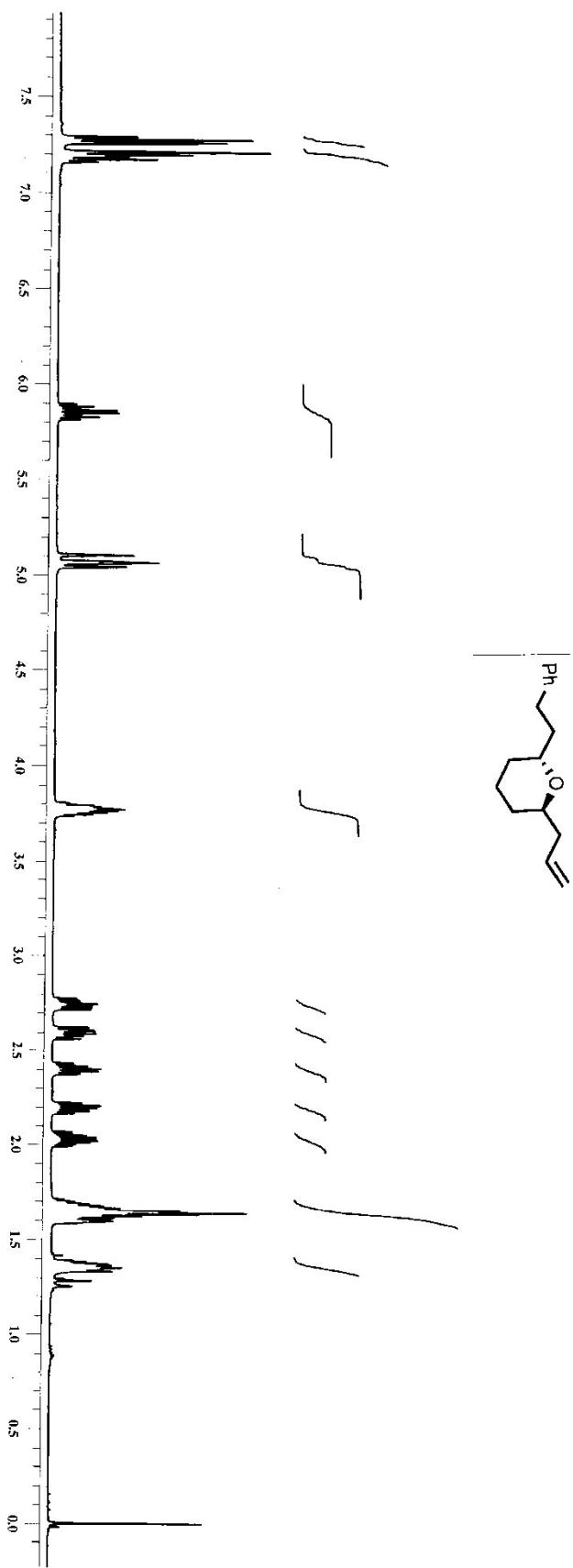


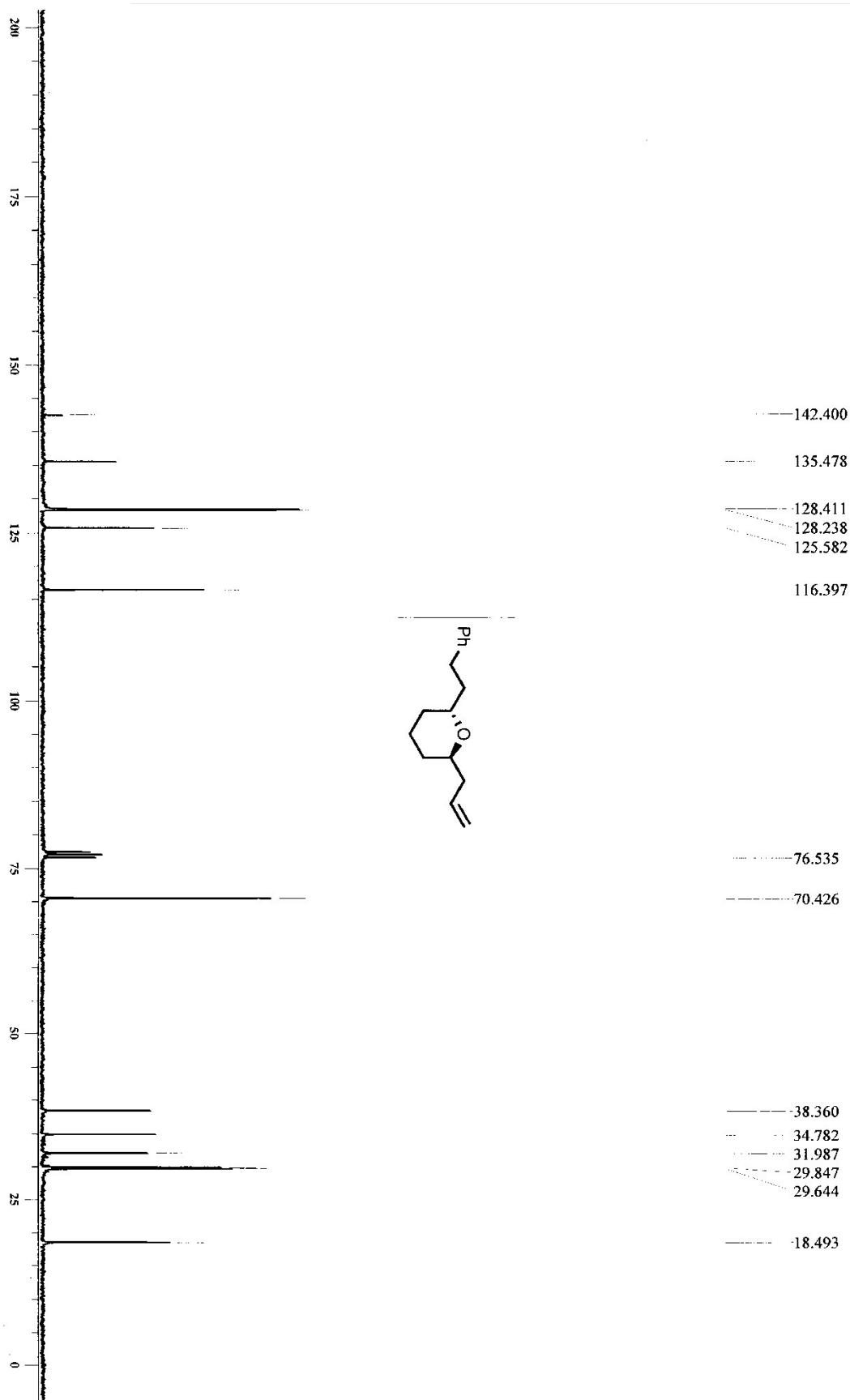
Supporting Information for

Gold-Catalyzed Diastereoselective Synthesis of 2,6-trans-Disubstituted Tetrahydropyran Derivatives: Application for the Synthesis of C1-C13 Fragment of Bistramide A and B

Birakishore Padhi, D. Srinivas Reddy and Debendra K. Mohapatra*



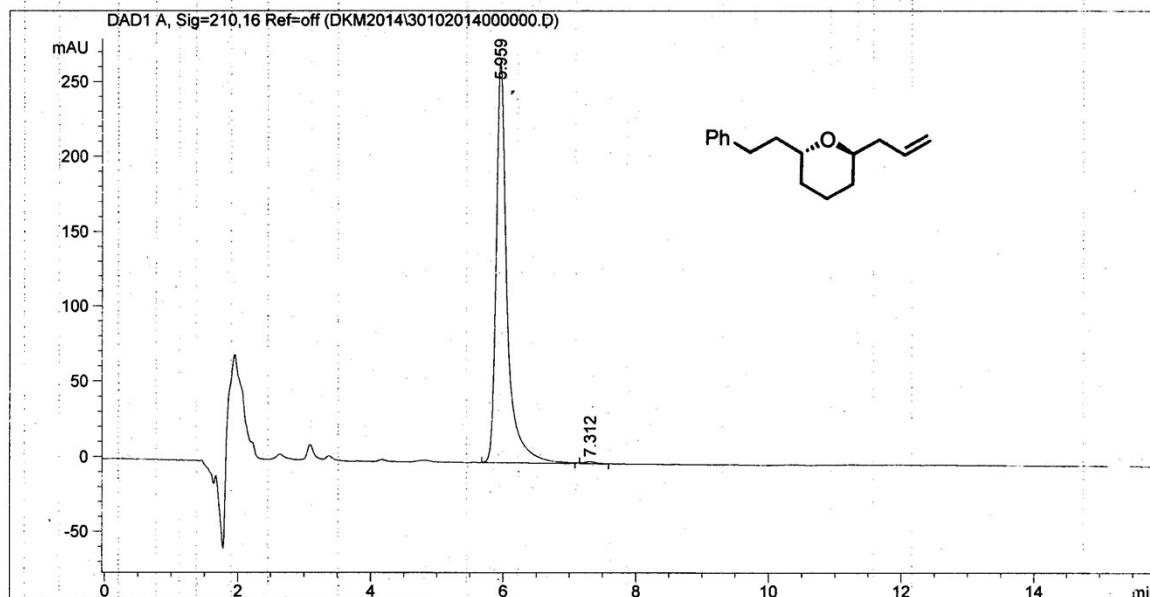
^1H NMR of 2 (300 MHz, CDCl_3)



^{13}C NMR of 2 (75 MHz, CDCl_3)

Data File D:\DATA\DKM2014\30102014000000.D
 Sample Name: DKM-DSR-PH 230

```
=====
Acq. Operator : V.RAJESH          Seq. Line : 1
Acq. Instrument : Instrument 1   Location : Vial 31
Injection Date : 10/30/2014 11:33:43 AM Inj : 1
                                                Inj Volume : 20 µl
Acq. Method : D:\METHODS\LCM.m
Last changed : 10/30/2014 11:31:48 AM by V.RAJESH
Analysis Method : D:\METHODS\LCM.m
Last changed : 11/10/2014 11:06:17 AM by V.RAJESH
Method Info : COLUMN:ATLANTIS C18, 4.6 X 150MM 5U
               MOBILE PHASE:80% ACN IN WATER(0.1% F.A)
               DECTION:210NM
               FLOWRATE:1ML/MIN
```



=====

Area Percent Report

=====

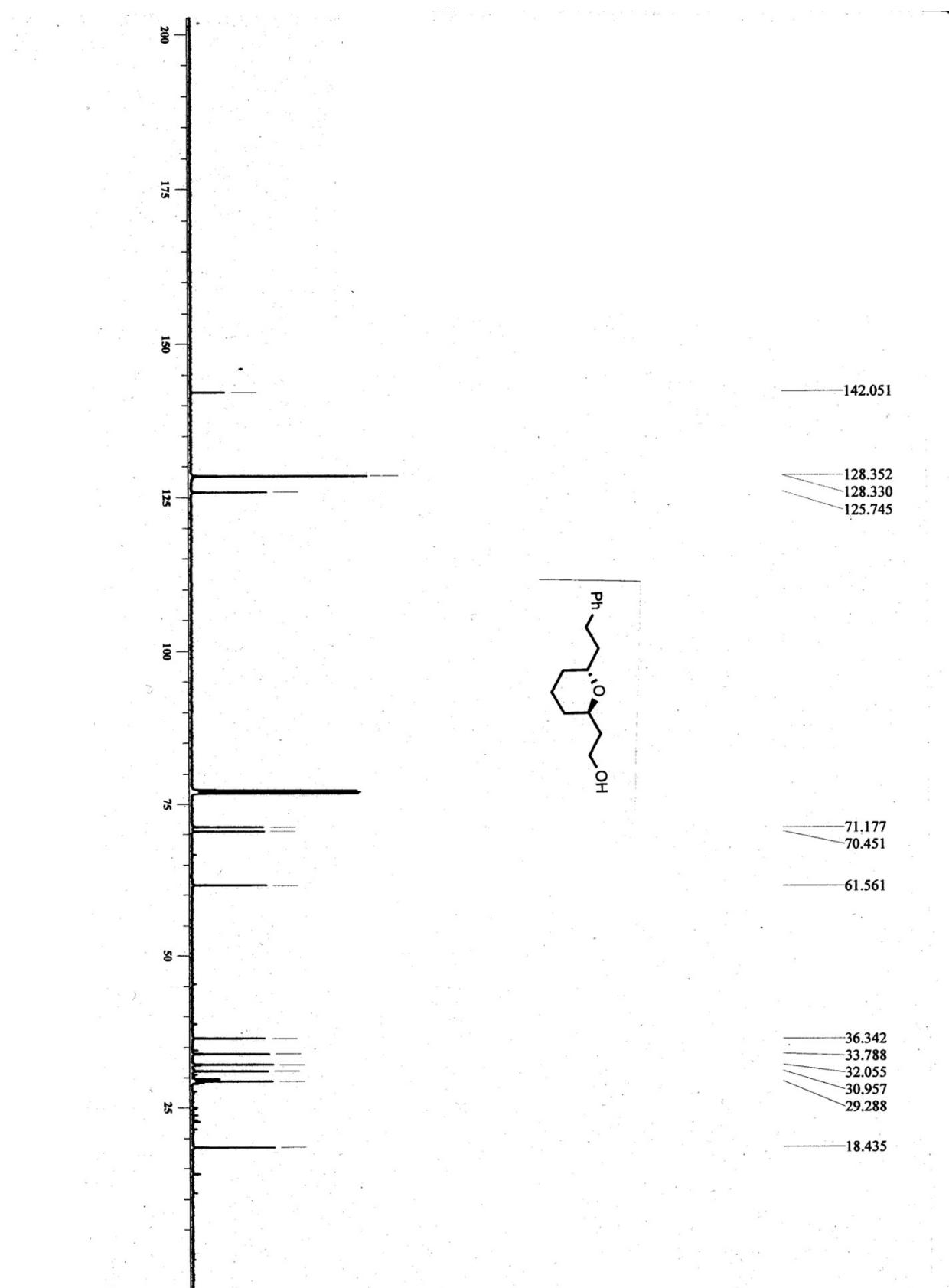
```
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=210,16 Ref=off

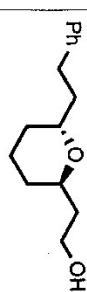
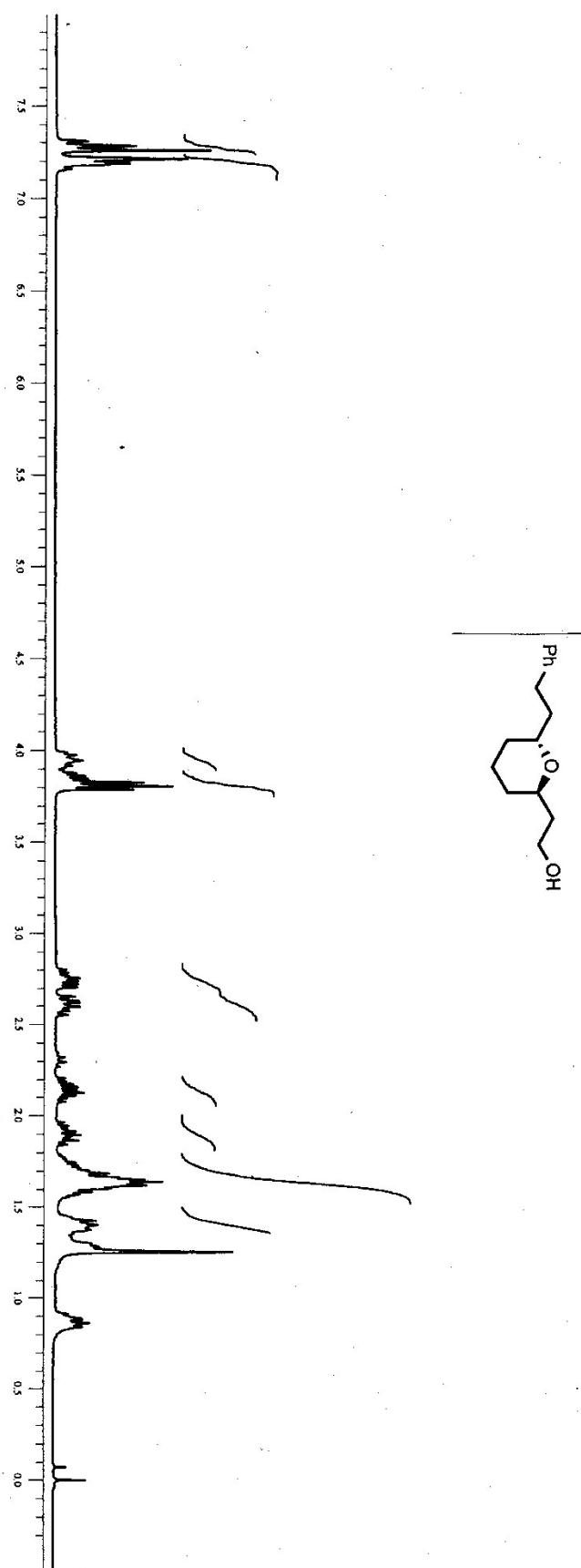
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.959	BB	0.1699	3063.78369	266.95361	99.4472
2	7.312	BB	0.1906	17.03193	1.33744	0.5528
Totals :					3080.81563	268.29105

=====

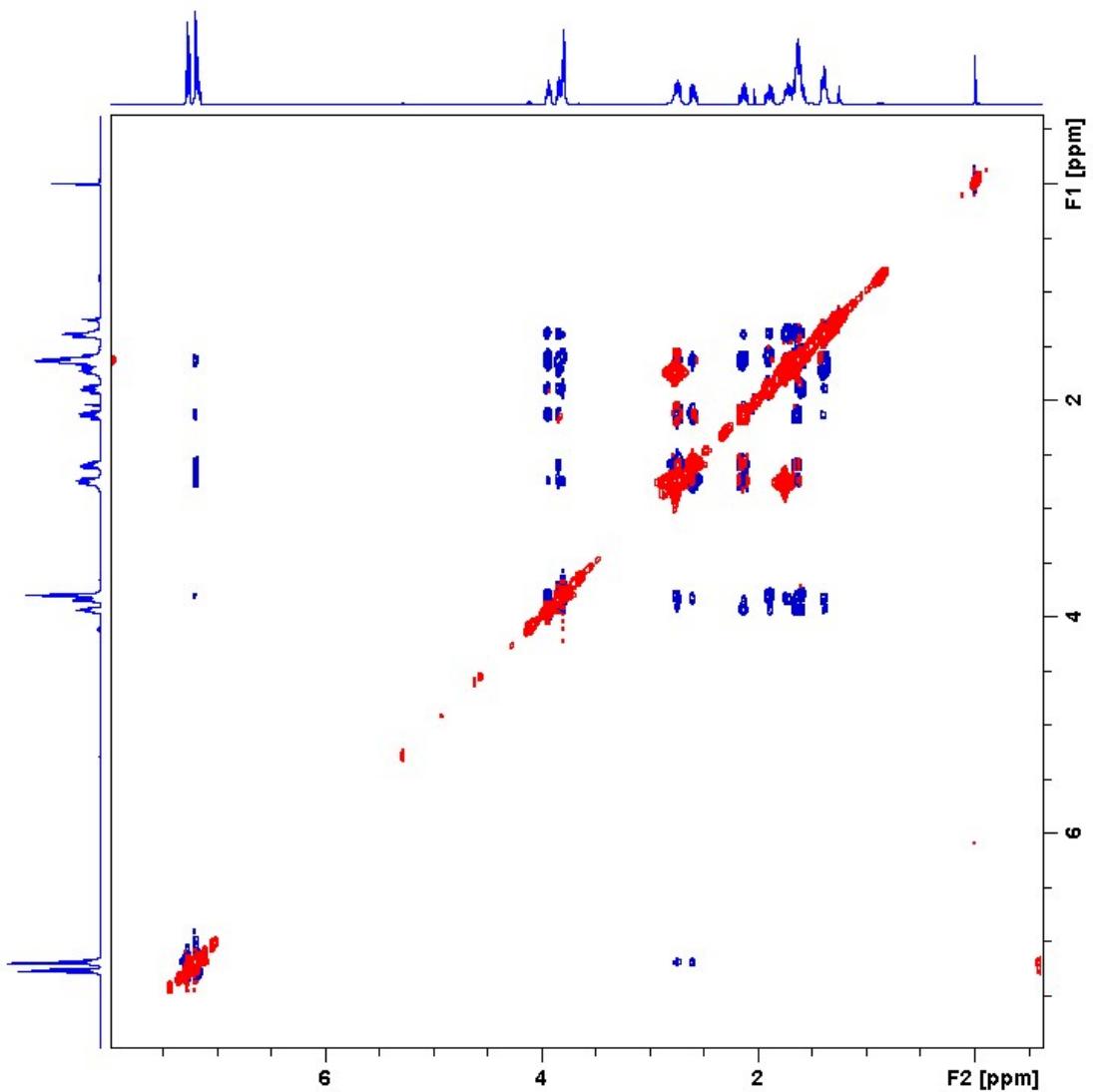
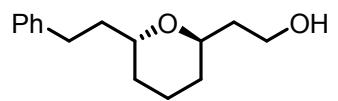
*** End of Report ***



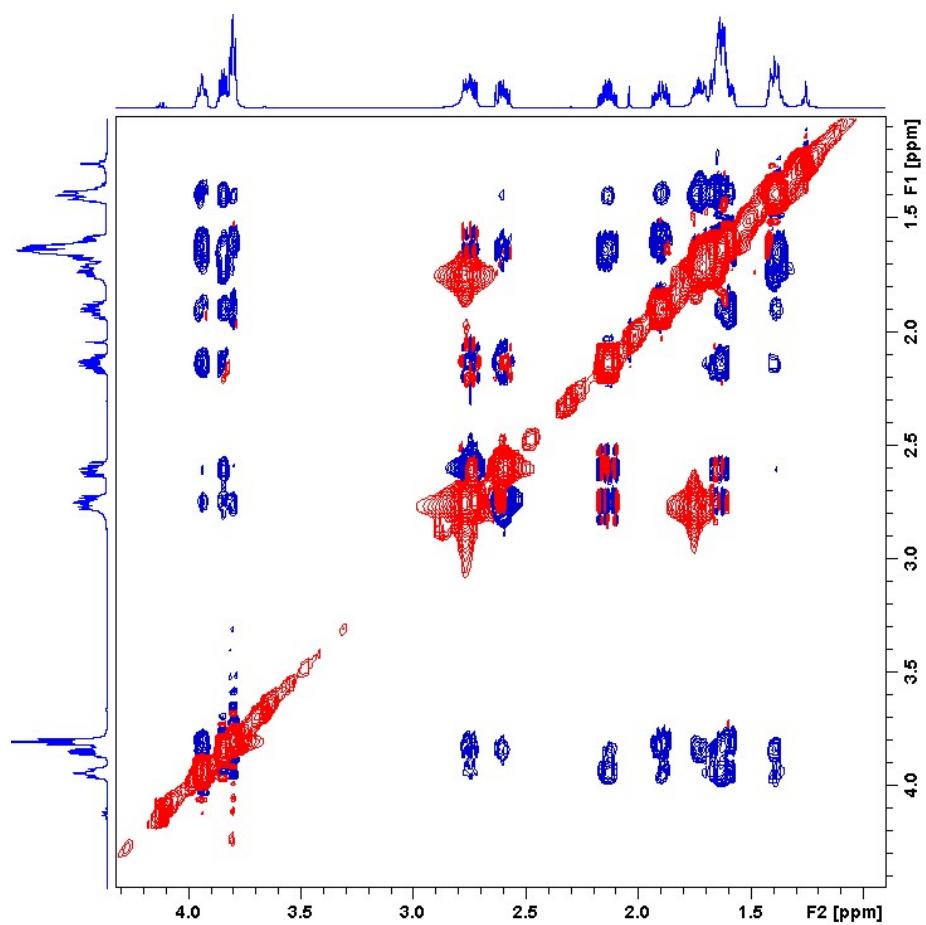
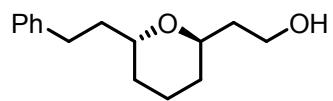
¹H NMR of 4 (300 MHz, CDCl₃)



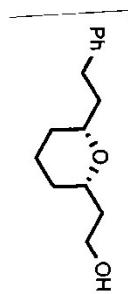
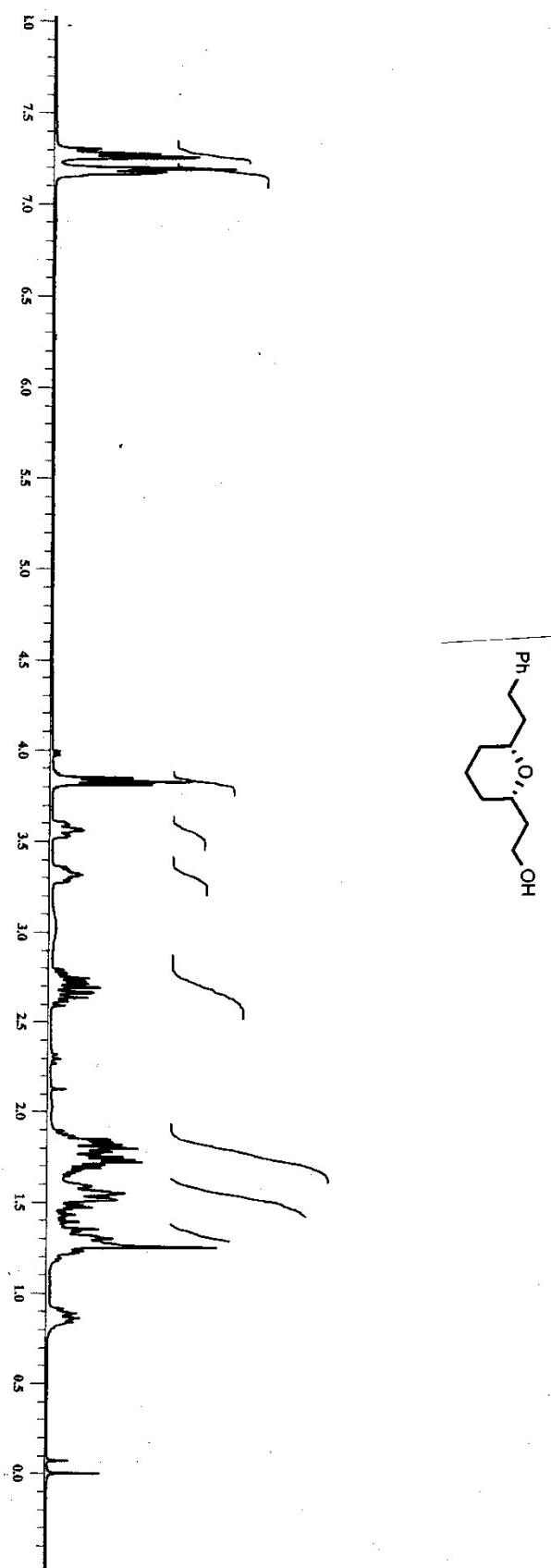
^{13}C NMR of 4 (75 MHz, CDCl_3)



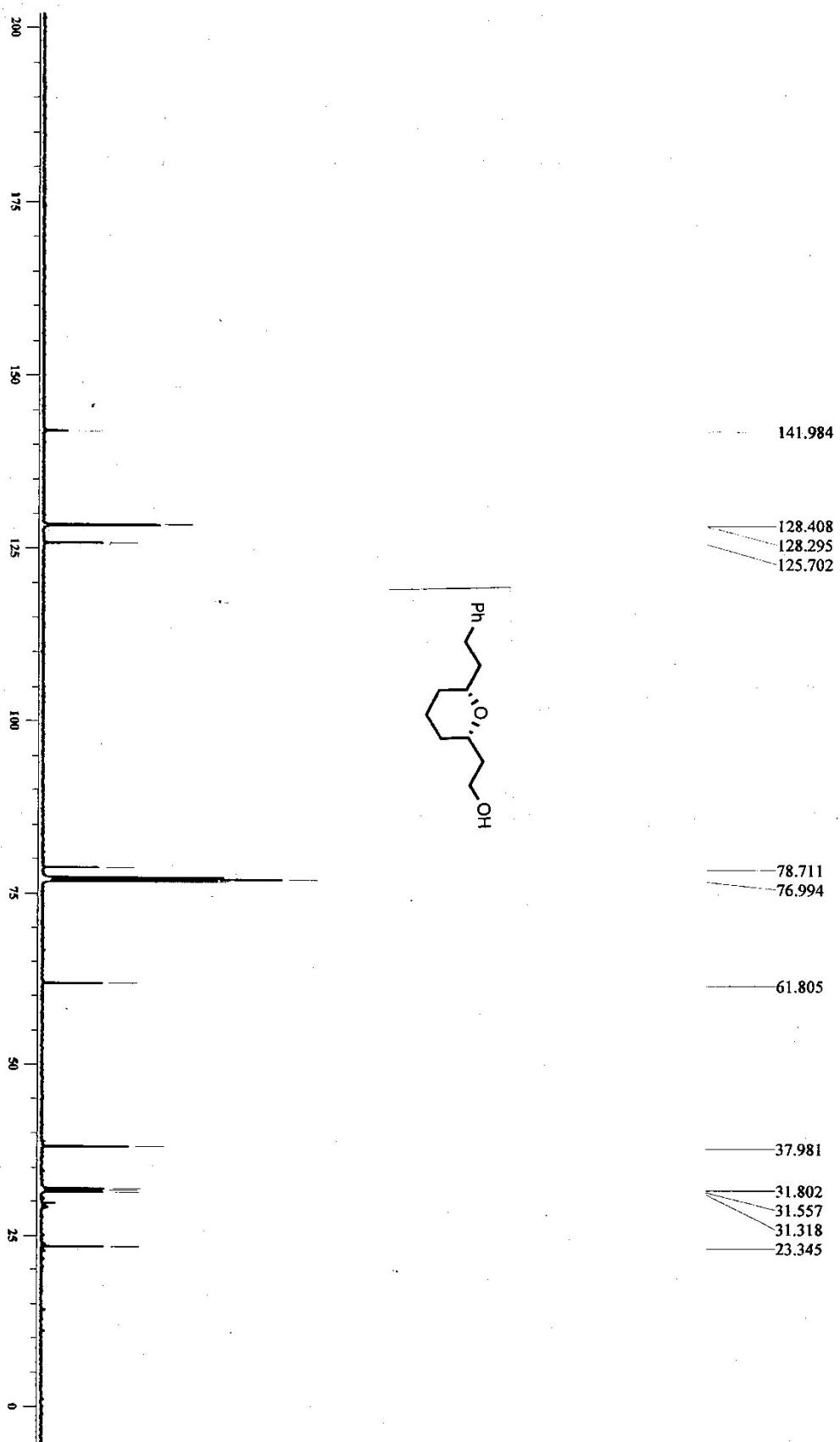
2D ^1H NOE of 4 (500 MHz, CDCl_3)



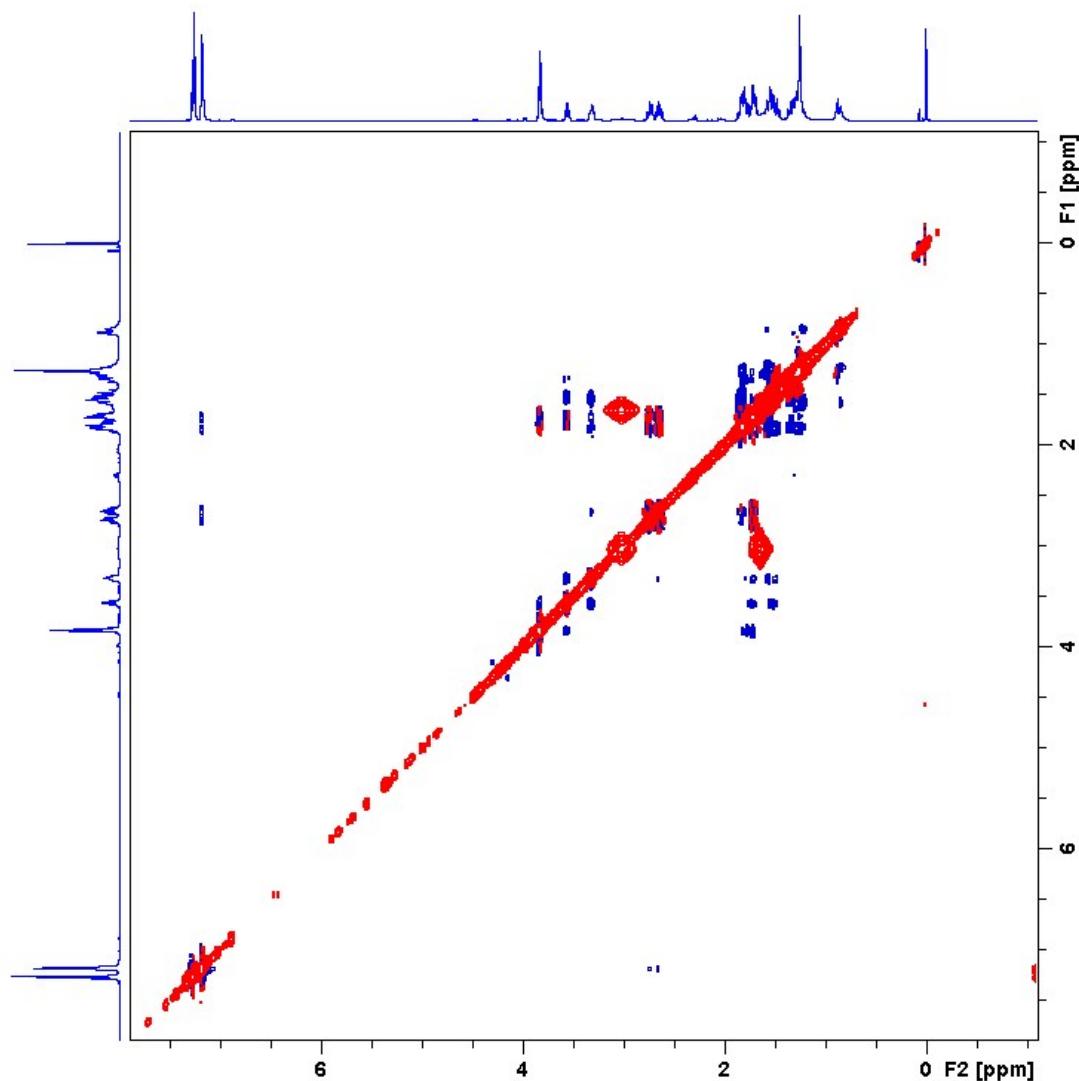
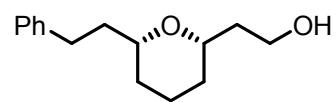
Expanded 2D ^1H NOE of 4 (500 MHz, CDCl_3)



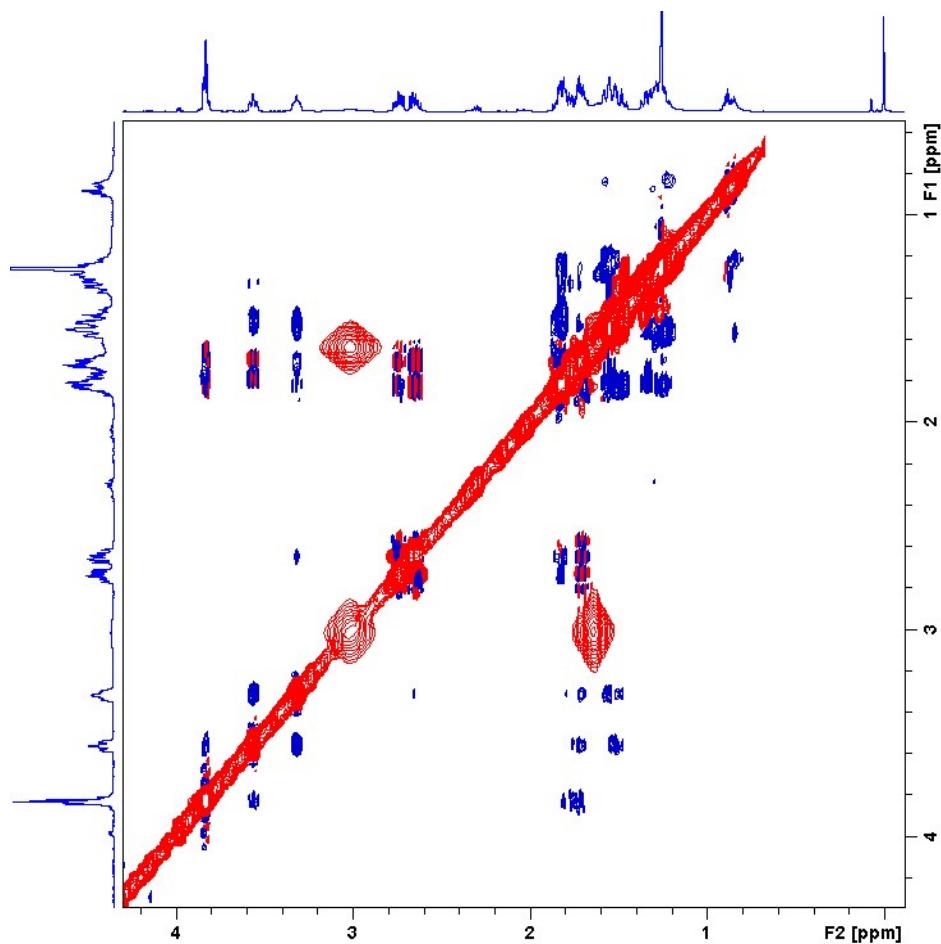
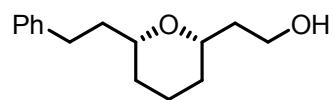
^1H NMR of 5 (300 MHz, CDCl_3)



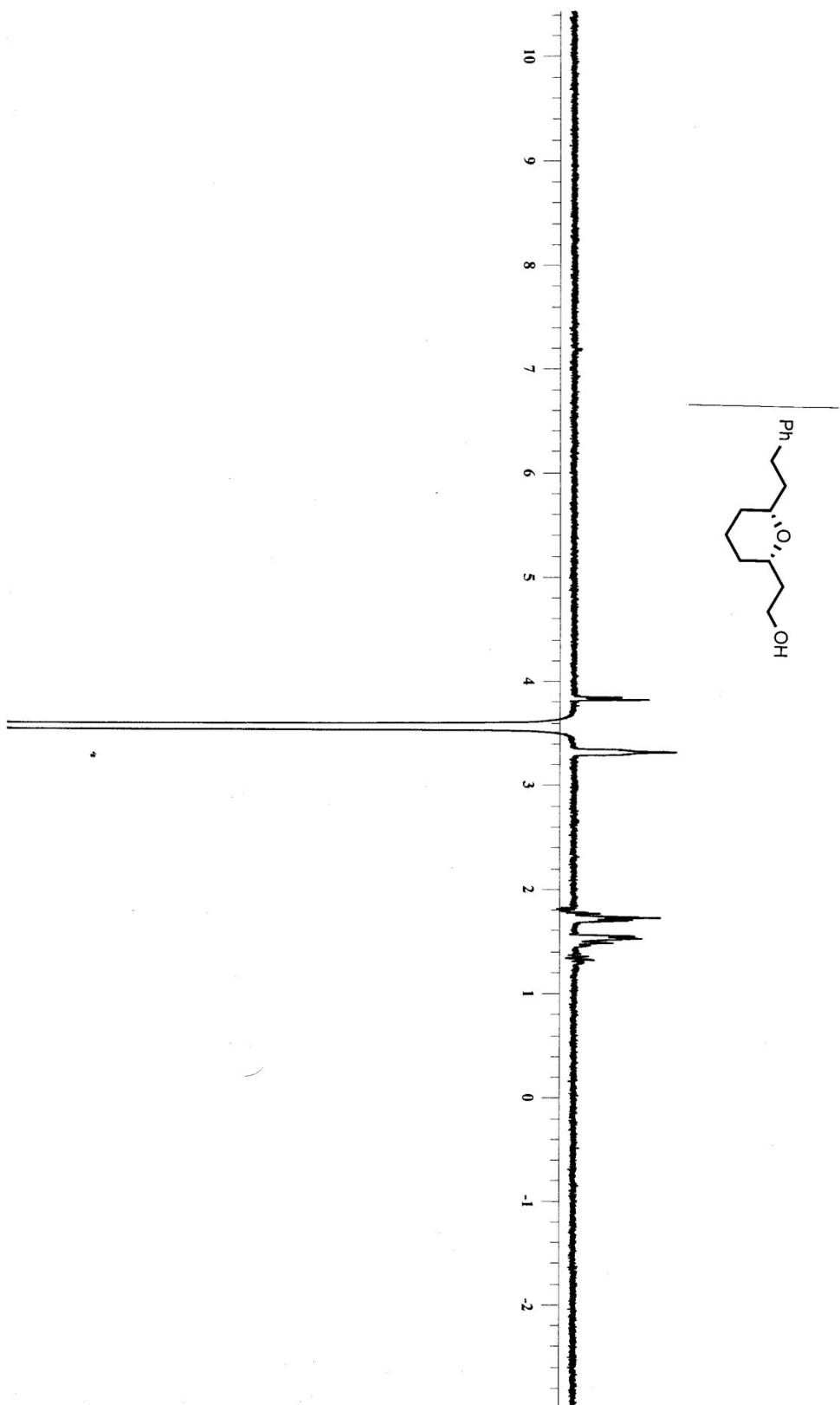
^{13}C NMR of 5 (75 MHz, CDCl_3)



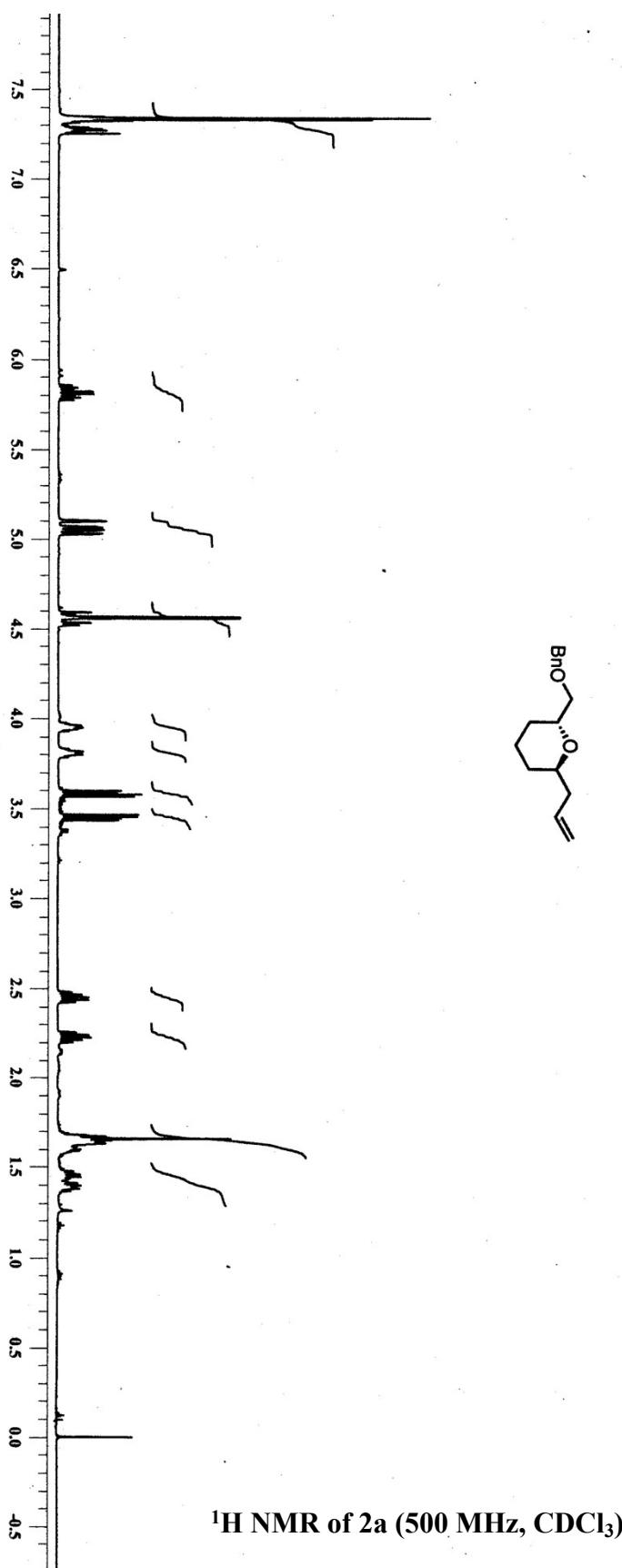
2D ¹H NOE of 5 (500 MHz, CDCl₃)

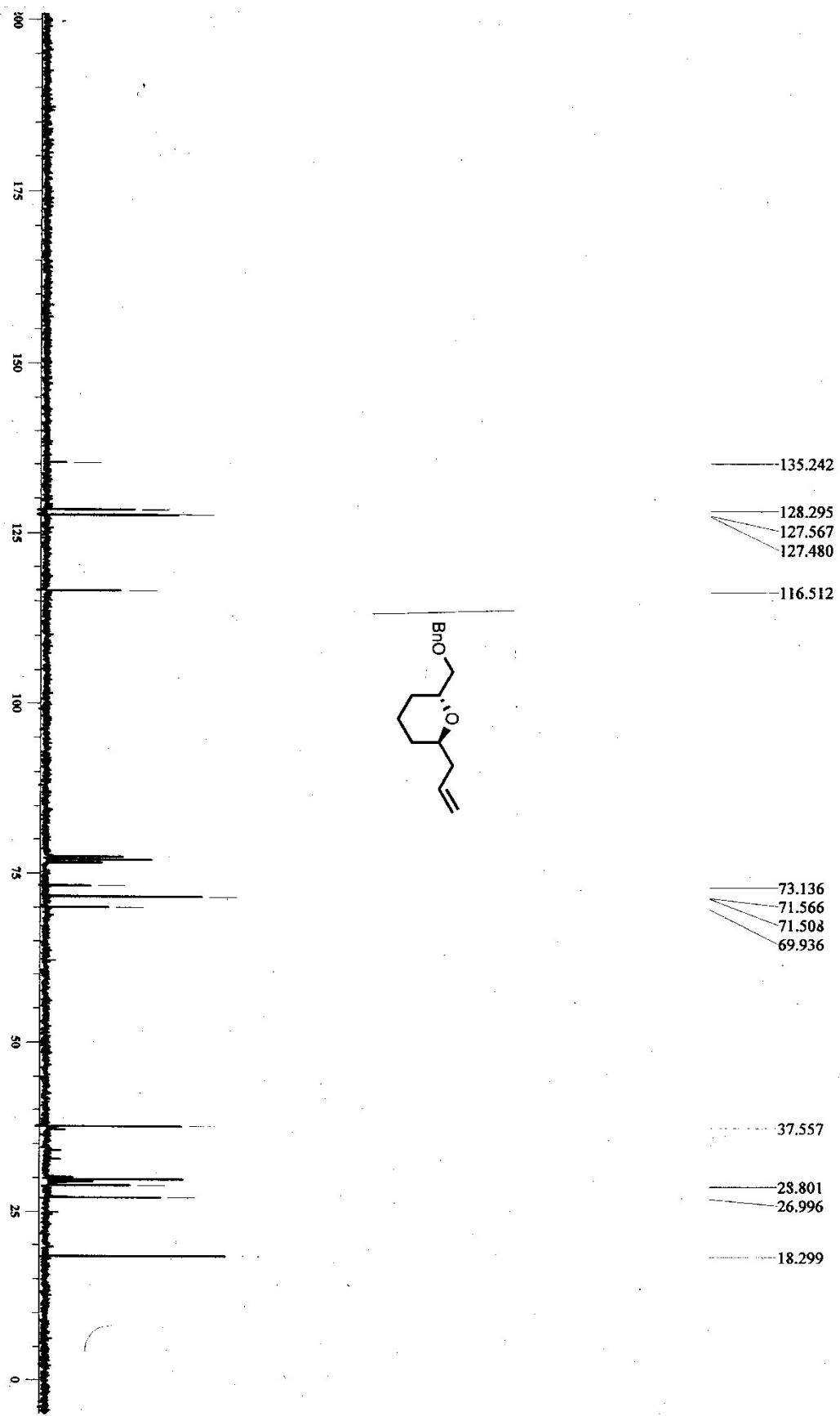


Expanded 2D ¹H NOE of 5 (500 MHz, CDCl₃)



1D ^1H NOE of 5 at 3.56 ppm

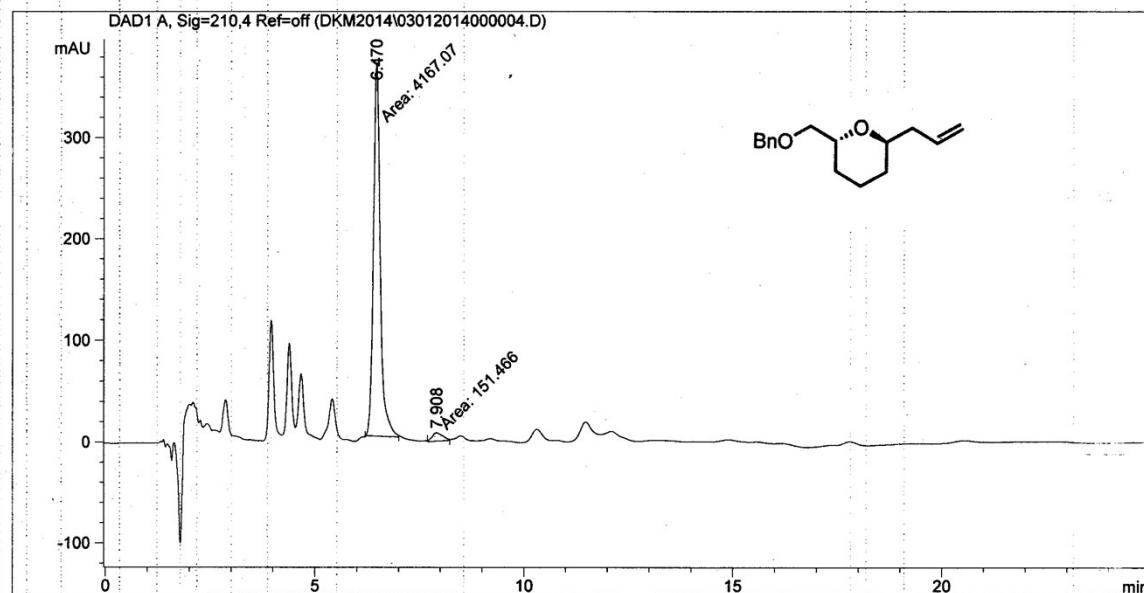




^{13}C NMR of 2a (75 MHz, CDCl_3)

Data File D:\DATA\DKM2014\03012014000004.D
Sample Name: DKM-DL-CYC-2 246

```
=====
Acq. Operator : V.RAJESH
Acq. Instrument : Instrument 1
Injection Date : 1/3/2014 12:16:33 PM
Location : Vial 22
Inj Volume : 20 µl
Acq. Method : D:\METHODS\LCM.m
Last changed : 1/3/2014 12:36:29 PM by V.RAJESH
Analysis Method : D:\METHODS\DATURA.m
Last changed : 1/3/2014 12:44:04 PM by V.RAJESH
Method Info : COLUMN:ATLANTIS C181 50 X4.6MM,5U
MOBILEPHASE:70% ACN IN H2O (0.1 F.A)
FLOWRATE:1.0ML/MIN
DETECTION:210NM
Sample Info :
```



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

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

Signal 1: DAD1 A, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.470	MM	0.1884	4167.06836	368.72510	96.4926
2	7.908	MM	0.2983	151.46640	8.46200	3.5074
Totals :					4318.53476	377.18710

Instrument 1 1/3/2014 12:44:13 PM V.RAJESH

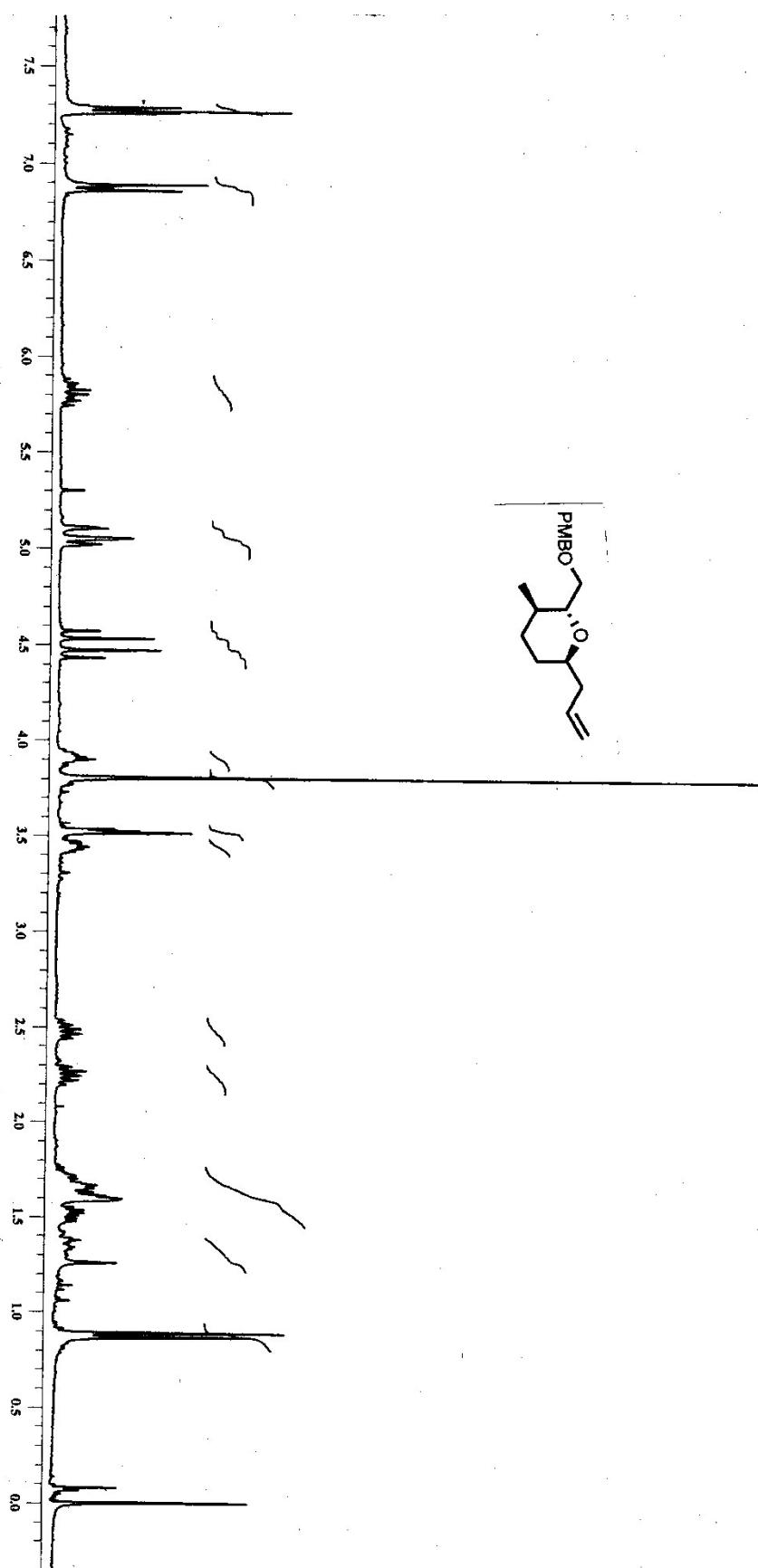
Page 1 of 2

HPLC of compound 2a

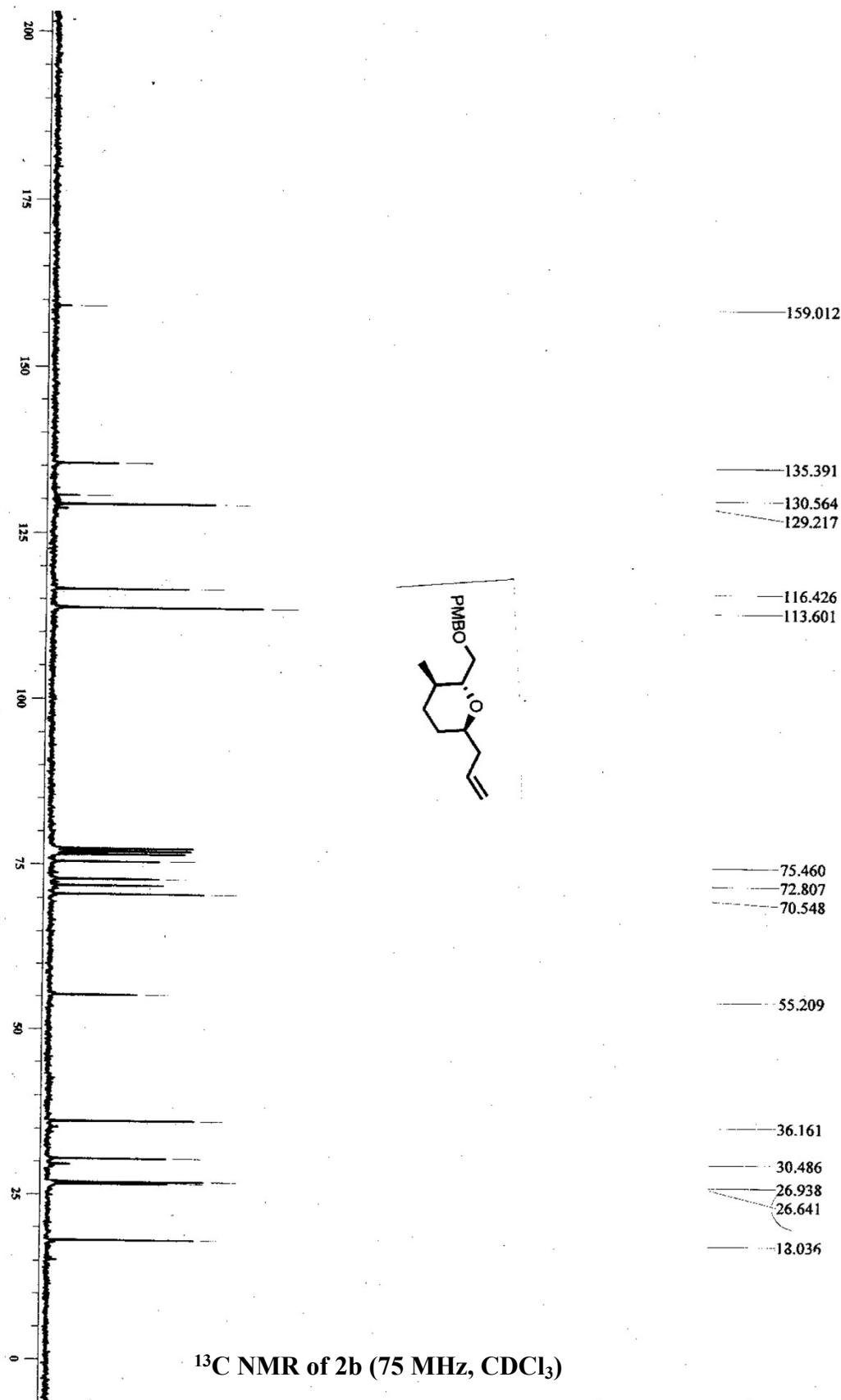
Display Report - Selected Window Selected Analysis

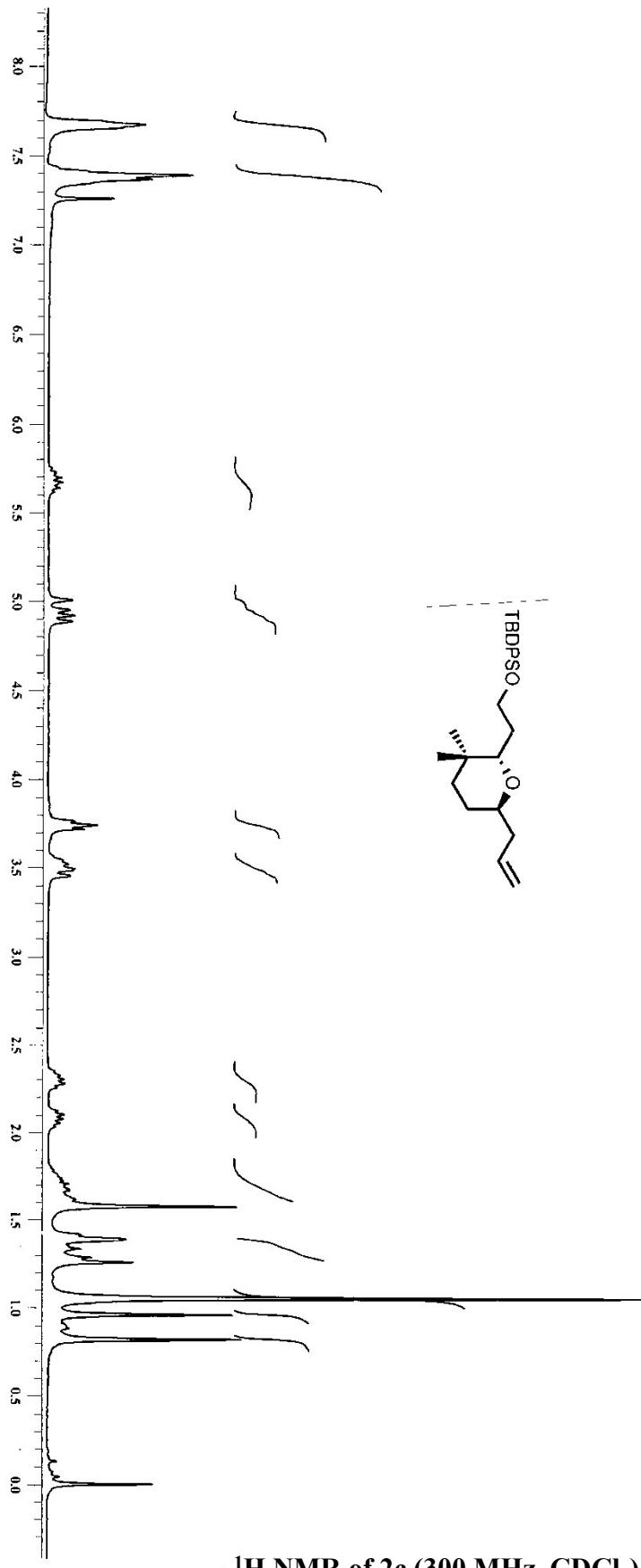
Analysis Name: 03012014000004.D

Method: LCM.M

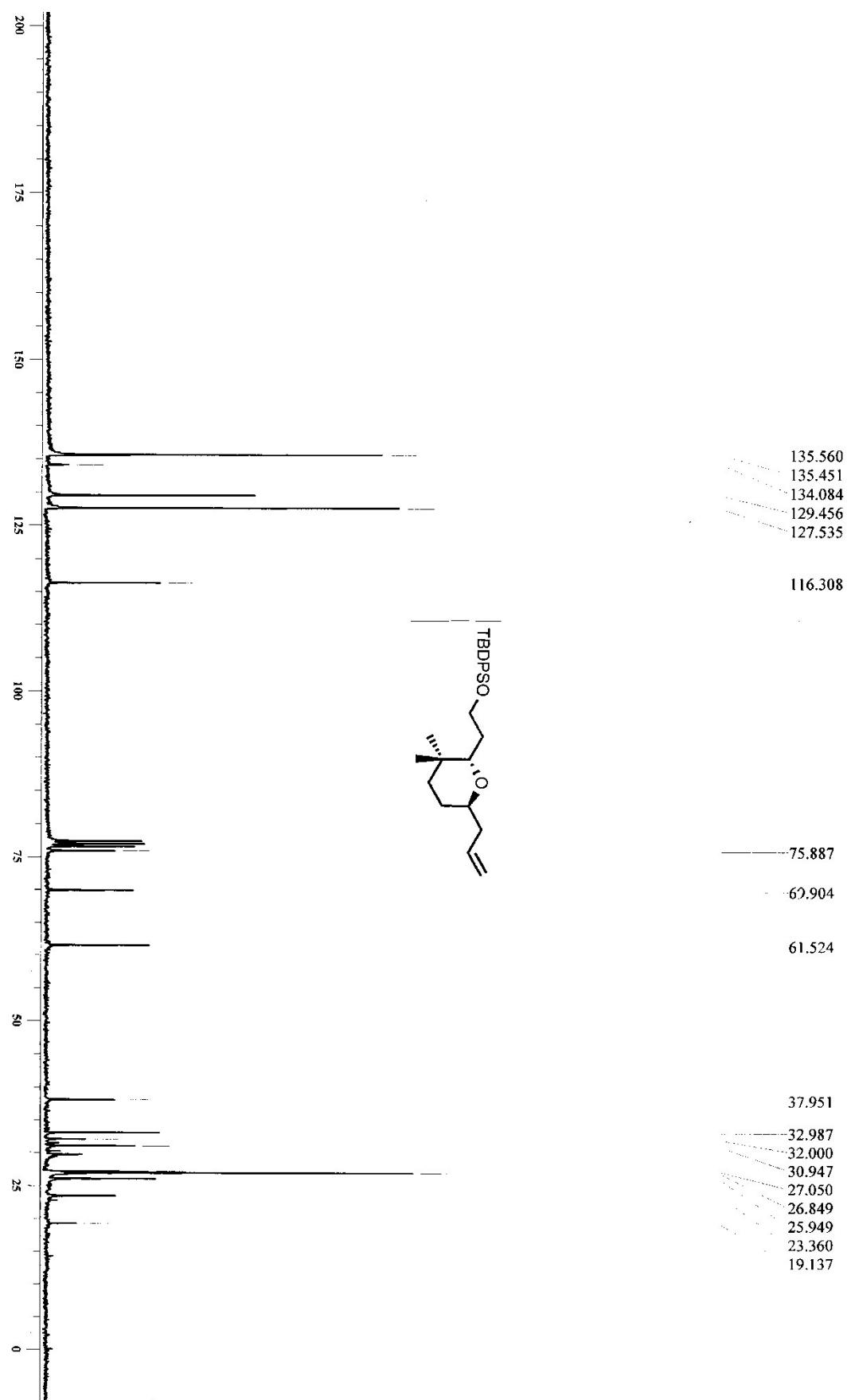


^1H NMR of 2b (300 MHz, CDCl_3)





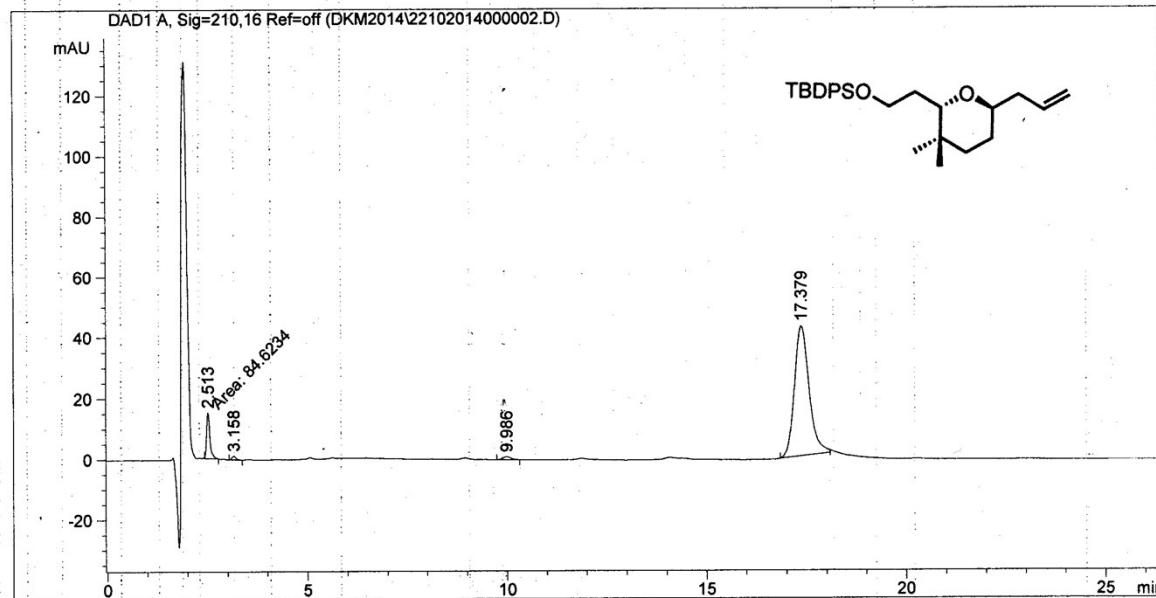
^1H NMR of 2c (300 MHz, CDCl_3)



^{13}C NMR of 2c (75 MHz, CDCl_3)

Data File D:\DATA\DKM2014\22102014000002.D
 Sample Name: DKM-BKR-TBDB 436

```
=====
Acq. Operator : V.RAJESH          Seq. Line : 1
Acq. Instrument : Instrument 1   Location : Vial 32
Injection Date : 10/22/2014 10:46:50 AM Inj : 1
                                                Inj Volume : 20 µl
Acq. Method : D:\METHODS\LCM.m
Last changed : 10/22/2014 10:59:19 AM by V.RAJESH
Analysis Method : D:\METHODS\LCM.m
Last changed : 10/22/2014 10:05:22 AM by V.RAJESH
Method Info : COLUMN: ATLANTIS C18, 4.6 X 150MM, 5U
               MOBILEPHASE: 80% ACN IN WATER(0.1% F.A)
               DECTION: 210NM
               FLOWRATE: 1ML/MIN
```



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

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

Signal 1: DAD1 A, Sig=210,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.513	MM	0.0933	84.62338	15.11111	7.0739
2	3.158	BB	0.1124	8.84328	1.22229	0.7392
3	9.986	BB	0.2119	14.44650	1.00388	1.2076
4	17.379	BB	0.3892	1088.36340	42.71683	90.9792
Totals :				1196.27657	60.05410	

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

Instrument 1 10/22/2014 11:16:22 AM V.RAJESH

Page 1 of 1

HPLC of compound 2c

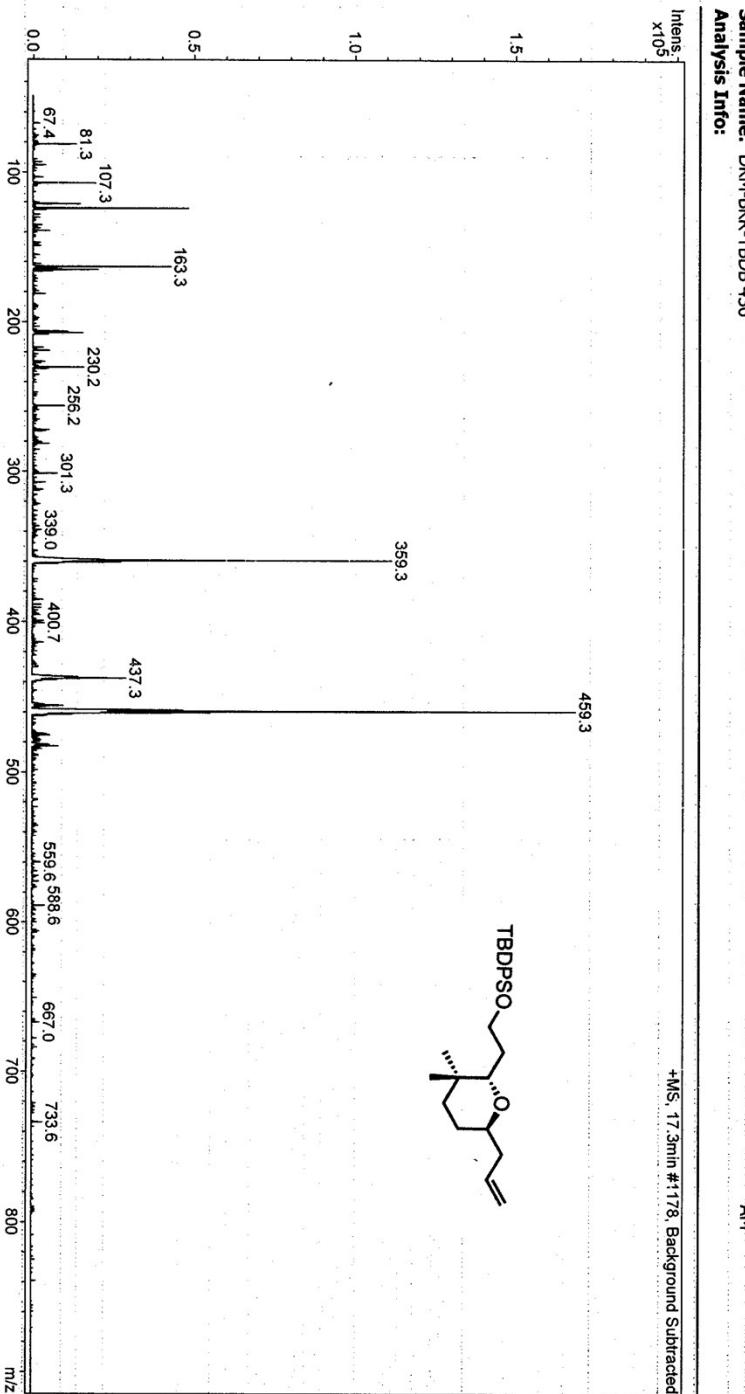
Display Report - Selected Window Selected Analysis

Analysis Name: 22102014000002.D

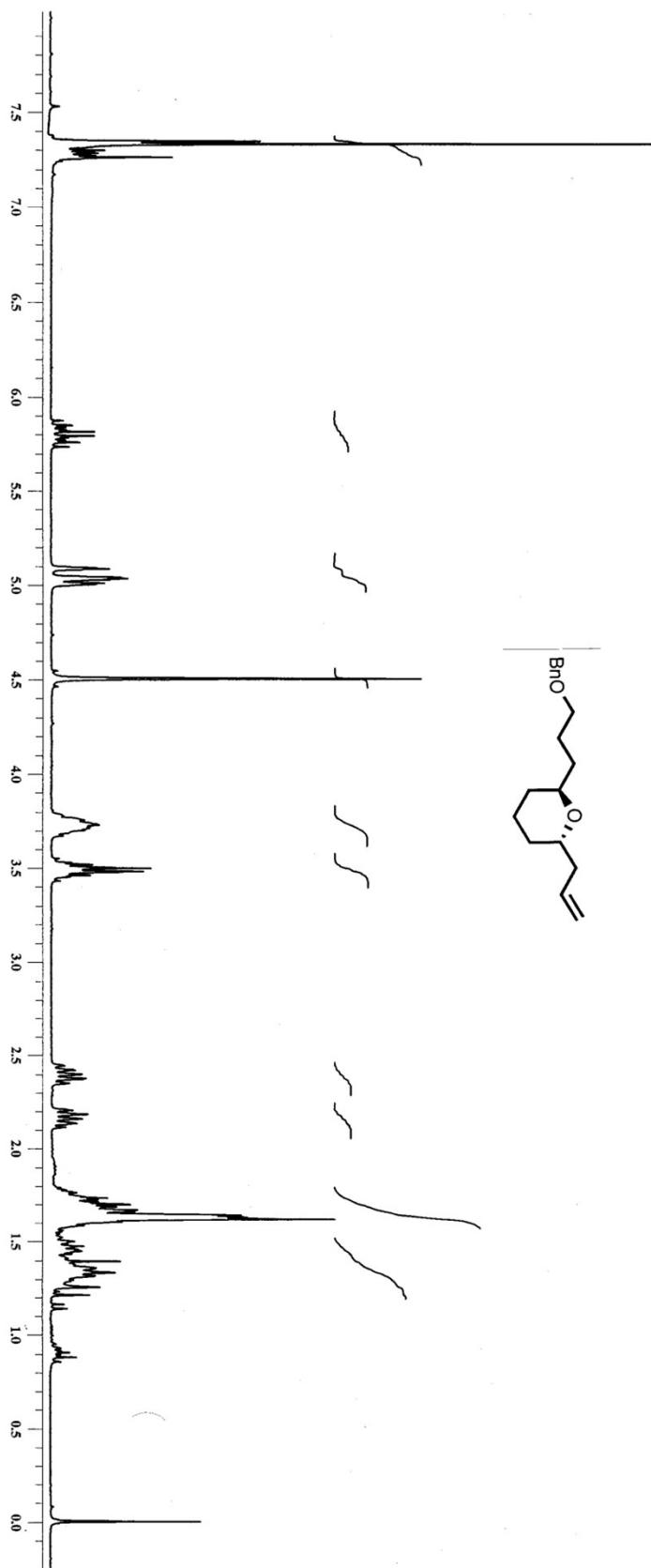
Instrument: LC-MSD-Trap-SL

Print Date: 10/22/2011 11:17:51

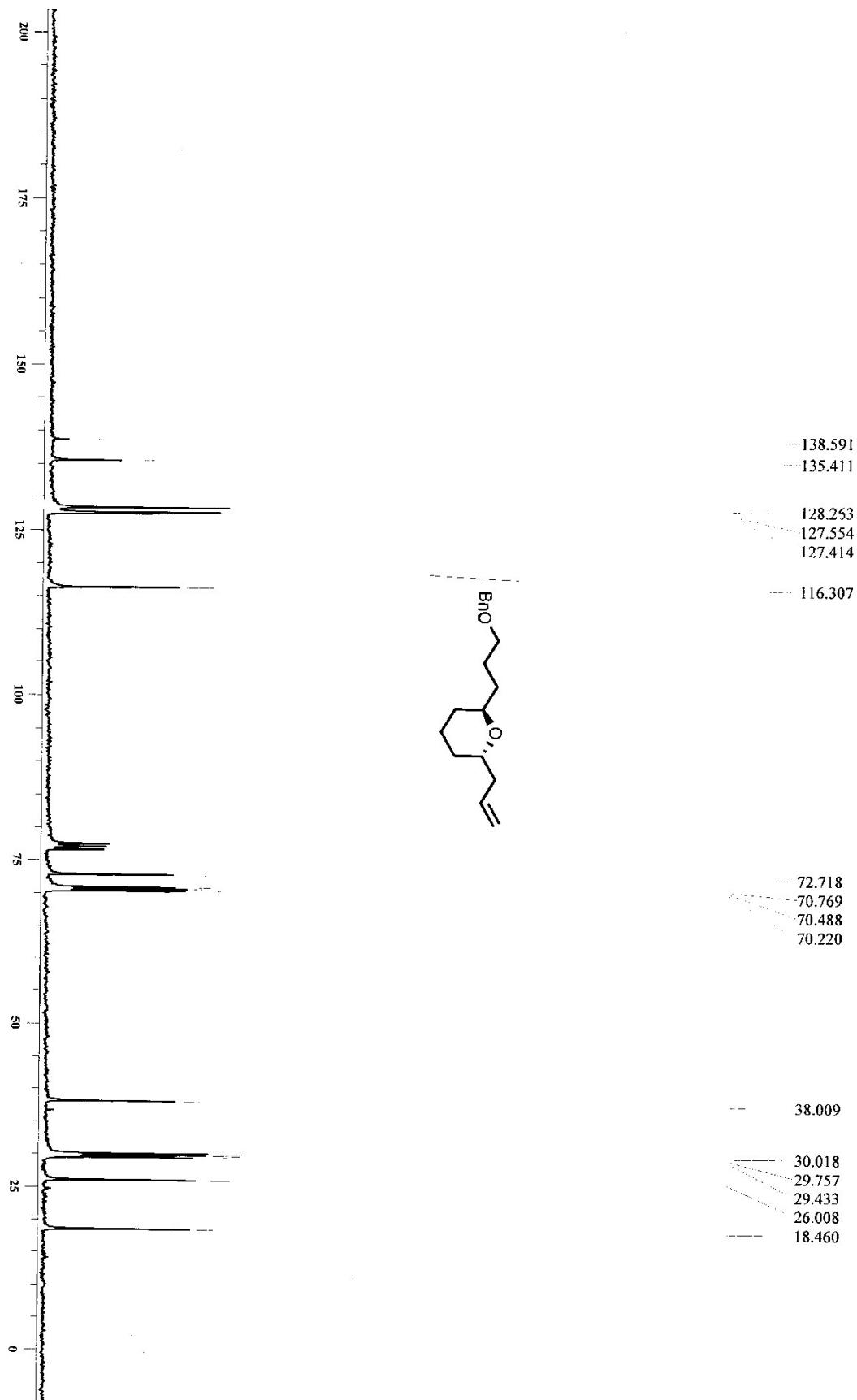
Print Date: 10/22/2011 11:17:51



LC-MS of compound 2c



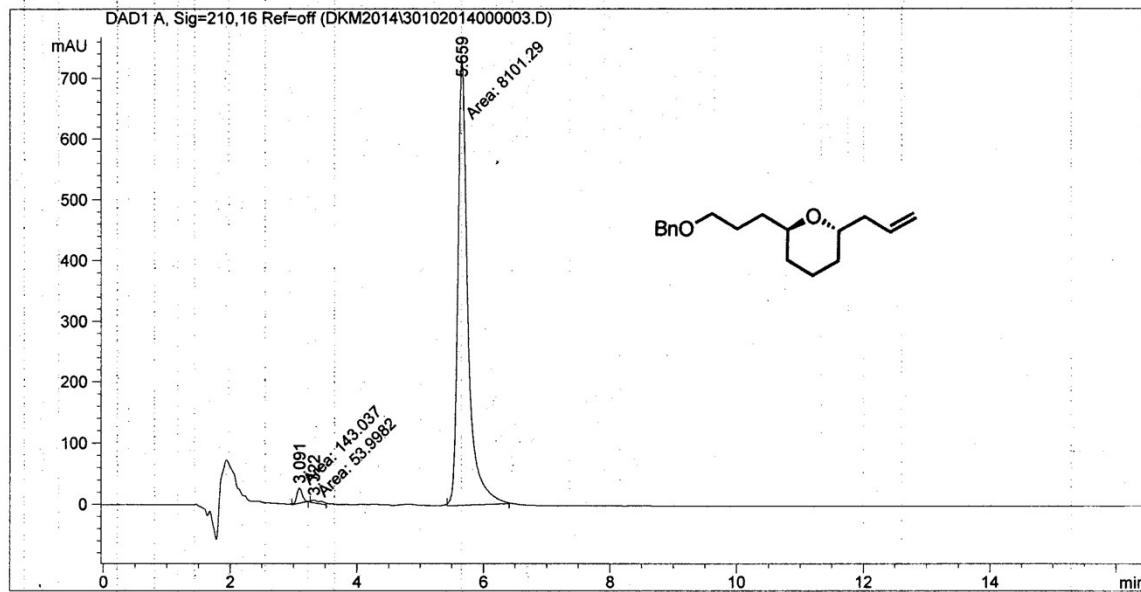
^1H NMR of 2d (300 MHz, CDCl_3)



^{13}C NMR of 2d (75 MHz, CDCl_3)

Data File D:\DATA\DKM2014\30102014000003.D
 Sample Name: DKM-DSR-BN 274

```
=====
Acq. Operator : V.RAJESH          Seq. Line : 1
Acq. Instrument : Instrument 1   Location : Vial 32
Injection Date : 10/30/2014 12:23:50 PM    Inj : 1
                                                Inj Volume : 20 µl
Acq. Method : D:\METHODS\LCM.m
Last changed : 10/30/2014 12:21:45 PM by V.RAJESH
Analysis Method : D:\METHODS\LCM.m
Last changed : 11/10/2014 11:06:17 AM by V.RAJESH
Method Info : COLUMN:ATLANTIS C18, 4.6 X 150MM 5U
               MOBILE PHASE:80% ACN IN WATER(0.1% F.A)
               DECTION:210NM
               FLOWRATE:1ML/MIN
```



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

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

Signal 1: DAD1 A, Sig=210,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.091	MM	0.0962	143.03712	24.77896	1.7237
2	3.322	MM	0.1974	53.99820	4.55960	0.6507
3	5.659	MM	0.1844	8101.28857	732.35425	97.6256

Totals : 8298.32390 761.69281

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

Display Report - Selected Window Selected Analysis

Analysis Name: 30102014000003.D

Instrument: LC-MSD-Trap-SL

Print Date: 11/10/2014 11:07:37

Method: LCM.M

Operator: SARNA

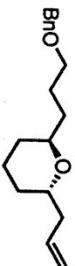
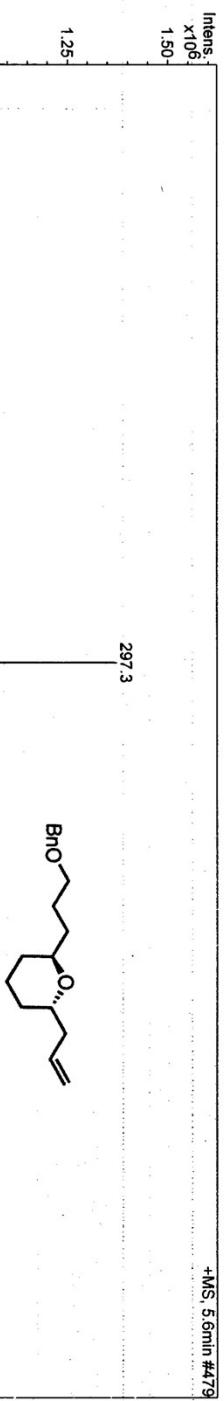
Acq. Date: 10/30/2014 24:24:27

Sample Name: DKM-DSR-BN 274

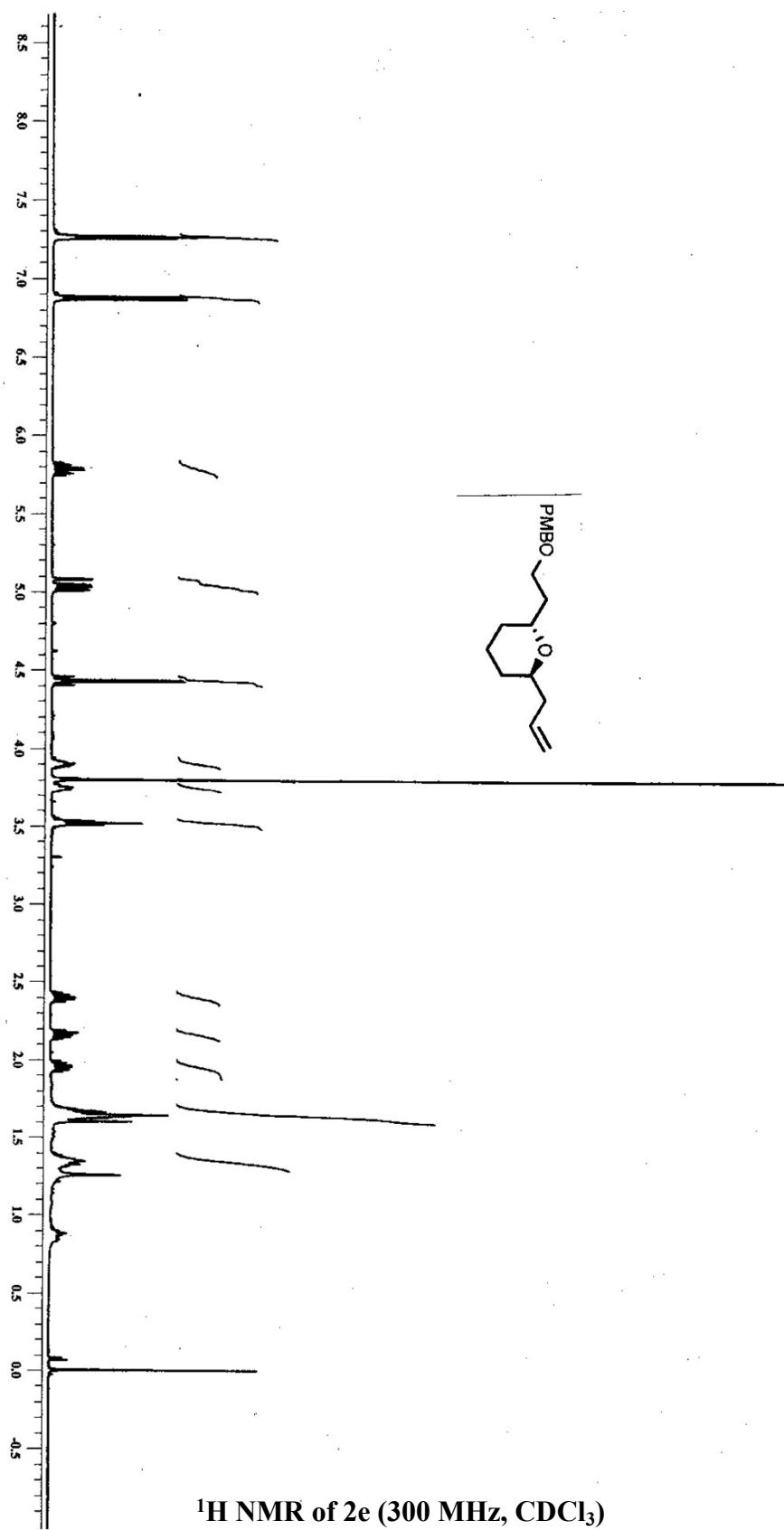
PM

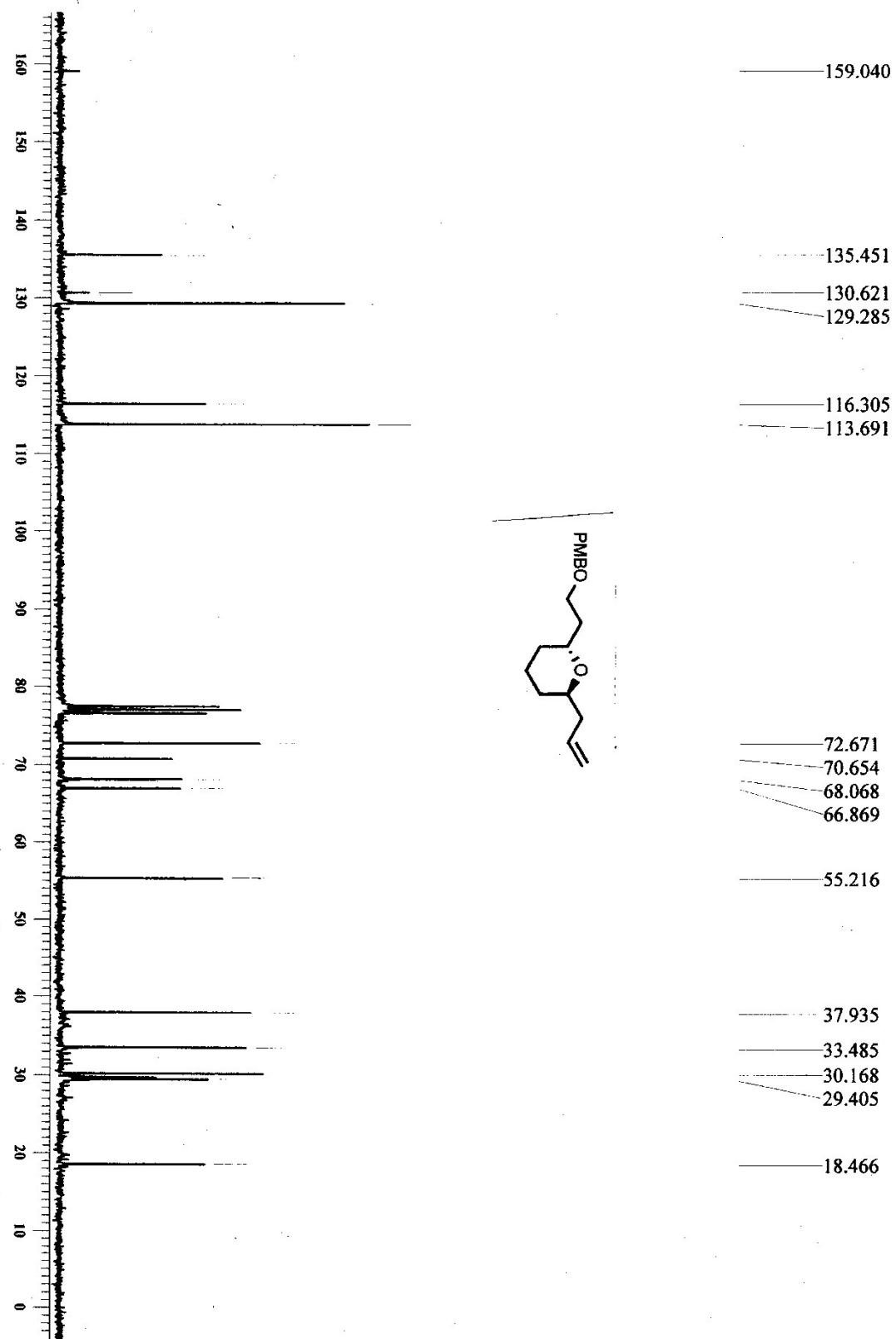
Analysis Info:

PM



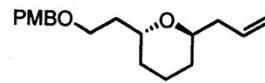
LC-MS of compound 2d





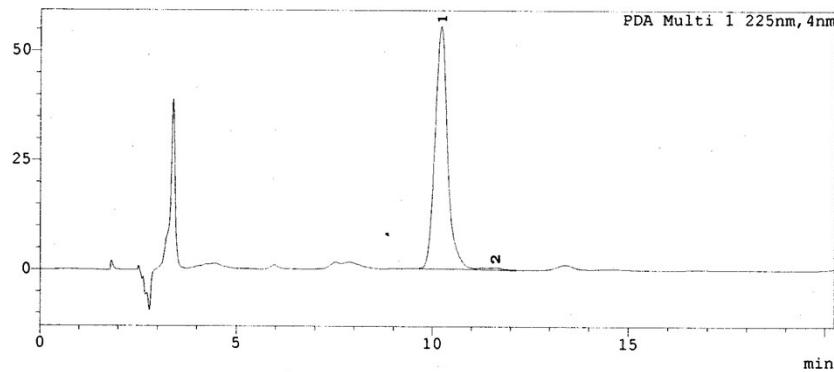
^{13}C NMR of 2e (75 MHz, CDCl_3)

Sample Code : BKP-290
Data File : 071015....6.lcd
Method : LC-MS Lab Method.lcm
Injection Volume : 20
Date Acquired : 10/7/2015 11:45:09 AM
Report File : LC-MS Data Report.lsr
Chromatographic Conditions : Column: Phenosphere ODS (2) (250 X 4.6mm, 5.0 u)
Mobile Phase: 70% MeOH in 0.1% FA, 1.0 mL/min



mAU

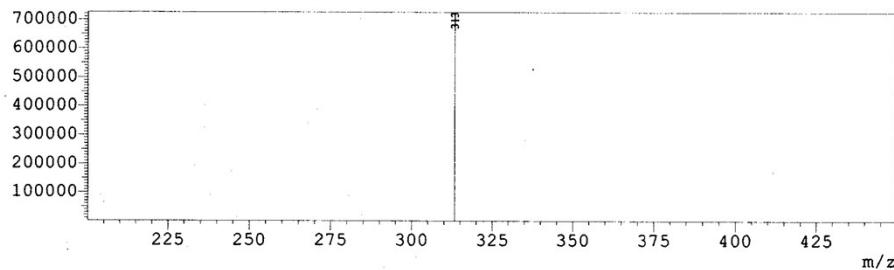
Chromatogram



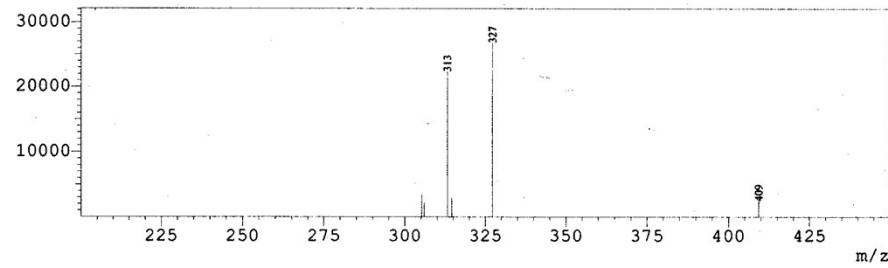
PDA Ch1 225nm

Peak#	Ret. Time	Peak Start	Peak End	Area	Area%
1	10.222	9.557	11.072	1241411	98.723
2	11.612	11.072	12.160	16053	1.277
Total				1257464	100.000

Q1 Scan Positive+

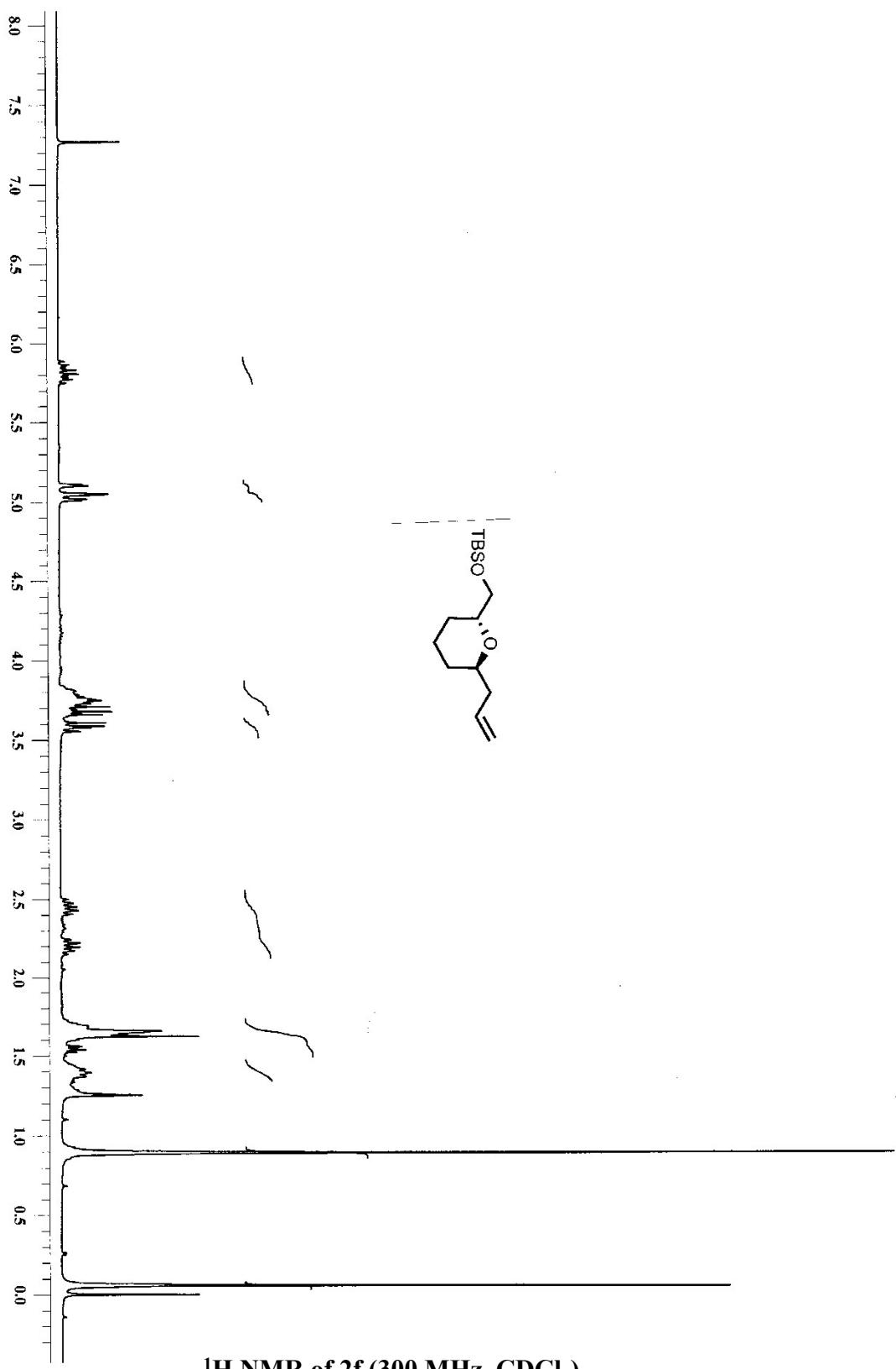
\$If\$(SpPrTab==SpPrTab) Spectrum Mode:Averaged 9.960-10.693(3985-4278)
BG Mode:Averaged 3.063-9.760(1226-3905)

Q1 Scan Positive+

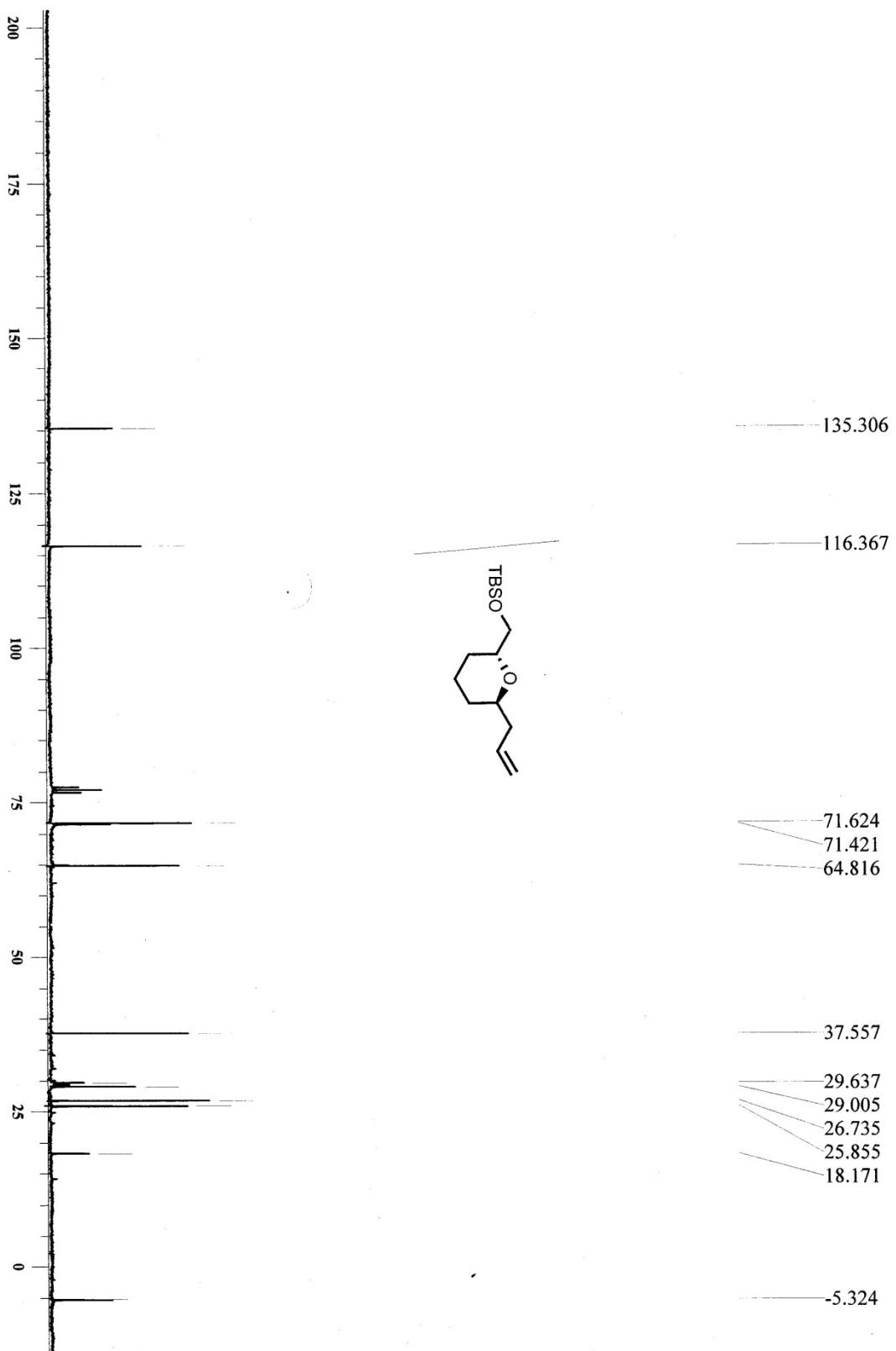
\$If\$(SpPrTab==SpPrTab) Spectrum Mode:Averaged 11.260-12.025(4505-4811)
BG Mode:Averaged 12.193-20.255(4878-8103)

LC-MS REPORT

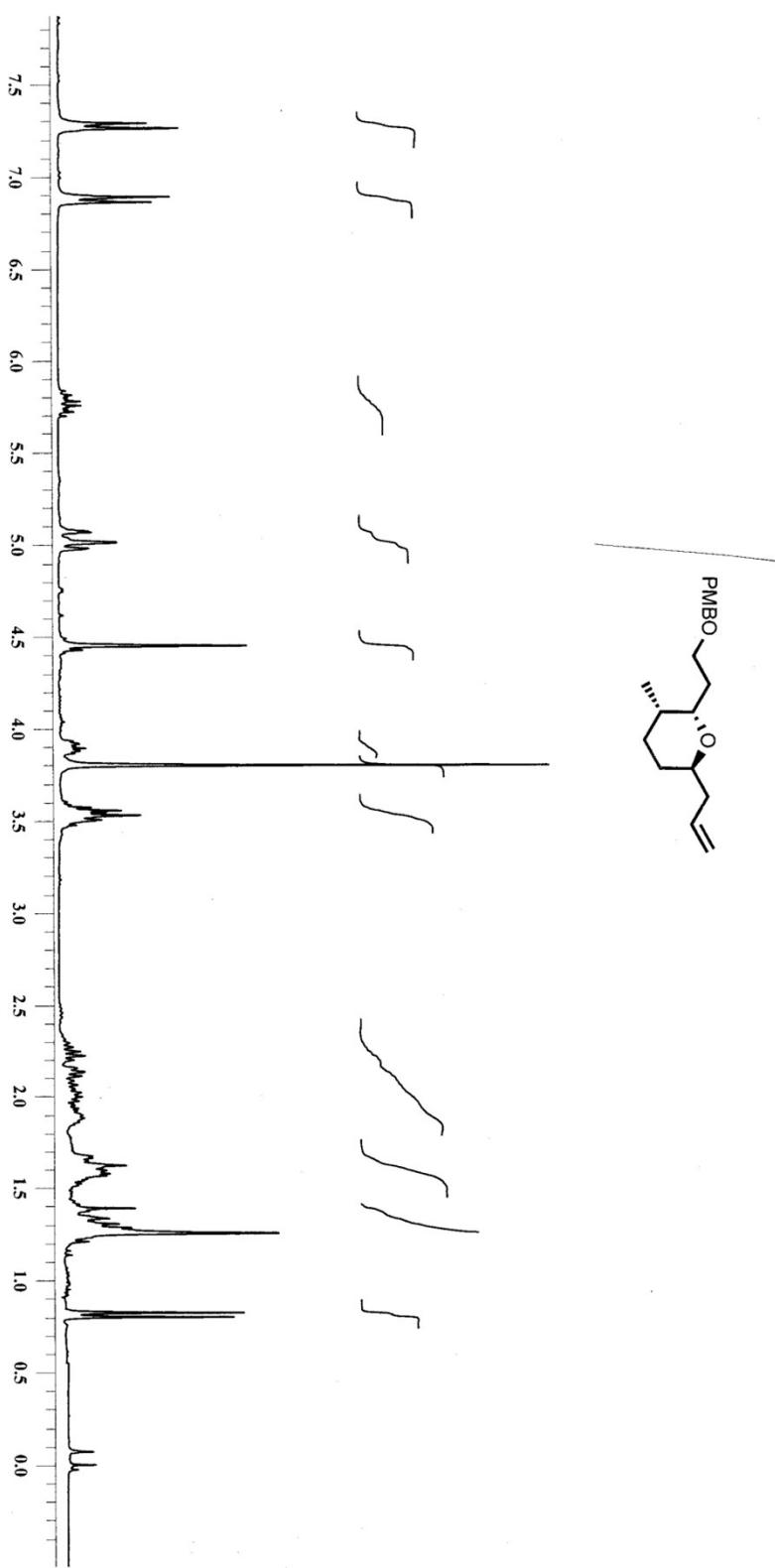
HPLC of compound 2e



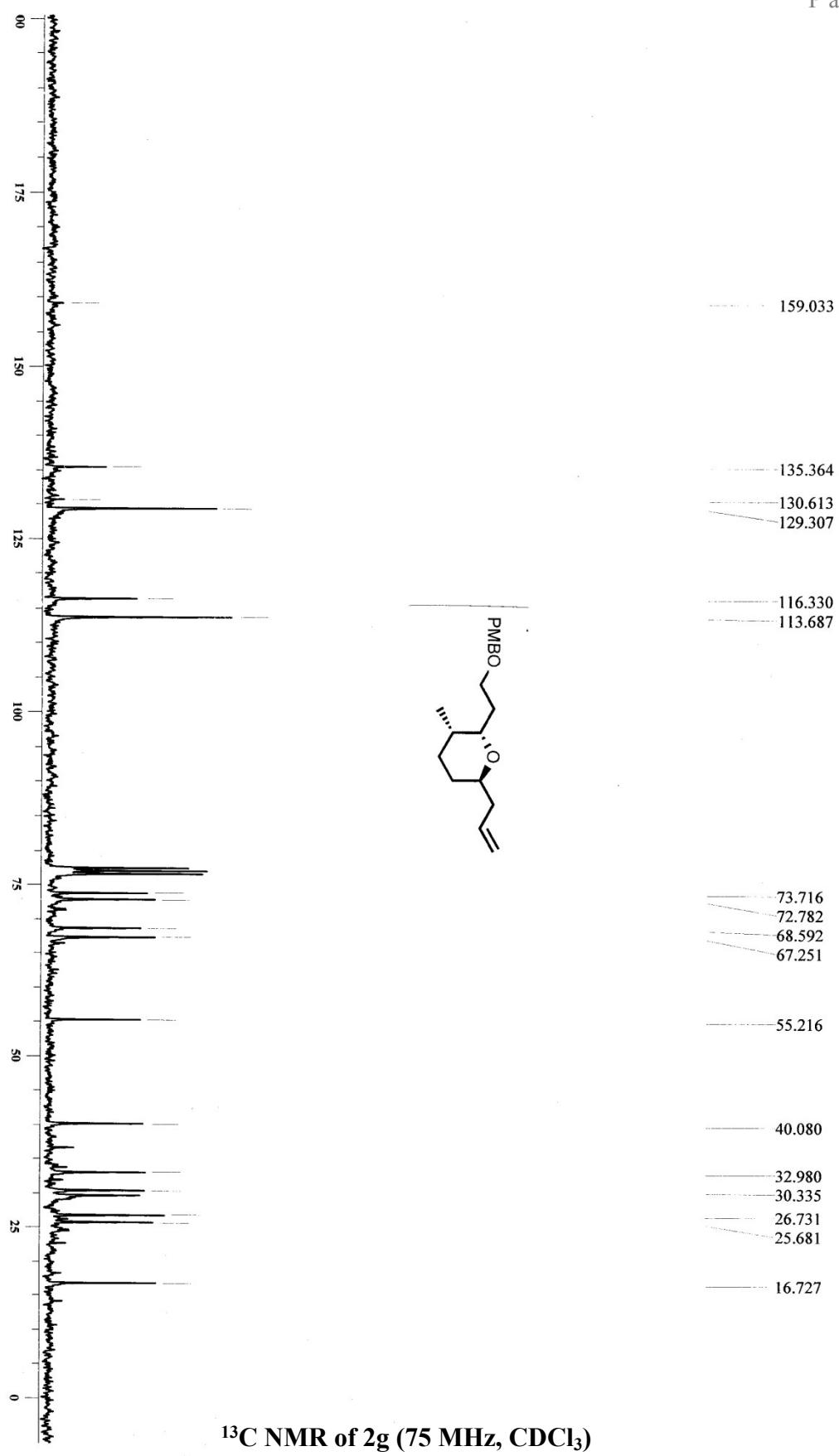
^1H NMR of 2f (300 MHz, CDCl_3)



^{13}C NMR of **2f** (75 MHz, CDCl_3)



^1H NMR of 2g (300 MHz, CDCl_3)

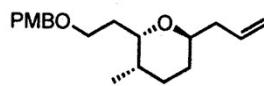




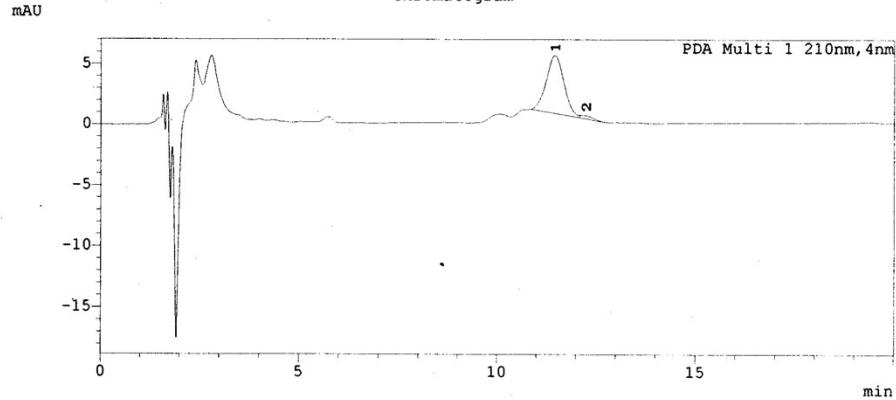
LC-MS DATA REPORT (IICT-NPL)

IICT-NPL

Sample Code : DKM-BKP-304
 Data File : 131015.2.lcd
 Method : LC-MS Lab Method.lcm
 Injection Volume : 20
 Date Acquired : 10/13/2015 11:20:16 AM
 Report File : LC-MS Data Report.lsr
 Chromatographic Conditions : Column: Atlantis C18 (150 X 4.6mm, 5.0u)
 Mobile Phase: 75% MeOH in 0.1% FA
 Flowrate: 1.0 mL/min



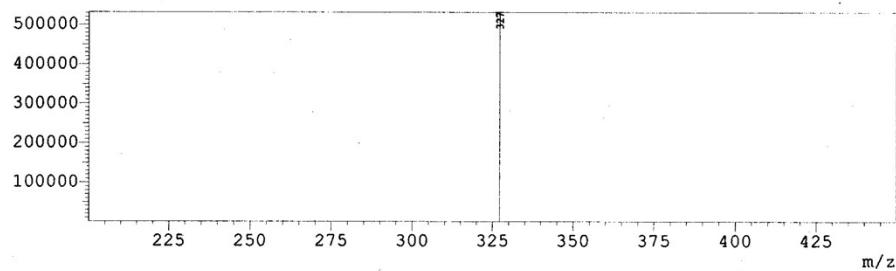
Chromatogram



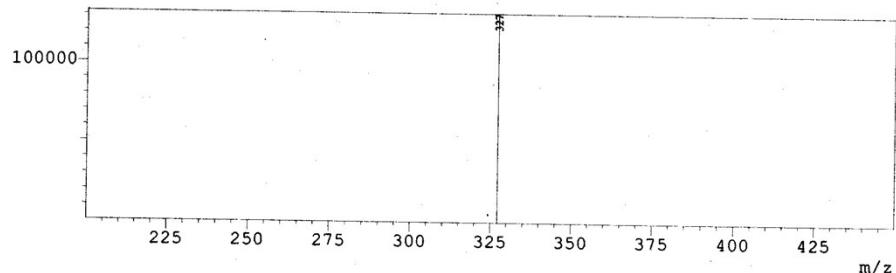
Peak Table

PDA Ch1 210nm					
Peak#	Ret. Time	Peak Start	Peak End	Area	Area%
1	11.462	10.869	12.085	147913	96.579
2	12.245	12.085	12.704	5239	3.421
Total				153152	100.000

Q1 Scan Positive+
 \$If\$(SpPrTab==SpPrTab) Spectrum Mode:Averaged 10.887-11.873(6533-7125)
 BG Mode:Averaged 1.085-10.788(652-6474)

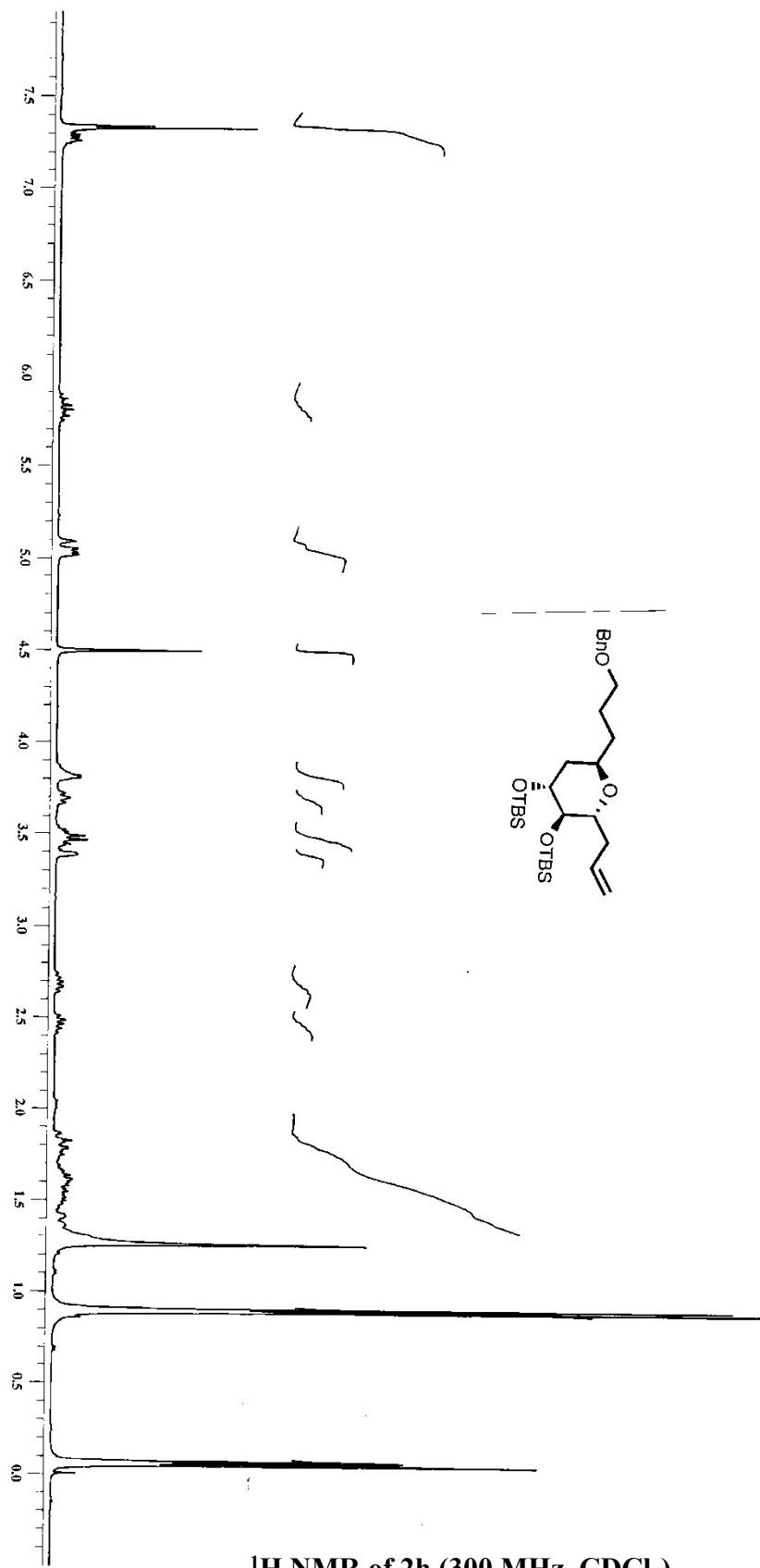


Q1 Scan Positive+
 \$If\$(SpPrTab==SpPrTab) Spectrum Mode:Averaged 11.873-12.498(7125-7500)
 BG Mode:Averaged 0.855-11.807(514-7085)

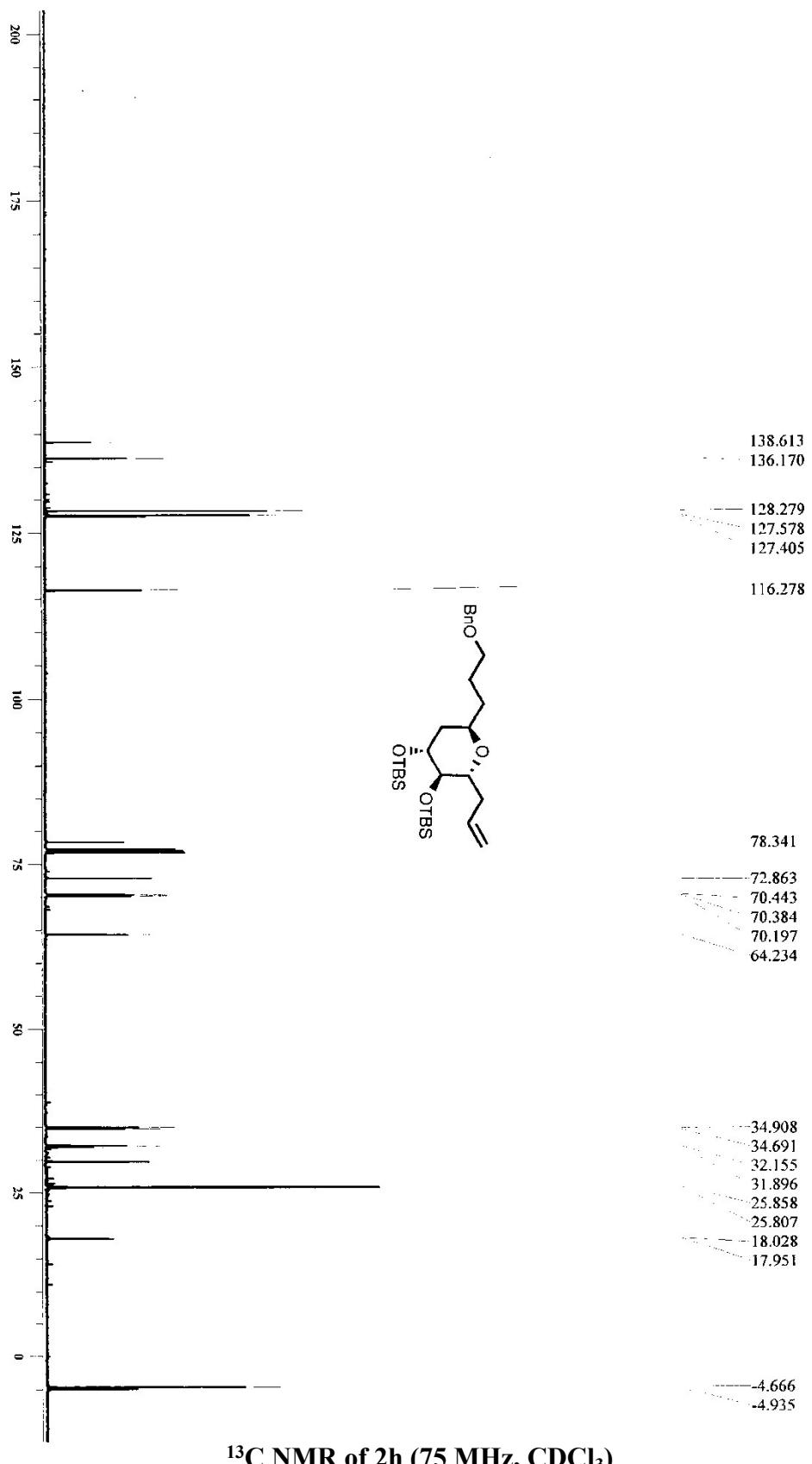


LC-MS REPORT

HPLC of compound 2g



¹H NMR of 2h (300 MHz, CDCl₃)

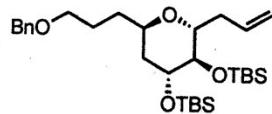




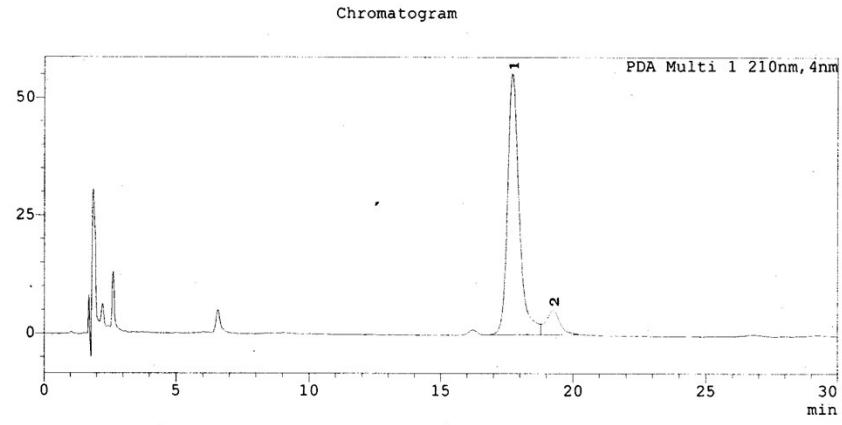
LC-MS DATA REPORT (IICCT-NPL)

IICCT-NPL

Sample Code : BKP-534
 Data File : 061015....3.lcd
 Method : LC-MS Lab Method.lcm
 Injection Volume : 20
 Date Acquired : 10/6/2015 7:31:06 PM
 Report File : LC-MS Data Report.lsr
 Chromatographic Conditions : Column: Atlantis C18 (150 X 4.6mm, 5.0 μ)
 Mobile Phase: 95% ACN in 0.1% FA, 1.0 mL/min



mAU

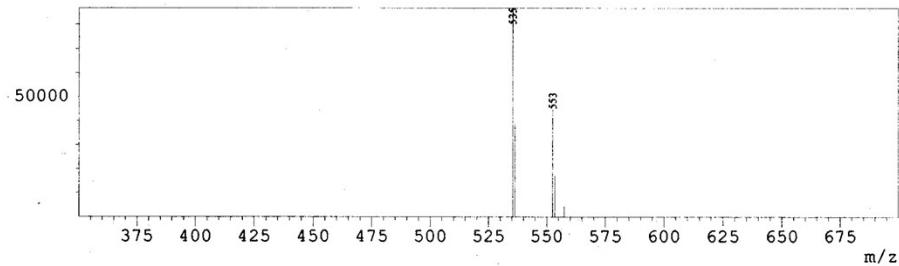


PDA Ch1 210nm

Peak#	Ret. Time	Peak Start	Peak End	Area	Area%
1	17.706	16.821	18.795	1703390	90.202
2	19.256	18.795	20.267	185026	9.798
Total				1888416	100.000

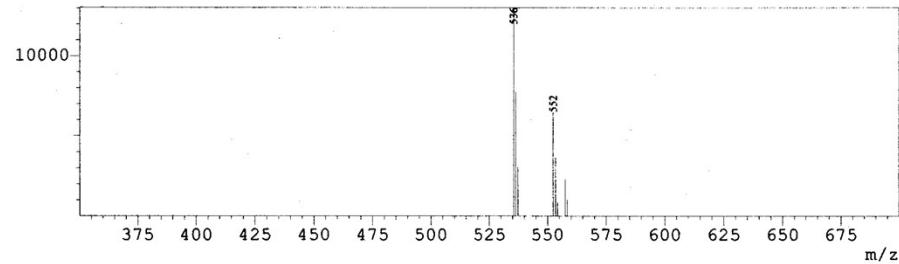
Q1 Scan Positive+

\$If\$(SpPrTab==SpPrTab) Spectrum Mode:Averaged 17.300-18.288(6921-7316)
 BG Mode:Averaged 5.195-17.235(2079-6895)



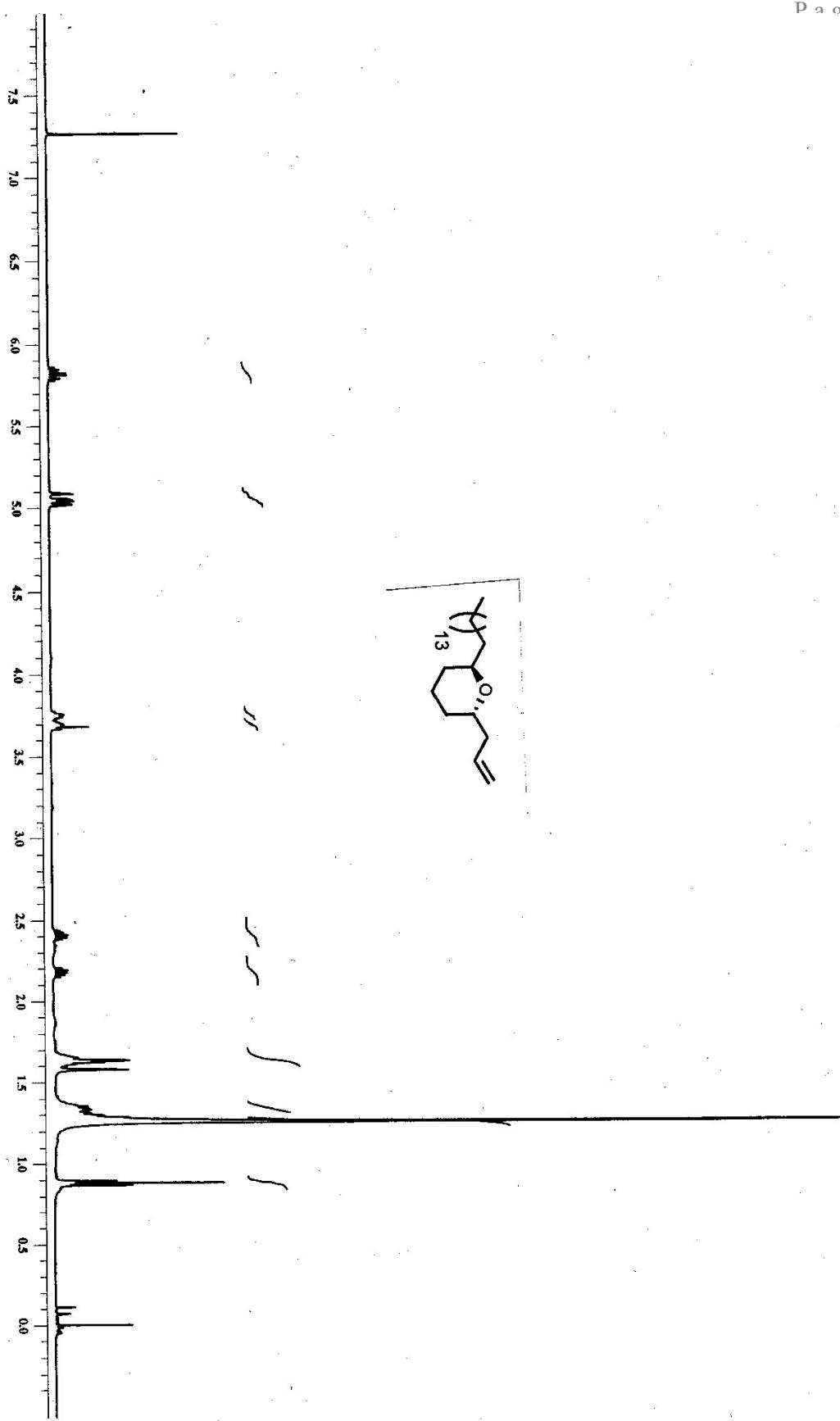
Q1 Scan Positive+

\$If\$(SpPrTab==SpPrTab) Spectrum Mode:Averaged 18.945-19.470(7579-7789)
 BG Mode:Averaged 19.470-33.090(7789-13237)

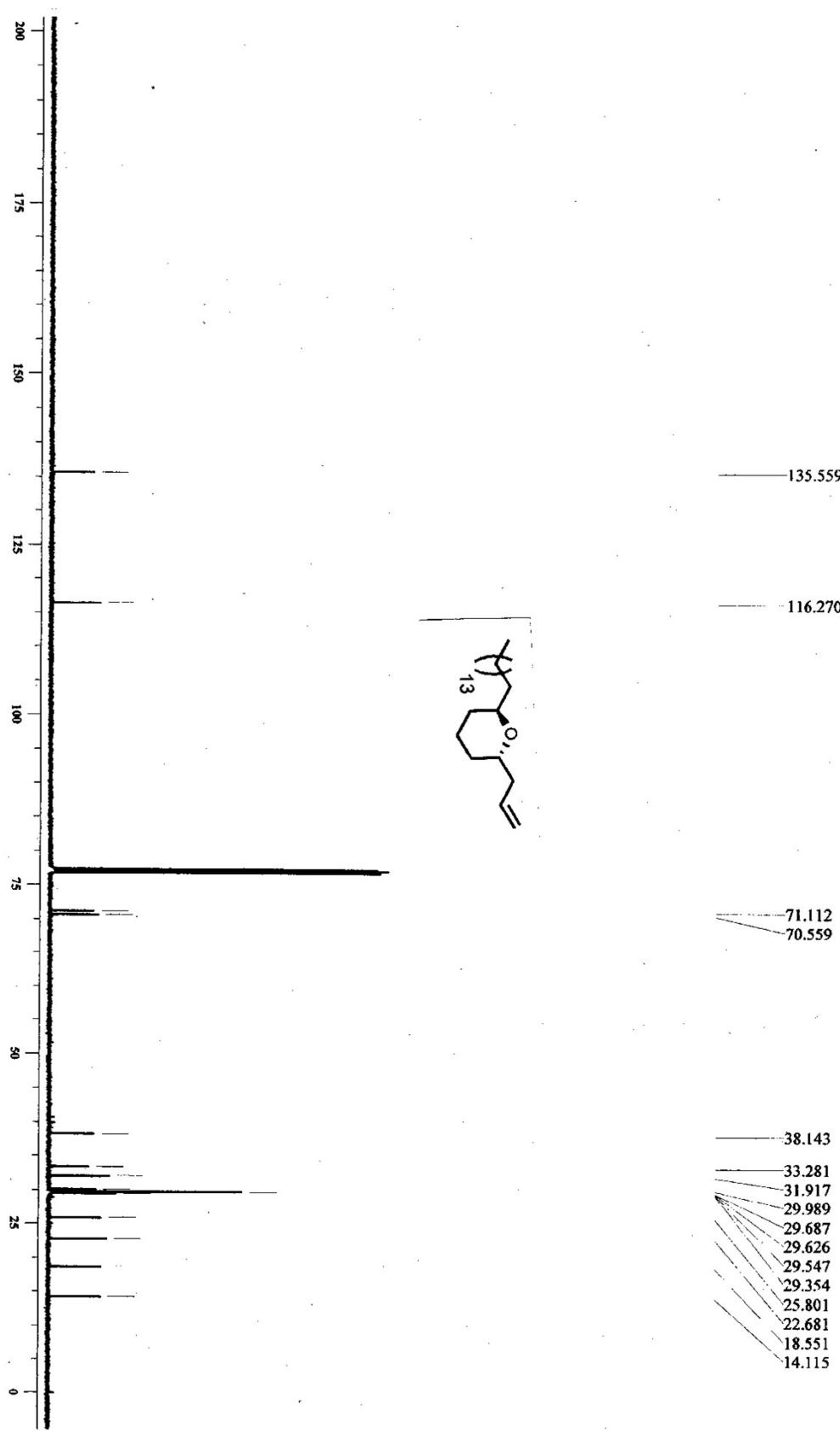


LC-MS REPORT

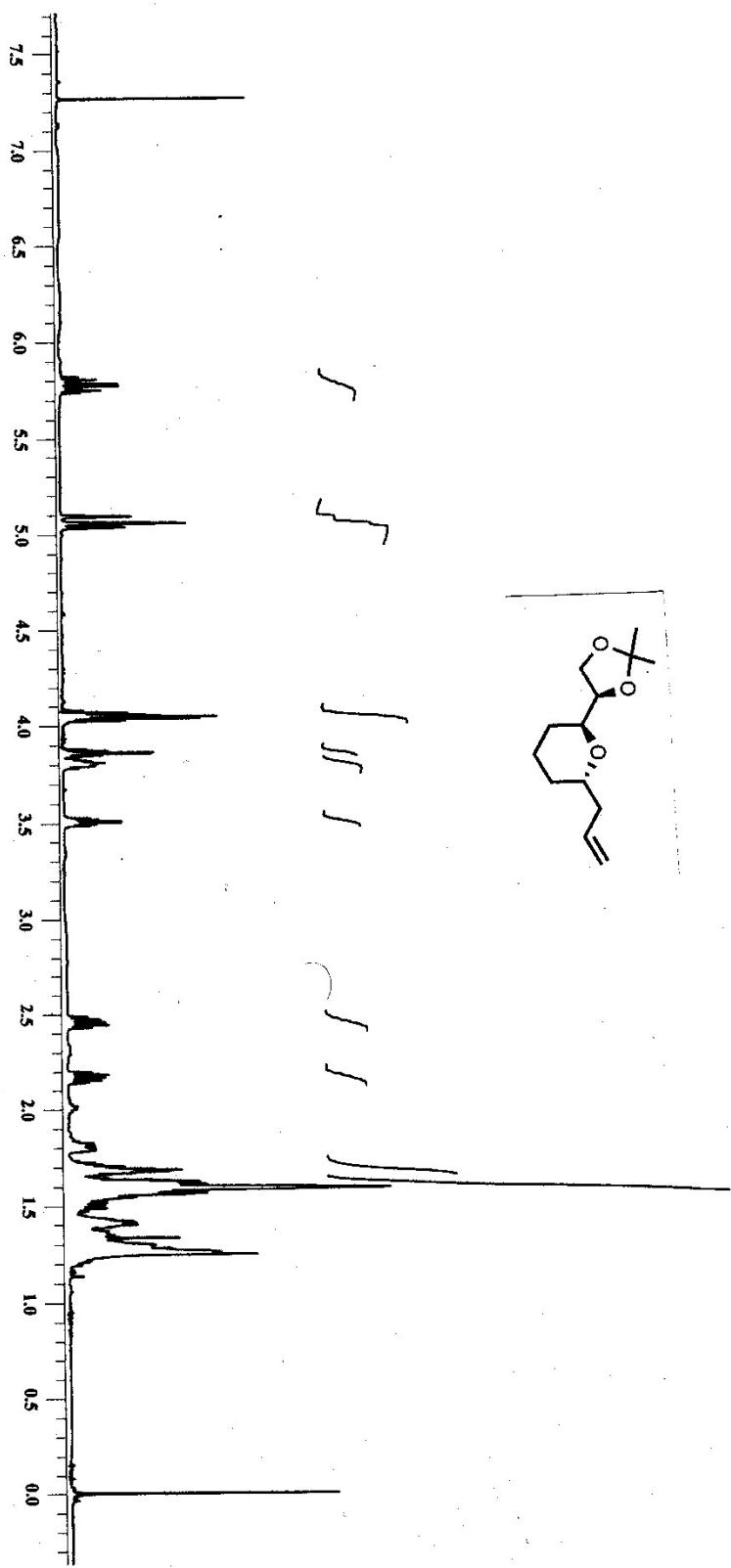
HPLC of compound 2h



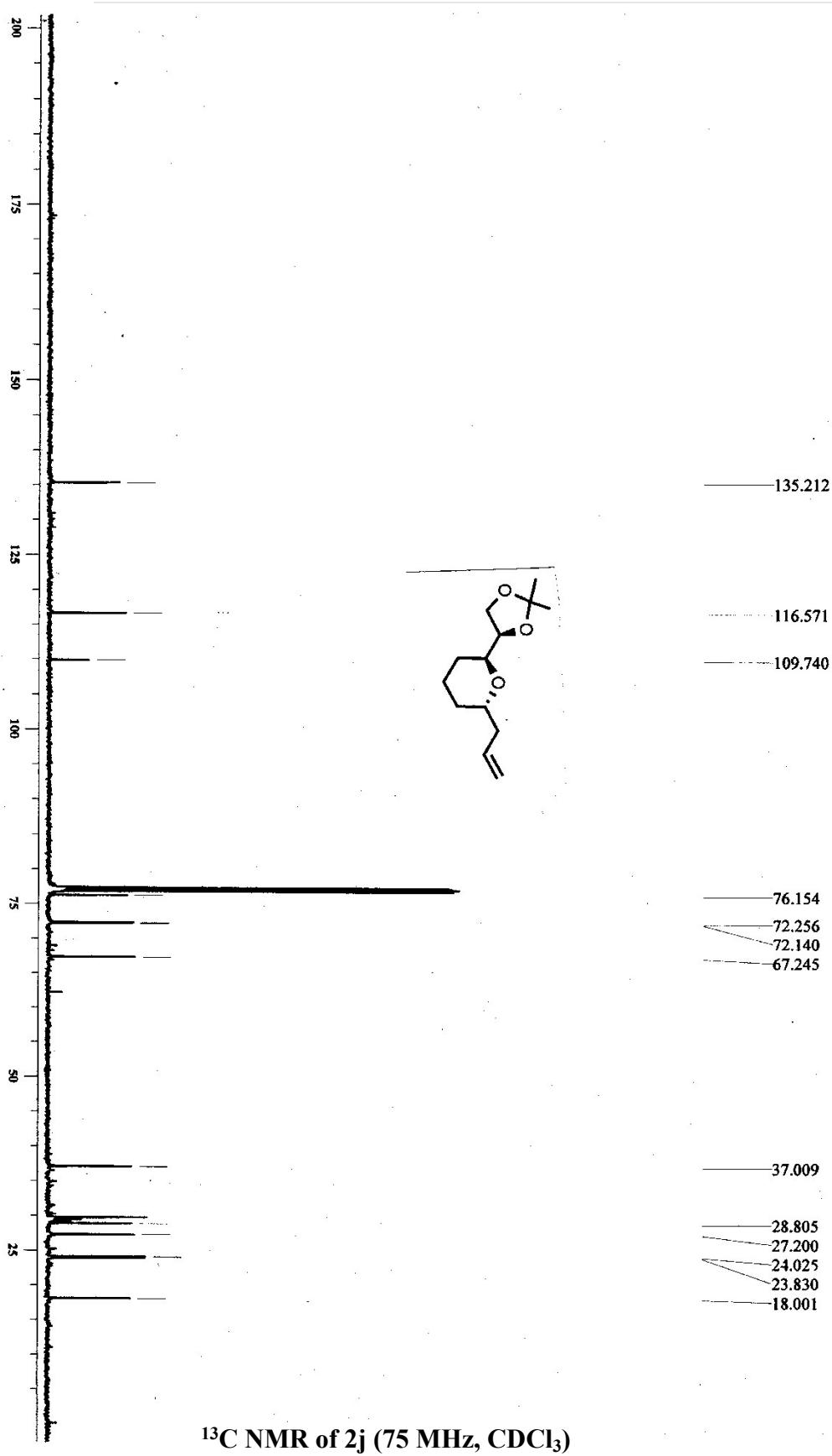
¹H NMR of 2i (300 MHz, CDCl_3)

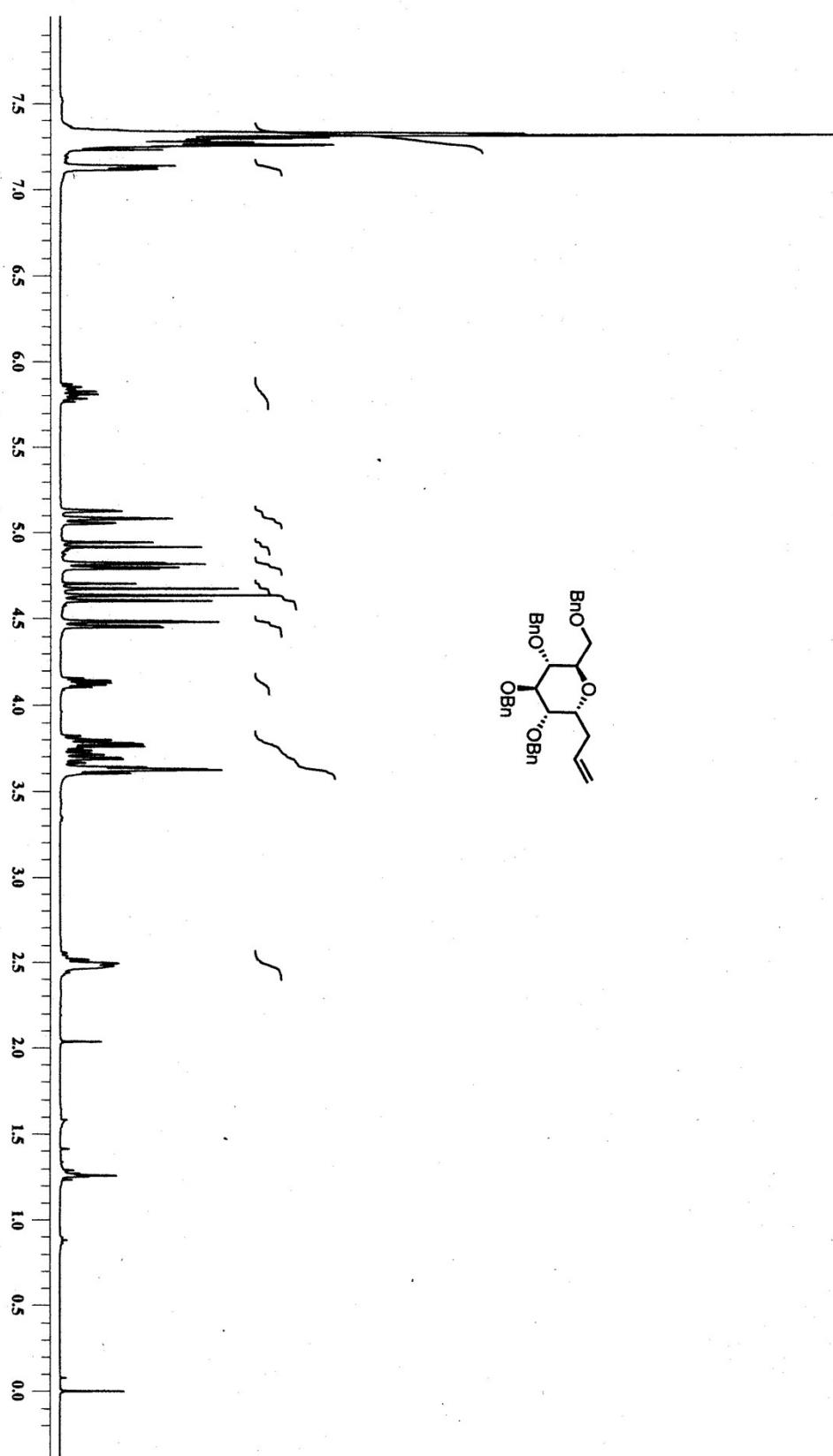


^{13}C NMR of 2i (75 MHz, CDCl_3)

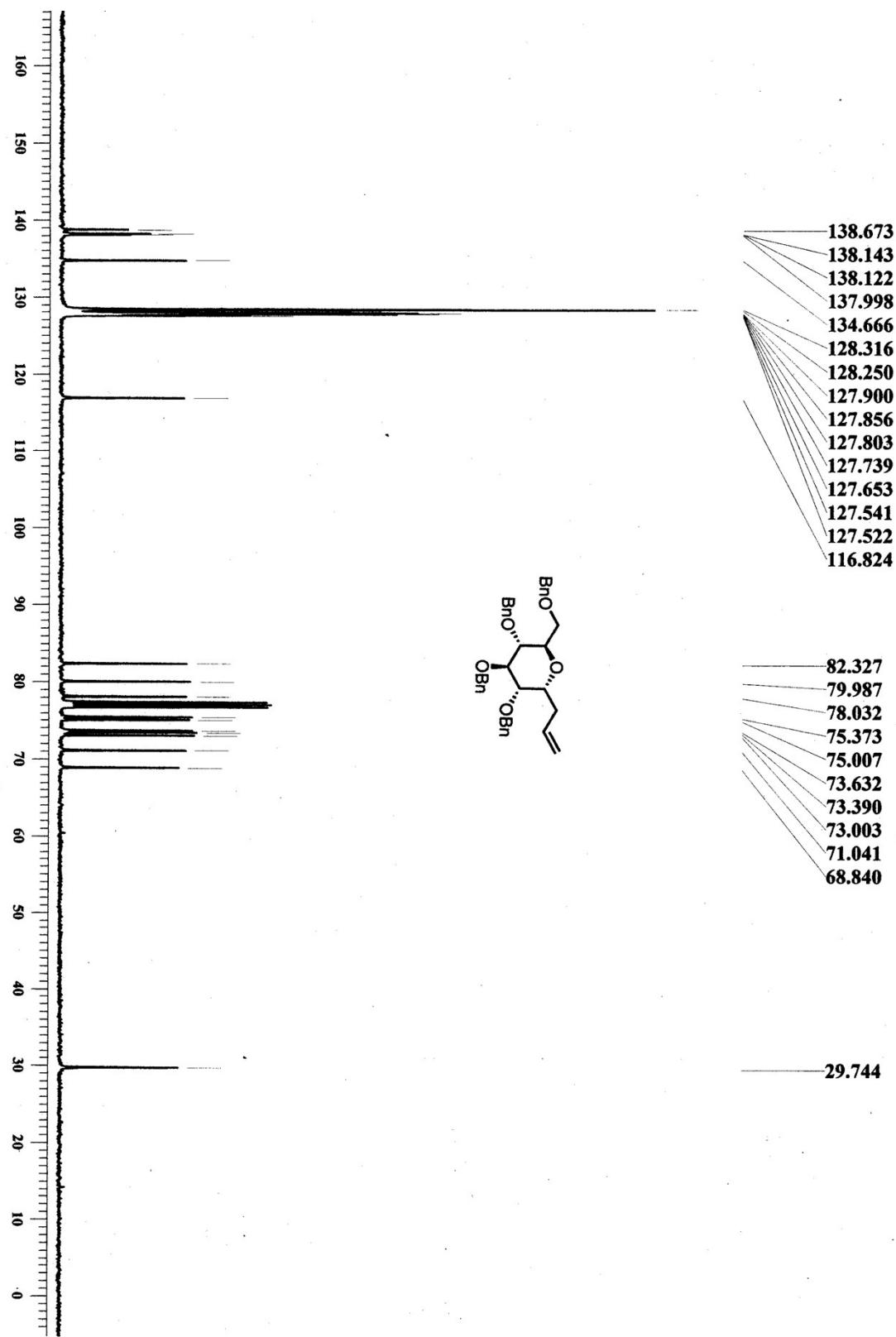


^1H NMR of 2j (300 MHz, CDCl_3)





¹H NMR of 2k (400 MHz, CDCl₃)

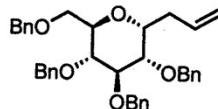




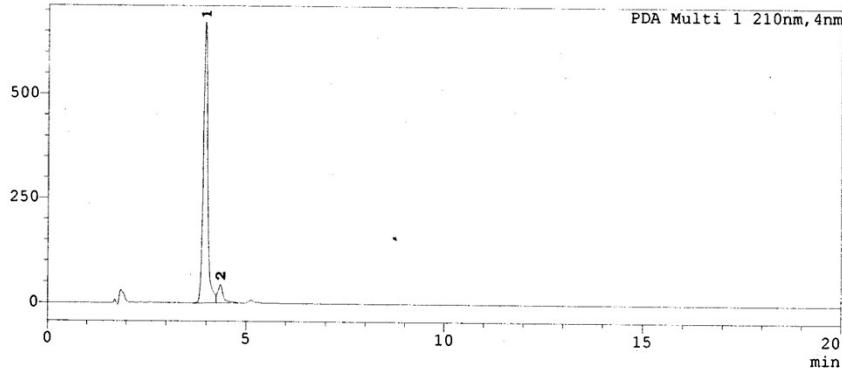
LC-MS DATA REPORT (IICT-NPL)

IICT-NPL

Sample Code : BKP-564
 Data File : 061015....5.lcd
 Method : LC-MS Lab Method.lcm
 Injection Volume : 20
 Date Acquired : 10/6/2015 8:52:27 PM
 Report File : LC-MS Data Report.lsr
 Chromatographic Conditions : Column: Atlantis C18 (150 X 4.6mm, 5.0 u)
 Mobile Phase: 95% ACN in 0.1% FA, 1.0 mL/min



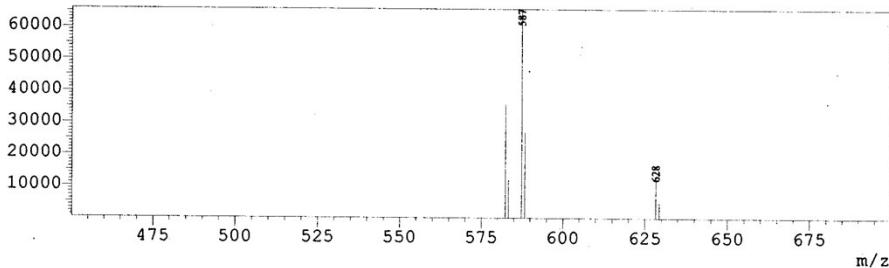
Chromatogram
mAU



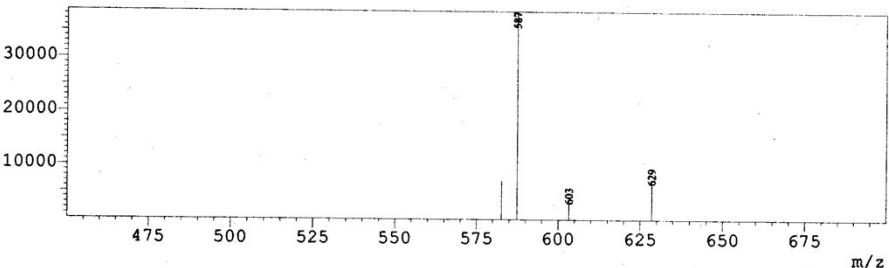
Peak Table

Peak#	Ret. Time	Peak Start	Peak End	Area	Area%
1	3.963	3.659	4.245	5197973	92.271
2	4.356	4.245	4.917	435425	7.729
Total				5633398	100.000

Q1 Scan Positive+
 \$If\$(SpPrTab==SpPrTab) Spectrum Mode:Averaged 3.828-4.105(1532-1643)
 BG Mode:Averaged 1.873-3.810(750-1525)

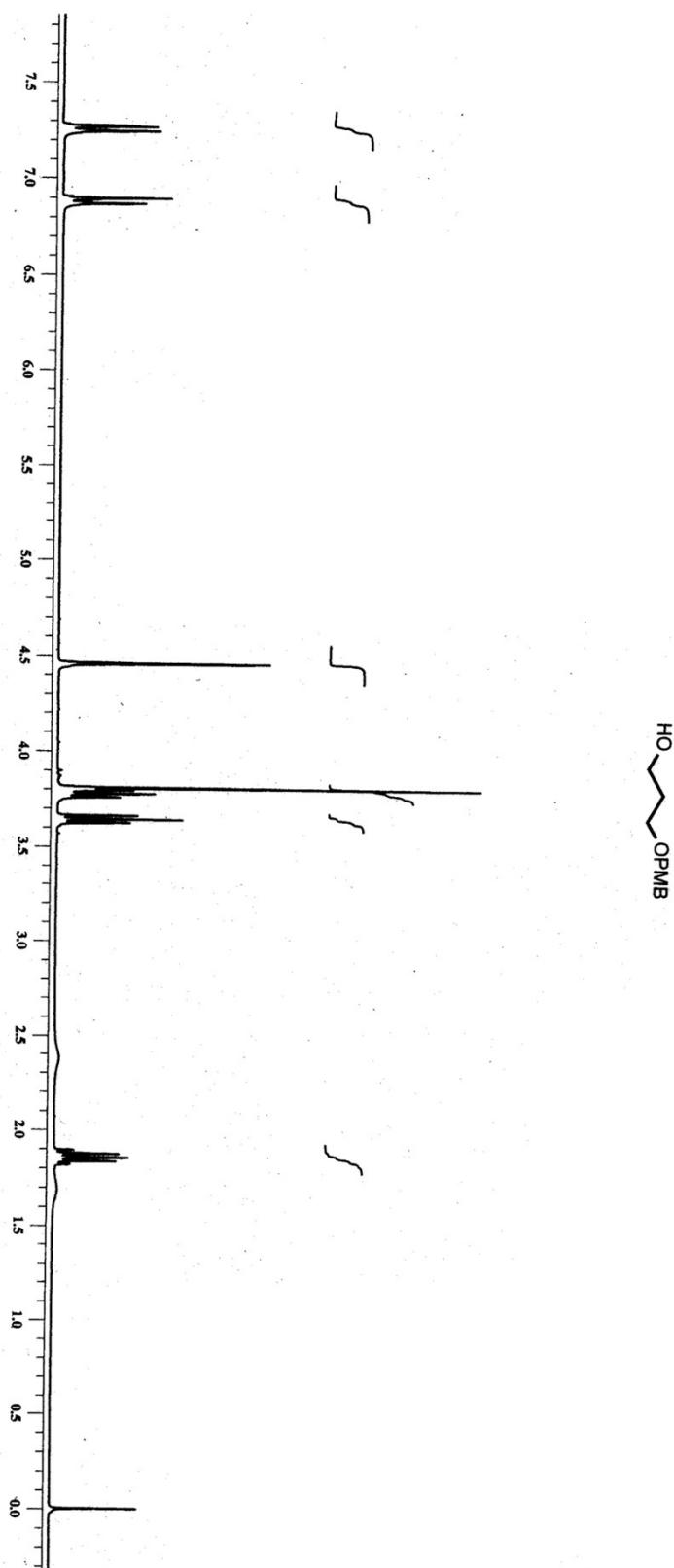


Q1 Scan Positive+
 \$If\$(SpPrTab==SpPrTab) Spectrum Mode:Averaged 4.193-4.418(1678-1768)
 BG Mode:Averaged 1.975-4.140(791-1657)

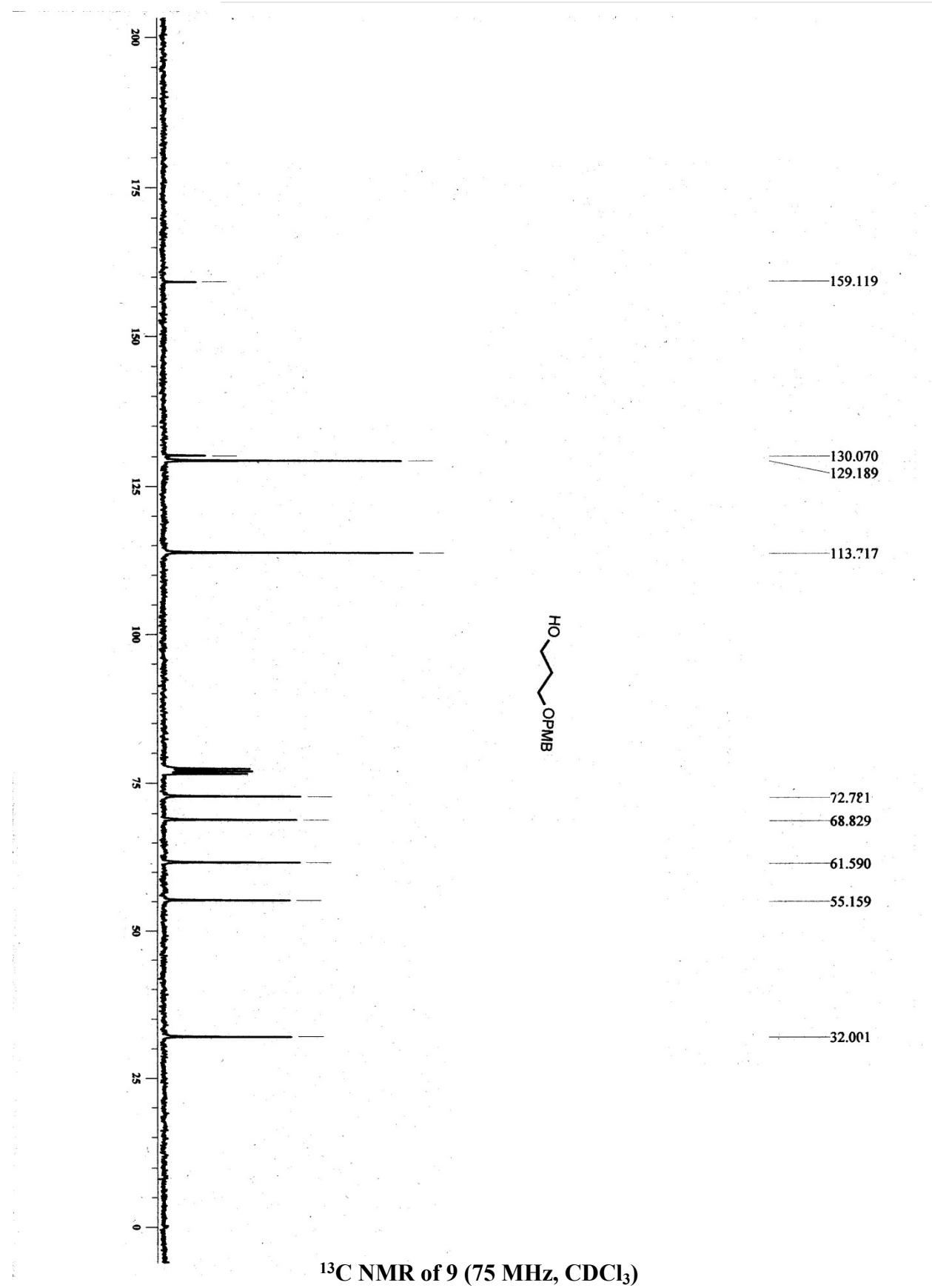


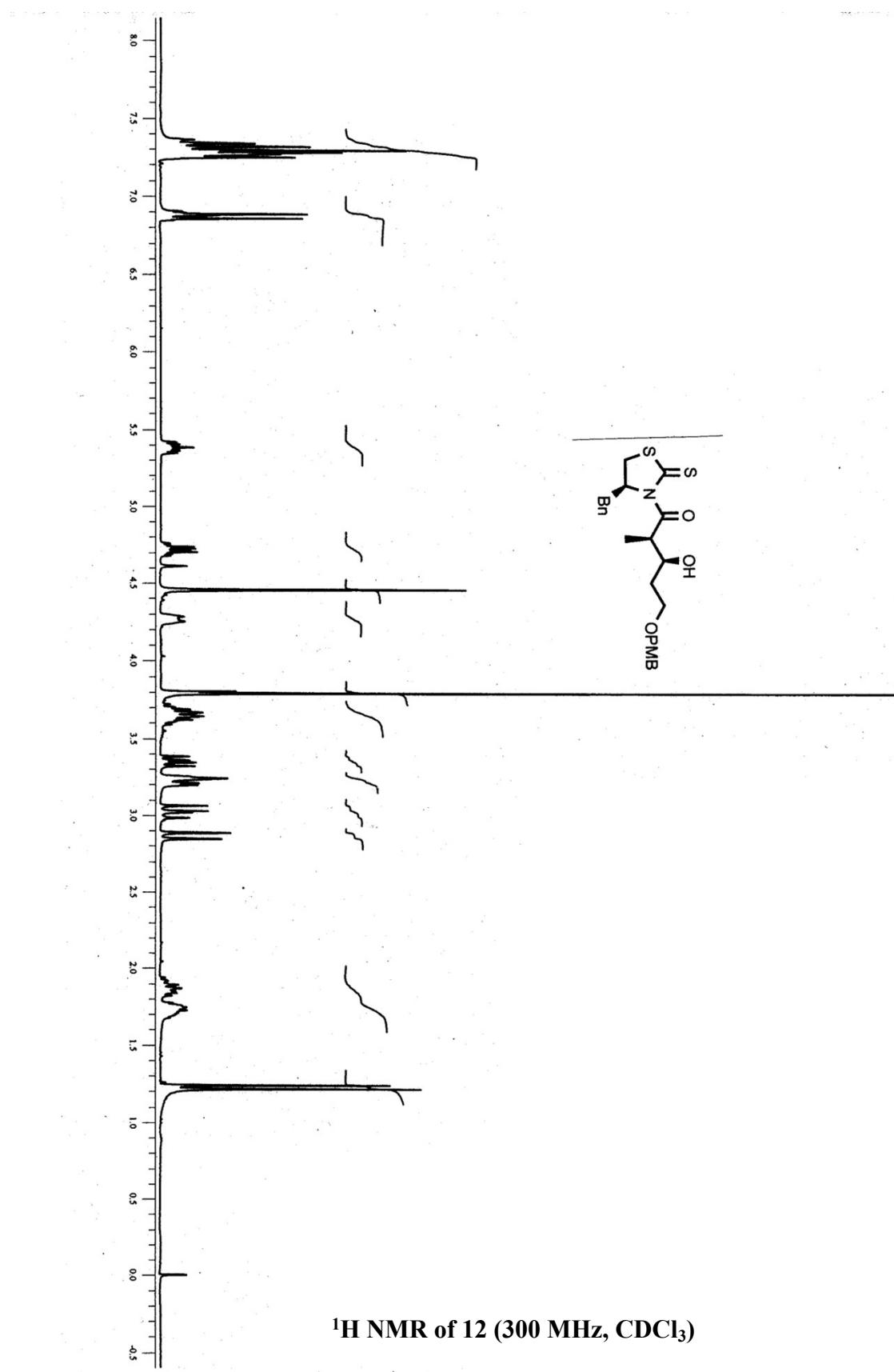
LC-MS REPORT

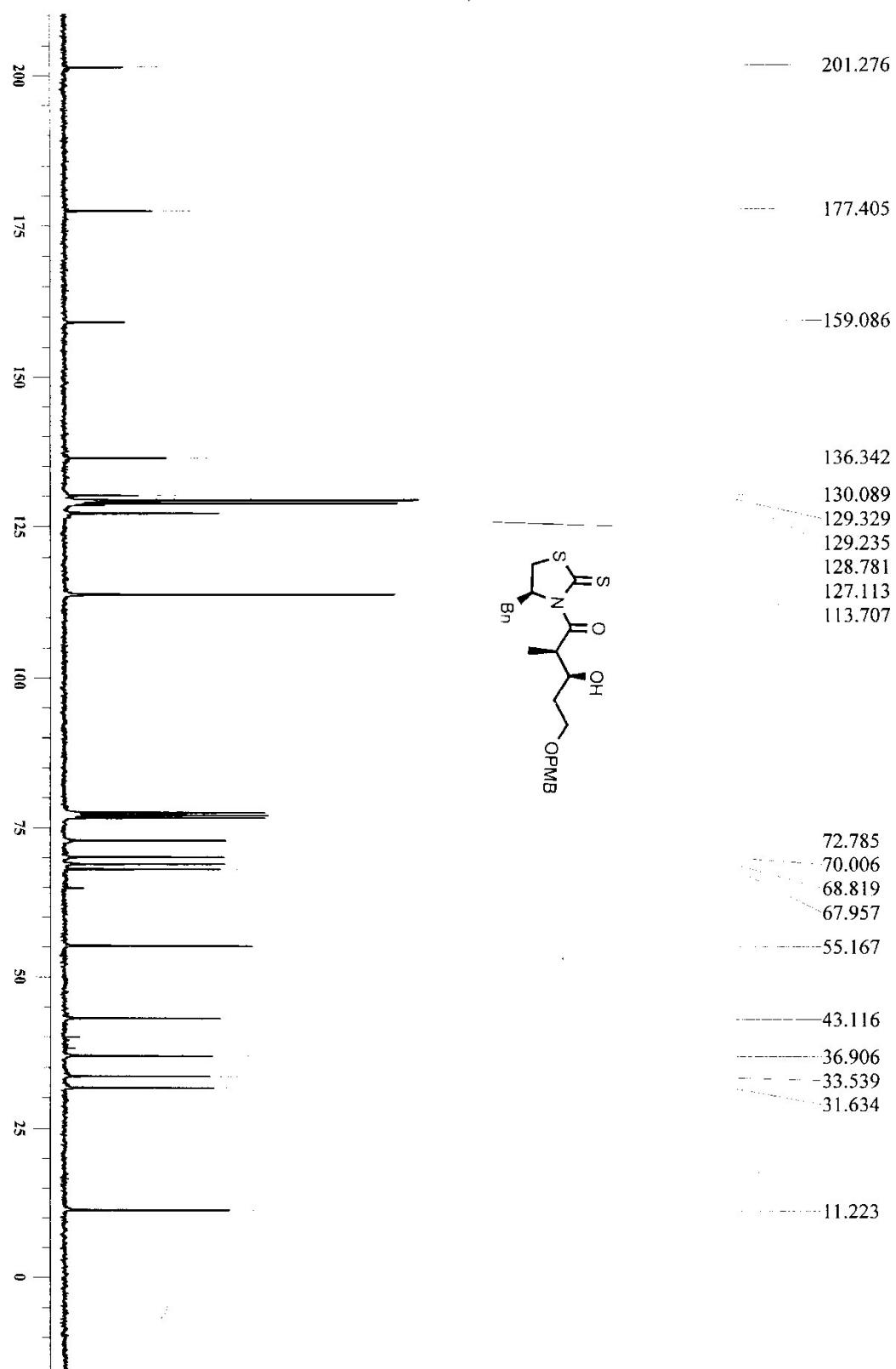
HPLC of compound 2k



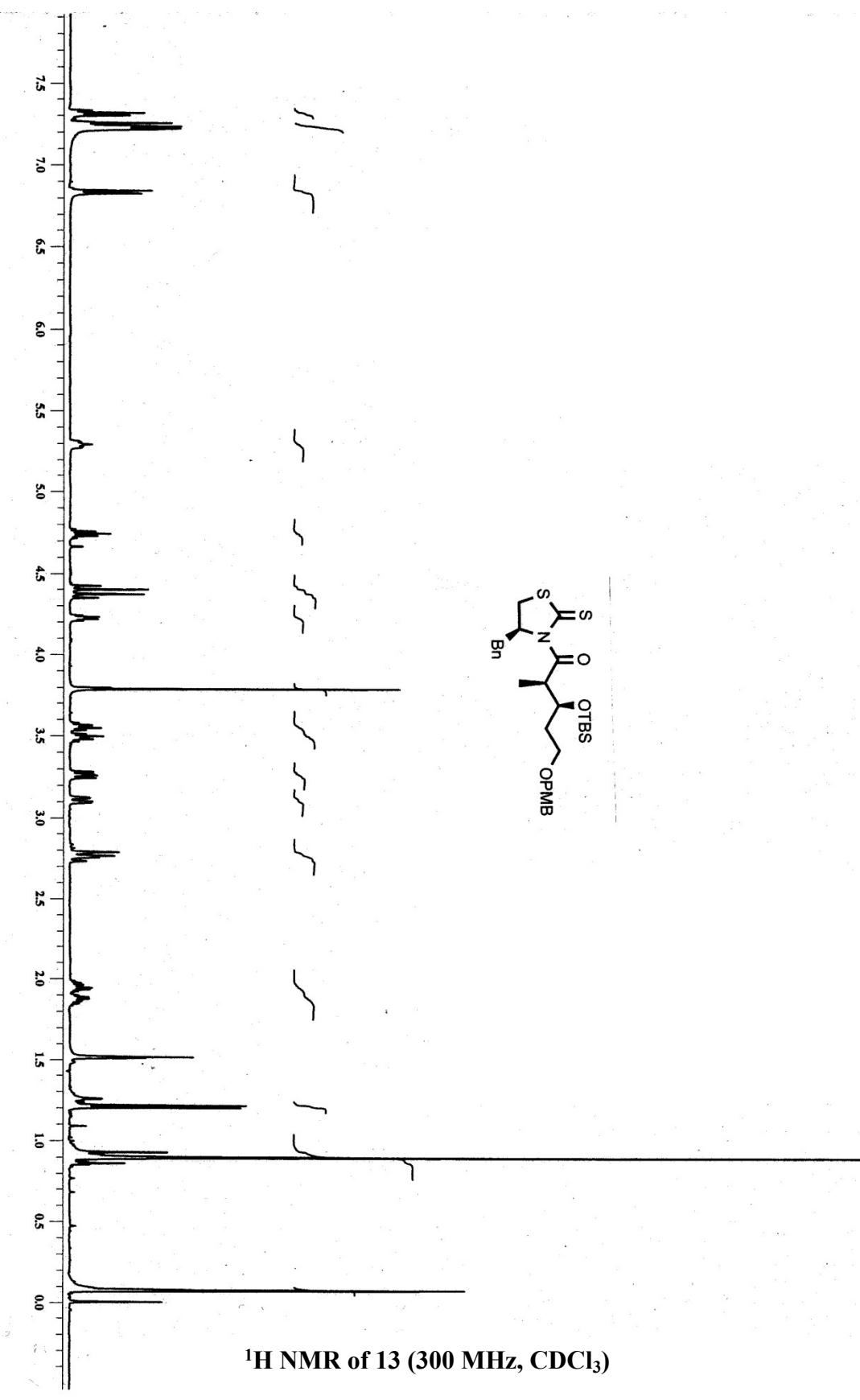
^1H NMR of 9 (300 MHz, CDCl_3)

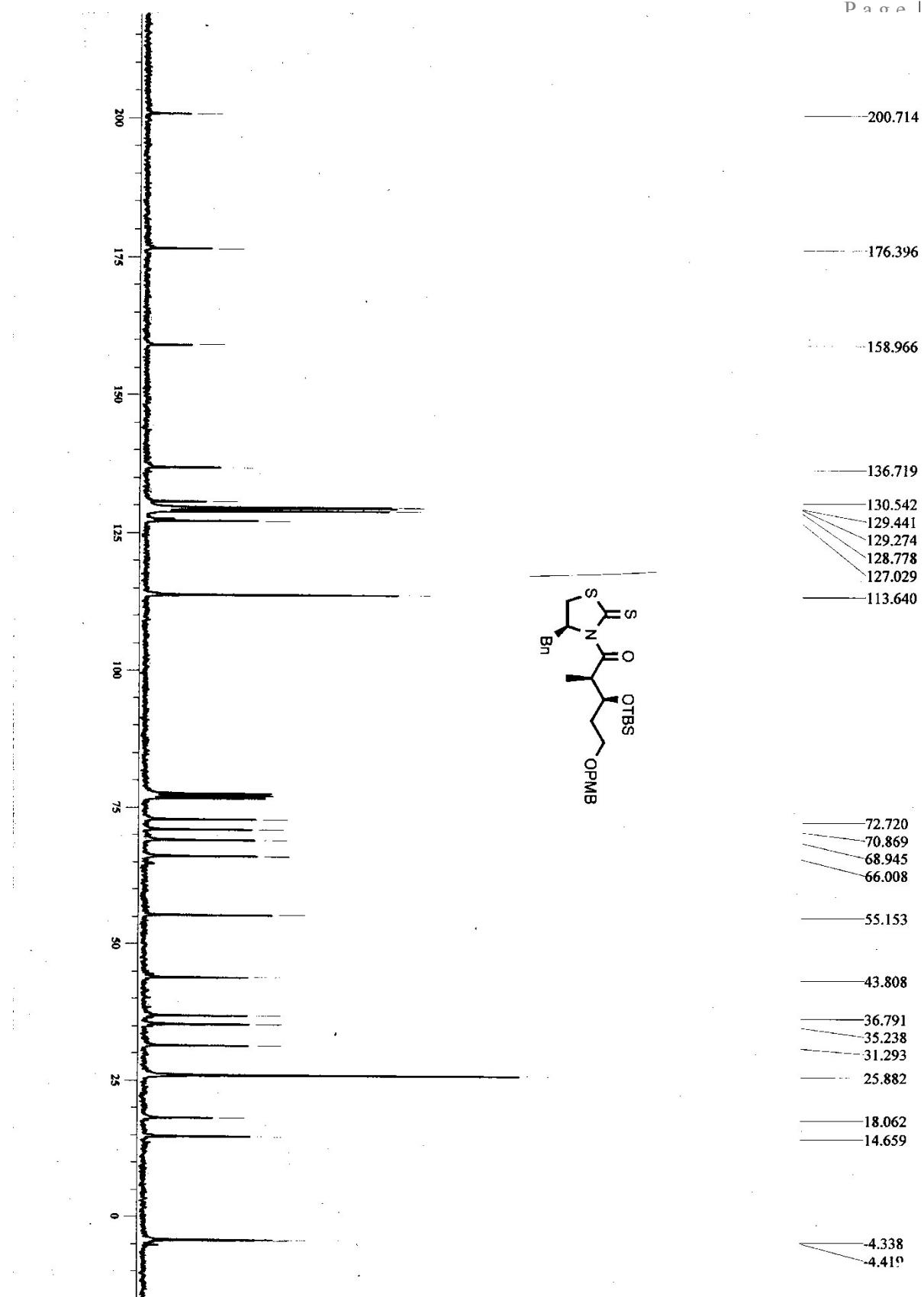




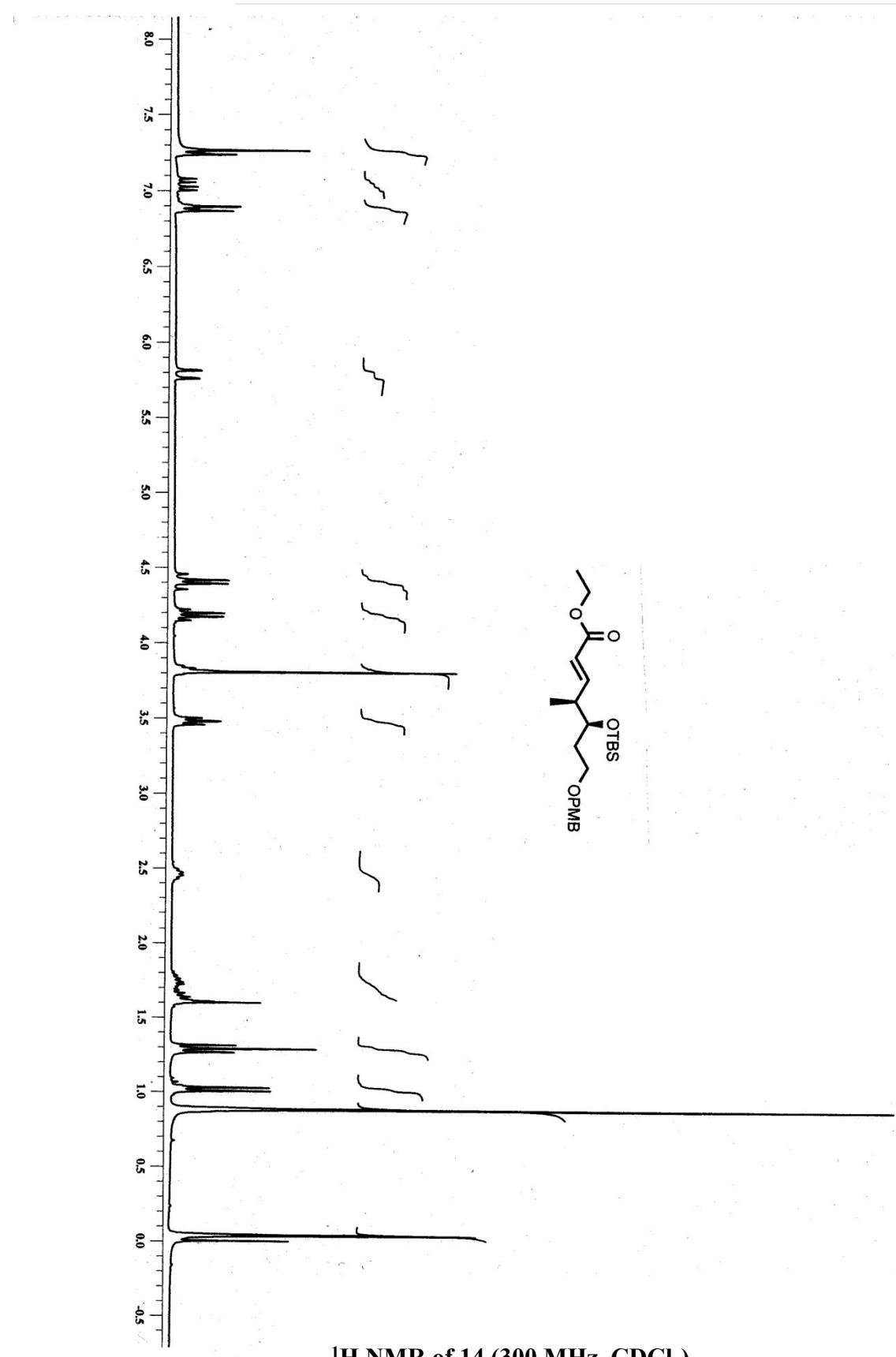


^{13}C NMR of 12 (75 MHz, CDCl_3)

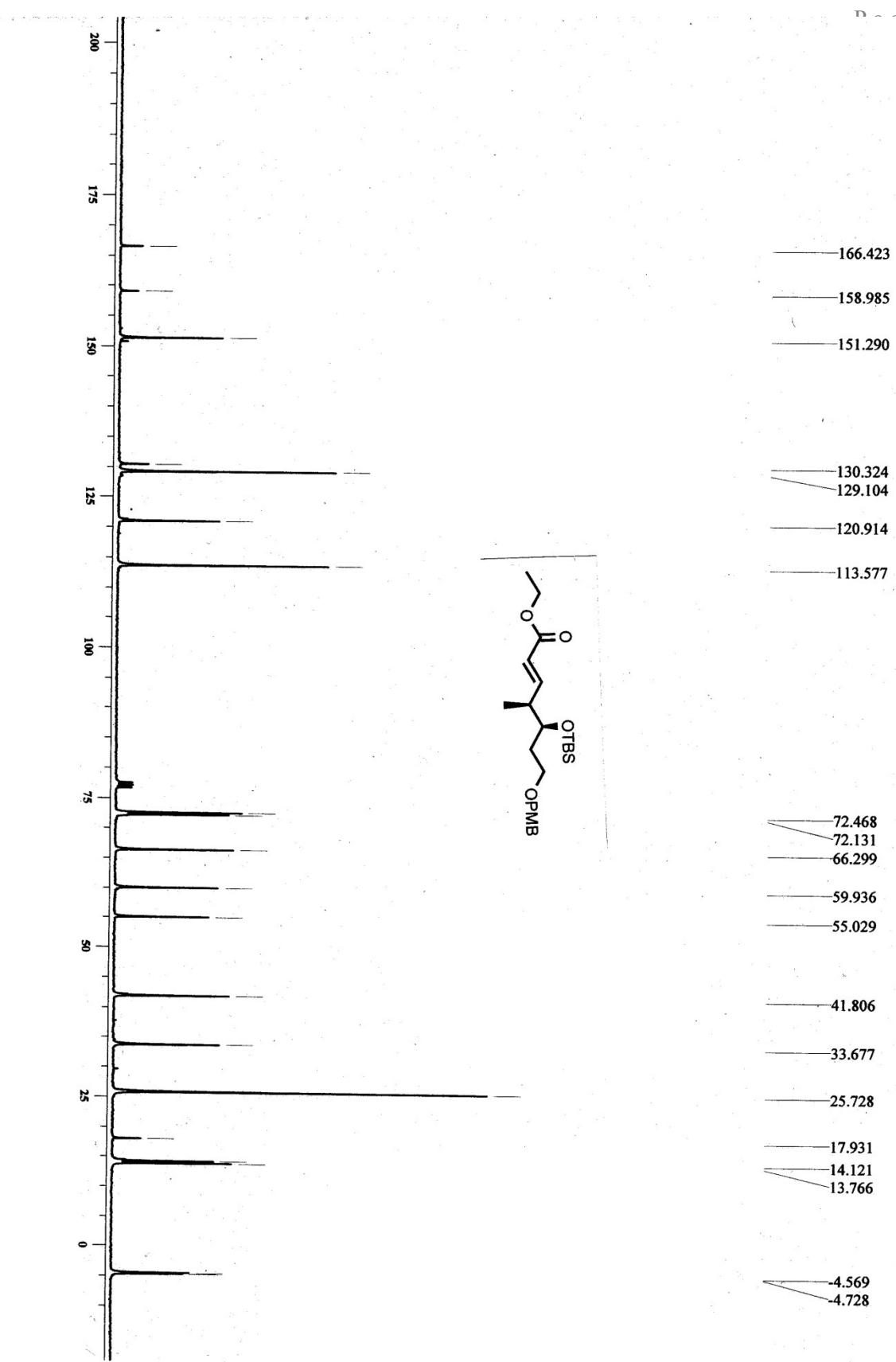


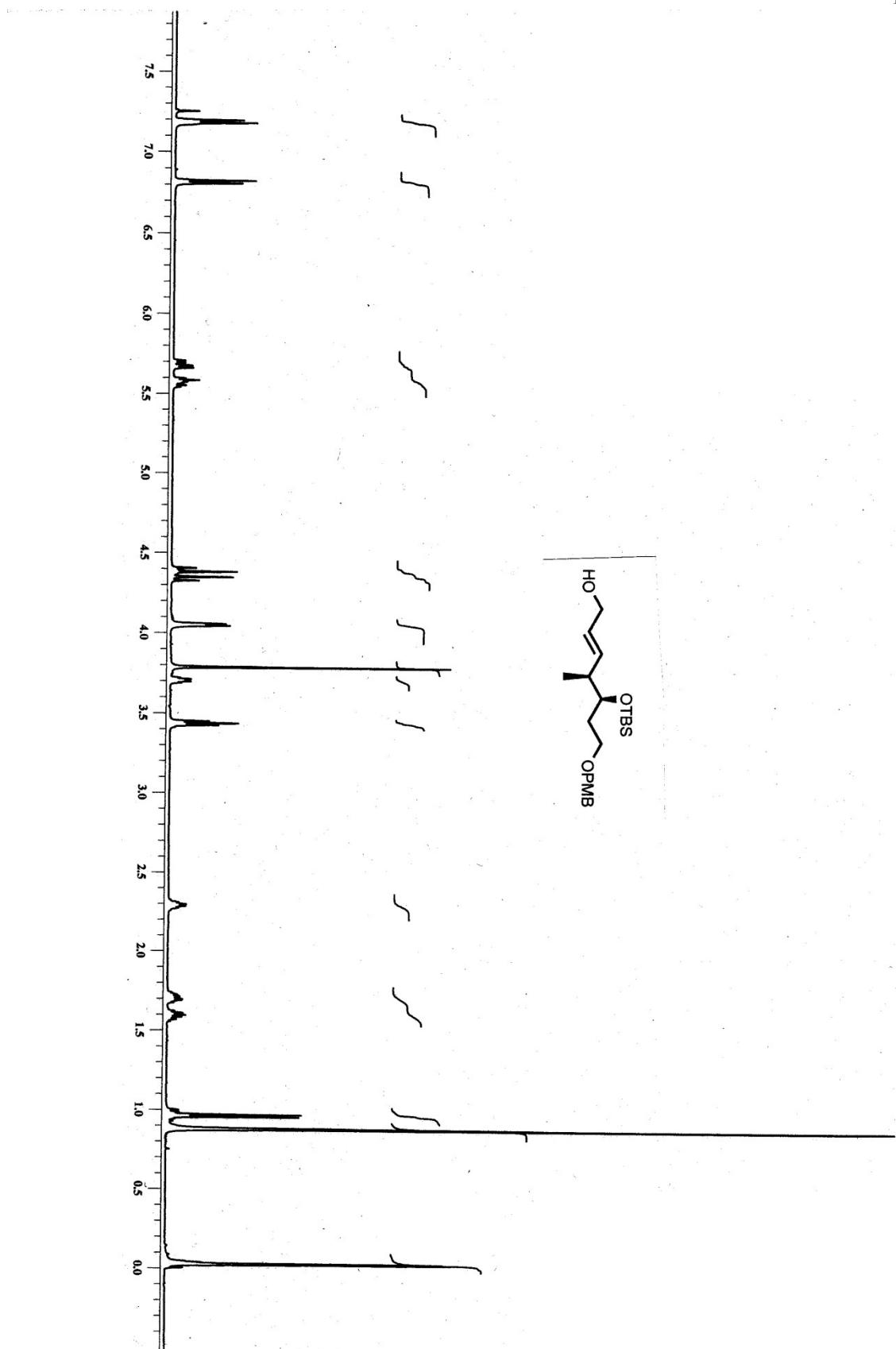


¹³C NMR of 13 (75 MHz, CDCl₃)

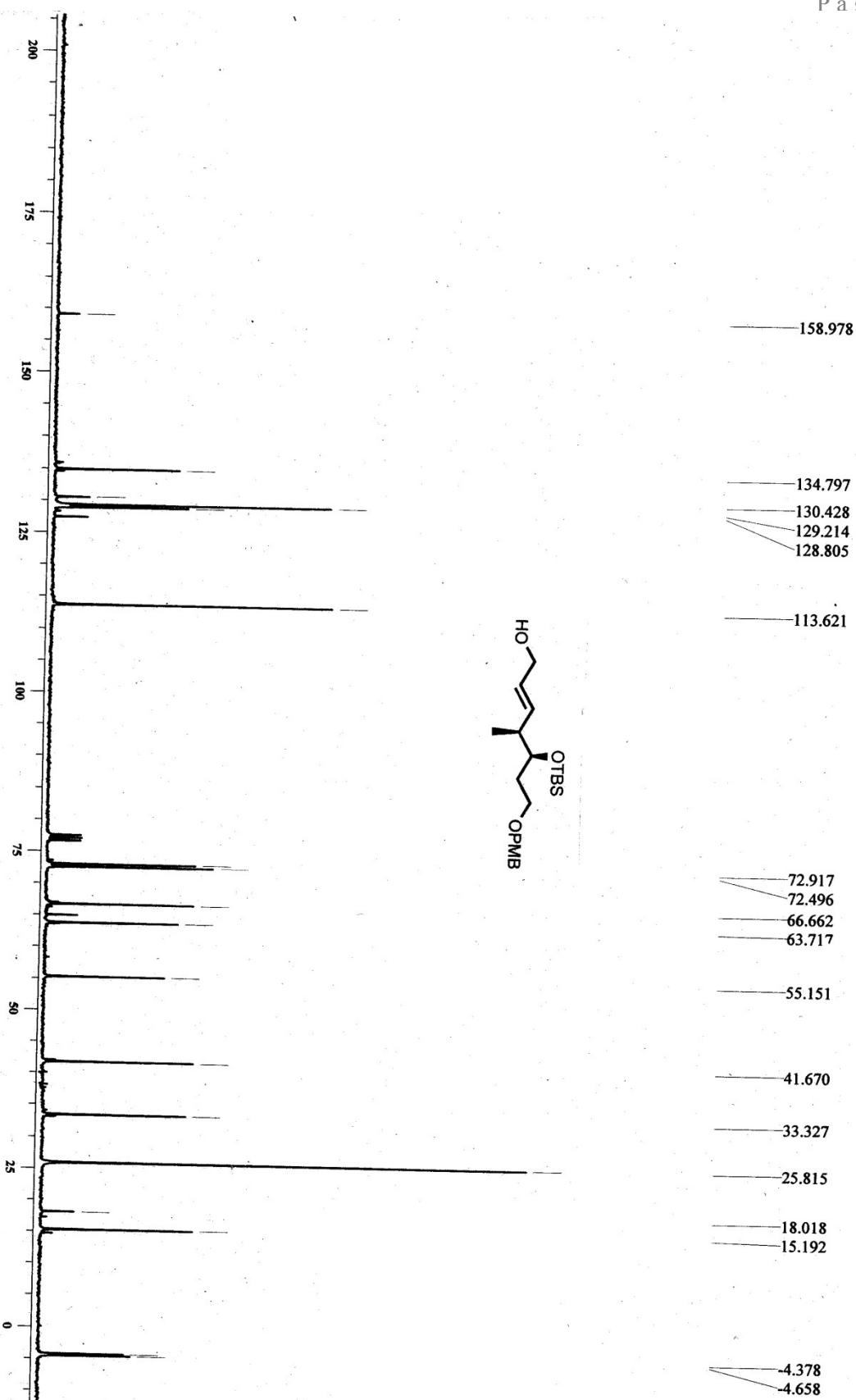


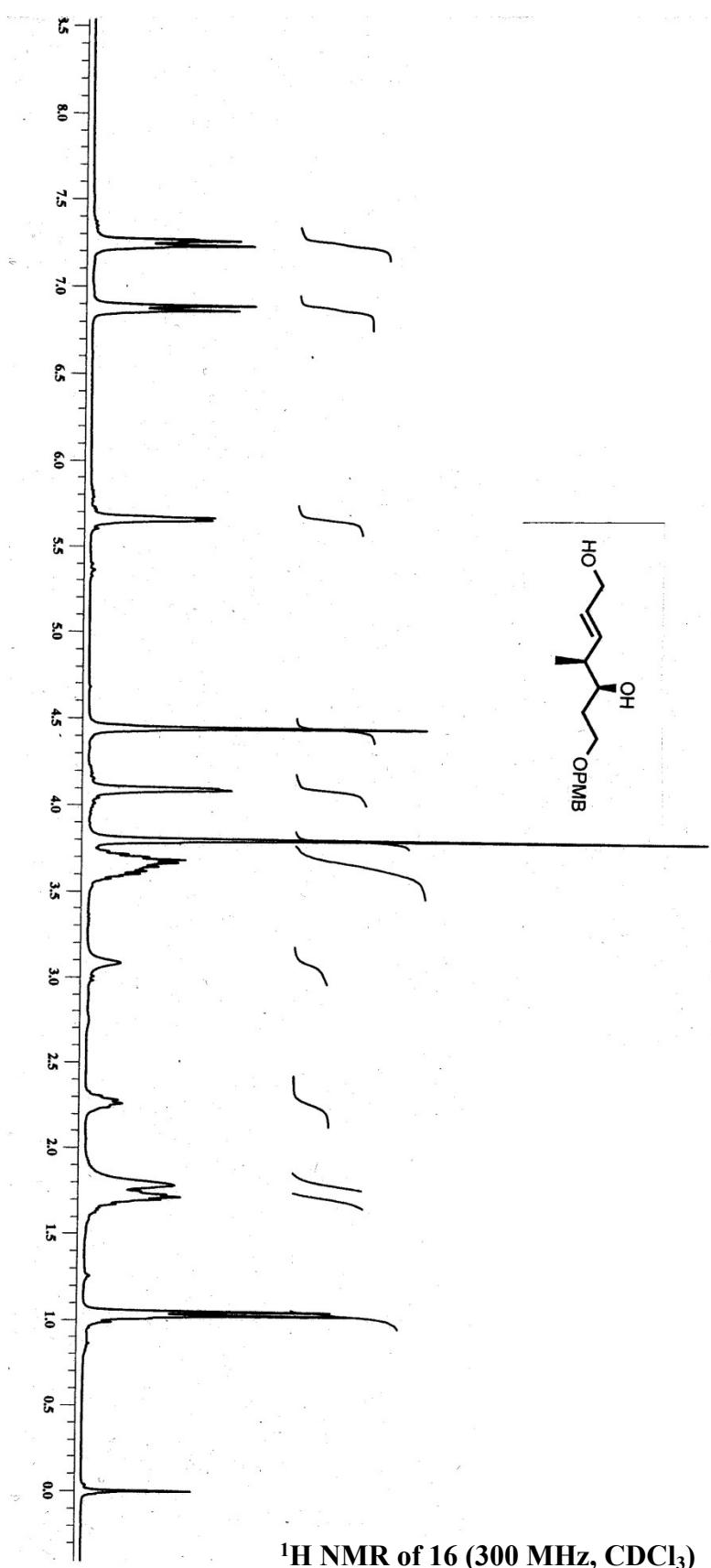
^1H NMR of 14 (300 MHz, CDCl_3)

¹³C NMR of 14 (75 MHz, CDCl₃)

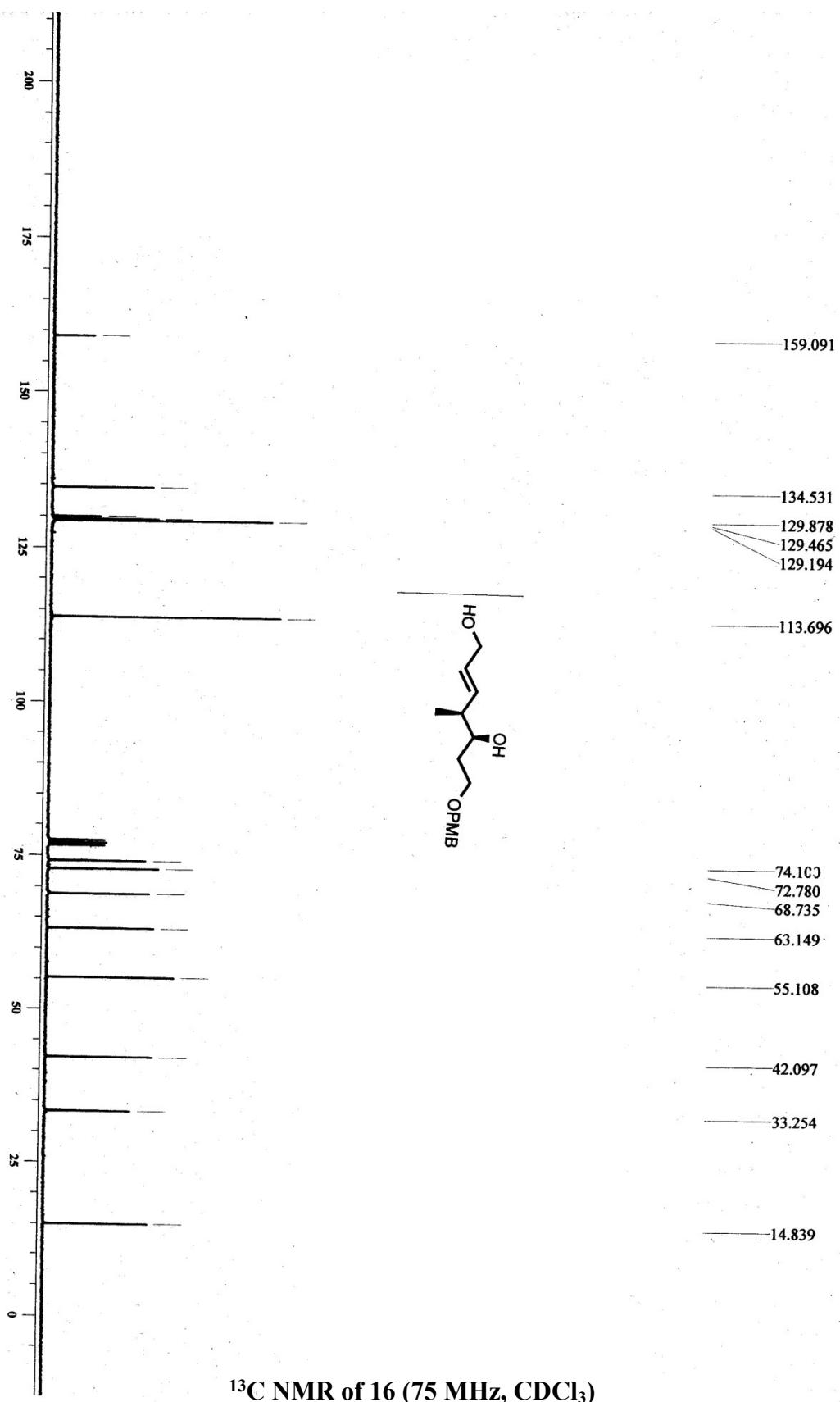


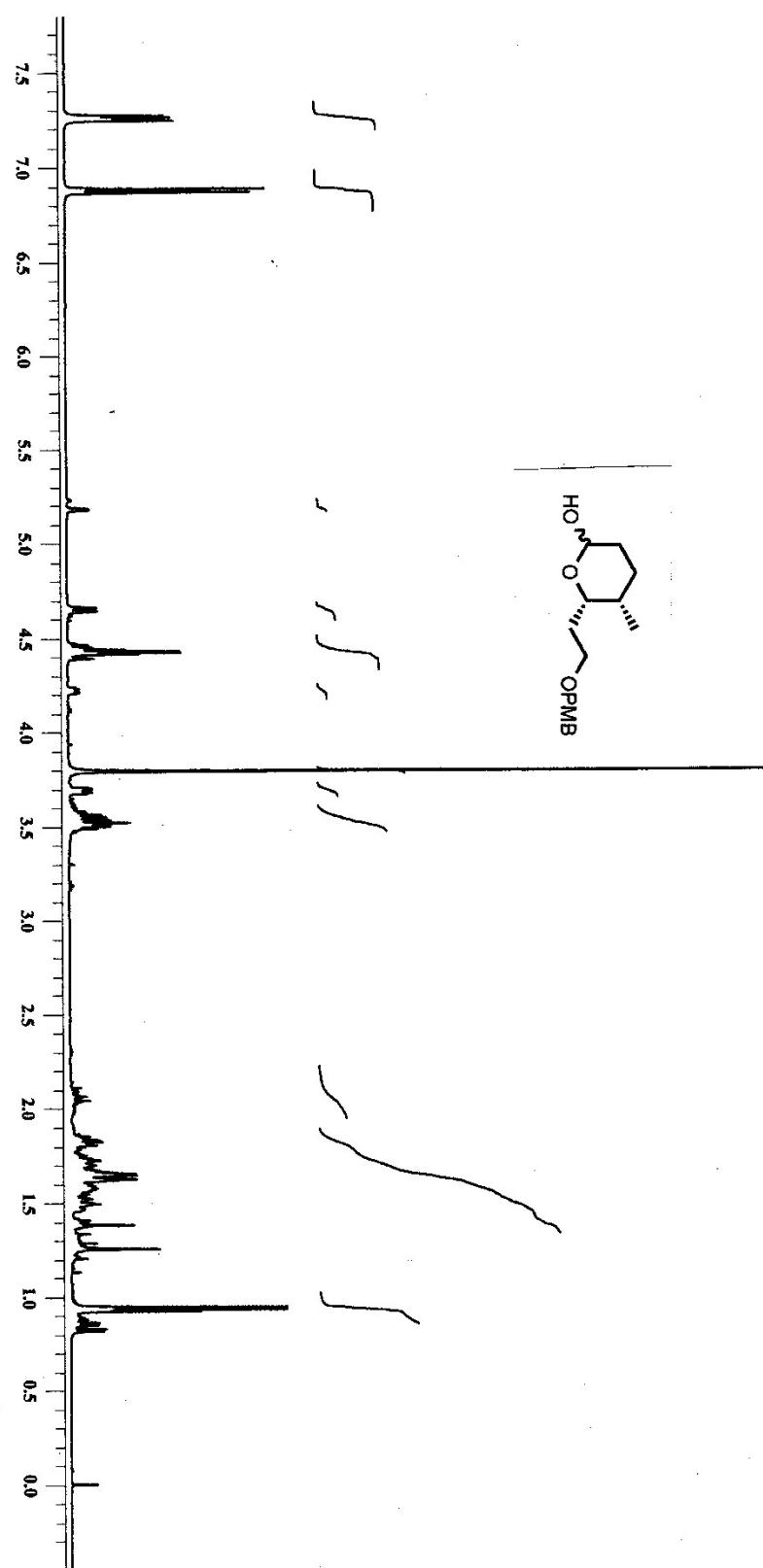
^1H NMR of 15 (300 MHz, CDCl_3)

 ^{13}C NMR of 15 (75 MHz, CDCl_3)

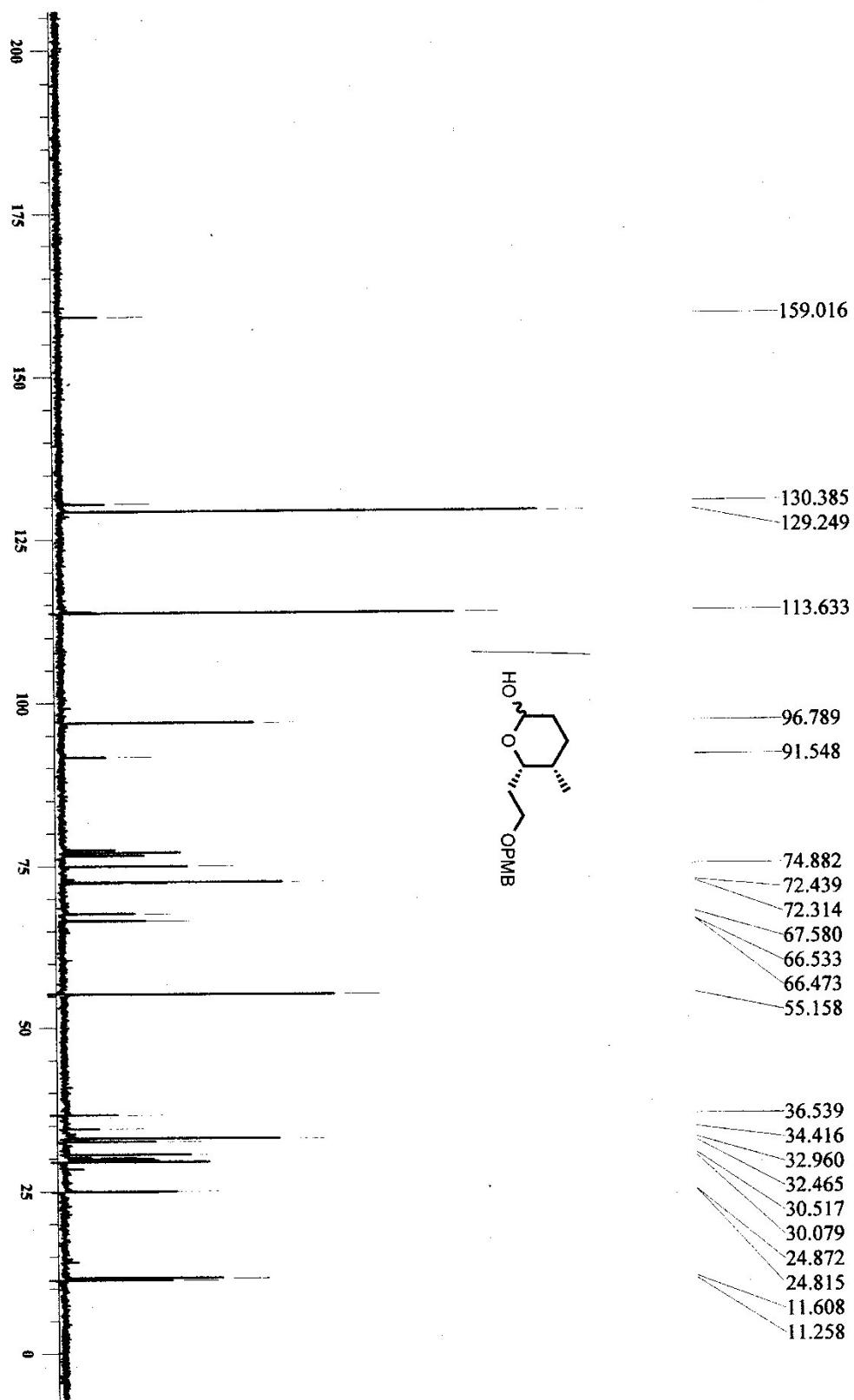


^1H NMR of 16 (300 MHz, CDCl_3)

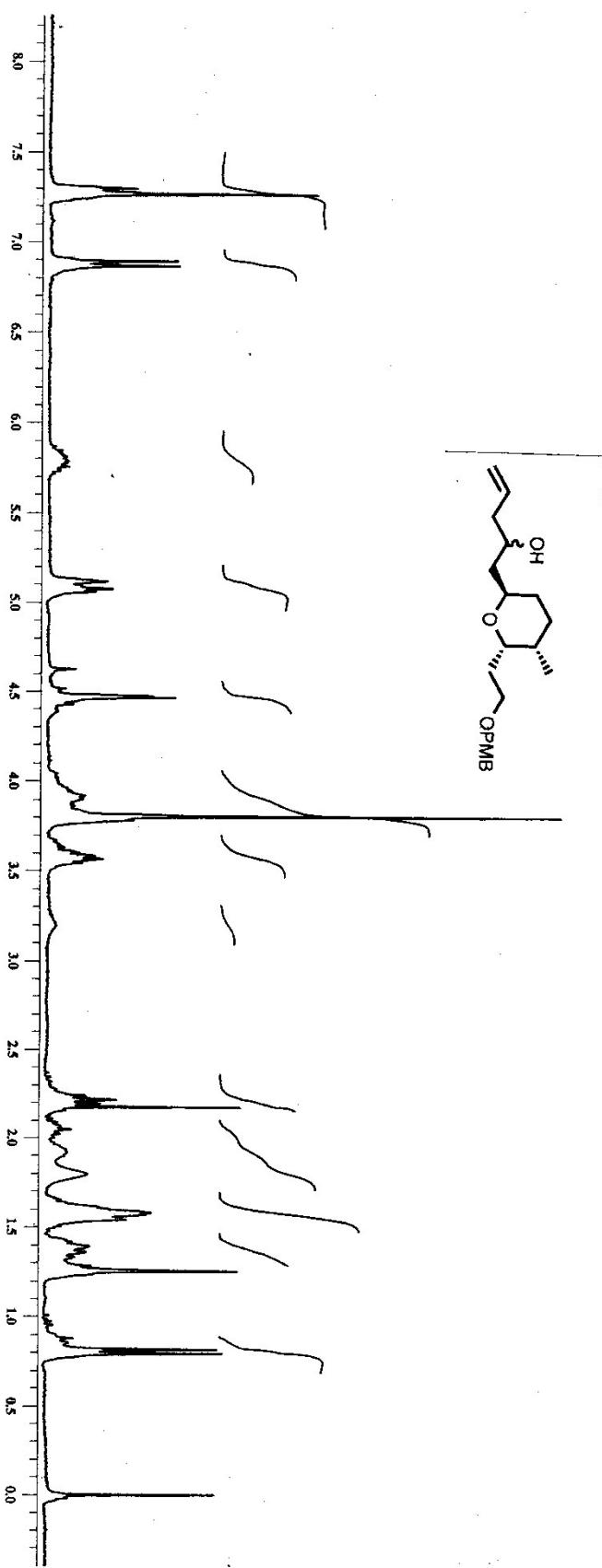




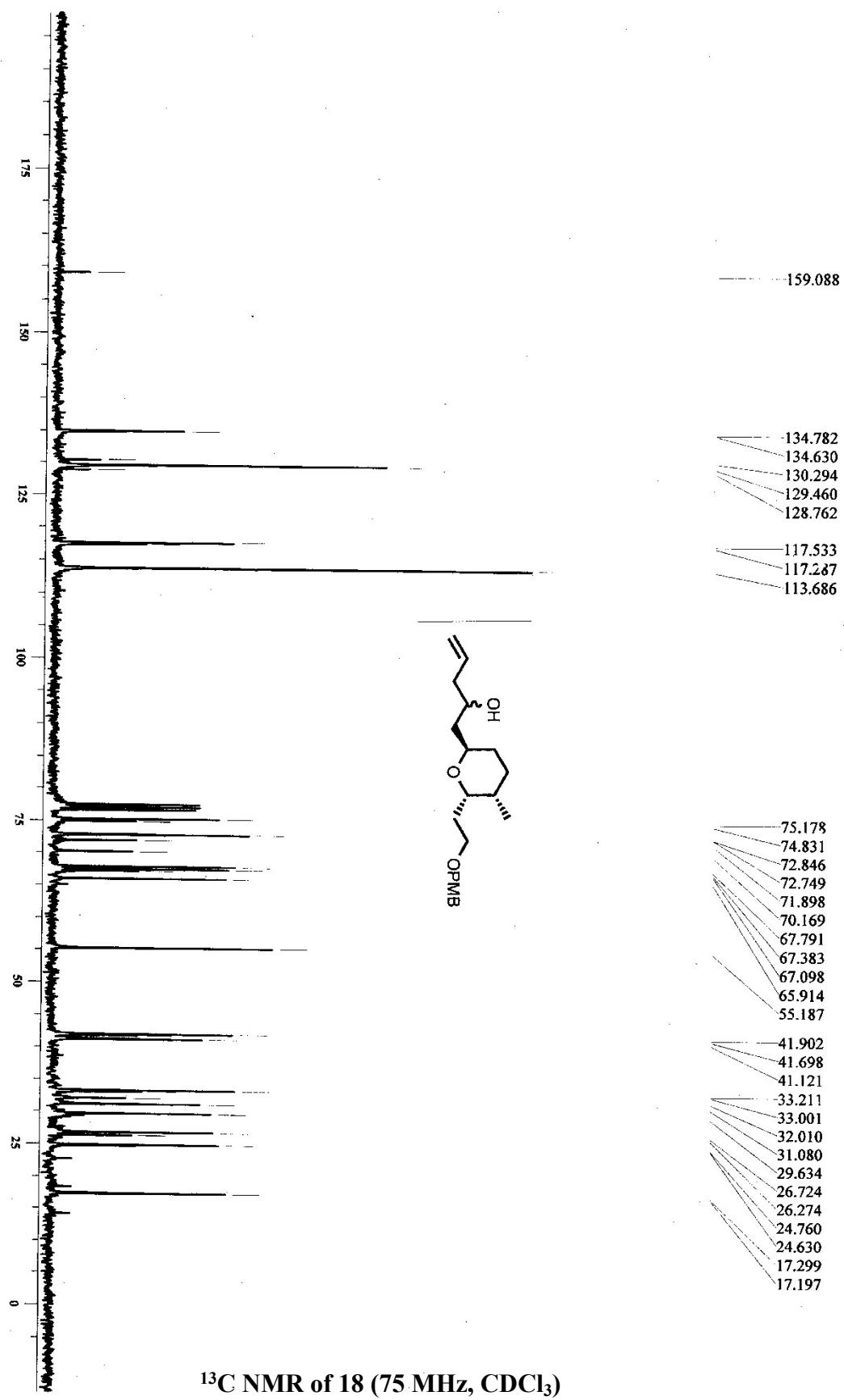
^1H NMR of 1g (300 MHz, CDCl_3)

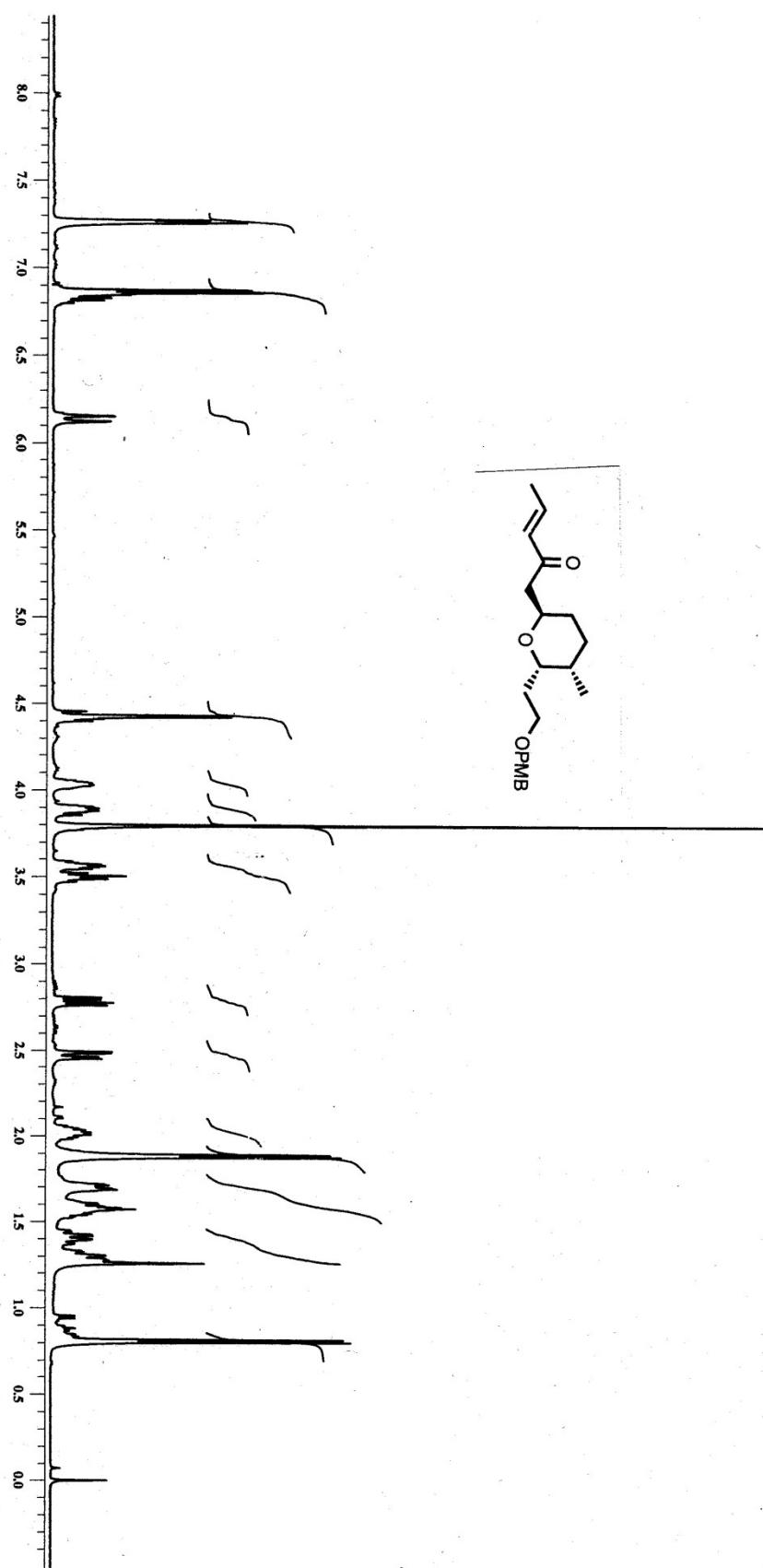


^{13}C NMR of **1g** (75 MHz, CDCl_3)

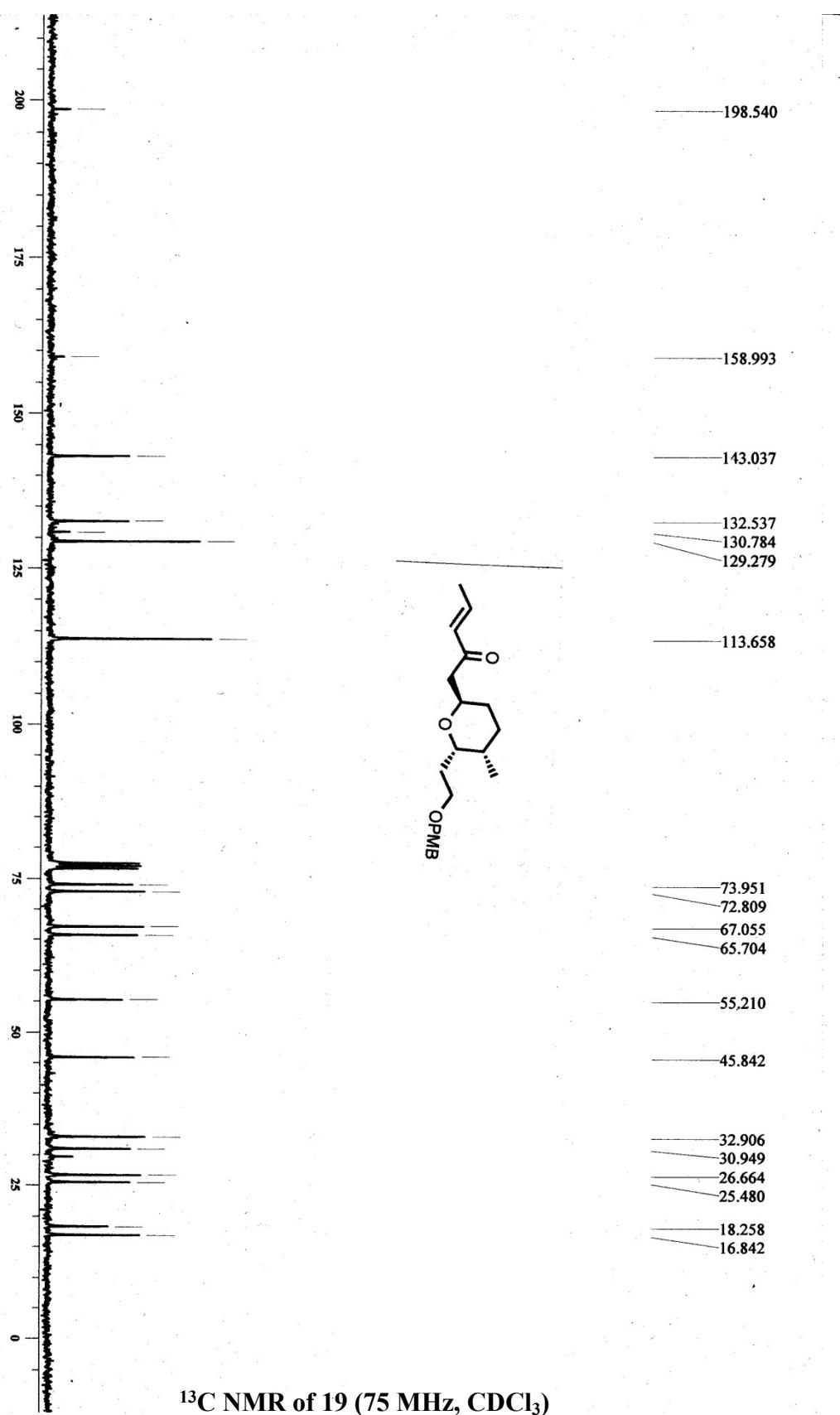


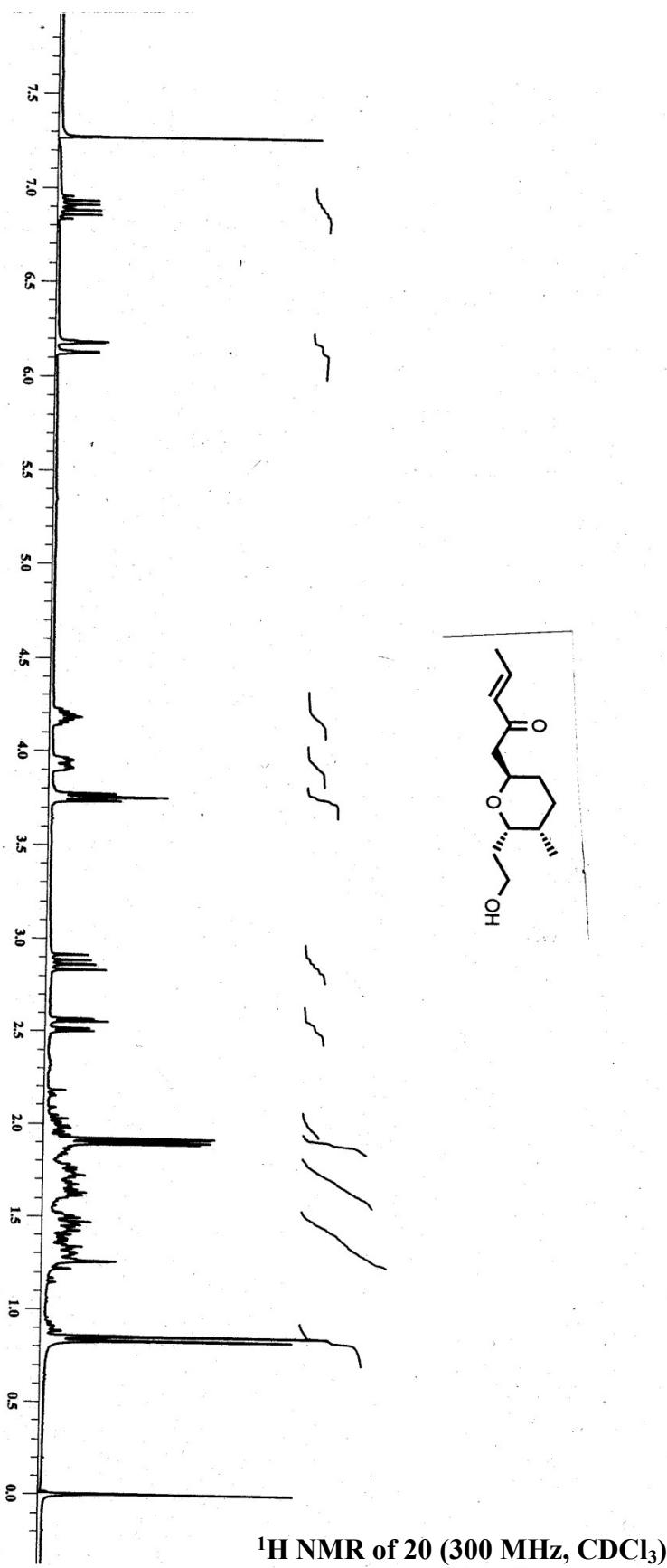
^1H NMR of 18 (300 MHz, CDCl_3)

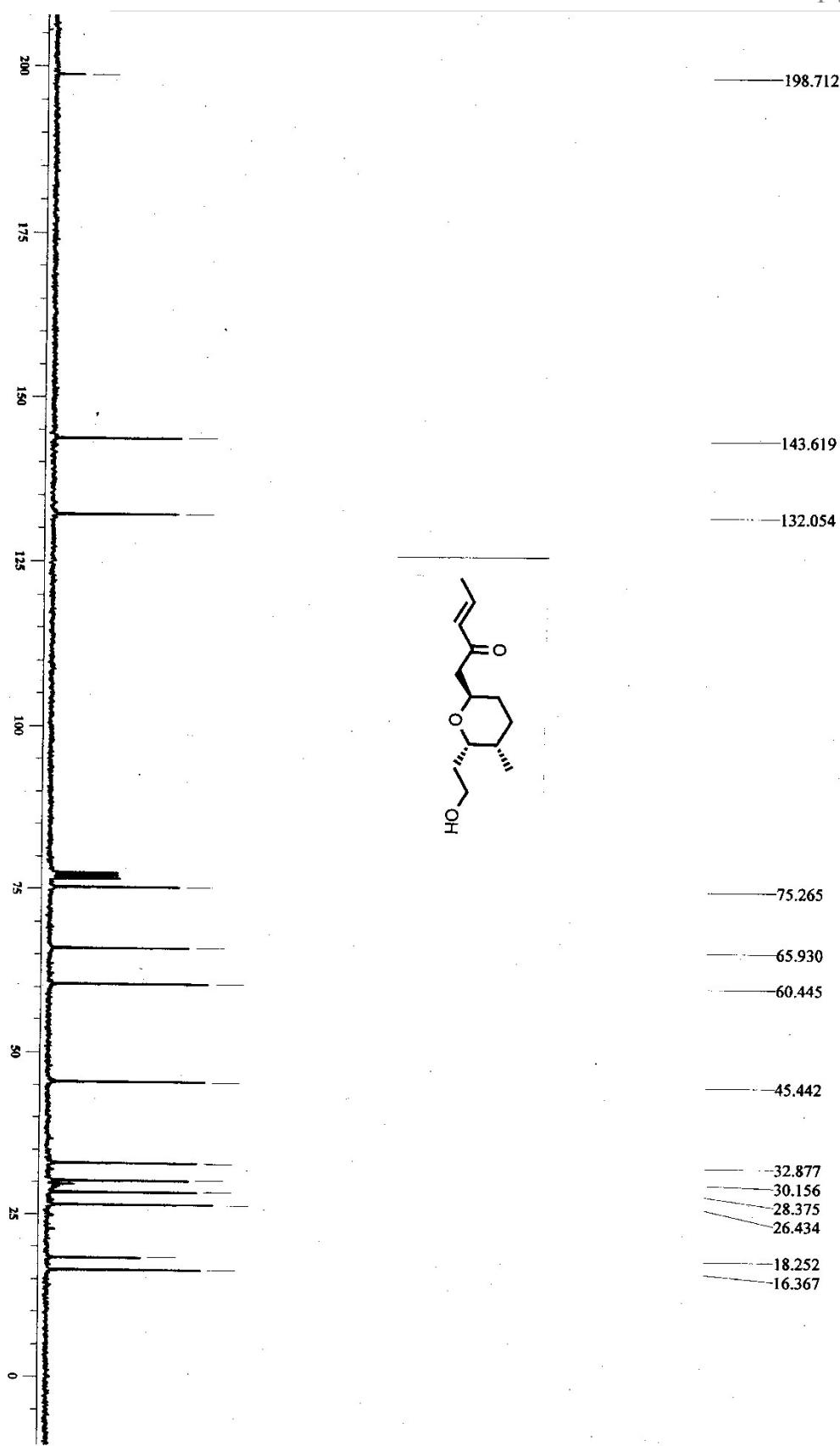




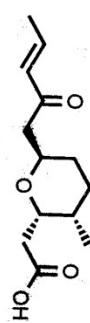
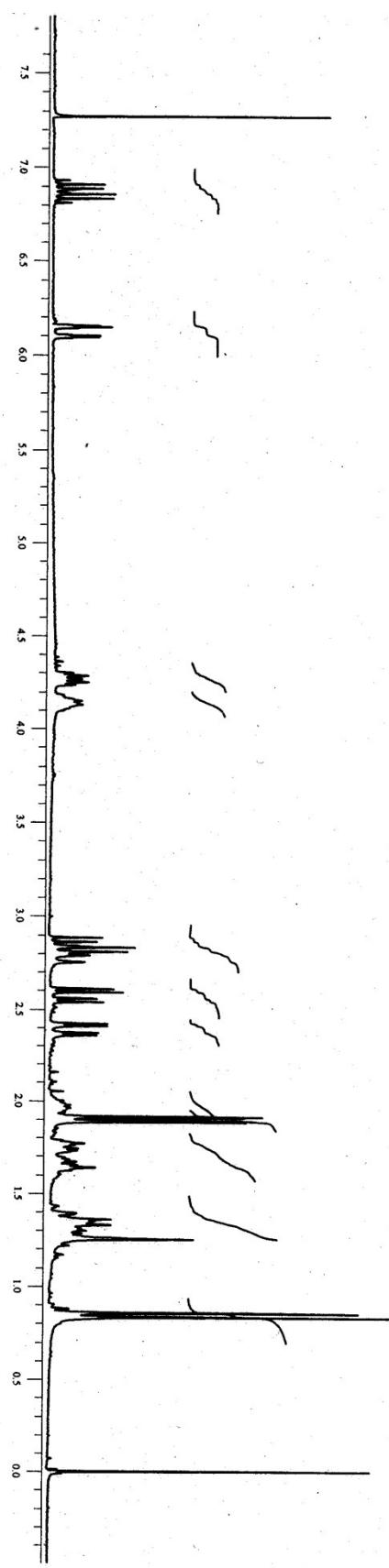
^1H NMR of 19 (500 MHz, CDCl_3)



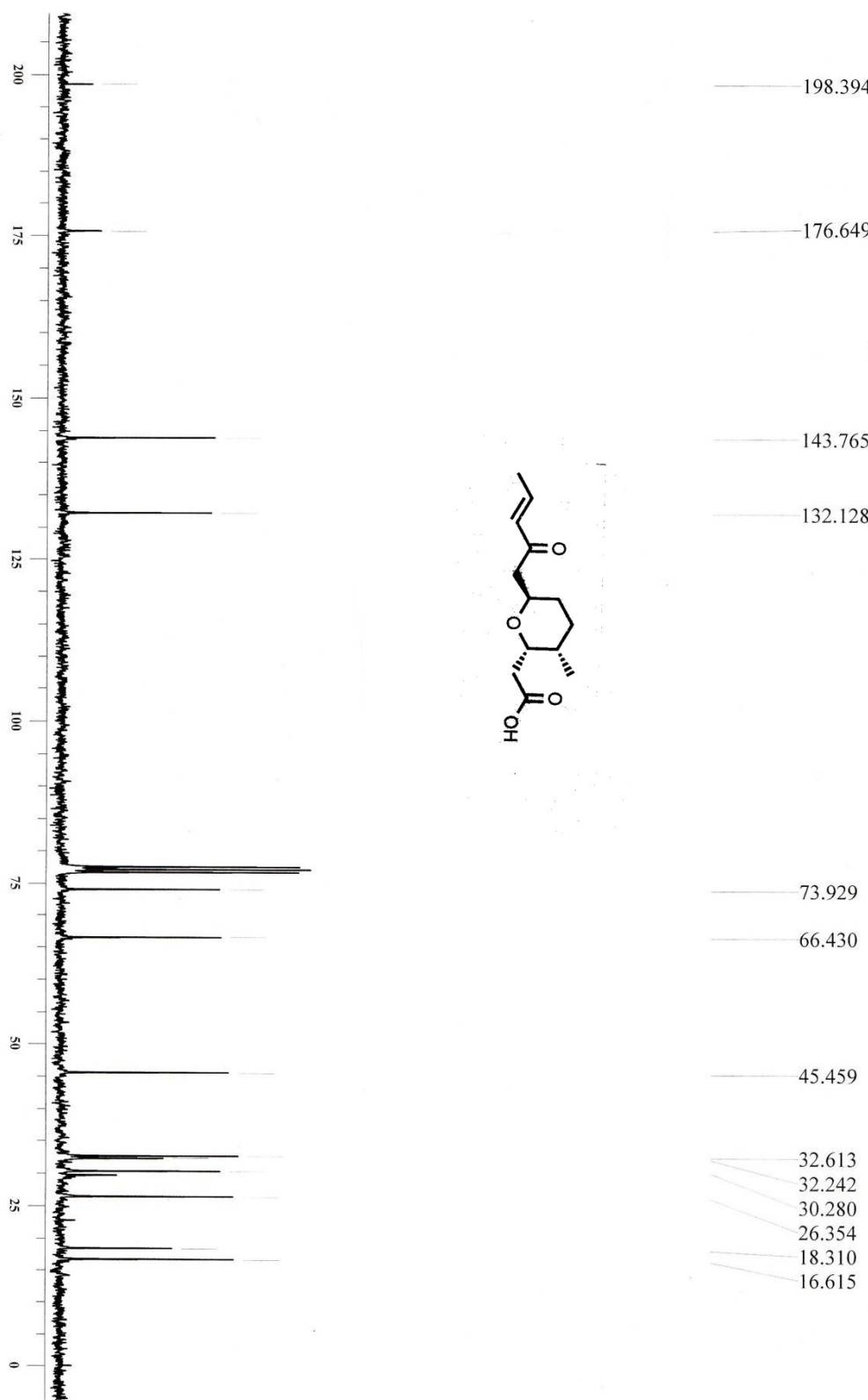




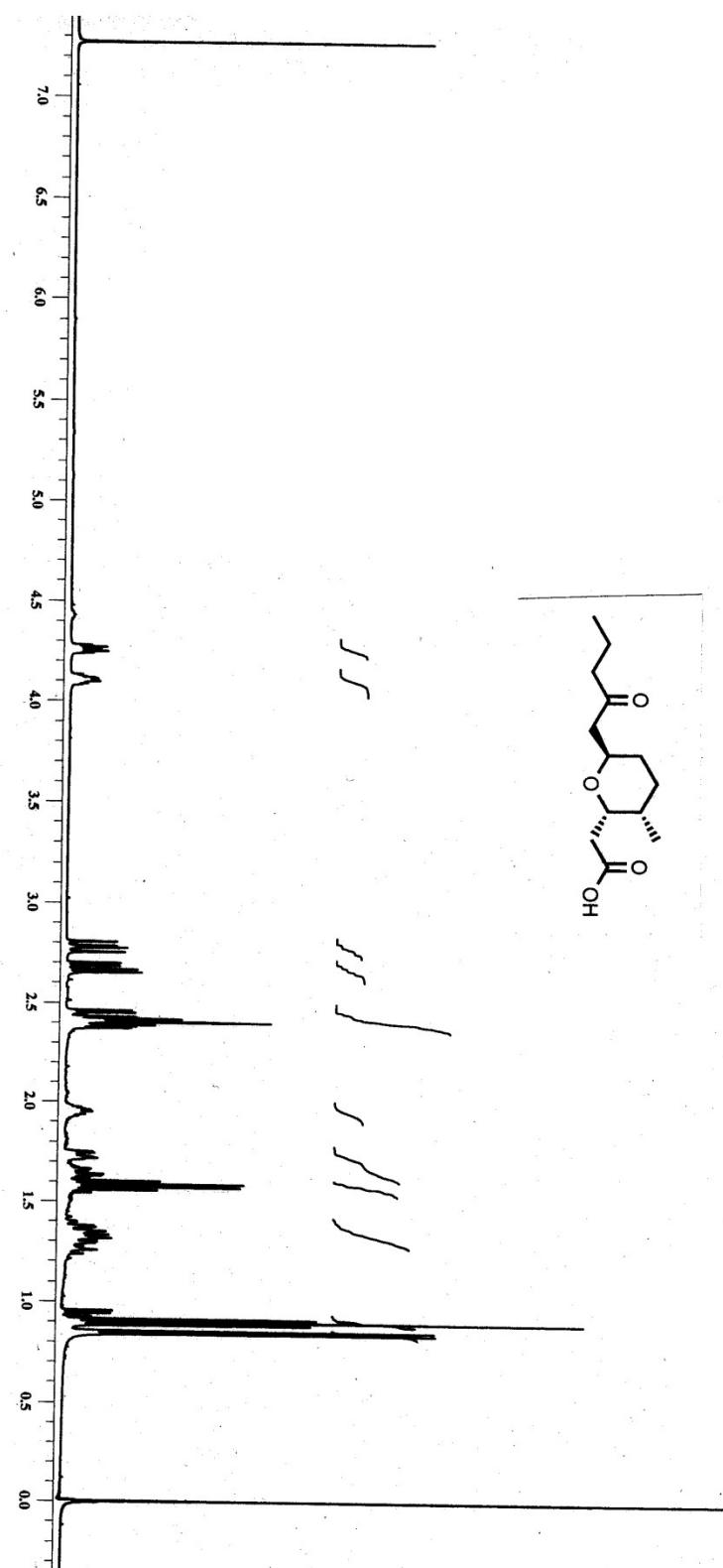
^{13}C NMR of 20 (75 MHz, CDCl_3)



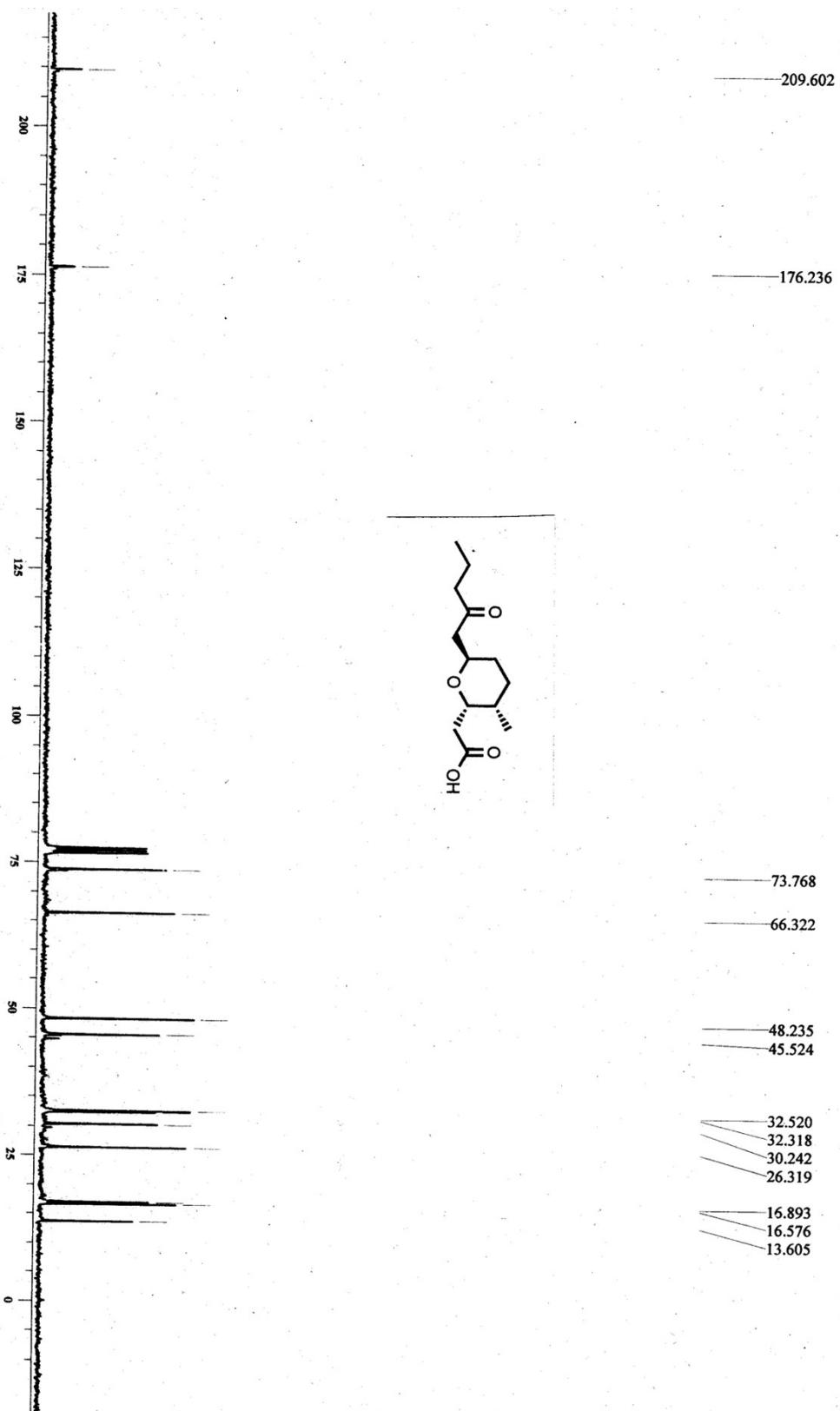
^1H NMR of 21 (300 MHz, CDCl_3)



¹H NMR of 21 (300 MHz, CDCl₃)



^1H NMR of 22 (500 MHz, CDCl_3)



^{13}C NMR of 22 (75 MHz, CDCl_3)