

Supporting Information

Rec. Nat. Prod. 7:2 (2013) 114-118

Maroccanin: a new γ -lactone and other constituents from *Centaurea maroccana* Ball. (Asteraceae)

Sabrina Bicha¹, Pierre Chalard^{2,3}, Leila Hammoud¹, Francisco León⁴, Ignacio Brouard⁴,
Victor Perez Garcia⁴, Annelise Lobstein⁵, Ali Bentamene¹, Samir Benayache⁶, Jaime Bermejo⁴
and Fadila Benayache^{1*}

¹Laboratoire de Phytochimie et Analyses Physico-Chimiques et Biologiques, Université Mentouri,
Route de Aïn El Bey, 25000 Constantine, Algeria.

²Institut de Chimie de Clermont-Ferrand (ICCF), Ecole Nationale Supérieure de Chimie de Clermont-
Ferrand (ENSCCF), BP10448, F-63000 Clermont-Ferrand (France)

³ UMR 6296, ICCF – CNRS. F-63171 Aubière

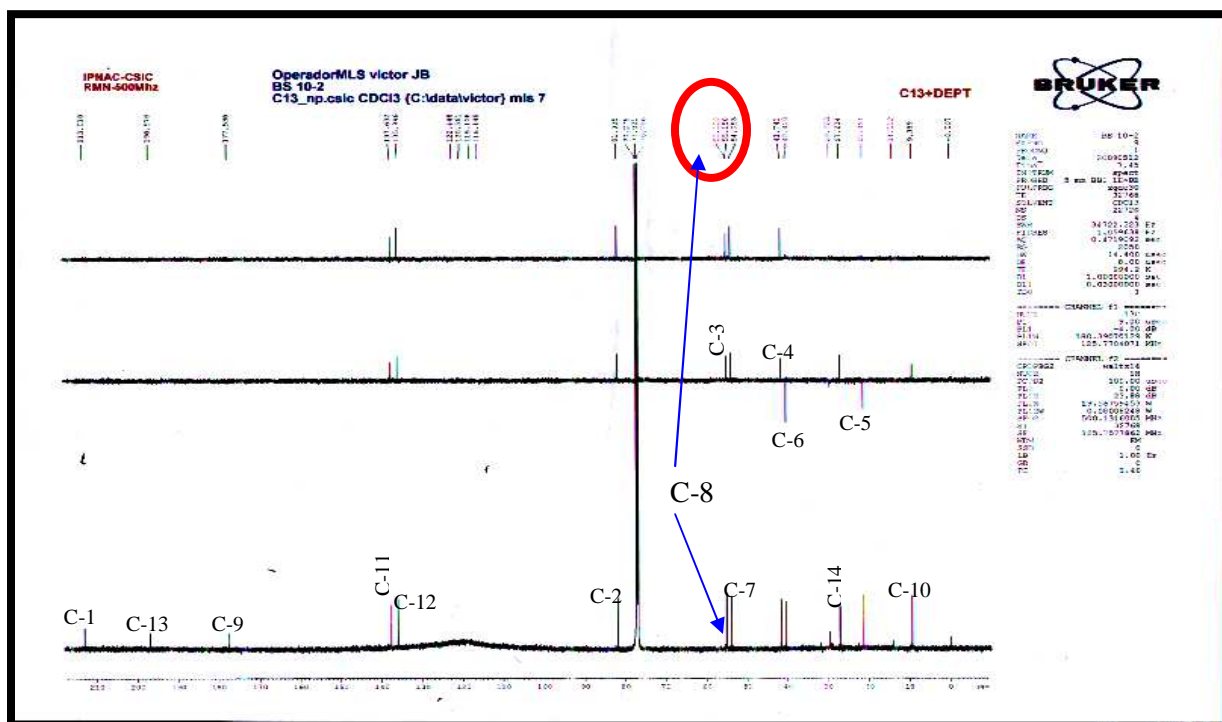
⁴Instituto de Productos Naturales y Agrobiología-CSIC, ³Instituto Universitario de Bio-orgánica
"Antonio González", Avda. Astrofísico Francisco Sánchez, 3, 38206 La Laguna, Tenerife, Spain.

⁵Laboratoire d'innovation thérapeutique UMR-CNRS 7200, Université de Strasbourg, Faculté de
Pharmacie, 74 Route du Rhin, 67401 Illkirch, France.

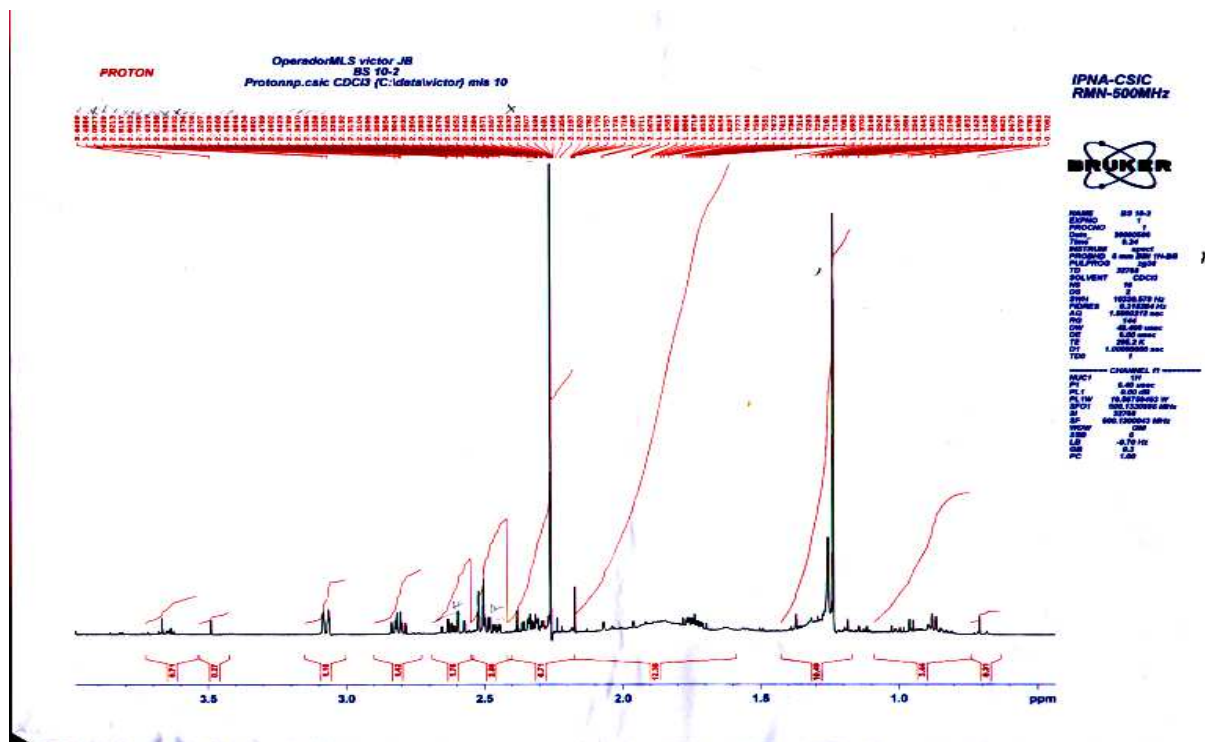
⁶Laboratoire de Valorisation des Ressources Naturelles et Synthèse de Substances Bioactives,
Université Mentouri, Route de Aïn El Bey, 25000 Constantine, Algeria

Table of contents	Page
S1 : ¹³ C and DEPT -NMR spectrum (CDCl ₃ , 125 MHz) of Compound 1 (Maroccanin)	3
S2 : ¹ H-NMR spectrum (CDCl ₃ , 500 MHz) of Compound 1 (Maroccanin)	4
S3 : Expansion, ¹ H-NMR spectrum (CDCl ₃ , 500 MHz) of Compound 1 (Maroccanin)	5
S4 : Expansion, ¹ H-NMR spectrum (CDCl ₃ , 500 MHz) of Compound 1 (Maroccanin)	6
S5 : COSY spectrum (CDCl ₃ , 500 MHz) of Compound 1 (Maroccanin)	7
S6 : Expansion, COSY spectrum (CDCl ₃ , 500 MHz) of Compound 1 (Maroccanin)	8
S7 : Expansion COSY spectrum (CDCl ₃ , 500 MHz) of Compound 1 (Maroccanin)	9

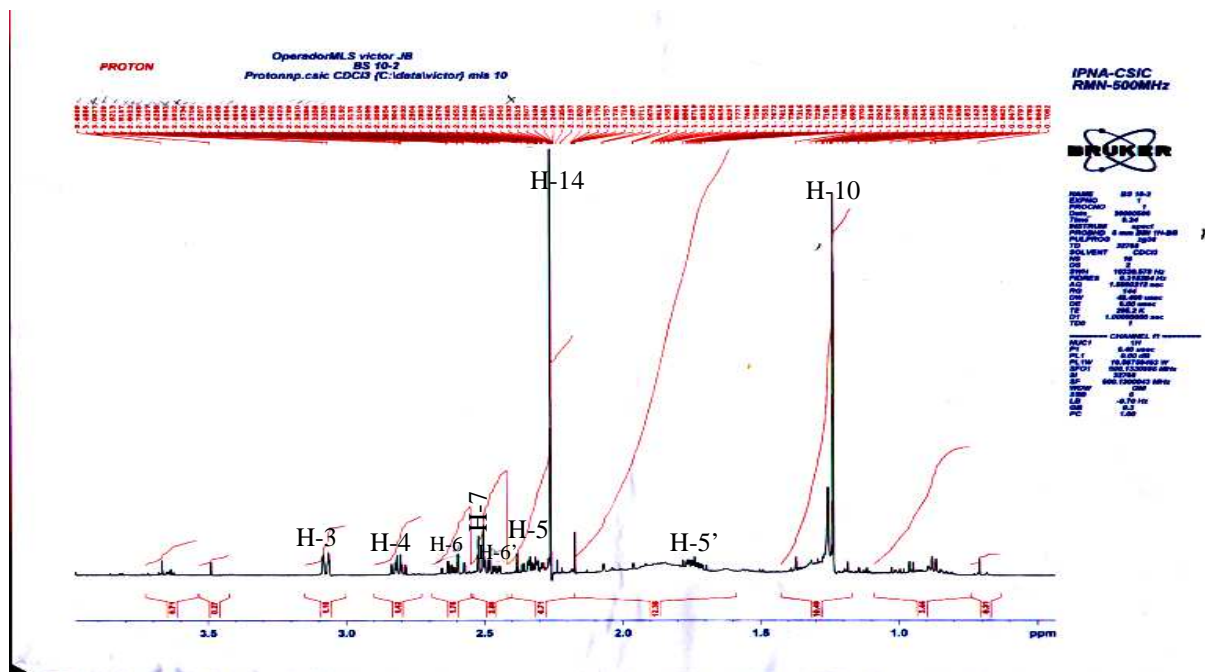
S8 : HSQC spectrum (CDCl ₃ , 500 MHz) of compound 1 (Maroccanin)	10
S9 : Expansion HSQC spectrum (CDCl ₃ , 500 MHz) of compound 1 (Maroccanin)	11
S10 : HMBC spectrum (CDCl ₃ , 500 MHz) of Compound 1 (Maroccanin)	12
S11 : Expansion HMBC spectrum (CDCl ₃ , 500 MHz) of Compound 1 (Maroccanin)	13
S12 : Expansion HMBC spectrum (CDCl ₃ , 500 MHz) of Compound 1 (Maroccanin)	14
S13 : Expansion HMBC spectrum (CDCl ₃ , 500 MHz) of Compound 1 (Maroccanin)	15
S14 : NOESY spectrum (CDCl ₃ , 500 MHz) of Compound 1 (Maroccanin)	16
S15 : High resolution Mass Spectrum (ESI+) of compound 1 (Maroccanin)	17



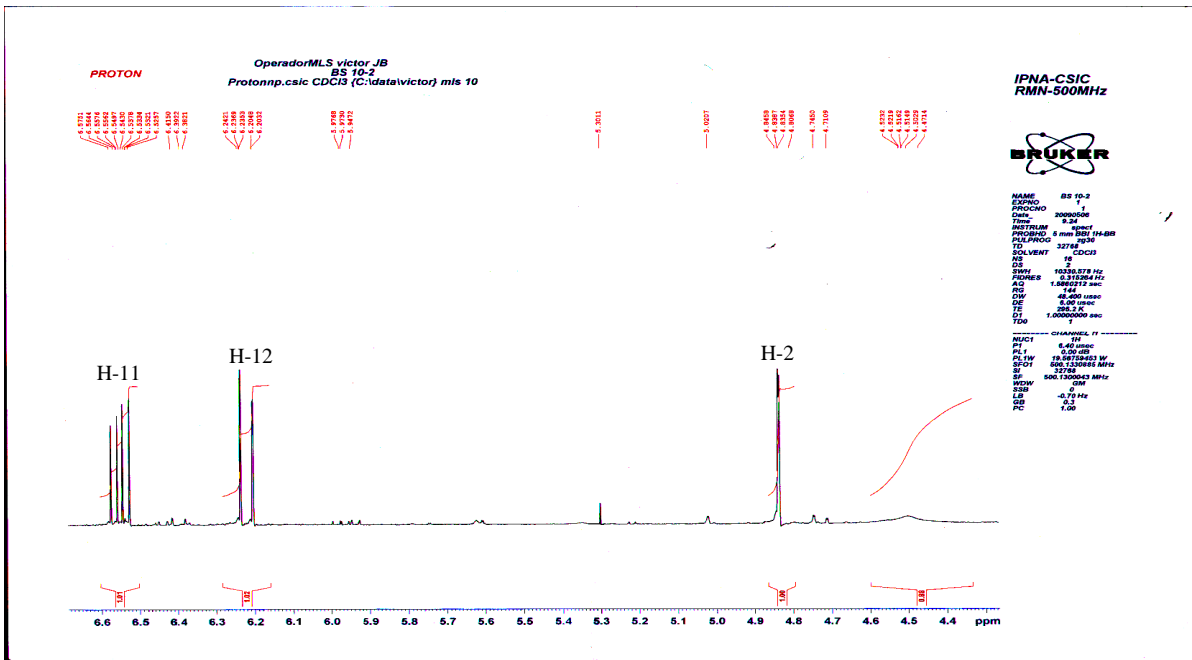
S1: ^{13}C and DEPT -NMR spectra (CDCl_3 , 125 MHz) of Compound 1 (Maroccanin)



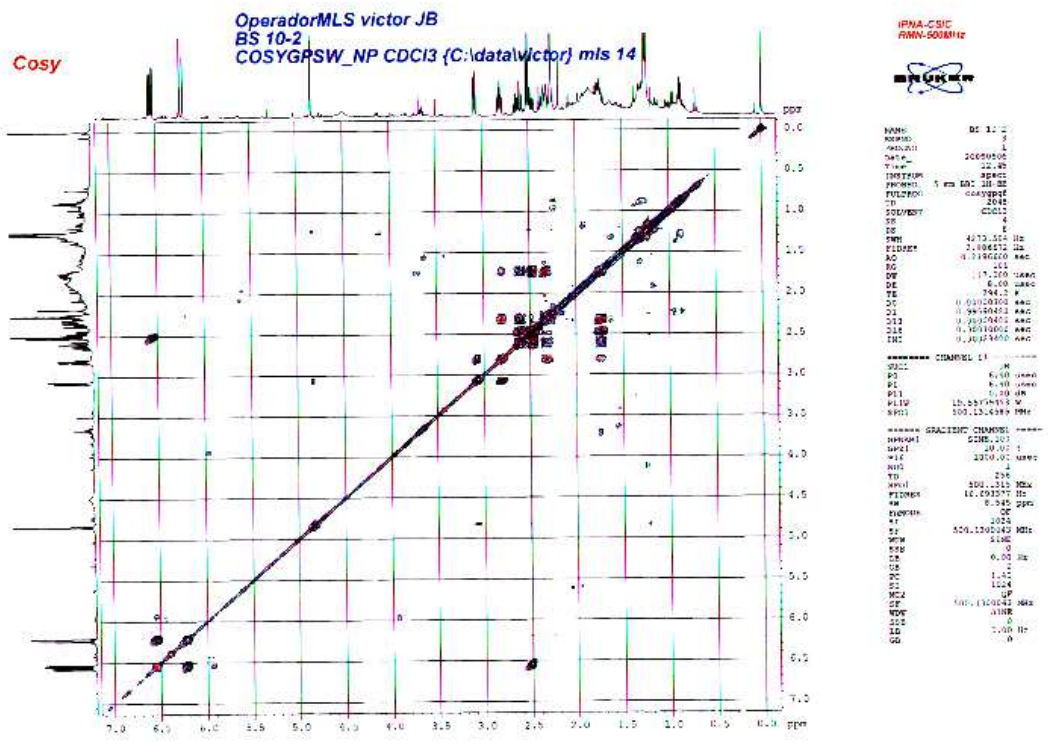
S2: ^1H -NMR spectrum (CDCl_3 , 500 MHz) of Compound **1** (Maroccanin)



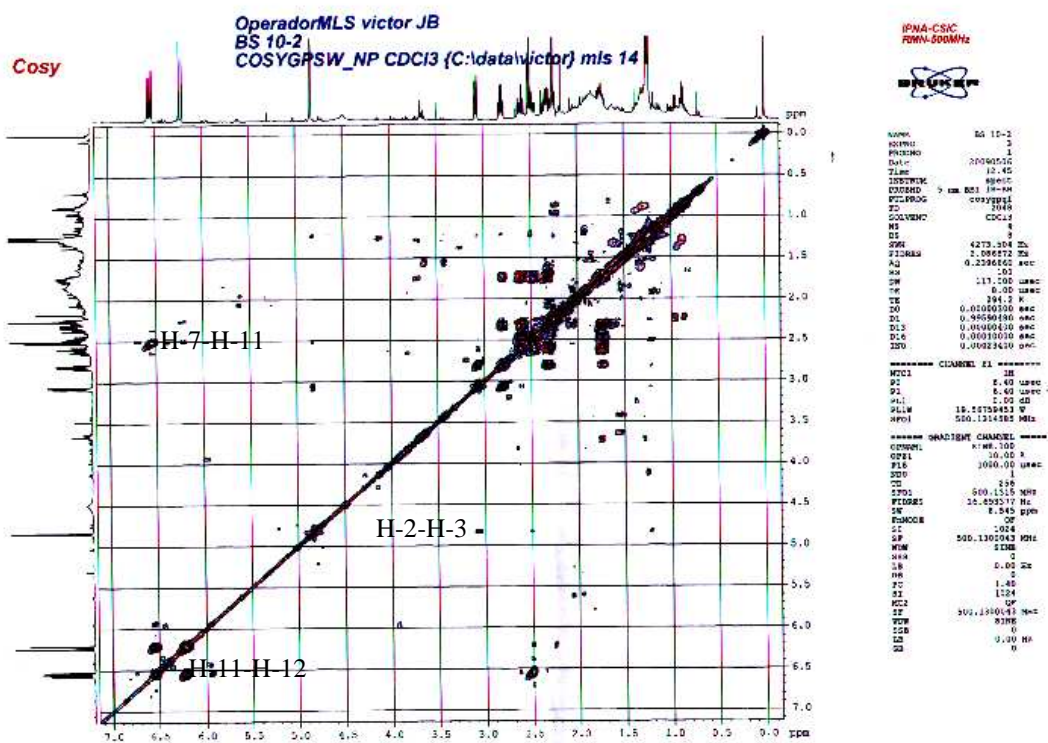
S3: Expansion, ^1H -NMR spectrum (CDCl_3 , 500 MHz) of Compound 1 (Maroccanin)



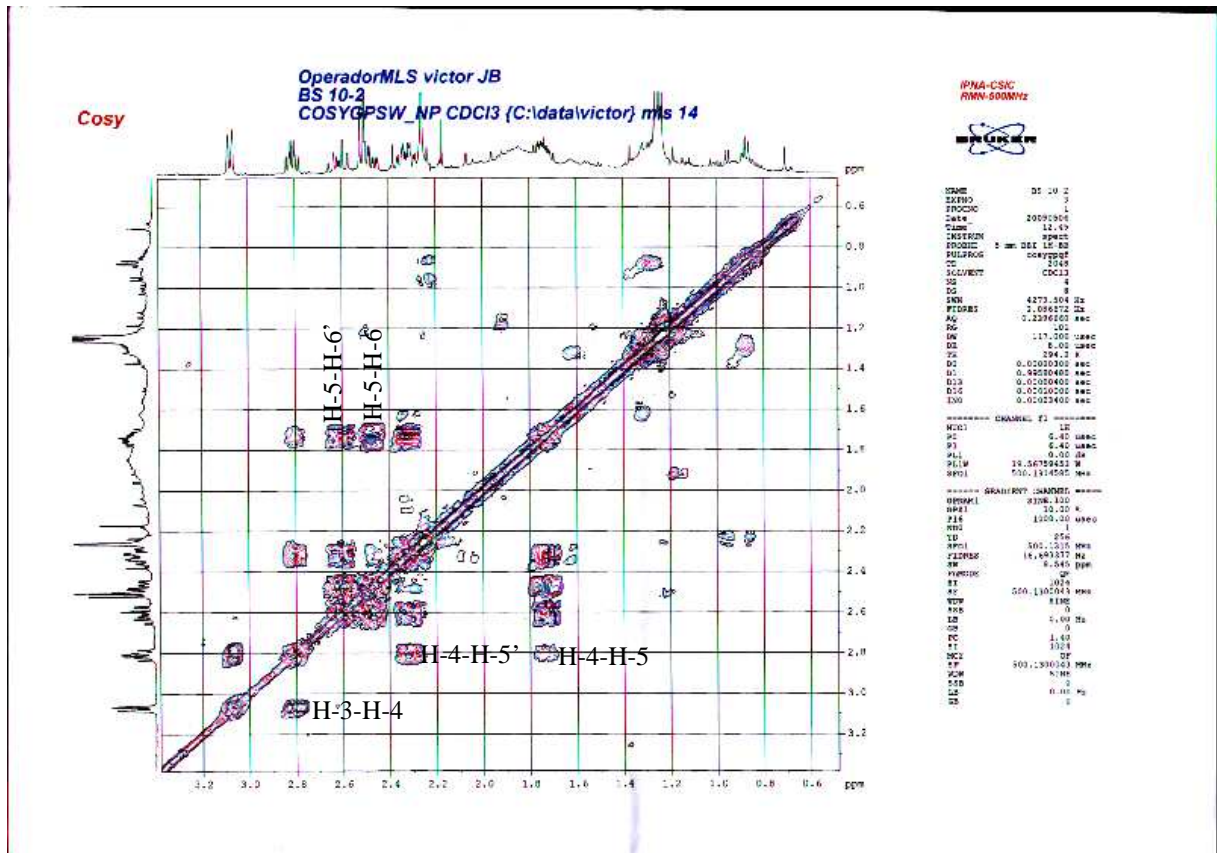
S4: Expansion, ¹H-NMR spectrum (CDCl₃, 500 MHz) of Compound 1 (Maroccanin)



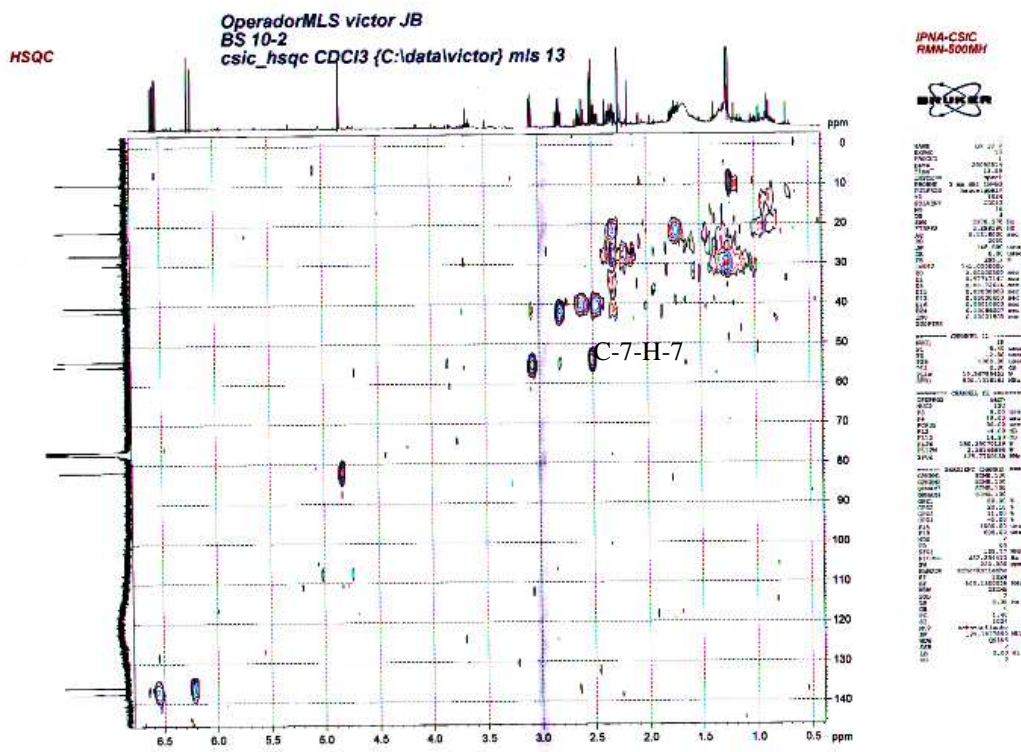
S5 : COSY spectrum (CDCl₃, 500 MHz) of Compound 1 (Maroccanin)



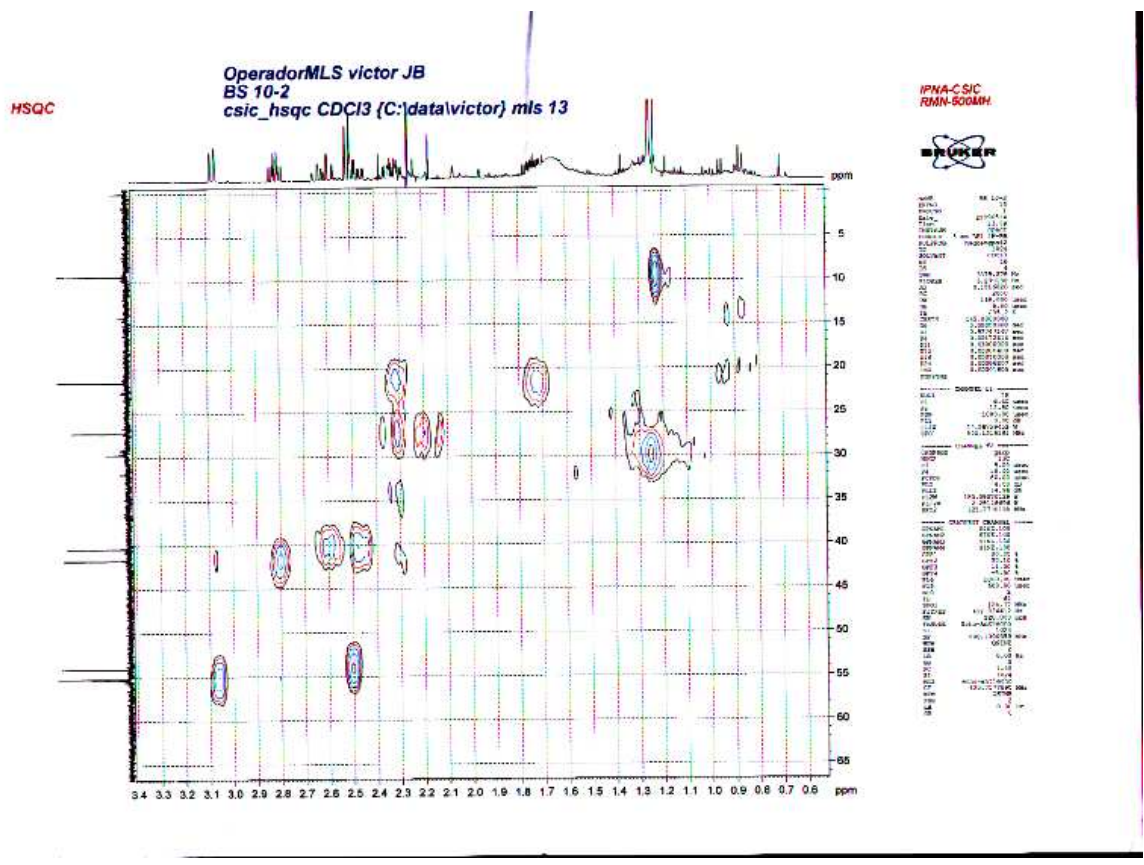
S6 : COSY spectrum (CDCl₃, 500 MHz) of Compound 1 (Maroccanin)



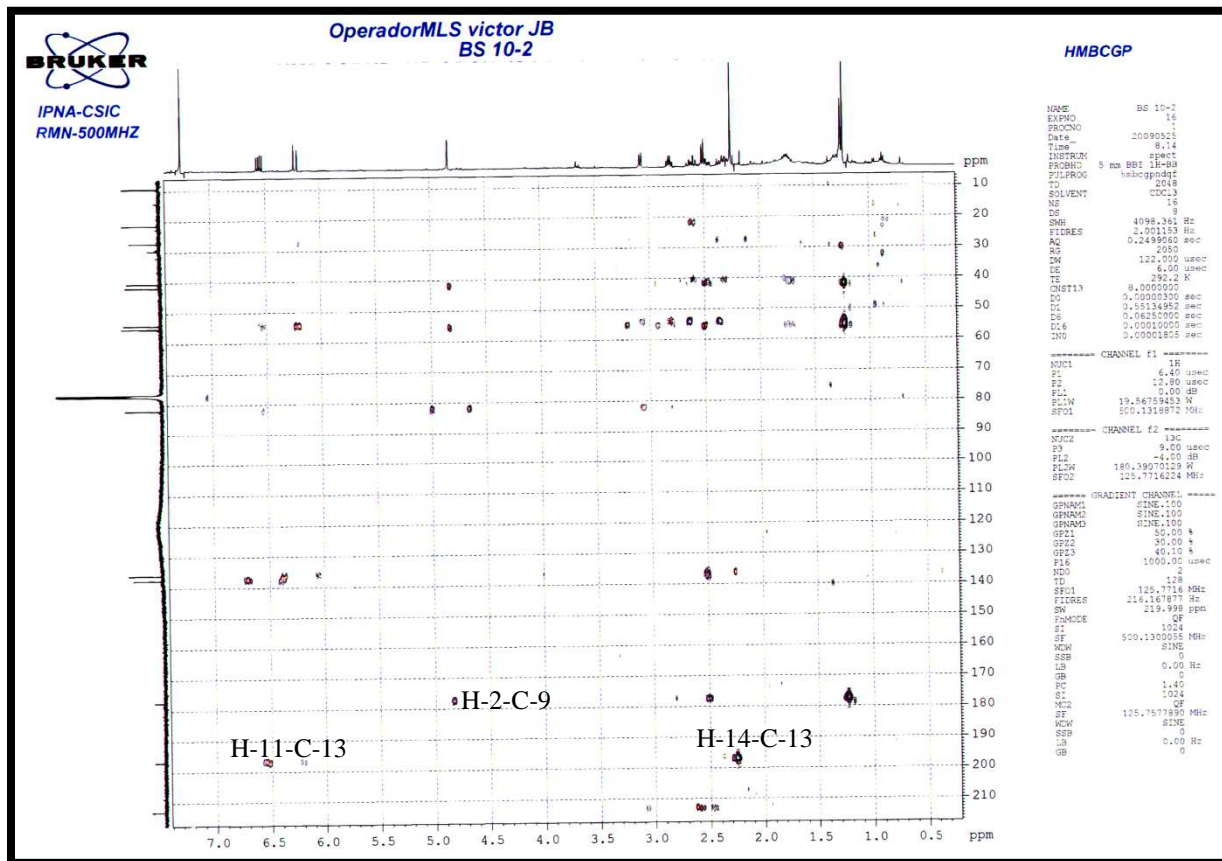
S7 : Expansion, COSY spectrum (CDCl₃, 500 MHz) of Compound 1 (Maroccanin)



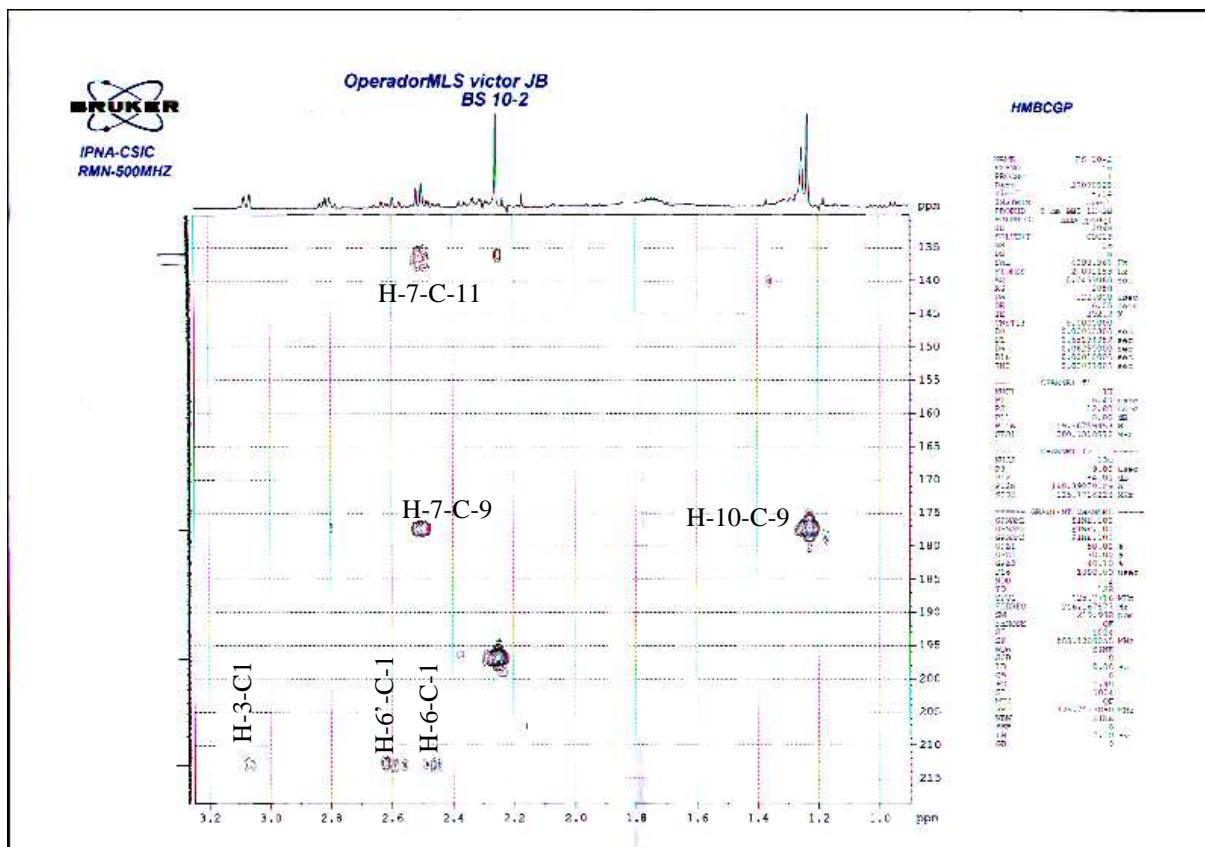
S8 : HSQC spectrum (CDCl₃, 500 MHz) of compound 1 (Maroccanin)



S9 : Expansion, HSQC spectrum (CDCl₃, 500 MHz) of compound 1 (Maroccanin)



S10: HMBC spectrum (CDCl₃, 500 MHz) of Compound **1** (Maroccanin)

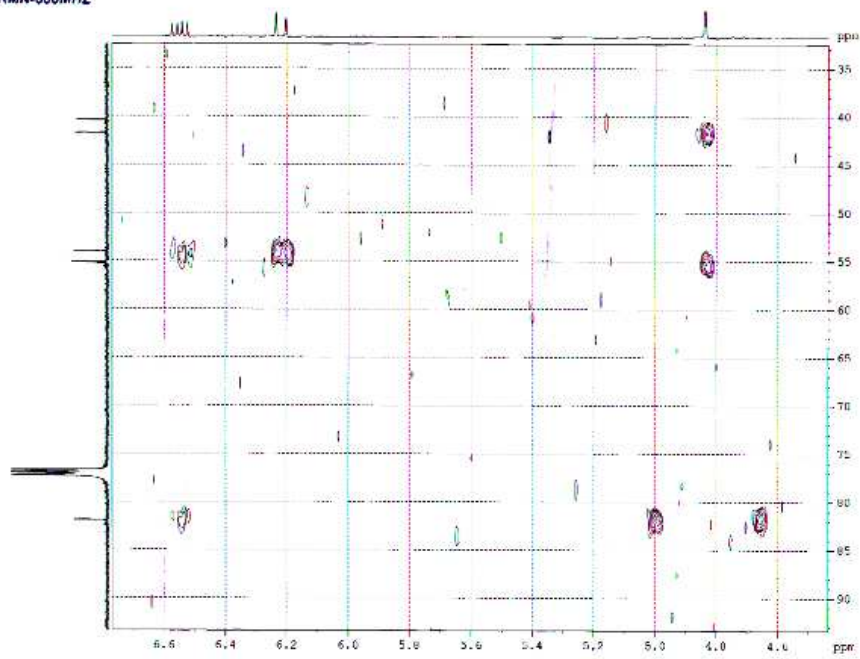


S11: Expansion, HMBC spectrum (CDCl₃, 500 MHz) of Compound 1(Maroccanin)



OperatorMLS victor JB
BS 10-2

HMBCGP

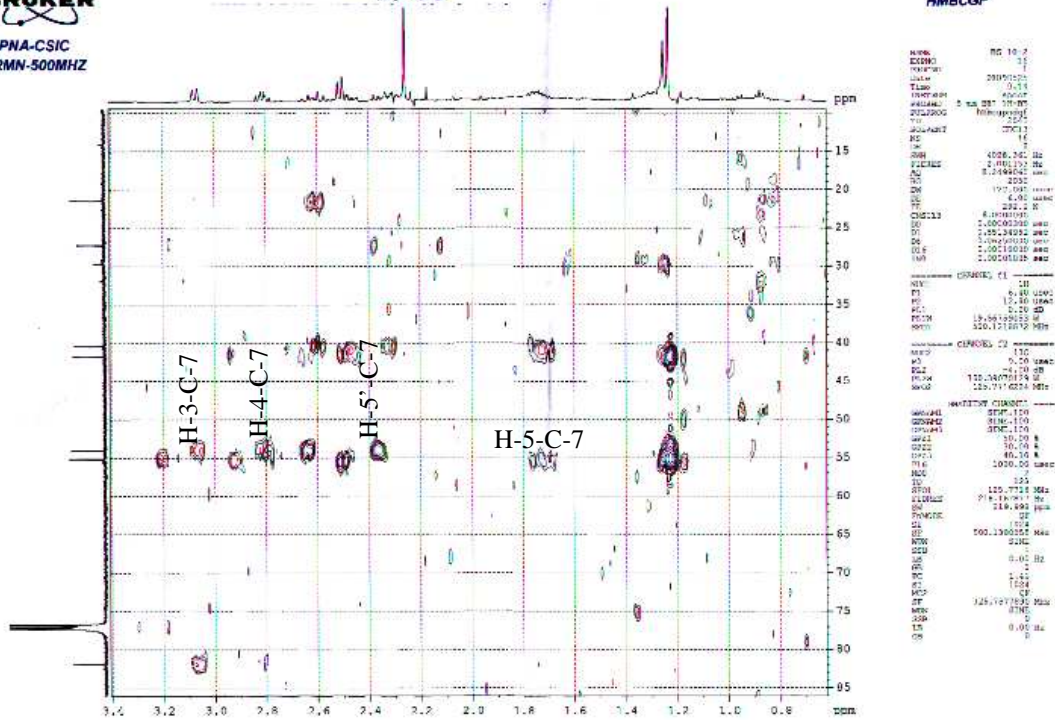


```

NAME      WS 11-2
EXPNO    34
PROCNO    16
Date_    20080828
Time     9.24
INSTRUM   spect
PROBHD    5 mm QNP 1H/13
PULPROG   zgpg30
PCPDPRG2  pgdd
PCPDPRG1  gddz
DE      20
AQ      0
DQ      0
CROSS    1200.1461 Hz
F1FINDP   4.903153 Hz
AQ       0.2259000 Hz
GQ       2000
LC       122.1000 usec
NUC1      13
NUC2      13
NUC3      13
DELTA     290.7 Y
DELTA2    4.1000000
DELTA3    1.0700000
DELTA4    1.4713000
DELTA5    1.0500000 usec
DELTA6    2.0700000 usec
DELTA7    2.0700000 usec
===== CHANNEL f1 =====
NUC1      13
P1        8.40 usec
PD        17.40 usec
PC1       1.20 dB
RF1CH1    130.1026000 MHz
SFO1      125.7614489 MHz
===== CHANNEL f2 =====
NUC2      13
P2        3.20 usec
PD        4.20 dB
RF2CH1    101.6231000 MHz
SFO2      125.7614489 MHz
===== CHANNEL f3 =====
NAME      MAROCANIN
INSTRUM   spect
PROBHD    5 mm QNP 1H/13
PULPROG   zgpg30
PCPDPRG2  pgdd
PCPDPRG1  gddz
DE      20
AQ      0
DQ      0
CROSS    1200.1461 Hz
F1FINDP   4.903153 Hz
AQ       0.2259000 Hz
GQ       2000
LC       122.1000 usec
NUC1      13
NUC2      13
NUC3      13
DELTA     290.7 Y
DELTA2    4.1000000
DELTA3    1.0700000
DELTA4    1.4713000
DELTA5    1.0500000 usec
DELTA6    2.0700000 usec
DELTA7    2.0700000 usec
===== CHANNEL f1 =====
NUC1      13
P1        8.40 usec
PD        17.40 usec
PC1       1.20 dB
RF1CH1    130.1026000 MHz
SFO1      125.7614489 MHz
===== CHANNEL f2 =====
NUC2      13
P2        3.20 usec
PD        4.20 dB
RF2CH1    101.6231000 MHz
SFO2      125.7614489 MHz
===== CHANNEL f3 =====
NAME      MAROCANIN
INSTRUM   spect
PROBHD    5 mm QNP 1H/13
PULPROG   zgpg30
PCPDPRG2  pgdd
PCPDPRG1  gddz
DE      20
AQ      0
DQ      0
CROSS    1200.1461 Hz
F1FINDP   4.903153 Hz
AQ       0.2259000 Hz
GQ       2000
LC       122.1000 usec
NUC1      13
NUC2      13
NUC3      13
DELTA     290.7 Y
DELTA2    4.1000000
DELTA3    1.0700000
DELTA4    1.4713000
DELTA5    1.0500000 usec
DELTA6    2.0700000 usec
DELTA7    2.0700000 usec
===== CHANNEL f1 =====
NUC1      13
P1        8.40 usec
PD        17.40 usec
PC1       1.20 dB
RF1CH1    130.1026000 MHz
SFO1      125.7614489 MHz
===== CHANNEL f2 =====
NUC2      13
P2        3.20 usec
PD        4.20 dB
RF2CH1    101.6231000 MHz
SFO2      125.7614489 MHz
===== CHANNEL f3 =====

```

S12: Expansion, HMBC spectrum (CDCl₃, 500 MHz) of Compound **1** (Maroccanin)

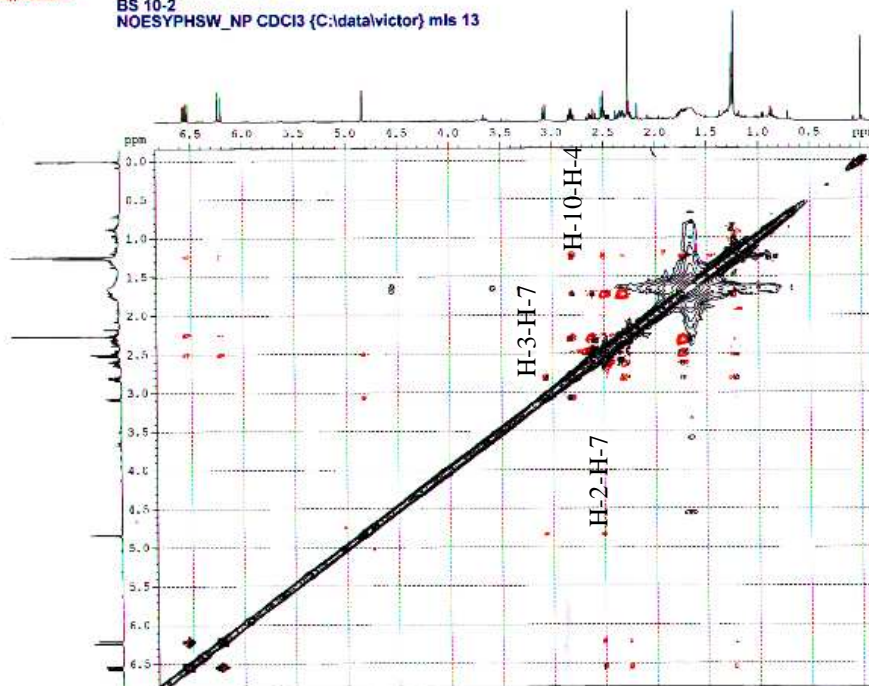


S13: Expansion, HMBC spectrum (CDCl₃, 500 MHz) of Compound 1(Maroccanin)

IPNA-CSIC
RMN-500MHz

OperadorMLS victor JB
BS 10-2
NOESYPSHW_NP CDCl3 {C:\data\victor} mls 13

noesyphsw



```
=====
NAME          ml 10-2
EXPNO         12
PROCNO        1
F2          500.131
TIME         12.52
INSTRUM       spect
PROBHD       5 mm BBI
PULPROG       zgpg30
TD           65536
SFO         500.131 MHz
AQ         0.0011198 sec
RG           692
WDW           EM
SSB           0
GB           0.0018517 sec
PC           0.0003002 sec
SC           0.0001001 sec
SD           0.0003450 sec
=====
=====
NAME          ml
EXPNO         6
PROCNO        1
F2          500.131
TIME         12.52
INSTRUM       spect
PROBHD       5 mm BBI
PULPROG       zgpg30
TD           65536
SFO         500.131 MHz
AQ         0.0011198 sec
RG           692
WDW           EM
SSB           0
GB           0.0018517 sec
PC           0.0003002 sec
SC           0.0001001 sec
SD           0.0003450 sec
=====
=====
NAME          ml
EXPNO         12
PROCNO        1
F2          500.131
TIME         12.52
INSTRUM       spect
PROBHD       5 mm BBI
PULPROG       zgpg30
TD           65536
SFO         500.131 MHz
AQ         0.0011198 sec
RG           692
WDW           EM
SSB           0
GB           0.0018517 sec
PC           0.0003002 sec
SC           0.0001001 sec
SD           0.0003450 sec
=====
=====
NAME          ml
EXPNO         12
PROCNO        1
F2          500.131
TIME         12.52
INSTRUM       spect
PROBHD       5 mm BBI
PULPROG       zgpg30
TD           65536
SFO         500.131 MHz
AQ         0.0011198 sec
RG           692
WDW           EM
SSB           0
GB           0.0018517 sec
PC           0.0003002 sec
SC           0.0001001 sec
SD           0.0003450 sec
=====
```

S14: NOESY spectrum (CDCl₃, 500 MHz) of Compound 1 (Maroccanin)

Elemental Composition Report

Multiple Mass Analysis: 45 mass(es) processed

Tolerance = 5.0 PPM / DBE: min = 0.0, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

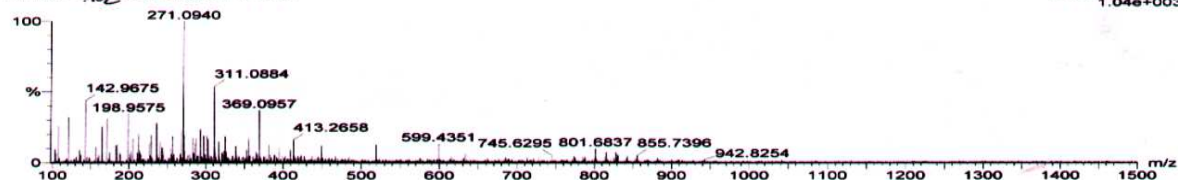
8633 formula(e) evaluated with 23 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-90 H: 0-150 N: 0-1 O: 0-40 Na: 0-1

Inaki (B345) 30 (1.075) Cm (27:32)

2: TOF MS ES+
1.04e+003



Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
801.6837	10.03	801.6820	1.7	2.1	1.5	4.3	C47 H93 O9
599.4351	13.53	---	---	---	---	---	---
519.1981	12.65	519.1995	-1.4	-2.7	12.5	7.2	C28 H32 O8 Na
449.3686	12.14	519.1960	2.1	4.0	24.5	14.3	C37 H27 O3
413.2658	16.10	413.2668	-1.0	-2.4	5.5	2.6	C24 H38 O4 Na
395.1501	10.63	395.1495	0.6	1.5	12.5	10.3	C23 H23 O6
381.1711	12.78	381.1702	0.9	2.4	11.5	38.0	C23 H25 O5
369.0957	37.10	369.0950	0.7	1.9	9.5	7.6	C18 H18 O7 Na
356.0744	17.16	369.0974	-1.7	-4.6	12.5	11.2	C20 H17 O7
339.0844	11.78	356.0746	-0.2	-0.6	9.5	0.2	C16 H15 N O7 Na
325.0971	18.69	339.0845	-0.1	-0.3	9.5	25.3	C17 H16 O6 Na
317.1362	14.93	---	---	---	---	---	---
311.2930	10.83	317.1365	-0.3	-0.9	5.5	8.5	C16 H22 O5 Na
311.0884	53.73	---	---	---	---	---	---
303.1237	17.00	311.0895	-1.1	-3.5	8.5	6.5	C16 H16 O5 Na
301.1411	19.31	303.1232	0.5	1.6	8.5	23.7	C17 H19 O5
297.0823	19.09	301.1416	-0.5	-1.7	5.5	3.1	C16 H22 O4 Na
293.0784	23.62	297.0822	0.1	0.3	2.5	4.1	C10 H17 O10
287.0692	17.38	293.0790	-0.6	-2.0	9.5	9.8	C16 H14 O4 Na
286.9716	13.19	287.0684	0.8	2.8	11.5	5.4	C17 H12 O3 Na
283.0813	12.27	---	---	---	---	---	---
272.1001	19.21	283.0818	-0.5	-1.8	6.5	2.1	C13 H15 O7
271.0940	100.00	271.0946	-0.6	-2.2	6.5	27.1	C14 H16 O4 Na
269.0497	23.23	269.0509	-1.2	-4.5	2.5	0.7	C8 H13 O10
256.9896	9.68	---	---	---	---	---	---
254.9895	10.16	254.9906	-1.1	-4.5	9.5	14.6	C11 H4 O6 Na
244.9069	10.34	---	---	---	---	---	---
242.9832	10.82	---	---	---	---	---	---
241.0407	12.90	---	---	---	---	---	---
240.9837	14.83	240.9832	0.5	2.1	3.5	75.5	C5 H5 O11
236.0708	27.94	236.0712	-0.4	-1.7	11.5	5.0	C15 H10 N O2
228.9693	19.45	---	---	---	---	---	---
226.9874	14.23	---	---	---	---	---	---
212.9730	20.37	---	---	---	---	---	---
204.9138	16.87	---	---	---	---	---	---
198.9946	11.57	---	---	---	---	---	---
198.9575	34.46	---	---	---	---	---	---
184.9789	12.71	---	---	---	---	---	---
182.9618	12.03	---	---	---	---	---	---
170.9630	31.06	---	---	---	---	---	---

S15 : High resolution (ESI+) Mass Spectrum of compound 1(Maroccanin)