

# Supporting Information

*Rec. Nat. Prod.* 5:4 (2011) 300-304

## A New Labdane Diterpene and Other Constituents from

*Marrubium deserti* Noe ex coss.

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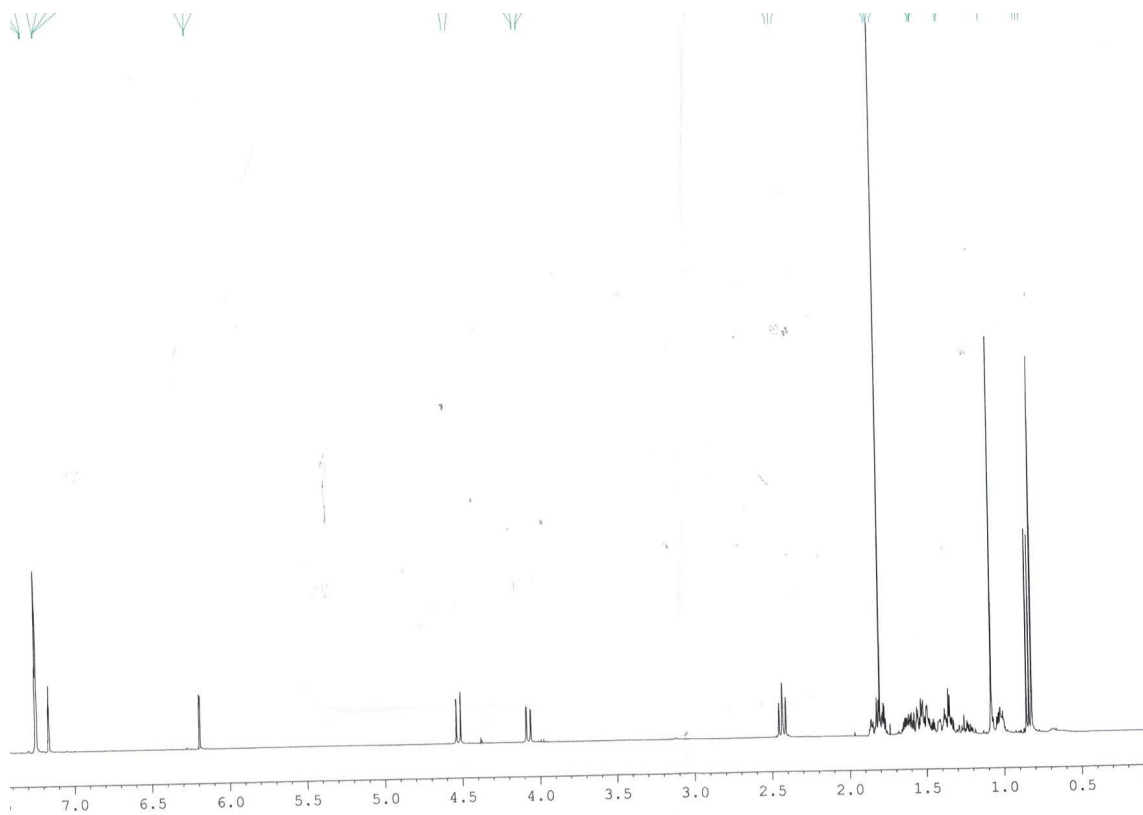
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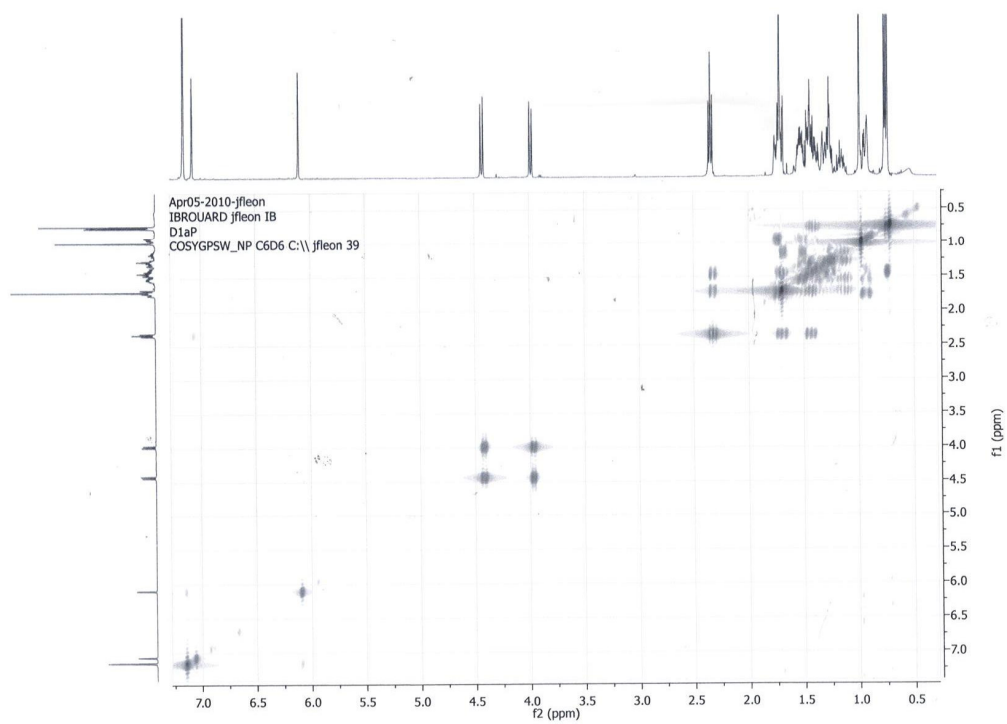
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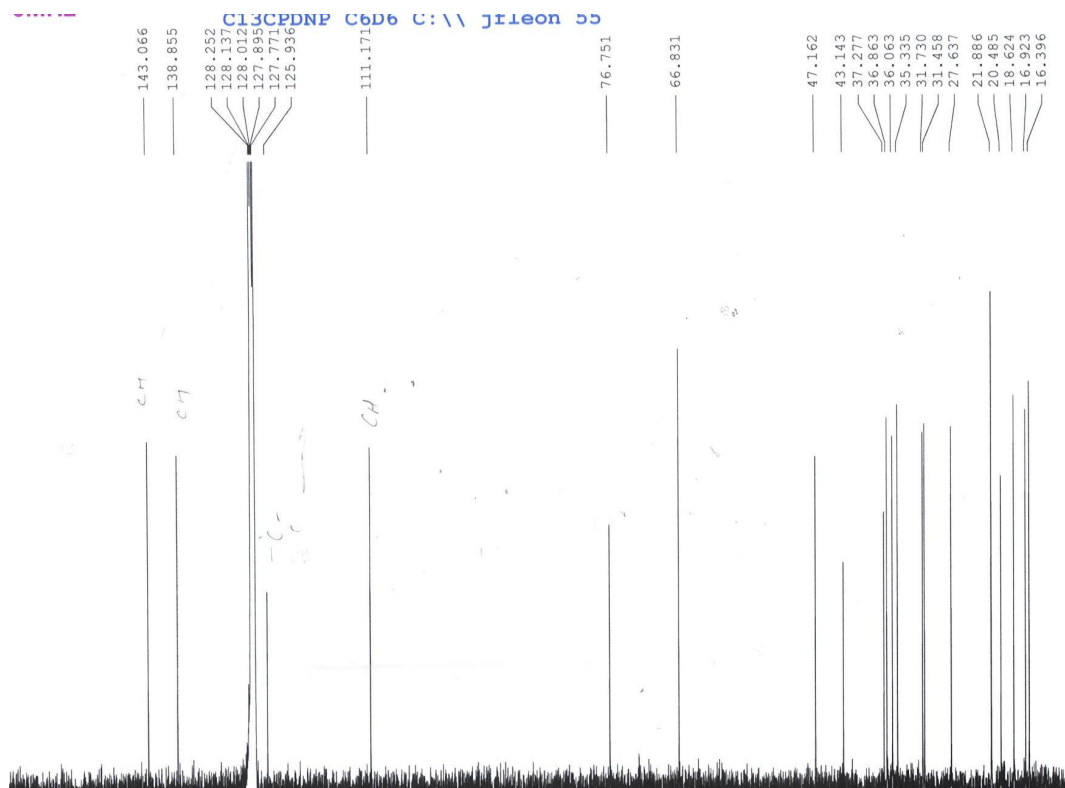
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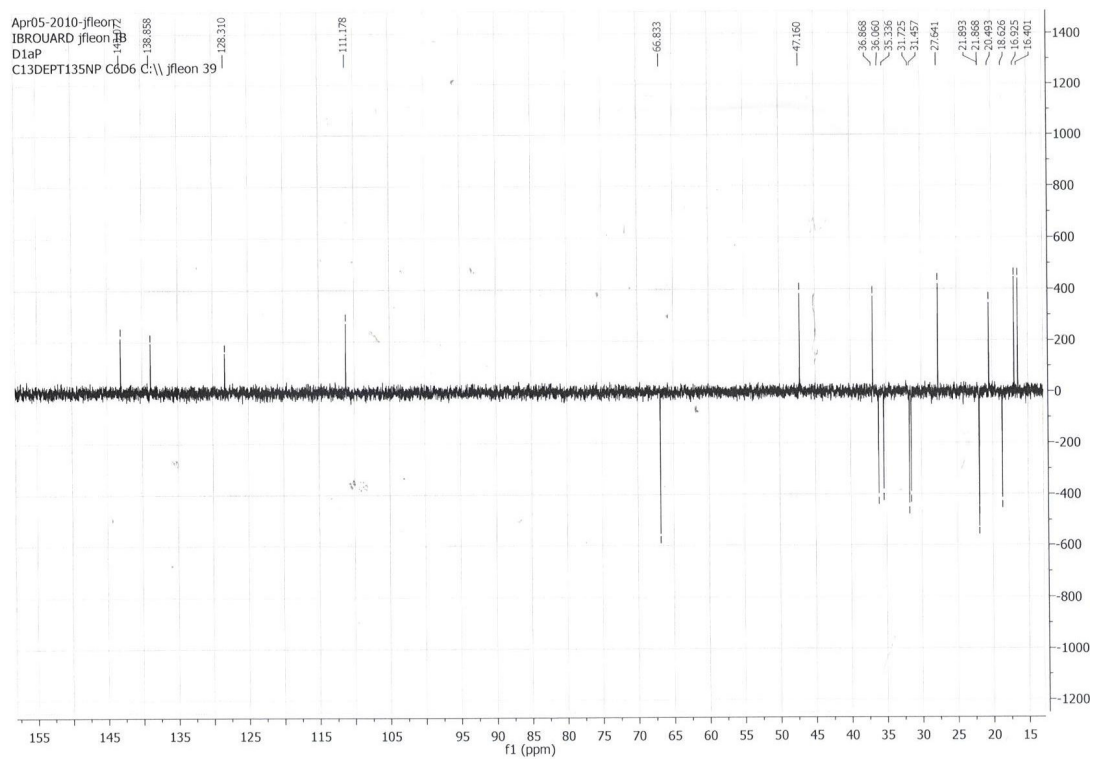
**S1:**  $^1\text{H}$ -NMR spectrum ( $\text{C}_6\text{D}_6$ , 500 MHz) of Compound **3** (6-dehydroxy-19-acetyl-marrubenol )



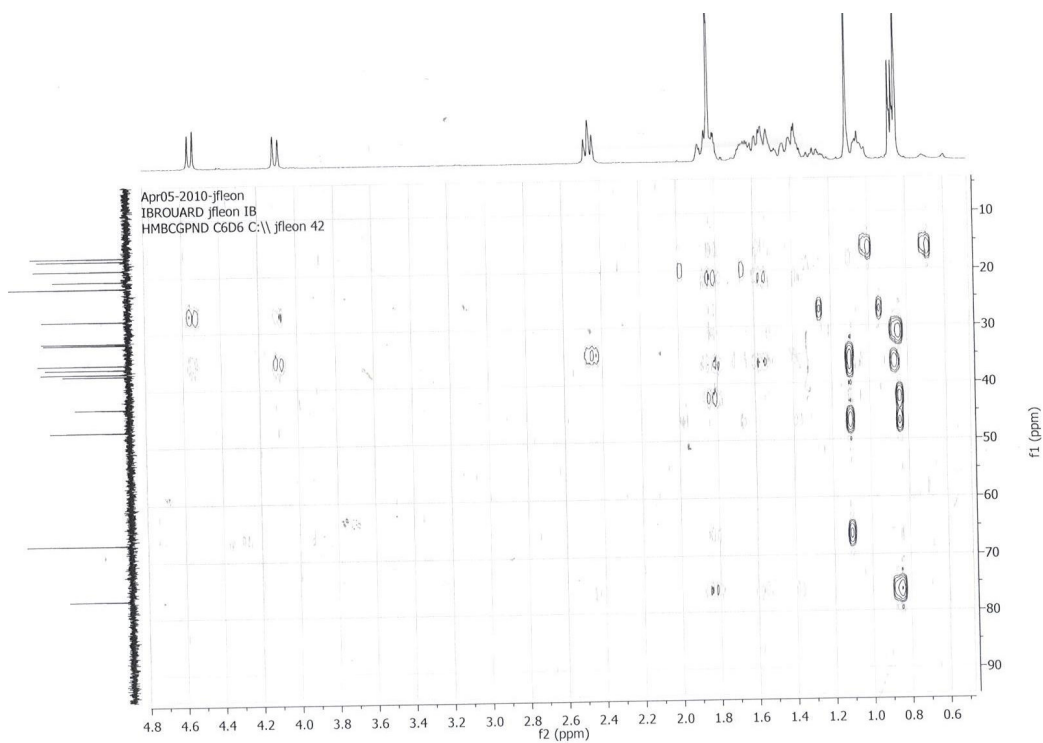
**S2:** COSY-NMR spectrum (C6D6, 500 MHz) of Compound **3** (6-dehydroxy-19-acetyl-marrubenol )



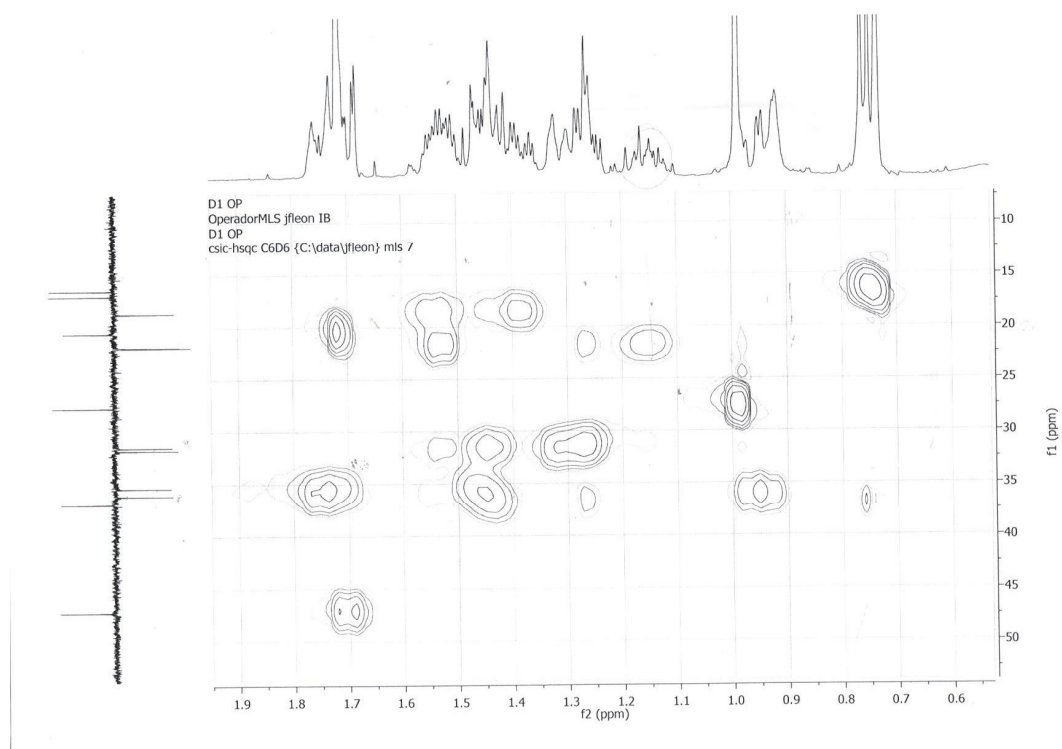
**S3:**  $^{13}\text{C}$ -NMR spectrum (C<sub>6</sub>D<sub>6</sub>, 125 MHz) of Compound **3** (6-dehydroxy-19-acetyl-marrubenol )



**S4:** DEPT 135-NMR (C6D6, 125 MHz) spectrum of Compound **3** (6-dehydroxy-19-acetyl-marrubenol )

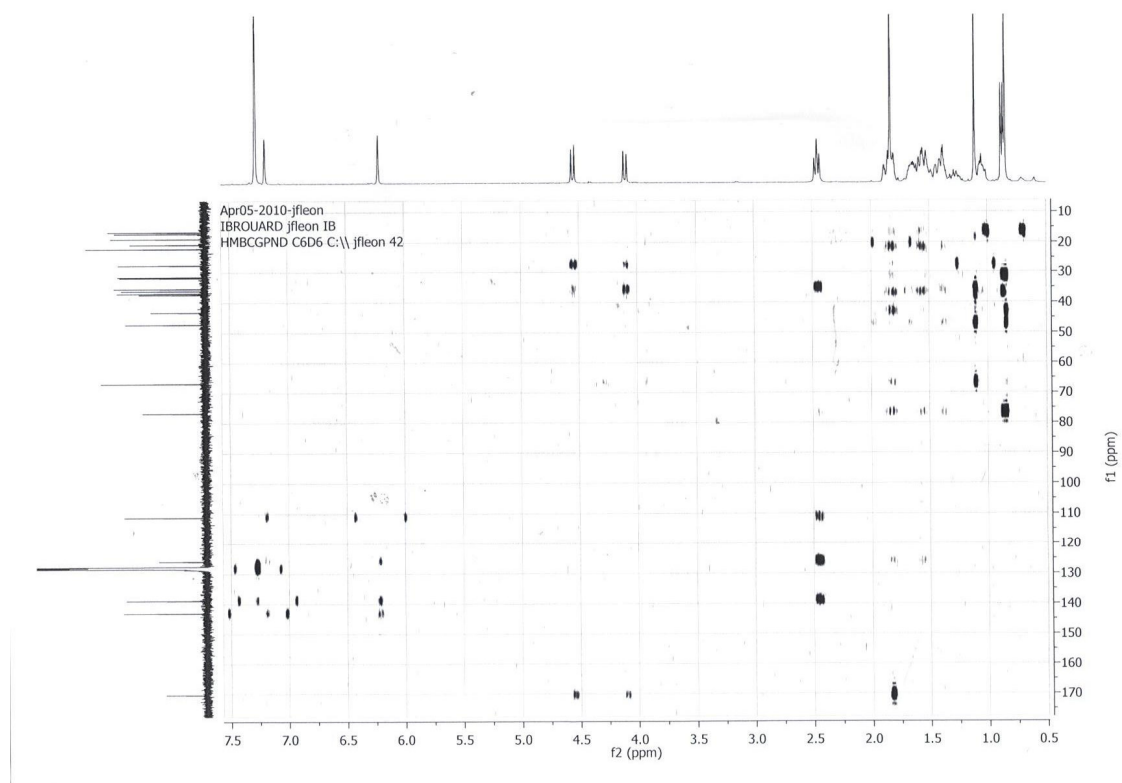


**S5:** Expansion HSQC-NMR (C6D6, 500 MHz) spectrum Compound **3** (6-dehydroxy-19-acetyl-marrubenol )

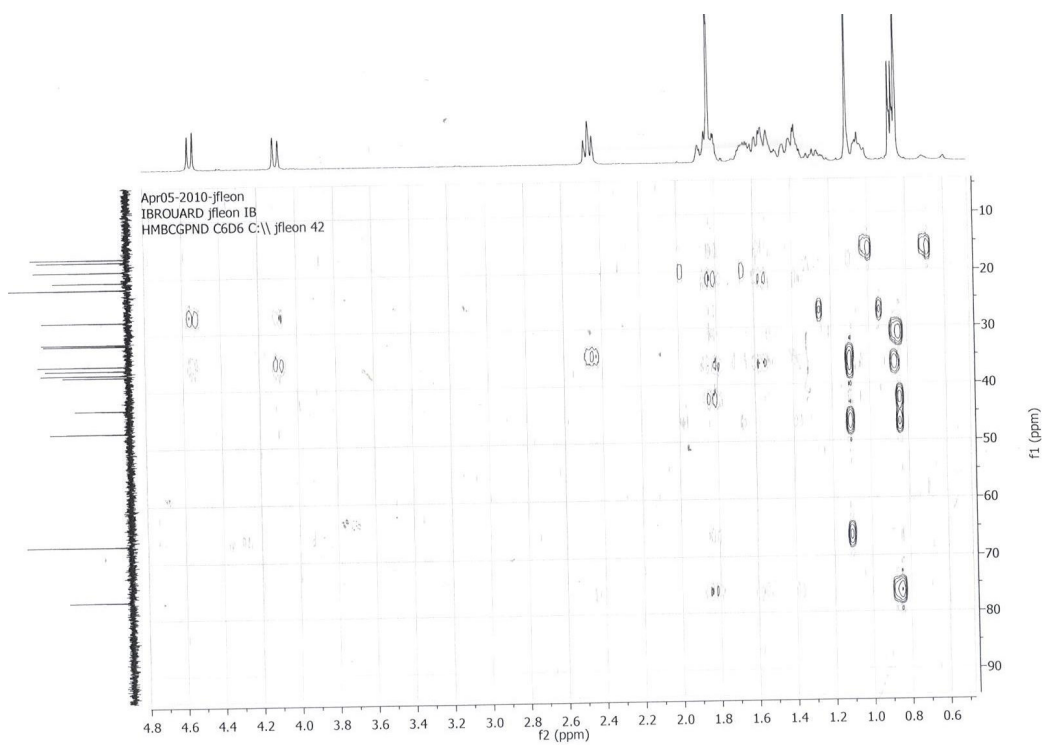


**S6:** Expansion HSQC-NMR ( C6D6, 500 MHz ) spectrum of Compound **3**  
(6-dehydroxy-19-acetyl-marrubenol )

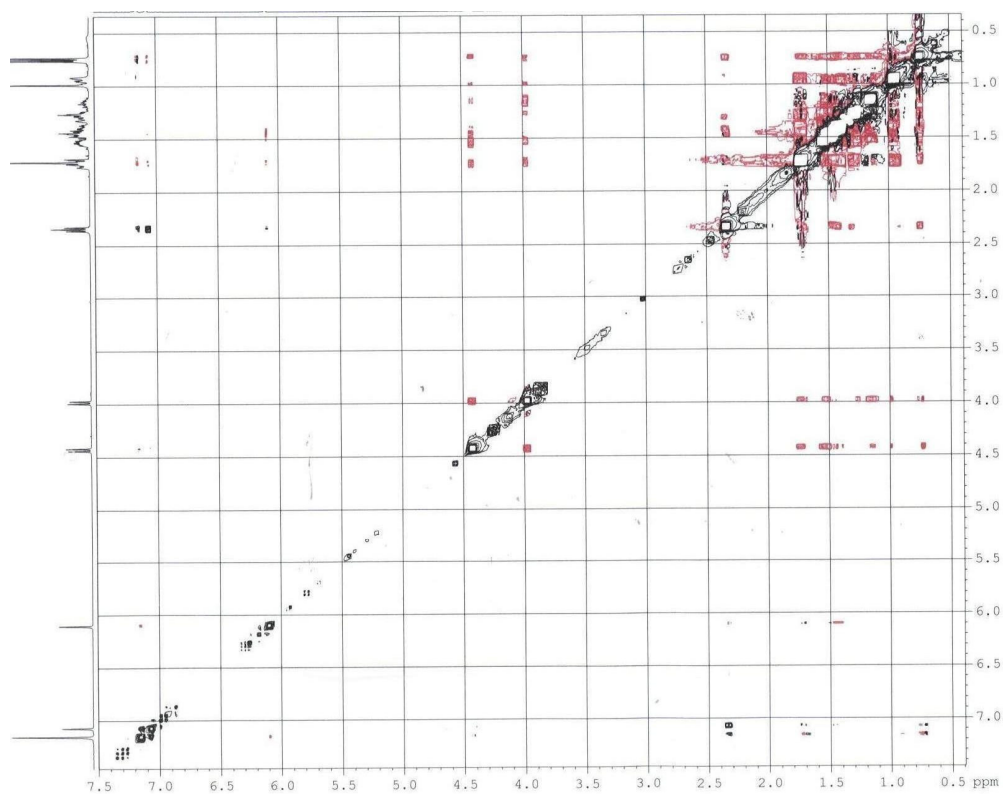




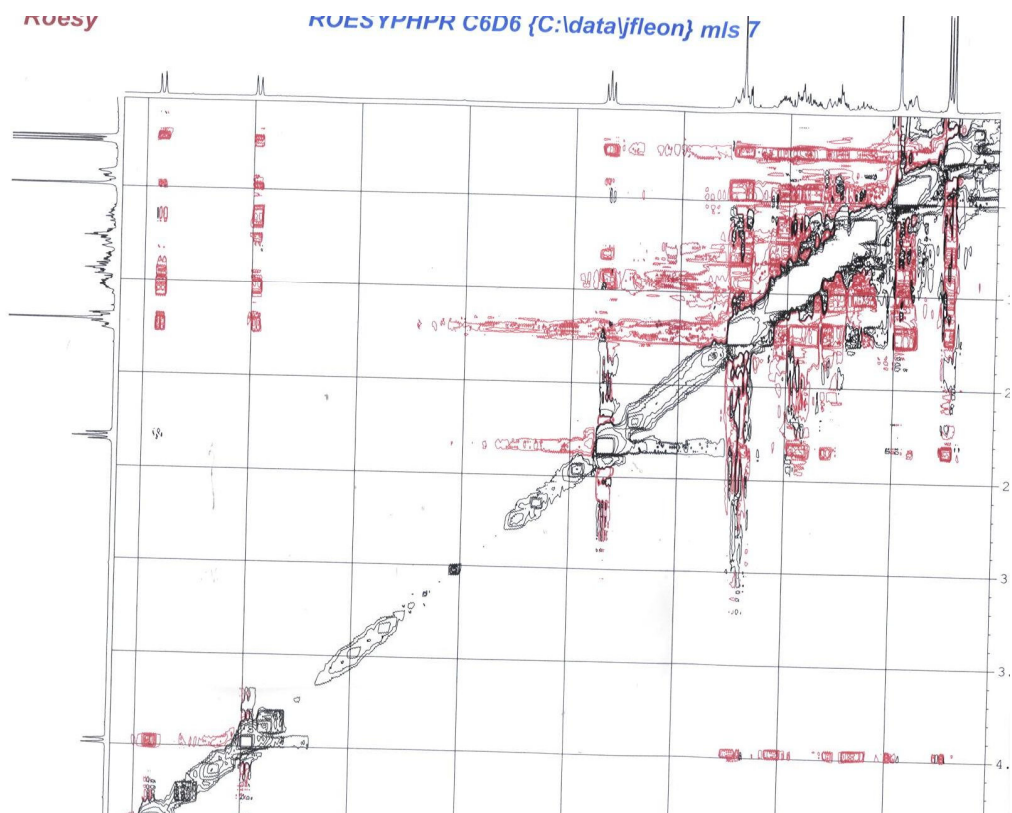
**S7:** HMBC-NMR spectrum of Compound **3** (6-dehydroxy-19-acetyl-marrubenol )



**S8:** Expansion HMBC -NMR spectrum ( C6D6, 500 MHz ) of Compound **3** (6-dehydroxy-19-acetyl-marrubenol )

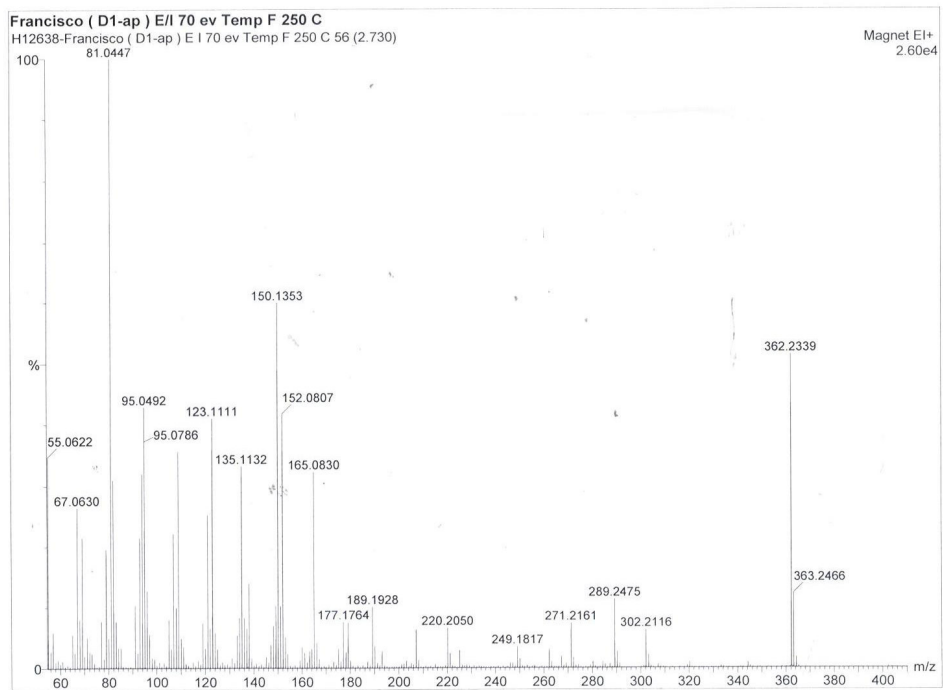


**S9:** ROESY -NMR spectrum ( C<sub>6</sub>D<sub>6</sub>, 500 MHz ) of Compound **3** (6-dehydroxy-

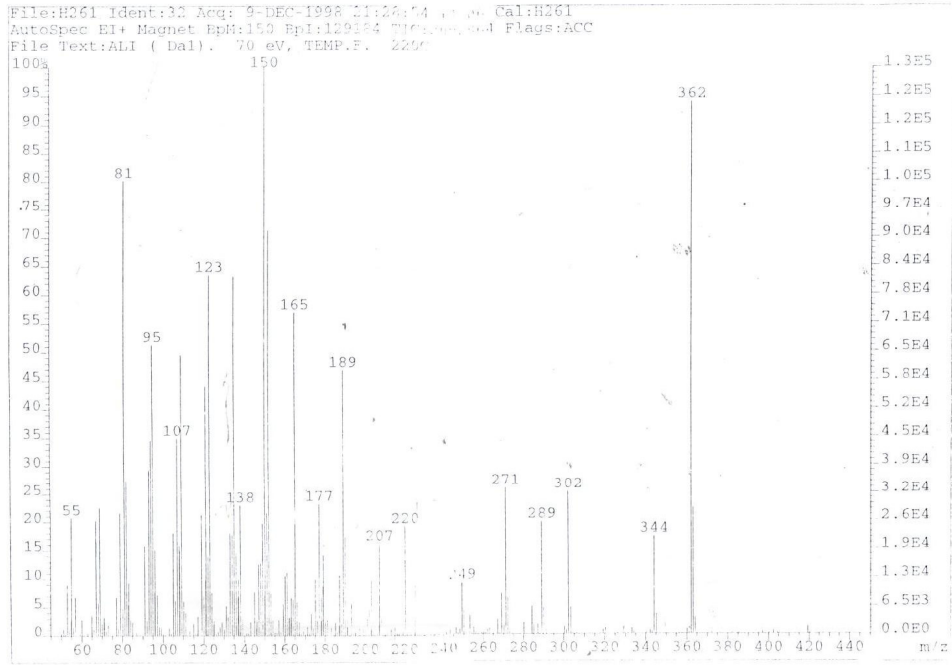


**S10:**ROESY -NMR spectrum ( C6D6, 500 MHz ) of Compound **3** ( 6-dehydroxy-19-acetyl-marrubanol )





**S12** : HRMS- spectrum of Compound **3** ( 6-dehydroxy-19-acetyl-marrubanol )



**S13** : EIMS (70 ev) spectrum of Compound **3** ( 6-dehydroxy-19-acetylmarrubanol)

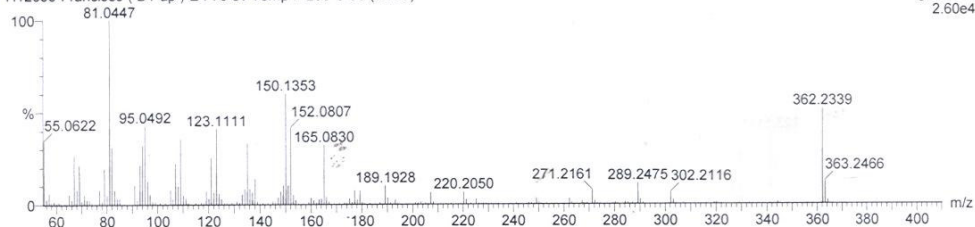
Elemental Composition Report

Multiple Mass Analysis: 1457 mass(es) processed - displaying only valid results  
 Tolerance = 5.0 PPM / DBE: min = 1.5, max = 50.0  
 Isotope matching not enabled

Monoisotopic Mass, Odd and Even Electron Ions  
 20118 formula(e) evaluated with 69 results within limits (up to 50 closest results for each mass)

Francisco (D1-ap) E/I 70 ev Temp F 250 C  
 H12638-Francisco (D1-ap) E/I 70 ev Temp F 250 C 56 (2.730)

Magnet EI+  
 2.60e4



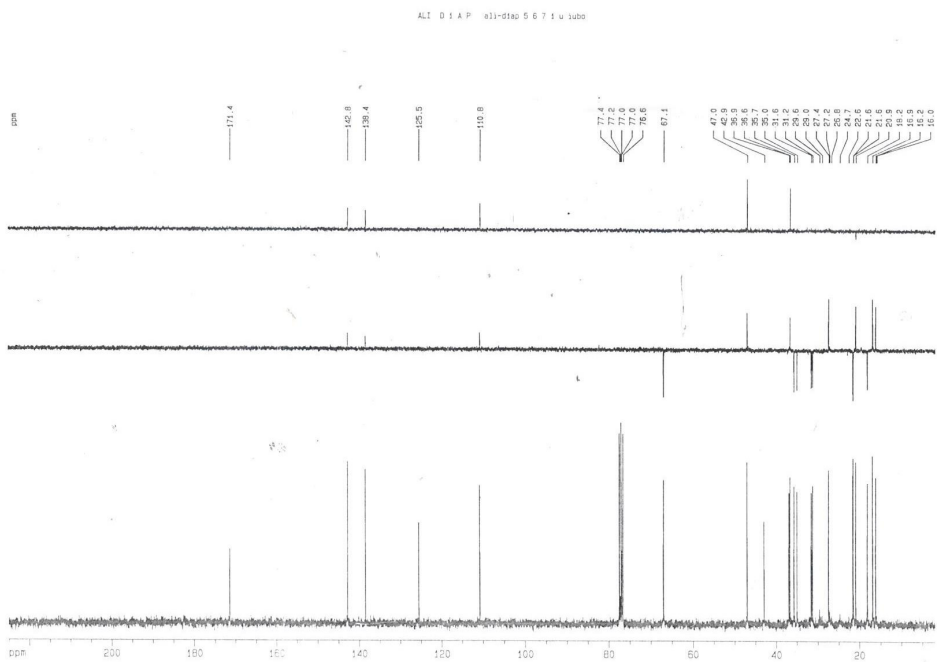
Minimum: 3.00  
 Maximum: 100.00

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
363.2675	7.46	363.2688	-1.3	-3.6	9.5	C26 H35 O
363.2549	11.10	363.2535	1.4	3.8	5.5	C22 H35 O4
363.2383	12.11	363.2383	0.0	0.1	1.5	C18 H35 O7
363.2341	11.27	363.2324	1.7	4.7	10.5	C25 H31 O2
363.2174	6.47	363.2171	0.3	0.7	6.5	C21 H31 O5
362.2838	5.67	362.2821	1.7	4.7	5.0	C23 H38 O3
362.2464	45.19	362.2457	0.7	1.9	6.0	C22 H34 O4
362.2297	50.50	362.2305	-0.8	-2.1	2.0	C18 H34 O7
362.2256	47.34	362.2246	1.0	2.8	11.0	C25 H30 O2
362.2089	31.92	362.2093	-0.4	-1.2	7.0	C21 H30 O5
362.2047	27.47	362.2035	1.2	3.4	16.0	C28 H26
362.1880	11.92	362.1882	-0.2	-0.5	12.0	C24 H26 O3
362.1714	4.38	362.1729	-1.5	-4.2	8.0	C20 H26 O6
302.2255	6.30	302.2246	0.9	3.0	6.0	C20 H30 O2
302.2082	6.12	302.2093	-1.1	-3.7	2.0	C16 H30 O5
302.2047	5.47	302.2035	1.2	4.1	11.0	C23 H26
289.2541	10.61	289.2531	1.0	3.3	4.5	C20 H33 O
289.2176	4.90	289.2168	0.8	2.9	5.5	C19 H29 O2
271.2285	5.61	271.2273	1.2	4.3	1.5	C16 H31 O3
271.2068	7.12	271.2062	0.6	2.2	6.5	C19 H27 O
271.1912	3.74	271.1909	0.3	1.0	2.5	C15 H27 O4
249.1846	3.43	249.1855	-0.9	-3.4	4.5	C16 H25 O2
207.1741	6.26	207.1749	-0.8	-3.8	3.5	C14 H23 O
179.1805	5.65	179.1800	0.5	2.9	2.5	C13 H23
179.1065	3.33	179.1072	-0.7	-3.9	4.5	C11 H15 O2
177.1642	4.78	177.1643	-0.1	-0.7	3.5	C13 H21
165.0697	14.82	165.0704	-0.7	-4.4	9.5	C13 H9
160.0886	3.40	160.0888	-0.2	-1.3	6.0	C11 H12 O
152.0842	39.68	152.0837	0.5	3.1	4.0	C9 H12 O2
152.0632	12.53	152.0626	0.6	3.9	9.0	C12 H8
151.1482	7.09	151.1487	-0.5	-3.1	2.5	C11 H19
151.0754	7.74	151.0759	-0.5	-3.3	4.5	C9 H11 O2
150.1405	52.00	150.1409	-0.4	-2.3	3.0	C11 H18
150.0682	3.23	150.0681	0.1	0.8	5.0	C9 H10 O2
149.1327	4.50	149.1330	-0.3	-2.2	3.5	C11 H17
149.0454	3.35	149.0450	0.4	2.7	1.5	C5 H9 O5
149.0386	4.16	149.0391	-0.5	-3.5	10.5	C12 H5
148.1248	7.04	148.1252	-0.4	-2.7	4.0	C11 H16
138.1412	8.87	138.1409	0.3	2.5	2.0	C10 H18
135.1178	30.70	135.1174	0.4	3.1	3.5	C10 H15
135.0806	6.94	135.0810	-0.4	-2.9	4.5	C9 H11 O
124.1249	3.91	124.1252	-0.3	-2.4	2.0	C9 H16
123.1168	31.75	123.1174	-0.6	-4.7	2.5	C9 H15

S14 : Table HRMS of Compound 3 ( 6-dehydroxy-19-acetyl-marrubenol )

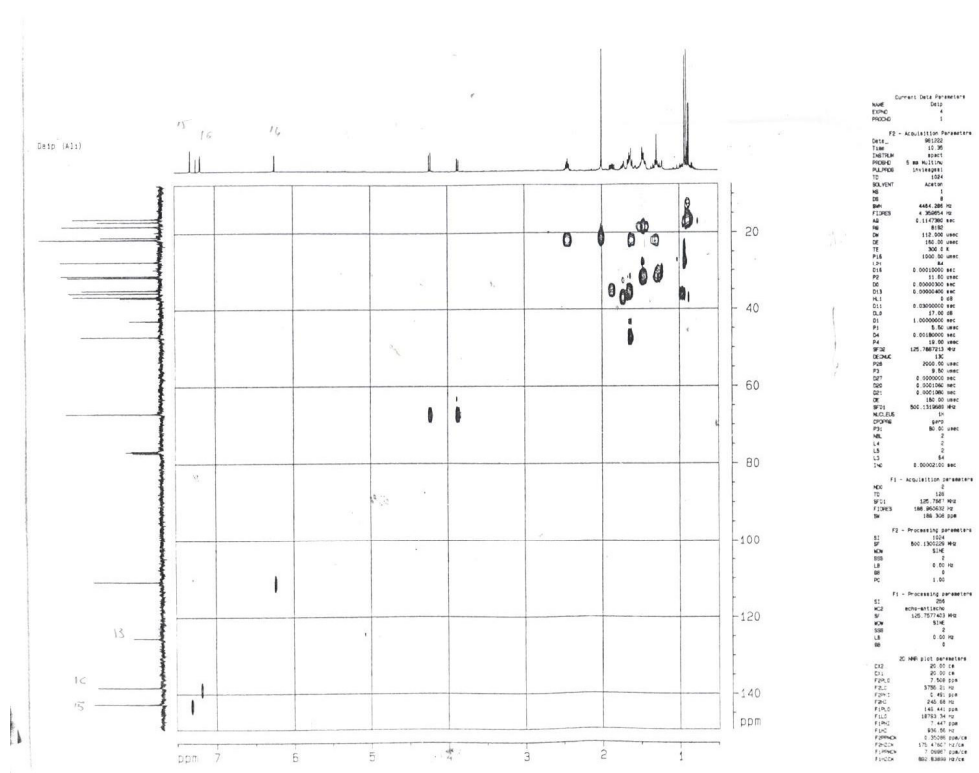






**S16** :  $^{13}\text{C}$  and DEPT -NMR spectrum ( $\text{CDCl}_3$ , 125 MHz) of Compound **3** ( 6-dehydroxy-19-acetyl-marrubenol )

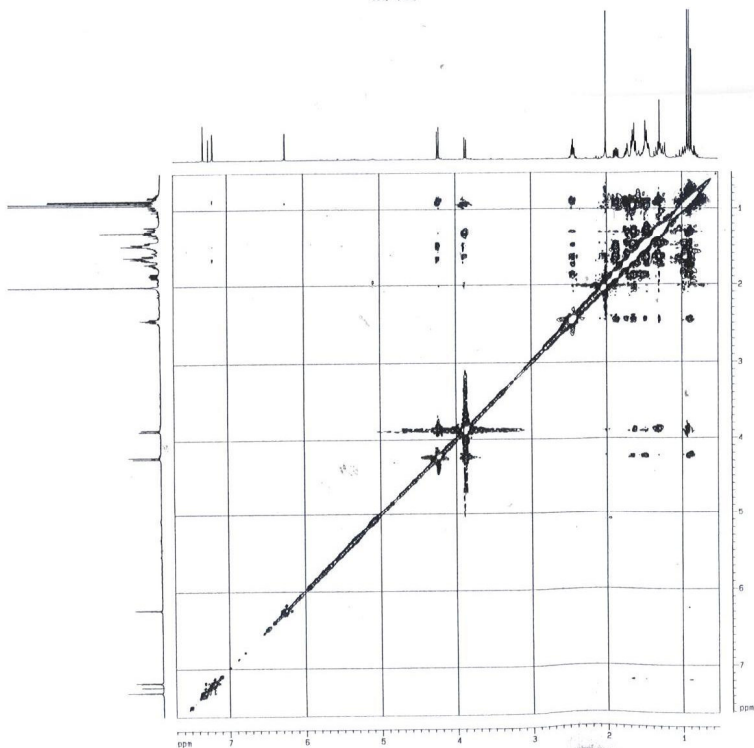




**S18** : HSQC-NMR spectrum (Aceton-d<sub>6</sub> , 500 MHz) of compound **3** (6-dehydroxy-19-acetyl-marrubanol **3**)

Figure S20 : NOESY-NMR spectrum of 6-dehydroxy-19-acetyl-marrubenol **3** in DMSO-d<sub>6</sub> recorded on Bruker DRX 500 MHz

Date (A11)



```
Current Data Parameters
NAME      Date
EXPNO    8
PROCNO   1

F2 - Acquisition Parameters
Date_    981222
Time     11:07
INSTRUM  spect
PROBHD   5 mm Multinu
PULPROG  mzgpg30
TD       1024
SOLVENT  DMSO
NS       8
DS       2
SWH      4664.200 Hz
FIDRES   4.259264 Hz
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RG       32
DN       112.000 usec
DC       160.00 usec
TE       300.0 K
HL1      0 dB
DI1      1.0000000 sec
PI1      5.50 usec
DD       0.0000300 sec
DB       0.5000000 sec
DE       160.00 usec
SFO1     500.1319689 MHz
NUCLEUS  1H
INNO     0.00011208 sec

F1 - Acquisition parameters
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SFO1   500.130 MHz
FIDRES 17.426458 Hz
DS     0.920 ppm

F2 - Processing parameters
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SF     500.1302000 MHz
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SSB    2
LB     0.00 Hz
GB     0
PC     1.00

F1 - Processing parameters
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HC2    1001
SF     500.1302000 MHz
WDW    SINE
SSB    2
LB     0.00 Hz
GB     0

2D NMR plot parameters
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CX1    20.00 cm
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F2LO   3893.54 Hz
F2PHI  0.522 ppm
F2HI   281.18 Hz
F2FL0  7.628 ppm
F1LO   3813.46 Hz
F1PHI  0.526 ppm
F1HI   262.82 Hz
F2HYCN  0.35914 ppm/cm
F2FSM  178.8170 Hz/cm
F1HYCN  0.35497 ppm/cm
F1FSM  177.53004 Hz/cm
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