

Supporting Information

Total Synthesis of Marine Natural Products Separacenes A and B

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1. NMR Comparison Tables: S(2-4)

2. Copies of Spectra: S(5-22)

Table S1: ^1H and ^{13}C NMR comparison of Separacenes A (natural, $\text{C}_5\text{D}_5\text{N}$, 900 MHz), compound 1 ($\text{C}_5\text{D}_5\text{N}$, 300 MHz) δ in ppm.

^1H NMR of Natural Separacenes A	^1H NMR of Compound 1	^{13}C NMR of Natural Separacenes A	^{13}C NMR of Compound 1
6.79, dd (15.5, 10.5), 1H 6.75, dd (15.5, 10.5), 1H	6.83-6.72, m, 2H	139.8	139.8
6.44, dd (15.5, 10.5), 1H 6.42, dd (15.5, 10.5), 1H 6.40, m, 1H 6.35, ddd (17.5, 10.5, 5.5), 1H 6.38, m, 1H	6.42-6.28, m, 5H	135.9	(Merged with $\text{C}_5\text{D}_5\text{N}$ signal)
6.26, dd (15.5, 6.0), 1H 6.21, dd (15.5, 6.0), 1H	6.22, dd (15.0, 6.0), 2H	135.7	(Merged with $\text{C}_5\text{D}_5\text{N}$ signal)
5.66, dd (17.5, 1.0), 1H	5.69, dd (17.4, 1.5), 1H	133.3	133.4
5.30, dd (10.5, 1.0), 1H	5.31, d (10.8), 1H	133.3	133.3
4.58, dd (9.5, 6.0), 1H	4.65-4.57, m, 1H	132.9	132.9
4.54, dd (9.5, 5.5), 1H	4.59-4.55, m, 1H	132.8	132.8
4.43, dd (10.0, 6.0), 1H	4.46, t (6.3), 1H	131.6	131.7
4.11, m, 1H	4.18-4.09, m, 1H	131.5	131.6

1.43, d (6.5), 3H	1.45, d (6.3), 3H	115.4	115.5
		77.3	77.4
		76.5	76.7
		76.0	76.1
		71.2	71.3
		19.6	19.6

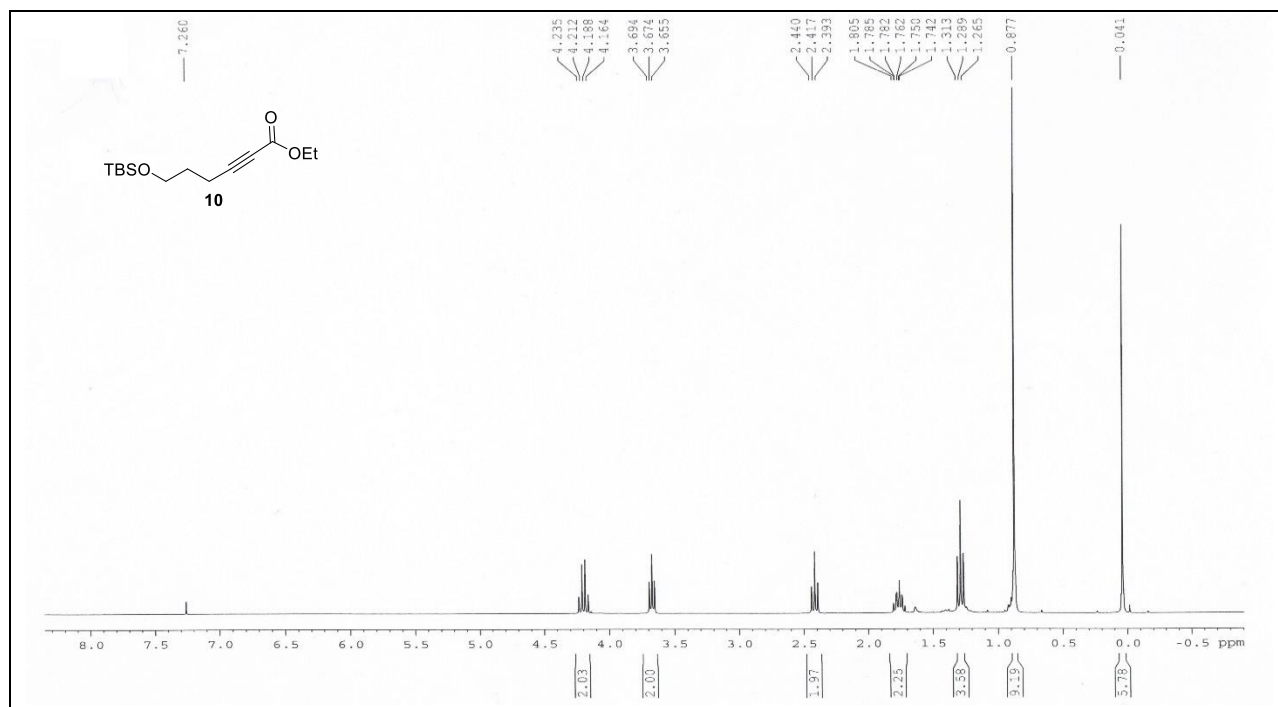
Table S2: ^1H and ^{13}C NMR comparison of Separacenes B (natural, $\text{C}_5\text{D}_5\text{N}$, 600 MHz), compound 1 ($\text{C}_5\text{D}_5\text{N}$, 300 MHz) δ in ppm.

^1H NMR of Natural Separacenes B	^1H NMR of Compound 1	^{13}C NMR of Natural Separacenes B	^{13}C NMR of Compound 1
6.80, dd (15.5, 10.0), 1H 6.76, dd (15.5, 9.5), 1H	6.82-6.71, m, 2H	142.4	140.9
6.44, dd (14.5, 9.5), 1H 6.42, dd (15.5, 10.0), 1H 6.40, m, 1H 6.38, m, 1H 6.36, ddd (17.5, 10.5, 5.5), 1H	6.43-6.35, m, 5H	137.8	137.4
6.35, dd (15.5, 6.5), 1H	6.33-6.31, m, 1H	136.8	(Merged with $\text{C}_5\text{D}_5\text{N}$ signal)
6.26, dd (15.5, 6.0), 1H	6.26, dd (15.3, 5.7), 1H	134.3	134.5
5.70, dd (17.5, 1.0), 1H	5.69, dd (17.1, 0.6), 1H	134.0	134.4

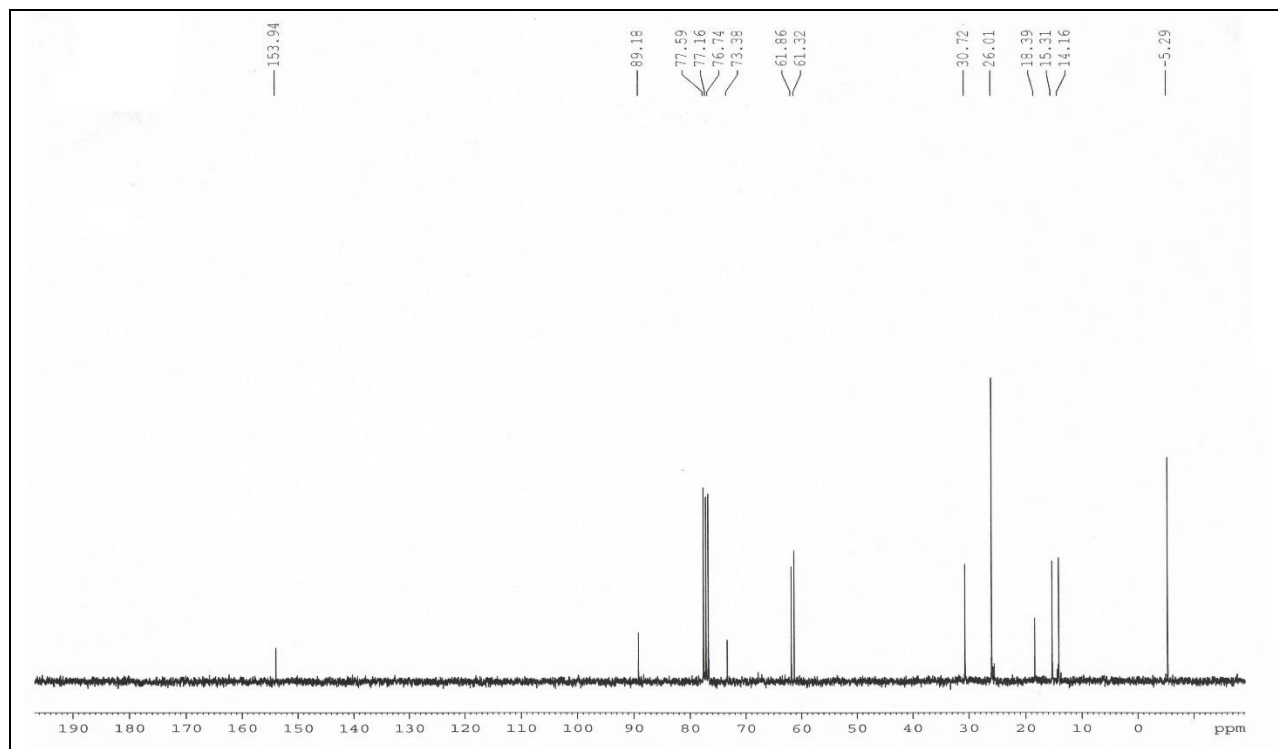
5.31, dd (10.5, 1.0), 1H	5.31, dd (10.8, 0.9), 1H	133.7	134.0
4.63, dd (11.0, 6.0), 1H	4.65-4.61, m, 1H	133.3	133.9
4.58, m, 1H 4.56, m, 1H	4.59-4.55, m, 2H	132.8	132.8
4.24, dd (11.0, 5.5), 1H	4.27-4.19, m, 1H	132.3	132.7
1.54, d (5.5), 3H	1.54, d (6.3), 3H	115.4	116.6
		78.2	78.4
		77.8	77.8
		77.3	77.2
		72.6	72.4
		20.7	20.7

Spectra (^1H NMR, ^{13}C NMR, HRMS) of compounds:

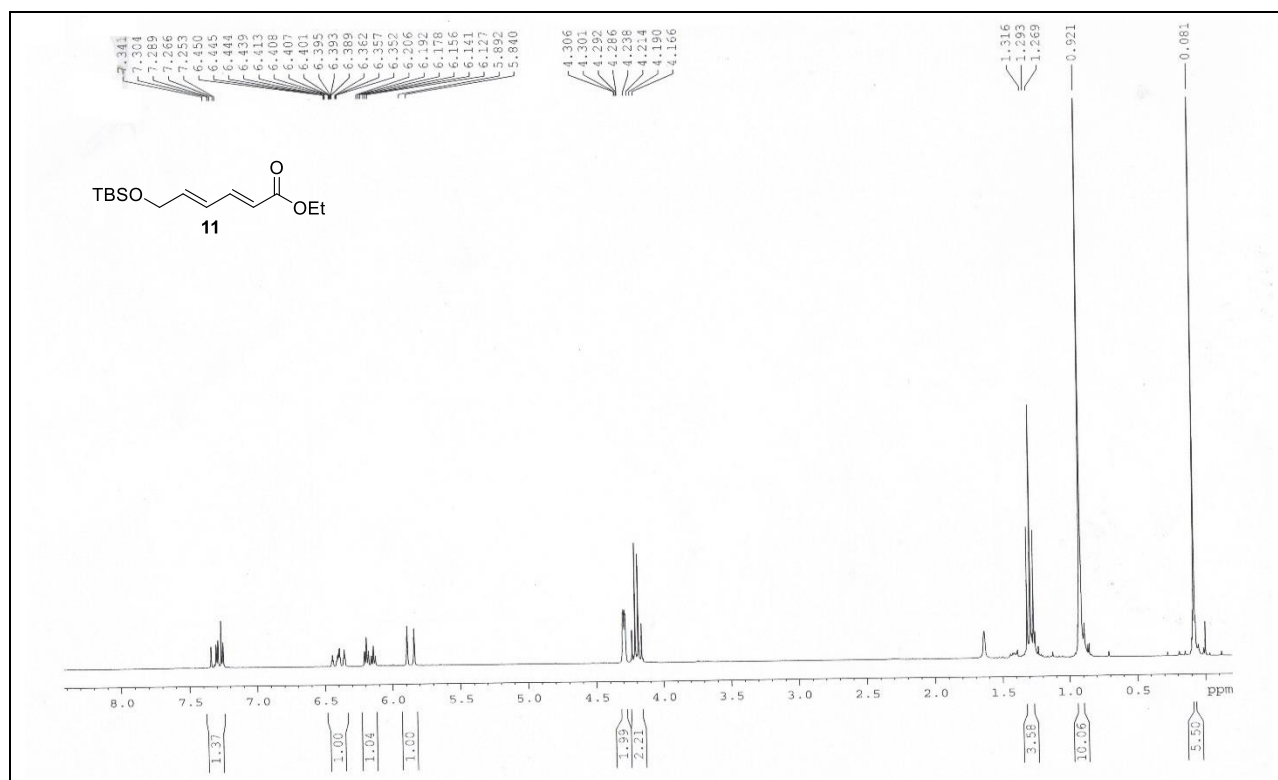
^1H NMR spectrum of 10 (300 MHz, CDCl_3):



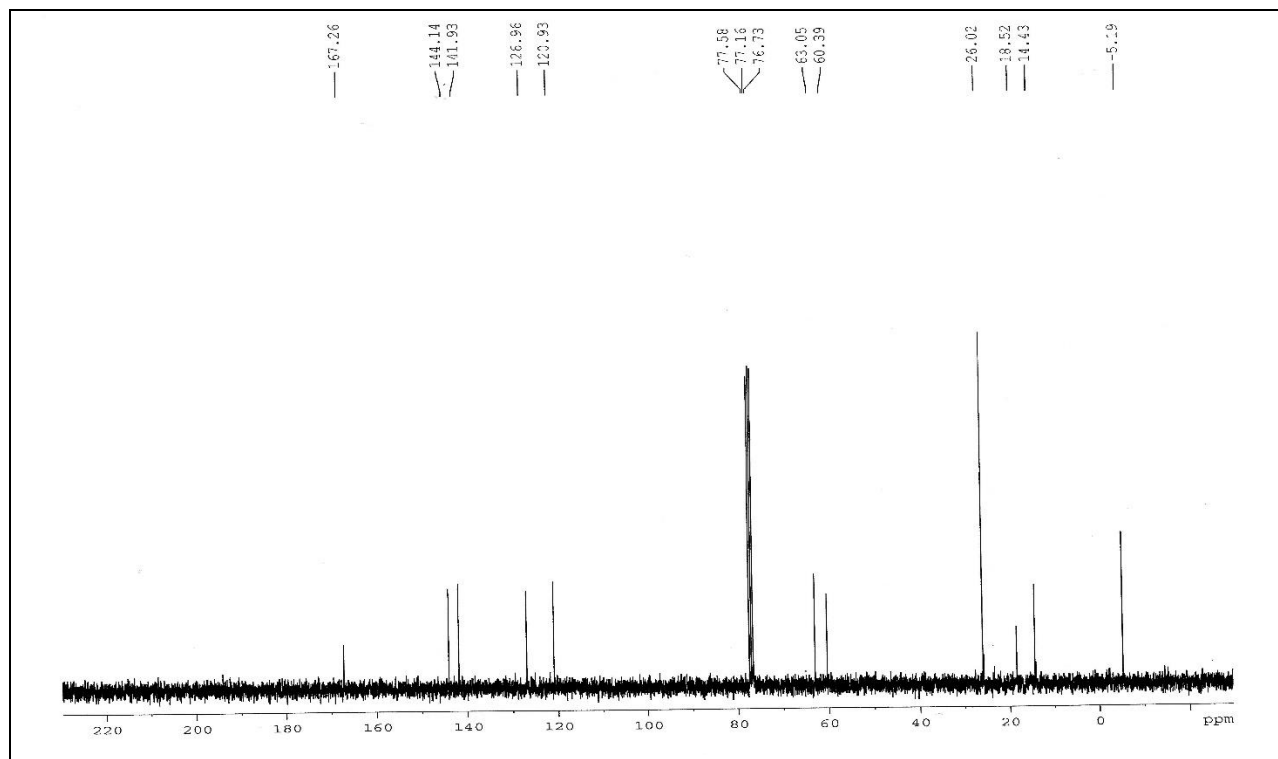
^{13}C NMR spectrum of 10 (75 MHz, CDCl_3):



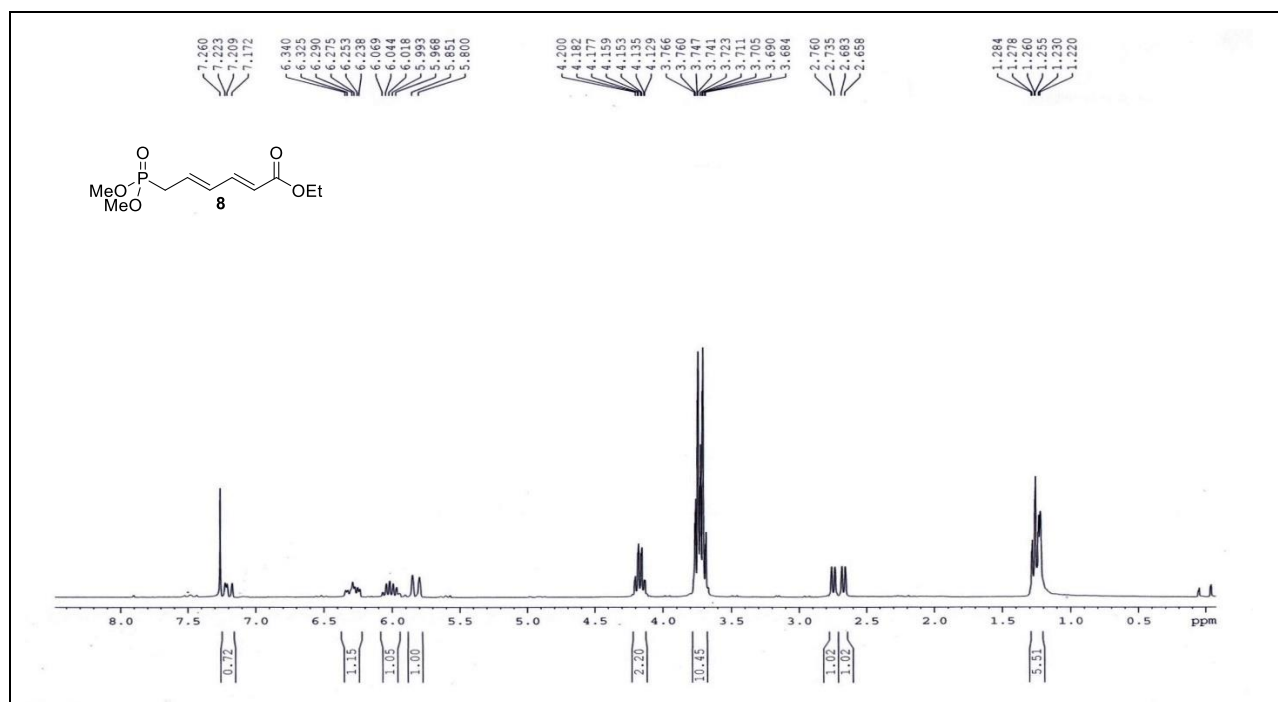
^1H NMR spectrum of 11 (300 MHz, CDCl_3):



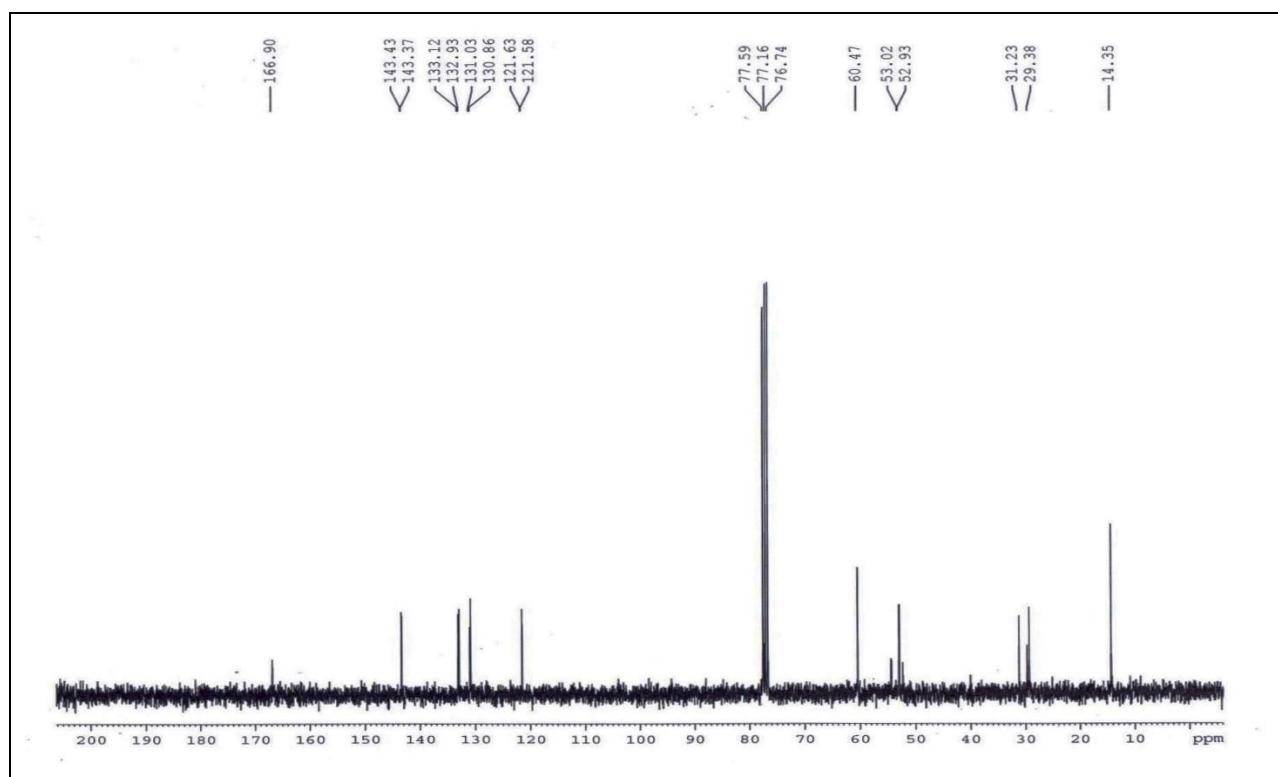
^{13}C NMR spectrum of 11 (75 MHz, CDCl_3):



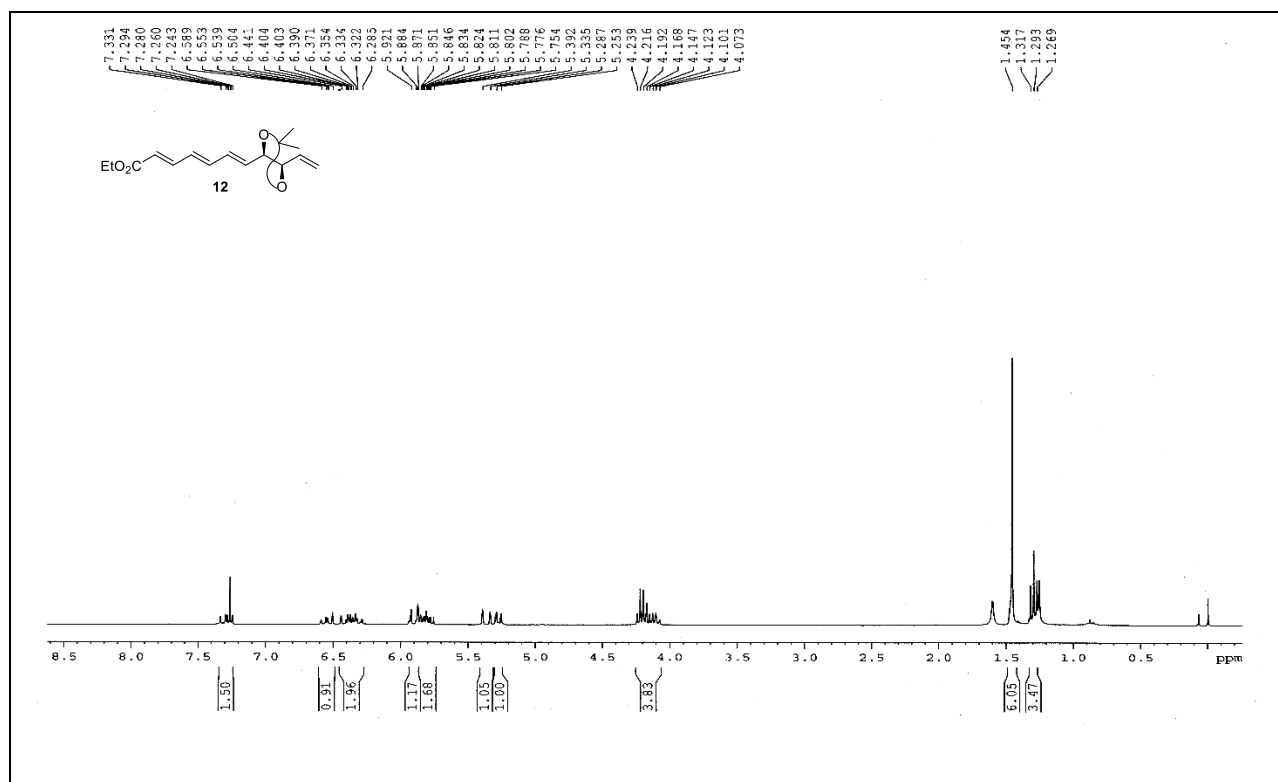
^1H NMR spectrum of 8 (300 MHz, CDCl_3):



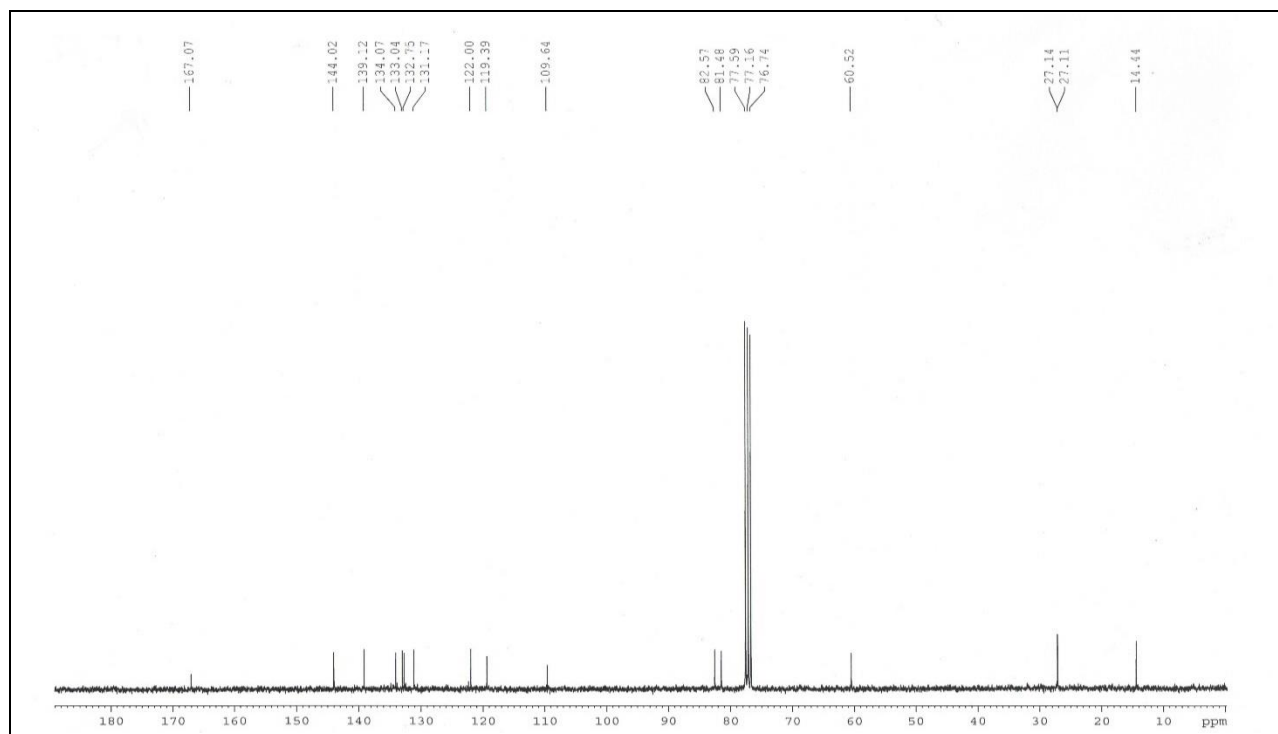
^{13}C NMR spectrum of 8 (75 MHz, CDCl_3):



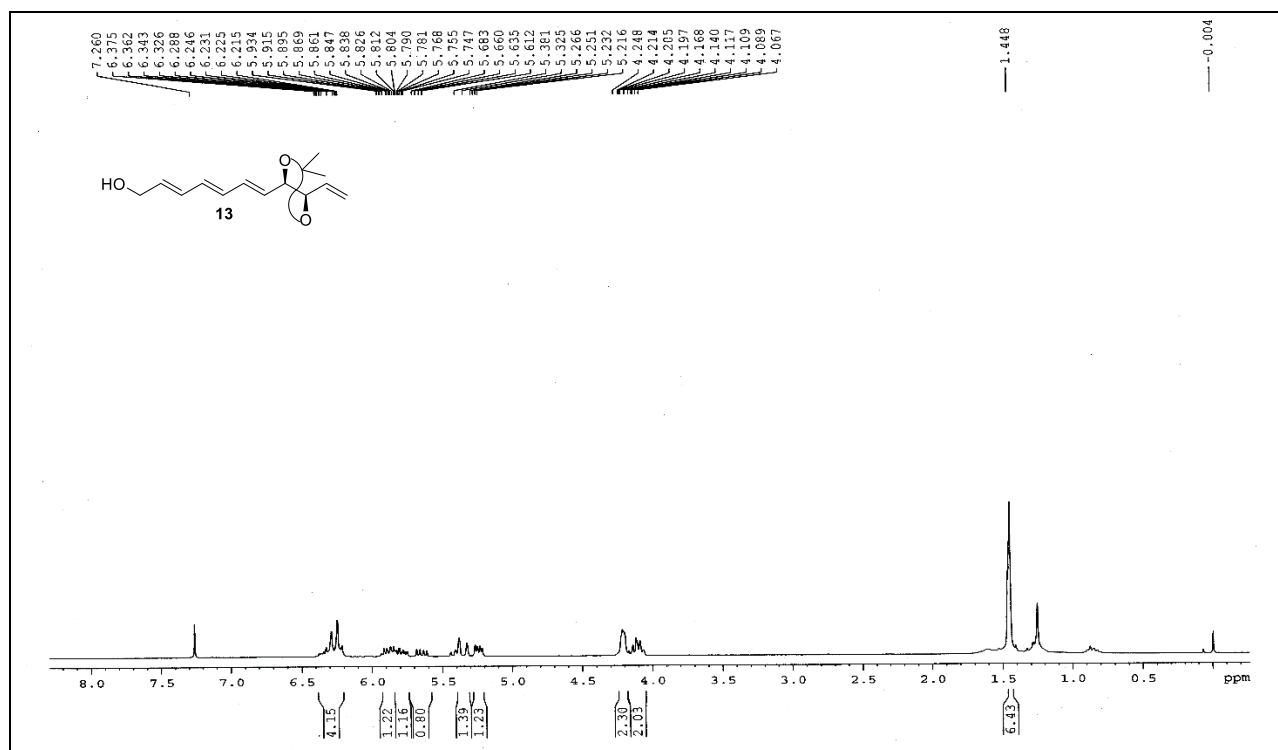
^1H NMR spectrum of 12 (300 MHz, CDCl_3):



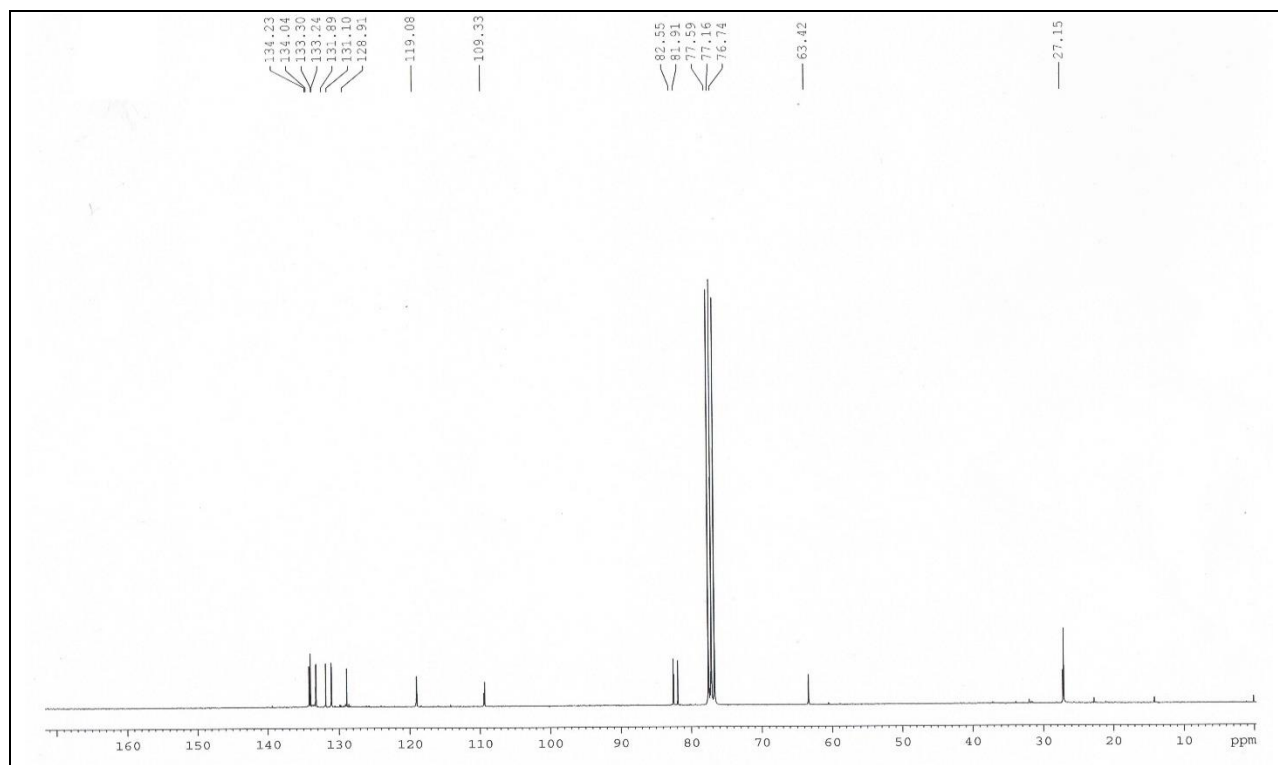
^{13}C NMR spectrum of 12 (75 MHz, CDCl_3):



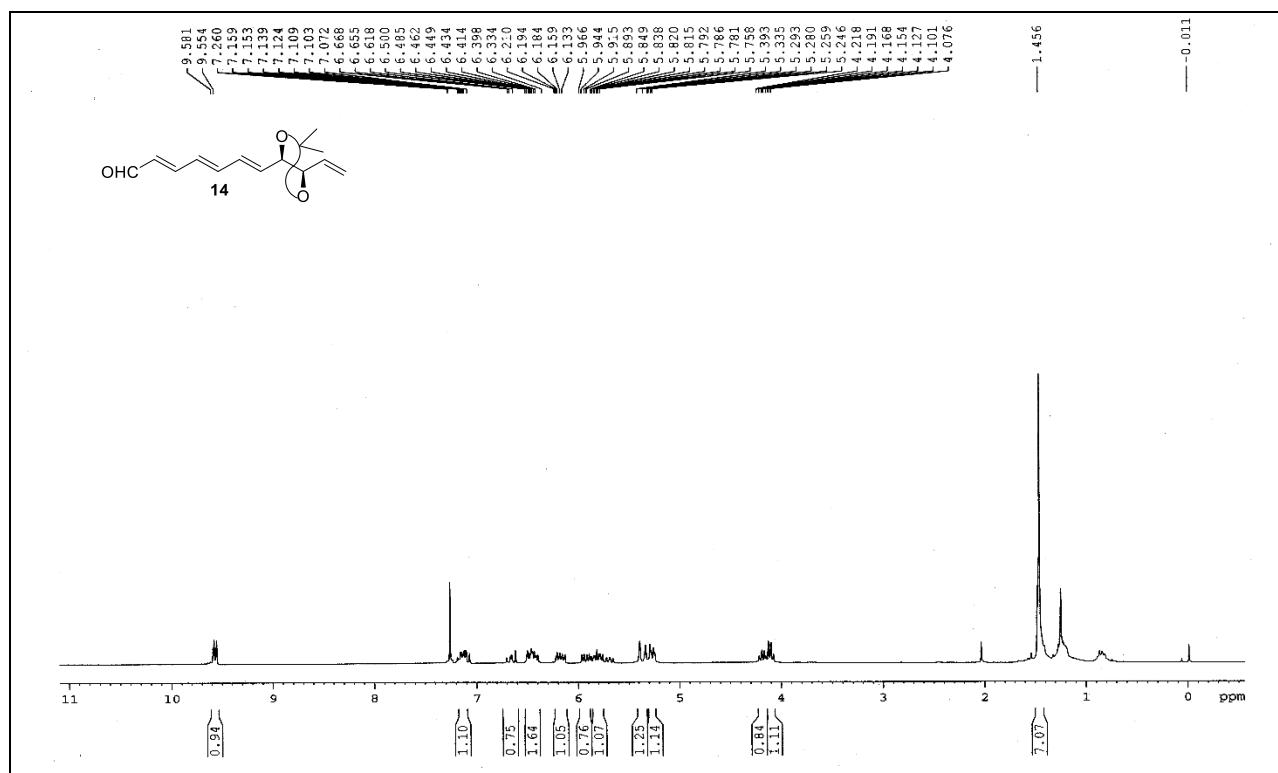
^1H NMR spectrum of 13 (300 MHz, CDCl_3):



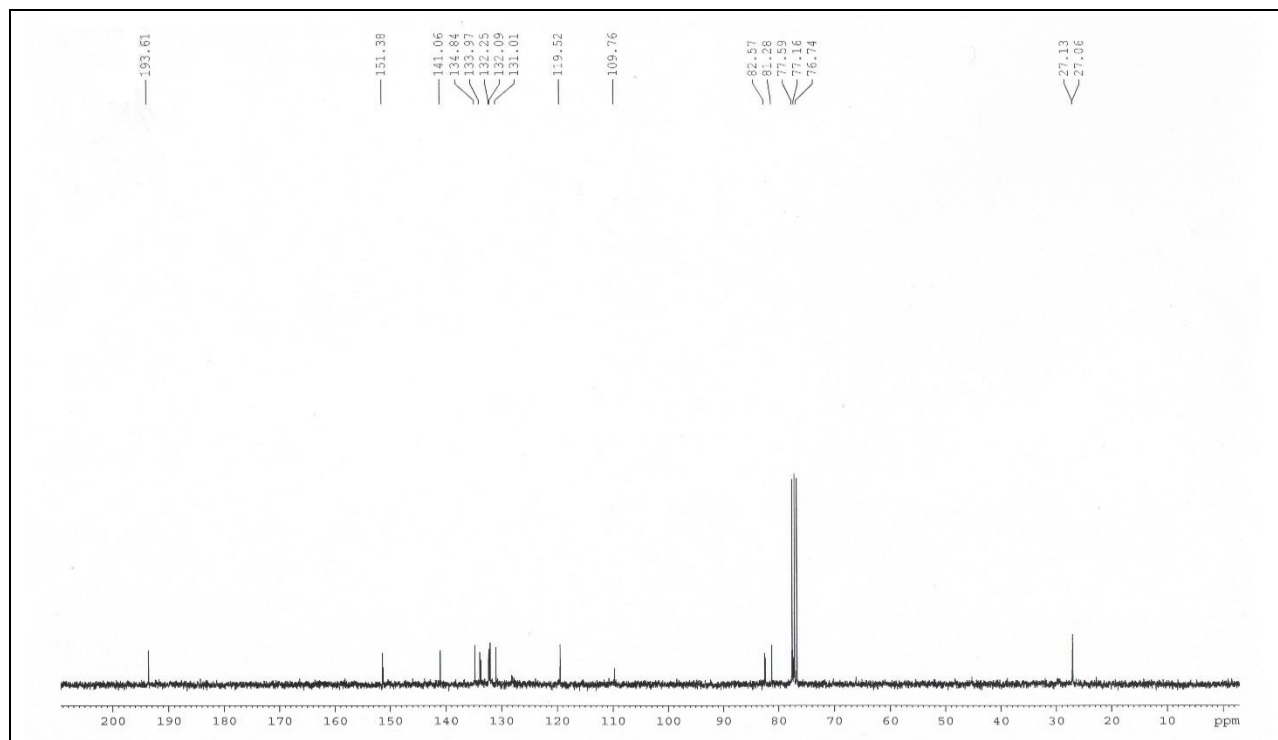
^{13}C NMR spectrum of 13 (75 MHz, CDCl_3):



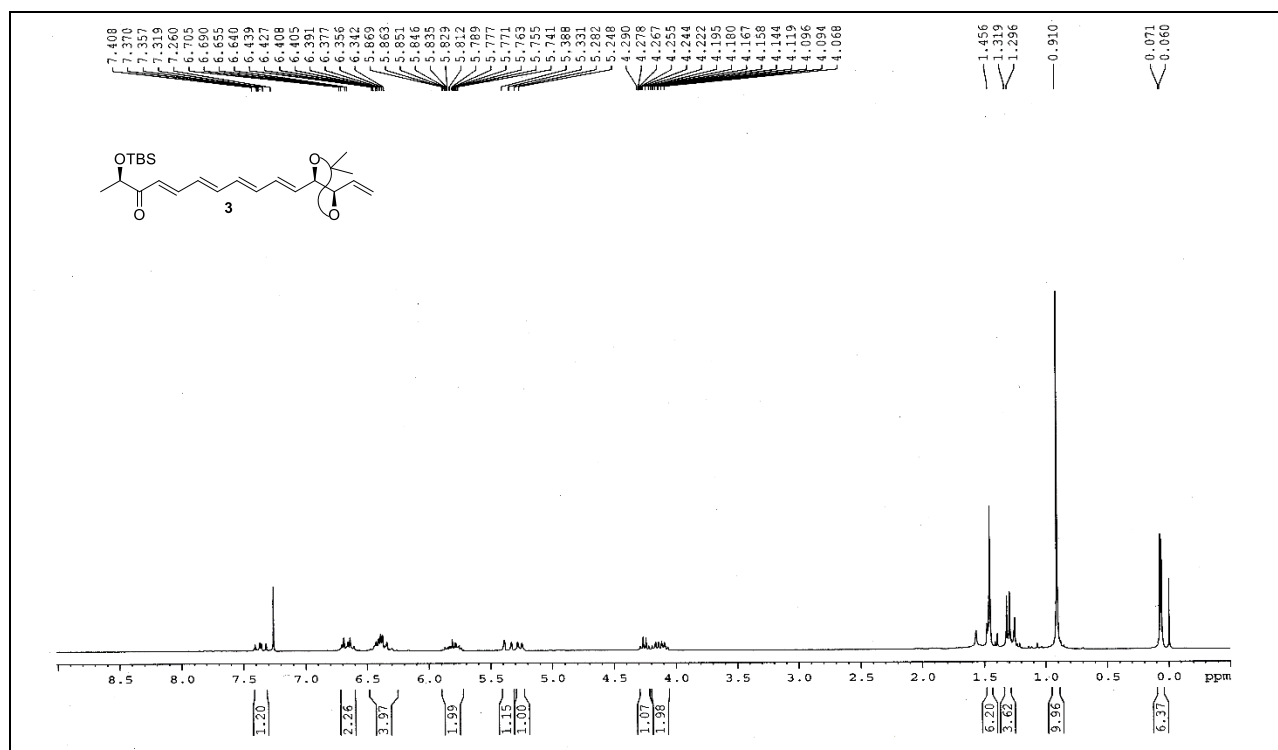
^1H NMR spectrum of 14 (300 MHz, CDCl_3):



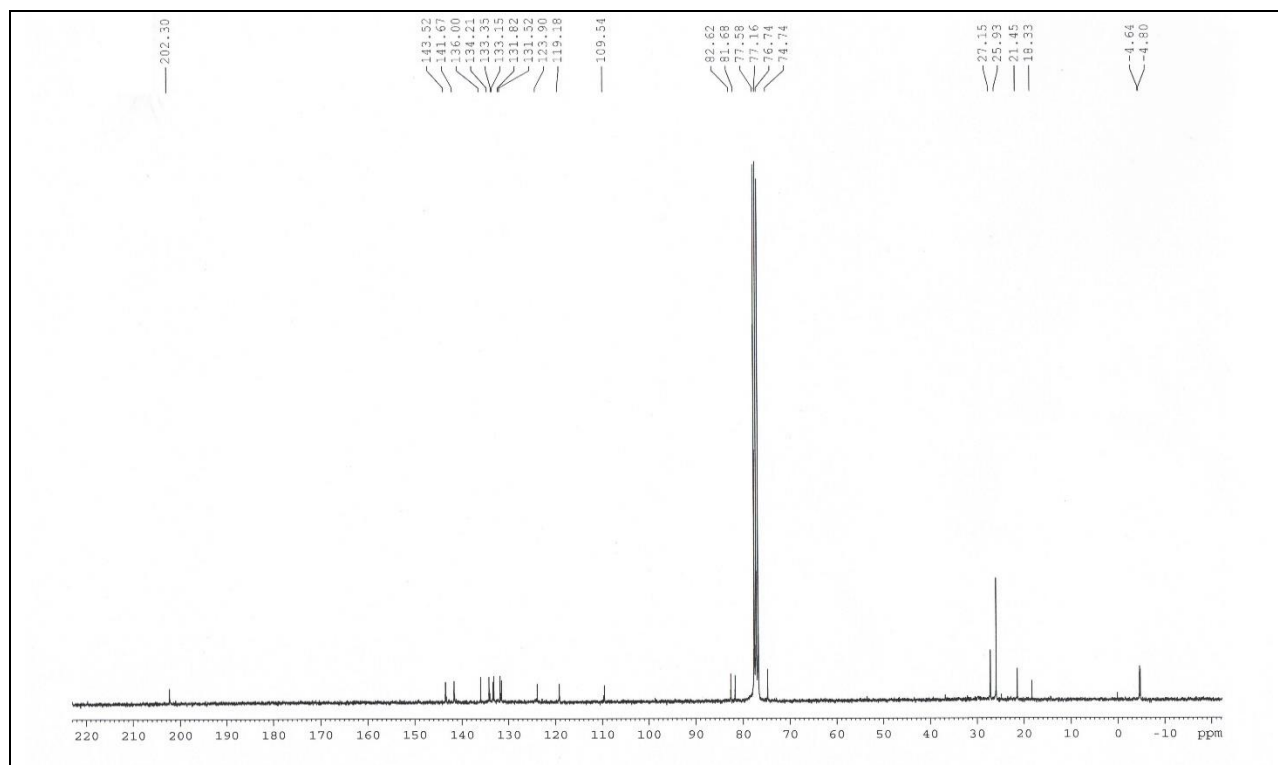
^{13}C NMR spectrum of 14 (75 MHz, CDCl_3):



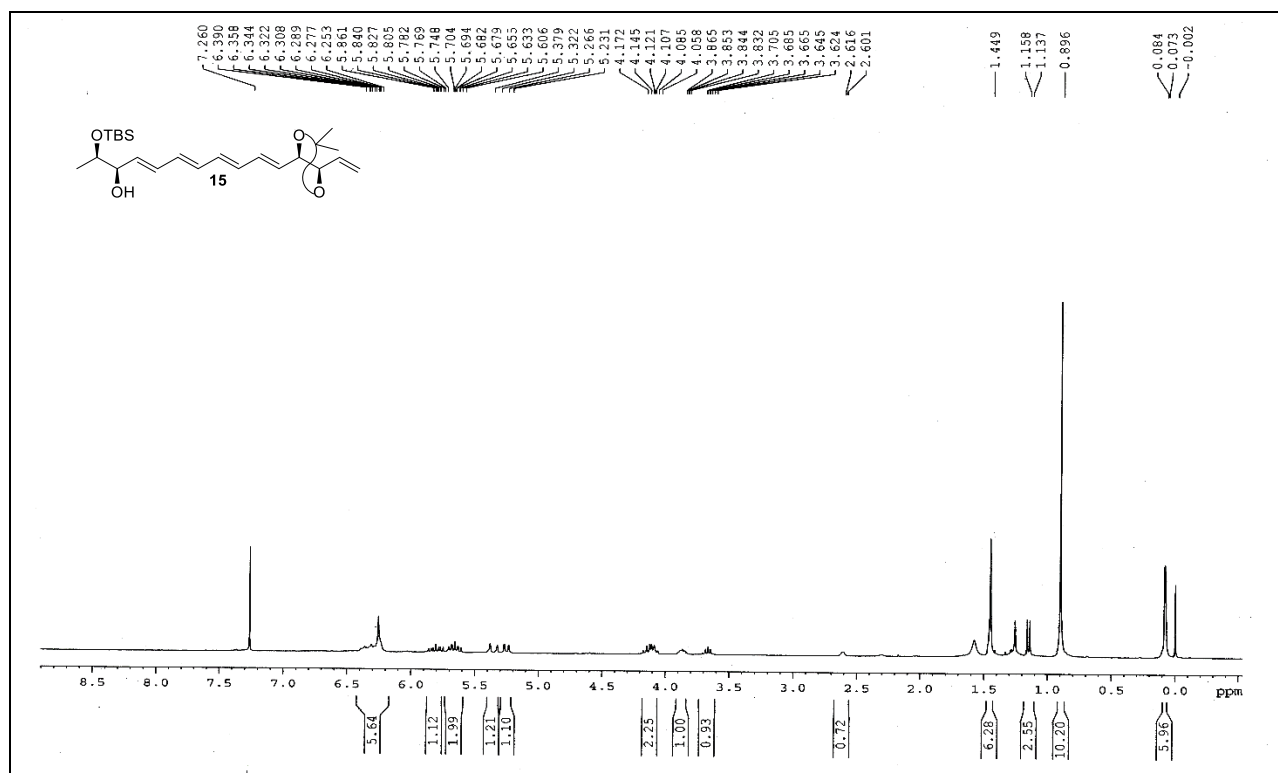
^1H NMR spectrum of 3 (300 MHz, CDCl_3):



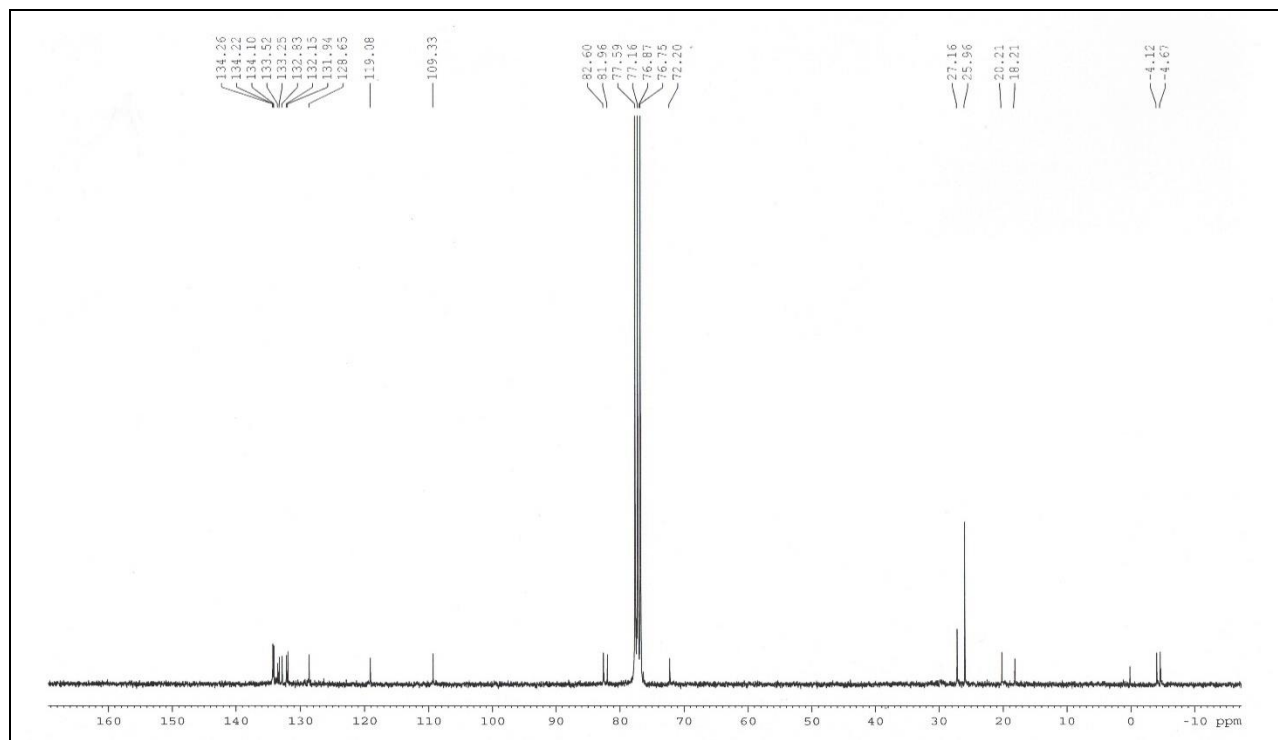
^{13}C NMR spectrum of 3 (75 MHz, CDCl_3):



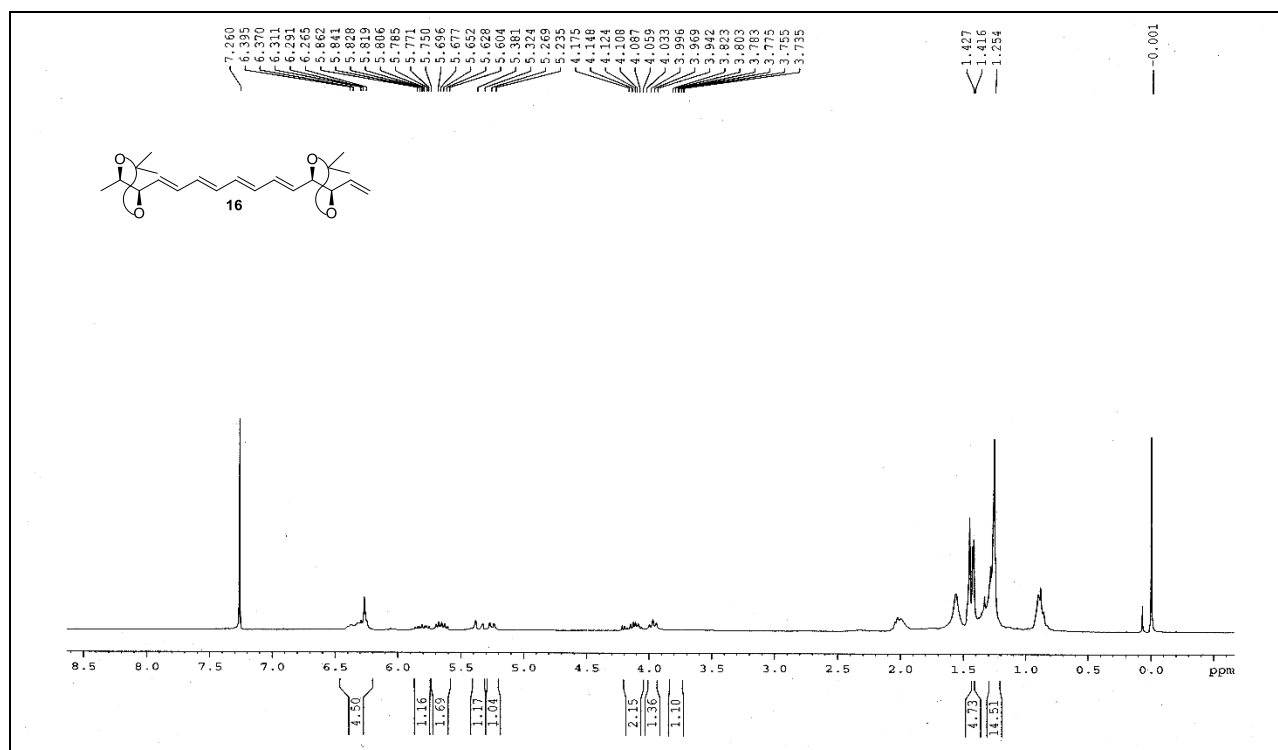
¹H NMR spectrum of 15 (300 MHz, CDCl₃):



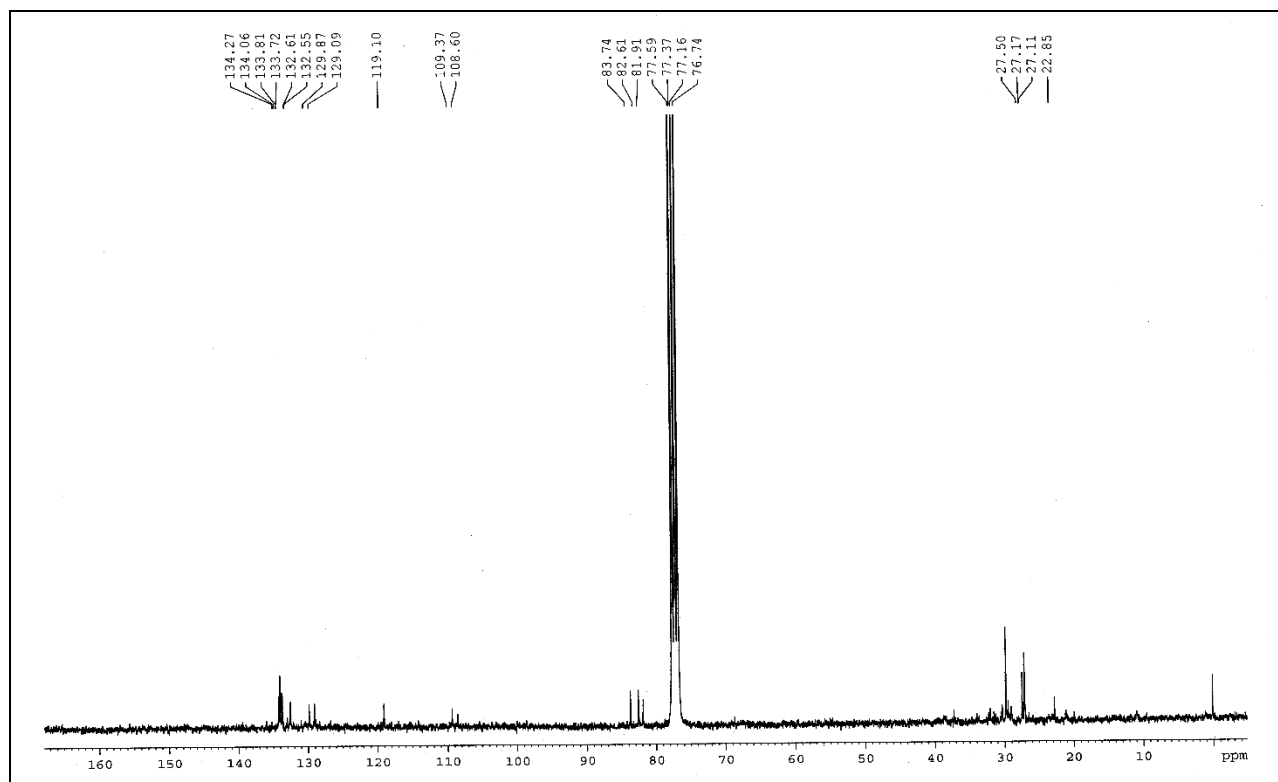
^{13}C NMR spectrum of 15 (75 MHz, CDCl_3):



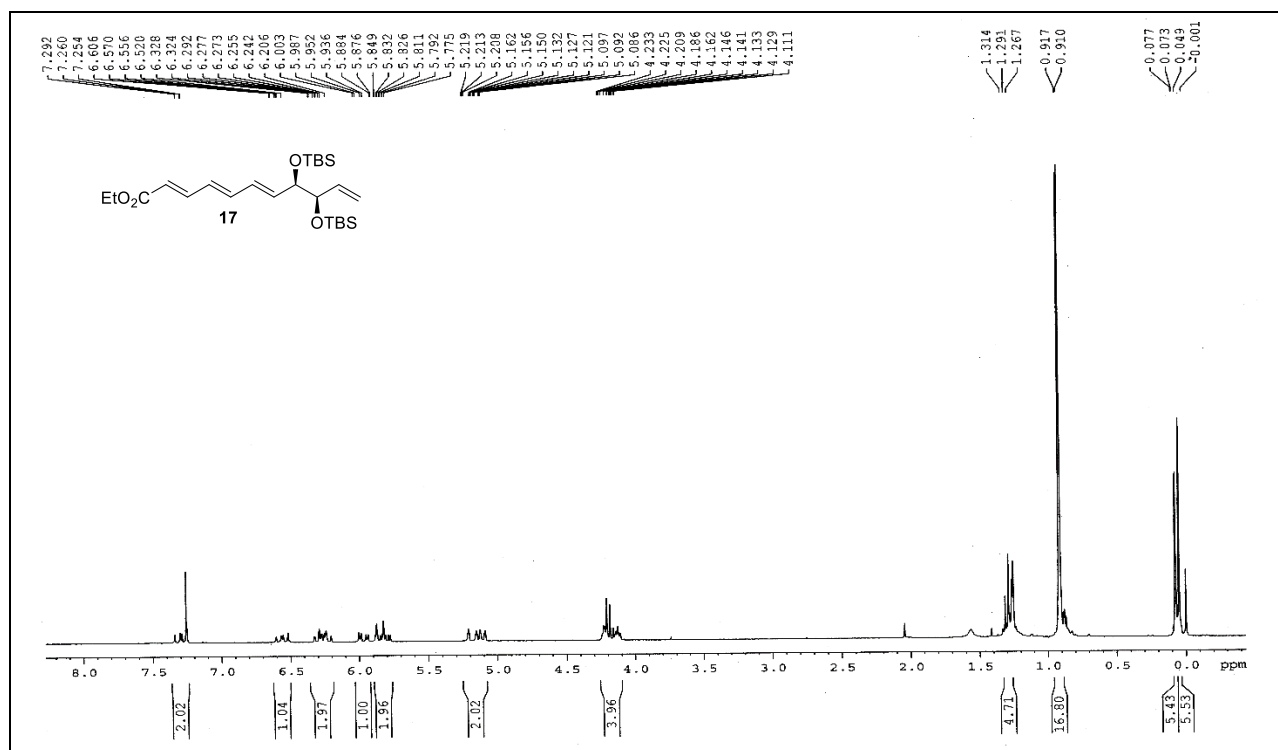
^1H NMR spectrum of 16 (300 MHz, CDCl_3):



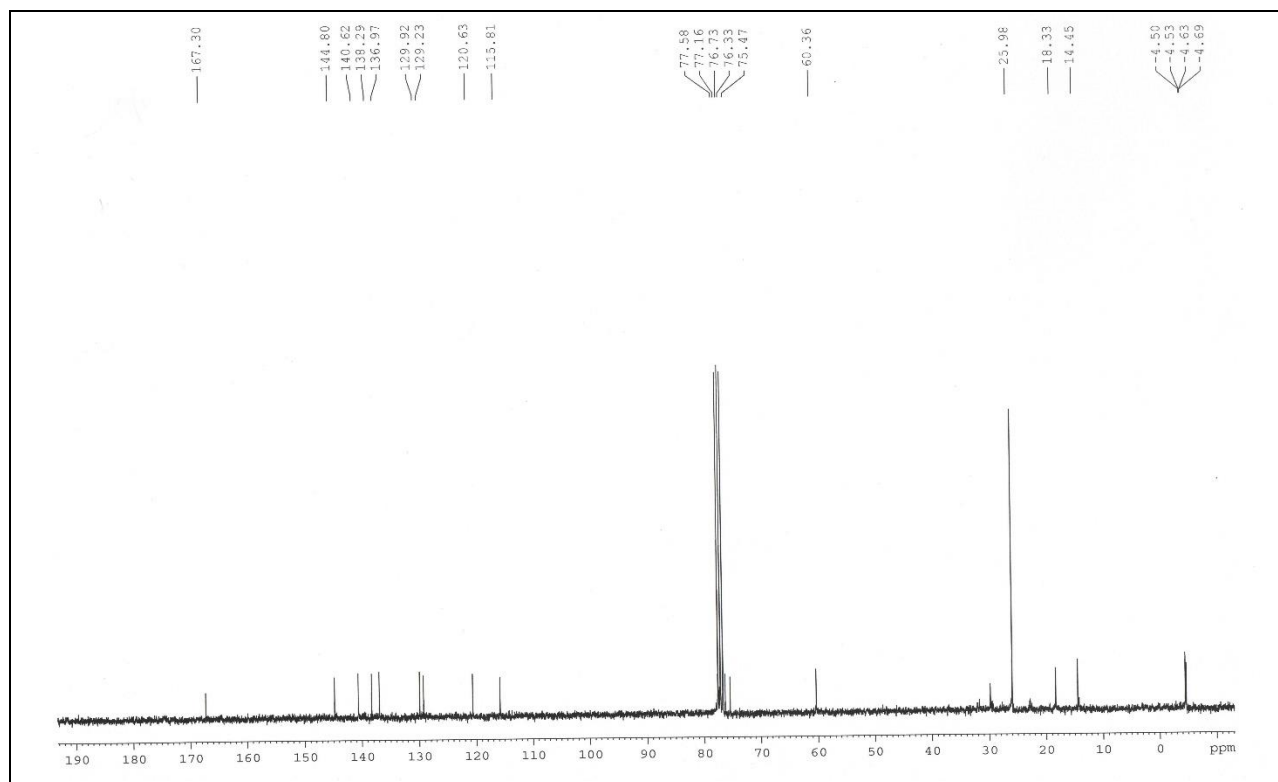
^{13}C NMR spectrum of 16 (75 MHz, CDCl_3):



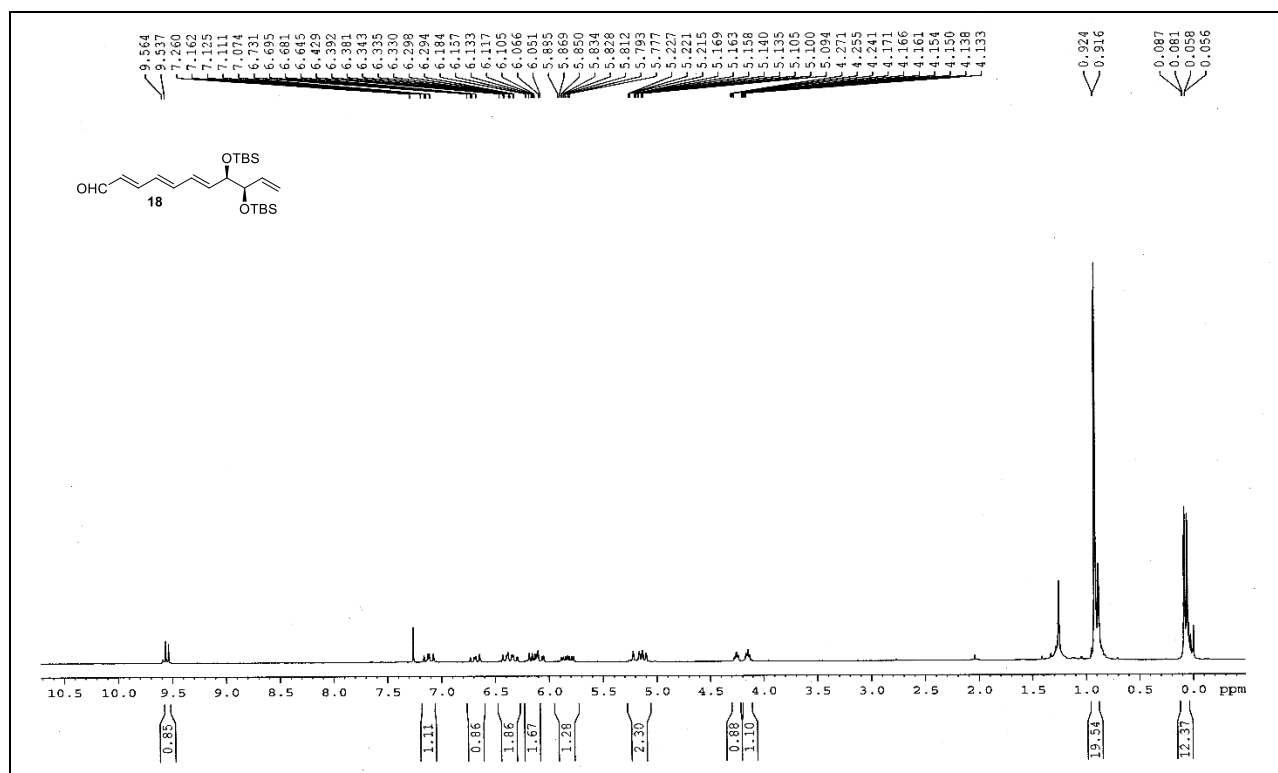
^1H NMR spectrum of 17 (300 MHz, CDCl_3):



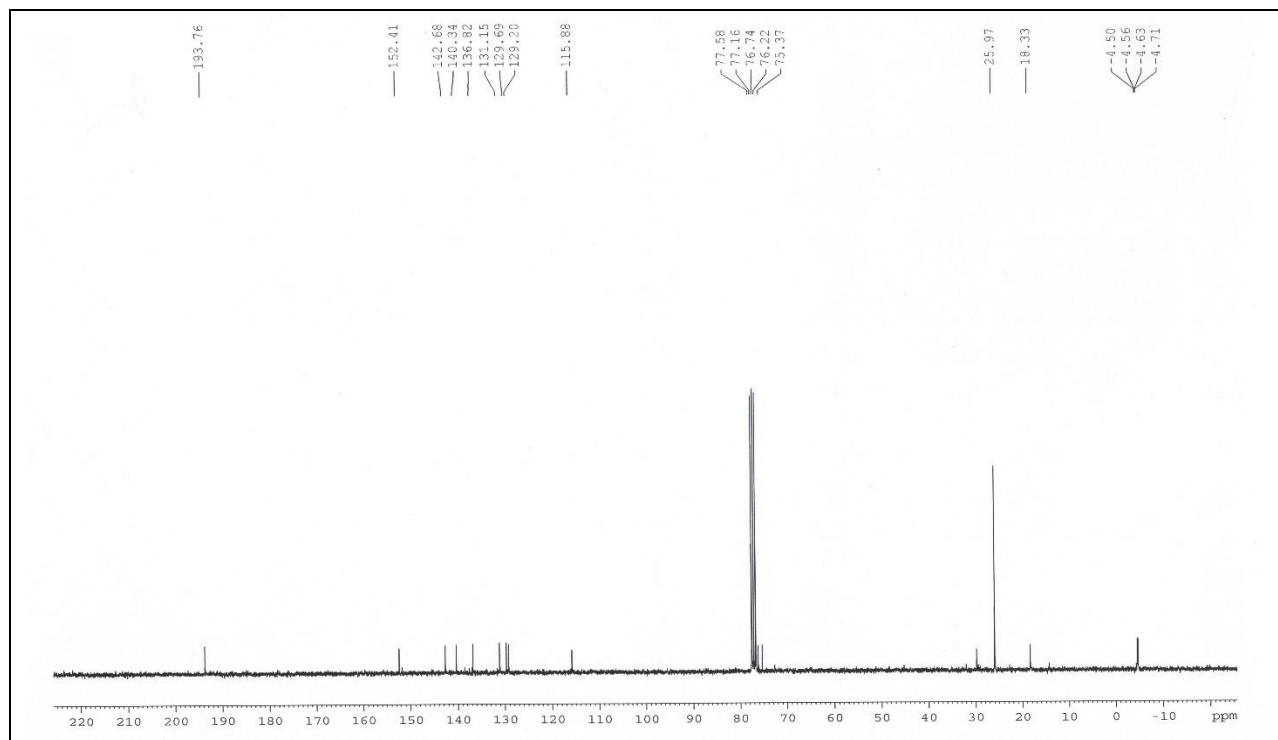
^{13}C NMR spectrum of 17 (75 MHz, CDCl_3):



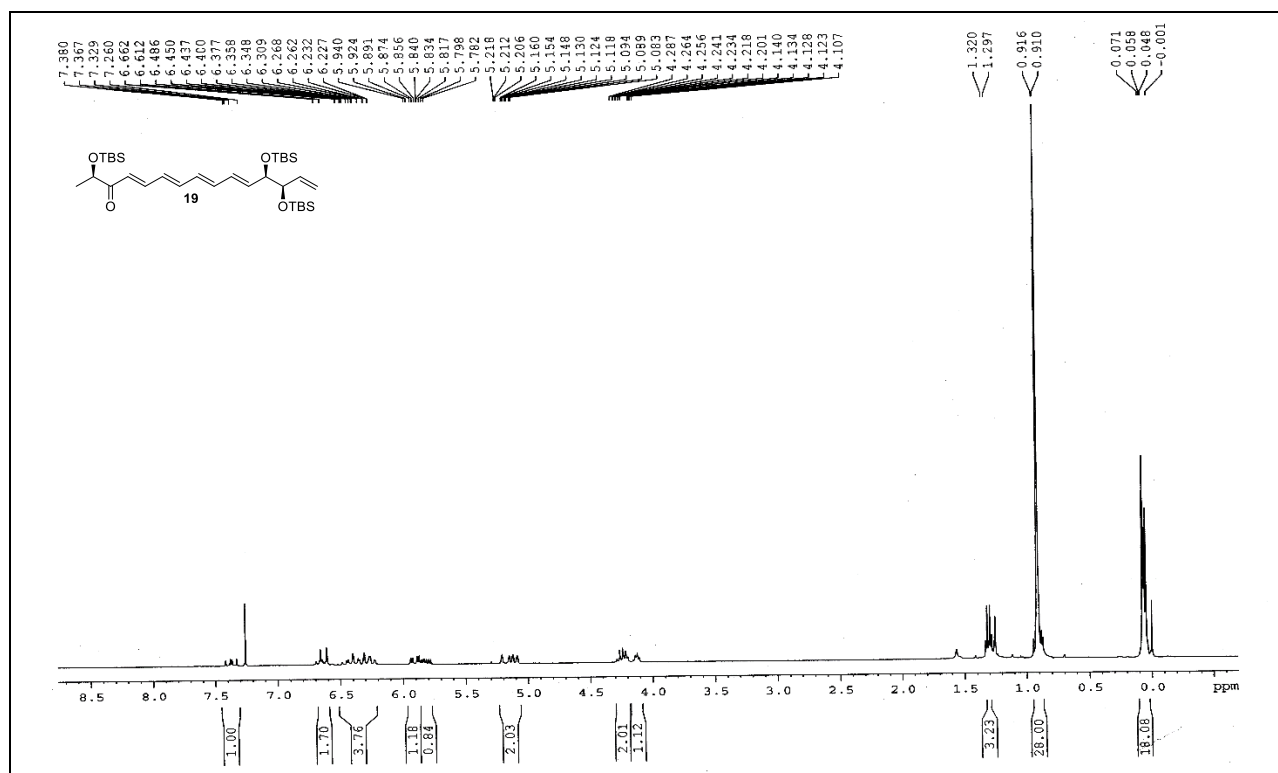
¹H NMR spectrum of 18 (300 MHz, CDCl₃):



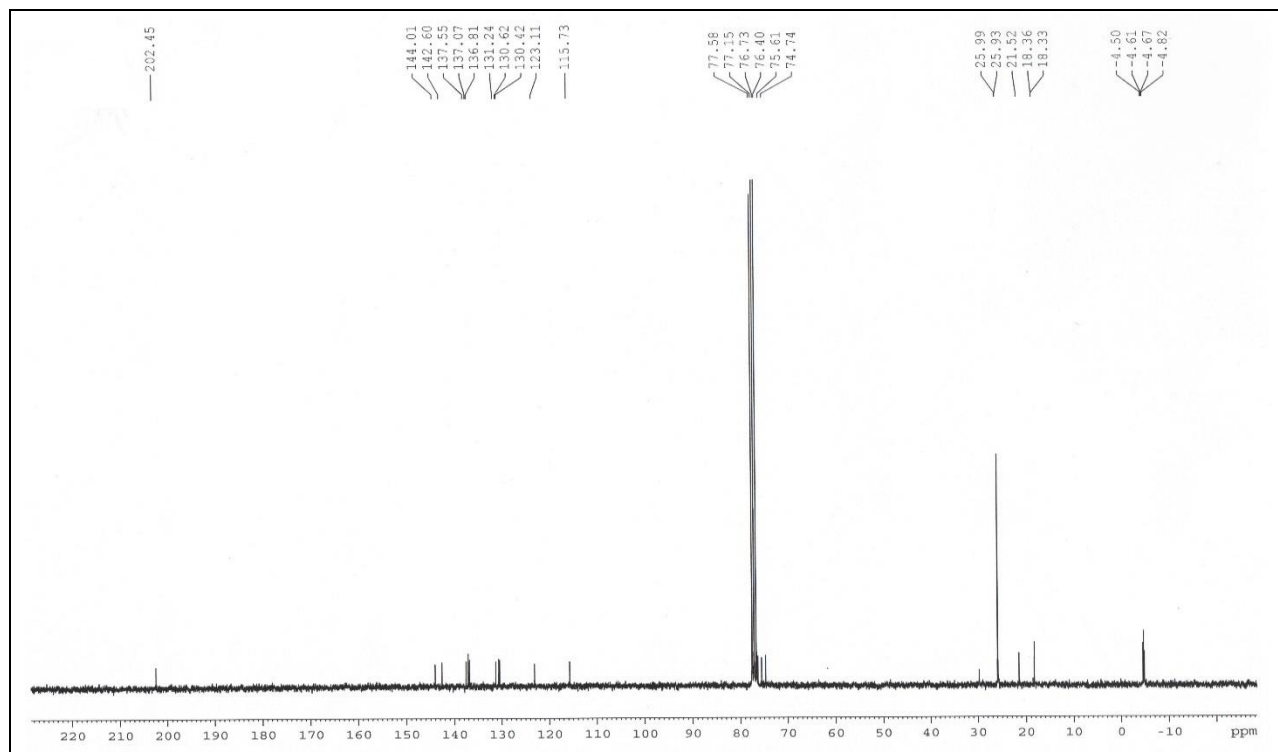
¹³C NMR spectrum of 18 (75 MHz, CDCl₃):



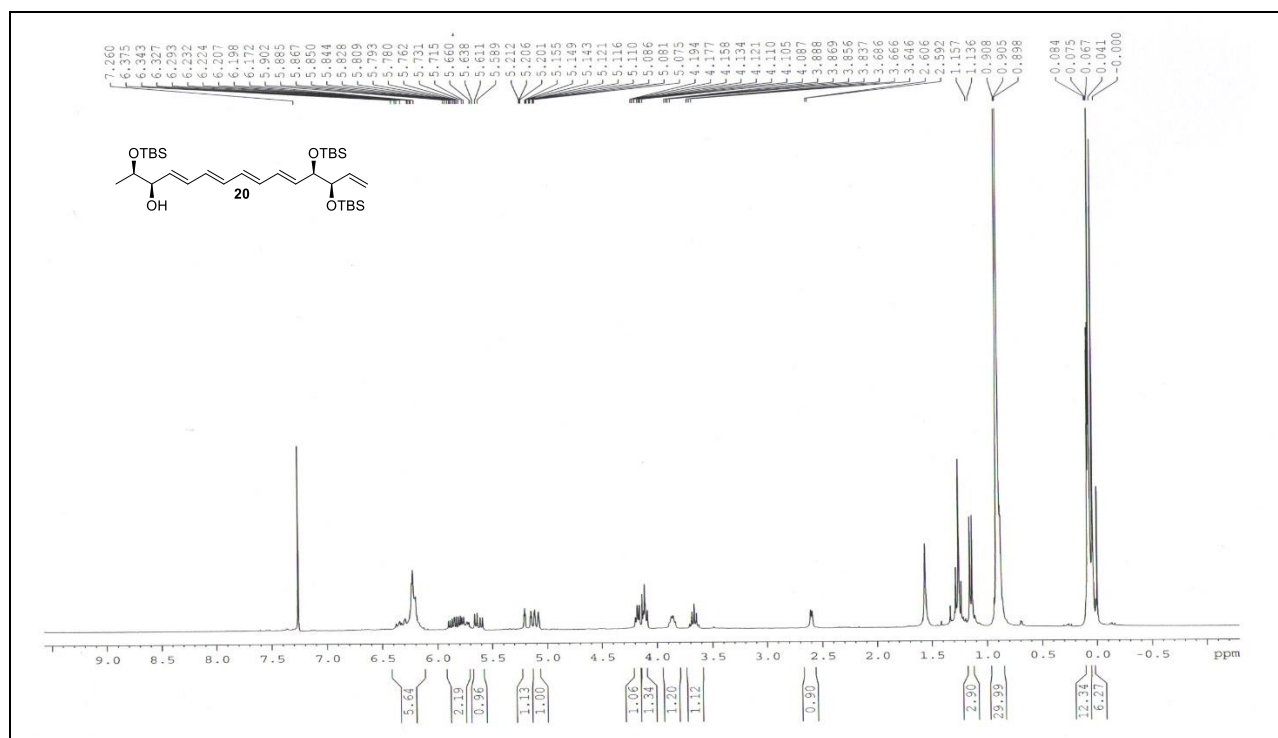
^1H NMR spectrum of 19 (300 MHz, CDCl_3):



^{13}C NMR spectrum of 19 (75 MHz, CDCl_3):



^1H NMR spectrum of 20 (300 MHz, CDCl_3):



Chemical structure of compound **21** is shown above the spectrum. The structure is a long-chain polyene with two OTBS groups and a terminal hydroxyl group.

¹H NMR spectrum (CDCl₃) of compound **21**. The x-axis represents chemical shift (ppm) from 9.0 to -0.5. The spectrum shows several peaks, with integrations provided below the baseline and a list of chemical shifts (δ) on the right.

Chemical shifts (ppm) listed on the right:

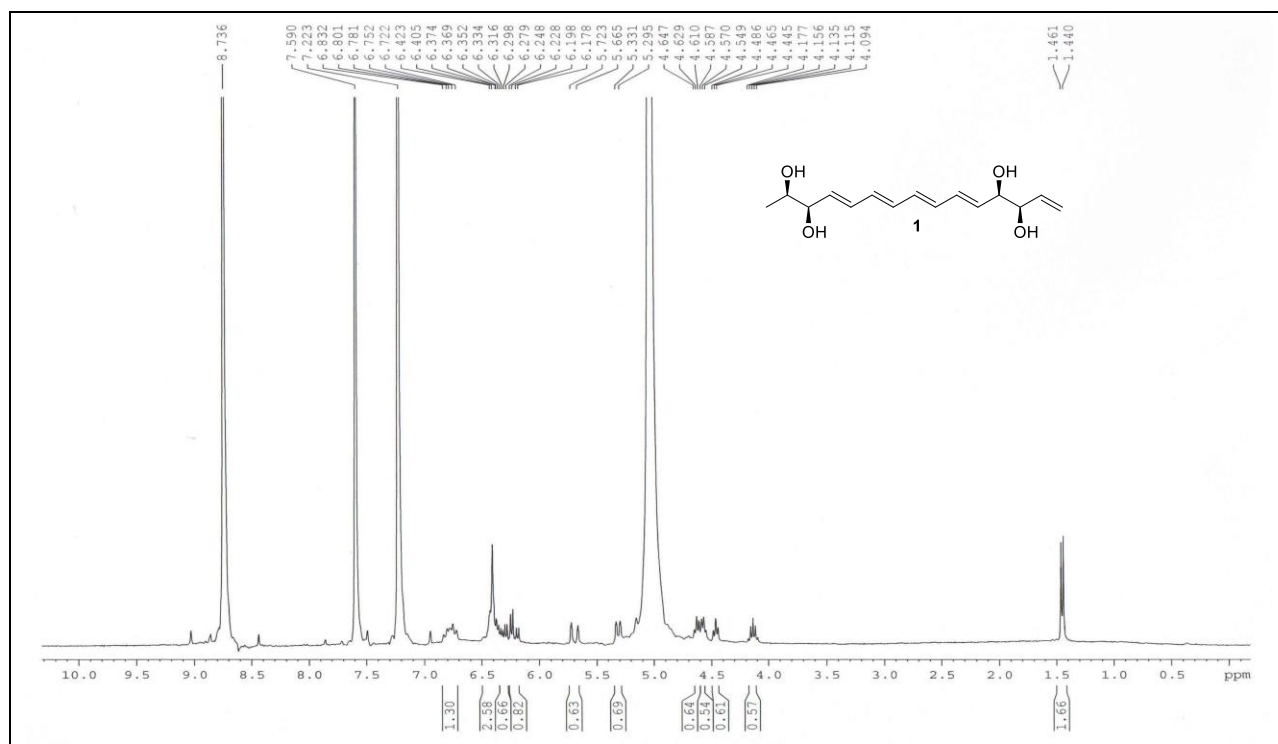
- 7.260, 6.375, 6.344, 6.327, 6.294, 6.225, 3.922, 3.869, 3.851, 3.845, 5.828, 5.610, 5.793, 5.760, 5.730, 5.713, 5.664, 5.642, 5.615, 5.593, 5.571, 5.507, 5.501, 5.155, 5.149, 5.143, 5.122, 5.117, 5.087, 5.082, 4.997, 4.180, 4.164, 4.125, 4.081, 4.092, 3.862, 3.692, 3.672, 3.652, 2.579
- 1.159, 1.139, 0.911, 0.908, 0.902
- 0.086, 0.078, 0.069, 0.043, 0.003

Integrations (from left to right):

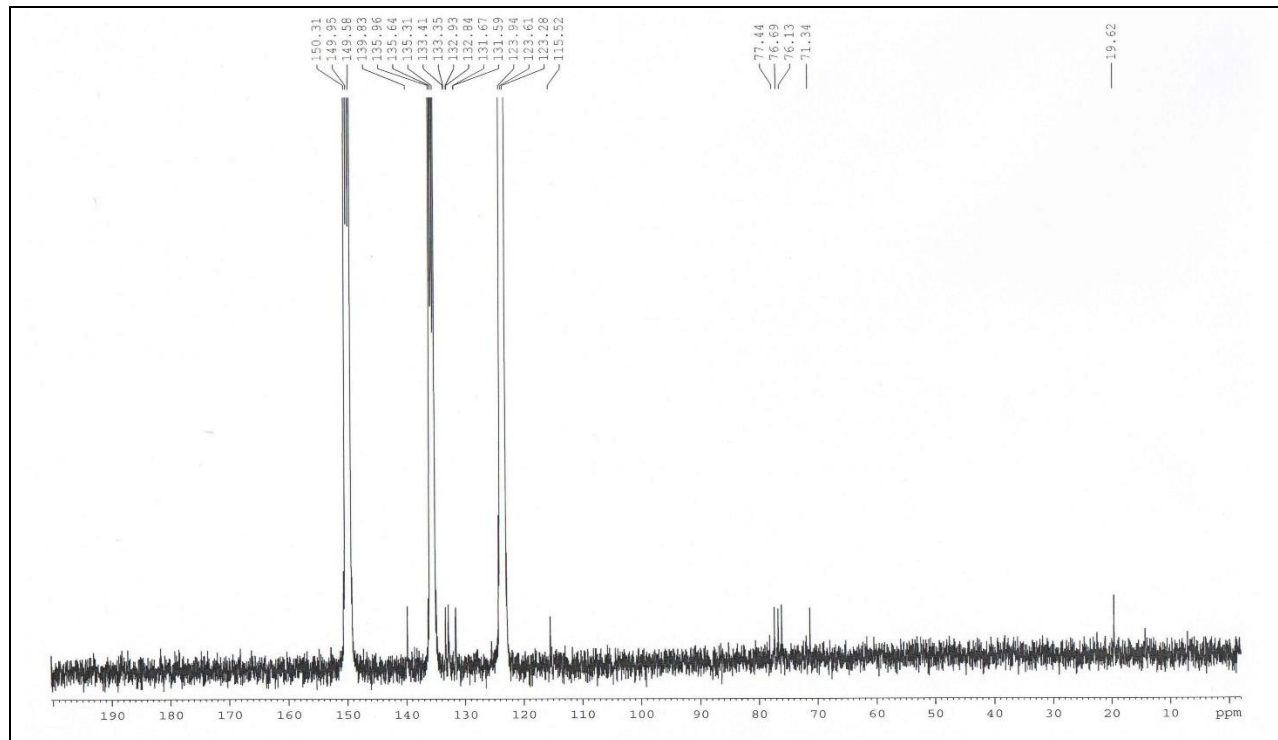
- 5.28
- 2.00
- 0.92
- 2.10
- 1.01, 1.06, 1.07, 1.13
- 1.02
- 3.09, 28.47
- 11.36, 6.06

137.30
133.44
133.47
133.33
132.57
132.52
131.99
131.93
130.94
— 115.58
77.59
77.16
77.00
76.84
76.44
72.82
72.55
26.01
25.96
20.23
18.36
18.19
-4.09
-4.48
-4.59
-4.54

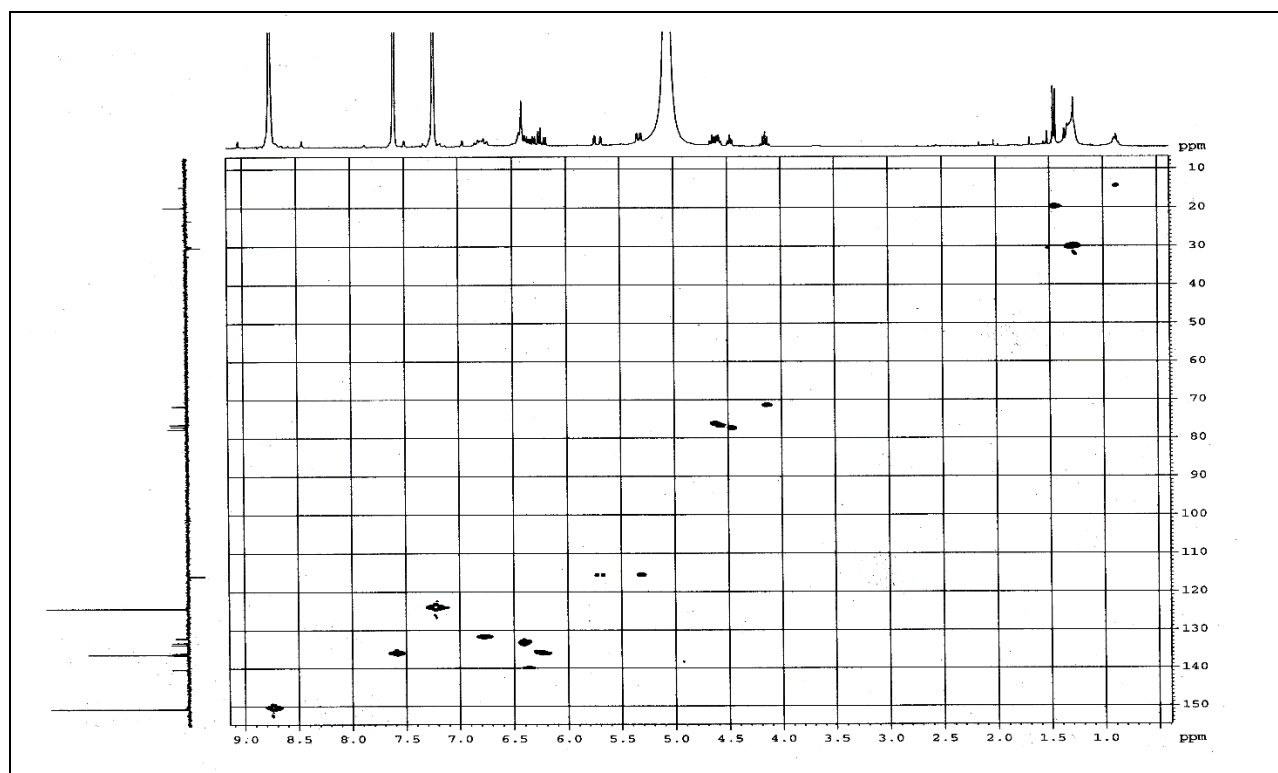
^1H NMR spectrum of 1 (300 MHz, $\text{C}_5\text{D}_5\text{N}$):



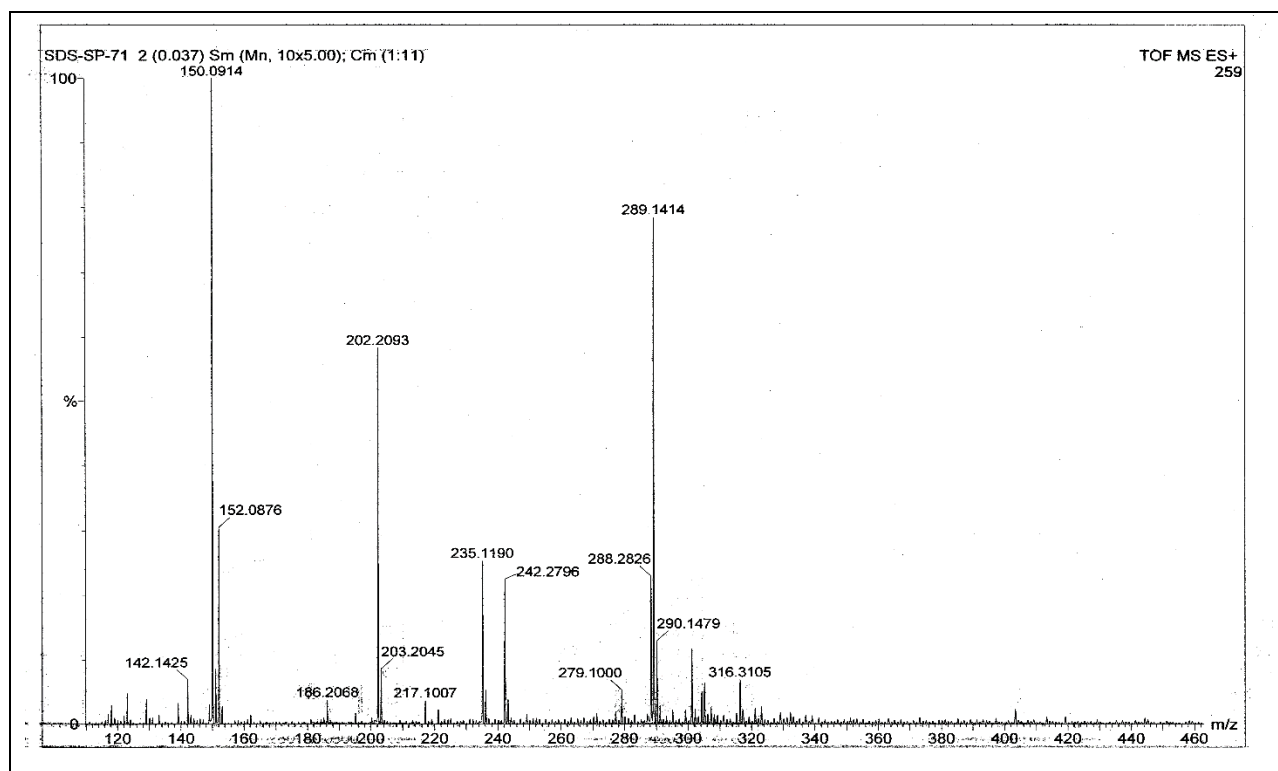
^{13}C NMR spectrum of 1 (75 MHz, $\text{C}_5\text{D}_5\text{N}$):



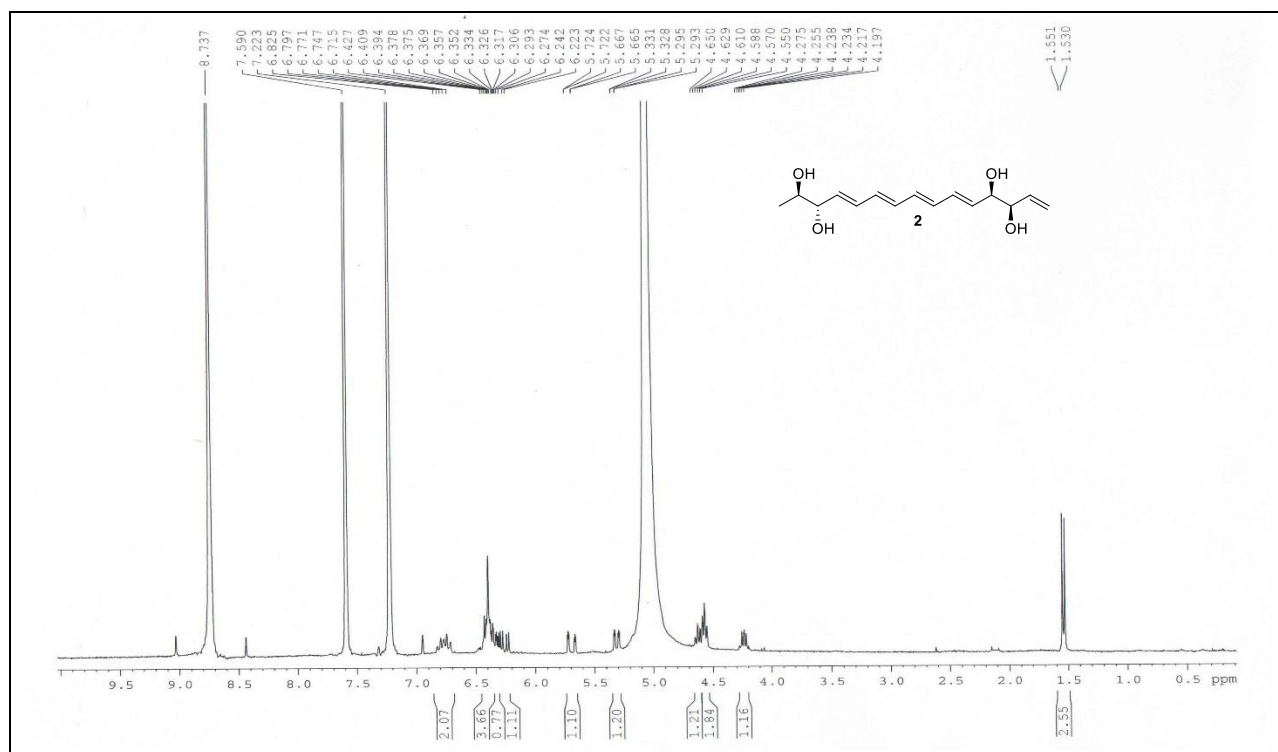
HSQC spectrum of 1:



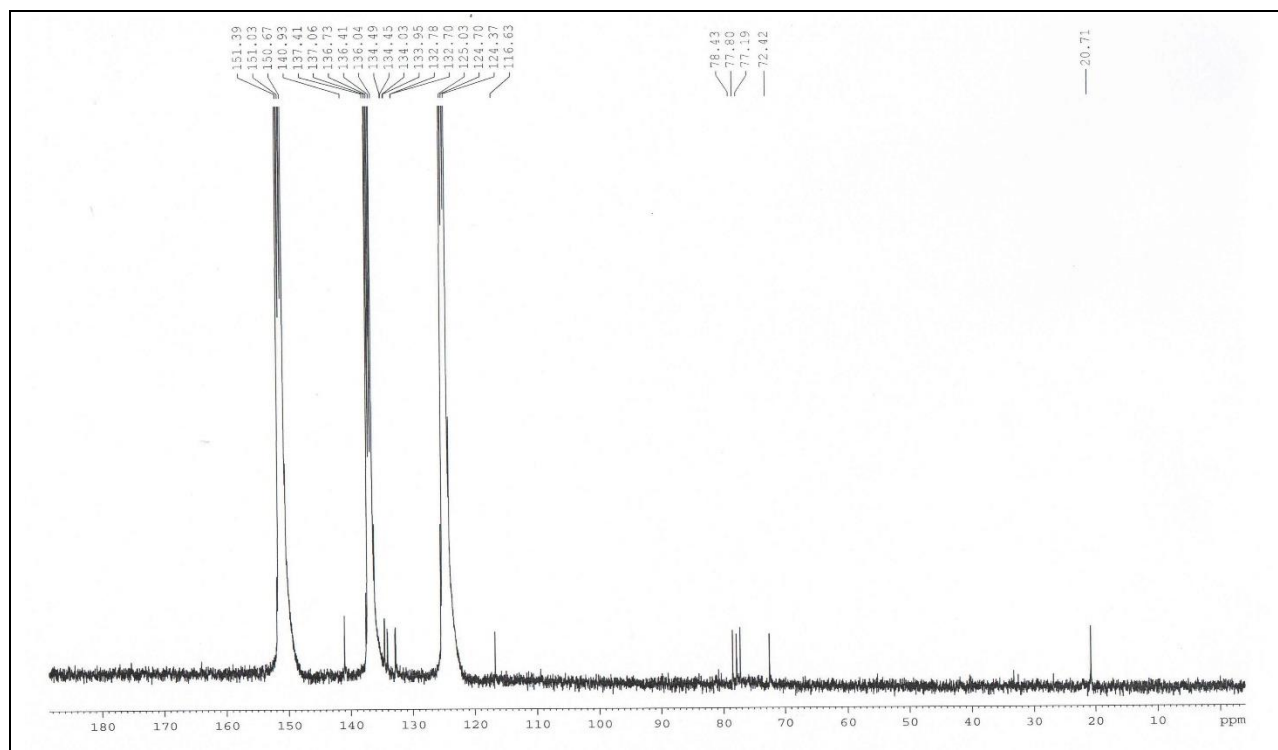
HRMS spectrum of 1:



^1H NMR spectrum of 2 (300 MHz, $\text{C}_5\text{D}_5\text{N}$):



^{13}C NMR spectrum of 21 (75 MHz, $\text{C}_5\text{D}_5\text{N}$):



HSQC spectrum of 2:

