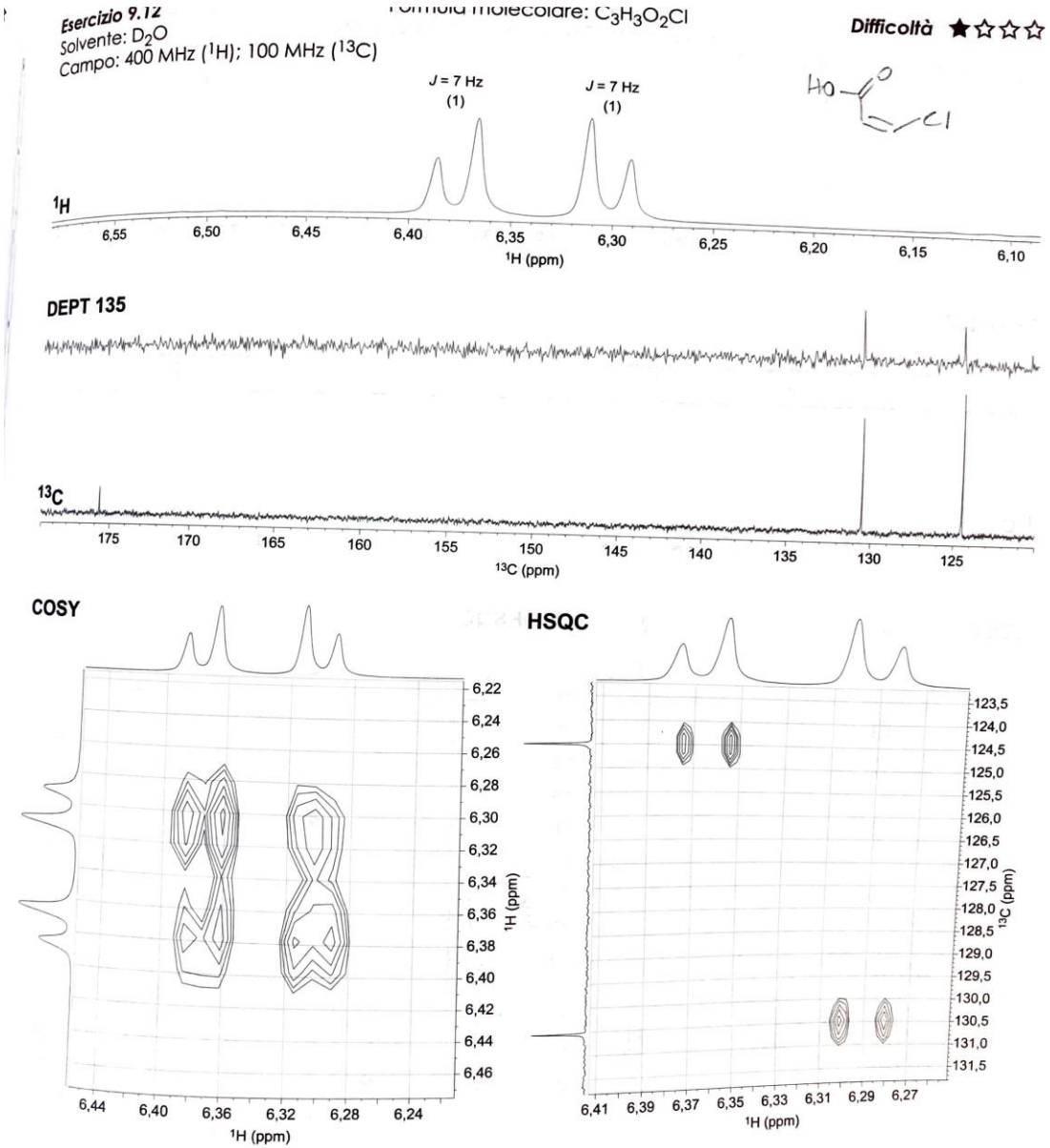
TABLE 10.1 Spectroscopic Techniques and Their Applications in Structure Elucidation of Organic Molecules

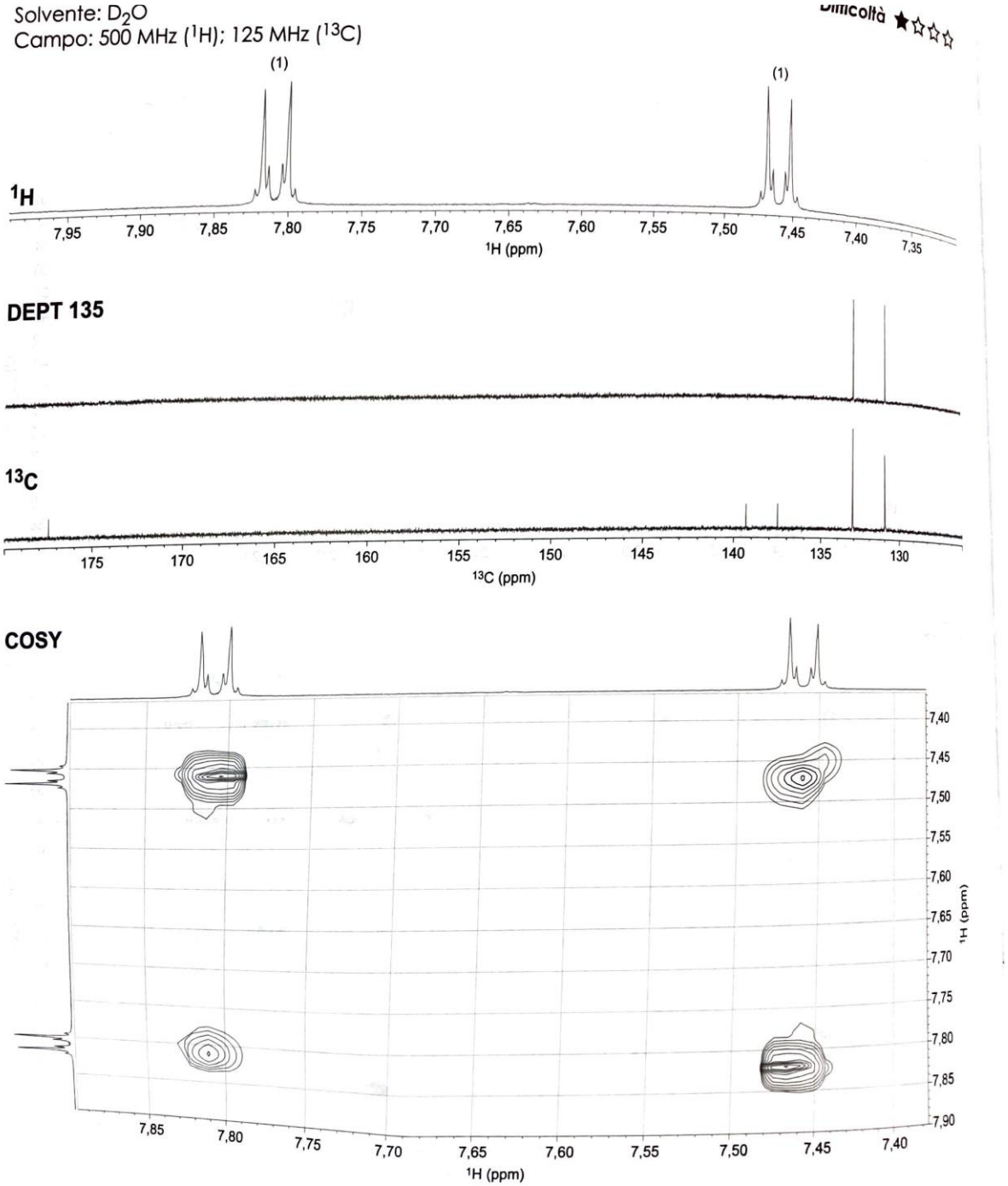
Spectroscopic Techniques	Radiation Absorbed	Effect on the Molecule	Structural Information Deduced
Ultraviolet/Visible spectrophotometry	Ultraviolet-visible λ, 190–400 nm and 400–800 nm	Changes in electronic energy levels within the molecule	Extent of π-electron systems. Presence of conjugated unsaturation, and conjugation with nonbonding electrons
Infrared spectrophotometry	Infrared (mid infrared) λ, 2.5–25 mm ν, 400–4000 cm^{-1}	Changes in the vibrational and rotational movements of the molecule	Detection of functional groups, which have specific vibration frequencies, for example, C═O, NH_2, OH, etc.
NMR spectroscopy	Radiofrequency λ, 25 cm	Nuclei placed under the static magnetic field change their spins after absorption of radiofrequency radiations	The electronic environment of nuclei, their numbers and number of neighboring atoms
Circular dichroism (CD)/Optical rotatory dispersion (ORD)	Ultraviolet/visible λ 200–600 nm	Changes in the electronic energy level of a molecule	Identification of absolute configurations of small molecules in solution
Mass spectrometry	High-speed electron beam and other ionization sources	Effects of ionization and fragmentation of charged particles	Relative masses of molecular ions and fragments

TECNICHE NMR PIU' USATE E LORO UTILIZZO

1. 1D-^{1}H NMR: informazioni su chemical shift, J e integrali.
2. 2D-COSY: identifica gli accoppiamenti H-H
3. 1D ^{13}C NMR (BBD) permette di identificare il numero di atomi di C e il loro chemical shift
4. 1D-DEPT: informazioni sulla molteplicità
5. 2D-HSQC (HMQC) identifica gli accoppiamenti C-H diretti (^{1}J)
6. 2D-HMBC identifica accoppiamenti C-H geminali (^{2}J) o vicinali (^{3}J)
7. 2D-NOESY, 1D DIFNOE o 1D NOESY: informazioni su relazioni spaziali
8. 2D-NOESY e ROESY: identifica relazioni attraverso scambi chimici o conformazionali
9. TOCSY: Identifica sistemi di spin separati
10. INADEQUATE: informazioni su connettività ^{13}C-^{13}C



Sistema di spin?



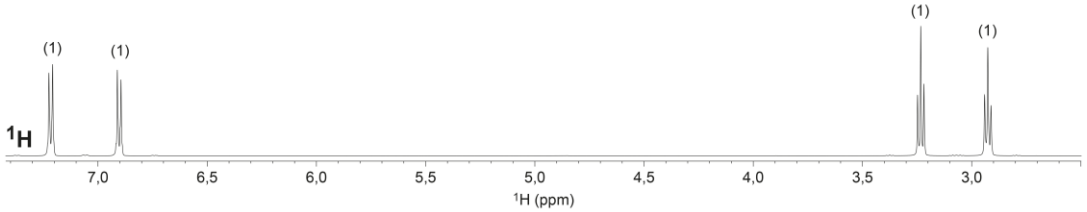
Acido p-clorobenzoico

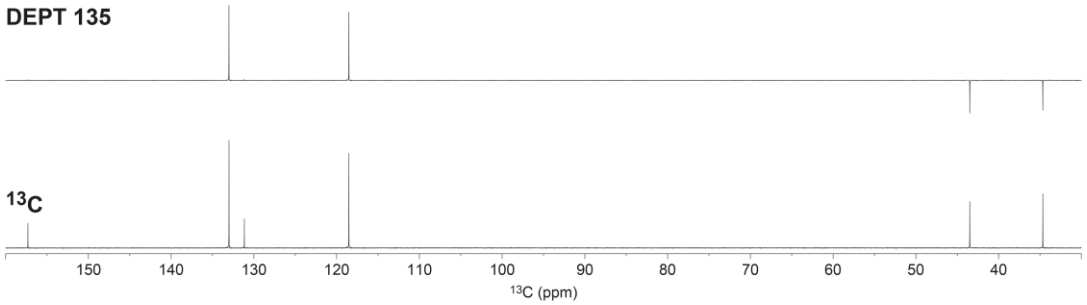
Sistema di spin?

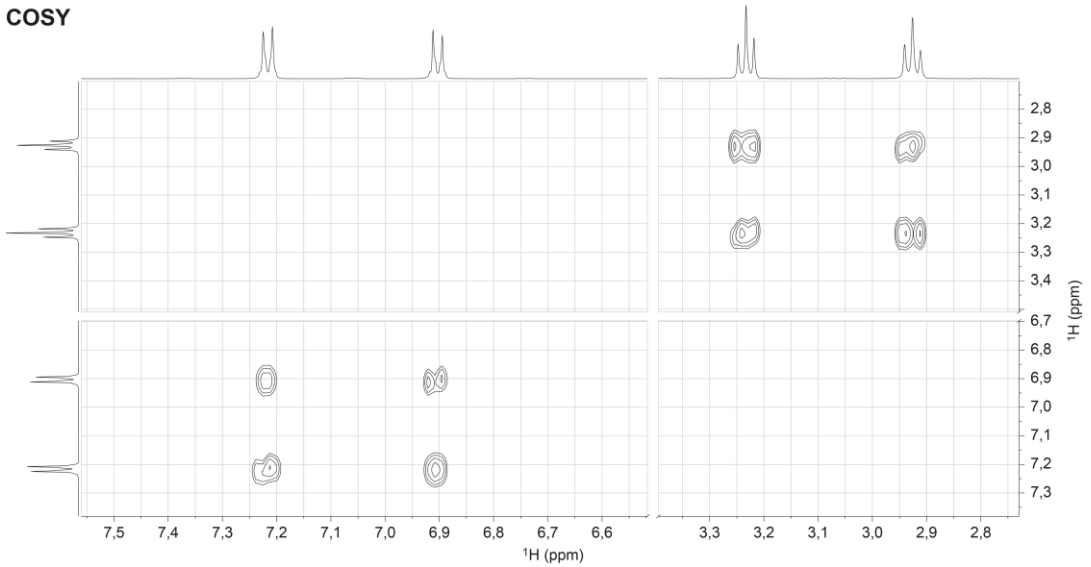
Esercizio 8.11 Formula molecolare: $C_8H_{11}NO$ ***Difficoltà*** ★★☆☆

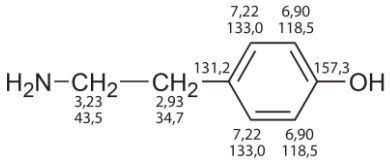
Solvente: D_2O

Campo: 500 MHz (^1H); 125 MHz (^{13}C)



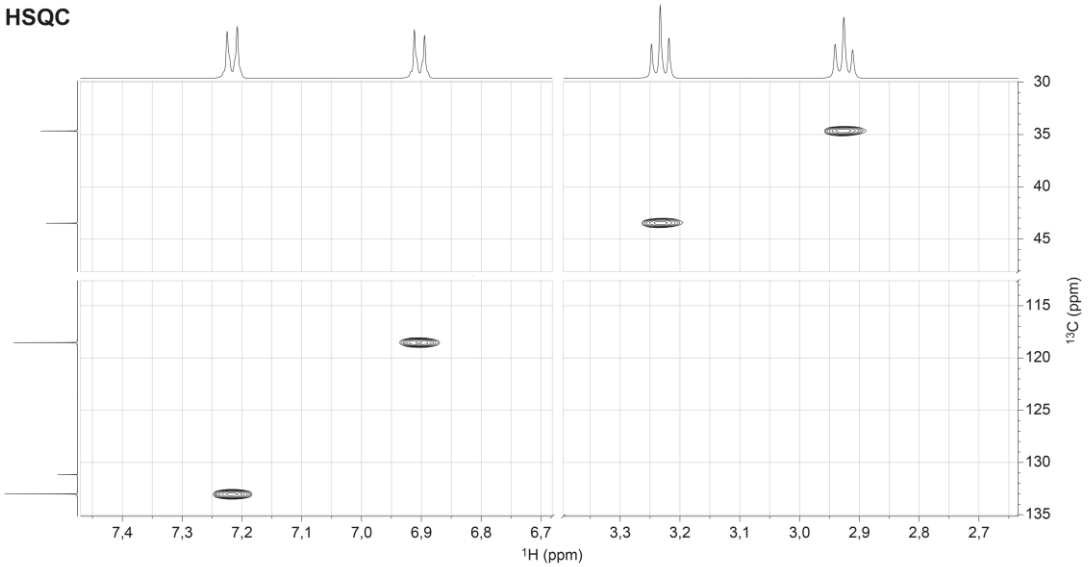


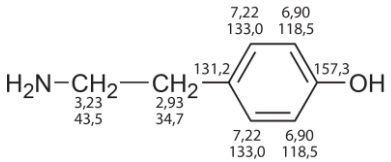




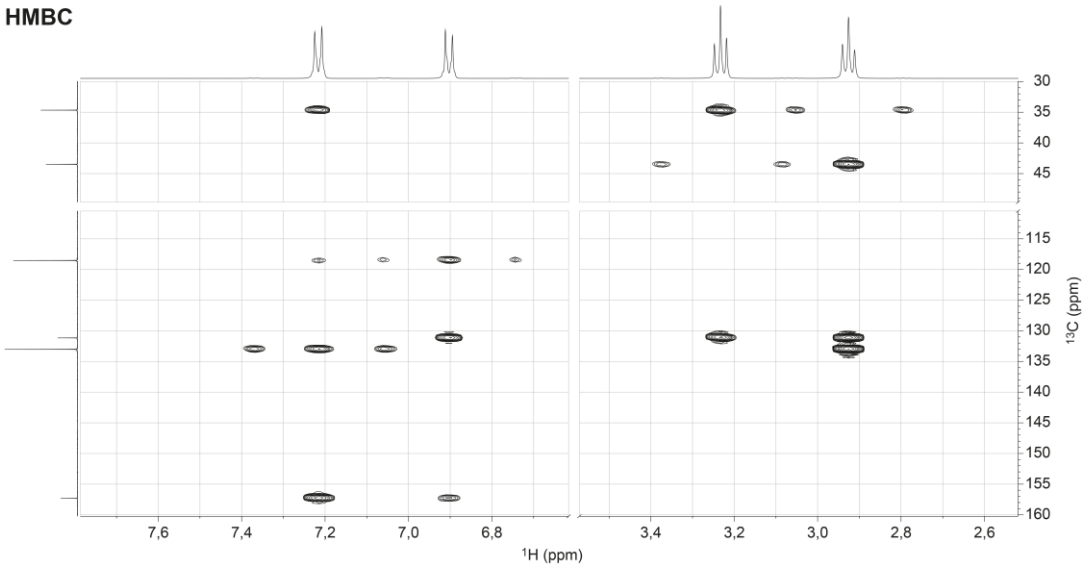
Sistemi di spin?

HSQC


30
35
40
45
115
120
125
130
135
7,4
7,3
7,2
7,1
7,0
6,9
6,8
6,7
3,3
3,2
3,1
3,0
2,9
2,8
2,7
^{1}H (ppm)
^{13}C (ppm)


7,22
133,0
6,90
118,5
131,2
157,3
$H_2N-CH_2-CH_2$
OH
3,23
43,5
2,93
34,7
7,22
133,0
6,90
118,5

HMBC


30
35
40
45
115
120
125
130
135
140
145
150
155
160
7,6
7,4
7,2
7,0
6,8
3,4
3,2
3,0
2,8
2,6
^{1}H (ppm)
^{13}C (ppm)

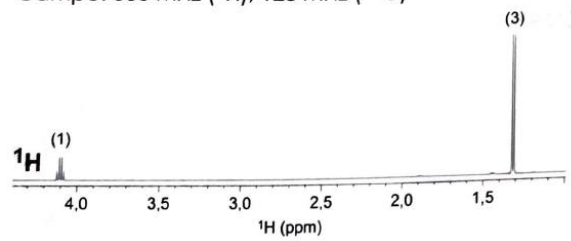
Esercizio 9.11

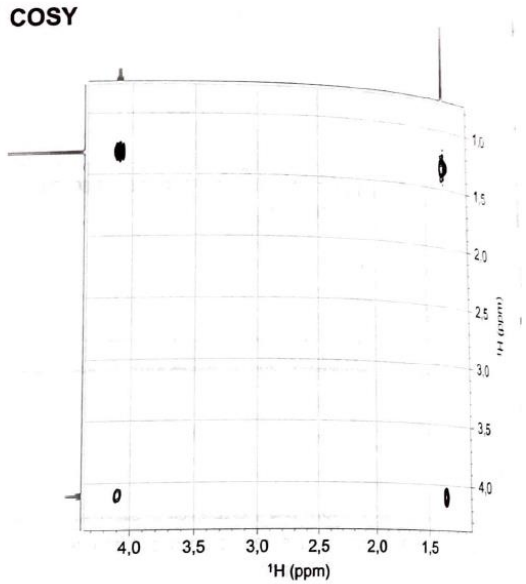
Formula molecolare: $C_3H_6O_3$

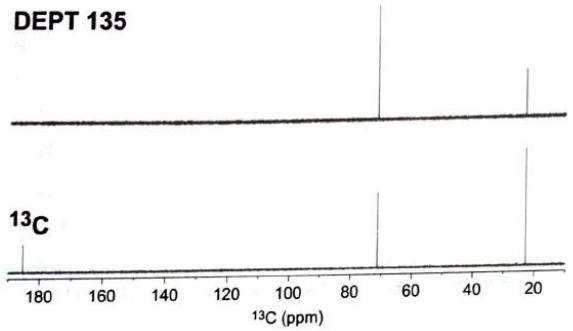
Difficoltà ★☆☆☆

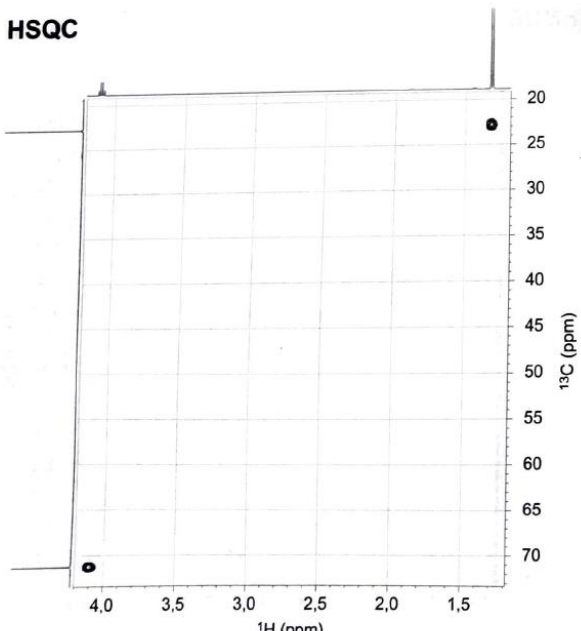
Solvente: D_2O

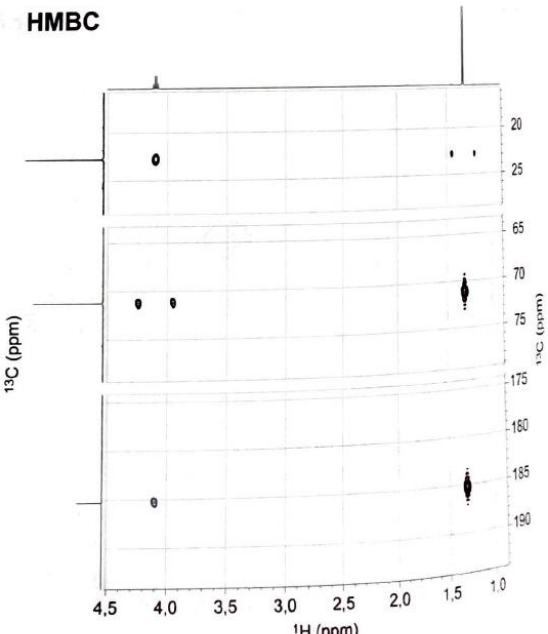
Campo: 500 MHz (^1H); 125 MHz (^{13}C)









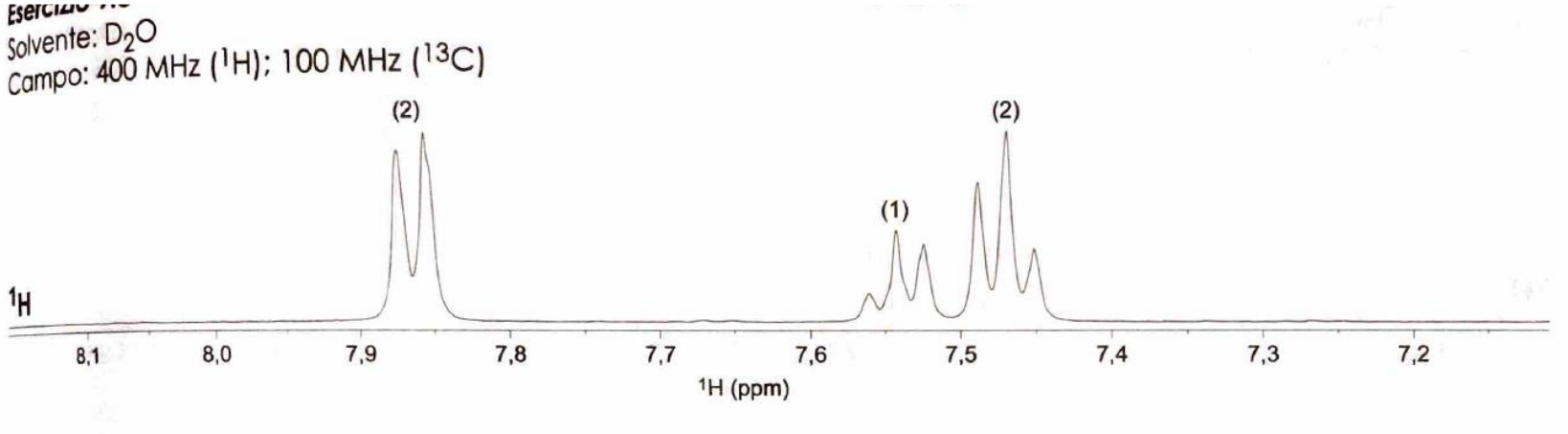


$CH_3CHCOOH$

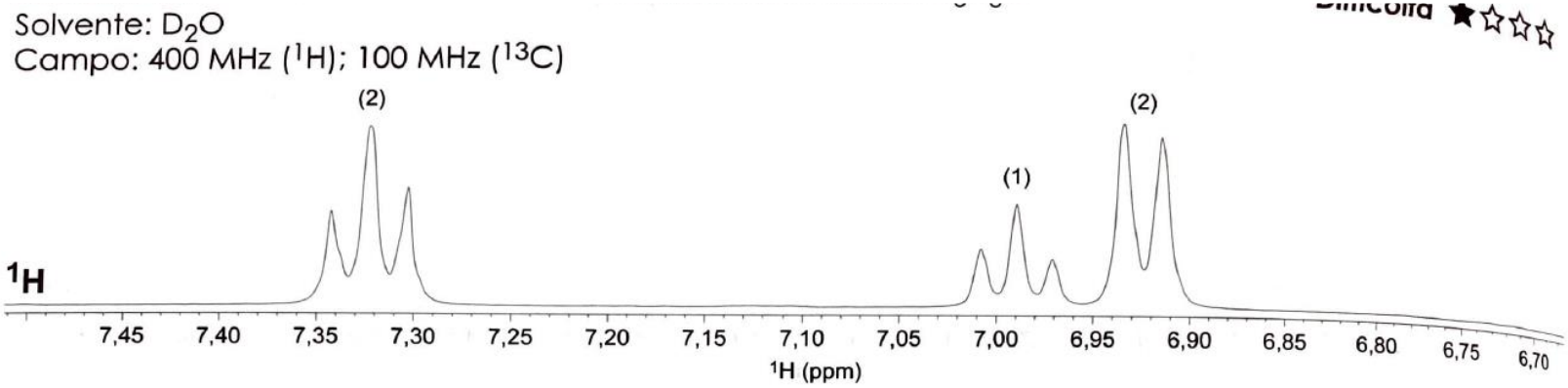
OH

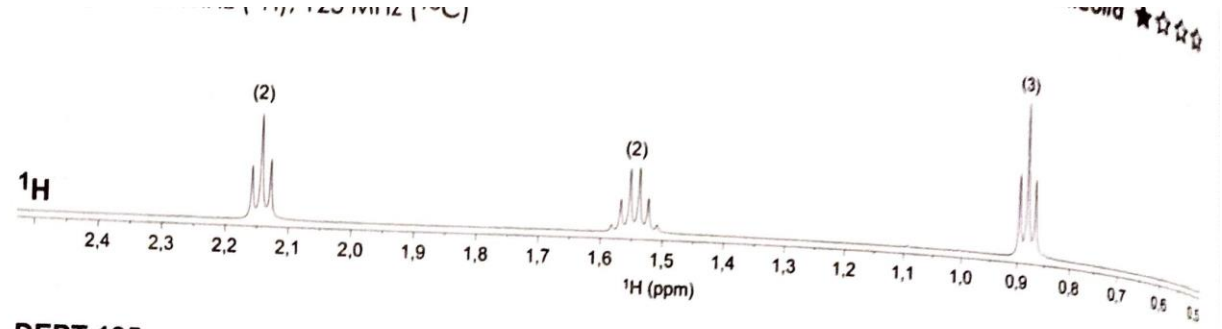
In D2O

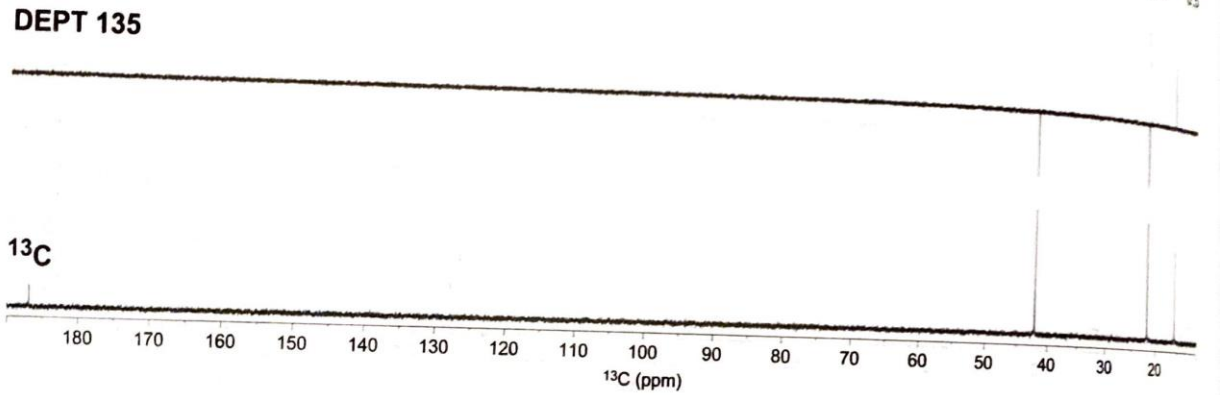
Sistema di spin?

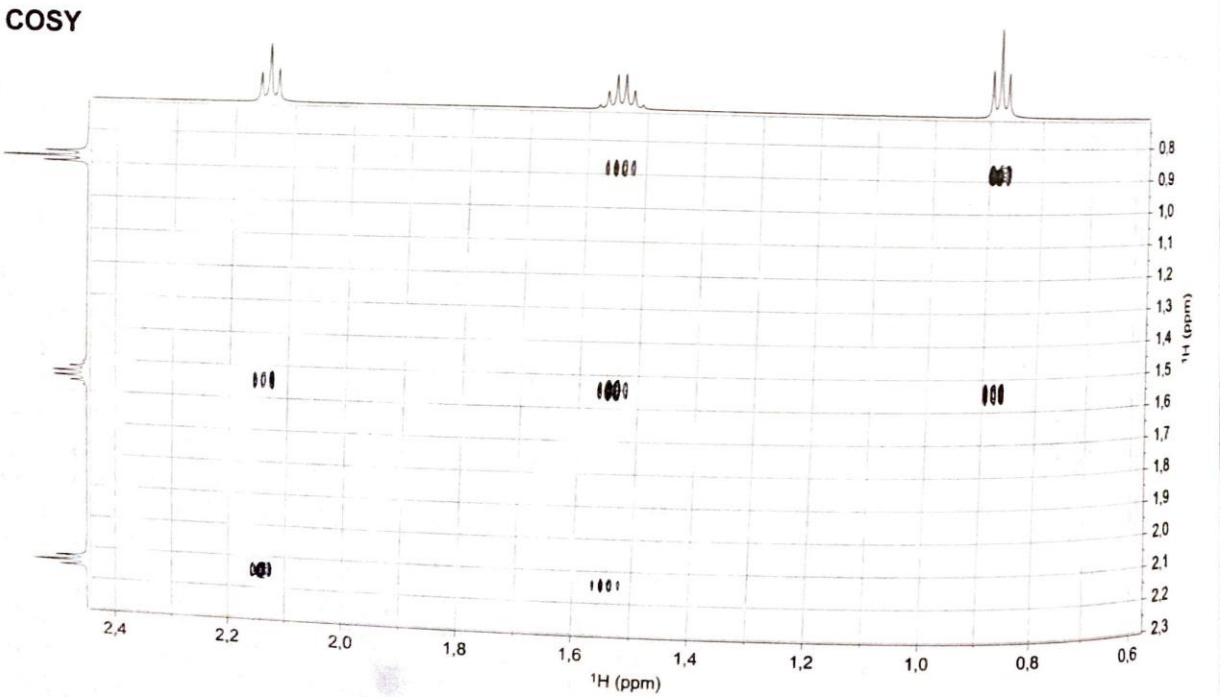


Fenolo o acido benzoico?







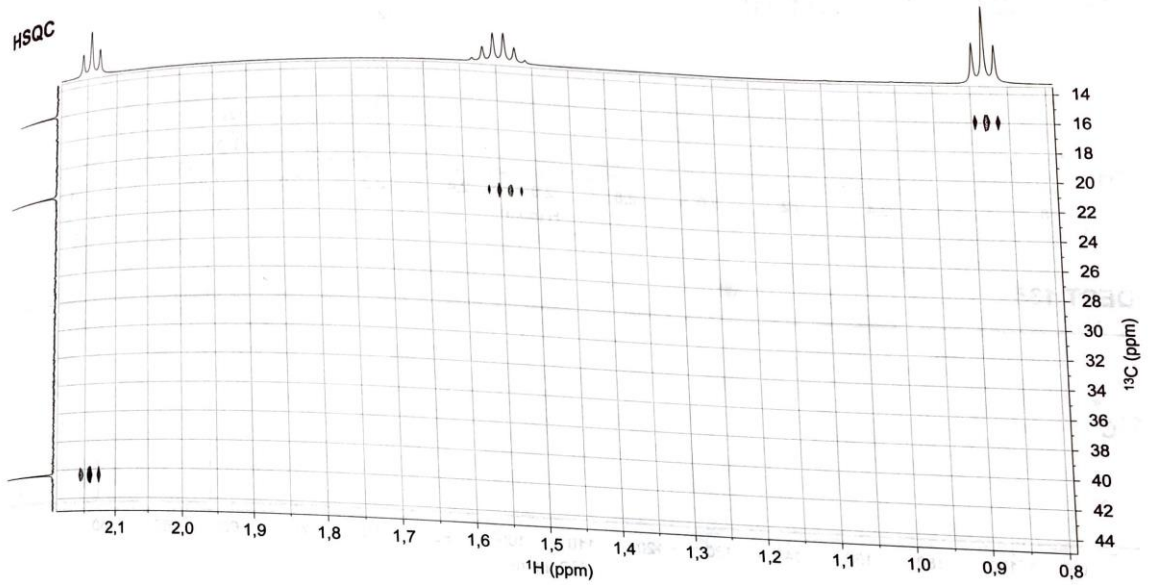


$CH_3CH_2CH_2COOH$

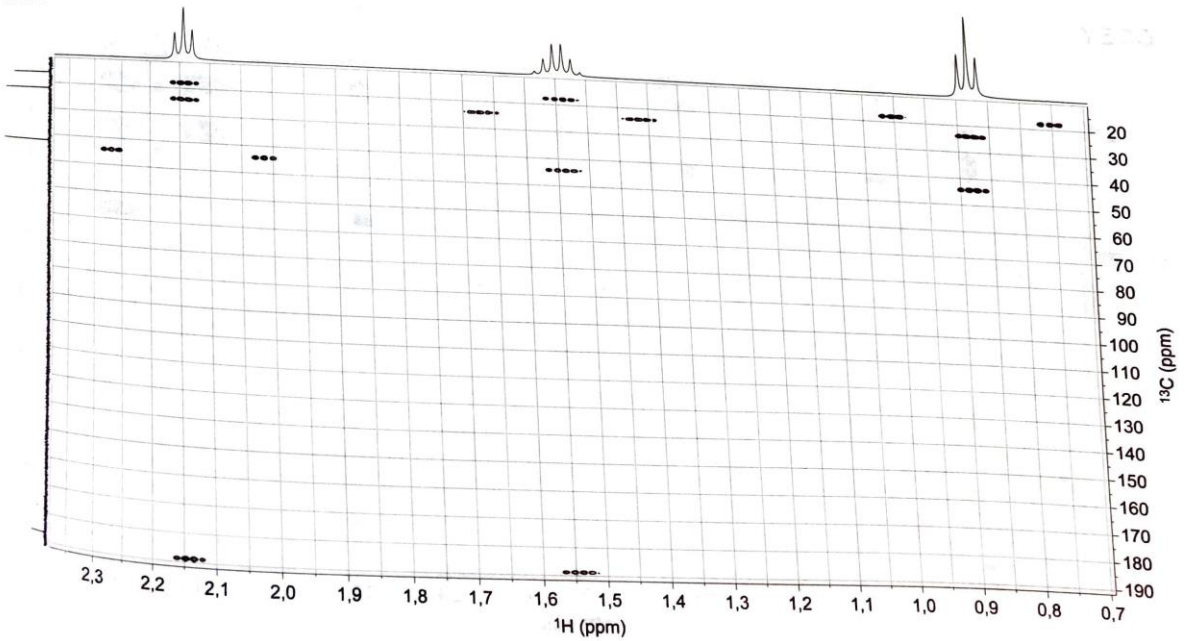
In D2O


OH

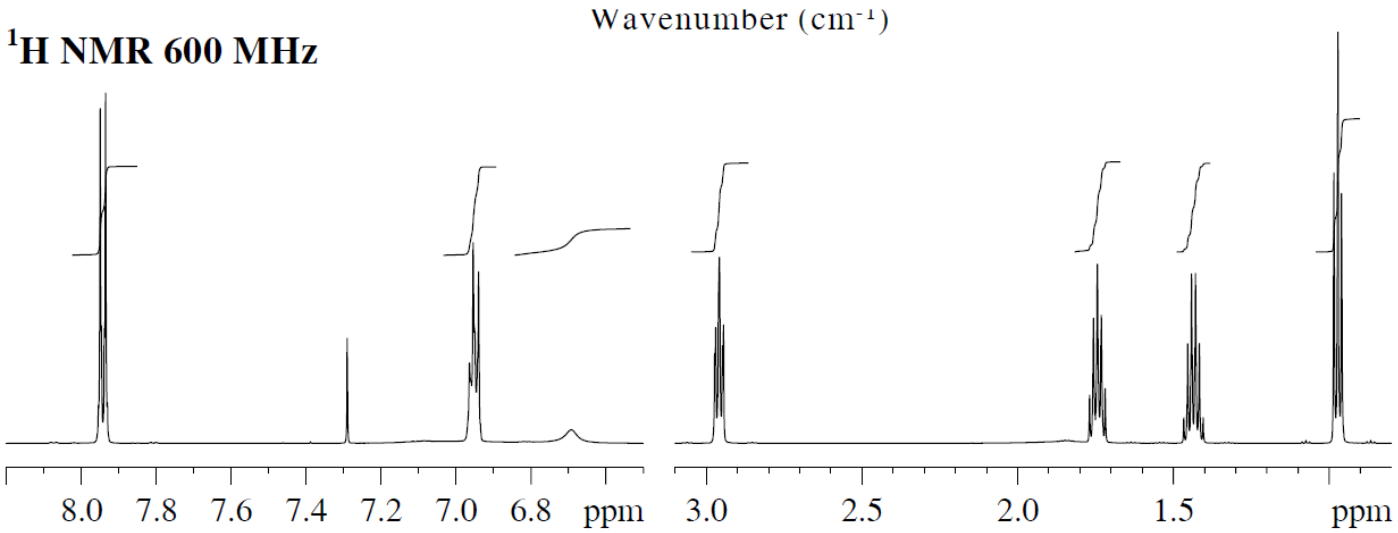
HSQC


14
16
18
20
22
24
26
28
30
32
34
36
38
40
42
44
13C (ppm)
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
1H (ppm)

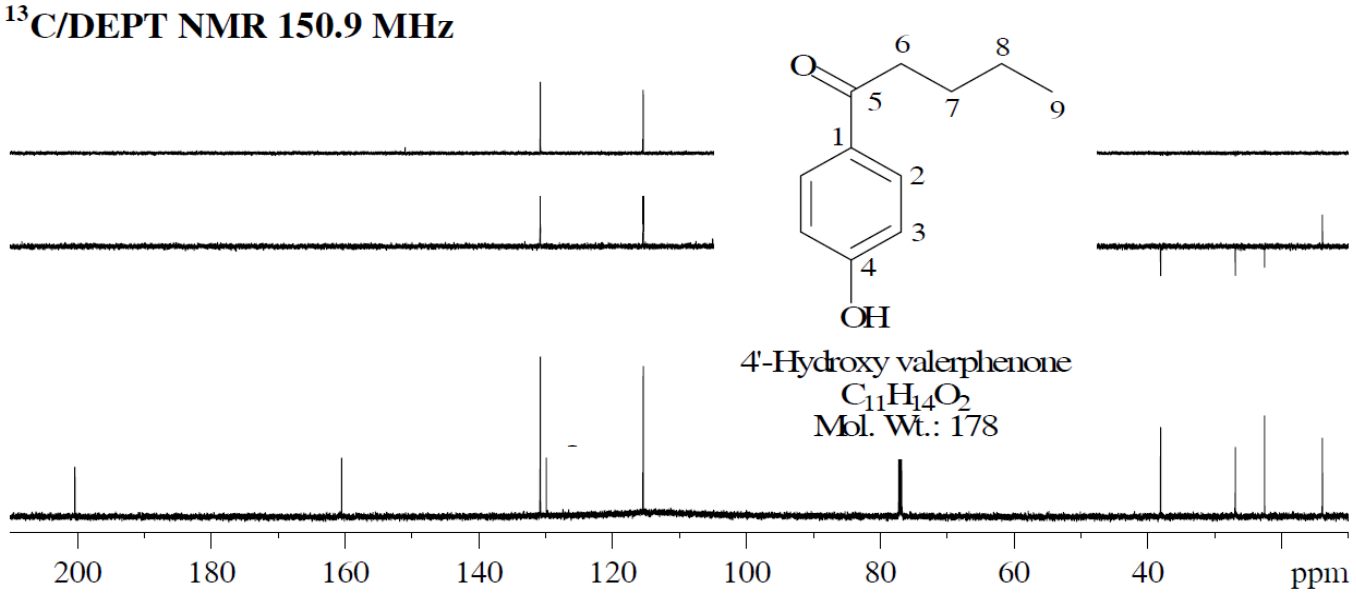
HMBC


20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
13C (ppm)
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
0,7
1H (ppm)

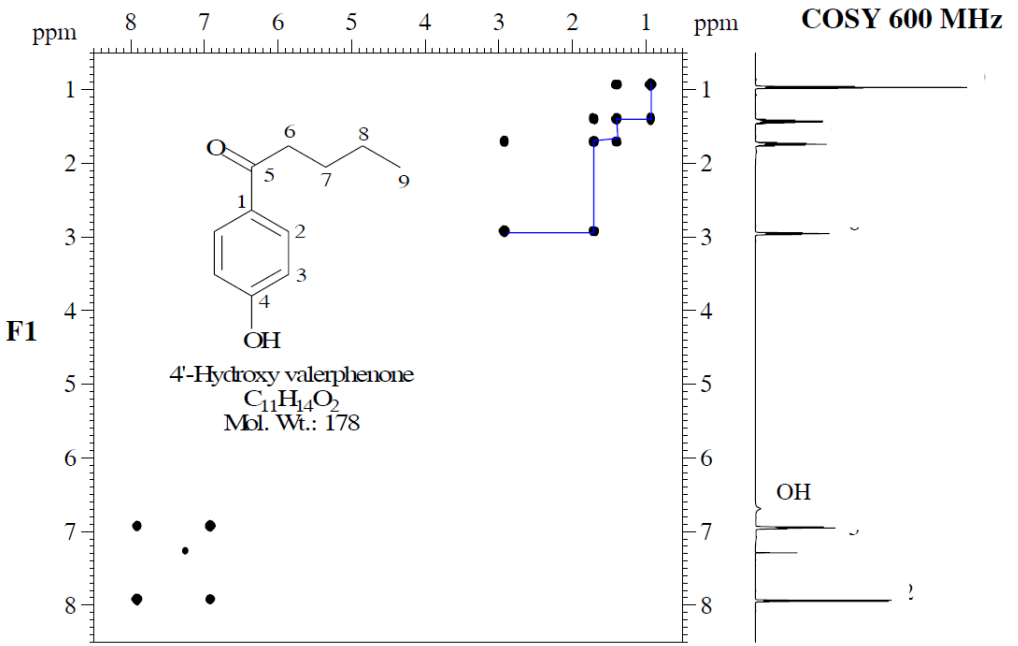
ES. 1

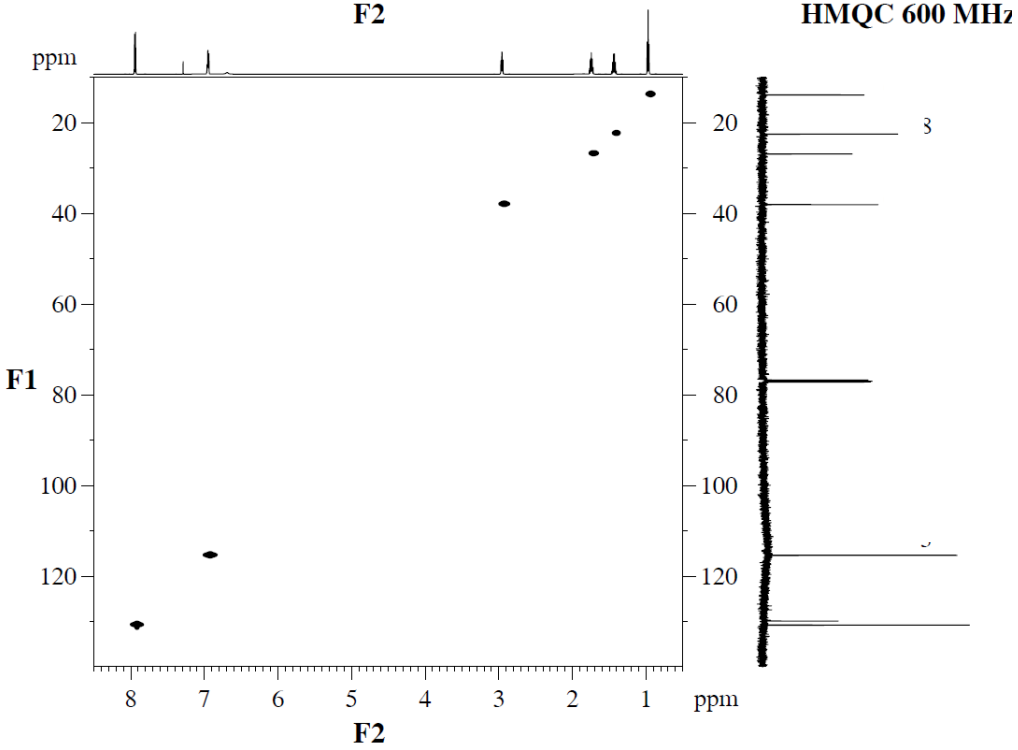

Wavenumber (cm⁻¹)
¹H NMR 600 MHz
8.0 7.8 7.6 7.4 7.2 7.0 6.8 ppm
3.0 2.5 2.0 1.5 ppm

In CDCl3

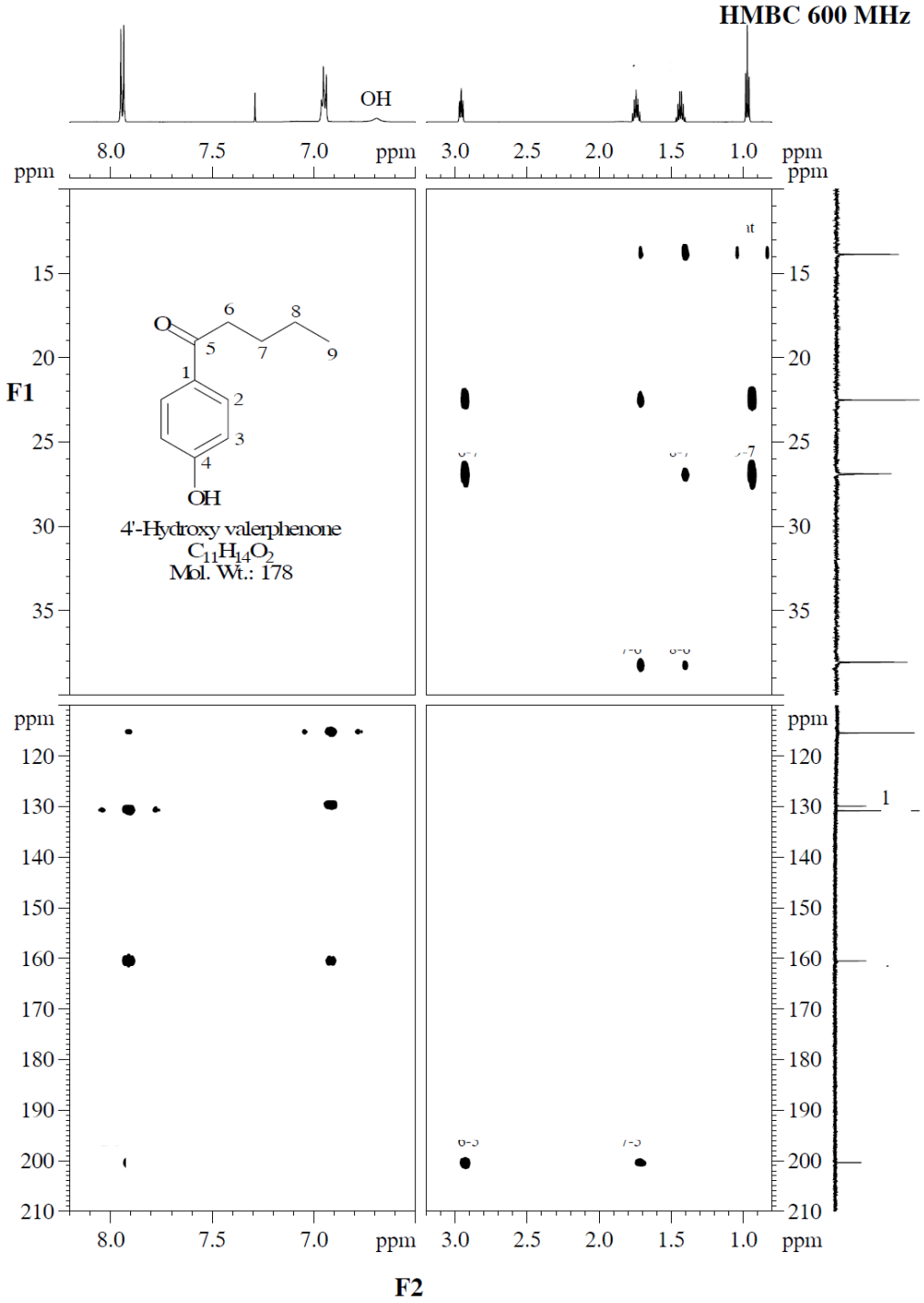

¹³C/DEPT NMR 150.9 MHz
O
OH
1 2 3 4 5 6 7 8 9
4'-Hydroxy valerphenone
C11H14O2
Mol. Wt.: 178
200 180 160 140 120 100 80 60 40 ppm

ES. 1


COSY 600 MHz
ppm
F1
F2
OH
4'-Hydroxy valerphenone
$C_{11}H_{14}O_2$
Mol. Wt.: 178


HMQC 600 MHz
ppm
F1
F2

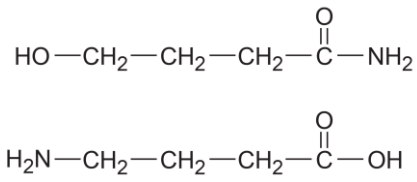
ES. 1


HMBC 600 MHz
OH
8.0
7.5
7.0
ppm
3.0
2.5
2.0
1.5
1.0
F1
F2
4'-Hydroxy valerphenone
$C_{11}H_{14}O_2$
Mol. Wt.: 178

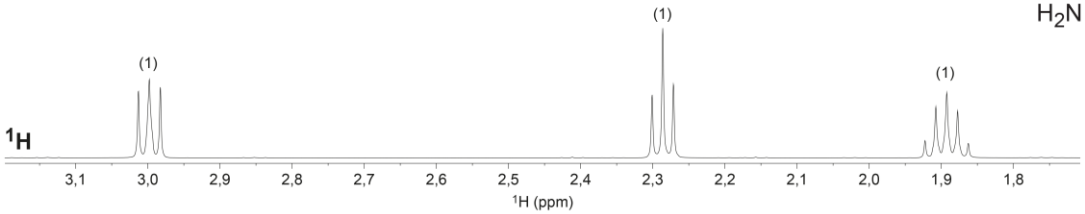
Esercizio 8.9 Formula molecolare: $C_4H_9NO_2$ ***Difficoltà*** ★

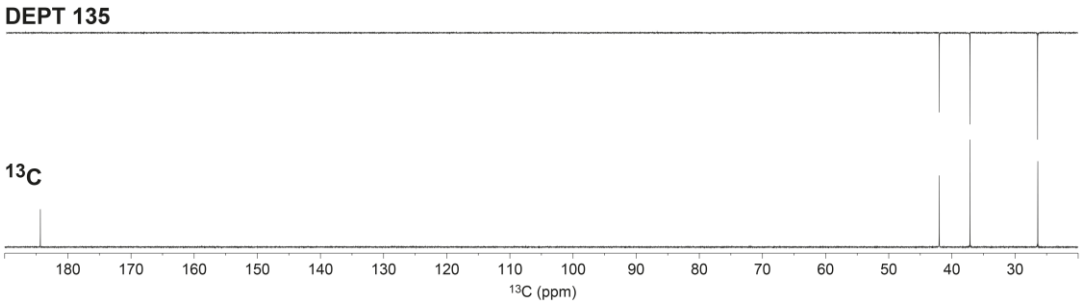
Solvente: D_2O

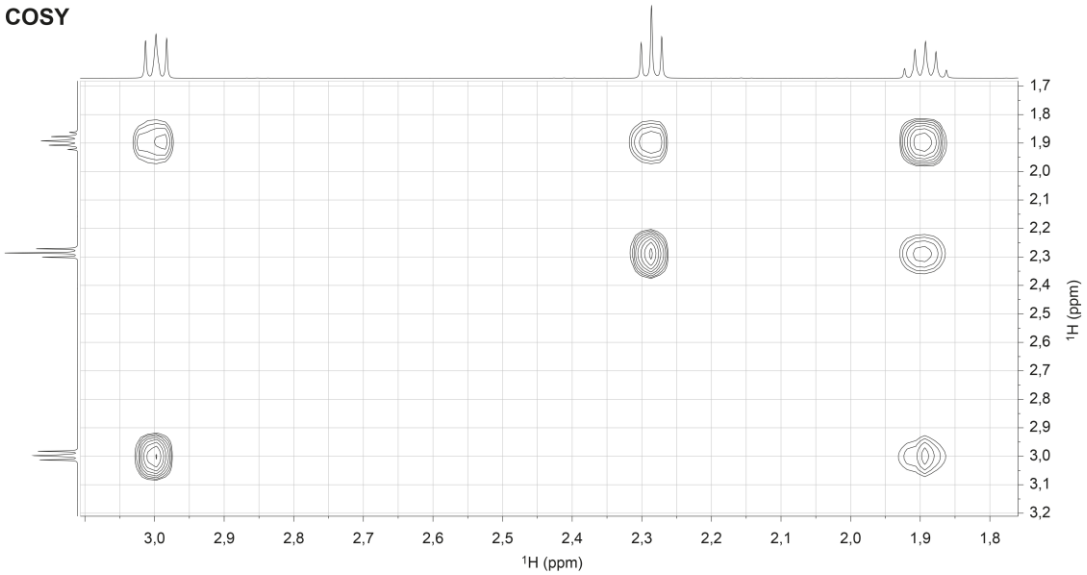
Campo: 500 MHz (^1H); 125 MHz (^{13}C)



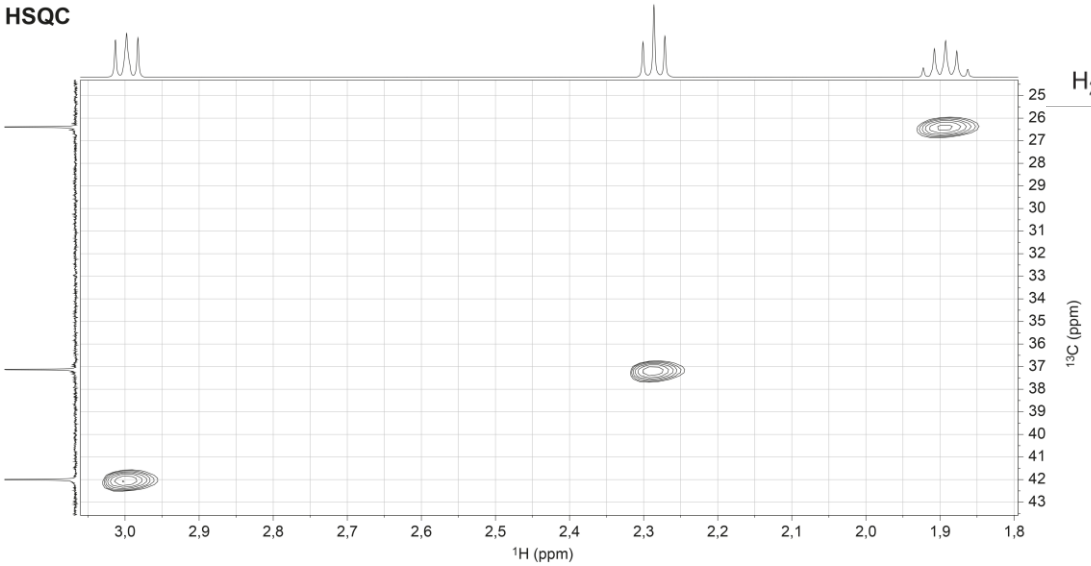
In D2O

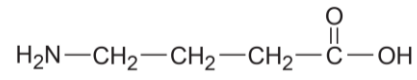




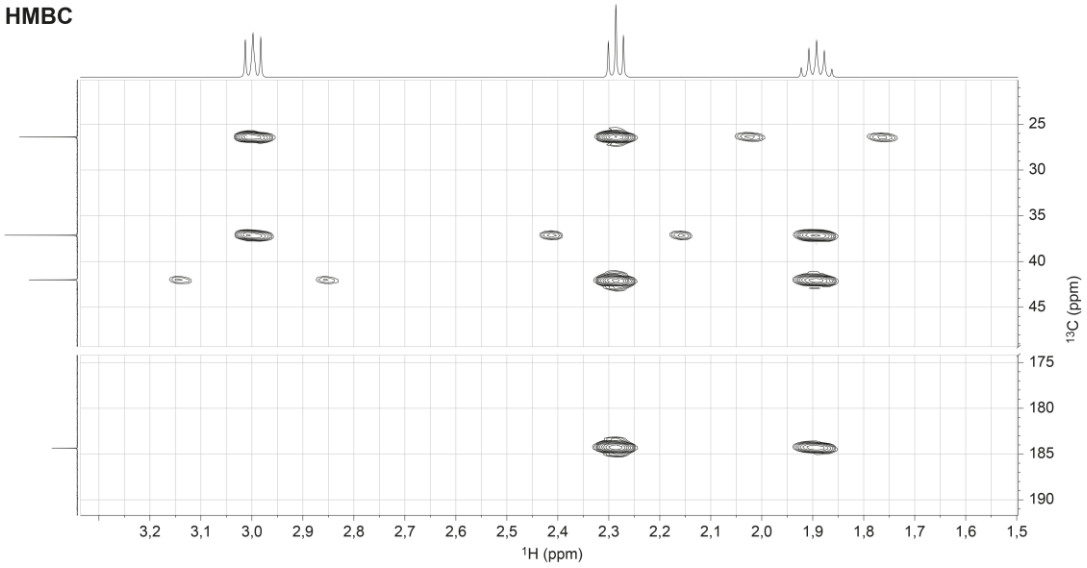


HSQC


25
26
27
28
29
30
31
32
33
34
35
36
37
38
39
40
41
42
43
^{13}C (ppm)
3,0
2,9
2,8
2,7
2,6
2,5
2,4
2,3
2,2
2,1
2,0
1,9
1,8
^{1}H (ppm)

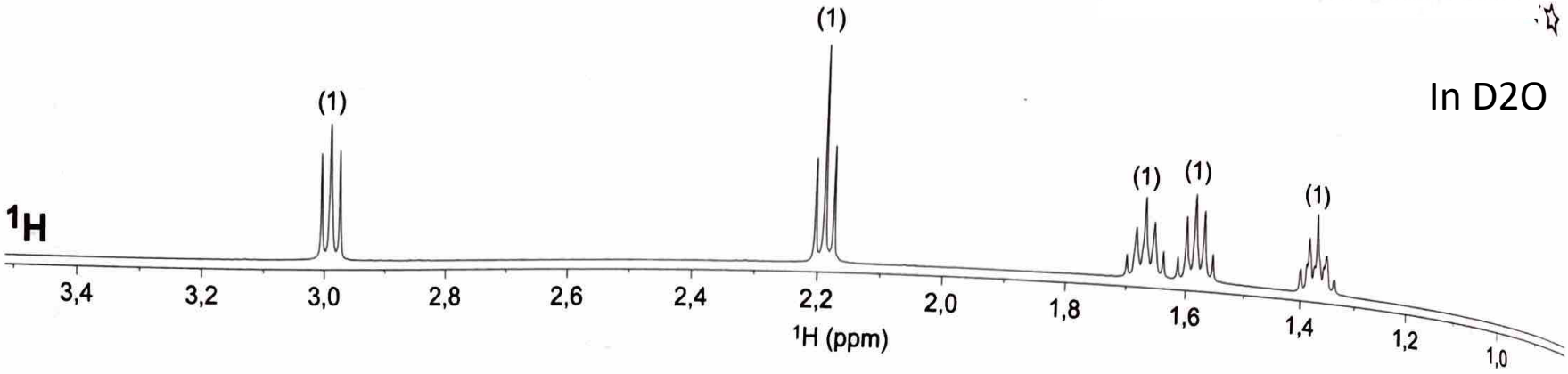

O
H_2N—CH_2—CH_2—CH_2—C—OH

HMBC

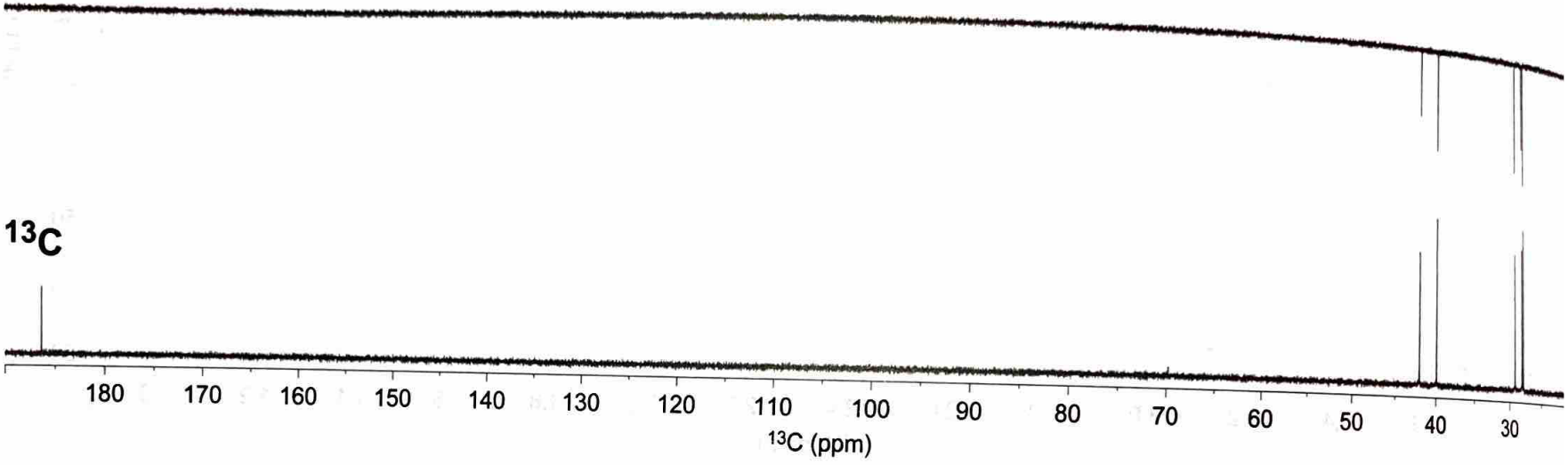

25
30
35
40
45
175
180
185
190
^{13}C (ppm)
3,2
3,1
3,0
2,9
2,8
2,7
2,6
2,5
2,4
2,3
2,2
2,1
2,0
1,9
1,8
1,7
1,6
1,5
^{1}H (ppm)

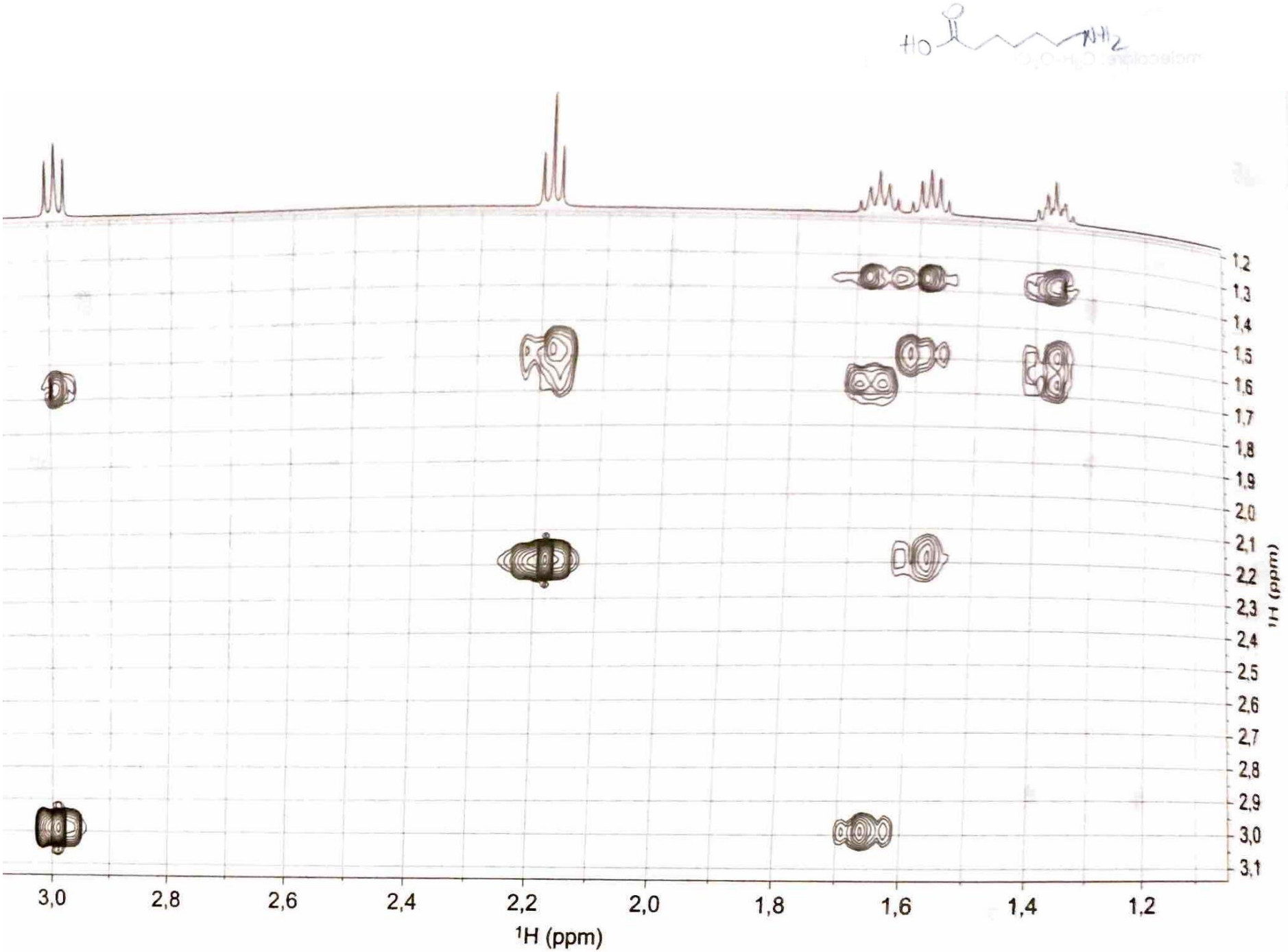
Campo: 500 MHz (^{1}H); 125 MHz (^{13}C)

In D2O

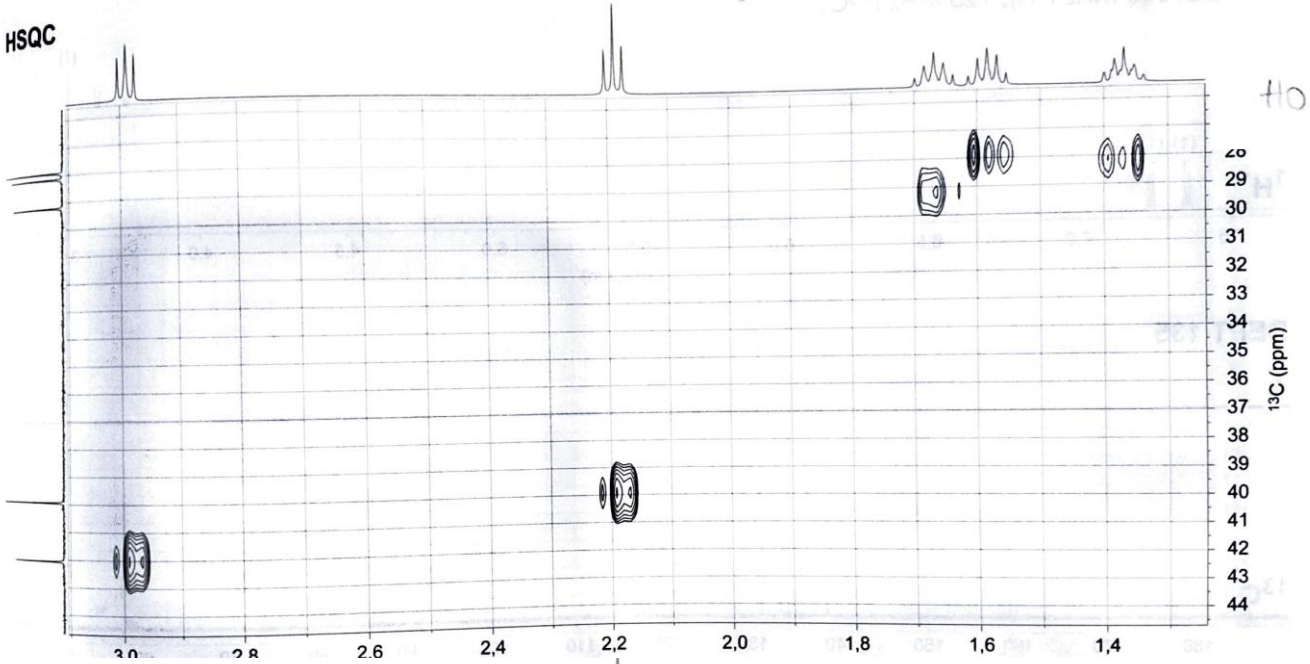


DEPT 135



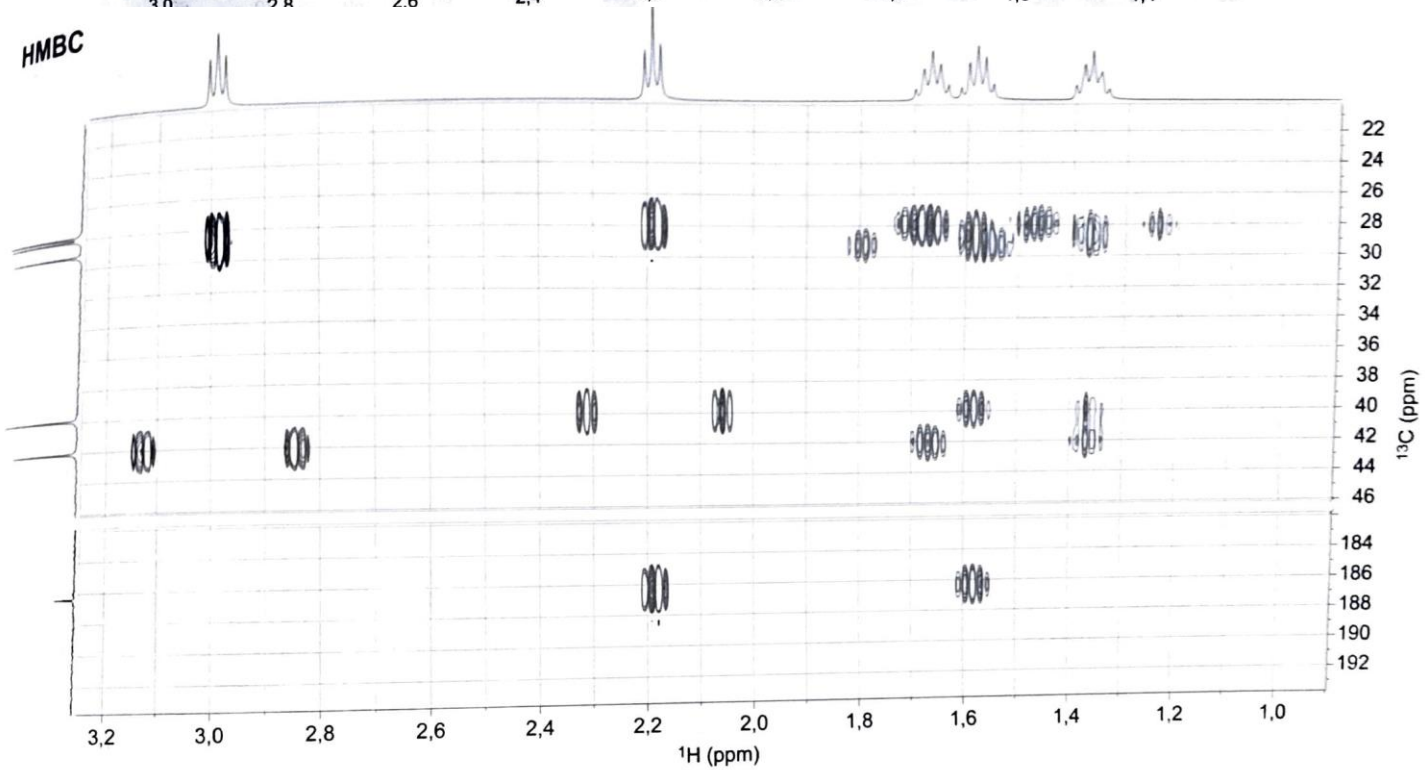

HO
NH2
3,0
2,8
2,6
2,4
2,2
2,0
1,8
1,6
1,4
1,2
¹H (ppm)
1,2
1,3
1,4
1,5
1,6
1,7
1,8
1,9
2,0
2,1
2,2
2,3
2,4
2,5
2,6
2,7
2,8
2,9
3,0
3,1
¹H (ppm)

HSQC


28
29
30
31
32
33
34
35
36
37
38
39
40
41
42
43
44
^{13}C (ppm)
3,0
2,8
2,6
2,4
2,2
2,0
1,8
1,6
1,4

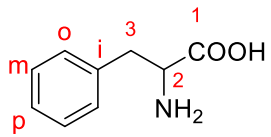

HO
NH2

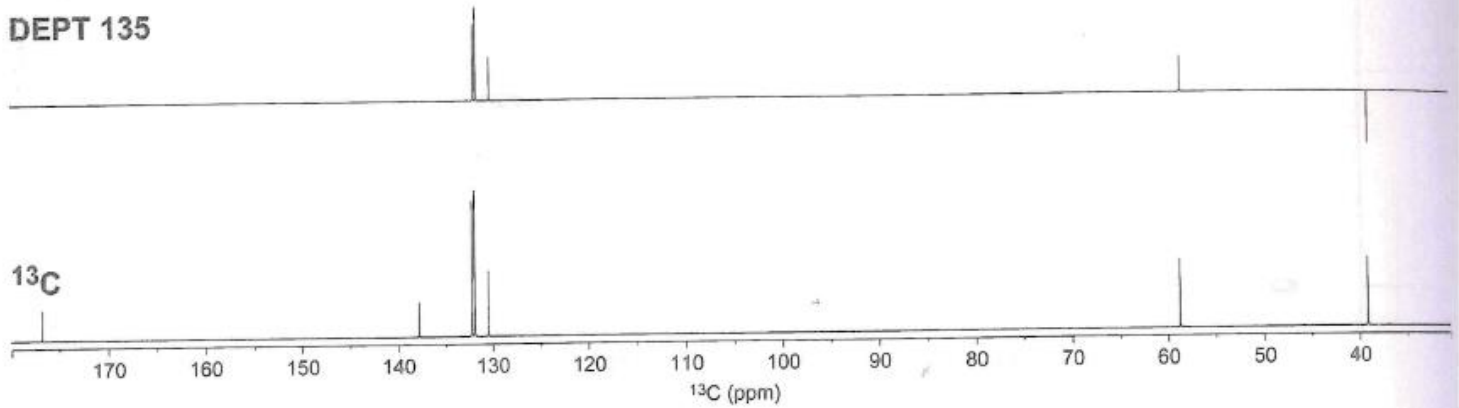
HMBC

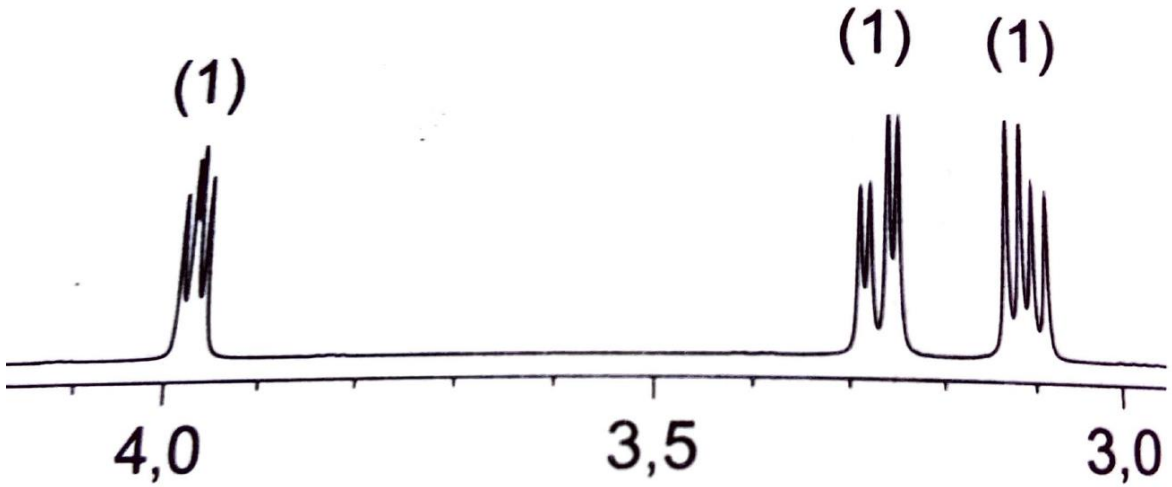

22
24
26
28
30
32
34
36
38
40
42
44
46
184
186
188
190
192
^{13}C (ppm)
3,2
3,0
2,8
2,6
2,4
2,2
2,0
1,8
1,6
1,4
1,2
1,0
^{1}H (ppm)

Solvente: D_2O Suggerimento: È un amminoacido naturale

Campo: 500 MHz (^1H); 125 MHz (^{13}C)



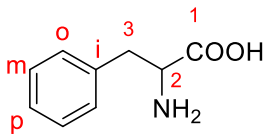


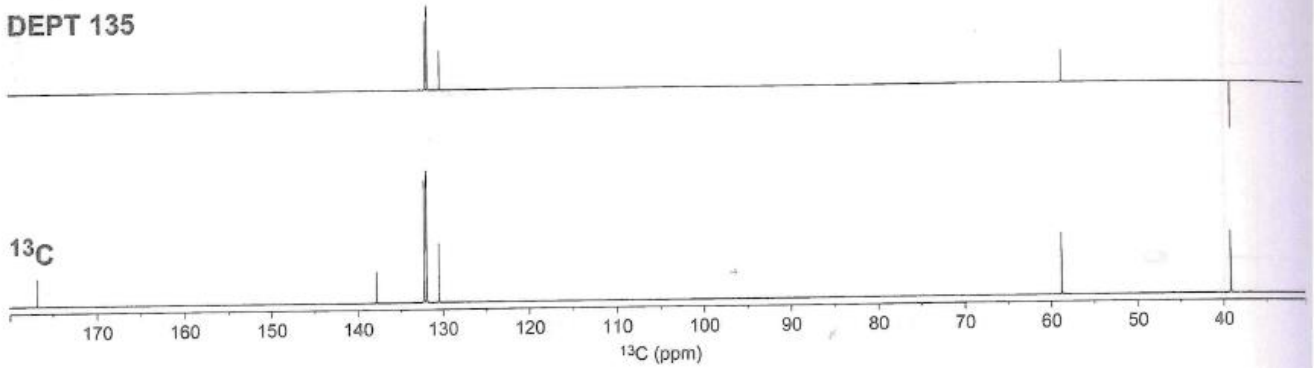


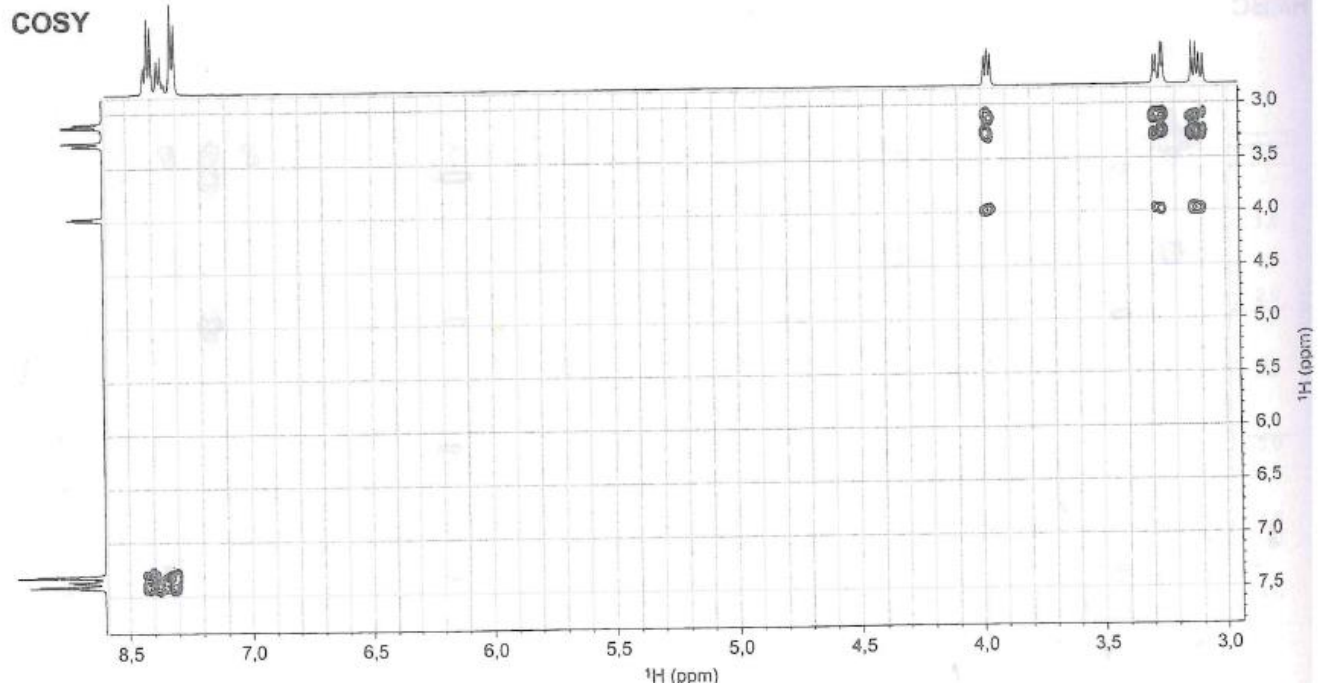
Sistema di spin?

Solvente: D_2O Suggerimento: È un amminoacido naturale

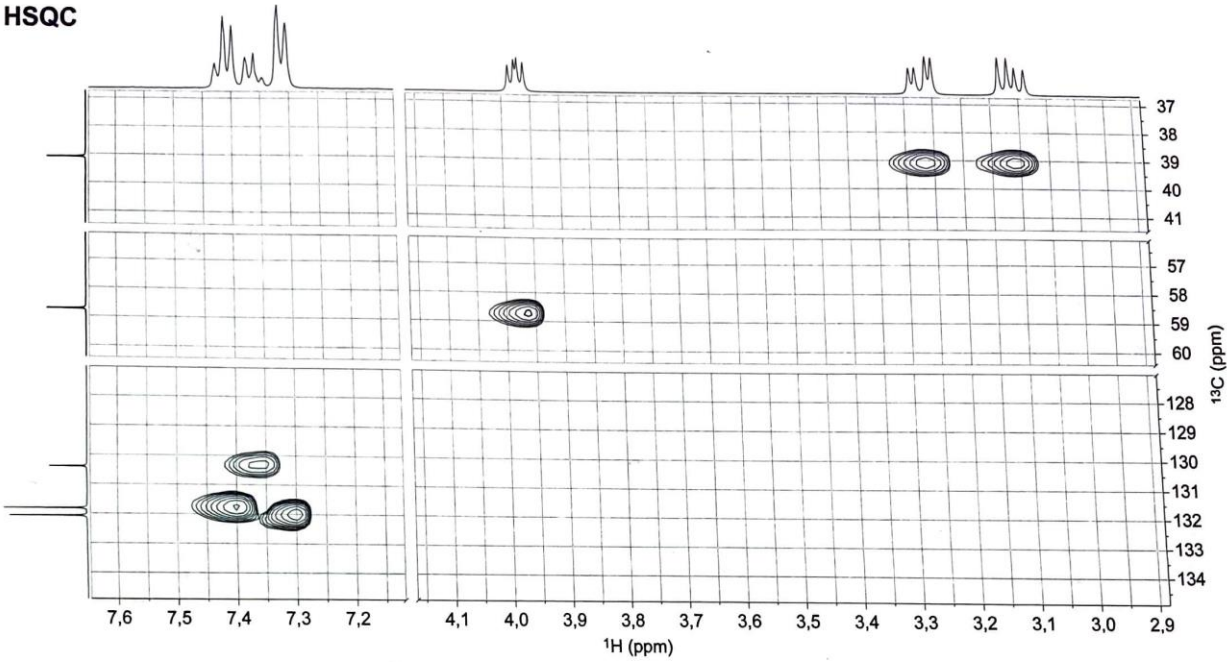
Campo: 500 MHz (^1H); 125 MHz (^{13}C)

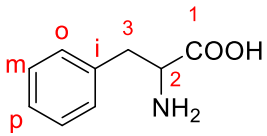




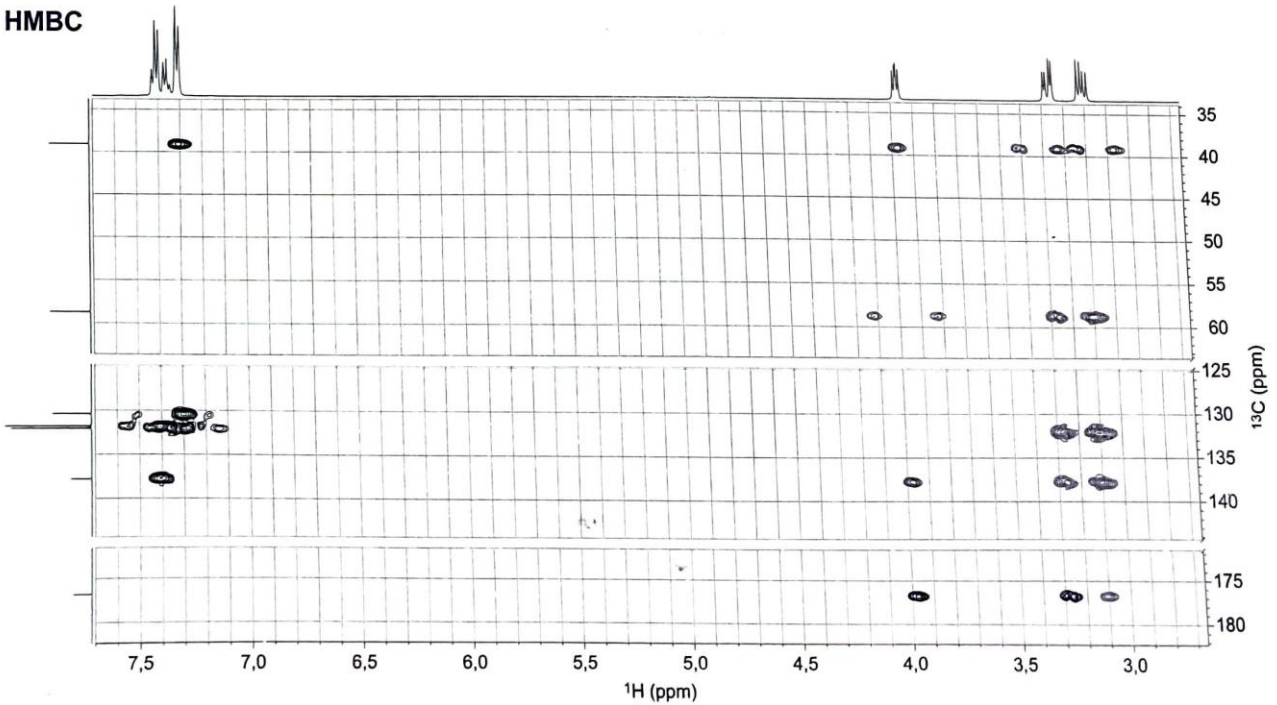


HSQC


37
38
39
40
41
57
58
59
60
128
129
130
131
132
133
134
13C (ppm)
7,6 7,5 7,4 7,3 7,2
4,1 4,0 3,9 3,8 3,7 3,6 3,5 3,4 3,3 3,2 3,1 3,0 2,9
1H (ppm)

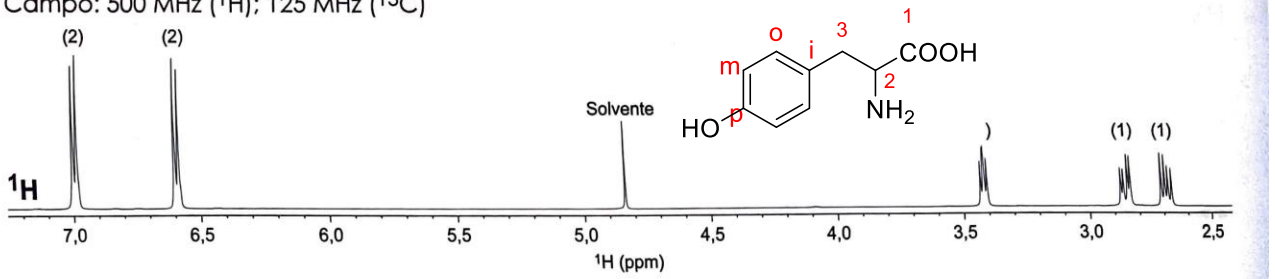

o
i
3
1
COOH
m
2
NH2
p

HMBC

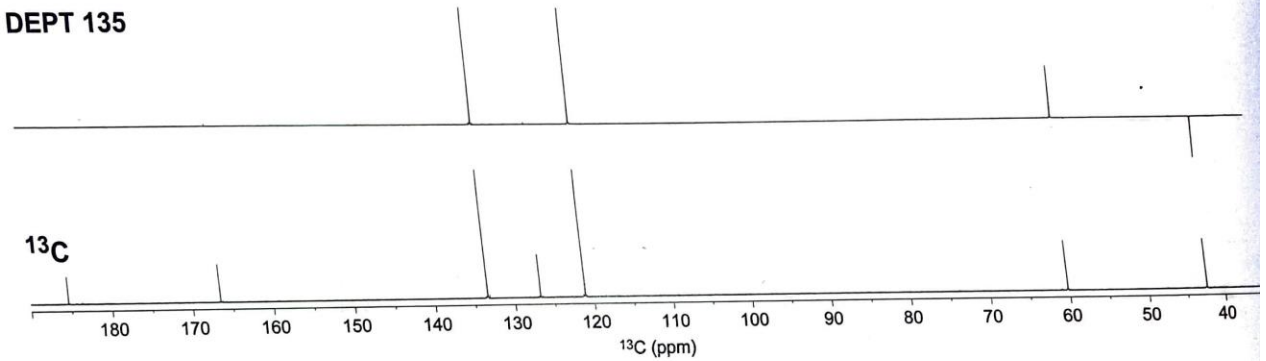

35
40
45
50
55
60
125
130
135
140
175
180
13C (ppm)
7,5 7,0 6,5 6,0 5,5 5,0 4,5 4,0 3,5 3,0
1H (ppm)

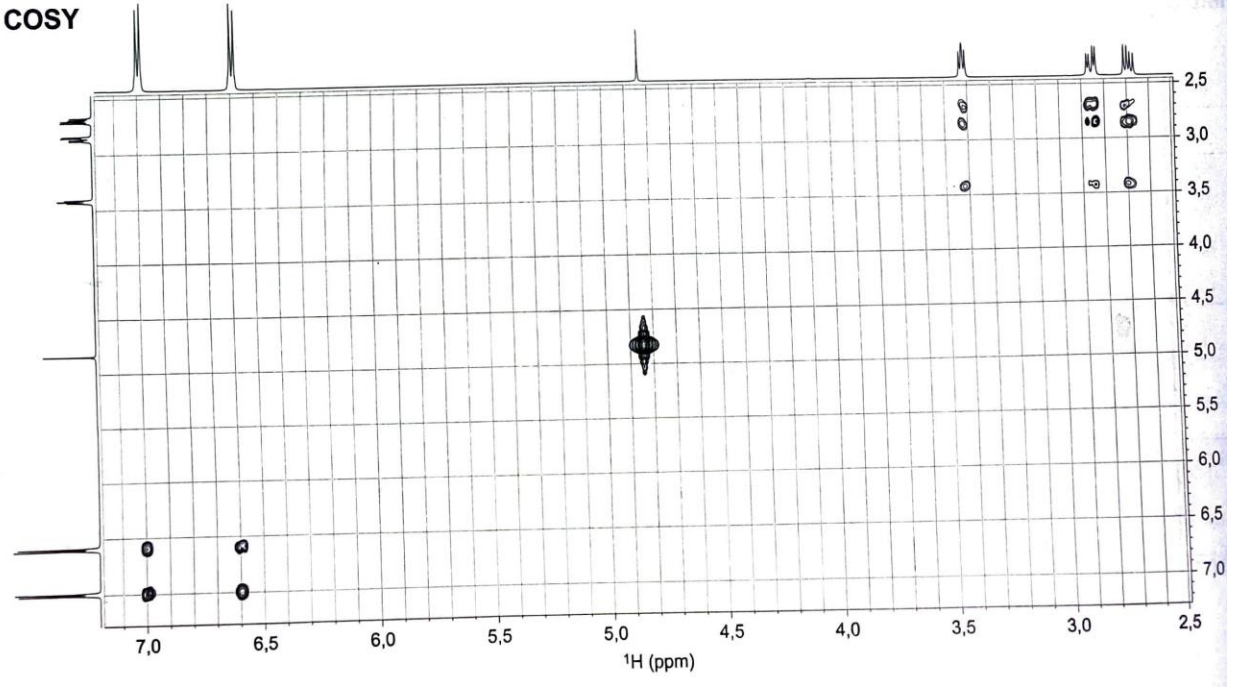
Solvente: D_2O Suggerimento: È un amminoacido naturale

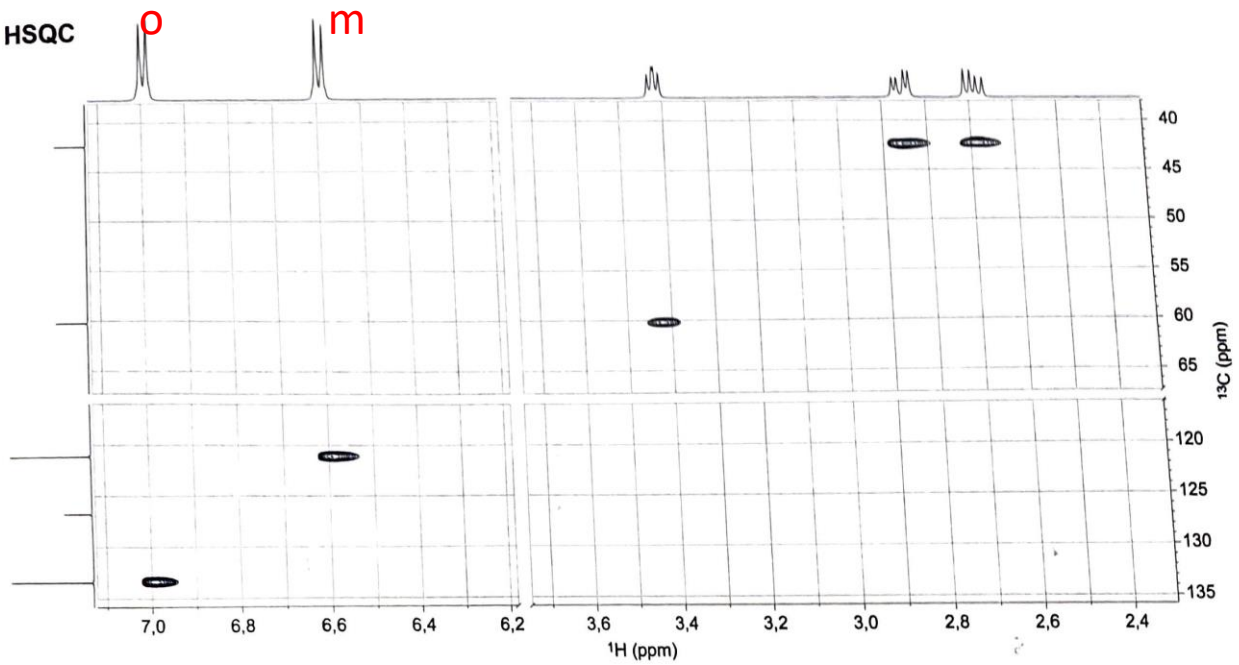
Campo: 500 MHz (^1H); 125 MHz (^{13}C)

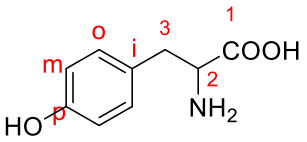


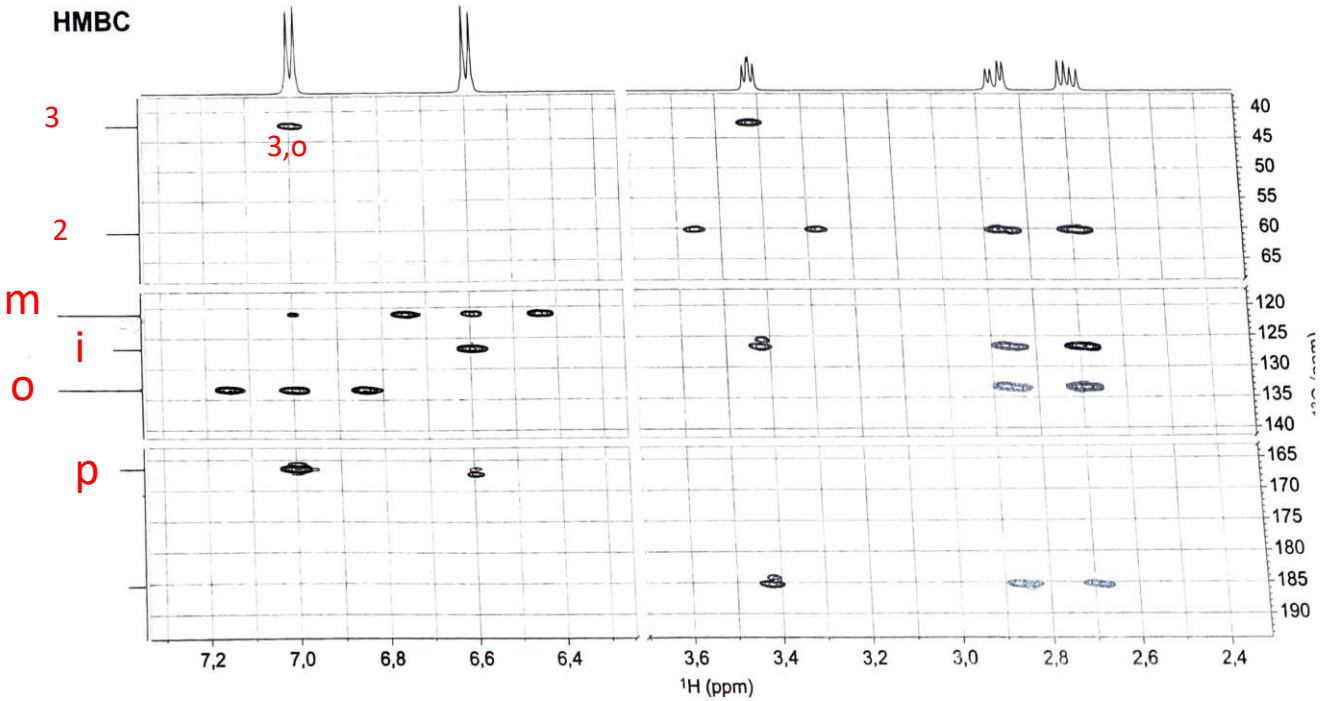
In D2O



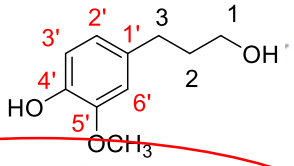


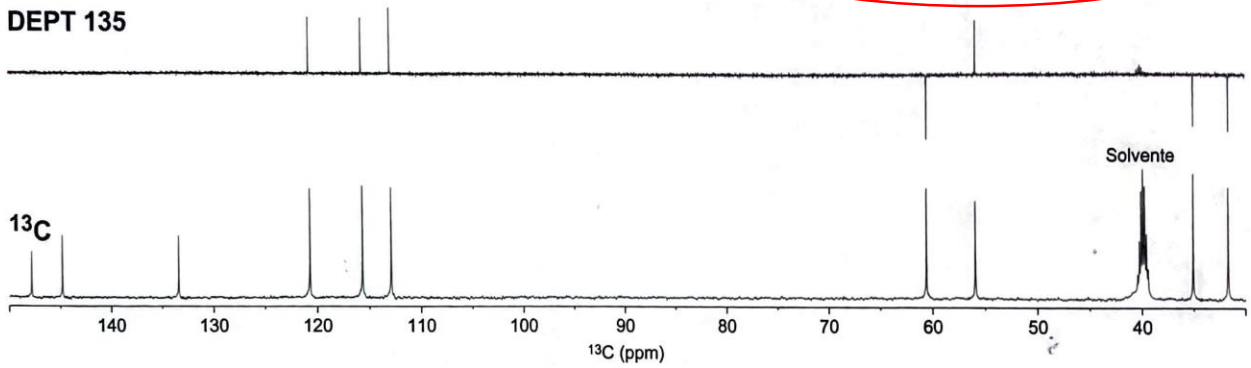

HSQC
o
m
40
45
50
55
60
65
120
125
130
135
13C (ppm)
7,0
6,8
6,6
6,4
6,2
3,6
3,4
3,2
3,0
2,8
2,6
2,4
1H (ppm)

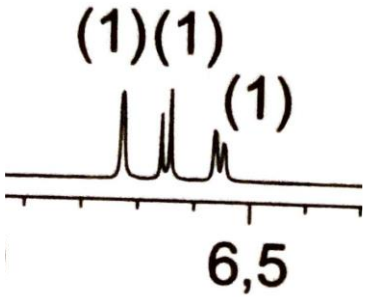

o
i
3
1
m
2
COOH
p
HO
NH2

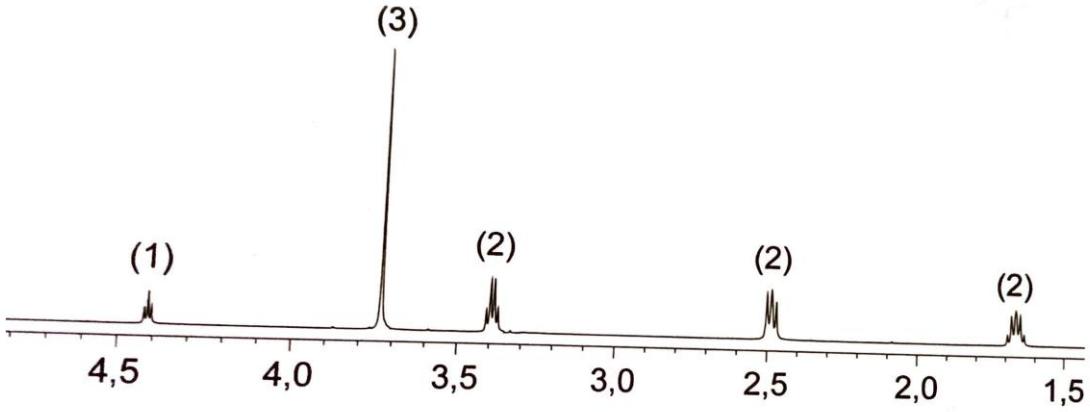

HMBC
3
2
m
i
o
p
3,o
40
45
50
55
60
65
120
125
130
135
140
165
170
175
180
185
190
7,2
7,0
6,8
6,6
6,4
3,6
3,4
3,2
3,0
2,8
2,6
2,4
1H (ppm)

Solvente: CD_3-SO-CD_3
Campo: 500 MHz (^1H); 125 MHz (^{13}C)

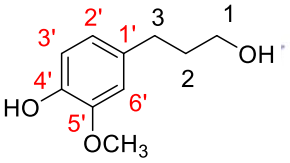






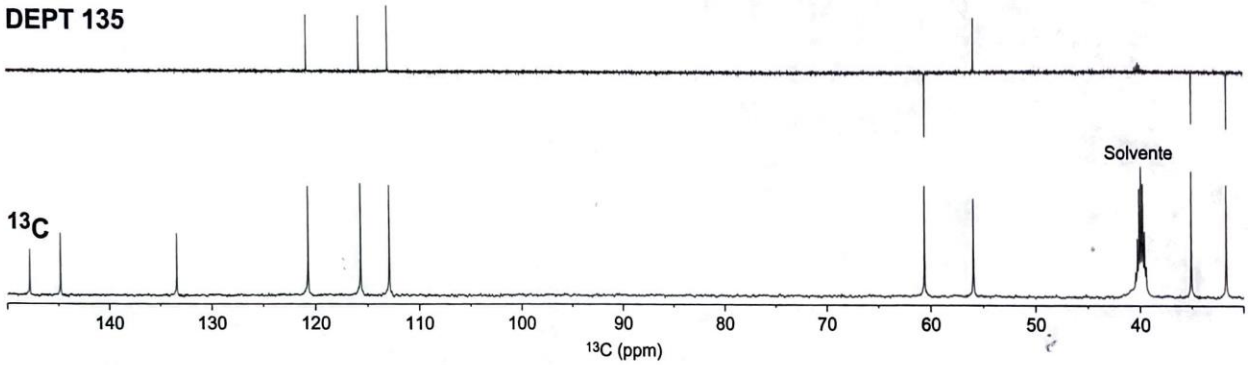


Solvente: CD_3-SO-CD_3
Campo: 500 MHz (^1H); 125 MHz (^{13}C)

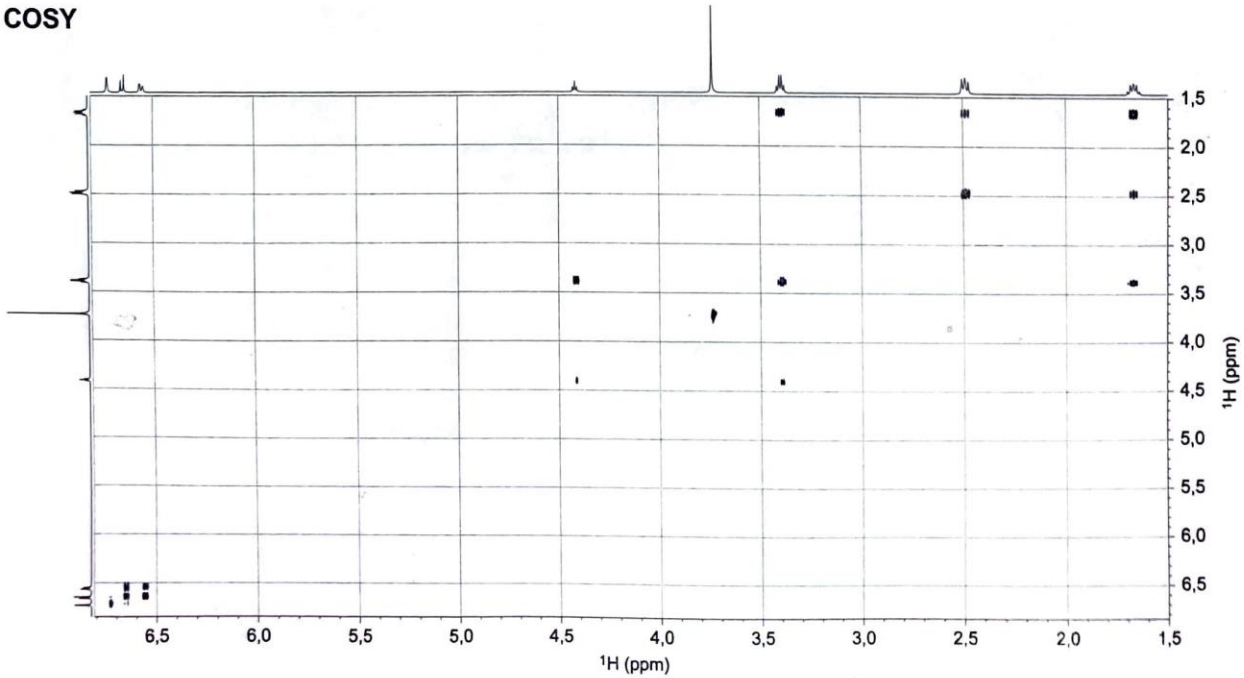


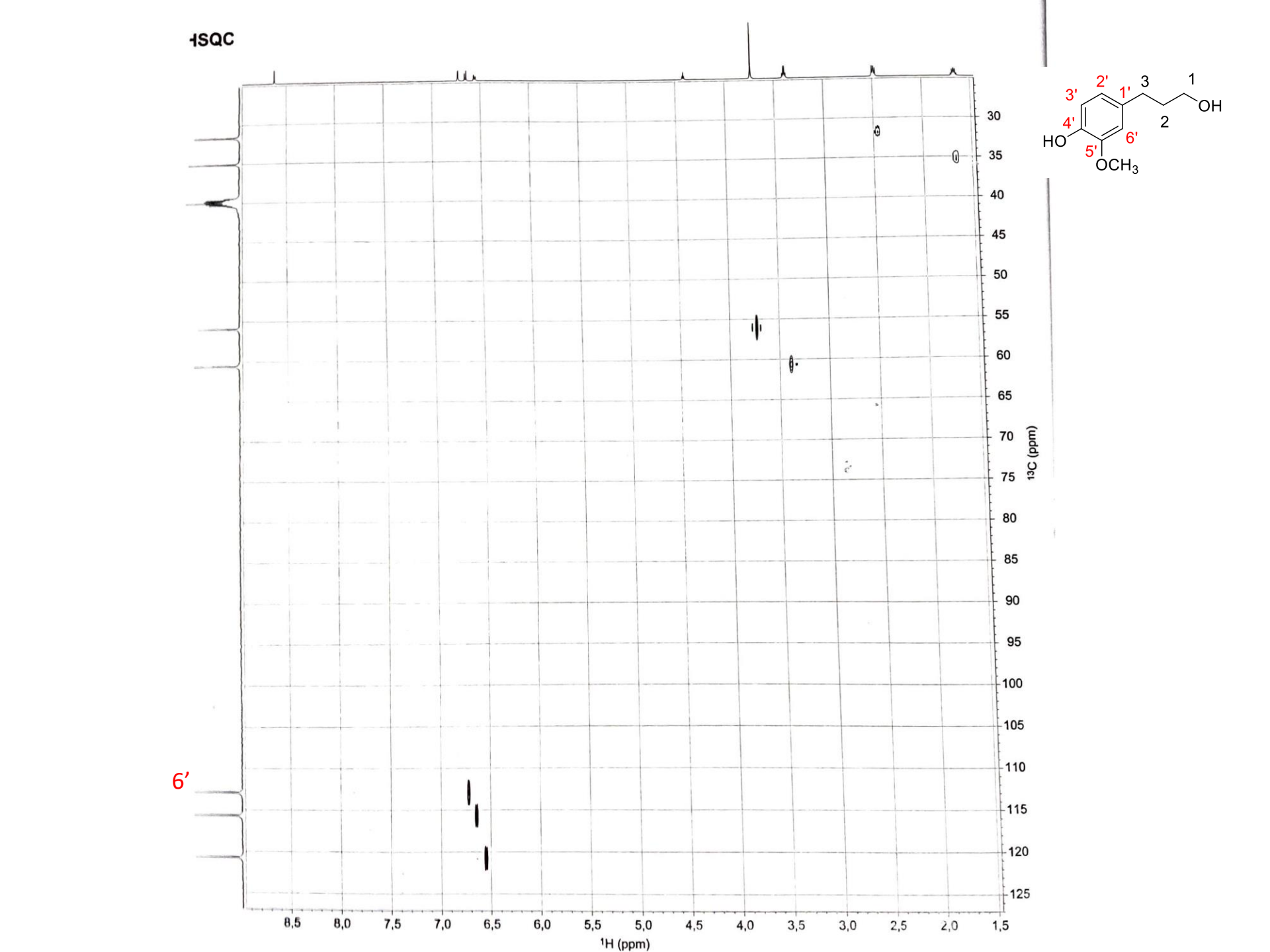
^1H

DEPT 135

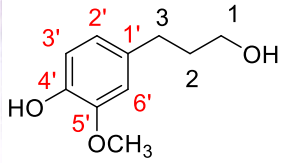


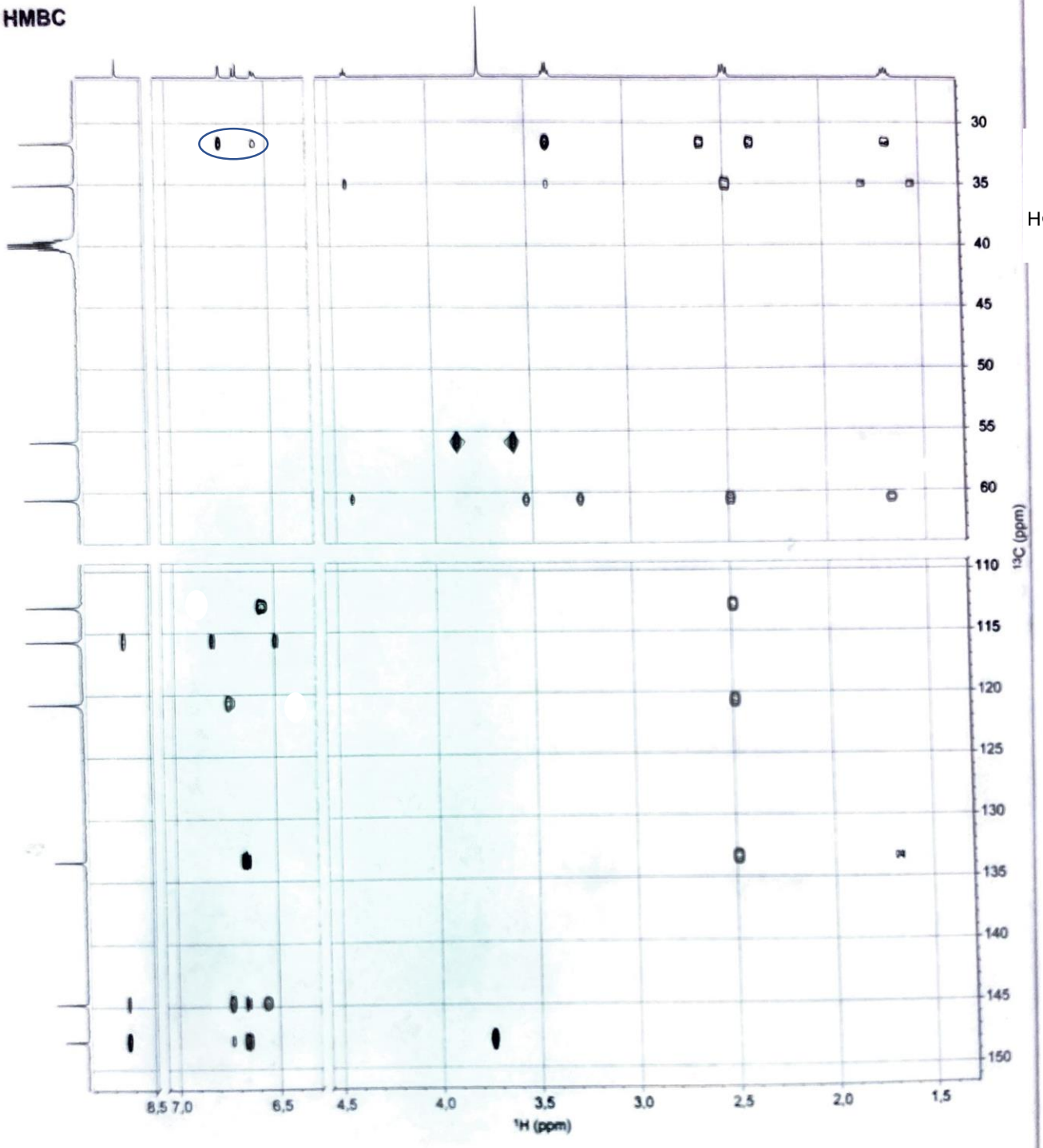
COSY




HSQC
6’
^{13}C (ppm)
^{1}H (ppm)
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
30
35
40
45
50
55
60
65
70
75
80
85
90
95
100
105
110
115
120
125
2'
3'
1'
3
1
OH
4'
2
HO
6'
5'
OCH3

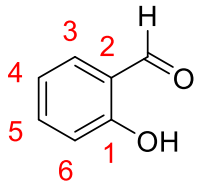
HMBC


1
2
3
OH
1'
2'
3'
4'
5'
6'
HO
OCH3


30
35
40
45
50
55
60
110
115
120
125
130
135
140
145
150
13C (ppm)
8,5
7,0
6,5
4,5
4,0
3,5
3,0
2,5
2,0
1,5
1H (ppm)

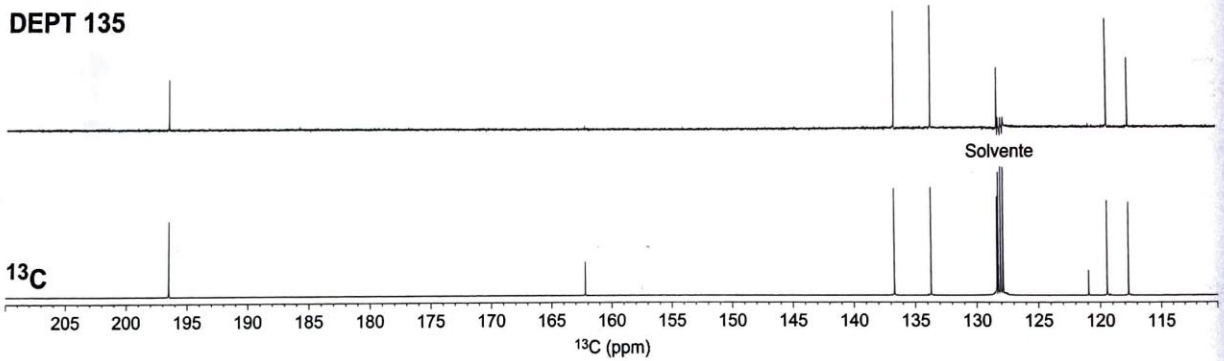
Solvente: C_6D_6

Campo: 500 MHz (^1H); 125 MHz (^{13}C)

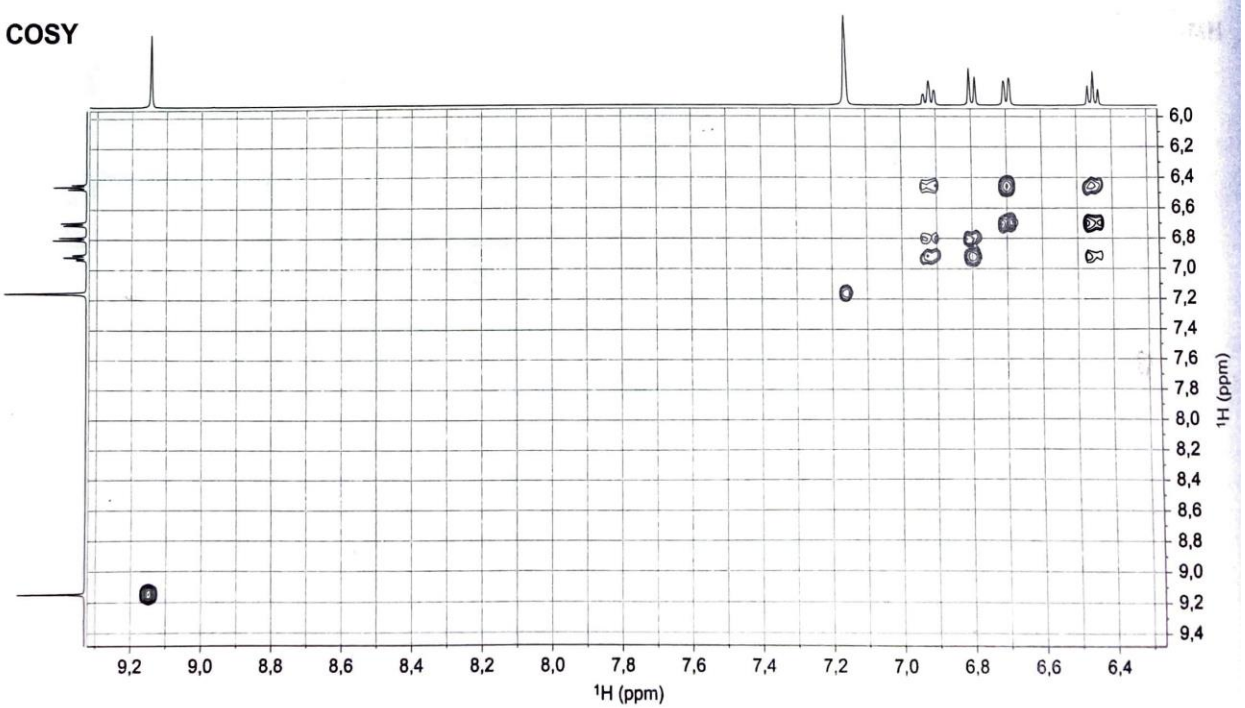


^1H

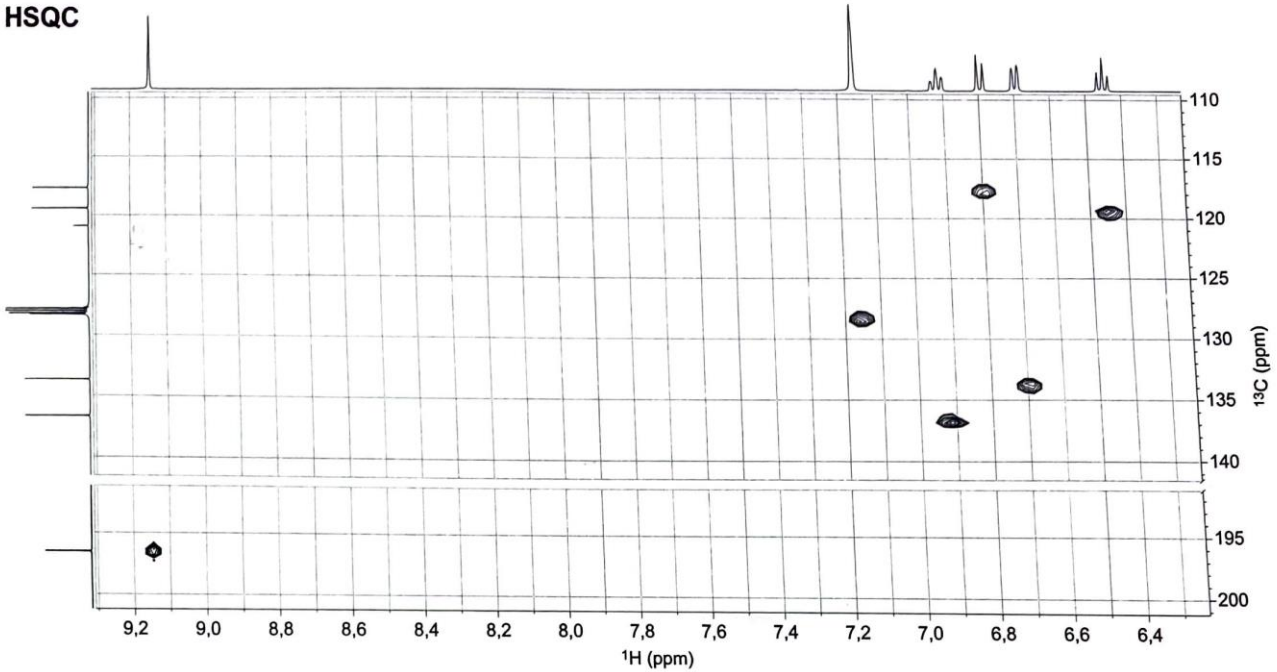
DEPT 135

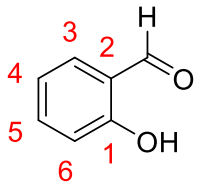


COSY

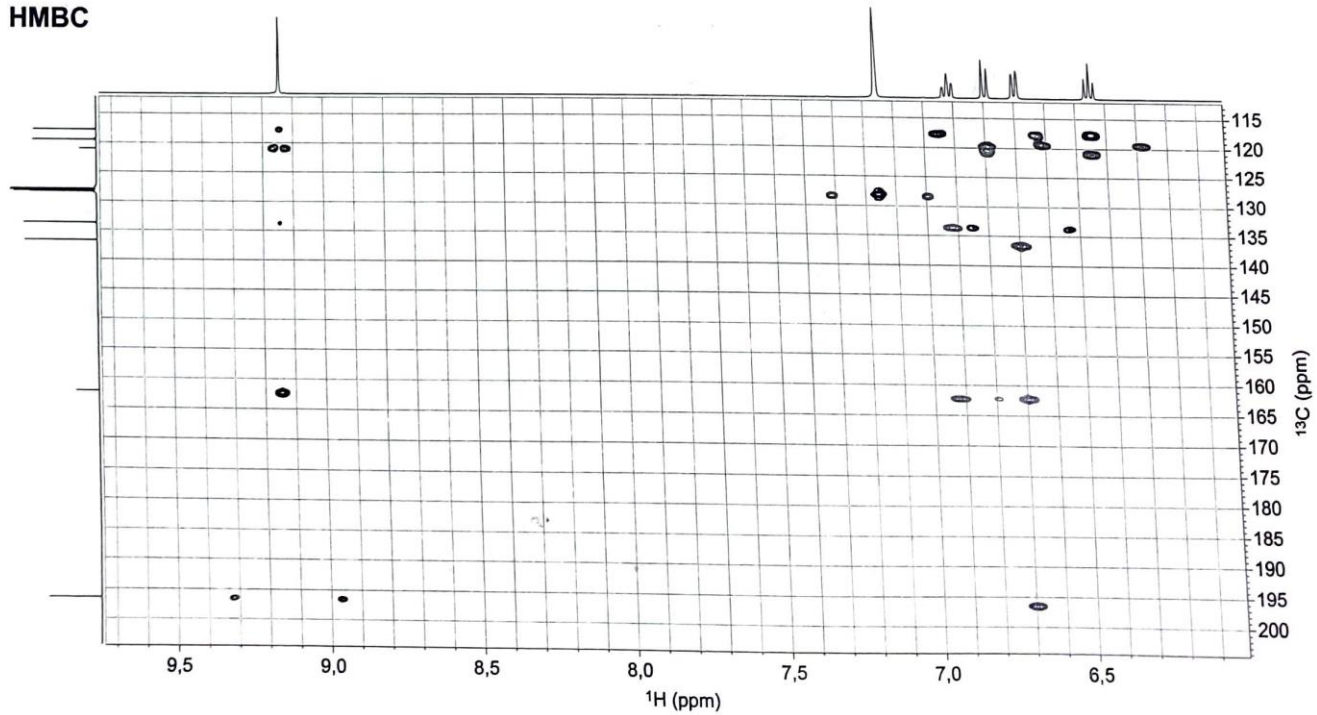


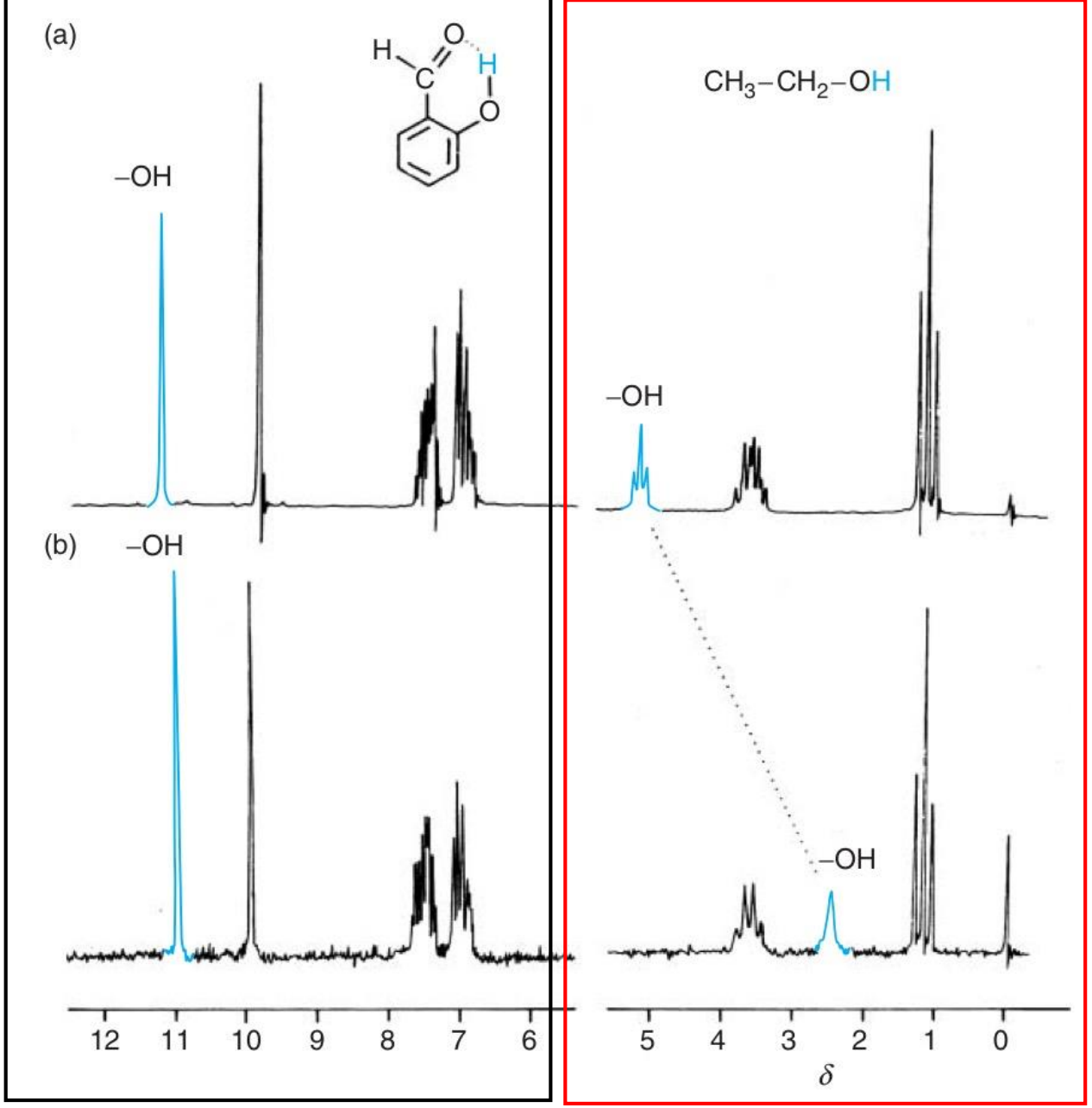
HSQC


110
115
120
125
130
135
140
195
200
9,2
9,0
8,8
8,6
8,4
8,2
8,0
7,8
7,6
7,4
7,2
7,0
6,8
6,6
6,4
¹H (ppm)
¹³C (ppm)


H
O
OH
1
2
3
4
5
6

HMBC


115
120
125
130
135
140
145
150
155
160
165
170
175
180
185
190
195
200
9,5
9,0
8,5
8,0
7,5
7,0
6,5
¹H (ppm)
¹³C (ppm)

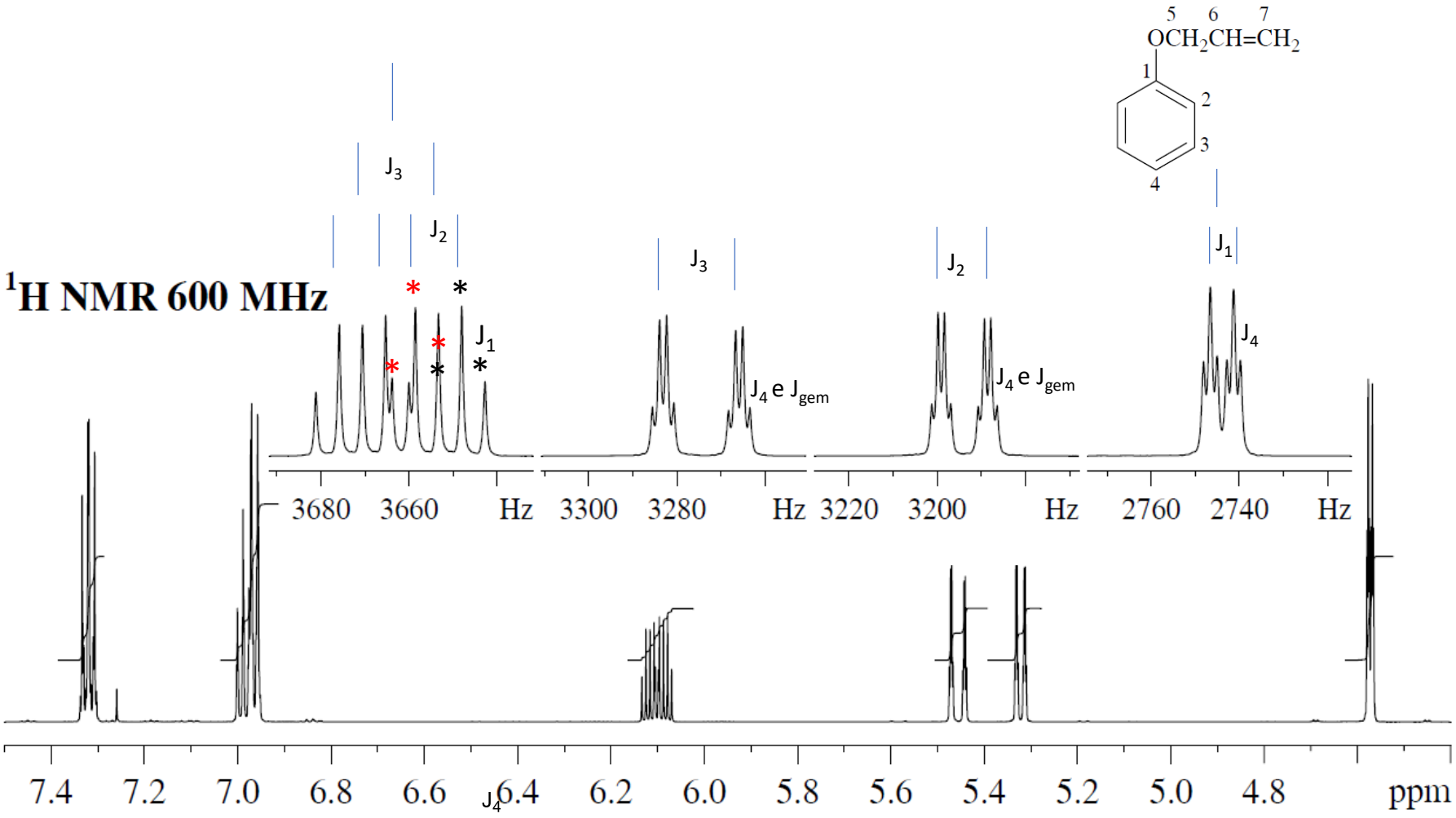


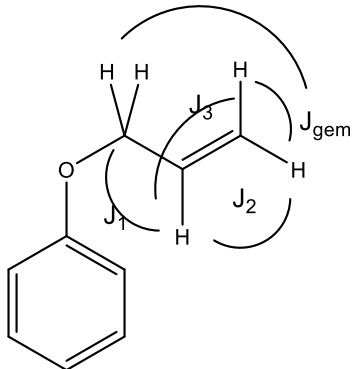
Spiegare le variazioni degli spettri con i solventi

a) DMSO d6

b) CDCl3

Figure 5.21 Concentration dependence of the proton resonance frequency of the hydroxyl protons of salicylaldehyde and ethanol: (a) neat and (b) 5% by volume in CCl_4.





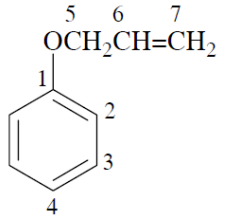
$J_1 = J_{5,6}$

$J_2 = J_{6,7\ cis}$

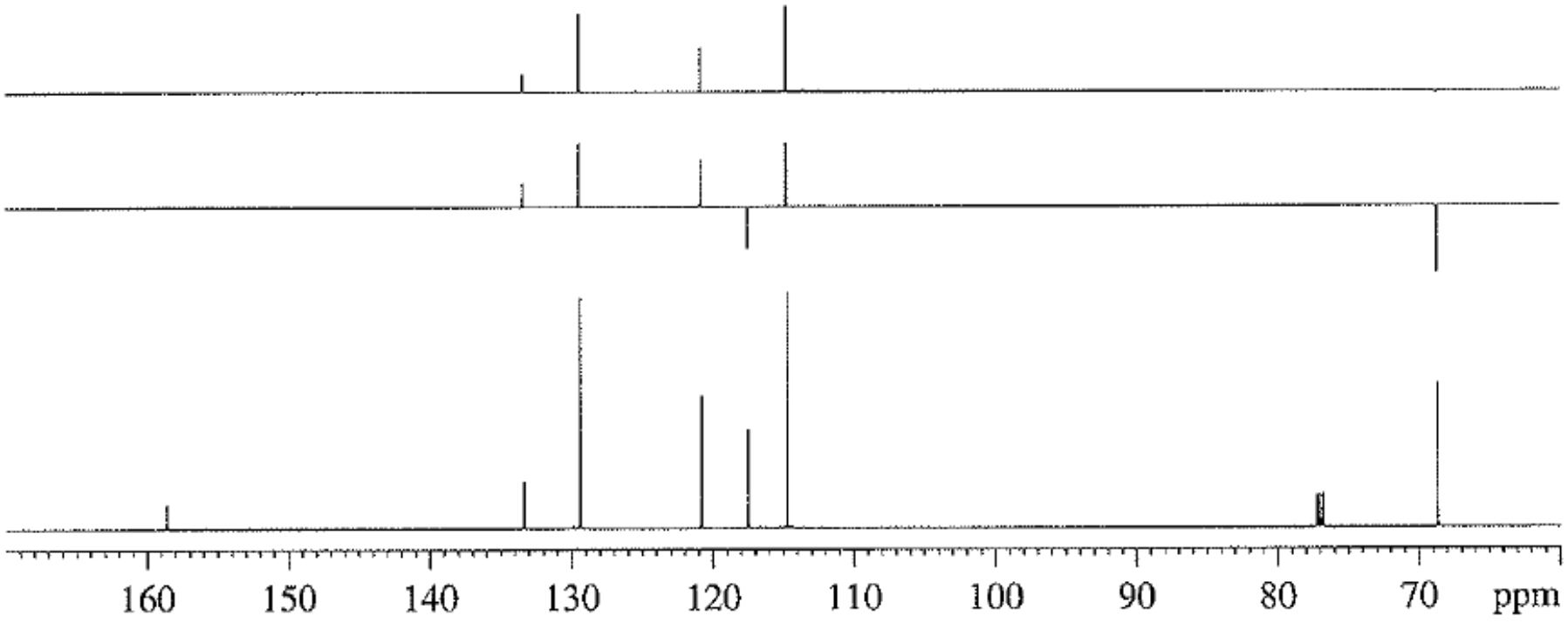
$J_3 = J_{6,7\ trans}$

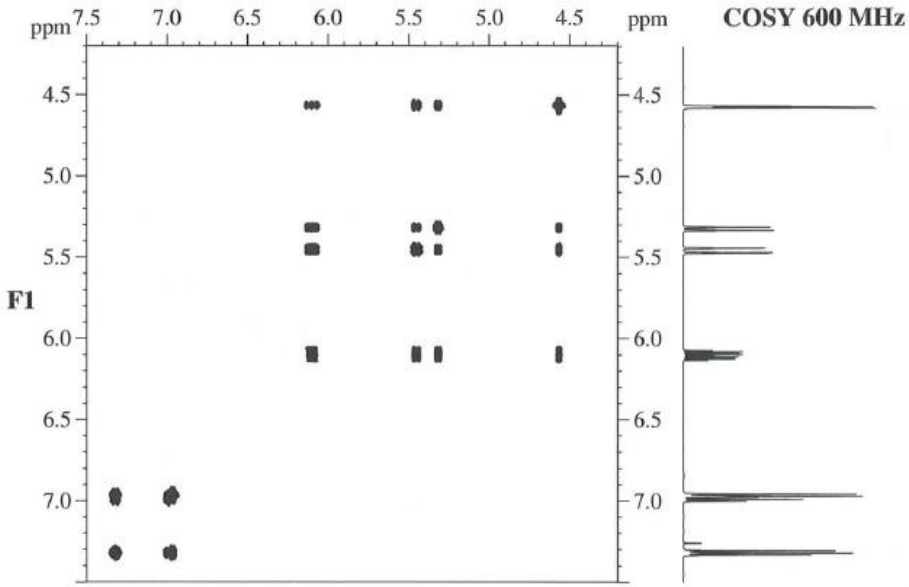
$J_4 = J_{5,7}$

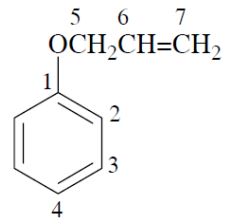
$J_5 = J_{gem}$

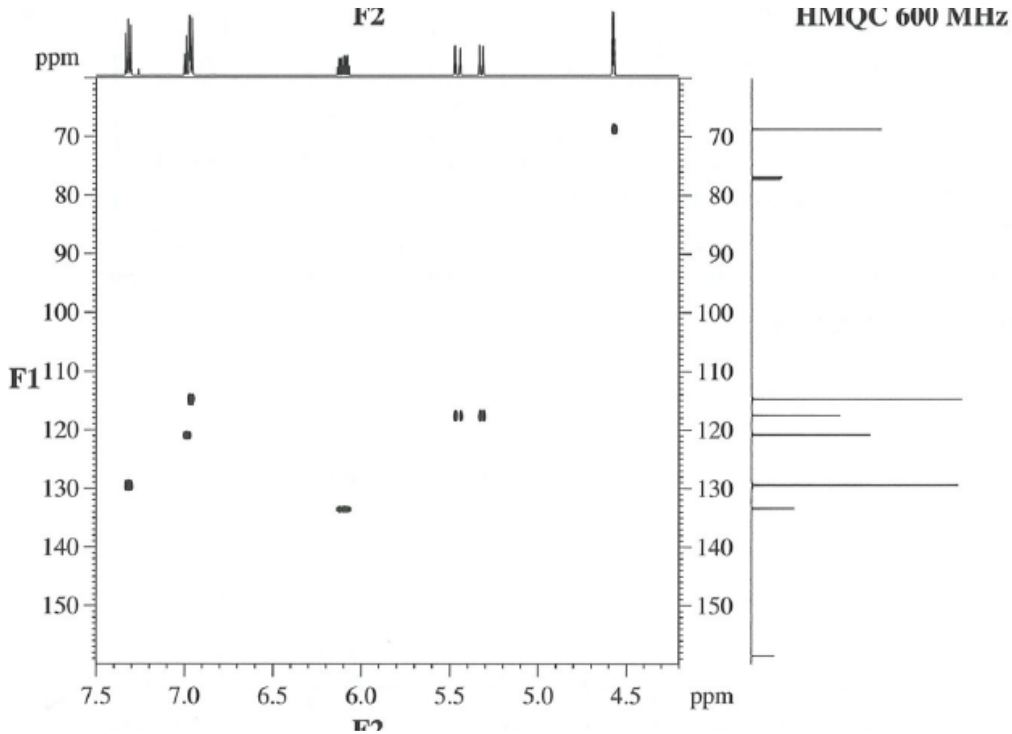


^{13}C/DEPT NMR 150.9 MHz

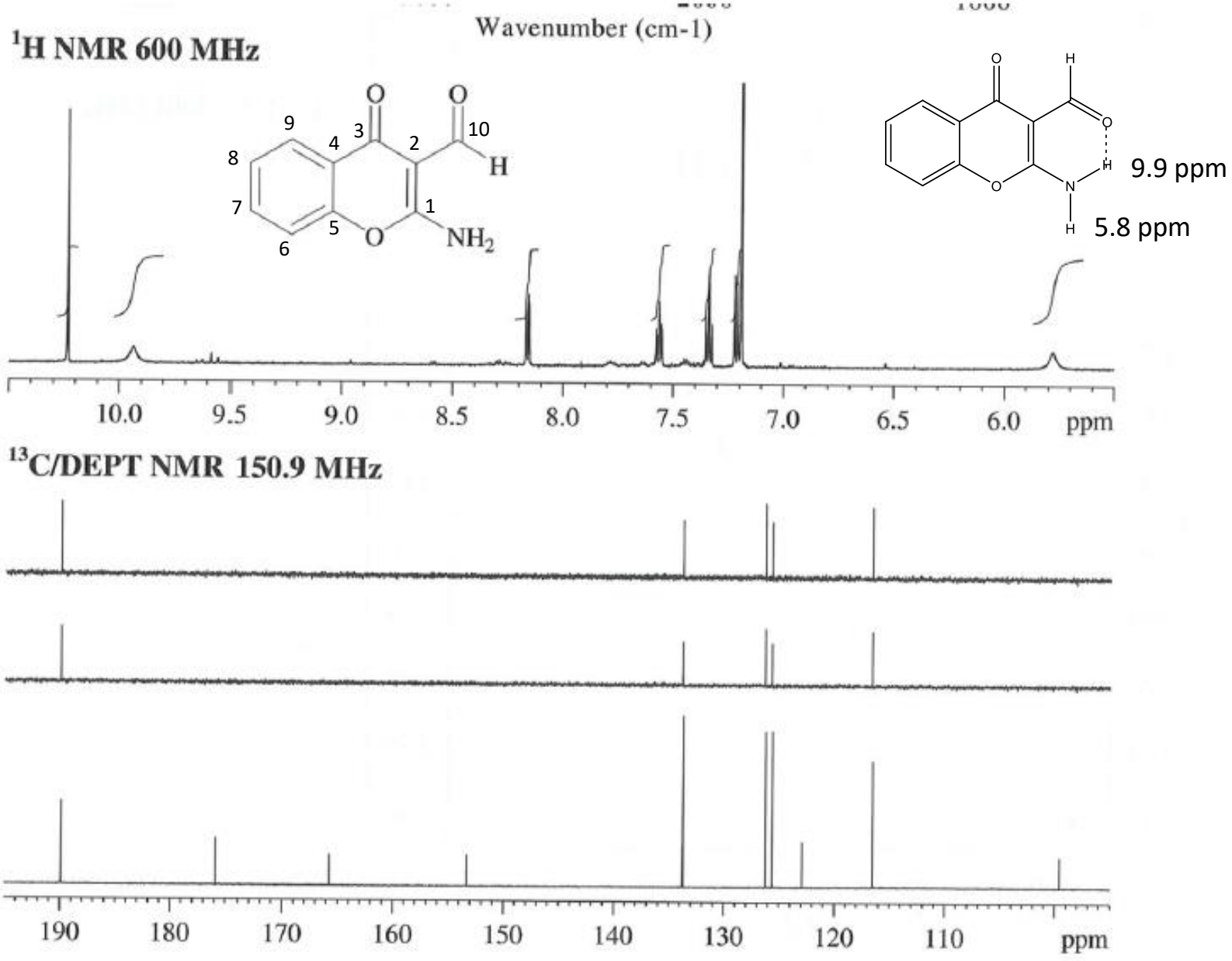



COSY 600 MHz
ppm
7.5
7.0
6.5
6.0
5.5
5.0
4.5
F1

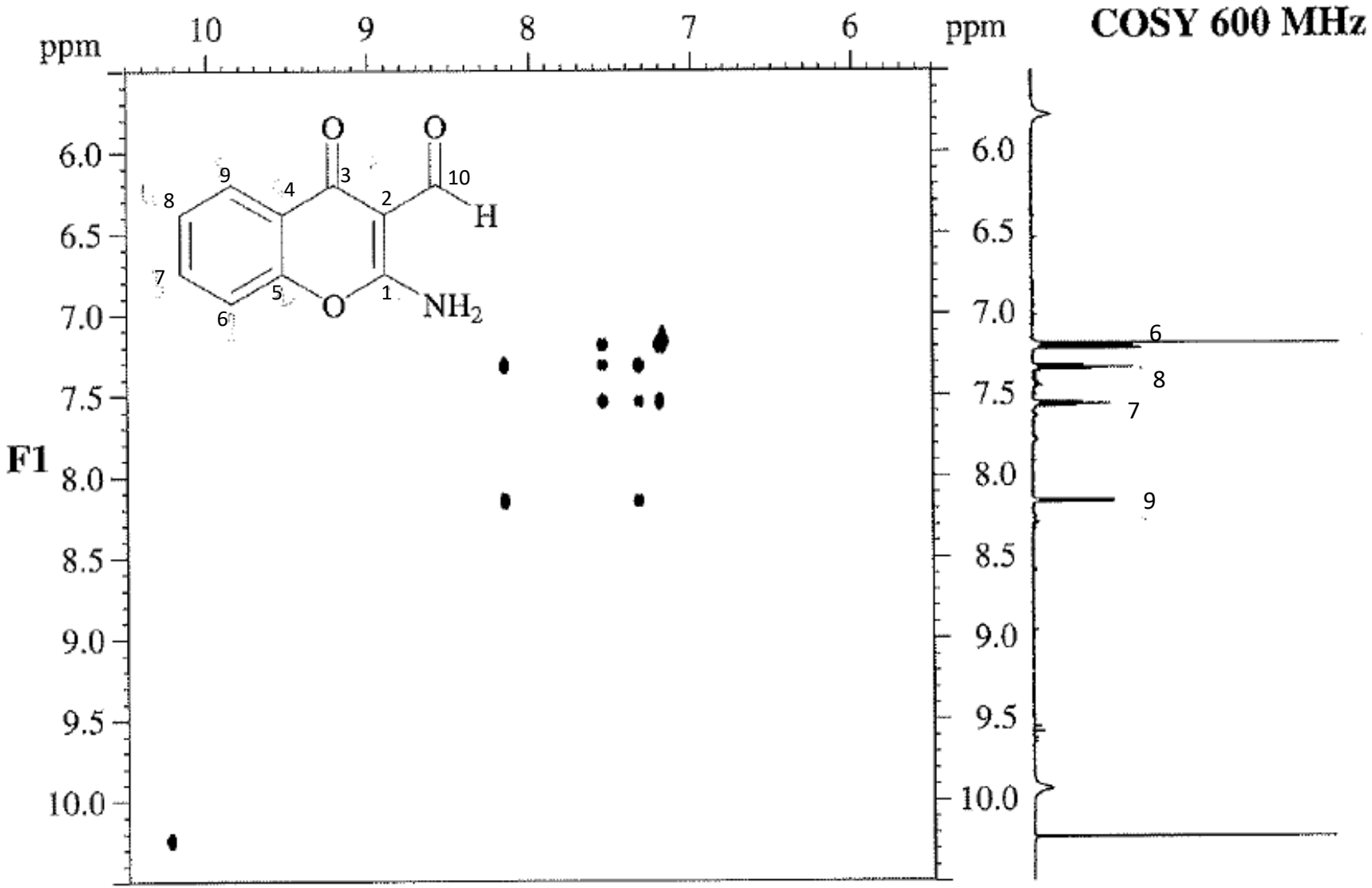

5 6 7
OCH2CH=CH2
1
2
3
4


HMQC 600 MHz
F2
ppm
70
80
90
100
110
120
130
140
150
F1
7.5
7.0
6.5
6.0
5.5
5.0
4.5

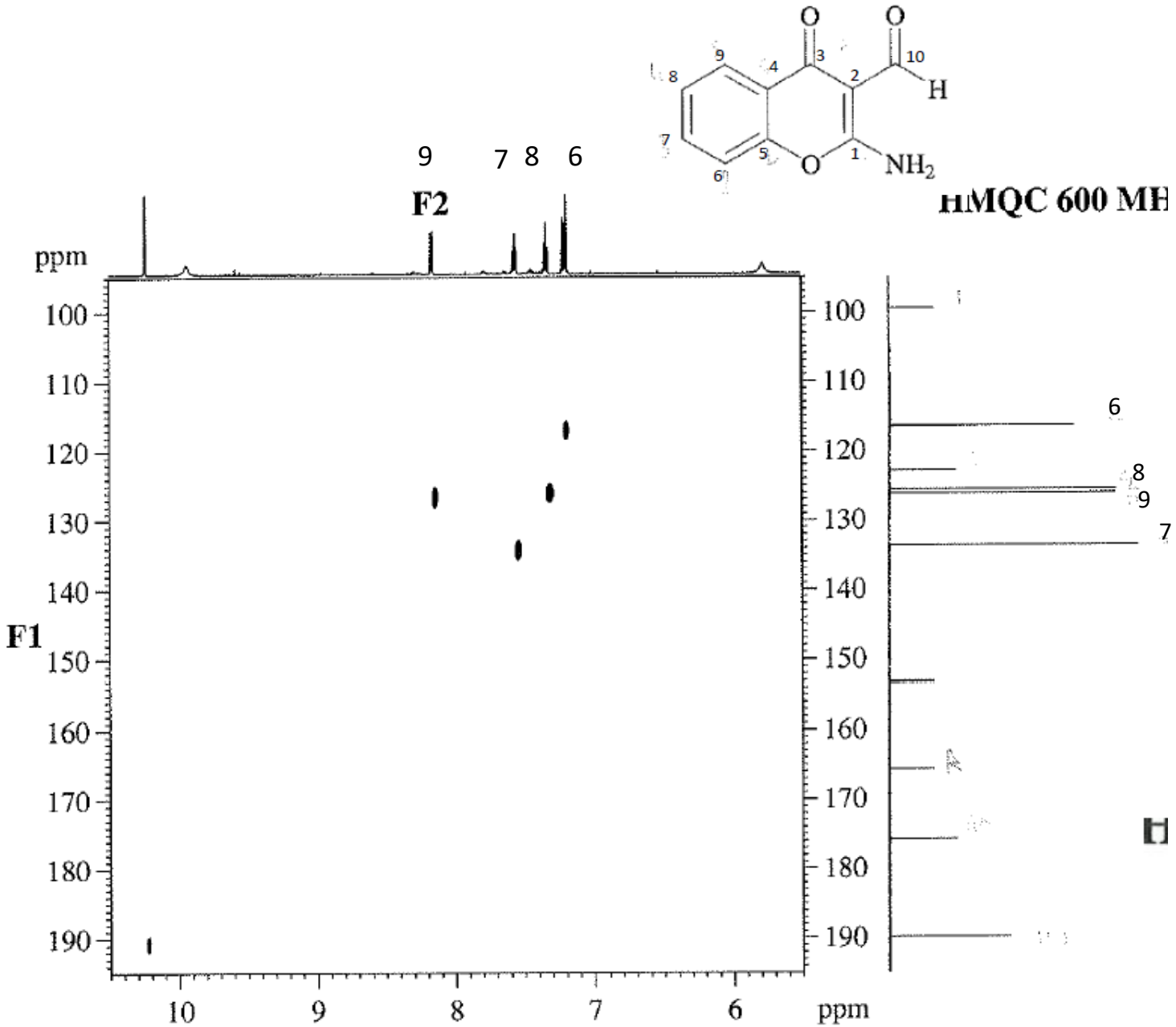
ES.3


Wavenumber (cm-1)
^{1}H NMR 600 MHz
9.9 ppm
5.8 ppm
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
ppm
^{13}C/DEPT NMR 150.9 MHz
190
180
170
160
150
140
130
120
110
ppm

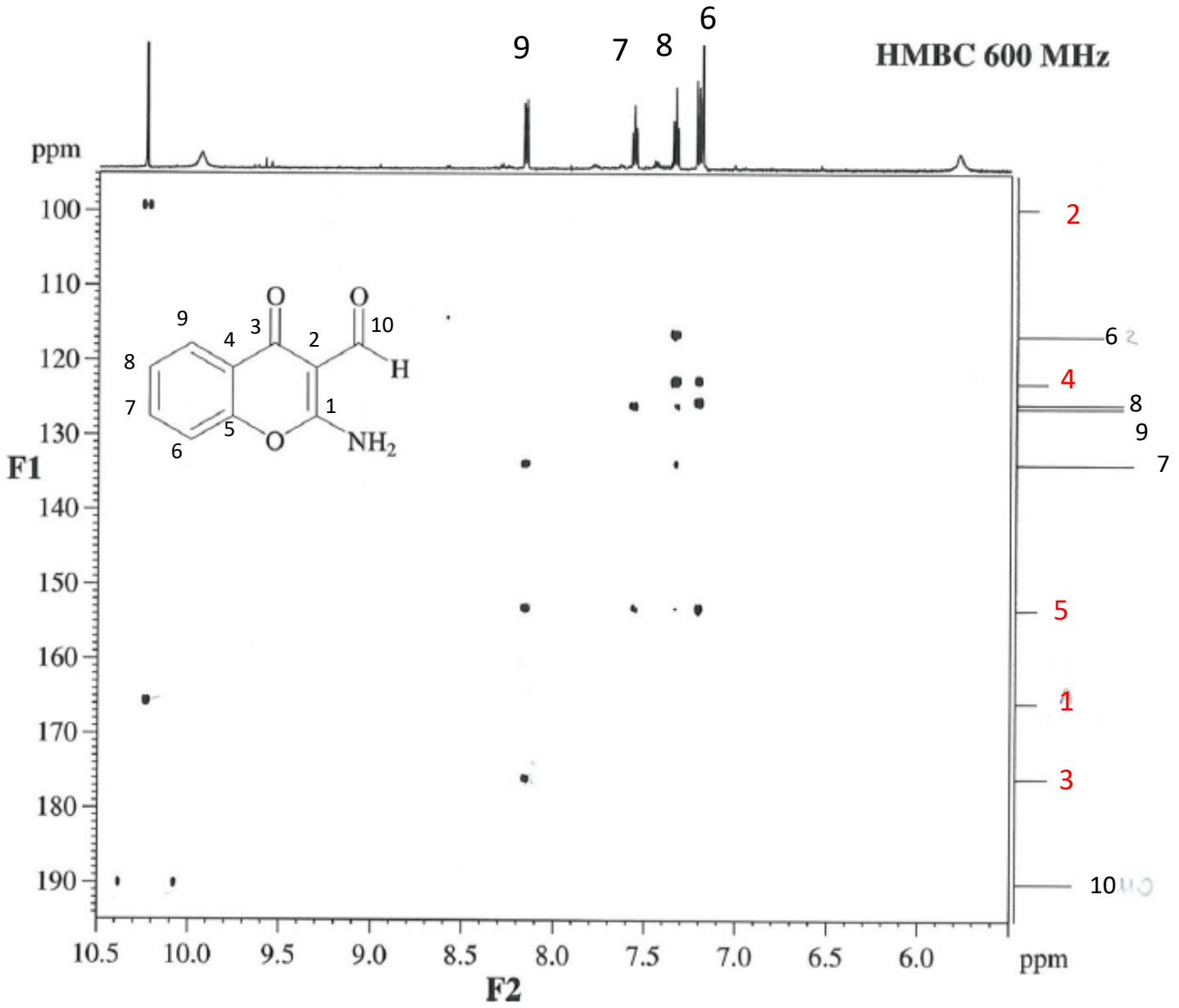
ES.3


COSY 600 MHz
ppm
10
9
8
7
6
ppm
F1
6.0
6.5
7.0
7.5
8.0
8.5
9.0
9.5
10.0
O
O
3
10
H
9
4
2
8
7
5
1
6
O
NH2
6
8
7
9

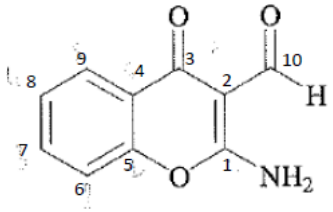
ES.3

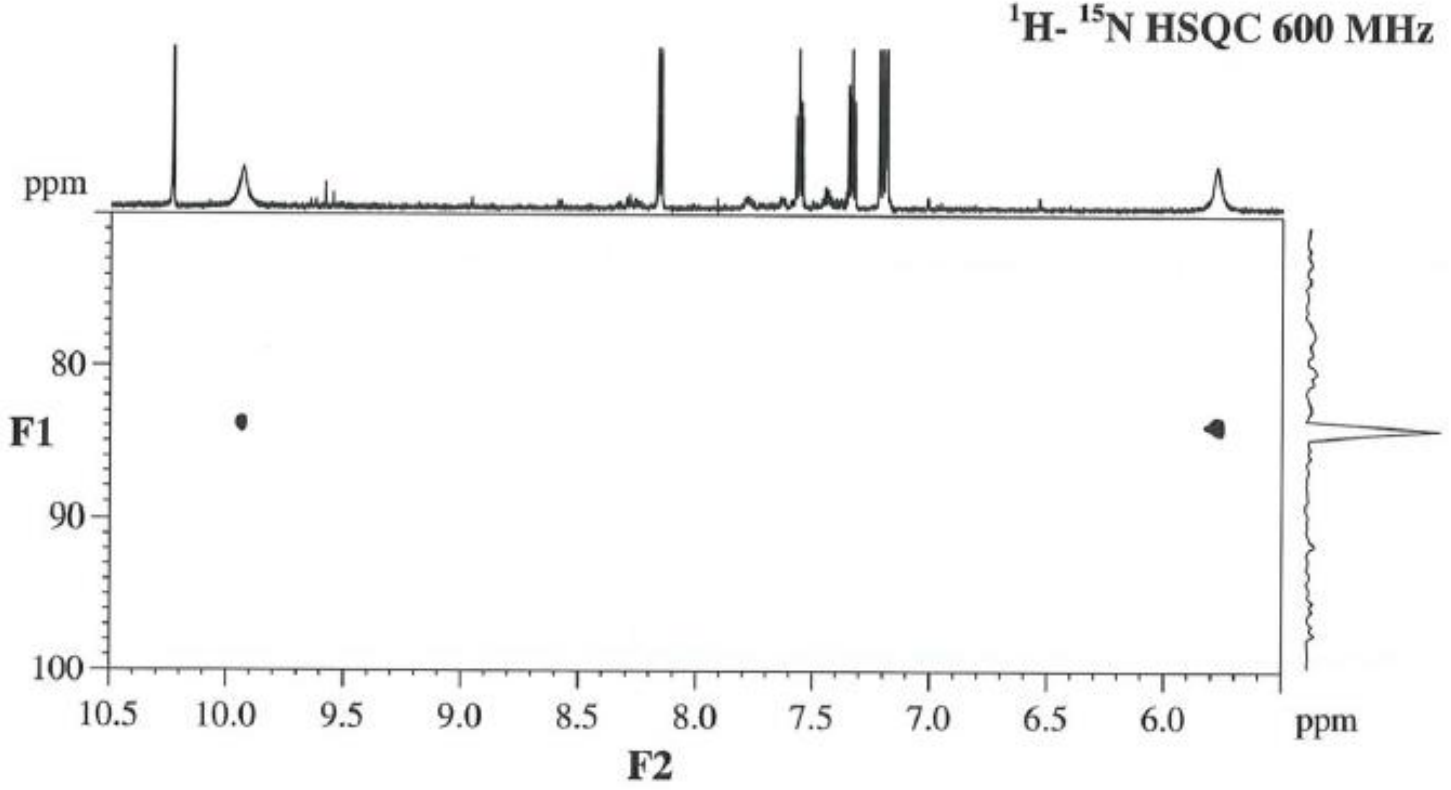

O
O
9
4
3
2
10
8
H
7
5
O
1
NH2
6
9
7 8
6
F2
HMQC 600 MH
ppm
100
110
120
130
140
F1
150
160
170
180
190
10
9
8
7
6
ppm
6
8
9
7

ES.3

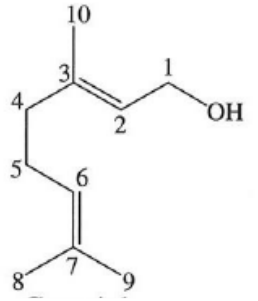

HMBC 600 MHz
9
7
8
6
ppm
100
110
120
130
140
150
160
170
180
190
F1
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
F2
ppm
2
6
4
8
9
7
5
1
3
10
O
O
O
O
NH2
H
1
2
3
4
5
6
7
8
9
10

ES.3

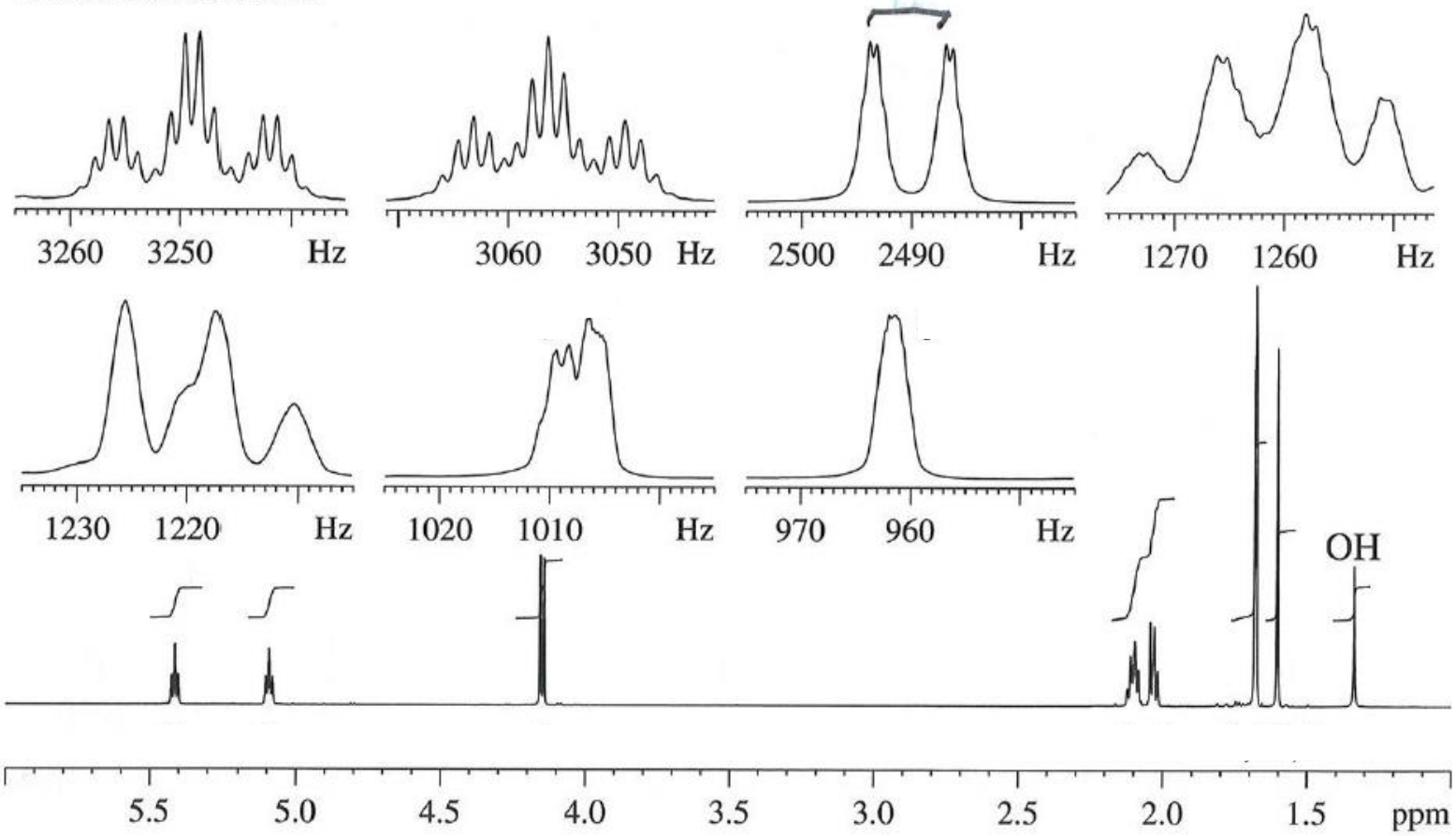

O
O
10
H
9
4
3
2
8
7
5
1
6
O
NH2


1H- 15N HSQC 600 MHz
ppm
80
90
100
F1
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
ppm
F2

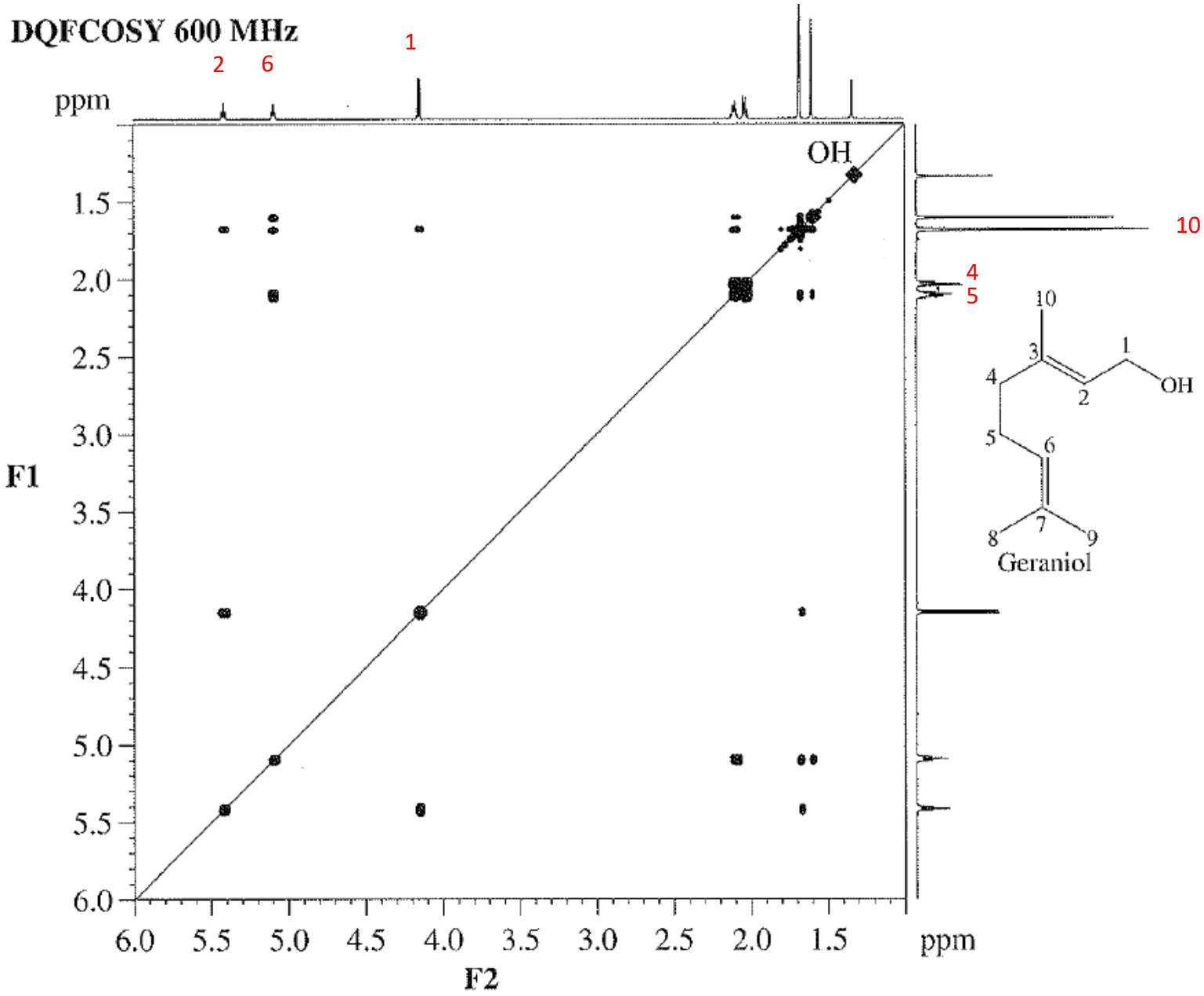
Es. 4



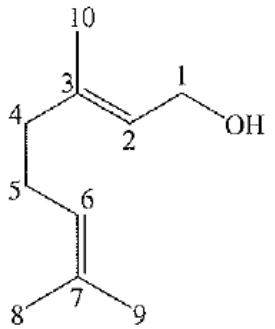
^{1}H NMR 600 MHz



Es. 4

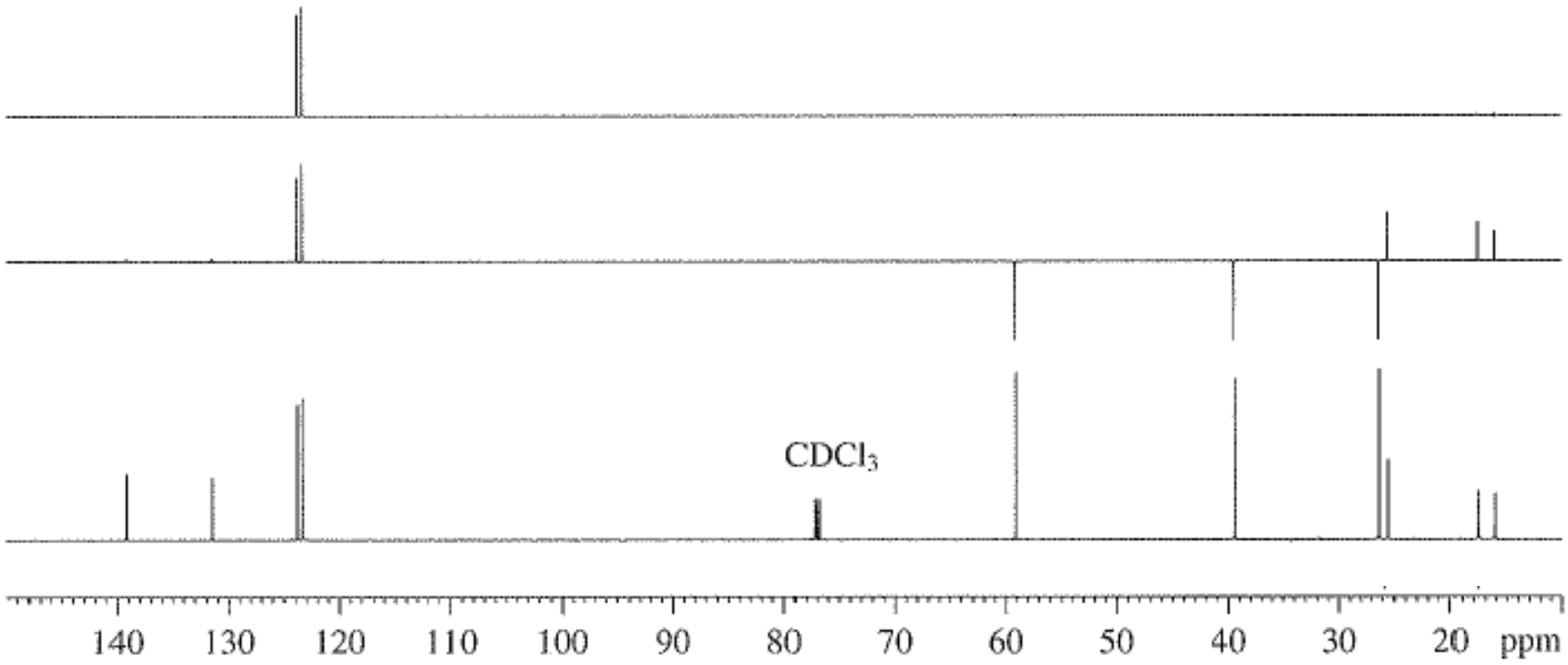

DQFCOSY 600 MHz
ppm
1
2
6
OH
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
F1
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm
F2
10
4
5
10
3
1
4
2
OH
5
6
8
7
9
Geraniol

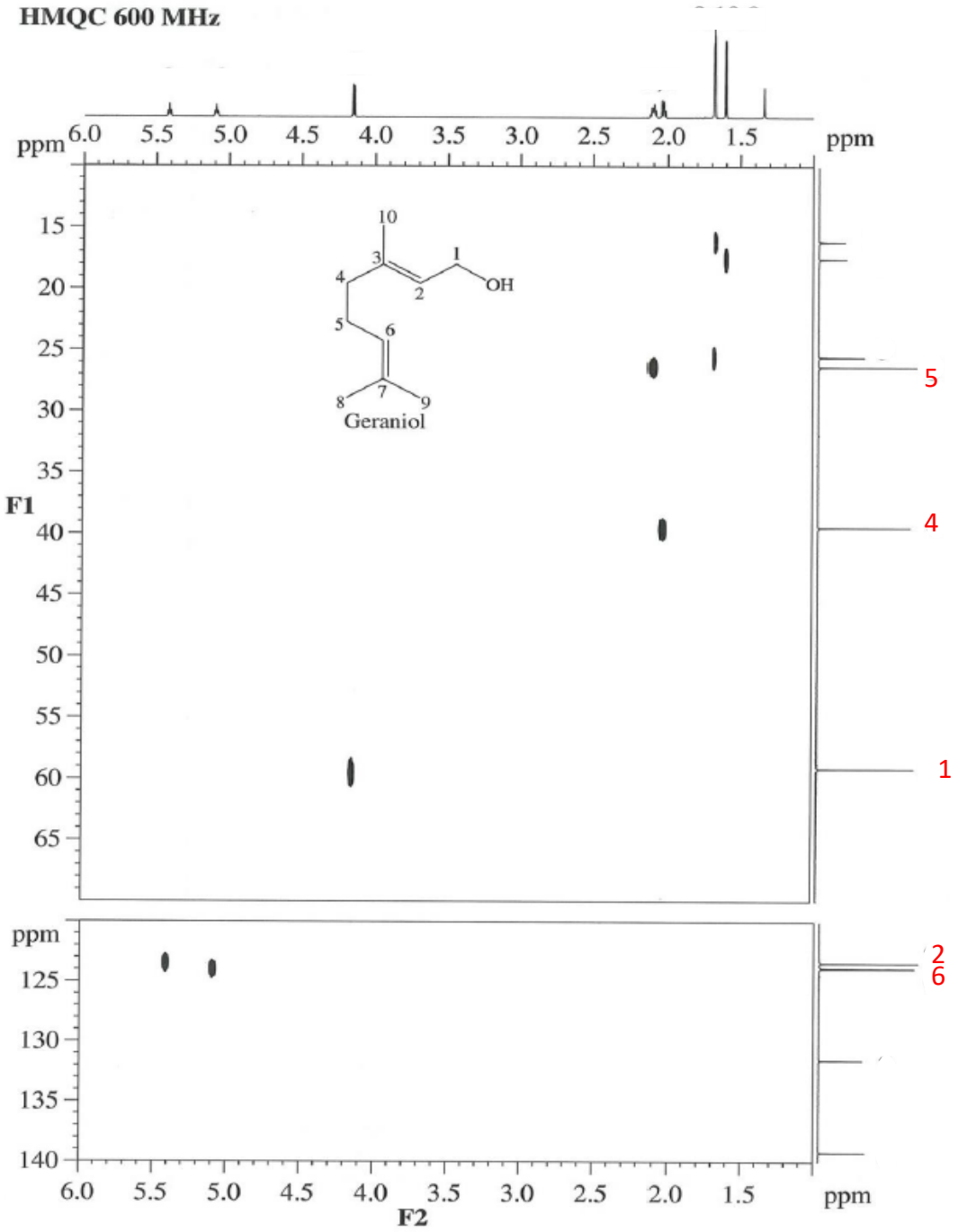
Es. 4



Geraniol

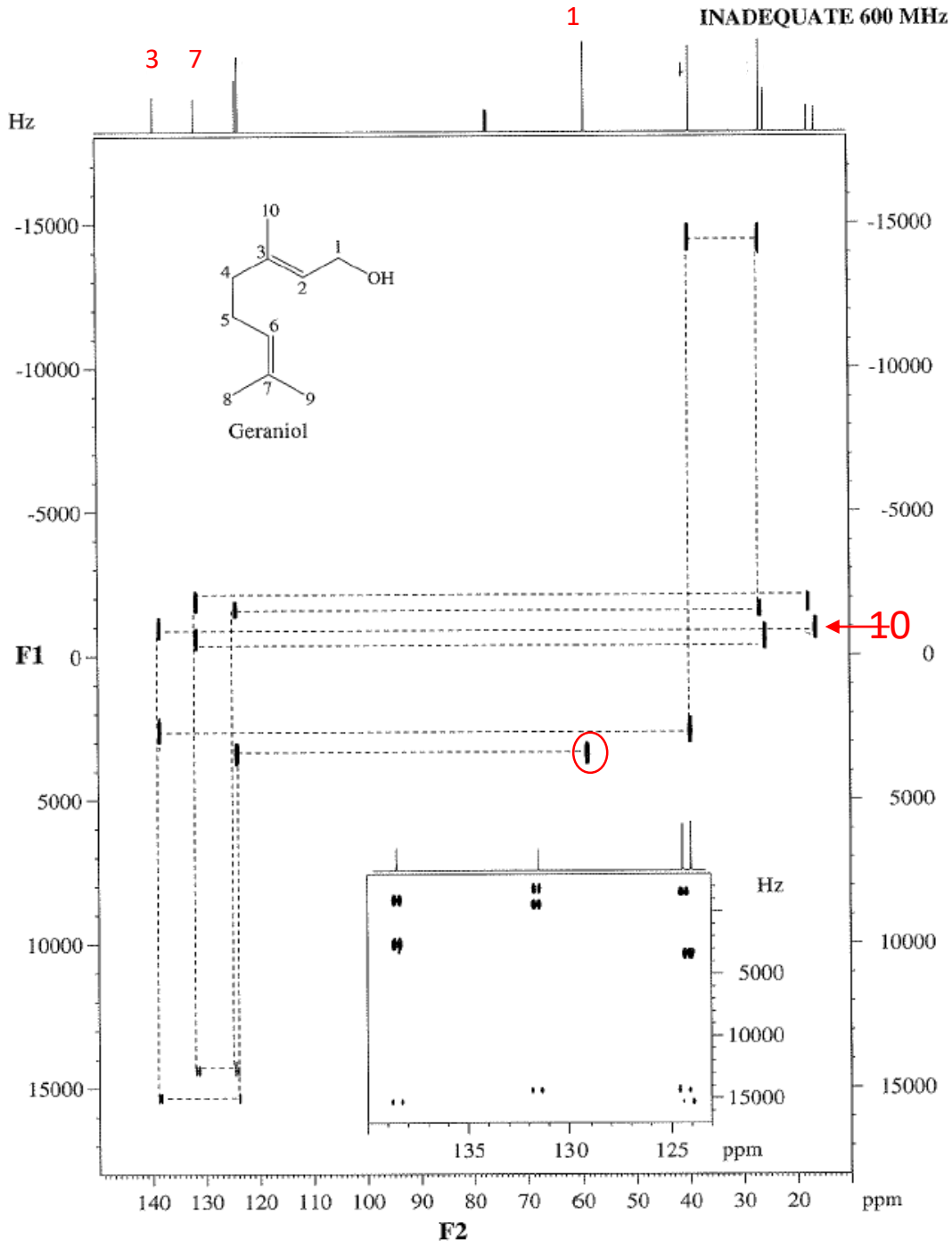
^{13}C/DEPT NMR 150.9 MHz





ES. 4

Mancano 3,7,8,9 e 10



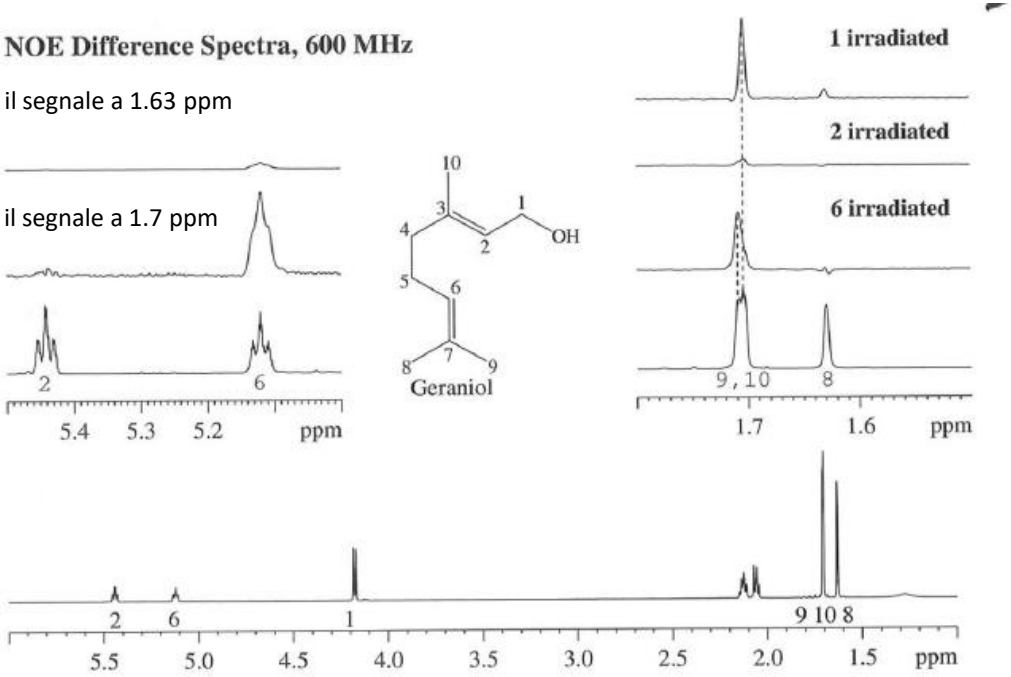
ES. 4

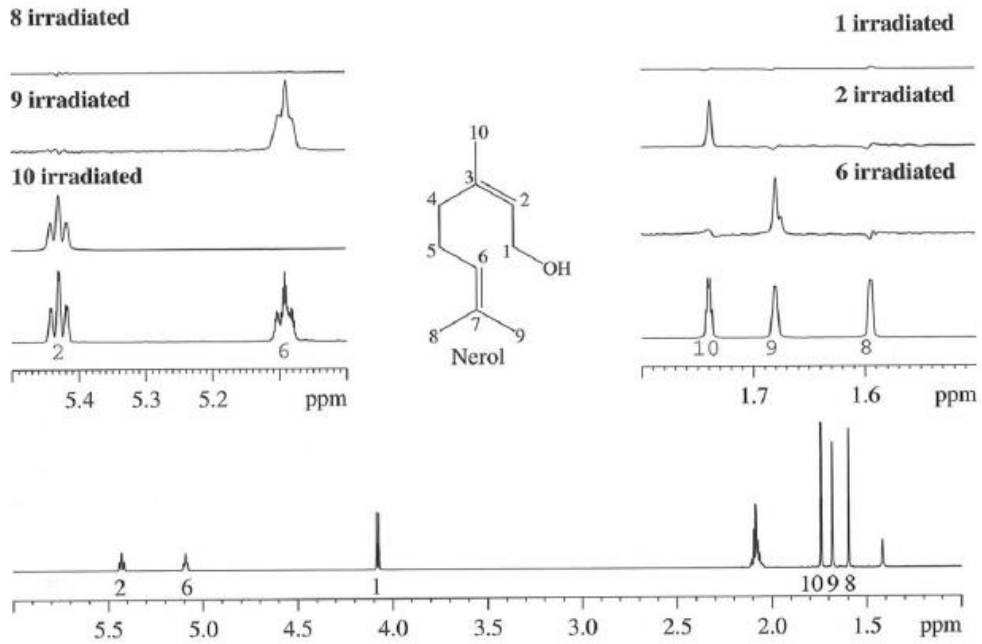
Mancano 8 e 9

ES.4

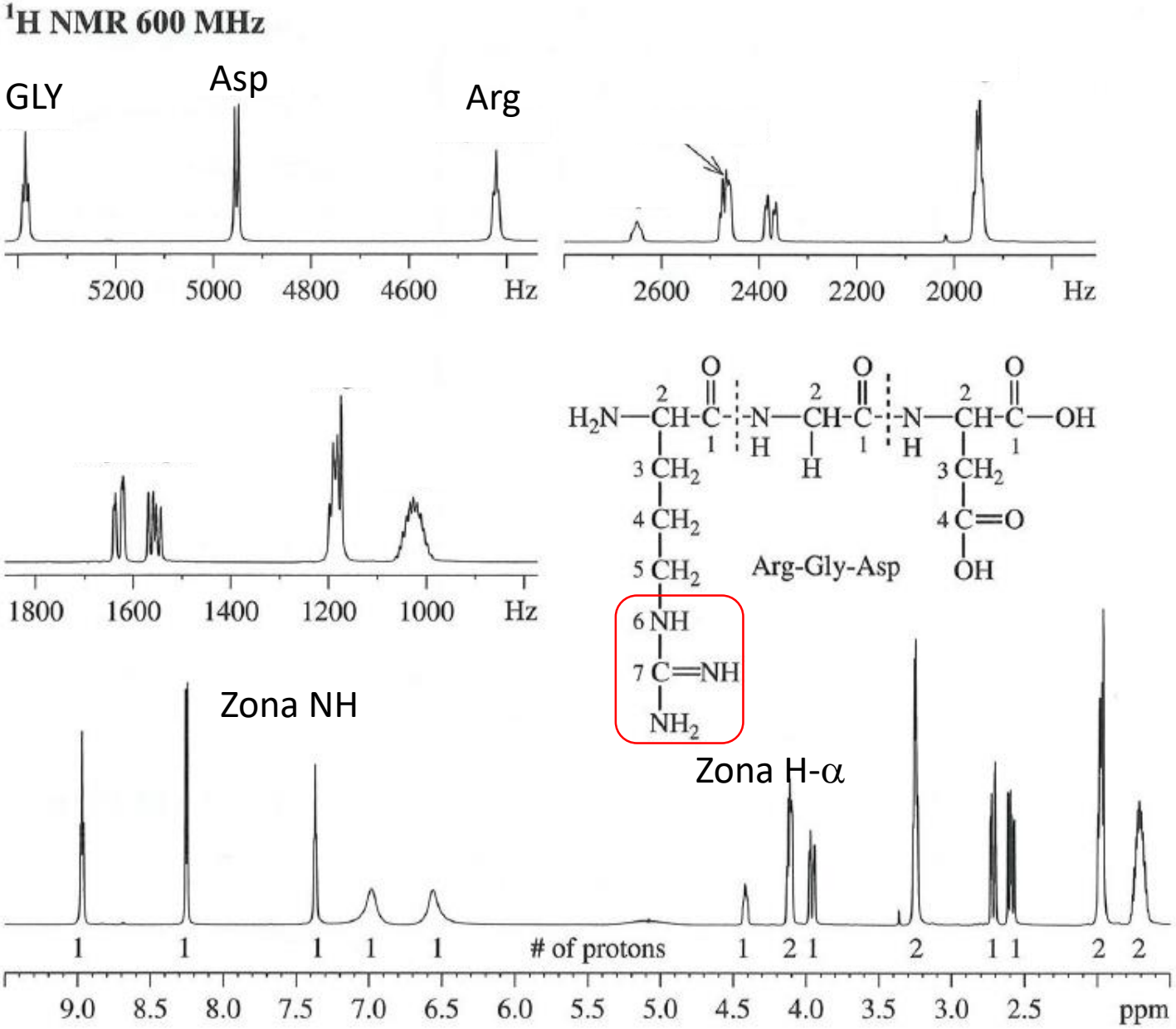
Irraggiato il segnale a 1.63 ppm

Irraggiato il segnale a 1.7 ppm

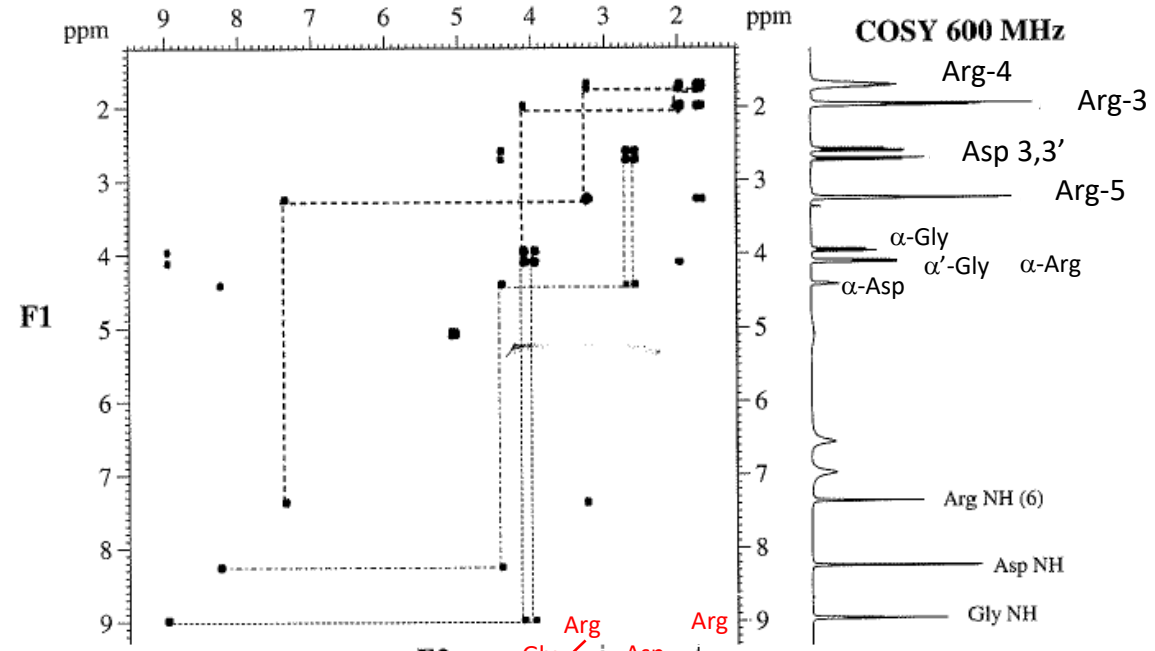


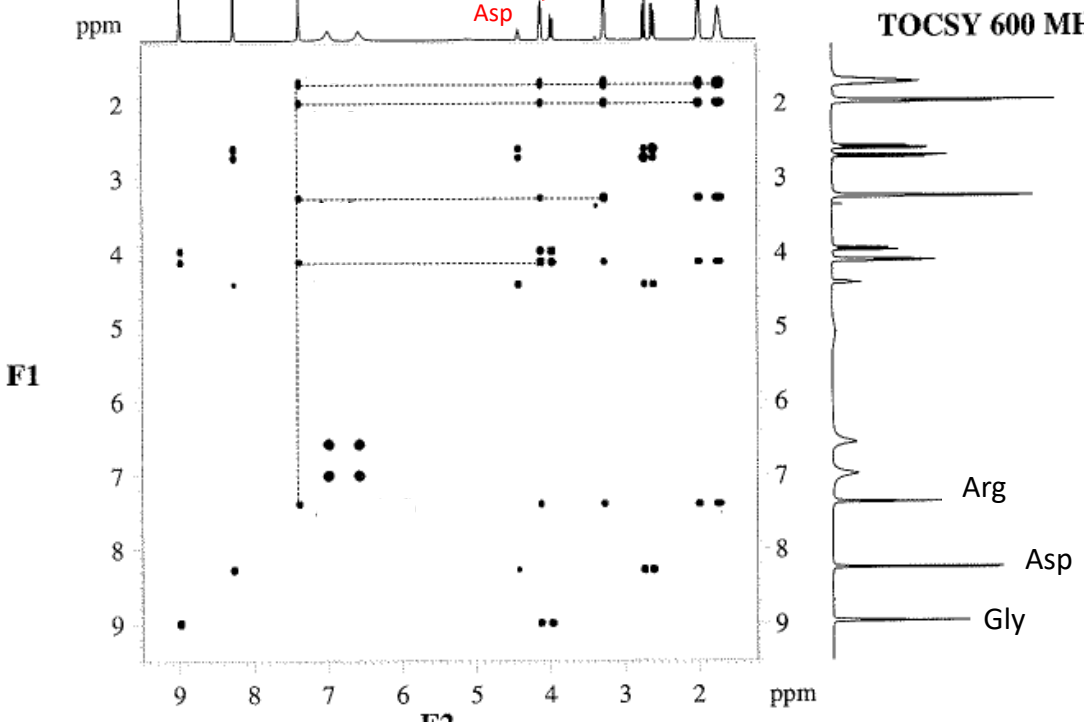


ES.5

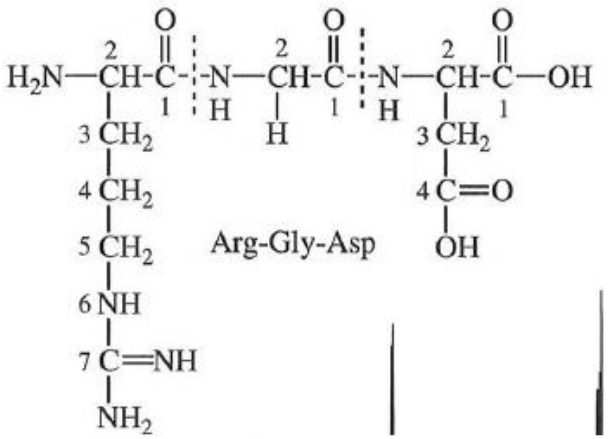

^{1}H NMR 600 MHz
GLY
Asp
Arg
5200
5000
4800
4600
Hz
2600
2400
2200
2000
Hz
1800
1600
1400
1200
1000
Hz
Arg-Gly-Asp
Zona NH
Zona H-α
1
1
1
1
1
# of protons
1
2
1
2
1
1
2
2
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm

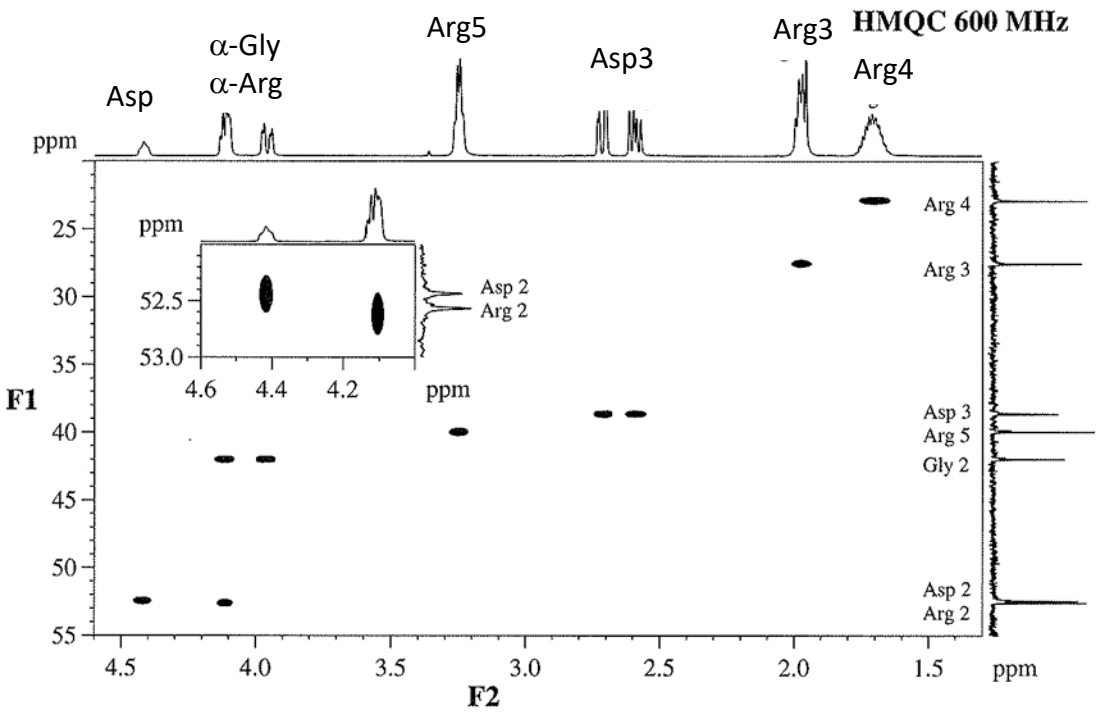
ES

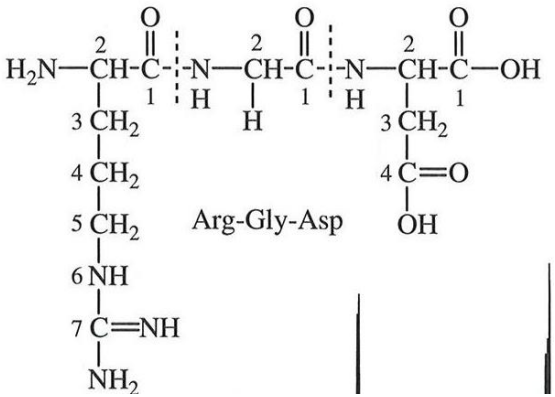




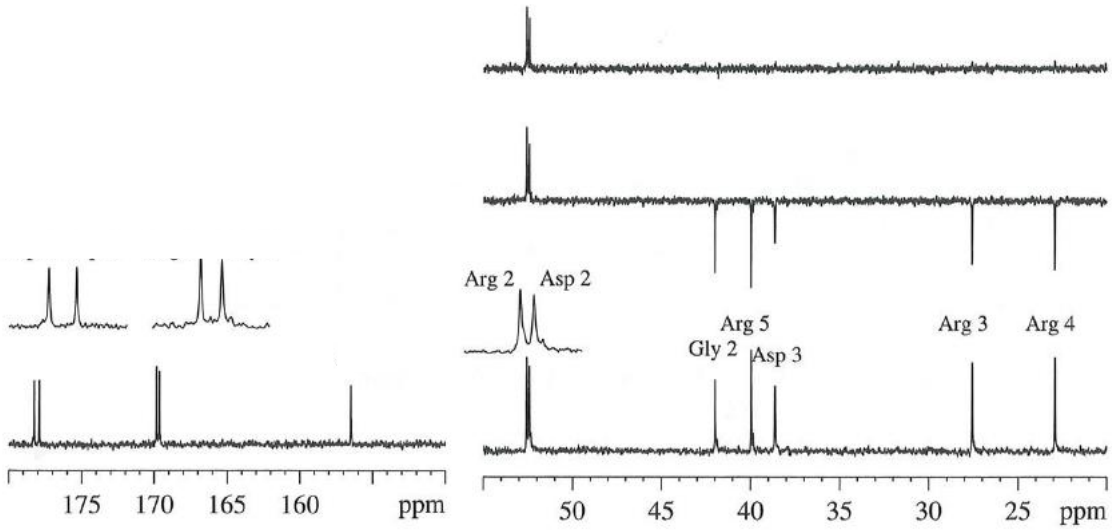
NH Gly -> α,α' Gly
NH Asp -> α Asp -> 3,3' Asp
NH (6) Arg -> CH2 (Arg) -> α-Arg




HMQC 600 MHz
Asp
α-Gly
α-Arg
Arg5
Asp3
Arg3
Arg4
ppm
F1
F2
Arg 4
Arg 3
Asp 3
Arg 5
Gly 2
Asp 2
Arg 2
52.5
53.0
4.6
4.4
4.2

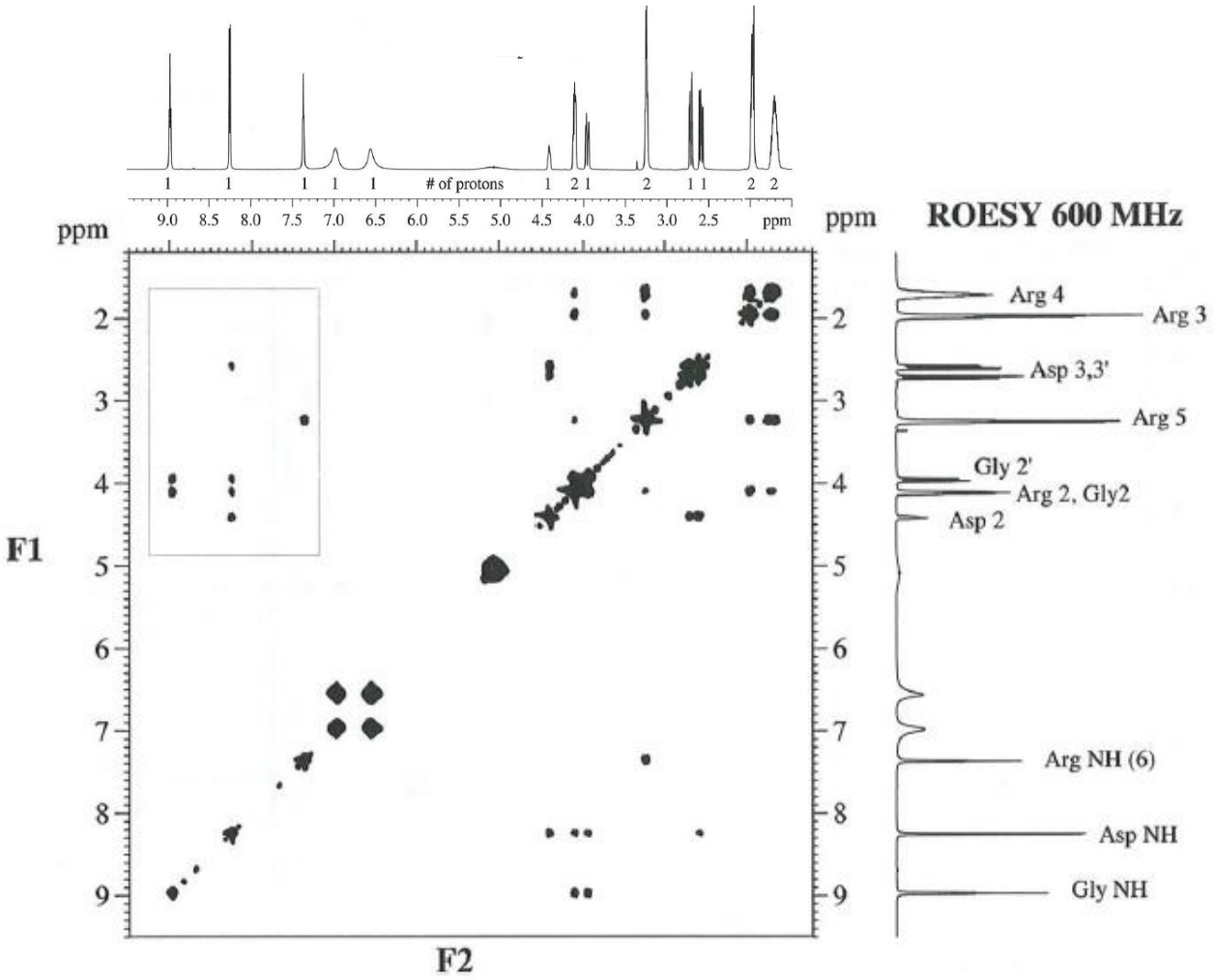

Arg-Gly-Asp

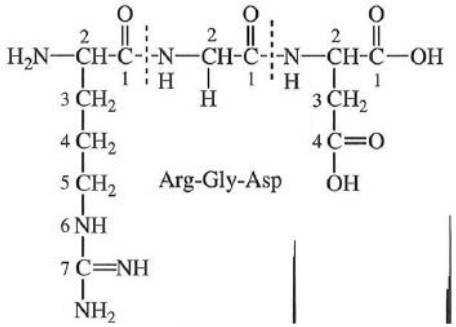
ES.5


13C/DEPT NMR 150.9 MHz
Arg 2
Asp 2
Arg 5
Gly 2
Asp 3
Arg 3
Arg 4
175
170
165
160
ppm
50
45
40
35
30
25
ppm

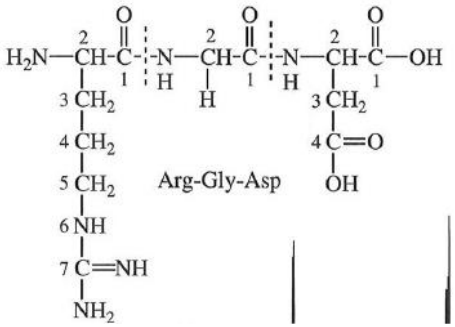
ES.5

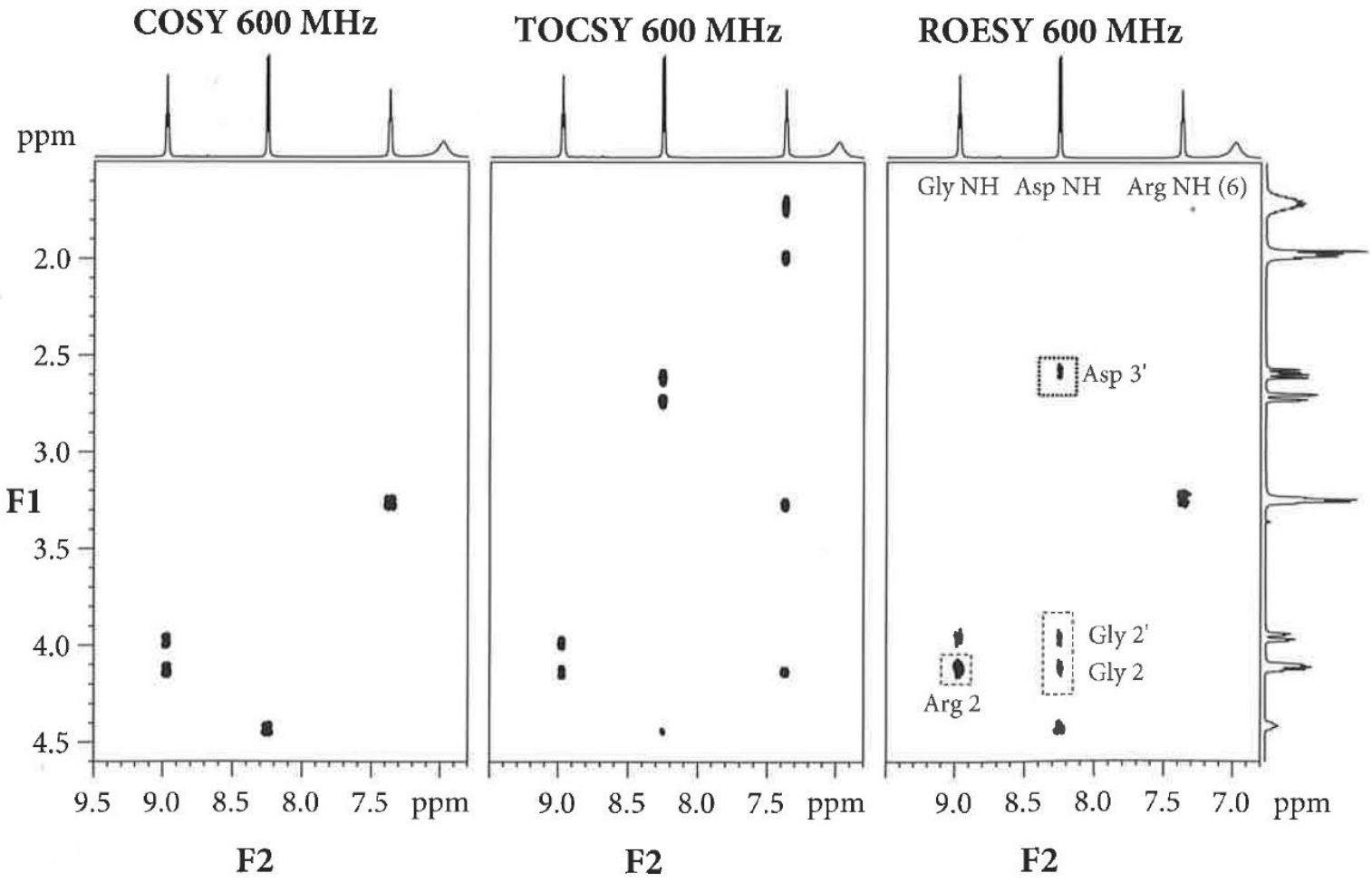
Tutti i cross peaks in antifase tranne quelli a 6.5 e 7.0 ppm



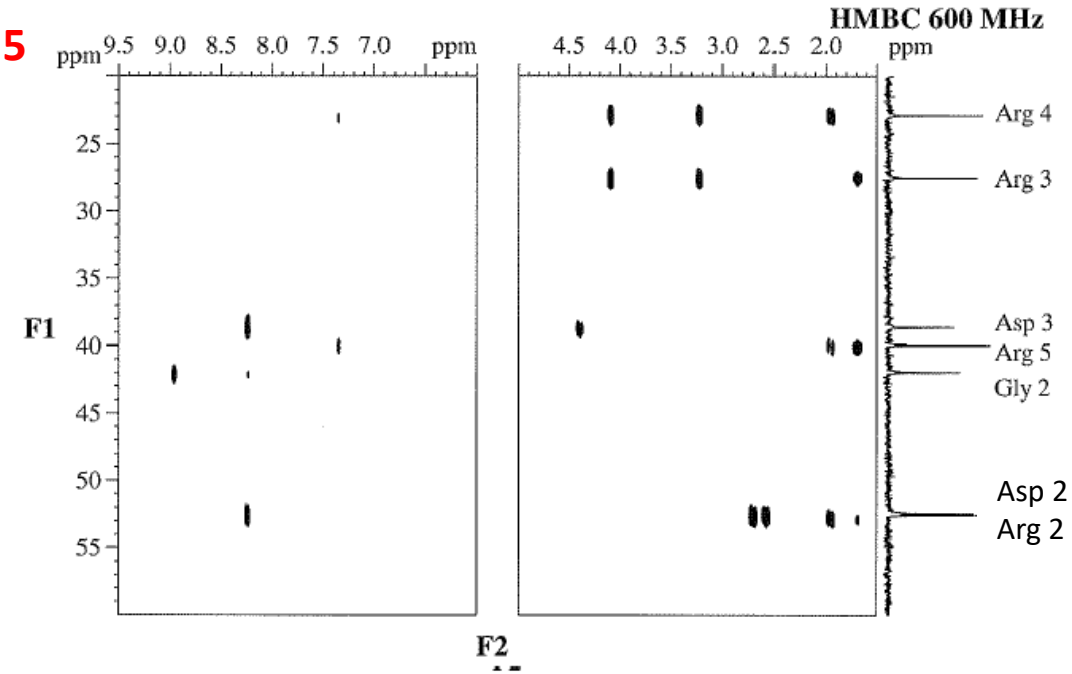


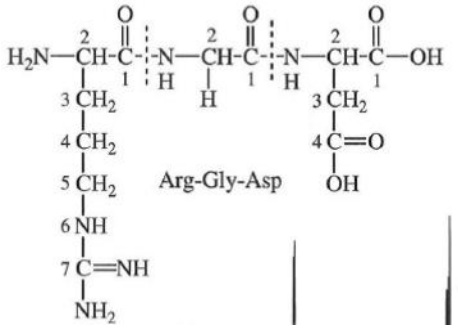
ES.5

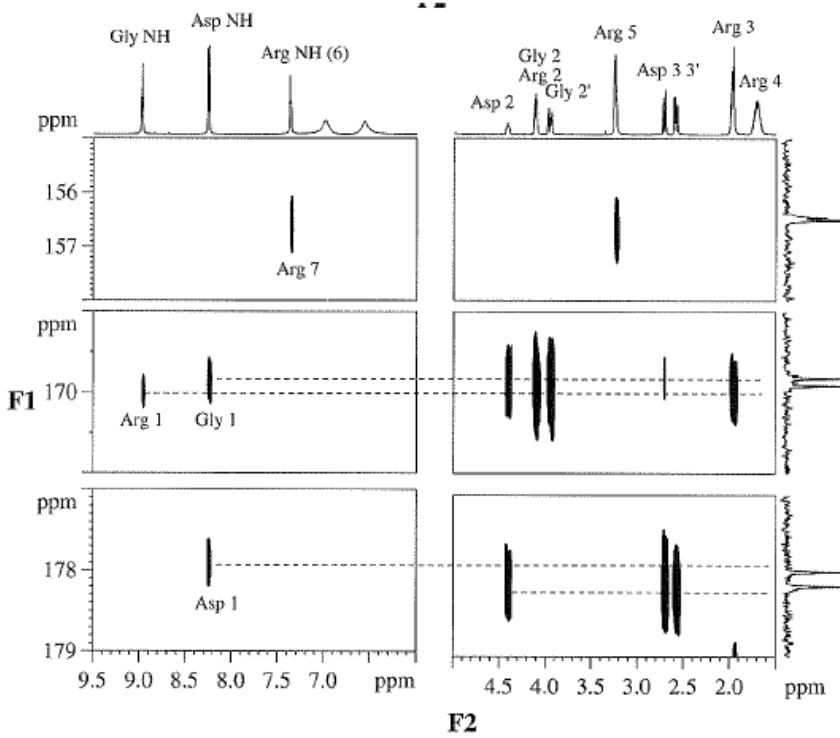

Arg-Gly-Asp


COSY 600 MHz
TOCSY 600 MHz
ROESY 600 MHz
ppm
F1
F2
Gly NH
Asp NH
Arg NH (6)
Asp 3'
Gly 2'
Gly 2
Arg 2

ES.5







Arg 7

Gly 1 (long range su Asp3)

Arg1

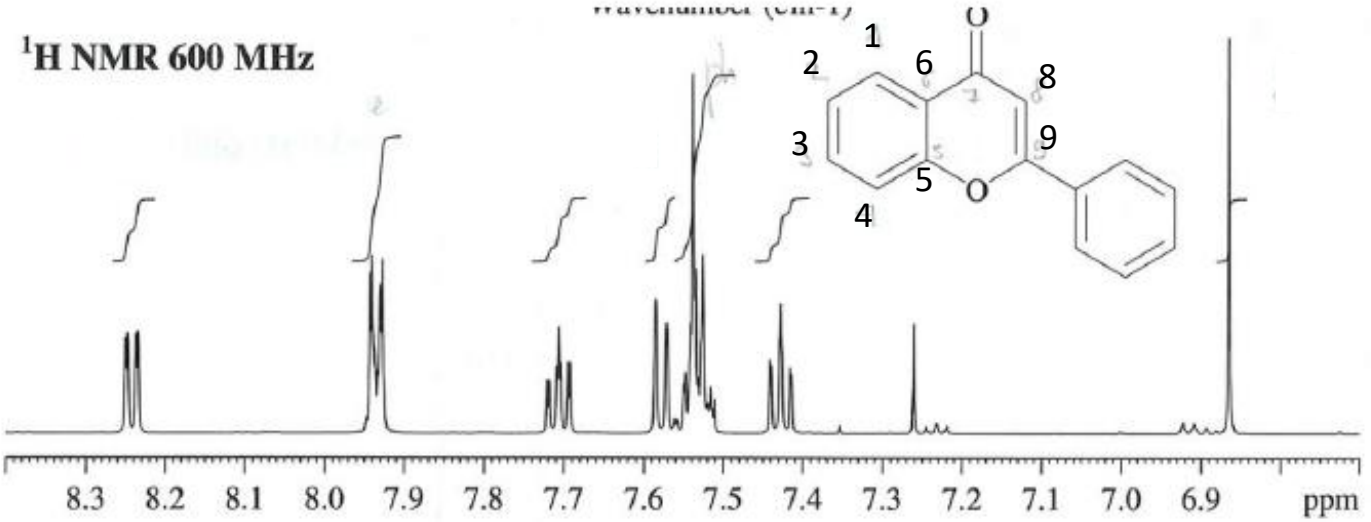
Zona CONH

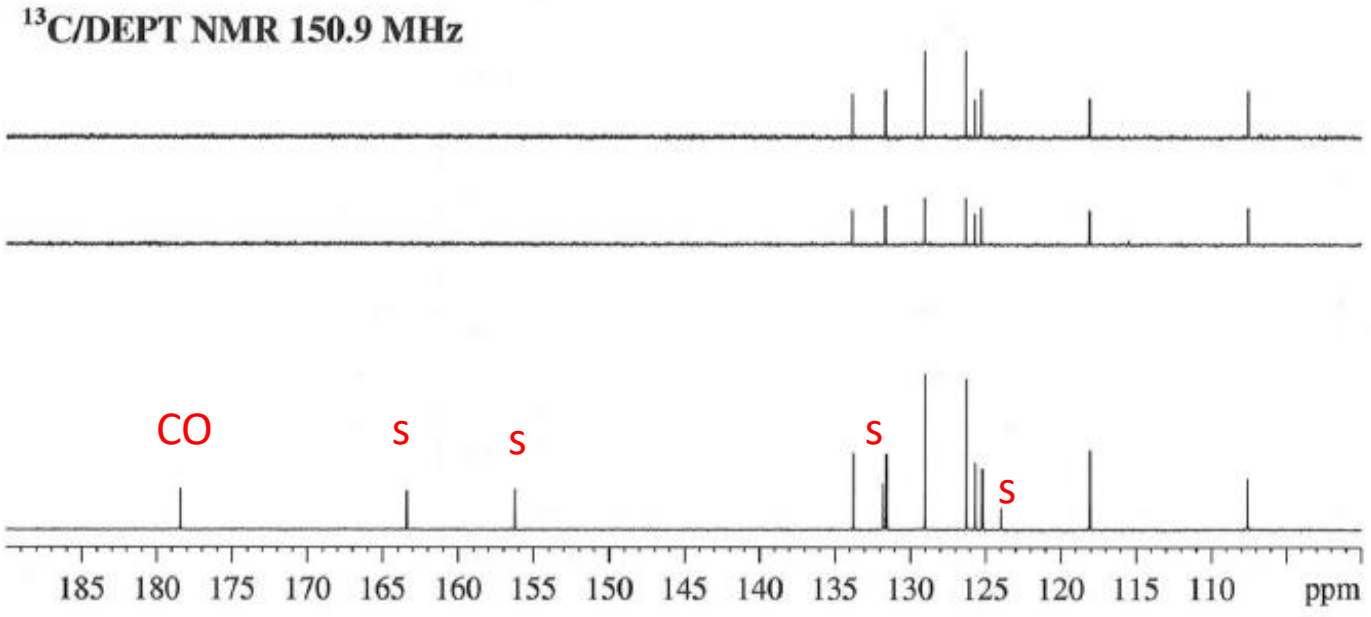
Asp1

Asp4

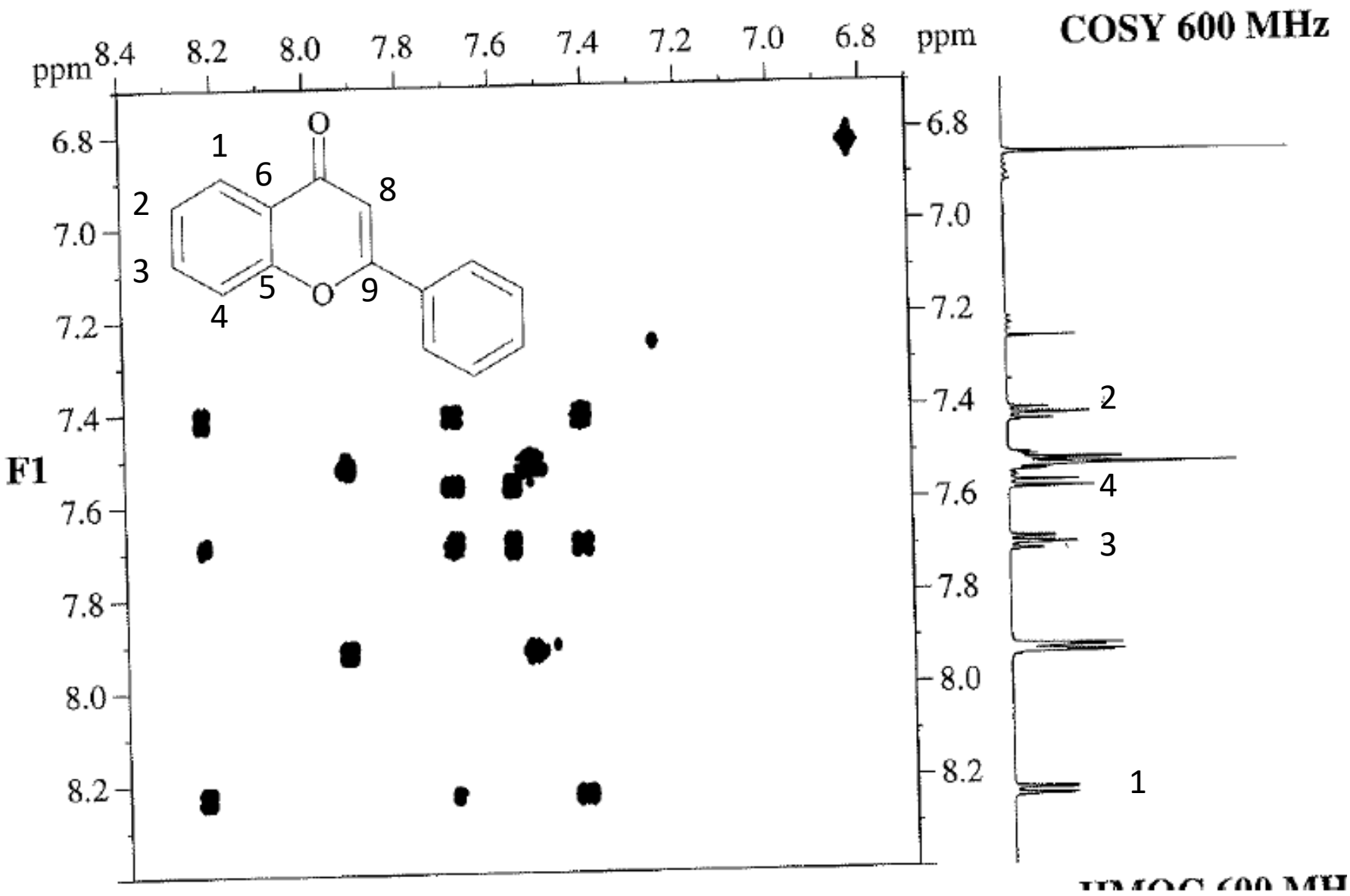
Zona COOH

ES.6


^{1}H NMR 600 MHz
1
2
6
8
3
9
5
O
4
8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 ppm

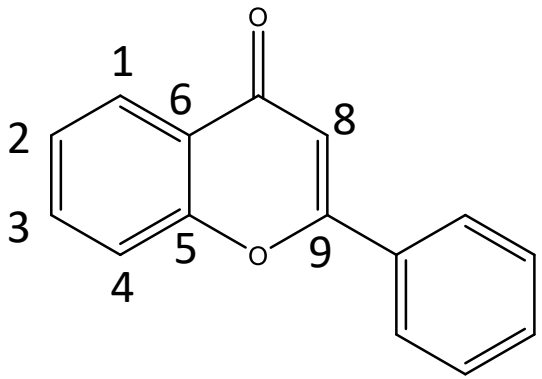

^{13}C/DEPT NMR 150.9 MHz
CO
s
s
s
s
185 180 175 170 165 160 155 150 145 140 135 130 125 120 115 110 ppm

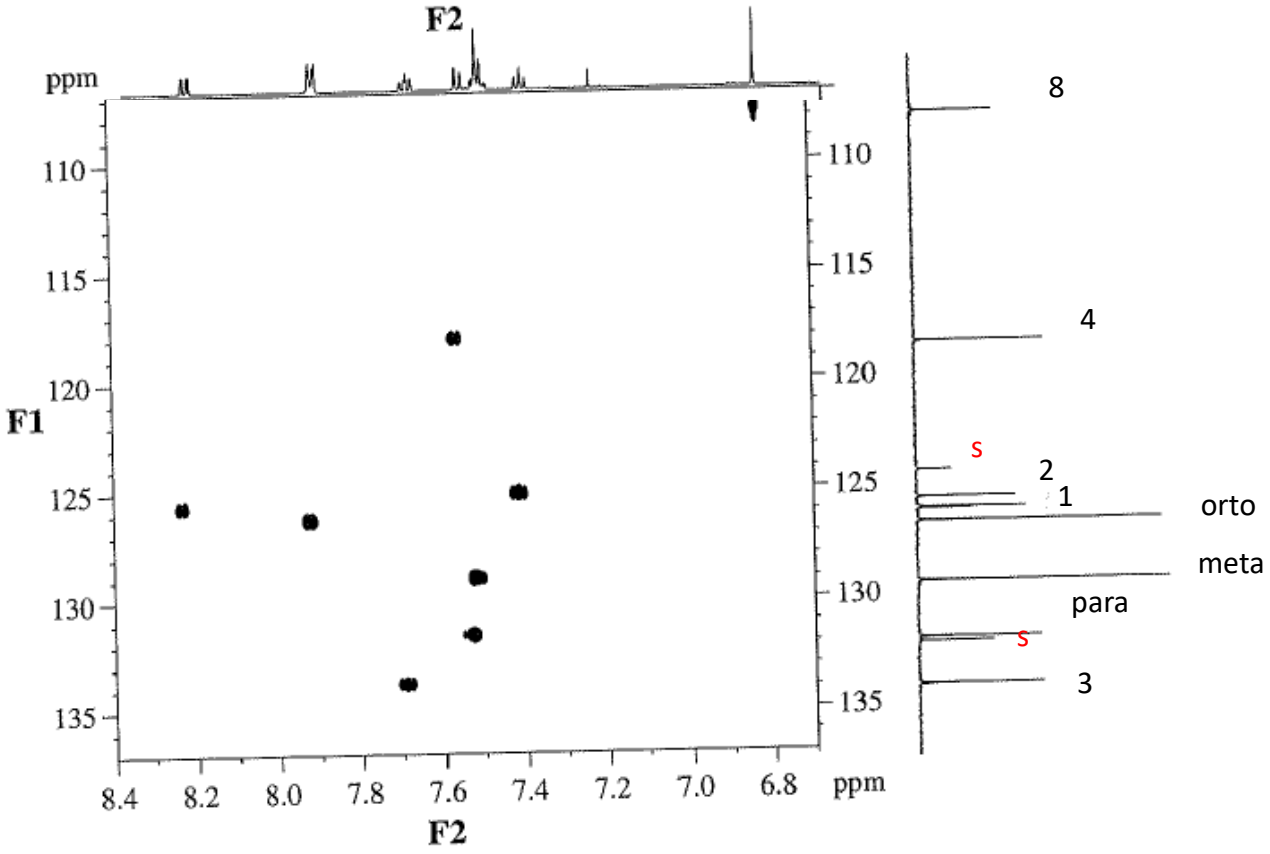
ES.6


COSY 600 MHz
ppm 8.4 8.2 8.0 7.8 7.6 7.4 7.2 7.0 6.8 ppm
F1
6.8
7.0
7.2
7.4
7.6
7.8
8.0
8.2
1
2
3
4
5
6
8
9
O
O
2
4
3
1

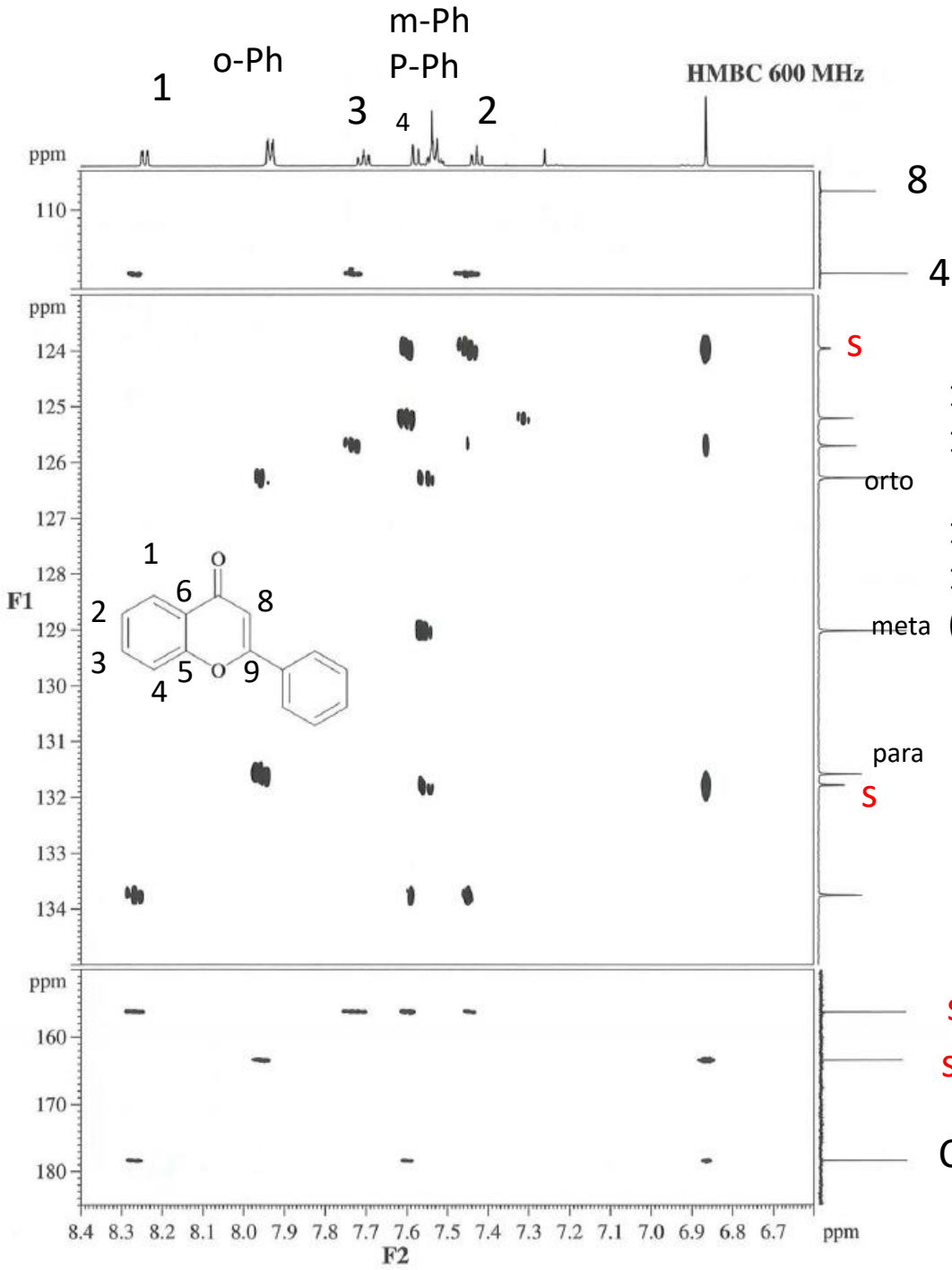
ES.6

HSQC


O
1
6
8
2
3
5
O
9
4


F2
ppm
110
115
120
125
130
135
F1
8.4
8.2
8.0
7.8
7.6
7.4
7.2
7.0
6.8
ppm
F2
8
4
s
2
1
orto
meta
para
s
3

ES.6

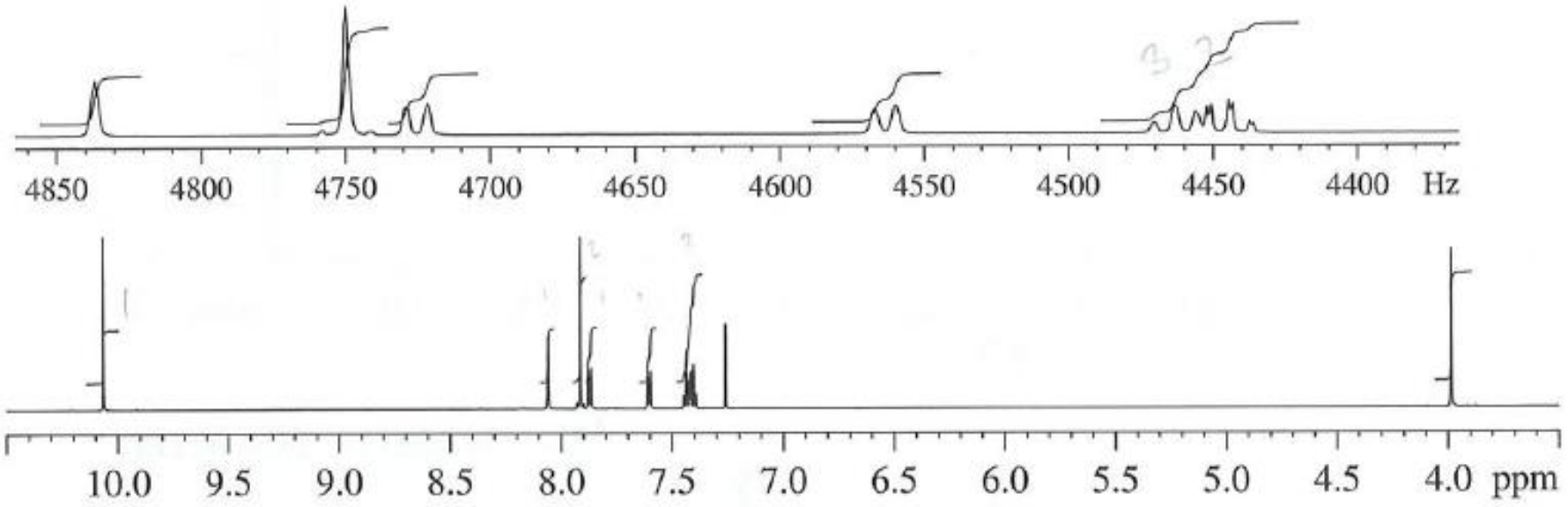


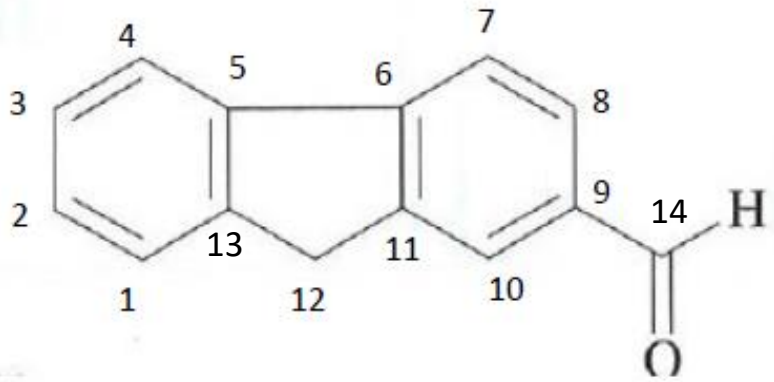
125 accoppia con 8, 2, 4 : 6
132 accoppia con 8, o, m,p : 10

162 accoppia con 8, o-Ph: 9
155 accoppia con 1,2,3,4: 5
(per esclusione)

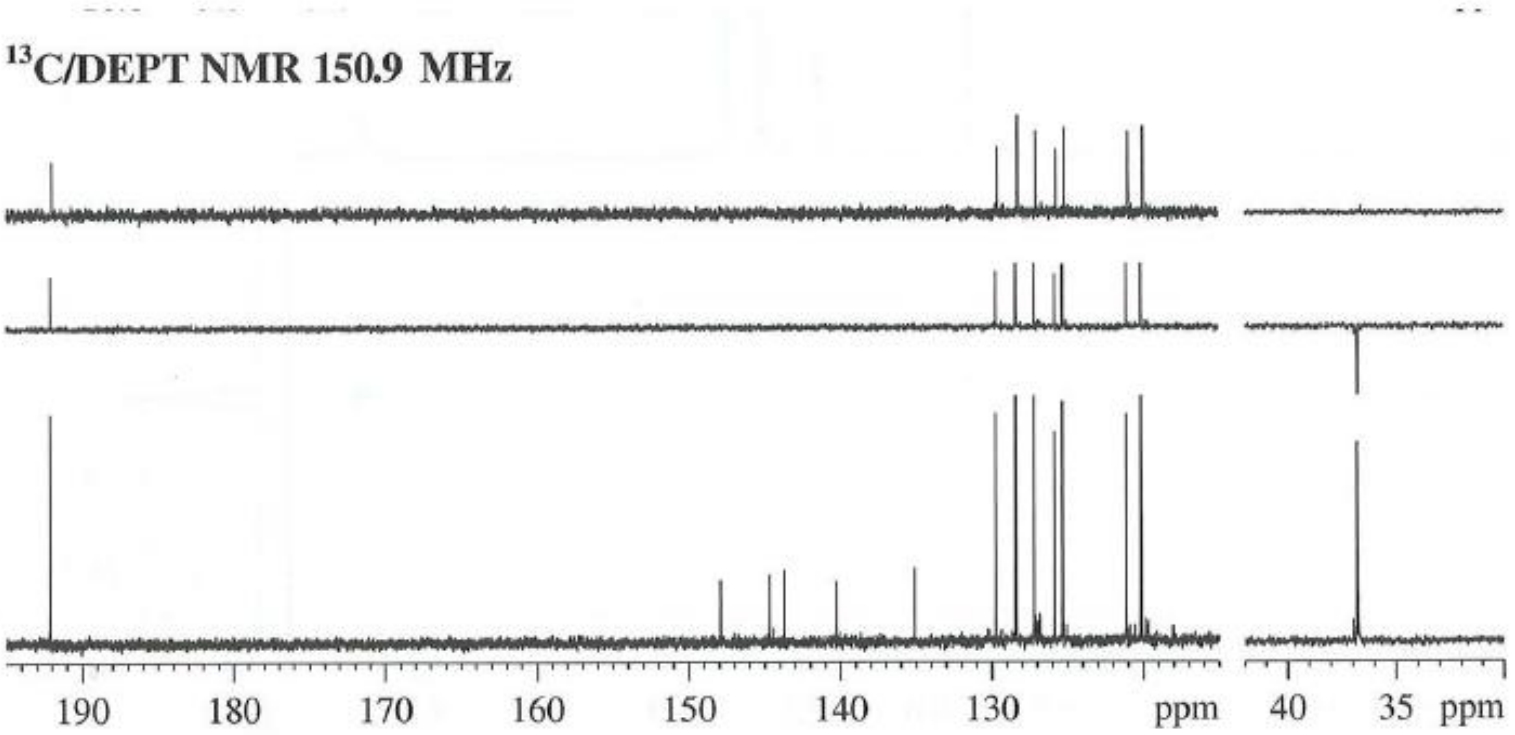
ES.7

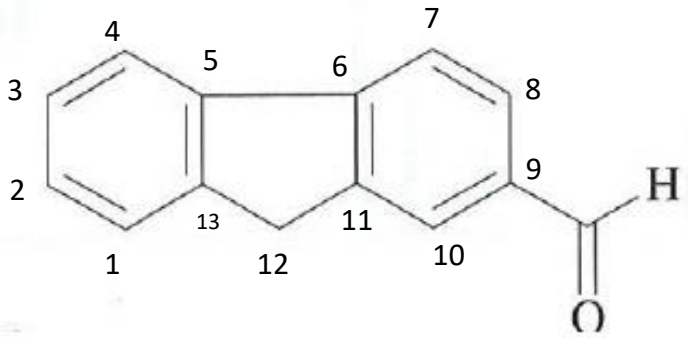
^{1}H NMR 600 MHz



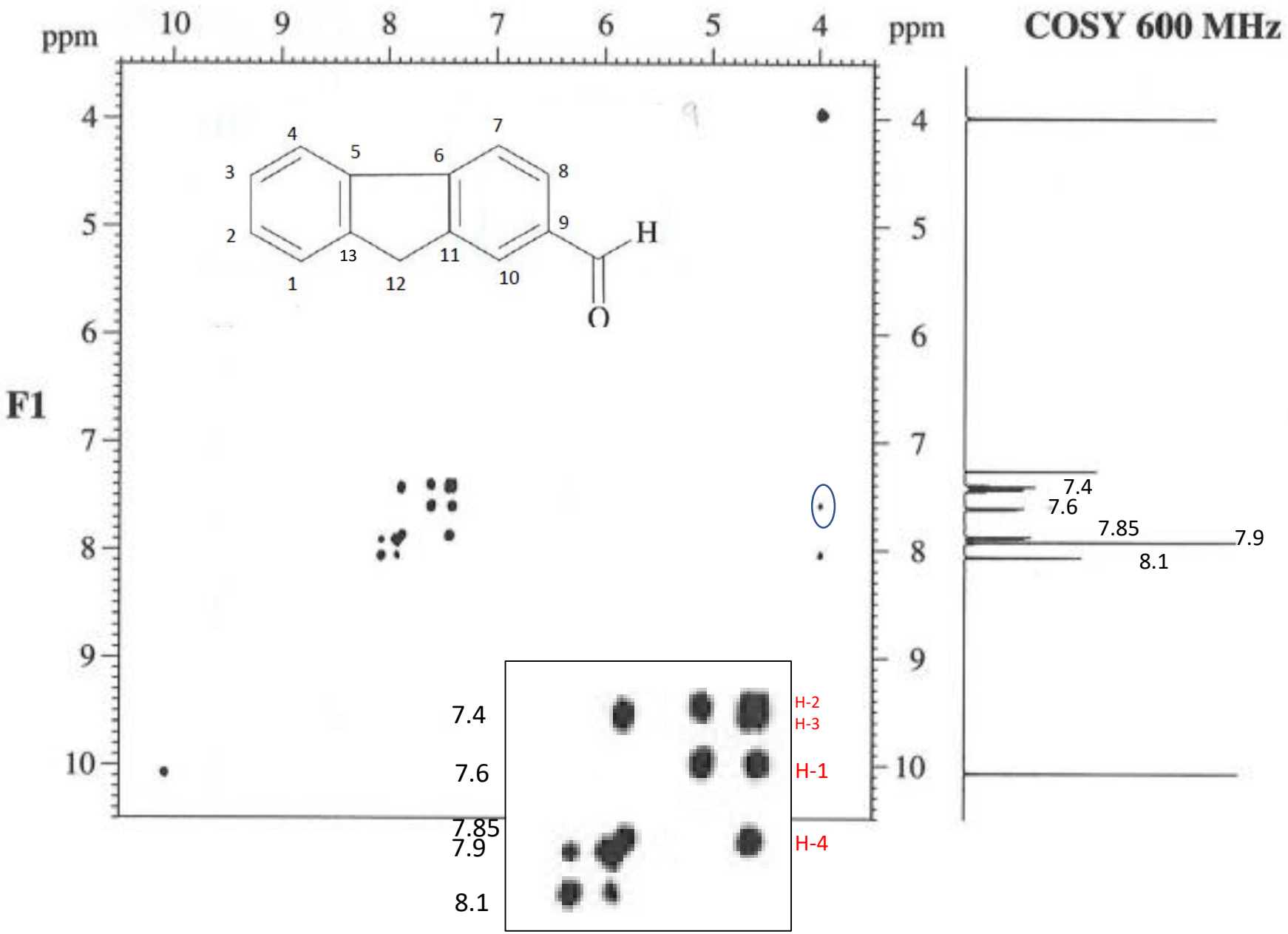


ES.7

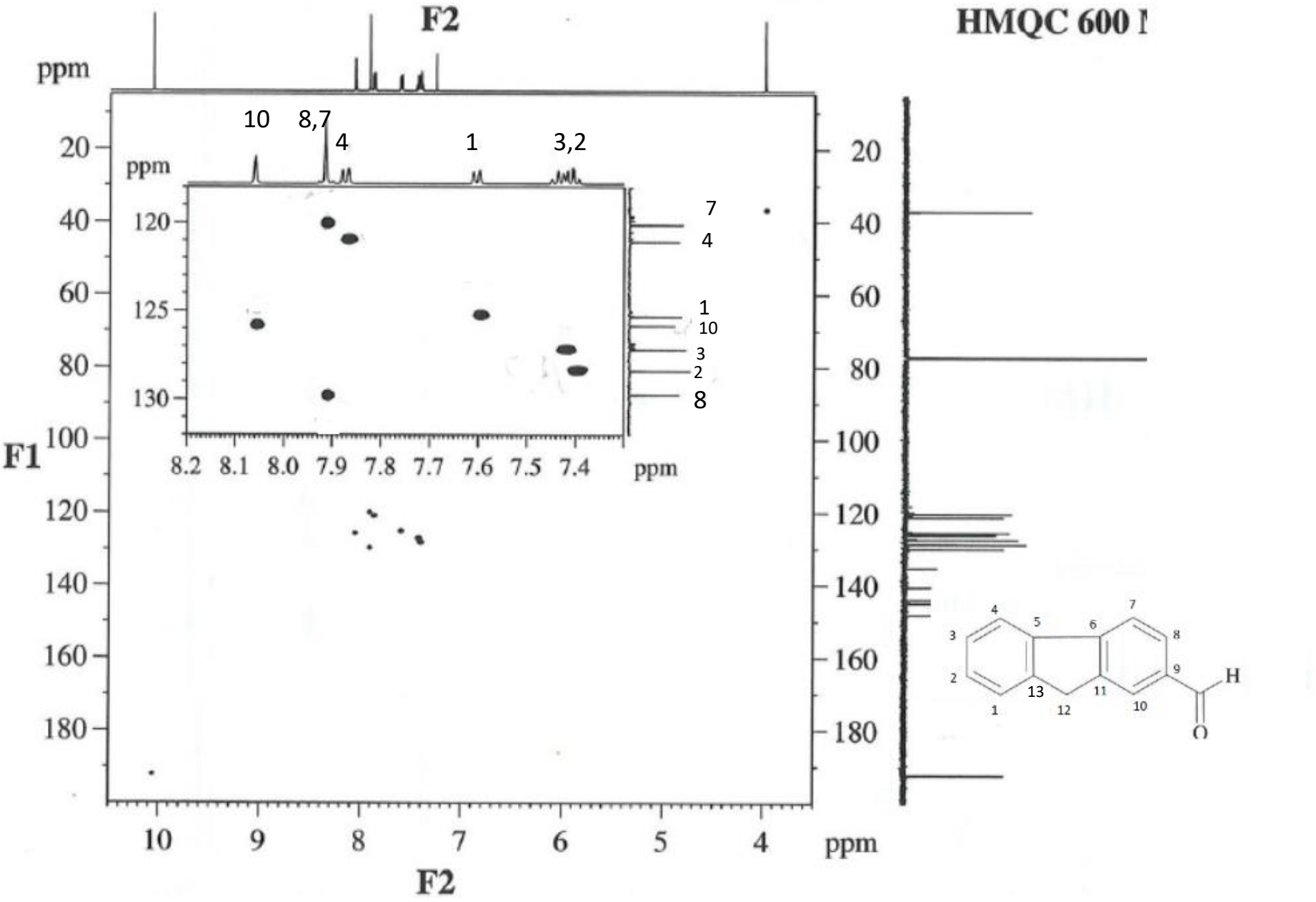

^{13}C/DEPT NMR 150.9 MHz
190
180
170
160
150
140
130
ppm
40
35
ppm


1
2
3
4
5
6
7
8
9
10
11
12
13
H
O

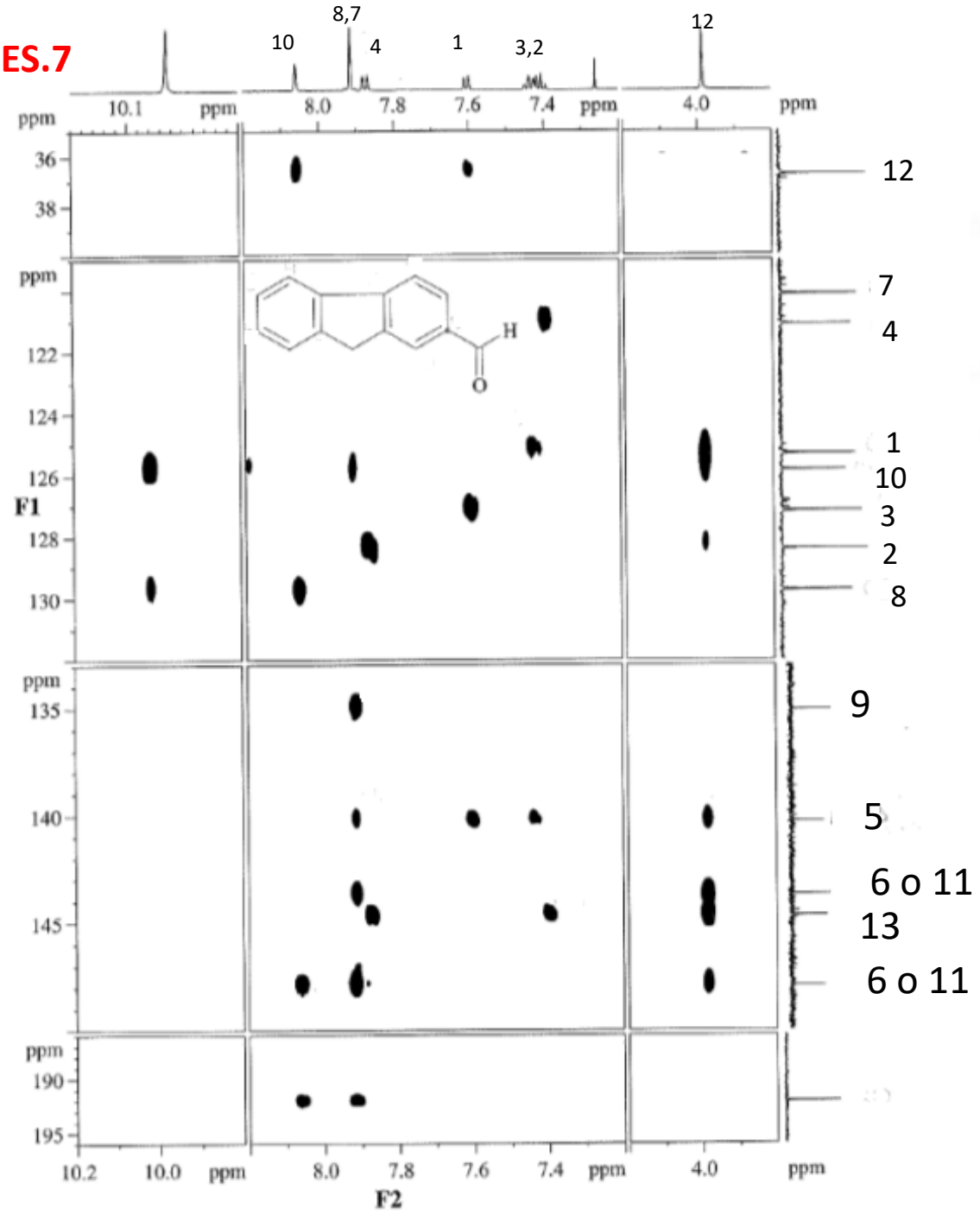
ES.7


COSY 600 MHz
ppm
10
9
8
7
6
5
4
F1
4
5
6
7
8
9
10
7.4
7.6
7.85
7.9
8.1
H-2
H-3
H-1
H-4
H
O

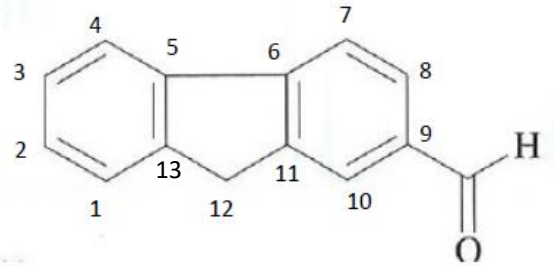
ES.7


HMQC 600 I
F2
ppm
F1
10
8,7
4
1
3,2
7
4
1
10
3
2
8
8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4
20 40 60 80 100 120 140 160 180
10 9 8 7 6 5 4 ppm
F2

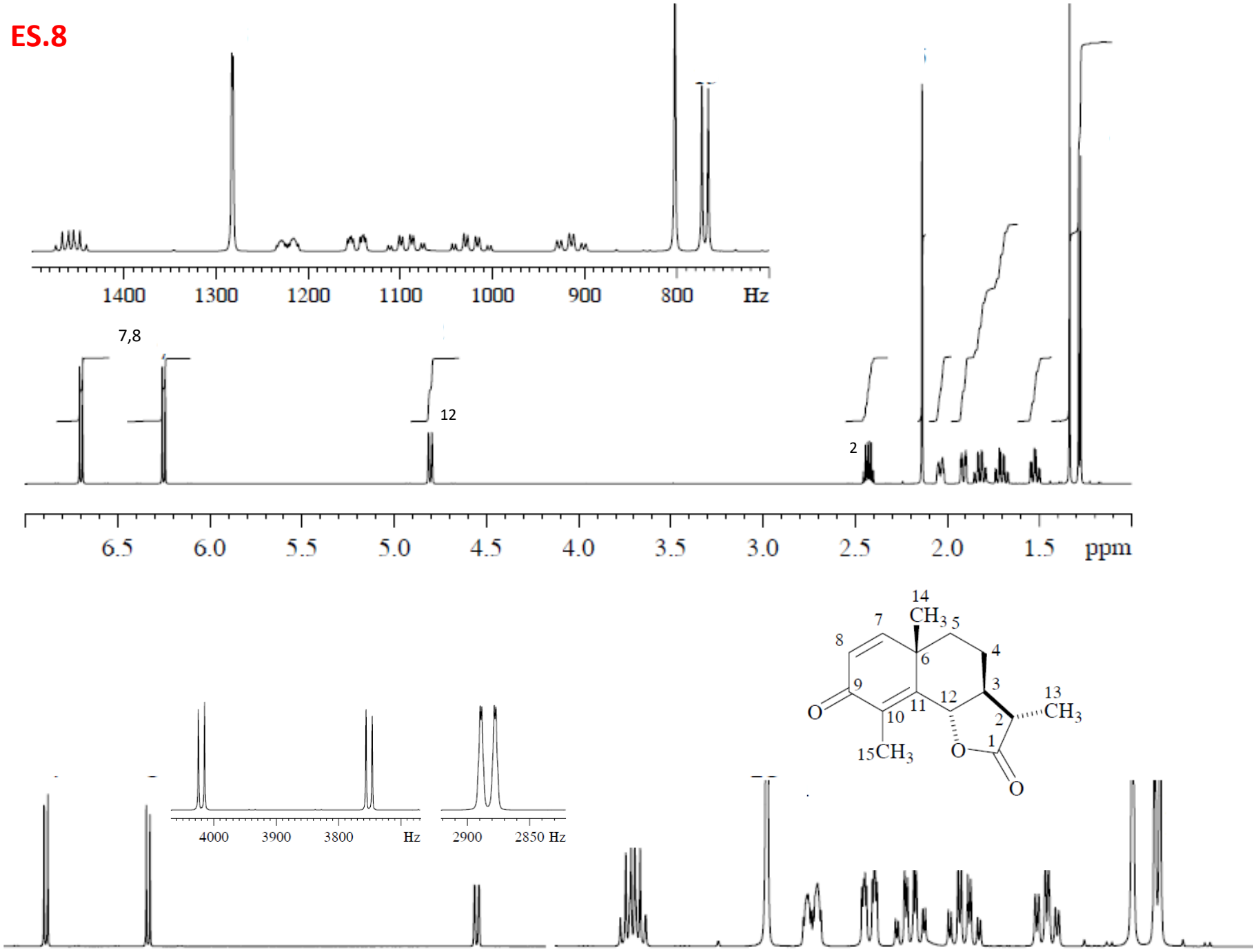
ES.7


10
8,7
4
1
3,2
12
ppm
10.1
8.0
7.8
7.6
7.4
4.0
36
38
122
124
126
128
130
F1
135
140
145
190
195
10.2
10.0
8.0
7.8
7.6
7.4
4.0
F2
12
7
4
1
10
3
2
8
9
5
6 o 11
13
6 o 11

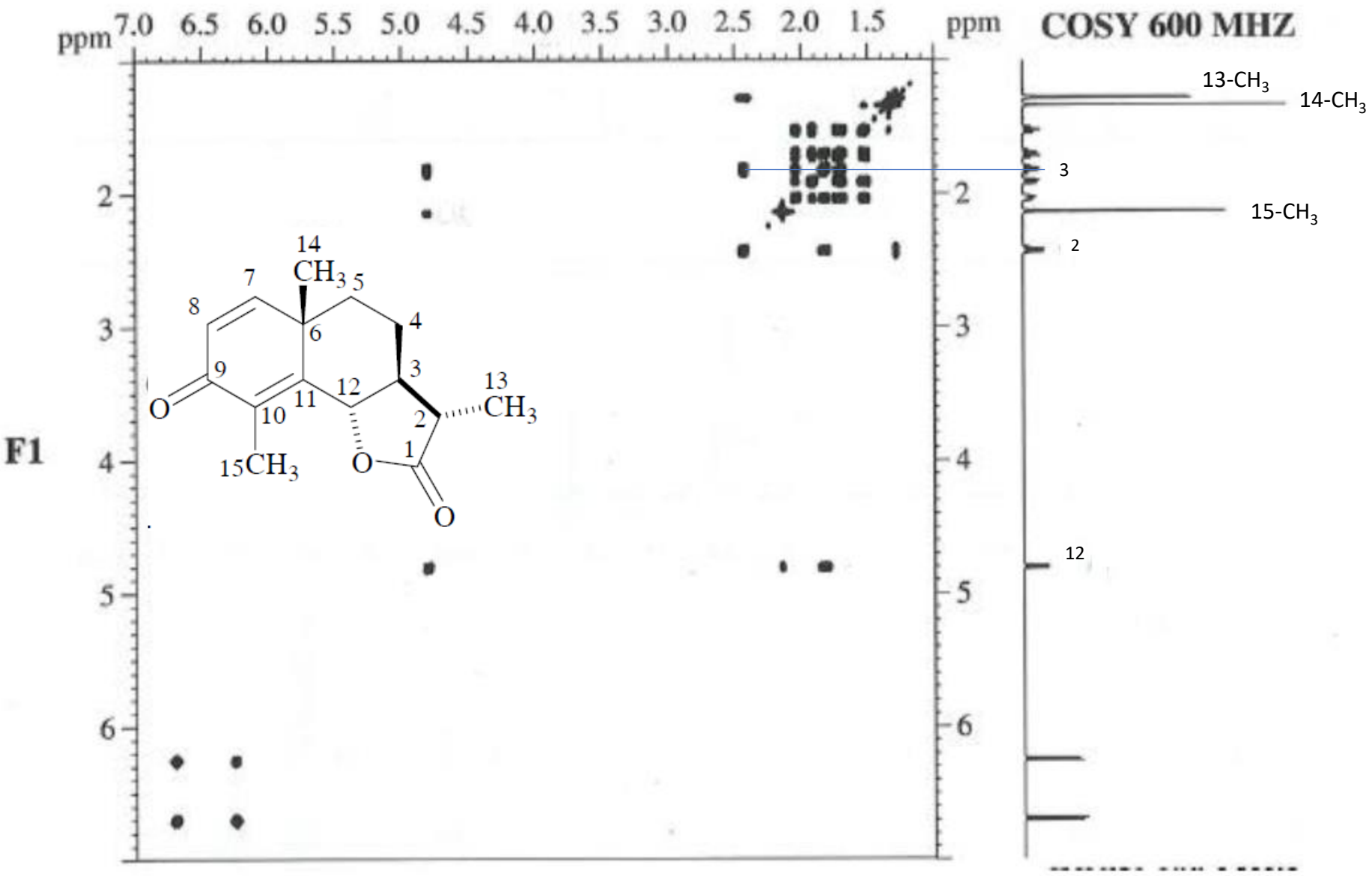
HMBC


4
5
6
7
3
8
2
9
H
13
11
1
12
10
O

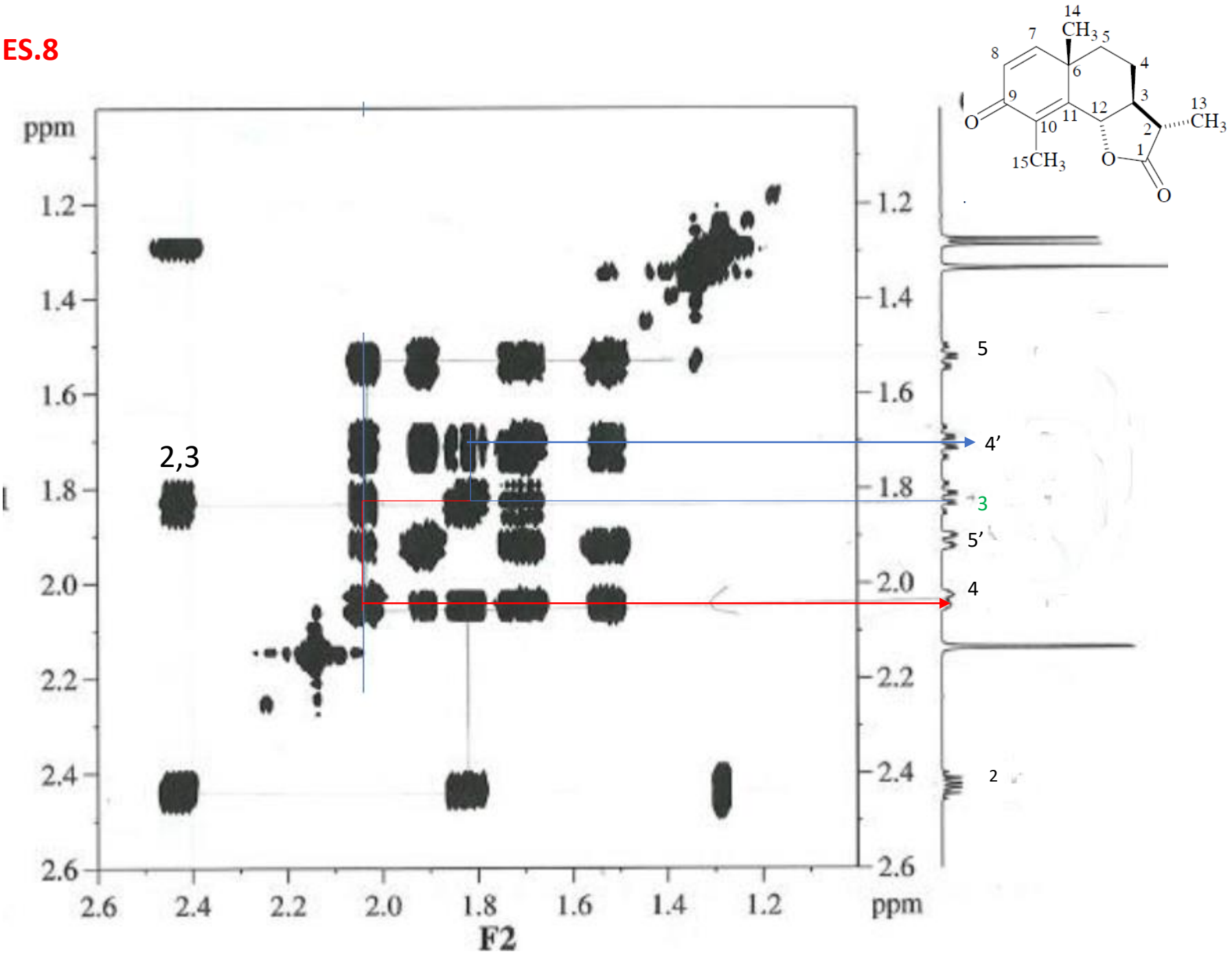
ES.8


1400
1300
1200
1100
1000
900
800
Hz
7,8
12
2
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm
14
CH3
7
5
8
6
4
9
3
13
CH3
O
10
11
12
2
15CH3
1
O
O
4000
3900
3800
Hz
2900
2850 Hz

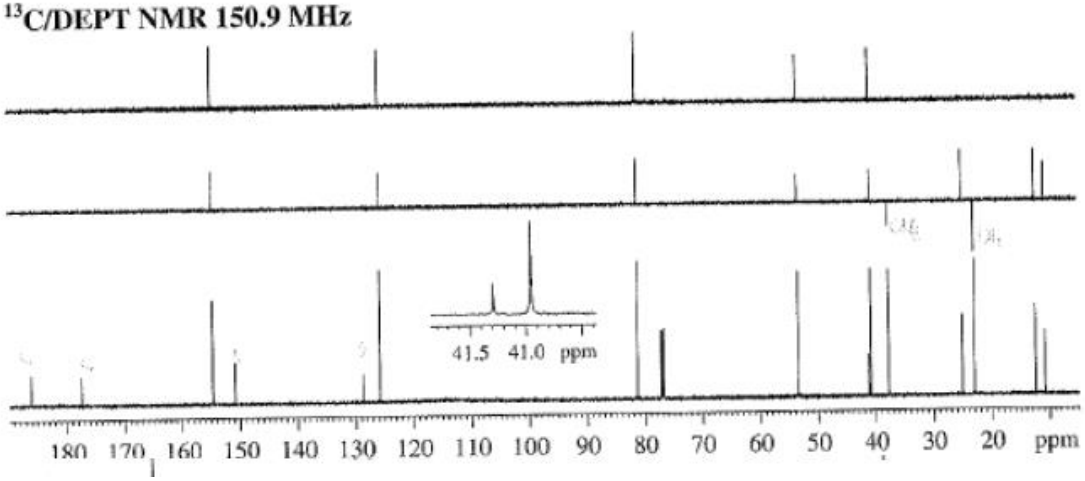
ES.8

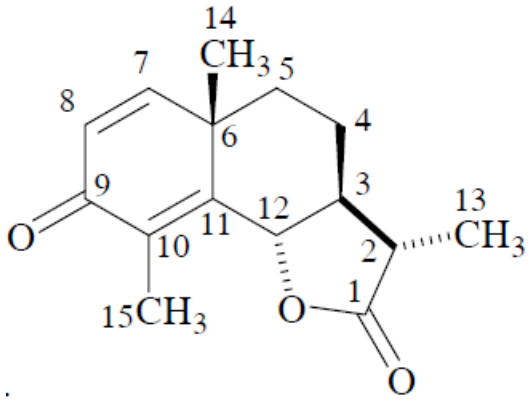

COSY 600 MHZ
ppm
7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5
F1
2
3
4
5
6
13-CH3
14-CH3
15-CH3
3
2
12
14
CH3
7
5
8
6
4
9
3
12
13
CH3
O
11
10
2
1
15CH3
O
O

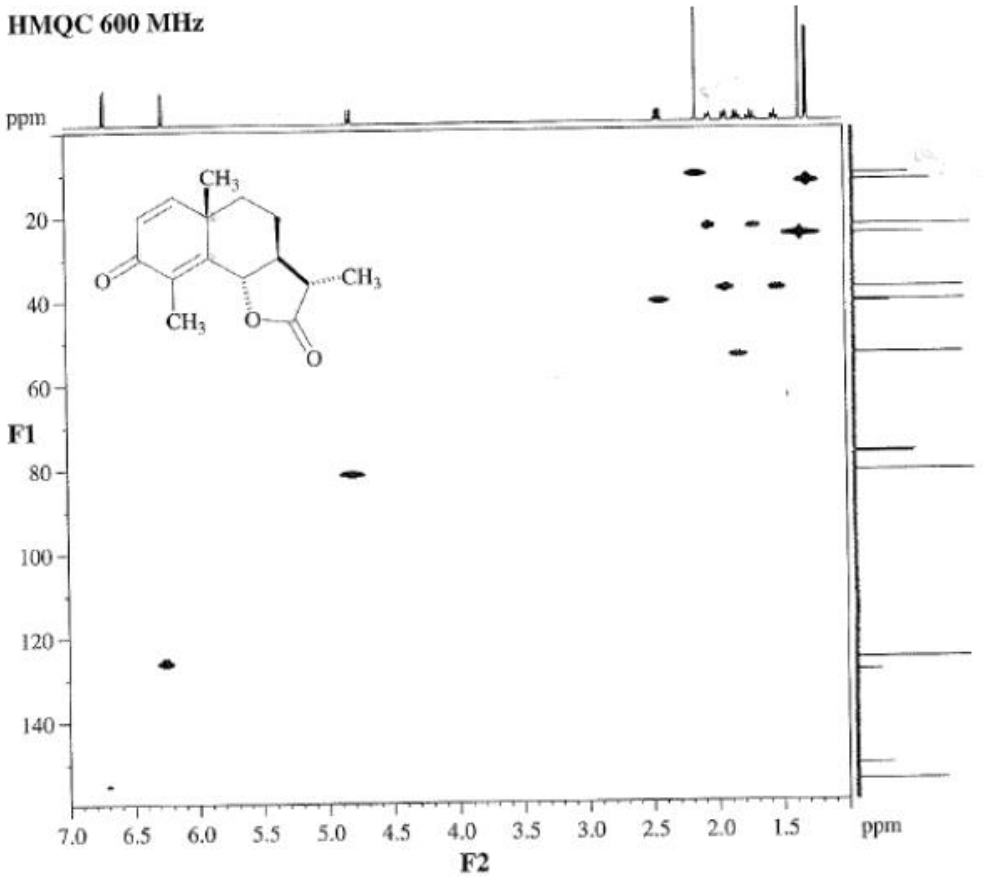
ES.8


ppm
1.2
1.4
1.6
1.8
2.0
2.2
2.4
2.6
2,3
F2
5
4’
3
5’
4
2
14
CH3
7
8
6
5
4
9
3
13
CH3
O
10
11
12
2
1
15CH3
O
O

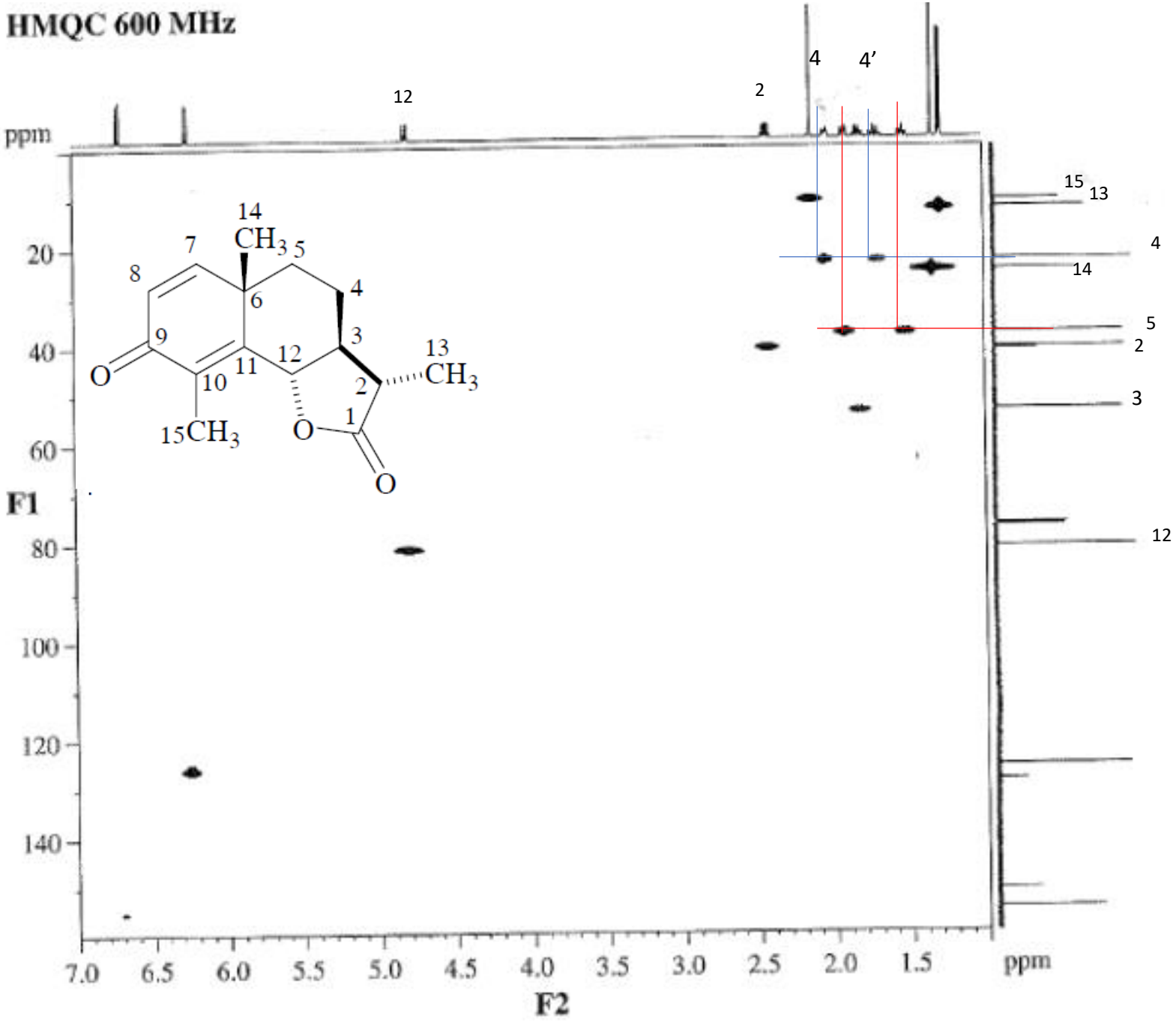
ES.8


^{13}C/DEPT NMR 150.9 MHz
41.5 41.0 ppm
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

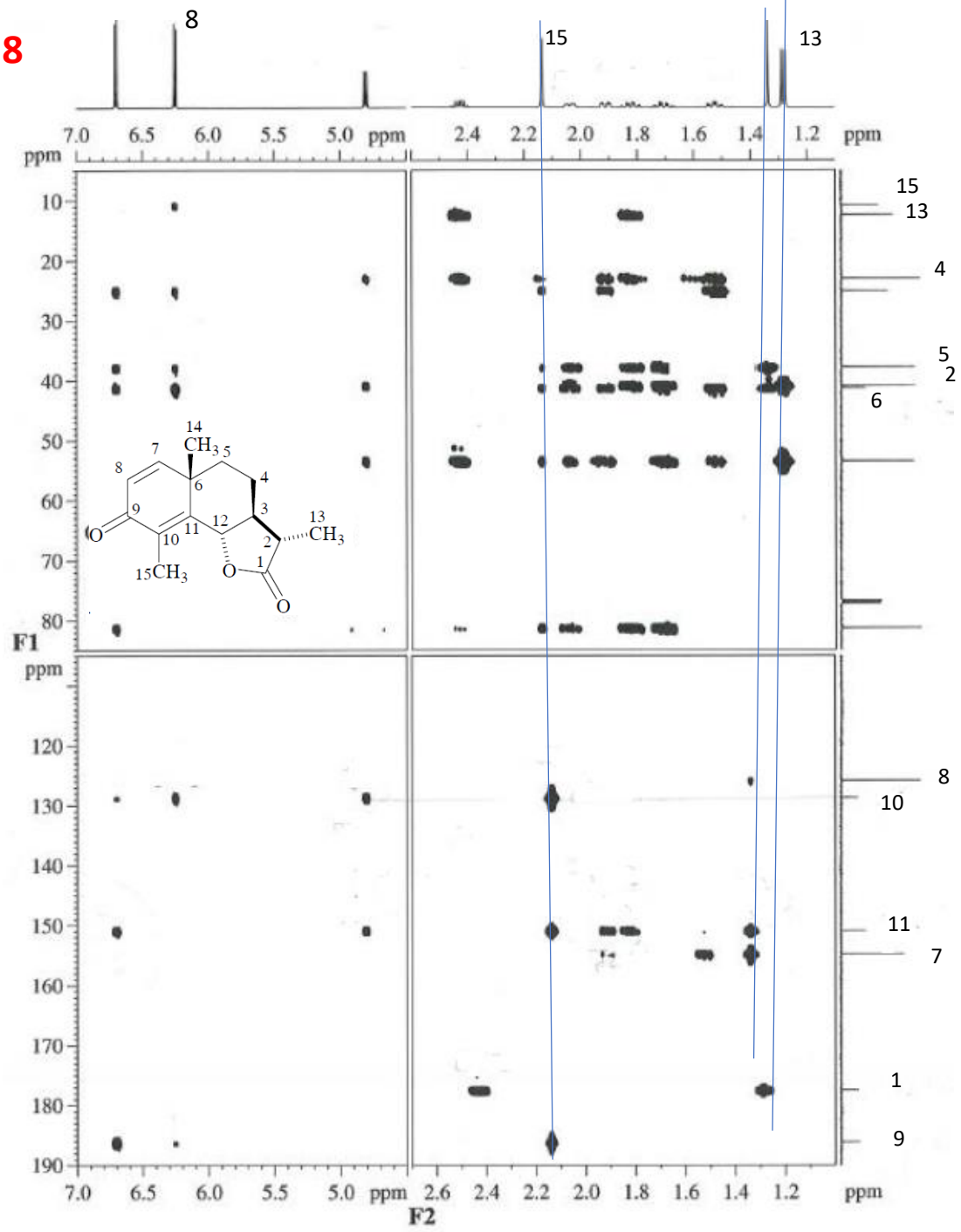

14
CH3
7
5
8
6
4
9
3
13
O
11
12
CH3
10
2
15CH3
1
O
O


HMQC 600 MHz
ppm
CH3
20
CH3
O
40
CH3
O
O
60
F1
80
100
120
140
7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 ppm
F2

ES.8 **HMQC 600 MHz**



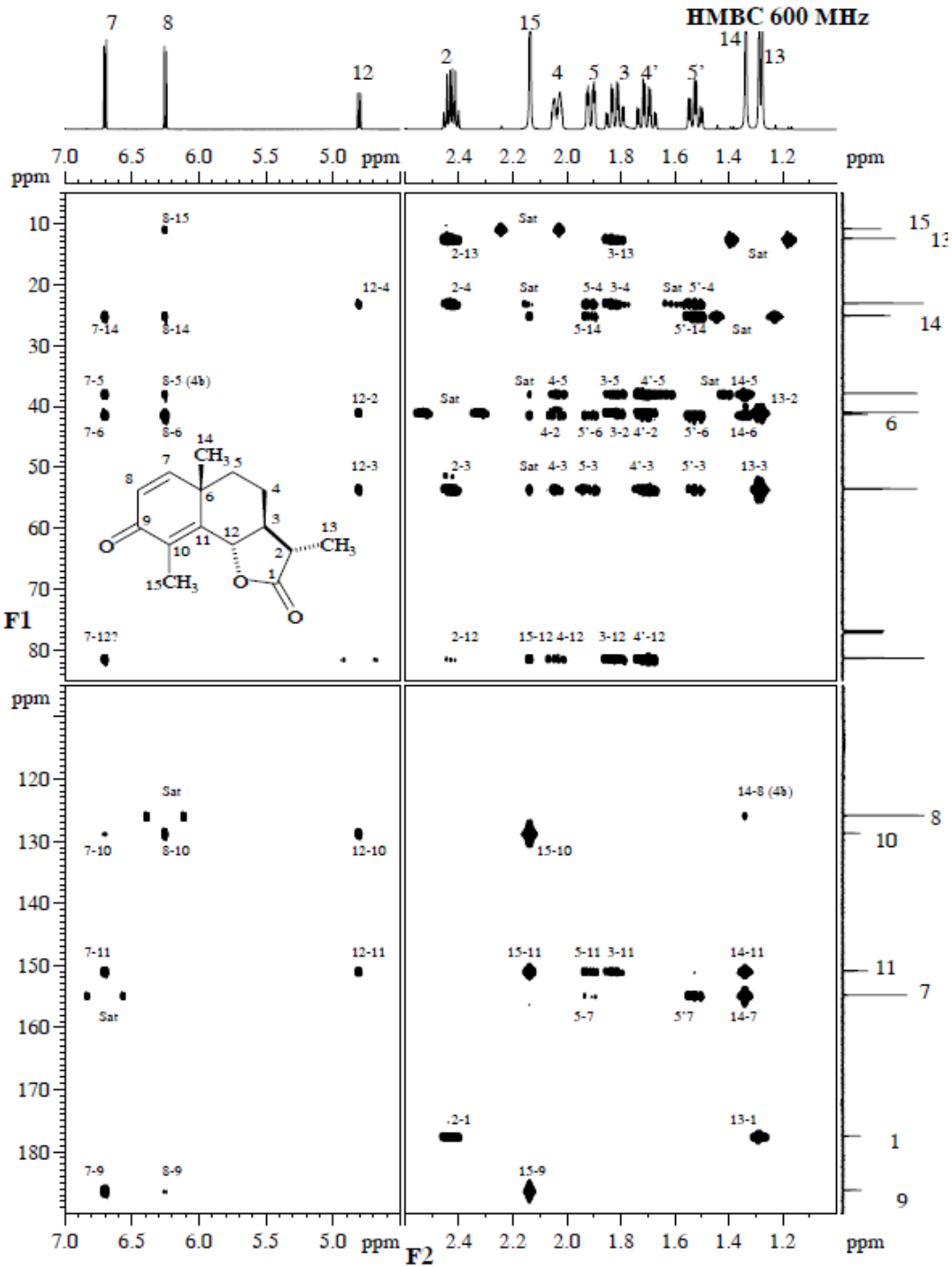
ES.8



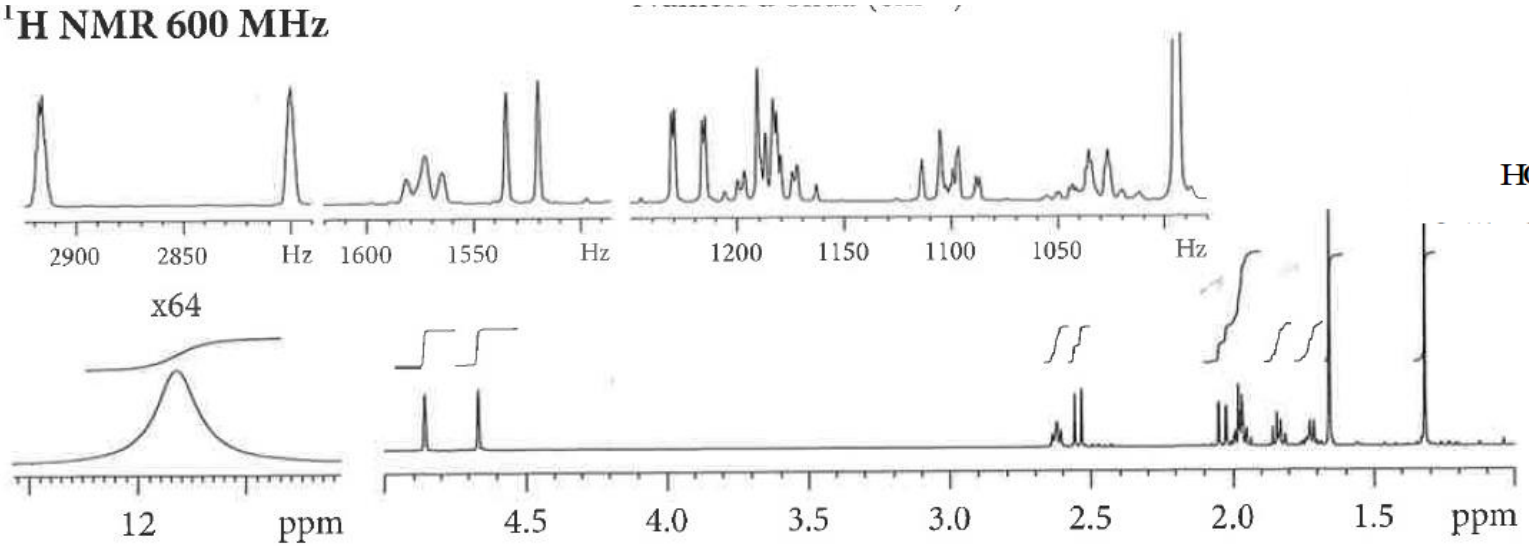
HMBC

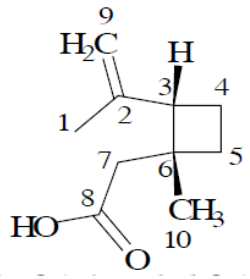
Assegnare 7,8, 1, 9, 6, 10, 11

ES.8



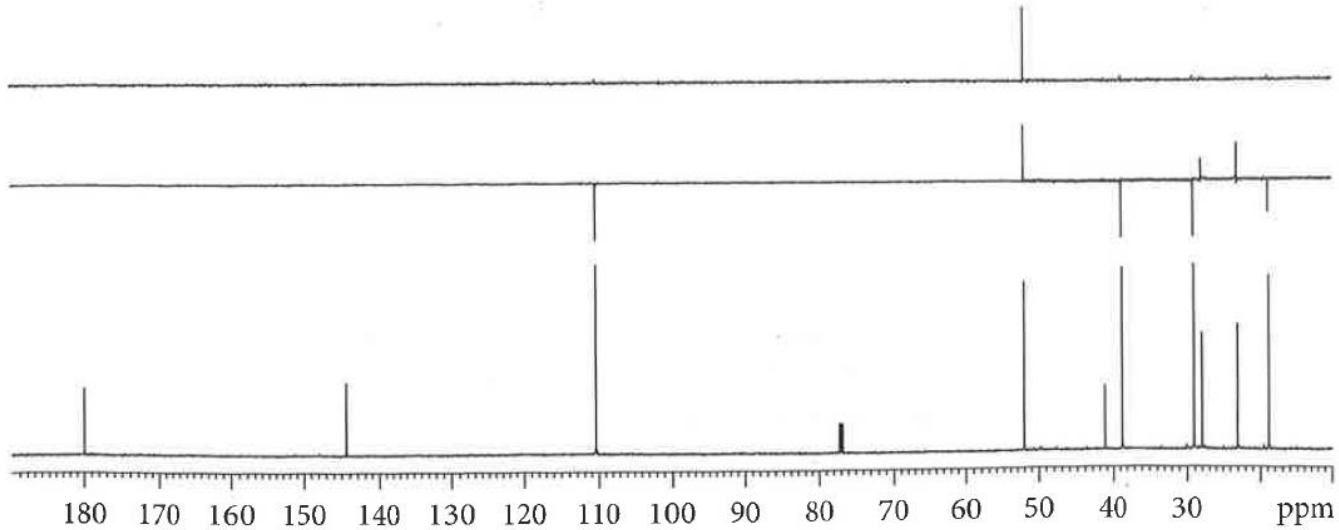
^{1}H NMR 600 MHz

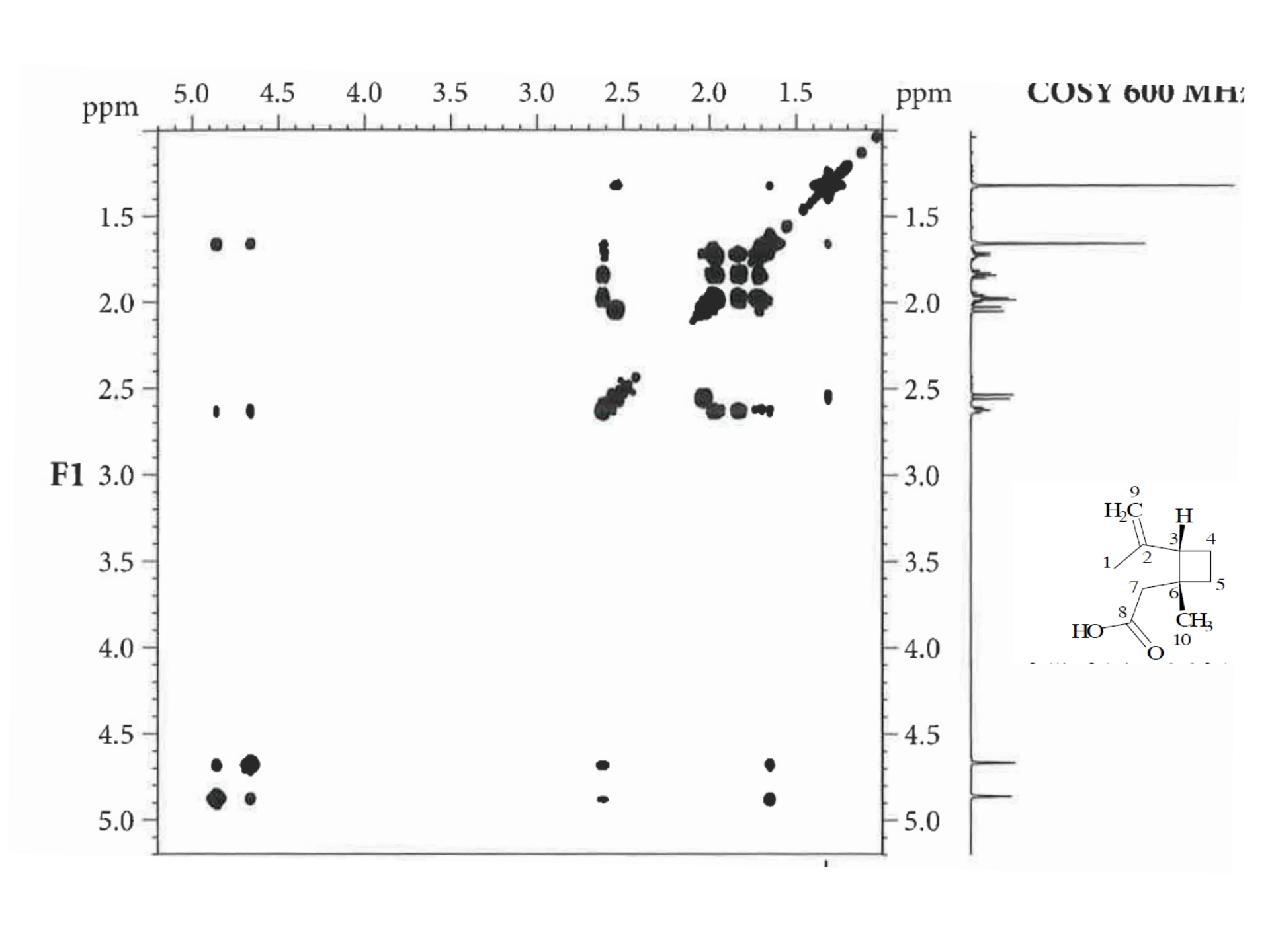


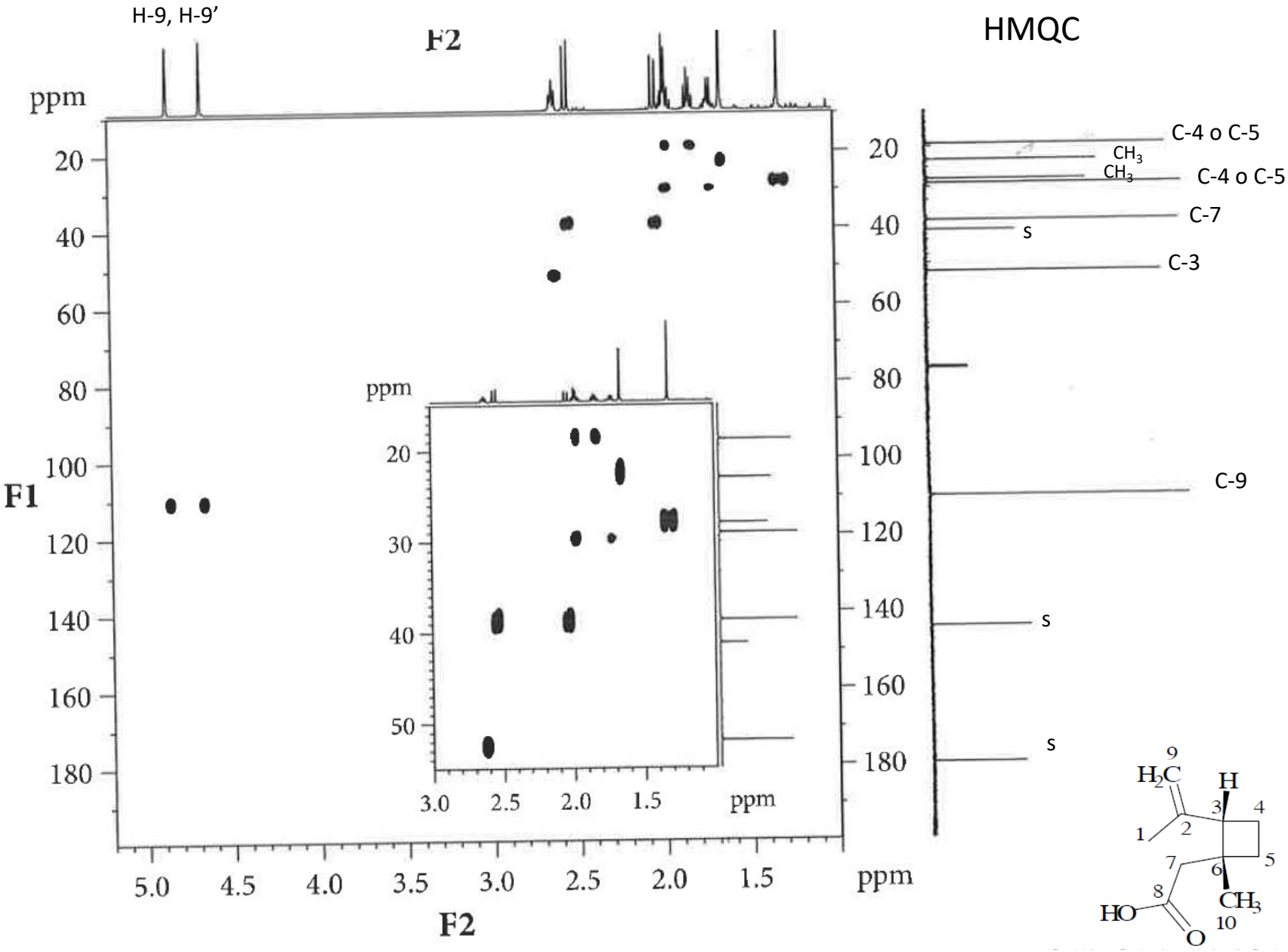


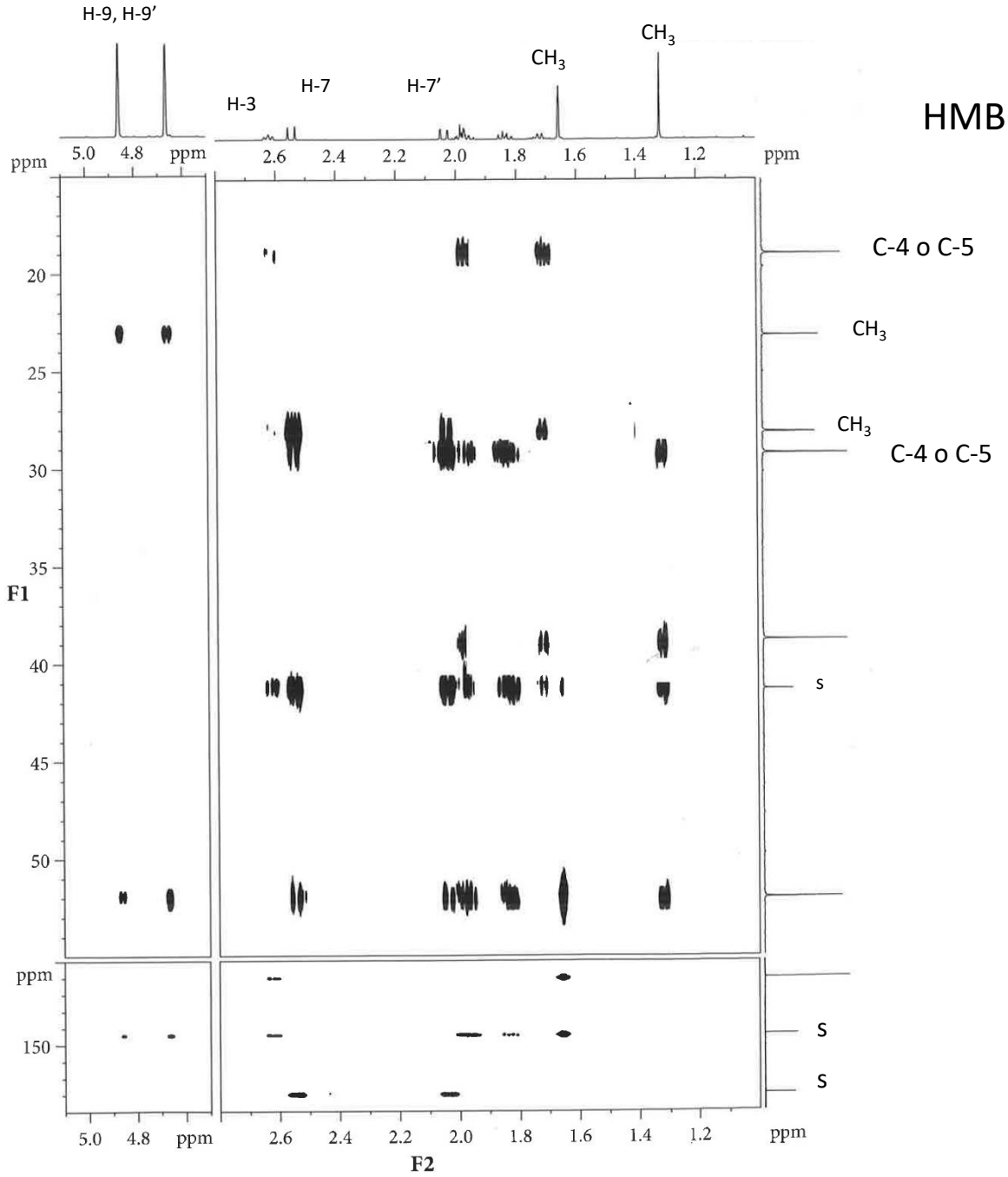
ES.10

^{13}C/DEPT NMR 150.9 MHz

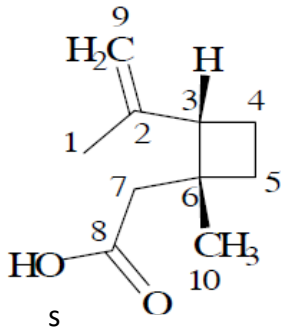


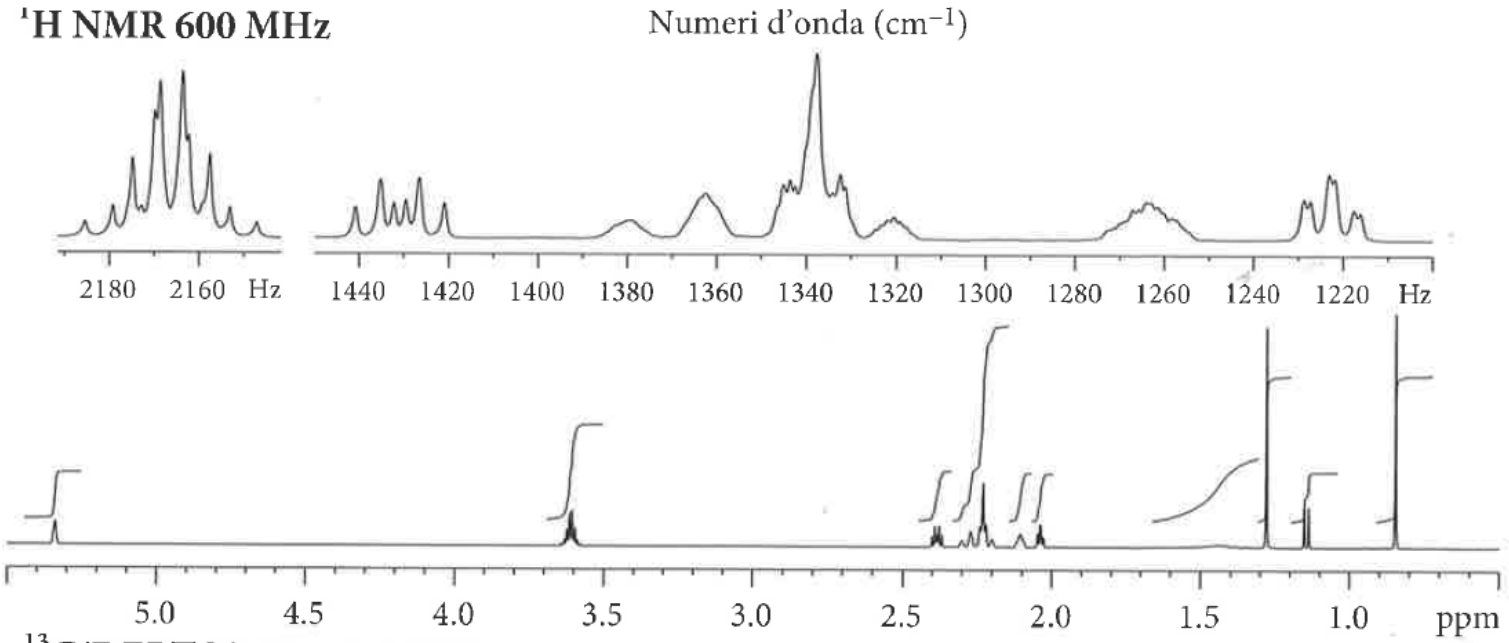

COSY 600 MH
ppm
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm
F1
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
9
H2C
H
3
4
1
2
7
6
5
8
HO
CH3
10
O

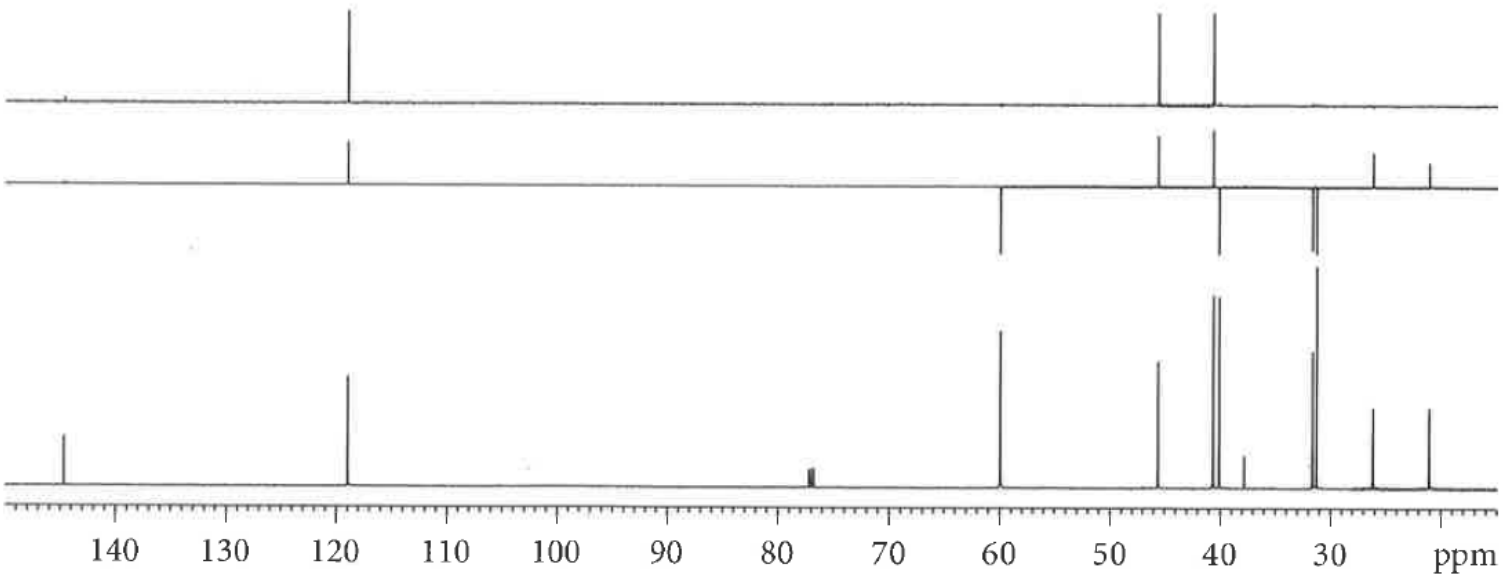

HMQC
H-9, H-9'
F2
ppm
F1
F2
ppm
C-4 o C-5
CH3
CH3
C-4 o C-5
C-7
s
C-3
C-9
s
s
H2C
H
HO
CH3
O

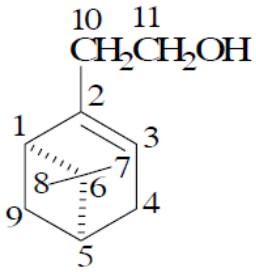

H-9, H-9'
H-3
H-7
H-7'
CH3
CH3
ppm
5.0
4.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
F1
F2
20
25
30
35
40
45
50
150
C-4 o C-5
CH3
CH3
C-4 o C-5
s
s
s

HMBC


9
H2C
H
1
2
3
4
5
6
7
8
HO
O
CH3
10

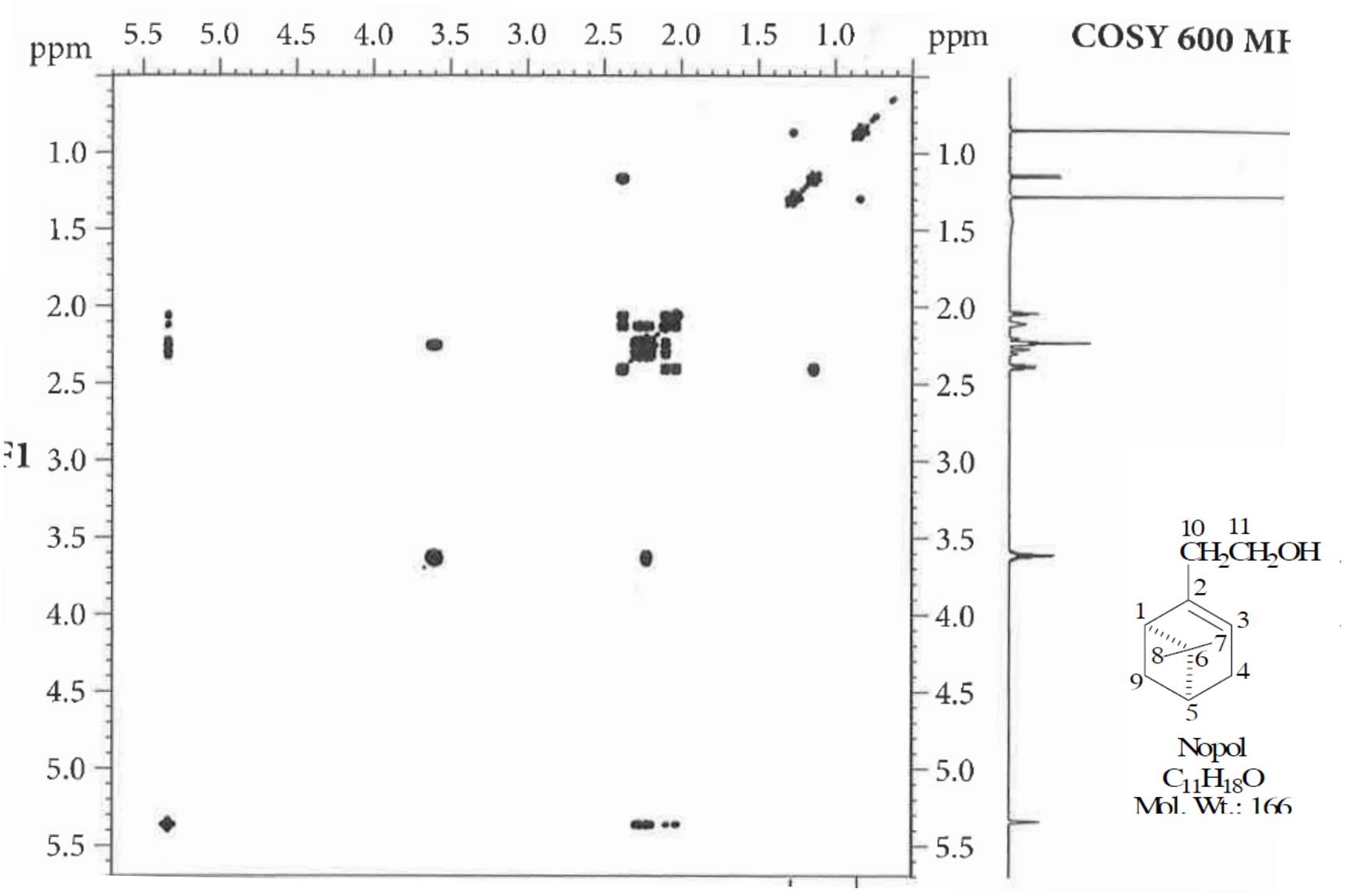


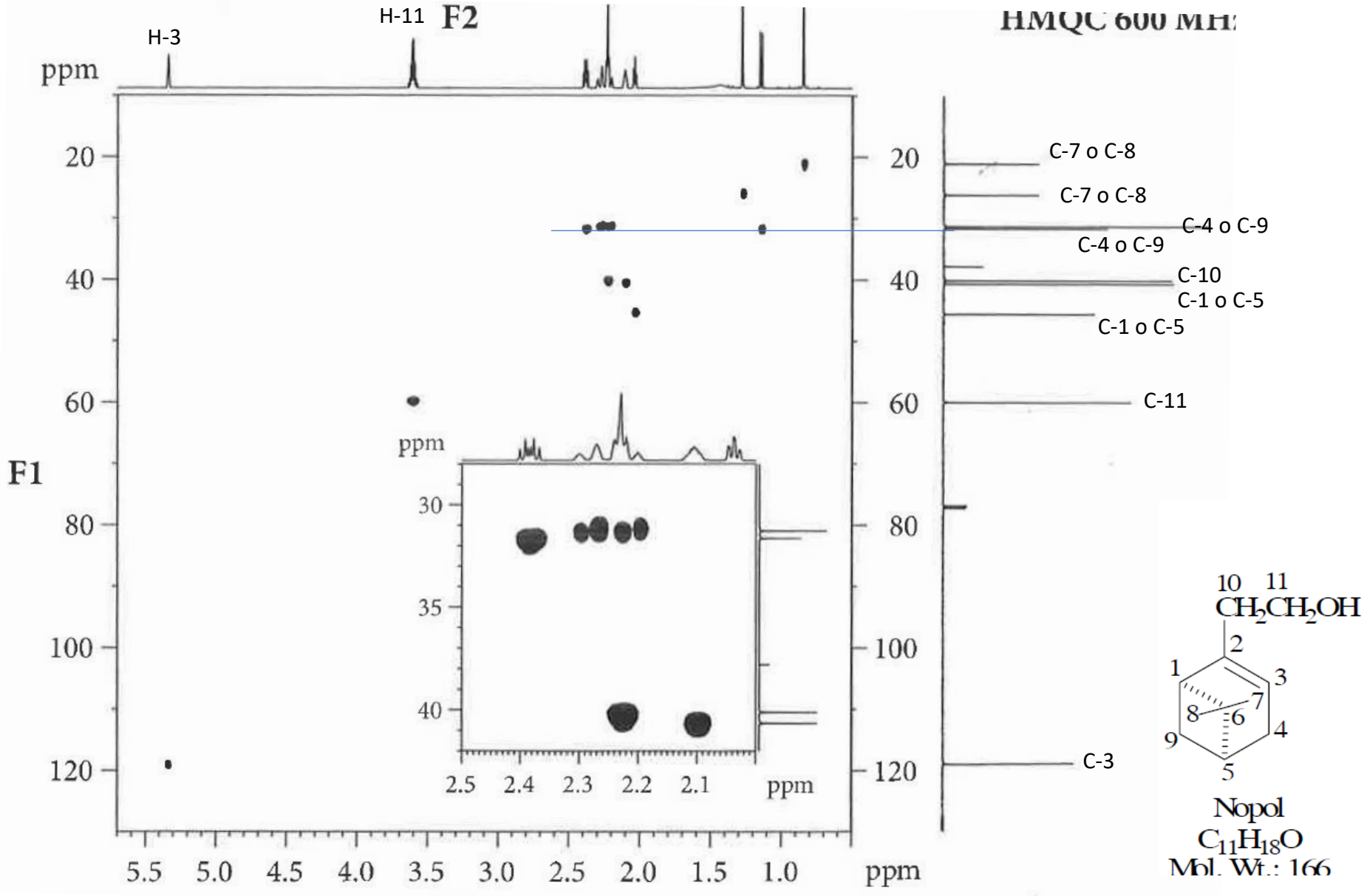


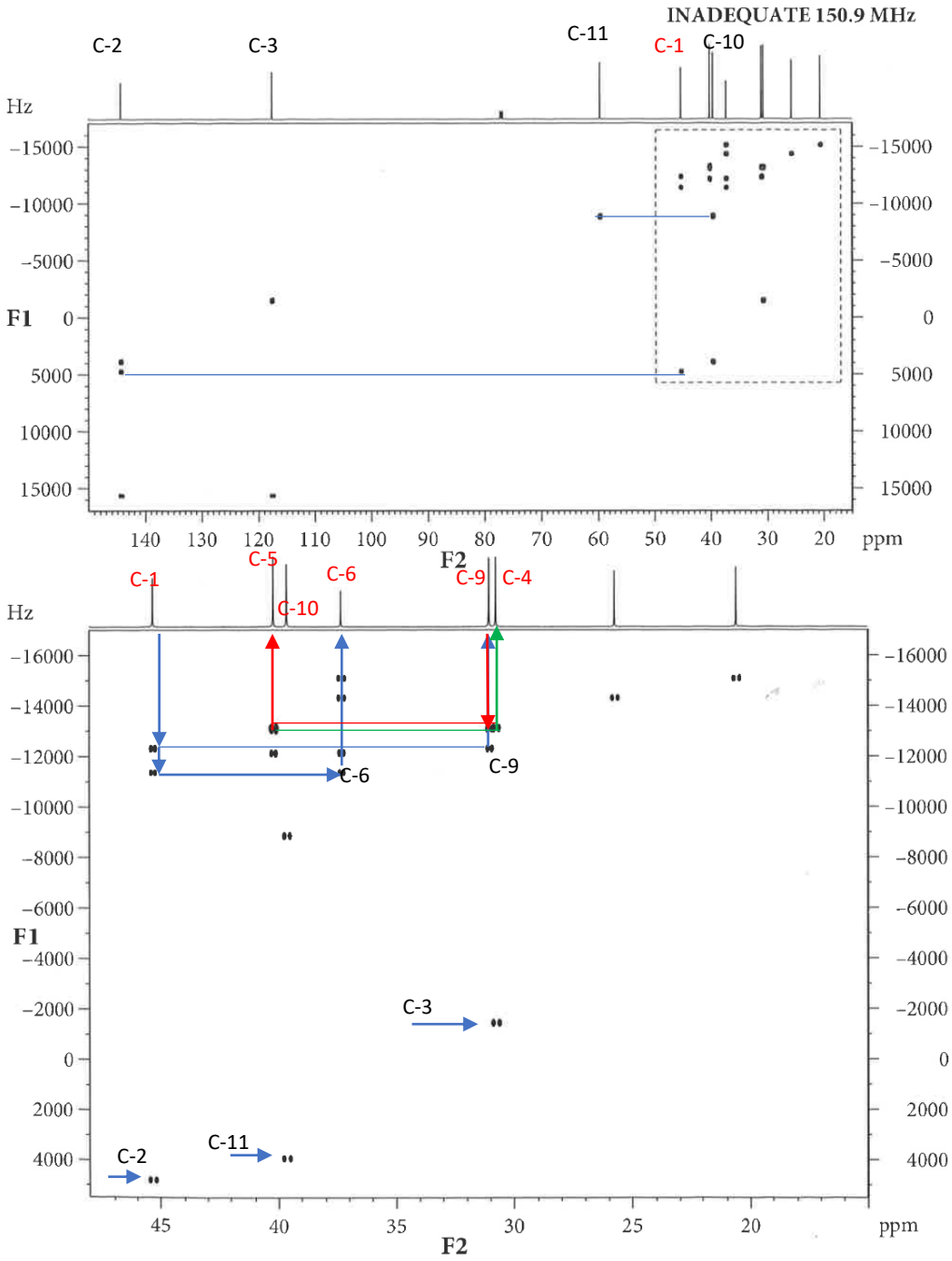


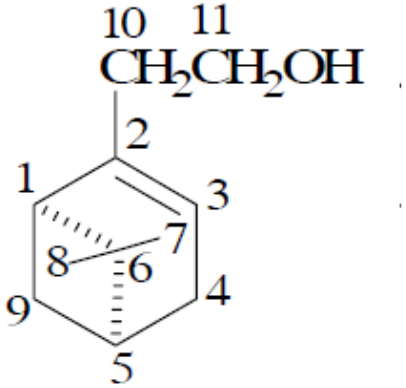
Nopol
$C_{11}H_{18}O$
Mol. Wt.: 166

ES.11


COSY 600 MH
ppm
5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
F1
1.0 1.5 2.0 2.5 3.0 3.5 4.0 4.5 5.0 5.5
10 11
CH2CH2OH
1 2 3 4 5 6 7 8 9
Nopol
C11H18O
Mol. Wt.: 166


H-3
H-11
F2
HMQC 600 MH
ppm
F1
20
40
60
80
100
120
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
C-7 o C-8
C-7 o C-8
C-4 o C-9
C-4 o C-9
C-10
C-1 o C-5
C-1 o C-5
C-11
C-3
30
35
40
2.5
2.4
2.3
2.2
2.1
10 11
CH2CH2OH
2
1
3
7
8
6
4
9
5
Nopol
C11H18O
Mol. Wt.: 166



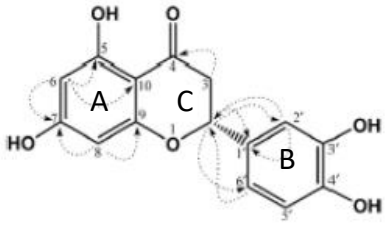


Nopol

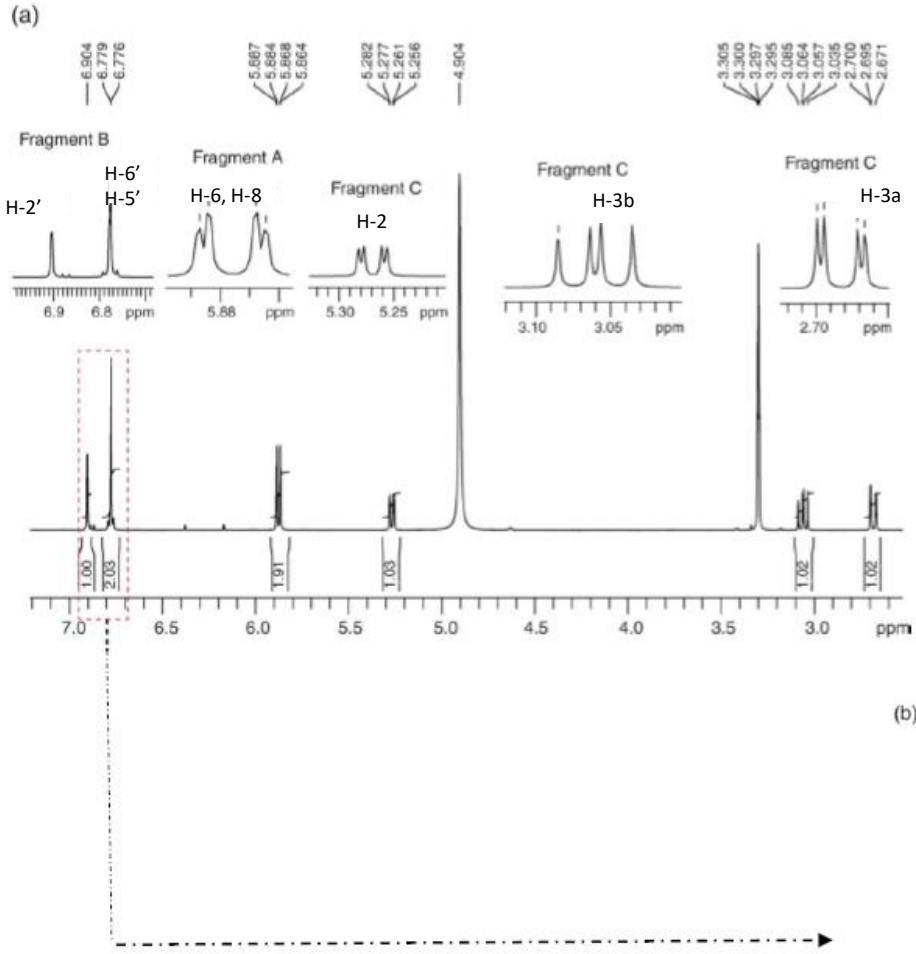
$C_{11}H_{18}O$

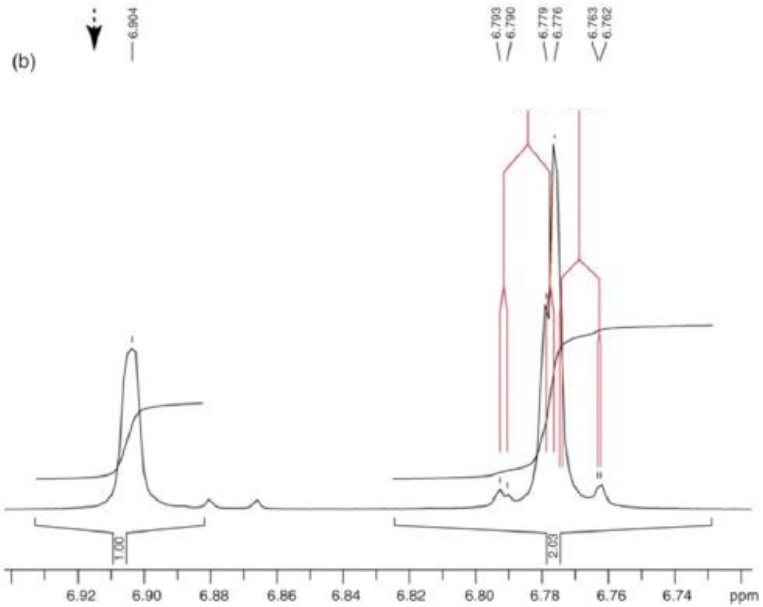
Mol. Wt.: 166

ES.9



Solvente: MeOD





Solvente: piridina-D5

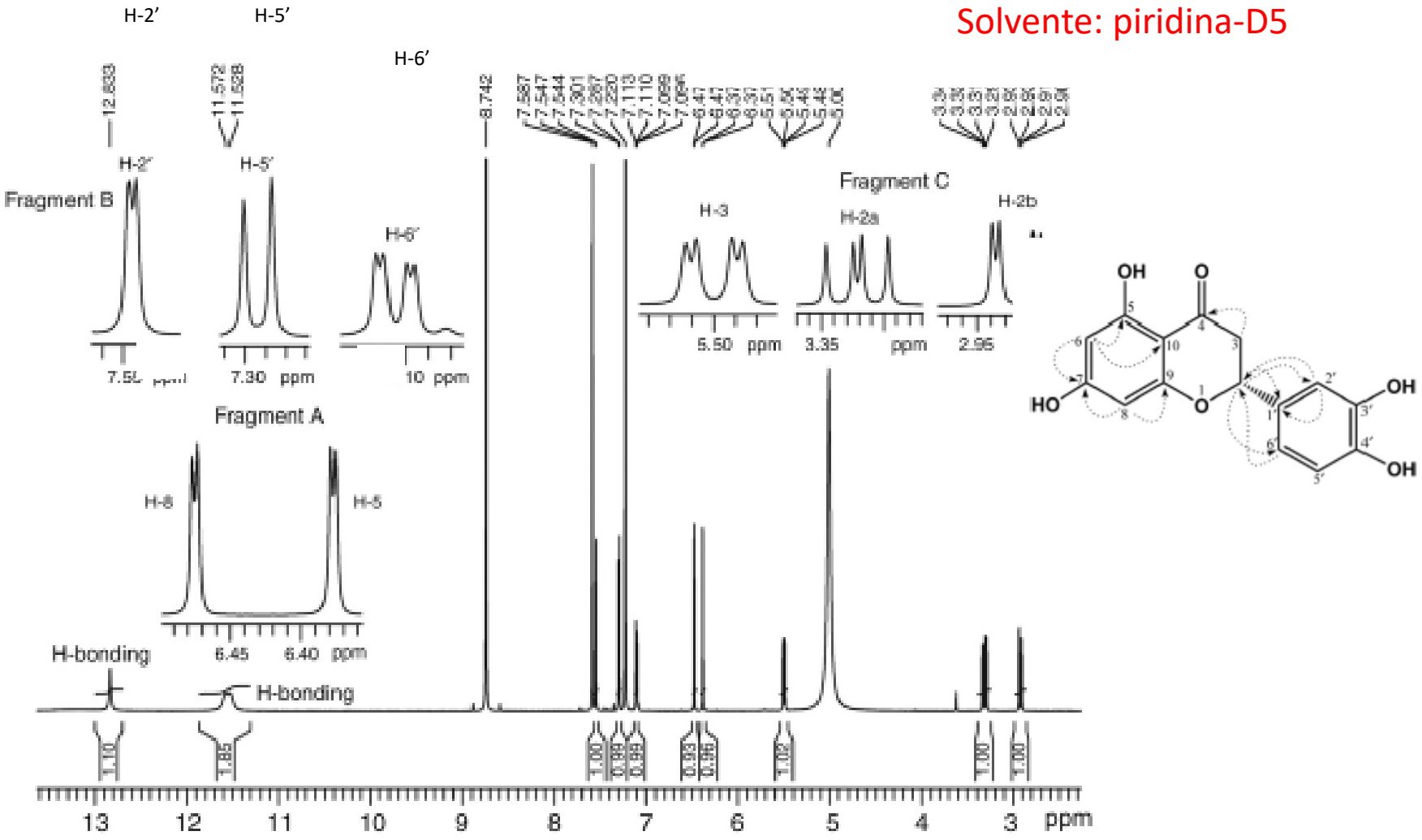


FIGURE 10.51 ^{1}H-NMR spectrum (C_5D_5N) of **4** showing protons of fragments A, B, and C.

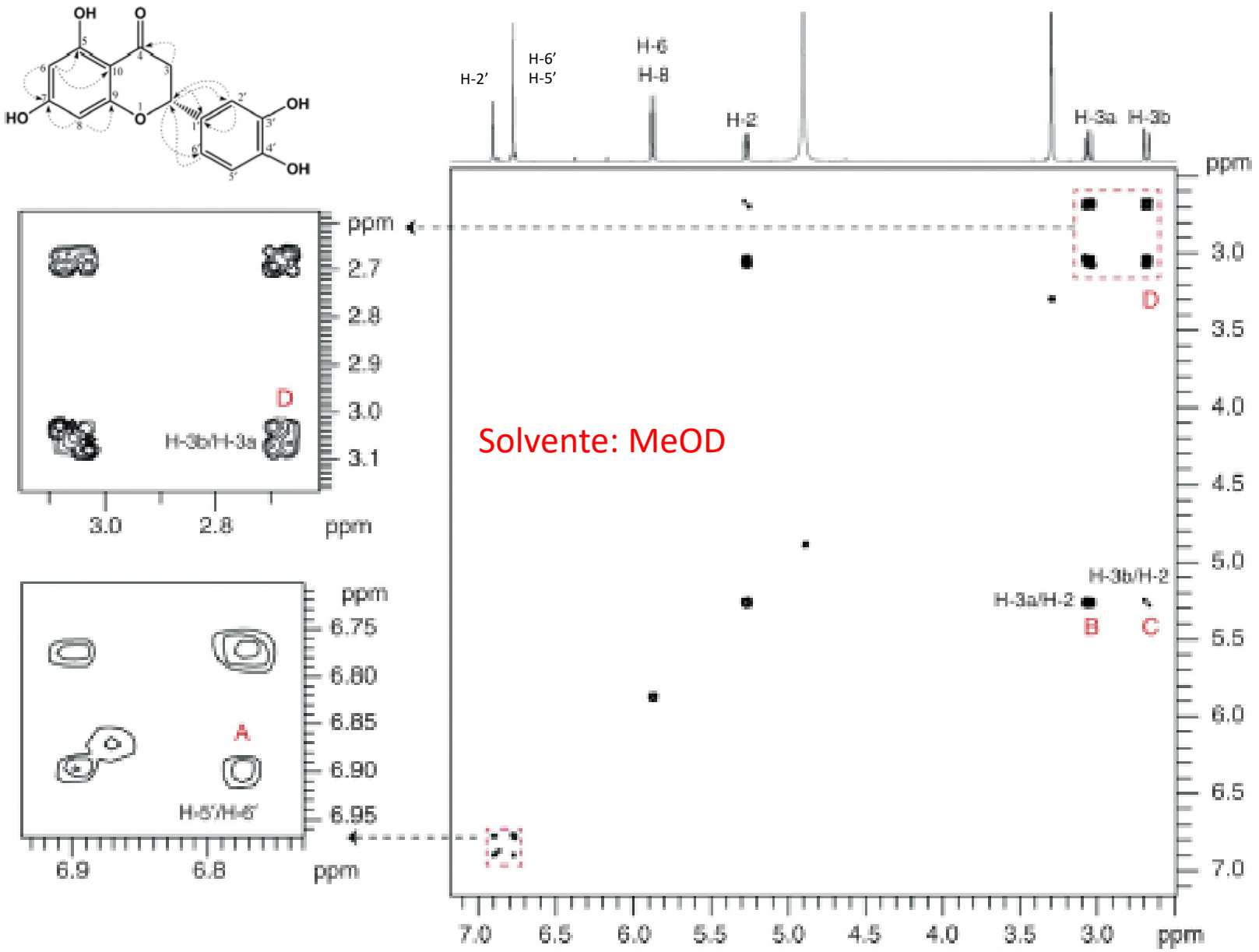
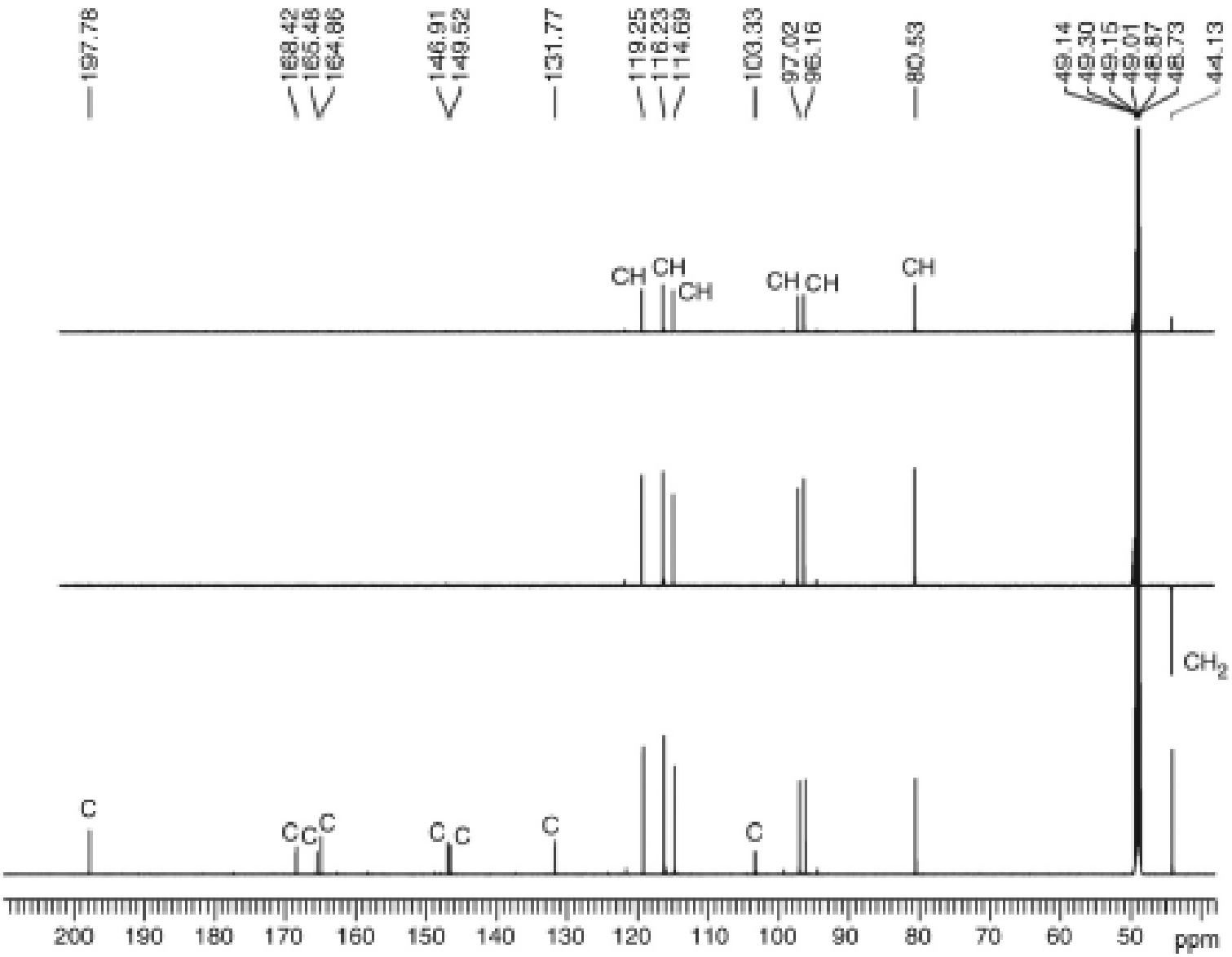
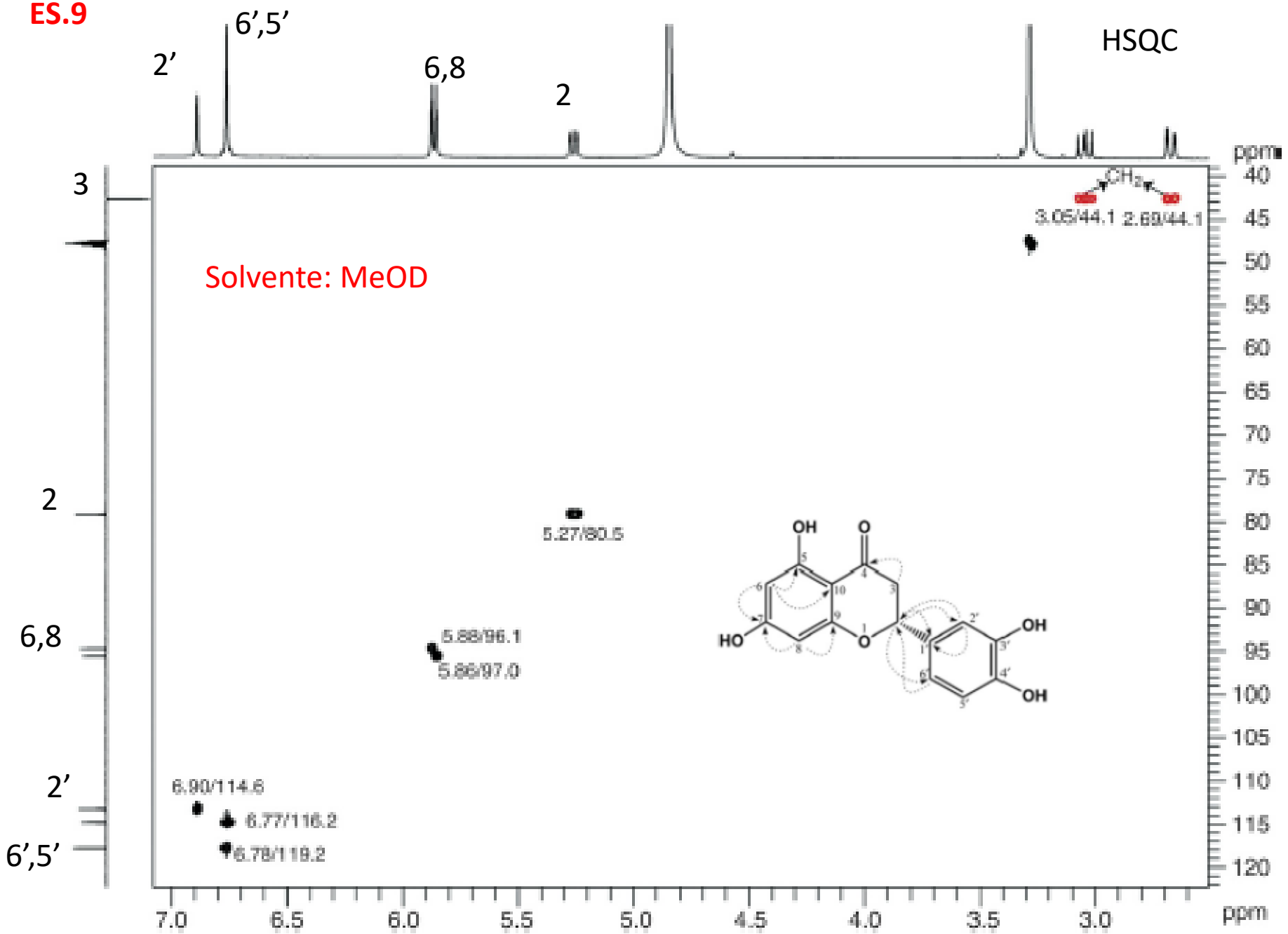


FIGURE 10.52 COSY-45° spectrum of **4** showing correlations between various vicinal and geminal protons.

ES.9


197.76
168.42
165.48
164.86
146.91
149.52
131.77
119.25
116.23
114.69
103.33
97.02
96.16
80.53
49.14
49.30
49.15
49.01
48.87
48.73
44.13
CH
CH
CH
CH
CH
CH
CH2
C
C
C
C
C
C
C
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
ppm

ES.9


2’
6’,5’
6,8
2
HSQC
ppm
3
2
6,8
2’
6’,5’
Solvente: MeOD
3.05/44.1
2.69/44.1
5.27/80.5
5.88/96.1
5.86/97.0
6.90/114.6
6.77/116.2
6.78/119.2
OH
O
HO
OH
OH
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
ppm

ES.9

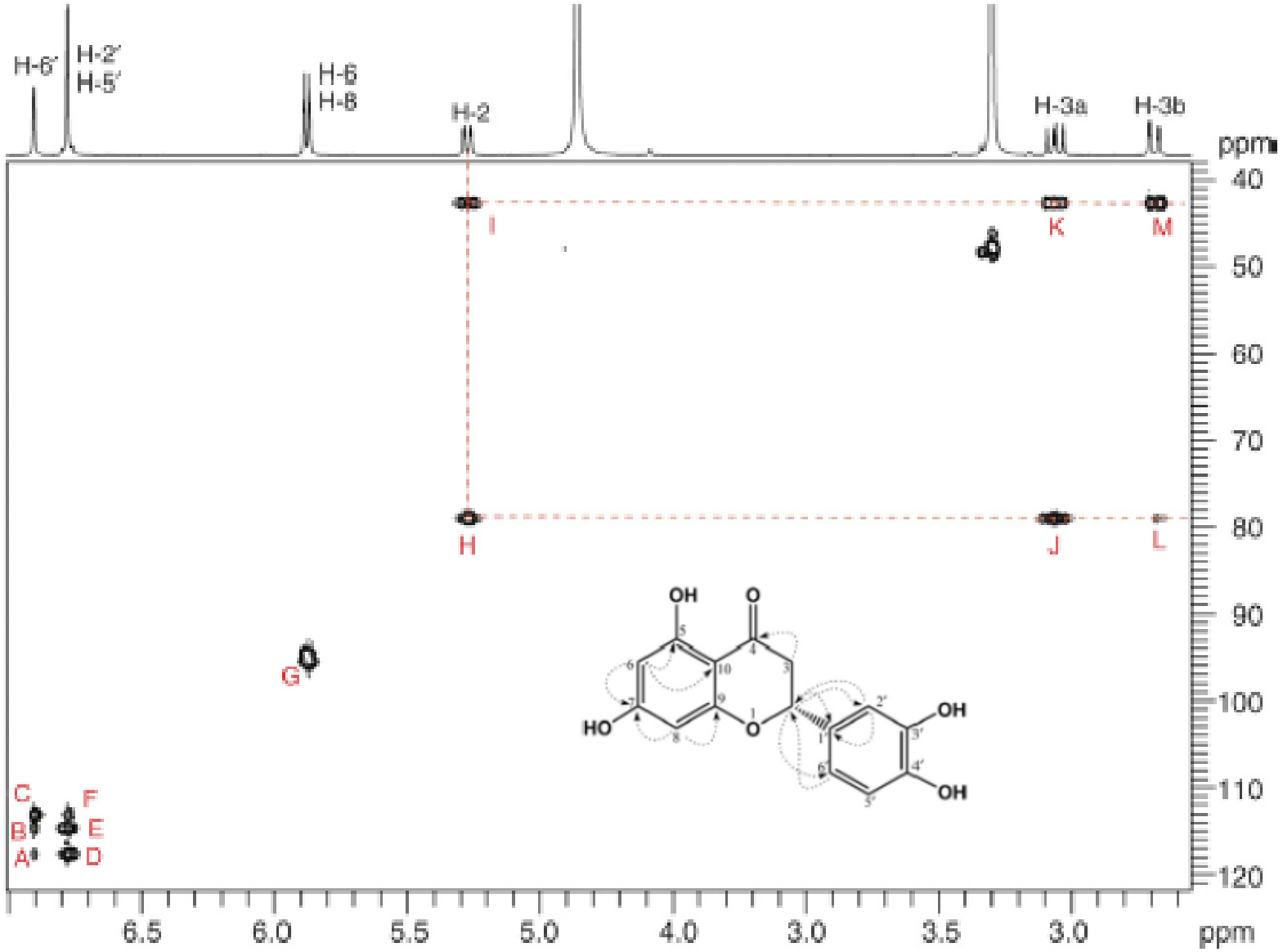
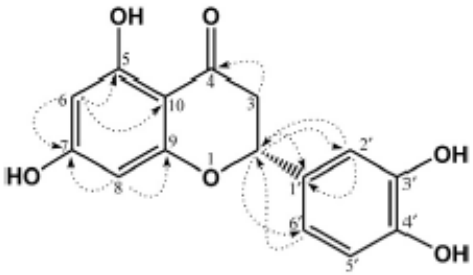
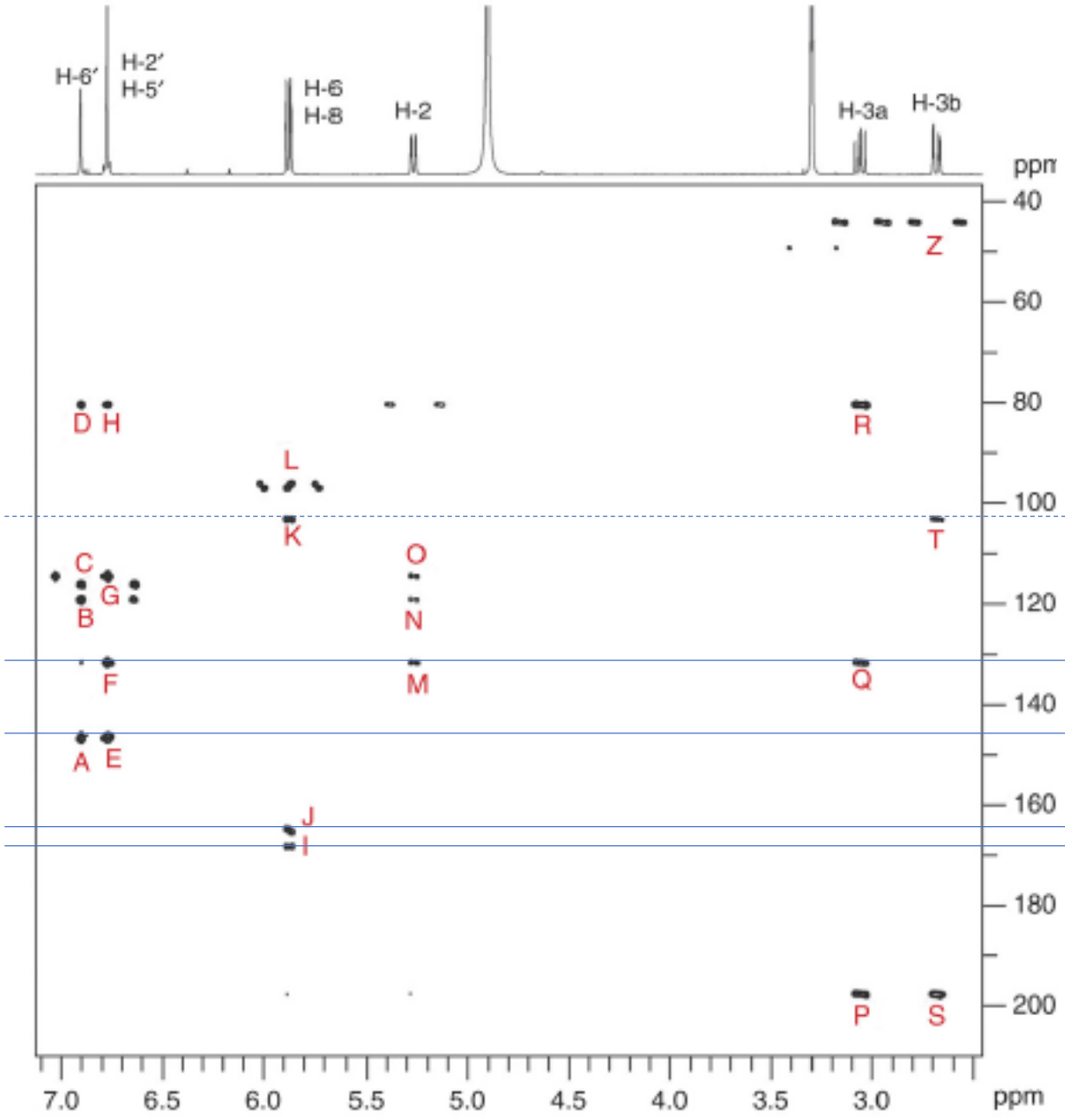


FIGURE 10.57 HSQC–TOCSY spectrum (concatenated NMR experiment) of compound **4** indicating one spin system F_1 plane and carbon connectivity in F_2 plane.

ES.9





103.3 10

131.7 1’

146.5
146.9 3’,4’

164.9, 165.5
166.5 5,7,9

CO

FIGURE 10.59 HMBC spectrum showing long-range $^{1}H/^{13}C$ correlations in compound **4**.