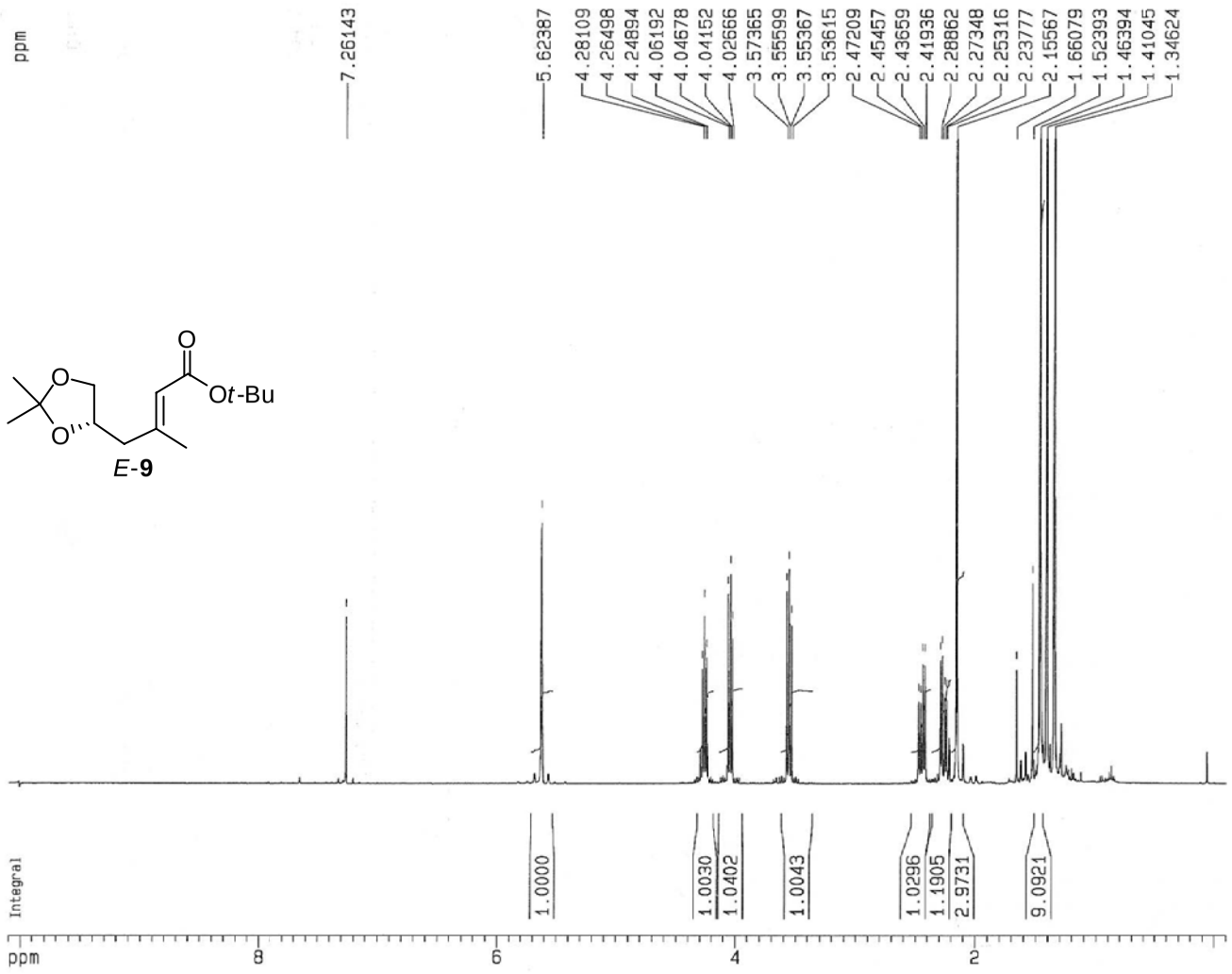
# Supplementary Information

# Total Synthesis of Potent Antitumor Macrolide, (-)-Zampanolide: An Oxidative Intramolecular Cyclization-Based Strategy

**Arun K. Ghosh,* Xu Cheng, Ruoli Bai, and Ernest Hamel**

$^{1}$H NMR and $^{13}$C NMR spectra for all new compounds and HPLC traces for compounds **1**, **16** and **39**.

H1 standard parameters, CDCl3, QNP probe.




*E*-**9**

Current Data Parameters

| | |
|---|---|
| NAME | chx_10_027_02 |
| EXPNO | 1 |
| PROCNO | 1 |

F2 - Acquisition Parameters

| | |
|---|---|
| Date_ | 20120127 |
| Time | 20.32 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H |
| PULPROG | zg30 |
| TD | 16384 |
| SOLVENT | CDCl3 |
| NS | 10 |
| DS | 2 |
| SWH | 5617.978 Hz |
| FIDRES | 0.342894 Hz |
| AQ | 1.4582260 sec |
| RG | 360 |
| DW | 89.000 usec |
| DE | 127.14 usec |
| TE | 300.0 K |
| D1 | 2.00000000 sec |
| P1 | 9.50 usec |
| SFO1 | 400.1694000 MHz |
| NUCLEUS | 1H |

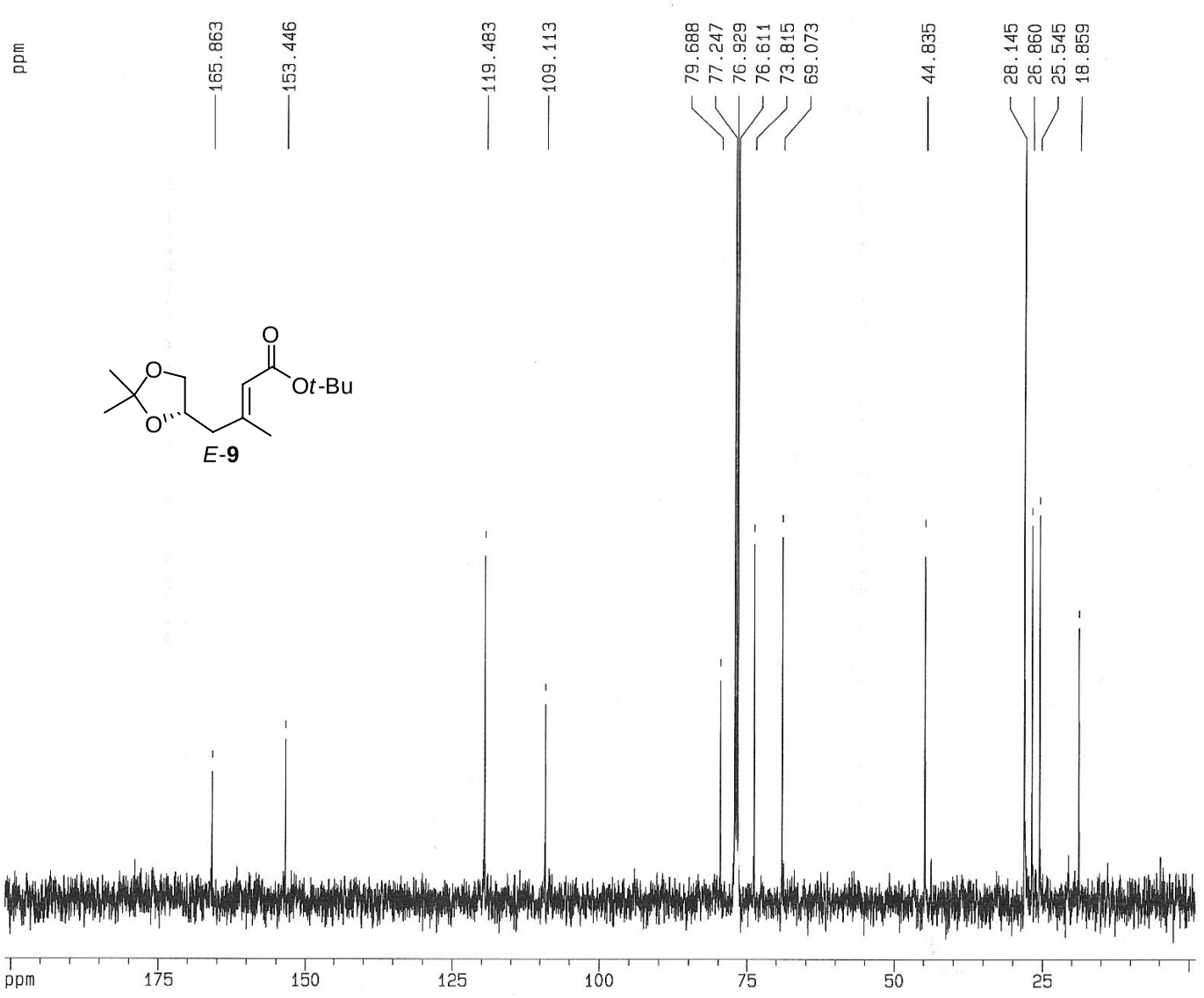
F2 - Processing parameters

| | |
|---|---|
| SI | 8192 |
| SF | 400.1669660 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |

1D NMR plot parameters

| | |
|---|---|
| CX | 20.00 cm |
| F1P | 10.100 ppm |
| F1 | 4041.69 Hz |
| F2P | -0.100 ppm |
| F2 | -40.02 Hz |
| PPMCM | 0.51000 ppm/cm |
| HZCM | 204.08516 Hz/cm |

C13 standard parameters, CDCl3, QNP probe.




Current Data Parameters

| | |
|---|---|
| NAME | chx_10_027_02 |
| EXPNO | 2 |
| PROCNO | 2 |

F2 - Acquisition Parameters

| | |
|---|---|
| Date_ | 20120127 |
| Time | 20.34 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H |
| PULPROG | zgpg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 83 |
| DS | 2 |
| SWH | 23809.523 Hz |
| FIDRES | 0.726609 Hz |
| AQ | 0.6881780 sec |
| RG | 16384 |
| DW | 21.000 usec |
| DE | 30.00 usec |
| TE | 300.0 K |
| D12 | 0.0000200 sec |
| DL6 | 21.00 dB |
| D1 | 2.00000000 sec |
| CPDPRG | waltz16 |
| P31 | 76.00 usec |
| D11 | 0.0300000 sec |
| DL5 | 18.00 dB |
| P1 | 8.60 usec |
| SFO1 | 100.6330000 MHz |
| NUCLEUS | 13C |

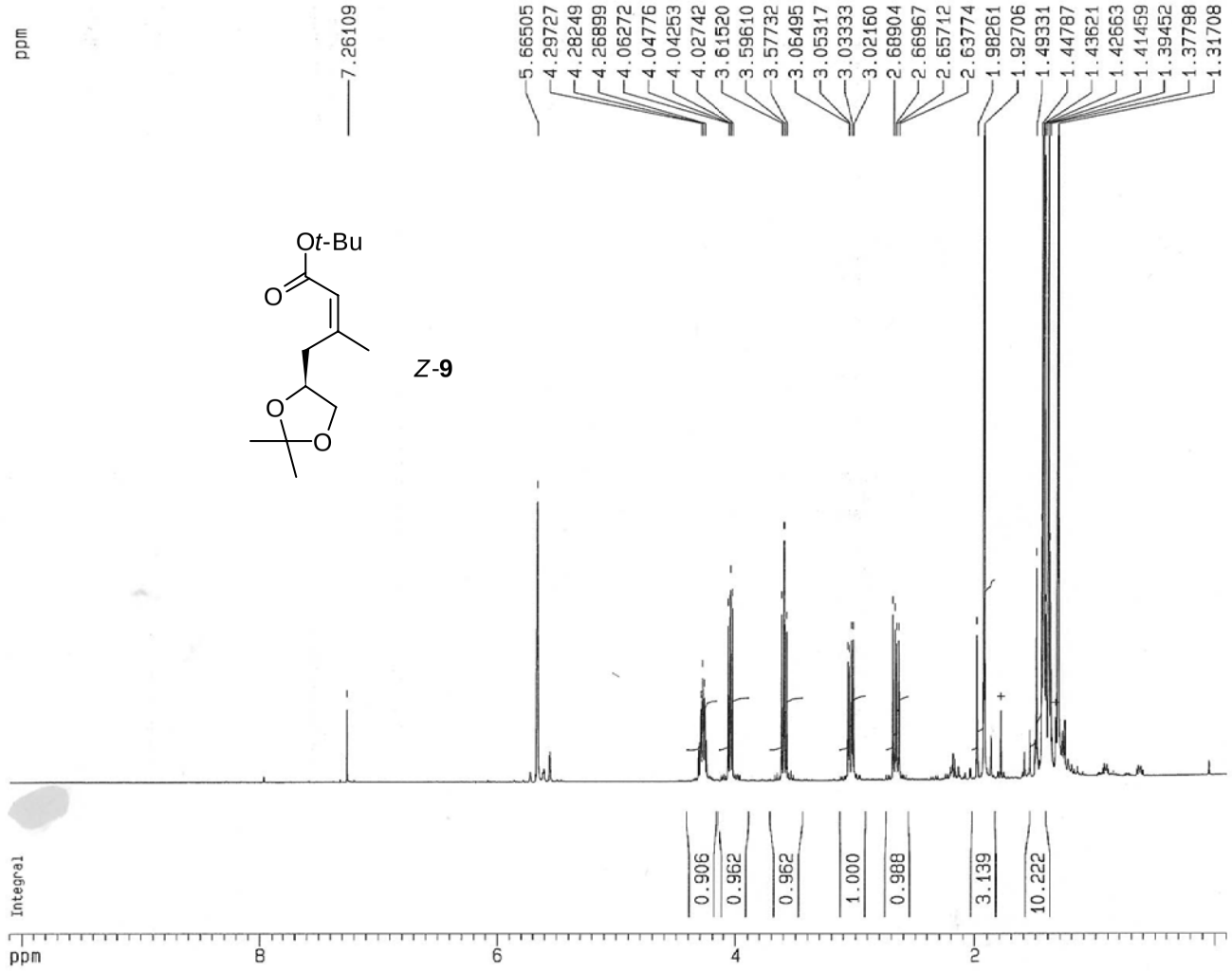
F2 - Processing parameters

| | |
|---|---|
| SI | 16384 |
| SF | 100.6220720 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 3.00 Hz |
| GB | 0 |
| PC | 1.00 |

1D NMR plot parameters

| | |
|---|---|
| CX | 20.00 cm |
| F1P | 201.000 ppm |
| F1 | 20225.04 Hz |
| F2P | -1.000 ppm |
| F2 | -100.62 Hz |
| PPMCM | 10.10000 ppm/cm |
| HZCM | 1016.28290 Hz/cm |

H1 standard parameters, CDCl3, QNP probe.







*Z*-**9**

Current Data Parameters

| | |
|---|---|
| NAME | chx_10_027_01 |
| EXPNO | 1 |
| PROCNO | 1 |

F2 - Acquisition Parameters

| | |
|---|---|
| Date_ | 20120127 |
| Time | 20.19 |
| INSTRUM | spect |
| PROBHD | 5 mm GNP 1H |
| PULPROG | zg30 |
| TD | 16384 |
| SOLVENT | CDCl3 |
| NS | 16 |
| DS | 2 |
| SWH | 5617.978 Hz |
| FIDRES | 0.342894 Hz |
| AQ | 1.4582260 sec |
| RG | 128 |
| DW | 89.000 usec |
| DE | 127.14 usec |
| TE | 300.0 K |
| D1 | 2.00000000 sec |
| P1 | 9.50 usec |
| SFO1 | 400.1694000 MHz |
| NUCLEUS | 1H |

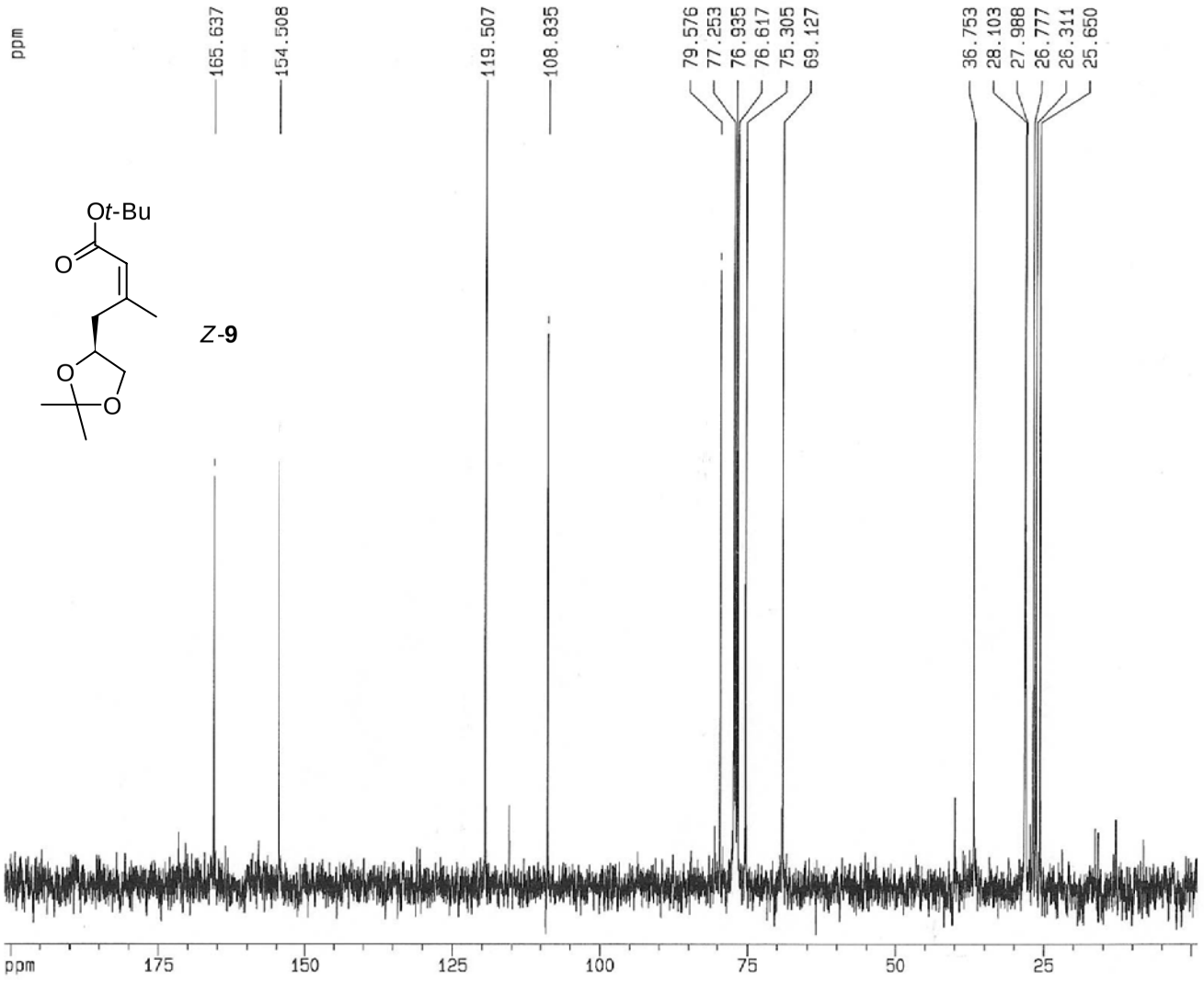
F2 - Processing parameters

| | |
|---|---|
| SI | 8192 |
| SF | 400.1669660 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |

1D NMR plot parameters

| | |
|---|---|
| CX | 20.00 cm |
| F1P | 10.100 ppm |
| F1 | 4041.69 Hz |
| F2P | -0.100 ppm |
| F2 | -40.02 Hz |
| PPMCM | 0.51000 ppm/cm |
| HZCM | 204.08516 Hz/cm |

C13 standard parameters, CDCl3, QNP probe.




Current Data Parameters

| | |
|---|---|
| NAME | chx_10_027_01 |
| EXPNO | 2 |
| PROCNO | 2 |

F2 - Acquisition Parameters

| | |
|---|---|
| Date_ | 20120127 |
| Time | 20.22 |
| INSTRUM | spect |
| PROBHD | 5 mm GNP 1H |
| PULPROG | zgpg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 130 |
| DS | 2 |
| SWH | 23809.523 Hz |
| FIDRES | 0.726609 Hz |
| AQ | 0.6881780 sec |
| RG | 16384 |
| DW | 21.000 usec |
| DE | 30.00 usec |
| TE | 300.0 K |
| D12 | 0.0000200 sec |
| DL6 | 21.00 dB |
| D1 | 2.00000000 sec |
| CPDPRG | waltz16 |
| P31 | 76.00 usec |
| D11 | 0.0300000 sec |
| DL5 | 18.00 dB |
| P1 | 8.60 usec |
| SFO1 | 100.6330000 MHz |
| NUCLEUS | 13C |

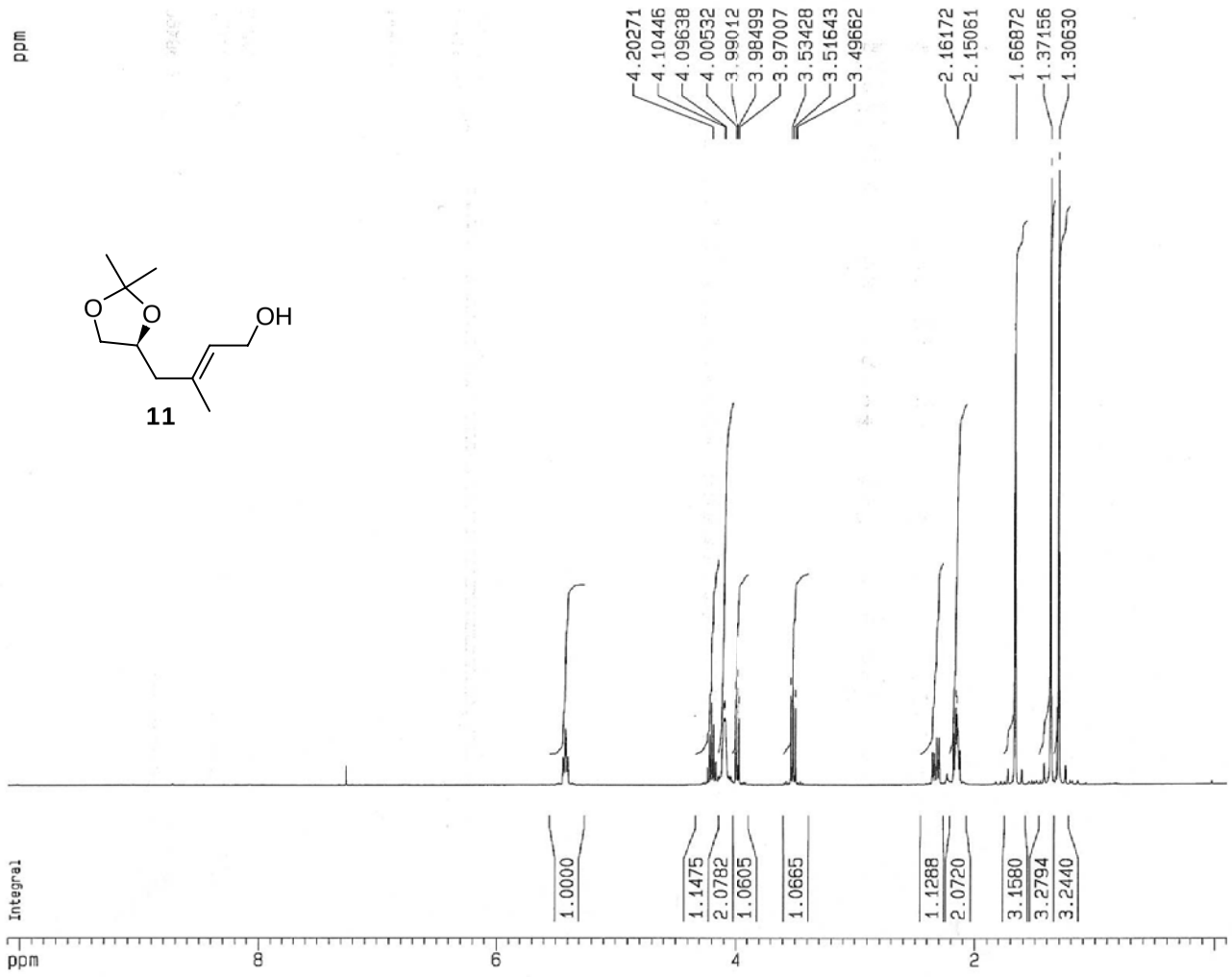
F2 - Processing parameters

| | |
|---|---|
| SI | 16384 |
| SF | 100.6220720 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 3.00 Hz |
| GB | 0 |
| PC | 1.00 |

1D NMR plot parameters

| | |
|---|---|
| CX | 20.00 cm |
| F1P | 201.000 ppm |
| F1 | 20225.04 Hz |
| F2P | -1.000 ppm |
| F2 | -100.62 Hz |
| PPMCM | 10.10000 ppm/cm |
| HZCM | 1016.28290 Hz/cm |

H1 standard parameters, CDCl3, GNP probe.




Current Data Parameters

| | |
|---|---|
| NAME | chx_10_028_02 |
| EXPNO | 1 |
| PROCNO | 1 |

F2 - Acquisition Parameters

| | |
|---|---|
| Date_ | 20120128 |
| Time | 18.47 |
| INSTRUM | spect |
| PROBHD | 5 mm GNP 1H |
| PULPROG | zg30 |
| TD | 16384 |
| SOLVENT | CDCl3 |
| NS | 13 |
| DS | 2 |
| SWH | 5617.978 Hz |
| FIDRES | 0.342894 Hz |
| AQ | 1.4582260 sec |
| RG | 128 |
| DW | 89.000 usec |
| DE | 127.14 usec |
| TE | 300.0 K |
| D1 | 2.00000000 sec |
| P1 | 9.50 usec |
| SFO1 | 400.1694000 MHz |
| NUCLEUS | 1H |

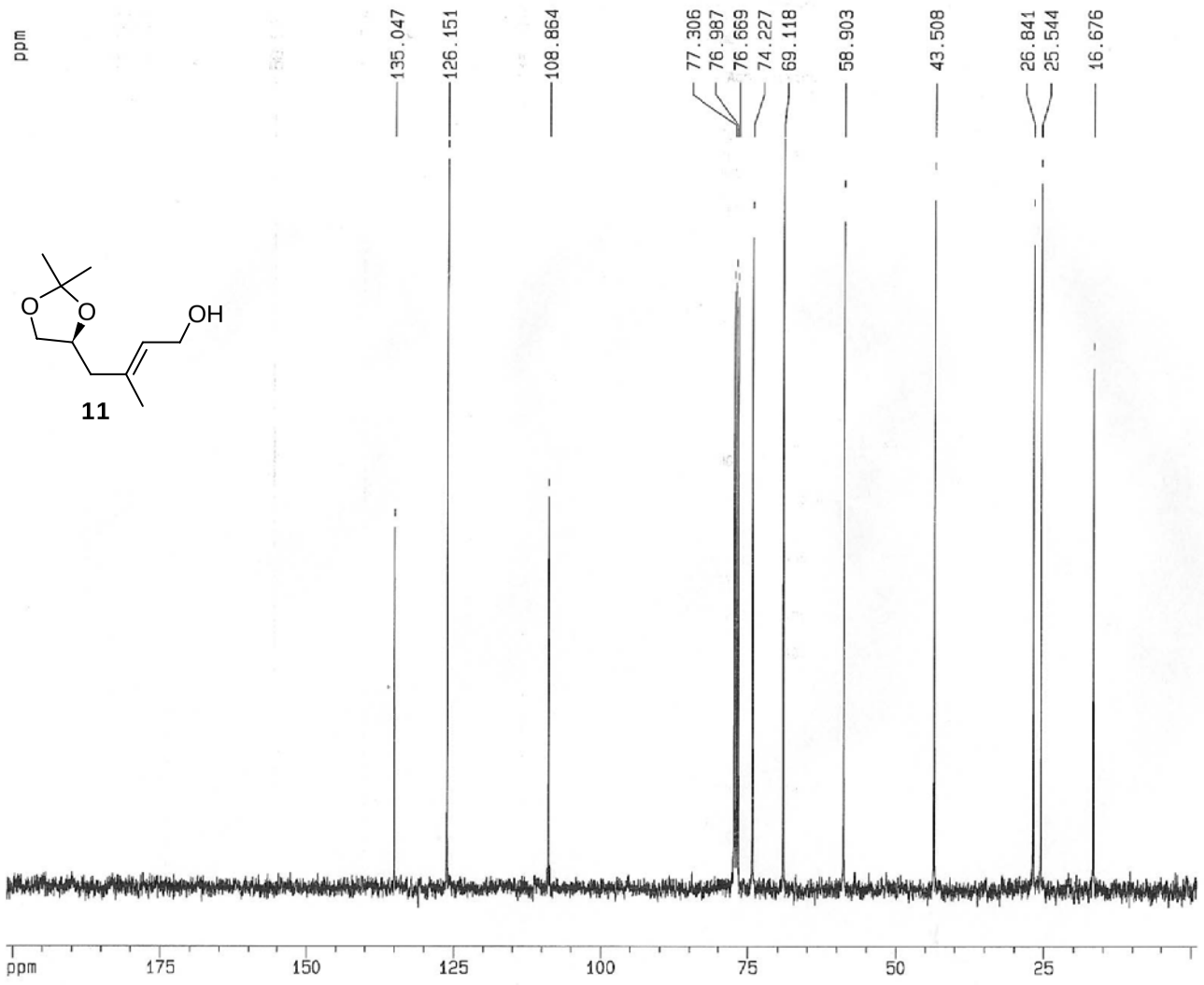
F2 - Processing parameters

| | |
|---|---|
| SI | 8192 |
| SF | 400.1669660 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |

1D NMR plot parameters

| | |
|---|---|
| CX | 20.00 cm |
| F1P | 10.100 ppm |
| F1 | 4041.69 Hz |
| F2P | -0.100 ppm |
| F2 | -40.02 Hz |
| PPMCM | 0.51000 ppm/cm |
| HZCM | 204.08516 Hz/cm |

C13 standard parameters, CDCl3, QNP probe.




Current Data Parameters

| | |
|---|---|
| NAME | chx_10_028_02 |
| EXPNO | 2 |
| PROCNO | 2 |

F2 - Acquisition Parameters

| | |
|---|---|
| Date_ | 20120128 |
| Time | 18.49 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H |
| PULPROG | zgpg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 131 |
| DS | 2 |
| SWH | 23809.523 Hz |
| FIDRES | 0.726609 Hz |
| AQ | 0.6881780 sec |
| RG | 16384 |
| DW | 21.000 usec |
| DE | 30.00 usec |
| TE | 300.0 K |
| D12 | 0.0000200 sec |
| DL6 | 21.00 dB |
| D1 | 2.00000000 sec |
| CPDPRG | waltz16 |
| P31 | 76.00 usec |
| D11 | 0.0300000 sec |
| DL5 | 18.00 dB |
| P1 | 8.60 usec |
| SFO1 | 100.6330000 MHz |
| NUCLEUS | 13C |

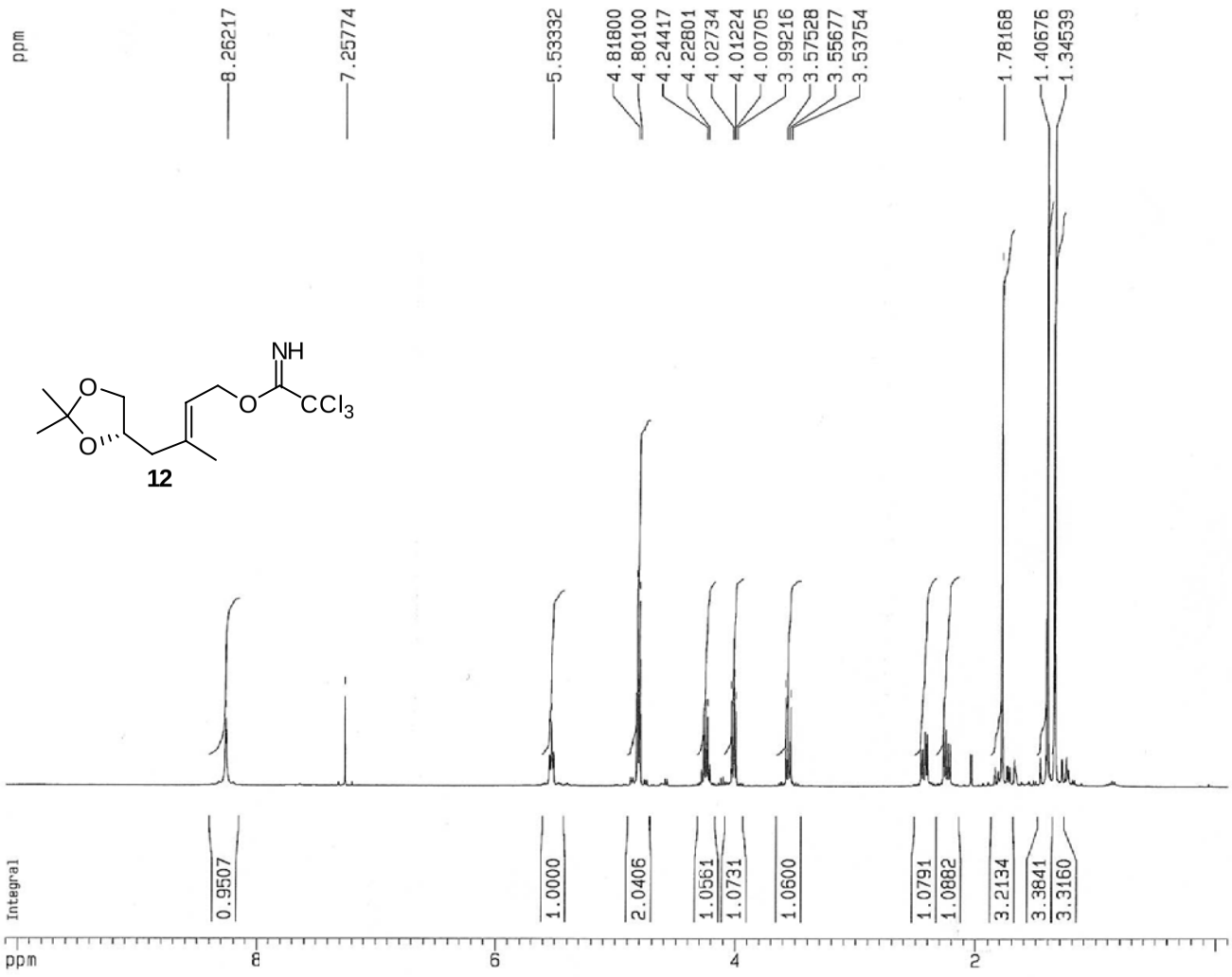
F2 - Processing parameters

| | |
|---|---|
| SI | 16384 |
| SF | 100.6220720 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 3.00 Hz |
| GB | 0 |
| PC | 1.00 |

1D NMR plot parameters

| | |
|---|---|
| CX | 20.00 cm |
| F1P | 201.000 ppm |
| F1 | 20225.04 Hz |
| F2P | -1.000 ppm |
| F2 | -100.62 Hz |
| PPMCM | 10.10000 ppm/cm |
| HZCM | 1016.28290 Hz/cm |

H1 standard parameters, CDCl3, QNP probe.




Current Data Parameters

| | |
|---|---|
| NAME | chx_10_030_01 |
| EXPNO | 1 |
| PROCNO | 1 |

F2 - Acquisition Parameters

| | |
|---|---|
| Date_ | 20120131 |
| Time | 21.16 |
| INSTRUM | spect |
| PROBHD | 5 mm GNP 1H |
| PULPROG | zg30 |
| TD | 16384 |
| SOLVENT | CDCl3 |
| NS | 8 |
| DS | 2 |
| SWH | 5617.978 Hz |
| FIDRES | 0.342894 Hz |
| AQ | 1.4582260 sec |
| RG | 715 |
| DW | 89.000 usec |
| DE | 127.14 usec |
| TE | 300.0 K |
| D1 | 2.00000000 sec |
| P1 | 9.50 usec |
| SFO1 | 400.1694000 MHz |
| NUCLEUS | 1H |

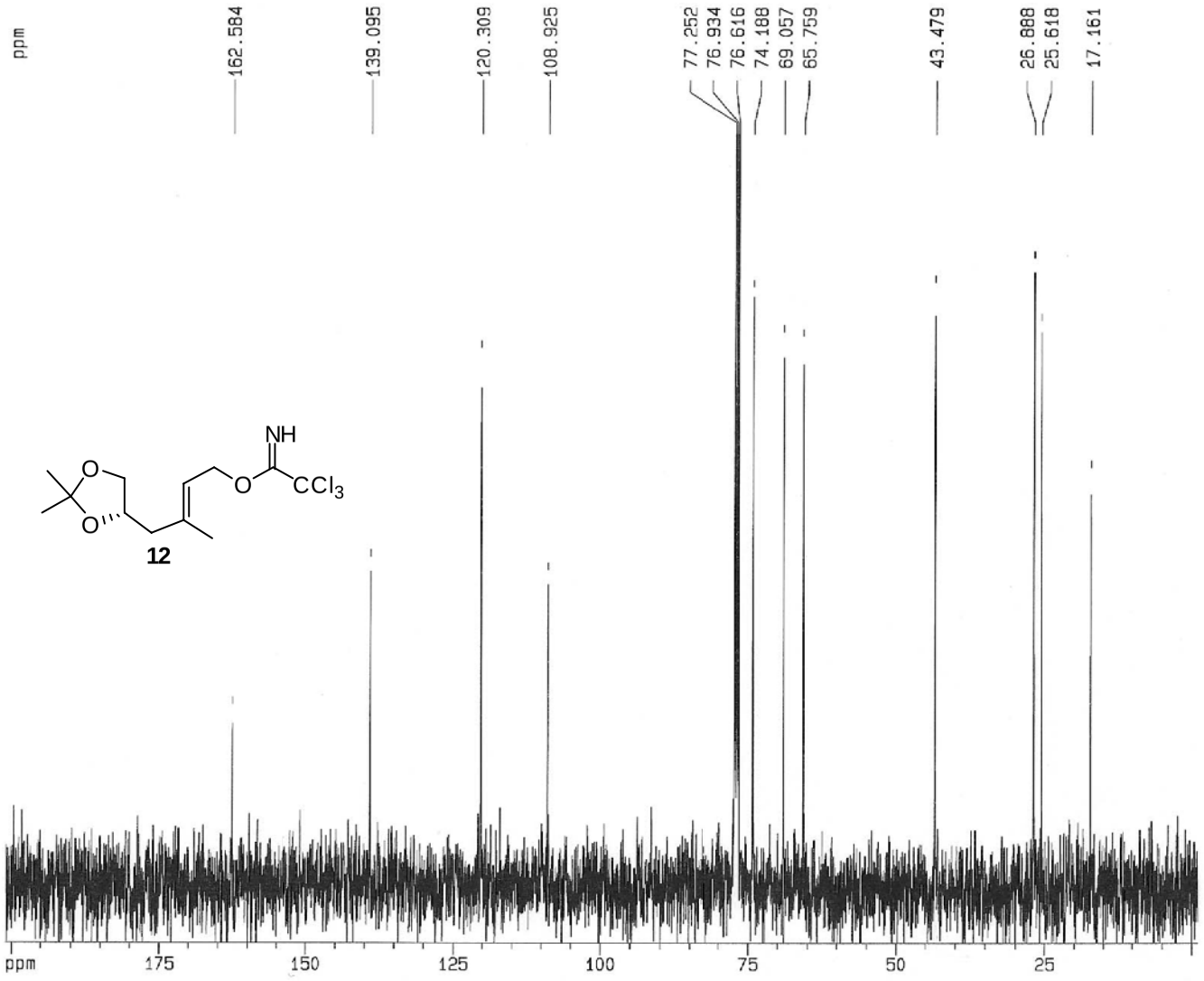
F2 - Processing parameters

| | |
|---|---|
| SI | 8192 |
| SF | 400.1669660 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |

1D NMR plot parameters

| | |
|---|---|
| CX | 20.00 cm |
| F1P | 10.100 ppm |
| F1 | 4041.69 Hz |
| F2P | -0.100 ppm |
| F2 | -40.02 Hz |
| PPMCM | 0.51000 ppm/cm |
| HZCM | 204.08516 Hz/cm |

C13 standard parameters, CDCl3, QNP probe.




Current Data Parameters

| | |
|---|---|
| NAME | chx_10_030_01 |
| EXPNO | 2 |
| PROCNO | 2 |

F2 - Acquisition Parameters

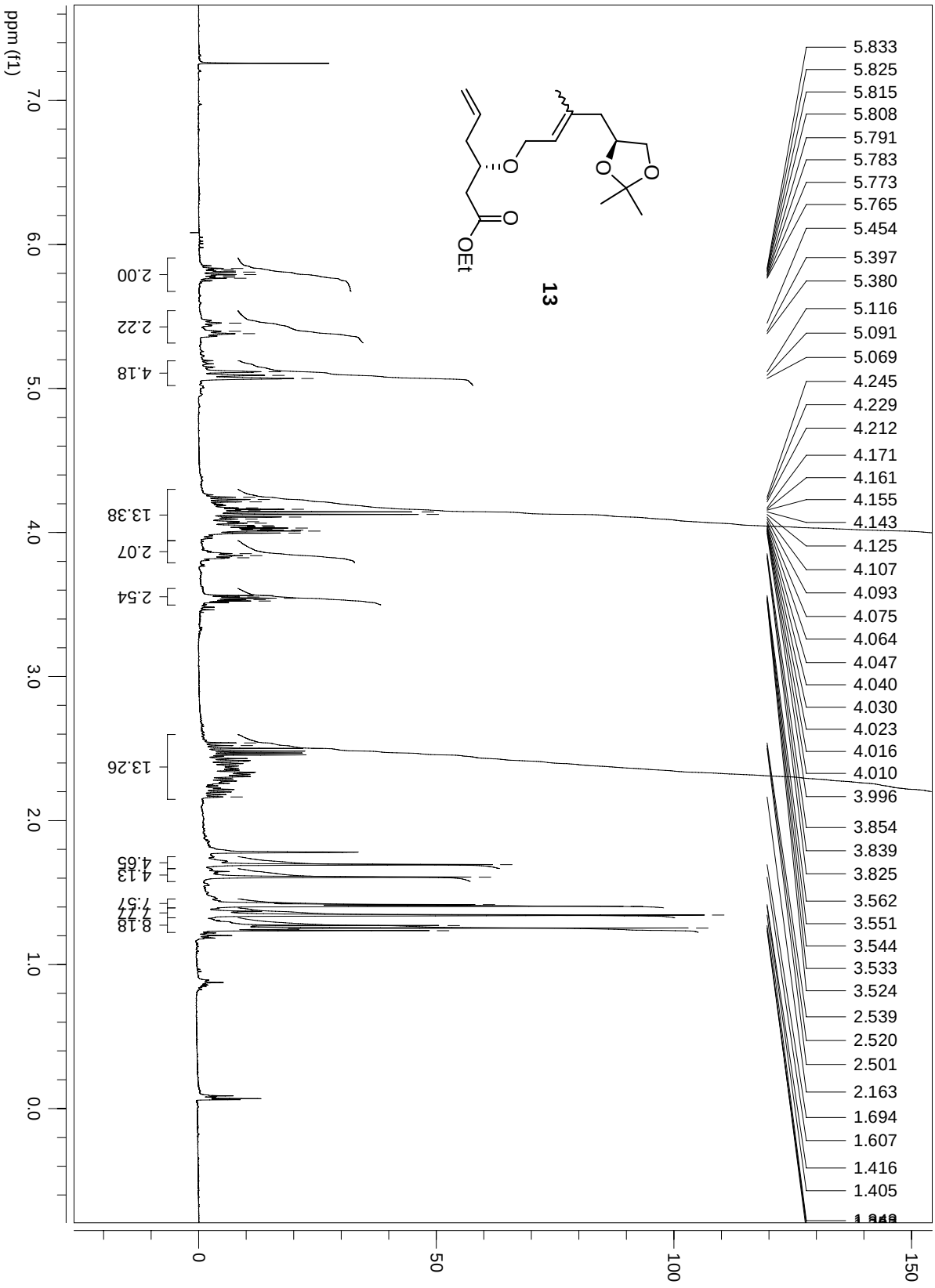
| | |
|---|---|
| Date_ | 20120131 |
| Time | 21.17 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H |
| PULPROG | zgpg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 73 |
| DS | 2 |
| SWH | 23809.523 Hz |
| FIDRES | 0.726609 Hz |
| AQ | 0.6881780 sec |
| RG | 16384 |
| DW | 21.000 usec |
| DE | 30.00 usec |
| TE | 300.0 K |
| D12 | 0.0000200 sec |
| DL6 | 21.00 dB |
| D1 | 2.00000000 sec |
| CPDPRG | waltz16 |
| P31 | 76.00 usec |
| D11 | 0.0300000 sec |
| DL5 | 18.00 dB |
| P1 | 8.50 usec |
| SFO1 | 100.6330000 MHz |
| NUCLEUS | 13C |

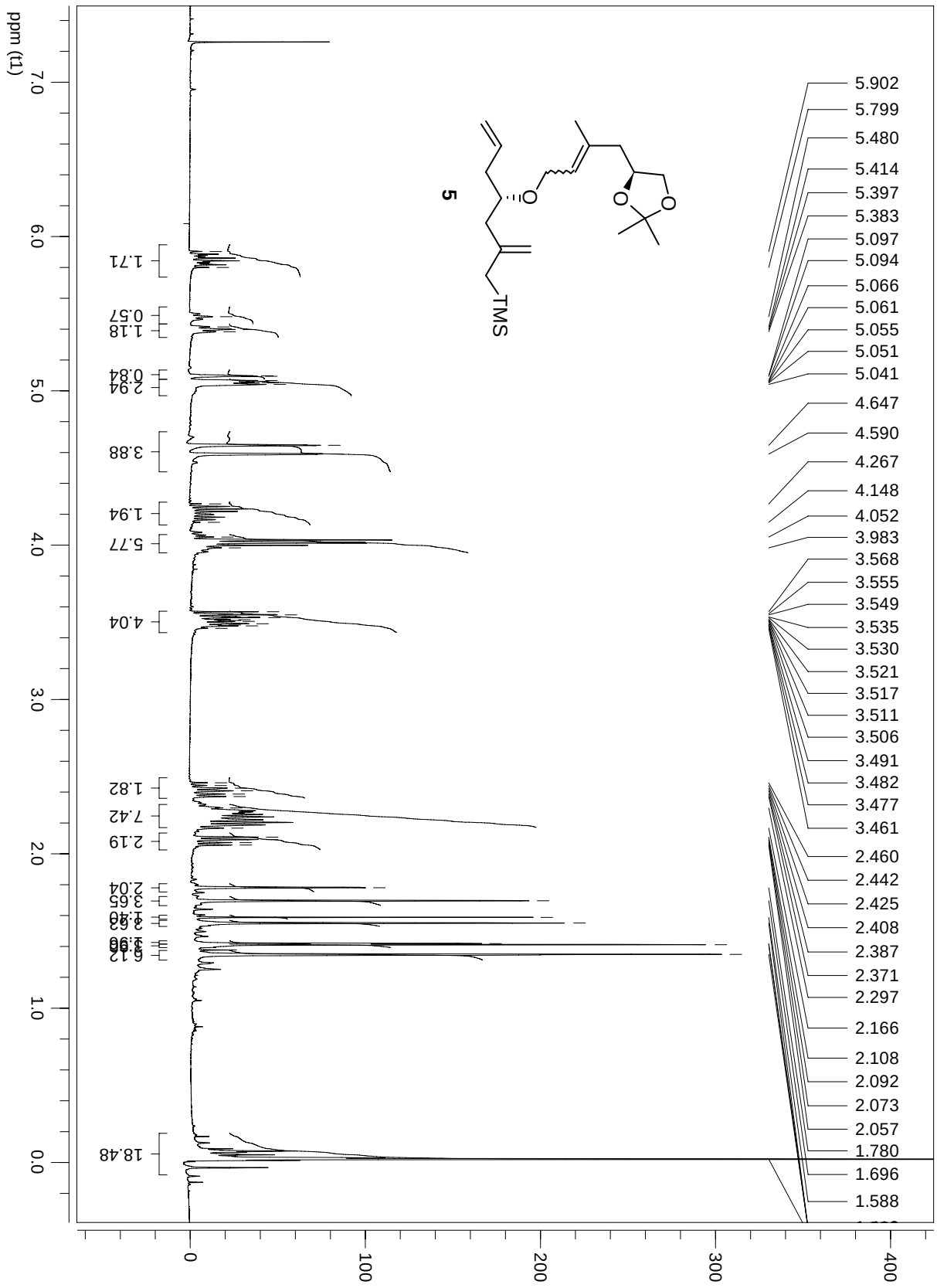
F2 - Processing parameters

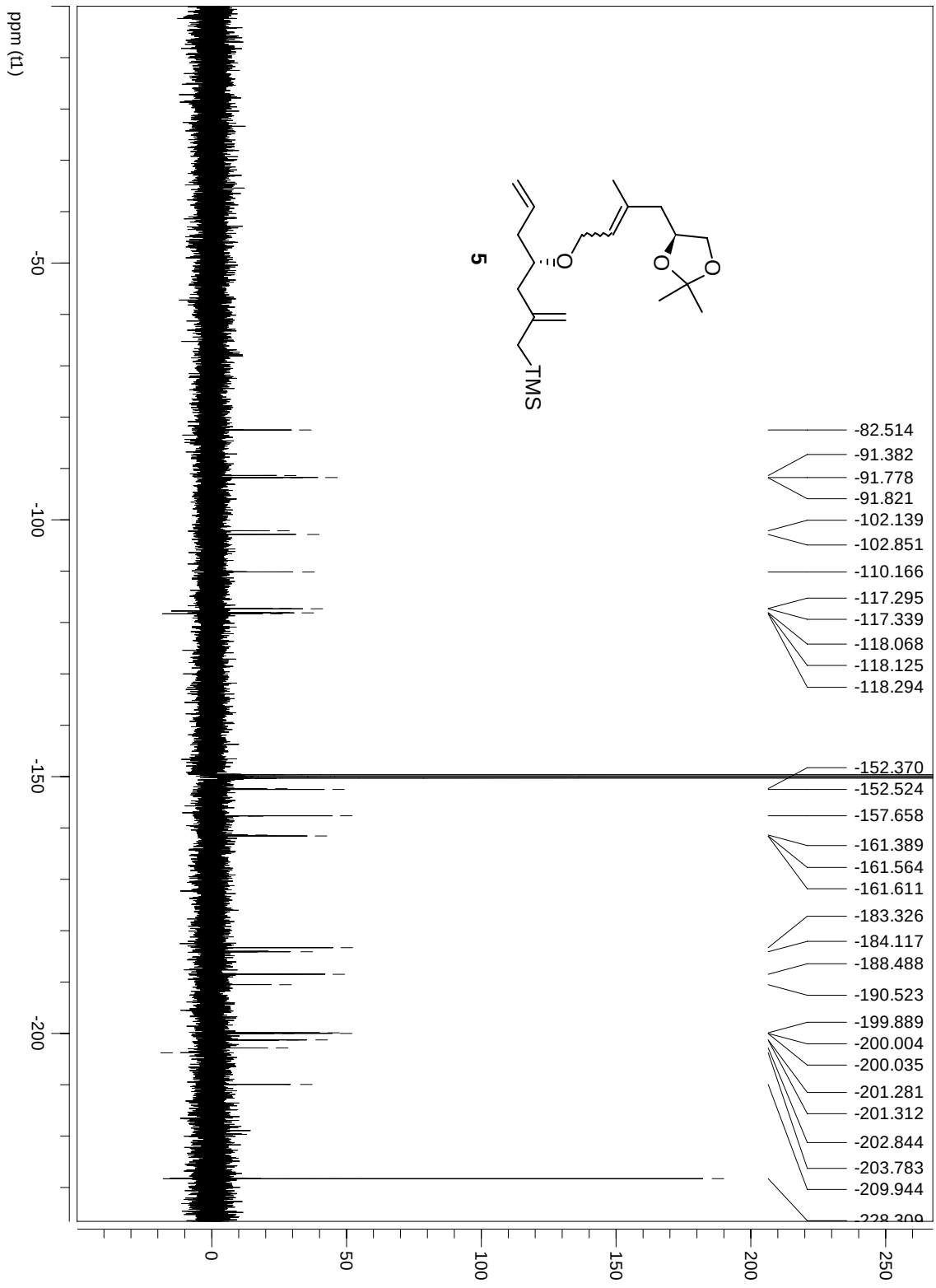
| | |
|---|---|
| SI | 16384 |
| SF | 100.6220720 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 3.00 Hz |
| GB | 0 |
| PC | 1.00 |

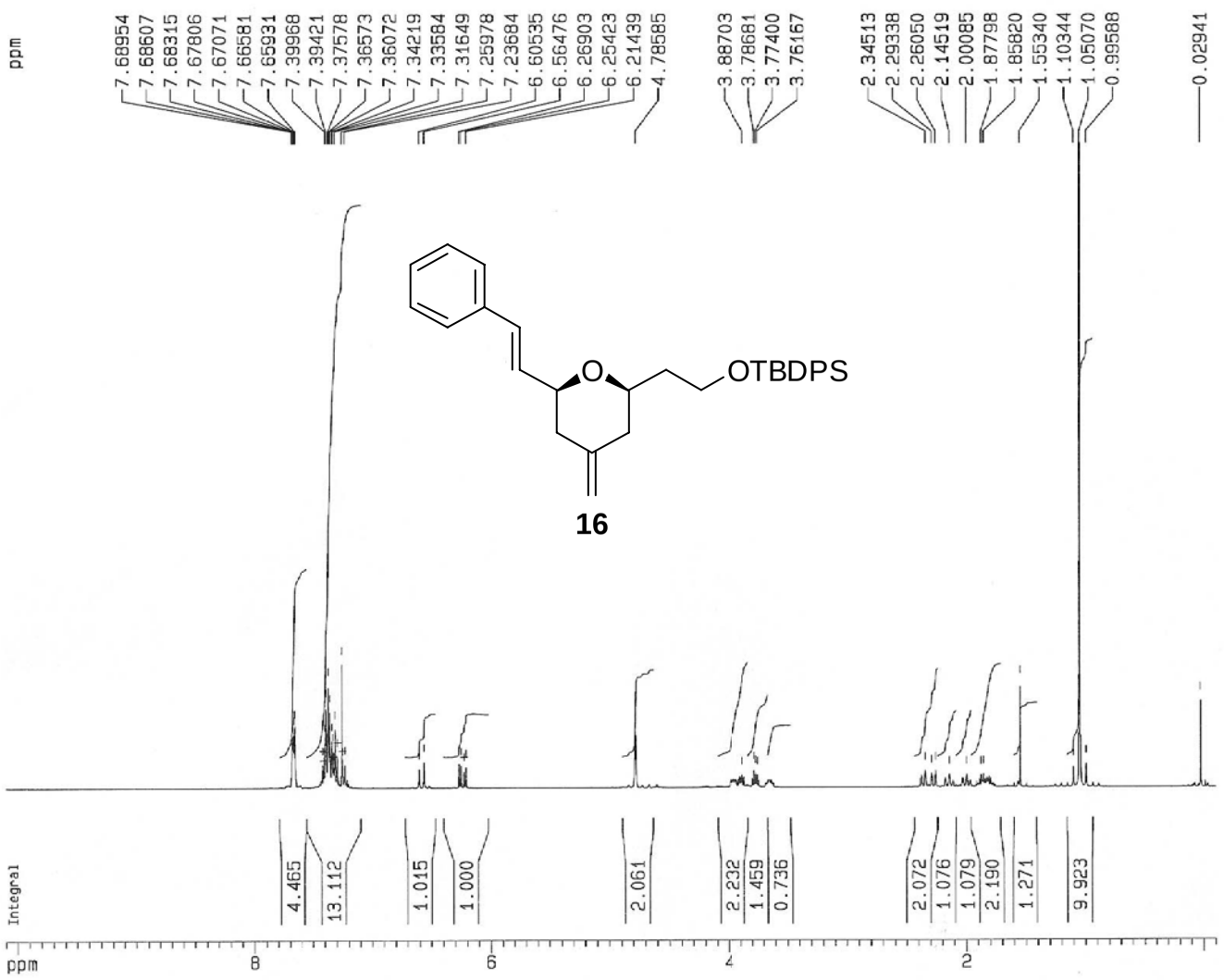
1D NMR plot parameters

| | |
|---|---|
| CX | 20.00 cm |
| F1P | 201.000 ppm |
| F1 | 20225.04 Hz |
| F2P | -1.000 ppm |
| F2 | -100.62 Hz |
| PPMCM | 10.10000 ppm/cm |
| HZCM | 1016.28290 Hz/cm |


ppm (f1)
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
50
100
150
OEt
O
O
O
O
13
2.00
2.22
4.18
13.38
2.07
2.54
13.26
4.65
4.13
7.57
7.77
8.18
5.833
5.825
5.815
5.808
5.791
5.783
5.773
5.765
5.454
5.397
5.380
5.116
5.091
5.069
4.245
4.229
4.212
4.171
4.161
4.155
4.143
4.125
4.107
4.093
4.075
4.064
4.047
4.040
4.030
4.023
4.016
4.010
3.996
3.854
3.839
3.825
3.562
3.551
3.544
3.533
3.524
2.539
2.520
2.501
2.163
1.694
1.607
1.416
1.405


ppm (t1)
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
100
200
300
400
5
TMS
18.48
1.82
7.42
2.19
4.04
1.94
5.77
3.88
2.94
0.84
1.18
0.57
1.71
5.902
5.799
5.480
5.414
5.397
5.383
5.097
5.094
5.066
5.061
5.055
5.051
5.041
4.647
4.590
4.267
4.148
4.052
3.983
3.568
3.555
3.549
3.535
3.530
3.521
3.517
3.511
3.506
3.491
3.482
3.477
3.461
2.460
2.442
2.425
2.408
2.387
2.371
2.297
2.166
2.108
2.092
2.073
2.057
1.780
1.696
1.588


ppm (t1)
-50
-100
-150
-200
0
50
100
150
200
250
5
TMS
-82.514
-91.382
-91.778
-91.821
-102.139
-102.851
-110.166
-117.295
-117.339
-118.068
-118.125
-118.294
-152.370
-152.524
-157.658
-161.389
-161.564
-161.611
-183.326
-184.117
-188.488
-190.523
-199.889
-200.004
-200.035
-201.281
-201.312
-202.844
-203.783
-209.944
-228.309




Current Data Parameters

| | |
|---|---|
| NAME | chx_05_082_02 |
| EXPNO | 1 |
| PROCNO | 1 |

F2 - Acquisition Parameters

| | |
|---|---|
| Date_ | 20101021 |
| Time | 17.13 |
| INSTRUM | spect |
| PROBHD | 5 mm GNP 1H |
| PULPROG | zg30 |
| TD | 16384 |
| SOLVENT | CDCl3 |
| NS | 32 |
| DS | 2 |
| SWH | 5617.978 Hz |
| FIDRES | 0.342894 Hz |
| AQ | 1.4582260 sec |
| RG | 2048 |
| DW | 89.000 usec |
| DE | 127.14 usec |
| TE | 300.0 K |
| D1 | 2.00000000 sec |
| P1 | 9.50 usec |
| SFO1 | 400.1694000 MHz |
| NUCLEUS | 1H |

F2 - Processing parameters

| | |
|---|---|
| SI | 8192 |
| SF | 400.1669660 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |

1D NMR plot parameters

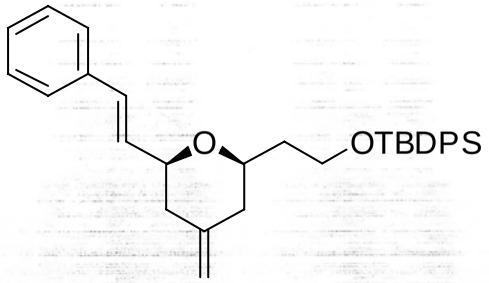
| | |
|---|---|
| CX | 20.00 cm |
| F1P | 10.100 ppm |
| F1 | 4041.69 Hz |
| F2P | -0.100 ppm |
| F2 | -40.02 Hz |
| PPMCM | 0.51000 ppm/cm |
| HZCM | 204.08516 Hz/cm |

chx_05_082_02_c

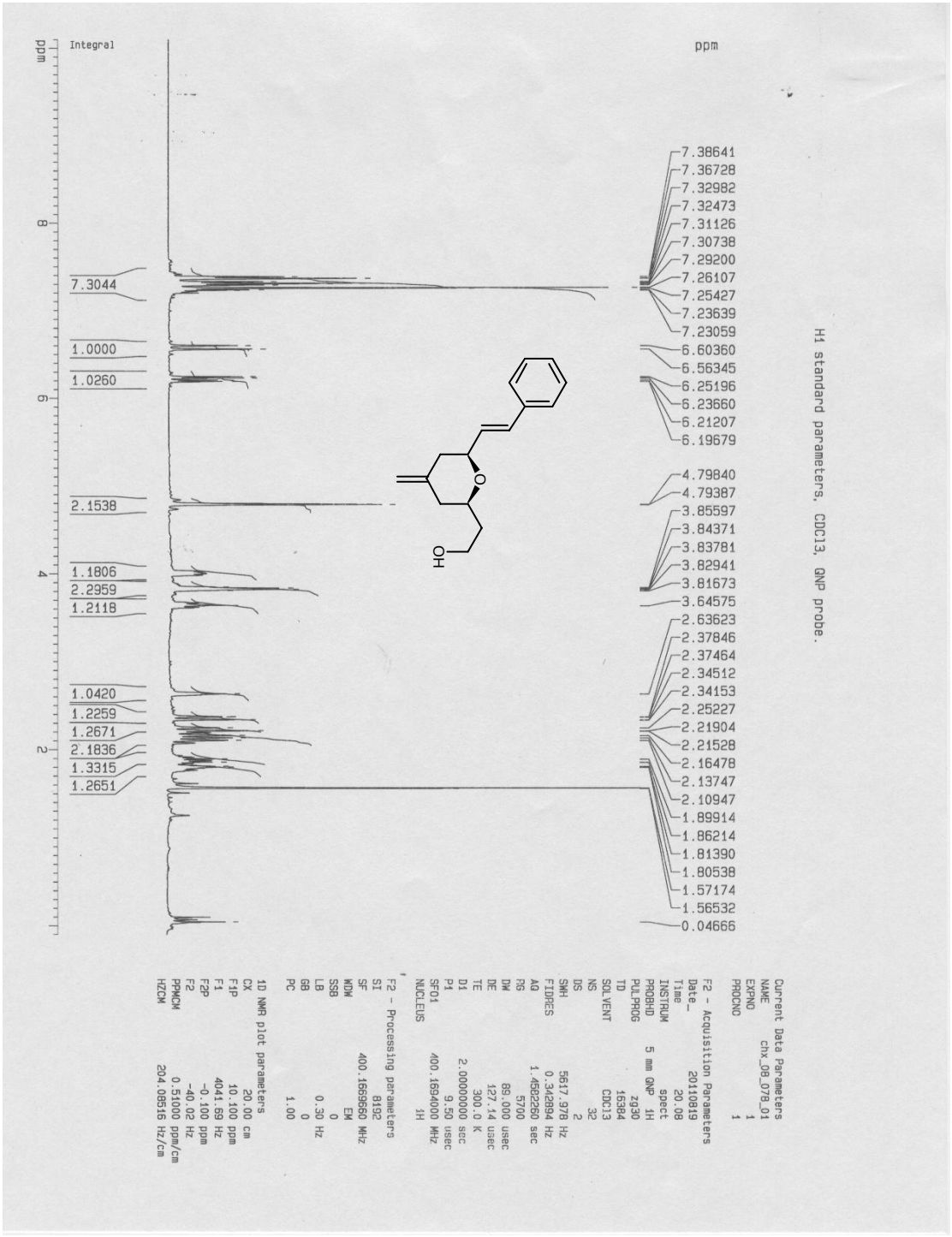
Data Collected on: inova300-1-inovah1freq
Archive directory: /mnt/d2/pinmrf/harwood/vnmrsys/data
Sample directory:
File: chx_05_082_02_c

Pulse Sequence: s2pul
Solvent: CDCl3

Relax. delay 2.000 sec
Pulse 33.8 degrees
Acq. time 0.468 sec
Width 17497.8 Hz
800 repetitions
OBSERVE C13, 75.4249826 MHz
DECOUPLE H1, 299.9613803 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 32768
Total time 10 min




**16**


H1 standard parameters, CDCl3, QNP probe.
ppm
Integral
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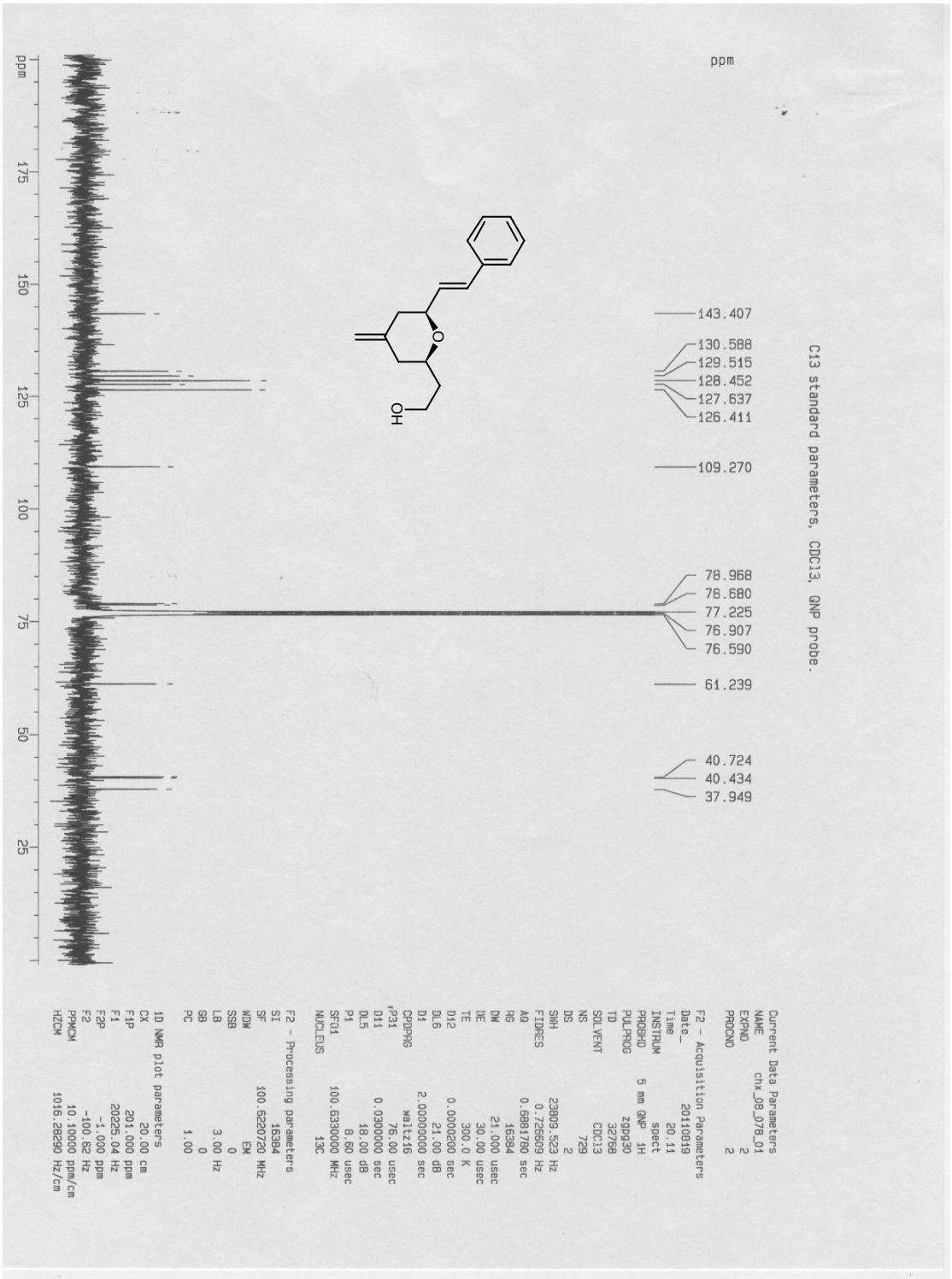
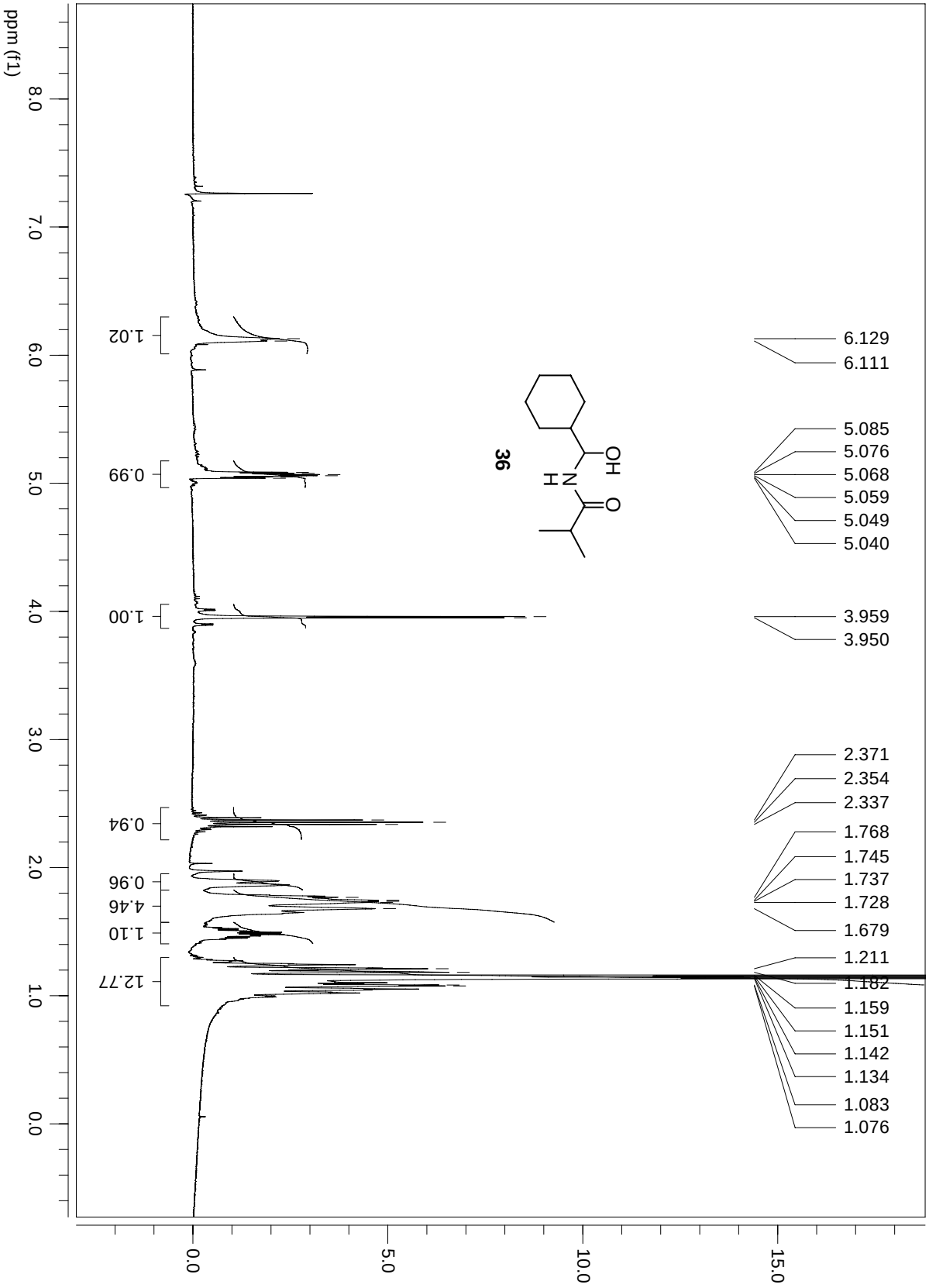
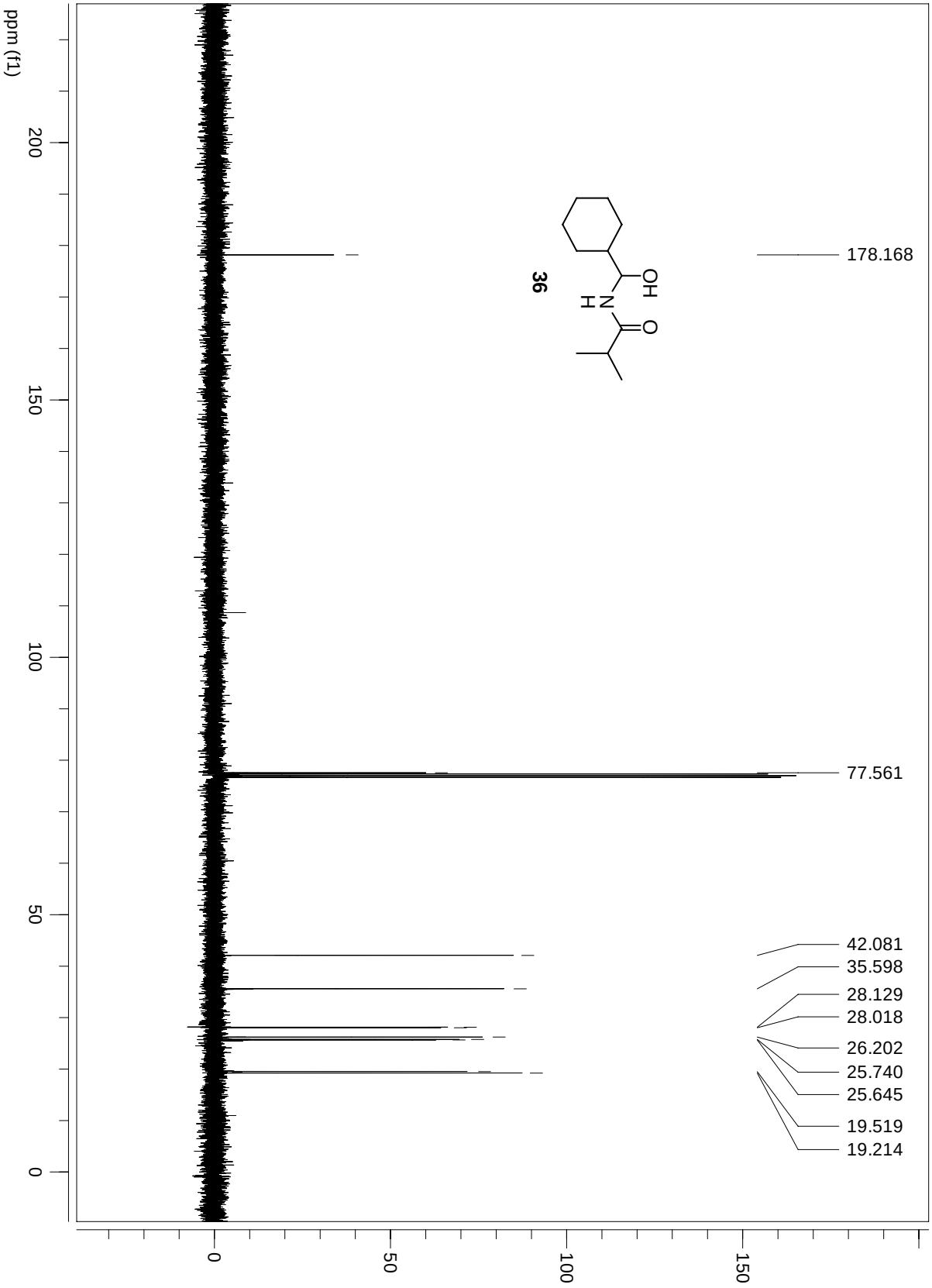
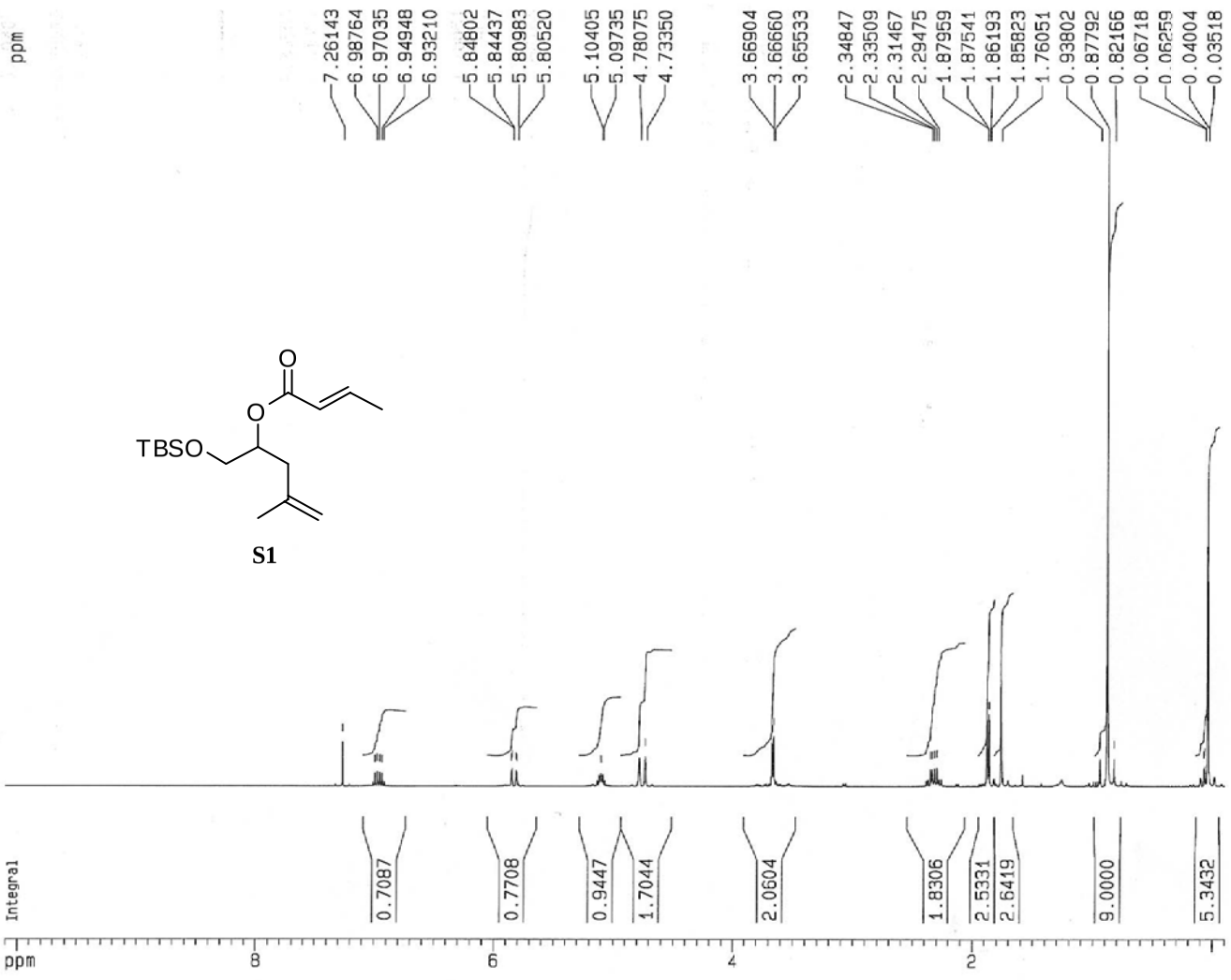
7.36728
7.32982
7.32473
7.31126
7.30738
7.29200
7.26107
7.25427
7.23639
7.23059
6.60360
6.56345
6.25196
6.23660
6.21207
6.19679
4.79840
4.79387
3.85597
3.84371
3.83781
3.82941
3.81673
3.64575
2.63623
2.37846
2.37464
2.34512
2.34153
2.25227
2.21904
2.21528
2.16478
2.13747
2.10947
1.89914
1.86214
1.81390
1.80538
1.57174
1.56532
0.04666
7.3044
1.0000
1.0260
2.1538
1.1806
2.2959
1.2118
1.0420
1.2259
1.2671
2.1836
1.3315
1.2651
OH
Current Data Parameters
NAME chx_08_07B_01
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110819
Time 20.08
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 32
DS 2
SWH 5617.978 Hz
FIDRES 0.342894 Hz
AQ 1.4582260 sec
RG 5700
DW 89.000 usec
DE 127.14 usec
TE 300.0 K
D1 2.00000000 sec
P1 9.50 usec
SFO1 400.1694000 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 8192
SF 400.1669660 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.100 ppm
F1 4041.69 Hz
F2P -0.100 ppm
F2 -40.02 Hz
PPMCM 0.51000 ppm/cm
HZCM 204.08516 Hz/cm


C13 standard parameters, CDCl3, QNP probe.
ppm
143.407
130.588
129.515
128.452
127.637
126.411
109.270
78.968
78.680
77.225
76.907
76.590
61.239
40.724
40.434
37.949
OH
ppm
175
150
125
100
75
50
25
Current Data Parameters
NAME chx_08_078_01
EXPNO 2
PROCNO 2
F2 - Acquisition Parameters
Date_ 20110819
Time 20.11
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 729
DS 2
SWH 23809.523 Hz
FIDRES 0.726609 Hz
AQ 0.6881780 sec
RG 16384
DW 21.000 usec
DE 30.00 usec
TE 300.0 K
D12 0.0000200 sec
DL6 21.00 dB
D1 2.00000000 sec
CPDPRG waltz16
P31 76.00 usec
D11 0.0300000 sec
DL5 18.00 dB
P1 8.60 usec
SFO1 100.6330000 MHz
NUCLEUS 13C
F2 - Processing parameters
SI 16384
SF 100.6220720 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 201.000 ppm
F1 20225.04 Hz
F2P -1.000 ppm
F2 -100.62 Hz
PPMCM 10.10000 ppm/cm
HZCM 1016.28290 Hz/cm


ppm (f1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
1.02
0.99
1.00
0.94
0.96
4.46
1.10
12.77
0.0
5.0
10.0
15.0
36
OH
N
H
O
6.129
6.111
5.085
5.076
5.068
5.059
5.049
5.040
3.959
3.950
2.371
2.354
2.337
1.768
1.745
1.737
1.728
1.679
1.211
1.182
1.159
1.151
1.142
1.134
1.083
1.076


ppm (f1)
200
150
100
50
0
0
50
100
150
36
OH
N
H
O
178.168
77.561
42.081
35.598
28.129
28.018
26.202
25.740
25.645
19.519
19.214

H1 standard parameters, CDCl3, GNP probe.



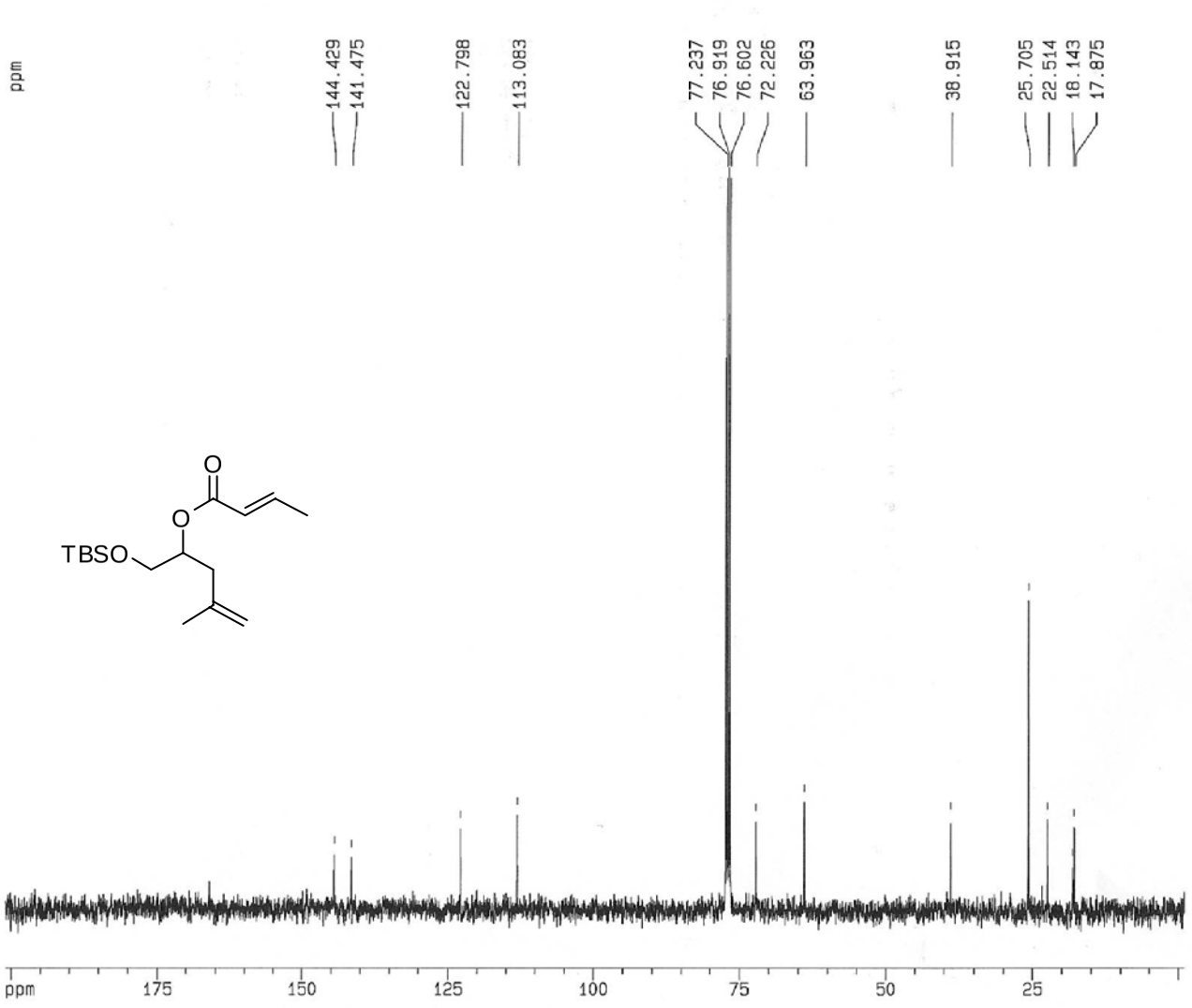




**S1**

```
Current Data Parameters
NAME        chx_10_036_04
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120210
Time               17.34
INSTRUM            spect
PROBHD     5 mm GNP  1H
PULPROG            zg30
TD                16384
SOLVENT           CDCl3
NS                   24
DS                    2
SWH           5617.978 Hz
FIDRES        0.342894 Hz
AQ            1.4582260 sec
RG                 1024
DW               89.000 usec
DE              127.14 usec
TE                300.0 K
D1           2.00000000 sec
P1              9.50 usec
SF01        400.1694000 MHz
NUCLEUS              1H

F2 - Processing parameters
SI                 8192
SF          400.1669660 MHz
WDW                  EM
SSB                   0
LB               0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
F1P              10.100 ppm
F1              4041.69 Hz
F2P              -0.100 ppm
F2               -40.02 Hz
PPMCM          0.51000 ppm/cm
HZCM         204.08516 Hz/cm
```

C13 standard parameters, CDCl3 QNP probe.




Current Data Parameters

| | |
|---|---|
| NAME | chx_10_036_04 |
| EXPNO | 2 |
| PROCNO | 2 |

F2 - Acquisition Parameters

| | |
|---|---|
| Date_ | 20120210 |
| Time | 17.36 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H |
| PULPROG | zgpg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 129 |
| DS | 2 |
| SWH | 23809.523 Hz |
| FIDRES | 0.726609 Hz |
| AQ | 0.6881780 sec |
| RG | 16384 |
| DW | 21.000 usec |
| DE | 30.00 usec |
| TE | 300.0 K |
| D12 | 0.0000200 sec |
| DL6 | 21.00 dB |
| D1 | 2.00000000 sec |
| CPDPRG | waltz16 |
| P31 | 76.00 usec |
| D11 | 0.0300000 sec |
| DL5 | 18.00 dB |
| P1 | 8.60 usec |
| SFO1 | 100.6330000 MHz |
| NUCLEUS | 13C |

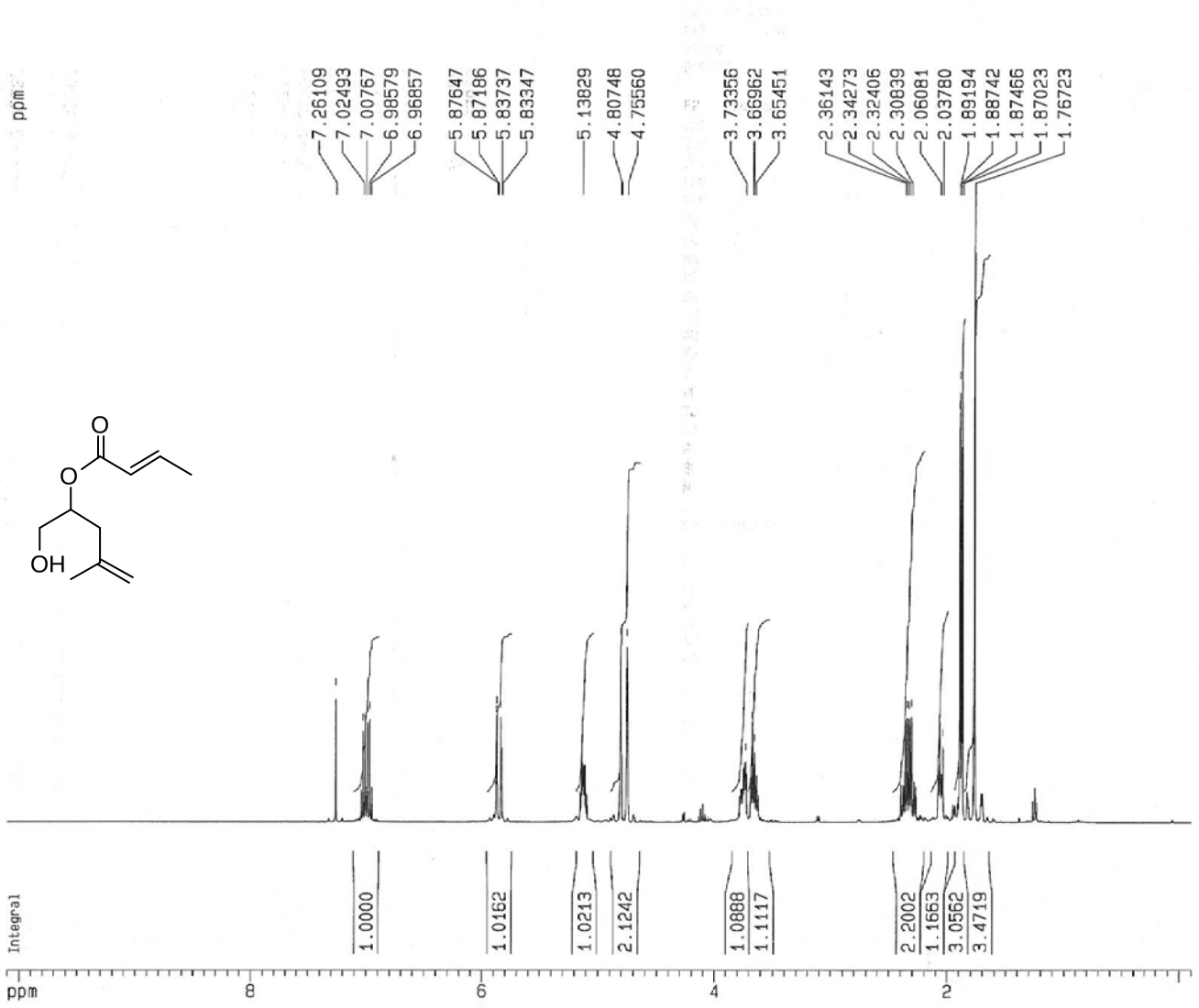
F2 - Processing parameters

| | |
|---|---|
| SI | 16384 |
| SF | 100.6220720 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 3.00 Hz |
| GB | 0 |
| PC | 1.00 |

1D NMR plot parameters

| | |
|---|---|
| CX | 20.00 cm |
| F1P | 201.000 ppm |
| F1 | 20225.04 Hz |
| F2P | -1.000 ppm |
| F2 | -100.62 Hz |
| PPMCM | 10.10000 ppm/cm |
| HZCM | 1016.28290 Hz/cm |

H1 standard parameters, CDCl3, QNP probe.




Current Data Parameters

| | |
|---|---|
| NAME | chx_10_037_02 |
| EXPNO | 1 |
| PROCNO | 1 |

F2 - Acquisition Parameters

| | |
|---|---|
| Date_ | 20120213 |
| Time | 15.05 |
| INSTRUM | spect |
| PROBHD | 5 mm GNP 1H |
| PULPROG | zg30 |
| TD | 16384 |
| SOLVENT | CDCl3 |
| NS | 17 |
| DS | 2 |
| SWH | 5617.978 Hz |
| FIDRES | 0.342894 Hz |
| AQ | 1.4582260 sec |
| RG | 1430 |
| DW | 89.000 usec |
| DE | 127.14 usec |
| TE | 300.0 K |
| D1 | 2.00000000 sec |
| P1 | 9.50 usec |
| SFO1 | 400.1694000 MHz |
| NUCLEUS | 1H |

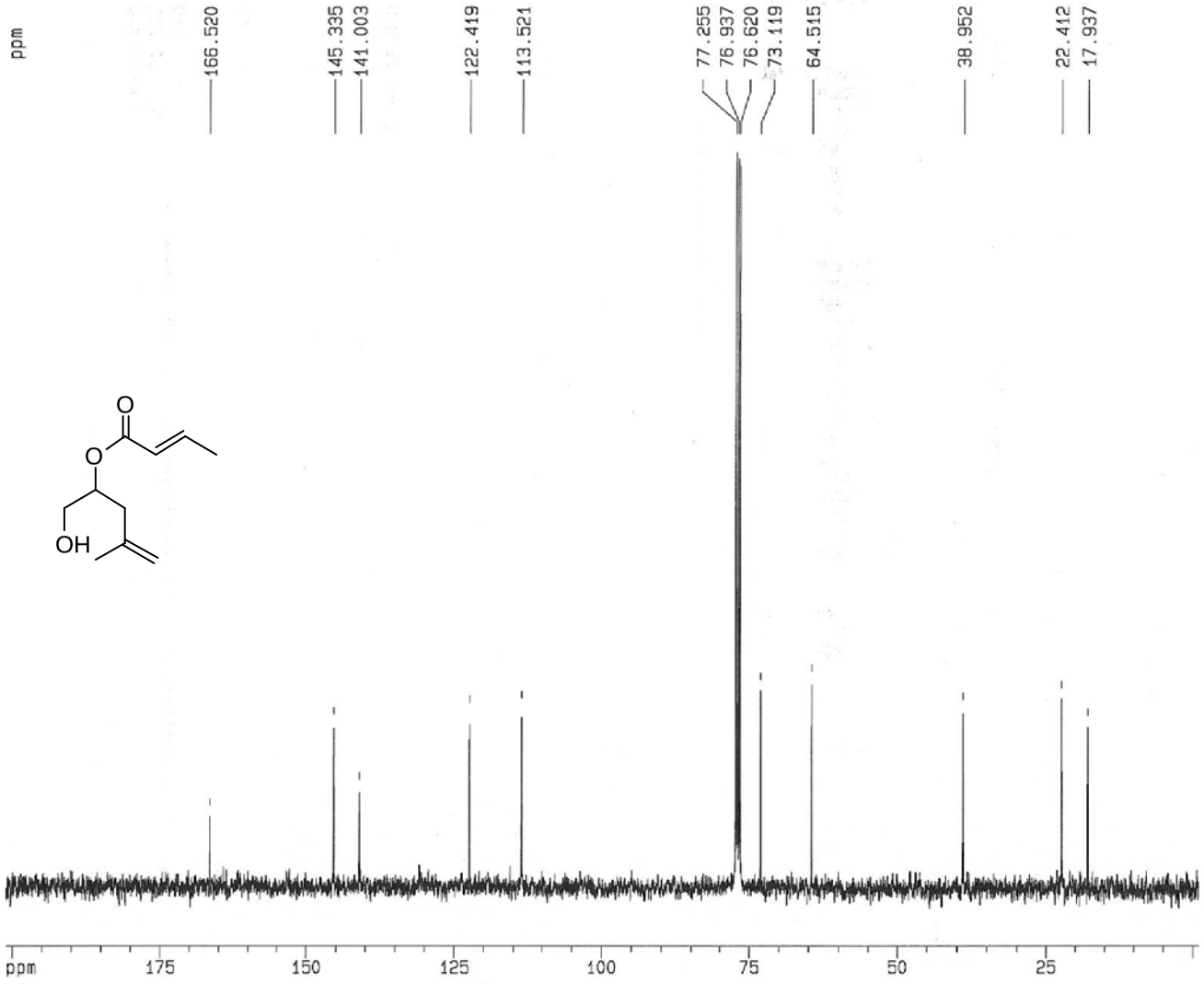
F2 - Processing parameters

| | |
|---|---|
| SI | 8192 |
| SF | 400.1669660 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |

1D NMR plot parameters

| | |
|---|---|
| CX | 20.00 cm |
| F1P | 10.100 ppm |
| F1 | 4041.69 Hz |
| F2P | -0.100 ppm |
| F2 | -40.02 Hz |
| PPMCM | 0.51000 ppm/cm |
| HZCM | 204.08516 Hz/cm |

C13 standard parameters, CDCl3, QNP probe.




Current Data Parameters

| | |
|---|---|
| NAME | chx_10_037_02 |
| EXPNO | 2 |
| PROCNO | 2 |

F2 - Acquisition Parameters

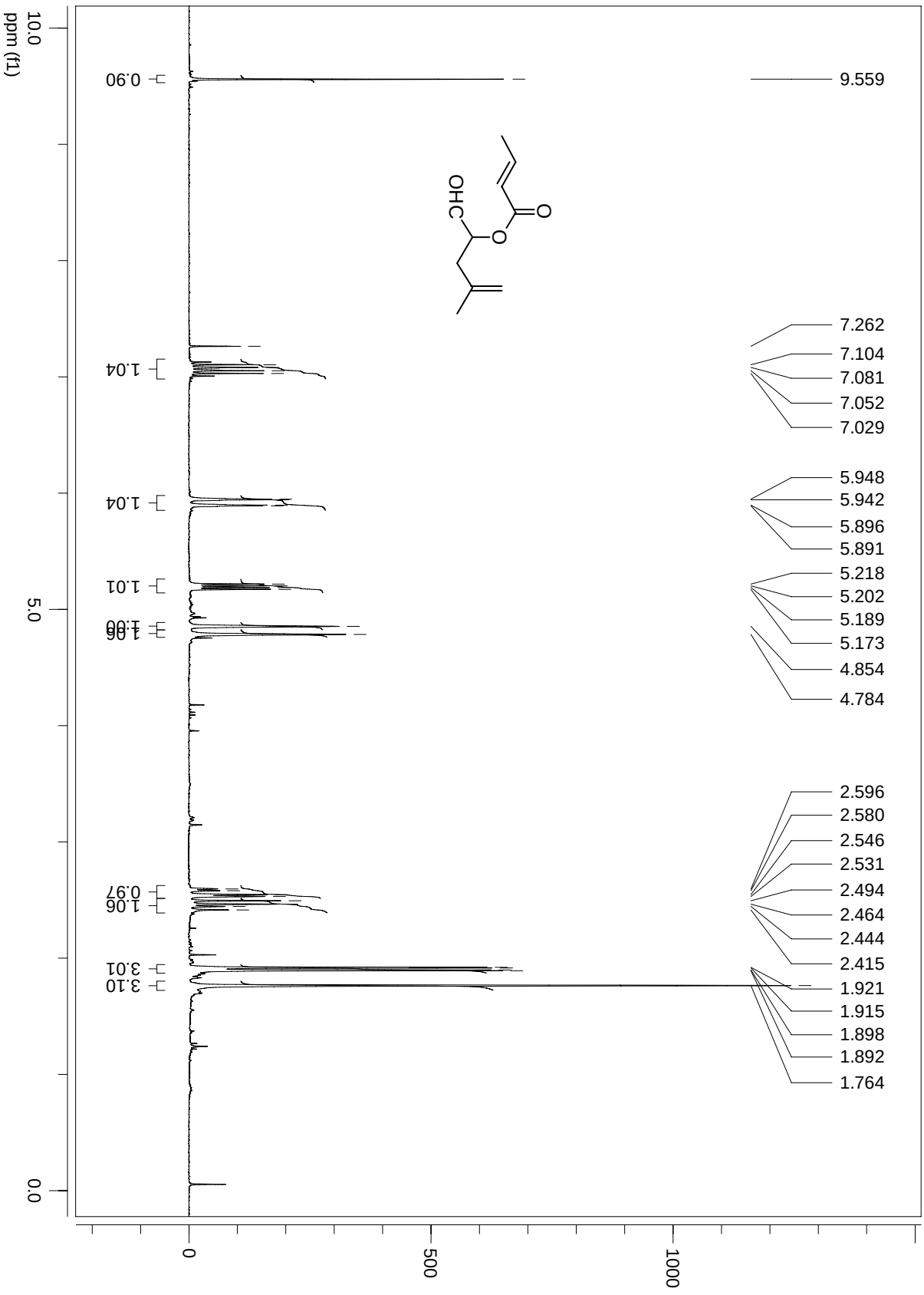
| | |
|---|---|
| Date_ | 20120213 |
| Time | 15.08 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H |
| PULPROG | zgpg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 162 |
| DS | 2 |
| SWH | 23809.523 Hz |
| FIDRES | 0.726609 Hz |
| AQ | 0.6881780 sec |
| RG | 16384 |
| DW | 21.000 usec |
| DE | 30.00 usec |
| TE | 300.0 K |
| D12 | 0.0000200 sec |
| DL6 | 21.00 dB |
| D1 | 2.00000000 sec |
| CPDPRG | waltz16 |
| P31 | 76.00 usec |
| D11 | 0.0300000 sec |
| DL5 | 18.00 dB |
| P1 | 8.60 usec |
| SFO1 | 100.6330000 MHz |
| NUCLEUS | 13C |

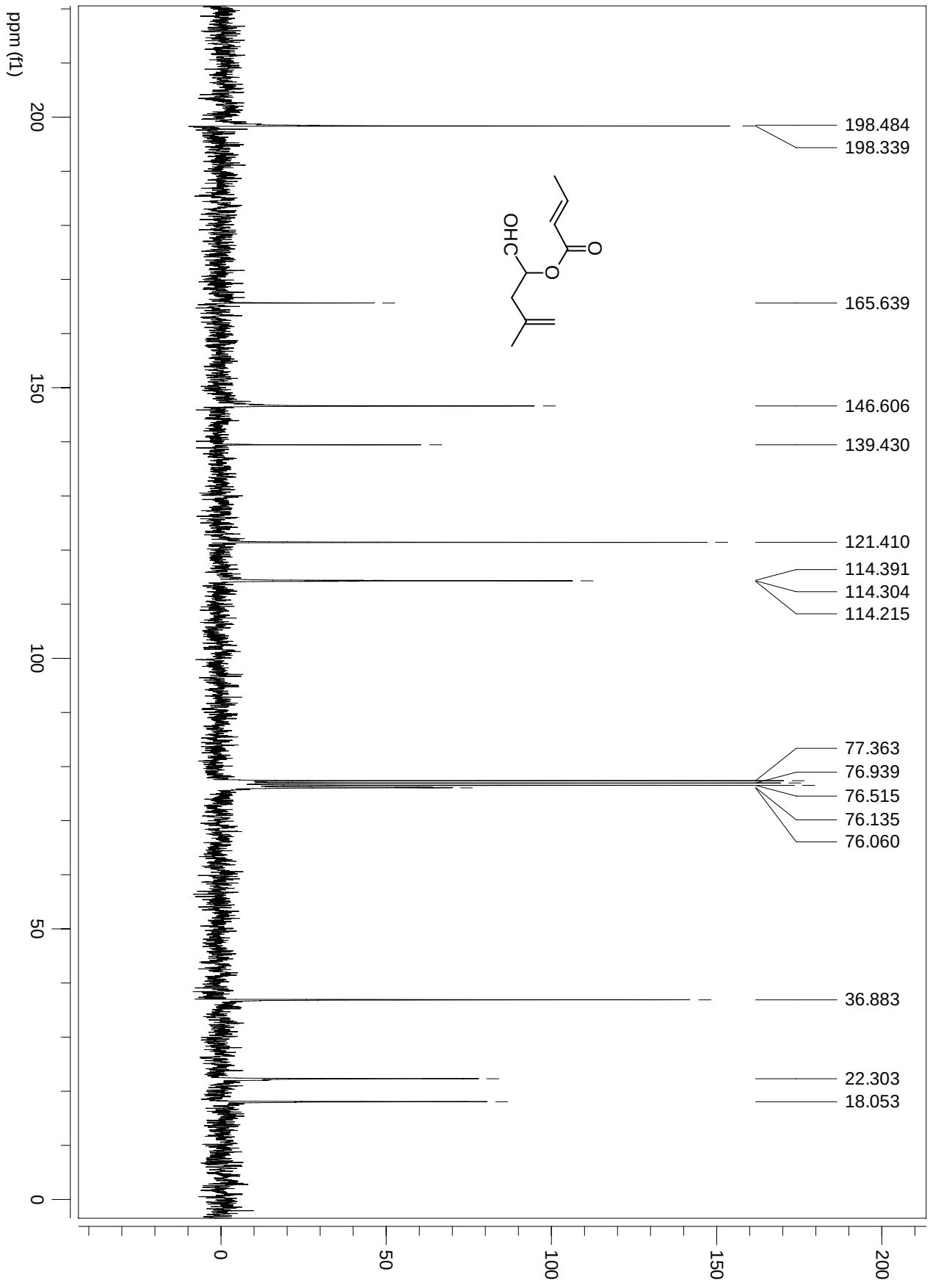
F2 - Processing parameters

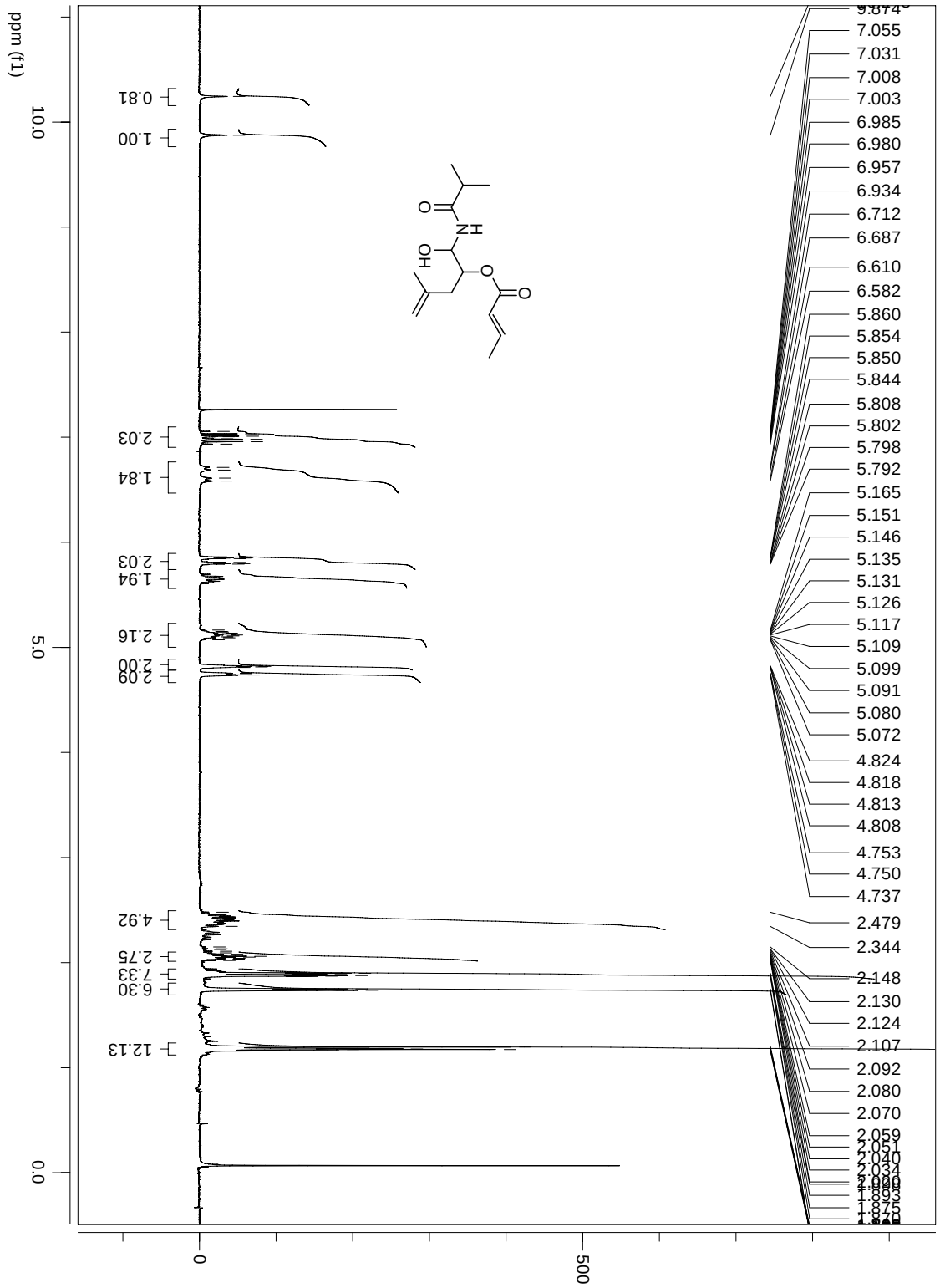
| | |
|---|---|
| SI | 16384 |
| SF | 100.6220720 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 3.00 Hz |
| GB | 0 |
| PC | 1.00 |

1D NMR plot parameters

| | |
|---|---|
| CX | 20.00 cm |
| F1P | 201.000 ppm |
| F1 | 20225.04 Hz |
| F2P | -1.000 ppm |
| F2 | -100.62 Hz |
| PPMCM | 10.10000 ppm/cm |
| HZCM | 1016.28290 Hz/cm |

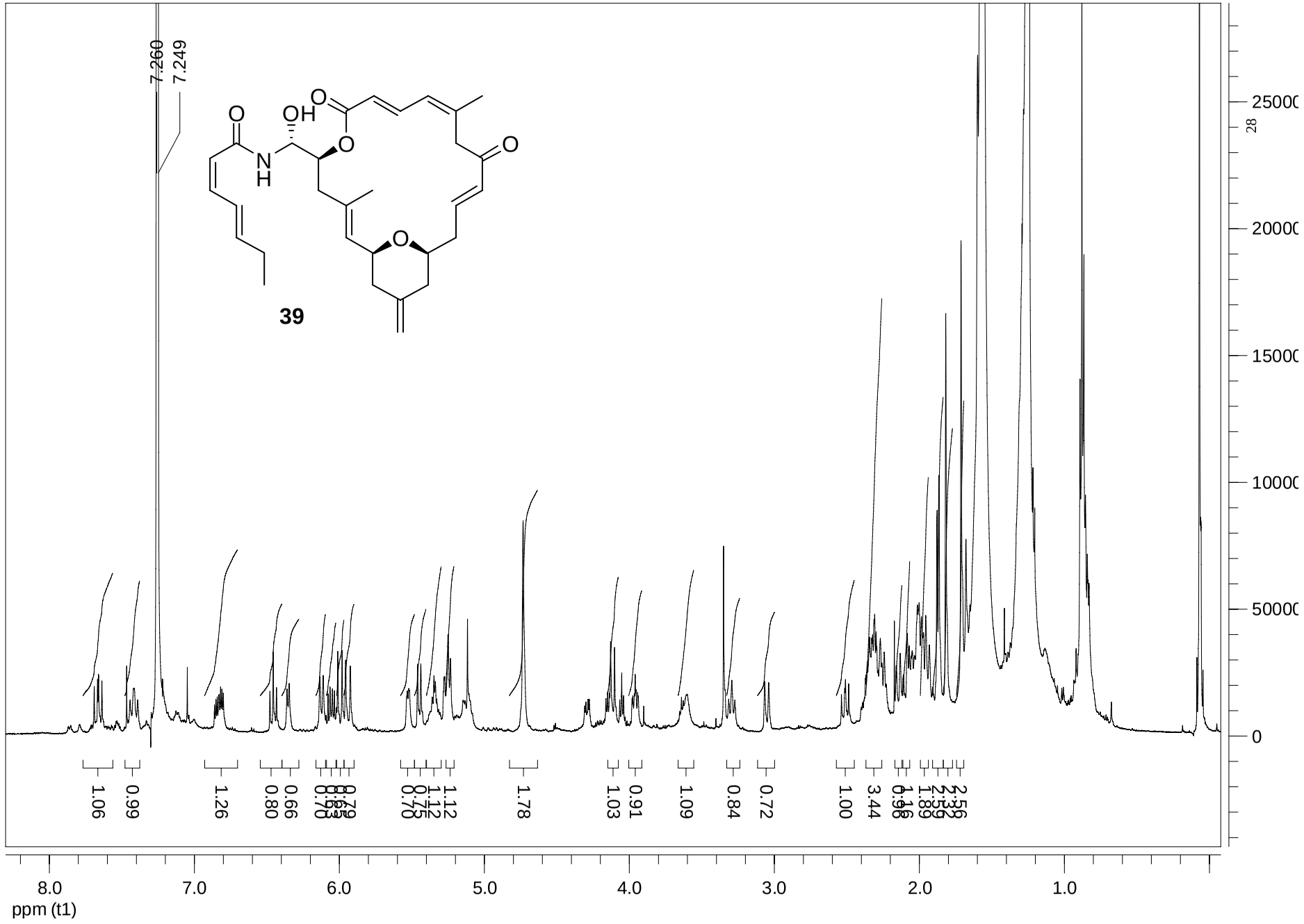

10.0
ppm (f1)
5.0
0.0
0
500
1000
0.90
1.04
1.04
1.01
1.00
1.06
0.97
3.01
3.10
9.559
7.262
7.104
7.081
7.052
7.029
5.948
5.942
5.896
5.891
5.218
5.202
5.189
5.173
4.854
4.784
2.596
2.580
2.546
2.531
2.494
2.464
2.444
2.415
1.921
1.915
1.898
1.892
1.764
OHC
O
O

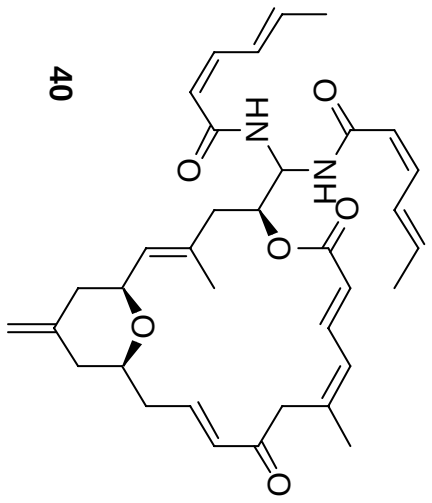

ppm (f1)
200
150
100
50
0
198.484
198.339
165.639
146.606
139.430
121.410
114.391
114.304
114.215
77.363
76.939
76.515
76.135
76.060
36.883
22.303
18.053
OHC
O
O
0
50
100
150
200


ppm (f1)
10.0
5.0
0.0
0
500
0.81
1.00
2.03
1.84
2.03
1.94
2.16
2.00
2.09
4.92
2.75
7.33
6.30
12.13
O
HN
OH
O
O
9.874
7.055
7.031
7.008
7.003
6.985
6.980
6.957
6.934
6.712
6.687
6.610
6.582
5.860
5.854
5.850
5.844
5.808
5.802
5.798
5.792
5.165
5.151
5.146
5.135
5.131
5.126
5.117
5.109
5.099
5.091
5.080
5.072
4.824
4.818
4.813
4.808
4.753
4.750
4.737
2.479
2.344
2.148
2.130
2.124
2.107
2.092
2.080
2.070
2.059
2.051
2.040
2.034
1.893
1.875

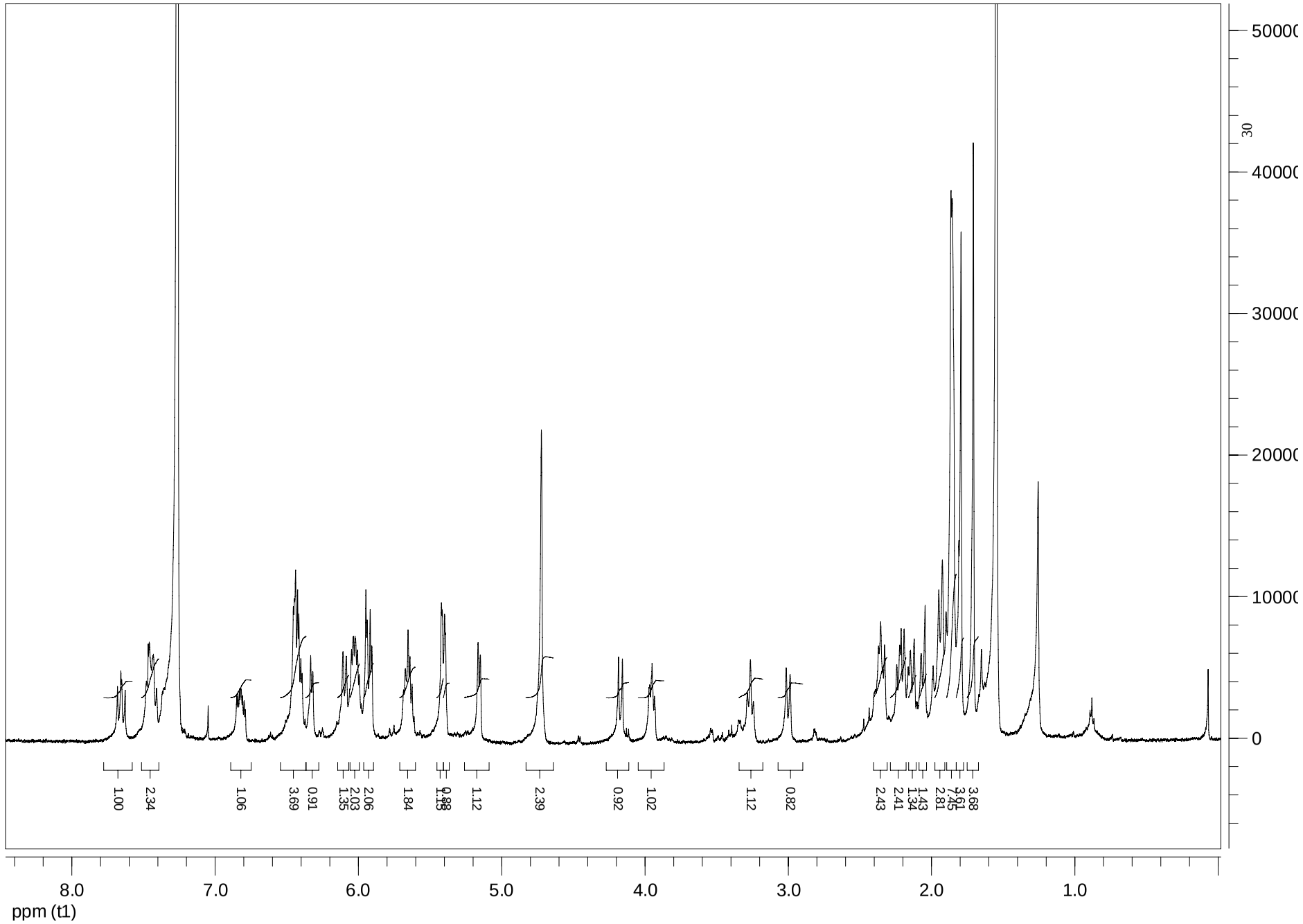
ppm (t1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
1.00
1.02
0.95
0.86
0.80
1.01
0.84
1.86
1.83
1.04
1.04
1.99
1.06
0.97
0.91
0.91
0.80
1.09
1.41
1.90
1.13
1.29
2.28
6.860
6.856
6.847
6.837
6.828
6.519
6.497
6.474
6.365
6.349
6.156
6.133
6.118
6.104
6.088
6.074
6.003
5.990
5.973
5.957
5.488
5.466
5.348
5.345
5.337
5.334
5.326
5.315
5.240
5.224
4.769
4.181
4.154
4.019
4.014
4.003
3.997
3.992
3.980
3.976
3.664
3.344
3.323
3.302
3.091
3.064
2.467
2.439
2.408
2.405
2.399
2.384
2.379
1
O
N
H
OH
O
O
O
O

ppm (t1)
200
150
100
50
0
1
197.931
167.566
166.831
146.323
143.786
143.604
143.534
140.215
140.125
132.004
131.360
129.932
128.110
125.239
120.177
116.836
109.142
77.256
77.000
76.748
76.456
75.818
75.341
71.360
45.094
41.797
40.887
40.571
40.148
23.572
22.680
18.645
16.610


7.260
7.249
O
OH
O
N
H
O
O
O
39
1.06
0.99
1.26
0.80
0.66
1.12
1.78
1.03
0.91
1.09
0.84
0.72
1.00
3.44
25000
20000
15000
10000
50000
0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)



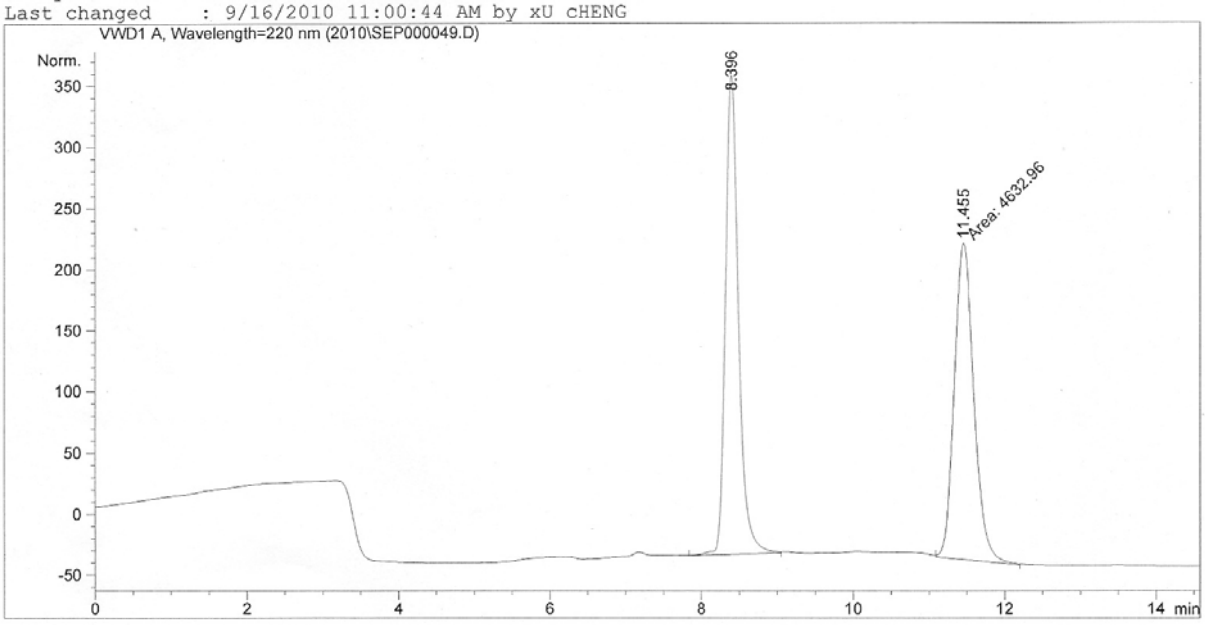
**40**


50000
40000
30000
20000
10000
0
03
1.00
2.34
1.06
3.69
0.91
1.35
2.03
2.06
1.84
1.12
2.39
0.92
1.02
1.12
0.82
2.43
2.41
1.34
1.43
2.81
3.68
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

HPLC traces:

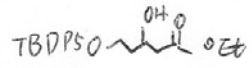
Data File C:\CHEM32\2\DATA\2010\SEP000049.D
Sample Name: BPSO RAC

```
Acq. Operator   : xU cHENG                          Seq. Line :   1
Acq. Instrument : Instrument 2                      Location : Vial 31
Injection Date  : 9/15/2010 10:13:16 PM                 Inj :   1
                                                   Inj Volume : 0.3 µl
Acq. Method     : C:\CHEM32\2\METHODS\CHIRAL.M
Last changed    : 9/15/2010 9:51:00 PM by xU cHENG
Analysis Method : C:\CHEM32\2\METHODS\CHIRAL.M
Last changed    : 9/16/2010 11:00:44 AM by xU cHENG
```



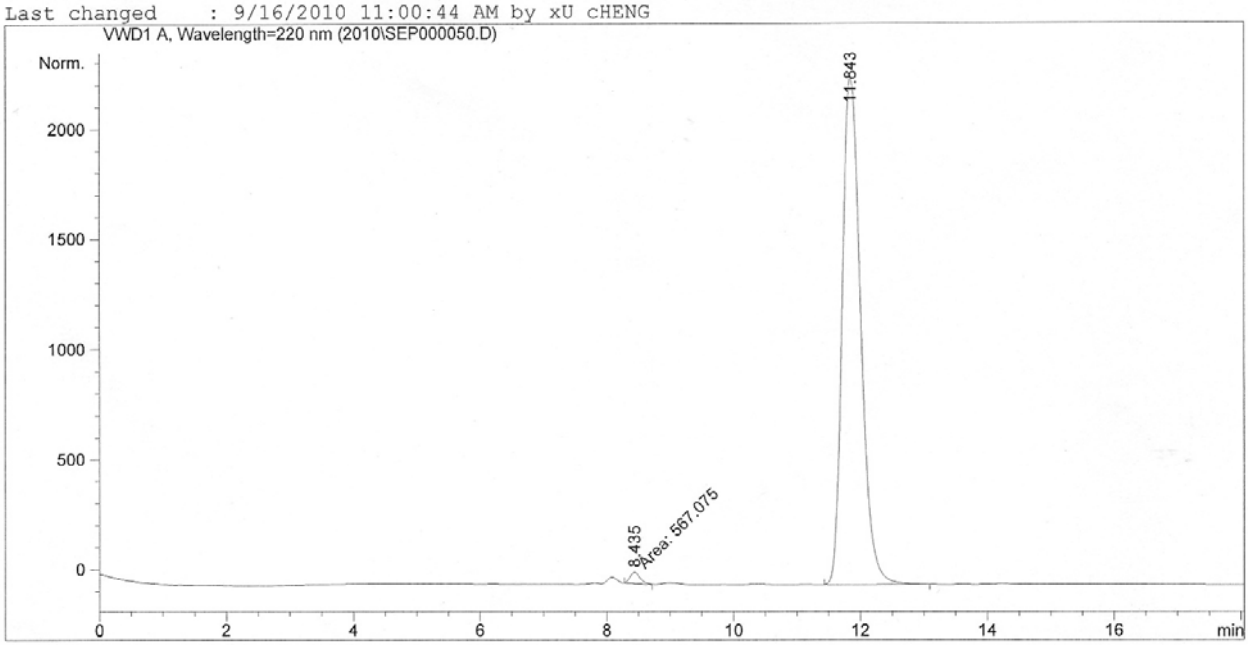

```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   8.396 BV    0.1834 4691.69922  391.63406  50.3149
   2  11.455 MM    0.2987 4632.96338  258.53027  49.6851

Totals :                  9324.66260  650.16434

                        *** End of Report ***
```

racemic



Data File C:\CHEM32\2\DATA\2010\SEP000050.D
Sample Name: BPSO CHIRAL

```
Acq. Operator   : xU cHENG                          Seq. Line :   1
Acq. Instrument : Instrument 2                      Location : Vial 31
Injection Date  : 9/16/2010 11:02:03 AM                 Inj :   1
                                                   Inj Volume : 1 µl
Method          : C:\CHEM32\2\METHODS\CHIRAL.M
Last changed    : 9/16/2010 11:00:44 AM by xU cHENG
```

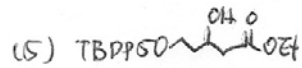



Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=220 nm

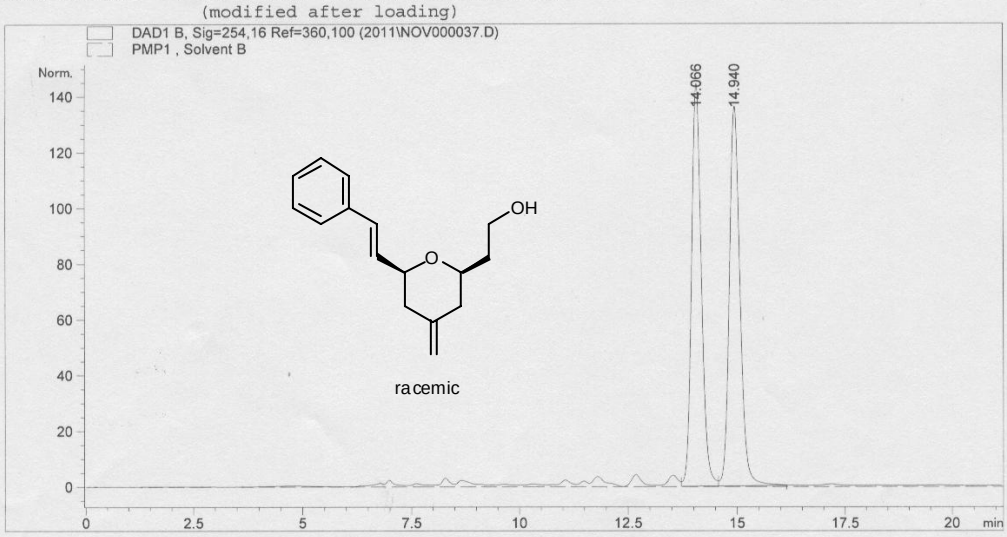
| Peak # | RetTime [min] | Type | Width [min] | Area mAU *s | Height [mAU ] | Area % |
|---|---|---|---|---|---|---|
| 1 | 8.435 | MM | 0.1851 | 567.07489 | 51.06468 | 1.2000 |
| 2 | 11.843 | BBA | 0.3184 | 4.66893e4 | 2300.13892 | 98.8000 |
| Totals : | | | | 4.72564e4 | 2351.20360 | |

(5) TBDPSO 

*** End of Report ***

Data File C:\CHEM32\1\DATA\2011\NOV000037.D
Sample Name: chx_09_085_01

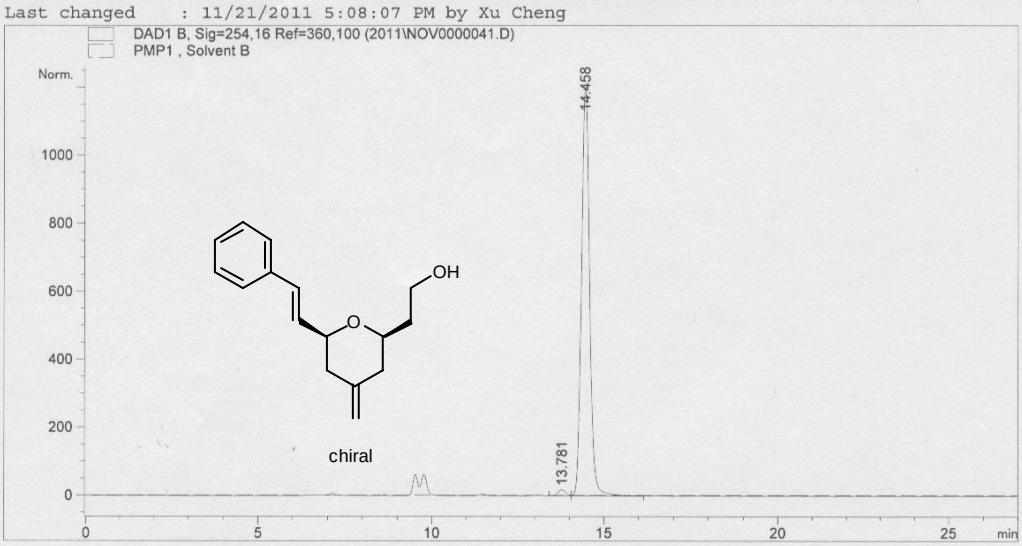
```
    ======================================================================
    Acq. Operator   : Xu Cheng                      Seq. Line :   1
    Acq. Instrument : Instrument 1                   Location : Vial 62
    Injection Date  : 11/19/2011 5:20:36 PM             Inj :   1
                                                   Inj Volume : 1.0 µl
    Acq. Method     : C:\CHEM32\1\METHODS\USER METHODS\XU CHENG\90TO1005254.M
    Last changed    : 11/19/2011 5:08:16 PM by Xu Cheng
    Analysis Method : C:\CHEM32\1\METHODS\USER METHODS\XU CHENG\90TO1005254.M
    Last changed    : 11/22/2011 4:02:22 PM by Xu Cheng
                      (modified after loading)
```




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

    Sorted By             :      Signal
    Multiplier:                   :      1.0000
    Dilution:                     :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: DAD1 B, Sig=254,16 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]    [mAU*s]    [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  14.066 VV    0.2437 2271.43188  144.24863  49.4556
       2  14.940 VB    0.2631 2321.43994  136.02289  50.5444

    Totals :                  4592.87183  280.27151


    ======================================================================
                            *** End of Report ***

Instrument 1 11/22/2011 4:08:34 PM Xu Cheng                    Page   1 of 1
```

Data File C:\CHEM32\1\DATA\2011\NOV0000041.D
Sample Name: chx_09_086_01

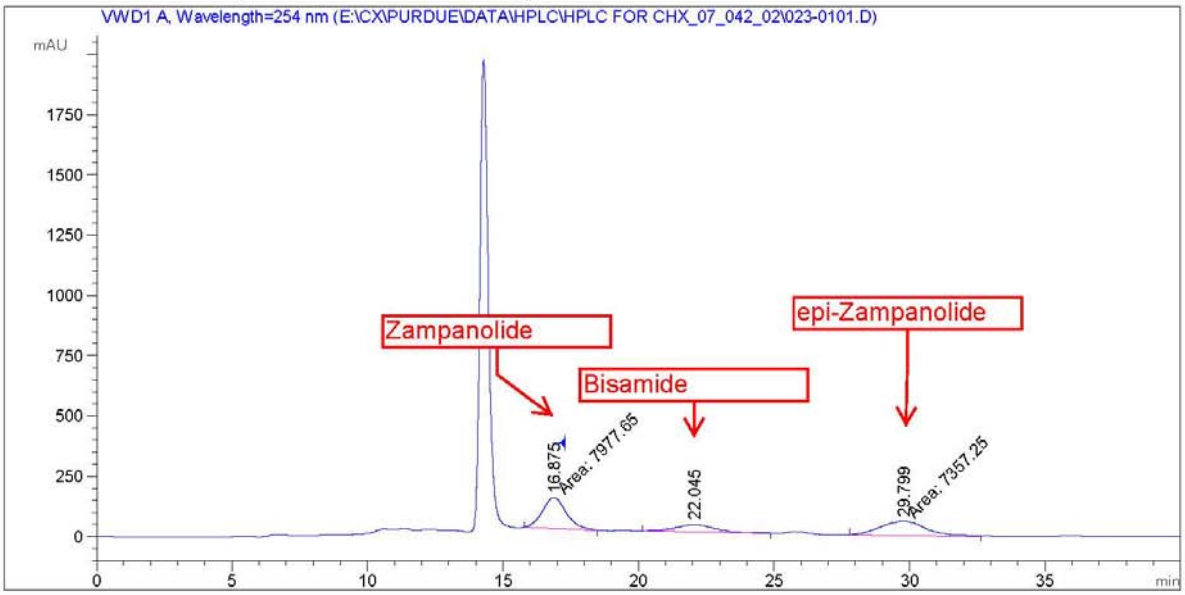
```
=====================================================================
Acq. Operator   : Xu Cheng                    Seq. Line :   1
Acq. Instrument : Instrument 1                 Location : Vial 62
Injection Date  : 11/21/2011 5:13:55 PM           Inj :   1
                                              Inj Volume : 0.3 µl
Method          : C:\CHEM32\1\METHODS\USER METHODS\XU CHENG\90TO1005254.M
Last changed    : 11/21/2011 5:08:07 PM by Xu Cheng
```




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

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1  13.781 VV    0.2305  266.81854   17.83802   1.3411
   2  14.458 VB    0.2505 1.96294e4  1202.26538  98.6589

Totals :                  1.98962e4  1220.10340

=====================================================================
                        *** End of Report ***
```

Addition of carboxamide to (-)-2 using diphenyl phosphoric acid as catalyst.

```
Data File E:\CX\PURDUE\DATA\HPLC\HPLC FOR CHX_07_042_02\023-0101.D
Sample Name: chx_07_042_02

==================================================================
Acq. Operator   : Xu Cheng                    Seq. Line :   1
Acq. Instrument : Instrument 2                 Location : Vial 23
Injection Date  : 3/24/2011 7:05:31 PM             Inj :   1
                                             Inj Volume : 8.000 µl
Acq. Method     : C:\CHEM32\1\METHODS\XU CHENG\50TO505254.M
Last changed    : 3/24/2011 7:00:53 PM by Xu Cheng
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 5/8/2011 12:35:14 AM
                  (modified after loading)
```

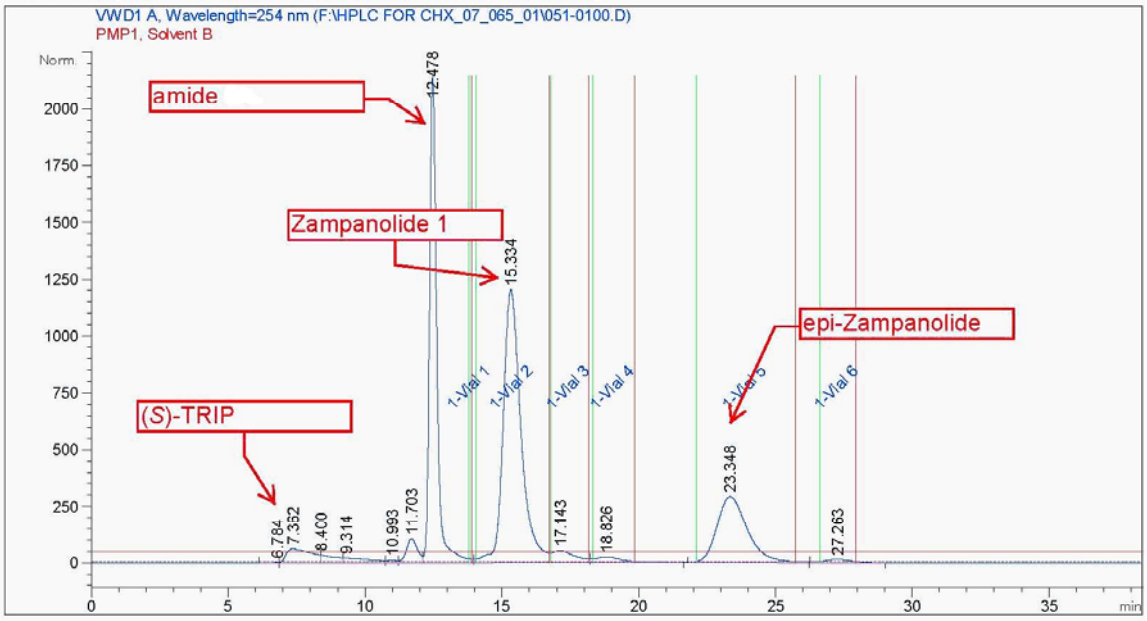



```
==================================================================
                      Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  16.875 MM    1.0359 7977.64941  128.35519  43.5451
   2  22.045 BB    1.4854 2985.51001   30.00826  16.2961
   3  29.799 MM    2.0446 7357.25000   59.97296  40.1588

Instrument 1 5/8/2011 12:54:21 AM                    Page   1 of 2
```

Addition of carboxamide to (-)-2 using (*S*)-TRIP as catalyst.

Data File F:\HPLC FOR CHX_07_065_01\051-0100.D
Sample Name: chx_07_065_01

```
Acq. Operator   : Xu Cheng                          Seq. Line :   1
Acq. Instrument : Instrument 2                      Location : Vial 51
Injection Date  : 4/21/2011 4:49:57 PM                   Inj :   1
                                                   Inj Volume : 2.000 µl
Acq. Method     : C:\CHEM32\1\METHODS\XU CHENG\50TO505254.M
Last changed    : 4/21/2011 4:48:26 PM by Xu Cheng
Analysis Method : C:\CHEM32\1\METHODS\USER METHODS\XU CHENG\50TO5005254.M
Last changed    : 3/10/2011 5:46:27 PM by Xu Cheng
```




Fraction Information

Fraction collection using a timetable

| Frac # | Well # | Location | Volume [µl] | BeginTime [min] | EndTime [min] | Reason | Mass |
|---|---|---|---|---|---|---|---|
| 1 | 1 | 1-Vial 1 | 77.19 | 13.8000 | 13.9085 | VWD1 | |
| 2 | 1 | 1-Vial 2 | 1364.27 | 14.0504 | 16.7331 | VWD1 | |
| 3 | 1 | 1-Vial 3 | 713.13 | 16.7920 | 18.1725 | VWD1 | |
| 4 | 1 | 1-Vial 4 | 786.04 | 18.3360 | 19.8623 | VWD1 | |
| 5 | 1 | 1-Vial 5 | 1824.17 | 22.1252 | 25.7277 | VWD1 | |
| 6 | 1 | 1-Vial 6 | 673.23 | 26.6266 | 27.9273 | VWD1 | |