SUPPLEMENTARY INFORMATION

Manuscript title: Lupane triterpenes from the leaves of the tropical rain forest tree *Hopea odorata* Roxb. and their cytotoxic activities.

Authors: Malai Satiraphan[a,b], Perayot Pamornsilpadaharm[b], Chavalit Sittisombut[b], Uthai Sotanaphun[b], Françoise Raynaud[c], Christiane Garbay[c], Sylvie Michel[a], Xavier Cachet[a,*]

Contents (S1 to S are the page numbers)

Evaluation of the cytotoxic activities of isolated lupanes: Experimental details and summary of results.

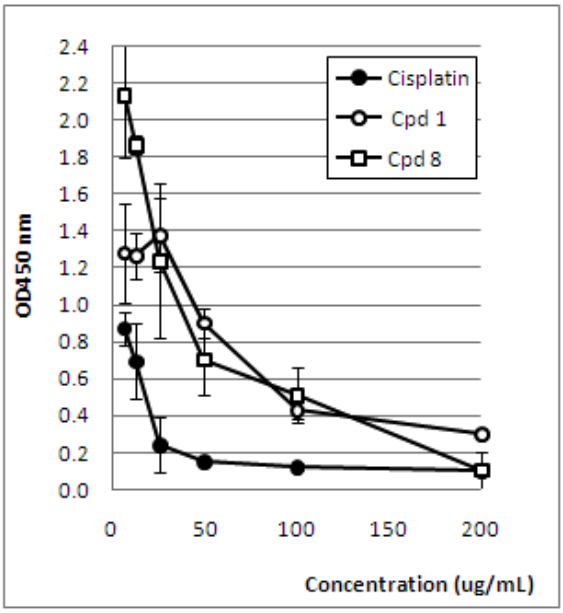
Materials. Dulbecco's Modified Eagle's Medium (DMEM), fetal bovine serum (FBS), penicillin, streptomycin, trypsin/EDTA were purchased from Invitrogen (Carlsbad, CA, USA), WST-1 solution were purchased from Roche Applied Science (Meylan Cedex, France).

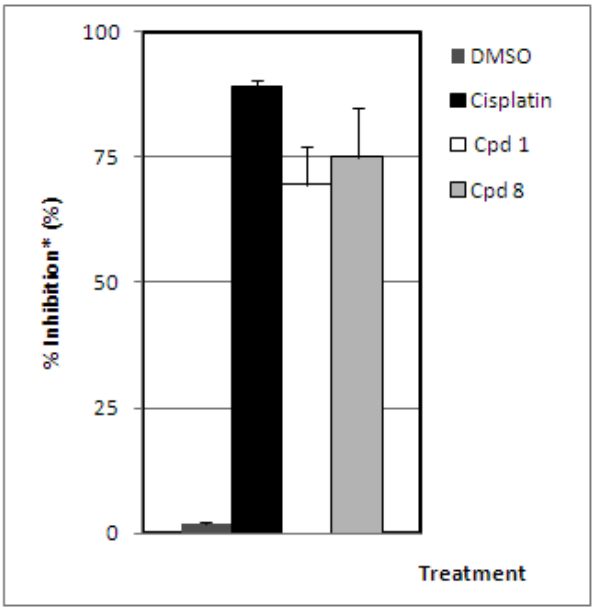
In vitro cytotoxic assay. The cytotoxic effects of isolated lupanes on various human cancer cell lines were evaluated using WST-1 method, as previously described (Ishiyama et al., 1996; Tsai et al., 2011). Different human cancer cell lines (*i.e.* prostate cancer cell line (PC3), human breast adenocarcinoma cell line (MDA-MB-231), colorectal adenocarcinoma cell line (HT-29) and colorectal carcinoma cell line (HCT 116)), were selected and seeded onto 96-well plates at a cell density of 5,000 cells/well in 5% CO_2 incubator at 37°C. After 24 h incubation, cells were exposed to various concentrations of each compound **1** to **8** (3 wells for each dilution) for 24 hr. Then, the tetrazolium salt WST-1 solution was added and cultured for further 4 h. To determine the cell survival, Optical Density (OD) was measured with a Bio-Rad Coda microplate analyzer at a wavelength of 450 nm (reference wavelength: 600 nm). Cisplatin was used as a positive control. Results presented in Table 2 were expressed as IC_{50}, the concentration required for 50% inhibition cell growth of treated cells compared to untreated controls. Dose-response curves of the active lupane triterpenes against human cancer cell line compared to cisplatin were shown in Figure 4.

Statistical analysis: IC_{50} are shown as mean ± standard deviation (SD) of triplicates from each independent experiment. Cell proliferation form WST-1 activities were analyzed using the student's t-test. P-values less than 0.05 were considered statistically significant.

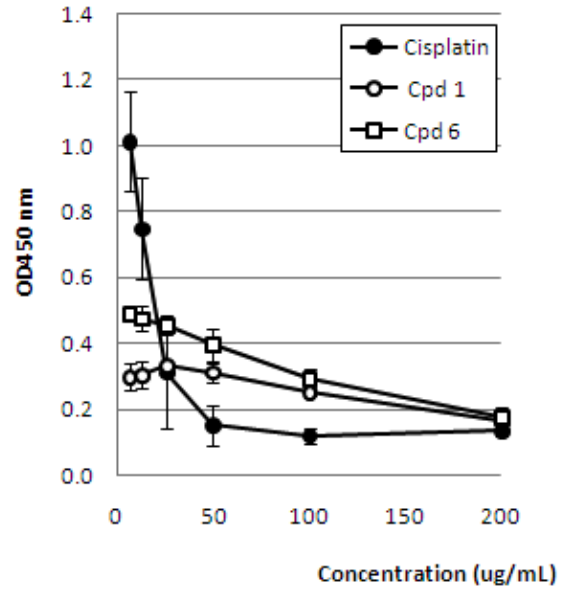
Fig. 4 Dose-response curve and % growth inhibition at 200 µg/mL of HCT116 cells to a 24-hour treatment with compounds **1** and **8** or cisplatine (positive control). (A); Dose-response curve and % growth inhibition at 200 µg/mL of PC3 cells to a 24-hour treatment with compounds **1** and **6** or cisplatine (positive control). (B)

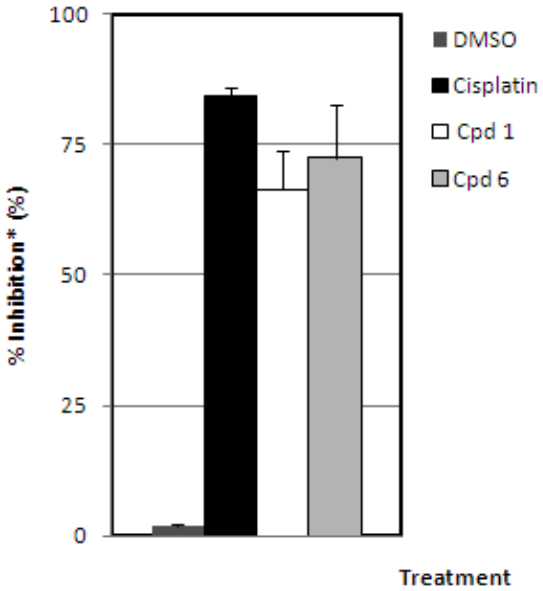
A) HCT116 cell line





B) PC3 cell line





Purification and identification of isolated compounds: Experimental with full physical and spectroscopic data for compounds **1-8**, including a complete assignment of ^{1}H and ^{13}C NMR data for lupanes **6-8**.

General methods

1D and 2D NMR spectra were recorded on Bruker AC300 (300 MHz) and Avance 400 (400 MHz). Chemical shifts are given in parts per million (ppm, δ) relative to solvent peaks as internal standards (δ: $CDCl_3$: 7.27 ppm (^{1}H), 77.0 ppm (^{13}C)); coupling constants are given in hertz (Hz, *J*). IR spectra were recorded using Nicolet 510 FT-IR spectrophotometer as film on NaCl pellets. Optical rotations were measured on a Perkin-Elmer Model 341 polarimeter at 20°C. Mass spectra were determined on Thermo Finnergan LCQ Advantage (ESI-ion trap) for low resolution MS and LCT Premier Waters® (ESI-TOF) for high resolution measurement. GC/MS analyses for sesquiterpenes and fatty acid were carried out with a GC chromatograph Hewlett Pack 6890 GC coupled to a 5975 quadrupole MS. Rough fractionation was performed on MPLC (medium pressure liquid chromatography) BUCHI684 Fraction collector. Further separation was carried out on silica gel column chromatography (Silica gel 60A C.C. 20-45μm chromagel SDS-CARLOERBA).

Betulonic acid (1) $C_{30}H_{46}O_3$; MW 454; white solid; $[\alpha]^{20}_{D}$ +12.2 ($CHCl_3$, c=0.09) ; IR (film) ν_{max} (cm^{-1}) 3500-2500 (br), 3066, 2917, 2849, 2863, 1703, 1694, 1462, 1377 and 757; ^{1}H and ^{13}C NMR ($CDCl_3$) data see Tables 1 and 2; MS (ESI-) *m/z* 453 $[M-H]^-$.

Betulinic acid (2) $C_{30}H_{48}O_3$; MW 456; colorless crystals; $[\alpha]^{20}_{D}$ +2.7° ($CHCl_3$, c=0.075) ; IR (film) ν_{max} (cm^{-1}) 3466-2500 (br), 3069, 2926, 2851 and 1687; ^{1}H NMR (δ_H, ppm, in $CDCl_3$) 0.70 (H5), 0.78 (H24), 0.82 (H25), 0.92 (H26), 0.96 (H23), 0.98 (H27), 1.71 (H30), 3.04 (H19), 3.21 (H3), 4.62, 4.75 (H29) ; ^{13}C NMR (δ_C, ppm, in $CDCl_3$) 38.7 (C1), 27.4 (C2), 79.0 (C-3), 38.8 (C4), 55.3 (C5), 18.3 (C6), 34.3 (C7), 40.7 (C8), 50.5 (C9), 37.2 (C10), 20.8 (C11), 25.5 (C12), 38.4 (C13), 42.4 (C14), 30.5 (C15), 32.1 (C16), 56.3 (C17), 49.2 (C18), 46.9 (C19), 150.4 (C20), 29.7 (C21), 37.1 (C22), 28.0

(C23), 15.4 (C24), 16.1 (C25), 16.1 (C26), 14.7 (C27), 180.5 (C28), 109.7 (C29), 19.4 (C30); MS (ES-) *m/z* 455 $[M-H]^-$.

Epibetulinic acid (3) $C_{30}H_{48}O_3$; MW 456; white solid; $[\alpha]^{20}_D$ -3.3 ($CHCl_3$, c=0.09) ; IR (film) ν_{max} (cm^{-1}) 3700-2500 (br), 3428, 3069, 2914, 2844, 1700, 1459, 1295 and 762; ^1H NMR (δ_H, ppm, in $CDCl_3$) 0.82 (H24), 0.84 (H25), 0.94 (H23+H26), 1.00 (H27), 1.70 (H30), 3.02 (H19), 3.40 (H3), 4.62, 4.75 (H29) ; ^{13}C NMR (δ_C, ppm, in $CDCl_3$) 33.2 (C1), 25.4 (C2),76.3 (C-3), 37.5 (C4), 49.0 (C5), 18.2 (C6), 34.2 (C7), 40.9 (C8), 50.3 (C9), 37.3 (C10), 20.7 (C11), 25.5 (C12), 38.4 (C13), 42.5 (C14), 30.6 (C15), 32.2 (C16), 56.4 (C17), 49.2 (C18), 46.9 (C19), 150.5 (C20), 29.6 (C21), 37.1 (C22), 28.3 (C23), 22.1 (C24), 15.9 (C25), 16.0 (C26), 14.8 (C27), 181.2 (C28), 109.7 (C29), 19.4 (C30); MS (ES-) *m/z* 455 $[M-H]^-$.

28-hydroxylup-20(29)-en-3-one (Betulone) (4) $C_{30}H_{48}O_2$; MW 440; white solid; $[\alpha]^{20}_D$ +21.7 ($CHCl_3$, c=0.12) ; IR (film) ν_{max} (cm^{-1}) 3435, 3065, 2917, 2847, 1705, 1463, 1376, 1025 and 757; ^1H NMR (δ_H, ppm, in $CDCl_3$) 0.93 (H25), 1.00 (H27), 1.03 (H24), 1.07 (H26), 1.08 (H23), 1.70 (H30), 3.36, 3.81 (H28), 4.59, 4.69 (H29) ; ^{13}C NMR (δ_C, ppm, in $CDCl_3$) 39.6 (C1), 34.2 (C2), 218.3 (C3), 47.4 (C4), 54.9 (C5), 19.7 (C6), 33.5 (C7), 40.9 (C8), 49.7 (C9), 36.9 (C10), 21.4 (C11), 25.2 (C12), 37.4 (C13), 42.8 (C14), 27.0 (C15), 29.3 (C16), 47.8 (C17), 48.7 (C18), 47.8 (C19), 150.4 (C20), 29.6 (C21), 34.0 (C22), 26.6 (C23), 21.1 (C24), 15.8 (C25), 16.0 (C26), 14.7 (C27), 60.5 (C28), 109.8 (C29), 19.1 (C30); MS (ES+) *m/z* 463 $[M+Na]^+$.

30-hydroxylup-20(29)-en-3-one (5) $C_{30}H_{48}O_2$; MW 440; white powder; $[\alpha]^{20}_D$ +14.4 ($CHCl_3$, c=0.18) ; IR (film) ν_{max} (cm^{-1}) 3448, 3085, 2937, 2851 and 1705; ^1H and ^{13}C NMR ($CDCl_3$) data see Tables 1 and 2; MS (ES+) *m/z* 463 $[M+Na]^+$.

3,30-dioxo-lup-20,29-en-28-oic acid, (6) $C_{30}H_{44}O_4$; MW 468.3240; colorless crystals; $[\alpha]^{20}_D$ +30.6 ($CHCl_3$, c=0.11) ; IR (film) ν_{max} (cm^{-1}) 3700-2500 (br), 3069, 2924, 2845, 1731, 1685, 1463 and 1276; ^1H and ^{13}C NMR ($CDCl_3$) data see Tables 1 and 2; MS (ESI-) *m/z* 467 $[M-H]^-$. HRMS (ESI-TOF) *m/z* (ESI-) : 467.3146 $[M-H]^-$ (calcd for $C_{30}H_{43}O_4$ 467.3161)

28,30-dihydroxy-3-oxolup-20(29)-ene, (7) $C_{30}H_{48}O_3$; MW 456; green oil; $[\alpha]^{20}_D$ +3.0 ($CHCl_3$, c=0.33) ; IR (film) ν_{max} (cm^{-1}) 3434, 1699 and 1456; ^1H and ^{13}C NMR ($CDCl_3$) data see Tables 1 and 2; MS (ESI+) *m/z* 479 $[M+Na]^+$.

Messagenic acid G, (8) $C_{30}H_{46}O_4$; MW 470; amorphous solid; $[\alpha]^{20}_D$ +14.3 ($CHCl_3$, c=0.14) ; IR (film) ν_{max} (cm^{-1}) 3700-2500 (br), 3074, 2938, 2864 and 1695; ^1H and ^{13}C NMR ($CDCl_3$) data see Tables 1 and 2; MS (ESI-) *m/z* 469 $[M-H]^+$.

Table 1. ^{1}H NMR data (400 MHz) of **1, 5, 6, 7, 8** (in $CDCl_3$, *J* in Hz, δ in ppm)

Position	**1**	**5**	**6**	**7**	**8**
	δ_H	δ_H	δ_H	δ_H	δ_H
1	1.38 (m), 1H 1.91 (m), 1H	1.38 (m), 1H 1.90 (m), 1H	1.37 (m), 1H 1.89 (m), 1H	1.38 (m), 1H 1.88 (m), 1H	1.39 (m,1H) 1.90 (m,1H)
2	2.42 (m), 1H 2.50 (m), 1H	2.43 (m), 1H 2.50 (m), 1H	2.41 (m), 1H 2.47 (m), 1H	2.42 (m), 1H 2.49 (m), 1H	2.43 (m, 1H) 2.50 (m, 1H)
3					
4					
5	1.35 (m), 1H	1.32 (m), 1H	1.31 (m), 1H	1.33 (m), 1H	1.33 (m), 1H
6	1.52 (m), 2H	1.47 (m), 2H	1.46 (m), 2H	1.47 (m), 2H	1.47 (m), 2H
7	1.44 (m), 2H	1.45 (m), 2H	1.43 (m), 2H	1.45 (m), 1H	1.43 (m), 2H
8					
9	1.38 (s), 1H	1.38 (m), 1H	1.35 (m), 1H	1.38 (m), 1H	1.38 (m), 1H
10					
11	1.34 (m), 1H 1.44 (m), 1H	1.28 (m), 1H 1.42 (m), 1H	1.31 (m), 1H 1.38 (m), 1H	1.27 (m), 1H 1.43 (m), 1H	1.32 (m), 1H 1.45 (m), 1H
12	1.06 (m), 1H 1.73 (m), 1H	1.13 (m), 1H 1,36 (m), 1H	0.91 (m), 1H 1.34 (m), 1H	1.02 (m), 1H 1.41 (m), 1H	1.48 (m), 1H
13	2.23 (m), 1H	1.68 (m), 1H	2.22 (m), 1H	1.67 (m), 1H	2.21 (m), 1H
14					
15	1.42 (m), 1H 1.99 (m), 1H	1.06 (m), 1H 1.71 (m), 1H	1.23 (m), 1H 1.55 (m), 1H	1.12 (m), 1H 1.73 (m), 1H	1.23 (m), 1H 1.54 (m), 1H
16	1.44 (m), 1H 2.28 (m), 1H	1.40 (m), 1H 1.52 (m), 1H	1.51 (m), 1H 2.32 (m), 1H	1.13 (m), 1H 1.90 (m), 1H	1.46 (m), 1H 2.31 (m), 1H
17					
18	1.64 (m), 1H	1.47 (m), 1H	2.02 (m), 1H	1.72 (m), 1H	1.77 (t, *J*=11.5), 1H
19	3.02 (td, *J*=10.7; 4.8), 1H	2.34 (td, *J*=5.3, 10.8), 1H	3.35 (td, J=11.2; 4.6), 1H	2.31 (m), 1H	2.90 (td, *J*= 11.0, 4.5), 1H
20					
21	1.22 (m), 1H 1.54 (m), 1H	1.33 (m), 1H 2.08 (m), 1H	1.42 (m), 1H 2.16 (m), 1H	1.31 (m), 1H 1.96 (m), 1H	1.42 (m), 1H 2.11 (m), 1H
22	1.47 (m), 1H 1.99 (m), 1H	1.28 (m), 1H 1.40 (m), 1H	1.75 (m), 1H 2.00 (m), 1H	1.43 (m), 1H 2.12 (m), 1H	1.57 (m), 1H 1.98 (m), 1H
23	1.08 (s), 3H	1.08 (s), 3H	1.07 (s), 3H	1.07 (s), 3H	1.08 (s), 3H
24	1.03 (s), 3H	1.03 (s), 3H	1.01 (s), 3H	1.02 (s), 3H	1.02 (s), 3H
25	0.94 (s), 3H	0.93 (s), 3H	0.91 (s), 3H	0.92 (s), 3H	0.92 (s), 3H
26	0.99 (s), 3H	1.07 (s), 3H	0.96 (s), 3H	1.06 (s), 3H	0.97 (s), 3H
27	1.00 (s), 3H	0.96 (s), 3H	0.96 (s), 3H	0.99 (s), 3H	1.02 (s), 3H
28		0.80 (s), 3H		3.33 (d, *J*=10.8), 1H 3.80 (d, *J*=10.8), 1H	
29	4.62 (s), 1H 4.75 (s), 1H	4.91 (s), 1H 4.95 (s), 1H	5.93 (s), 1H 6.31 (s), 1H	4.91 (s), 1H 4.96 (s), 1H	4.94 (s), 1H 4.99 (s), 1H
30	1.70 (s), 3H	4.15(br d, *J*=14.3), 1H 4.11 (br d, *J*=14.3), 1H)	9.53 (s), 1H	4.12 (m), 2H	4.14 (m), 2H

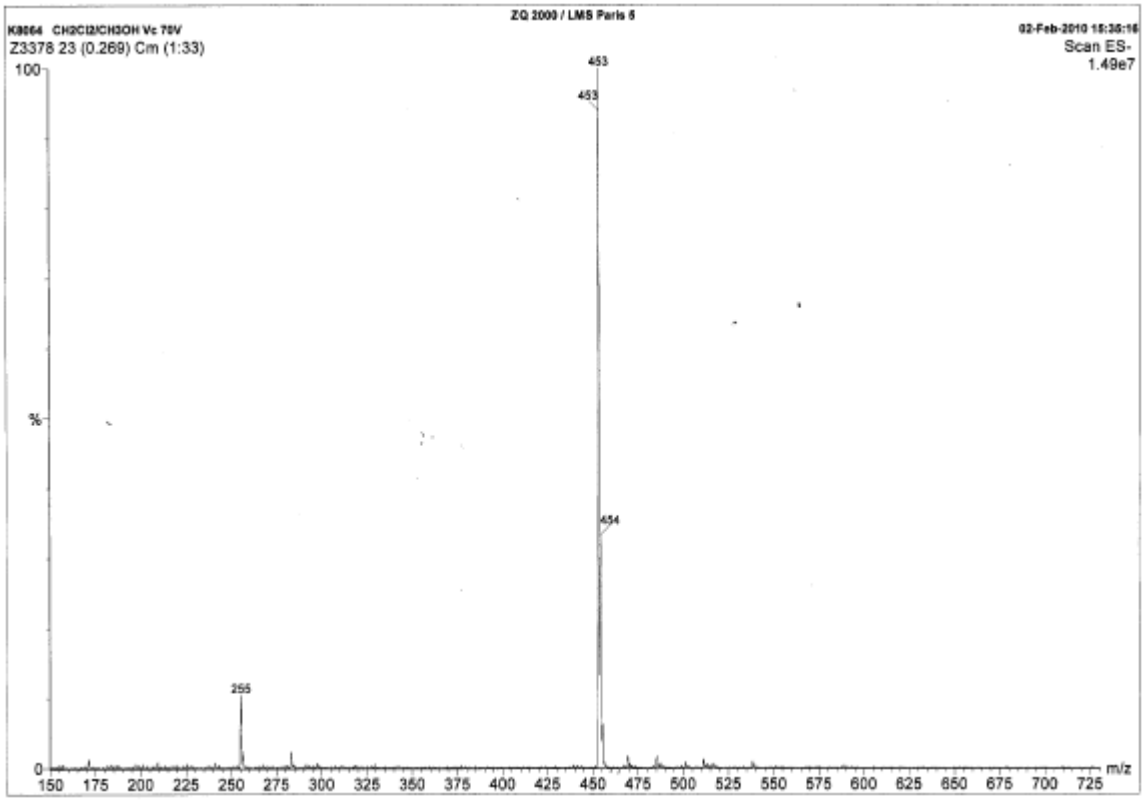
Table 2. ^{13}C NMR data (75 MHz) of **1, 5, 6, 7, 8** (in $CDCl_3$, δ in ppm)

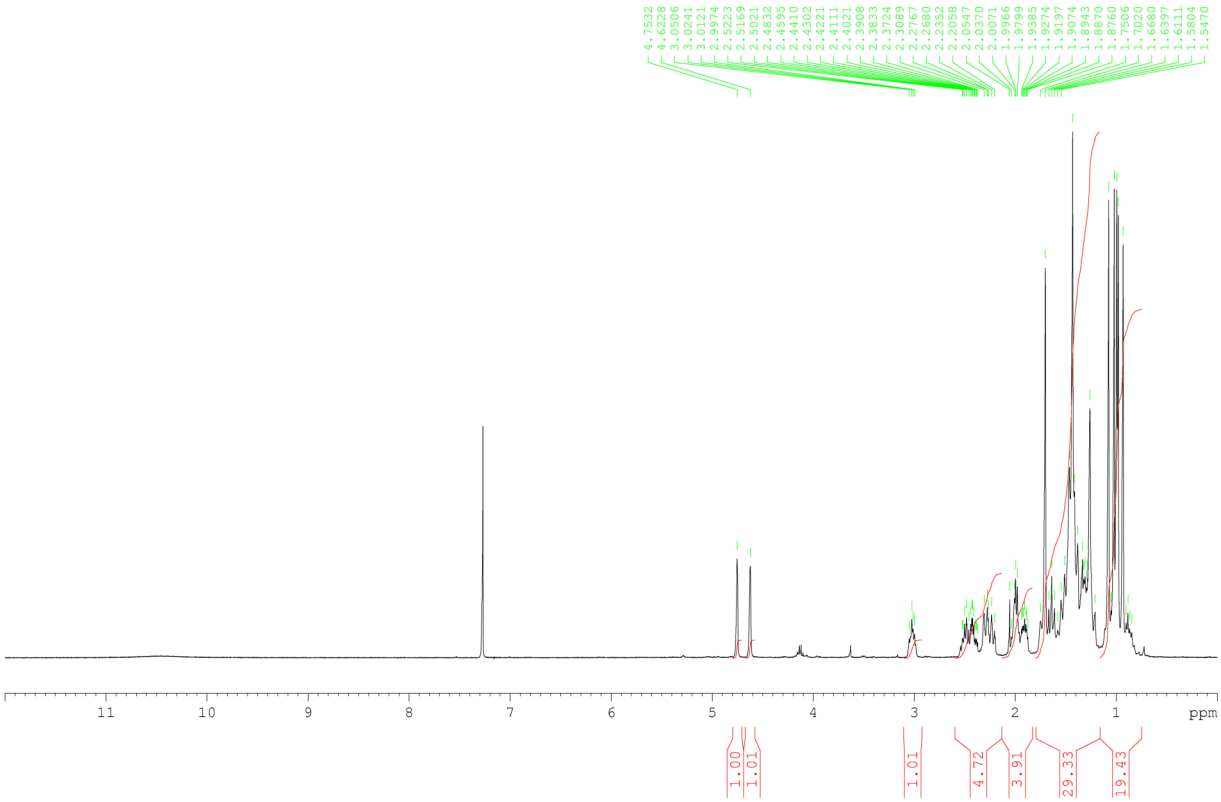
Position	**1**	**5**	**6**	**7**	**8**
	δ_C	δ_C	δ_C	δ_C	δ_C

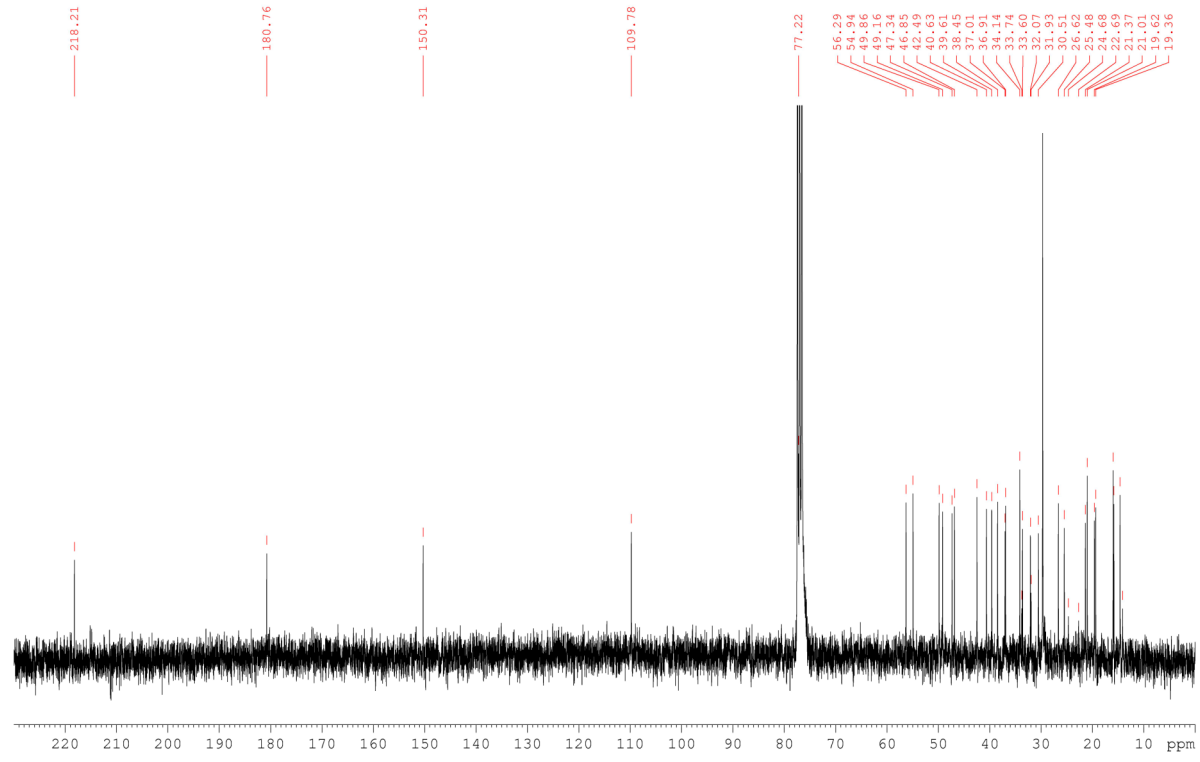
1	39.6	39.6	39.6	39.6	39.6
2	34.1	34.2	34.1	34.1	34.1
3	218.2	218.3	218.2	218.2	218.1
4	47.3	47.3	47.3	47.3	47.3
5	54.9	54.9	54.9	54.9	54.9
6	19.6	19.7	19.6	19.6	19.6
7	33.6	33.6	33.6	33.5	33.6
8	40.6	40.8	40.6	40.9	40.6
9	49.9	49.7	49.7	49.7	49.9
10	36.9	36.9	36.9	36.8	36.9
11	21.4	21.5	21.3	21.4	21.5
12	25.4	26.7	27.2	26.8	26.8
13	38.5	38.1	38.4	37.3	38.4
14	42.5	42.8	42.4	42.7	42.4
15	30.5	27.4	29.6	27.0	29.7
16	32.1	35.4	31.8	33.8	32.0
17	56.3	43.0	56.4	47.8	56.3
18	49.2	48.8	50.5	49.3	49.8
19	46.9	43.8	38.4	43.5	42.5
20	150.3	154.7	156.2	154.4	154.6
21	29.7	31.8	32.0	29.1	32.3
22	37.0	39.8	36.9	31.7	36.8
23	26.6	26.7	26.6	26.7	26.7
24	21.0	21.1	21.3	21.1	21.0
25	16.0	16.0	15.9	16.0	16.0
26	15.8	15.8	15.8	15.8	15.8
27	14.6	14.5	14.5	14.7	14.6
28	180.8	17.7	181.7	60.2	180.7
29	109.8	106.9	134.2	107.2	107.0
30	19.4	64.9	195.0	65.0	65.3

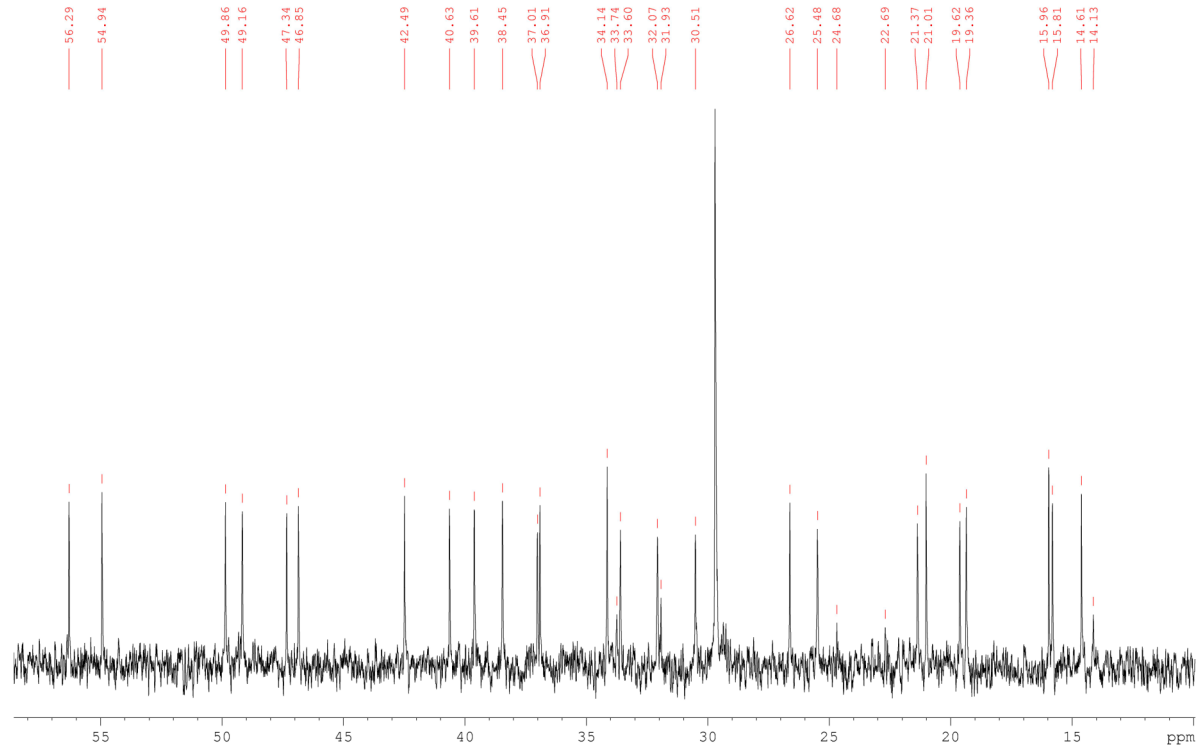
Original spectroscopic data:

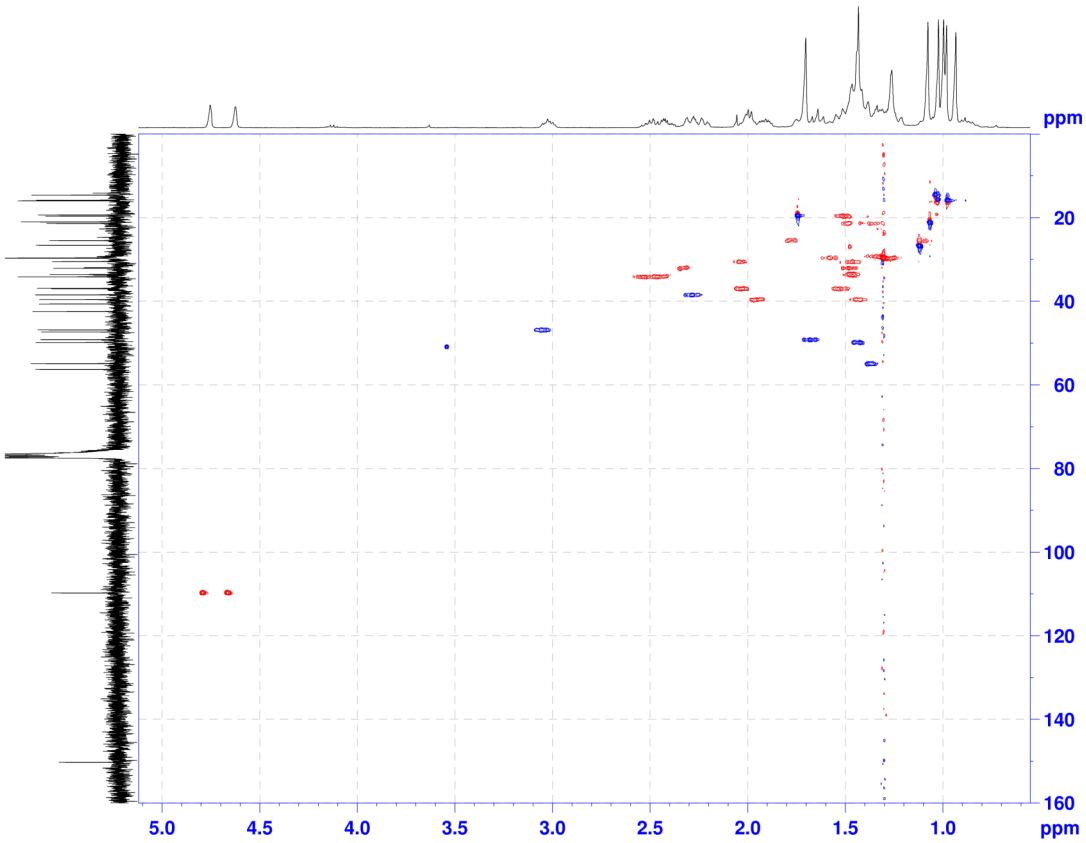
- Mass (ESI-: *m/z* 453 [M-H]$^-$) and NMR (^{1}H+^{13}C+HSQC edit) spectra of betulonic acid ($C_{30}H_{46}O_3$; MW 454) (1):



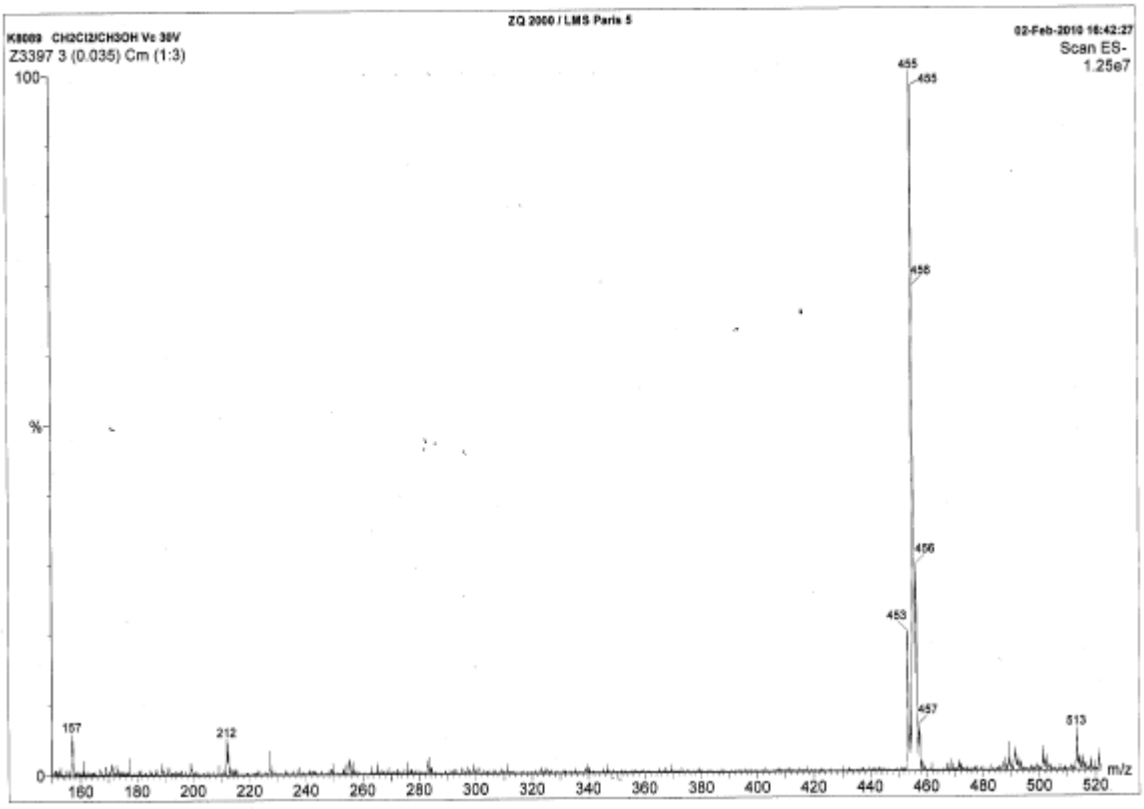

4.7532
4.6228
3.0506
3.0241
3.0121
2.9974
2.5223
2.5169
2.5021
2.4832
2.4435
2.4410
2.4302
2.4221
2.4111
2.4021
2.3908
2.3833
2.3724
2.3089
2.2767
2.2680
2.2352
2.2058
2.0547
2.0370
2.0071
1.9966
1.9789
1.9385
1.9274
1.9197
1.9074
1.8943
1.8870
1.8760
1.7506
1.7020
1.6680
1.6397
1.6111
1.5804
1.5470
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.01
1.01
4.72
3.91
29.33
19.43

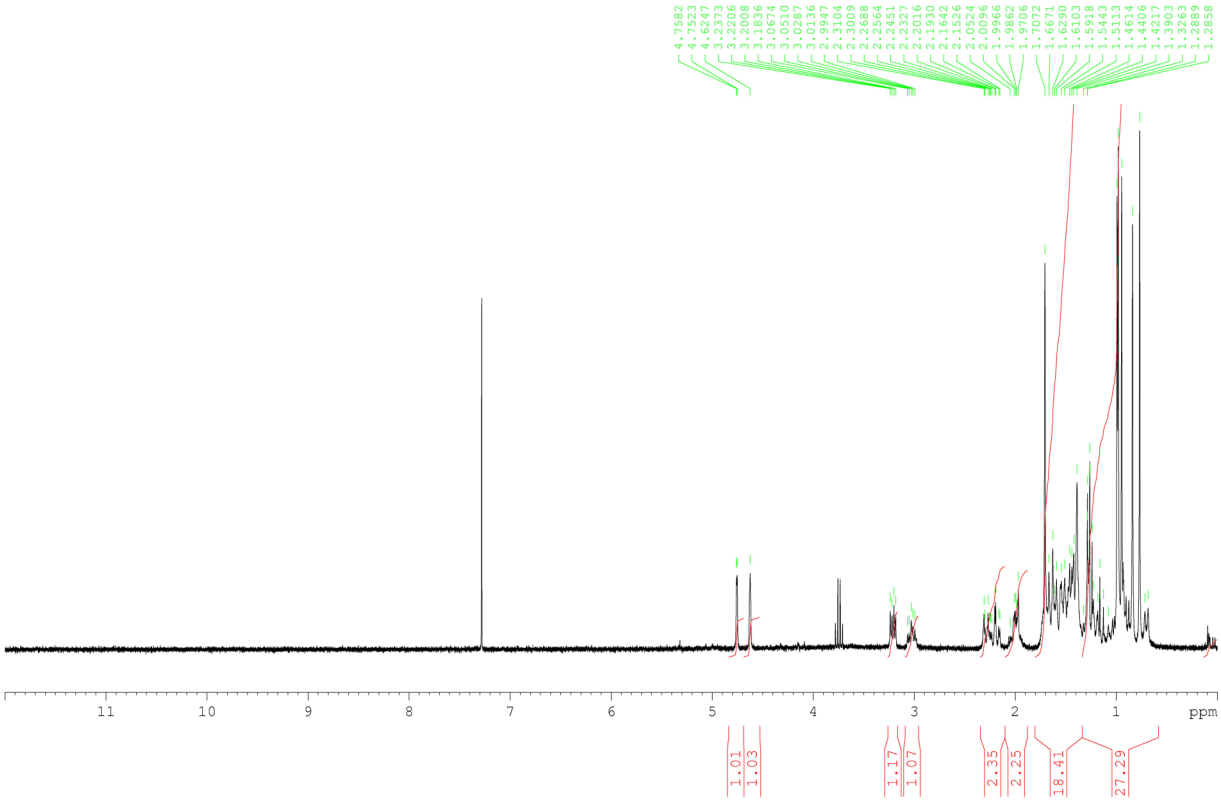

218.21
180.76
150.31
109.78
77.22
56.29
54.94
49.86
49.16
47.34
46.85
42.49
40.63
39.61
38.45
37.01
36.91
34.14
33.74
33.60
32.07
31.93
30.51
26.62
25.48
22.69
21.37
21.01
19.62
19.36
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

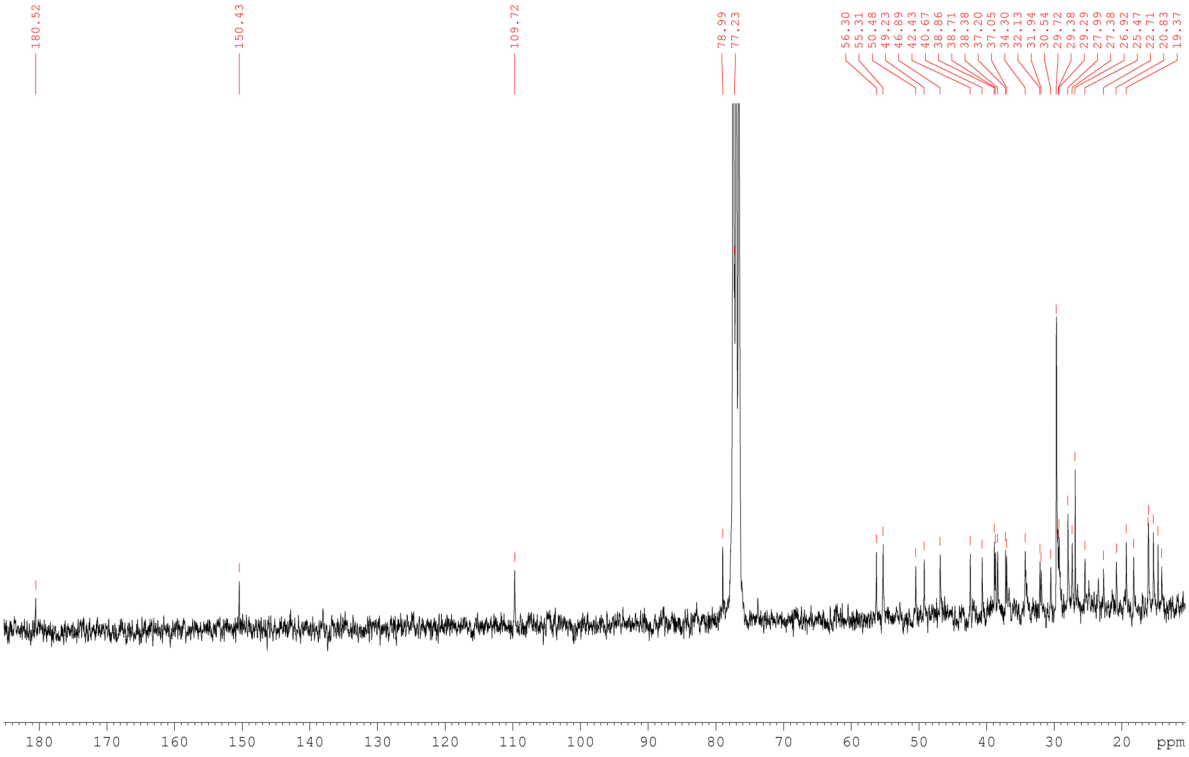

56.29
54.94
49.86
49.16
47.34
46.85
42.49
40.63
39.61
38.45
37.01
36.91
34.14
33.74
33.60
32.07
31.93
30.51
26.62
25.48
24.68
22.69
21.37
21.01
19.62
19.36
15.96
15.81
14.61
14.13
55
50
45
40
35
30
25
20
15
ppm

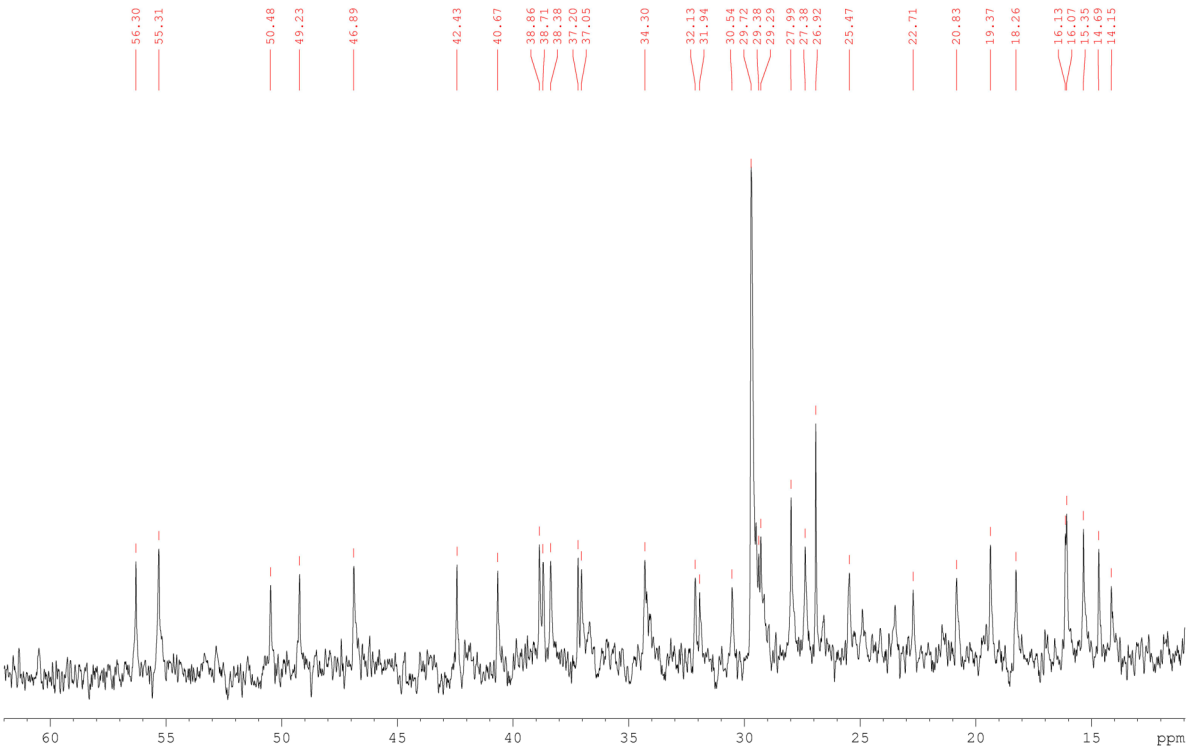

ppm
20
40
60
80
100
120
140
160
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

- Mass (ESI-: *m/z* 455 [M-H]$^-$) and NMR (^{1}H+^{13}C) spectra of betulinic acid ($C_{30}H_{48}O_3$; MW 456) (2):

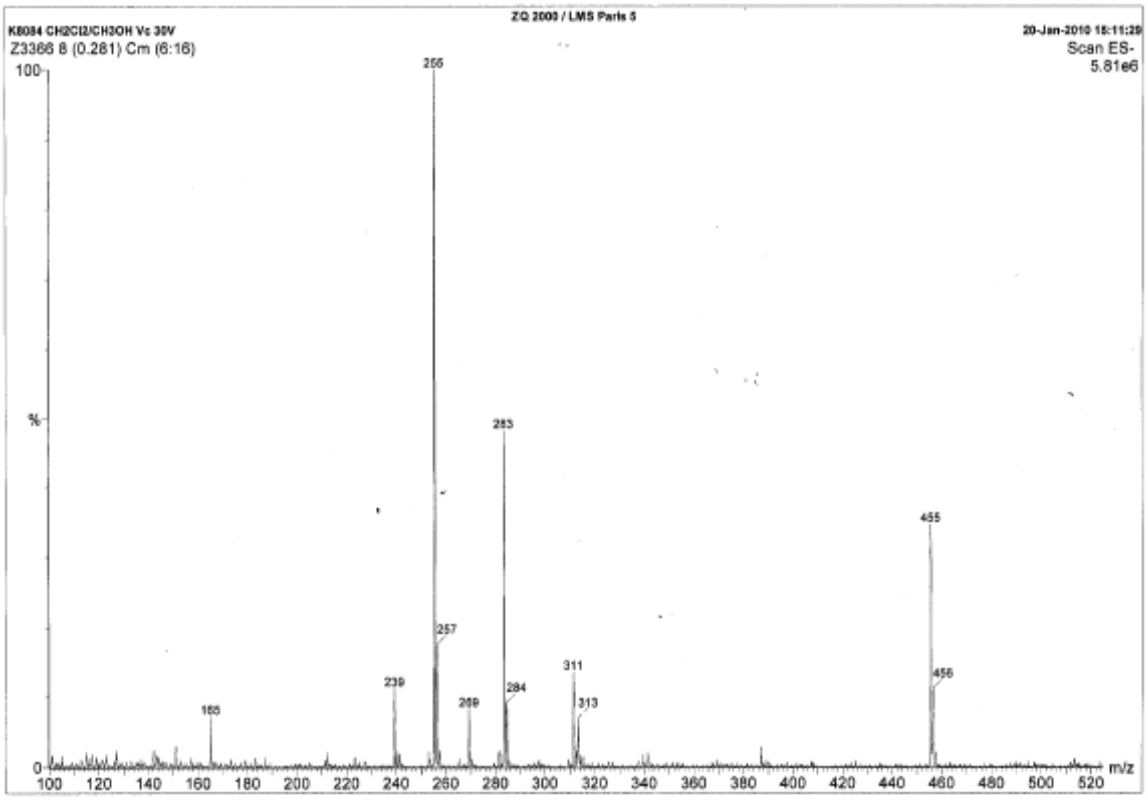


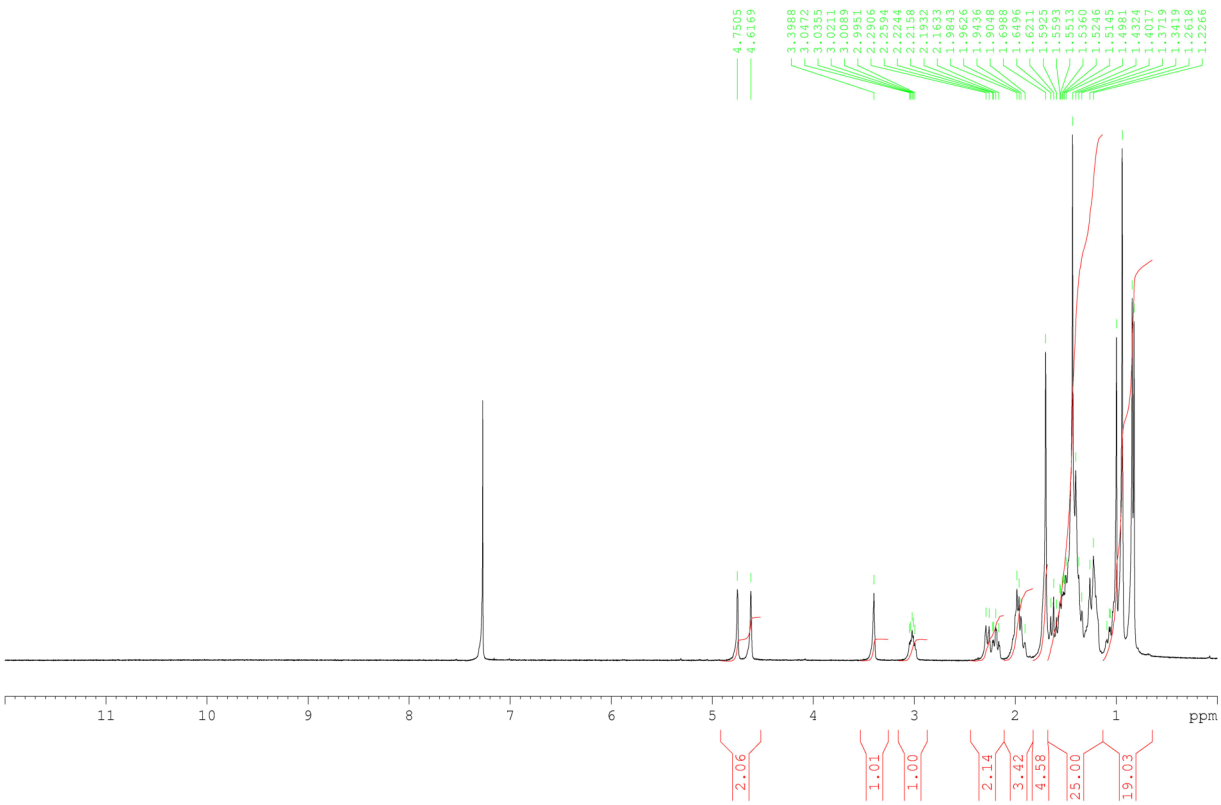


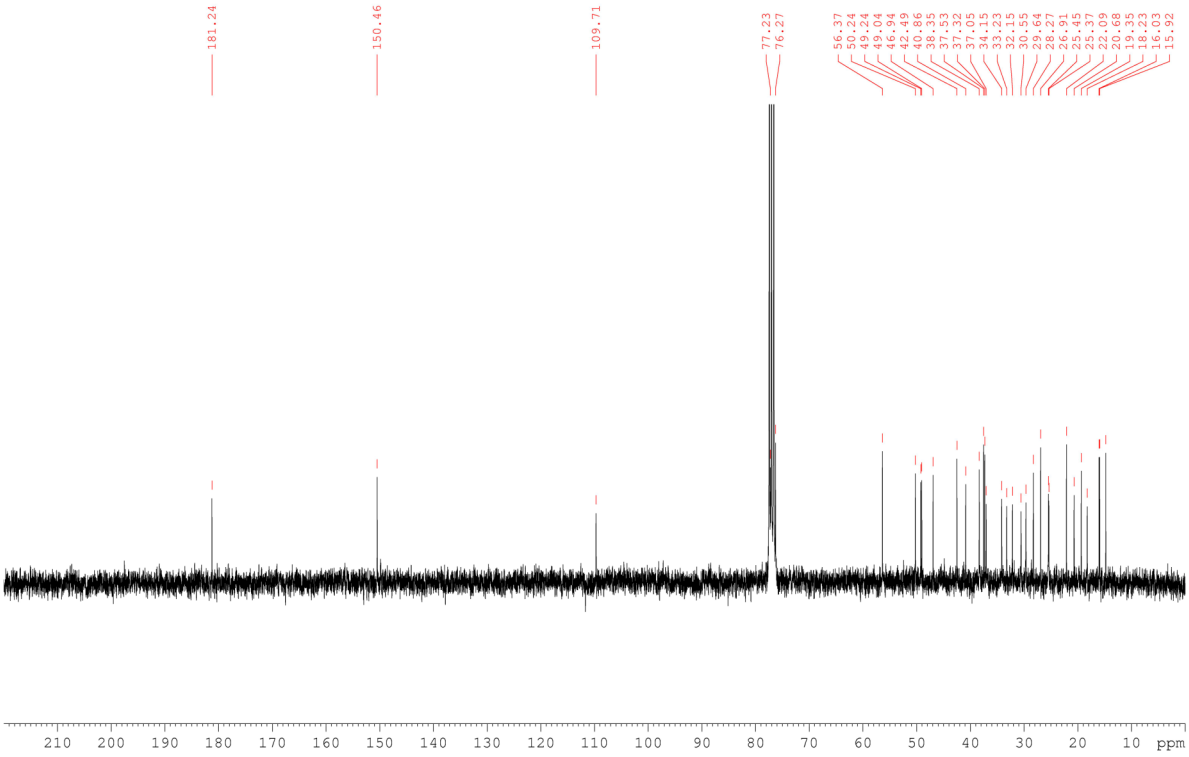

180.52
150.43
109.72
78.99
77.23
56.30
55.31
50.48
49.23
46.89
42.43
40.67
38.86
38.71
38.38
37.20
37.05
34.30
32.13
31.94
30.54
29.72
29.38
29.29
27.99
27.38
26.92
25.47
22.71
20.83
19.37
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

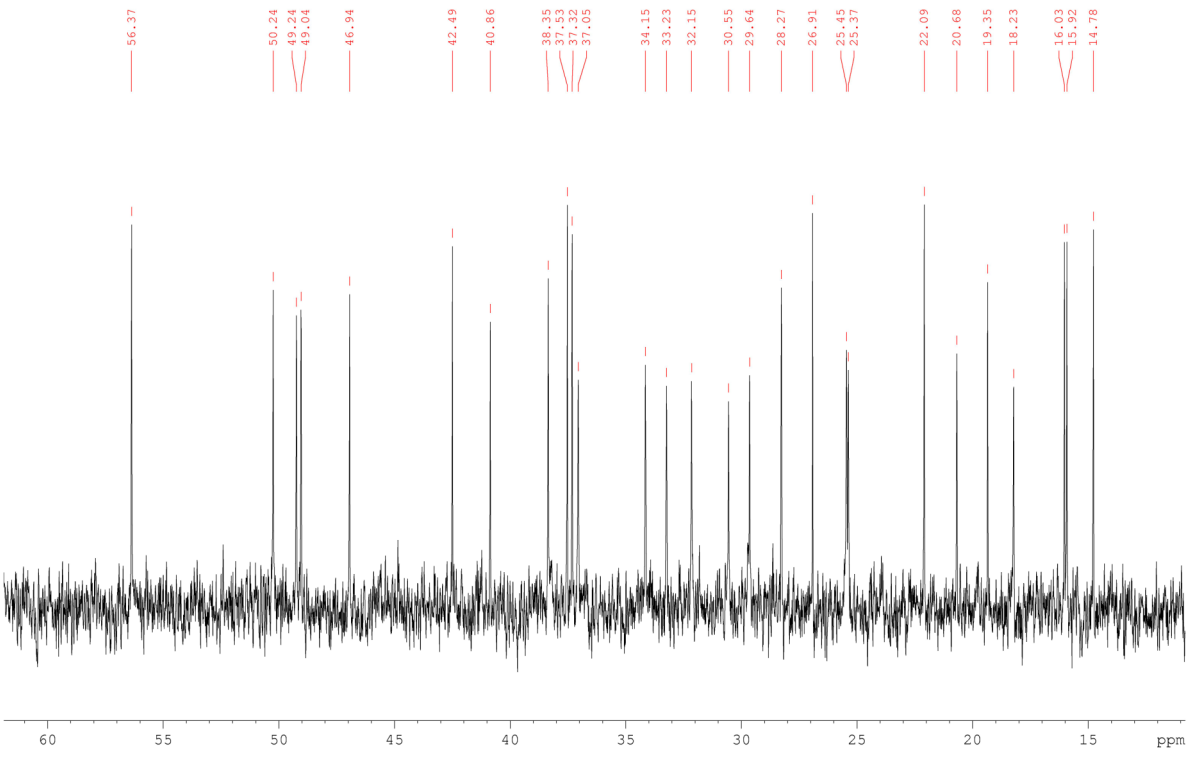

56.30
55.31
50.48
49.23
46.89
42.43
40.67
38.86
38.71
38.38
37.20
37.05
34.30
32.13
31.94
30.54
29.72
29.38
29.29
27.99
27.38
26.92
25.47
22.71
20.83
19.37
18.26
16.13
16.07
15.35
14.69
14.15
60 55 50 45 40 35 30 25 20 15 ppm

- Mass (ESI-: *m/z* 455 $[M-H]^-$) and NMR (^1H+^{13}C) spectra of epibetulinic acid ($C_{30}H_{48}O_3$; MW 456) (3):

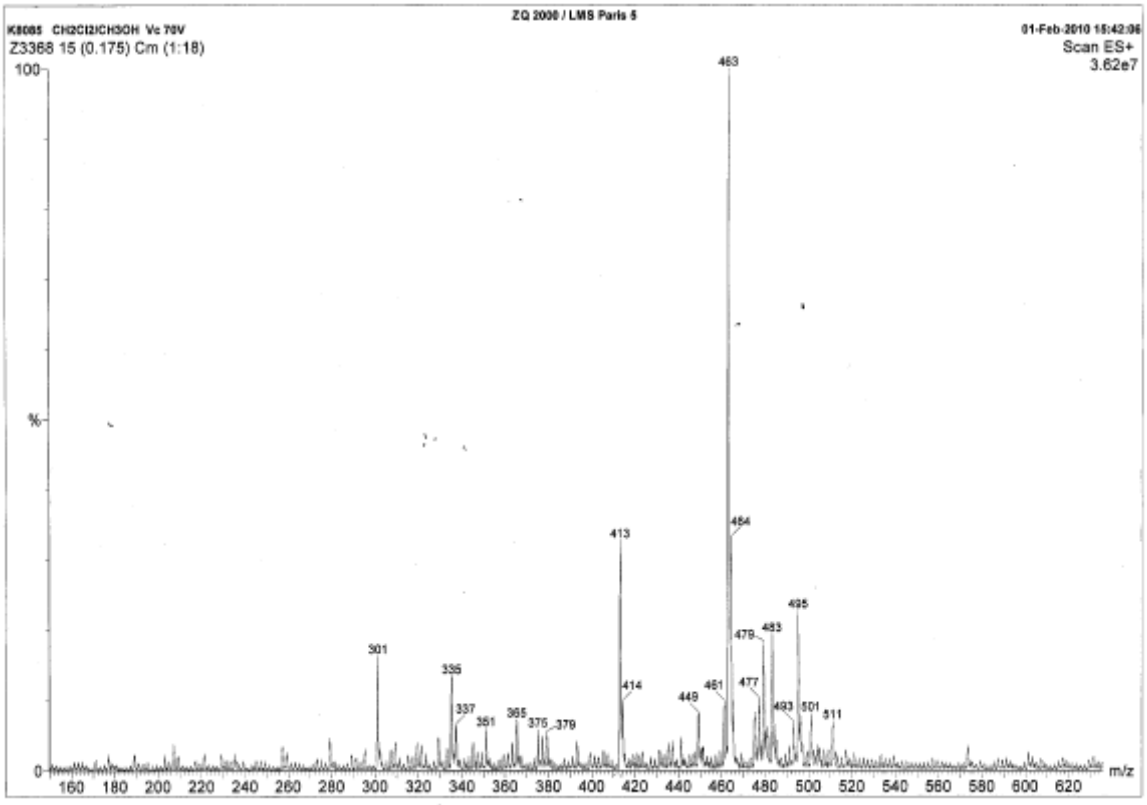


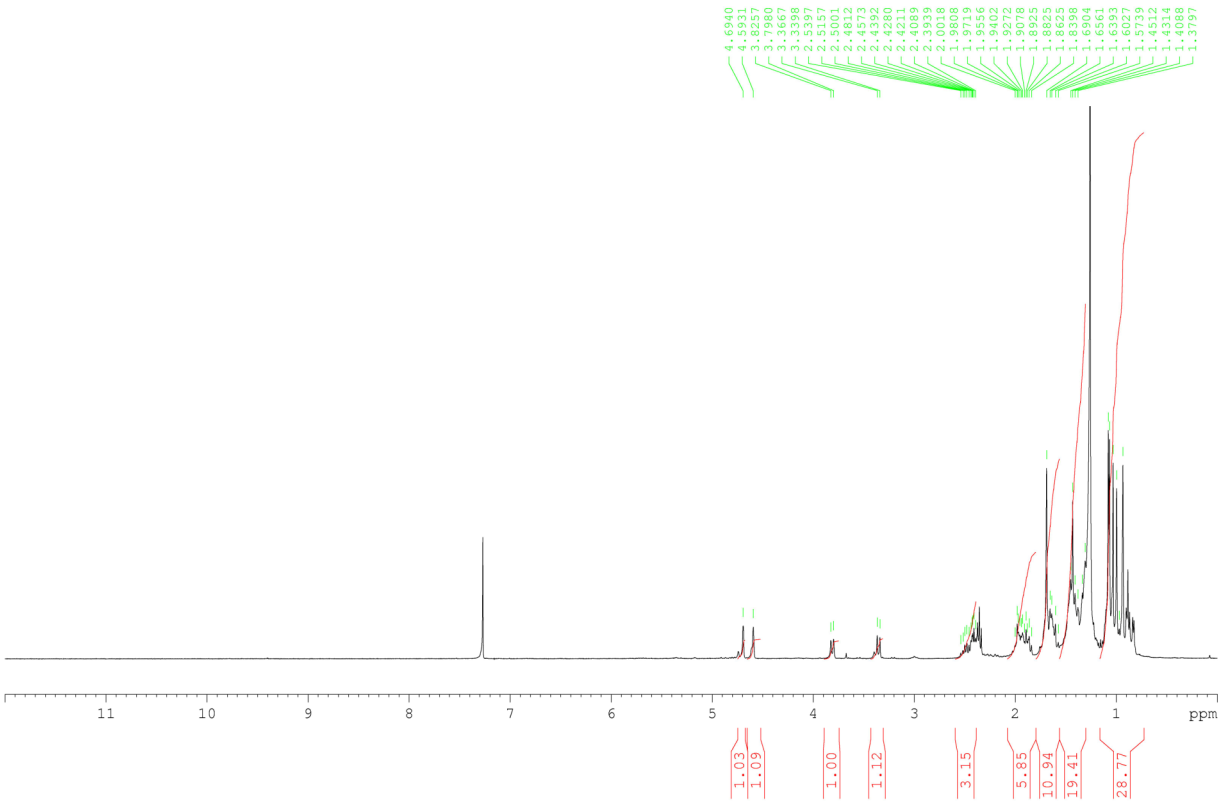


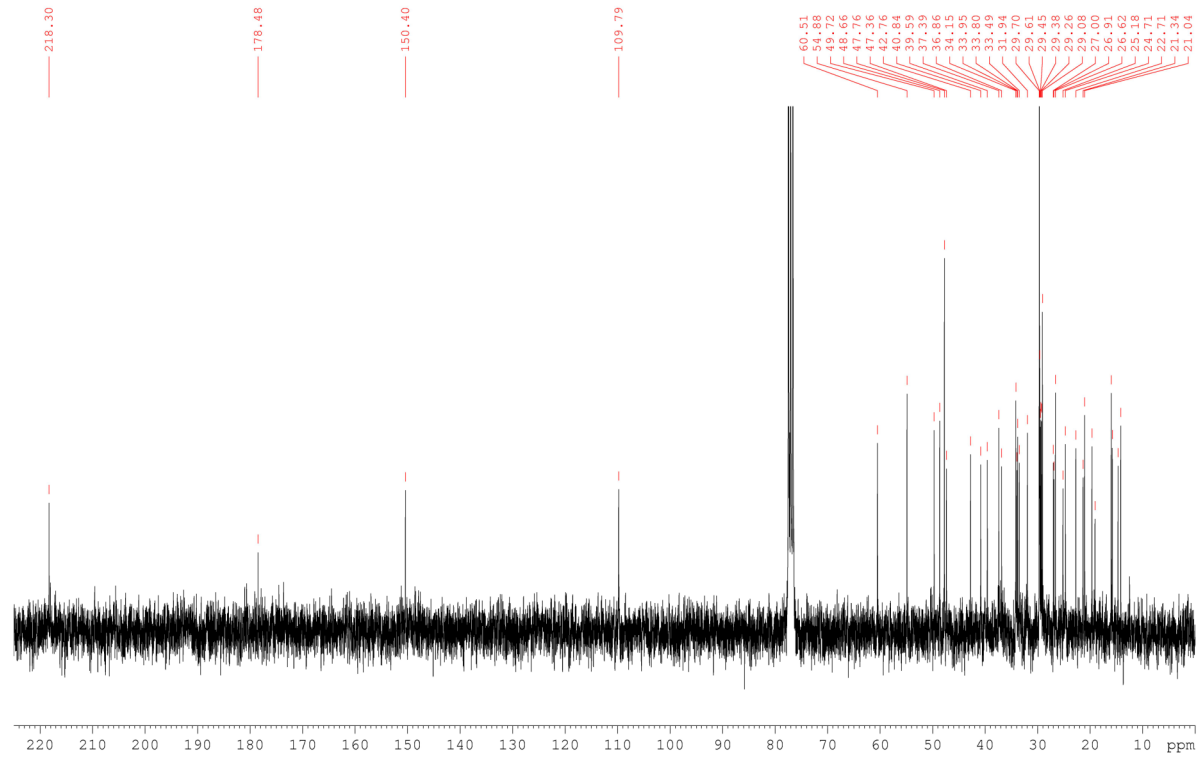

181.24
150.46
109.71
77.23
76.27
56.37
50.24
49.24
49.04
46.94
42.49
40.86
38.35
37.53
37.32
37.05
34.15
33.23
32.15
30.55
29.64
28.27
26.91
25.45
25.37
22.09
20.68
19.35
18.23
16.03
15.92
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

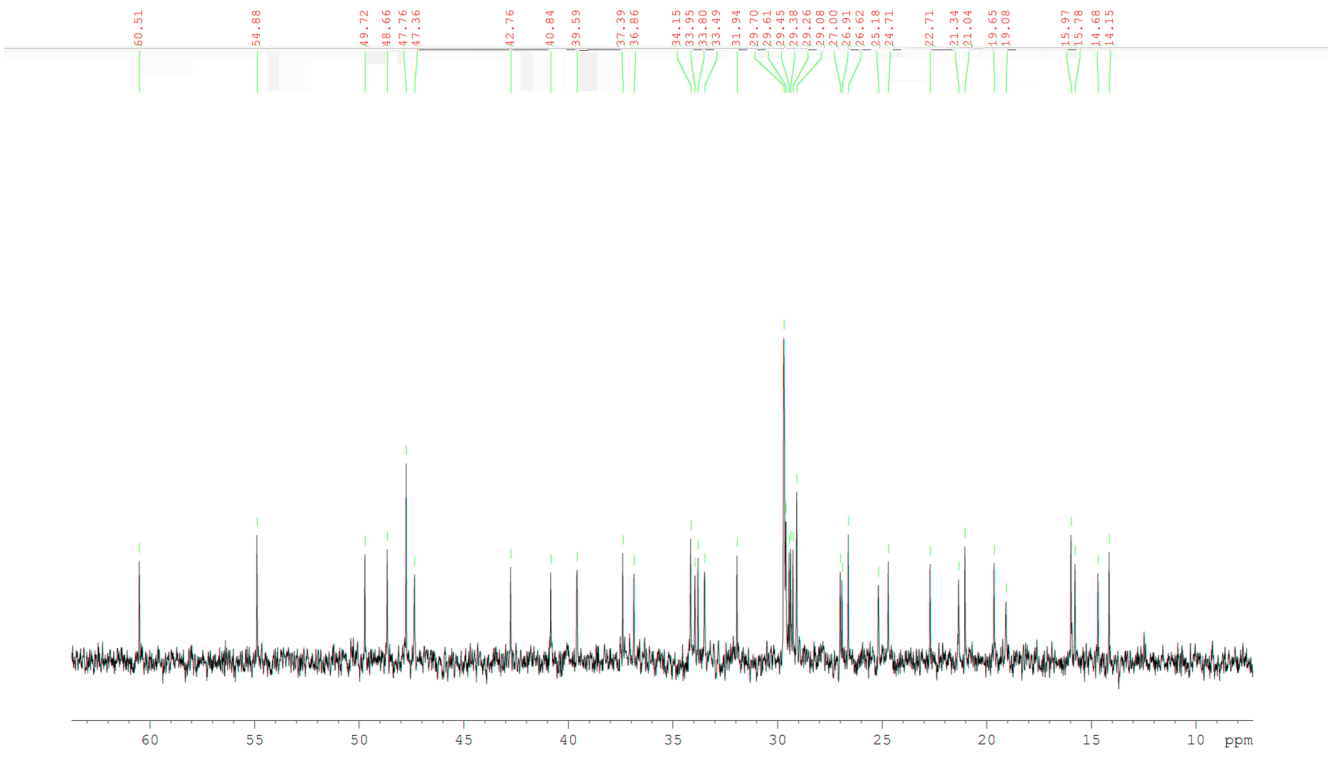

56.37
50.24
49.24
49.04
46.94
42.49
40.86
38.35
37.53
37.32
37.05
34.15
33.23
32.15
30.55
29.64
28.27
26.91
25.45
25.37
22.09
20.68
19.35
18.23
16.03
15.92
14.78
60 55 50 45 40 35 30 25 20 15 ppm

- Mass (ESI+: *m/z* 463 $[M+Na]^+$) and NMR ($^1H+^{13}C$) spectra of 28-hydroxylup-20(29)-en-3-one (= betulone) ($C_{30}H_{48}O_2$; MW 440) (4):

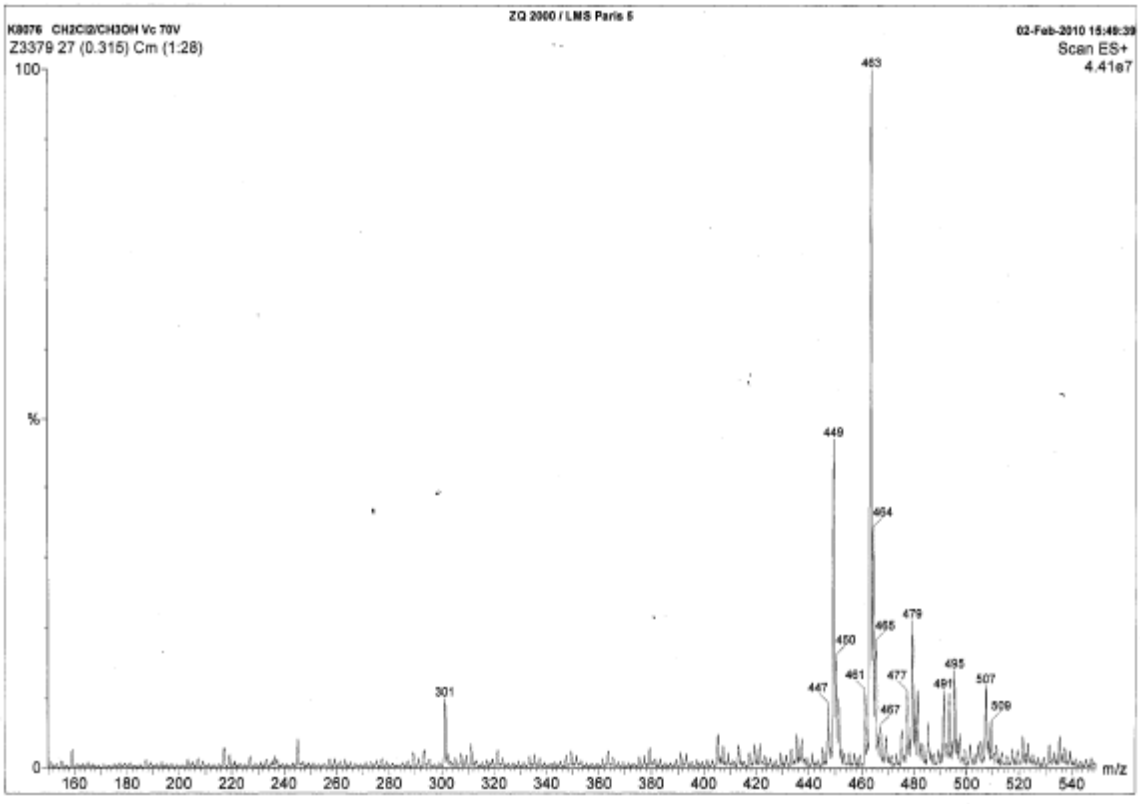


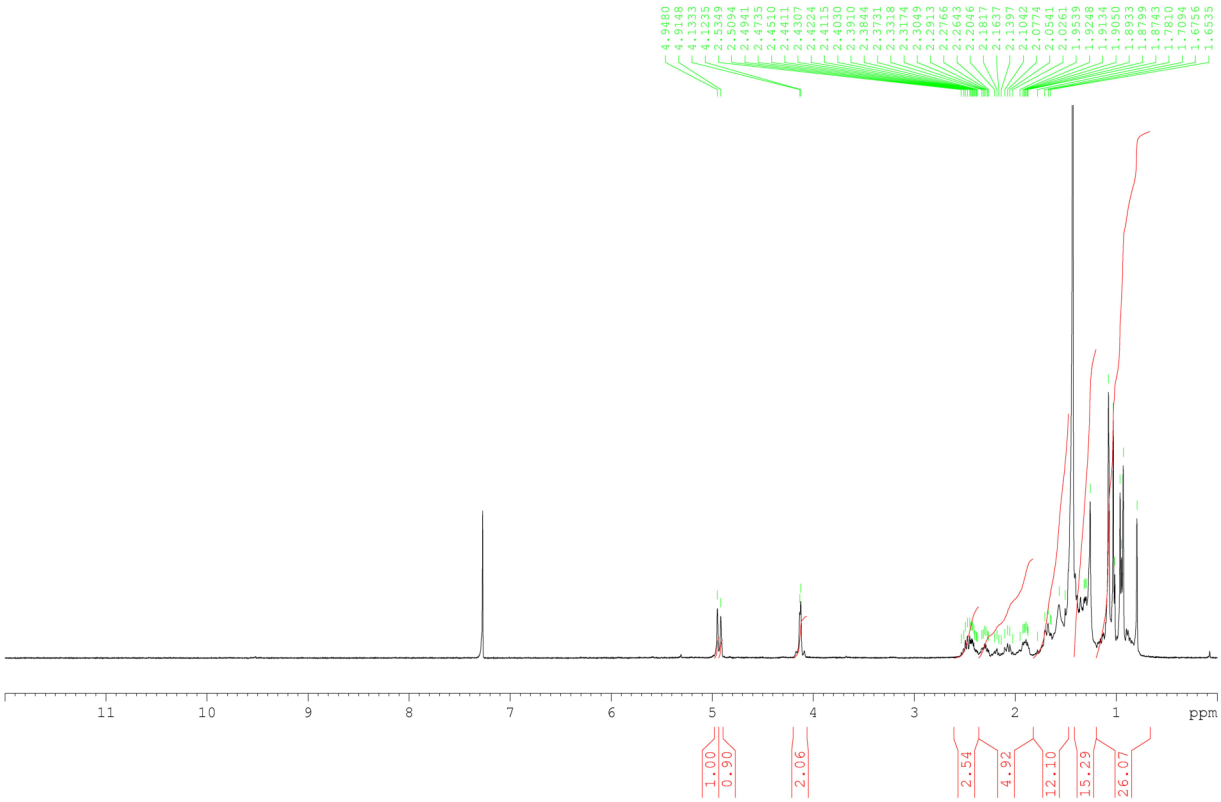

4.6940
4.5931
3.8257
3.7980
3.3667
3.3398
3.3337
2.5157
2.5001
2.4812
2.4573
2.4392
2.4280
2.4211
2.4089
2.3939
2.0018
1.9808
1.9719
1.9556
1.9402
1.9272
1.9078
1.8925
1.8825
1.8655
1.8398
1.6904
1.6561
1.6393
1.6027
1.5739
1.4512
1.4314
1.4088
1.3797
11
10
9
8
7
6
5
4
3
2
1
ppm
1.03
1.09
1.00
1.12
3.15
5.85
10.94
19.41
28.77

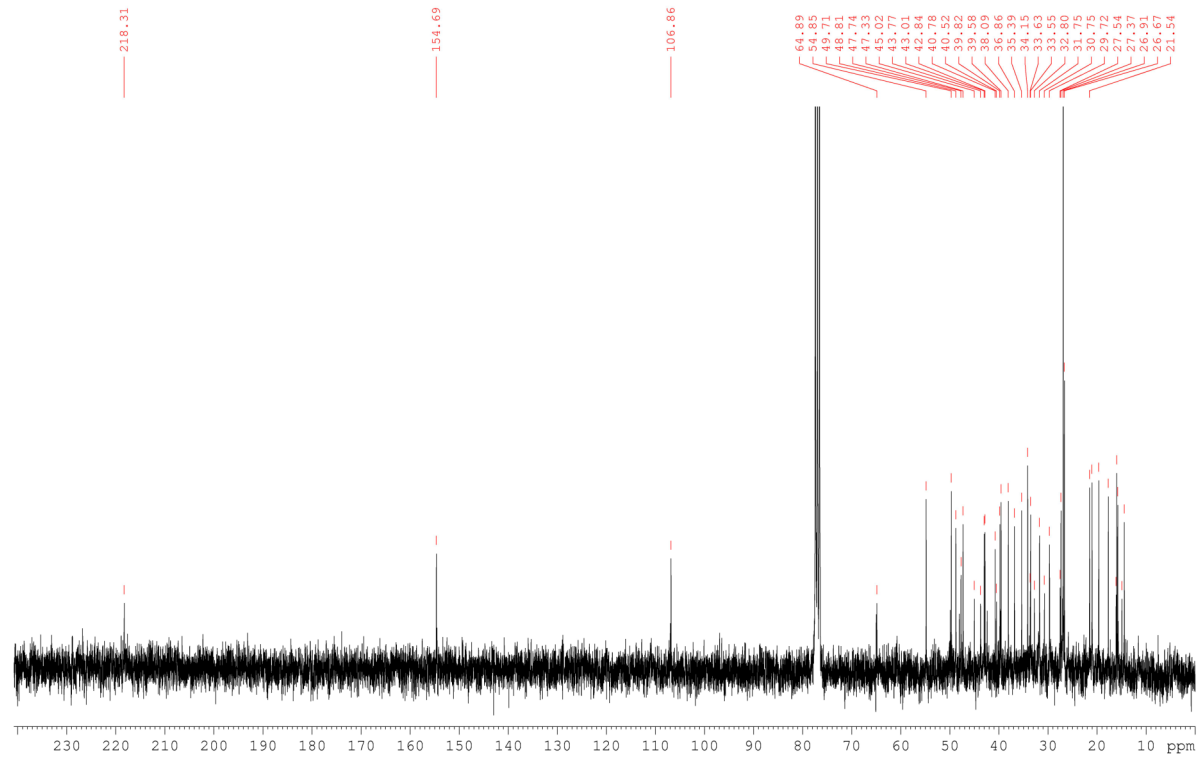

218.30
178.48
150.40
109.79
60.51
54.88
49.72
48.66
47.76
47.36
42.76
40.84
39.59
37.39
36.86
34.15
33.95
33.80
33.49
31.94
29.70
29.61
29.45
29.38
29.26
29.08
27.00
26.91
26.62
25.18
24.71
22.71
21.34
21.04
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

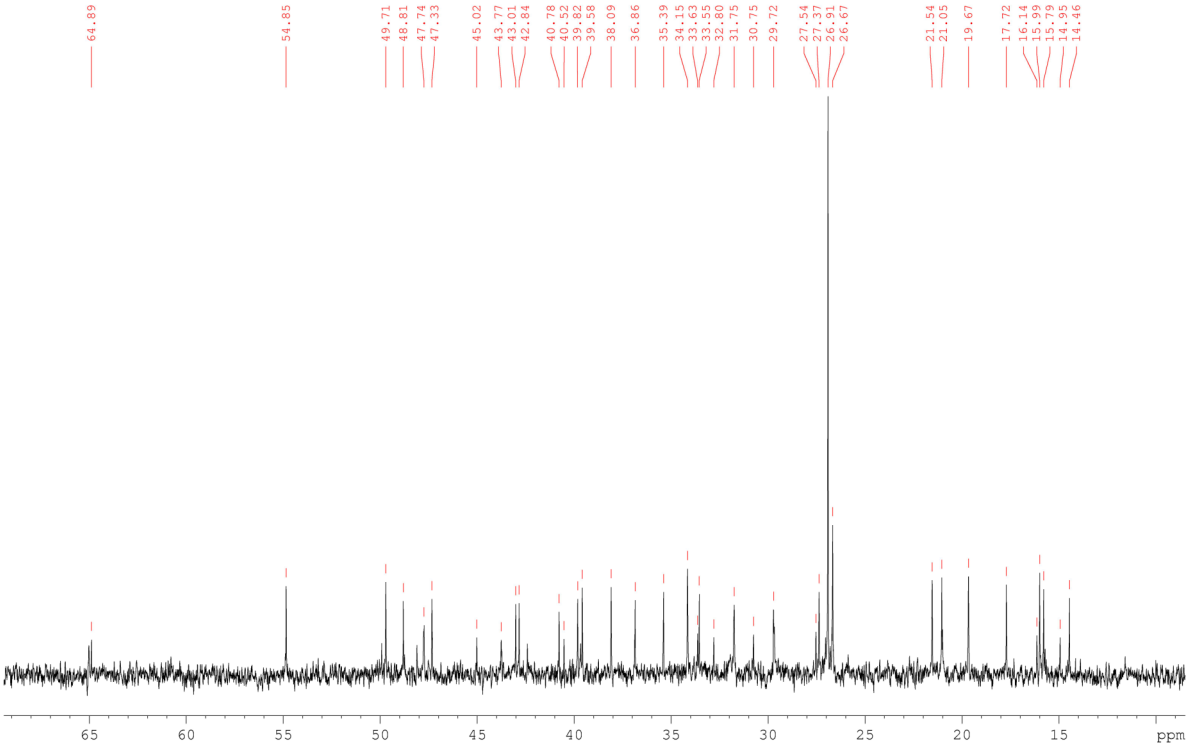


- Mass (ESI+: *m/z* 463 $[M+Na]^+$) and NMR (^1H+^{13}C) spectra of 30-hydroxy-3-lup-20(29)-ene ($C_{30}H_{48}O_2$; MW 440) (5):

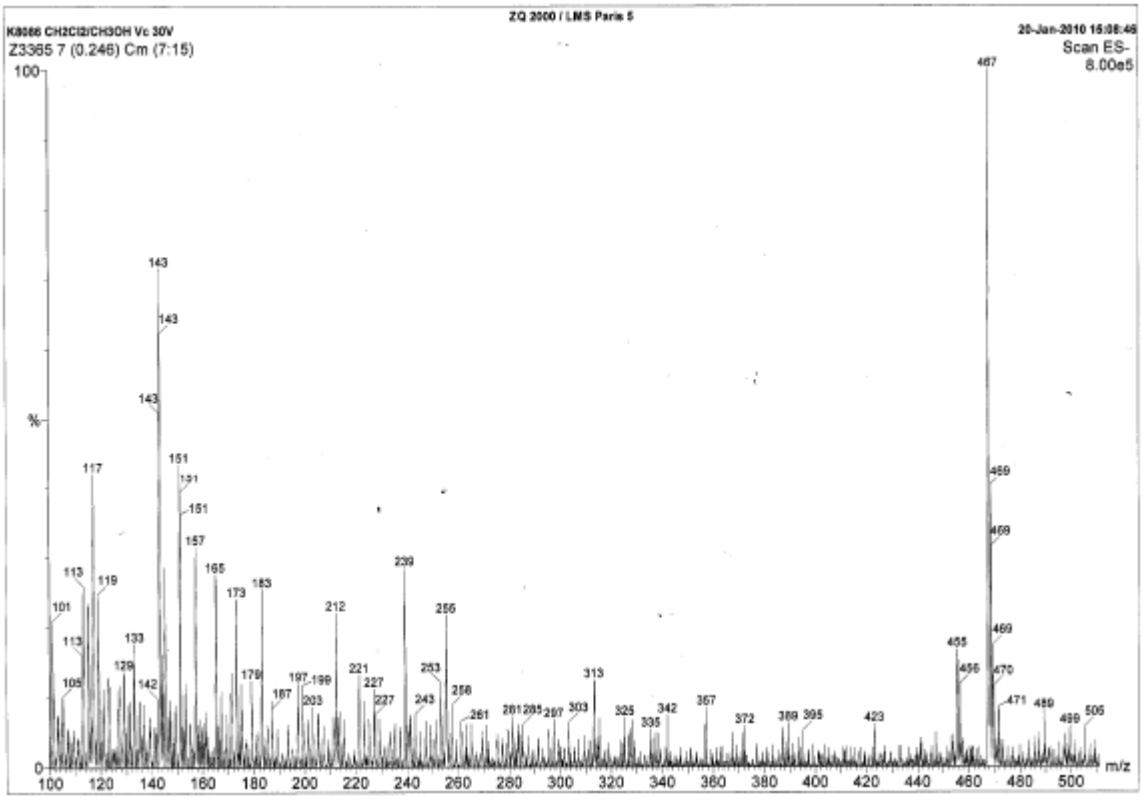


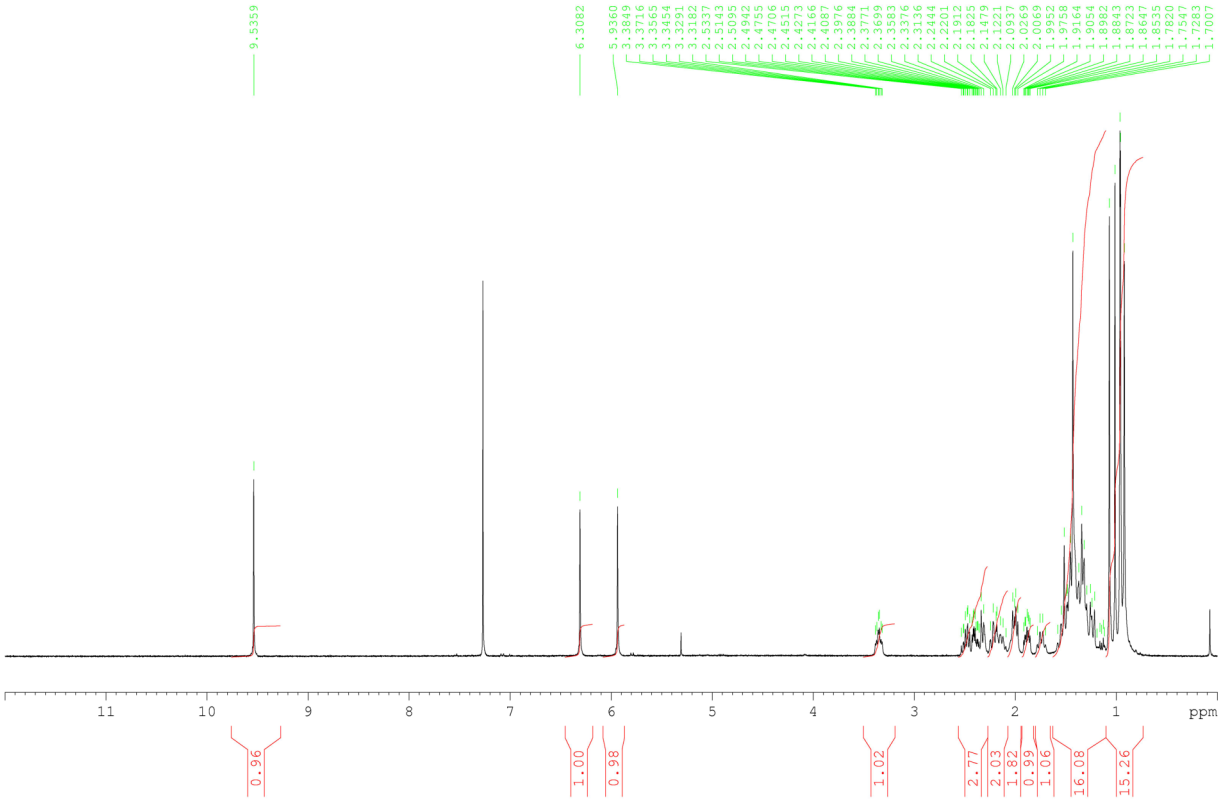

4.9480
4.9148
4.1333
4.1235
2.5349
2.5094
2.4941
2.4735
2.4510
2.4411
2.4307
2.4224
2.4115
2.4030
2.3900
2.3844
2.3731
2.3318
2.3174
2.3049
2.2913
2.2766
2.2643
2.2046
2.1817
2.1637
2.1397
2.1042
2.0774
2.0541
2.0261
1.9539
1.9298
1.9234
1.9050
1.8933
1.8799
1.8743
1.7810
1.7094
1.6756
1.6535
11 10 9 8 7 6 5 4 3 2 1 ppm
1.00
0.90
2.06
2.54
4.92
12.10
15.29
26.07

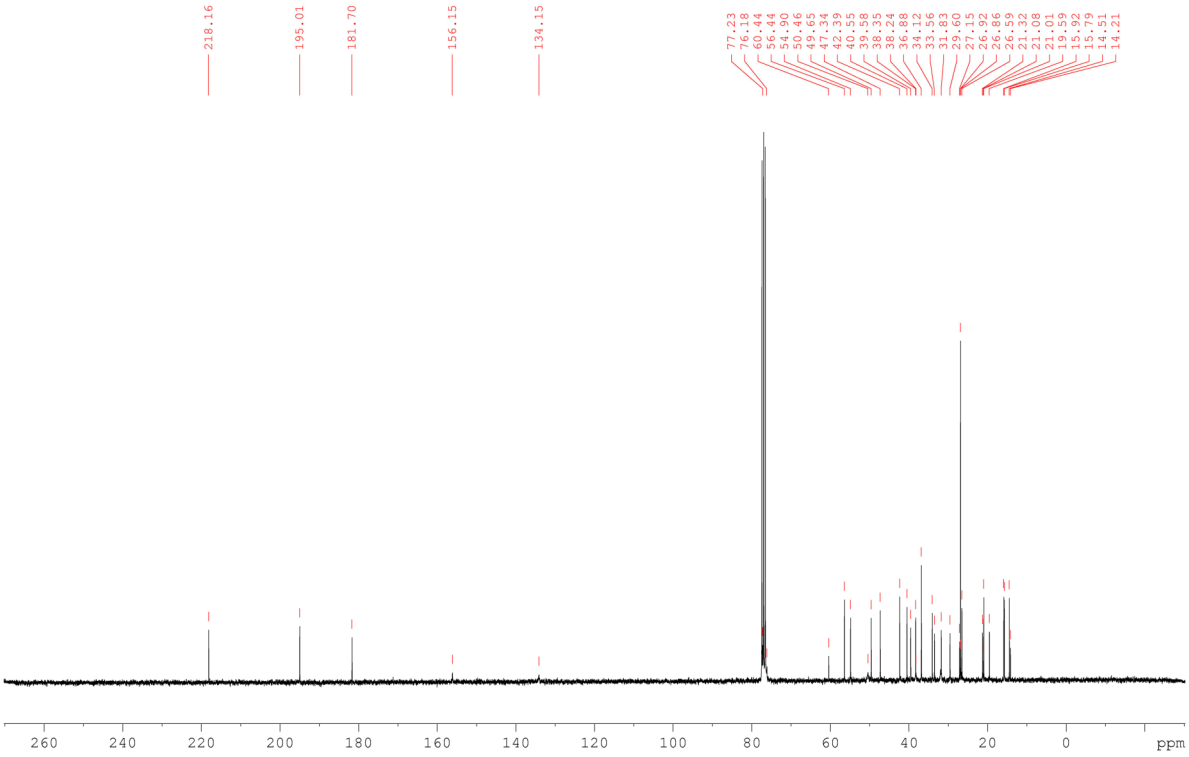

218.31
154.69
106.86
64.89
54.85
49.71
48.81
47.74
47.33
45.02
43.77
43.01
42.84
40.78
40.52
39.82
39.58
38.09
36.86
35.39
34.15
33.63
33.55
32.80
31.75
30.75
29.72
27.54
27.37
26.91
26.67
21.54
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

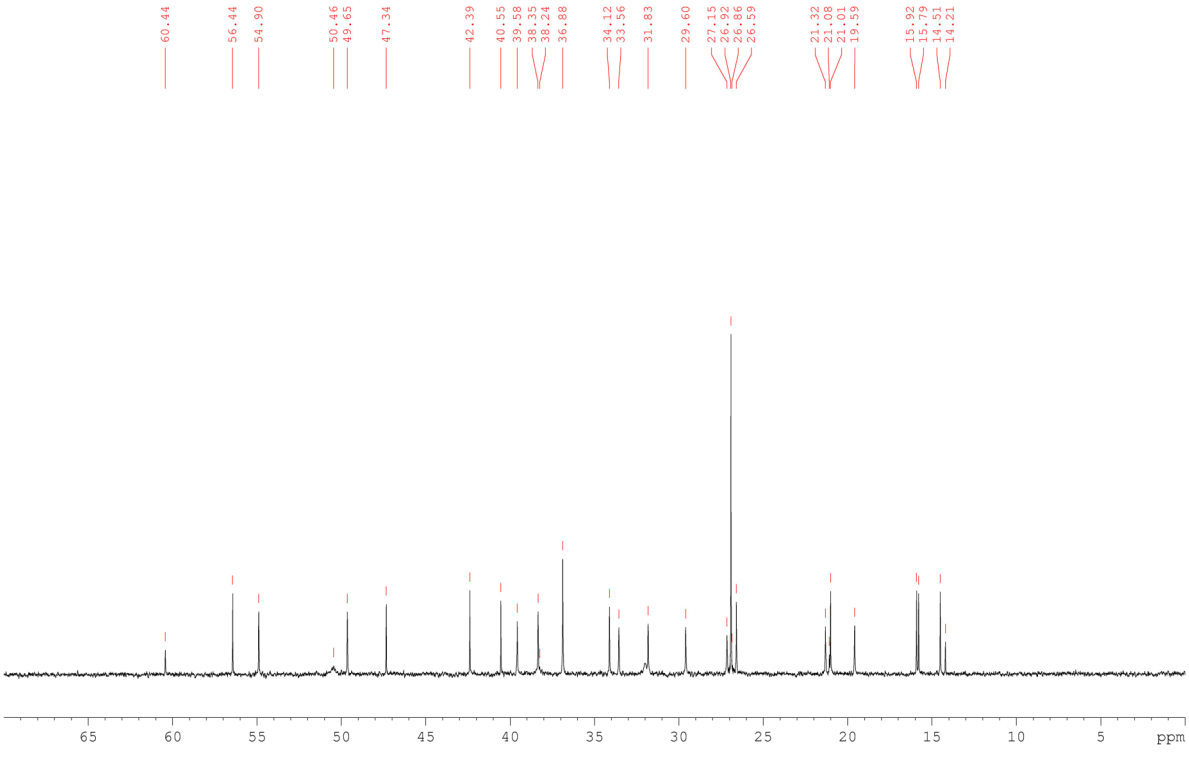

64.89
54.85
49.71
48.81
47.74
47.33
45.02
43.77
43.01
42.84
40.78
40.52
39.82
39.58
38.09
36.86
35.39
34.15
33.63
33.55
32.80
31.75
30.75
29.72
27.54
27.37
26.91
26.67
21.54
21.05
19.67
17.72
16.14
15.79
14.95
14.46
65
60
55
50
45
40
35
30
25
20
15
ppm

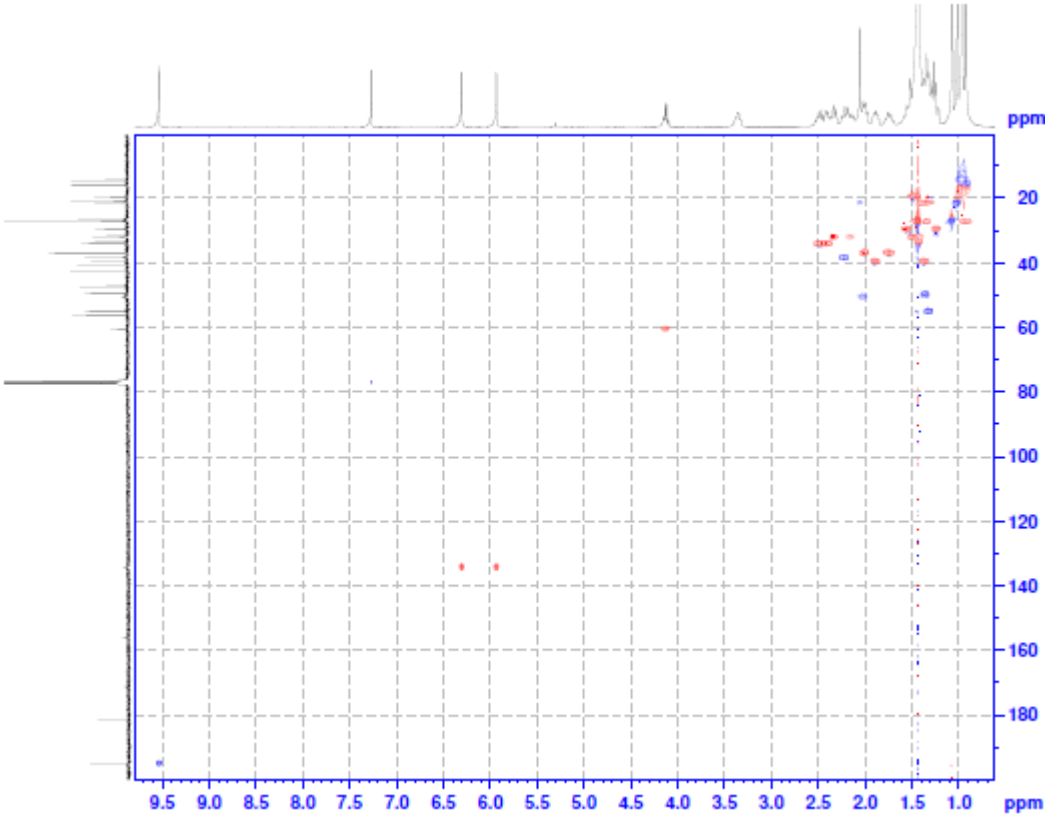
- Mass (ESI-: *m/z* 467 [M-H]$^-$) and NMR (^{1}H + ^{13}C + HSQC edit + HMBC + NOESY) spectra of 3,30-dioxolup-20(29)-en-28-oic acid ($C_{30}H_{44}O_4$; MW 468) (6):

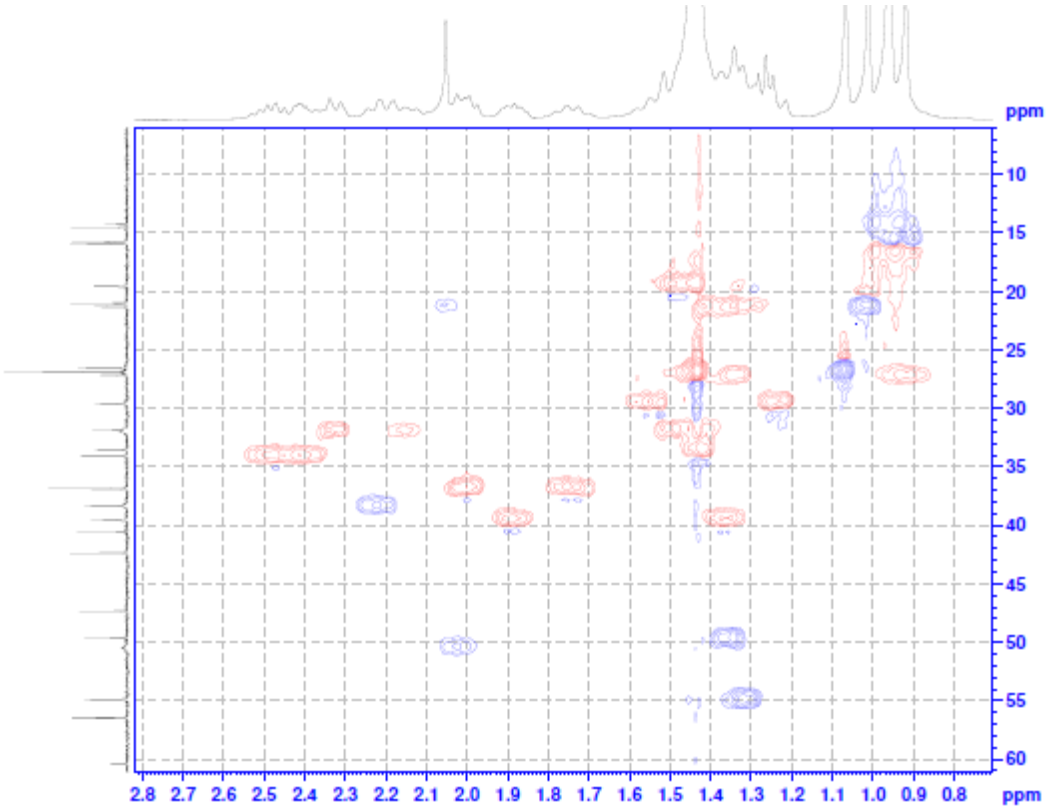


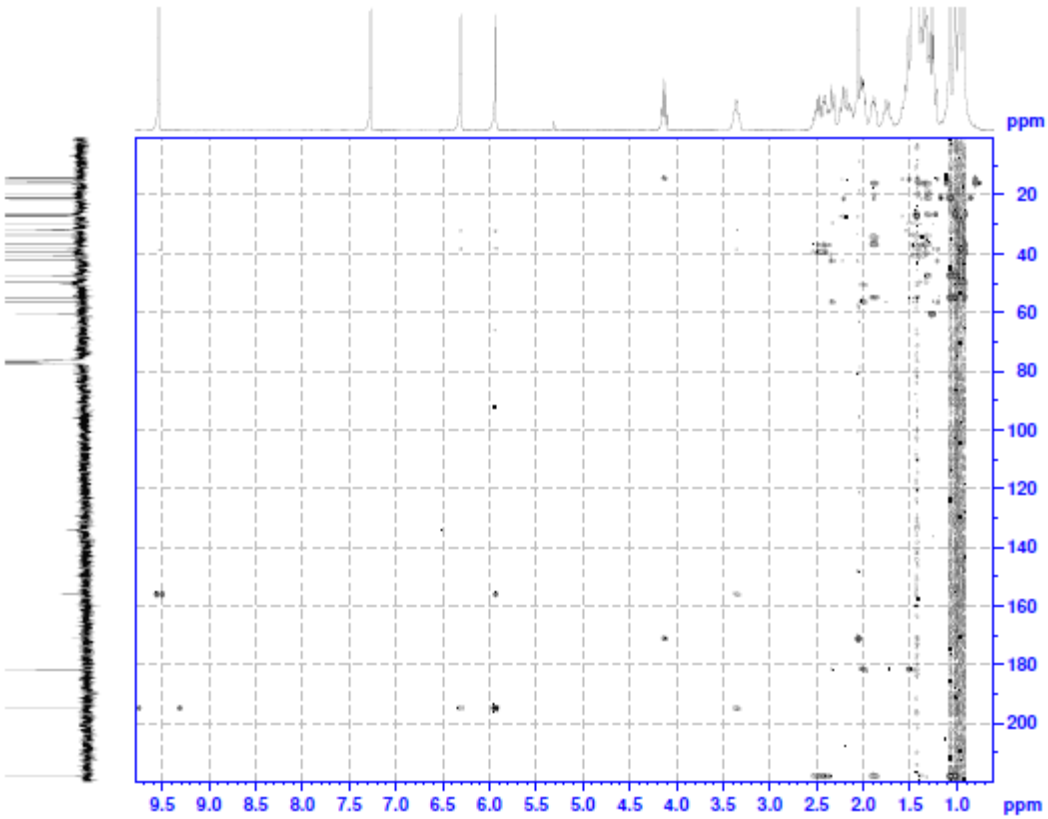


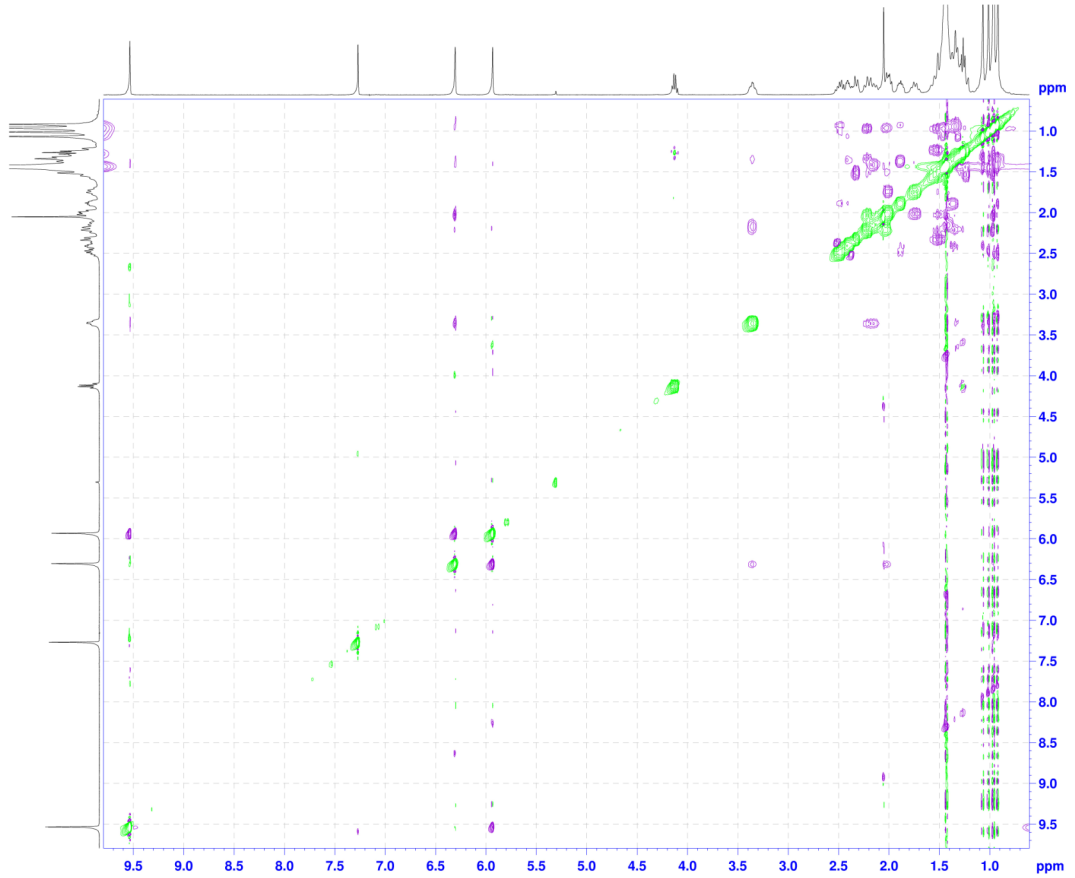

218.16
195.01
181.70
156.15
134.15
77.23
76.18
60.44
56.44
54.90
50.46
49.65
47.34
42.39
40.55
39.58
38.35
38.24
36.88
34.12
33.56
31.83
29.60
27.15
26.92
26.86
26.59
21.32
21.08
21.01
19.59
15.92
15.79
14.51
14.21
260
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm


60.44
56.44
54.90
50.46
49.65
47.34
42.39
40.55
39.58
38.35
38.24
36.88
34.12
33.56
31.83
29.60
27.15
26.92
26.86
26.59
21.32
21.08
21.01
19.59
15.92
15.79
14.51
14.21
65
60
55
50
45
40
35
30
25
20
15
10
5
ppm

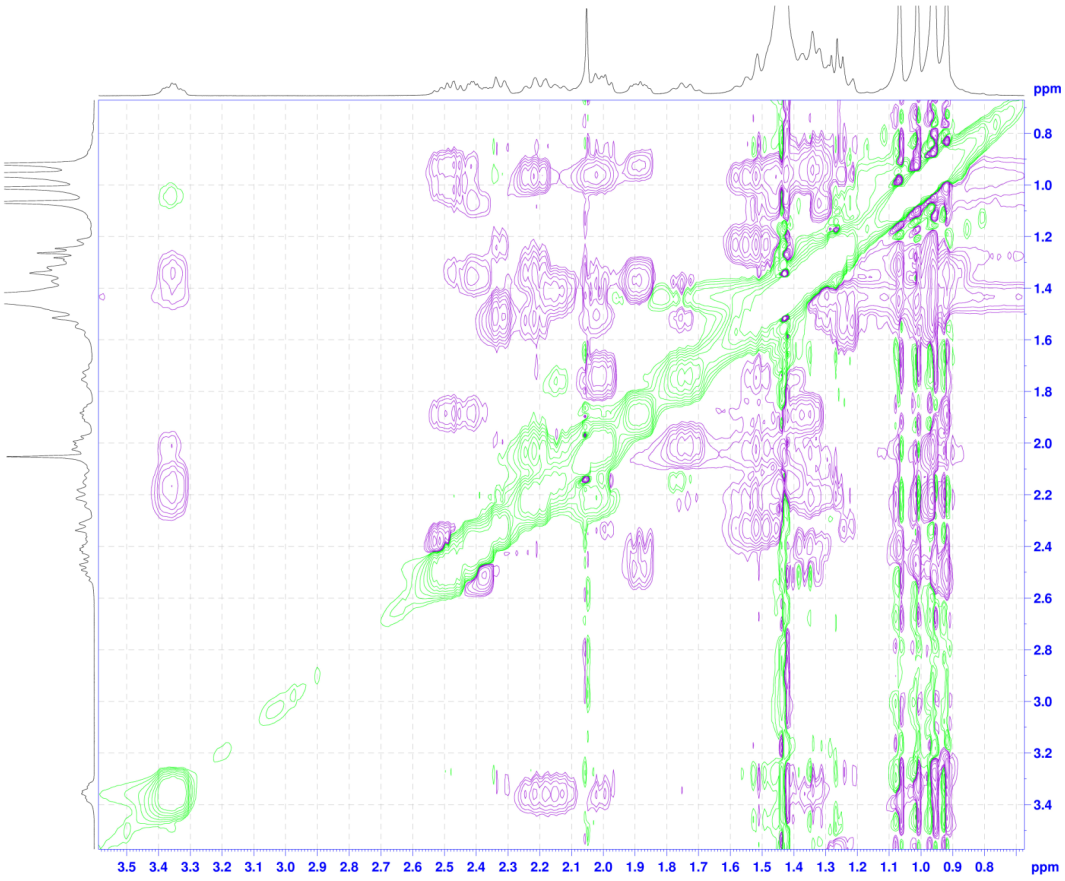

ppm
20
40
60
80
100
120
140
160
180
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
ppm


ppm
10
15
20
25
30
35
40
45
50
55
60
2.8 2.7 2.6 2.5 2.4 2.3 2.2 2.1 2.0 1.9 1.8 1.7 1.6 1.5 1.4 1.3 1.2 1.1 1.0 0.9 0.8
ppm

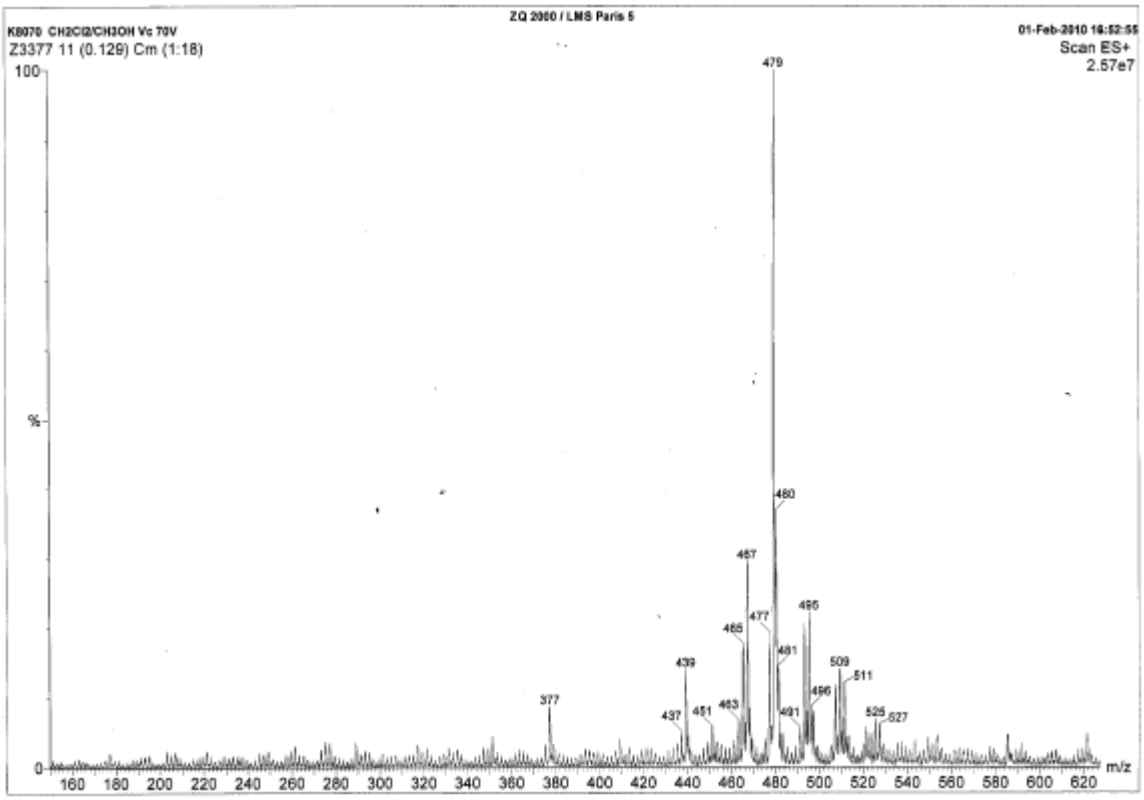


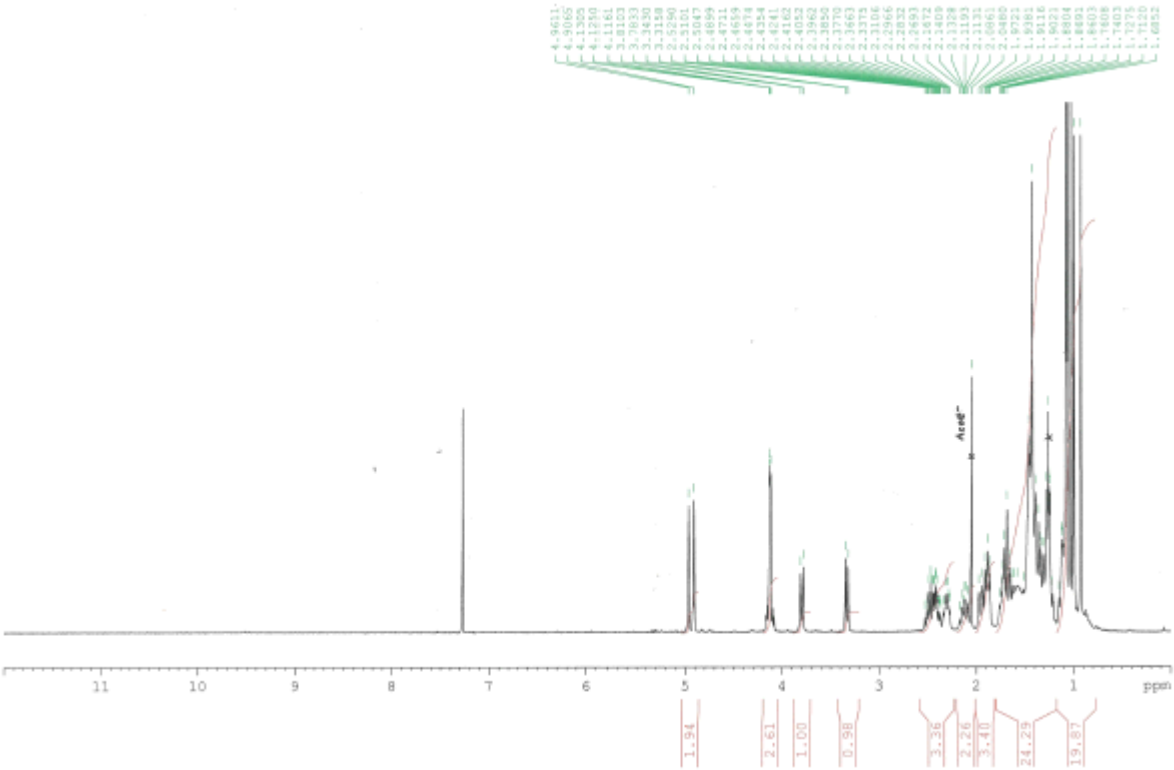


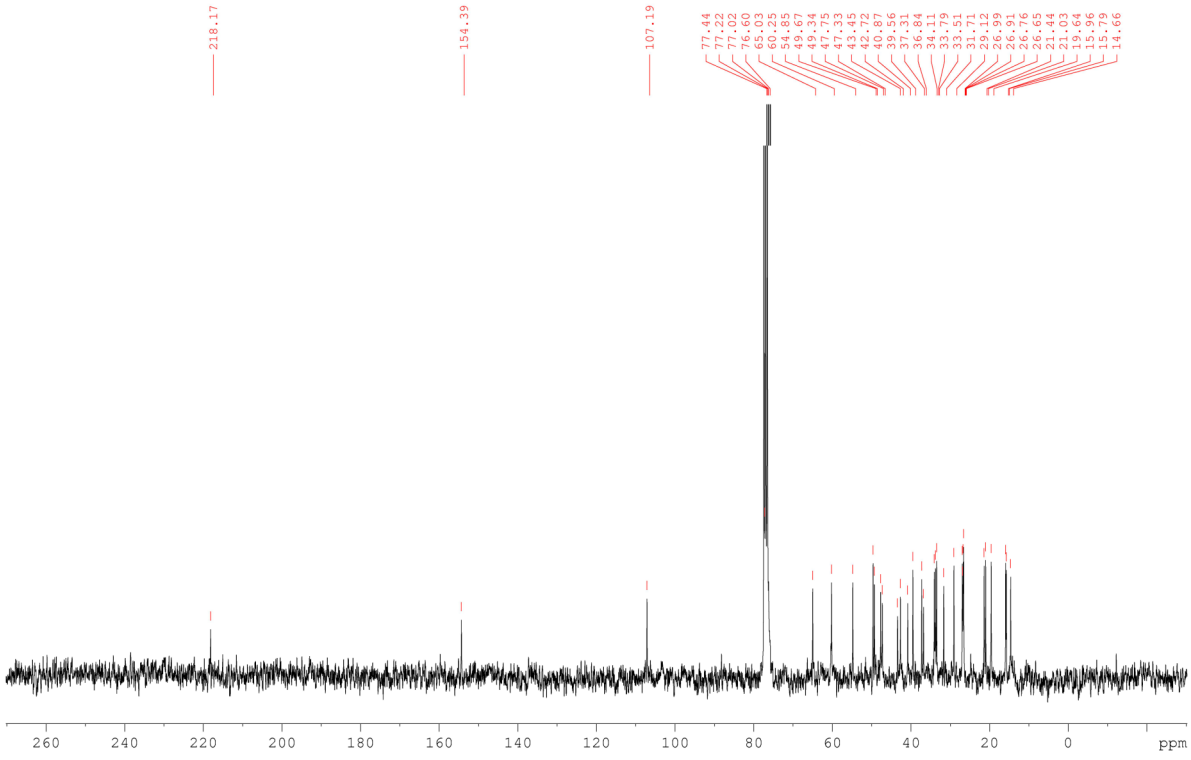

```
Current Data Parameters
NAME      1-xc-malai-ho-ald-02
EXPNO                        4
PROCNO                       1

F2 - Acquisition Parameters
Date_              20100126
Time                  17.39
INSTRUM                spect
PROBHD    5 mm Multinucl
PULPROG             noesyph
TD                     2048
SOLVENT                CDCl3
NS                       64
DS                       16
SWH                3676.471 Hz
FIDRES             1.795152 Hz
AQ                 0.2785780 sec
RG                       64
DW                  136.000 usec
DE                    7.00 usec
TE                  295.2 K
d0                 0.00012862 sec
D1                 2.00000000 sec
D8                 0.69999999 sec
IN0                0.00027200 sec
ST1CNT                  128

======== CHANNEL f1 ========
NUC1                     1H
P1                     5.80 usec
PL1                   -3.00 dB
SFO1            400.1320965 MHz

F1 - Acquisition parameters
ND0                      1
TD                     256
SFO1             400.1321 MHz
FIDRES          14.361214 Hz
SW                   9.188 ppm
FnMODE         States-TPPI

F2 - Processing parameters
SI                    1024
SF              400.1300152 MHz
WDW                   QSINE
SSB                       2
LB                     0.00 Hz
GB                        0
PC                     1.00

F1 - Processing parameters
SI                    1024
MC2            States-TPPI
SF              400.1300149 MHz
WDW                   QSINE
SSB                       2
LB                     0.00 Hz
GB                        0
```

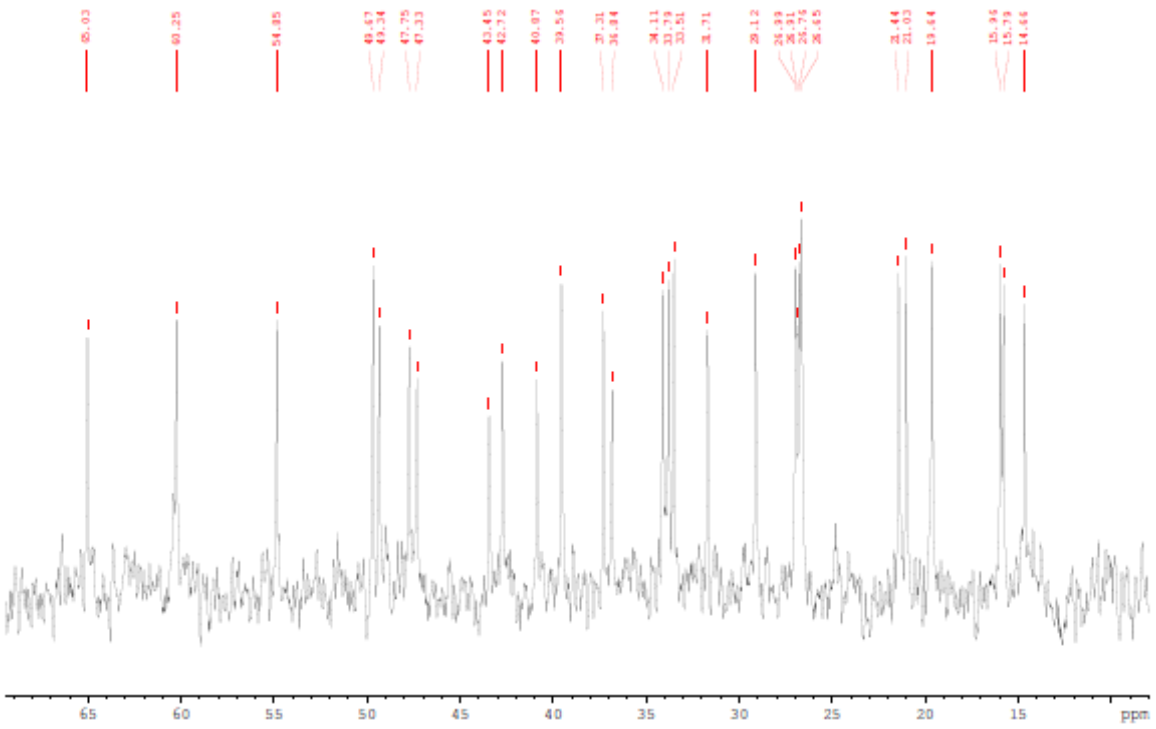


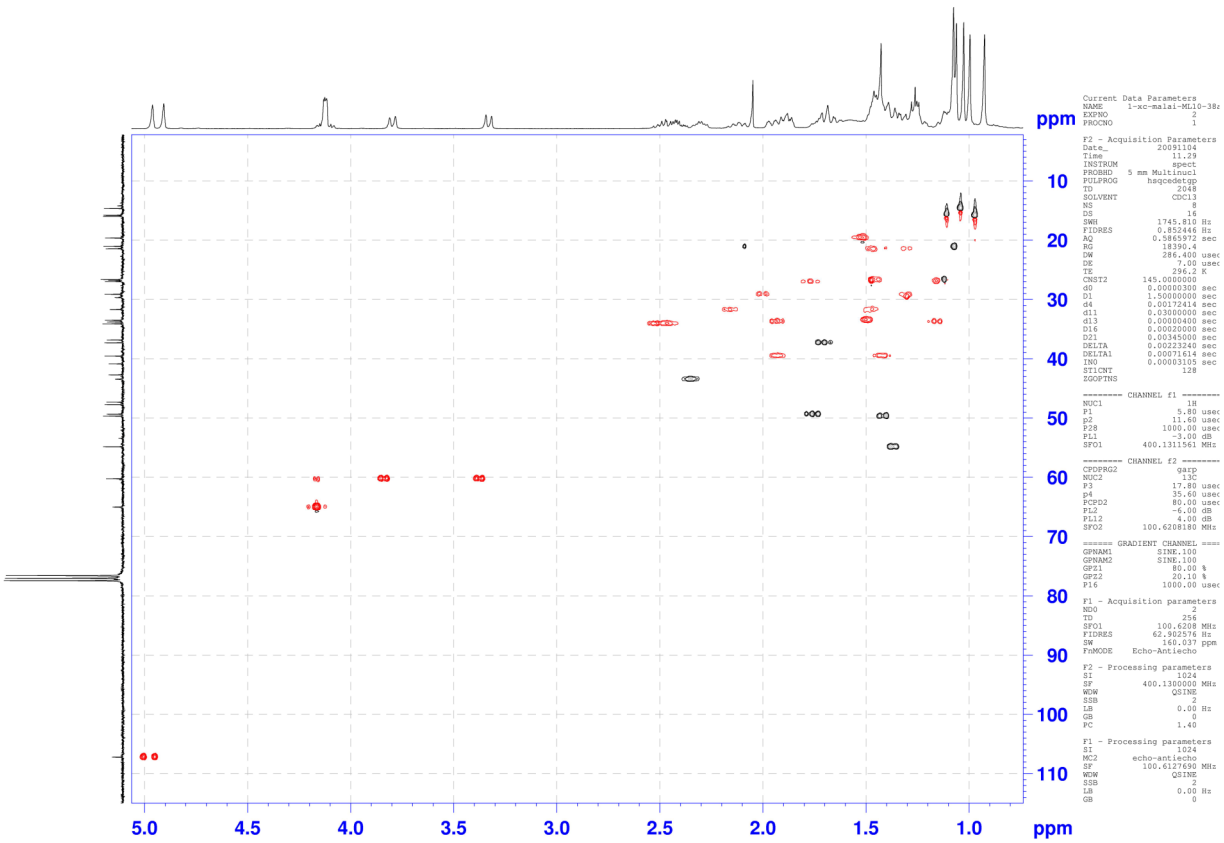
- Mass (ESI+: *m/z* 479 $[M+Na]^+$) and NMR (^1H + ^{13}C + HSQC edit + NOESY) spectra of 28,30-dihydroxy-3-oxolup-20(29)-ene ($C_{30}H_{48}O_3$; MW 456) (7):

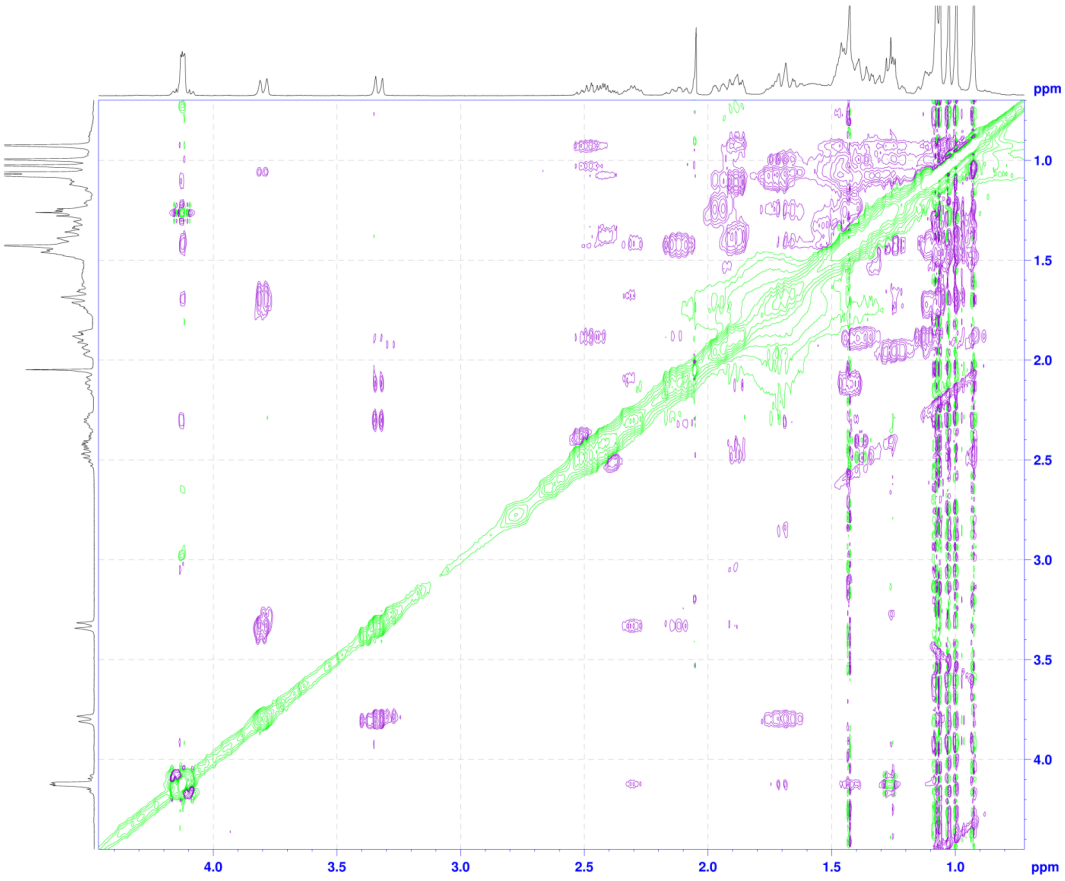



11
10
9
8
7
6
5
4
3
2
1
ppm
1.94
2.61
1.00
0.98
3.36
2.26
3.40
24.29
19.87

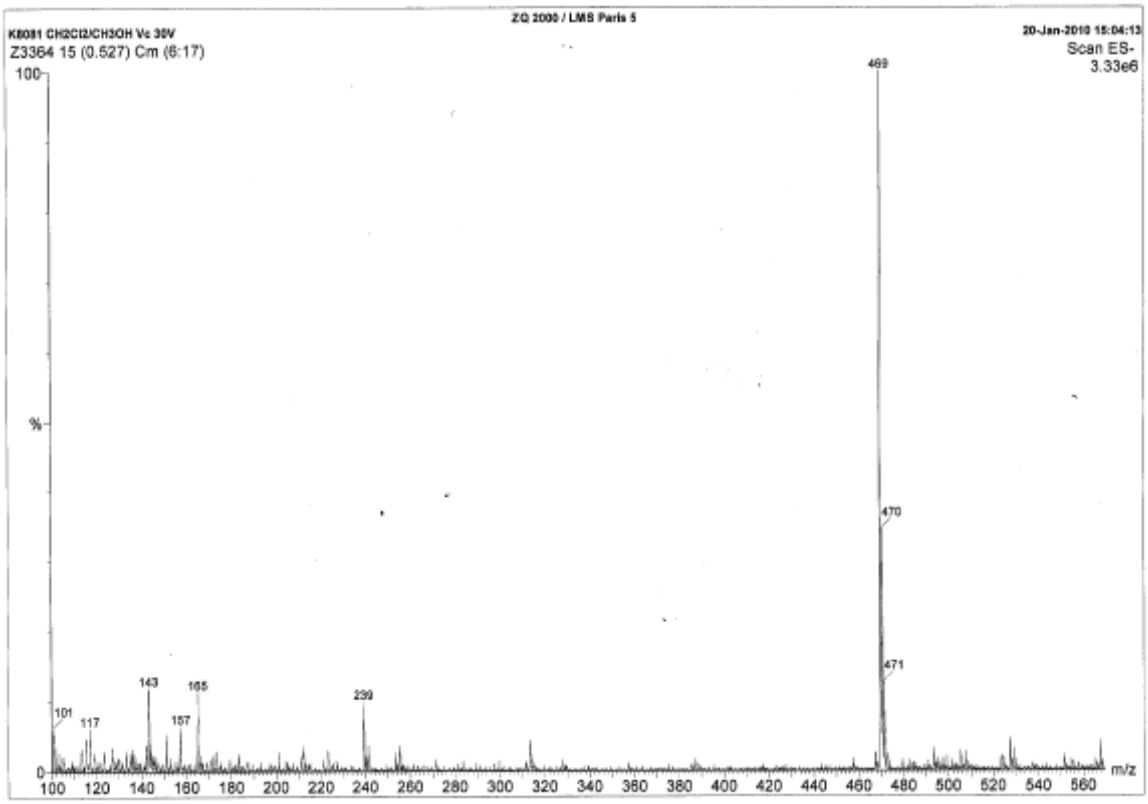

218.17
154.39
107.19
260
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm

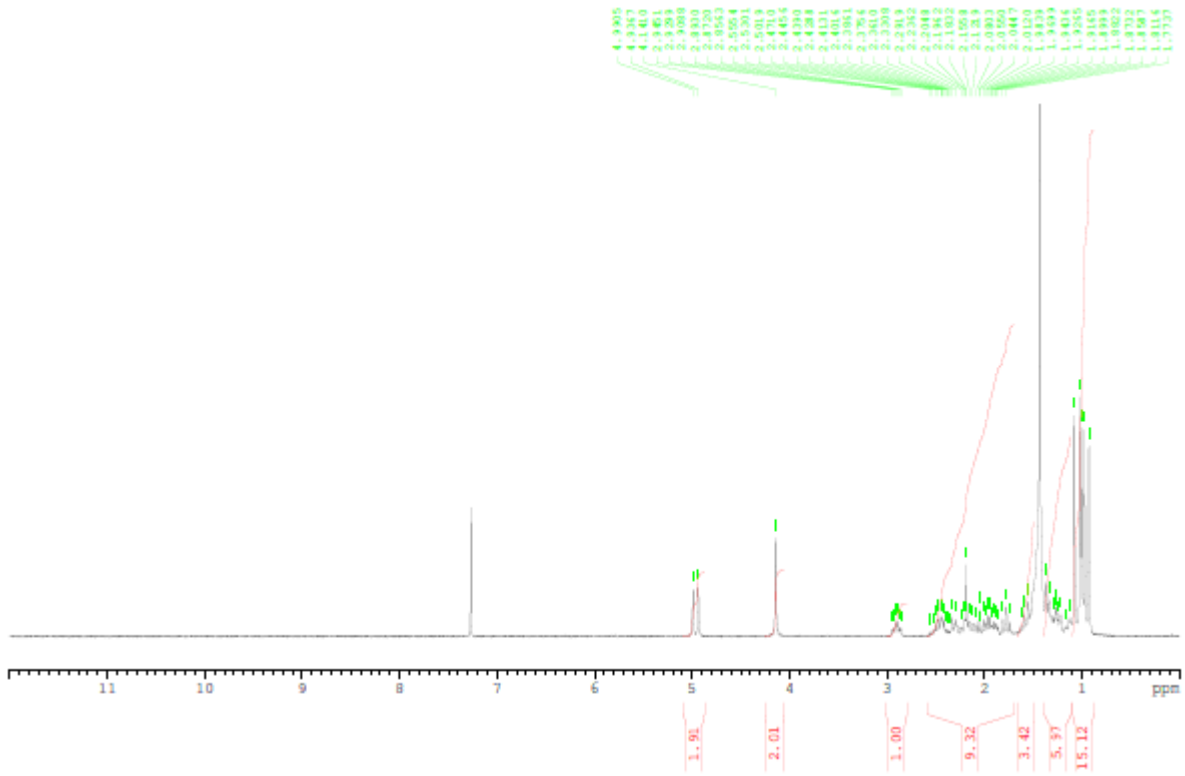

65.03
60.25
54.85
49.67
49.34
47.75
47.33
43.45
42.72
40.87
39.56
37.31
36.84
34.11
33.79
33.51
31.71
29.12
28.89
28.51
28.26
28.05
21.44
21.03
19.64
15.96
15.79
14.66
65
60
55
50
45
40
35
30
25
20
15
ppm

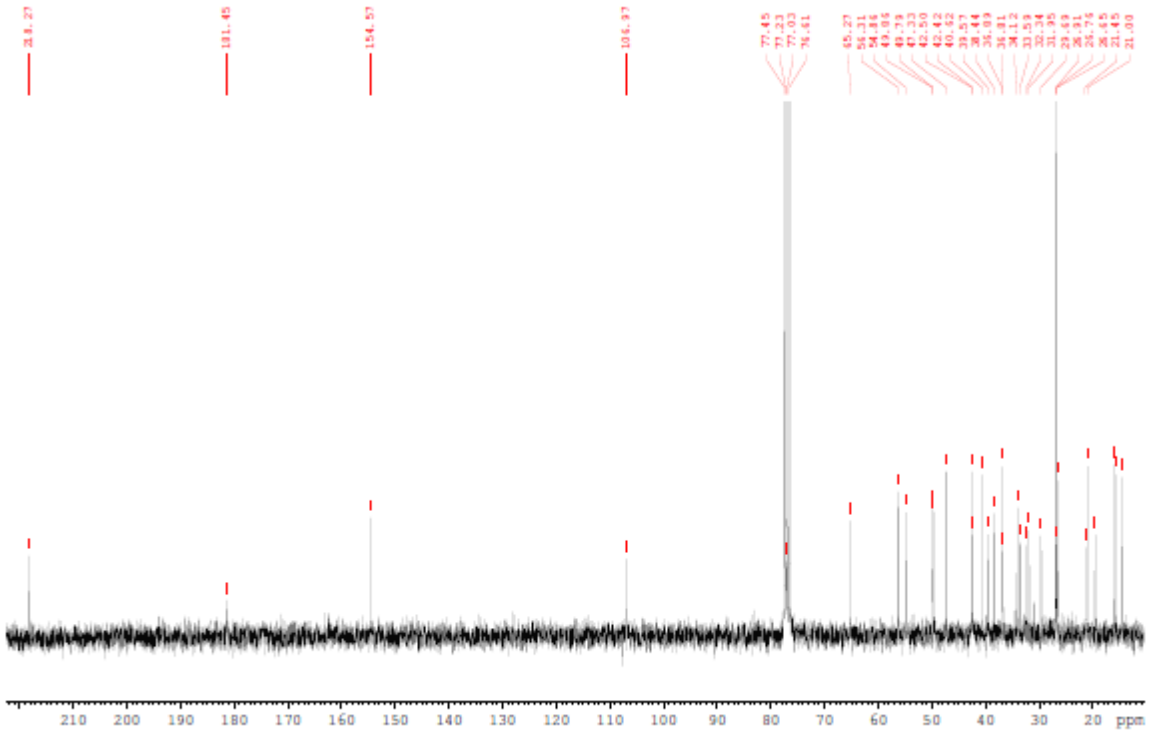

ppm
10
20
30
40
50
60
70
80
90
100
110
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

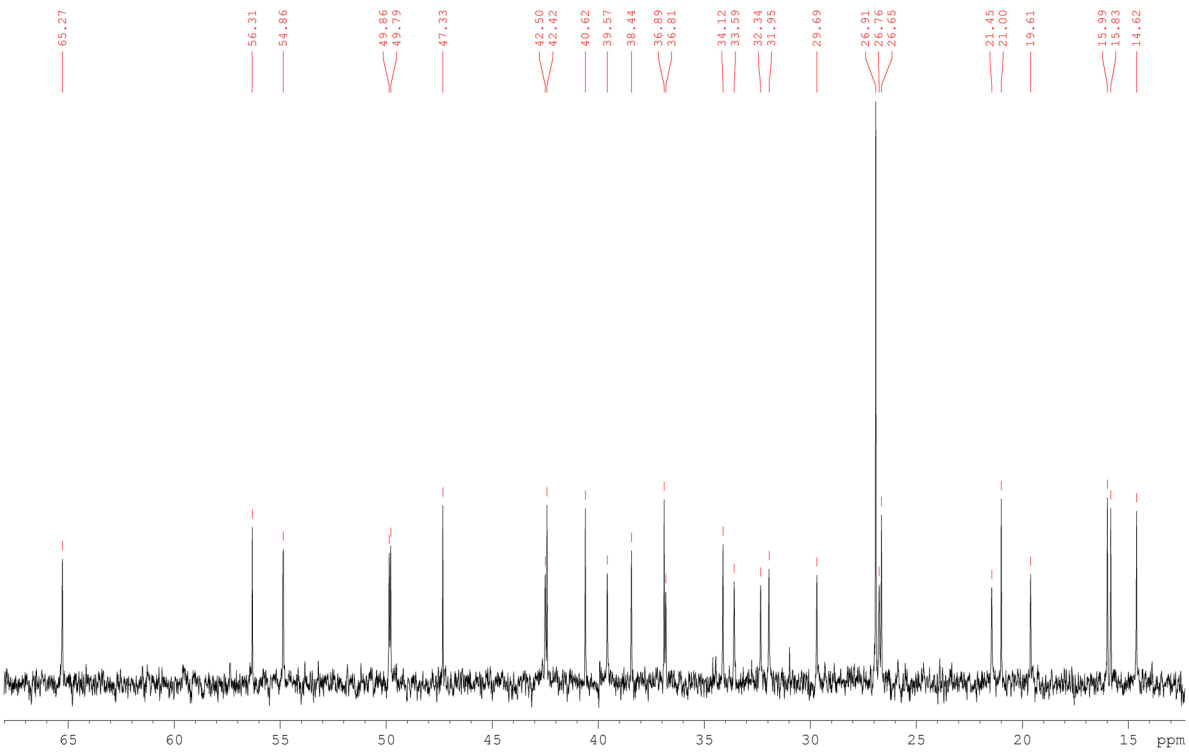


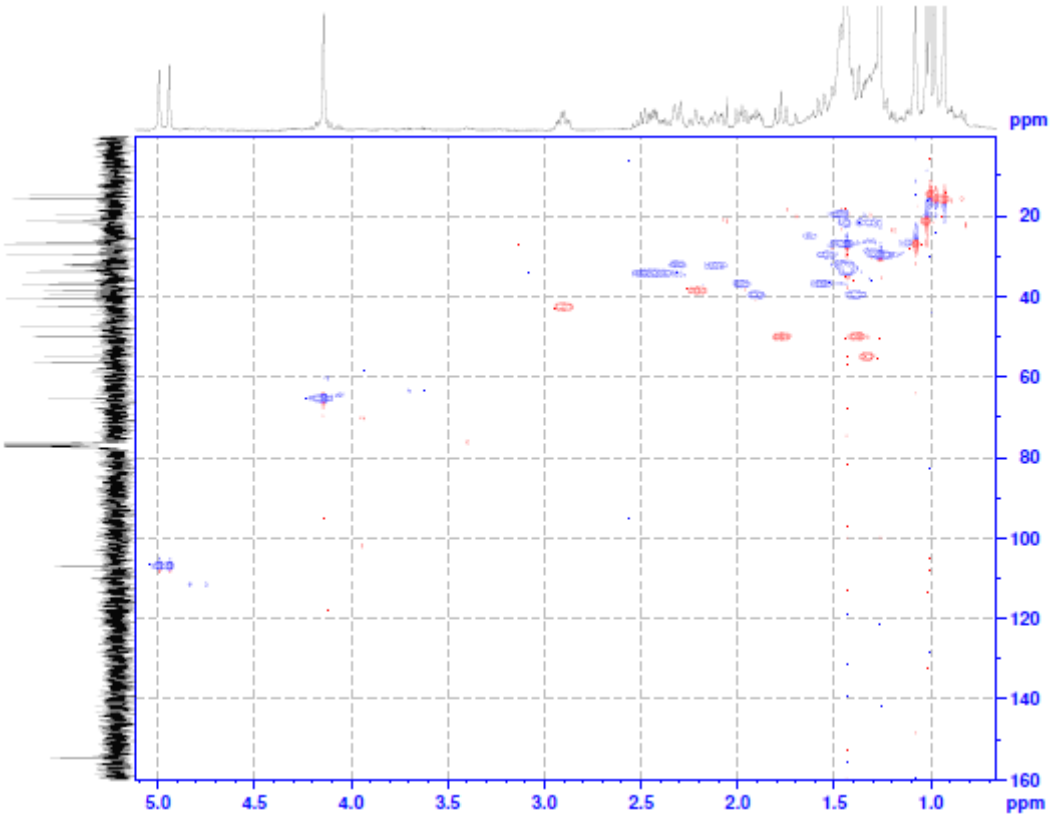
- Mass (ESI-: *m/z* 469 $[M-H]^-$) and NMR (^1H + ^{13}C + HSQC edit) spectra of Messagenic acid G ($C_{30}H_{46}O_4$; MW 470) (8):



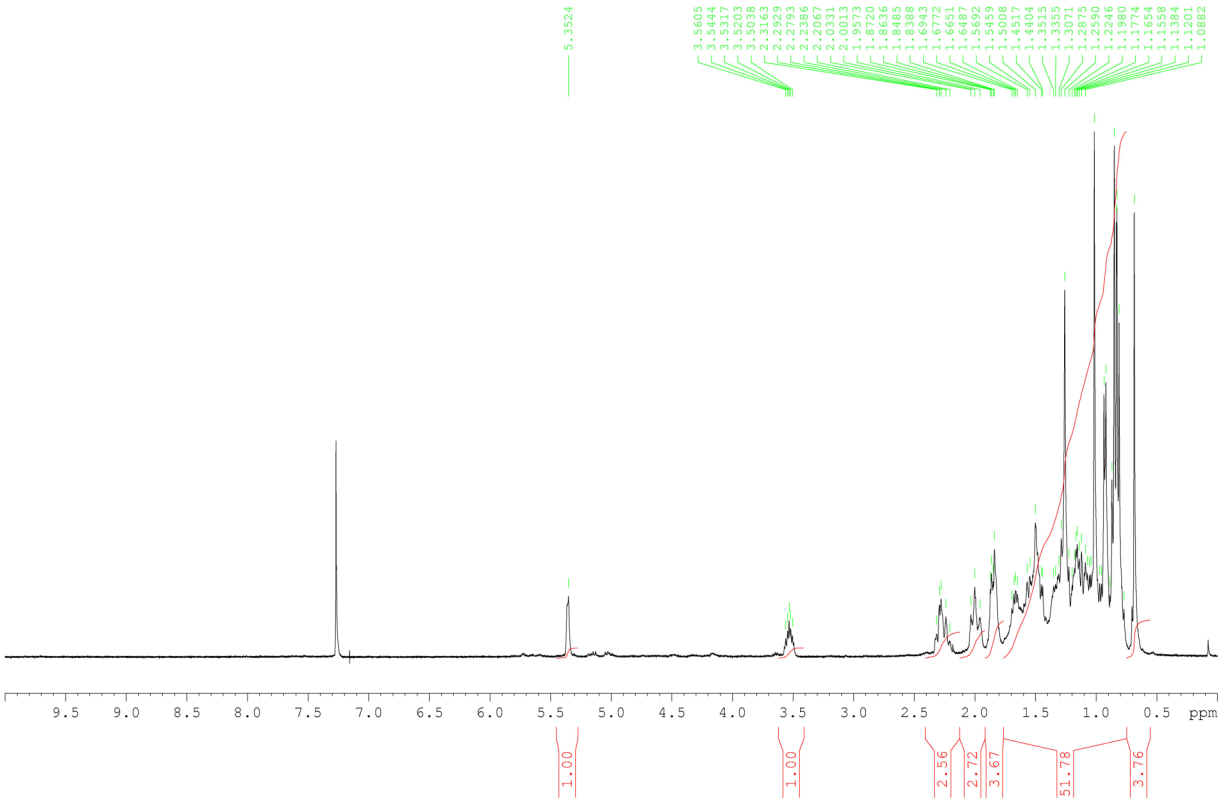

11
10
9
8
7
6
5
4
3
2
1
ppm
1.91
2.01
1.00
9.32
3.42
5.97
15.12

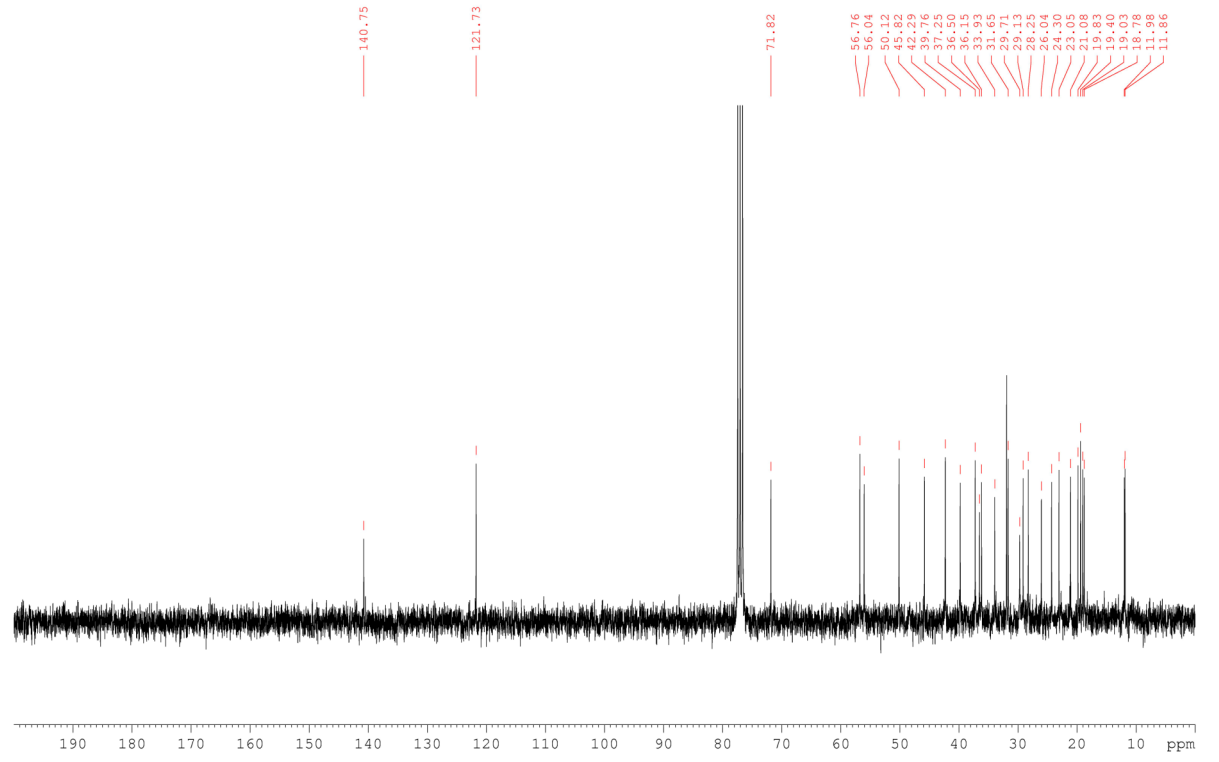

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

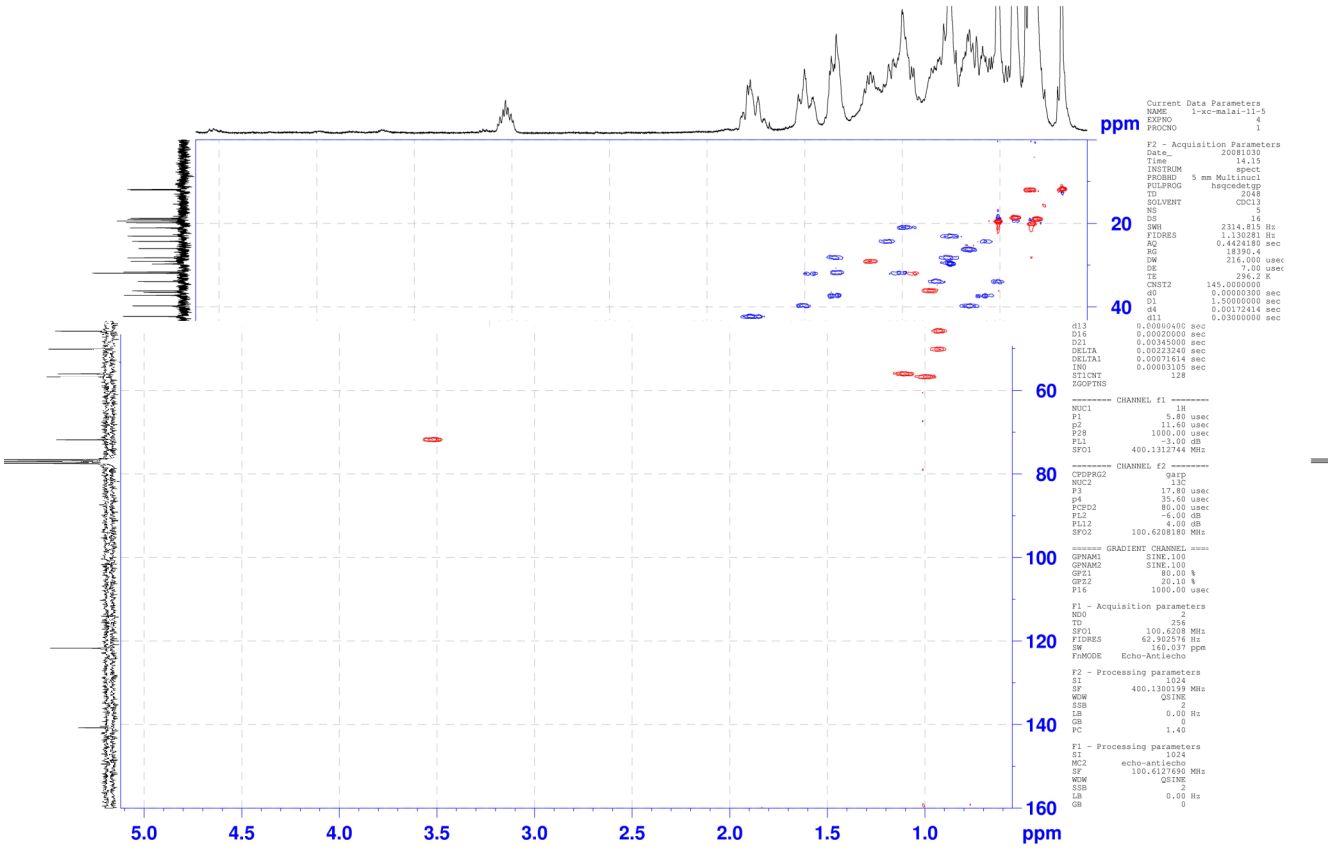




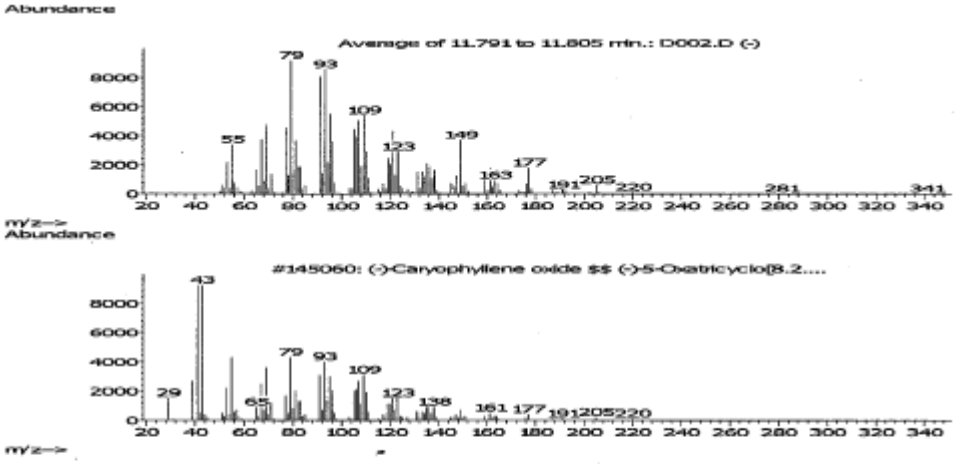
- Mass (EI: *m/z* 414 $[M]^+$) and NMR (^1H+^{13}C+HSQC edit) spectra of β-sitosterol ($C_{29}H_{50}O$) (9):


5.3524
3.5605
3.5444
3.5277
3.5203
3.5038
2.3163
2.2929
2.2793
2.2386
2.2067
2.0331
2.0013
1.9573
1.8720
1.8636
1.8485
1.8388
1.6943
1.6772
1.6631
1.6487
1.6322
1.5459
1.5008
1.4517
1.4404
1.3515
1.3355
1.3071
1.2875
1.2590
1.2246
1.1980
1.1774
1.1654
1.1558
1.1384
1.1201
1.0882
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
1.00
2.56
2.72
3.67
51.78
3.76

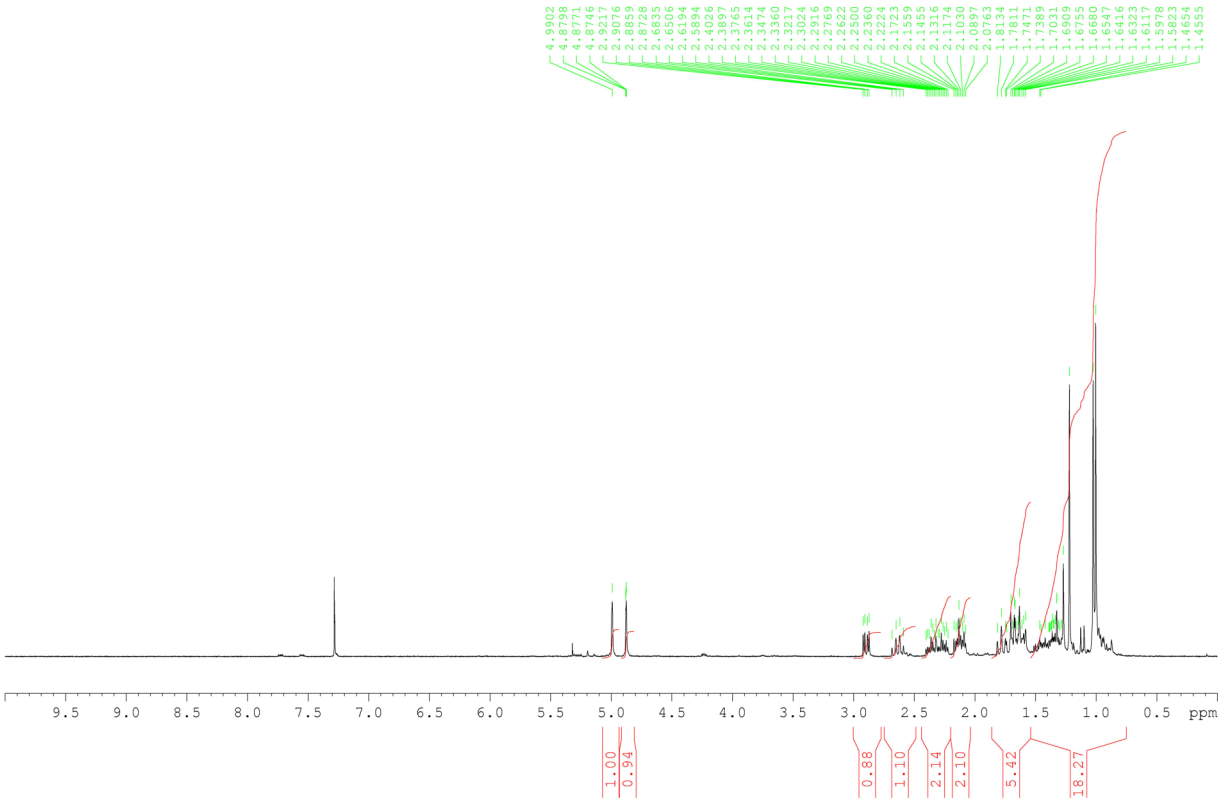

140.75
121.73
71.82
56.76
56.04
50.12
45.82
42.29
39.76
37.25
36.50
36.15
33.93
31.65
31.31
29.13
28.25
26.04
24.30
23.05
21.08
19.83
19.40
19.03
18.78
11.98
11.86
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

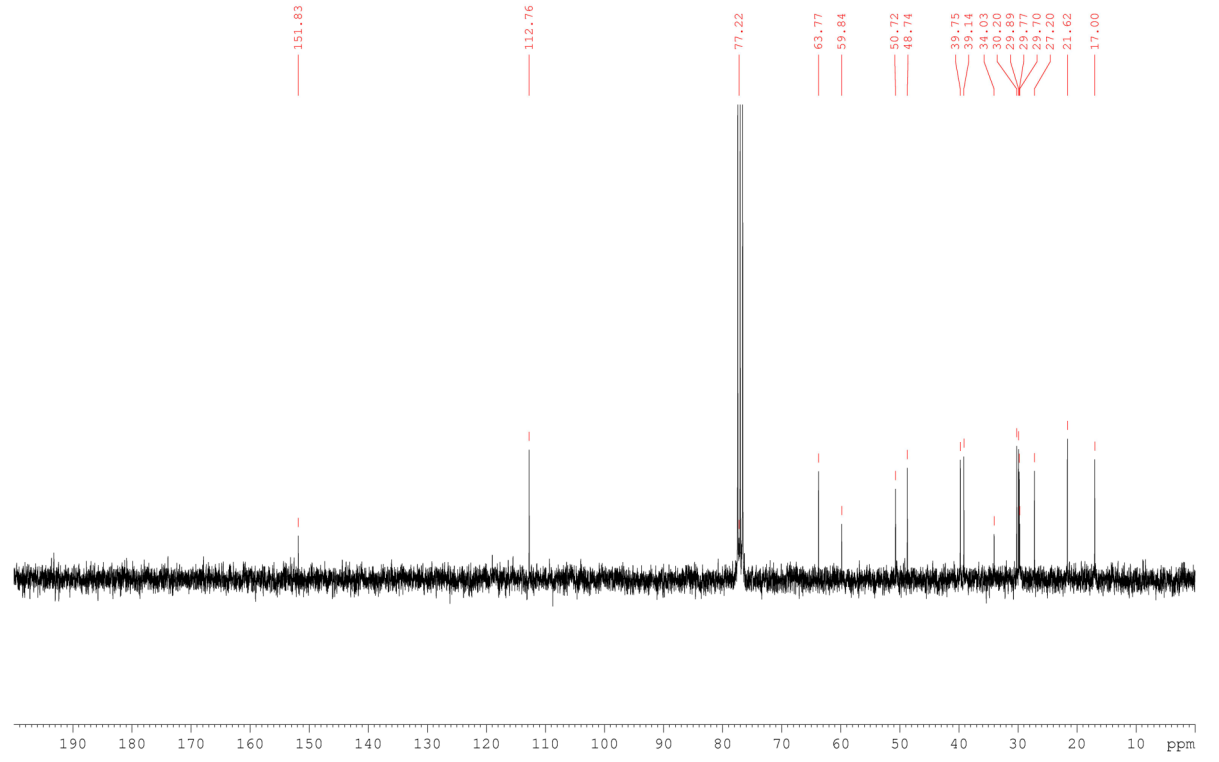

ppm
20
40
60
80
100
120
140
160
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

- Mass (EI: *m/z* 220 $[M]^+$) and NMR ($^1H+^{13}C$) spectra of β-caryophyllene oxide ($C_{15}H_{24}O$; MW 220) (10):



Match Quality 92
Name (-)-Caryophyllene oxide $$ (-)-5-Oxatricyclo[8.2.0.0(4,6)]dodecane,,12-trimethyl-9-methylene-, [1R-(1R*,4R*,6R*,10S*)]- (CAS) $$ (-)-.beta.-Caryophyllene epoxide $$ Caryophyllene oxide $$ (-)caryophyllene oxide $$ 5-Oxatricyclo[8.2.0.0(4,6)-]dodecane, 4,1
CAS Number 001139-30-6
Molecular Formula C15H24O
Molecular Weight 220.18


4.9902
4.8798
4.8771
4.8746
4.8217
2.9076
2.8859
2.8728
2.6835
2.6596
2.6194
2.5894
2.4026
2.3897
2.3765
2.3614
2.3474
2.3360
2.3217
2.3024
2.2916
2.2769
2.2622
2.2500
2.2360
2.2224
2.1723
2.1559
2.1435
2.1256
2.1174
2.1030
2.0897
2.0763
1.8134
1.7811
1.7471
1.7389
1.7031
1.6909
1.6755
1.6680
1.6547
1.6416
1.6323
1.6117
1.5978
1.5823
1.4654
1.4555
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
0.94
0.88
1.10
2.14
2.10
5.42
18.27


151.83
112.76
77.22
63.77
59.84
50.72
48.74
39.75
39.14
34.03
30.20
29.89
29.77
29.70
27.20
21.62
17.00
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm