

### *Supporting Information*

#### **Synthesis of the C45-C53 tetrahydropyran domain of Norhalichondrins and C14-C22 tetrahydrofuran domain of Halichondrin Family**

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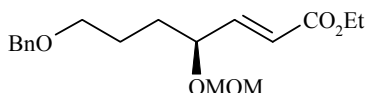
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## 1. General information

Unless otherwise mentioned, all reactions were carried out using standard syringe, septa and cannula techniques. All glassware was flame/oven-dried and cooled under an inert atmosphere of nitrogen unless otherwise stated. Column chromatography was performed using silica gel (60-120 mesh) and the column was usually eluted with ethyl acetate-Hexanes. The diastereomeric excess of the products were measured by chiral-phase HPLC using Chiralpak AS column. Analytical thin layer chromatography (TLC) was performed on precoated silica gel-60 F254 (0.5 mm) glass plates. Visualization of the spots on TLC plates was achieved either by exposure to iodine vapor or UV light or by dipping the plates to sulphuric acid- $\beta$ -naphthol or to ethanolic anisaldehyde-sulphuric acid-acetic acid and heating the plates at 120 °C.  $^1\text{H}$  NMR spectra were recorded at 300, 500, 600 MHz &  $^{13}\text{C}$  NMR spectra were recorded at 75, 125 MHz in  $\text{CDCl}_3$  using Tetramethylsilane as the reference standard. s, brs, d, dd, ddd, dt, t, q, qt, and m refer to singlet, broad singlet, doublet, doublet of doublet, doublet of doublet of doublet, doublet of triplet, triplet, quartet, quintet and multiplet respectively unless otherwise mentioned. Infrared spectra were recorded on Perkin-Elmer Infrared-683 spectrophotometer with NaCl optics. Spectra were calibrated against the polystyrene absorption at  $1610\text{ cm}^{-1}$ . Samples were scanned neat. The optical rotations were measured on JASCO DIP-360 Digital Polarimeter. Mass spectra were recorded on Micro Mass VG-7070H mass spectrometer for ESI and EI are given in mass units ( $m/z$ ). High-resolution mass spectra (HRMS) [ESI+] were obtained using either a TOF or a double focusing spectrometer.

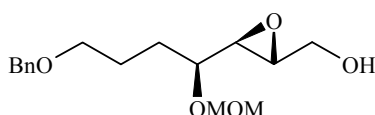
## 2. Physical and spectroscopic data of the products

### (*S,E*)-ethyl 7-(benzyloxy)-4-(methoxymethoxy)hept-2-enoate (7):



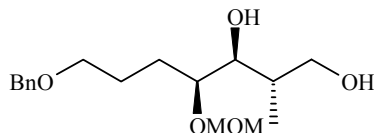
$[\alpha]_{\text{D}}^{25} = -40.4$  ( $c = 1.0$ ,  $\text{CHCl}_3$ ); IR (Neat): 2943, 1720, 1656, 1270, 1099, 1034  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  7.32-7.22 (m, 5H), 6.76 (dd,  $J = 5.9, 15.8$  Hz, 1H), 5.93 (d,  $J = 15.8$  Hz, 1H), 4.59 (d,  $J = 6.9$  Hz, 1H), 4.53 (d,  $J = 6.9$  Hz, 1H), 4.46 (s, 2H), 4.21-4.19 (m, 1H), 4.18 (q,  $J = 6.9$ , 2H), 3.48-3.43 (m, 2H), 3.33 (s, 3H), 1.75-1.63 (m, 4H), 1.30 (t,  $J = 6.9$  Hz, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta$  166.1, 147.5, 138.3, 128.2, 127.5, 127.4, 121.9, 94.5, 74.8, 72.8, 69.8, 60.3, 55.5, 31.4, 25.3, 14.1; ESI-MS:  $m/z = 345$  [ $\text{M}+\text{Na}$ ] $^+$ ; HRMS calcd for  $\text{C}_{18}\text{H}_{26}\text{O}_5\text{Na}$ : 345.16725; found: 345.16693.

### ((*2R,3R*)-3-((*S*)-4-(benzyloxy)-1-(methoxymethoxy)butyl)oxiran-2-yl)methanol (4):



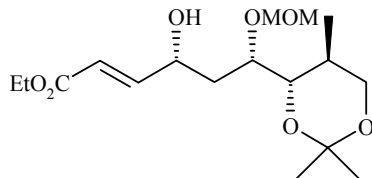
$[\alpha]_D^{25} = -15.6$  ( $c = 1.0$ ,  $\text{CHCl}_3$ ); IR (Neat): 3446, 2933, 1637, 1098, 1033  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz):  $\delta$  7.34-7.22 (m, 5H), 4.81 (d,  $J = 6.7$  Hz, 1H), 4.59 (d,  $J = 6.7$  Hz, 1H), 4.47 (s, 2H), 3.88-3.81 (m, 1H), 3.65-3.56 (m, 1H), 3.49-3.42 (m, 2H), 3.37-3.30 (m, 1H), 3.35 (s, 3H), 2.98 (dd,  $J = 2.2, 7.5$  Hz, 1H), 2.91 (q,  $J = 2.2$ , 1H), 1.77-1.57 (m, 4H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta$  138.1, 128.3, 127.6, 127.5, 95.3, 76.8, 72.8, 69.8, 61.1, 57.9, 55.5, 55.4, 28.9, 25.5; ESI-MS:  $m/z = 319$   $[\text{M}+\text{Na}]^+$ ; HRMS calcd for  $\text{C}_{16}\text{H}_{24}\text{O}_5\text{Na}$ : 319.15160; found: 319.15126.

**(2S,3S,4S)-7-(benzyloxy)-4-(methoxymethoxy)-2-methylheptane-1,3-diol (9):**



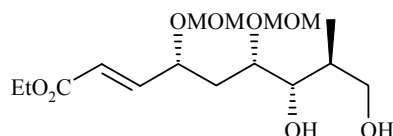
$[\alpha]_D^{25} = +31.6$  ( $c = 1.0$ ,  $\text{CHCl}_3$ ); IR (Neat): 3422, 2934, 2879, 1453, 1096, 1033  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  7.32-7.22 (m, 5H), 4.66 (q,  $J = 6.9$ , 2H), 4.46 (s, 2H), 3.69 (dd,  $J = 2.9, 10.8$  Hz, 1H), 3.64 (q,  $J = 5.9$ , 1H), 3.58 (dd,  $J = 5.9, 10.8$  Hz, 1H), 3.45 (t,  $J = 5.9$  Hz, 2H), 3.39 (s, 3H), 3.41-3.35 (m, 1H), 1.88-1.80 (m, 1H), 1.74-1.56 (m, 4H), 0.95 (d,  $J = 6.9$  Hz, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta$  138.2, 128.3, 127.6, 127.5, 96.3, 79.4, 77.8, 72.9, 70.0, 66.8, 55.9, 36.4, 27.5, 25.4, 14.1; ESI-MS:  $m/z = 335$   $[\text{M}+\text{Na}]^+$ ; HRMS calcd for  $\text{C}_{17}\text{H}_{28}\text{O}_5\text{Na}$ : 335.18290; found: 335.18201.

**(4R,6S,E)-ethyl 4-hydroxy-6-(methoxymethoxy)-6-((4S,5S)-2,2,5-trimethyl-1,3-dioxan-4-yl)hex-2-enoate (12):**



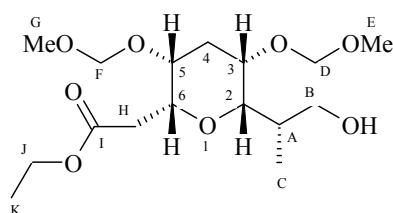
$[\alpha]_D^{25} = +21.3$  ( $c = 1.0$ ,  $\text{CHCl}_3$ ); IR (Neat): 3441, 2934, 1718, 1374, 1269, 1036  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz):  $\delta$  6.95 (dd,  $J = 3.7, 15.1$  Hz, 1H), 6.10 (dd,  $J = 2.2, 15.8$  Hz, 1H), 4.71 (q,  $J = 6.7$ , 2H), 4.56-4.46 (m, 1H), 4.21 (q,  $J = 7.5$  Hz, 2H), 3.99-3.92 (m, 1H), 3.74 (dd,  $J = 5.2, 11.3$  Hz, 1H), 3.62 (dd,  $J = 2.2, 10.5$  Hz, 1H), 3.57-3.51 (m, 1H), 3.42 (s, 3H), 2.19-2.02 (m, 2H), 1.81-1.69 (m, 1H), 1.45 (s, 3H), 1.43 (s, 3H); 1.29 (t,  $J = 7.5$  Hz, 3H), 0.84 (d,  $J = 6.0$  Hz, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta$  166.6, 149.8, 120.0, 98.6, 96.1, 76.0, 74.6, 67.7, 65.8, 60.3, 56.0, 37.2, 29.5, 29.3, 18.8, 14.1, 12.3; ESI-MS:  $m/z = 369$   $[\text{M}+\text{Na}]^+$ ; HRMS calcd for  $\text{C}_{17}\text{H}_{30}\text{O}_7\text{Na}$ : 369.18837; found: 369.18838.

**(4R,6S,7S,8S,E)-ethyl 7,9-dihydroxy-4,6-bis(methoxymethoxy)-8-methylnon-2-enoate (13):**



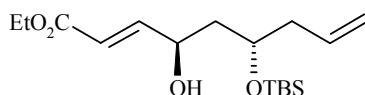
$[\alpha]_D^{25} = +87.3$  ( $c = 1.0$ ,  $\text{CHCl}_3$ ); IR (Neat): 3430, 2932, 1718, 1153, 1029  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz):  $\delta$  6.83 (dd,  $J = 6.7, 15.8$  Hz, 1H), 6.01 (d,  $J = 15.8$  Hz, 1H), 4.75-4.56 (m, 4H), 4.41-4.30 (m, 1H), 4.21 (q,  $J = 6.7$  Hz, 2H), 3.88-3.80 (m, 1H), 3.79-3.62 (m, 2H), 3.56-3.49 (m, 1H), 3.42 (s, 3H), 3.38 (s, 3H), 2.08-1.84 (m, 3H), 1.30 (t,  $J = 7.5$  Hz, 3H), 0.95 (d,  $J = 7.5$  Hz, 3H),  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta$  166.0, 147.0, 122.2, 96.2, 94.7, 77.5, 75.8, 72.6, 67.0, 60.5, 56.0, 55.9, 36.6, 36.0, 14.1, 13.8; ESI-MS:  $m/z = 373$   $[\text{M}+\text{Na}]^+$ ; HRMS calcd for  $\text{C}_{16}\text{H}_{30}\text{O}_8\text{Na}$ : 373.18329; found: 369.18333.

**Ethyl 2-((2R,3R,5S,6S)-6-((S)-1-hydroxypropan-2-yl)-3,5-bis(methoxymethoxy)-tetrahydro-2H-pyran-2-yl)acetate (1):**



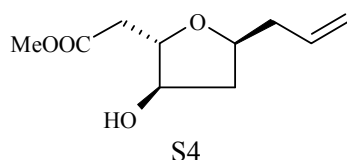
$[\alpha]_D^{25} = +8.0$  ( $c = 0.4$ ,  $\text{CHCl}_3$ ); IR (Neat): 3444, 2925, 2932, 1714, 1456, 1151, 1042  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 600 MHz):  $\delta$  4.81 (d,  $J = 7.1$  Hz,  $\text{H}_{\text{Da}}$ ), 4.70 (d,  $J = 6.9$  Hz,  $\text{H}_{\text{Fa}}$ ), 4.66 (d,  $J = 6.9$  Hz,  $\text{H}_{\text{Fb}}$ ), 4.63 (d,  $J = 7.1$  Hz,  $\text{H}_{\text{Db}}$ ), 4.40 (td,  $J = 4.0, 10.1$  Hz,  $\text{H}_6$ ), 4.22 (m,  $\text{H}_{\text{Ja}}$ ), 4.18 (m,  $\text{H}_{\text{Jb}}$ ), 3.82 (td,  $J = 4.0, 1.9$  Hz,  $\text{H}_3$ ), 3.75 (dd,  $J = 2.8, 11.3$  Hz,  $\text{H}_{\text{Ba}}$ ), 3.58 (q,  $J = 4.1$  Hz,  $\text{H}_5$ ), 3.53 (dd,  $J = 1.9, 10.1$  Hz,  $\text{H}_2$ ), 3.49 (dd,  $J = 5.2, 11.2$  Hz,  $\text{H}_{\text{Bb}}$ ), 3.42 (s,  $\text{H}_\text{E}$ ), 3.38 (s,  $\text{H}_\text{G}$ ), 2.73 (dd,  $J = 10.1, 14.3$  Hz,  $\text{H}_{\text{Ha}}$ ), 2.54 (dd,  $J = 4.0, 14.3$  Hz,  $\text{H}_{\text{Hb}}$ ), 2.24 (m,  $\text{H}_\text{A}$ ), 2.22 (m,  $\text{H}_{4\text{a}}$ ), 1.86 (td,  $J = 4.0, 15.2$  Hz,  $\text{H}_{4\text{b}}$ ), 1.28 (t,  $J = 7.0$  Hz,  $\text{H}_\text{K}$ ), 0.93 (d,  $J = 7.0$  Hz,  $\text{H}_\text{C}$ );  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  171.2, 94.6, 94.4, 75.0, 73.2, 70.2, 68.3, 65.8, 61.1, 55.9, 55.5, 36.2, 34.8, 27.9, 14.0, 12.9; ESI-MS:  $m/z = 351$   $[\text{M}+\text{H}]^+$ ; HRMS calcd for  $\text{C}_{16}\text{H}_{30}\text{O}_8\text{Na}$ : 373.18329; found: 373.18286

**(4R,6S,E)-ethyl 6-(tert-butyldimethylsilyloxy)-4-hydroxynona-2,8-dienoate (20):**



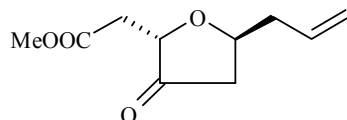
$[\alpha]_D^{25} = +12.1$  ( $c = 1.0$ ,  $\text{CHCl}_3$ ); IR (Neat): 3476, 3076, 2933, 2858, 1716, 1259, 1081  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz):  $\delta$  6.90 (dd,  $J = 3.9, 15.0$  Hz, 1H), 6.09 (dd,  $J = 2.0, 15.9$  Hz, 1H), 5.82-5.71 (m, 1H), 5.12-5.03 (m, 2H), 4.49-4.43 (m, 1H), 4.19 (q,  $J = 7.9$ , 2H), 4.06-4.00 (m, 1H), 3.38 (brs, OH), 2.34-2.28 (m, 2H), 1.78 (dt,  $J = 13.9, 2.9$  Hz, 1H), 1.67-1.60 (m, 1H), 1.28 (t,  $J = 6.9$  Hz, 3H), 0.92 (s, 9H), 0.14 (s, 3H), 0.13 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta$  166.6, 150.3, 134.0, 119.8, 117.8, 70.4, 67.8, 60.3, 41.0, 40.6, 25.7, 17.9, 14.2, -4.4, -4.8; ESI-MS:  $m/z = 351$   $[\text{M}+\text{Na}]^+$ .

**methyl 2-((2S,3R,5S)-5-allyl-3-hydroxytetrahydrofuran-2-yl)acetate (21):**

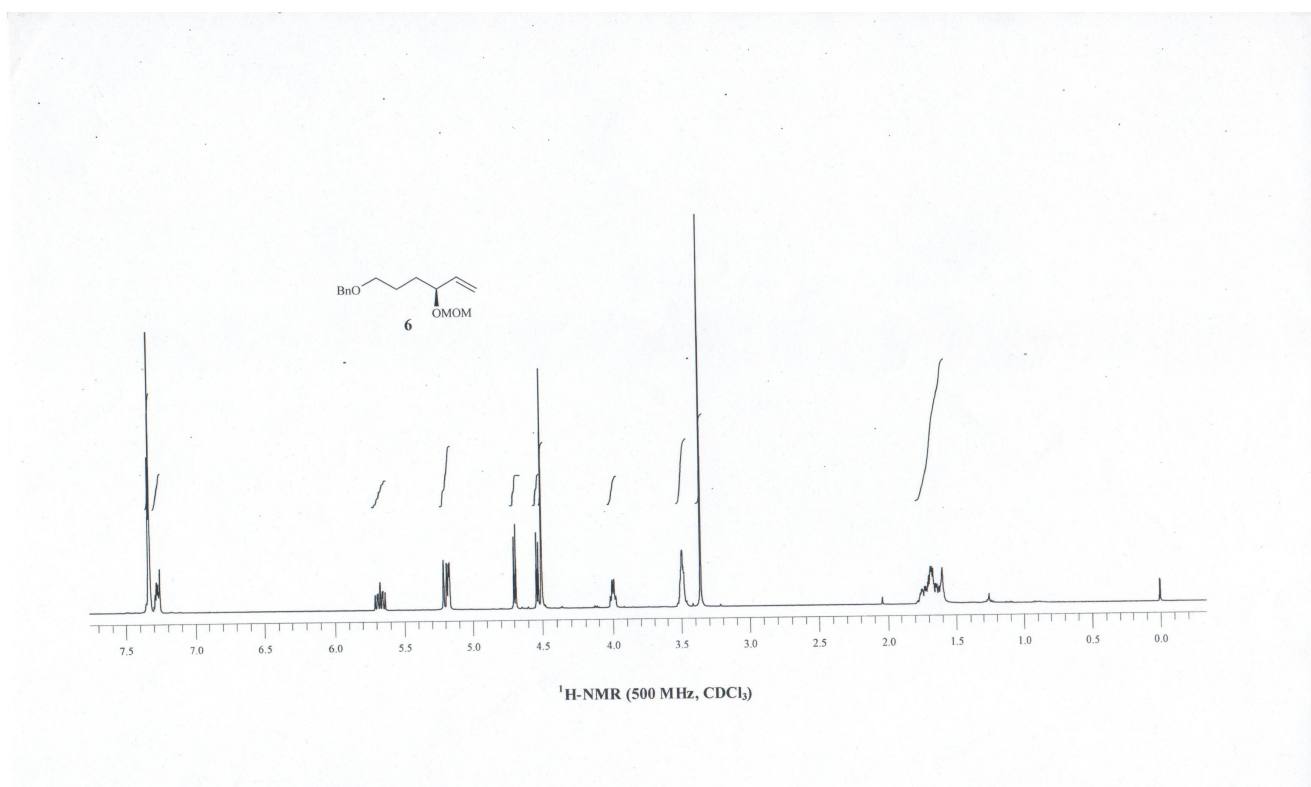


$[\alpha]_D^{25} = +11.0$  ( $c = 1.0$ ,  $\text{CHCl}_3$ ); IR (Neat): 3442, 3076, 2926, 1734, 1438, 1270, 1080  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  5.86-5.75 (m, 1H), 5.15-5.06 (m, 2H), 4.19-4.06 (m, 3H), 3.71 (s, 3H), 2.75 (dd,  $J = 6.0, 16.8\text{Hz}$ , 1H), 2.52 (dd,  $J = 7.9, 15.8\text{Hz}$ , 1H), 2.46-2.29 (m, 3H), 1.77-1.70 (m, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta$  172.5, 134.2, 117.4, 80.3, 76.9, 76.5, 51.9, 40.2, 39.3, 38.2; ESI-MS:  $m/z = 223$   $[\text{M}+\text{Na}]^+$ . HRMS calcd for  $\text{C}_{10}\text{H}_{16}\text{O}_4\text{Na}$ : 223.09408; found: 223.09404.

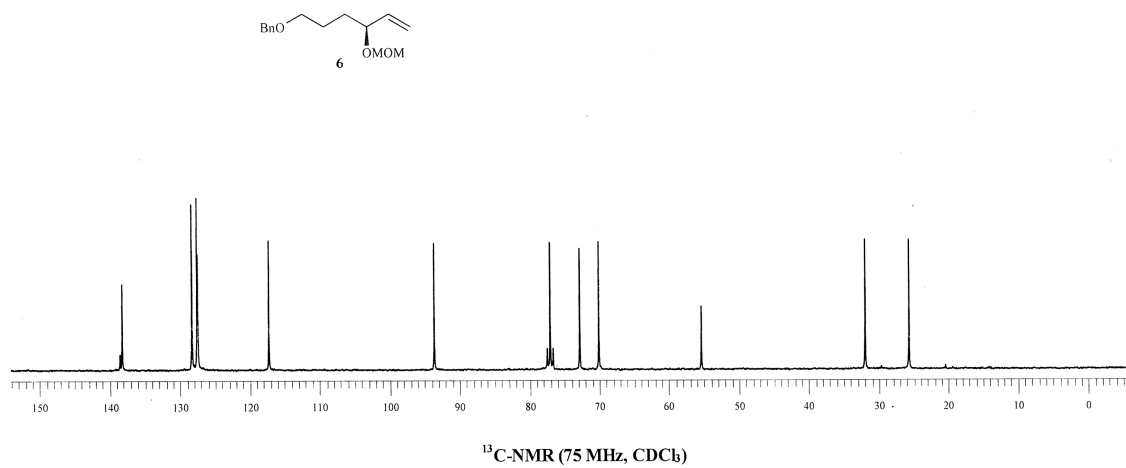
**methyl 2-((2S,5S)-5-allyl-3-oxotetrahydrofuran-2-yl)acetate (14):**

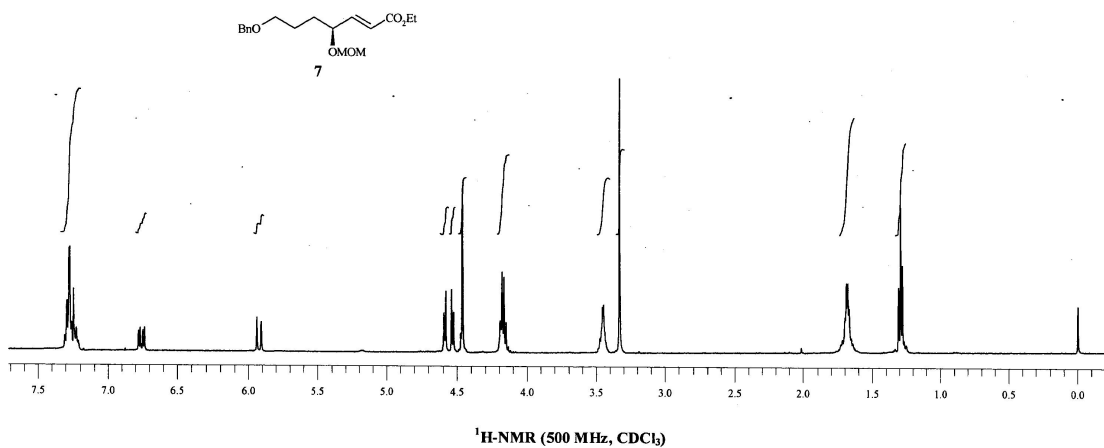


$[\alpha]_D^{25} = -17.5$  ( $c = 1.0$ ,  $\text{CHCl}_3$ ); IR (Neat): 2954, 1738, 1439, 1173, 1017  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz):  $\delta$  5.89-5.73 (m, 1H), 5.19-5.11 (m, 2H), 4.56 (qt,  $J = 6.0, 12.8\text{ Hz}$ , 1H), 4.23 (t,  $J = 4.5\text{ Hz}$ , 1H), 3.69 (s, 3H), 2.79 (d,  $J = 4.5\text{ Hz}$ , 2H), 2.74 (d,  $J = 7.5\text{ Hz}$ , 1H), 2.53-2.29 (m, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta$  215.0, 170.7, 133.0, 118.4, 75.7, 75.5, 52.0, 41.1, 40.1, 36.5; ESI-MS:  $m/z = 221$   $[\text{M}+\text{Na}]^+$ .

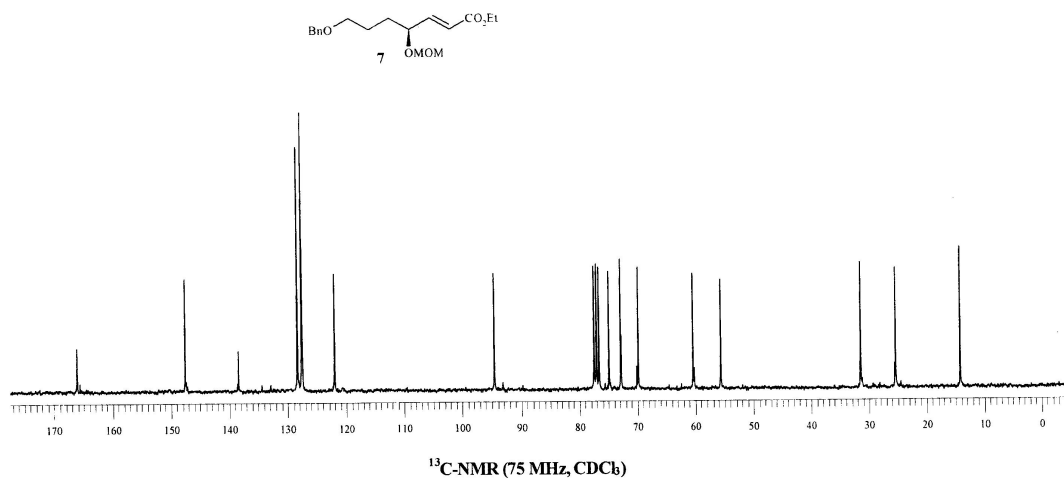


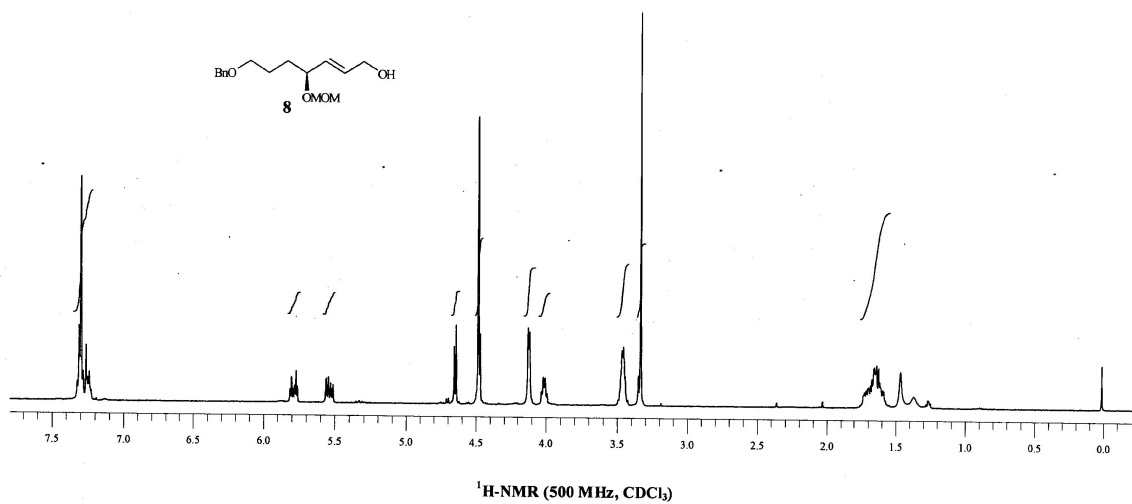
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128.246  
127.502  
127.402  
117.219  
93.578  
76.996  
72.773  
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31.922  
25.646



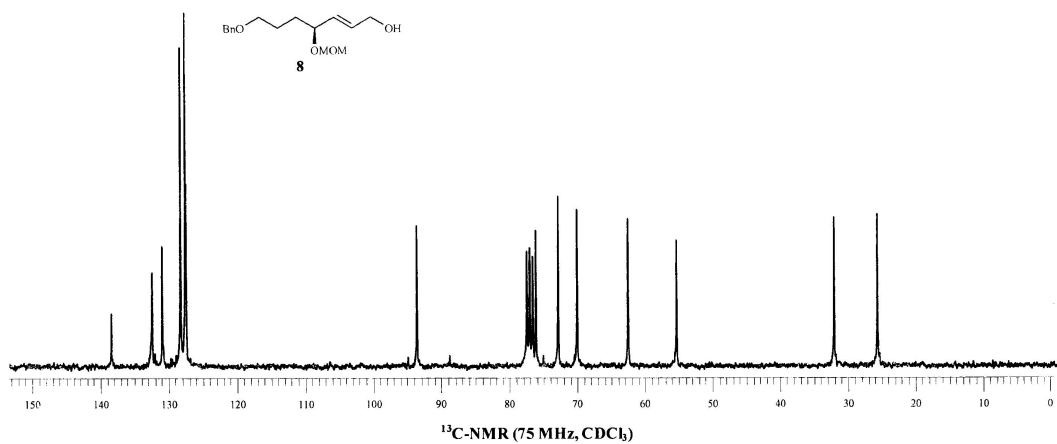


166.080  
147.544  
138.373  
128.268  
127.517  
127.452  
121.947  
94.516  
74.879  
72.836  
69.789  
60.372  
55.546  
31.448  
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14.125

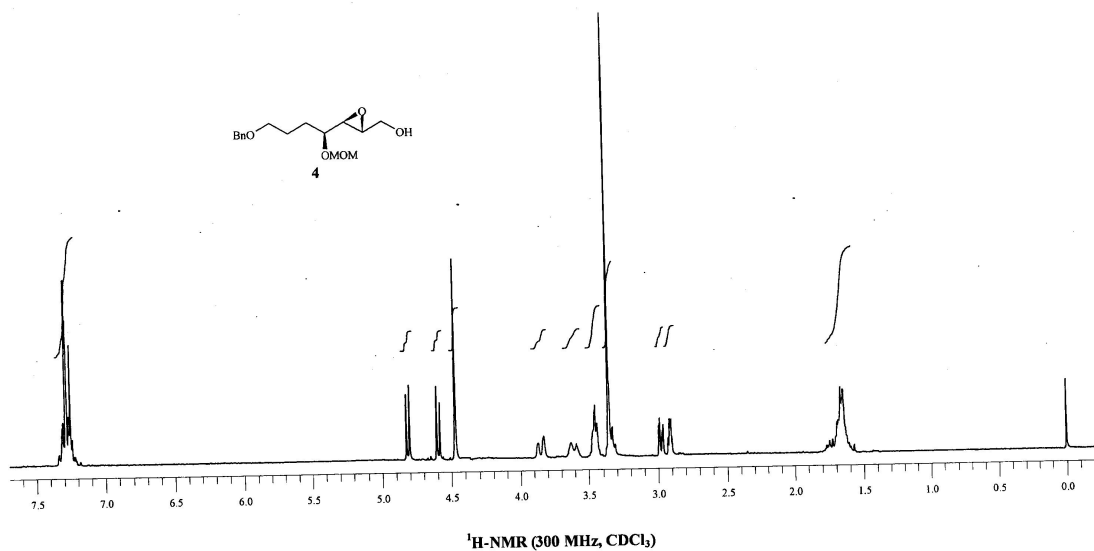




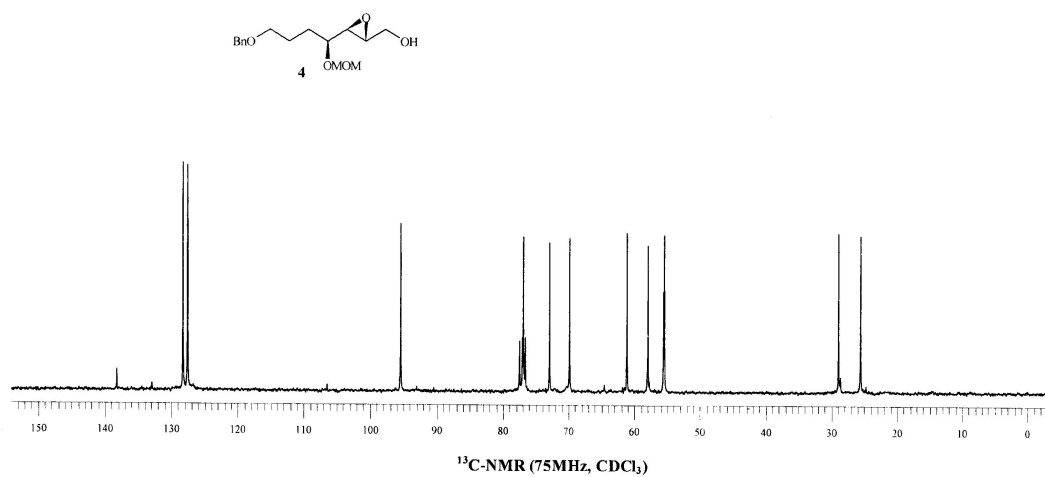
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128.246  
127.534  
127.438  
  
93.553  
  
76.097  
72.787  
70.001  
62.502  
55.299  
  
32.038  
25.607

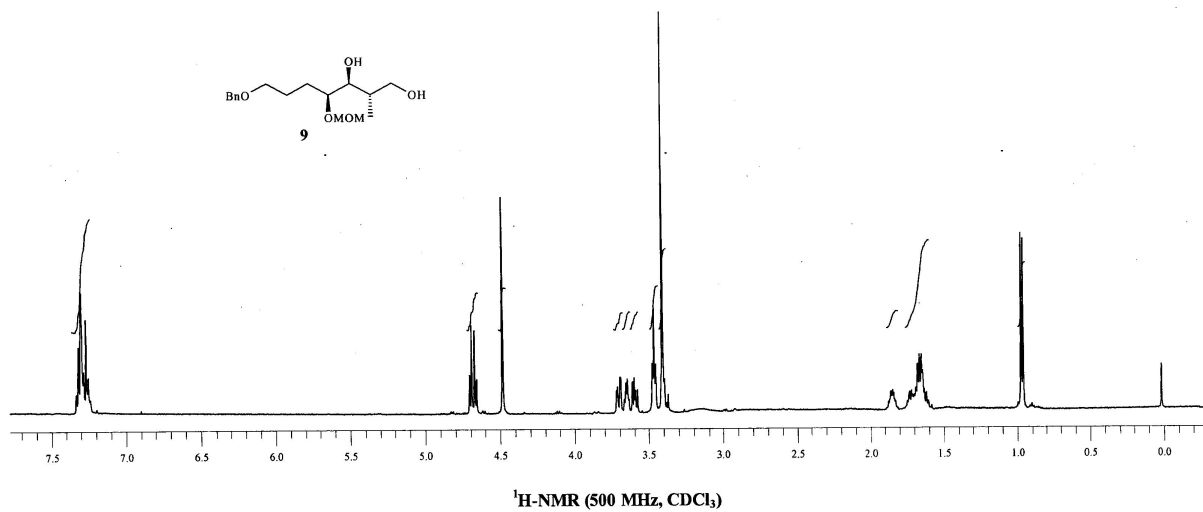




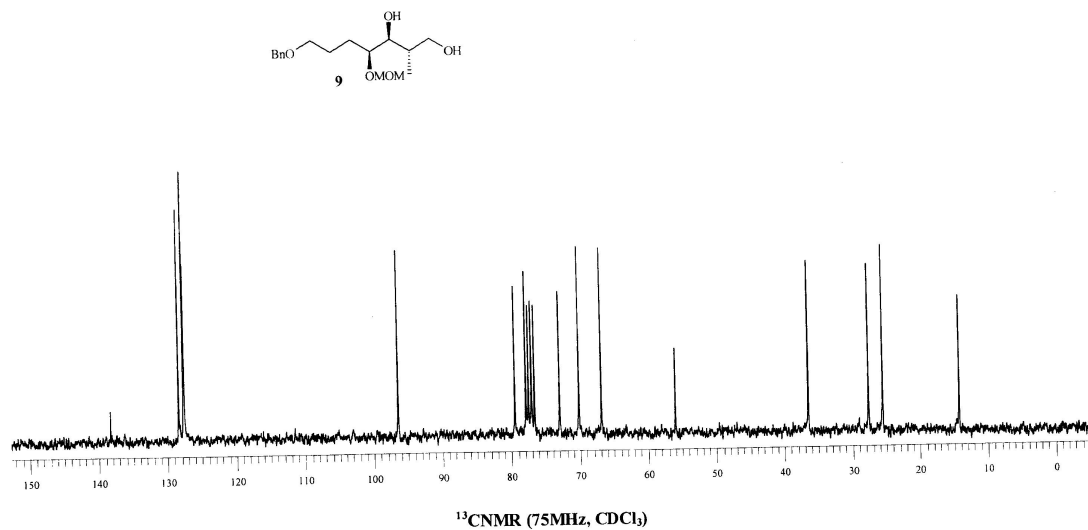


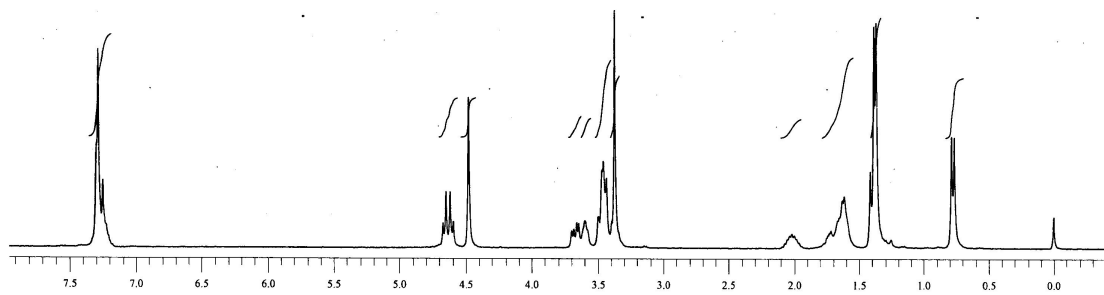
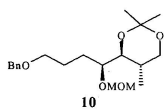
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128.296  
127.596  
127.548  
95.374  
76.874  
72.881  
69.849  
61.106  
57.914  
57.538  
55.427  
28.914  
25.569





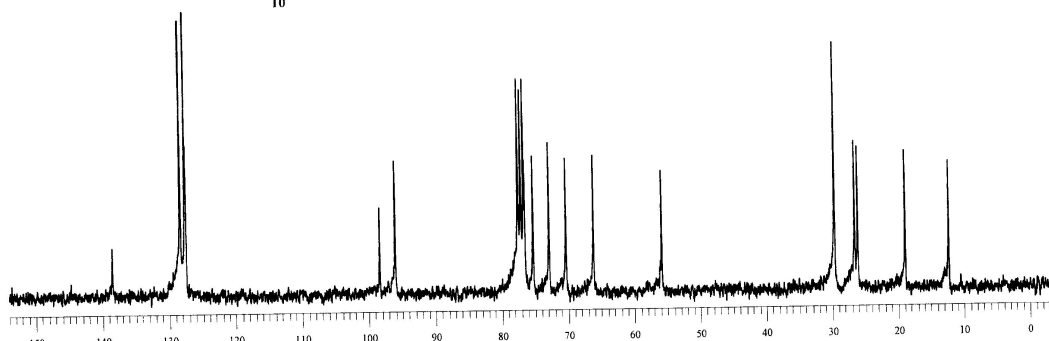
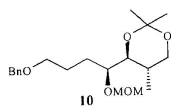
138.244  
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127.622  
127.527  
96.380  
79.384  
77.787  
72.920  
70.052  
66.795  
55.905  
36.394  
27.542  
25.424  
14.154



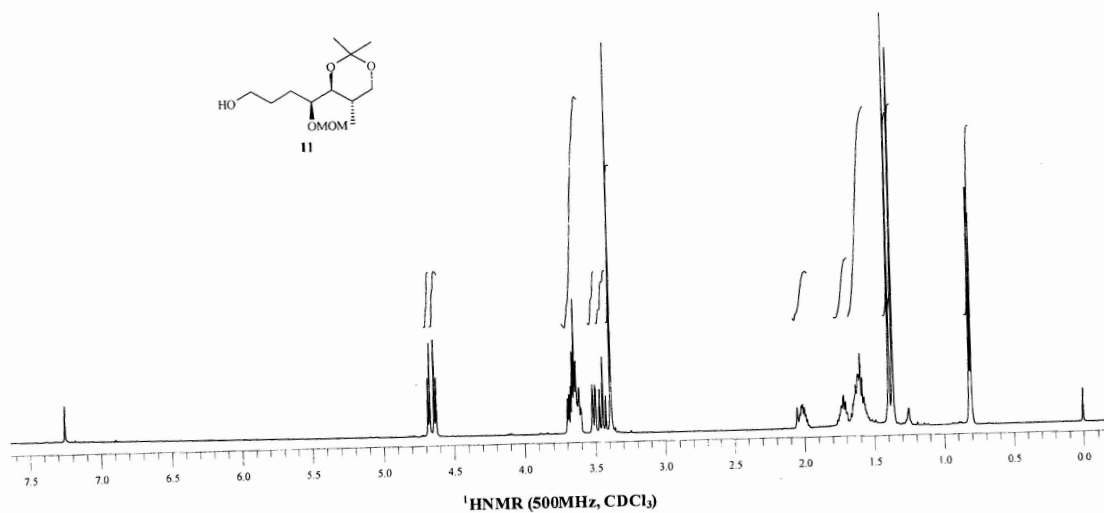


<sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>)

138.482  
128.268  
127.547  
127.445  
98.330  
95.999  
76.433  
75.208  
72.795  
70.258  
66.101  
55.816  
29.559  
26.527  
26.052  
18.841  
12.215



<sup>13</sup>C-NMR (75 MHz, CDCl<sub>3</sub>)



98.368  
96.004

76.569  
75.288

66.015  
62.645

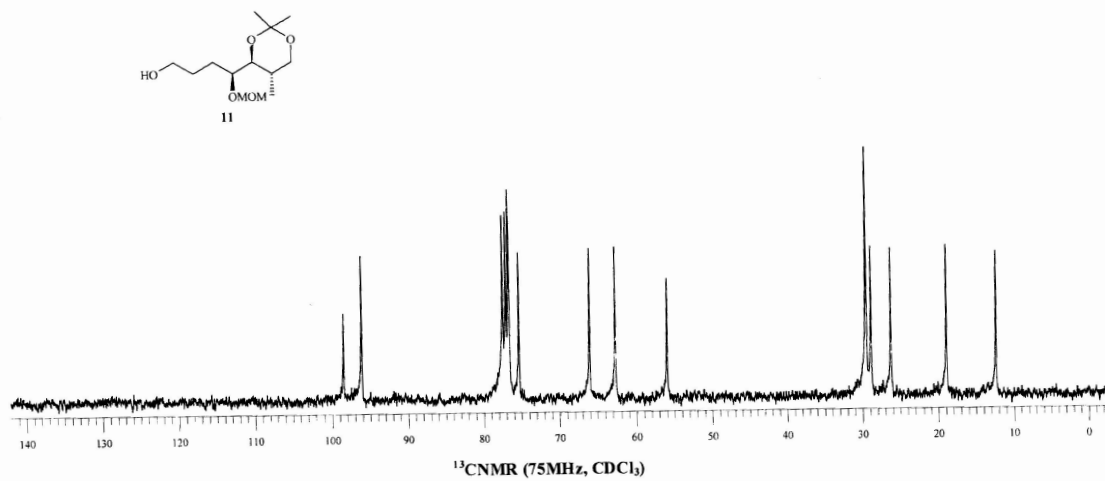
55.830

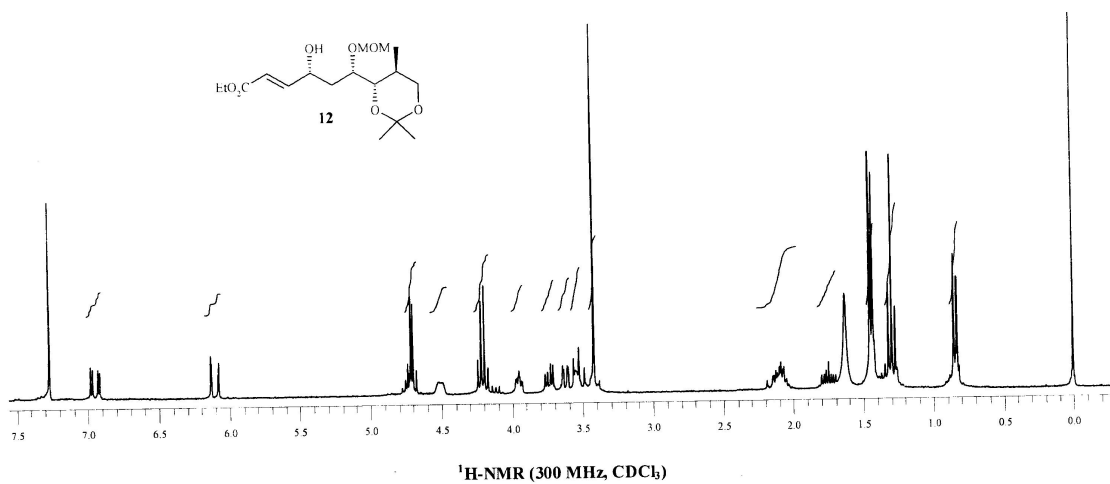
29.515  
28.845

26.214

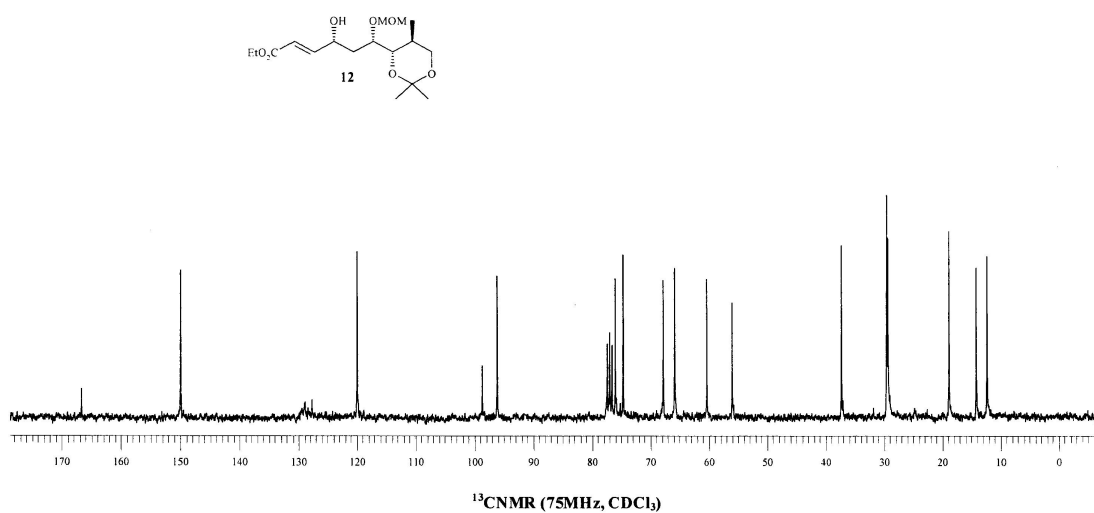
18.807

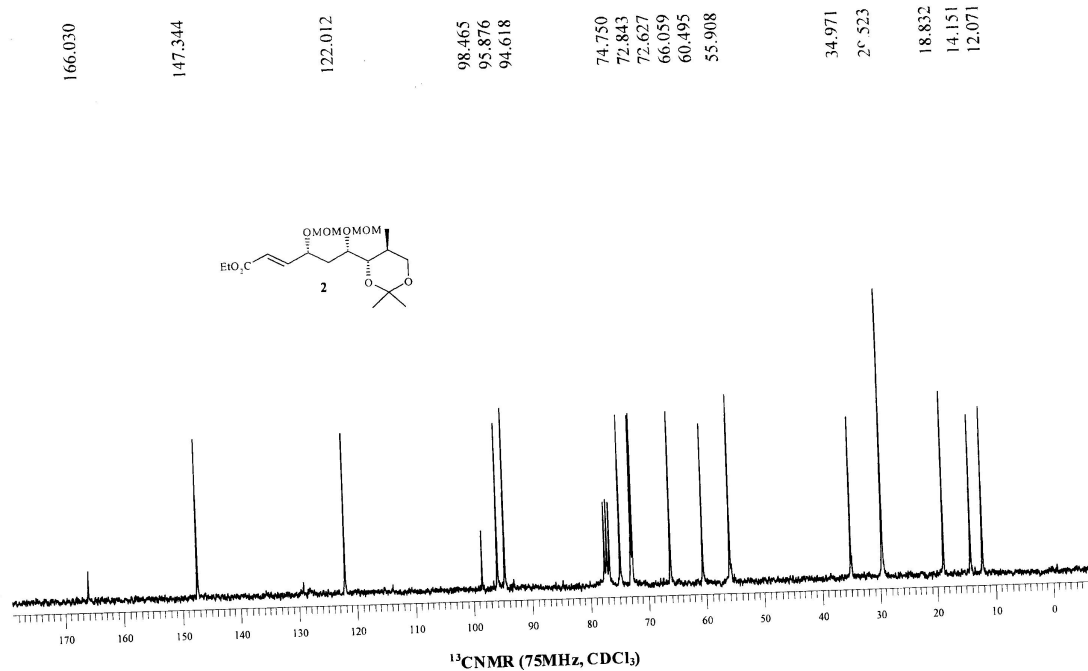
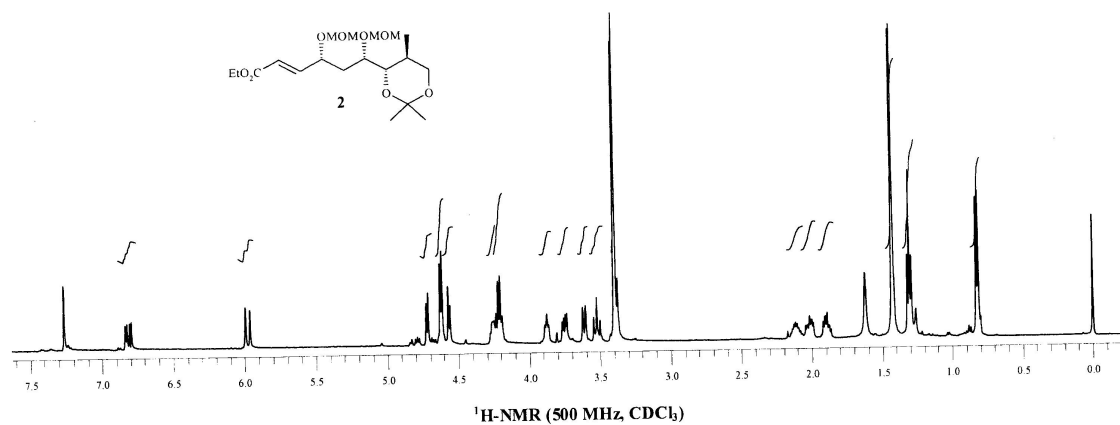
12.251

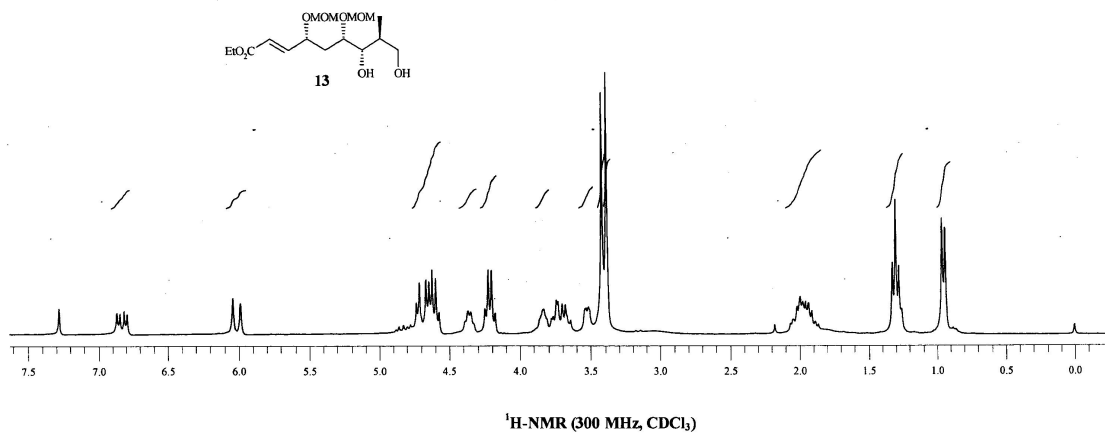




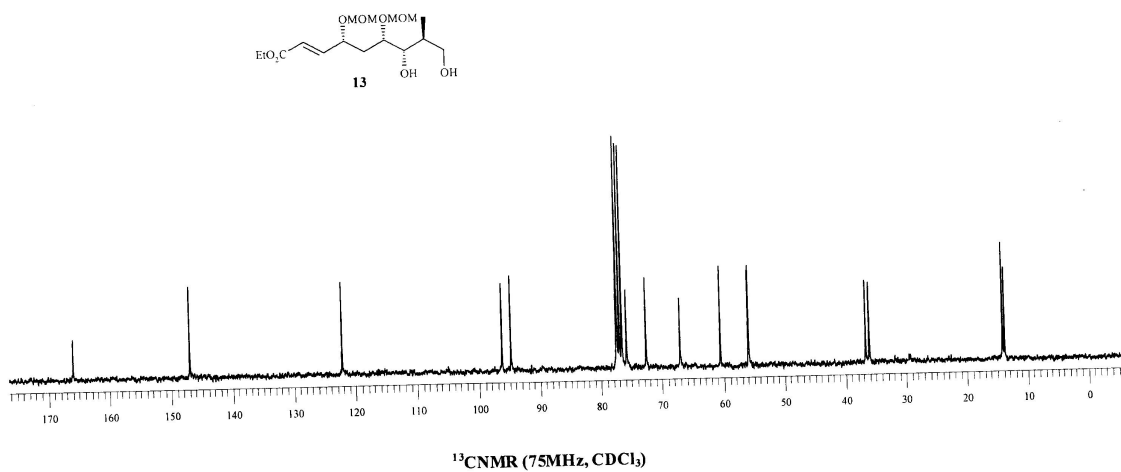
166.608  
149.849  
119.915  
98.677  
96.111  
75.977  
74.648  
67.776  
65.824  
60.330  
56.002  
37.267  
29.527  
29.317  
18.820  
14.137  
12.303

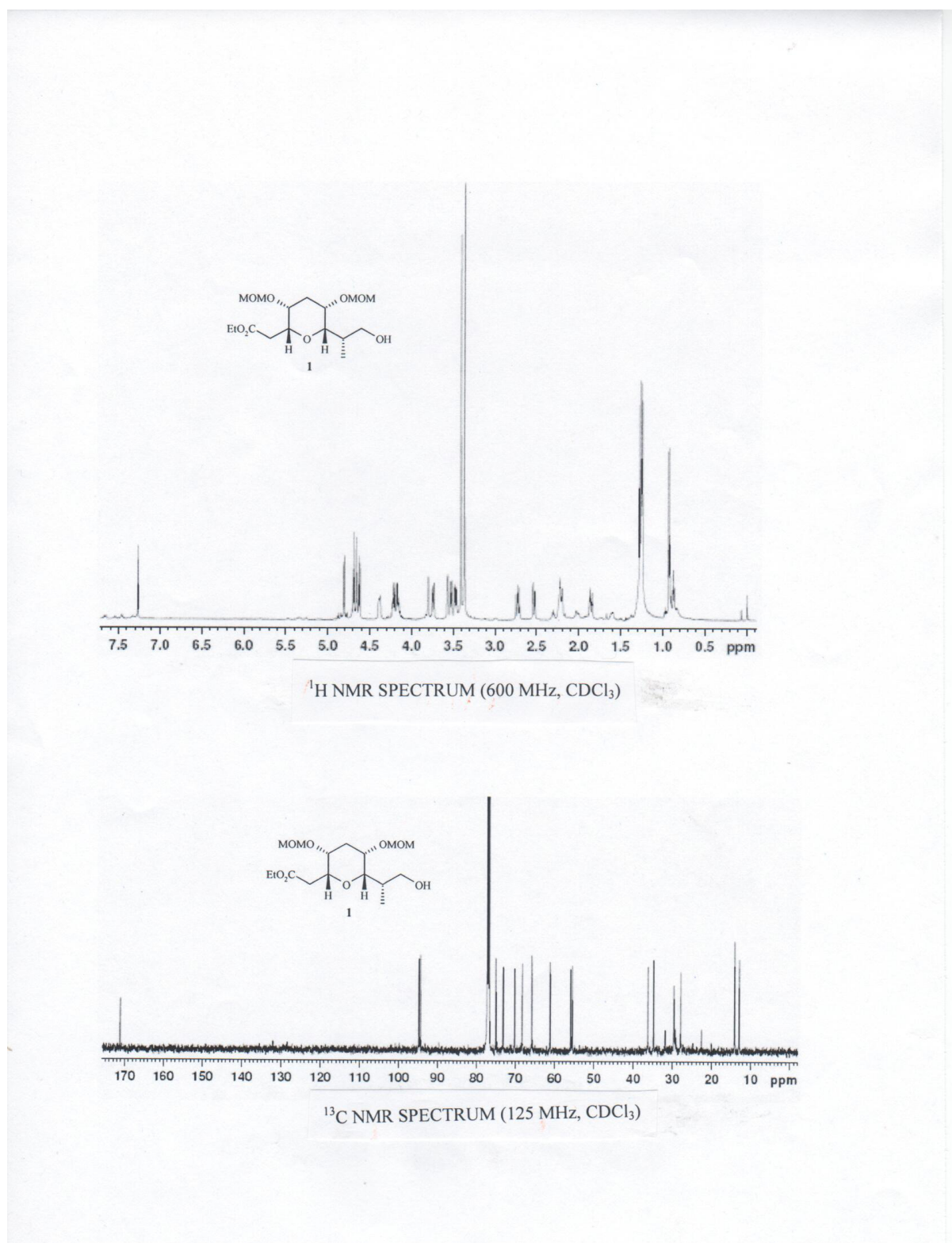






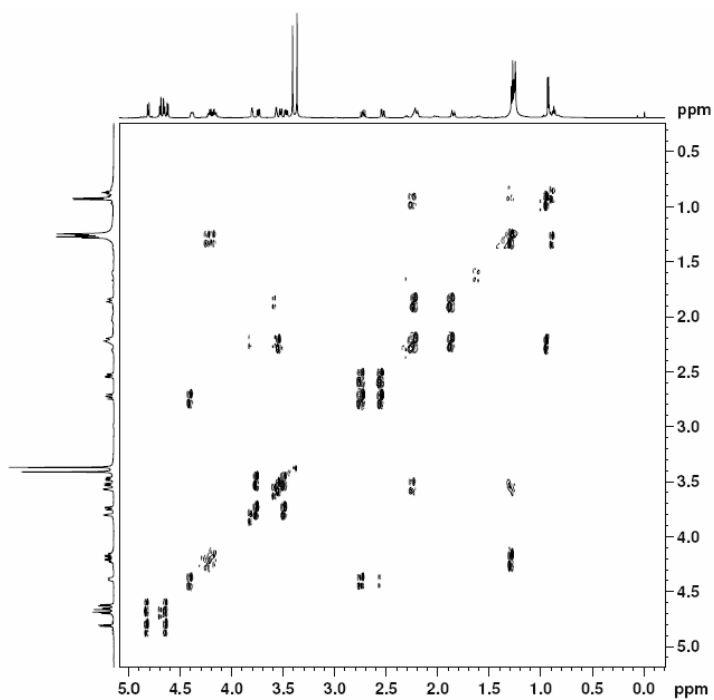
165.994  
146.996  
122.203  
96.230  
94.765  
77.497  
75.805  
72.589  
67.062  
60.532  
55.970  
55.897  
36.666  
36.083  
14.141  
13.818



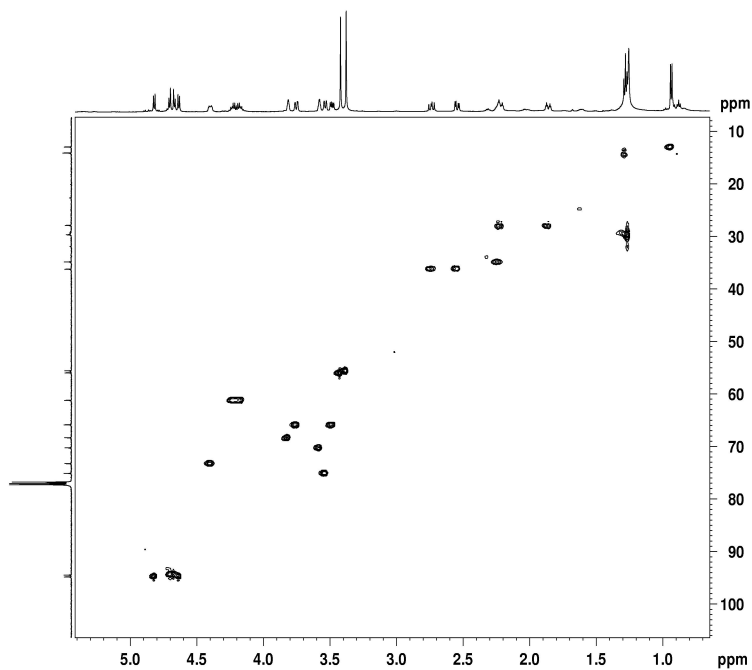




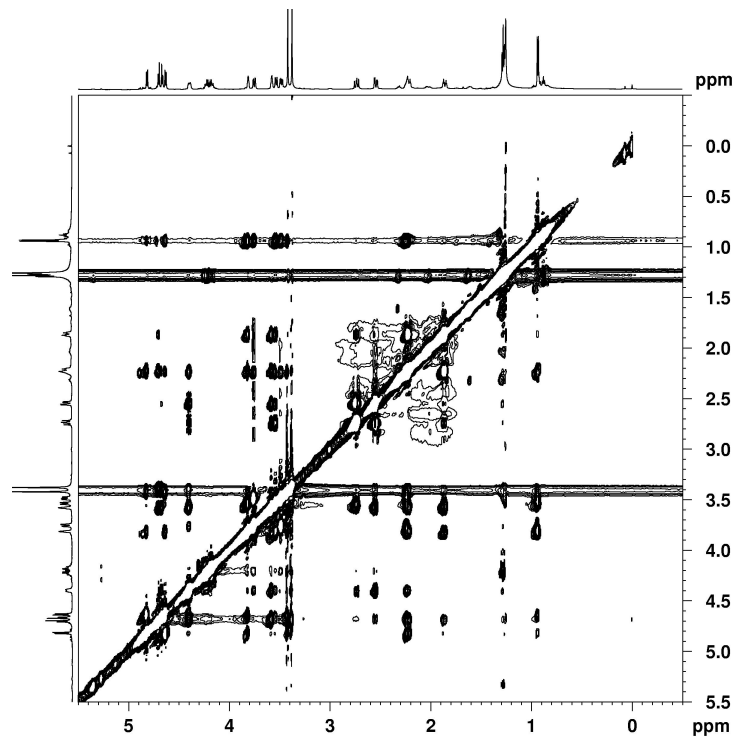
**COSY NMR Spectrum of compound 1 in CDCl<sub>3</sub> solution at 600 MHz**

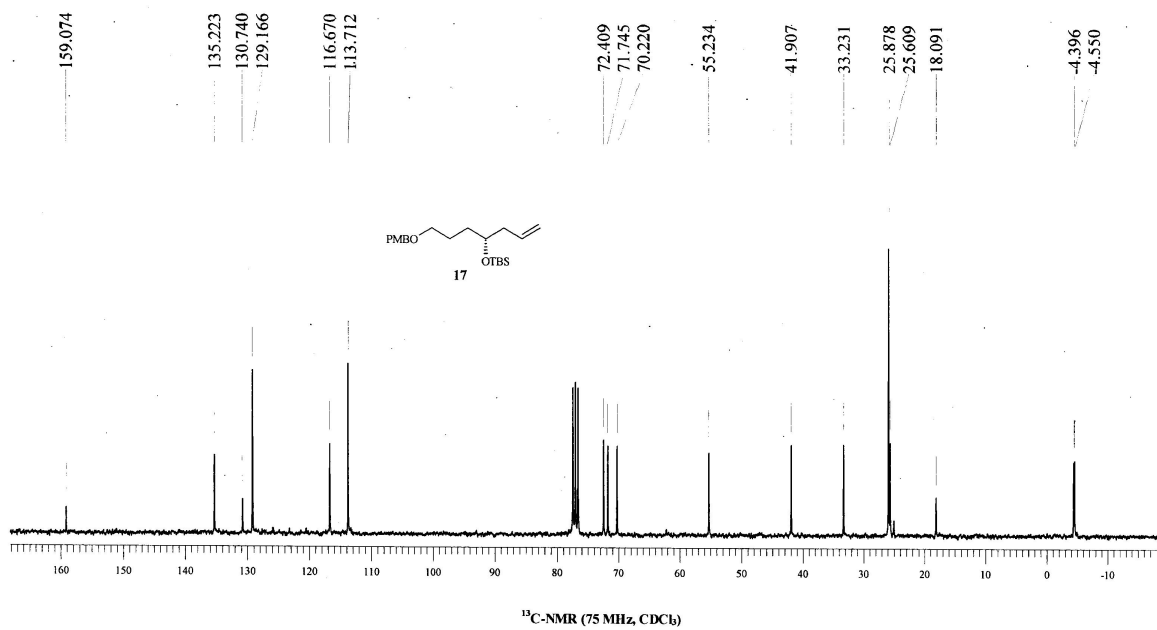
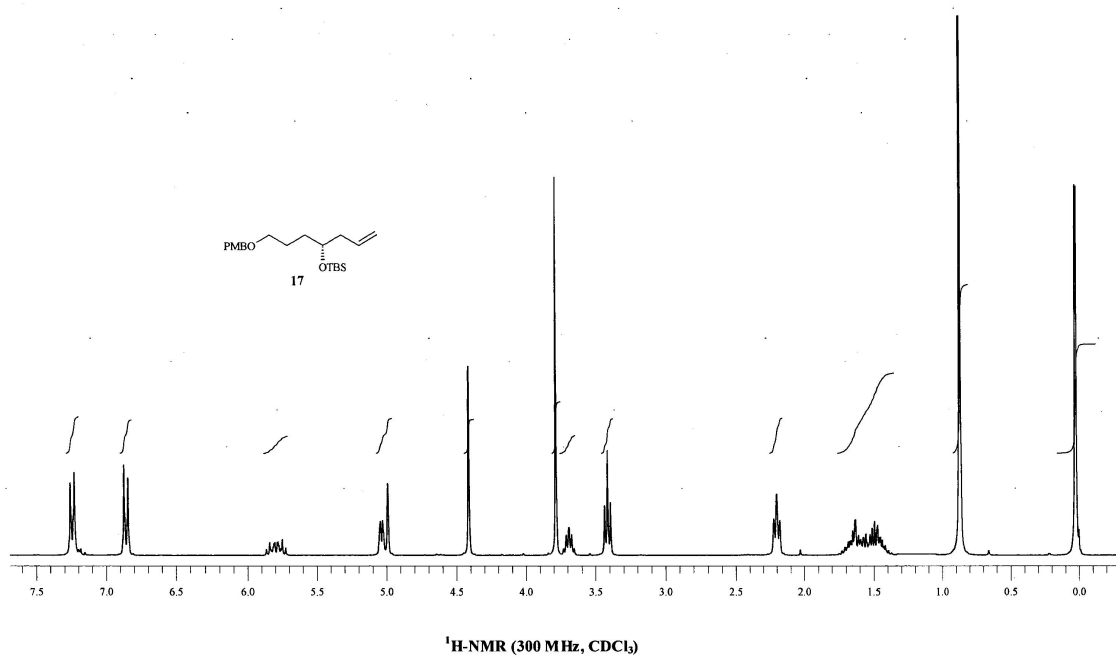


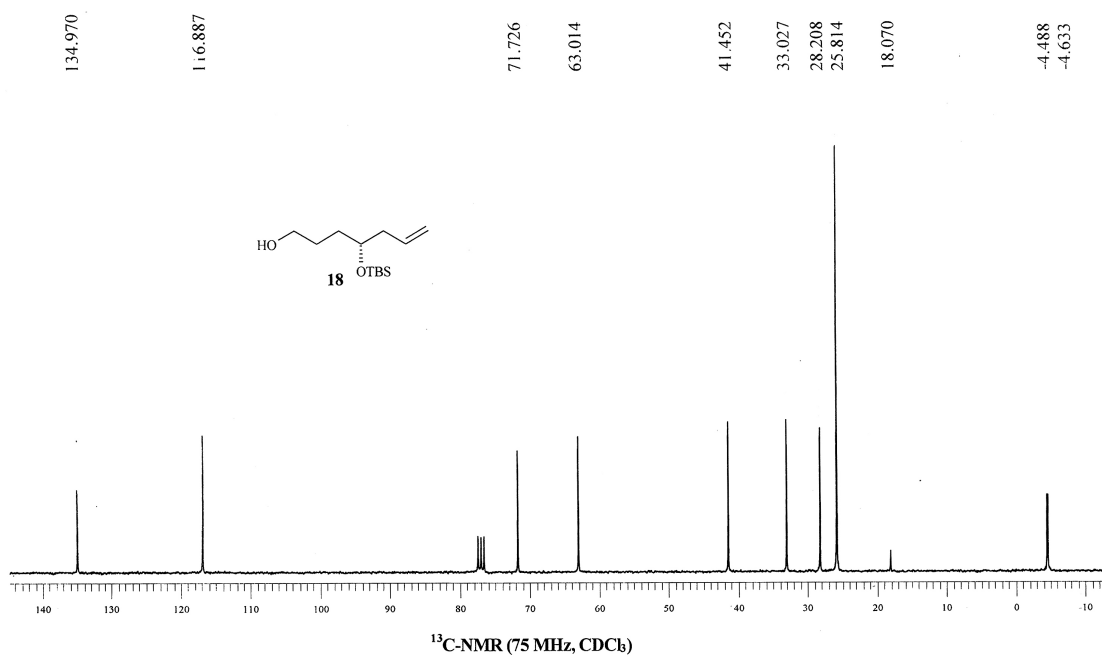
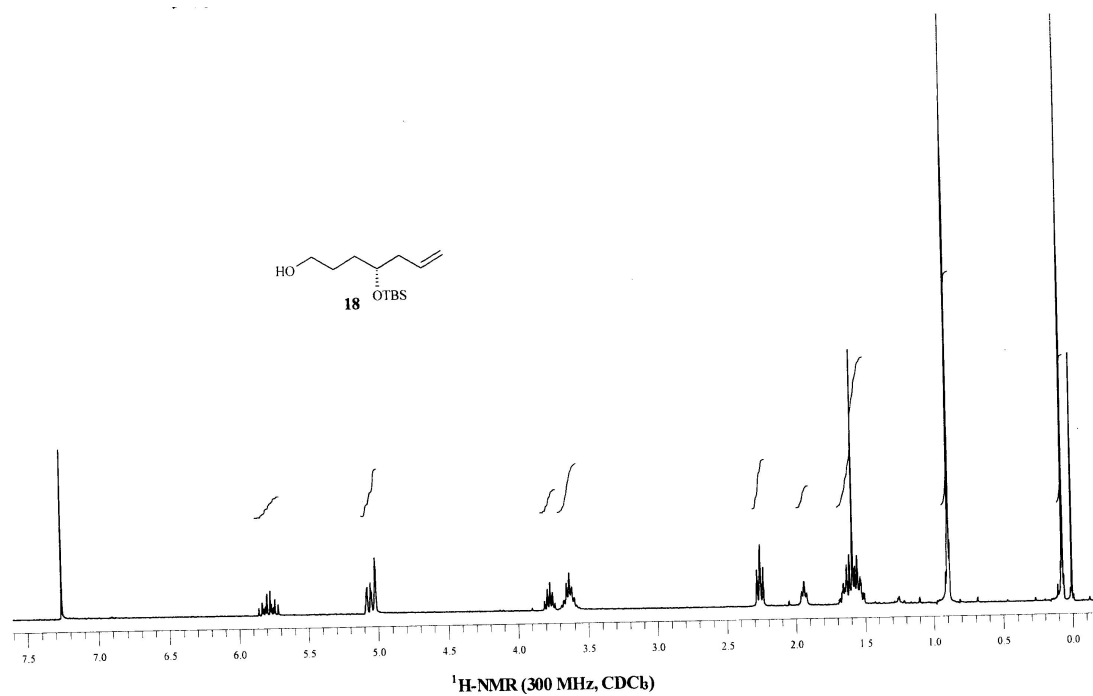
**HSQC NMR Spectrum of compound 1 in CDCl<sub>3</sub> solution at 600 MHz**

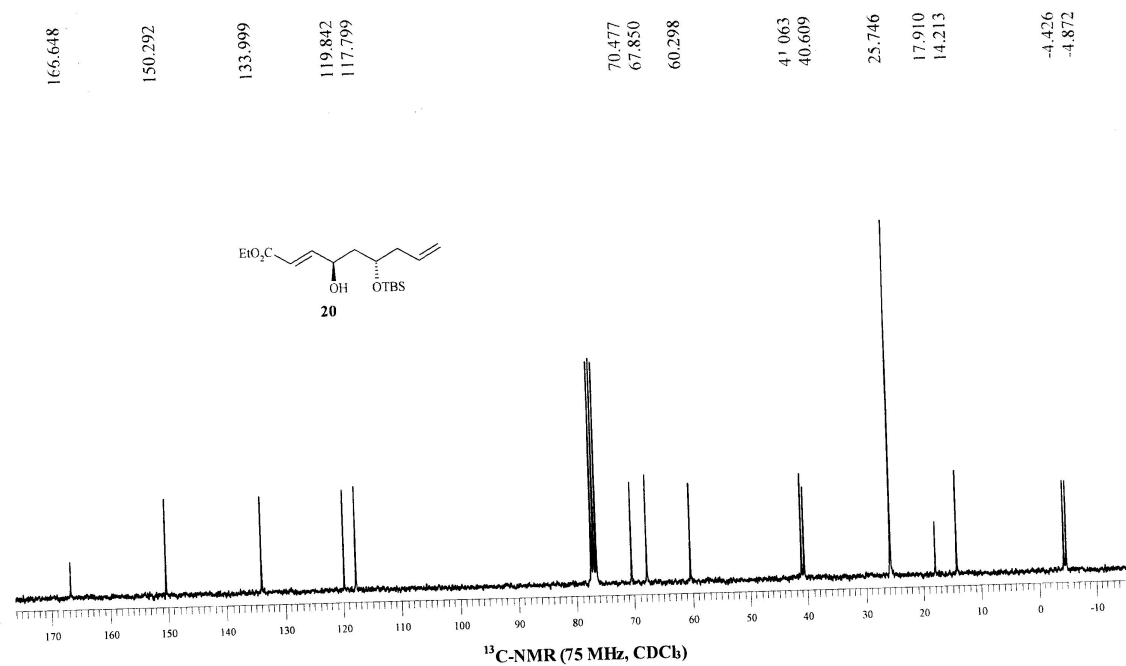
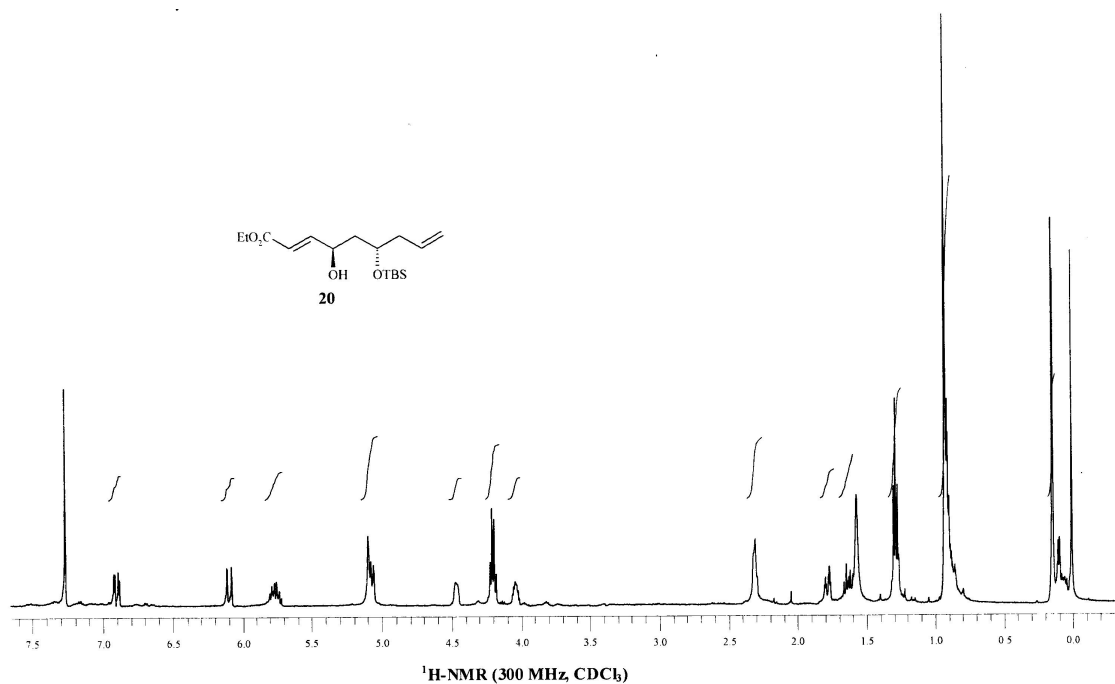


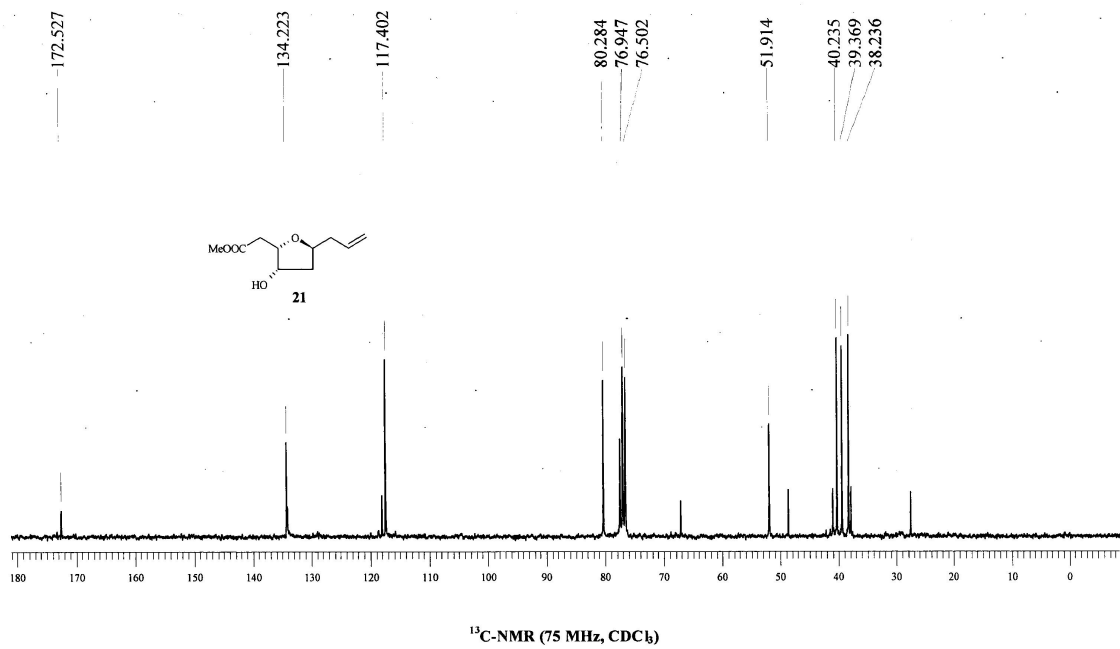
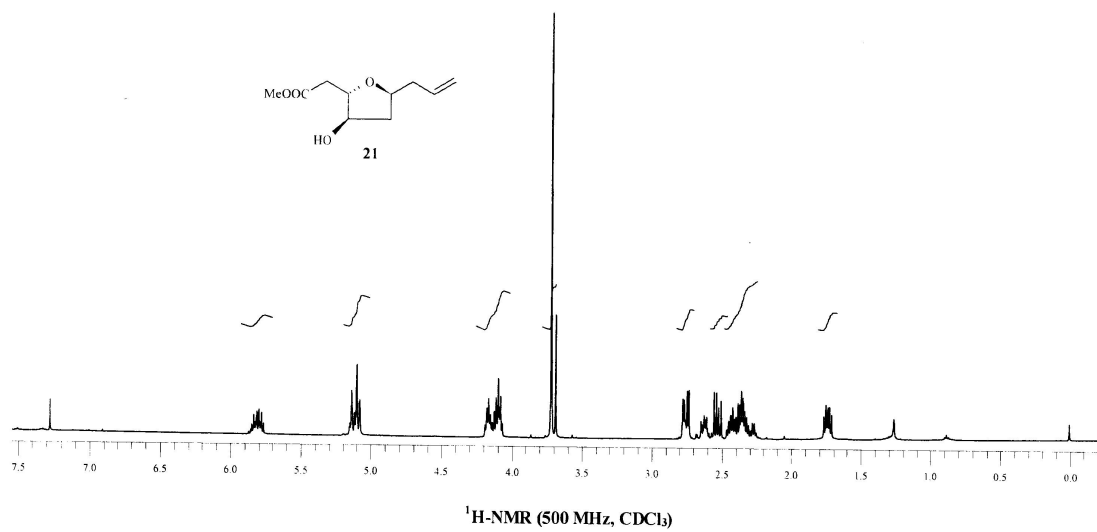
**Expanded NOESY NMR Spectrum of compound 1 in CDCl<sub>3</sub> solution at 600 MHz**



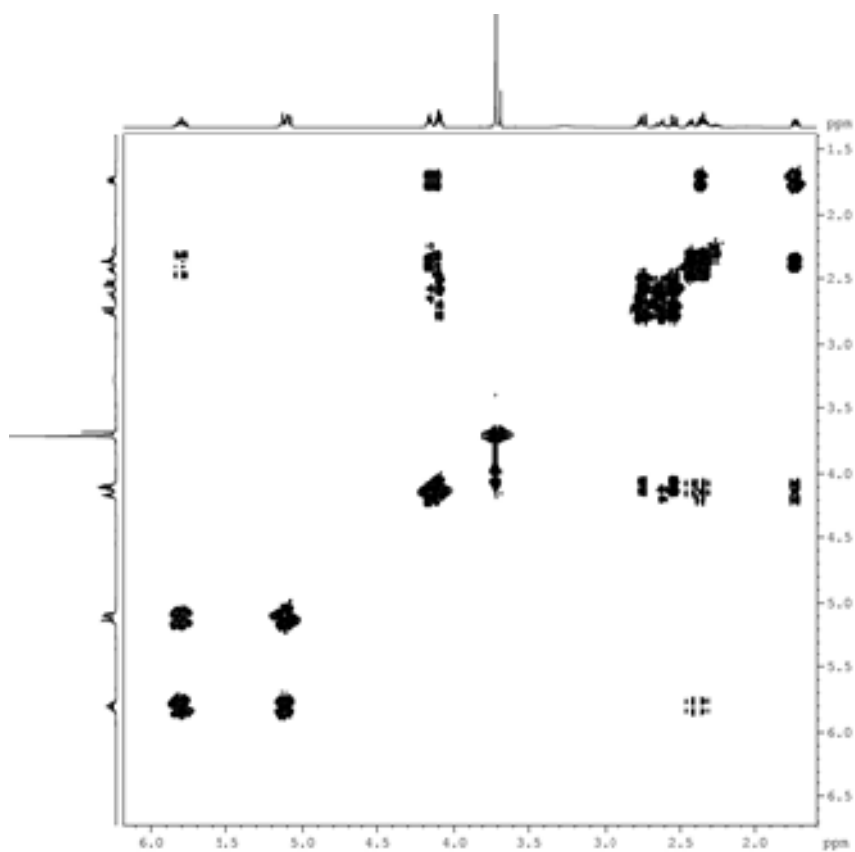




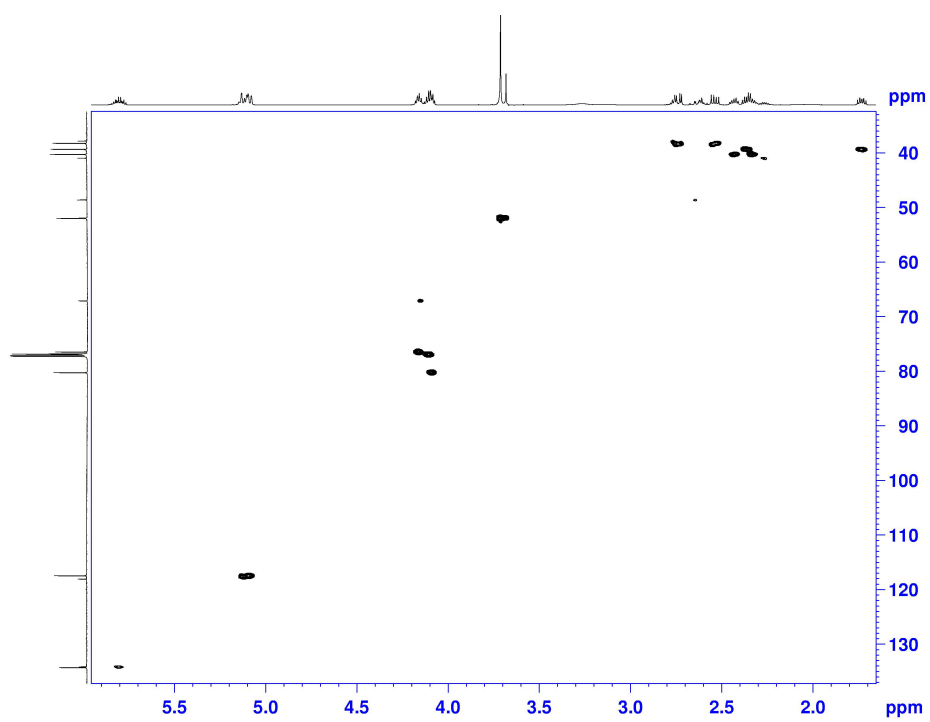




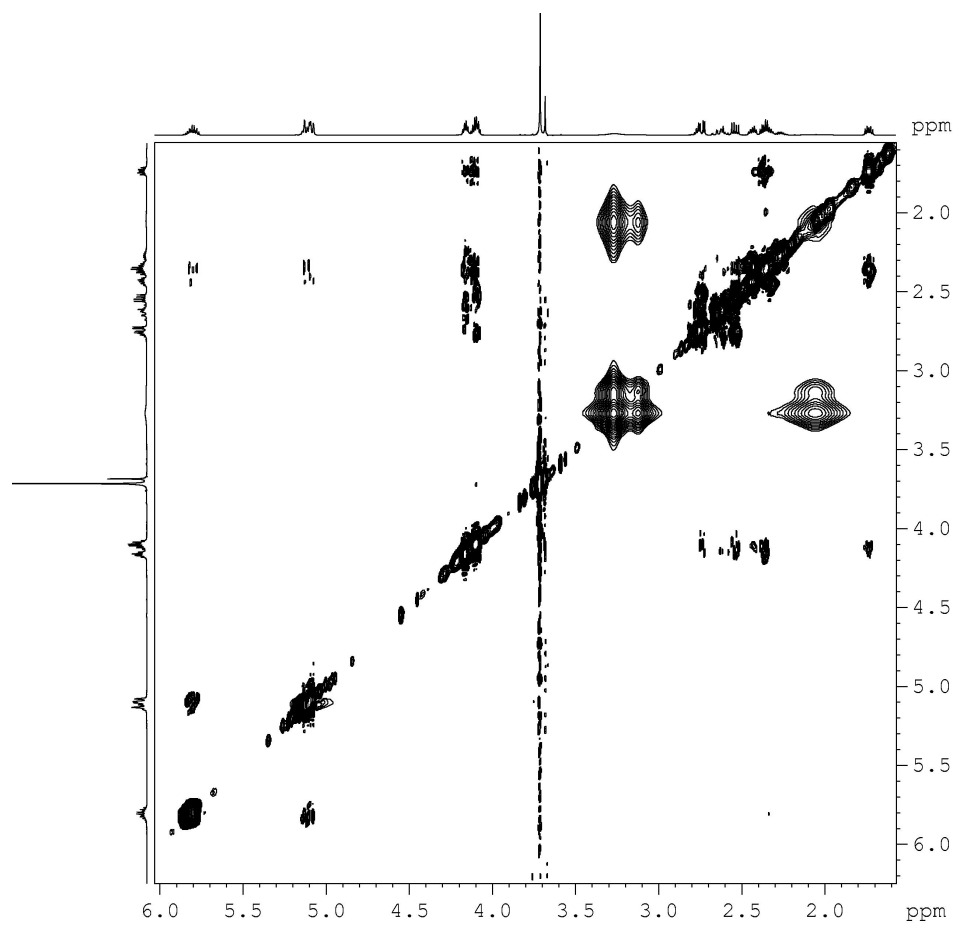
**COSY NMR Spectrum of compound 21 in CDCl<sub>3</sub> solution at 600MHz**



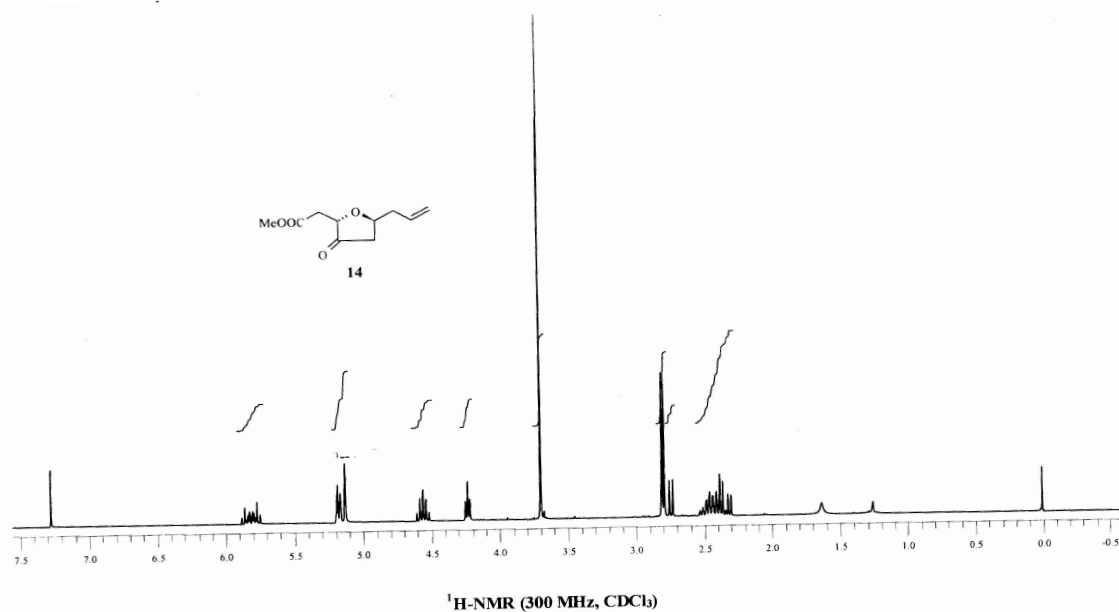
**HSQC NMR Spectrum of compound 21 in CDCl<sub>3</sub> solution at 600MHz**



**NOESY NMR Spectrum of compound 21 in CDCl<sub>3</sub> solution at 600MHz**







2.15.047

17.0.750

133.030

118.443

75.687

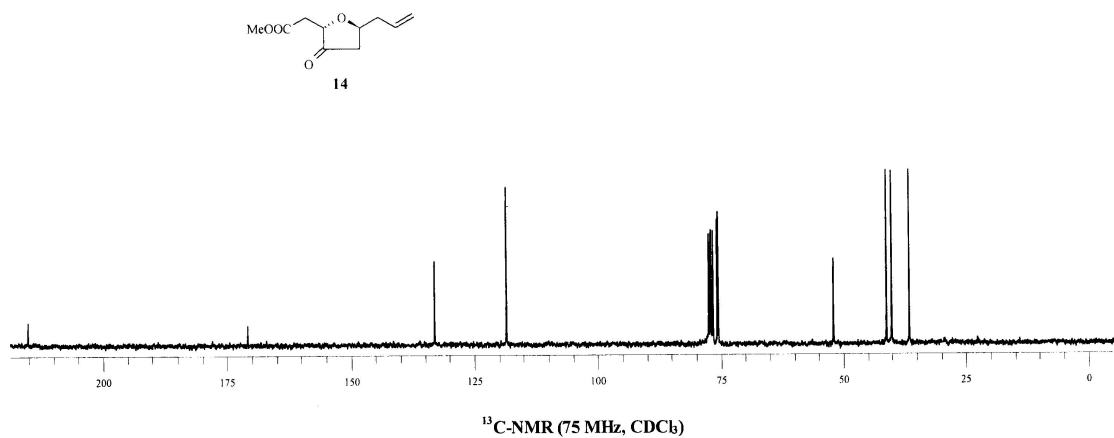
75.529

52.018

41.151

40.091

36.514



Data File D:\DATA\SABITHA\28061205.D

Sample Name: GSR-NITRO 1

HR C18: 40% ACN IN WATER

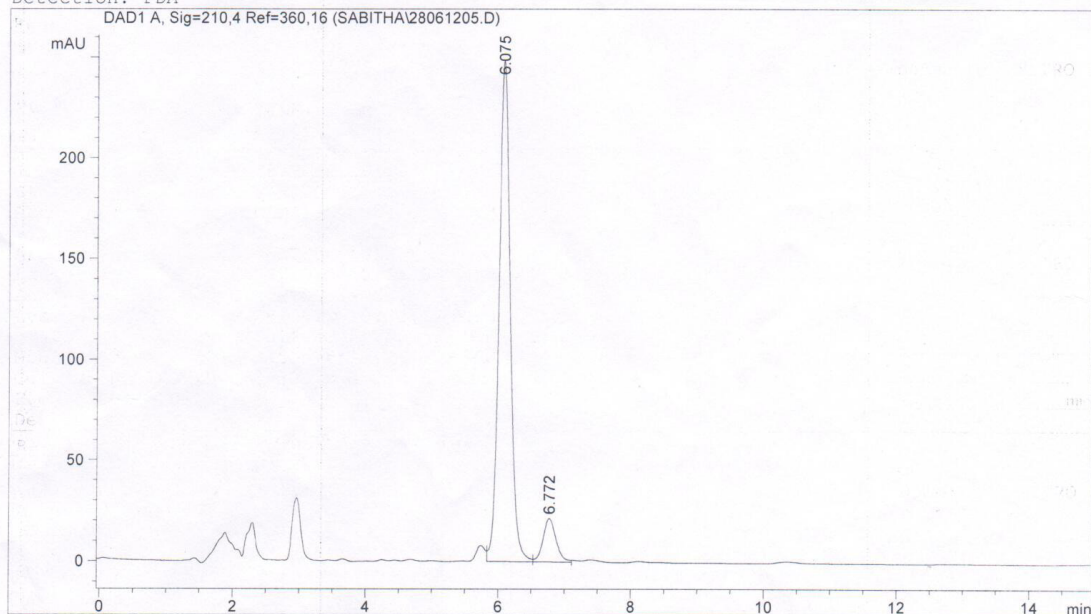
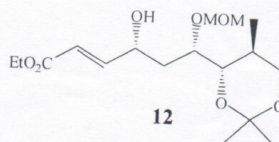
Injection Date : 6/28/2012 10:19:29 PM  
Sample Name : GSR-NITRO 1  
Acq. Operator : B.RAMESH

Location : Vial 12

Inj Volume : 10 µl

Acq. Method : D:\METHODS\GEN.M  
Last changed : 6/28/2012 10:34:55 PM by B.RAMESH  
Analysis Method : D:\METHODS\GEN.M  
Last changed : 6/28/2012 10:32:57 PM by RAJU  
(modified after loading)

Mobile phase: 40% Acetonitrile + 60% Water  
Column: WATERS HR C18 300 x 3.9mm 6,µm  
Flow rate: 1.0mL/min  
Detection: PDA



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=360,16

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.075	VV	0.1799	2941.80176	248.82742	90.9674
2	6.772	VB	0.2066	292.10458	21.48468	9.0326

Totals : 3233.90634 270.31211

Results obtained with enhanced integrator!

\*\*\* End of Report \*\*\*

Data File D:\DATA\SABITHA\28061201.D

Sample Name: GSR-NITRO 2

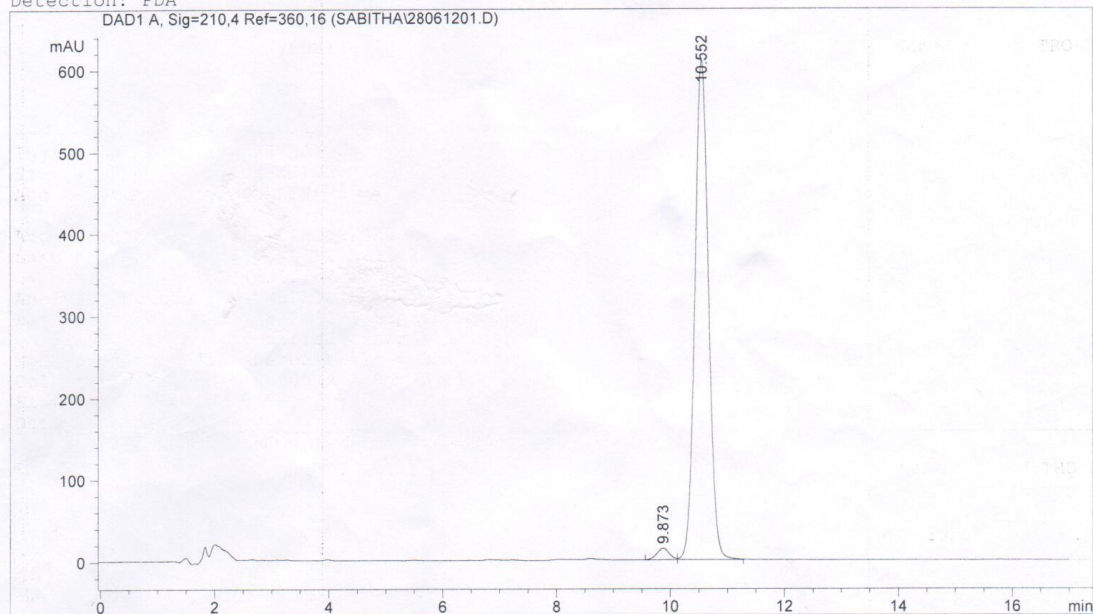
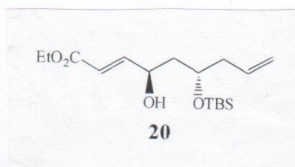
HR C18: 75% ACN IN WATER

=====  
Injection Date : 6/28/2012 8:54:06 PM  
Sample Name : GSR-NITRO 2  
Acq. Operator : B.RAMESH  
Location : Vial 11  
Inj Volume : 20 µl

Acq. Method : D:\METHODS\GEN.M  
Last changed : 6/28/2012 8:52:23 PM by B.RAMESH  
(modified after loading)

Analysis Method : D:\METHODS\GEN.M  
Last changed : 6/28/2012 10:14:44 PM by RAJU  
(modified after loading)

Mobile phase: 75% Acetonitrile + 25% Water  
Column: WATERS HR C18 300 x 3.9mm 6 micron  
Flow rate: 1.0mL/min  
Detection: PDA



=====  
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=360,16

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.873	BV	0.2422	215.65994	13.96120	2.0954
2	10.552	VB	0.2568	1.00762e4	609.80548	97.9046

Totals : 1.02919e4 623.76668

Results obtained with enhanced integrator!

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