

TRACHYLOBANE AND KAURANE DITERPENES FROM *Croton floribundus* SPRENG.

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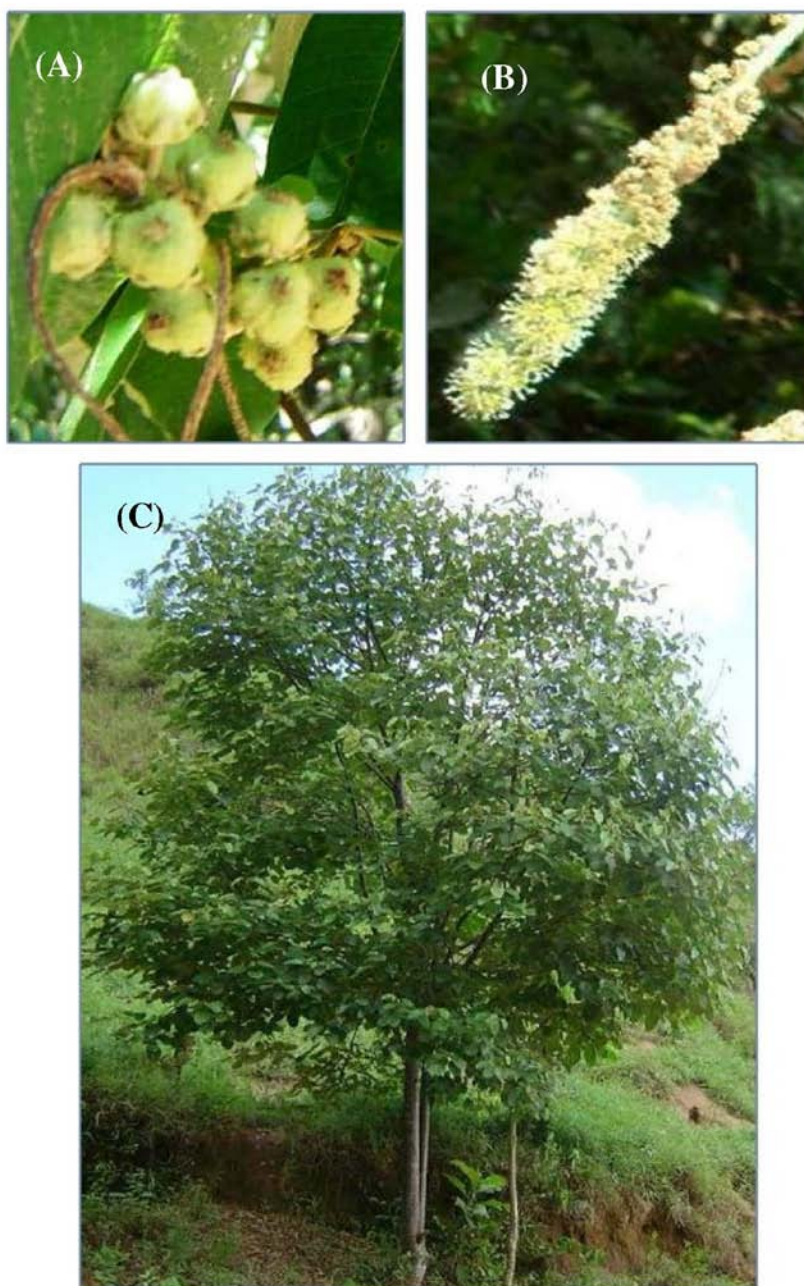


Figure 1S. *Croton floribundus*: fruits (A), flours (B) and entire plant (C)

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#Pesquisador Visitante Emérito – FAPERJ/UENF

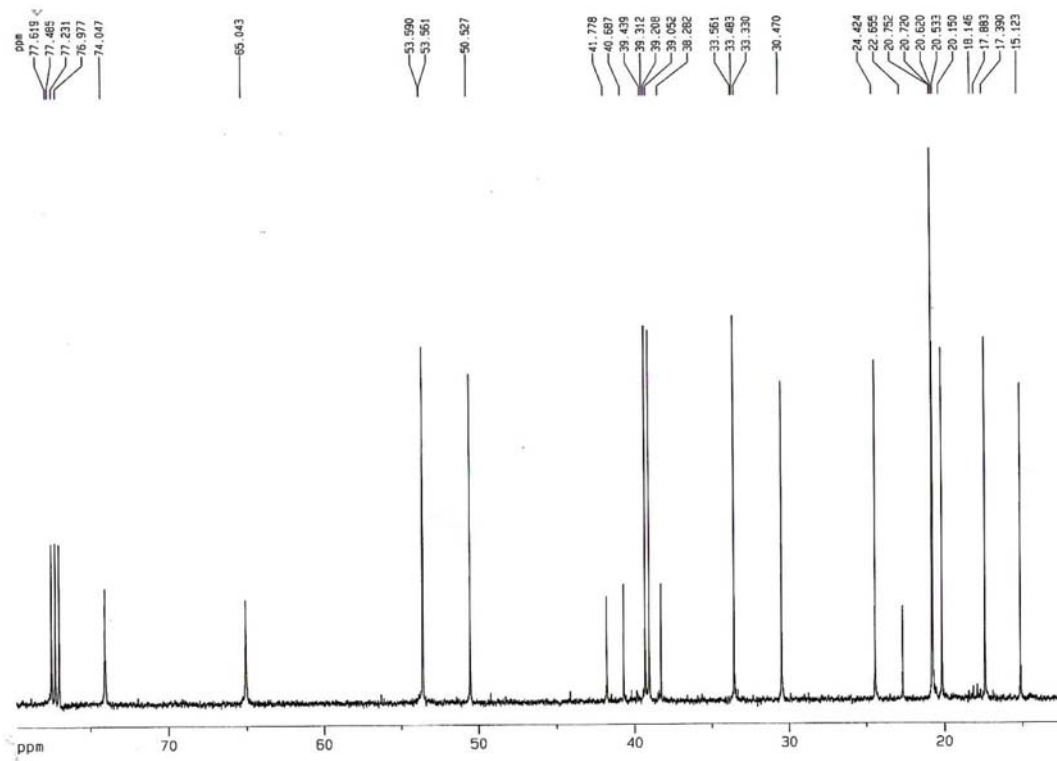


Figure 4S. NMR ^{13}C - CPD spectrum of **1** (125 MHz, CDCl_3)

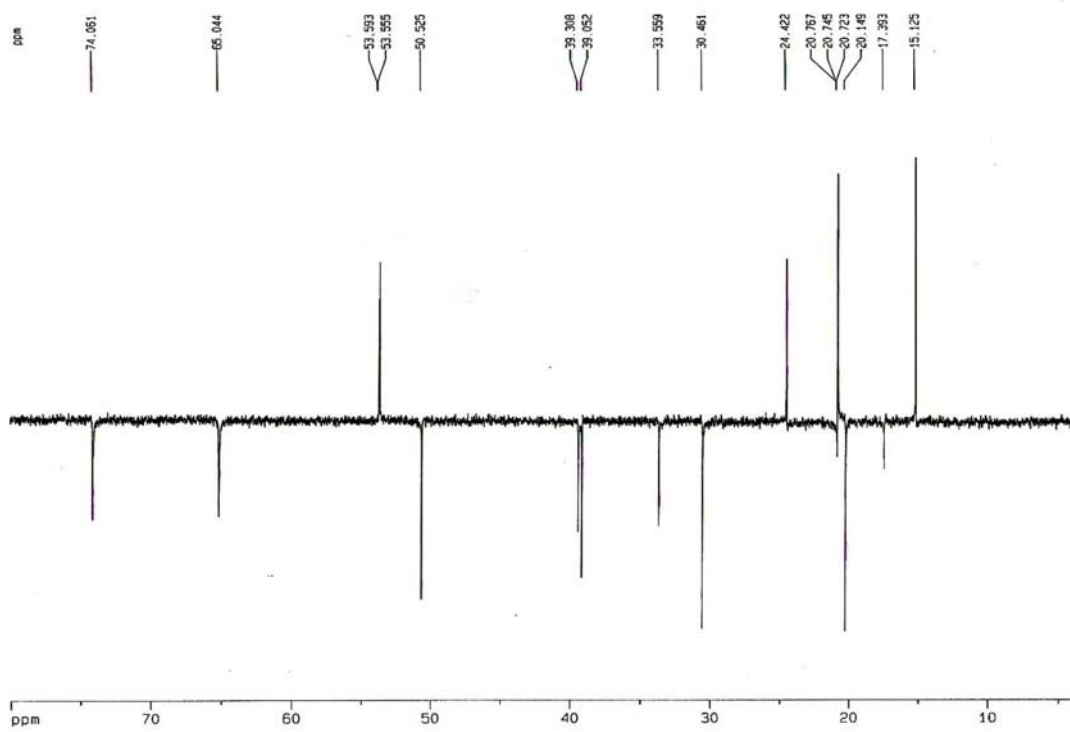


Figure 5S. NMR ^{13}C - DEPT 135° spectrum of **1** (125 MHz, CDCl_3)

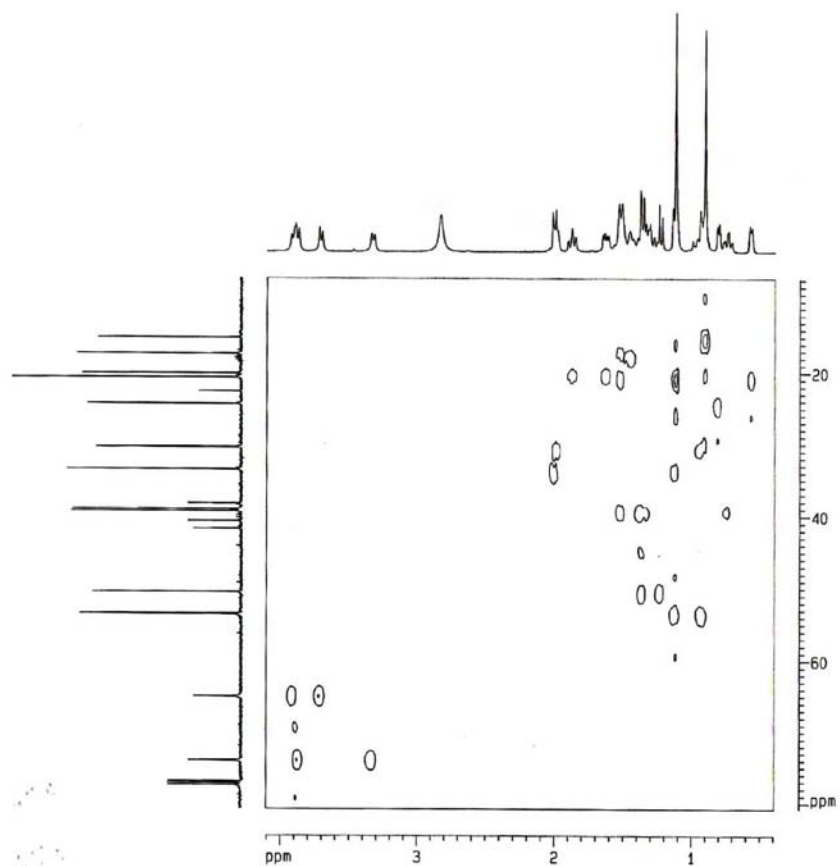


Figure 6S. NMR ^1H , ^{13}C - HSQC (500 x 125 MHz, CDCl_3) spectrum of **1**

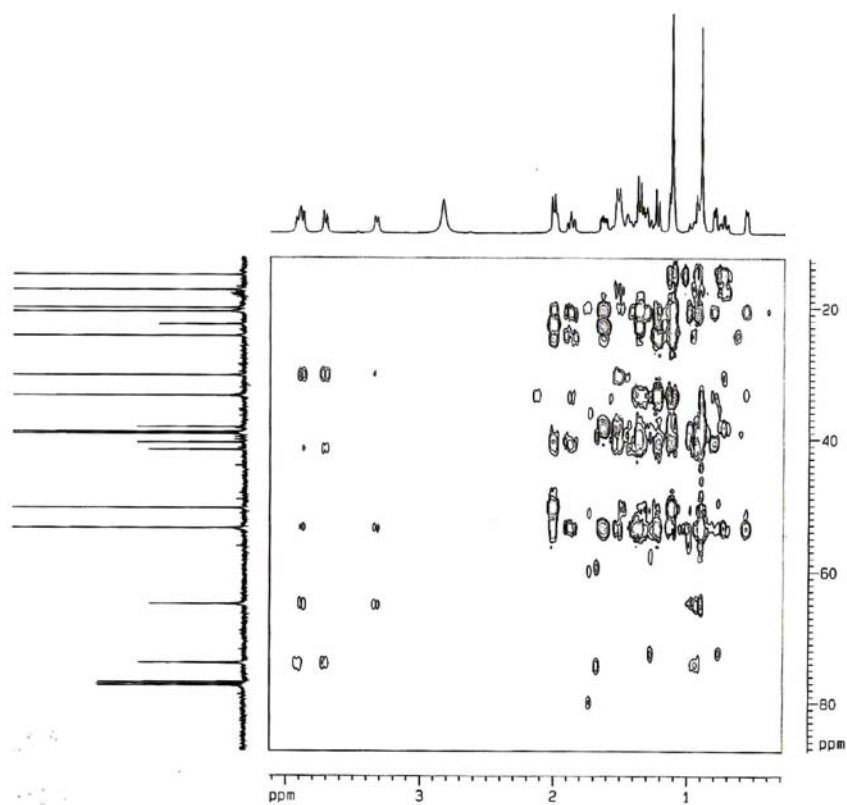
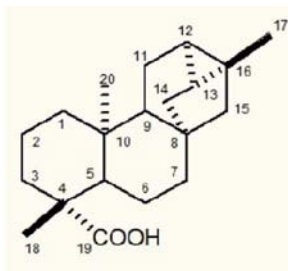


Figure 7S. NMR ^1H , ^{13}C - HMBC (500 x 125 MHz, CDCl_3) spectrum of **1**



ent-trachyloban-19-oic acid (2)

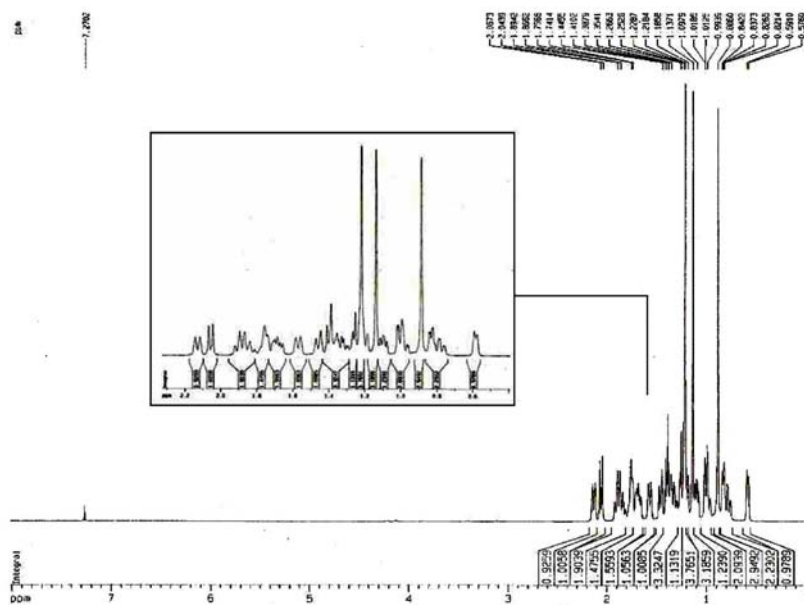


Figure 8S. NMR ¹H (500 MHz, CDCl₃) spectrum of 2

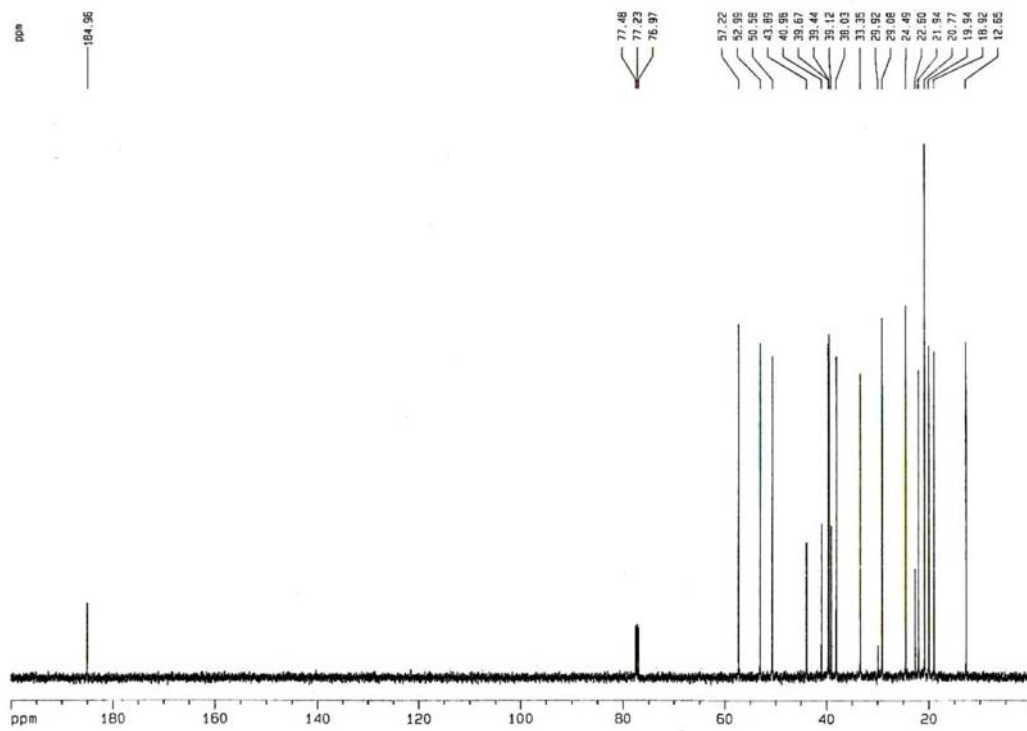
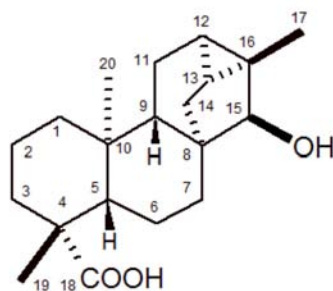


Figure 9S. NMR ¹³C - CPD (125 MHz, CDCl₃) of 2

15 β -hydroxy-*ent*-trachyloban-19-oic acid (**3**)**Table 1S.** NMR data of compound **3**

C	HSQC		HMBC	
	δ_c^a	δ_H^b	$^2J_{CH}$	$^3J_{CH}$
1	41,2	a - 1,57 (m, 1H); b - 0,87 (m, 1H)	–	3H-20
2	20,3	1,28 (m, 2H)	–	–
3	39,9	a - 2,90 (d, $J = 13,1$ Hz); b - 0,92 (m, 1H)	–	3H-18
4	45,2	–	3H-18	H-5
5	58,1	0,90 (m, 1H)	–	3H-18; 3H-20
6	23,0	1,79 (m, 2H)	–	–
7	37,9	a - 1,45 (m, 1H); b - 1,20 (m, 1H)	–	H-15
8	42,3	–	H-9; H-14a; H-15;	H-13
9	43,7	1,43 (m, 1H)	H-11b	H-14a; 3H-20
10	39,6	–	H-1a; 3H-20	–
11	19,5	a - 1,85 (t, $J = 2,9$ Hz, 1H); b - 1,63 (dd, $J = 7,5$ e $2,9$ Hz 1H)	–	–
12	21,3	0,64 (d, $J = 7,5$ Hz, 1H)	H-11b	H-15; 3H-17
13	22,3	0,81 (dd, $J = 7,5$ e $2,9$ Hz, 1H)	H-14a	H-11a; 3H-17
14	31,6	a - 2,17 (d, $J = 12,0$ Hz, 1H); b - 1,18 (m, 1H)	–	H-7a; H-9; H-15
15	84,6	3,28 (s, 1H)	–	H-14a; 3H-17
16	25,9	–	H-15; 3H-17	H-11a; H-14 ^a
17	18,6	1,13 (s, 3H)	–	H-15
18	29,9	1,15 (s, 3H)	–	–
19	183,4	–	–	H-5; 3H-18
20	13,8	0,95 (s, 3H)	–	H-5; H-9

^a 125 MHz; ^b 500 MHz.

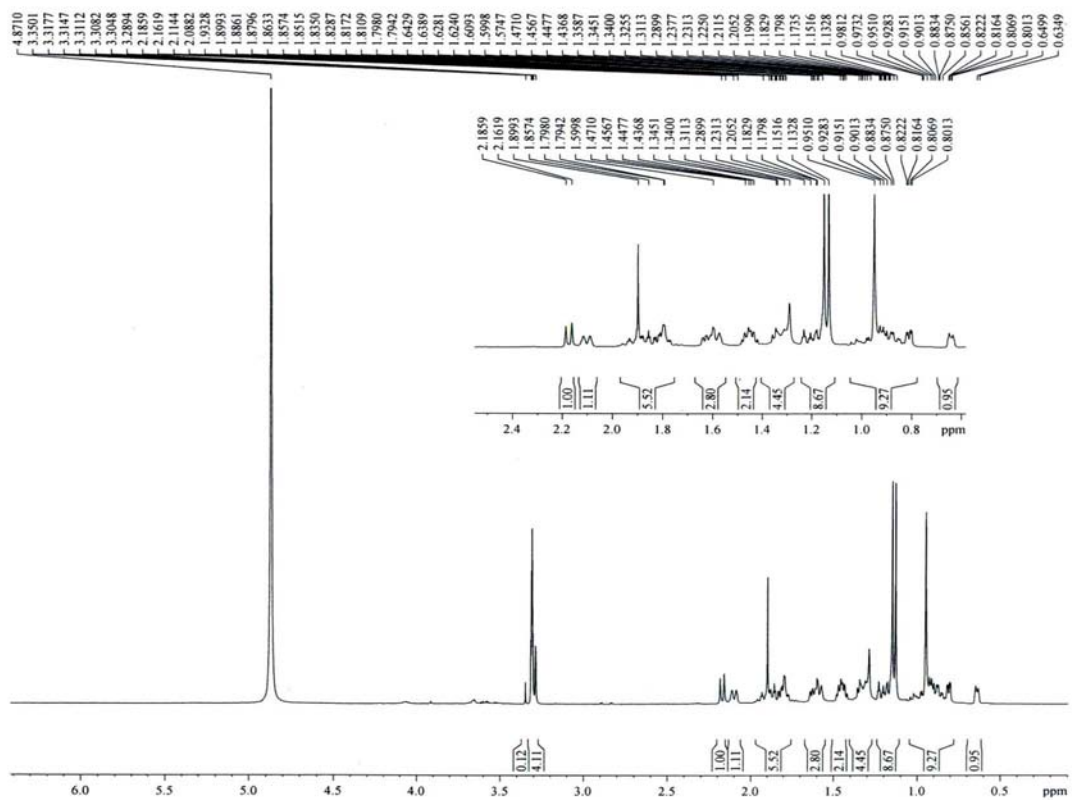


Figure 10S. NMR ^1H spectrum of **3** (500 MHz, CD_3OD)

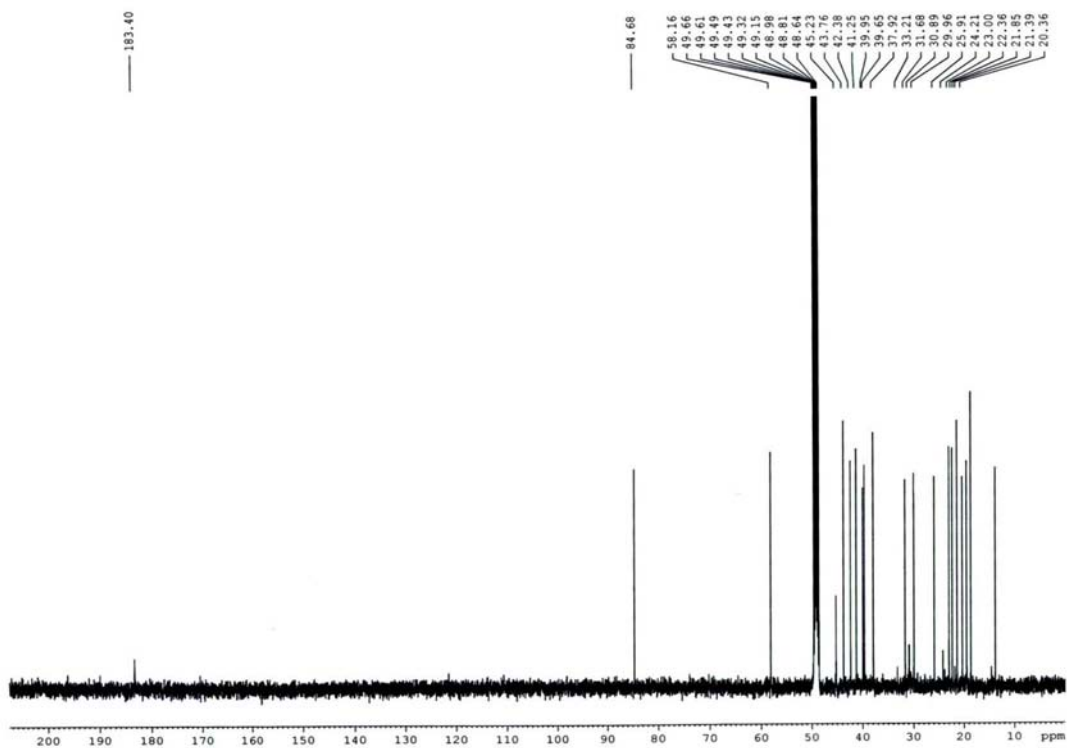
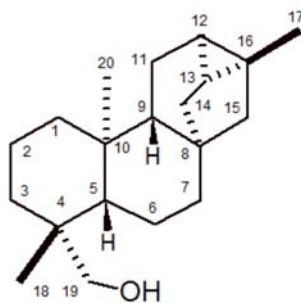


Figure 11S. NMR ^{13}C - CPD spectrum of **3** (125 MHz, CD_3OD)

*ent*-trachyloban-19-ol (**4**)**Table 2S.** NMR data of compound **4**

C	HSQC		HMBC	
	δ_c^a	δ_H^b	$^2J_{CH}$	$^3J_{CH}$
1	39,3	a - 1,34 (d, $J = 3,5$ Hz, 1H); b - 0,75 (td, $J = 13,5$ e $3,5$ Hz, 1H)	–	H-2b
2	17,8	a - 1,50 (m, 1H); b - 1,36 (m, 1H)	H-3b	–
3	35,7	a - 1,75 (d, $J = 13,7$ Hz); b - 0,88 (m, 1H)	–	3H-18; H-19b
4	38,3	–	3H-18	–
5	56,8	0,89 (sl, 1H)	–	3H-20; 3H-18
6	20,4	a - 1,58 (m, 1H); b - 1,55 (m, 1H)	–	–
7	39,5	a - 1,52 (m, 1H); b - 1,40 (m, 1H)	–	H-6b
8	40,7	–	H-9; H-15b	–
9	53,4	1,10 (m, 1H)	H-11b	–
10	38,3	–	3H-20	–
11	19,9	a - 1,88 (td, $J = 14,1$ e $2,5$ Hz, 1H); b - 1,64 (m, 1H)	–	–
12	20,6	0,57 (d, $J = 7,5$ Hz, 1H)	H-11b	H-15a; 3H-17
13	24,3	0,80 (dd, $J = 7,5$ e $2,5$ Hz, 1H)	–	H-15b
14	33,4	a - 2,02 (d, $J = 11,8$ Hz, 1H); b - 1,15 (m, 1H)	–	H-9; H-15b
15	50,4	a - 1,38 (m, 1H); b - 1,25 (m, 1H)	–	H-14a; 3H-17
16	22,4	–	3H-17	H-14a
17	20,5	1,10 (s, 3H)	–	H-15a
18	26,8	0,93 (s, 3H)	–	2H-19
19	65,6	a - 3,72 (d, $J = 10,9$ Hz, 1H); b - 3,42 (d, $J = 10,9$ Hz, 1H)	–	H-5; 3H-18
20	15,1	0,92 (s, 3H)	–	H-5; H-9

^a 125 MHz; ^b 500 MHz.

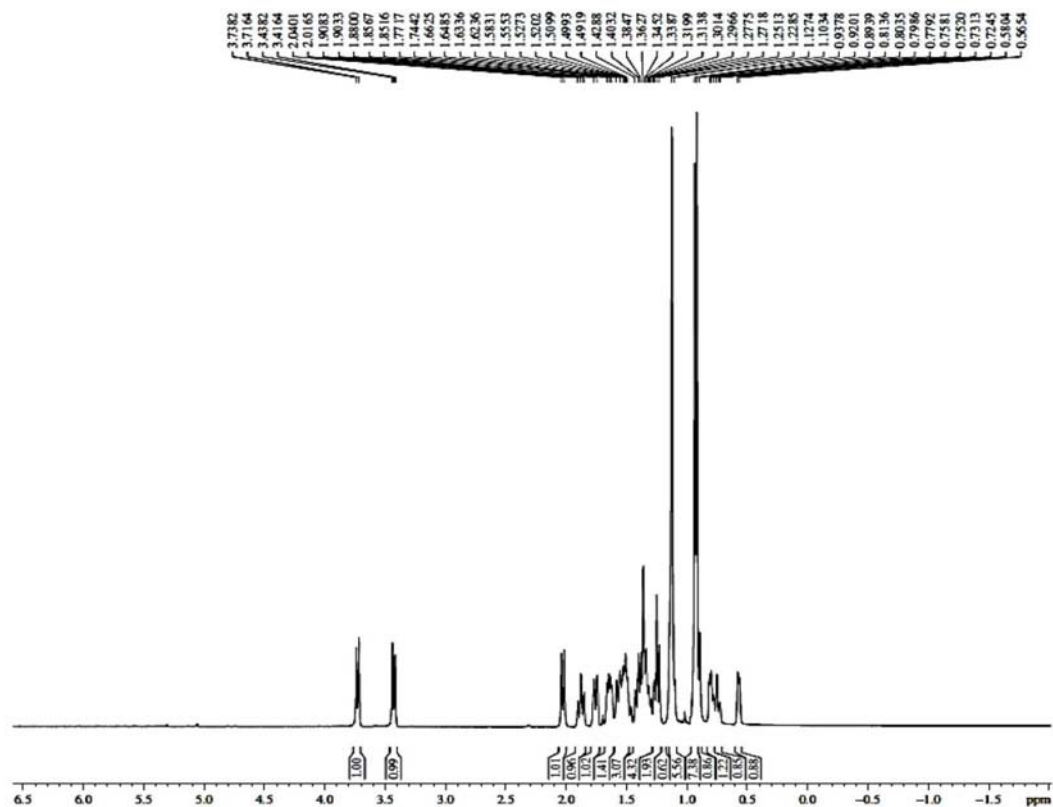


Figure 12S. NMR ^1H spectrum of **4** (500 MHz, CDCl_3)

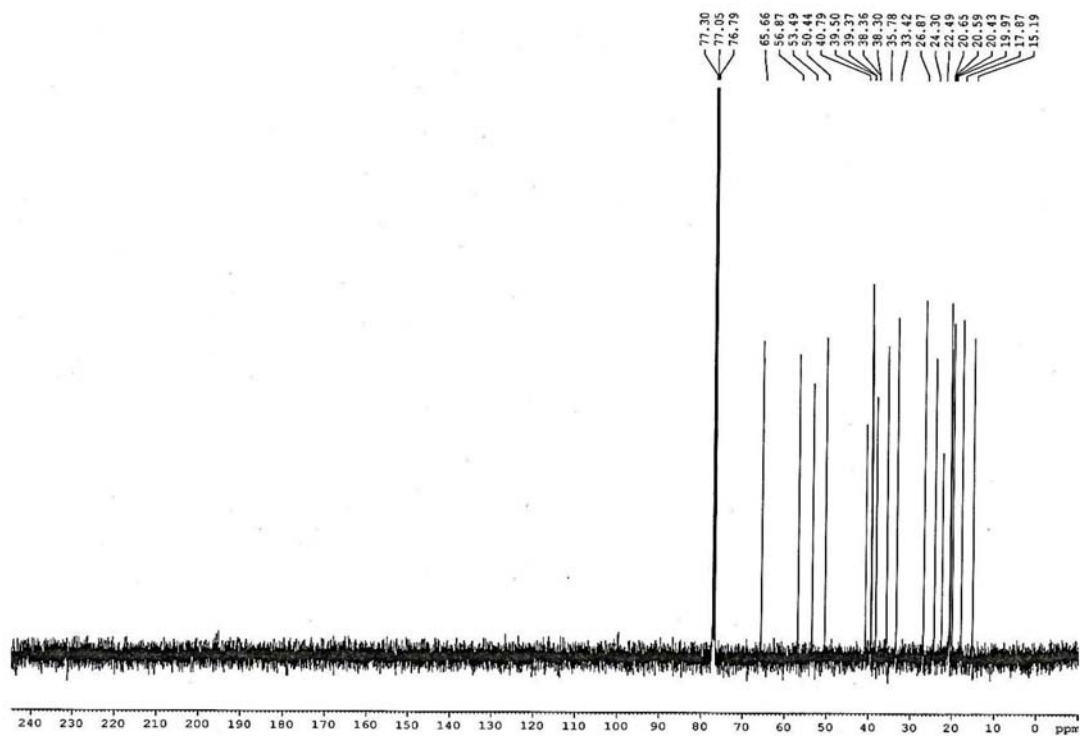


Figure 13S. NMR ^{13}C - CPD spectrum of **4** (125 MHz, CDCl_3)

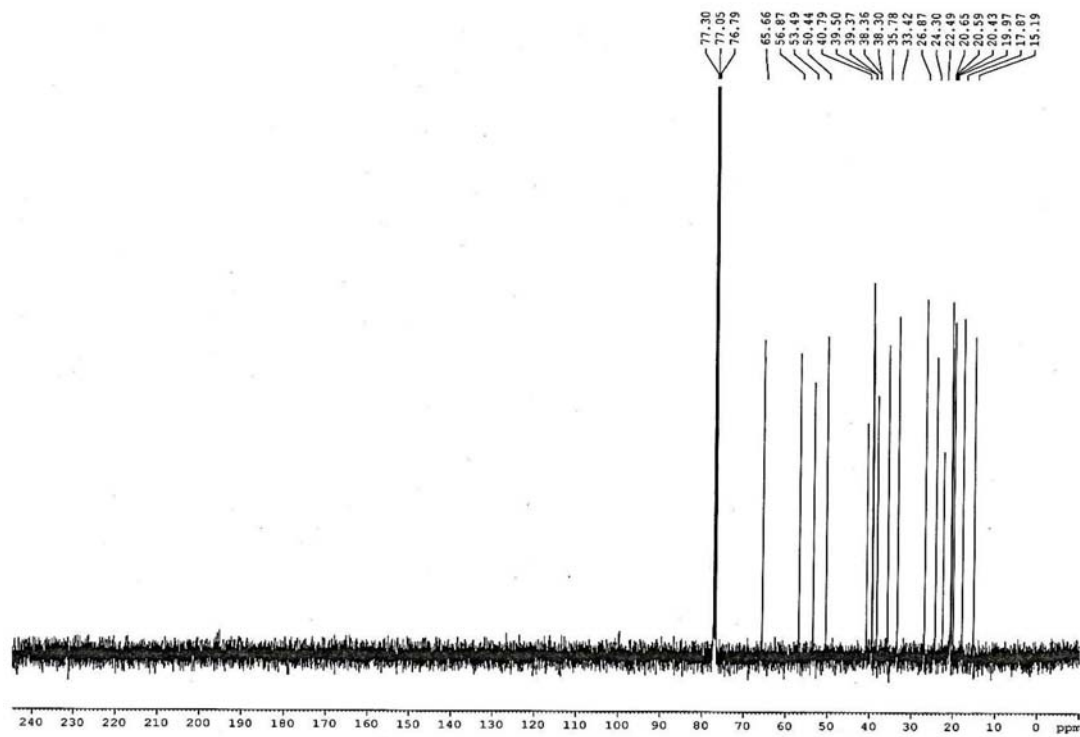
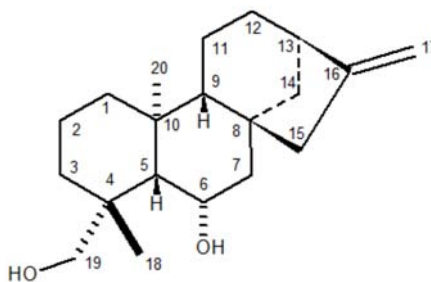


Figure 14S. NMR ^1H spectrum of **5** (500 MHz, CDCl_3)

*ent*-kaur-16-ene-6 α ,19-diol (**6**)**Table 3S.** NMR data of compound **6**

C	HSQC		HMBC	
	δ_c^a	δ_H^b	$^2J_{CH}$	$^3J_{CH}$
1	43,0	a - 1,85 (d, $J = 13,0$ Hz, 1H); b - 0,85 (m, 1H)	–	H-3b; 3H-20
2	18,3	a - 1,78 (m, 1H); b - 1,58 (m, 1H)	2H-1; H-3b	–
3	40,7	a - 1,52 (m, 1H); b - 1,21 (m, 1H)	–	3H-18; 2H-19
4	39,2	–	3H-18; 2H-19	H-6
5	57,1	1,04 (m, 1H)	H-6	3H-18; 2H-19; 3H-20
6	66,5	4,37 (sl, 1H)	H-5	–
7	47,1	1,78 - 1,69 (m, 2H)	H-6	–
8	42,1	–	H-14a; 2H-15	H-6; H-13
9	56,4	1,19 (m, 1H)	–	H-1b; H-5; H-14a; 2H-15; 3H-20
10	39,1	–	2H-1; H-9; 3H-20	H-6
11	18,6	a - 1,78 (m, 1H); b - 1,58 (m, 1H)	–	–
12	33,1	a - 1,75 (m, 1H); b - 1,55 (m, 1H)	–	H-14a
13	44,5	2,68 (s, 1H)	H-14a	H-17a
14	41,0	a - 2,39 (d, $J = 11,5$ Hz, 1H); b - 1,28 (m, 1H)	–	–
15	49,7	2,12 (m, 2H)	–	H-13; H-14a; H-17b
16	155,2	–	H-13; 2H-15; H-17a	H-14a
17	102,2	a - 4,84 (sl, 1H); b - 4,78 (sl, 1H)	–	H-13; 2H-15
18	27,9	1,05 (s, 3H)	–	H-3b; H-5; 2H-19
19	68,4	a - 4,28 (d, $J = 11,5$ Hz, 1H); b - 3,16 (d, $J = 11,5$ Hz, 1H)	–	3H-18
20	18,5	1,43 (s, 3H)	–	H-5; H-9

^a 125 MHz; ^b 500 MHz.

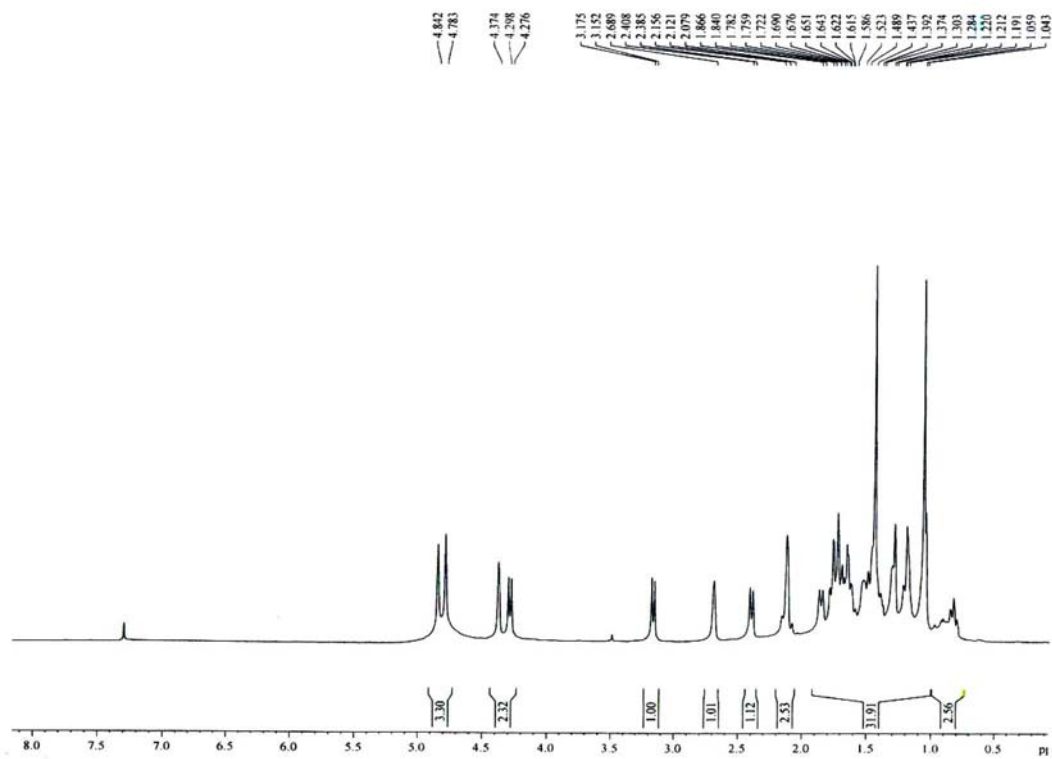


Figure 15S. NMR ^1H spectrum of **6** (500 MHz, CDCl_3)

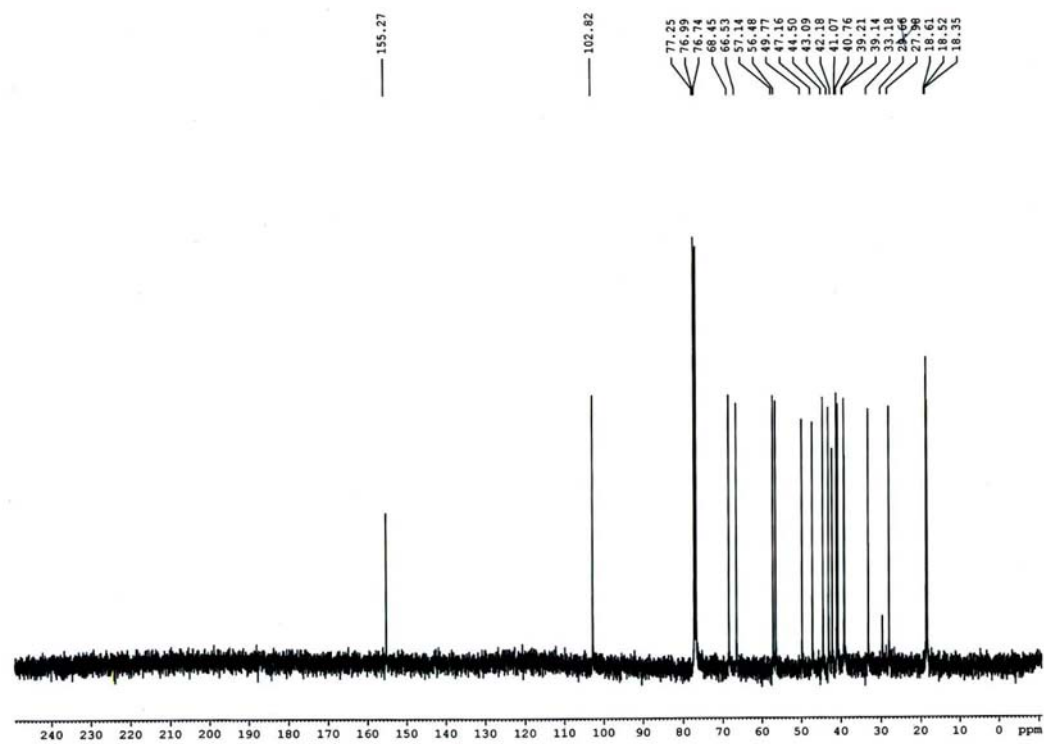
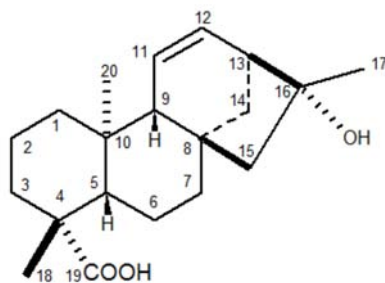


Figure 16S. NMR ^{13}C - CPD spectrum of **6** (125 MHz, CDCl_3)

*ent*-16 α -hydroxykaur-11-en-19-oic acid (7)**Table 4S.** NMR data of compound 7

C	HSQC		HMBC	
	δ_c^a	δ_H^b	$^2J_{CH}$	$^3J_{CH}$
1	41,6	a - 1,81 (m, 1H); b - 1,04 (m, 1H)	–	H-3b
2	20,5	a - 1,90 (m, 1H); b - 1,33 (m, 1H)	–	–
3	39,7	a - 2,13 (m, 1H); b - 1,00 (m, 1H)	–	3H-18
4	45,3	–	H-3b; H-5; 3H-18	–
5	57,4	1,11 (m, 1H)	–	3H-18; H-3b; 3H-20
6	23,4	1,72 (m, 2H)	H-5	–
7	42,7	1,51 (m, 2H)	–	H-15a
8	44,6	–	H-9; 2H-14; H-15b	H-11; H-13
9	63,0	1,40 (m, 1H)	H-11	H-12; 2H-15; 3H-20
10	40,1	–	H-5; H-9; 3H-20	–
11	128,4	5,56 (dd, $J = 9,7$ e $3,0$ Hz, 1H)	H-9	–
12	133,8	5,91 (t, $J = 9,7$ Hz, 1H)	–	H-9; 2H-14a
13	51,1	2,17 (m, 1H)	H-12; H-14b	H-11; 3H-17
14	35,3	a - 1,82 (m, 1H); b - 1,65 (m, 1H);	–	H-9; H-15b
15	59,6	a - 1,48 (m, 1H); b - 1,42 (m, 1H);	–	2H-7; H-14a; 3H-17
16	84,5	–	3H-17	H-14a
17	25,9	1,27 (s, 3H)	–	H-15b
18	29,9	1,17 (s, 3H)	–	–
19	184,7	–	–	H-3b; H-5; 3H-18
20	16,5	0,88 (s, 3H)	–	–

^a 125 MHz; ^b 500 MHz.

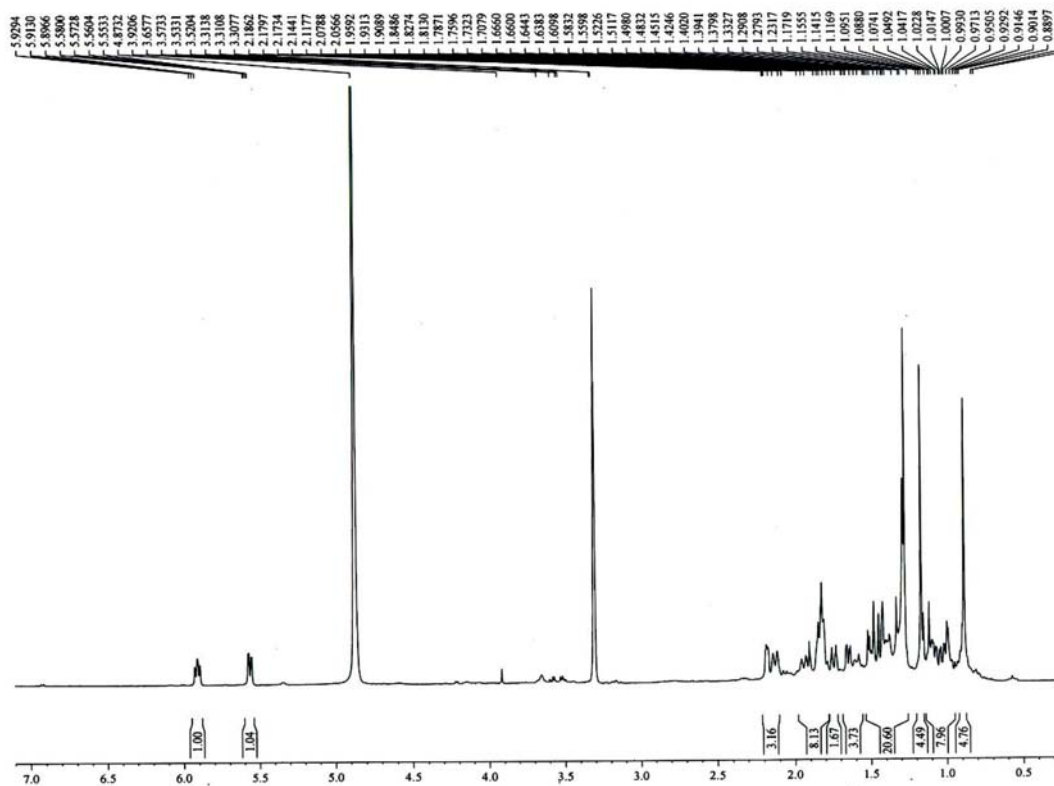


Figure 17S. NMR ^1H spectrum of 7 (500 MHz, CD_3OD)

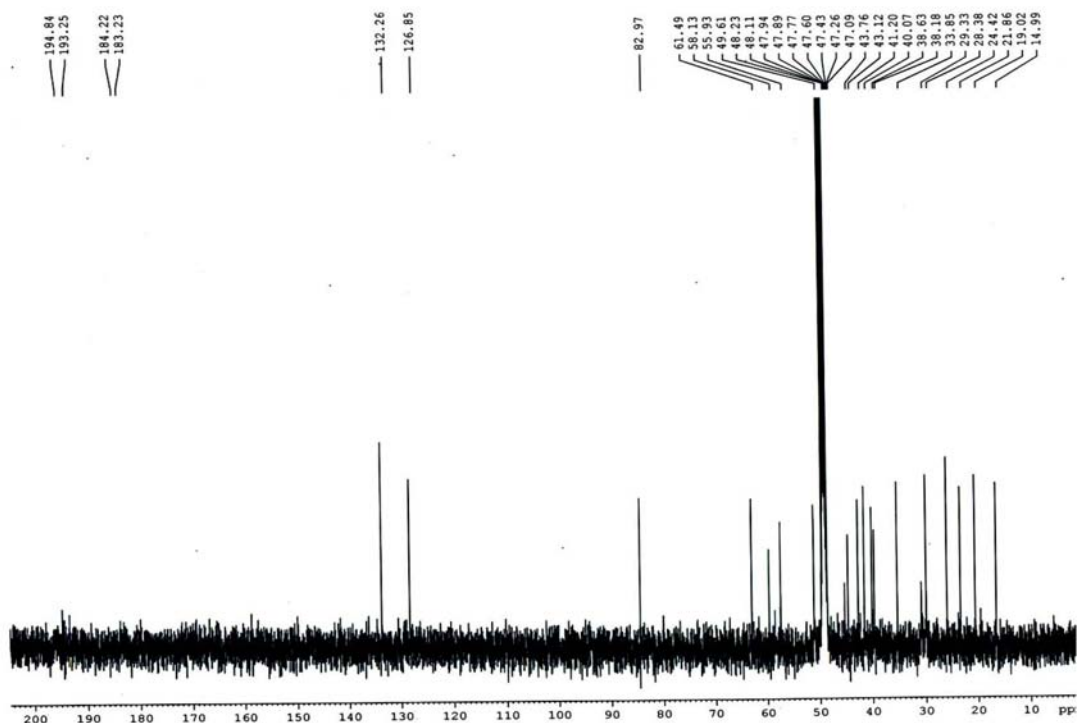
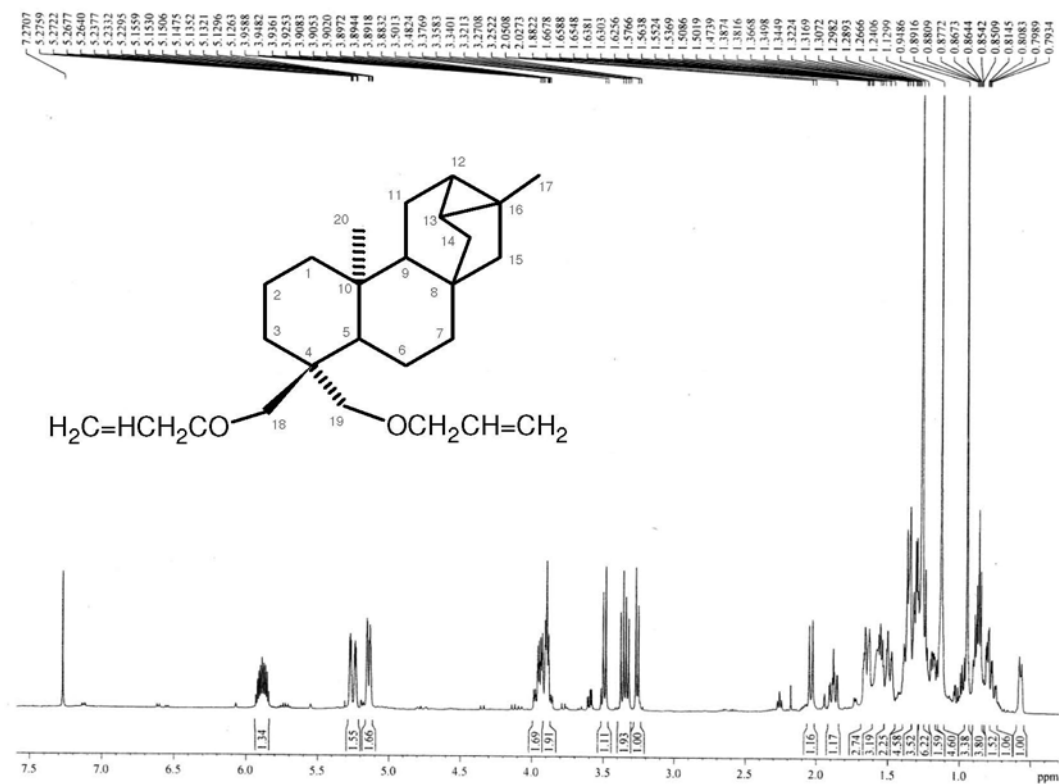
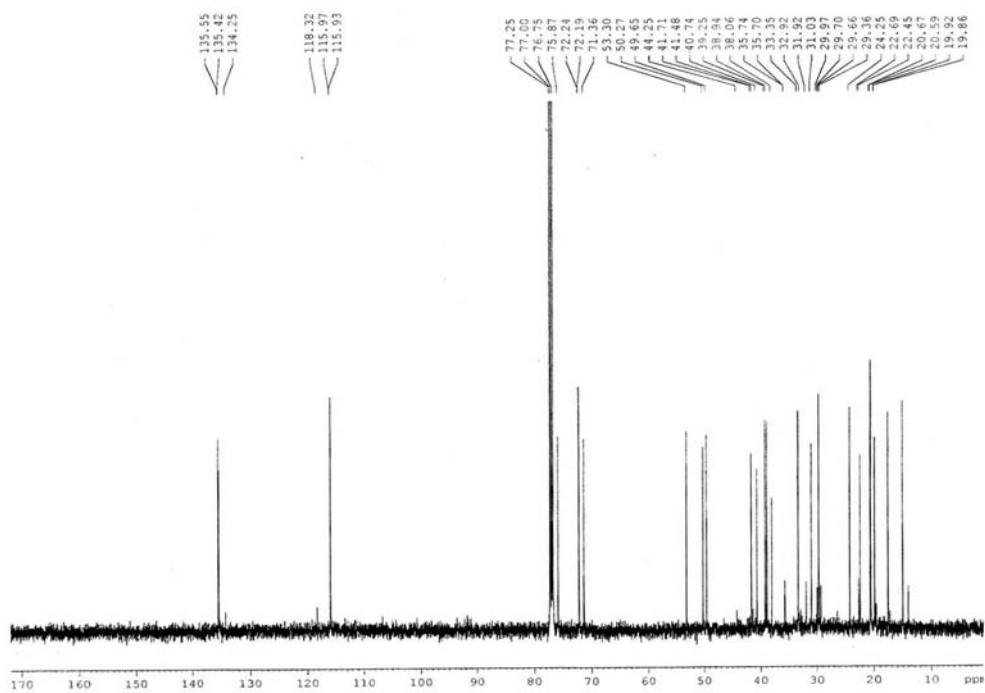


Figure 18S. NMR ^{13}C - CPD of 7 (125 MHz, CD_3OD)

Figure 23S. NMR ^1H spectrum of **10** (500 MHz, CDCl_3)Figure 24S. NMR ^{13}C -CPD spectrum of **10** (125 MHz, CDCl_3)

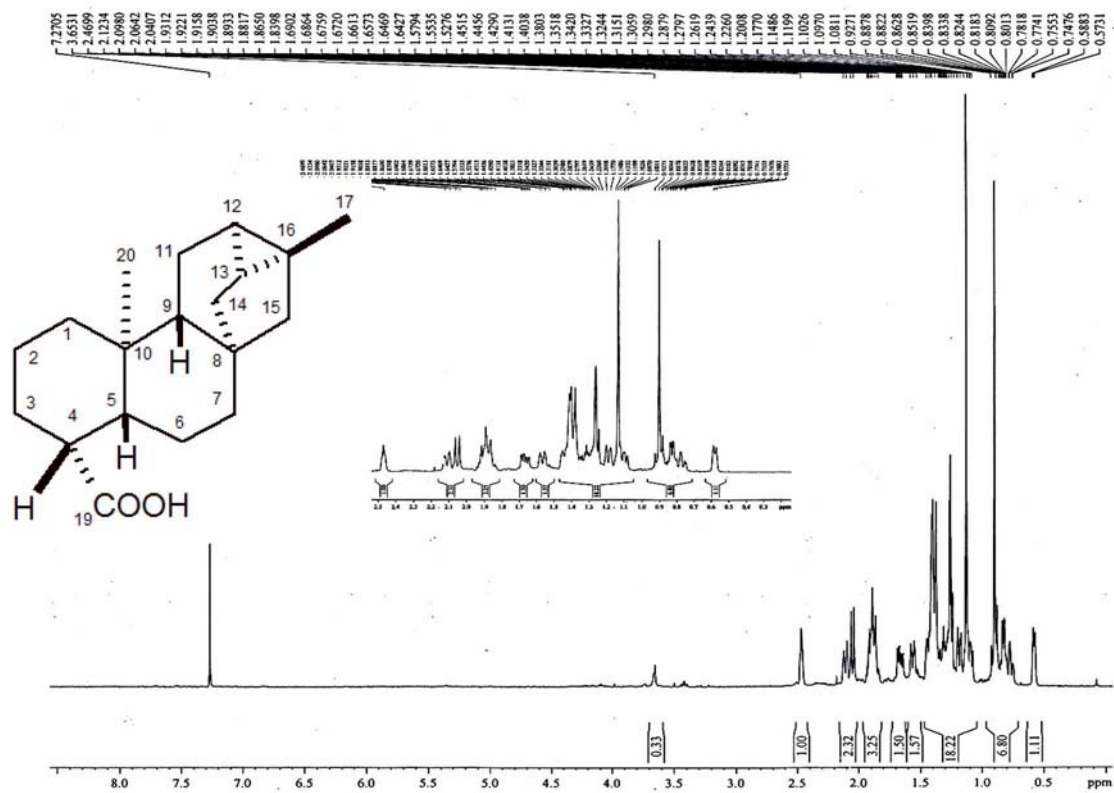


Figure 25S. NMR ^1H spectrum of **II** (500 MHz, CDCl_3)

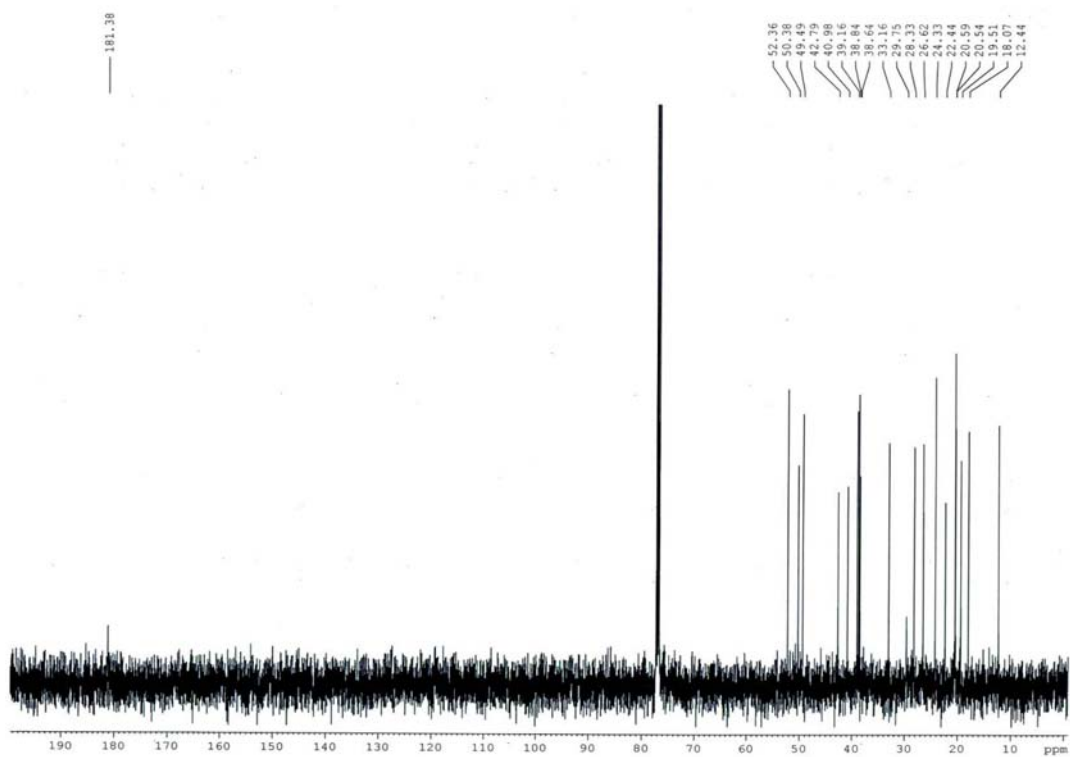


Figure 26S. NMR ^{13}C - CPD spectrum of **II** (125 MHz, CDCl_3)