Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

Synthesis of fully functionalized aglycon of lycoperdinoside A

By Balla Chandrasekhar, Sudhakar Athe, P. Purushotham Reddy, Subhash Ghosh*

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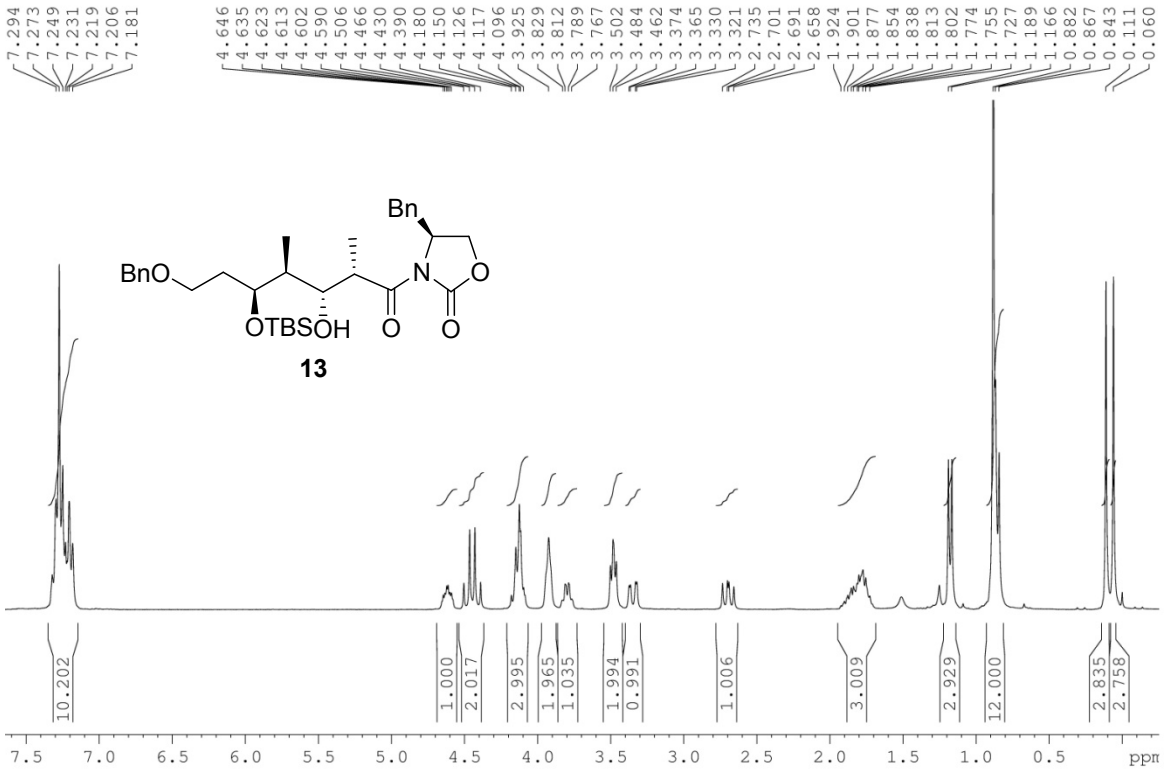
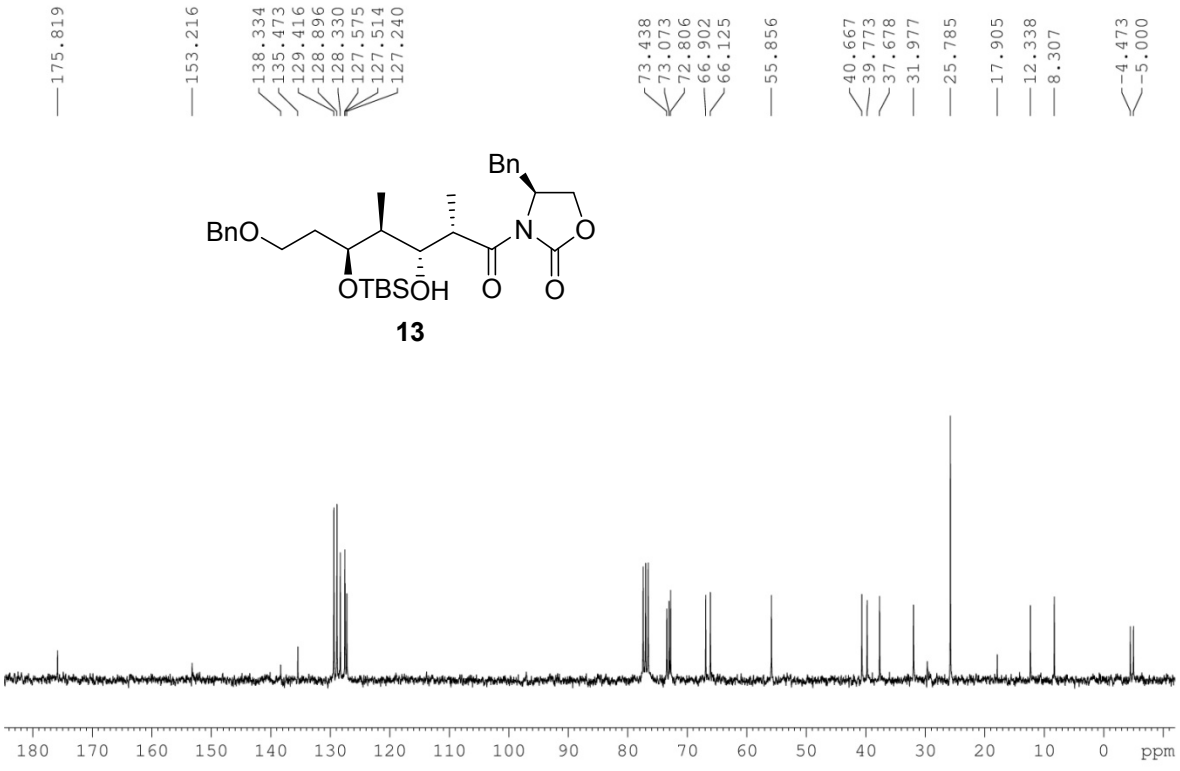
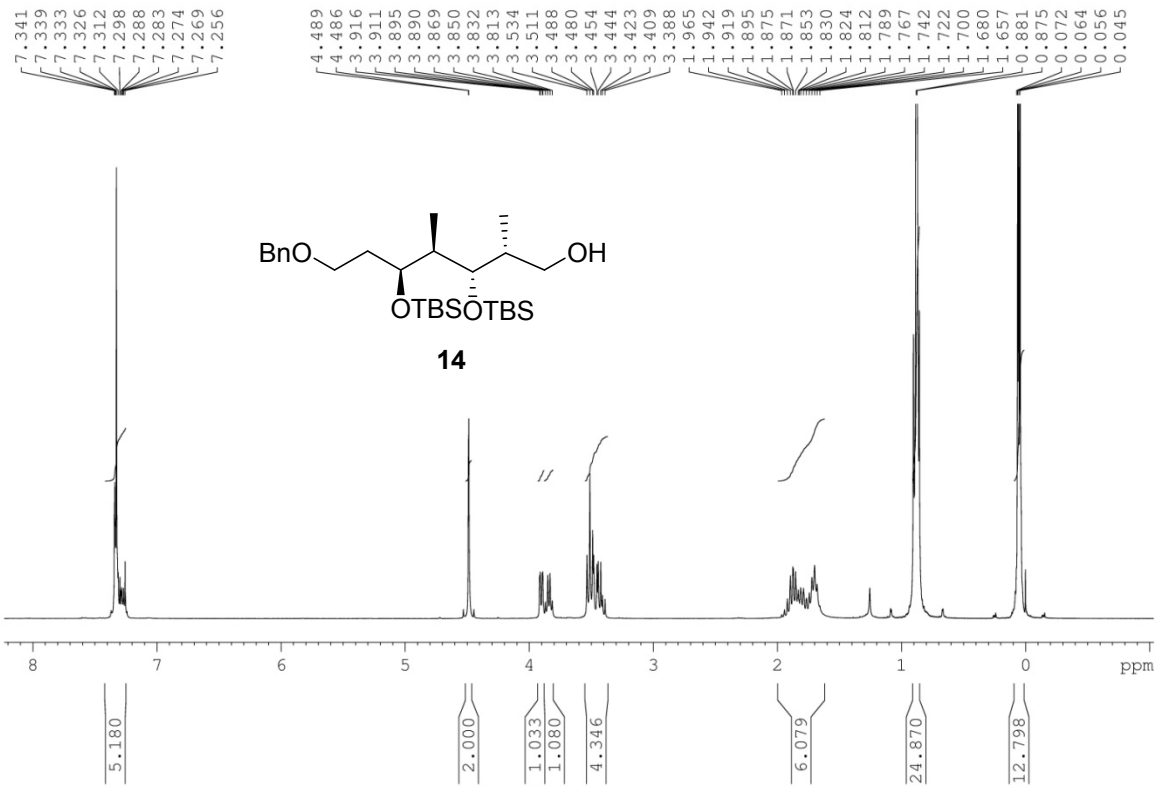


^{1}H NMR Spectrum of Compound 13 ($CDCl_3$, 300 MHz)



^{13}C NMR Spectrum of Compound 13 ($CDCl_3$, 75 MHz)



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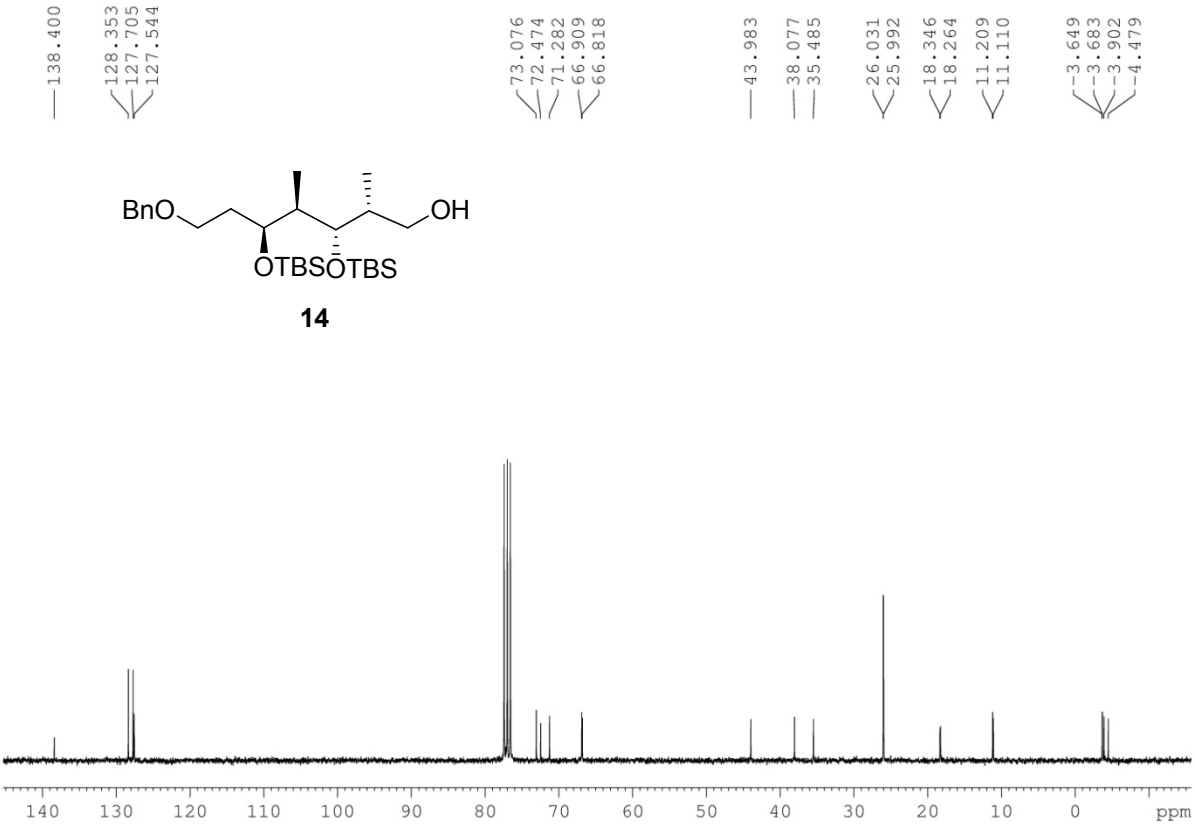
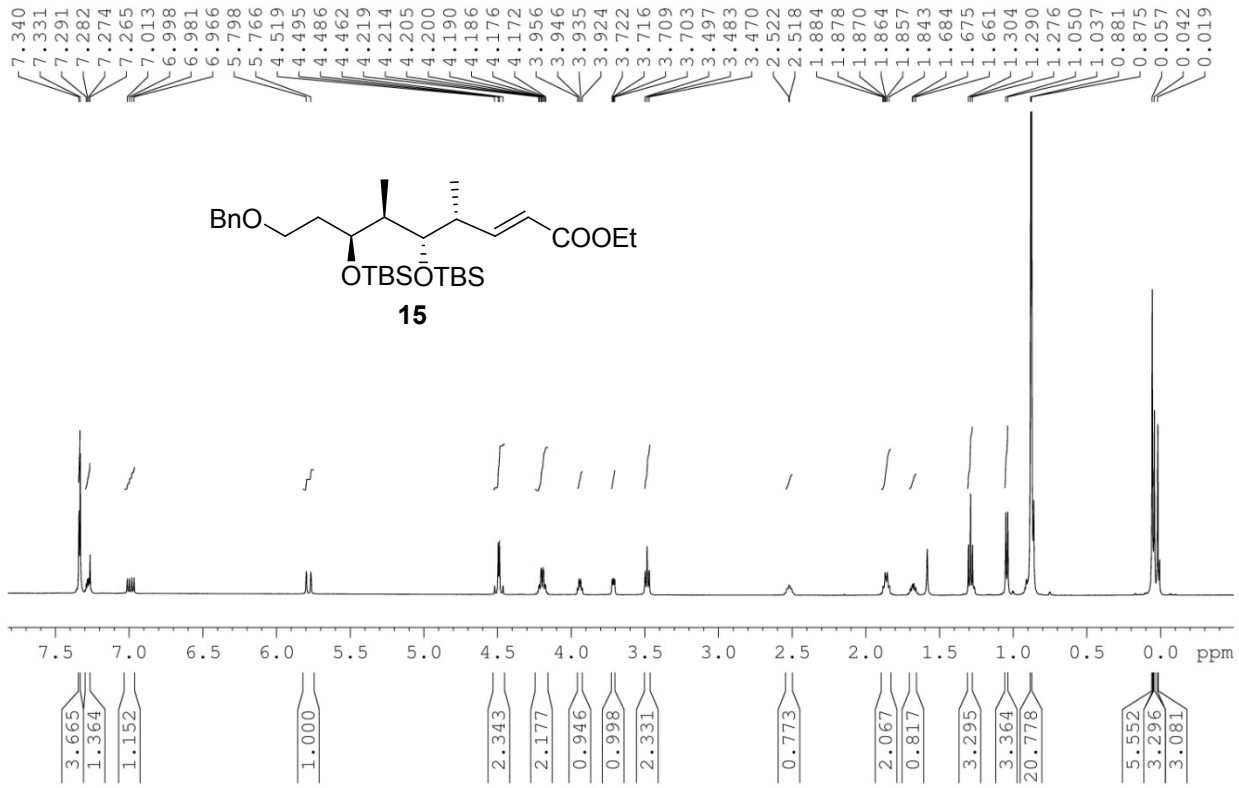
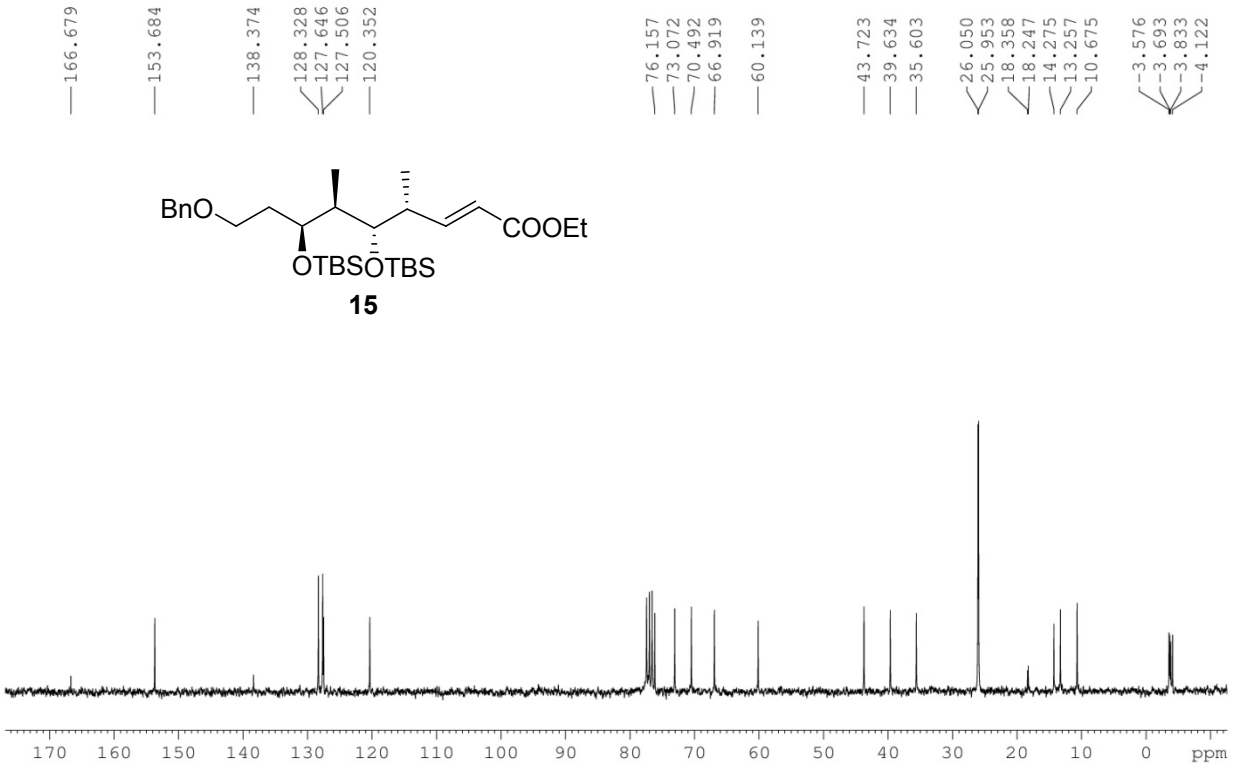


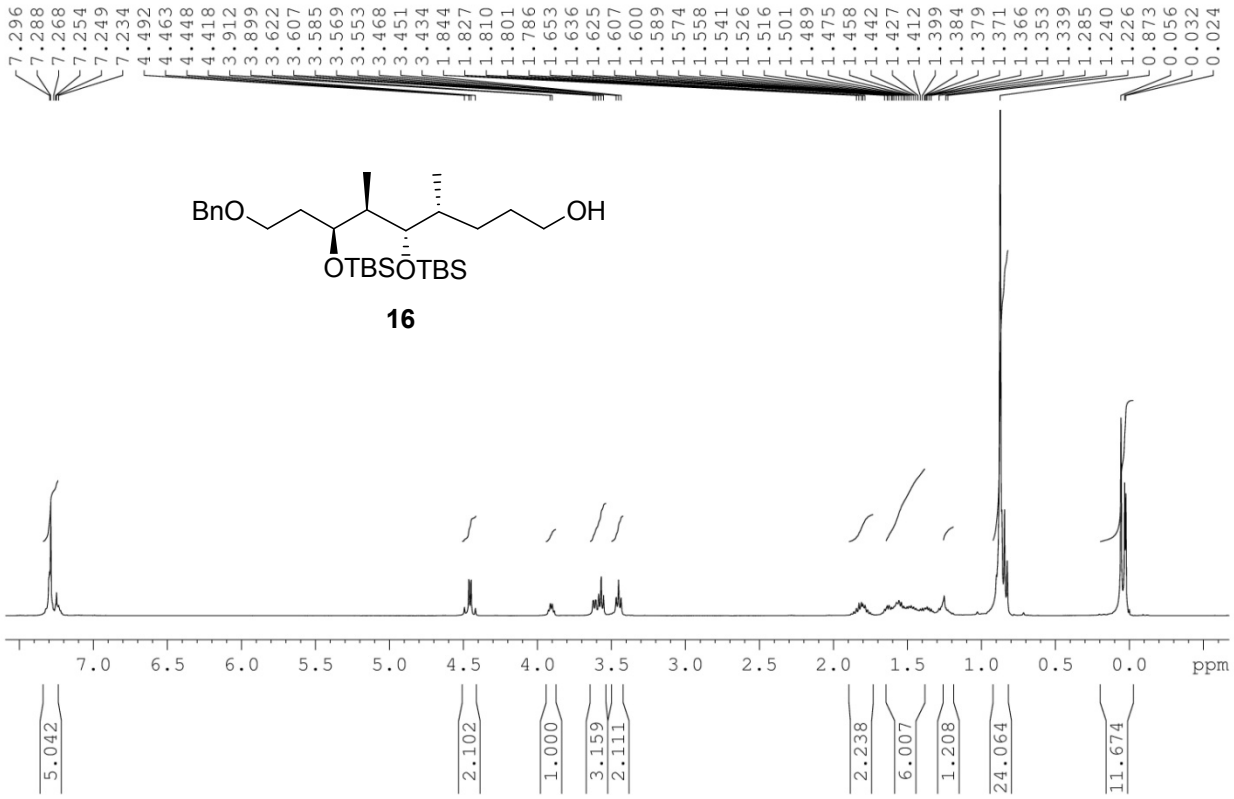
^{13}C NMR Spectrum of Compound 14 ($CDCl_3$, 75 MHz)



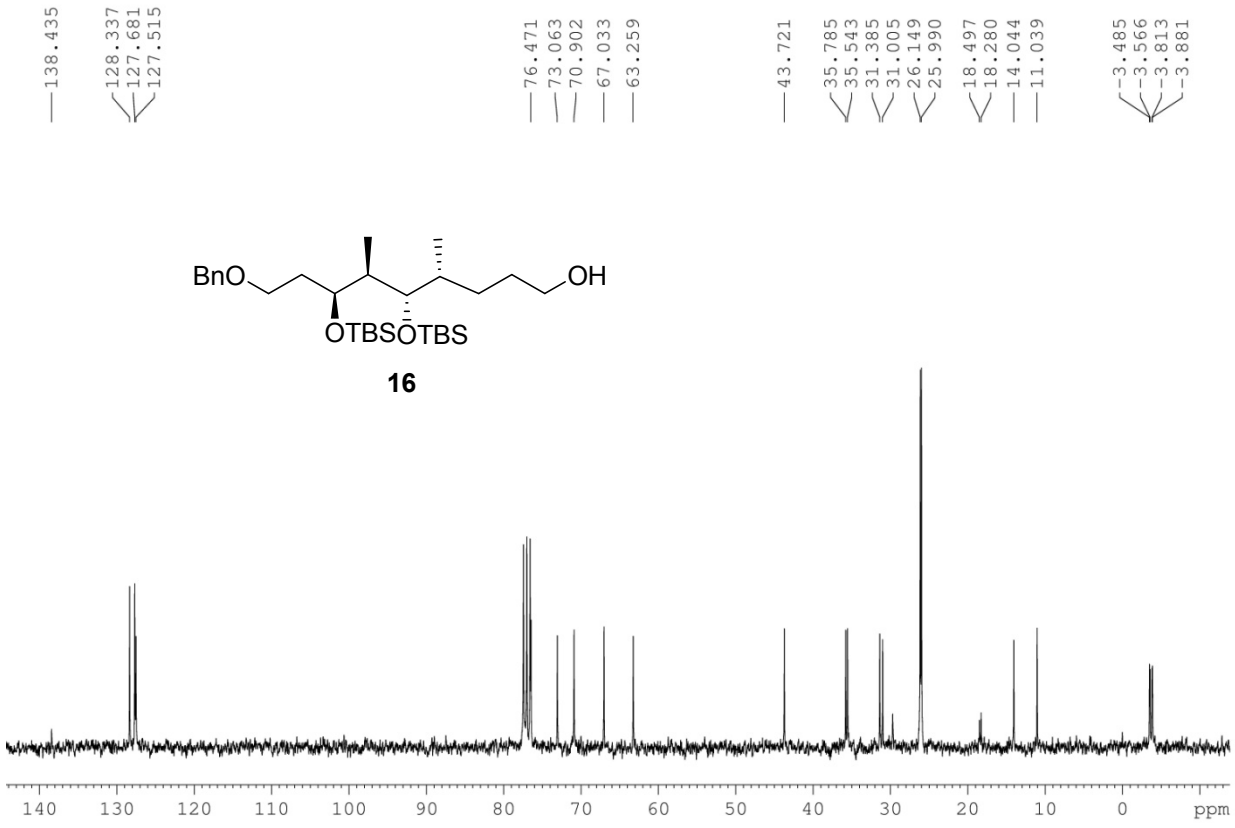
^{1}H NMR Spectrum of Compound 15 ($CDCl_3$, 500 MHz)



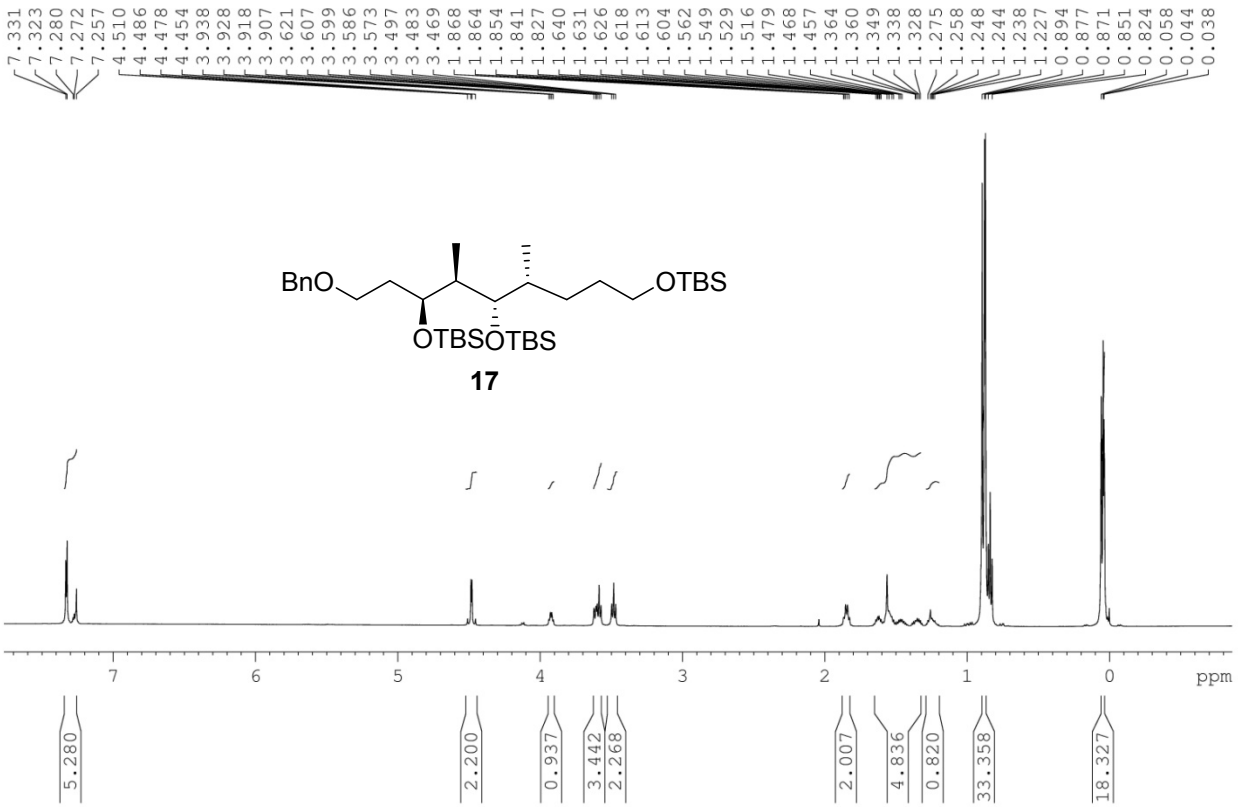
^{13}C NMR Spectrum of Compound 15 ($CDCl_3$, 75 MHz)



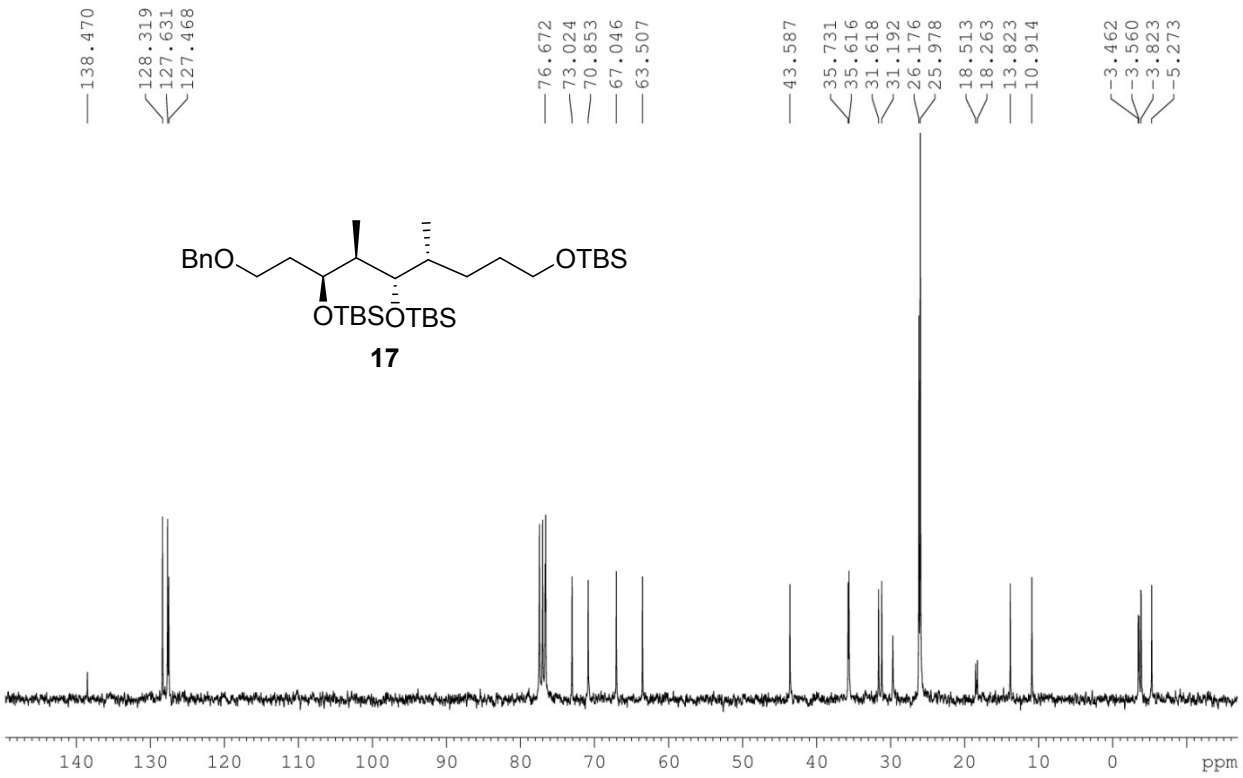
^{1}H NMR Spectrum of Compound 16 ($CDCl_3$, 400 MHz)



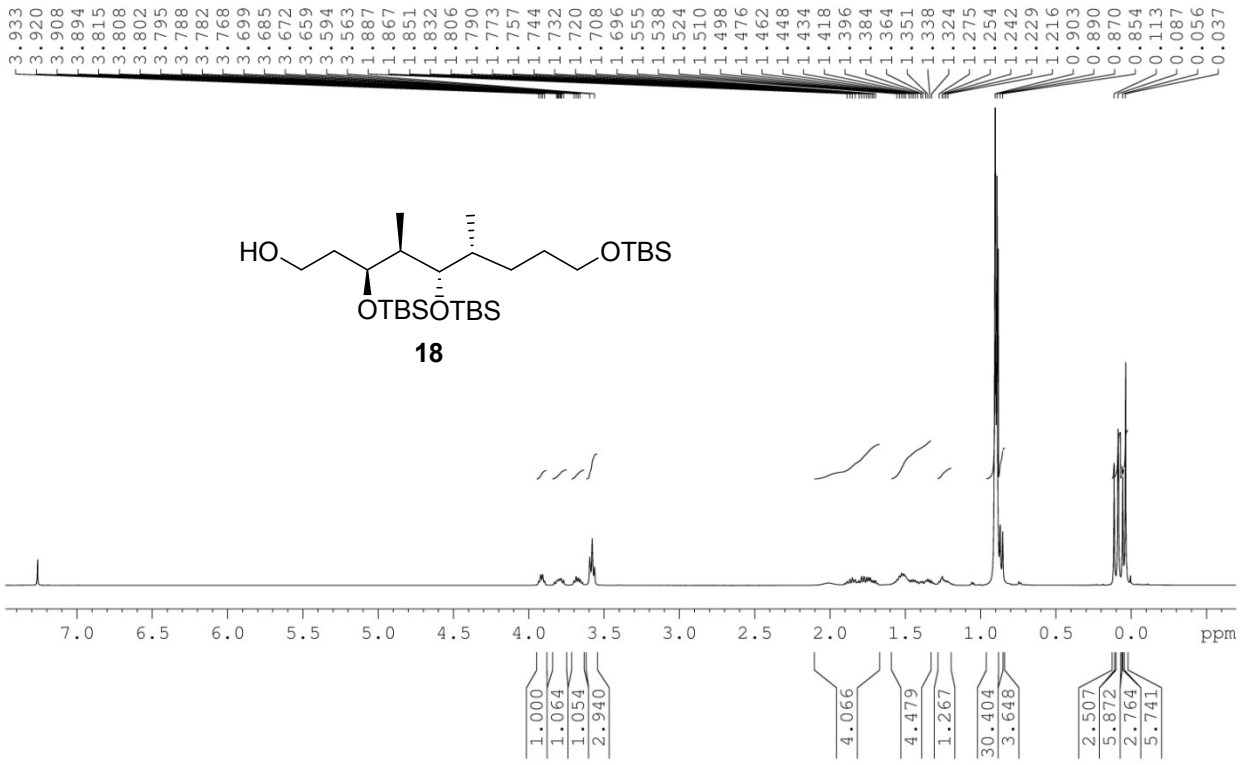
^{13}C NMR Spectrum of Compound 16 ($CDCl_3$, 75 MHz)



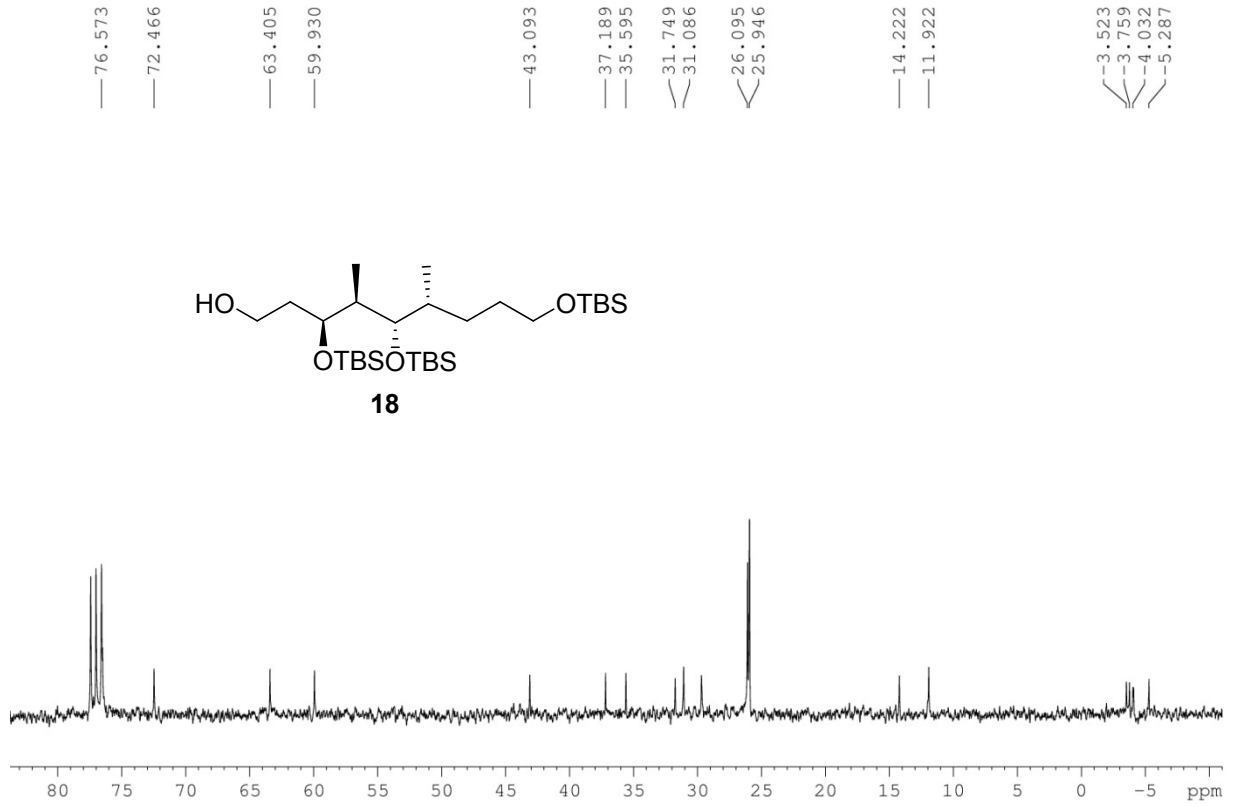
^{1}H NMR Spectrum of Compound 17 ($CDCl_3$, 500 MHz)



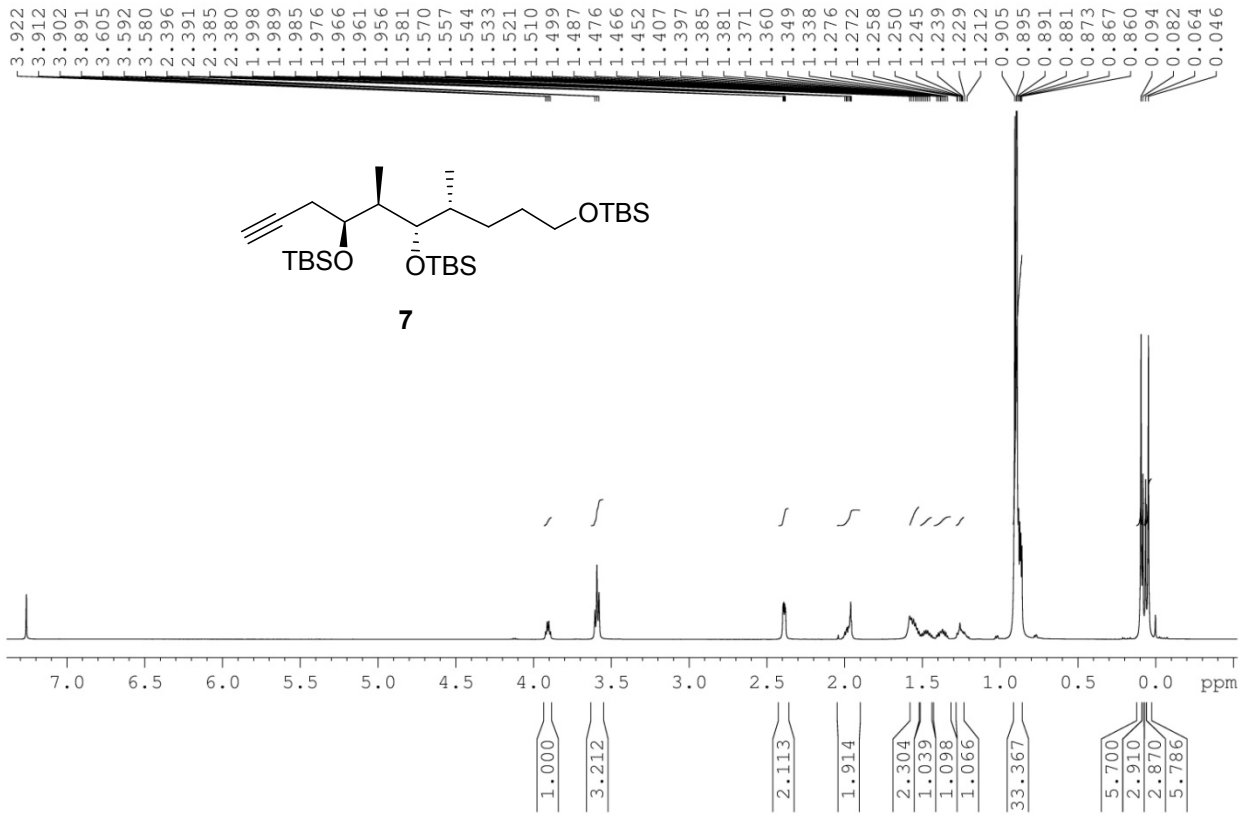
^{13}C NMR Spectrum of Compound 17 ($CDCl_3$, 75 MHz)



^{1}H NMR Spectrum of Compound 18 ($CDCl_3$, 400 MHz)



^{13}C NMR Spectrum of Compound 18 ($CDCl_3$, 75 MHz)



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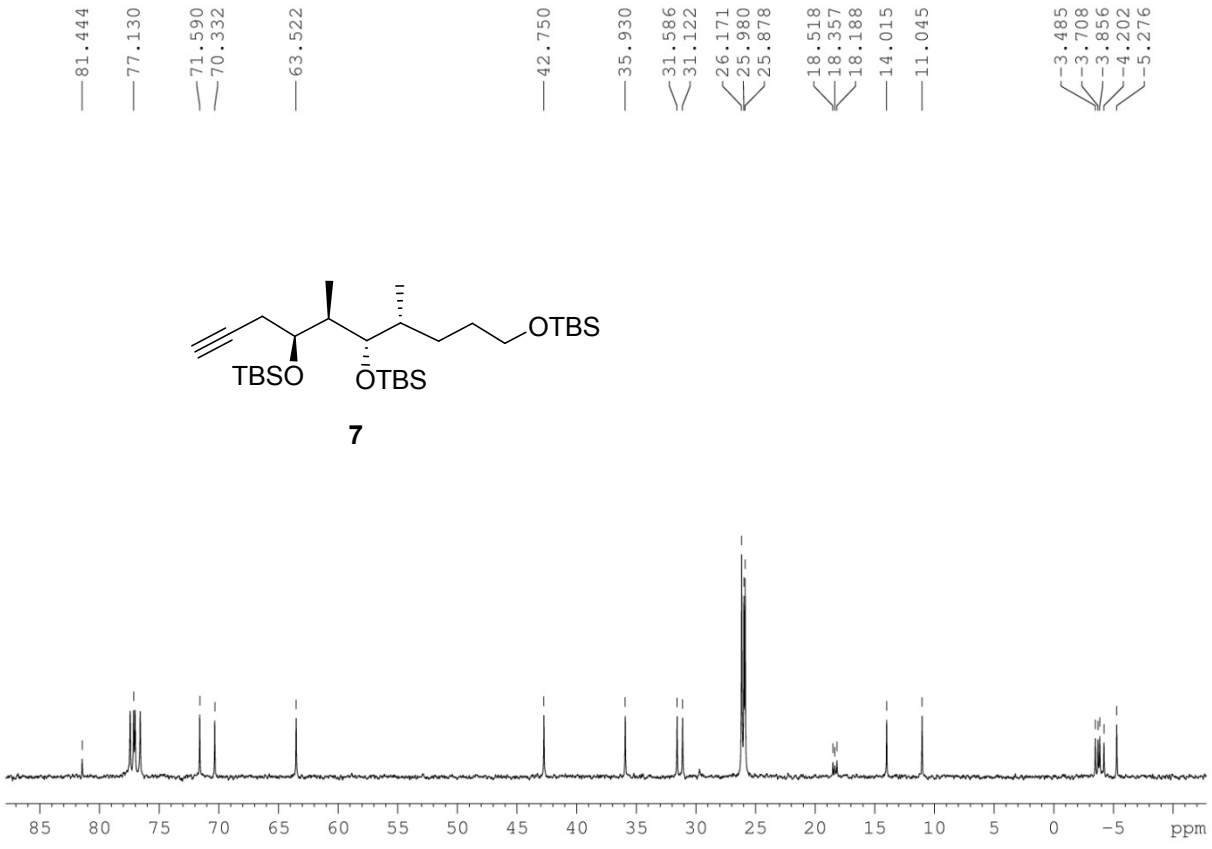
^{1}H NMR Spectrum of Compound 7 ($CDCl_3$, 500 MHz)



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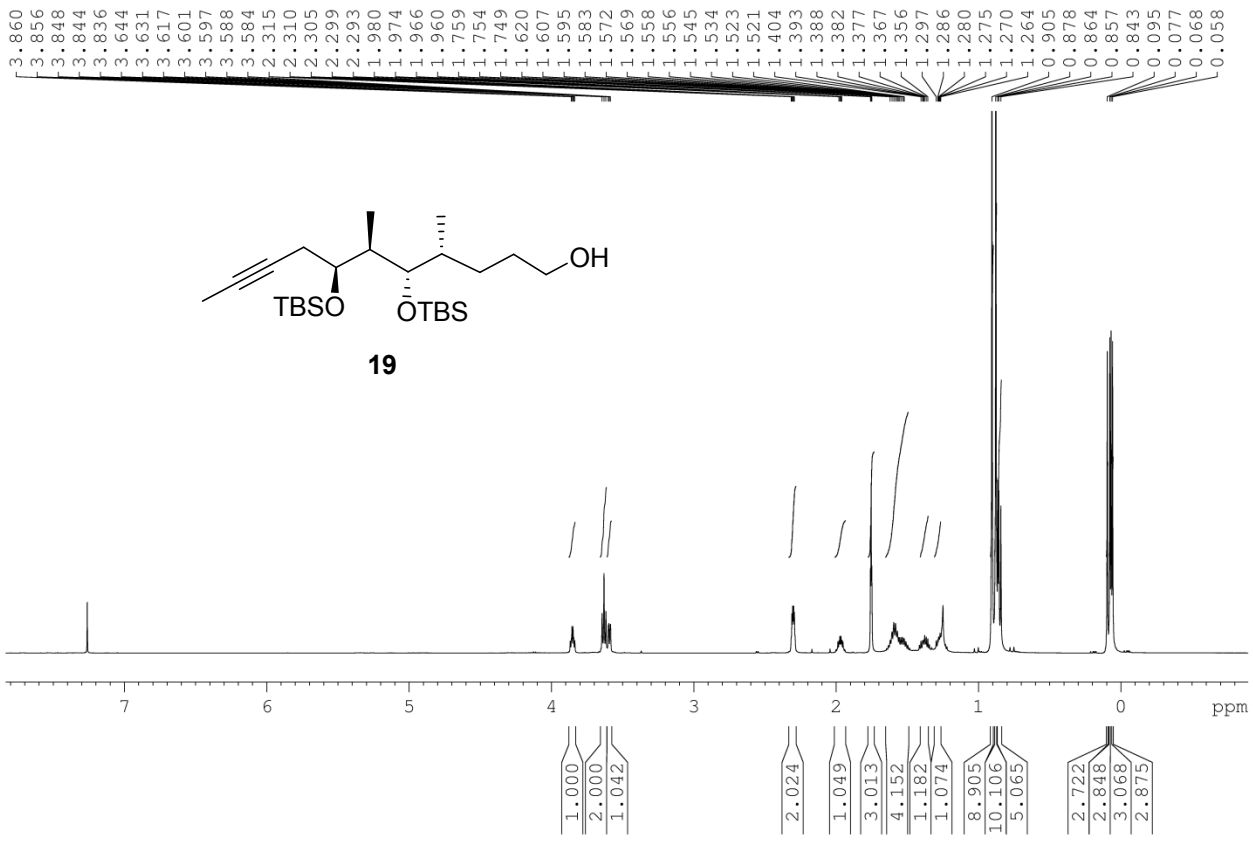
^{13}C NMR Spectrum of Compound 7 ($CDCl_3$, 75 MHz)



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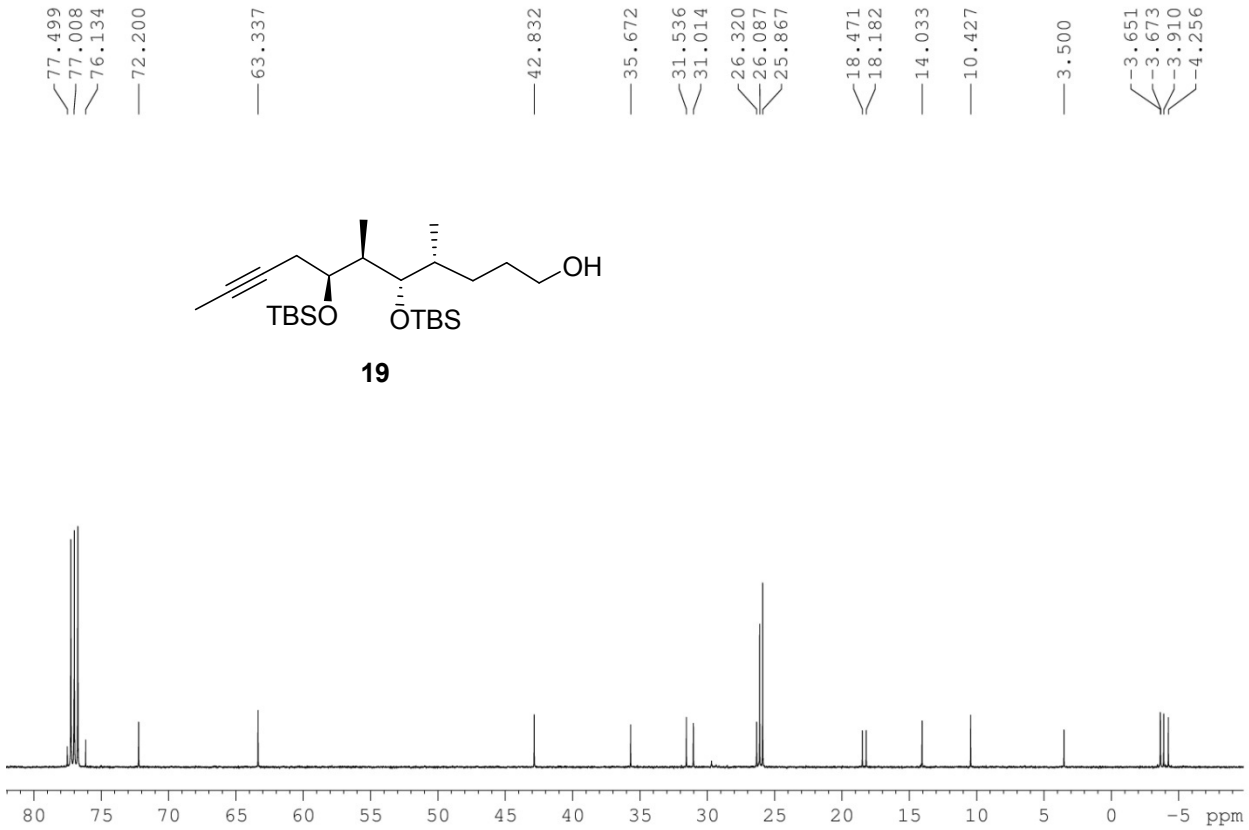
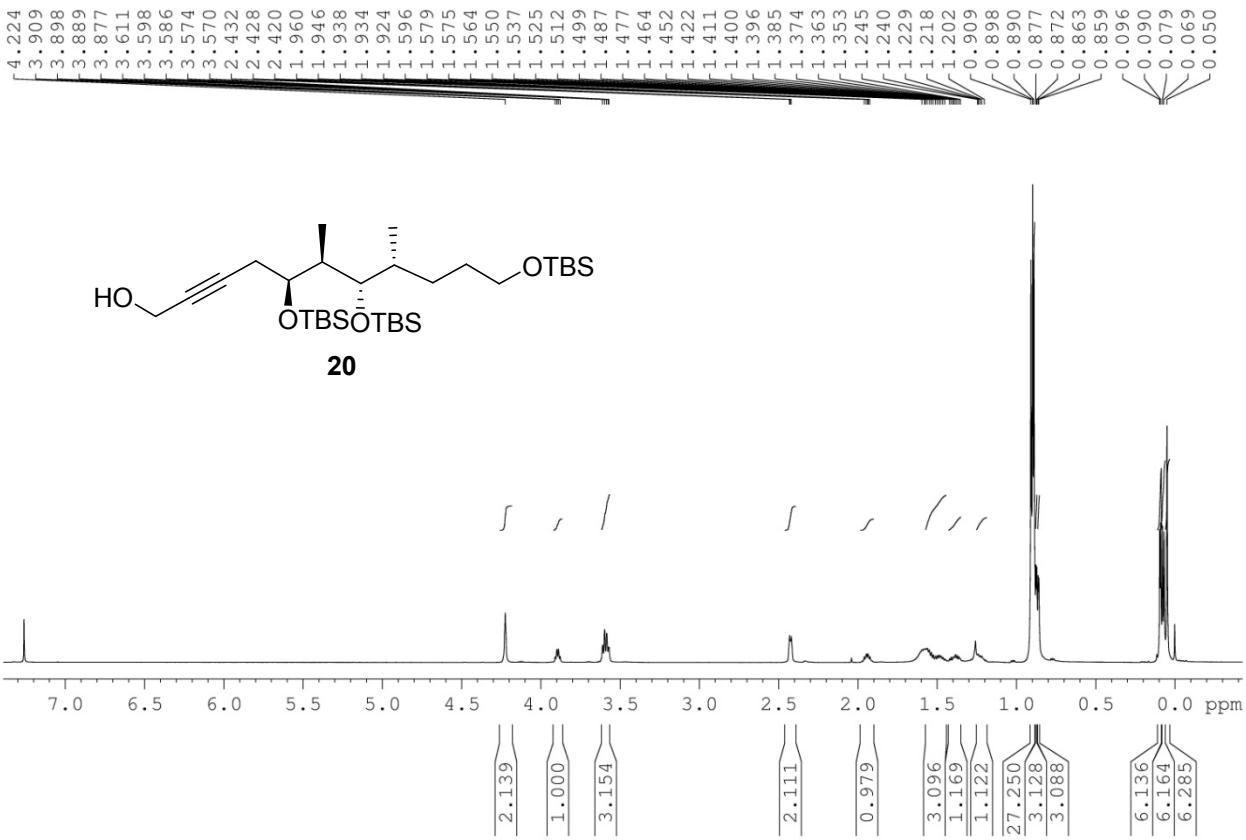


^{13}C NMR Spectrum of Compound 19 ($CDCl_3$, 125 MHz)



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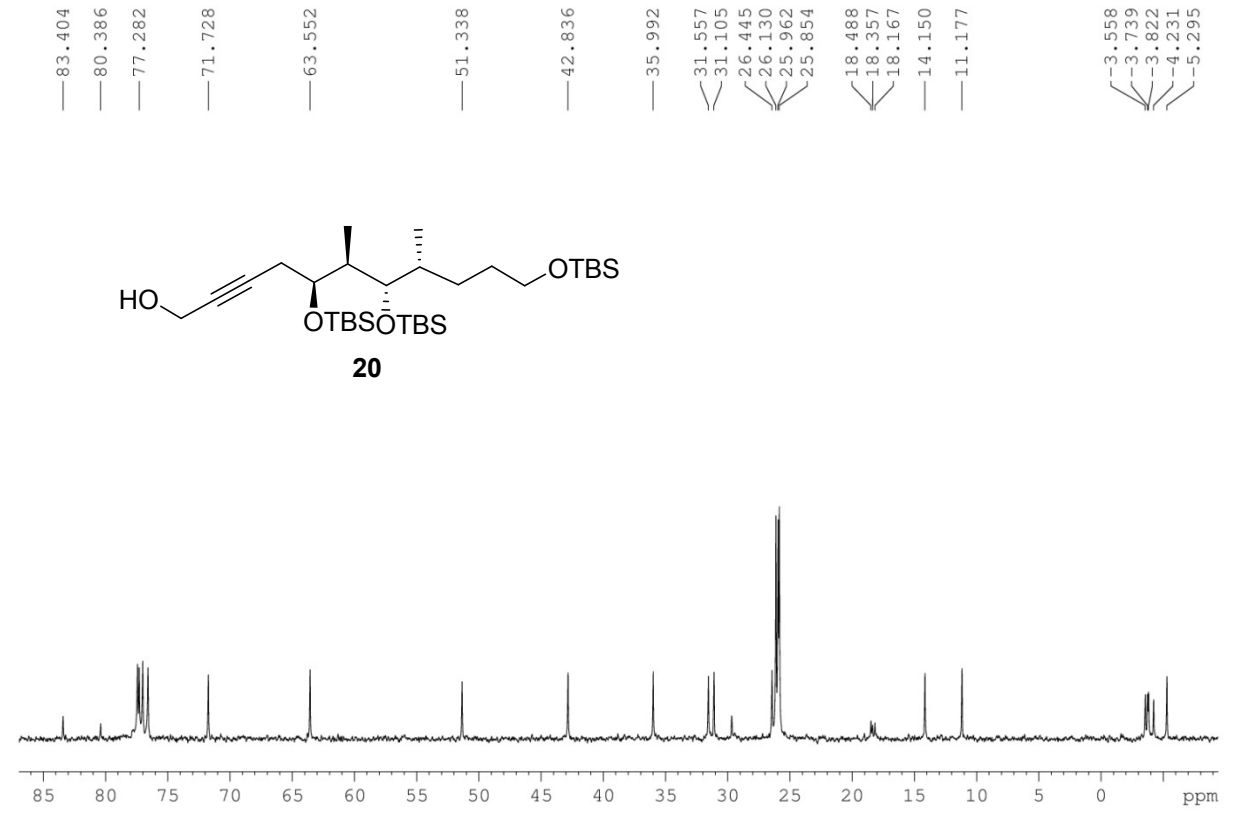
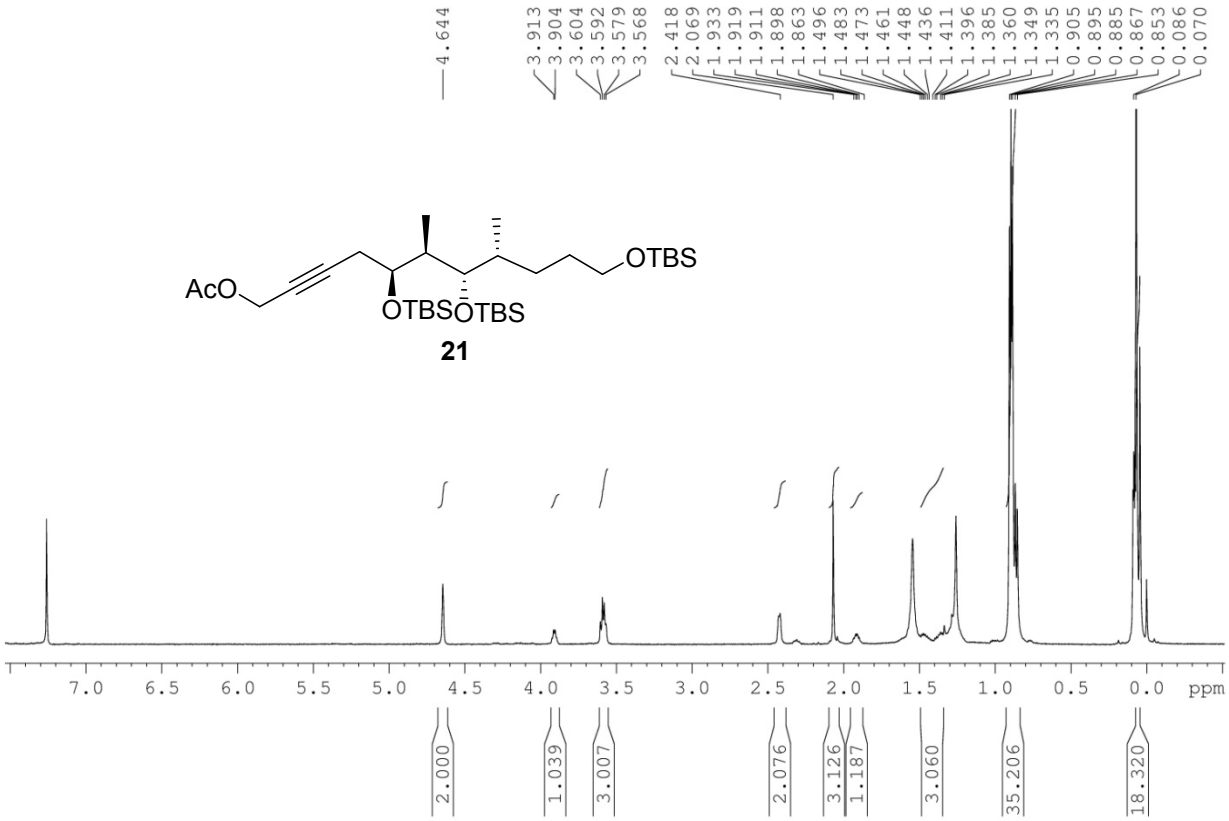


^{13}C NMR Spectrum of Compound 20 ($CDCl_3$, 75 MHz)



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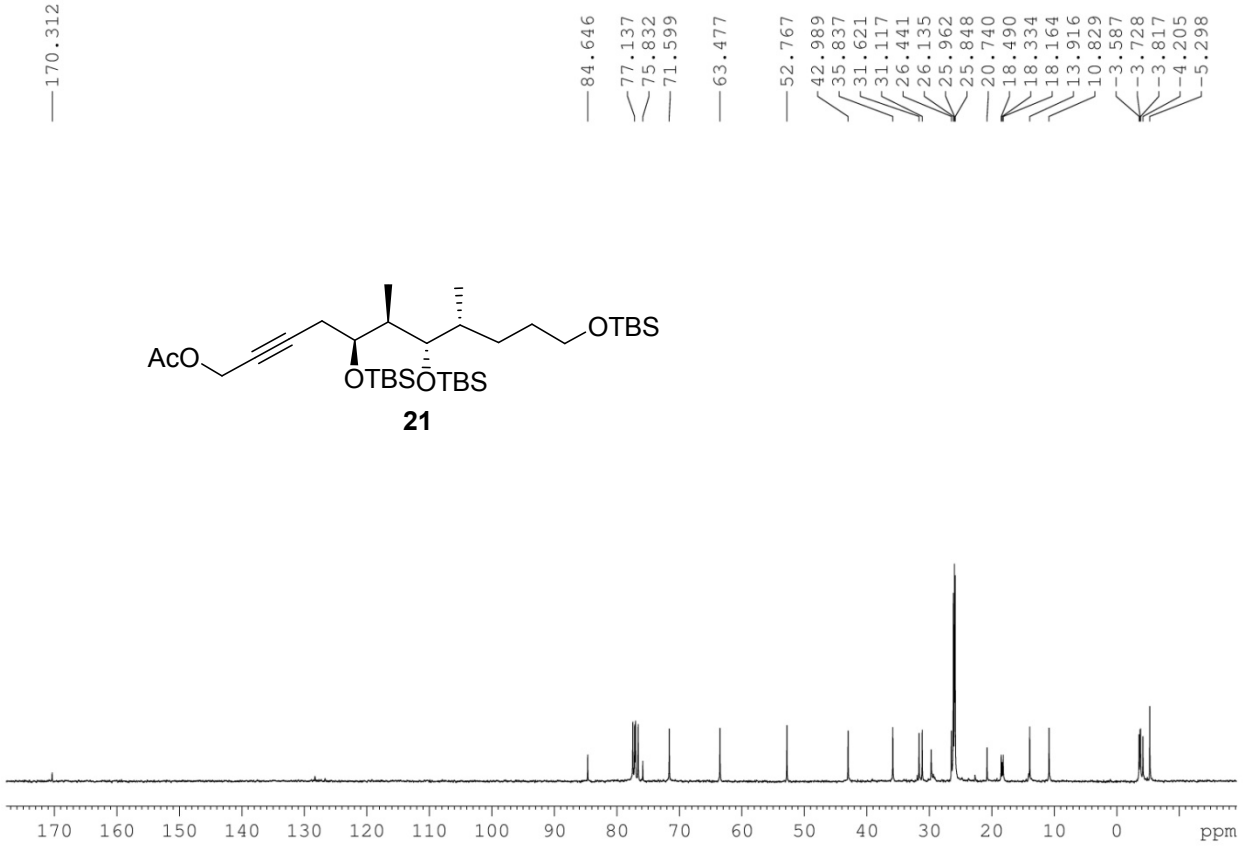
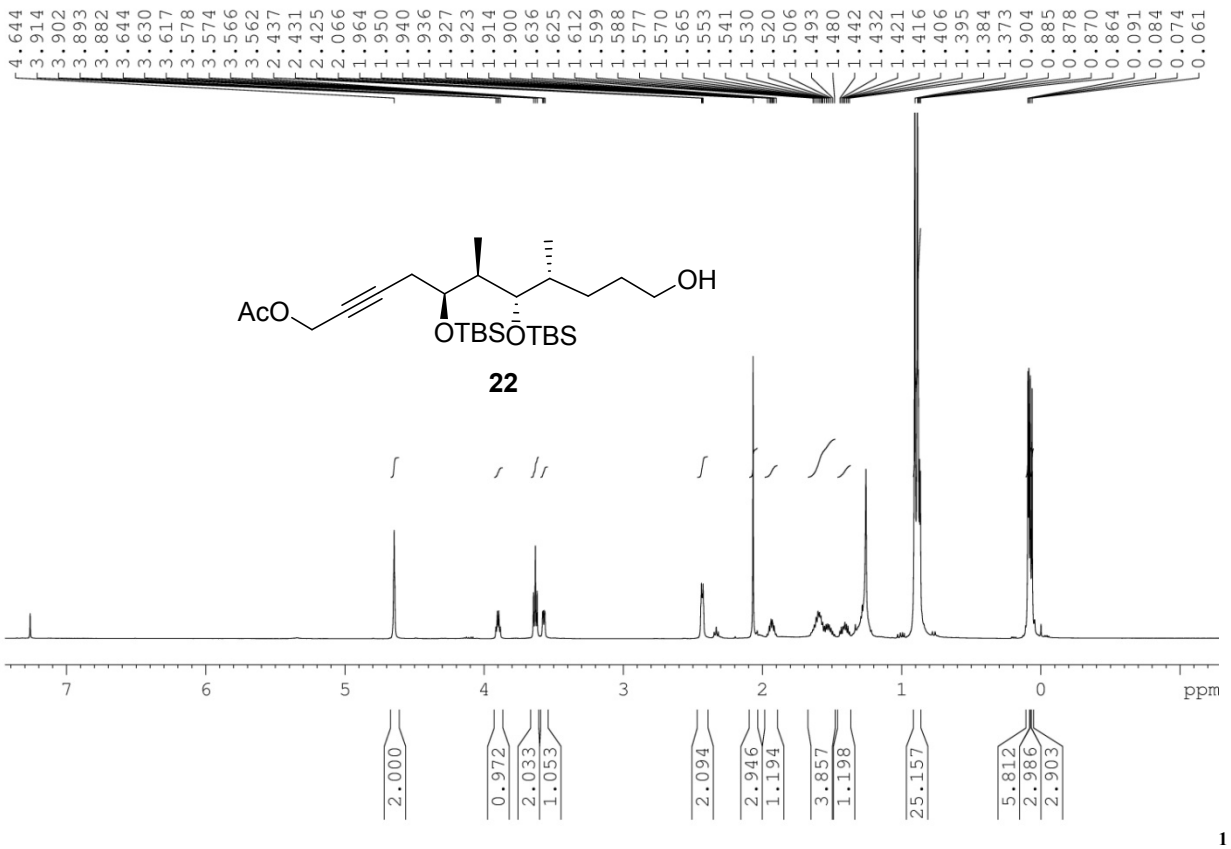
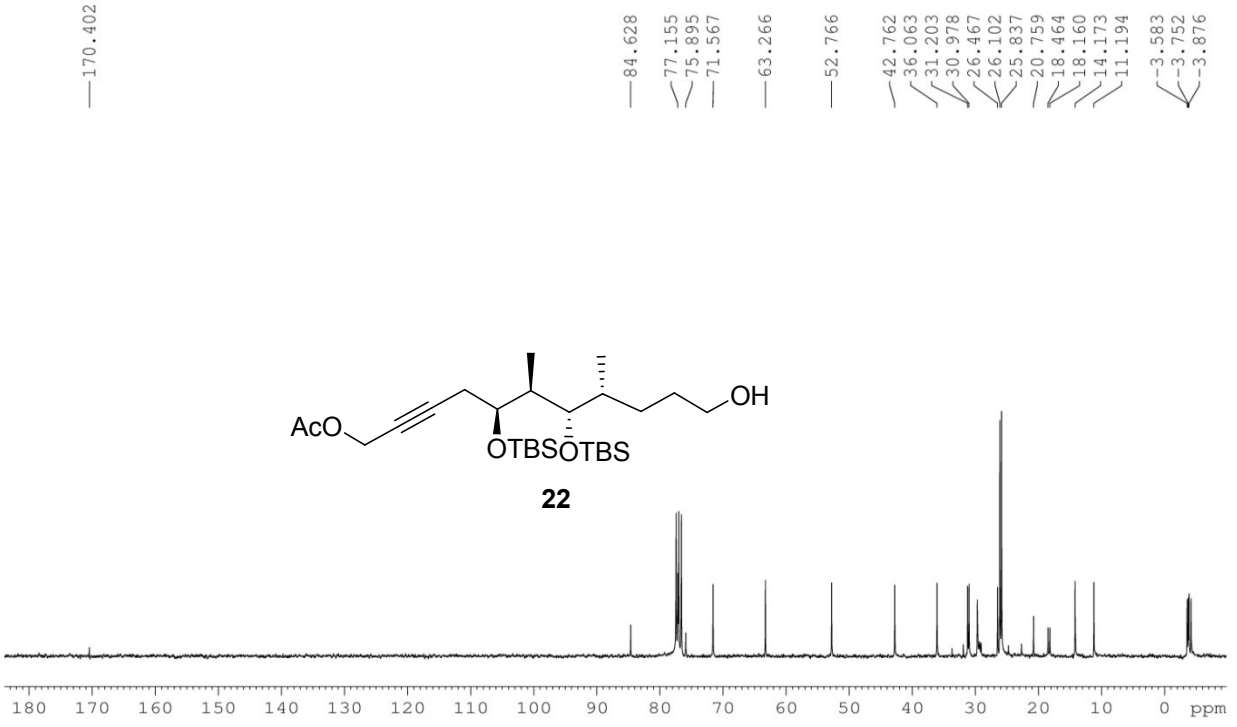


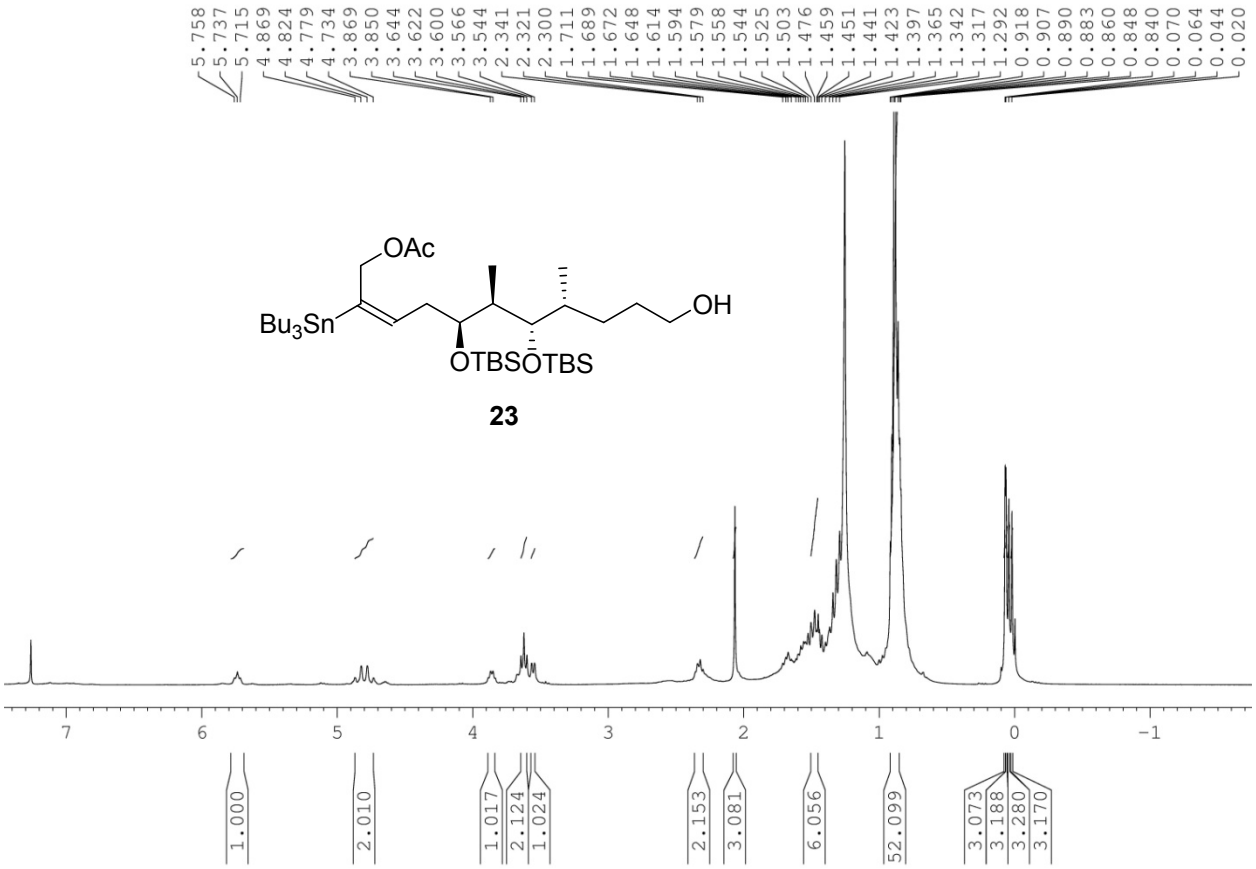
^{13}C NMR Spectrum of Compound 21 ($CDCl_3$, 75 MHz)



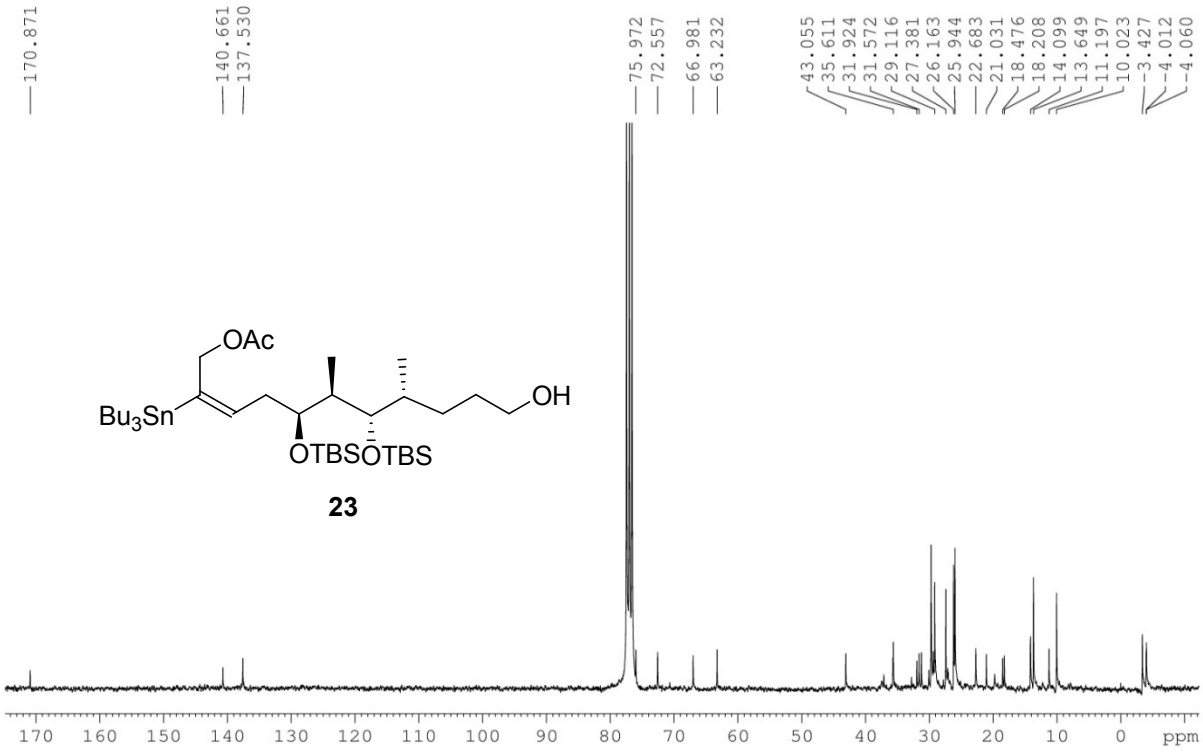
H NMR Spectrum of Compound 22 ($CDCl_3$, 500 MHz)



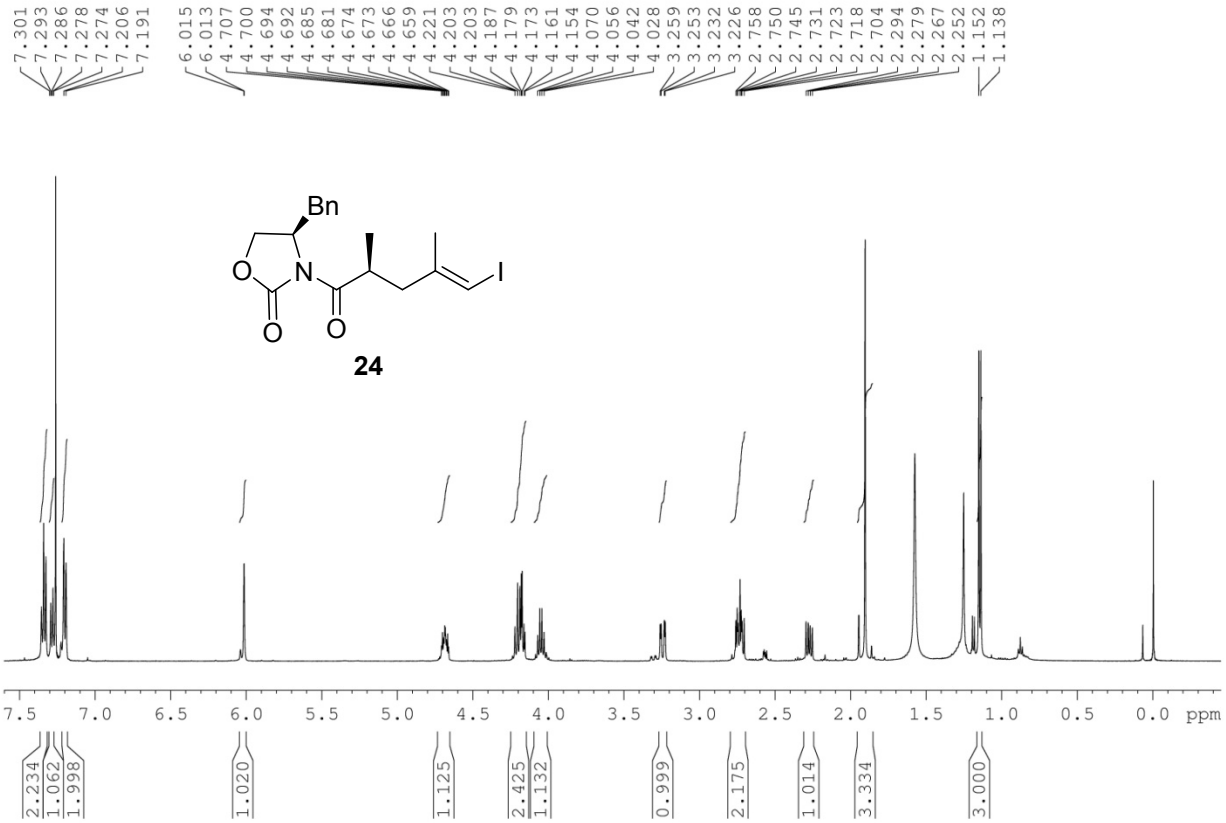
^{13}C NMR Spectrum of Compound 22 ($CDCl_3$, 75 MHz)



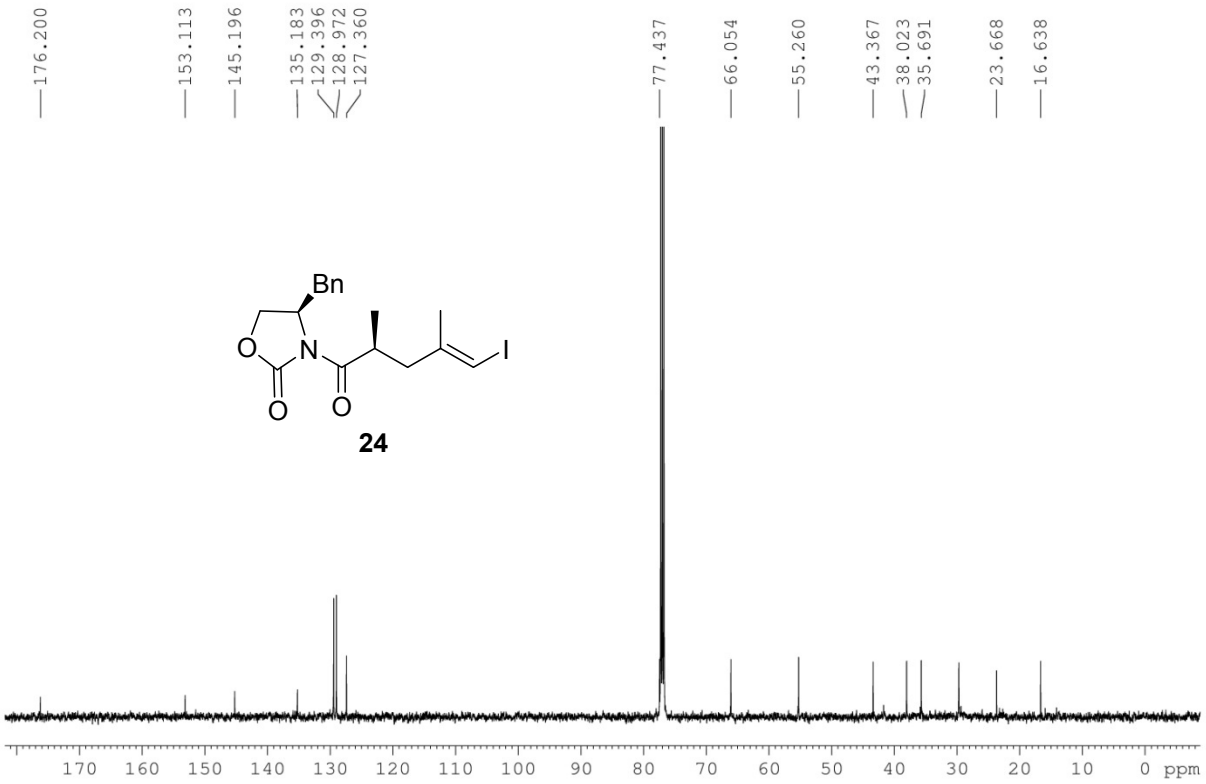
^{1}H NMR Spectrum of compound 23 ($CDCl_3$, 300 MHz)



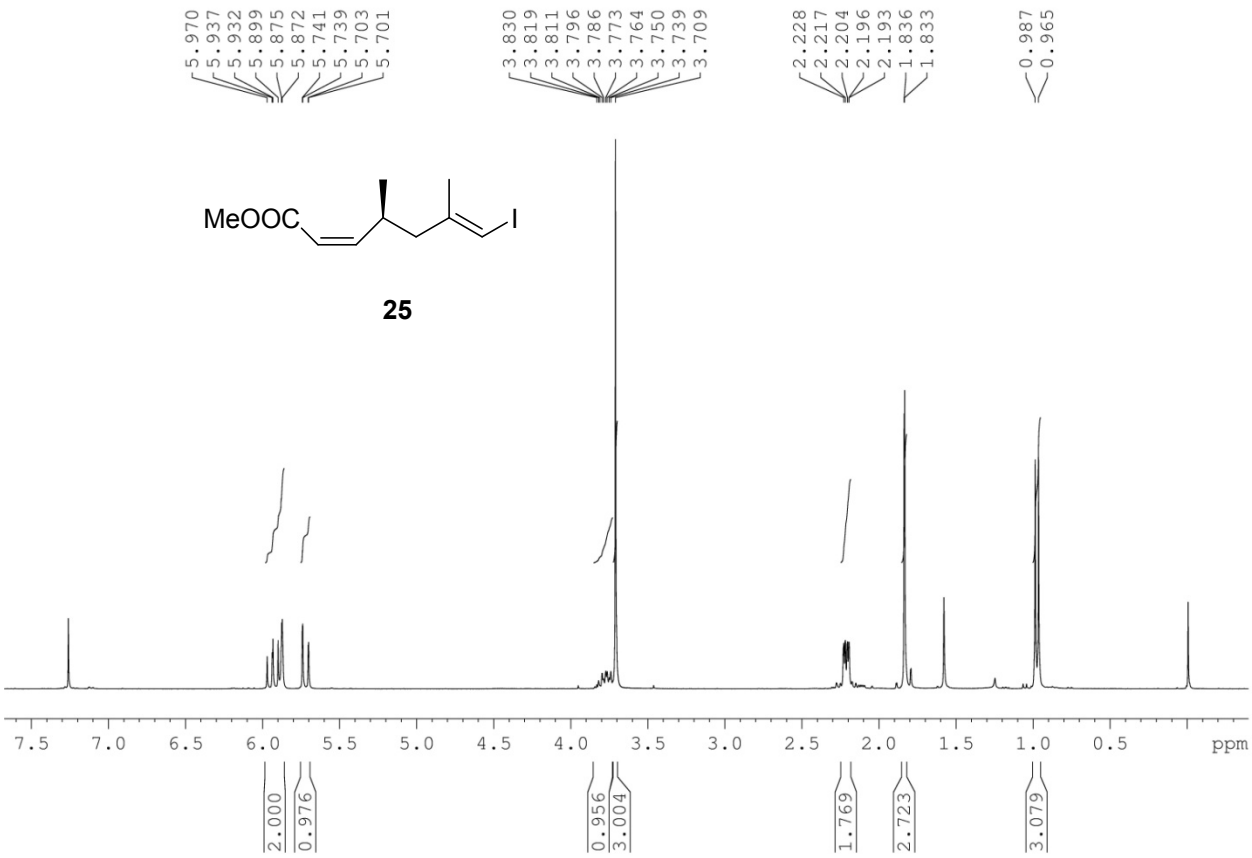
^{13}C NMR Spectrum of compound 23 ($CDCl_3$, 75 MHz)



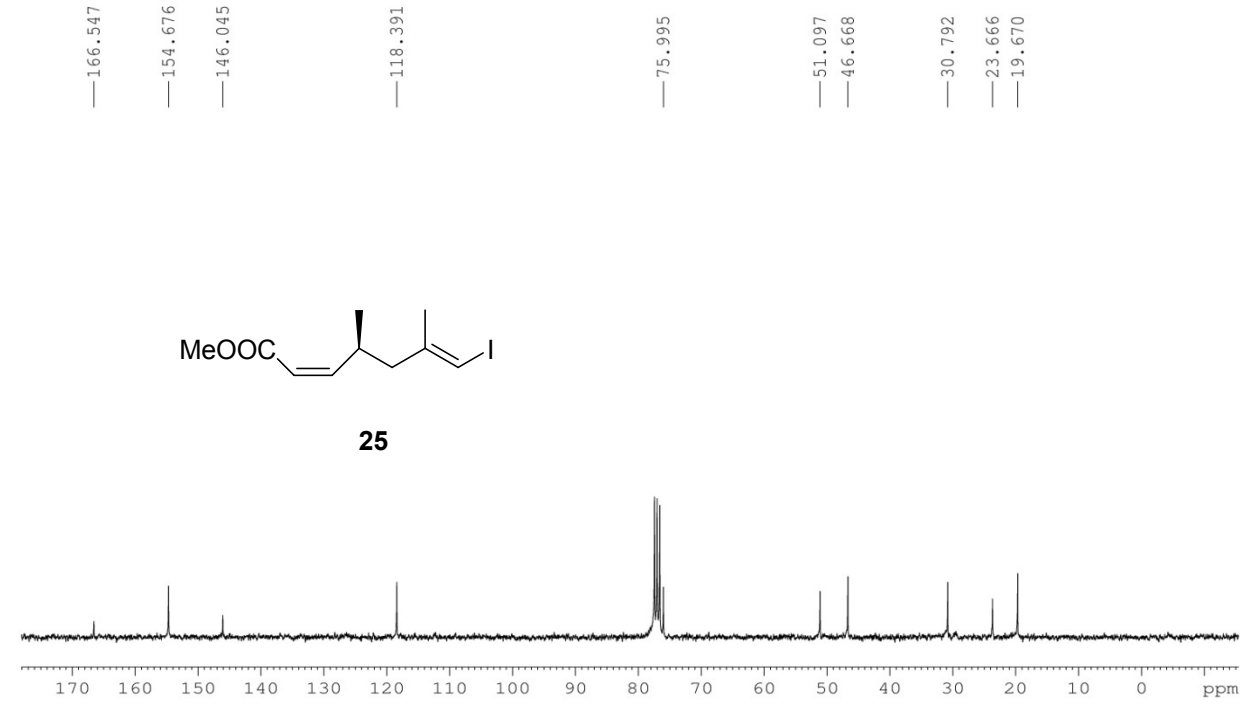
^{1}H NMR Spectrum of Compound 24 ($CDCl_3$, 300 MHz)



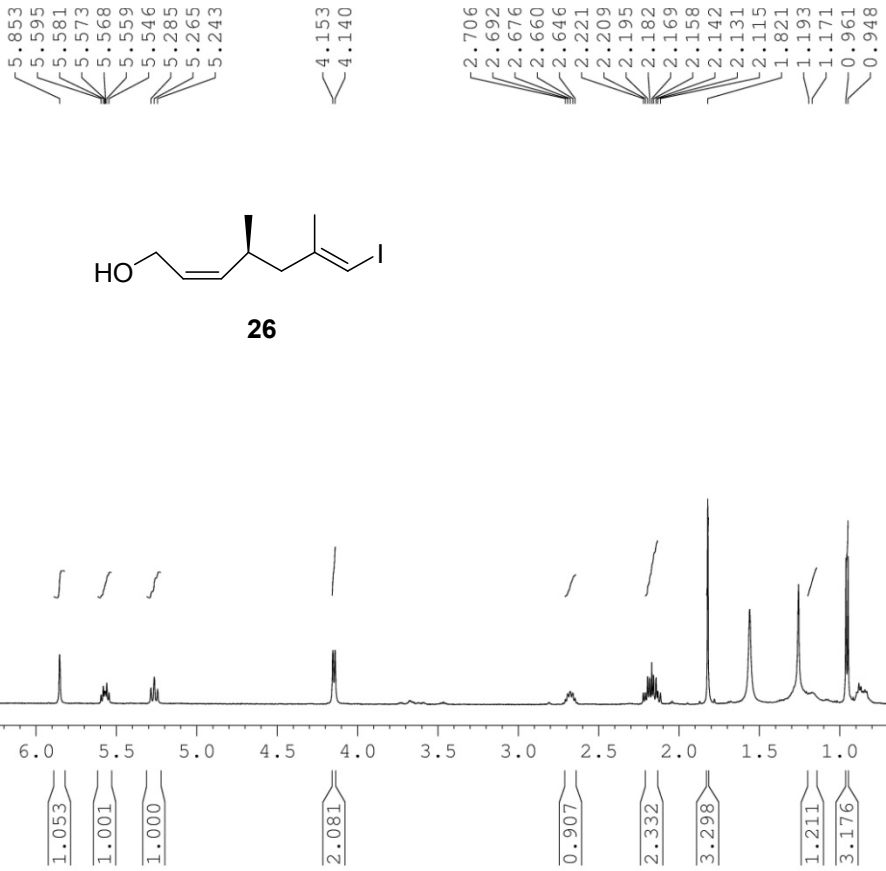
^{13}C NMR Spectrum of Compound 24 ($CDCl_3$, 125 MHz)



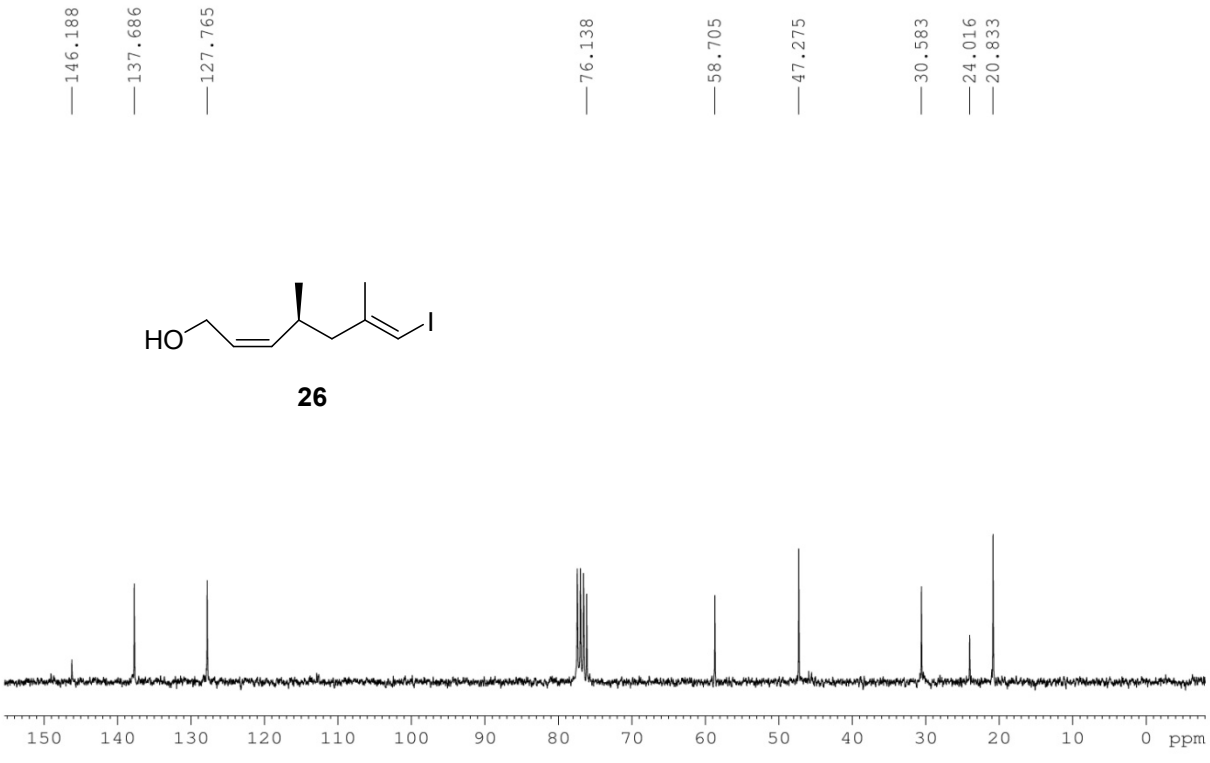
^{1}H NMR Spectrum of Compound 25 ($CDCl_3$, 300 MHz)



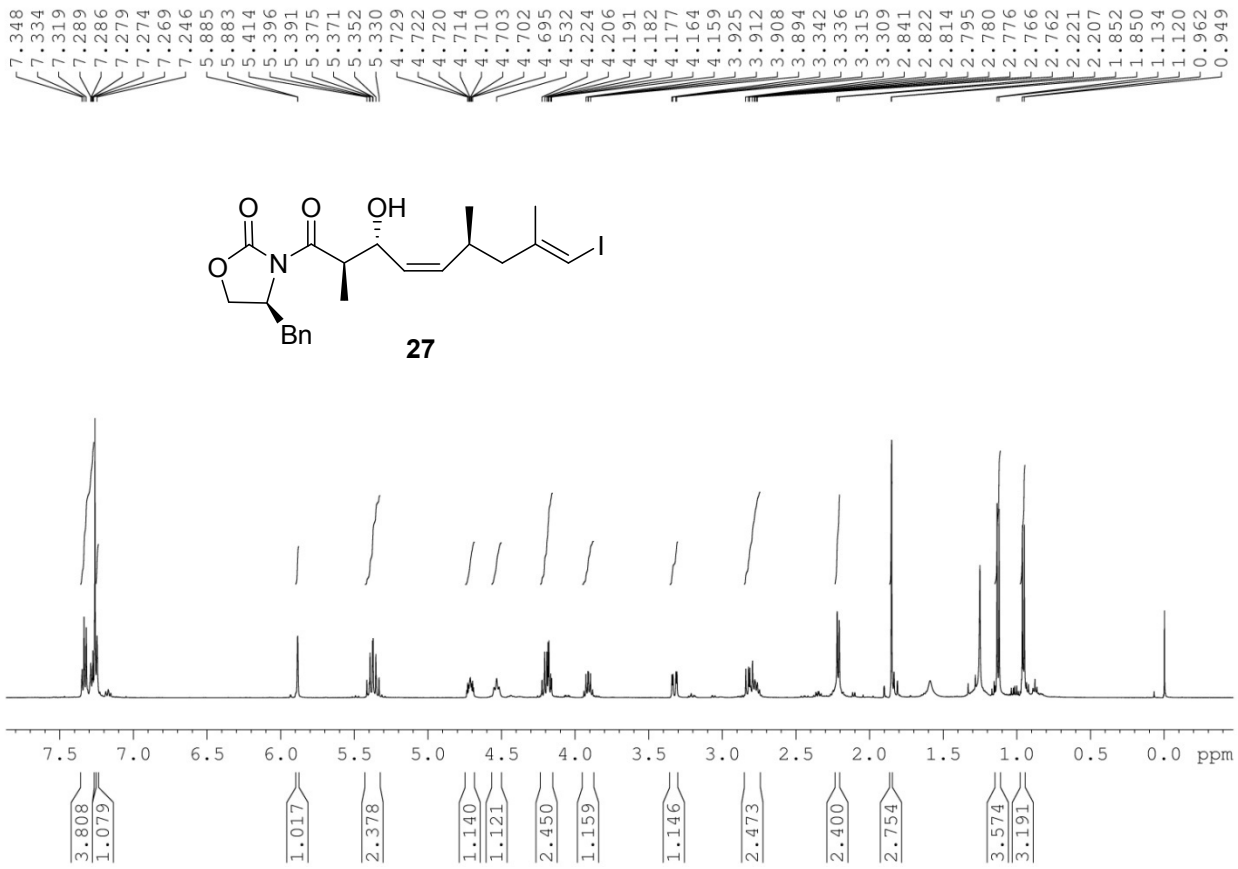
^{13}C NMR Spectrum of Compound 25 ($CDCl_3$, 75 MHz)



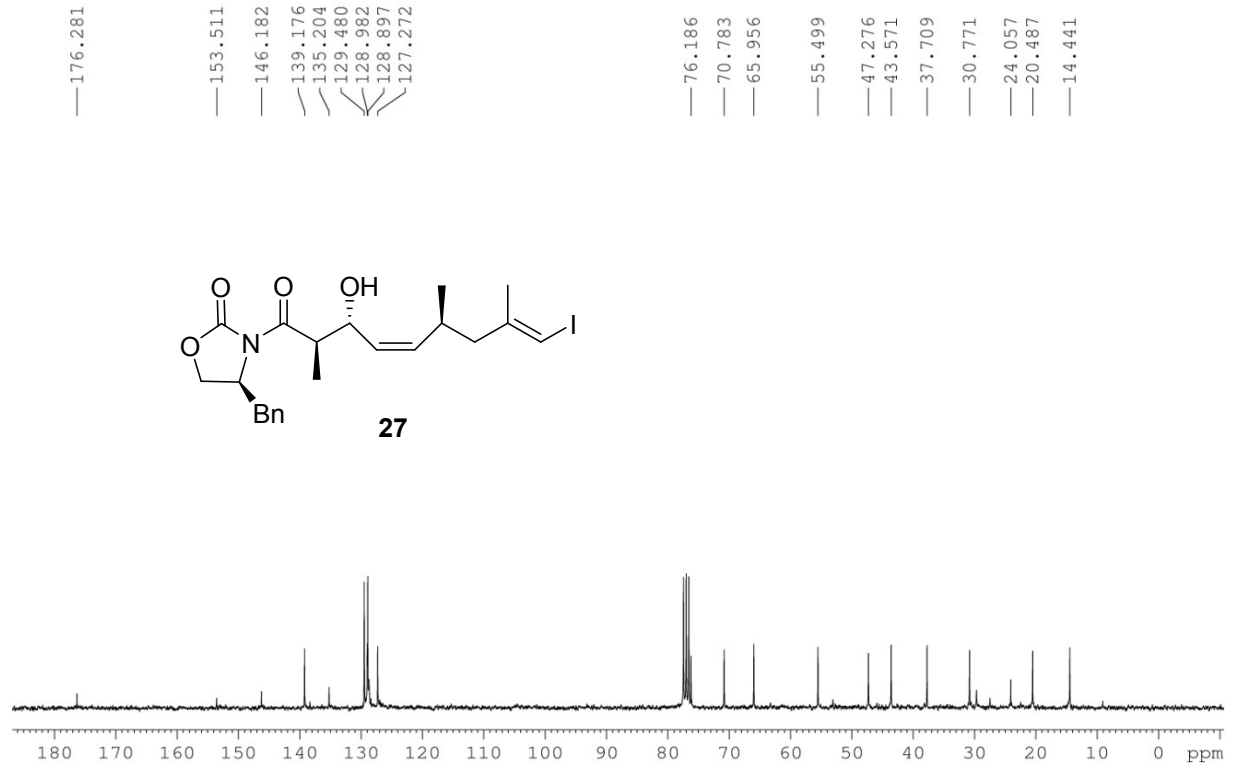
^{1}H NMR Spectrum of Compound 26 ($CDCl_3$, 500 MHz)



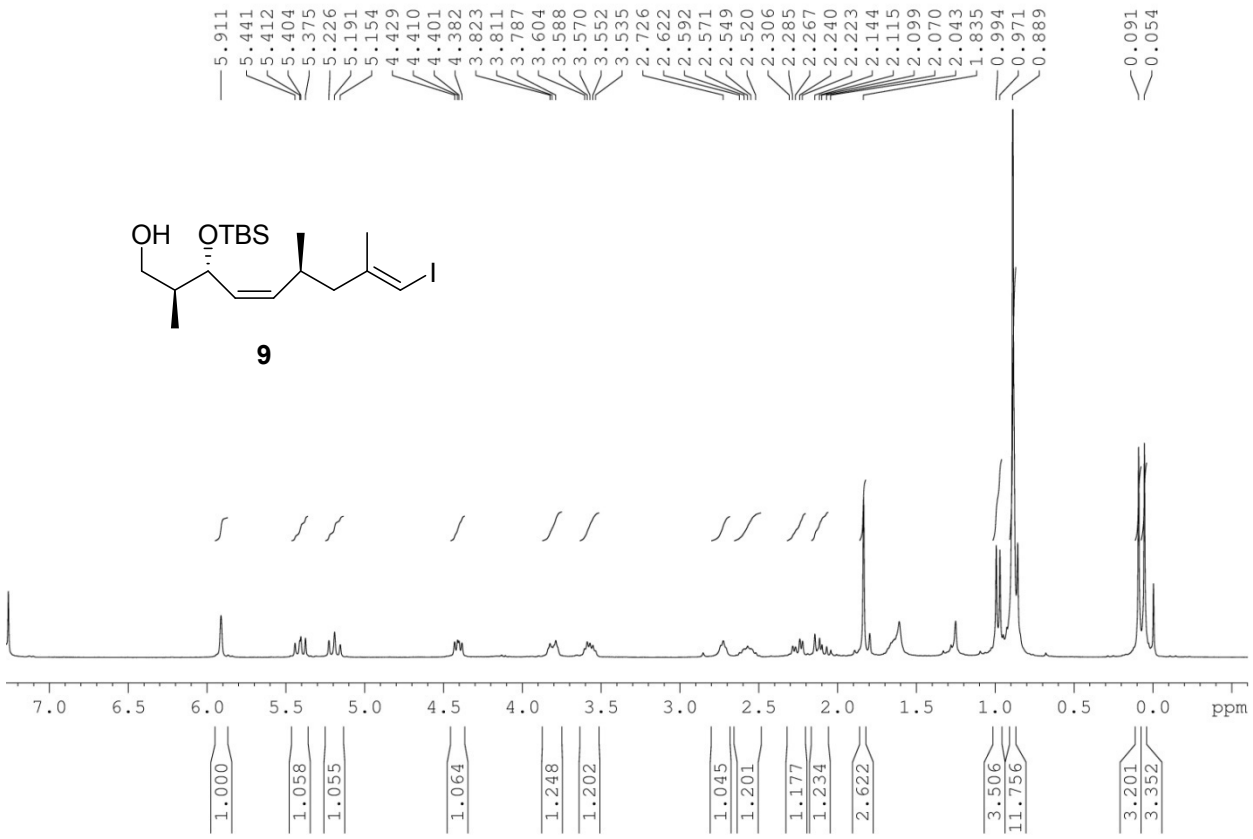
^{13}C NMR Spectrum of Compound 26 ($CDCl_3$, 75 MHz)



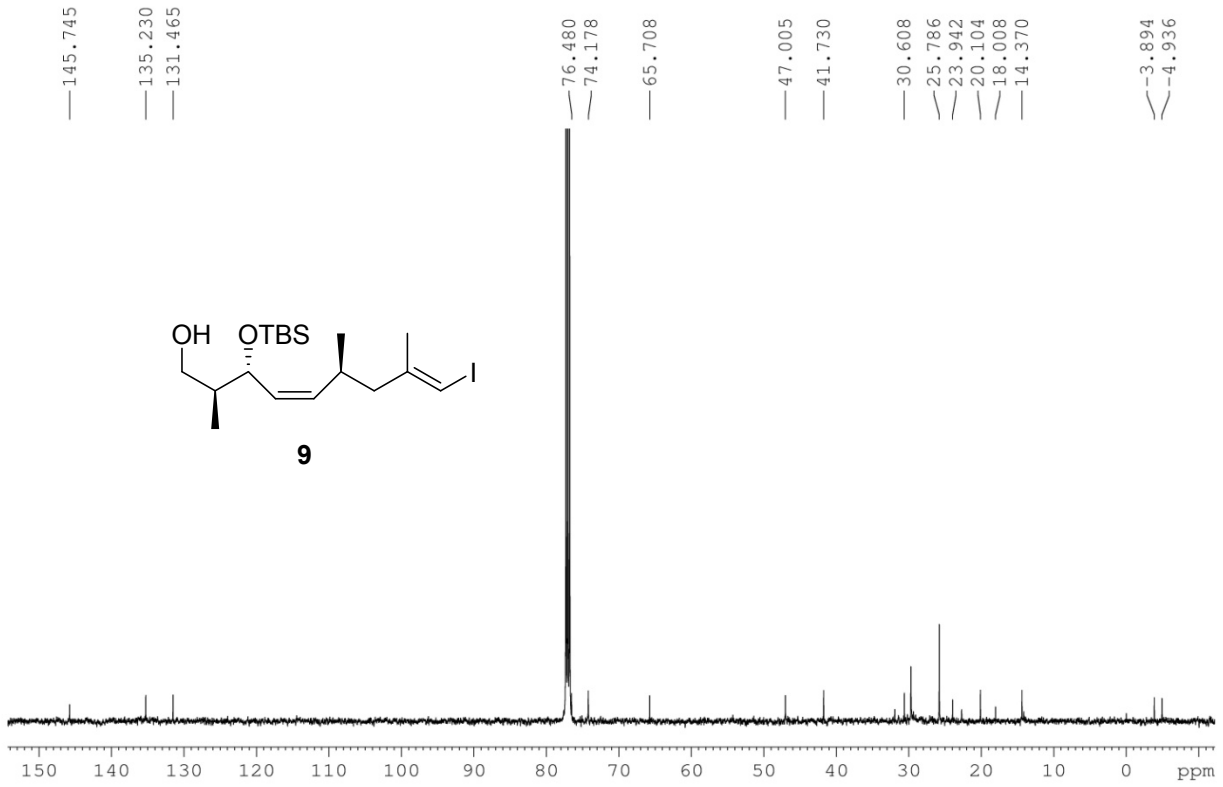
^{1}H NMR Spectrum of Compound 27 ($CDCl_3$, 300 MHz)



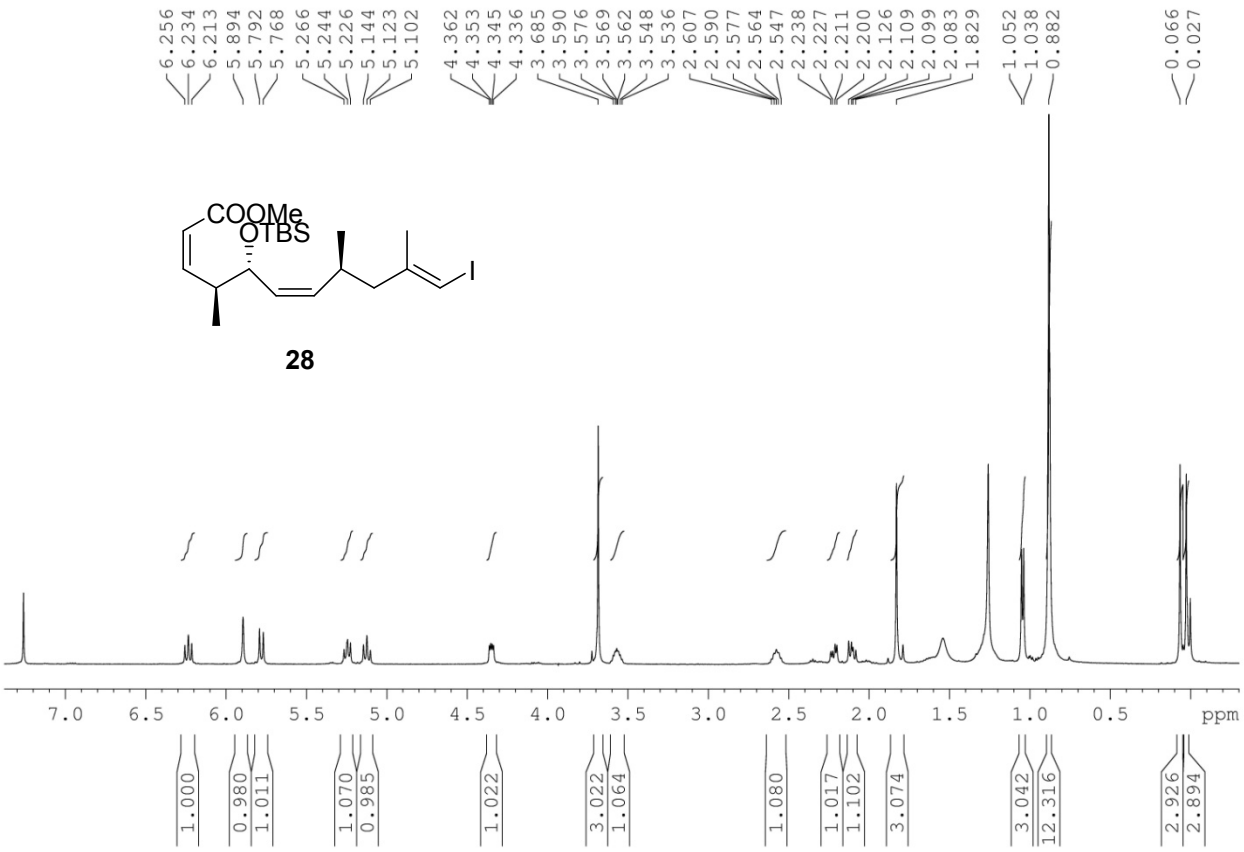
^{13}C NMR Spectrum of Compound 27 ($CDCl_3$, 75 MHz)



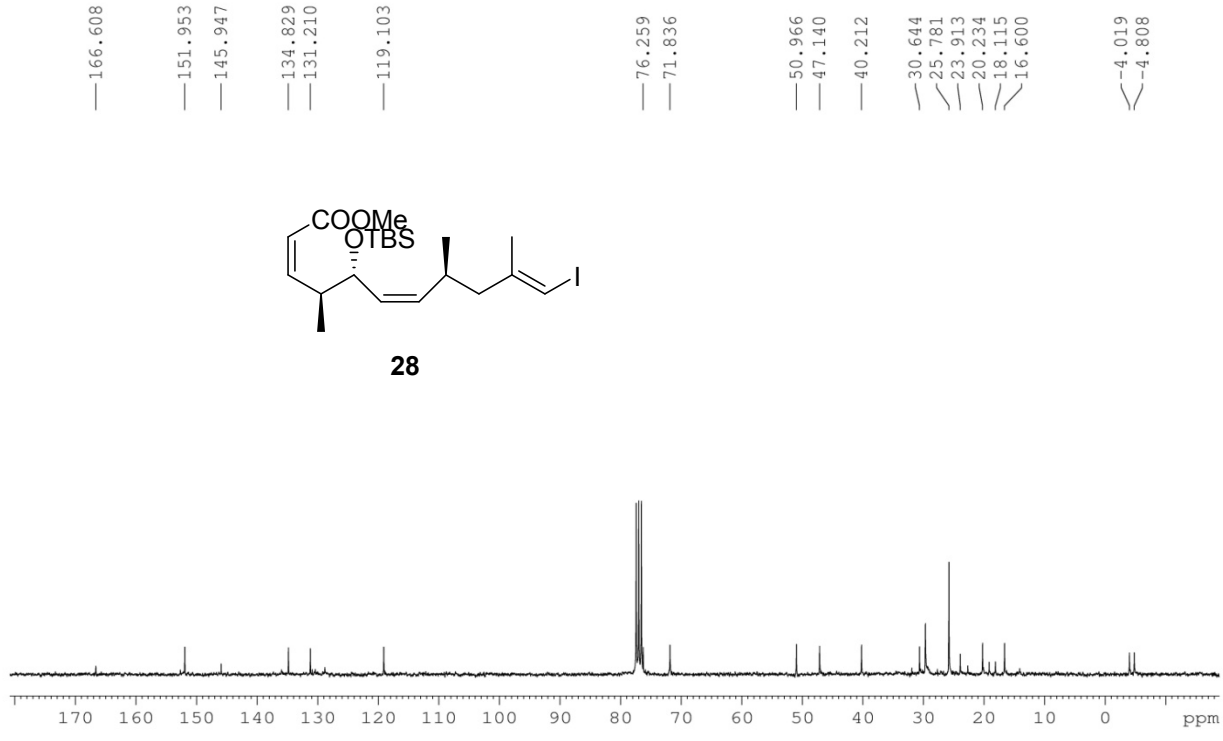
^{1}H NMR Spectrum of Compound 9 ($CDCl_3$, 500 MHz)



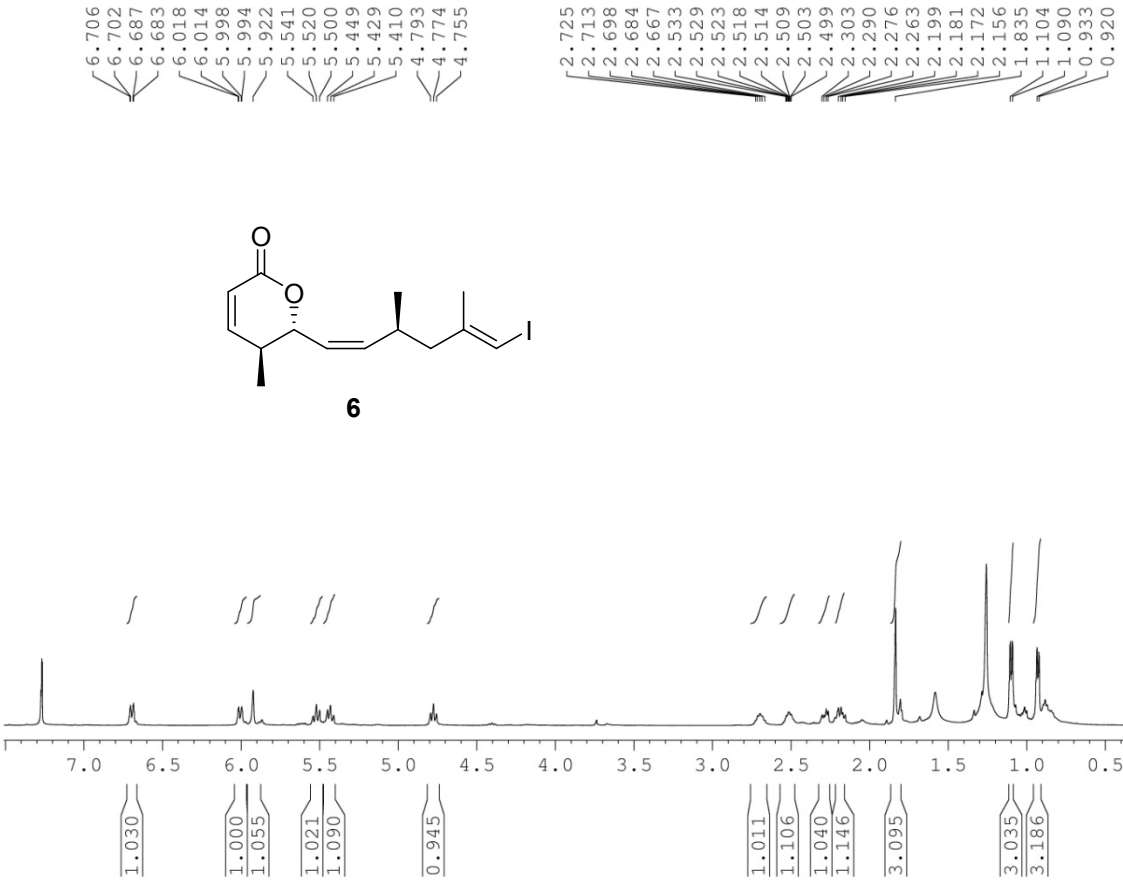
^{13}C NMR Spectrum of Compound 9 ($CDCl_3$, 125 MHz)



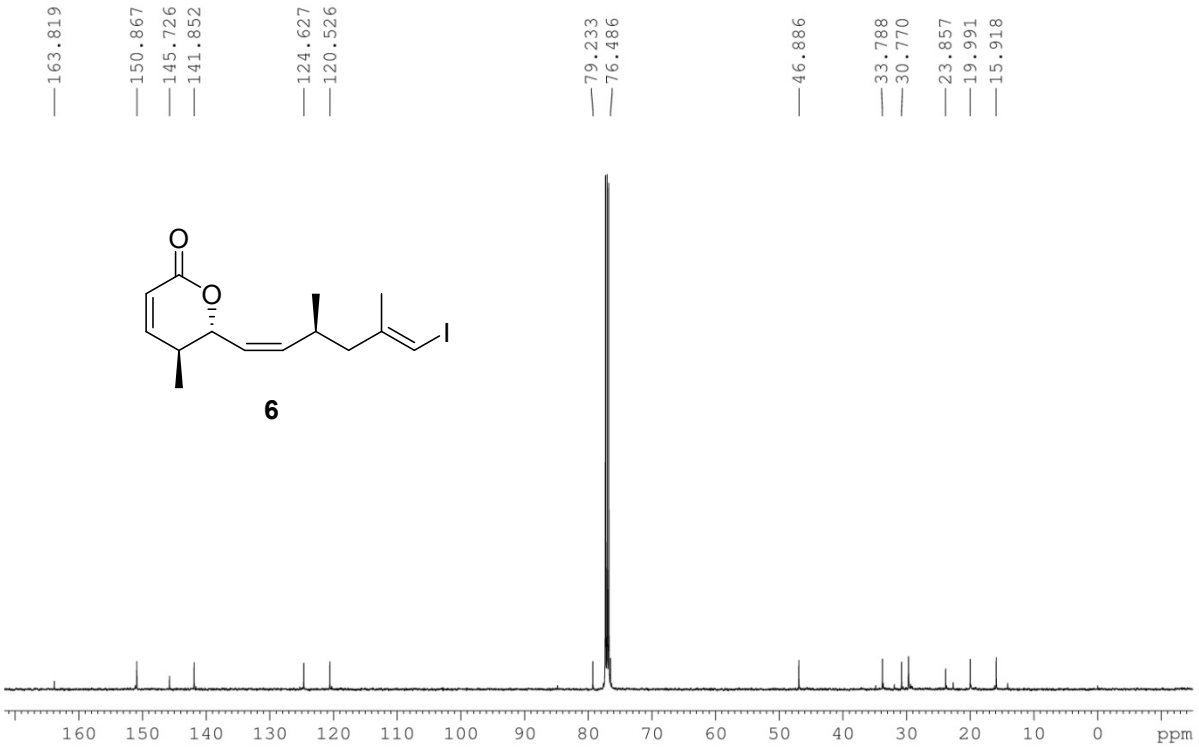
^{1}H NMR Spectrum of Compound 28 ($CDCl_3$, 500 MHz)



^{13}C NMR Spectrum of Compound 28 ($CDCl_3$, 125 MHz)



^{1}H NMR Spectrum of Compound 6 ($CDCl_3$, 500 MHz)



^{13}C NMR Spectrum of Compound 6 ($CDCl_3$, 125 MHz)

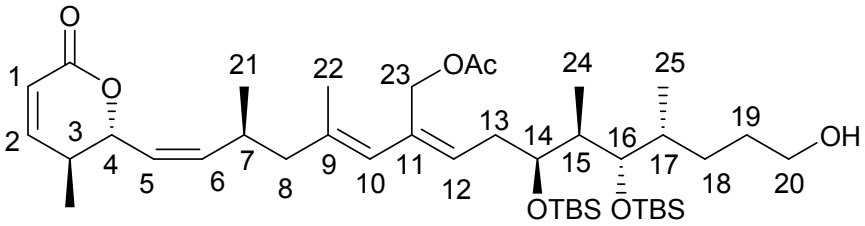
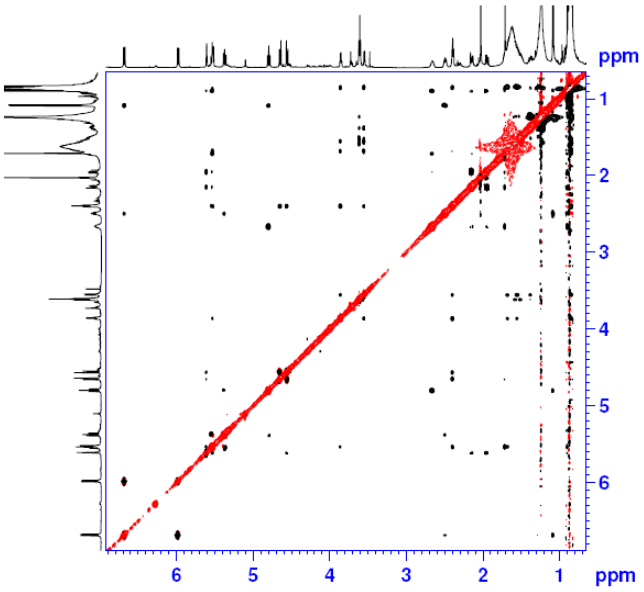


Table S1: Chemical shifts and coupling constant for compound **29**		
Proton S.No	Chemical shift Δppm	Coupling constant *j*
1	6.69(dd)	9.8, 2.2
2	5.99(dd)	9.8, 2.4
3	2.50(dqt)	10.0, 7.0, 2.5
4	4.80(t)	10.0
5	5.38(dd)	10.0, 10.7
6	5.54(t)	10.7
7	2.67(ddqd)	10.7,8.6, 7.0, 6.0
8	2.15(dd)	13.5, 6.0
8’	1.95(dd)	13.5, 8.6
9	-	-
10	5.61(d)	1.3
11	-	-
12	5.53(t)	6.7
13(2H)	2.40 (m)	
14	3.86(m)	
15	1.68(m)	
16	3.56(dd)	1.8, 6.8
17	1.68(m)	
18	--	
19	1.59(m), 1.51(m)	
20(2H)	3.62(m)	
21(3H)	0.90(d)	6.5
22(3H)	1.72(d)	1.3

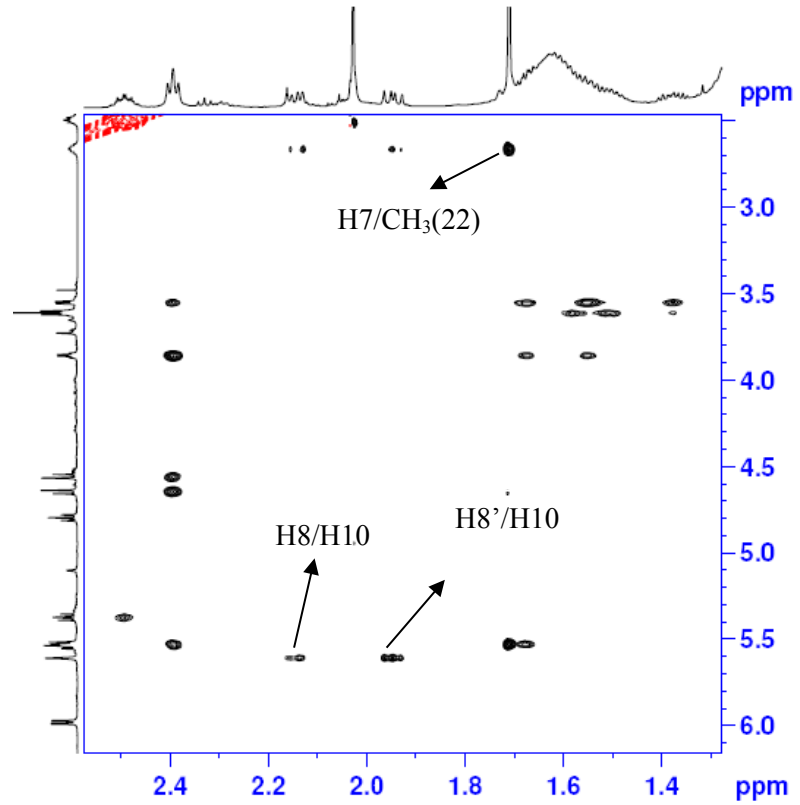
23H	4.56(d)	12.5
23'H	4.65(d)	12.5
24(3H)	0.85(d)	6.9
25(3H)	0.84(d)	6.7

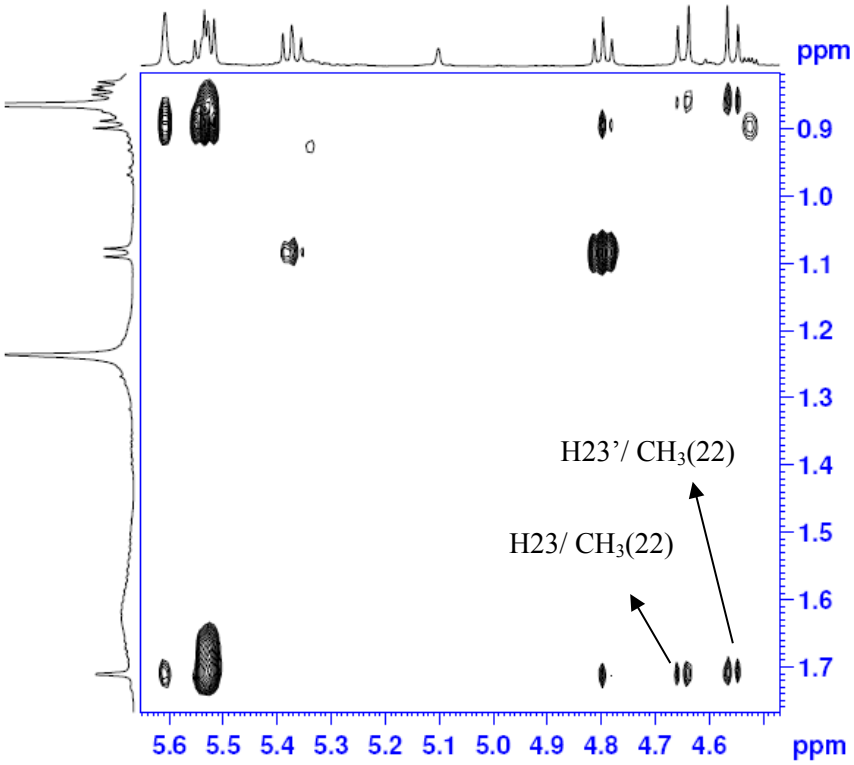
The structure of the compound **29** was characterized by extensive NMR experiment including 2-D double quantum filtered correlation spectroscopy (DQFCOSY) and nuclear Overhauser effect spectroscopy (NOESY). The assignments of the protons were initiated with the olefinic protons **1** and **2**, which are both doublet of doublets (dd), around 6-7 ppm. The coupling of the two major fragments, resulting in the formation of *trans*-olefinic bonds was supported by the NOESY correlations, H10/H12, CH_3(22)/H23 and CH_3(22)/H23'. Additionally, nOe correlations, H7/CH_3(22), H8/H10 and H8'/H10, further support the *trans* disposition of bond.

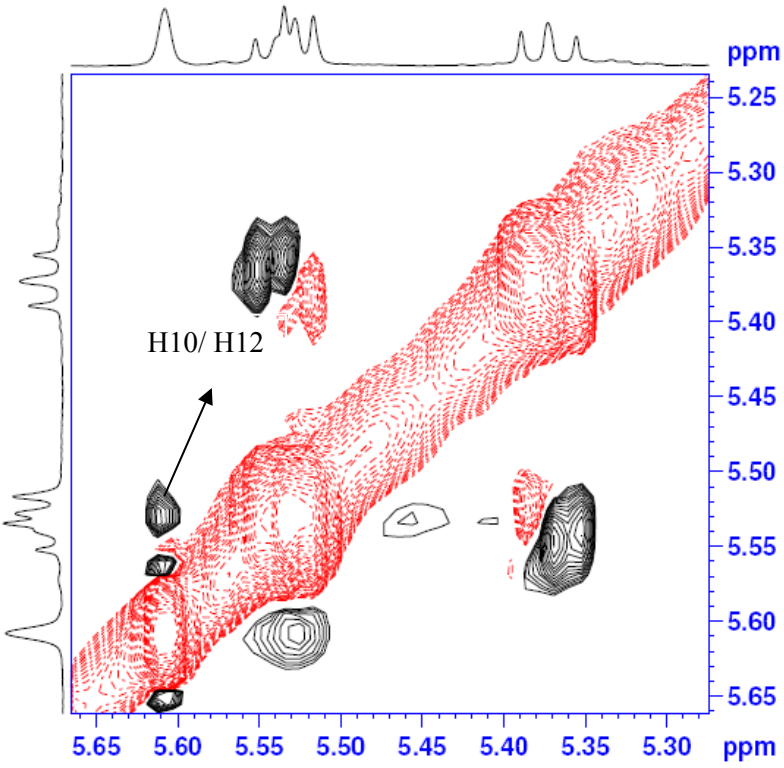


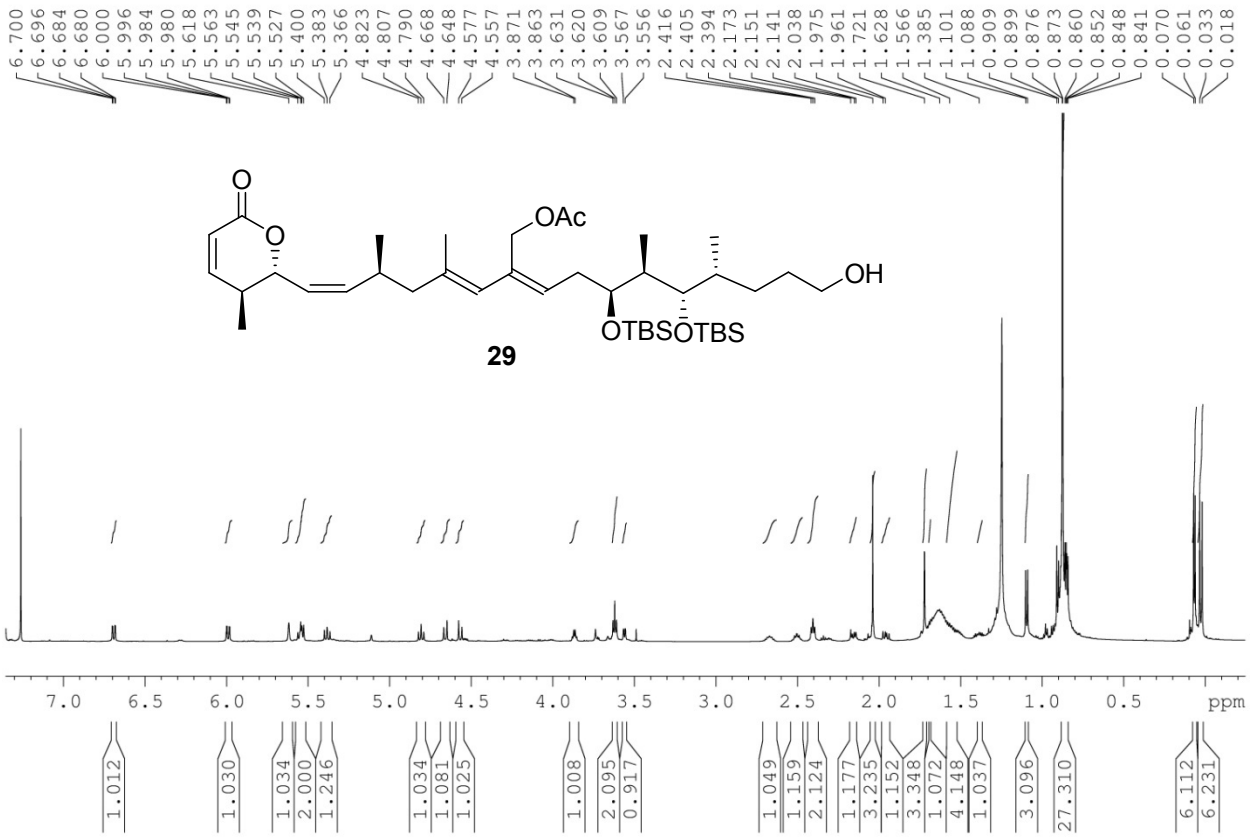
NOESY Spectrum of Compound 29 ($CDCl_3$, 600 MHz)

Expansions of NOESY spectra for compound **29**

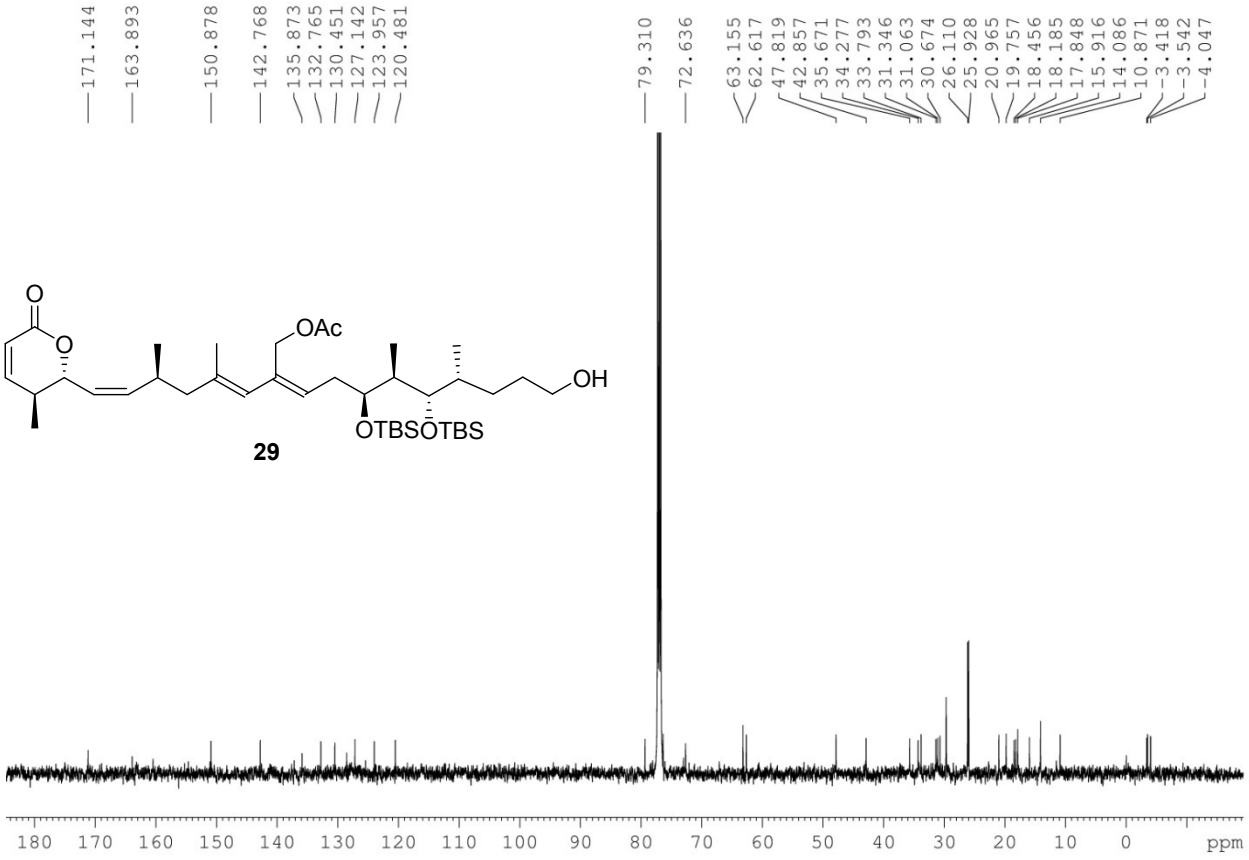

ppm
H7/CH3(22)
H8/H10
H8'/H10
3.0
3.5
4.0
4.5
5.0
5.5
6.0
2.4
2.2
2.0
1.8
1.6
1.4
ppm


ppm
H23'/ CH3(22)
H23/ CH3(22)
0.9
1.0
1.1
1.2
1.3
1.4
1.5
1.6
1.7
5.6
5.5
5.4
5.3
5.2
5.1
5.0
4.9
4.8
4.7
4.6
ppm


ppm
5.25
5.30
5.35
5.40
5.45
5.50
5.55
5.60
5.65
H10/ H12
5.65
5.60
5.55
5.50
5.45
5.40
5.35
5.30
ppm

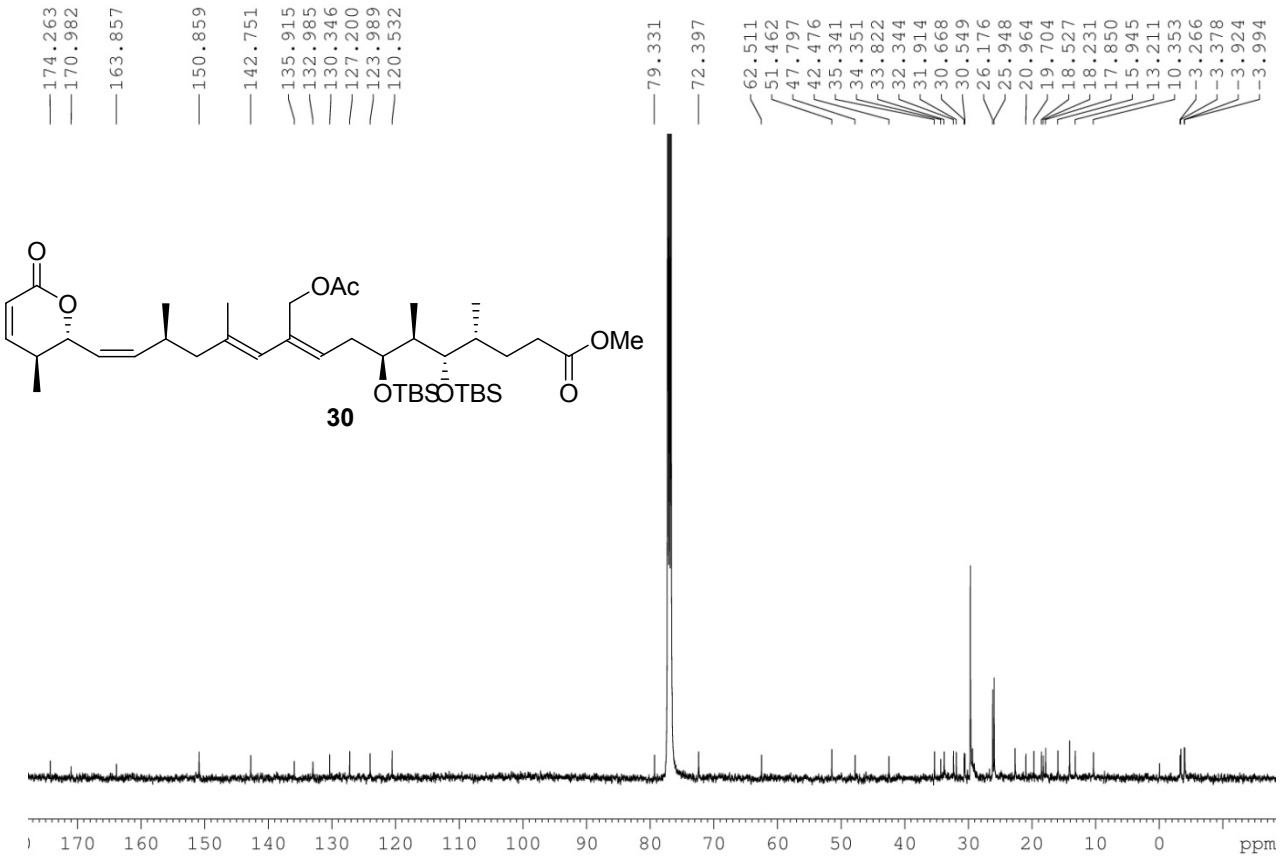


^{1}H NMR Spectrum of Compound 29 ($CDCl_3$, 600 MHz)



^{13}C NMR Spectrum of Compound 29 ($CDCl_3$, 75 MHz)

^{1}H NMR Spectrum of Compound 30 ($CDCl_3$, 500 MHz)



^{13}C NMR Spectrum of compound 30 ($CDCl_3$, 125 MHz)