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

Supporting Information

Total Synthesis of Marine Natural Products Separacenes A and B

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Table S1: ^{1}H and ^{13}C NMR comparison of Separacenes A (natural, C_5D_5N, 900 MHz), compound 1 (C_5D_5N, 300 MHz) δ in ppm.

^{1}H NMR of Natural Separacenes A	^{1}H 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 C_5D_5N 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 C_5D_5N 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

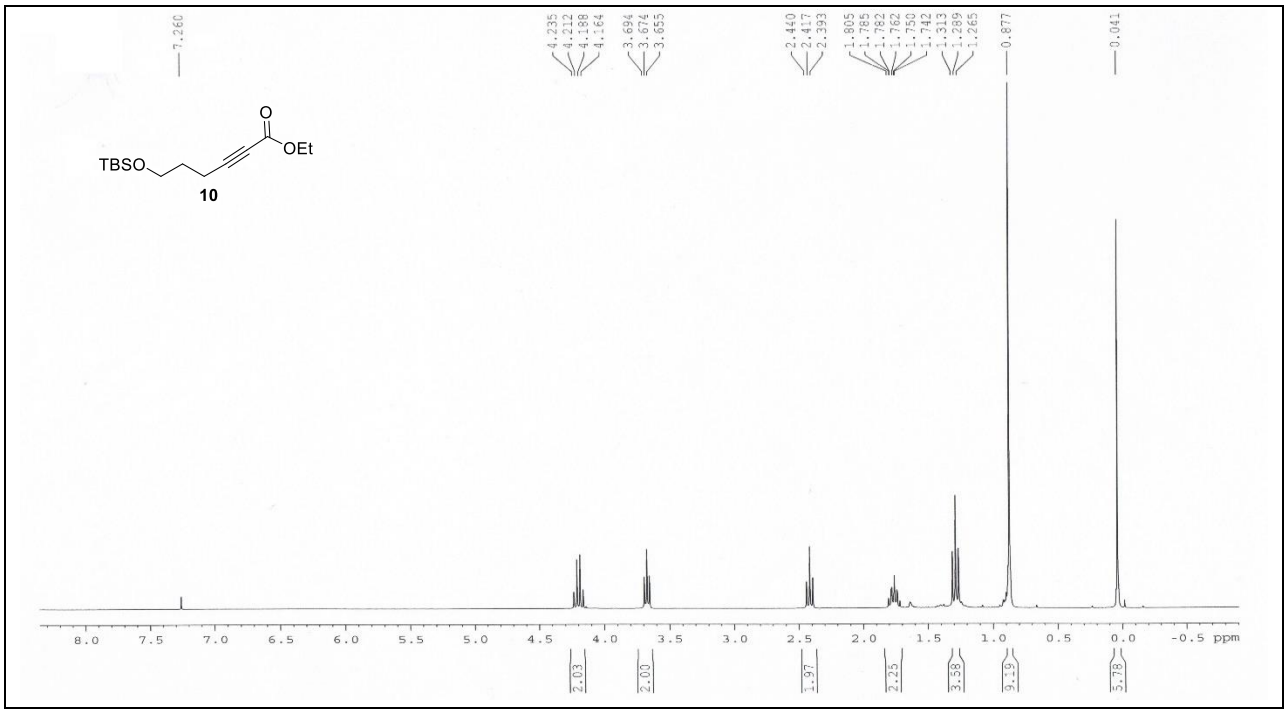
Table S2: ^{1}H and ^{13}C NMR comparison of Separacenes B (natural, C_5D_5N, 600 MHz), compound 1 (C_5D_5N, 300 MHz) δ in ppm.

^{1}H NMR of Natural Separacenes B	**^{1}H 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 C_5D_5N 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

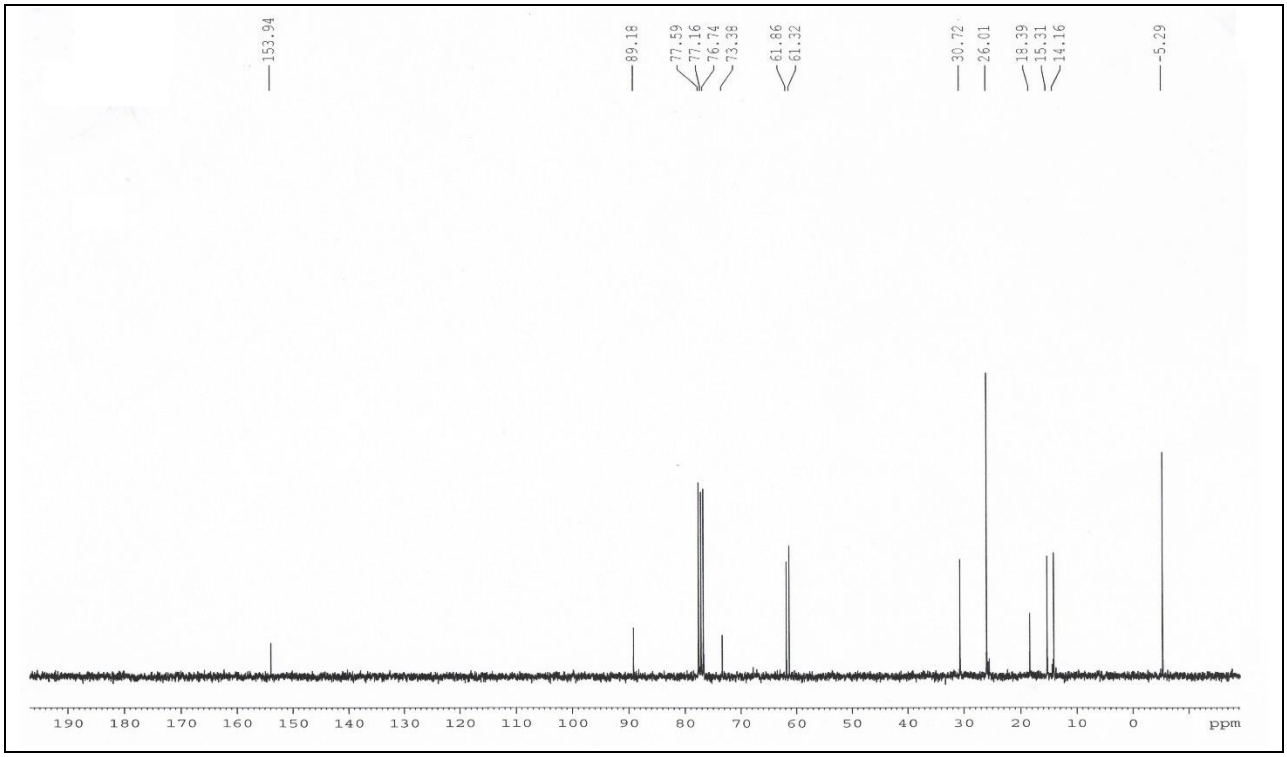
5.31, dd (10.5, 1.0), 1H	5.31, dd (10.8, 0.9), 1H	133.7	134.0
4.63, dd (11.0, 6.0), 1H	4.65-4.61, m, 1H	133.3	133.9
4.58, m, 1H 4.56, m, 1H	4.59-4.55, m, 2H	132.8	132.8
4.24, dd (11.0, 5.5), 1H	4.27-4.19, m, 1H	132.3	132.7
1.54, d (5.5), 3H	1.54, d (6.3), 3H	115.4	116.6
		78.2	78.4
		77.8	77.8
		77.3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

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

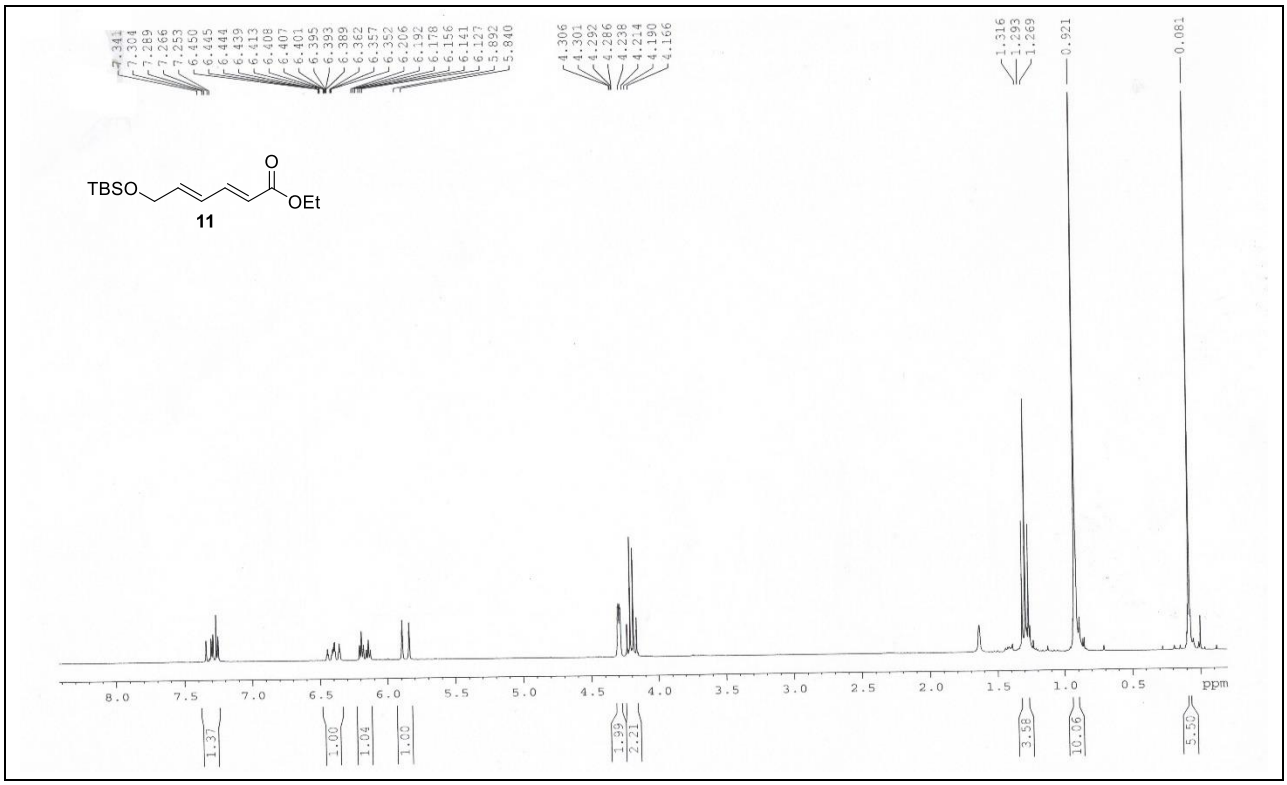
^{1}H NMR spectrum of 10 (300 MHz, $CDCl_3$):



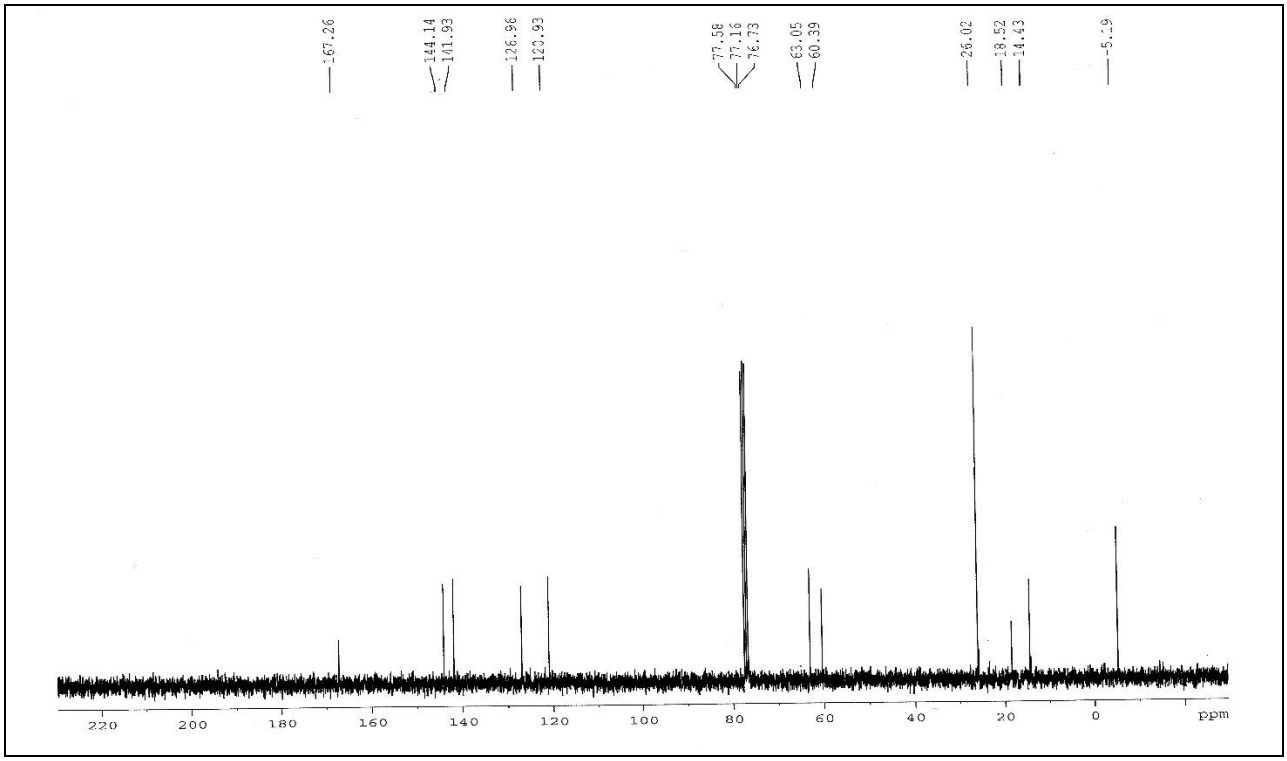
^{13}C NMR spectrum of 10 (75 MHz, $CDCl_3$):



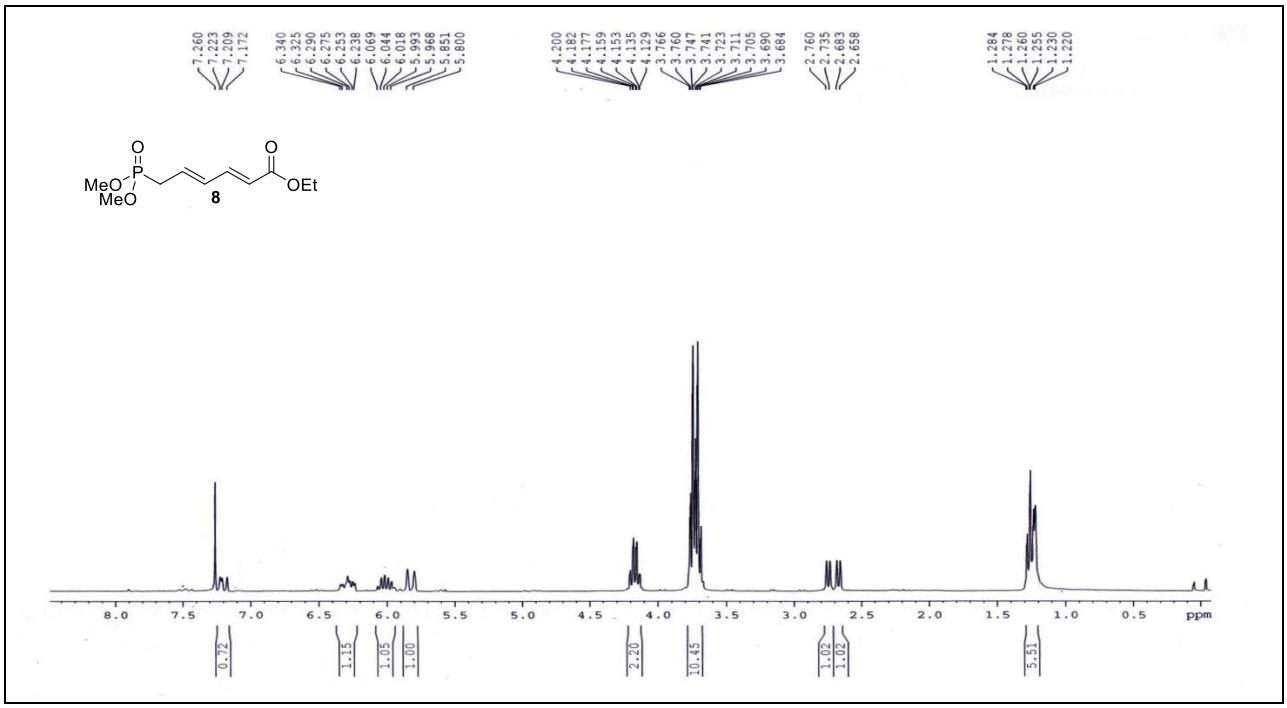
^{1}H NMR spectrum of 11 (300 MHz, $CDCl_3$):



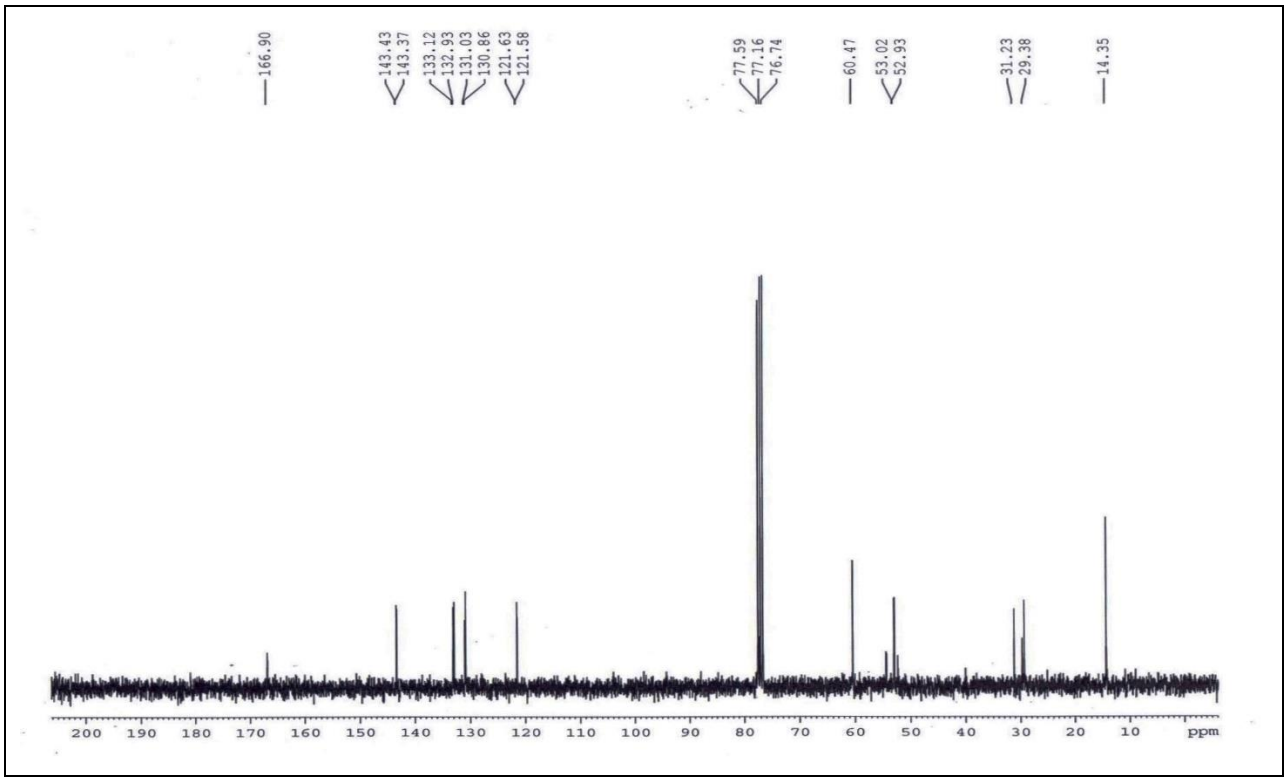
^{13}C NMR spectrum of 11 (75 MHz, $CDCl_3$):



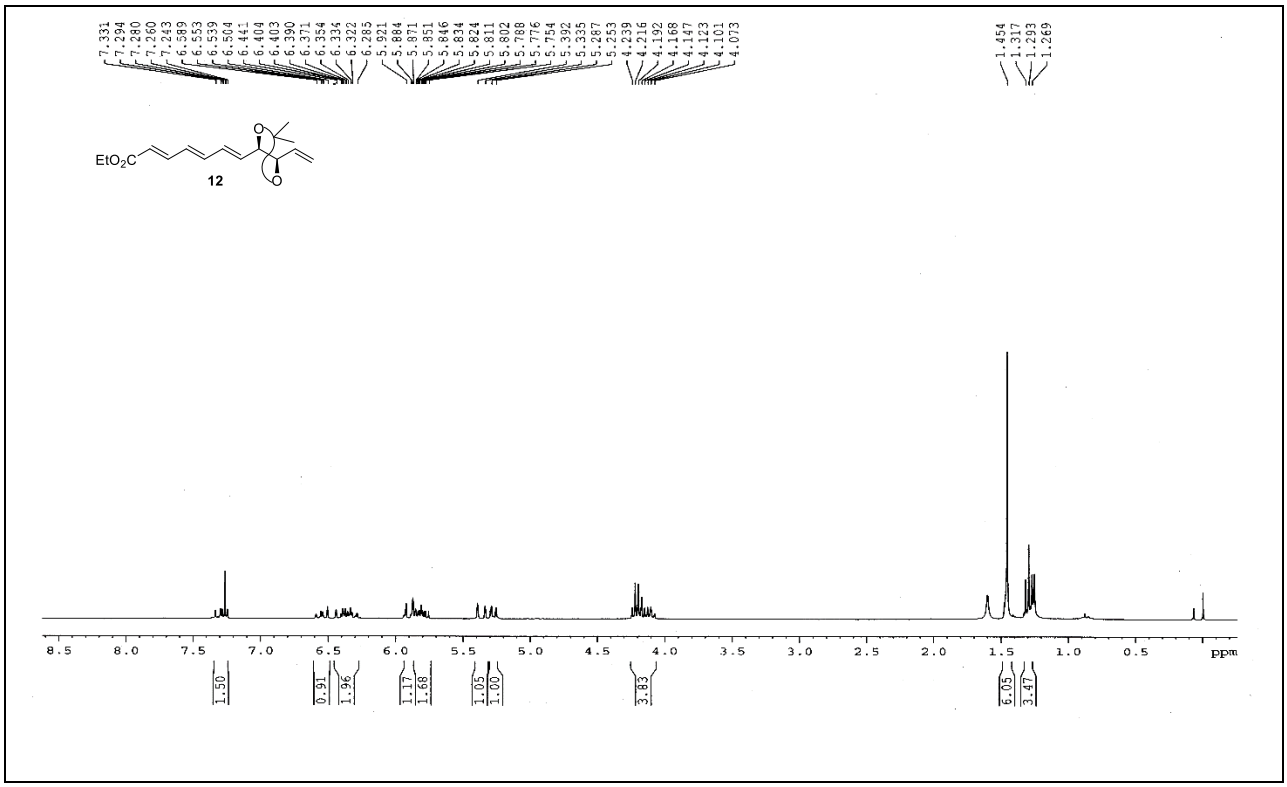
^{1}H NMR spectrum of 8 (300 MHz, $CDCl_3$):



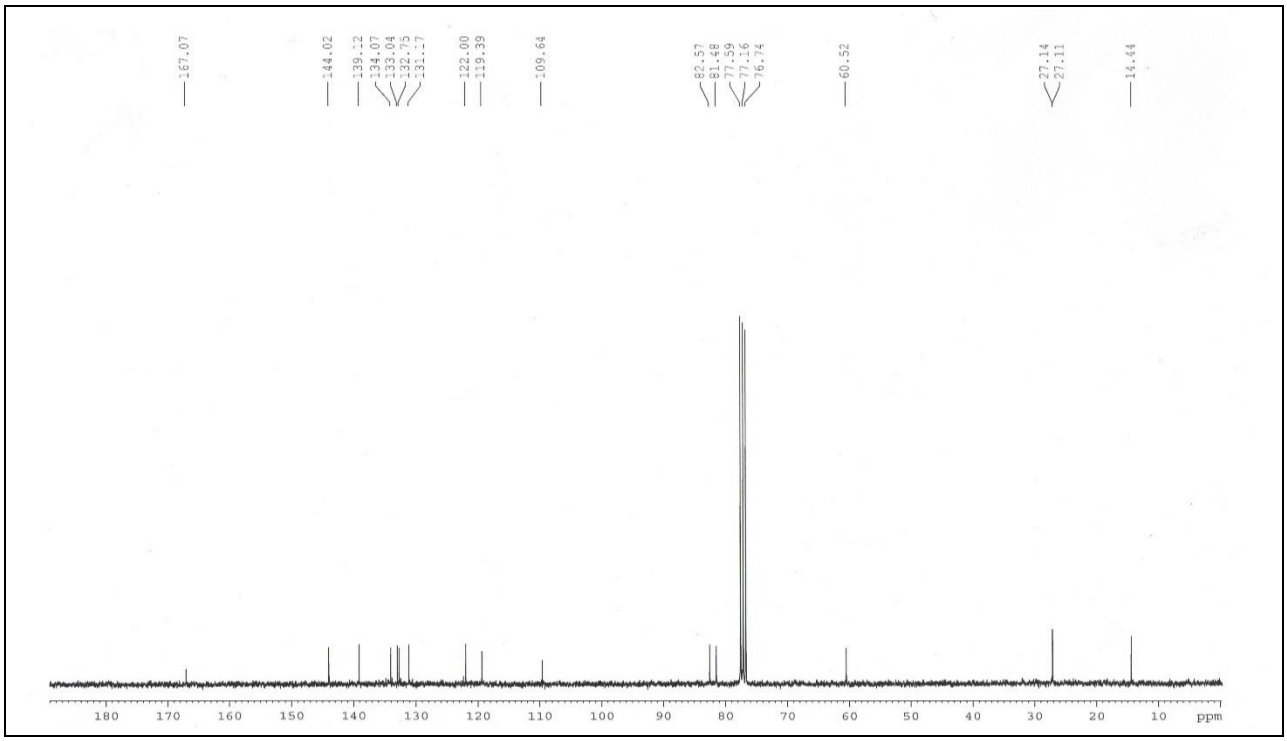
^{13}C NMR spectrum of 8 (75 MHz, $CDCl_3$):



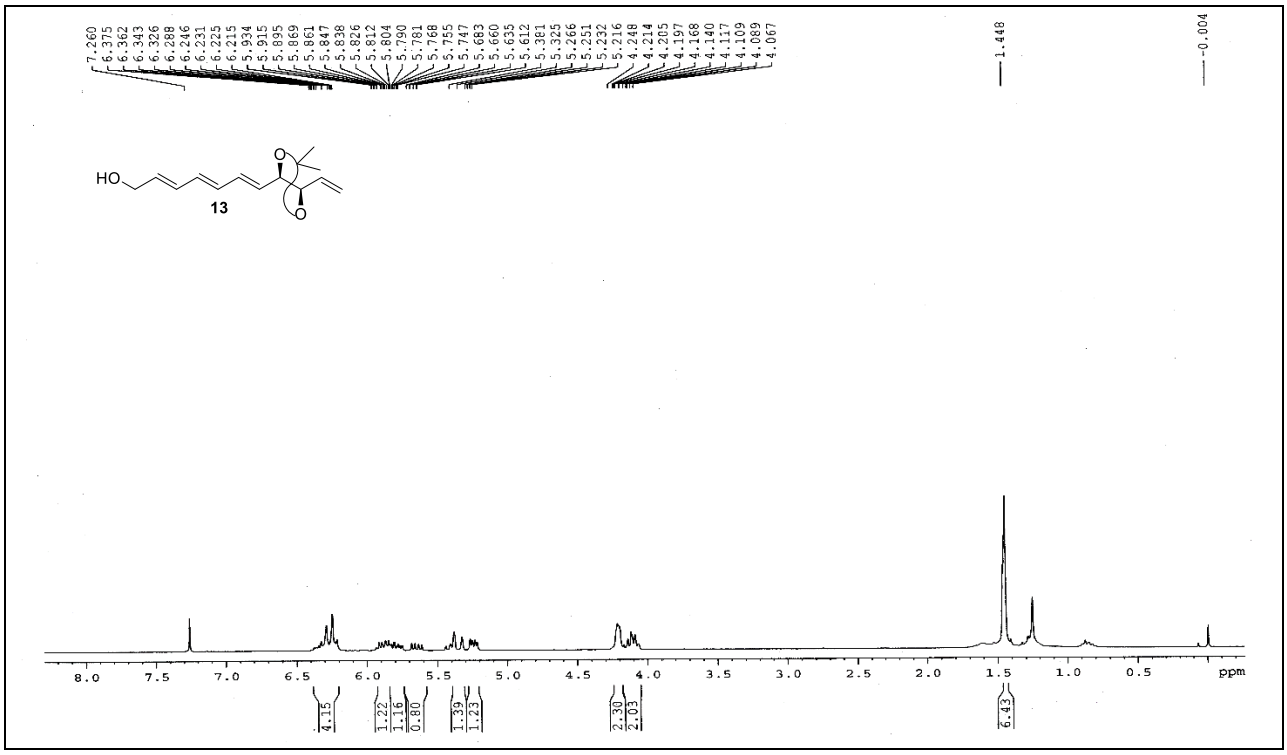
^{1}H NMR spectrum of 12 (300 MHz, $CDCl_3$):



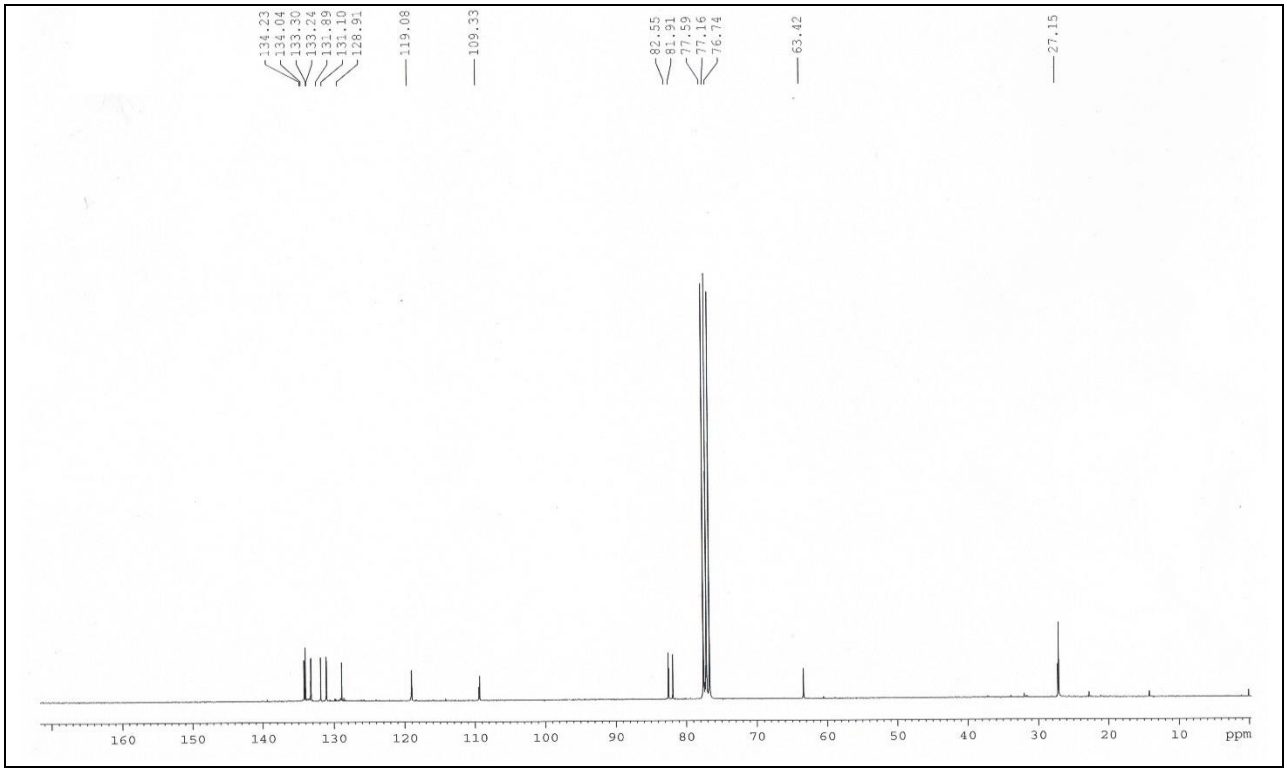
^{13}C NMR spectrum of 12 (75 MHz, $CDCl_3$):



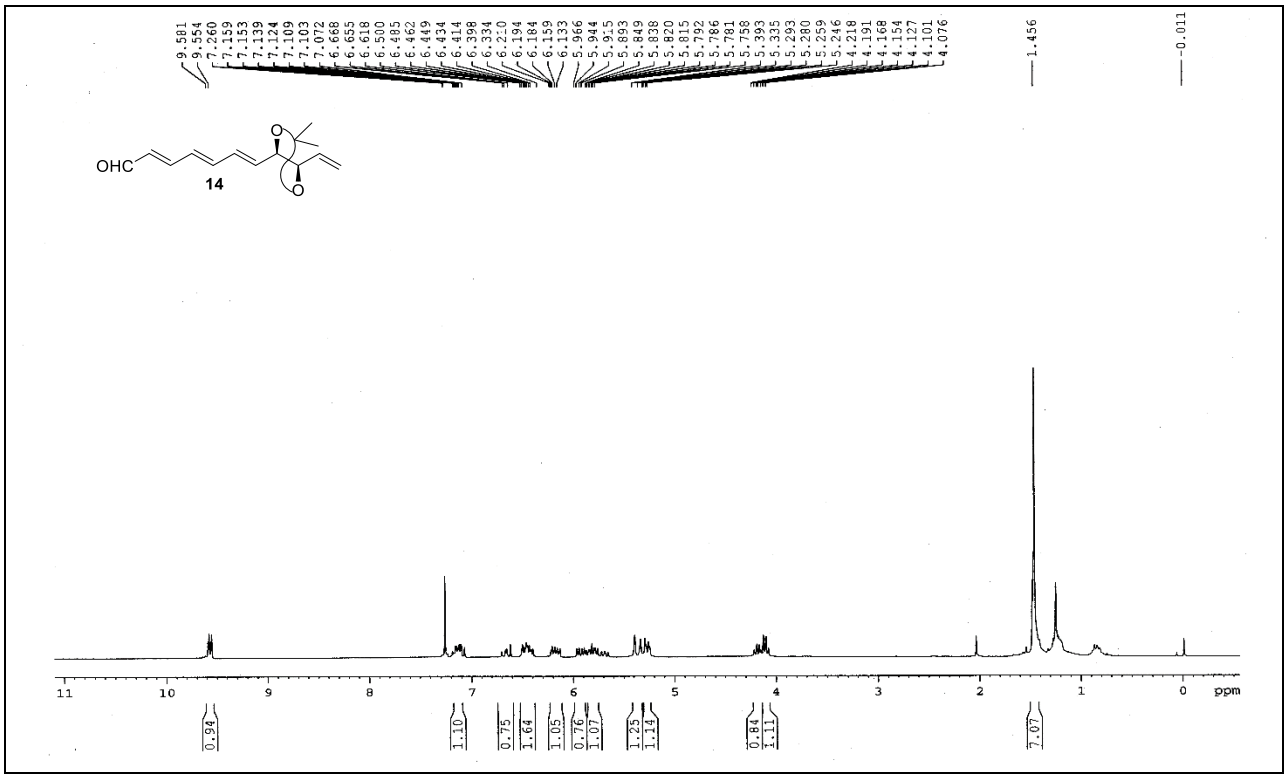
^{1}H NMR spectrum of 13 (300 MHz, $CDCl_3$):



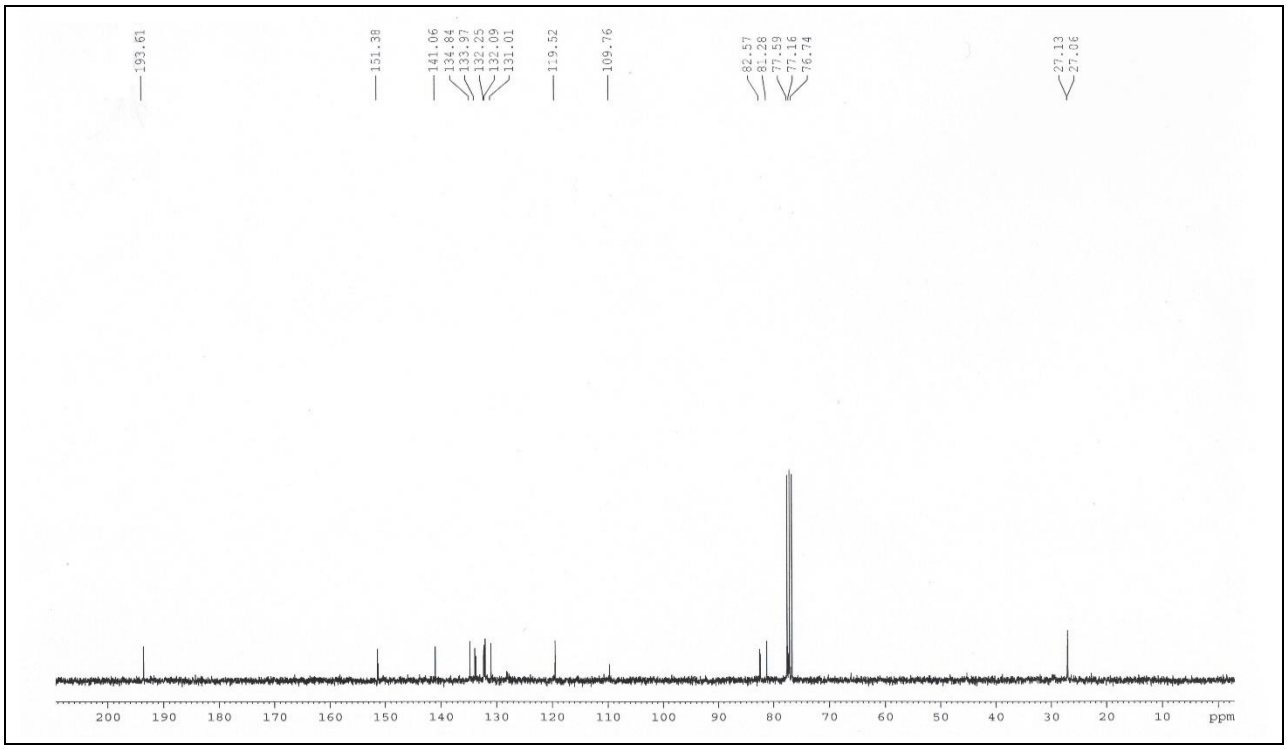
^{13}C NMR spectrum of 13 (75 MHz, $CDCl_3$):



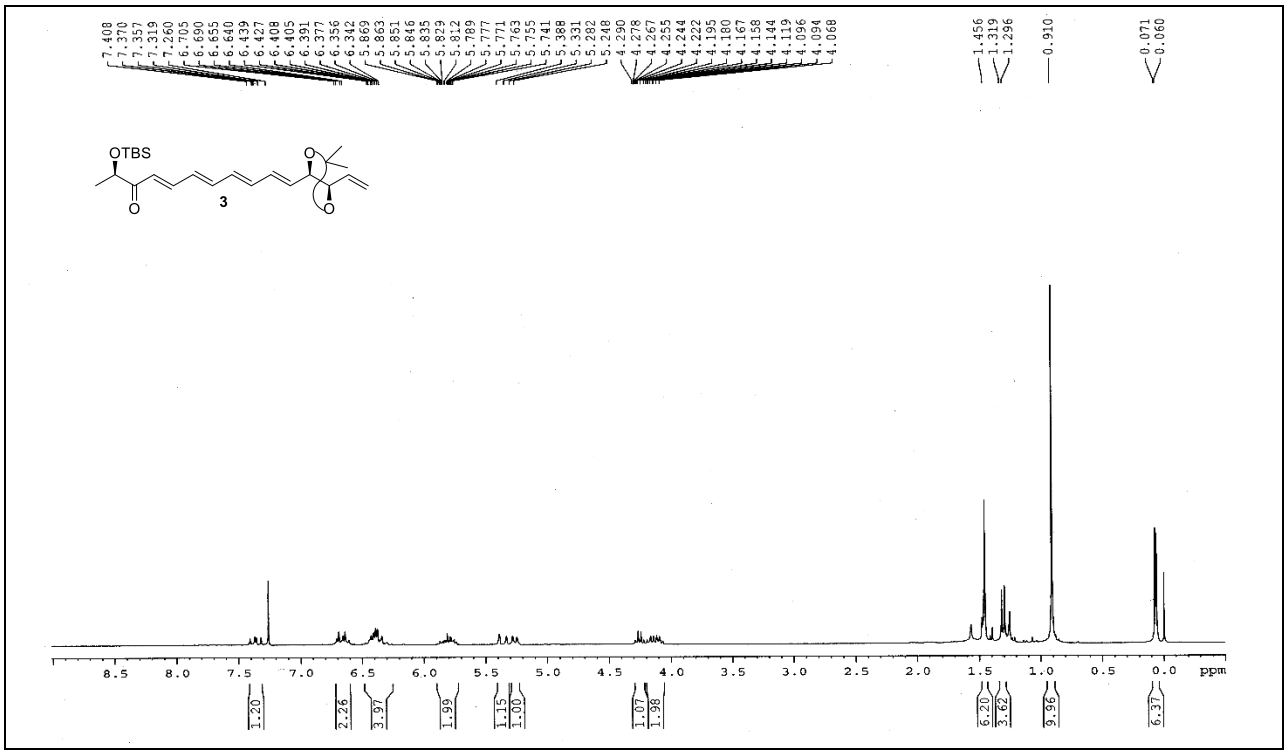
^{1}H NMR spectrum of 14 (300 MHz, $CDCl_3$):



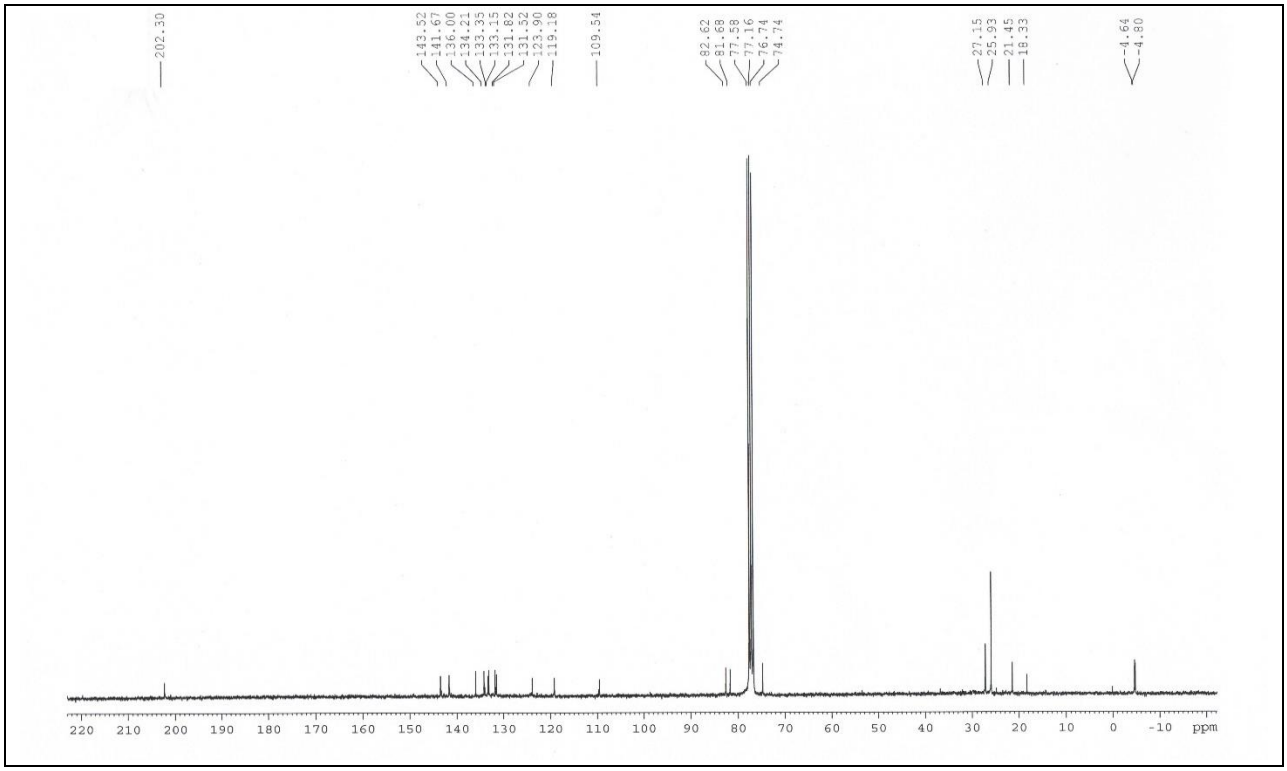
^{13}C NMR spectrum of 14 (75 MHz, $CDCl_3$):



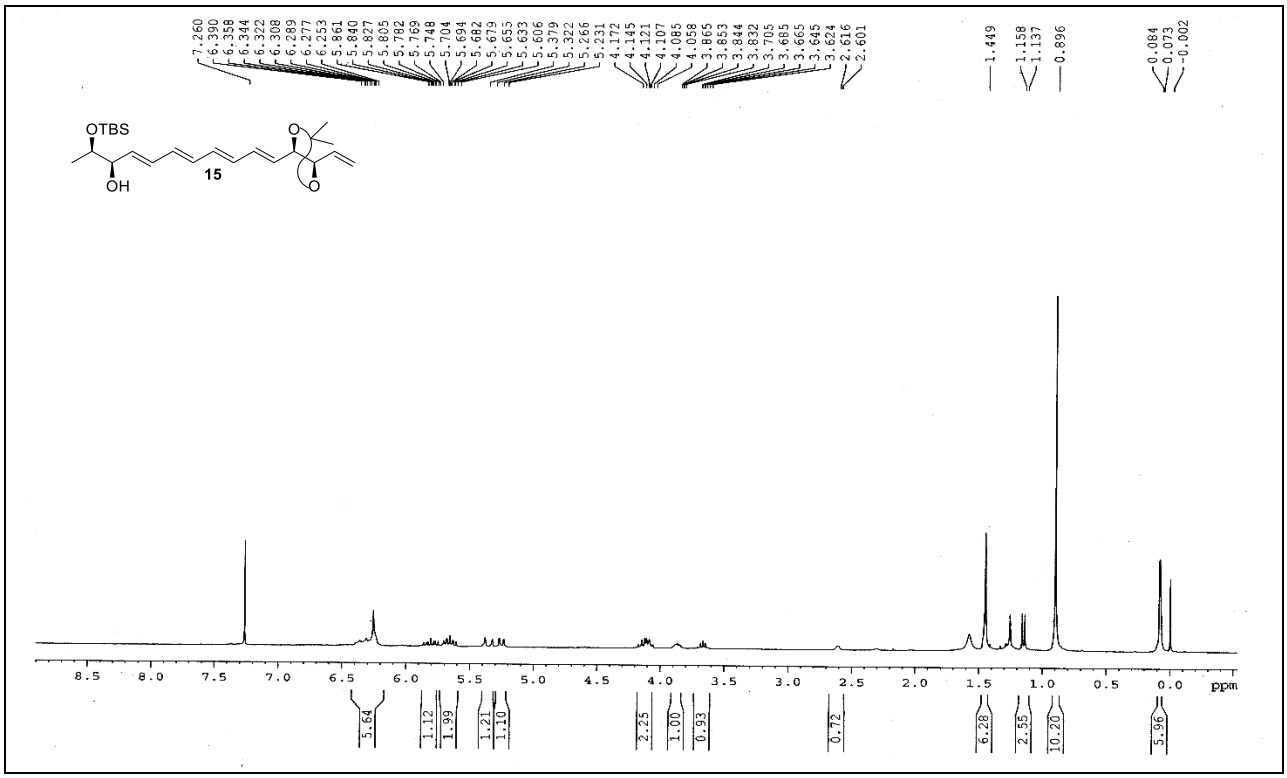
^{1}H NMR spectrum of 3 (300 MHz, $CDCl_3$):



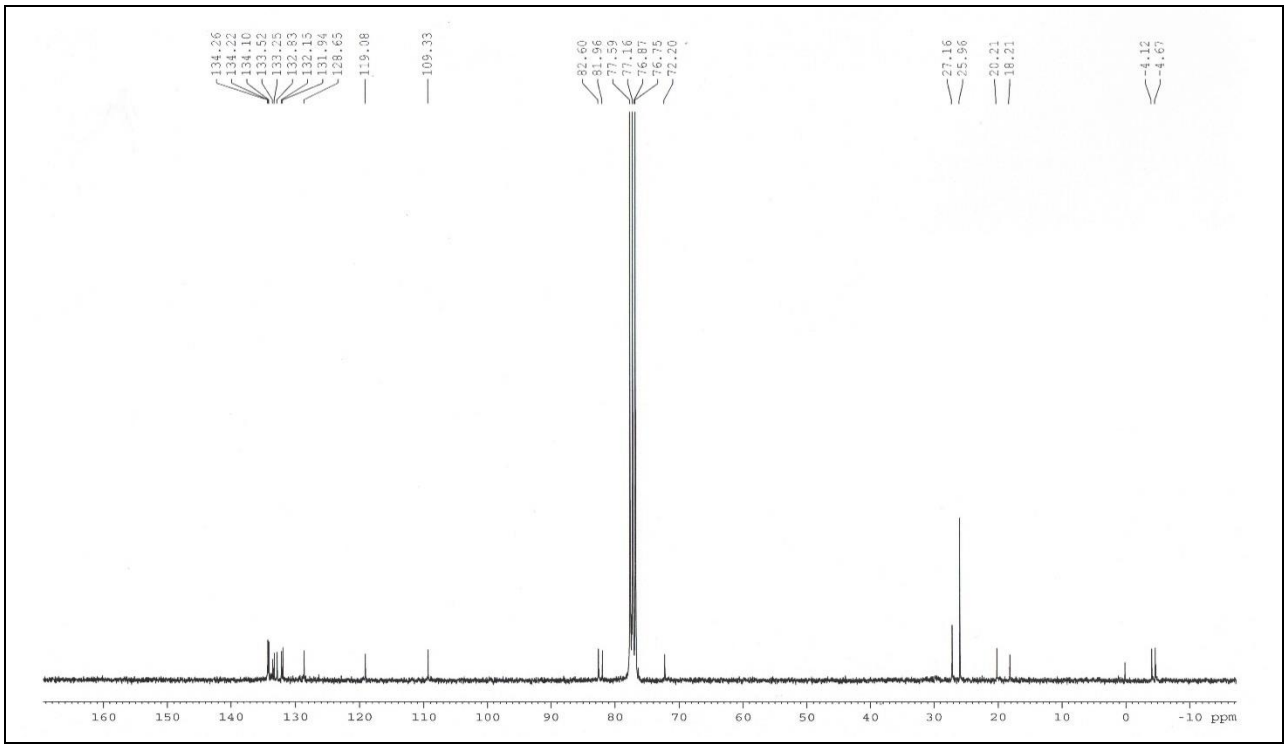
^{13}C NMR spectrum of 3 (75 MHz, $CDCl_3$):



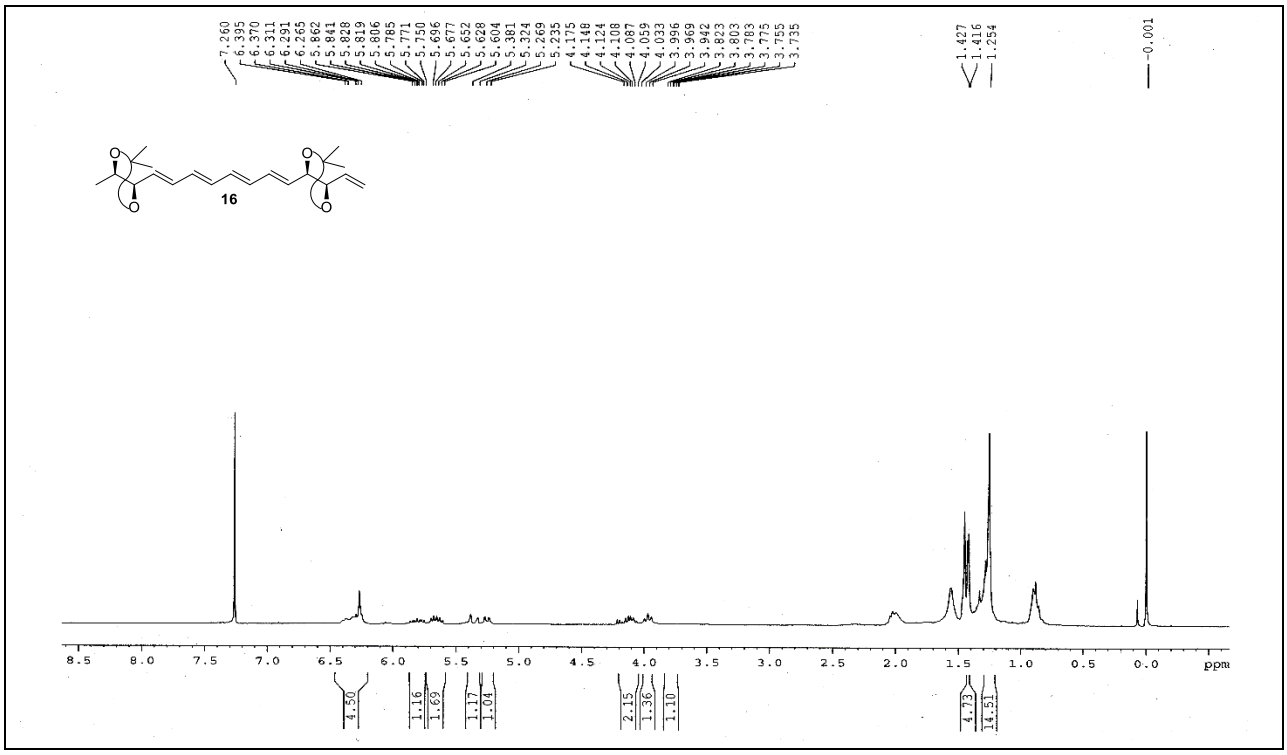
^{1}H NMR spectrum of 15 (300 MHz, $CDCl_3$):



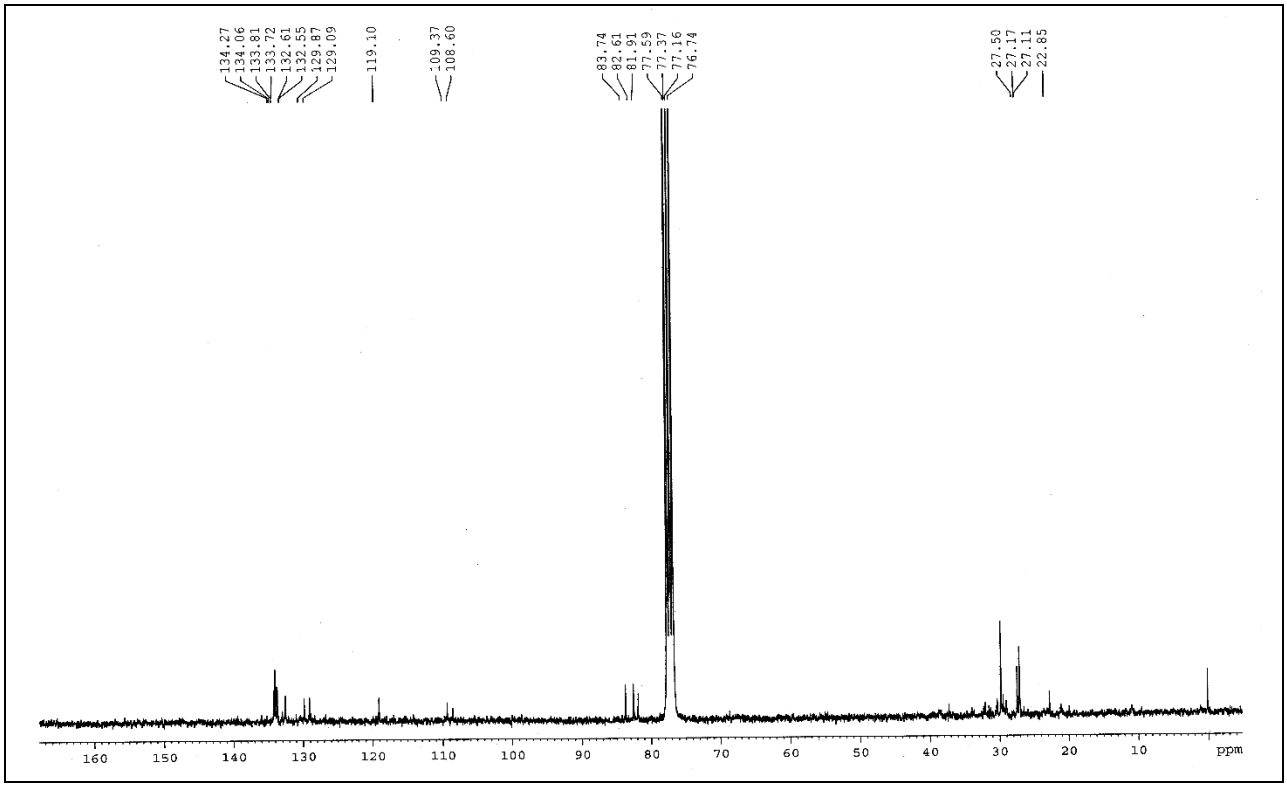
^{13}C NMR spectrum of 15 (75 MHz, $CDCl_3$):



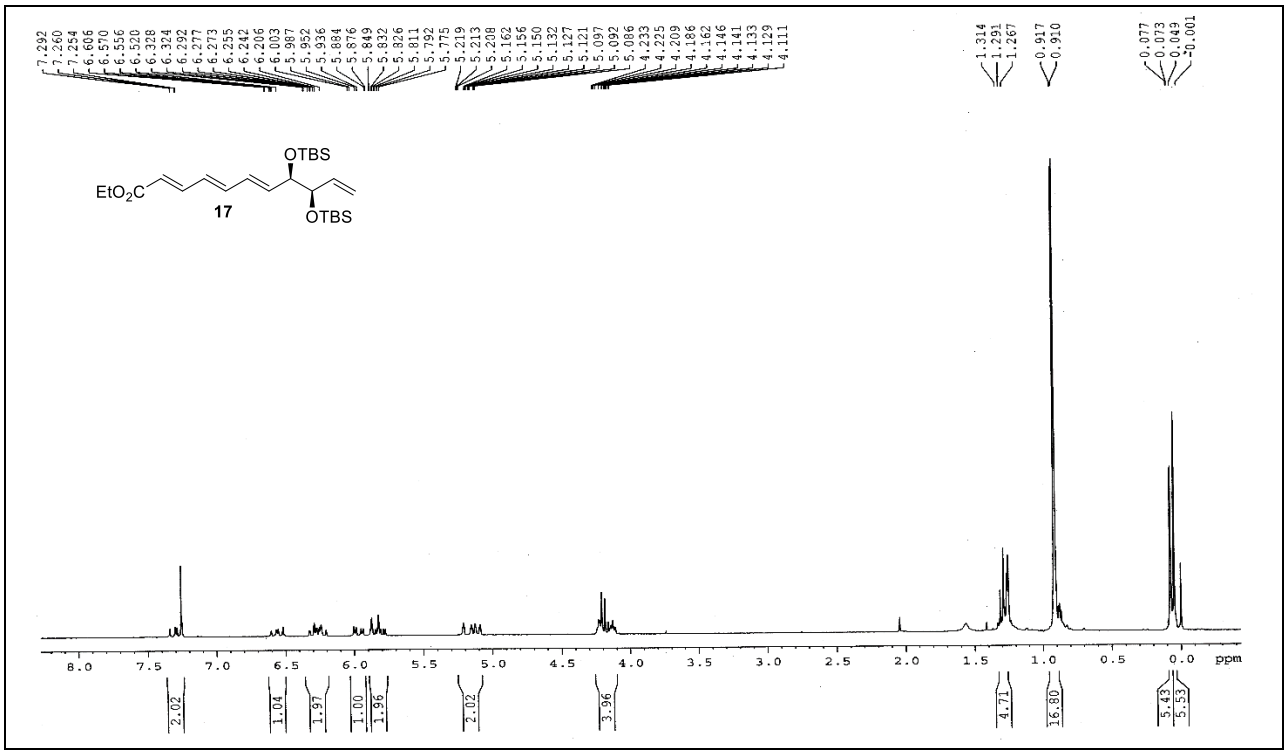
^{1}H NMR spectrum of 16 (300 MHz, $CDCl_3$):



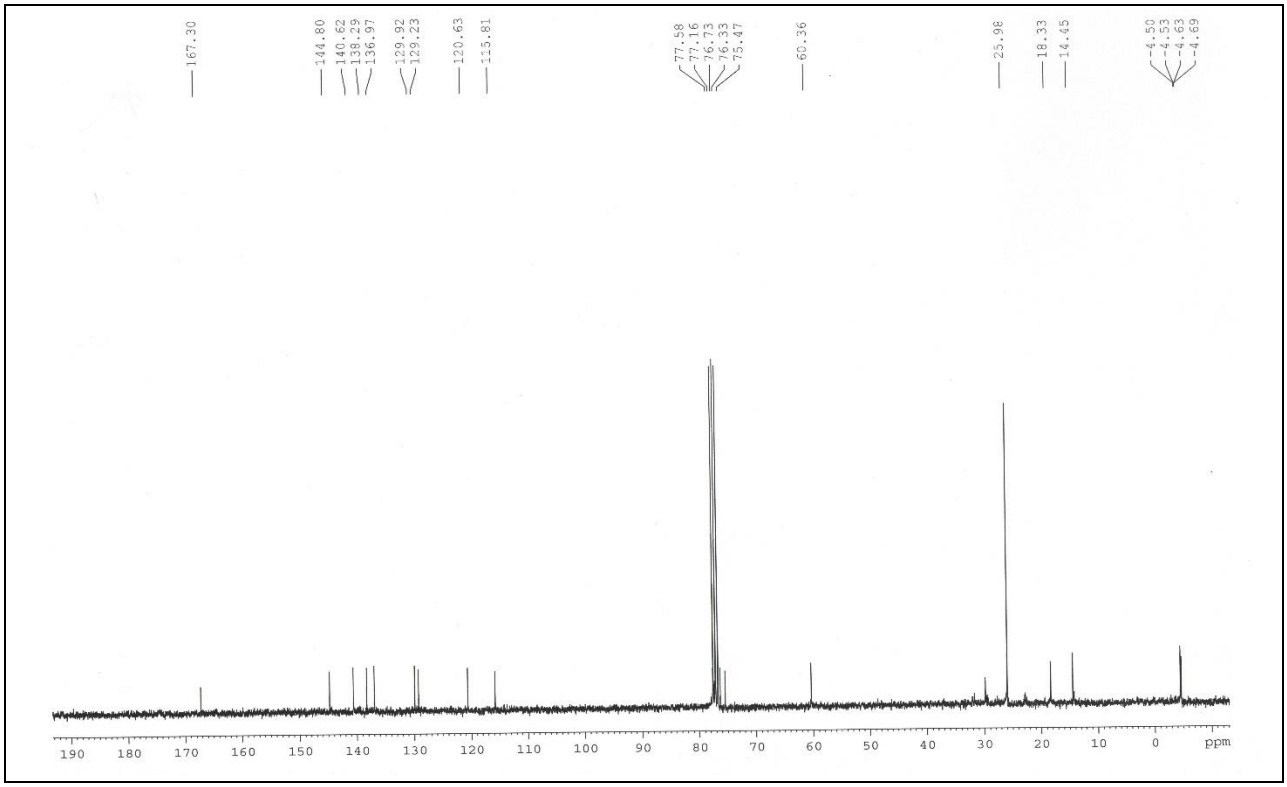
^{13}C NMR spectrum of 16 (75 MHz, $CDCl_3$):



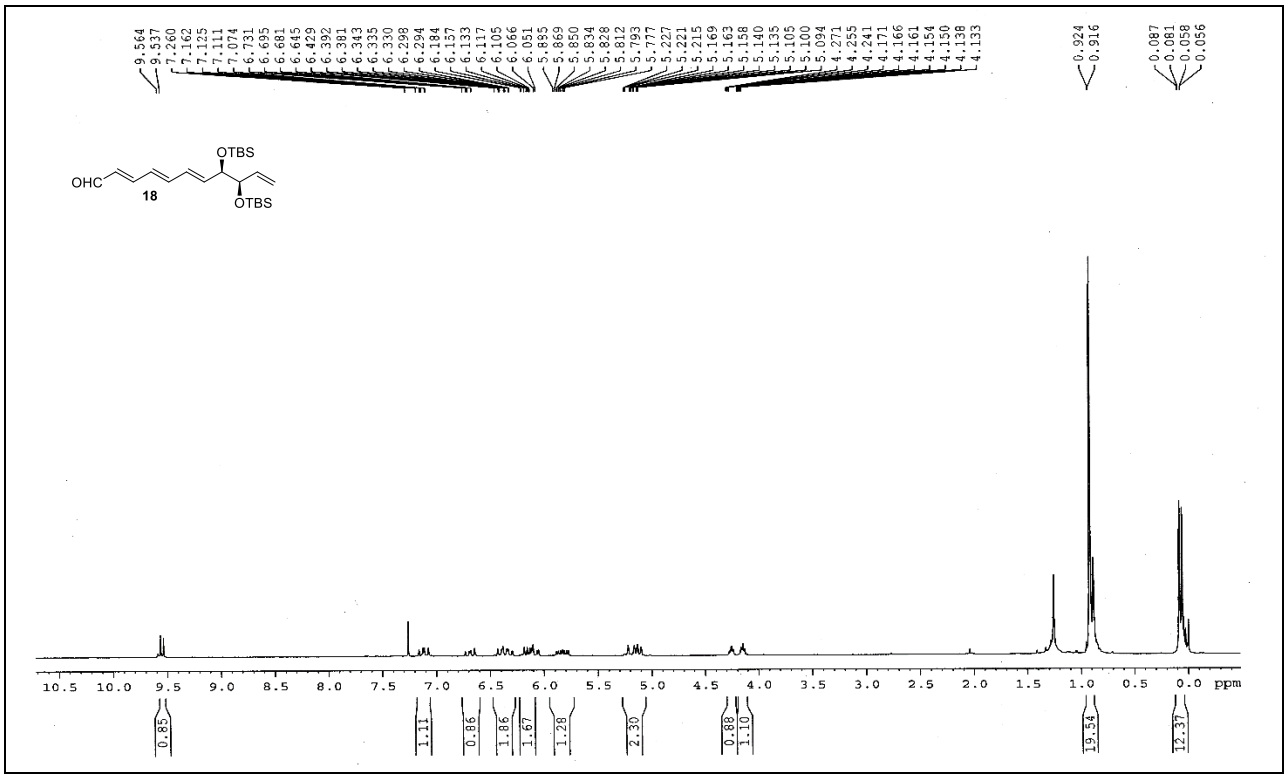
^{1}H NMR spectrum of 17 (300 MHz, $CDCl_3$):



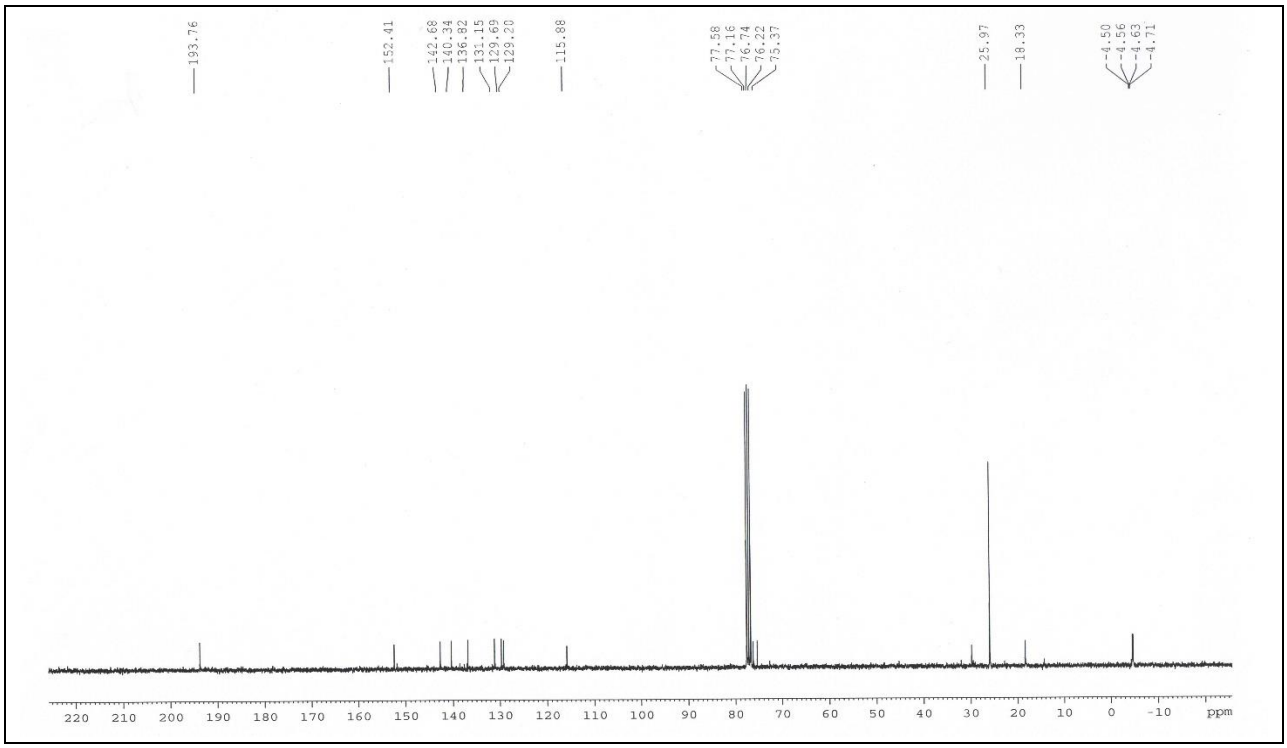
^{13}C NMR spectrum of 17 (75 MHz, $CDCl_3$):



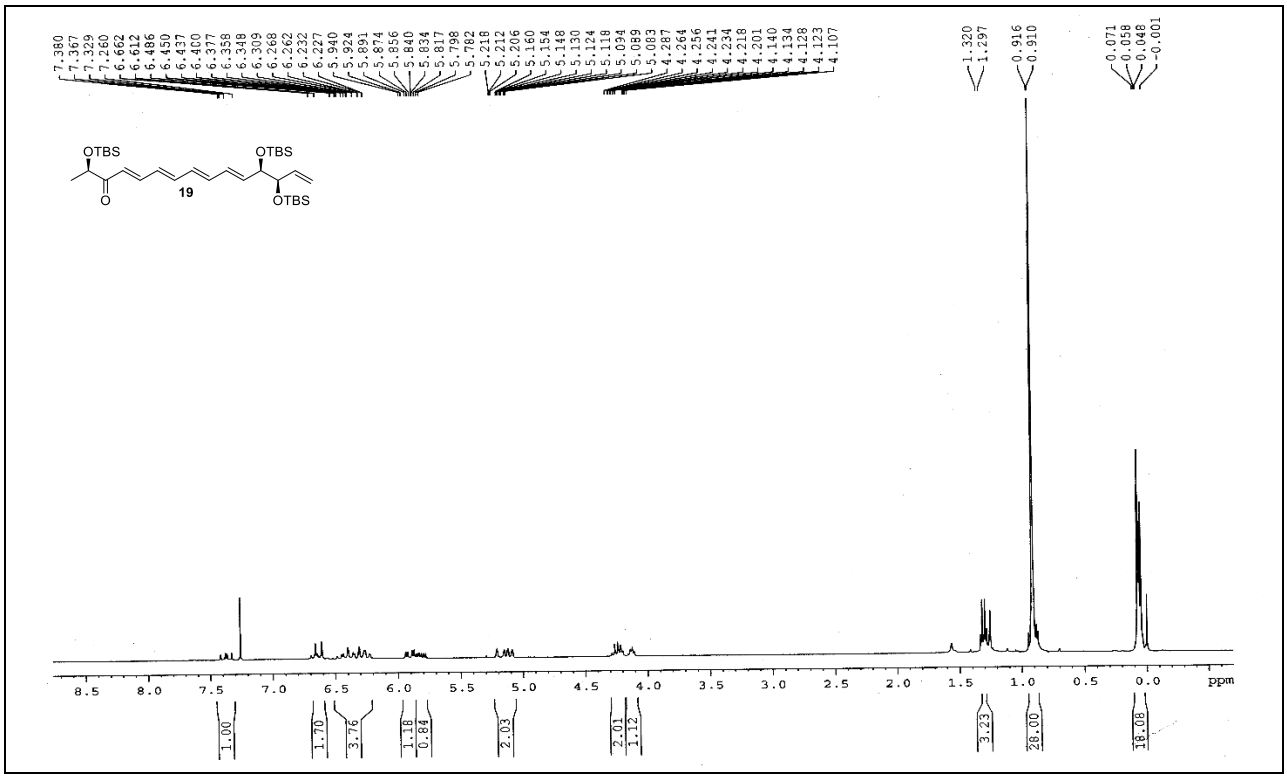
^{1}H NMR spectrum of 18 (300 MHz, $CDCl_3$):



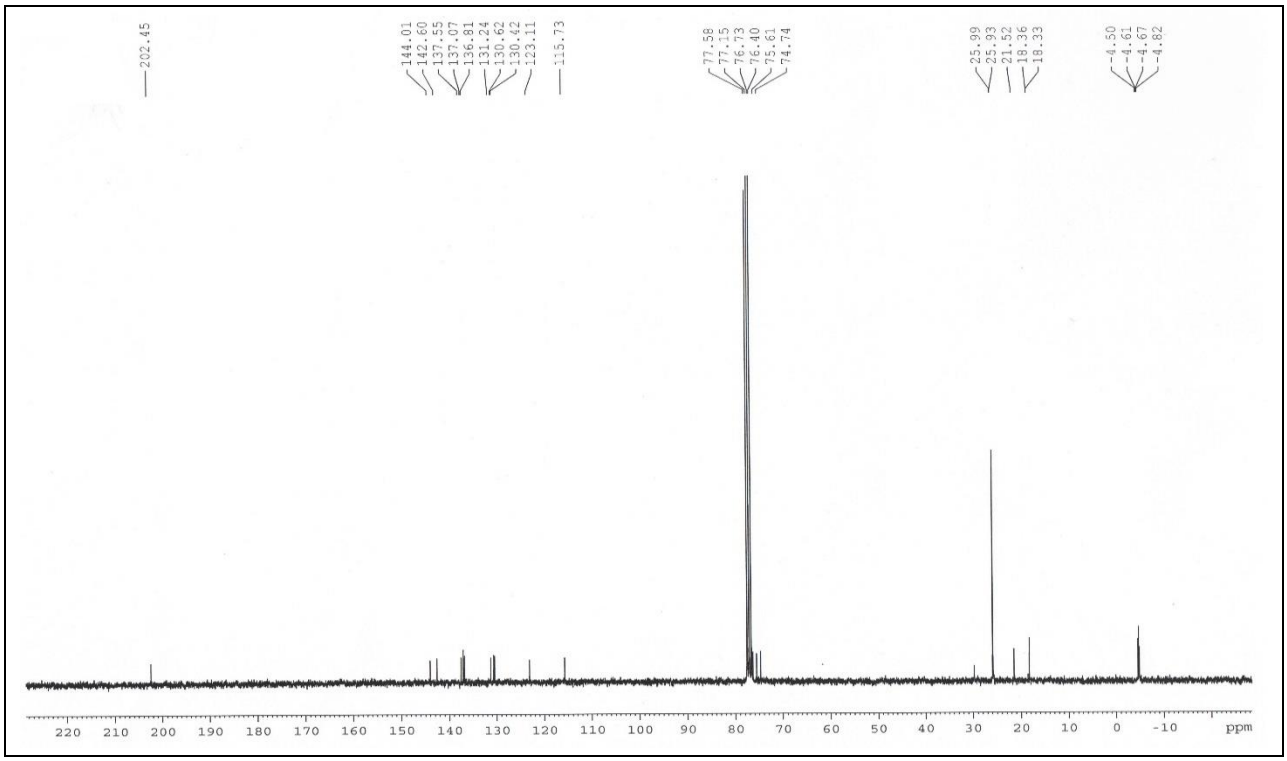
^{13}C NMR spectrum of 18 (75 MHz, $CDCl_3$):



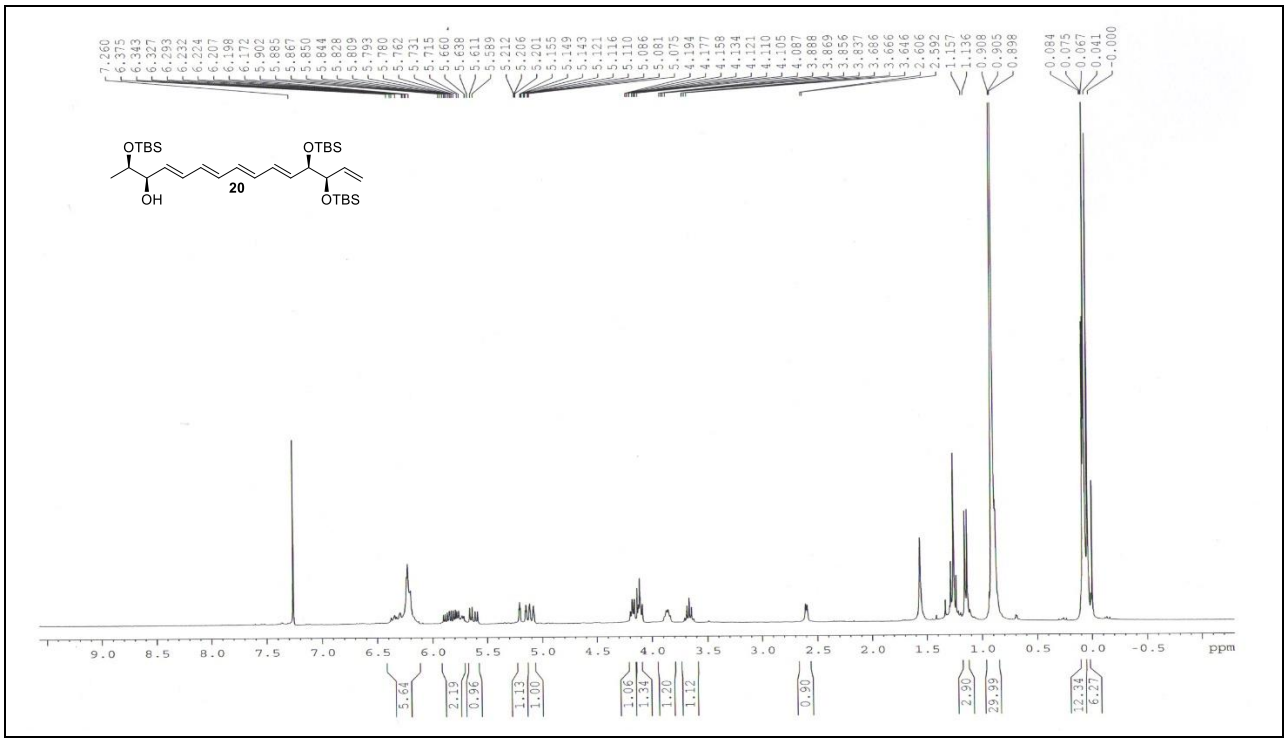
^{1}H NMR spectrum of 19 (300 MHz, $CDCl_3$):



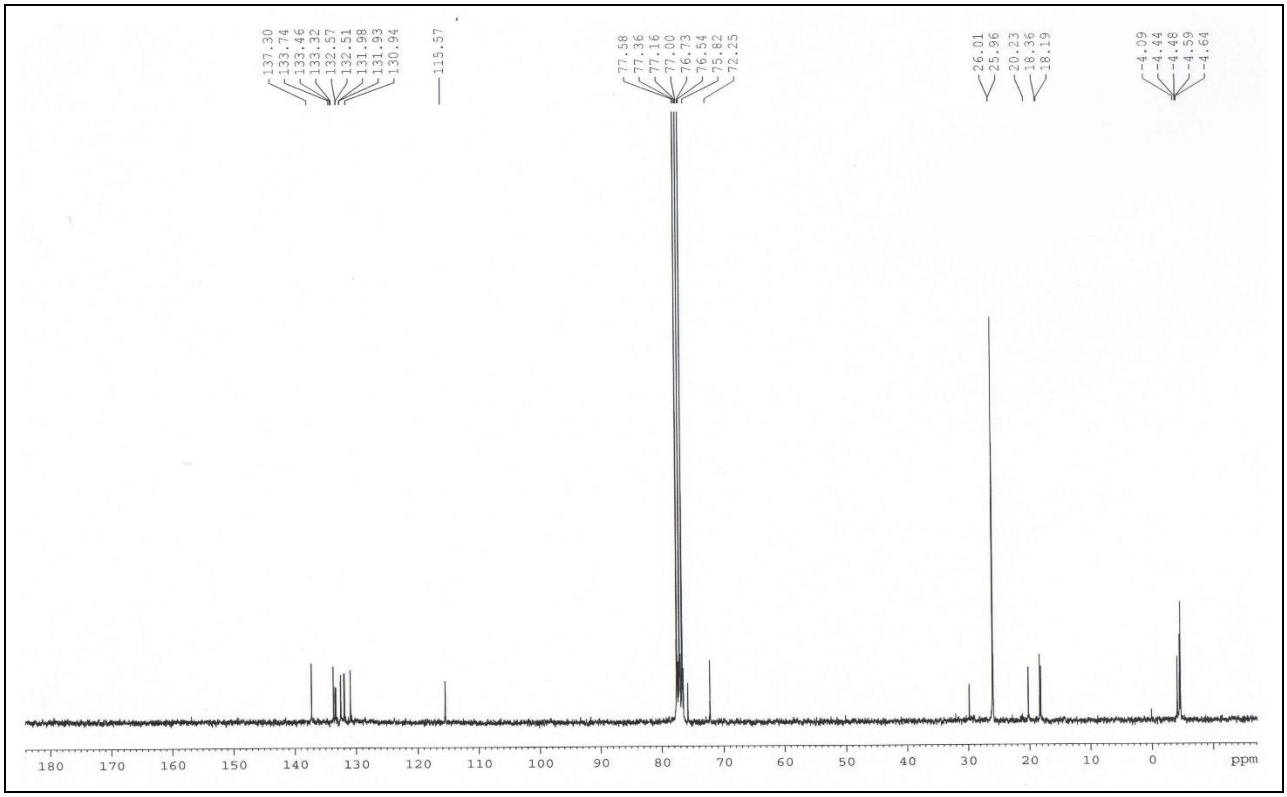
^{13}C NMR spectrum of 19 (75 MHz, $CDCl_3$):



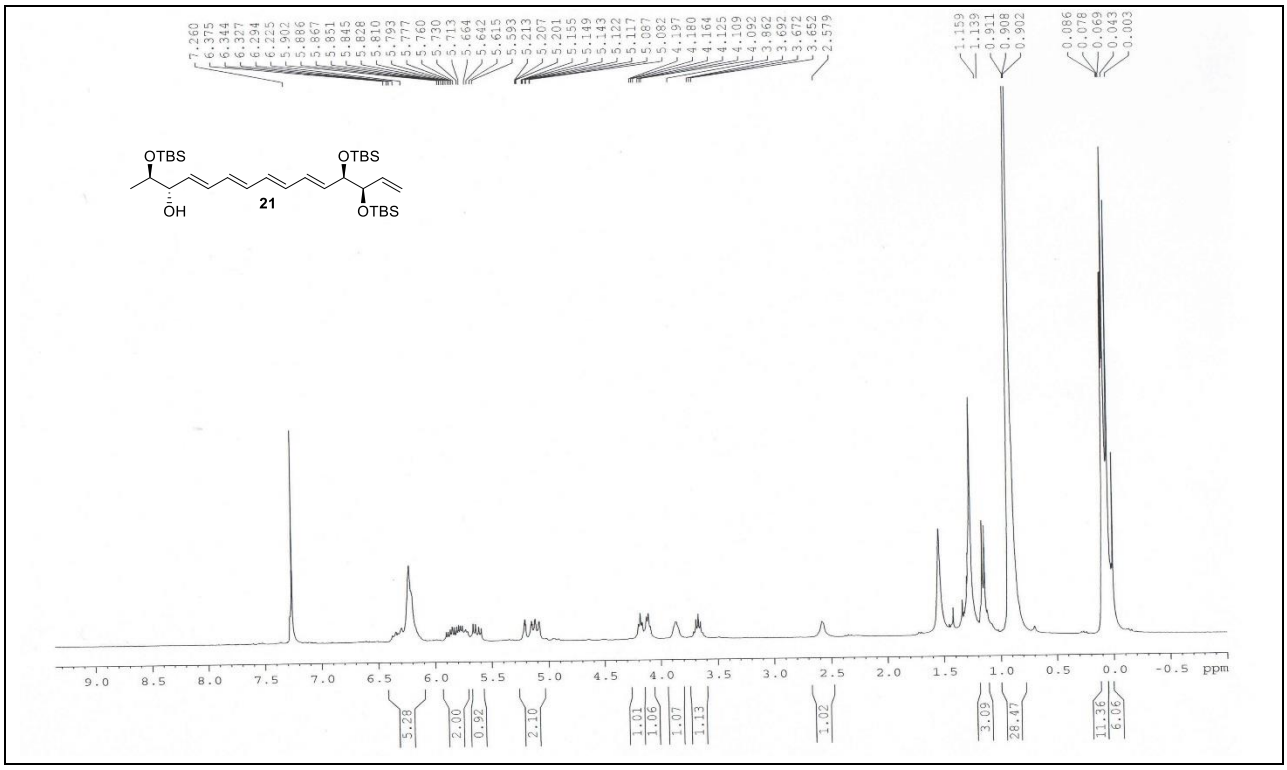
^{1}H NMR spectrum of 20 (300 MHz, $CDCl_3$):



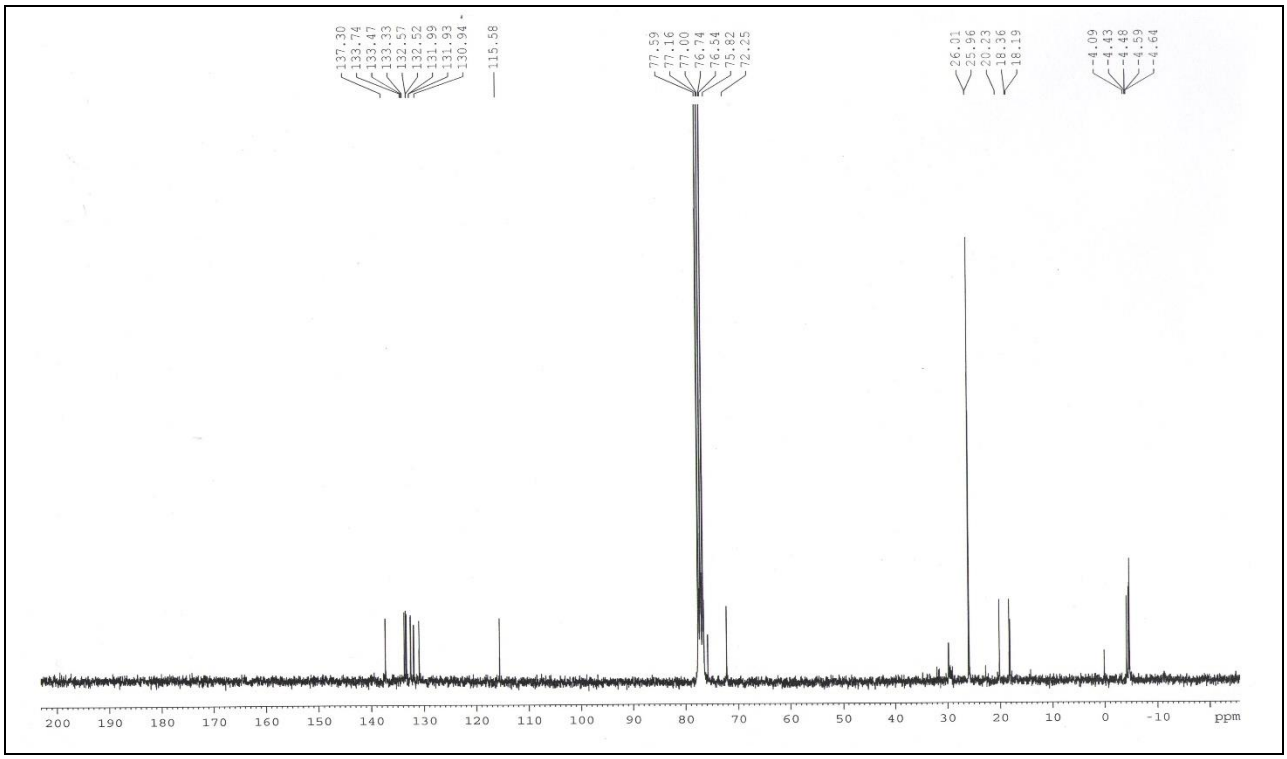
^{13}C NMR spectrum of 20 (75 MHz, $CDCl_3$):



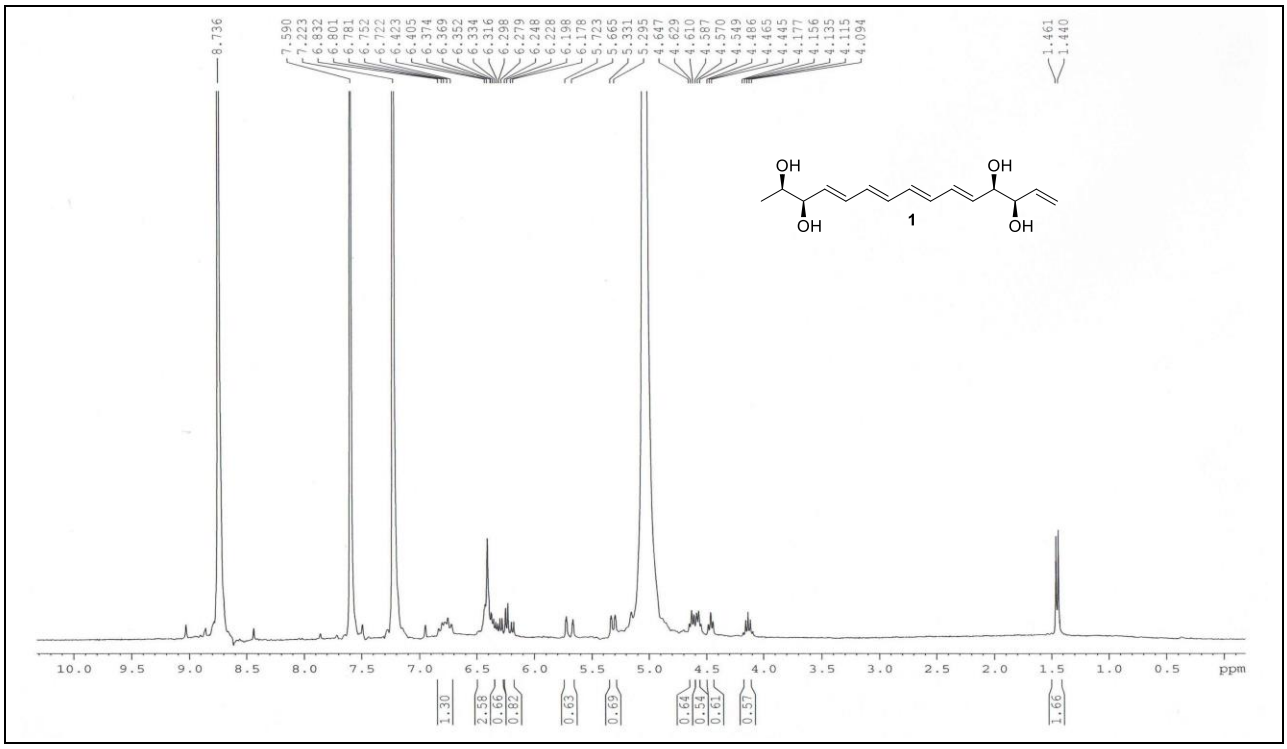
^{1}H NMR spectrum of 21 (300 MHz, $CDCl_3$):



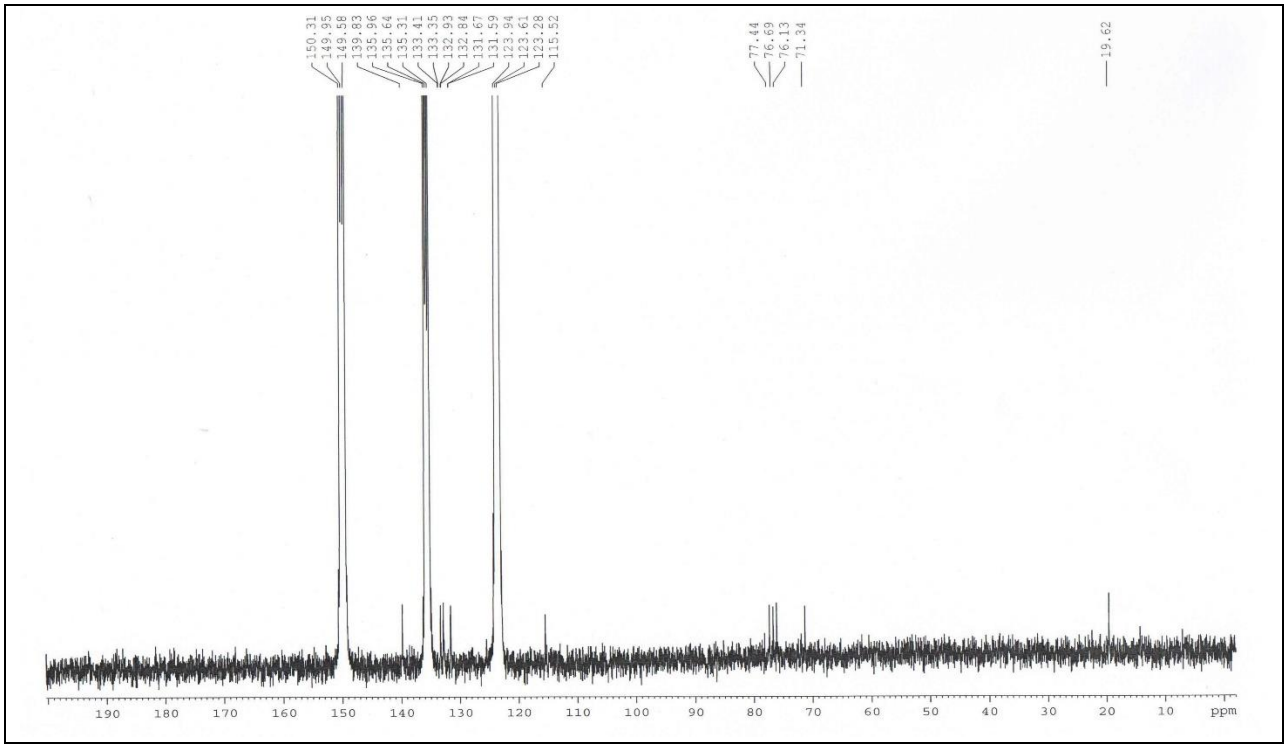
^{13}C NMR spectrum of 21 (75 MHz, $CDCl_3$):



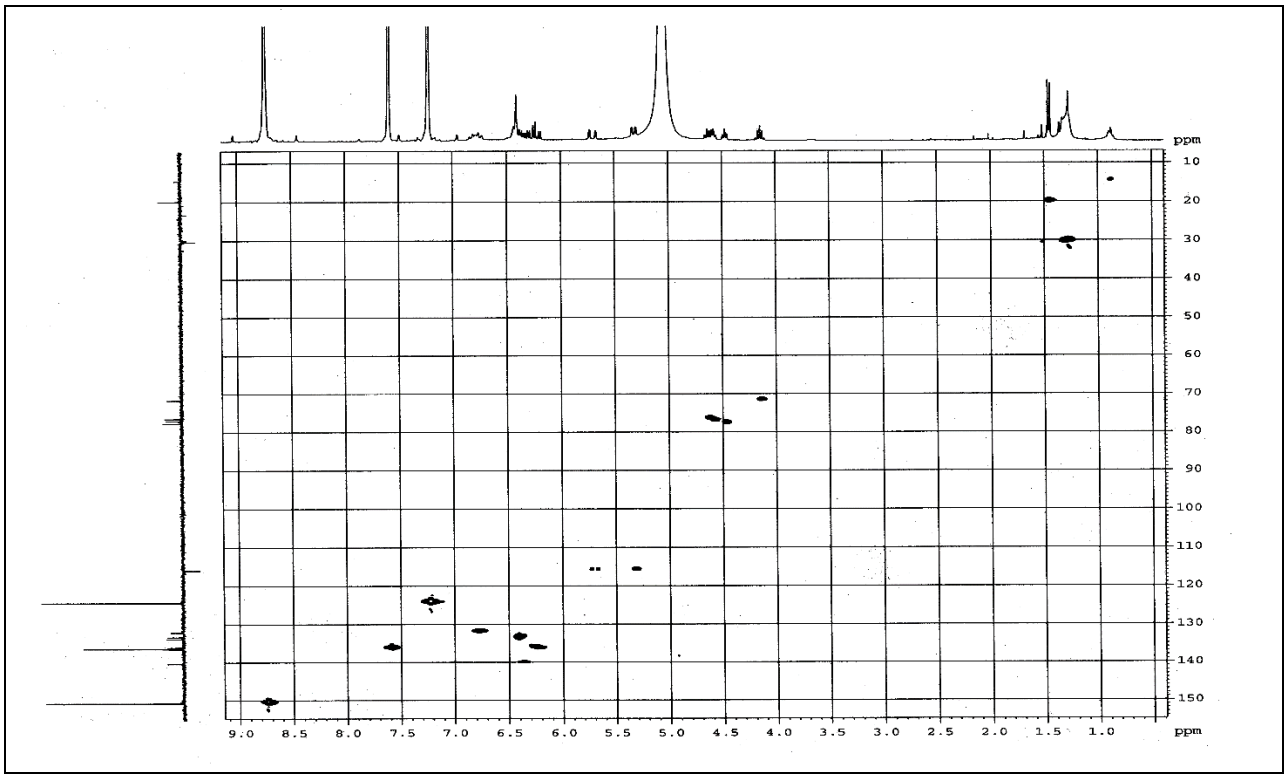
^{1}H NMR spectrum of 1 (300 MHz, C_5D_5N,):



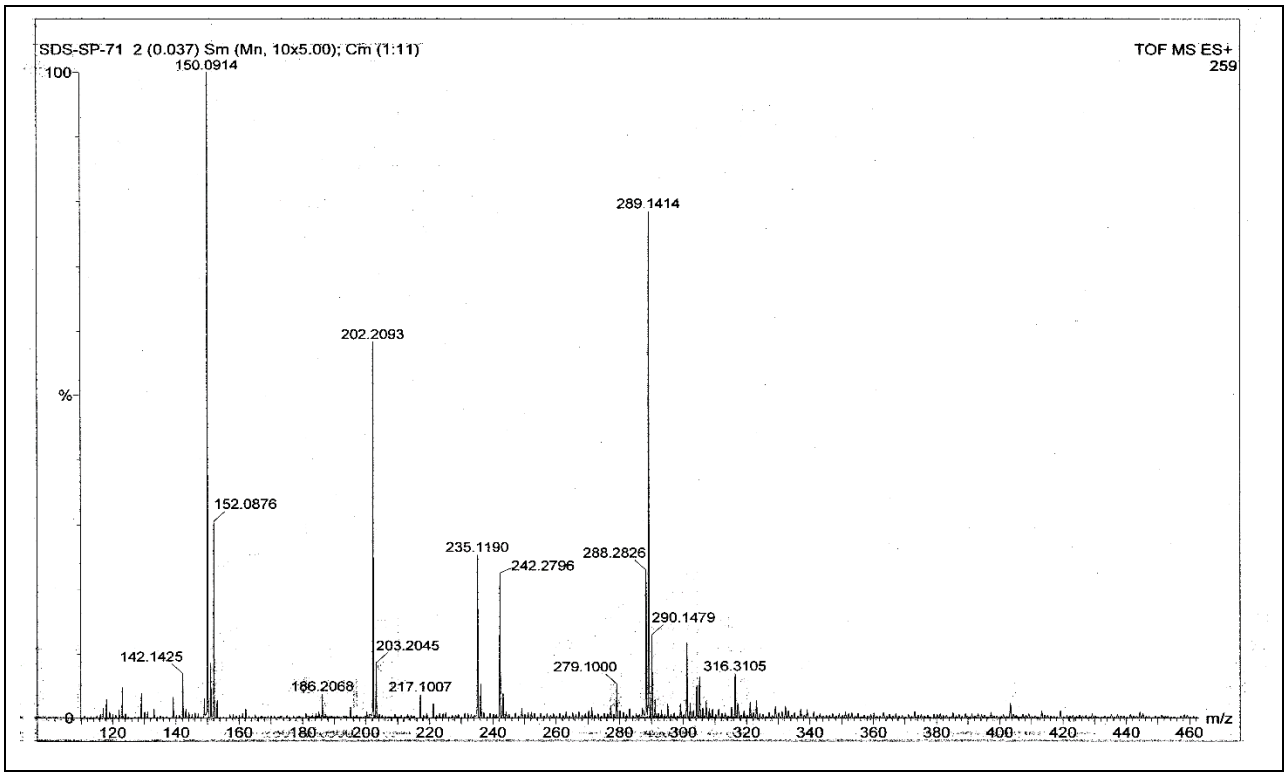
^{13}C NMR spectrum of 1 (75 MHz, C_5D_5N,):



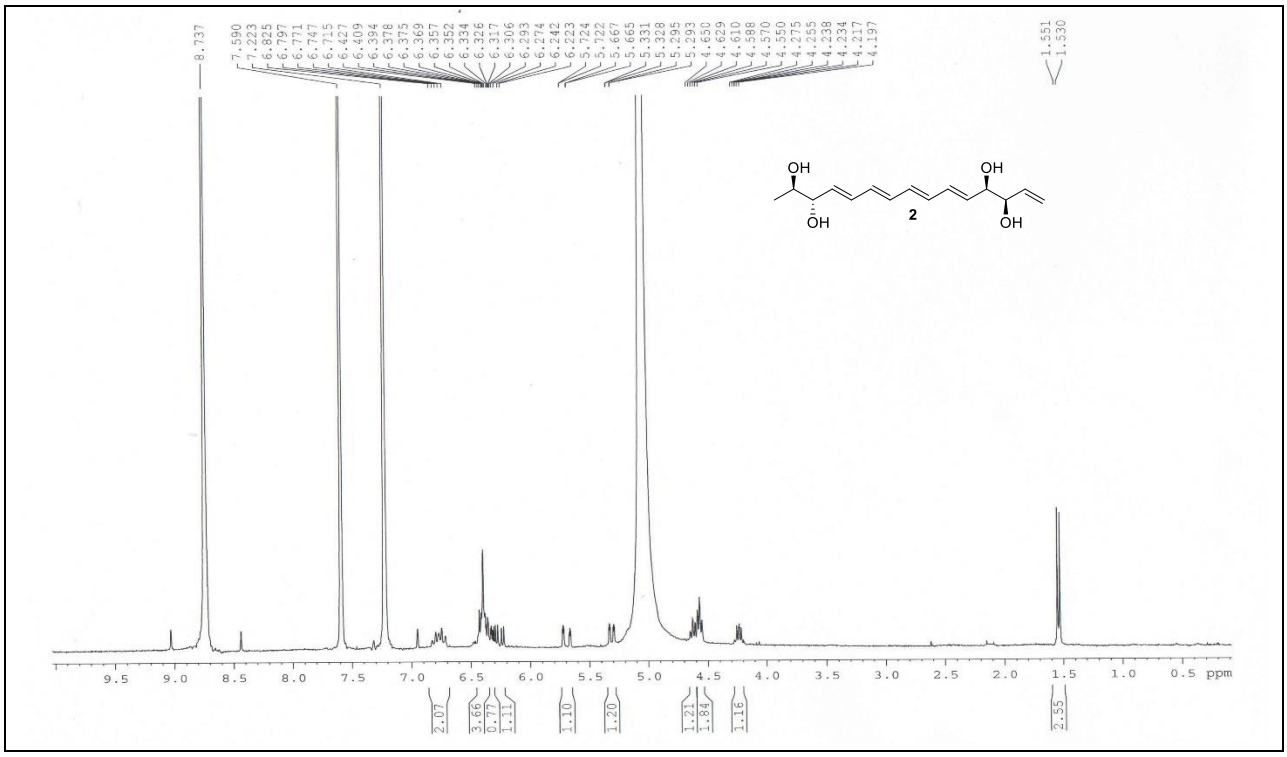
HSQC spectrum of 1:



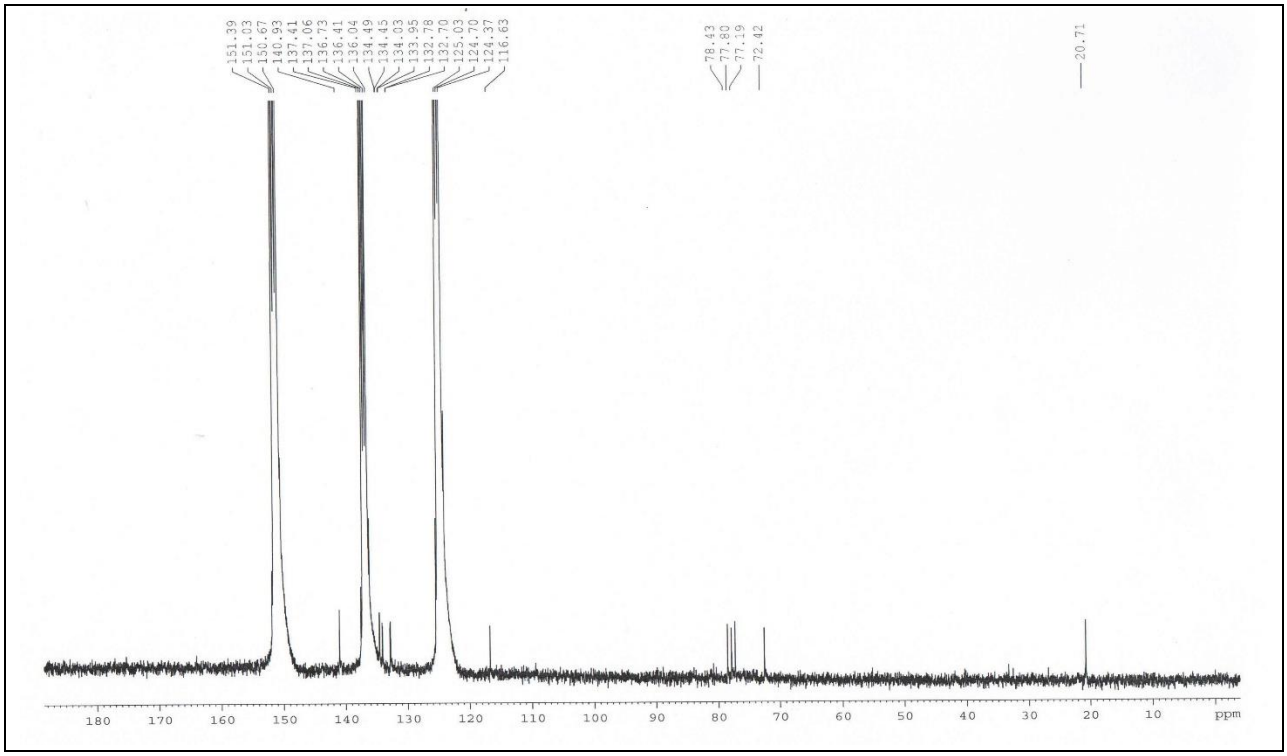
HRMS spectrum of 1:



^{1}H NMR spectrum of 2 (300 MHz, C_5D_5N,):



^{13}C NMR spectrum of 21 (75 MHz, C_5D_5N,):



HSQC spectrum of 2: