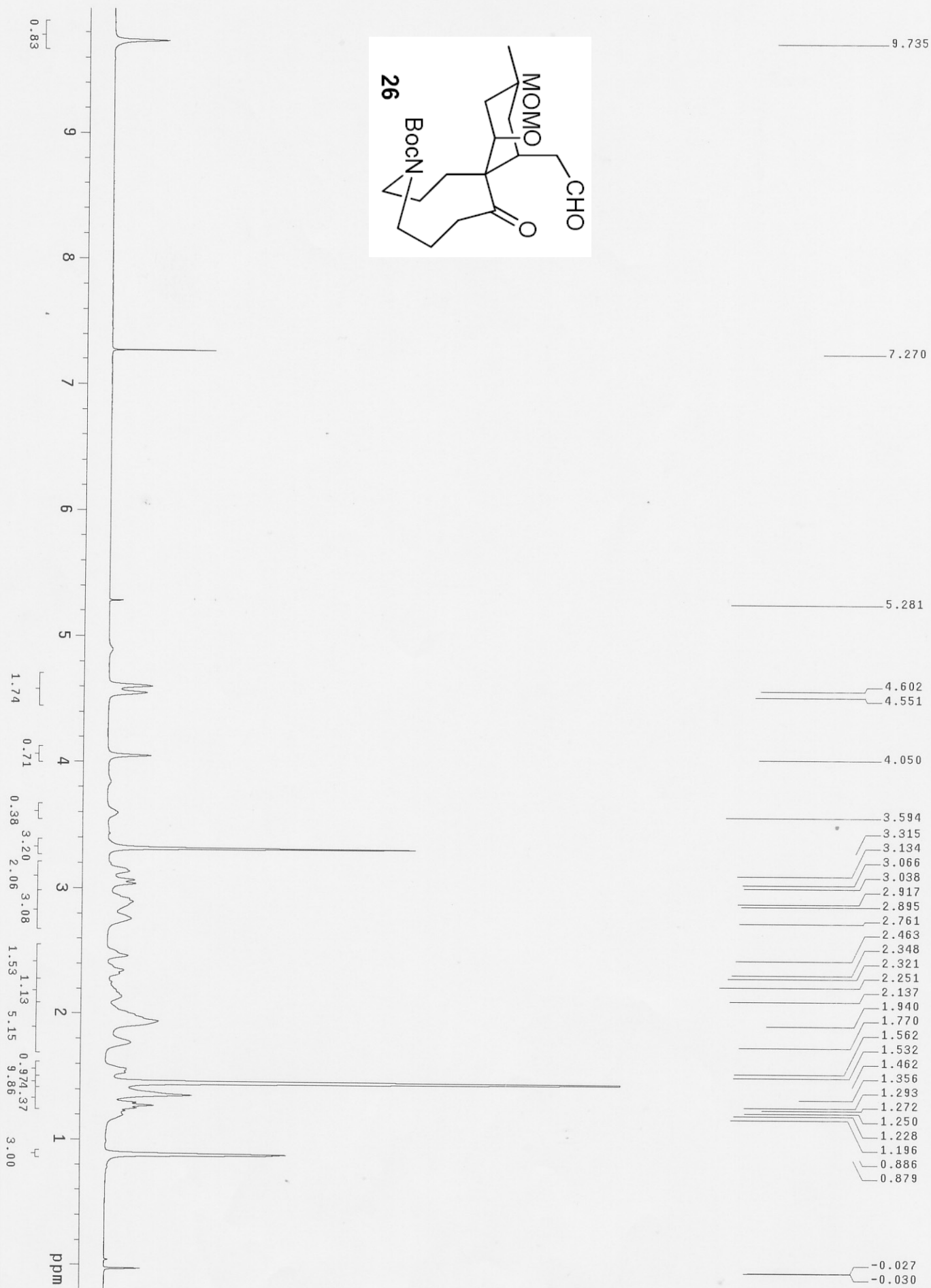
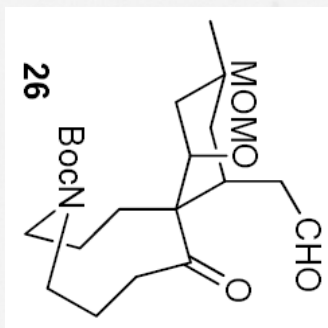


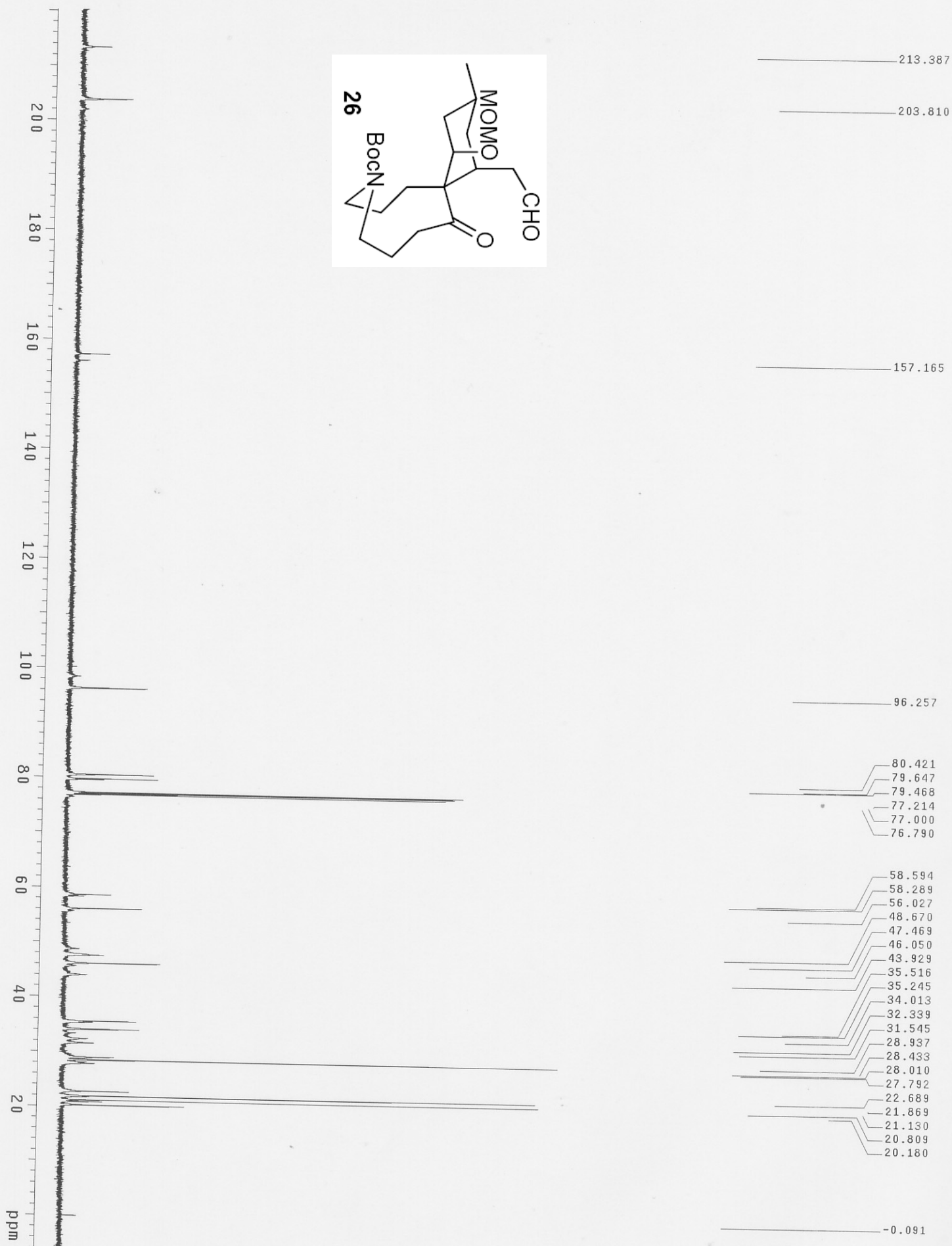
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

```
NAME zxm-sh-131
EXPNO 11
PROCNO 1
Date_ 20110612
Time 0.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 4
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 512
DW 19.800 usec
DE 6.50 usec
TE 296.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

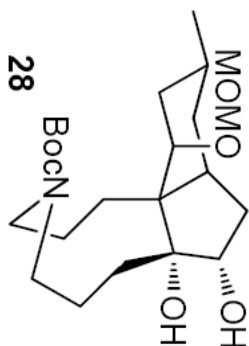
===== CHANNEL f1 =====
NUC1 13C
P1 9.70 usec
PL1 -2.00 dB
PL1W 56.13311005 W
SFO1 100.6238364 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.10 dB
PL12 13.90 dB
PL13 13.90 dB
PL2W 17.72104263 W
PL12W 0.44513249 W
PL13W 0.44513249 W
SFO2 400.1316003 MHz
SI 400.6127742 MHz
SF 32768
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
```





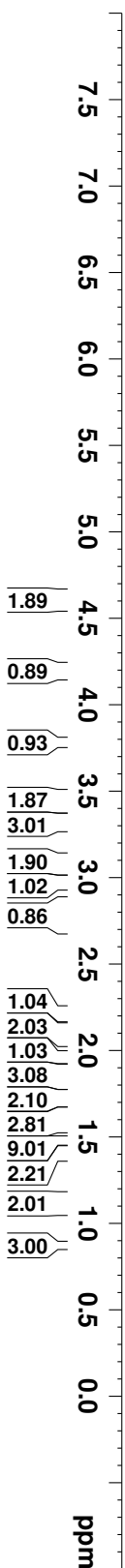
7.270



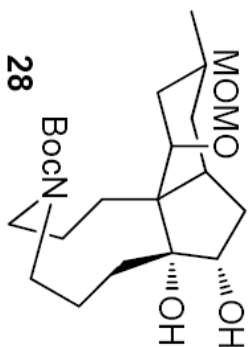
4.638
4.622
4.596
4.579
4.205
4.183
3.783
3.433
3.385
3.355
3.350
3.097
3.057
2.998
2.969
2.938
2.821
2.215
2.203
2.105
2.077
2.047
2.020
1.935
1.874
1.829
1.792
1.754
1.727
1.696
1.629
1.599
1.549
1.514
1.474
1.470
1.430
1.399
1.367
1.161
1.147
1.130
1.116
1.099
1.067
0.874
0.858
0.007
0.011

NAME zxm-sh-155
EXPNO 10
PROCNO 1
Date_ 20110718
Time 15.58
INSTRUM spect
PROBHD 5 mm PABBO BP-
PULPROG zg30
TD 65536
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 57
DW 60.800 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.70 usec
PL1 -1.00 dB
PL1W 13.75590801 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300008 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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



157.16

97.03

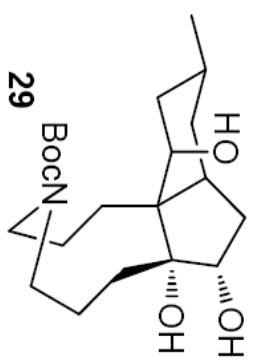
82.19
81.77
79.87
77.32
77.00
76.68

56.16
52.24
50.15
49.55
41.29
37.23
35.36
32.89
30.49
28.43
28.25
23.39
22.36
22.18
20.12

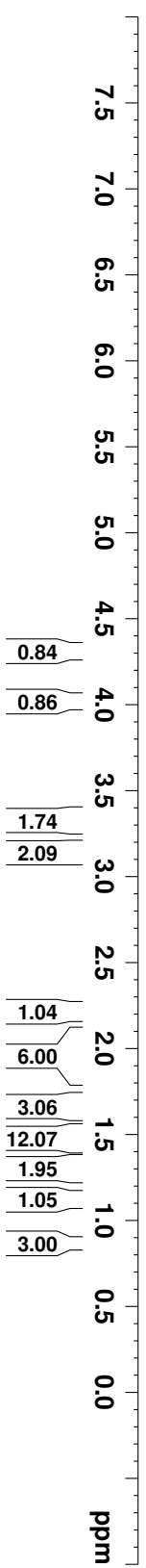
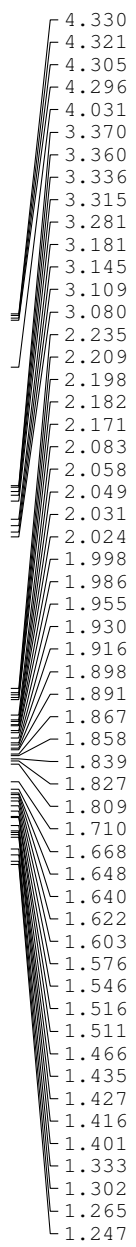
NAME	zxm-sh-155
EXPNO	11
PROCNO	1
Date_	20110718
Time	16.43
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	750
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	812
DW	20.800 usec
DE	6.50 usec
TE	296.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

===== CHANNEL f1 =====	
NUC1	13C
P1	9.70 usec
PL1	-2.00 dB
PL1W	56.13311005 W
SFO1	100.6228298 MHz

===== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-2.10 dB
PL12	13.90 dB
PL13	13.90 dB
PL2W	17.72104263 W
PL12W	0.44513249 W
PL13W	0.44513249 W
SFO2	400.1316003 MHz
SI	32768
SF	100.6127709 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



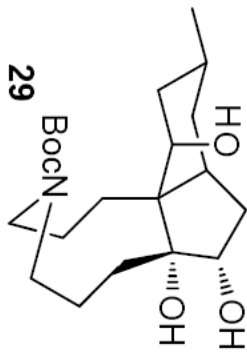
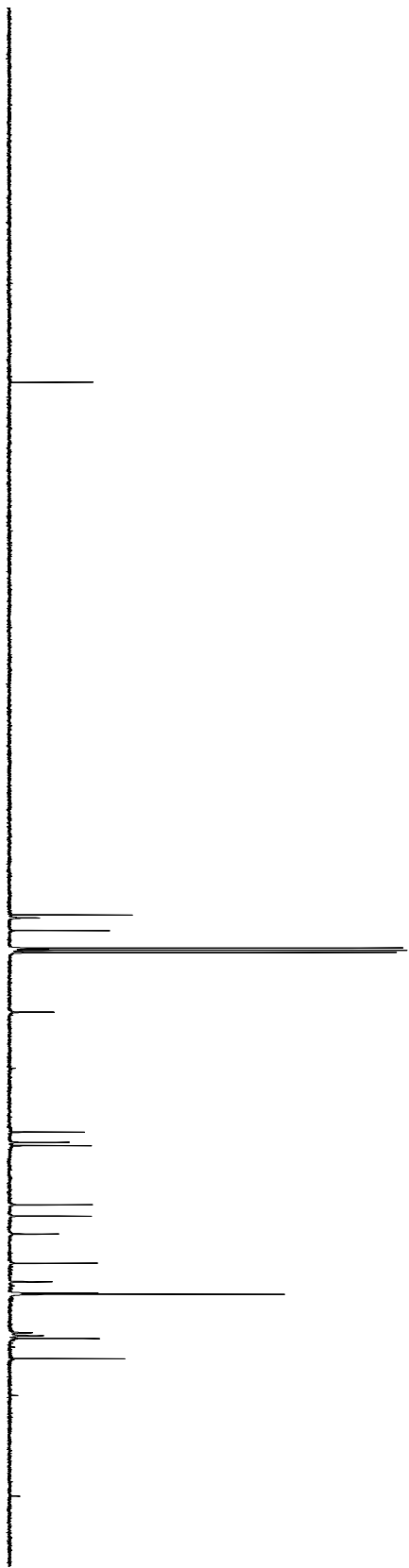
7.270



NAME zxm-sh-158
EXPNO 10
PROCNO 1
Date_ 20110720
Time 11.42
INSTRUM spect
PROBHD 5 mm PABBO BP-
PULPROG zg30
TD 65536
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 71.8
DW 60.800 usec
DE 6.50 usec
TE 297.3 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 -3.00 dB
PL1W 22.90425682 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300006 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

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



157.19

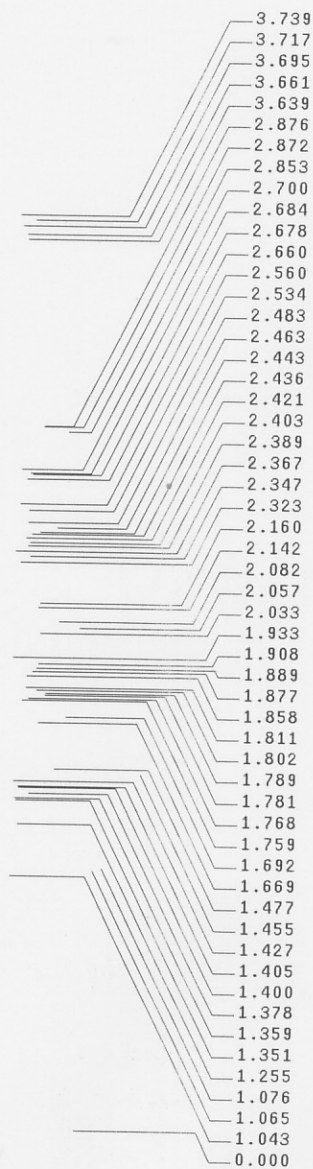
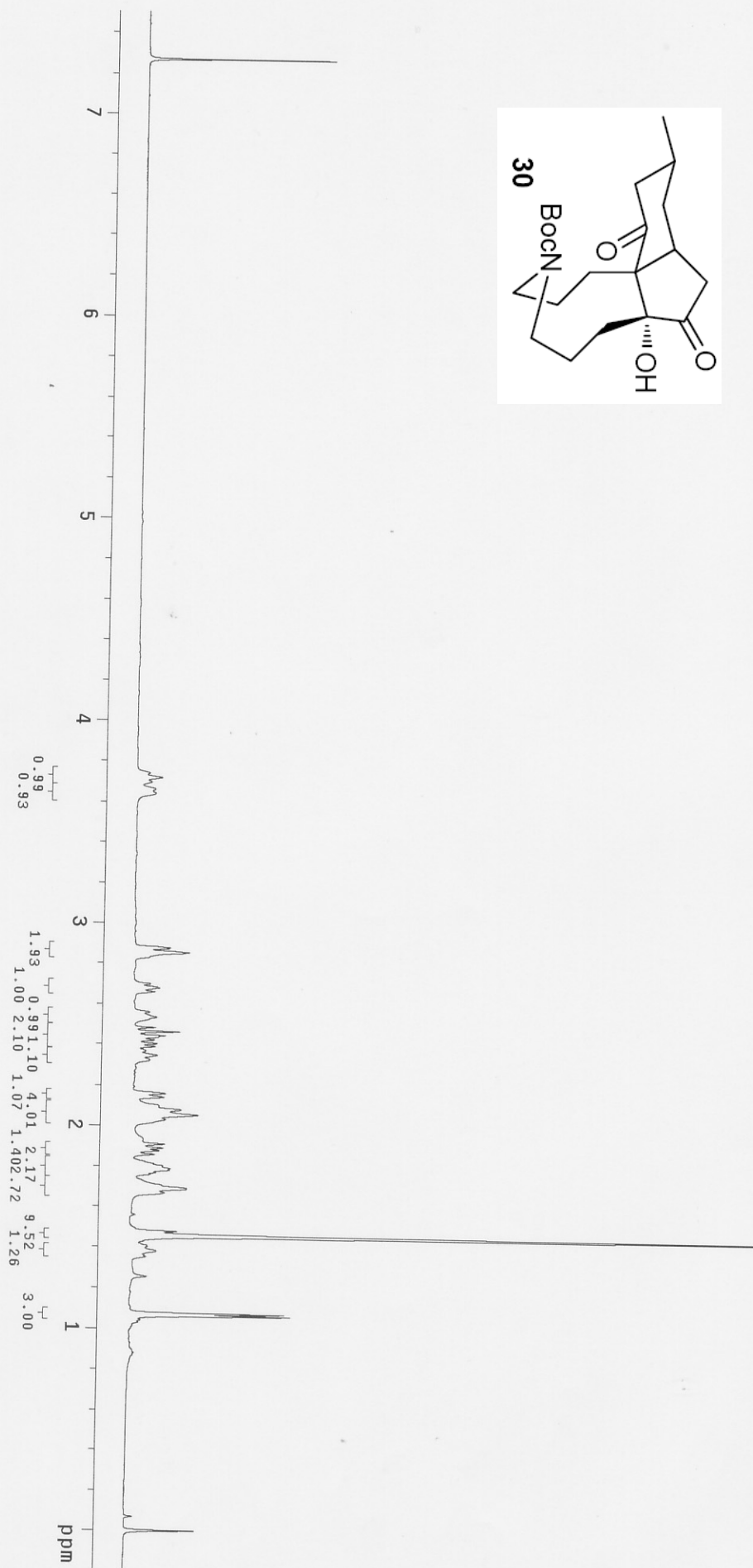
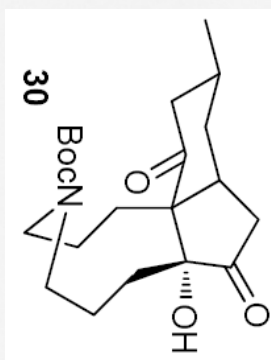
82.01
81.57
79.79
77.32
77.00
76.68
68.28

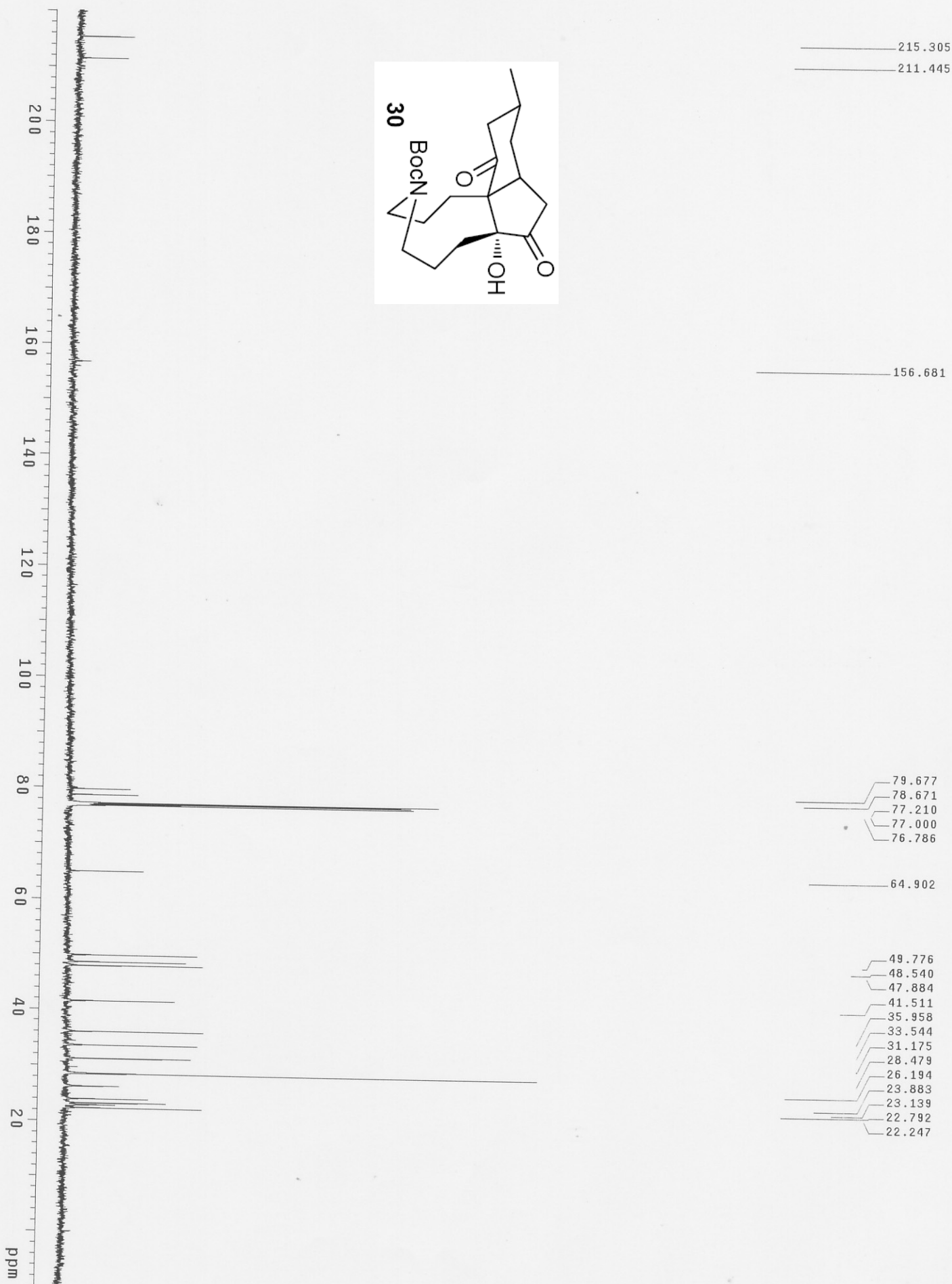
51.33
49.88
49.41
41.07
39.48
36.96
32.82
30.21
28.63
28.44
23.01
22.58
22.21
19.35

NAME	zxm-sh-158
EXPNO	11
PROCNO	1
Date_	20110720
Time	12.45
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1100
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	2050
DW	20.800 usec
DE	6.50 usec
TE	296.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

===== CHANNEL f1 =====	
NUC1	13C
P1	9.40 usec
PL1	-2.00 dB
PL1W	57.32743073 W
SFO1	100.6228298 MHz

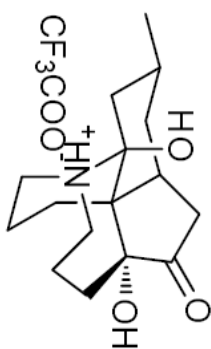
===== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-2.00 dB
PL12	15.50 dB
PL13	15.50 dB
PL2W	18.19349861 W
PL12W	0.32353121 W
PL13W	0.32353121 W
SFO2	400.1316003 MHz
SI	32768
SF	100.6127715 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



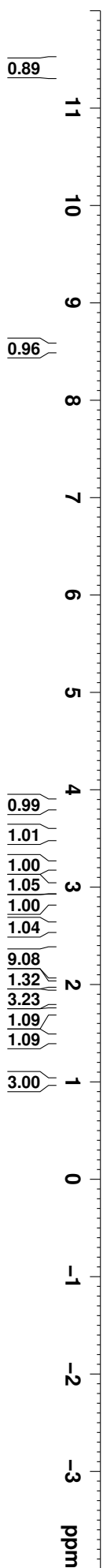


11.422

8.539
7.270
3.877
3.850
3.843
3.815
3.574
3.539
3.503
3.243
3.231
3.209
3.197
3.002
2.967
2.904
2.870
2.864
2.829
2.622
2.616
2.593
2.586
2.578
2.556
2.550
2.365
2.349
2.330
2.313
2.296
2.279
2.259
2.237
2.218
2.196
2.185
2.162
2.135
2.114
2.084
2.071
2.047
2.033
2.002
1.970
1.935
1.922
1.900
1.887
1.856
1.845
1.812
1.800
1.744
1.740
1.736
1.708
1.704
1.478
1.464
1.446
1.442
1.429
1.410
1.396
1.007
0.991
0.002



Alopecuridine·TFA 31

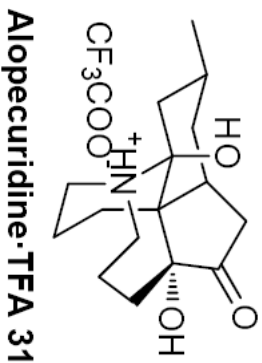


215.64

95.05

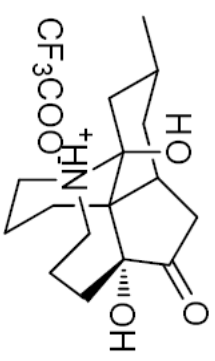
80.42
77.32
77.00
76.68

53.97
50.07
49.77
41.27
40.66
40.44
36.05
31.35
24.98
23.83
21.89
21.45
19.24



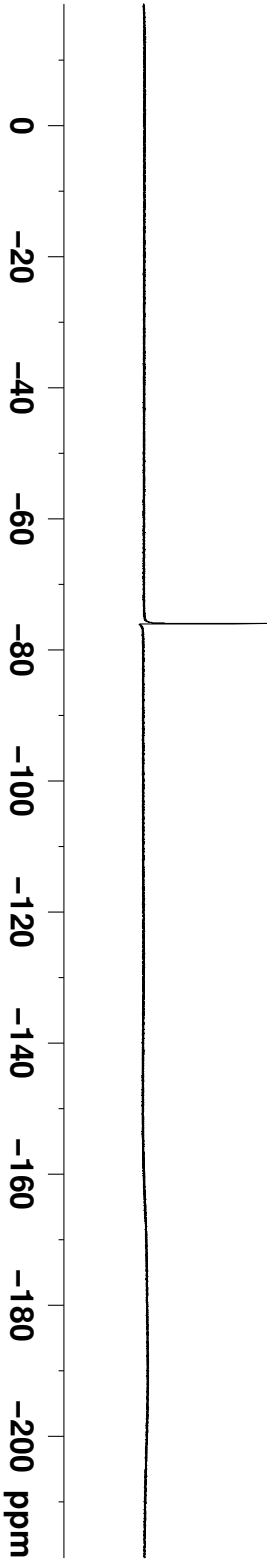
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

NAME	zxm-1161
EXPNO	11
PROCNO	1
Date_	20110718
Time	11.30
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
ID	65536
SOLVENT	CDCl3
NS	1100
DS	4
SWH	26041.666 Hz
FIDRES	0.397364 Hz
AQ	1.2383412 sec
RG	1030
DW	19.200 usec
DE	6.50 usec
TE	296.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
===== CHANNEL f1 =====	
NUC1	13C
P1	9.70 usec
PL1	-2.00 dB
PL1W	56.13311005 W
SFO1	100.6238364 MHz
===== CHANNEL f2 =====	
CEPPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-2.10 dB
PL12	13.90 dB
PL13	13.90 dB
PL12W	17.72104263 W
PL13W	0.44513249 W
SFO2	400.1316003 MHz
SI	32768
SF	100.6127689 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



Alopecuridine-TFA 31

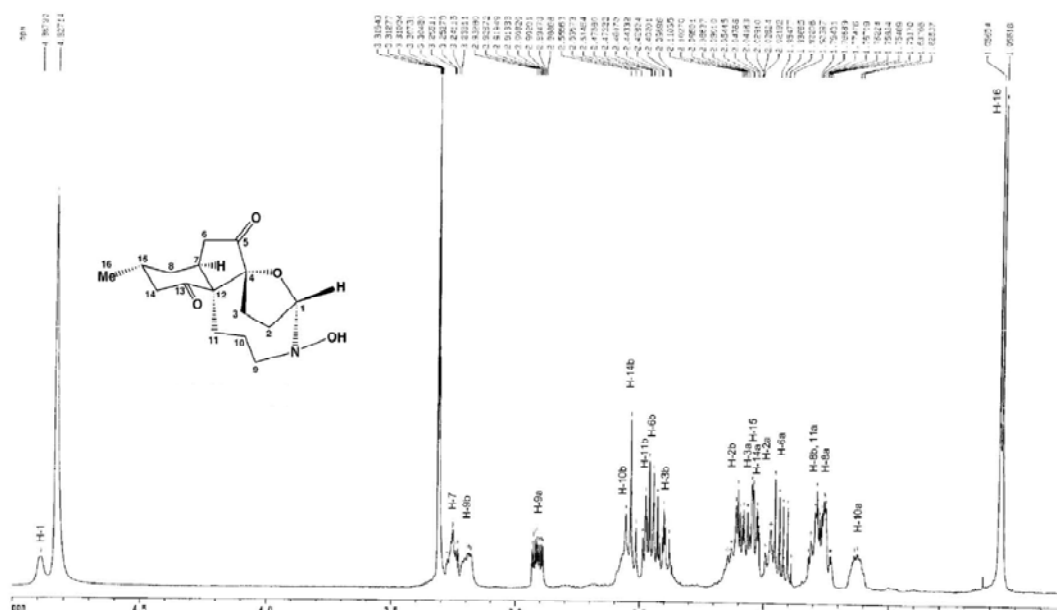
— -75.97



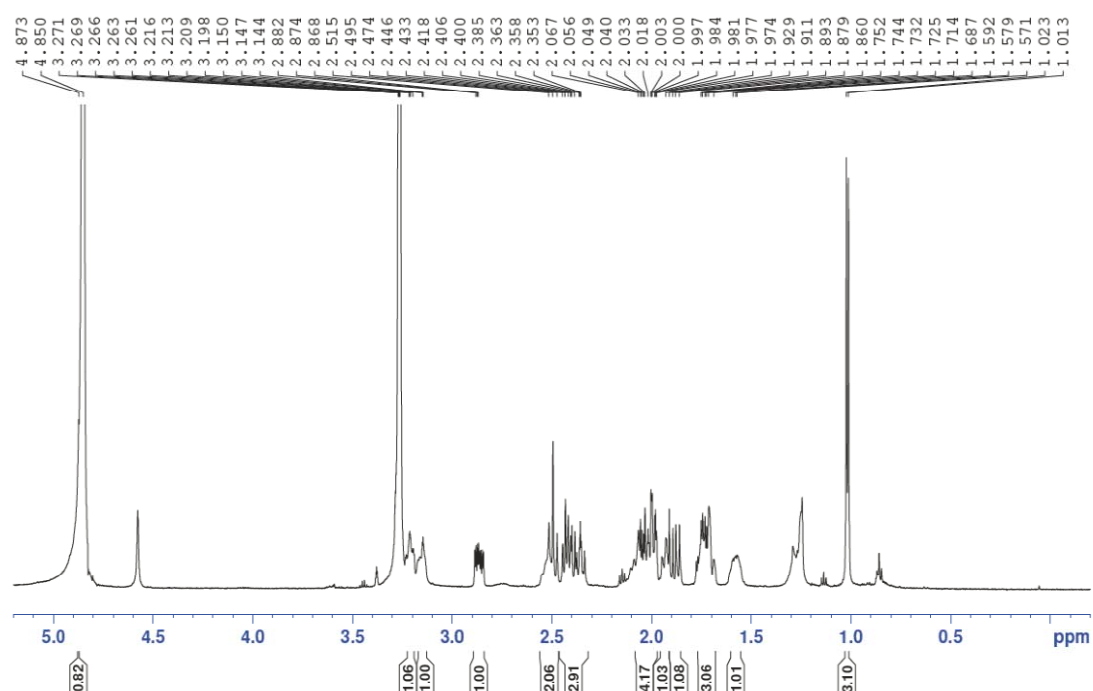
NAME	zxm-f
EXPNO	10
PROCNO	1
Date_	20120824
Time	3.20
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgfg1q
TD	131072
SOLVENT	CDCl3
NS	30
DS	4
SWH	89285.711 Hz
FIDRES	0.681196 Hz
AQ	0.7340532 sec
RG	2050
DW	5.600 usec
DE	6.50 usec
TE	295.7 K
D1	1.00000000 sec
TD0	1

===== CHANNEL f1 =====	
NUC1	19F
P1	15.00 usec
PL1	-3.20 dB
PL1W	17.63353157 W
SFO1	376.4607164 MHz
SI	65536
SF	376.4983660 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

¹H NMR of natural and synthetic sieboldine A (2)

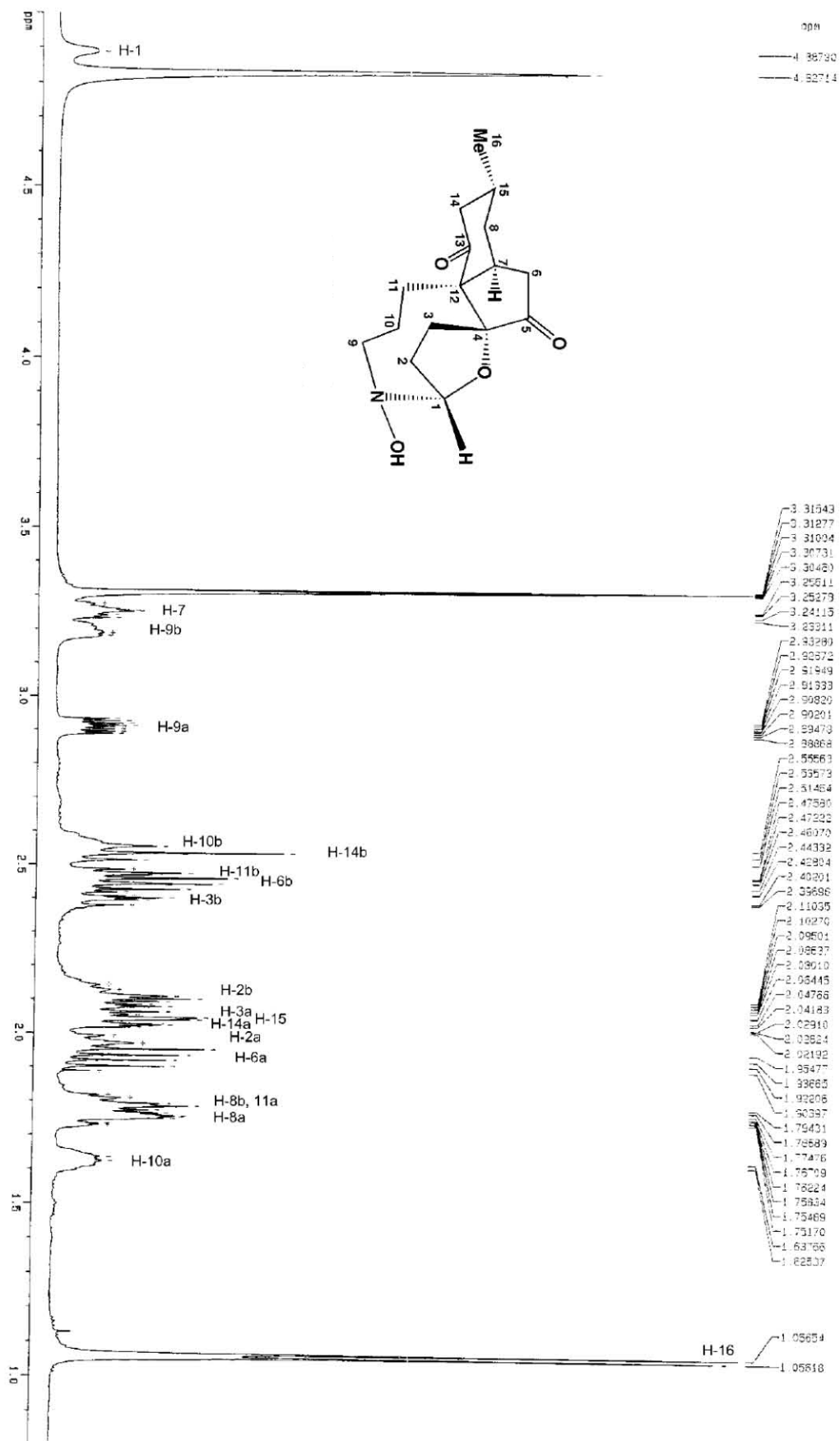


Natural sieboldine A (2), see: Y. Hirasawa, H. Morita, M. Shiro and J. Kobayashi, *Org. Lett.* 2003, **5**, 3991.

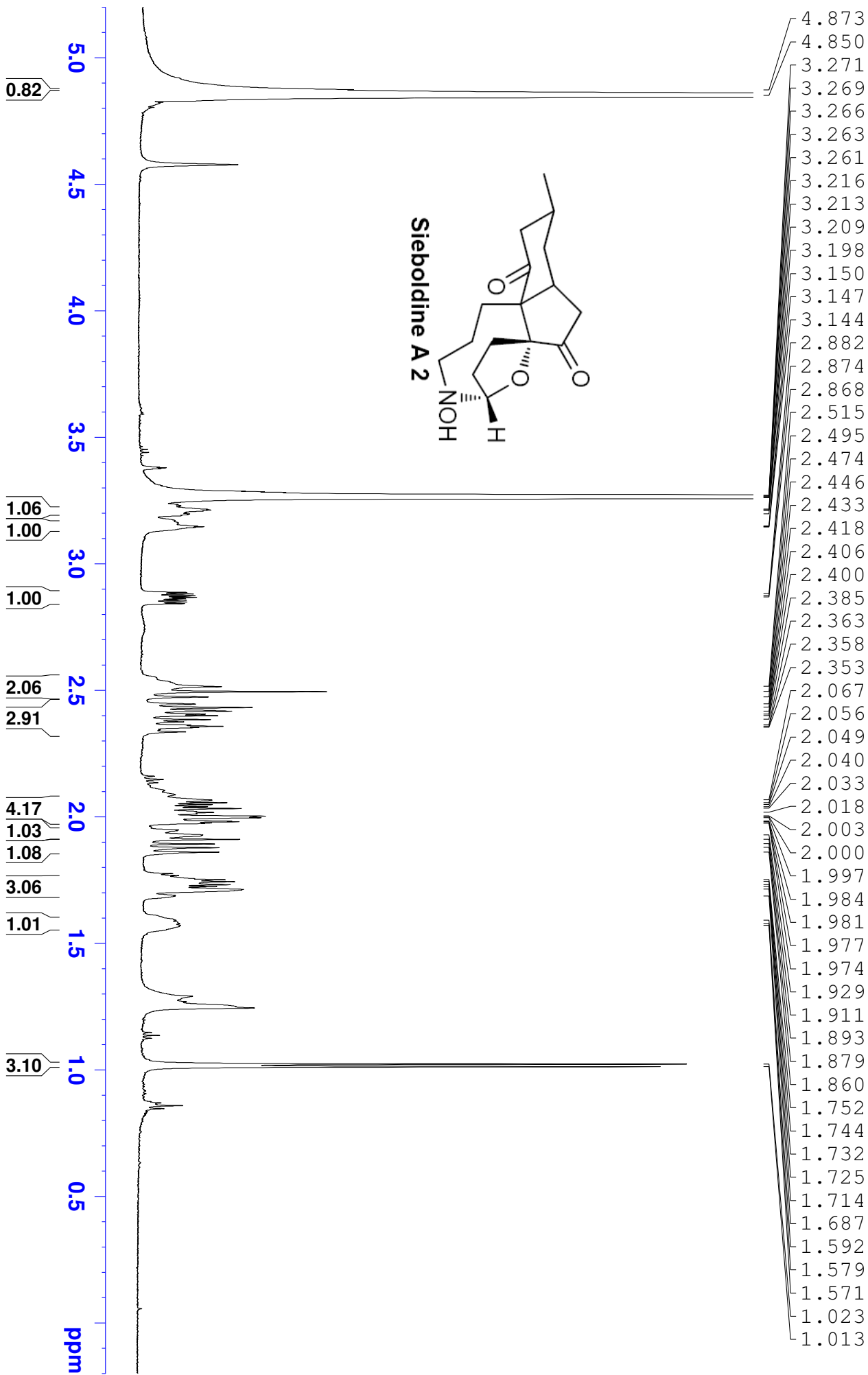


Our synthetic sieboldine A (2).

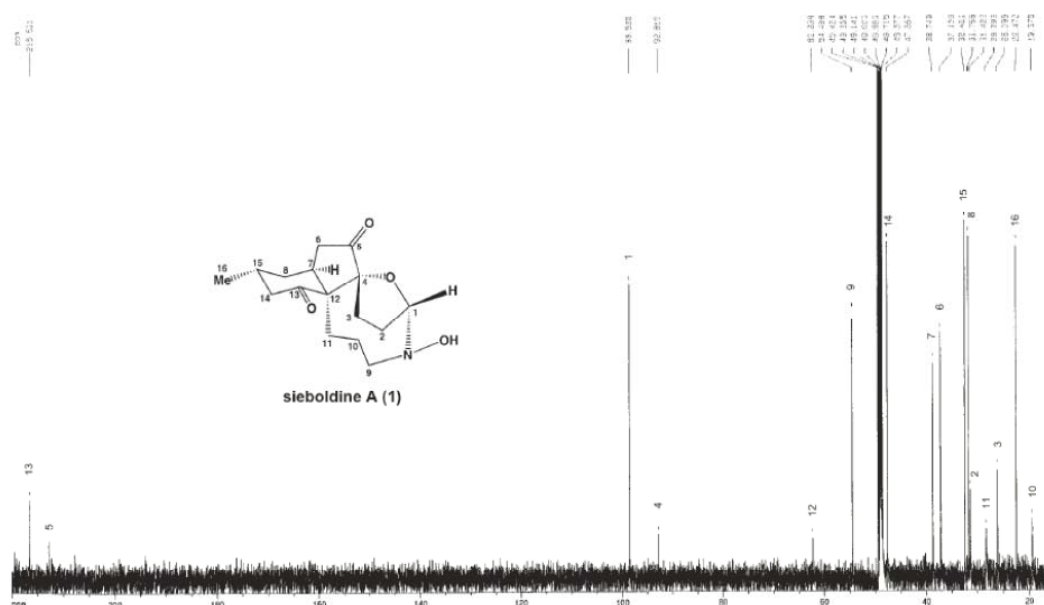
Natural Sieboldine A



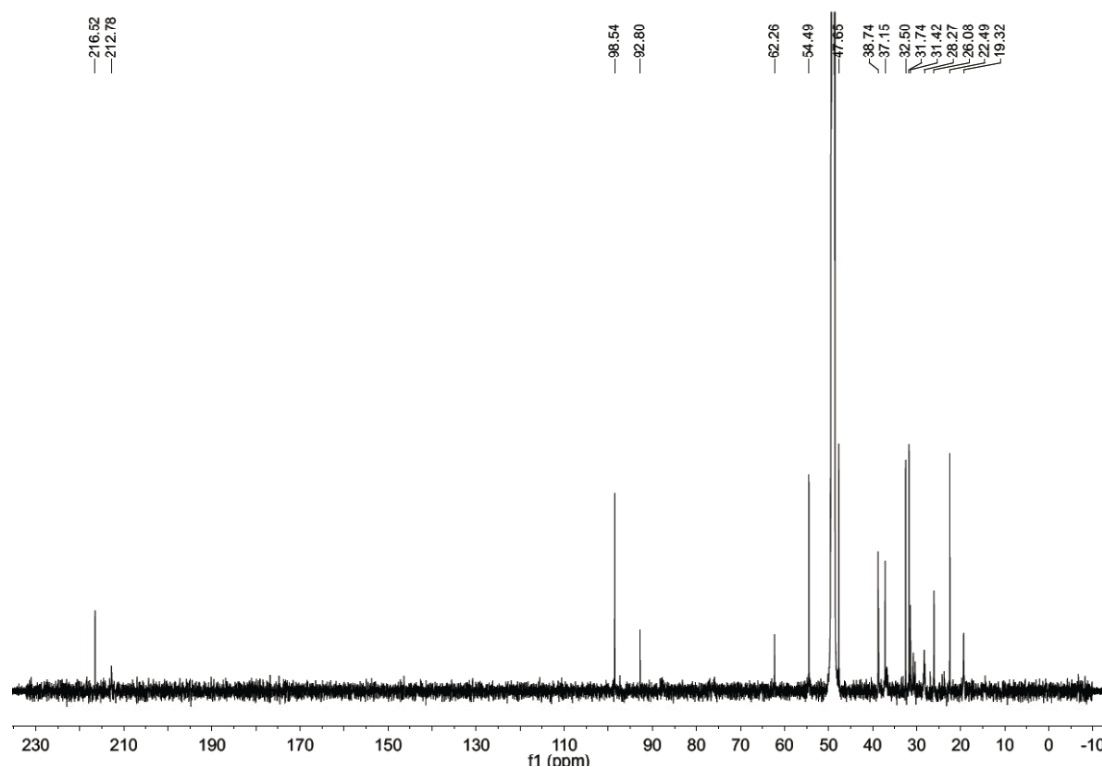
Synthetic Sieboldine A



¹³C NMR of natural and synthetic sieboldine A (2)

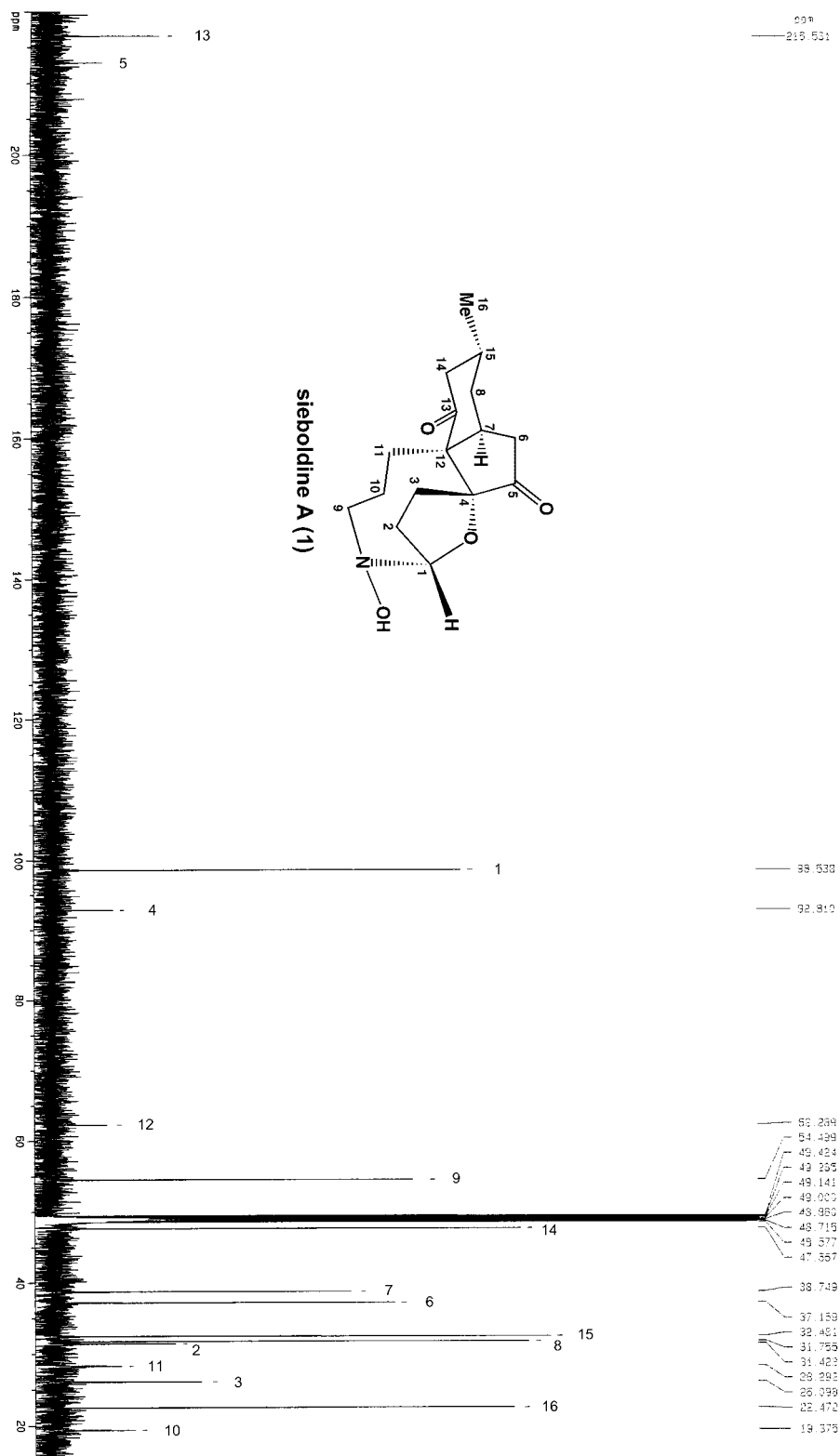


Natural sieboldine A (2), see: Y. Hirasawa, H. Morita, M. Shiro and J. Kobayashi, *Org. Lett.* 2003, **5**, 3991.

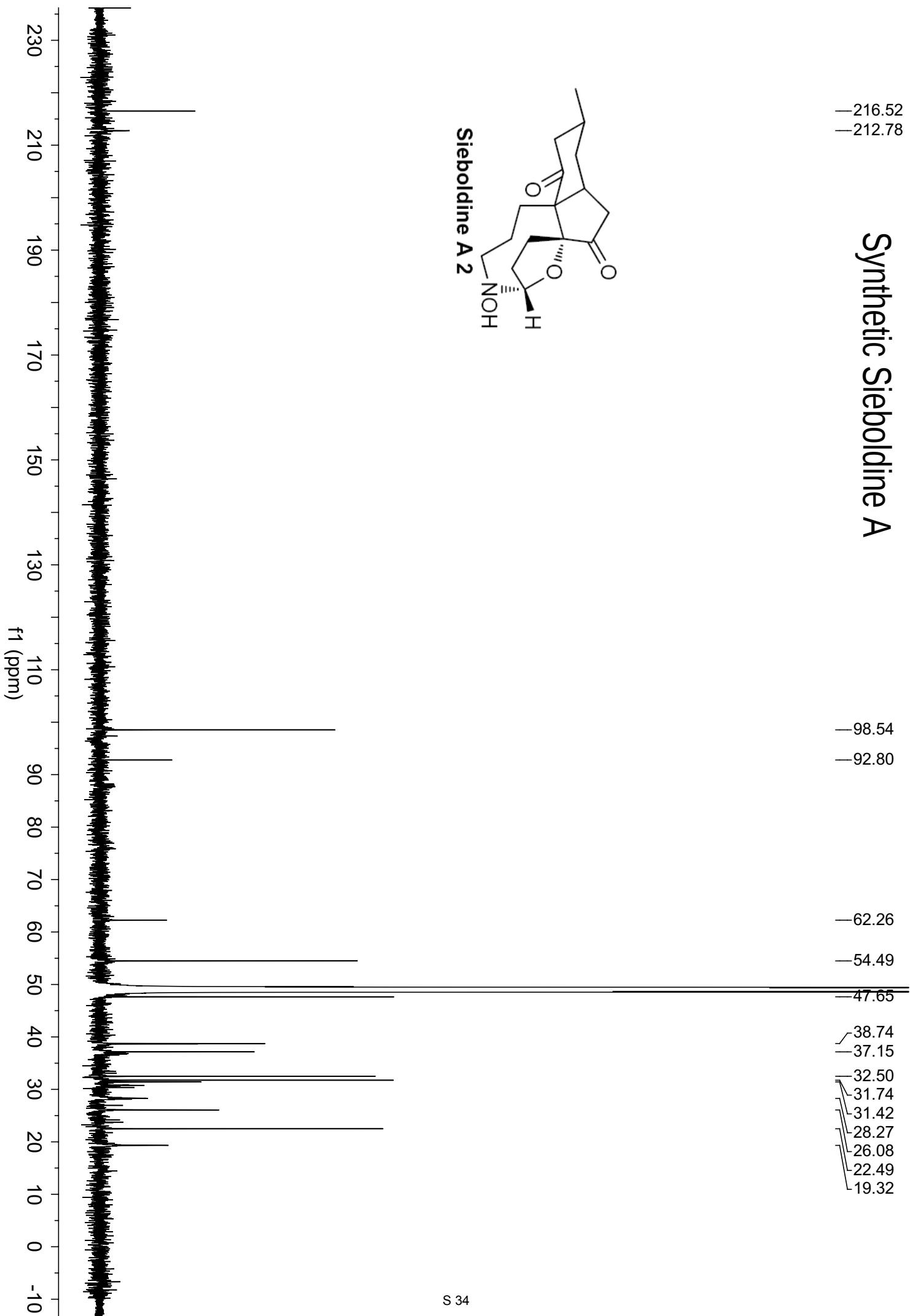
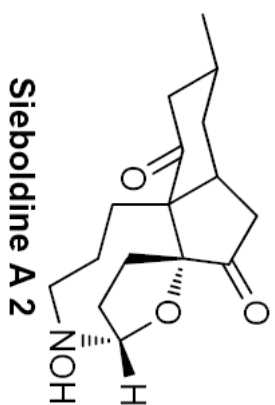


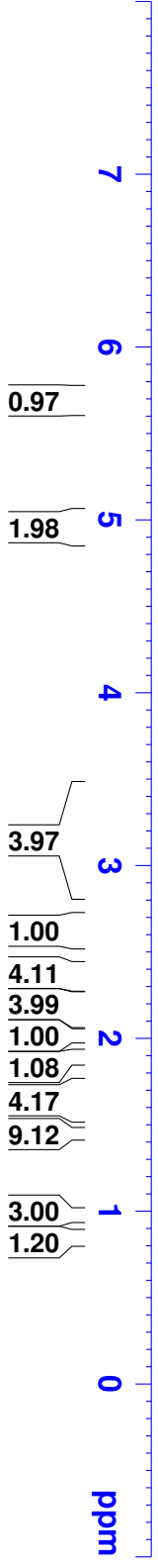
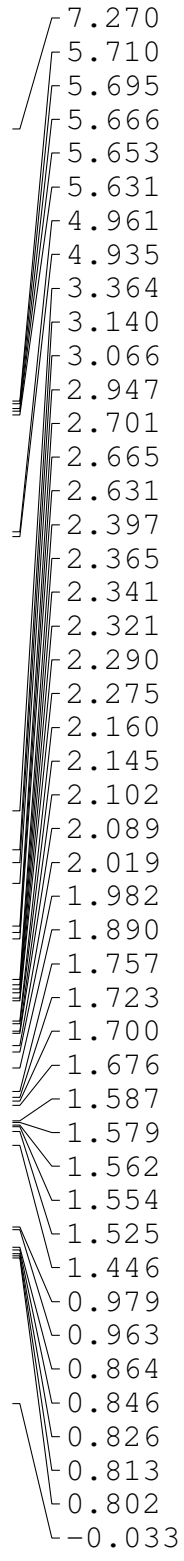
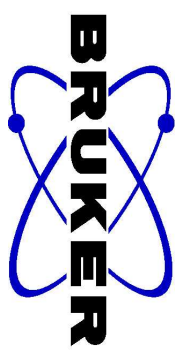
Our synthetic sieboldine A (2).

Natural Sieboldine A



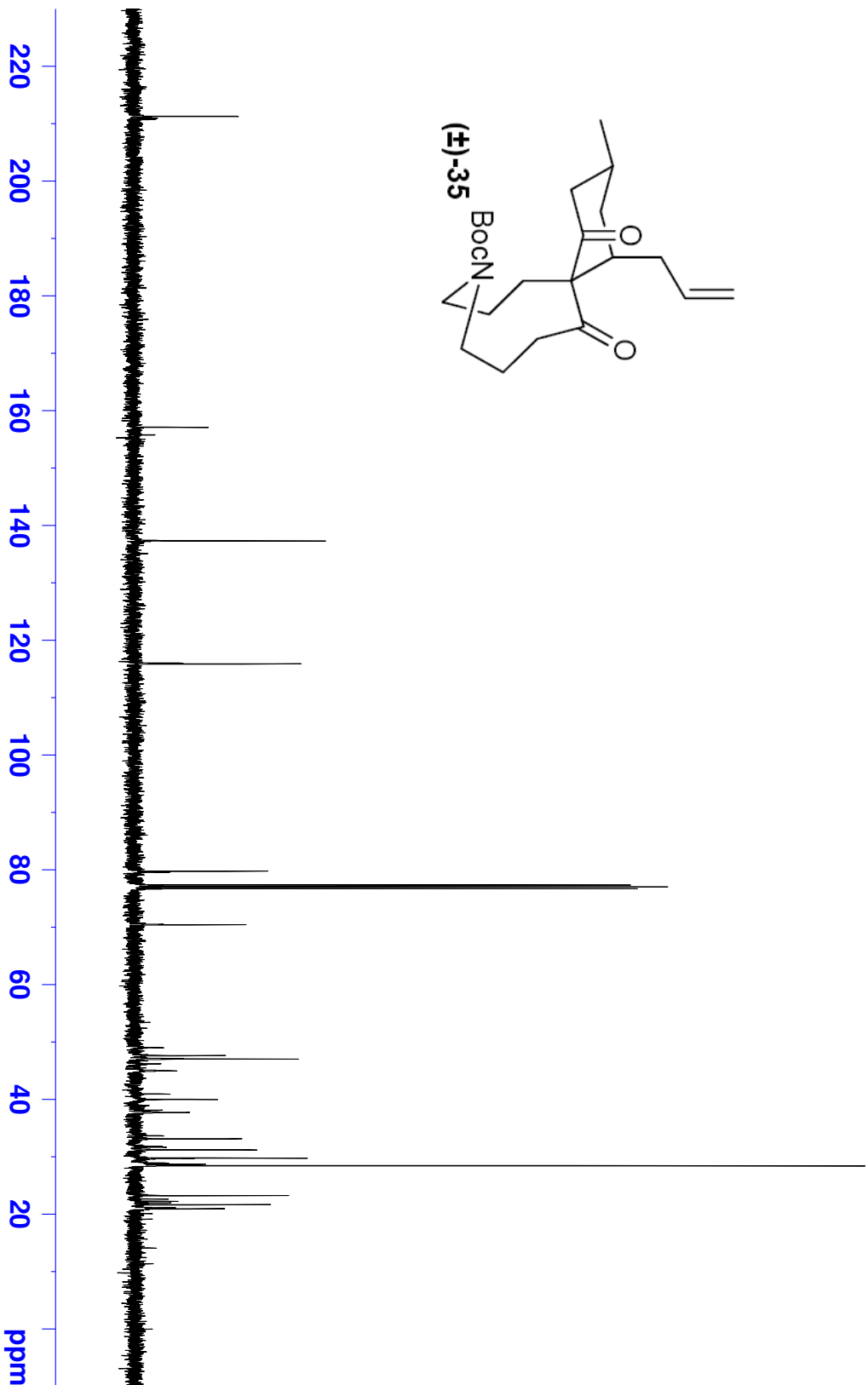
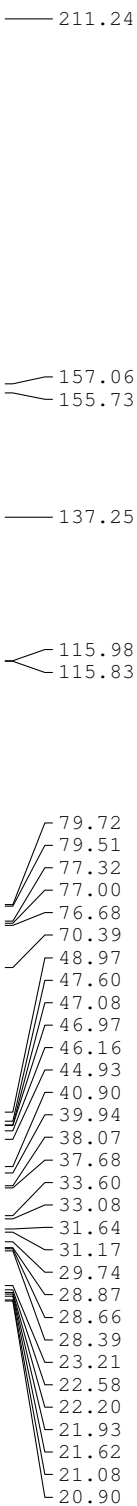
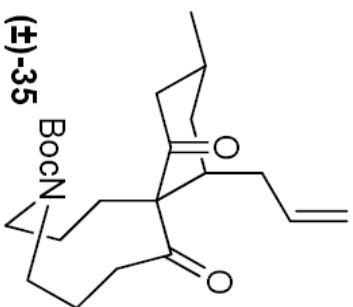
Synthetic Sieboldine A





NAME zxm-680
EXPNO 10
PROCNO 1
Date_ 20091103
Time 10.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
DS 16
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 40.3
DW 60.800 usec
DE 6.50 usec
TE 292.4 K
D1 1.00000000 sec
TD0 1

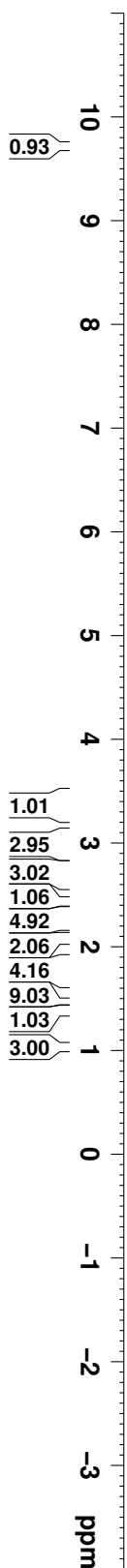
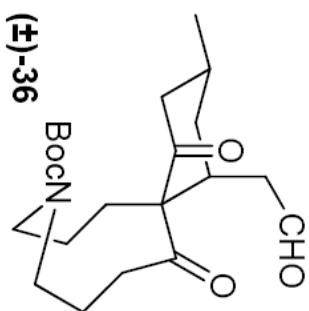
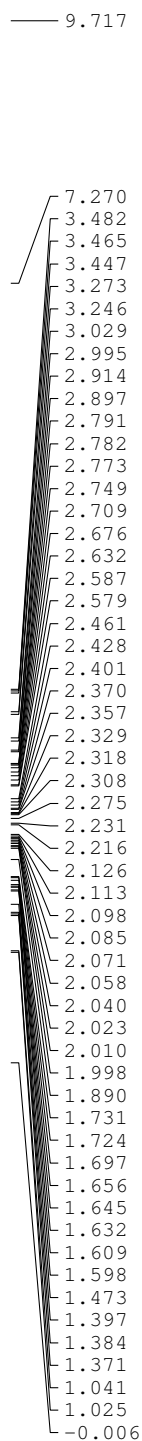
===== CHANNEL f1 =====
NUC1 1H
P1 14.70 usec
PL1 -1.00 dB
PL1W 13.75590801 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300011 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



NAME zxm-680
EXPNO 11
PROCNO 1
Date_ 20091103
Time 11.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 80
DS 4
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 s
RG 80.6
DW 19.800 s
DE 6.50 s
TE 293.8 K
D1 2.0000000 s
D11 0.03000000 s
TD0 1

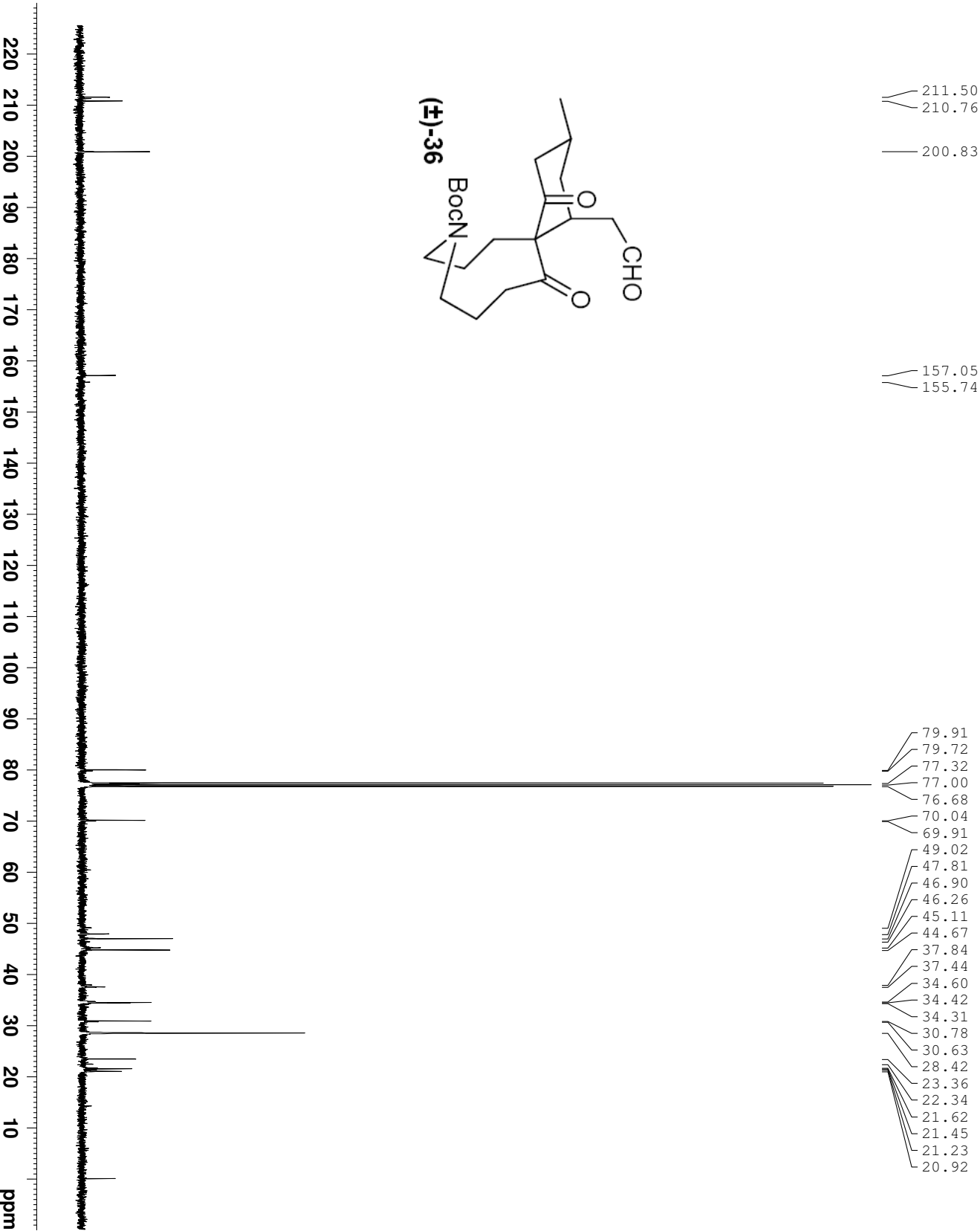
===== CHANNEL f1 =====
NUC1 13C
P1 9.70 s
PL1 -2.00 s
PL1W 56.13311005 V
SFO1 100.6238364 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 s
PL2 -2.10 s
PL12 13.90 s
PL13 13.90 s
PL2W 17.72104263 V
PL12W 0.44513249 V
PL13W 0.44513249 V
SFO2 400.1316005 MHz
SI 32768
SF 100.6127735 MHz
WDW EM
SSB 0
LB 1.00 MHz
GB 0
PC 1.40



NAME zxm-fail1-2
EXPNO 10
PROCNO 1
Date_ 20111114
Time 10.57
INSTRUM spect
PROBHD 5 mm PABBO BP-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 128
DW 60.800 usec
DE 6.50 usec
TE 293.1 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 -3.00 dB
PL1W 22.90425682 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300015 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



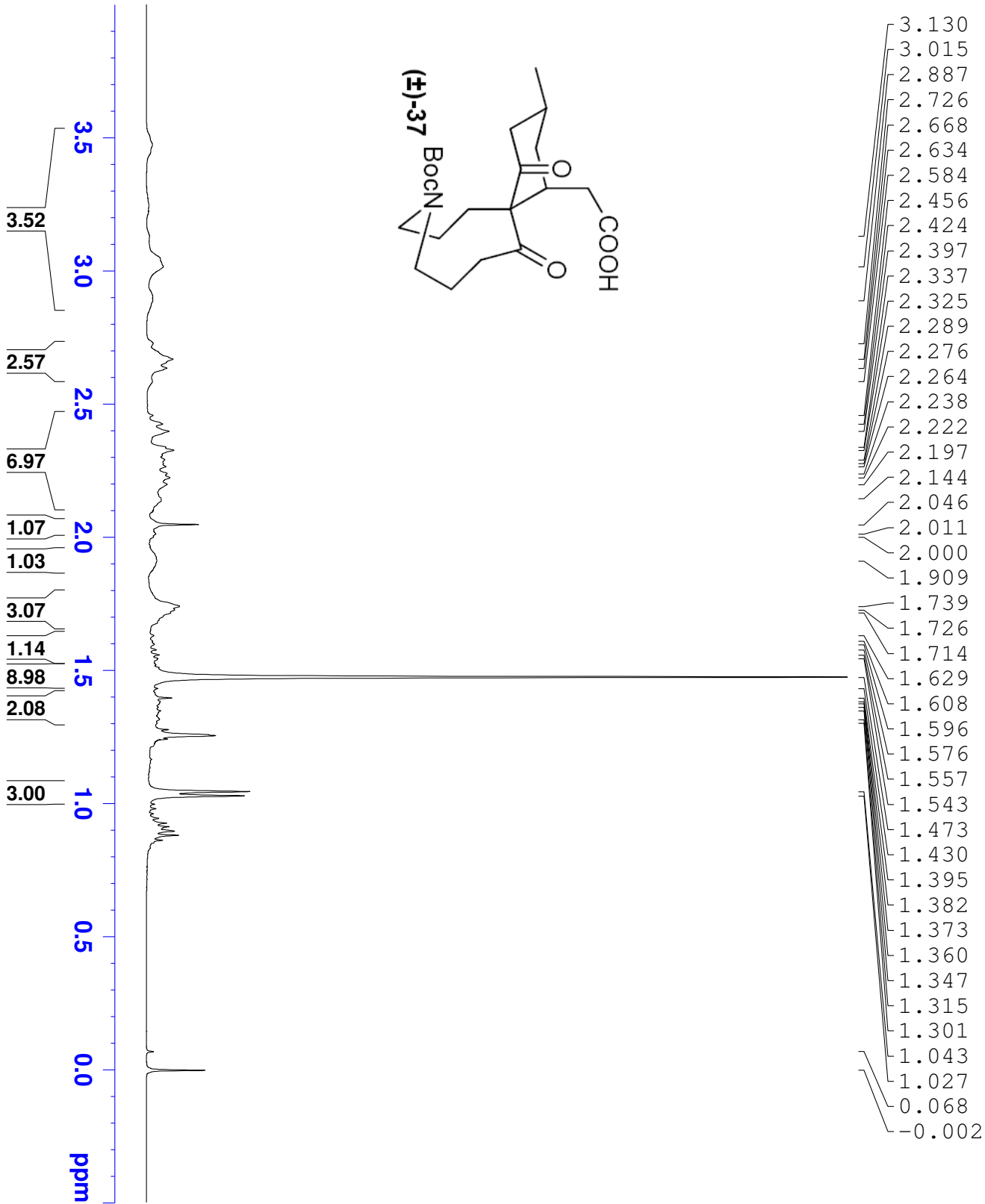
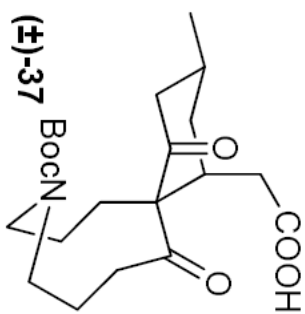
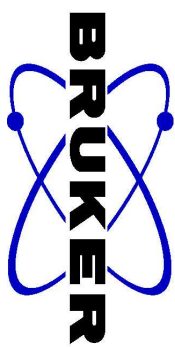
```

NAME          xzm-fa1-2
EXPNO         11
PROCNO        1
Date_         20111114
Time_         11.37
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            700
DS            4
SWH           25252.525 Hz
FIDRES        0.385323 Hz
AQ            1.2976229 sec
RG            2050
DM            19.800 usec
DE            6.50 usec
TE            293.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            9.40 usec
PL1           -2.00 dB
PL1W          57.32743073 W
SFO1          100.6228298 MHz

===== CHANNEL f2 =====
CEPPRG2       waltz16
NUC2          1H
PCPD2         90.00 usec
PL2           -2.00 dB
PL12          15.50 dB
PL13          15.50 dB
PL2W          18.19349861 W
PL12W         0.32353121 W
PL13W         0.32353121 W
SFO2          400.1316003 MHz
SI            32768
SR            100.6127710 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            0.80

```

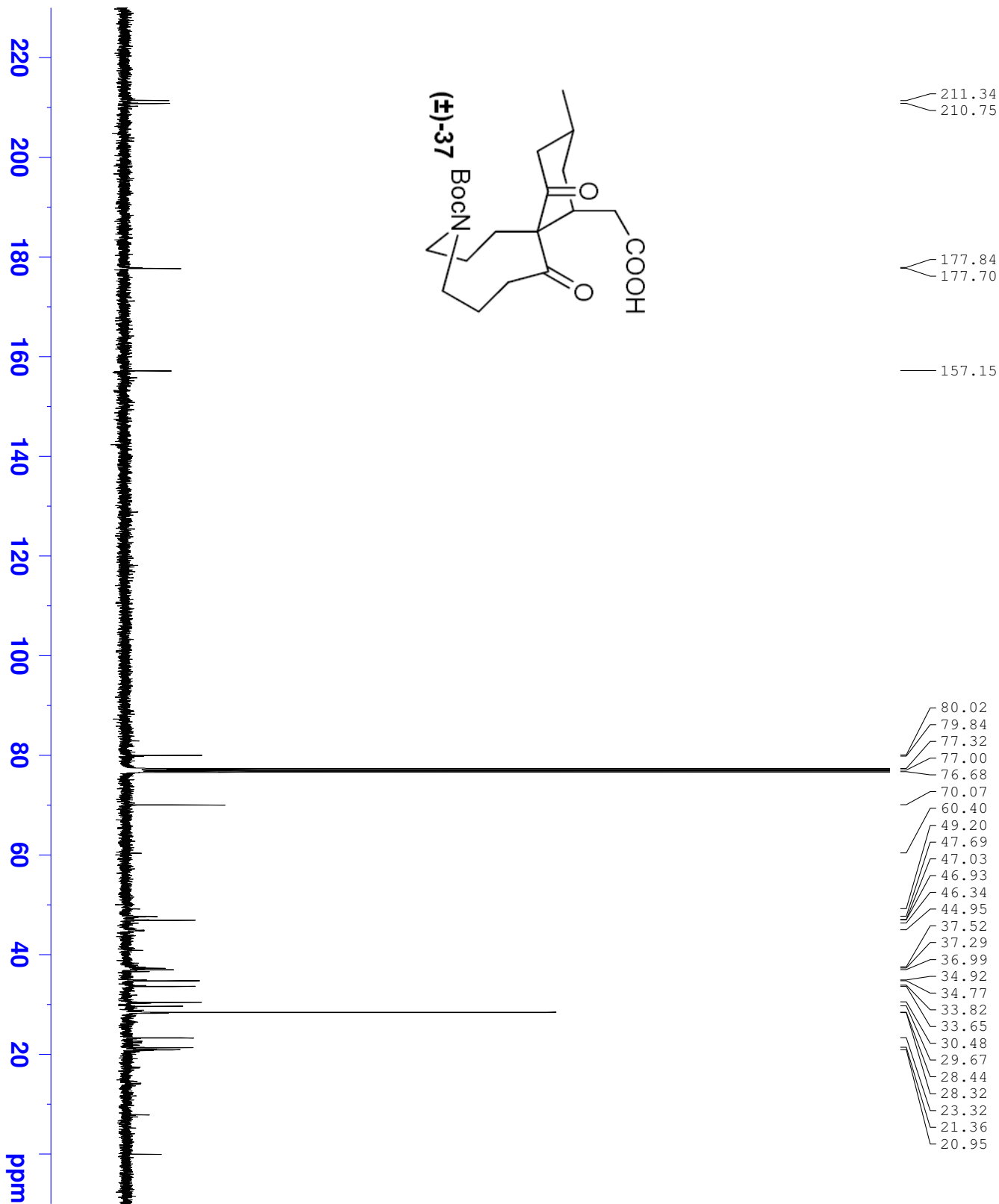
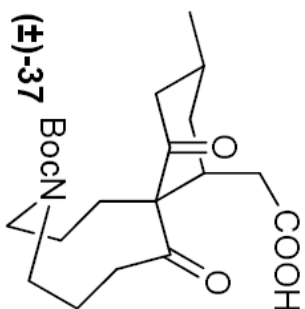
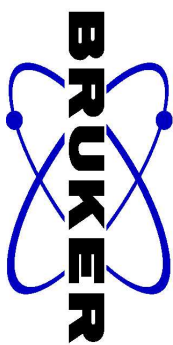


```

NAME          zxm-691
EXPNO         10
PROCNO        1
Date_         20091111
Time          22.27
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            101
DE            60.800 usec
TE            294.2 K
D1            1.00000000 sec
TD0           1
  
```

```

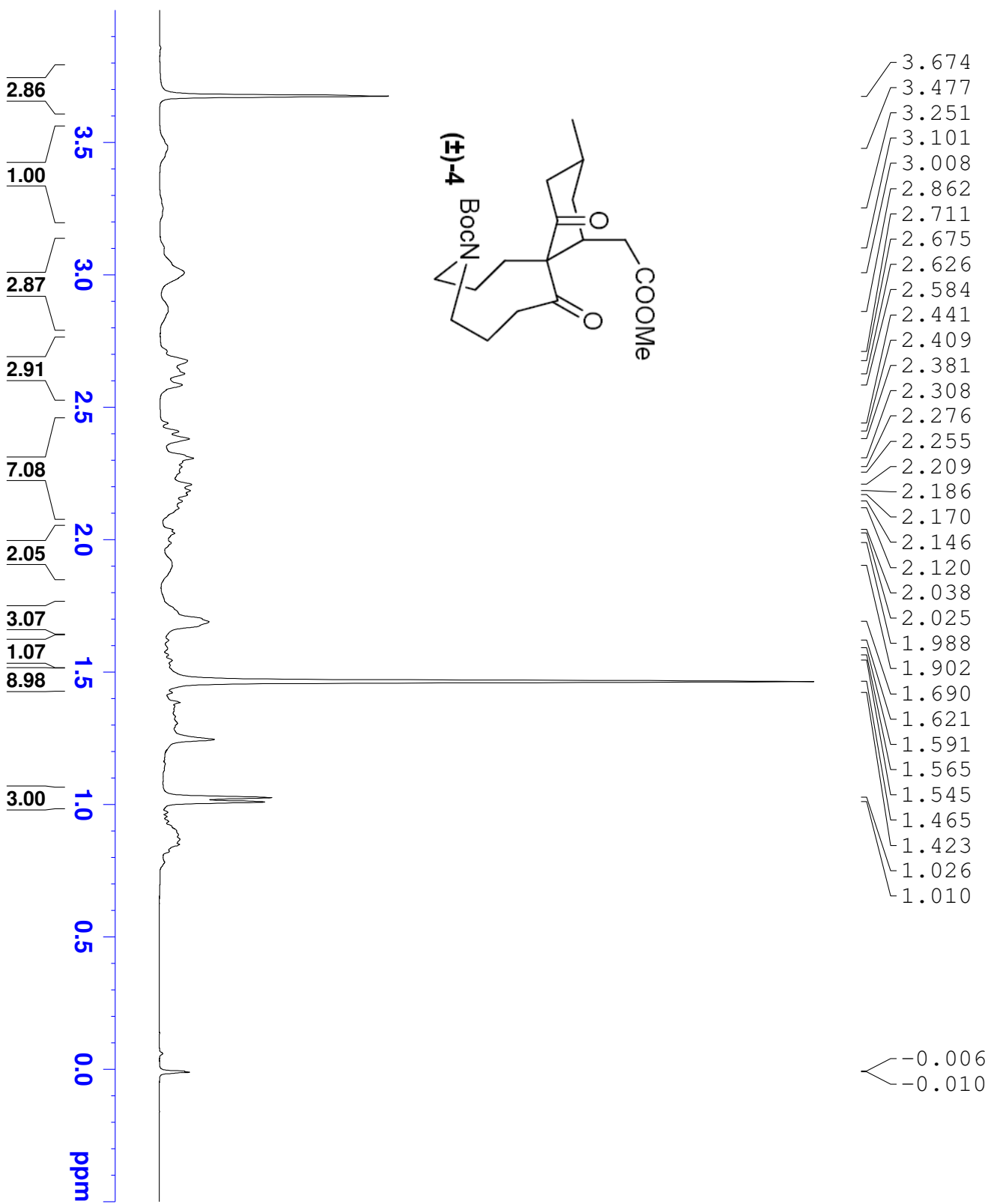
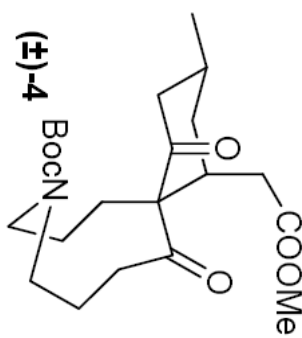
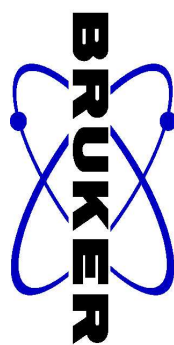
===== CHANNEL f1 =====
NUC1          1H
P1            14.70 usec
PL1          -1.00 dB
PL1W         13.75590801 W
SFO1         400.1324710 MHz
SI           32768
SF           400.1300009 MHz
WDW           EM
SSB           0
LB           0.30 Hz
GB           0
PC           1.00
  
```



NAME zxm-691
EXPNO 11
PROCNO 1
Date_ 20091111
Time 23.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 700
DS 4
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.297629 sec
RG 575
DW 19.800 usec
DE 6.50 usec
TE 296.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.70 usec
PL1 -2.00 dB
PL1W 56.1331005 W
SFO1 100.6238364 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.10 dB
PL12 13.90 dB
PL13 13.90 dB
PL2W 17.72104263 W
PL12W 0.44513249 W
PL13W 0.44513249 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127696 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.60



NAME zxm-729
EXPNO 10
PROCNO 1
Date_ 20091225
Time 9.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 80.6
DW 60.800 usec
DE 6.50 usec
TE 290.2 K
D1 1.00000000 sec
TD0 1

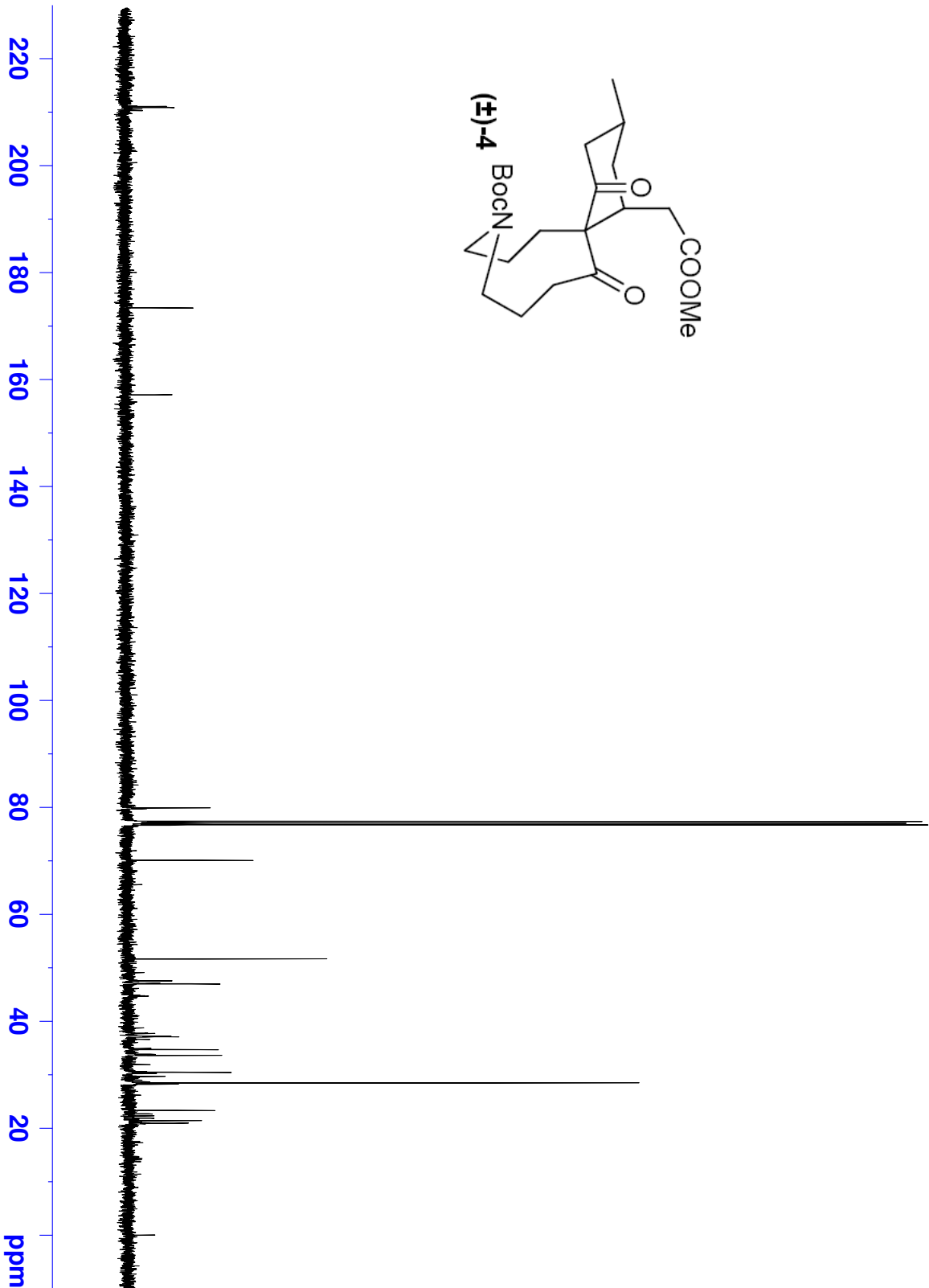
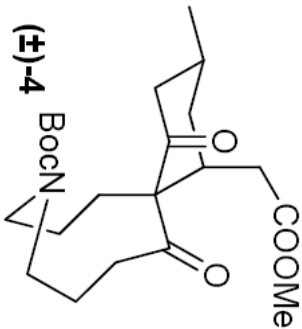
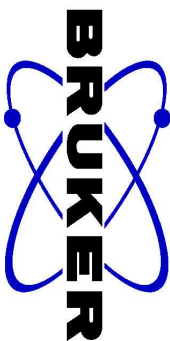
===== CHANNEL f1 =====
NUC1 1H
P1 14.70 usec
PL1 -1.00 dB
PL1W 13.75590801 W
SEOL 400.1324710 MHz
SI 32768
SF 400.1300008 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

211.04
210.85

173.35

157.11

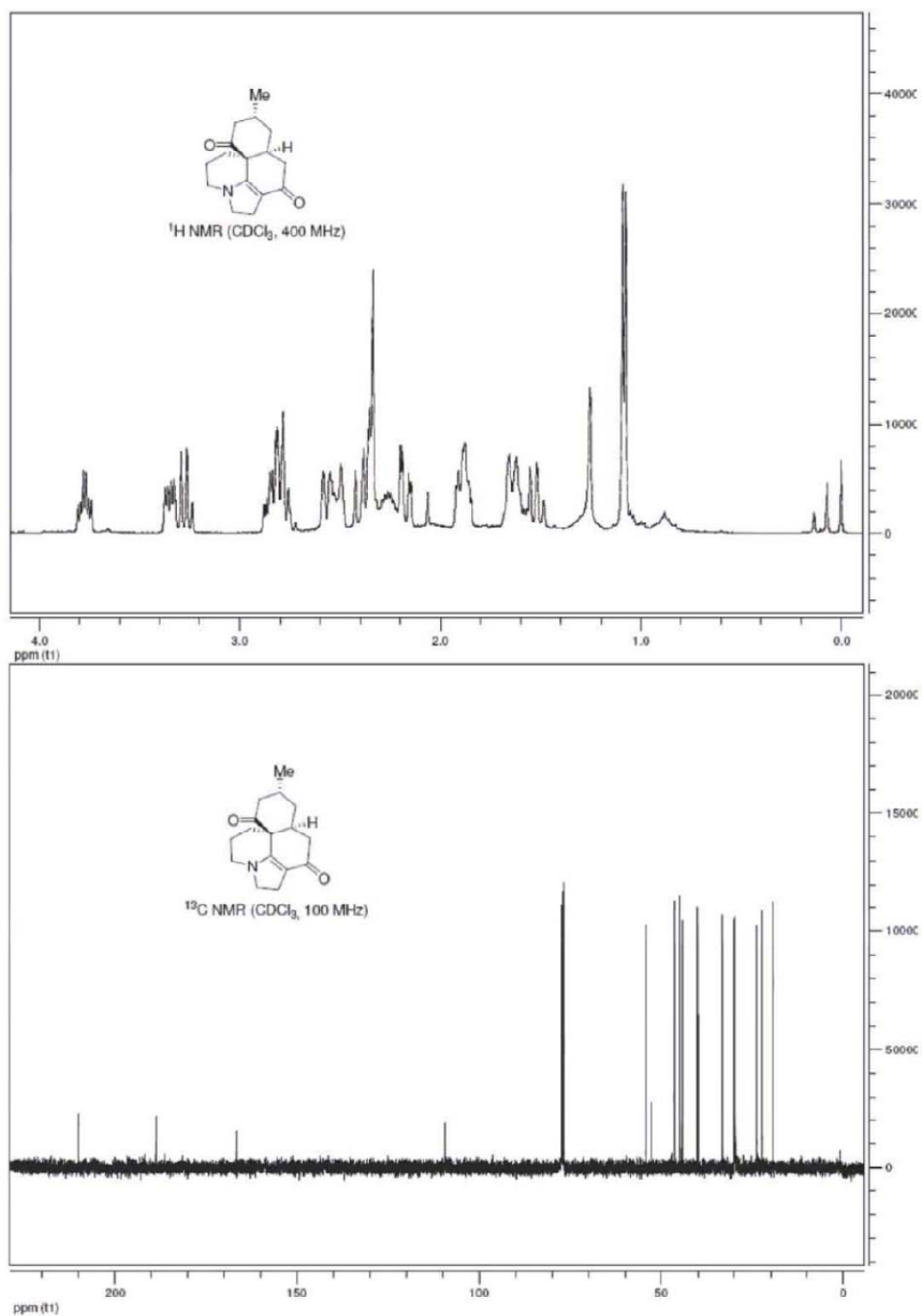
79.86
79.69
77.32
77.00
76.68
70.05
51.61
47.51
47.04
46.94
46.10
44.67
37.67
37.16
37.04
34.86
34.66
33.79
33.59
30.41
30.20
29.65
28.43
28.21
23.27
22.64
22.29
21.84
21.38
20.89



NAME zxm-729
EXPNO 11
PROCNO 1
Date_ 20091225
Time 9.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 280
DS 4
SWH 26041.666 Hz
FIDRES 0.397364 Hz
AQ 1.2583412 sec
RG 575
DW 19.200 usec
DE 6.50 usec
TE 291.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.70 usec
PL1 -2.00 dB
PL1W 56.13311005 W
SFO1 100.6228298 MHz

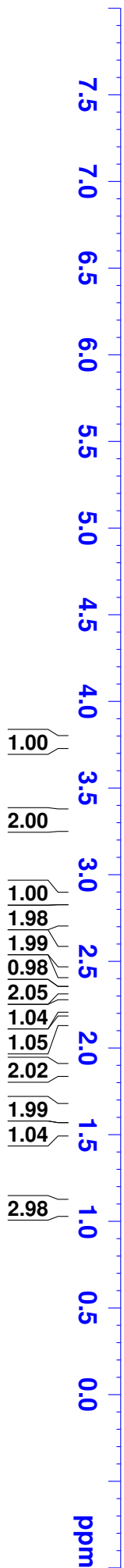
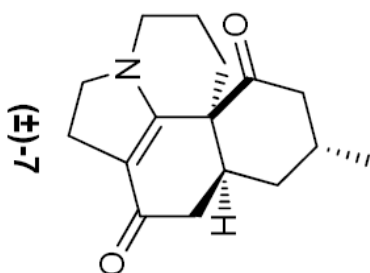
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.10 dB
PL12 13.90 dB
PL13 13.90 dB
PL2W 17.72104263 W
PL12W 0.44513249 W
PL13W 0.44513249 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127717 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.80

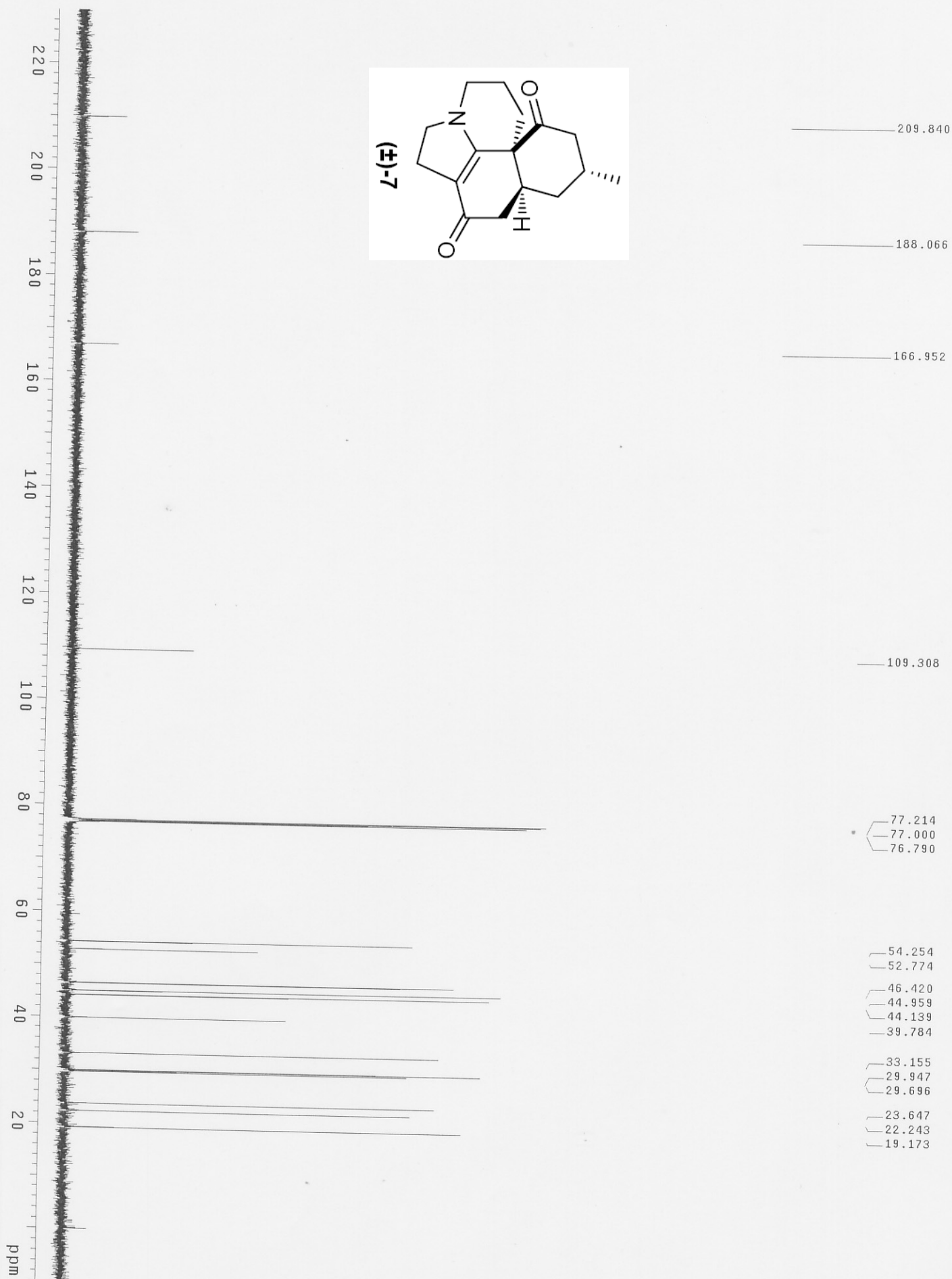


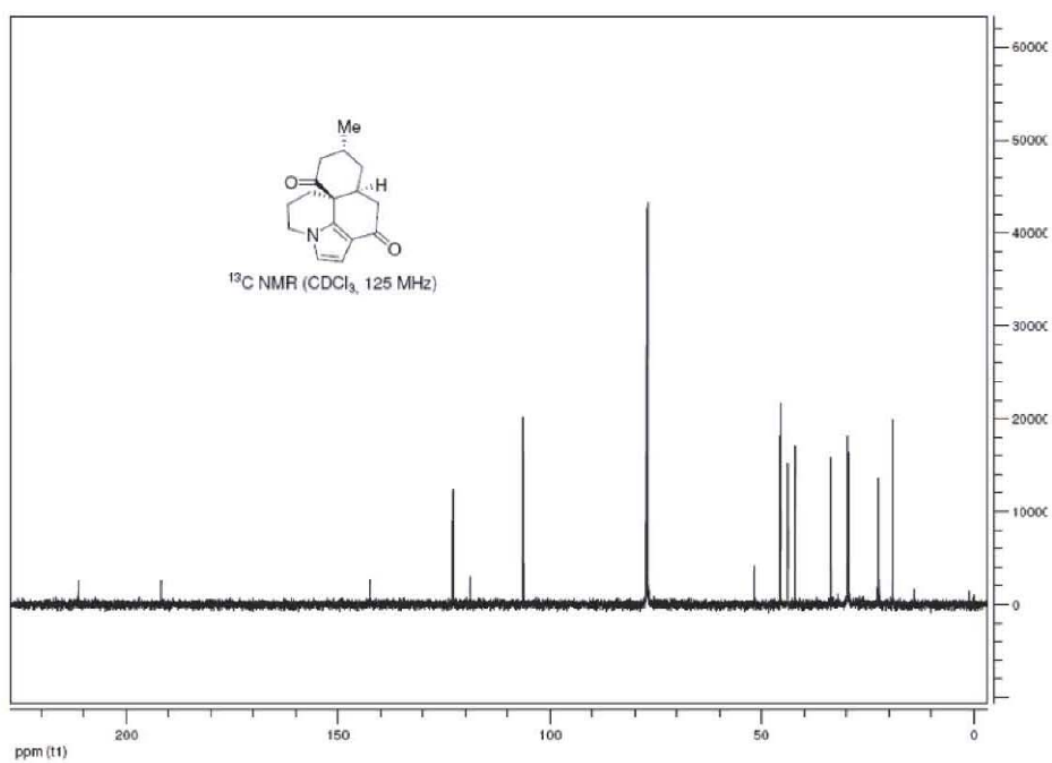
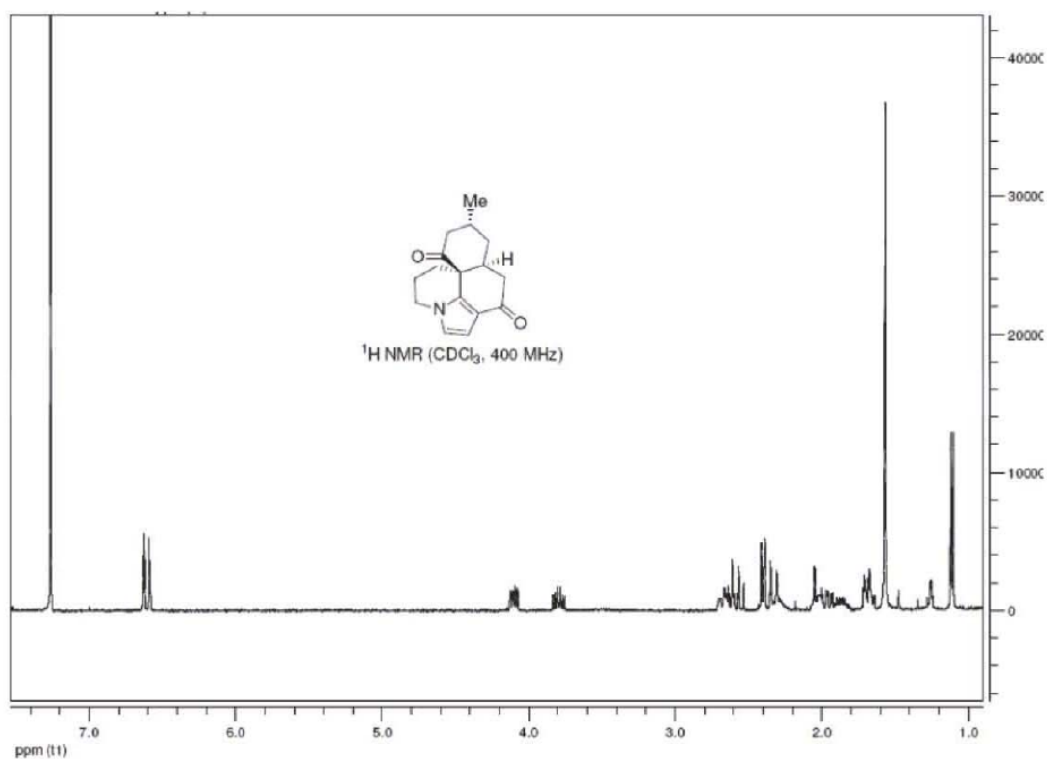
Reported compound **7** by Yang and co-workers, see: Y.-R. Yang, L. Shen, K. Wei and Q.-S. Zhao, *J. Org. Chem.* 2010, **75**, 1317.

STANDARD PROTON PARAMETERS

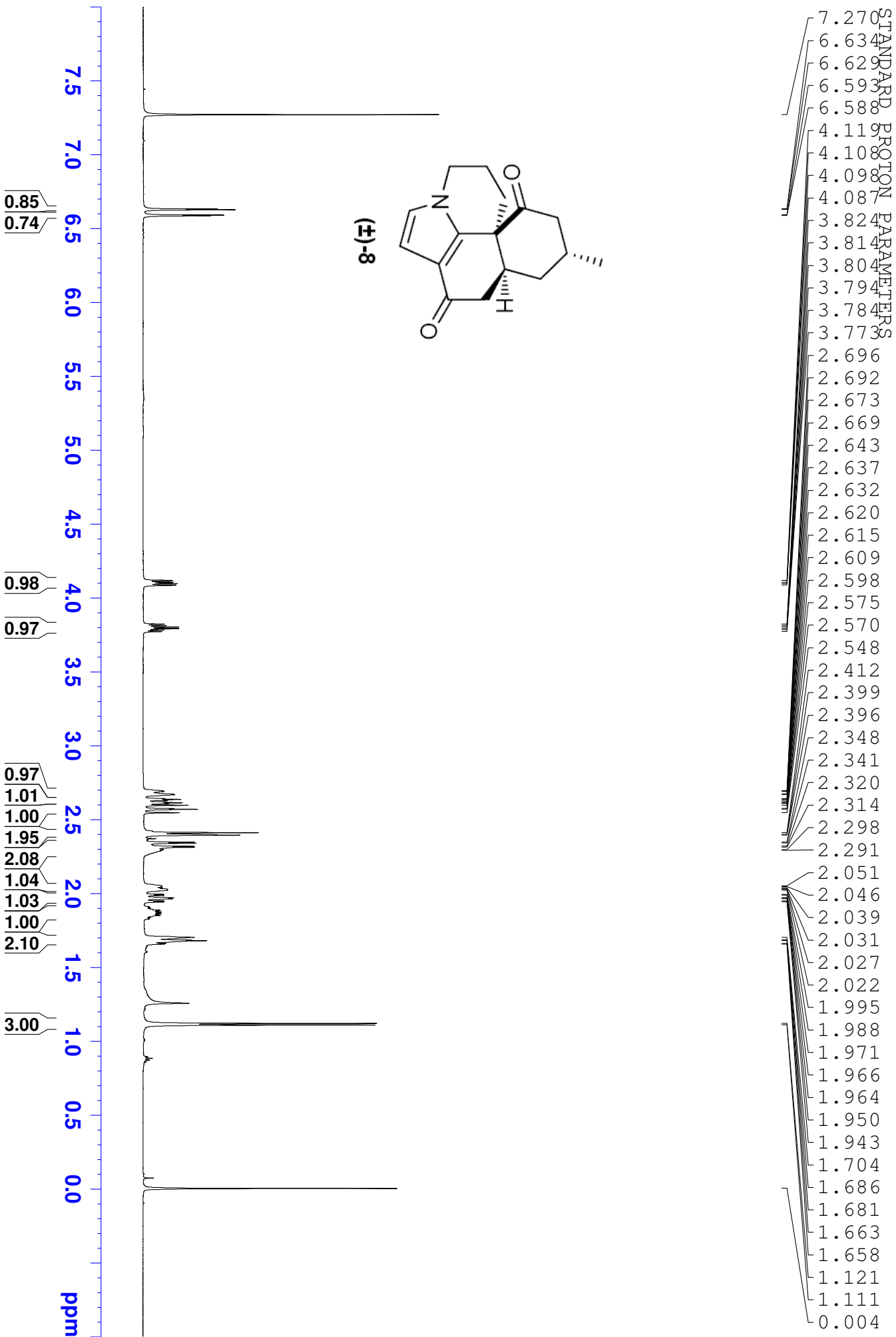
7.270
3.780
3.772
3.762
3.754
3.743
3.349
3.340
3.329
3.319
3.301
3.283
2.871
2.860
2.851
2.795
2.787
2.776
2.768
2.758
2.740
2.559
2.554
2.541
2.537
2.531
2.519
2.515
2.497
2.493
2.391
2.364
2.341
2.330
2.321
2.241
2.231
2.171
2.164
2.143
2.136
1.896
1.889
1.872
1.867
1.851
1.844
1.640
1.637
1.617
1.613
1.595
1.549
1.546
1.527
1.523
1.072
1.062
-0.018

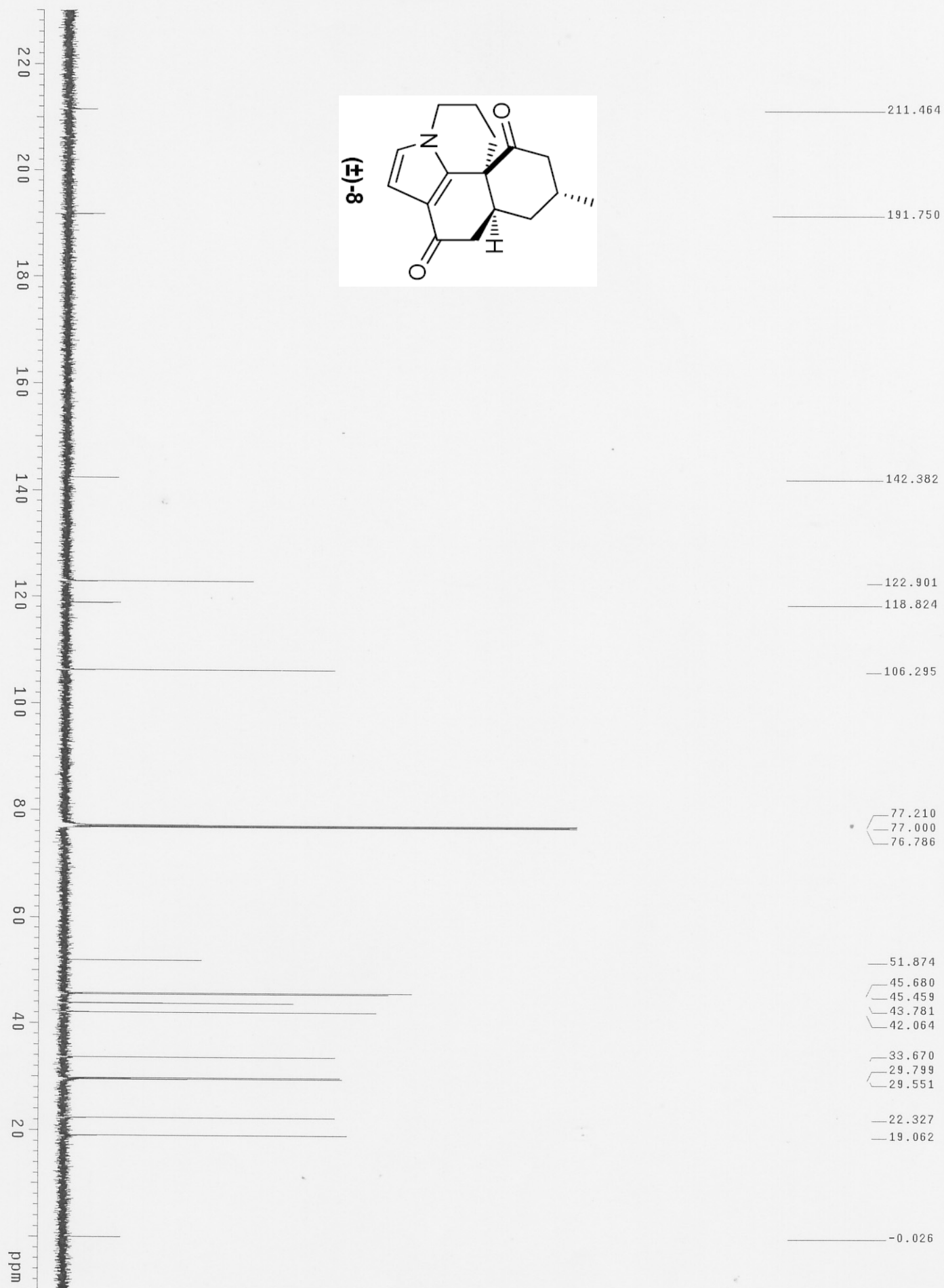




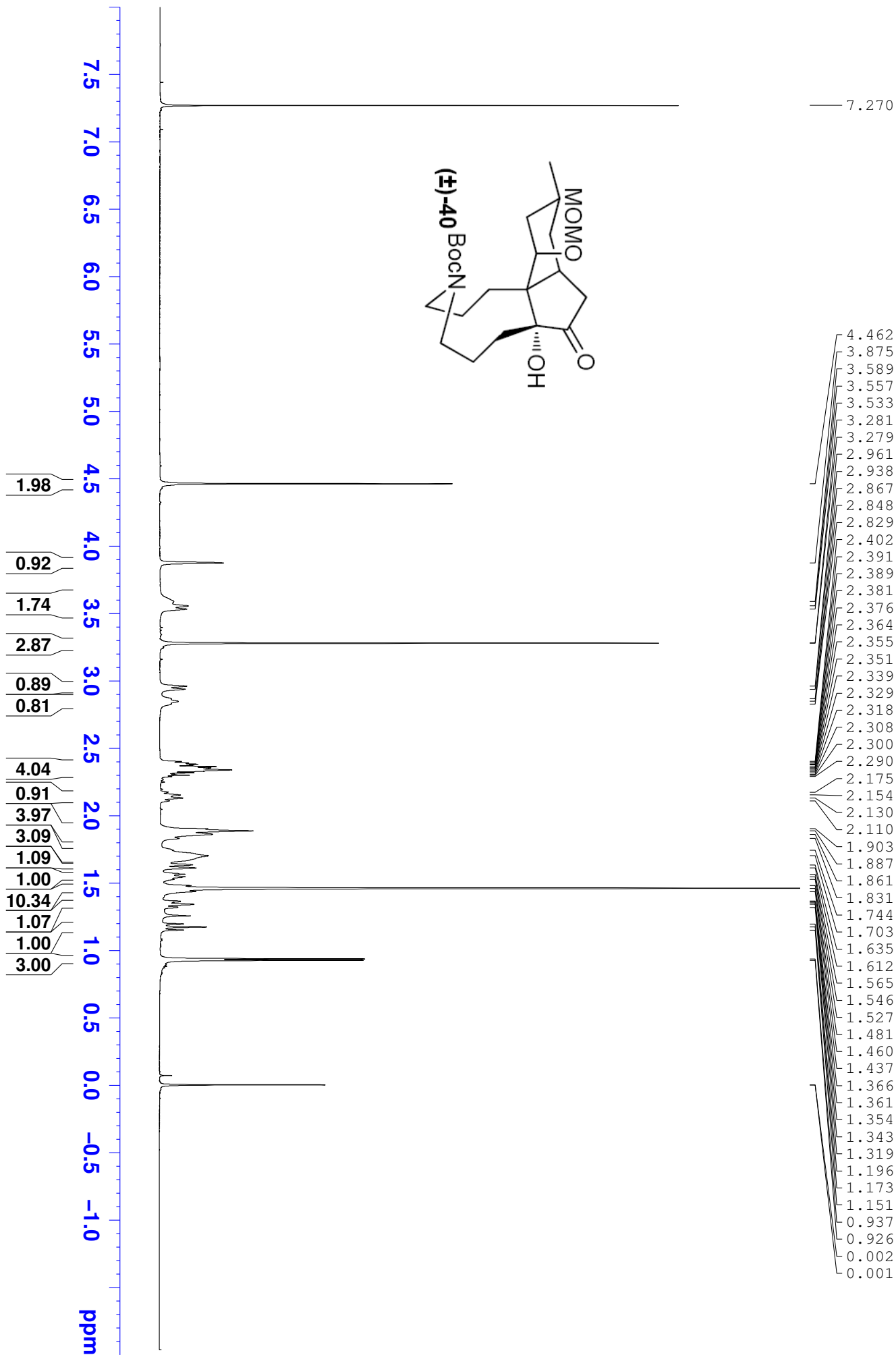


Reported compound **8** by Yang and co-workers, see: Y.-R. Yang, L. Shen, K. Wei and Q.-S. Zhao, *J. Org. Chem.* 2010, **75**, 1317.

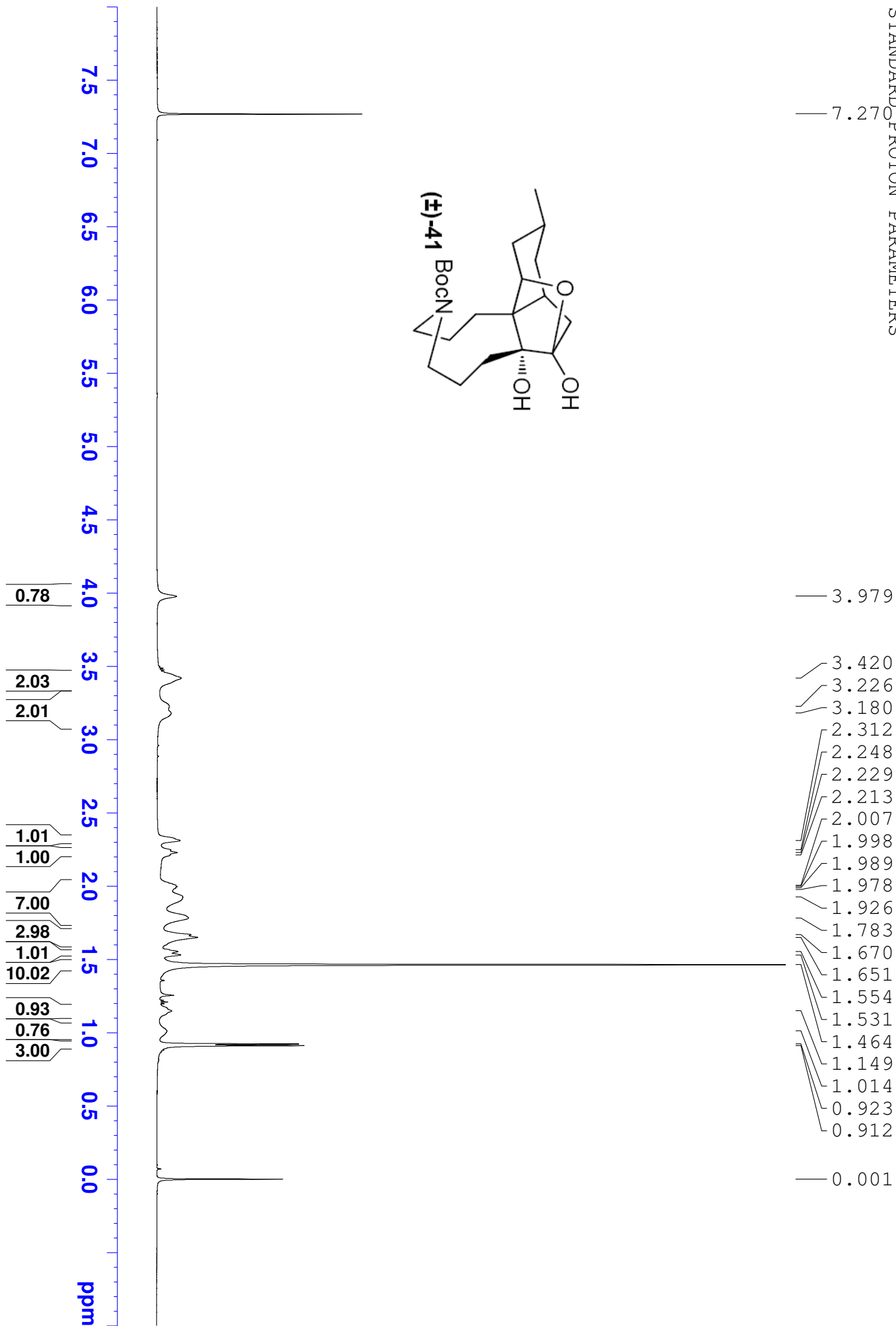
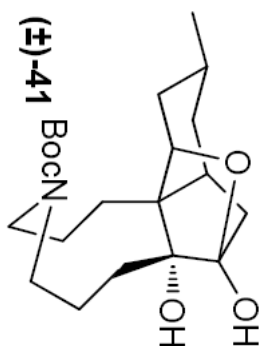


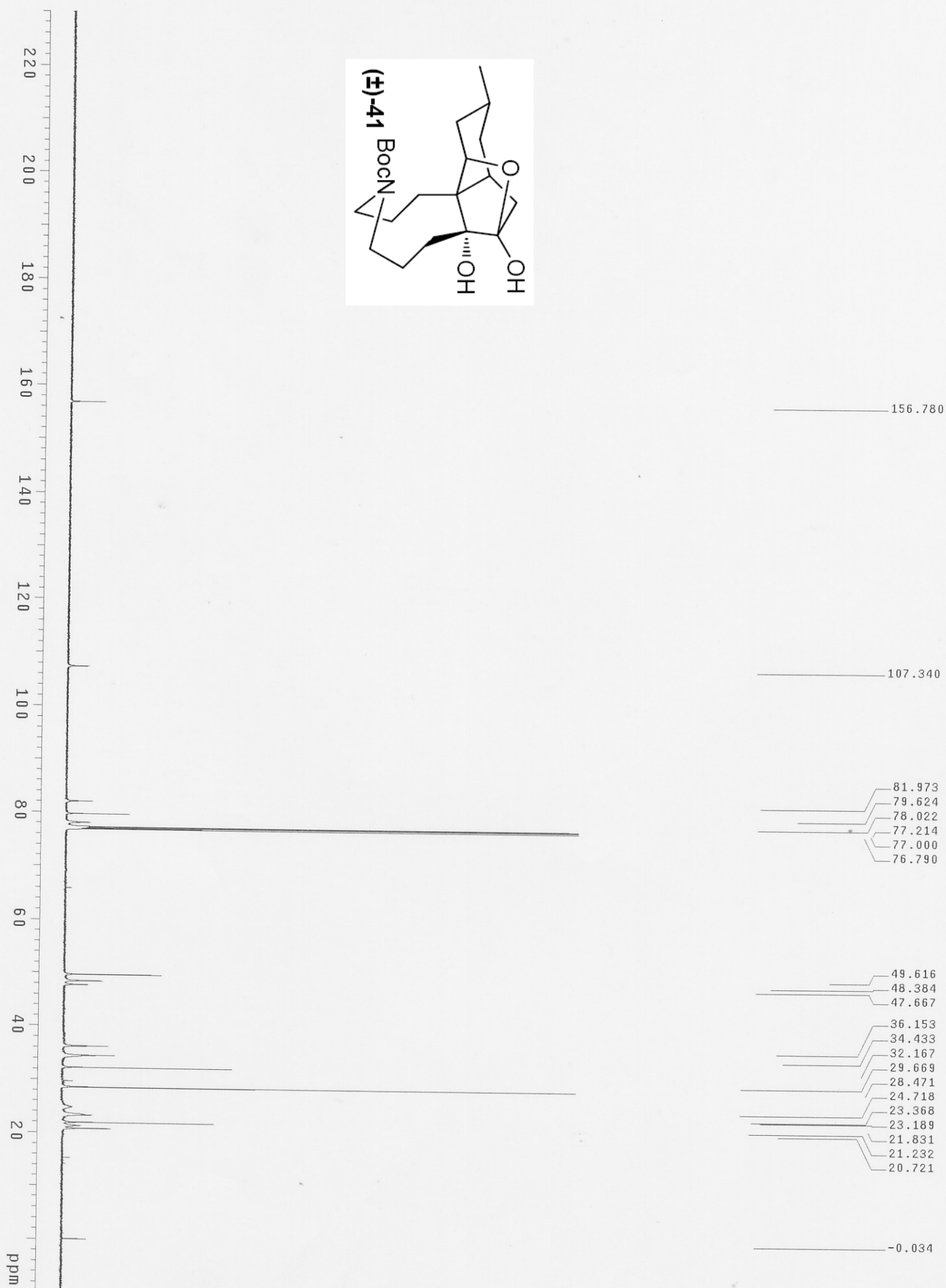
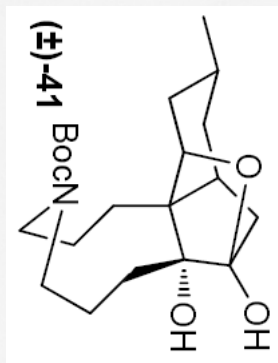


STANDARD PROTON PARAMETERS



STANDARD PROTON PARAMETERS





STANDARD PROTON PARAMETERS

7.270

4.875

3.475

3.190

3.061

2.451

2.383

2.358

2.272

2.069

2.046

1.966

1.752

1.743

1.620

1.607

1.584

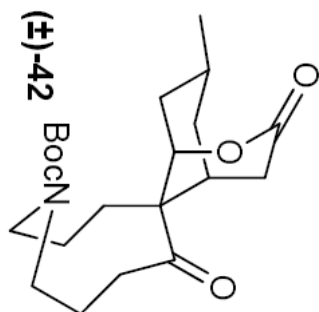
1.566

1.503

1.487

0.989

0.979



1.00

5.05

3.08

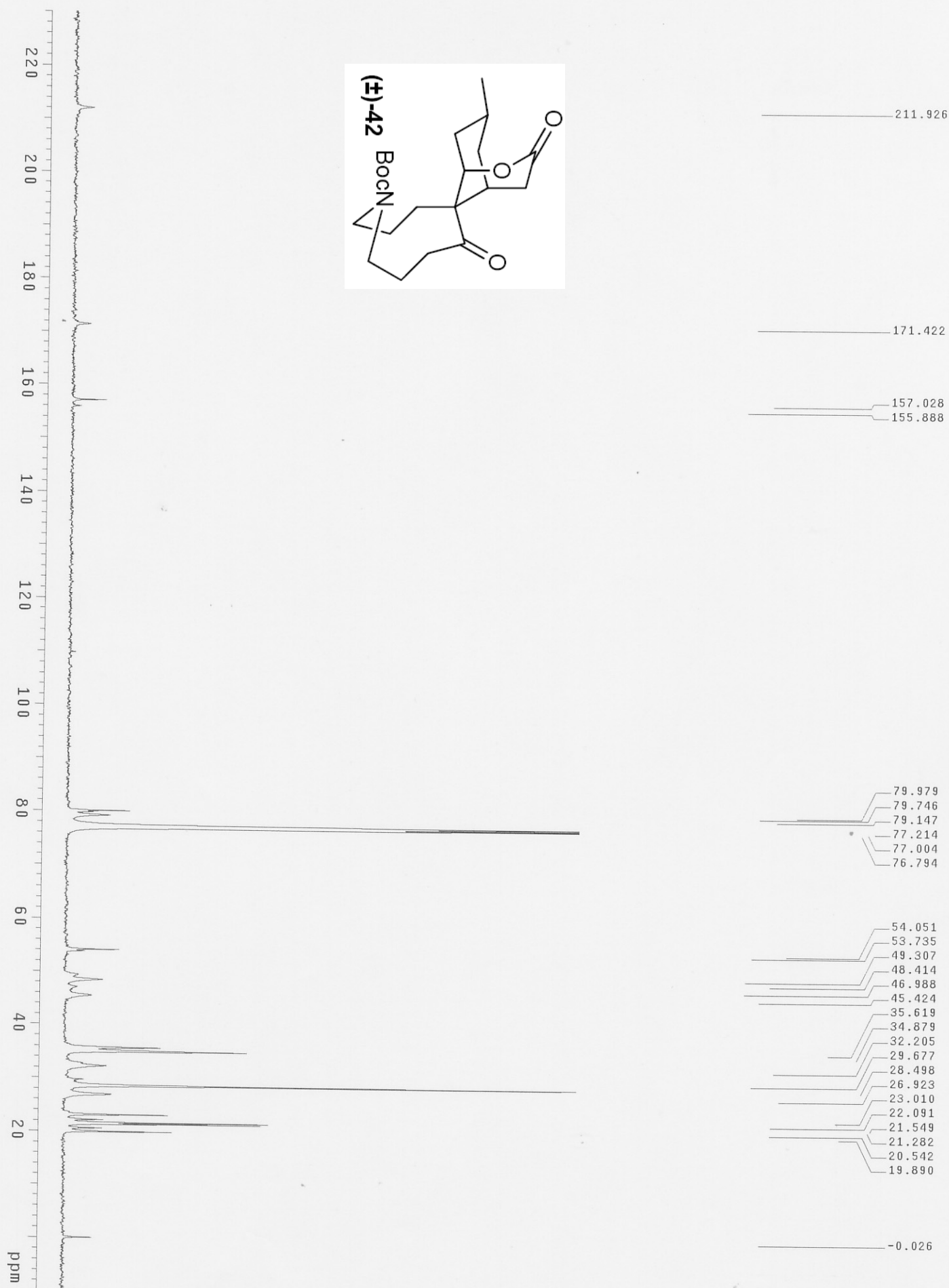
4.03

1.16

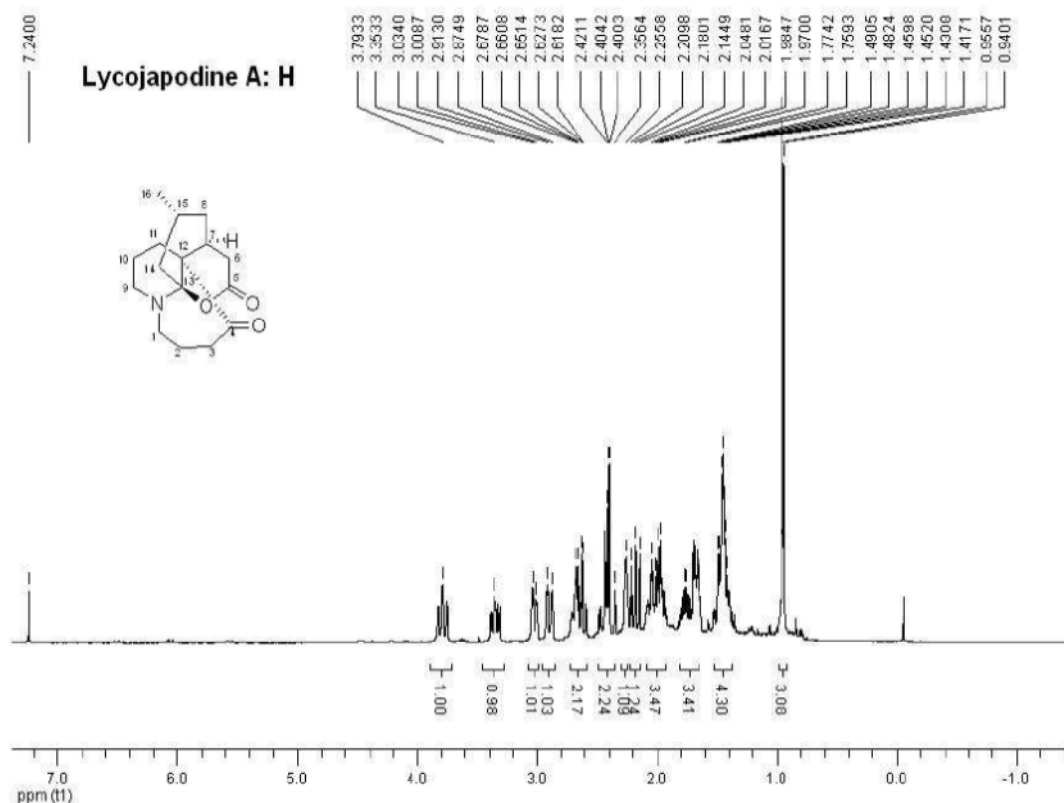
4.03

12.02

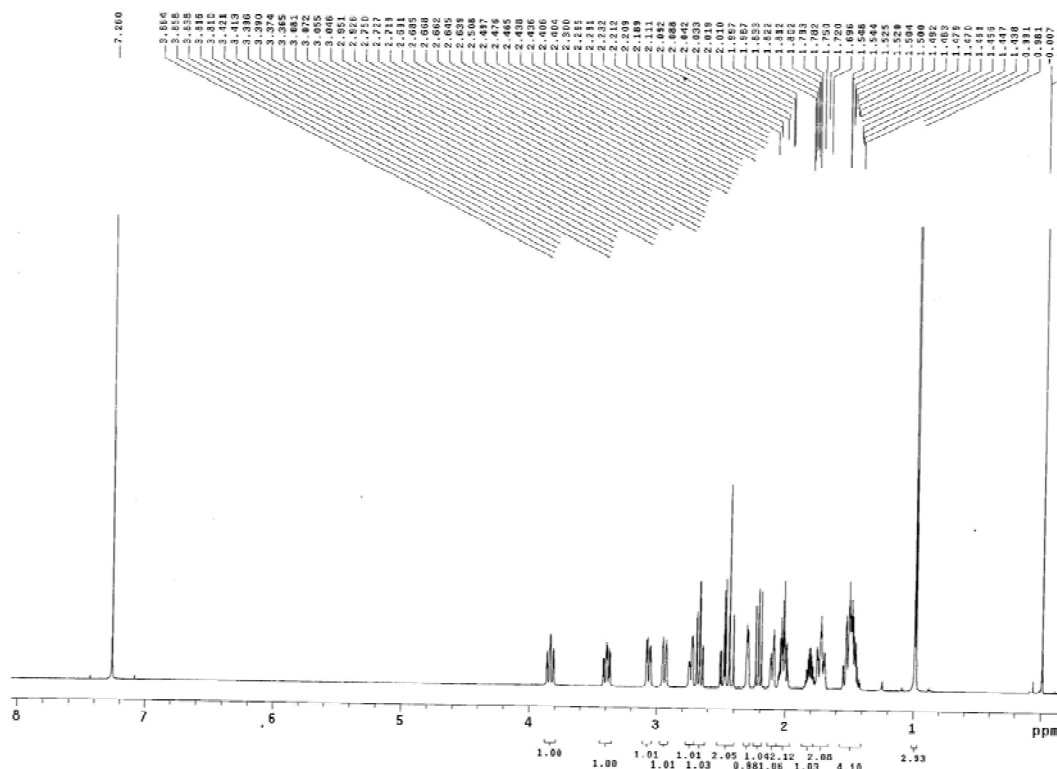
3.14



¹H NMR of natural and synthetic lycojapodine A (**3**)



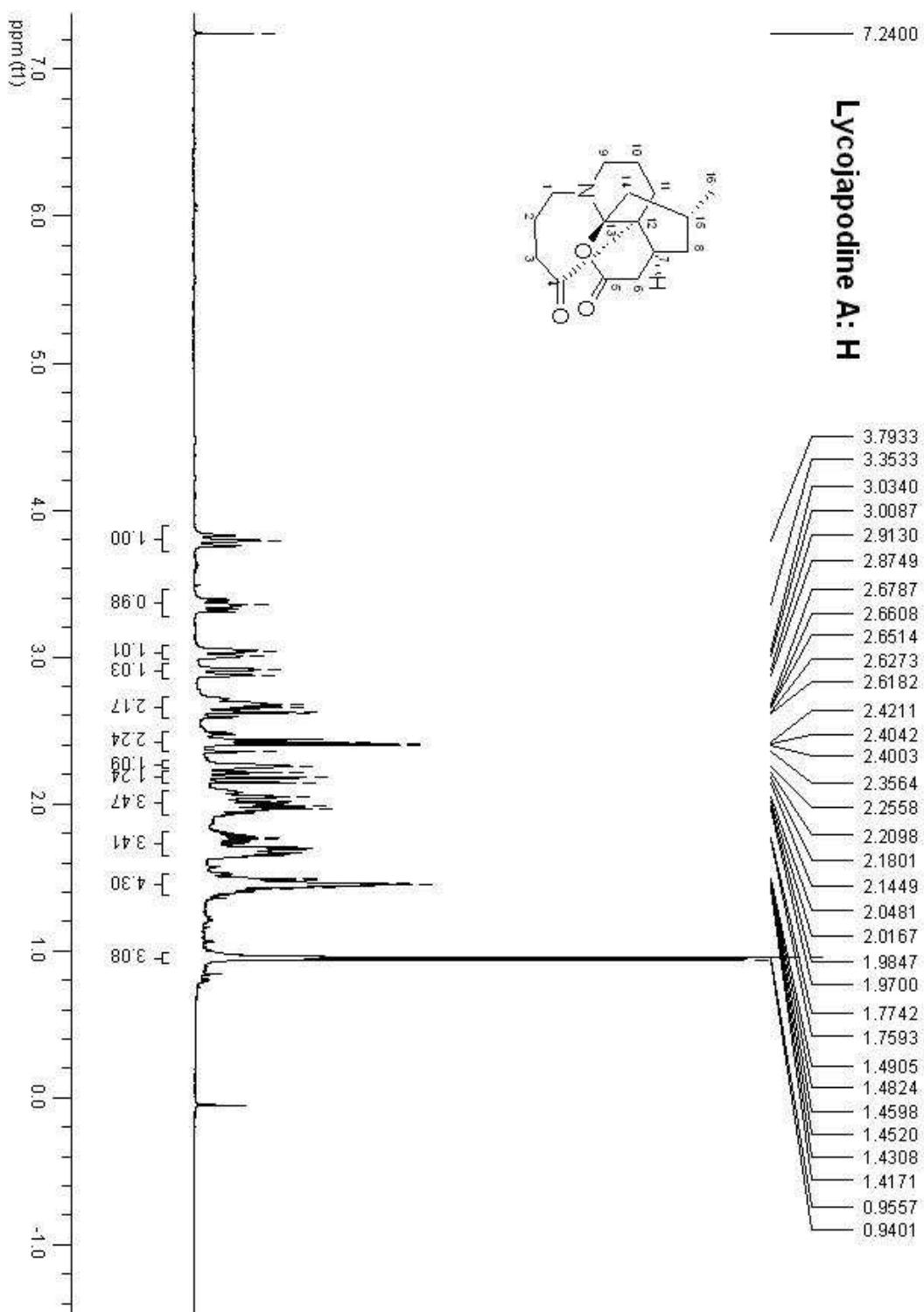
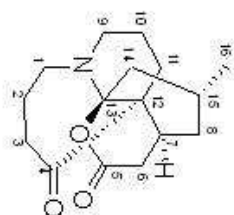
Natural lycojapodine A (**3**), see: J. He, X.-Q. Chen, M.-M. Li, Y. Zhao, G. Xu, X. Cheng, L.-Y. Peng, M.-J. Xie, Y.-T. Zheng, Y.-P. Wang and Q.-S. Zhao, *Org. Lett.* 2009, **11**, 1397.



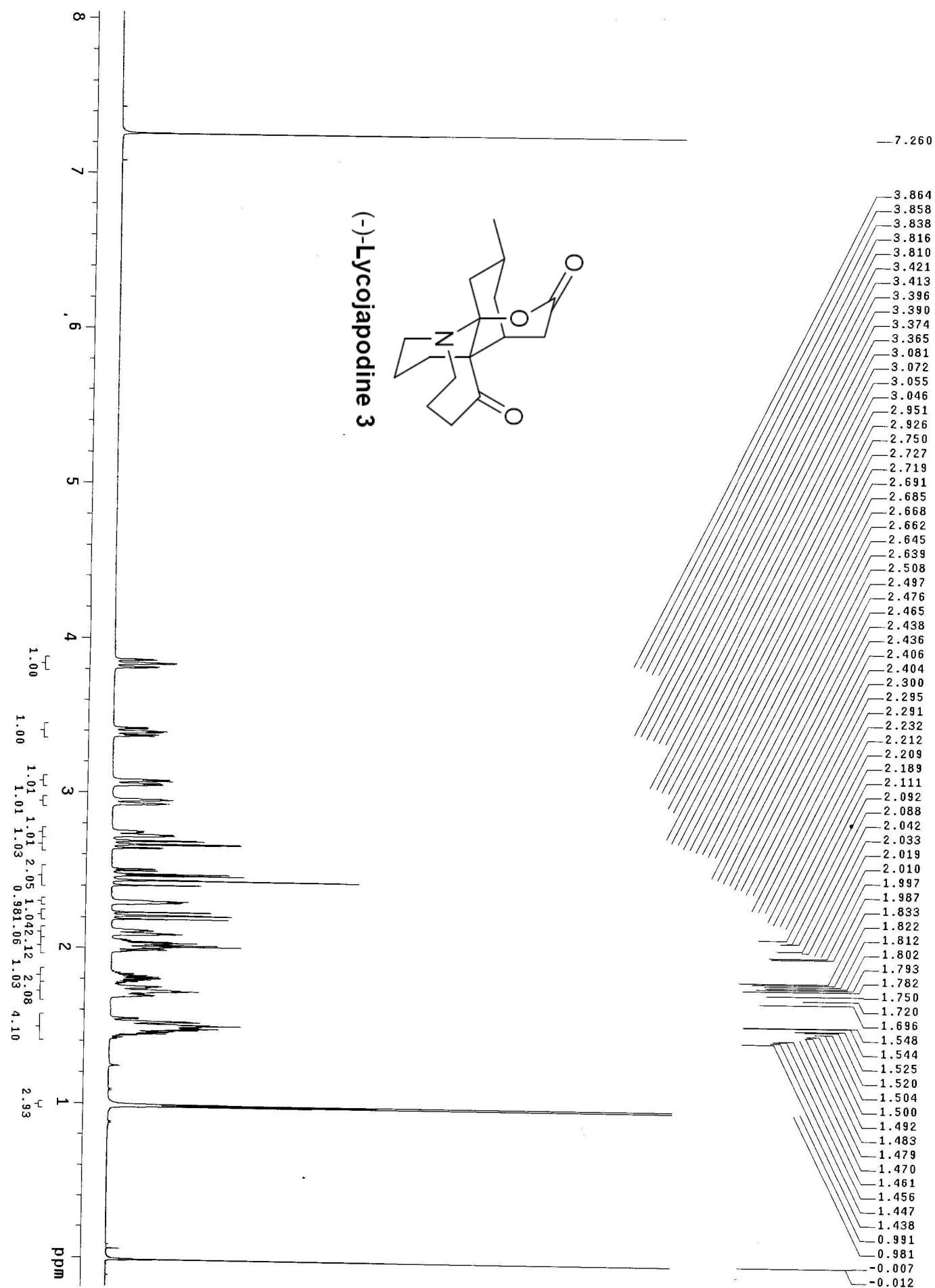
Our synthetic lycojapodine A (**3**).

Natural Lycopodium A

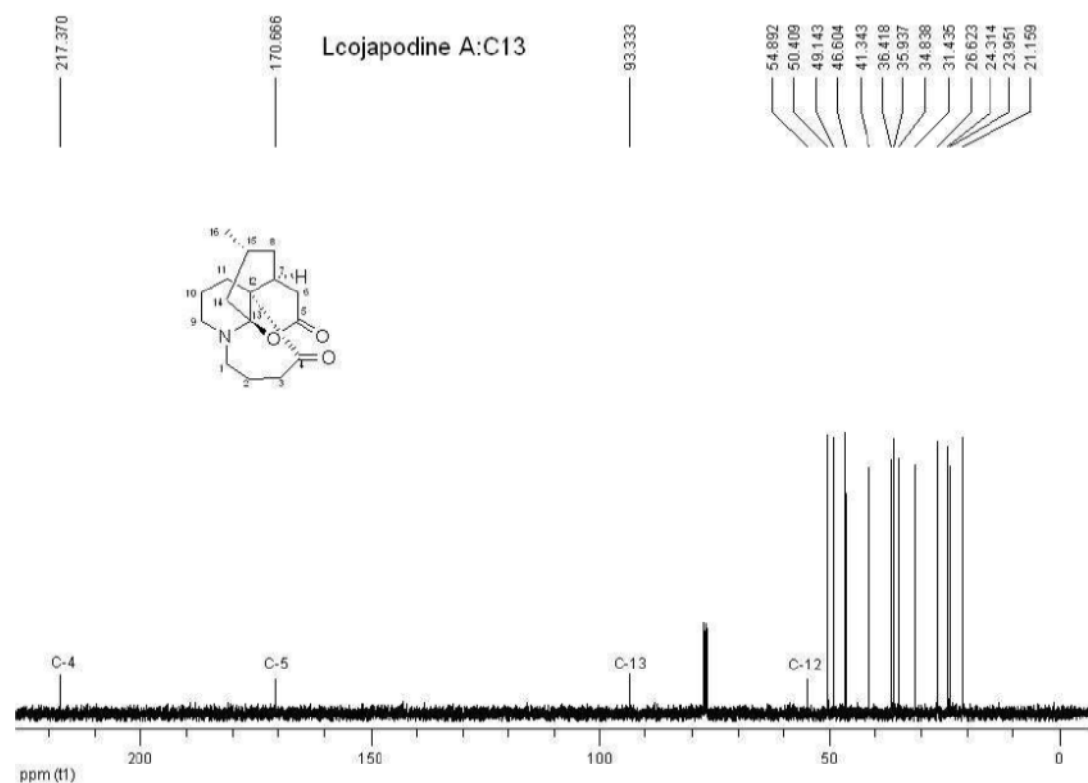
Lycopodium A: H



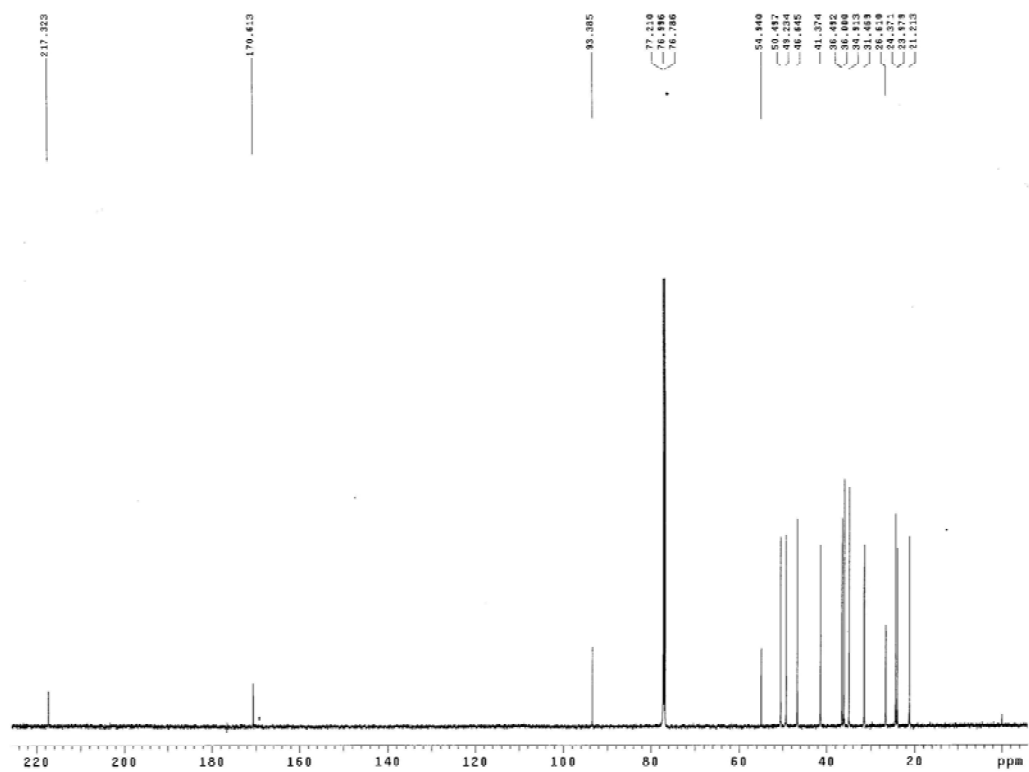
Synthetic Lycopodine A



¹³C NMR of natural and synthetic lycojapodine A (**3**)

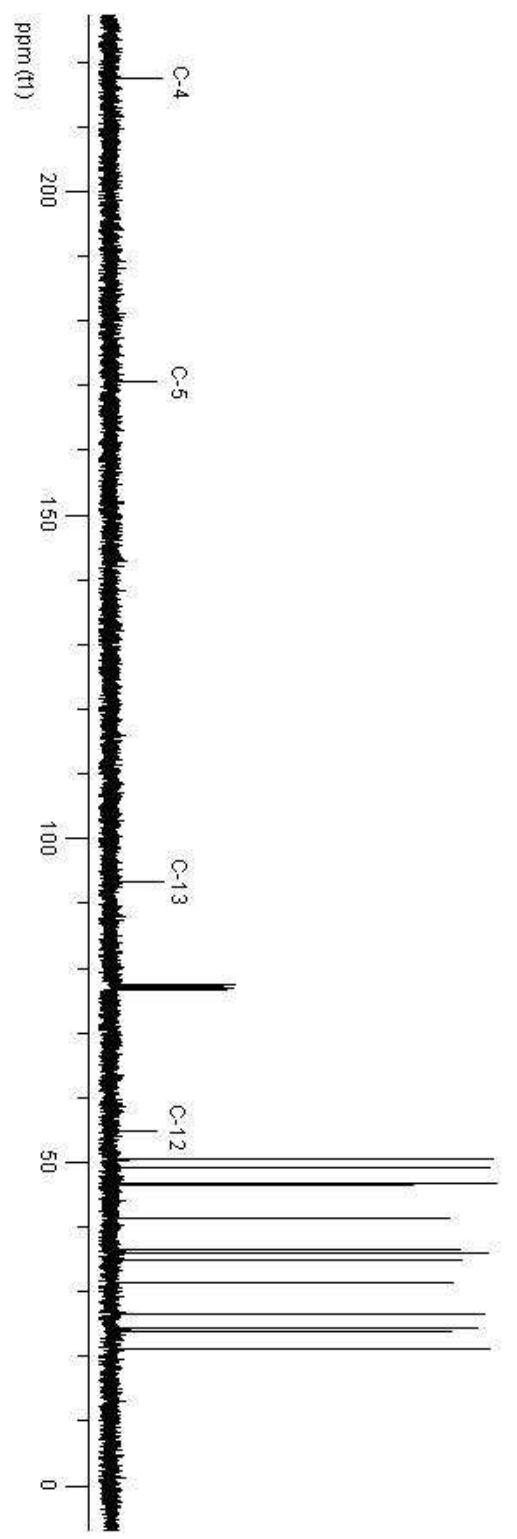
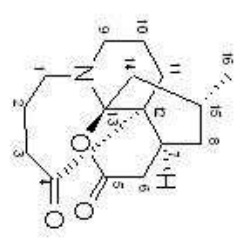


Natural lycojapodine A (**3**), see: J. He, X.-Q. Chen, M.-M. Li, Y. Zhao, G. Xu, X. Cheng, L.-Y. Peng, M.-J. Xie, Y.-T. Zheng, Y.-P. Wang and Q.-S. Zhao, *Org. Lett.* 2009, **11**, 1397.

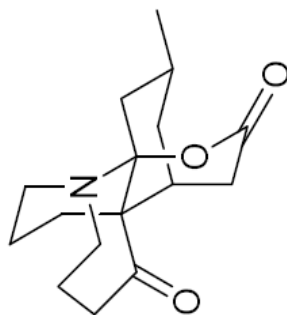


Our synthetic lycojapodine A (**3**).

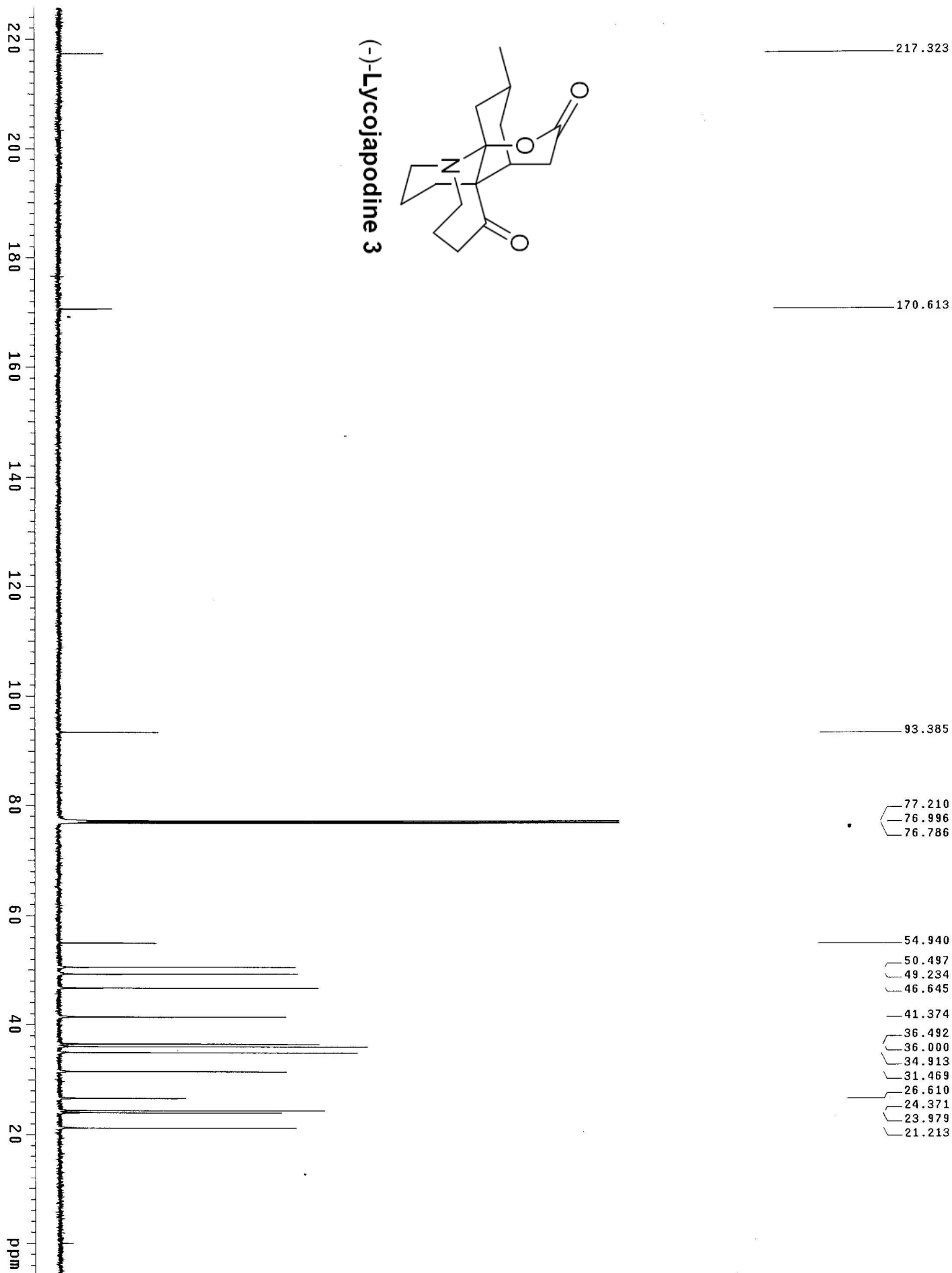
Natural Lycopodine A



Synthetic Lycojapodine A



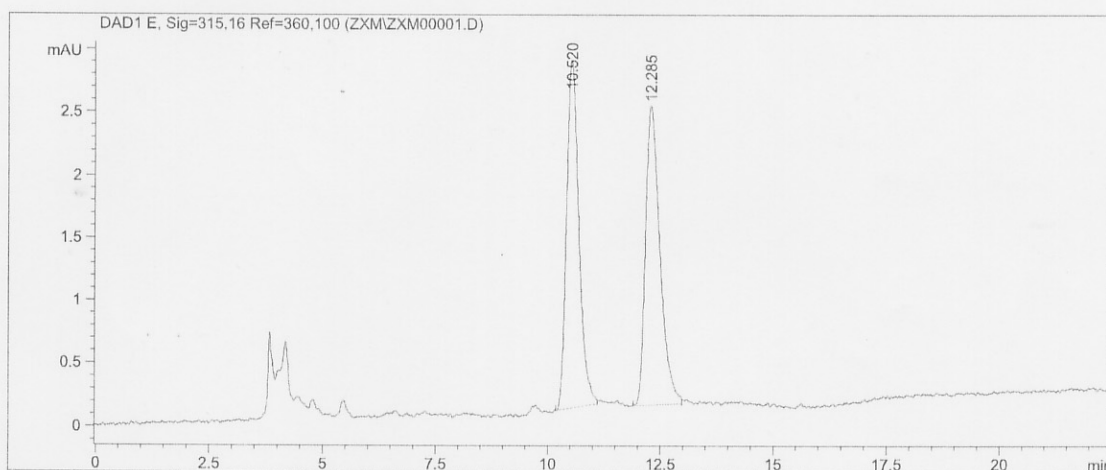
(-)-Lycojapodine 3



Data File C:\HPCHEM\1\DATA\ZXM\ZXM00001.D
IC hex:ipr=90:10 1ml/min

Sample Name: zxm-1133-1

=====
Injection Date : 7/25/2002 1:31:40 AM
Sample Name : zxm-1133-1 Location : Vial 1
Acq. Operator : zhangxiaoming
Acq. Method : C:\HPCHEM\1\METHODS\ZHANGQW.M
Last changed : 7/25/2002 12:51:45 AM by zhangxiaoming
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\ZHANGQW.M
Last changed : 7/25/2002 1:56:09 AM by zhangxiaoming
(modified after loading)
=====



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 E, Sig=315,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.520	PB	0.2906	52.71849	2.76242	49.9239
2	12.285	PB	0.3319	52.87917	2.37363	50.0761

Totals : 105.59766 5.13604

Results obtained with enhanced integrator!

=====
Summed Peaks Report
=====

Signal 1: DAD1 E, Sig=315,16 Ref=360,100

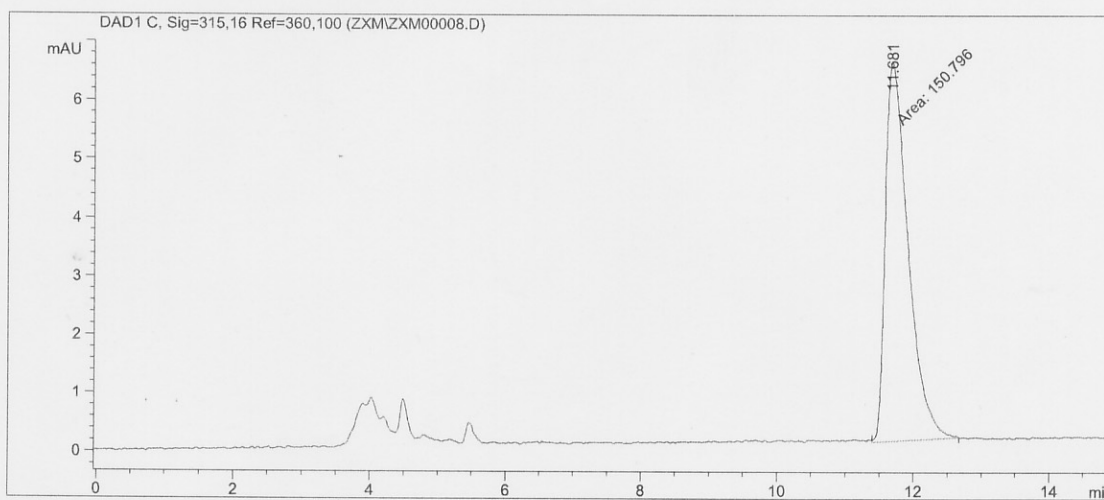
=====
Final Summed Peaks Report
=====

Signal 1: DAD1 E, Sig=315,16 Ref=360,100

Data File C:\HPCHEM\1\DATA\ZXM\ZXM00008.D
IC hex:ipr=90:10 1ml/min

Sample Name: zxm-1156-1

=====
Injection Date : 7/25/2002 12:52:37 AM
Sample Name : zxm-1156-1 Location : Vial 1
Acq. Operator : zhangxiaoming
Method : C:\HPCHEM\1\METHODS\ZHANGQW.M
Last changed : 7/25/2002 12:08:36 AM by zhangxiaoming
(modified after loading)
=====



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 C, Sig=315,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.681	MM T	0.3889	150.79610	6.46301	100.0000

Totals : 150.79610 6.46301

Results obtained with enhanced integrator!

=====
Summed Peaks Report
=====

Signal 1: DAD1 C, Sig=315,16 Ref=360,100

=====
Final Summed Peaks Report
=====

Signal 1: DAD1 C, Sig=315,16 Ref=360,100