Spektroskopska strukturna analiza
Dodatni zadaci

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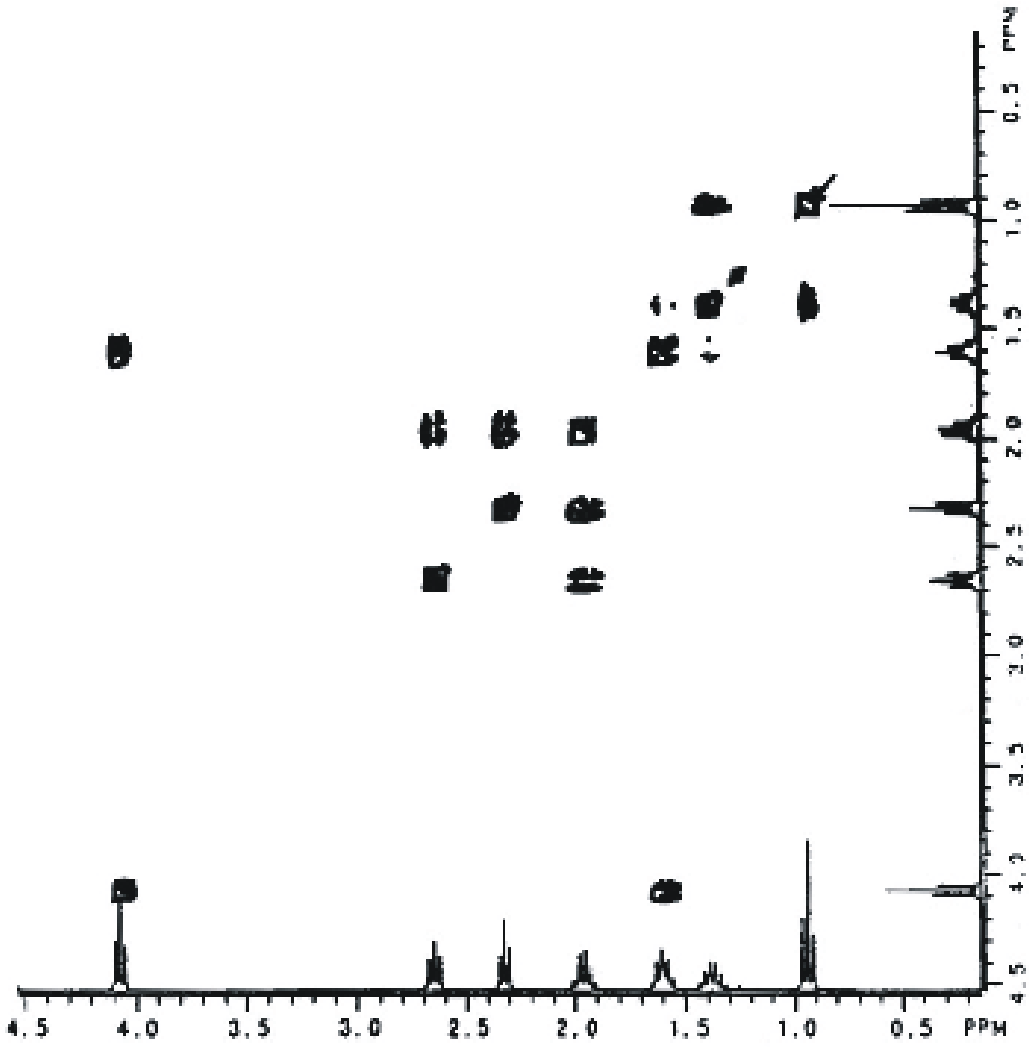
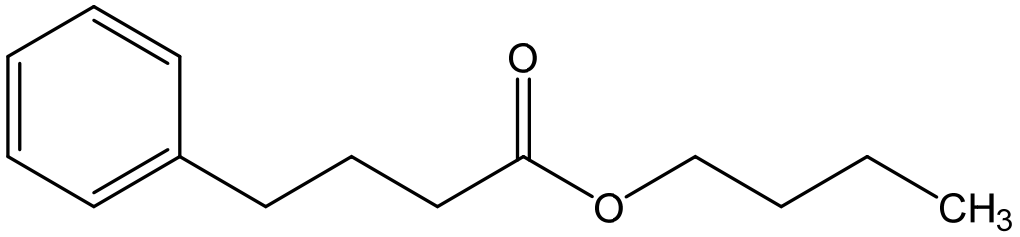
1. Odredite strukturu spoja molekulske formule $C_{14}H_{20}O_2$ na temelju njegovog COSY spektra. ^1H NMR spektar sadrži još široki singlet s integralom 5.



- indeks manjka vodika (*index of hydrogen deficiency,* IHD)

$$IHD = \frac{1}{2}[2C + 2 + N - (H + X)]$$

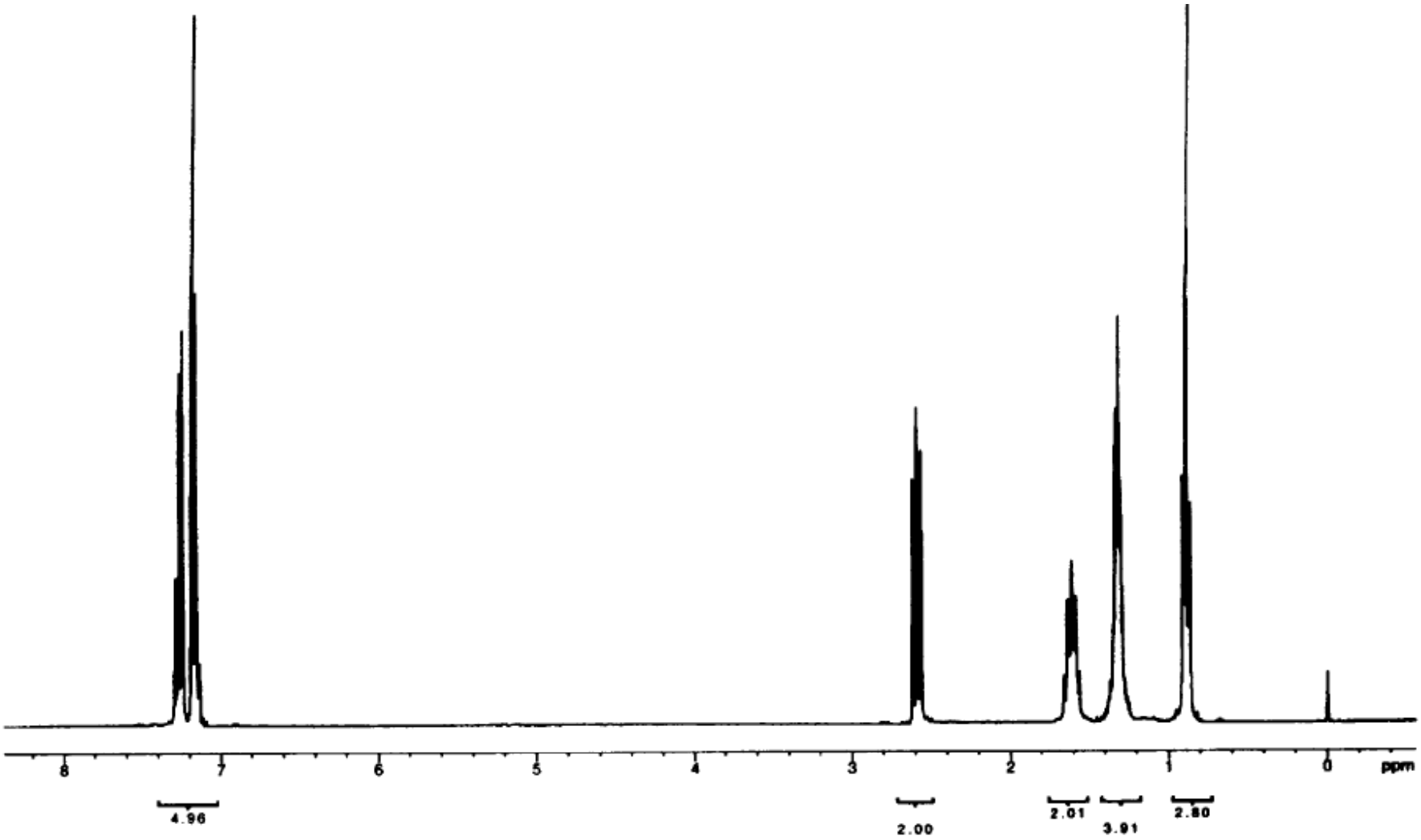
- za ovaj spoj: IHD = [2·14+2–20]/2 = 5



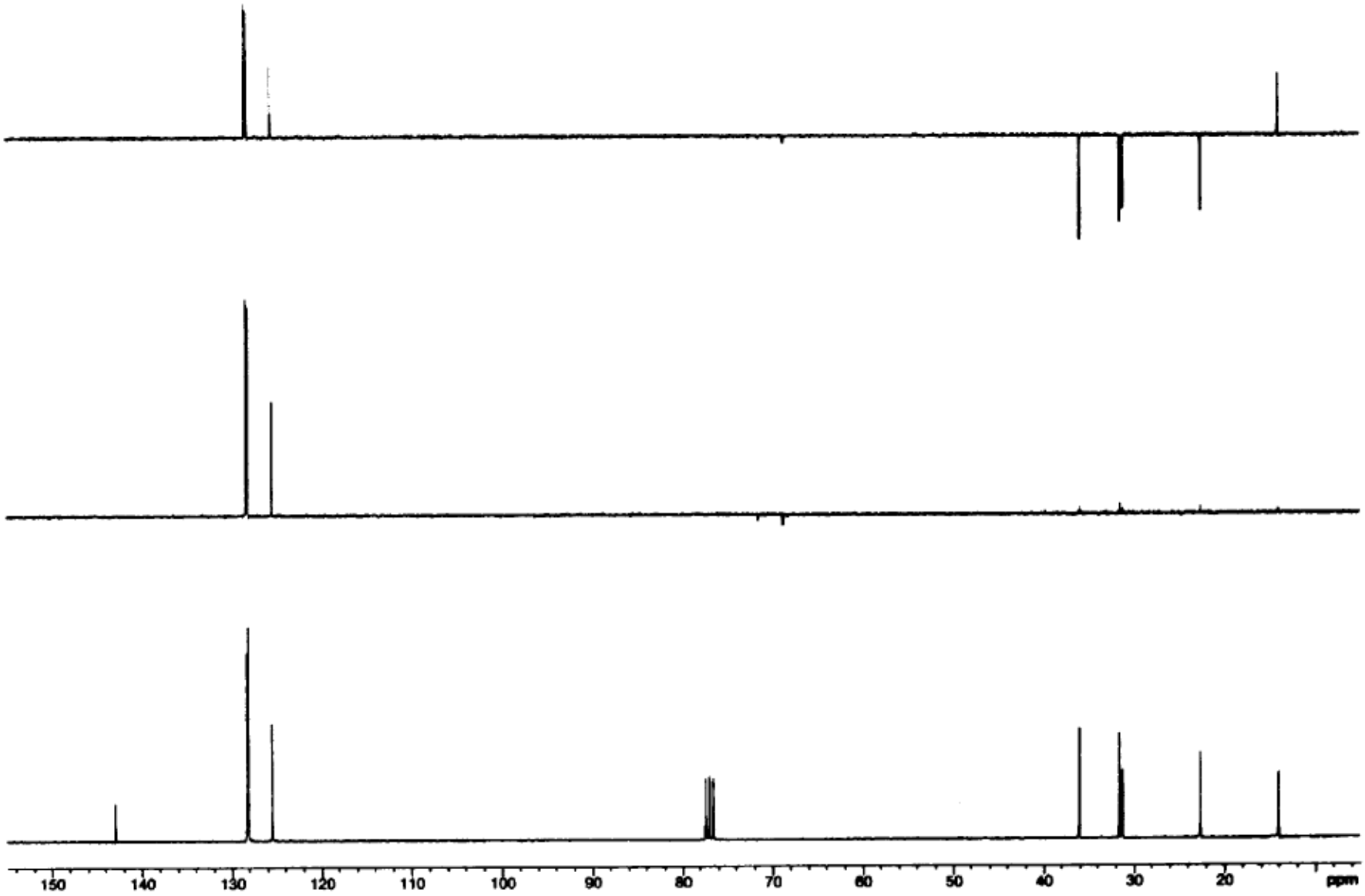
$\delta(^1H)$ / ppm	multiplet	H-atom
0,95	t	$-CH_3$
1,40	m(heptet)	$-CH_2-$
1,55	m(kvintet)	$-CH_2-$
1,95	m(kvintet)	$-CH_2-$
2,35	t	$-CH_2-$
2,65	t	$-CH_2-$
4,10	t	$-CH_2-$
	s	=CH–

2. Odredite strukturu spoja molekulske formule $C_{11}H_{16}$ na temelju njegovih ^{1}H NMR, ^{13}C NMR, COSY i HETCOR spektara.

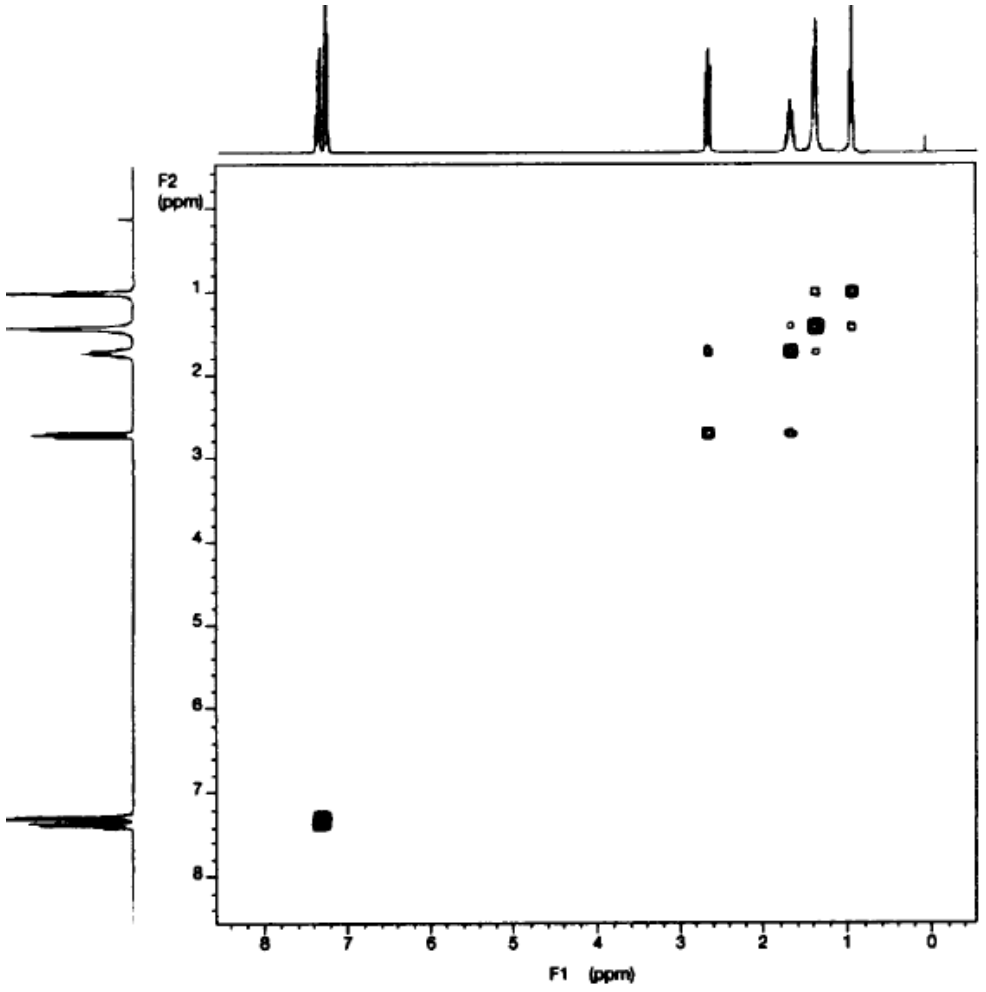
^{1}H NMR spektar

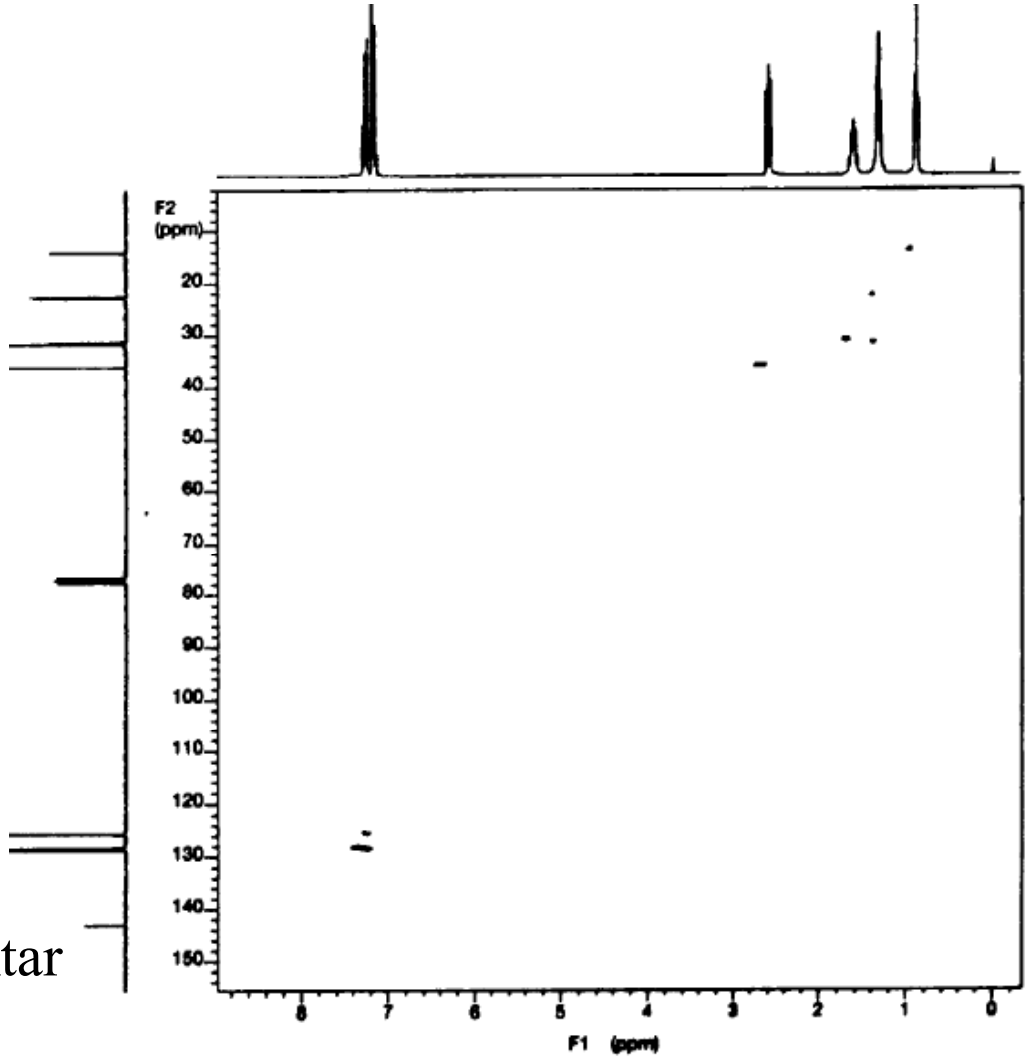


^{13}C NMR/DEPT spektar



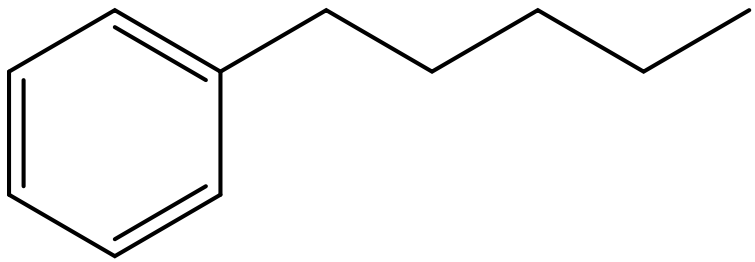
COSY spektar





HETCOR spektar

IHD = [2·11+2–16]/2 = 4



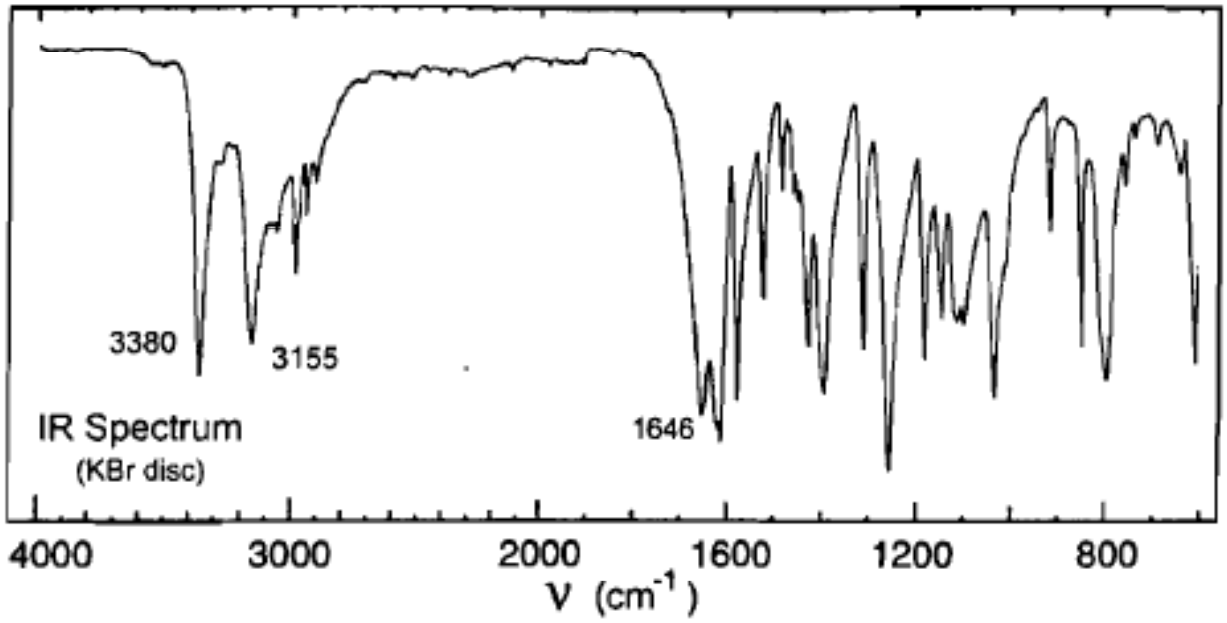
- informacije iz ^{1}H NMR:

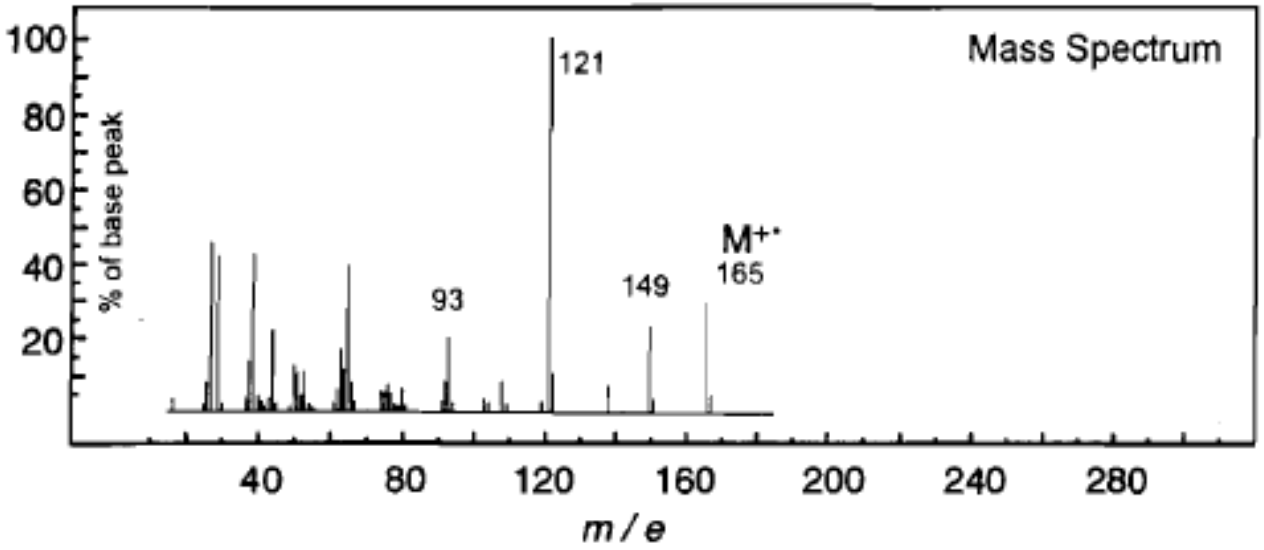
$\delta(^{1}H)$ / ppm	multiplet	H-atom
0,9	t	$–CH_3$
1,3	m	$–CH_2–$, $–CH_2–$
1,6	m(kvintet)	$–CH_2–$
2,6	t	$–CH_2–$
7,2	m	=CH–
7,3	m	=CH–

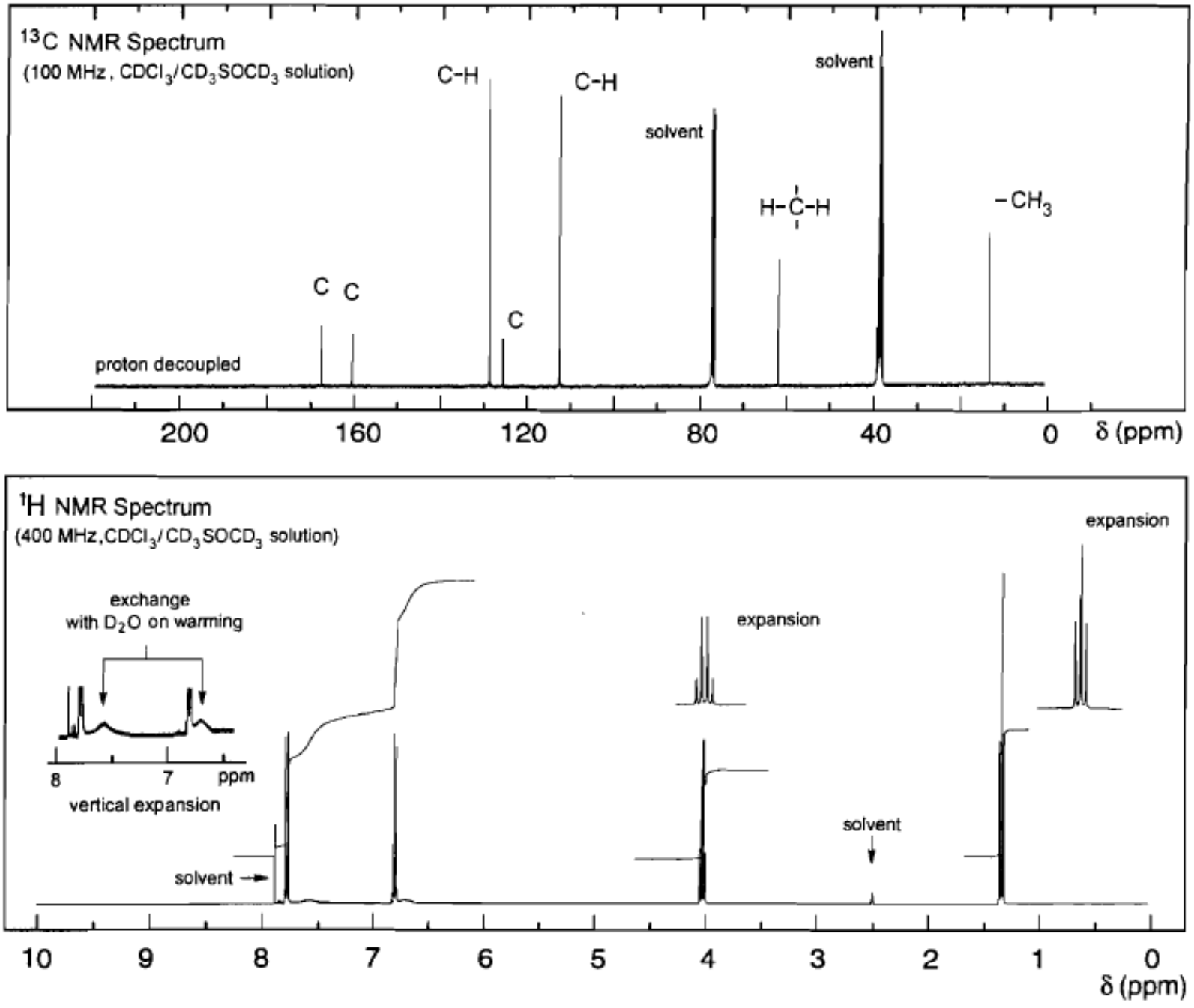
- informacije iz ^{13}C NMR:

$\delta(^{13}C)$ / ppm	C-atom
14	$–CH_3$
23	$–CH_2–$
31	$–CH_2–$
32	$–CH_2–$
36	$–CH_2–$
125	=CH–
128	=CH–
143	C

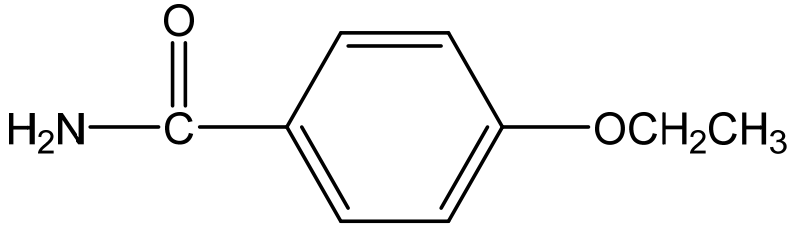
3. Odredite strukturu spoja na temelju njegovog IR, MS, ^{1}H NMR i ^{13}C NMR spektra.






^{13}C NMR Spectrum
(100 MHz, $CDCl_3/CD_3SOCD_3$ solution)
C-H
C-H
solvent
solvent
H-C-H
$-CH_3$
C C
C
proton decoupled
200
160
120
80
40
0
δ (ppm)
^{1}H NMR Spectrum
(400 MHz, $CDCl_3/CD_3SOCD_3$ solution)
expansion
exchange
with D_2O on warming
8
7
ppm
vertical expansion
expansion
solvent
solvent
10
9
8
7
6
5
4
3
2
1
0
δ (ppm)

- informacije iz MS:
  bazni pik: 121
  $M^+ = 165$



- informacije iz IR:
  3380 i 3155 cm^{-1} N–H istezanje
  1646 cm^{-1} C=O istezanje
  1250 cm^{-1} C–O istezanje

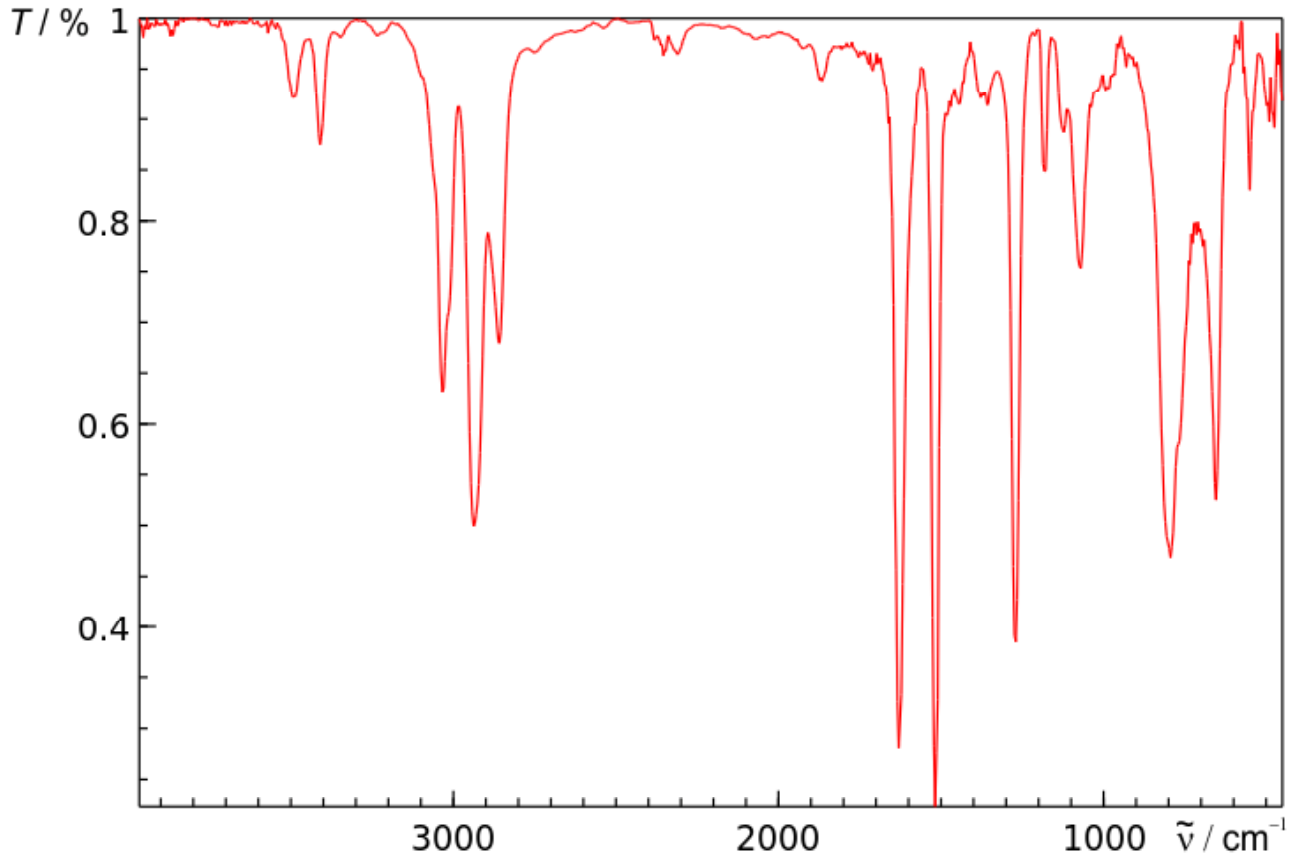
- informacije iz ^{1}H NMR:

$\delta(^1H)$ / ppm	multiplet	H-atom
1,4	t	$-CH_3$
4,0	q	$-CH_2-$
6,7	s	$-NH_2$
6,8	d	=CH–
7,6	s	$-NH_2$
7,8	d	=CH–

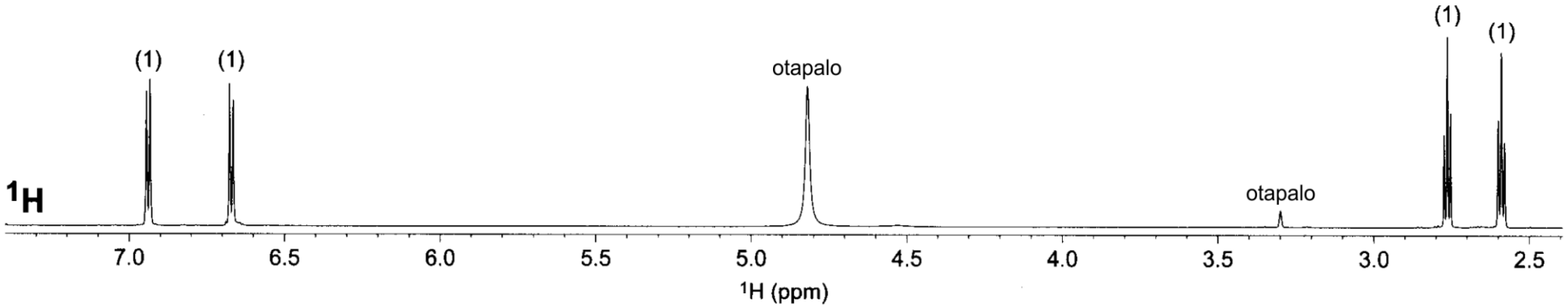
- informacije iz ^{13}C NMR:

$\delta(^{13}C)$ / ppm	C-atom
12	$-CH_3$
62	$-CH_2-$
110	=CH–
126	C
129	=CH–
160	C
168	C=O

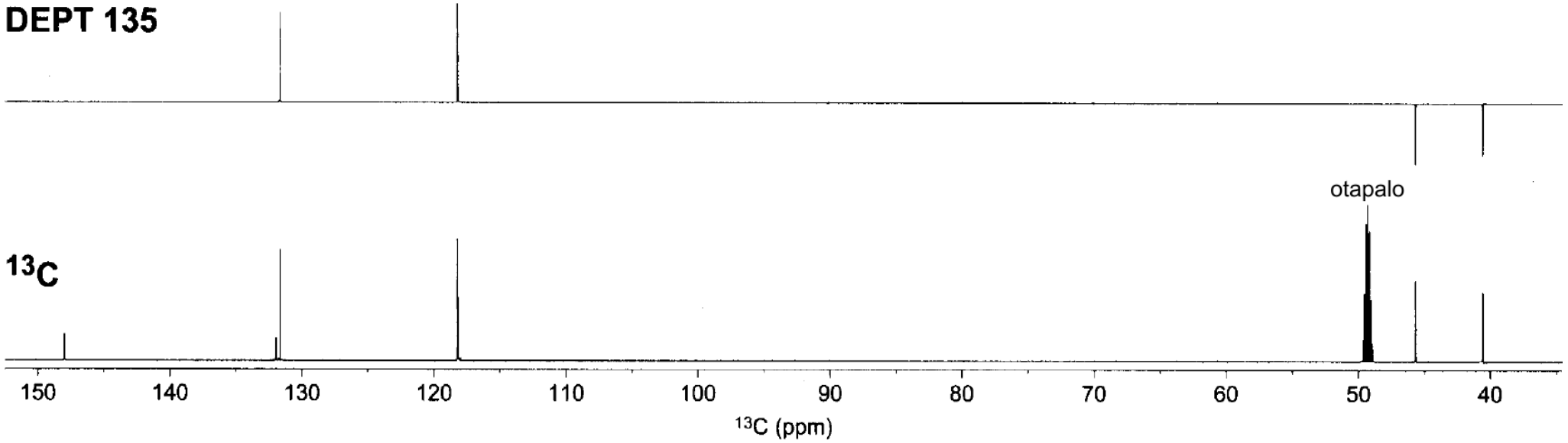
4. Odredite strukturu spoja molarne mase 136 g mol^{-1} na temelju njegovih IR, ^{1}H NMR ^{13}C NMR, COSY, HSQC i HMBC spektara.



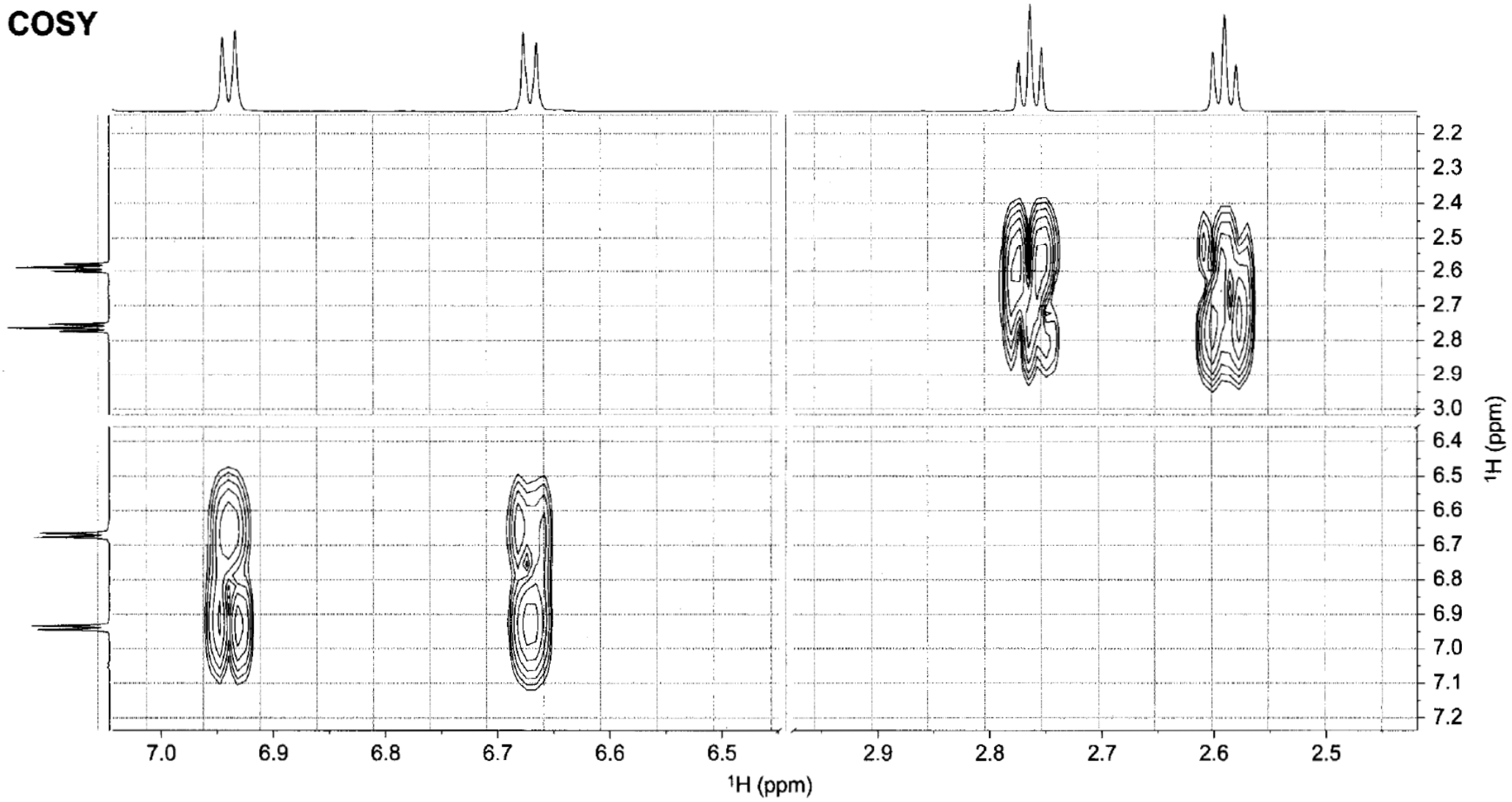
IR spektar spoja snimljen tehnikom KBr pastile.



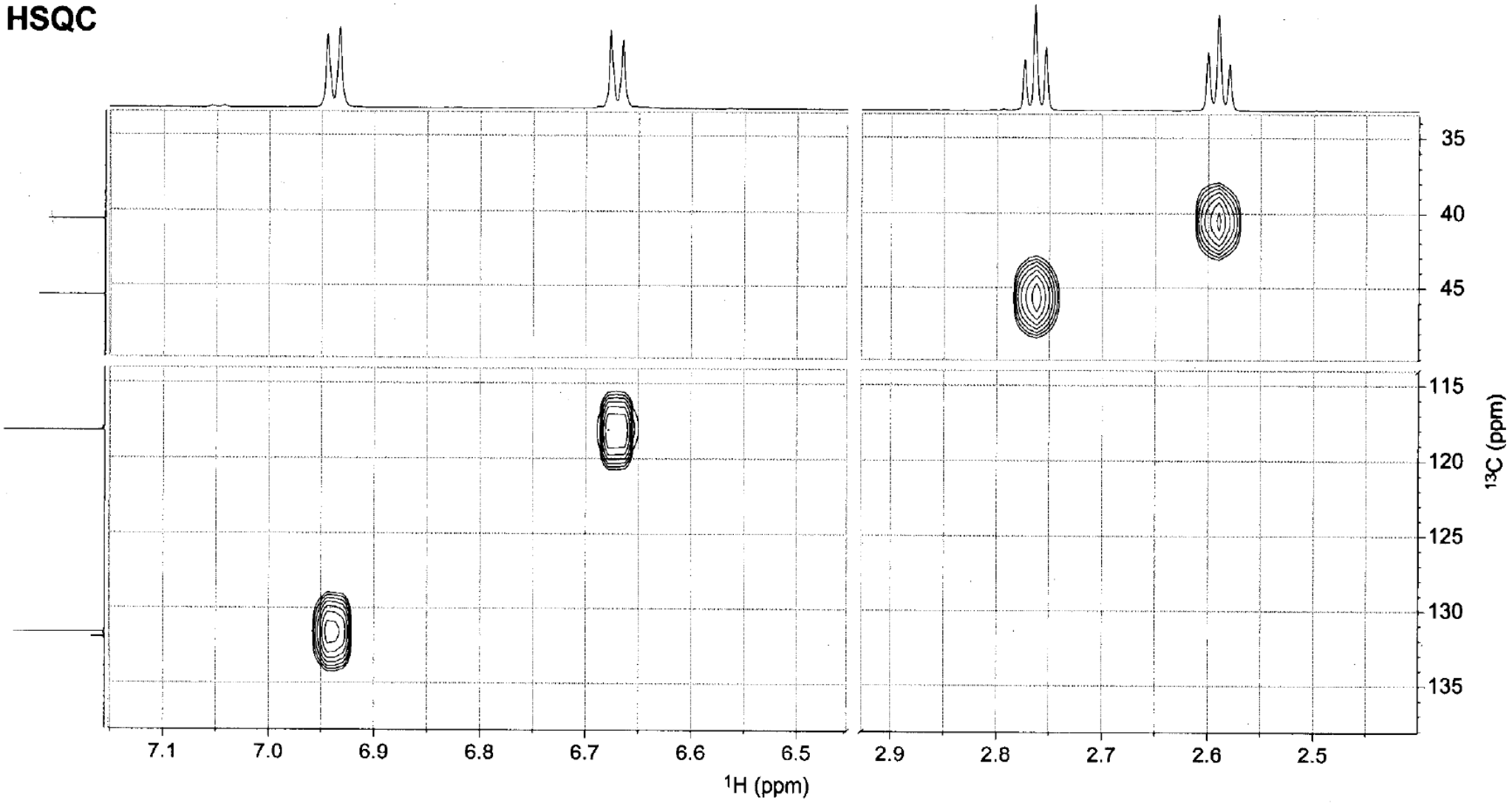
^{1}H NMR spektar spoja snimljen u CD_3OD. Spoj sadrži i protone koji se izmijene u CD_3OD.



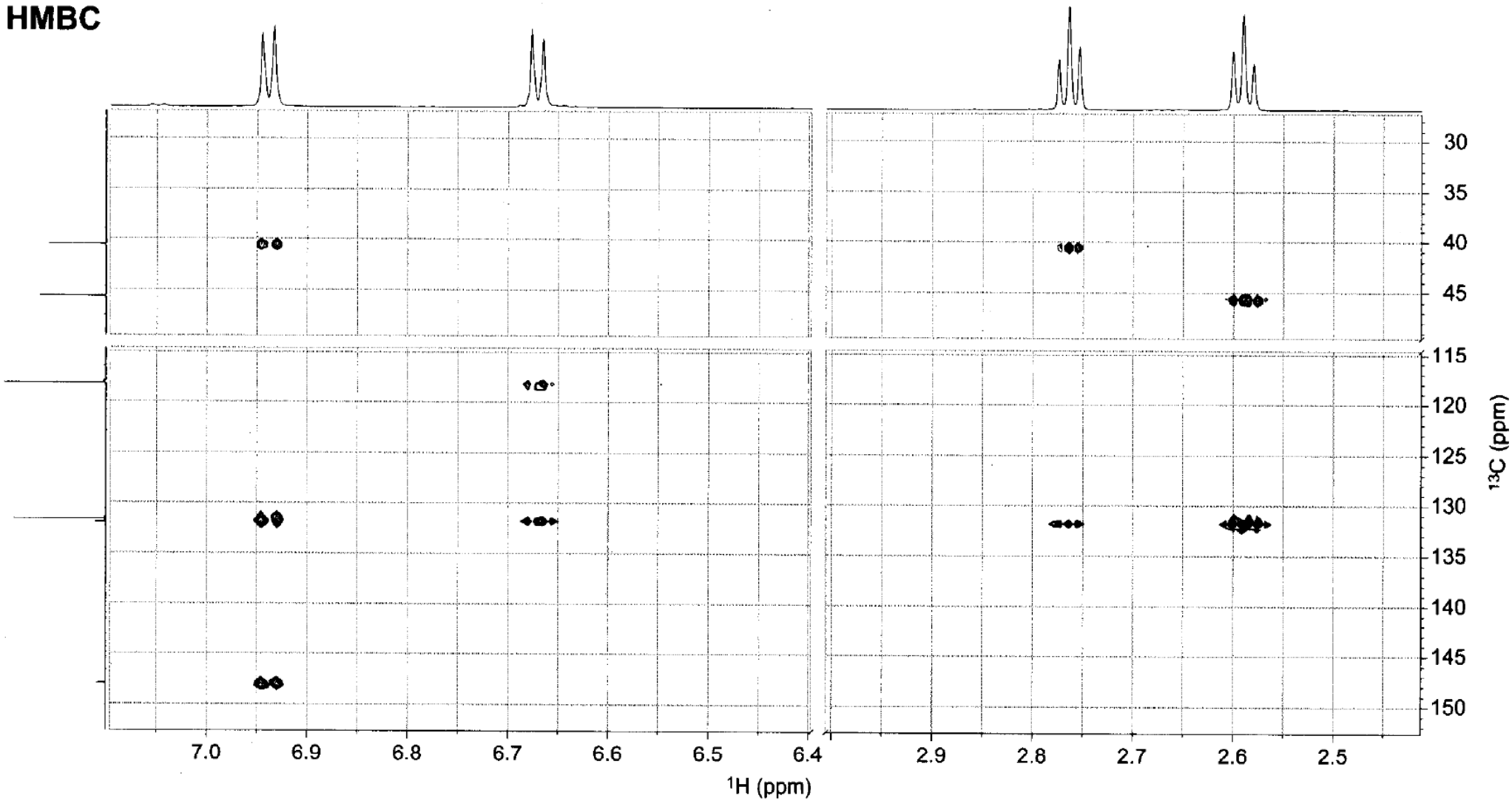
^{13}C NMR spektri spoja snimljeni u CD_3OD.



COSY spektar spoja snimljen u CD_3OD.

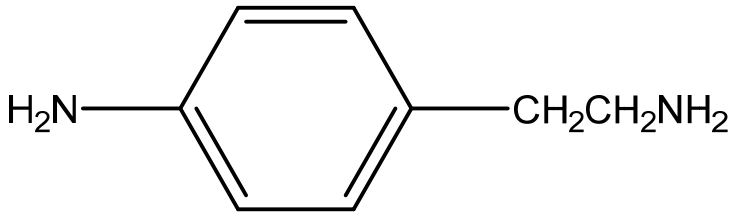


HSQC spektar spoja snimljen u CD_3OD.



HMBC spektar spoja snimljen u CD_3OD.

$M = 136\ \text{g mol}^{-1}$



- informacije iz IR:

  3400 i 3550 cm^{-1} N–H istezanje
  2900 i 2950 cm^{-1} C–H alifatsko istezanje
  3050 cm^{-1} C–H aromatsko istezanje
  1500 i 1400 cm^{-1} C–C istezanja
  800 cm^{-1} C–H aromatsko svijanje

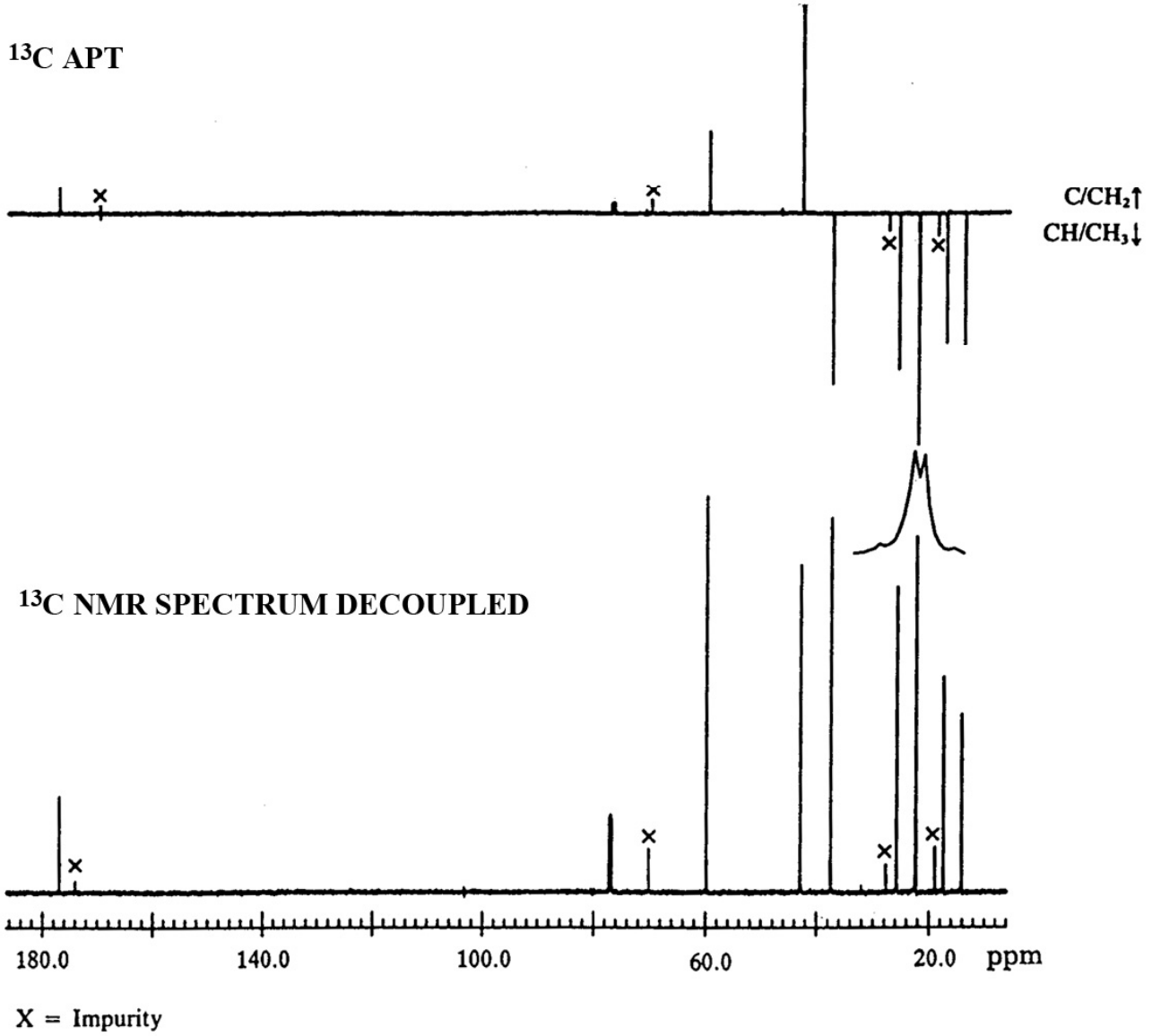
- informacije iz ^{1}H NMR:

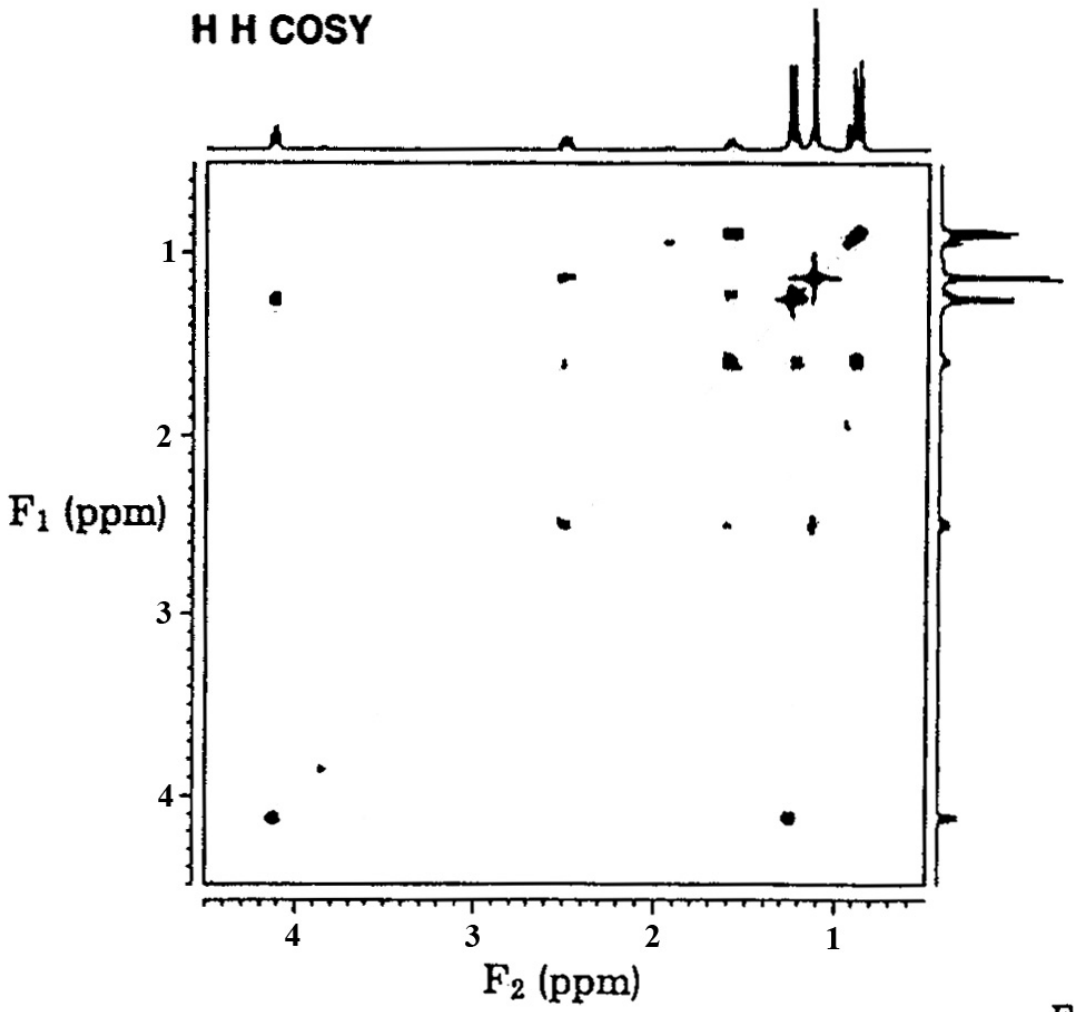
$\delta(^{1}\text{H})$ / ppm	multiplet	H-atom
2,59	t	$-CH_2-$
2,75	t	$-CH_2-$
6,68	d	=CH–
6,95	d	=CH–

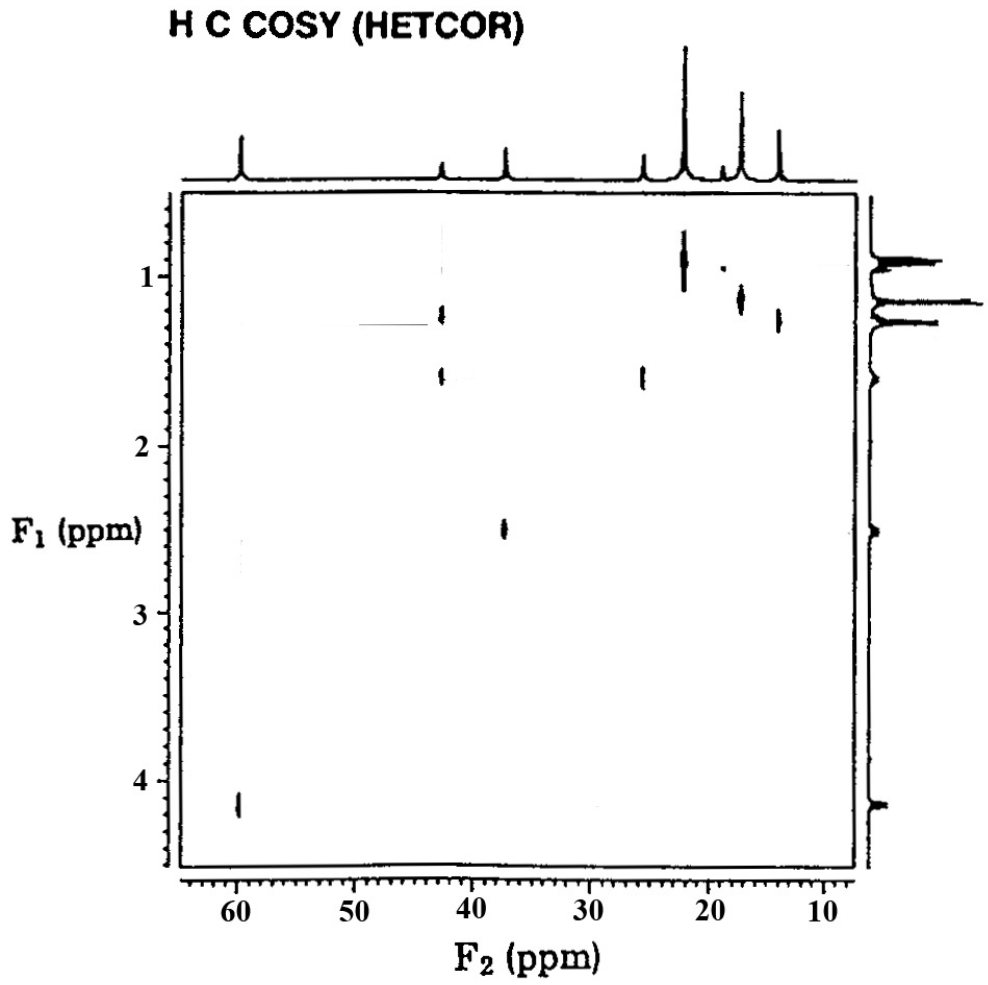
- informacije iz ^{13}C NMR:

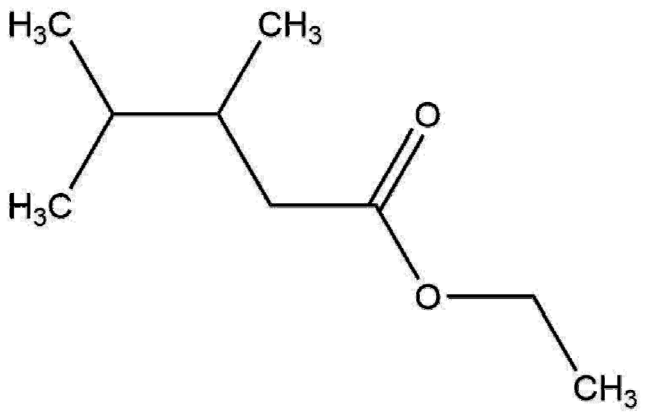
$\delta(^{13}\text{C})$ / ppm	C-atom
41	$-CH_2-$
46	$-CH_2-$
118	=CH–
131	=CH–
131,5	C
148	C

5. Odredite strukturu spoja molekulske formule $C_9H_{18}O_2$ na temelju njegovih ^{13}C NMR, COSY i HETCOR spektara.




H H COSY
1
2
3
4
F1 (ppm)
4
3
2
1
F2 (ppm)


H C COSY (HETCOR)
1
2
3
4
F1 (ppm)
60
50
40
30
20
10
F2 (ppm)



- informacije iz ^{1}H NMR:

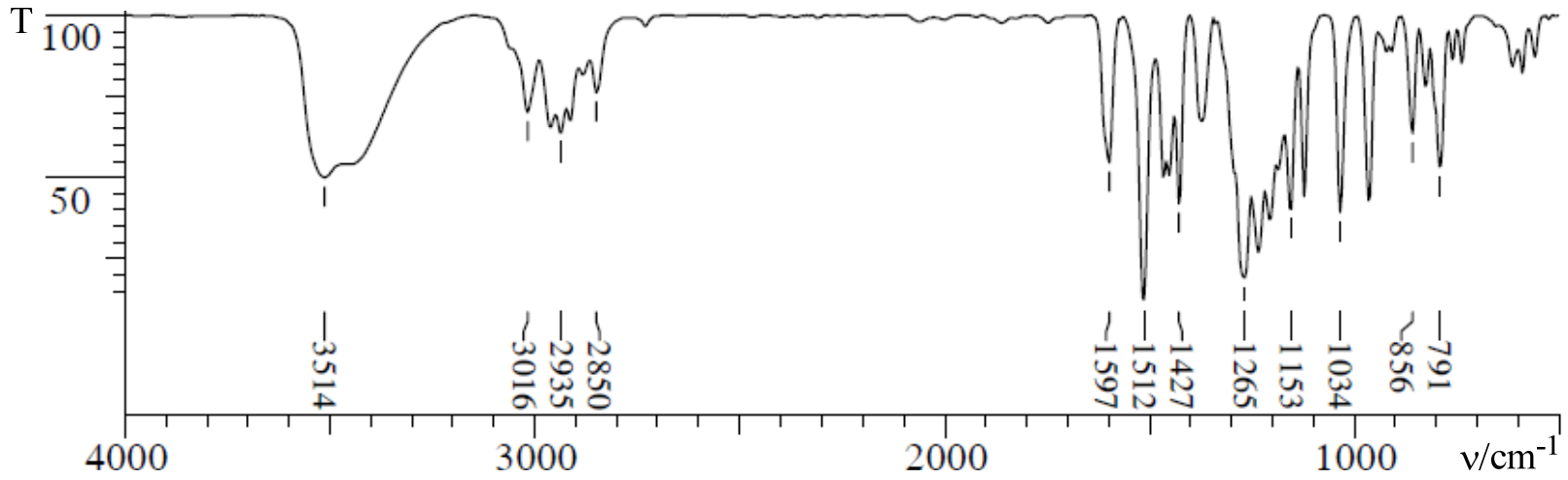
$\delta(^{1}H)$ / ppm	H-atom
0,9	$-CH_3$, $-CH_3$
1,2	$-CH_3$
1,3	$-CH_3$
1,7	$-CH_2-$, $=CH-$
2,5	$=CH-$
4,1	$-CH_2-$

- informacije iz ^{13}C NMR:

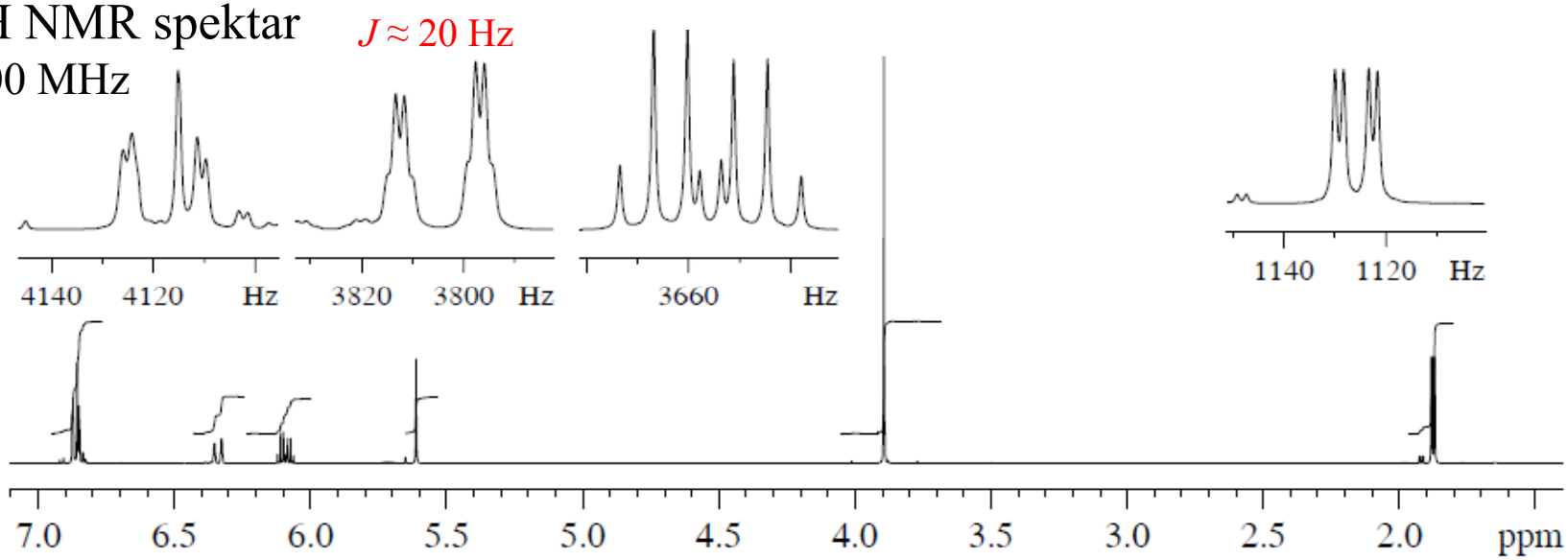
$\delta(^{13}C)$ / ppm	C-atom
17	$-CH_3$
19	$-CH_3$
20,9	$-CH_3$
21,1	$-CH_3$
27	$=CH-$
38	$=CH-$
43	$-CH_2-$
60	$-CH_2-$
178	C=O

6. Identificirajte spoj molarne mase 164 g/mol na temelju njegovih IR, ^{1}H NMR, ^{13}C NMR, COSY, HMQC i HMBC spektara.

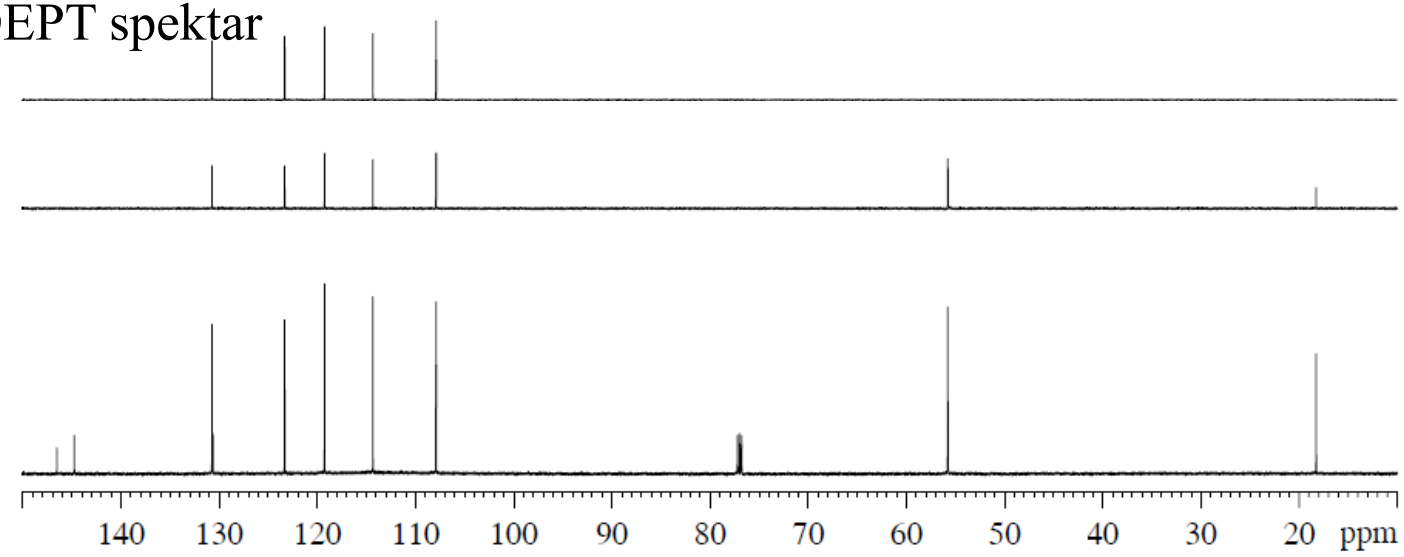
IR spektar



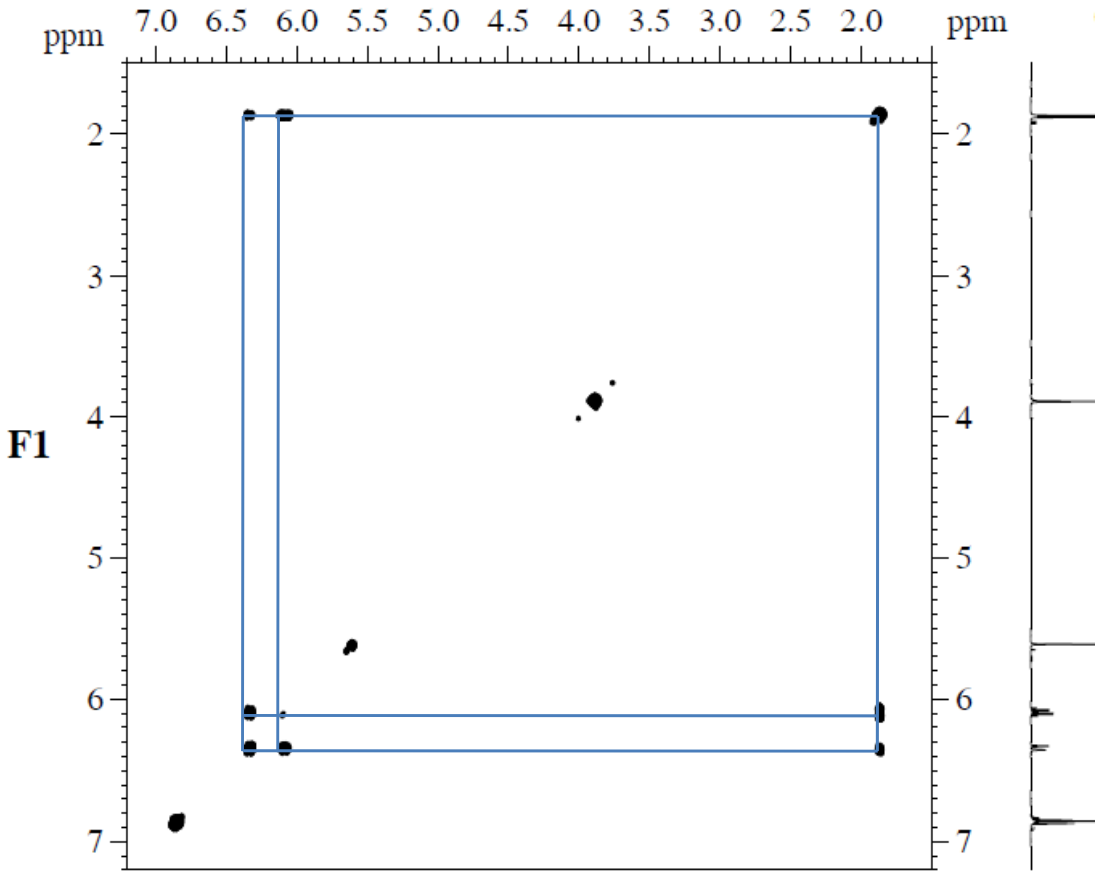
^{1}H NMR spektar
600 MHz



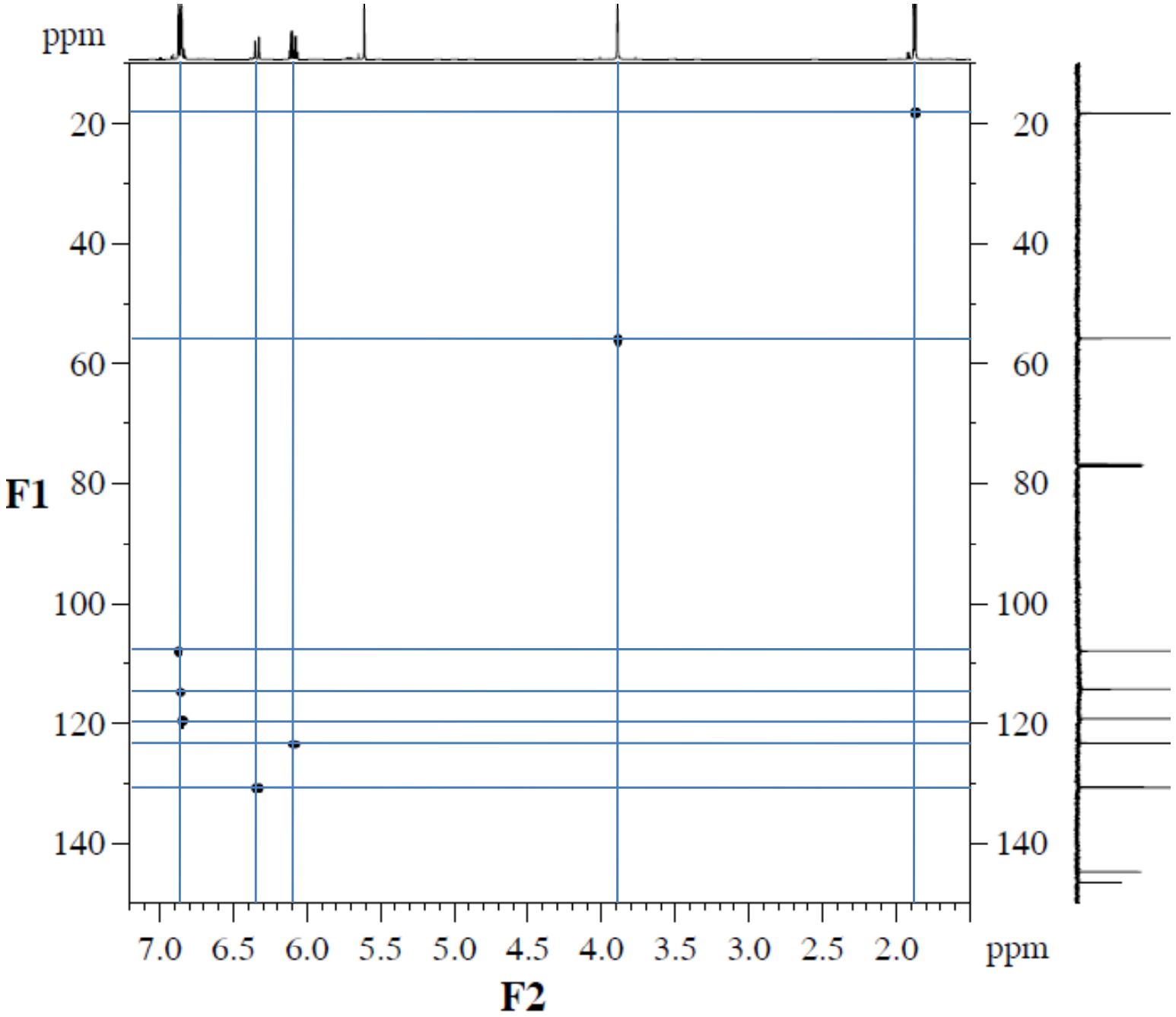
^{13}C NMR/DEPT spektar

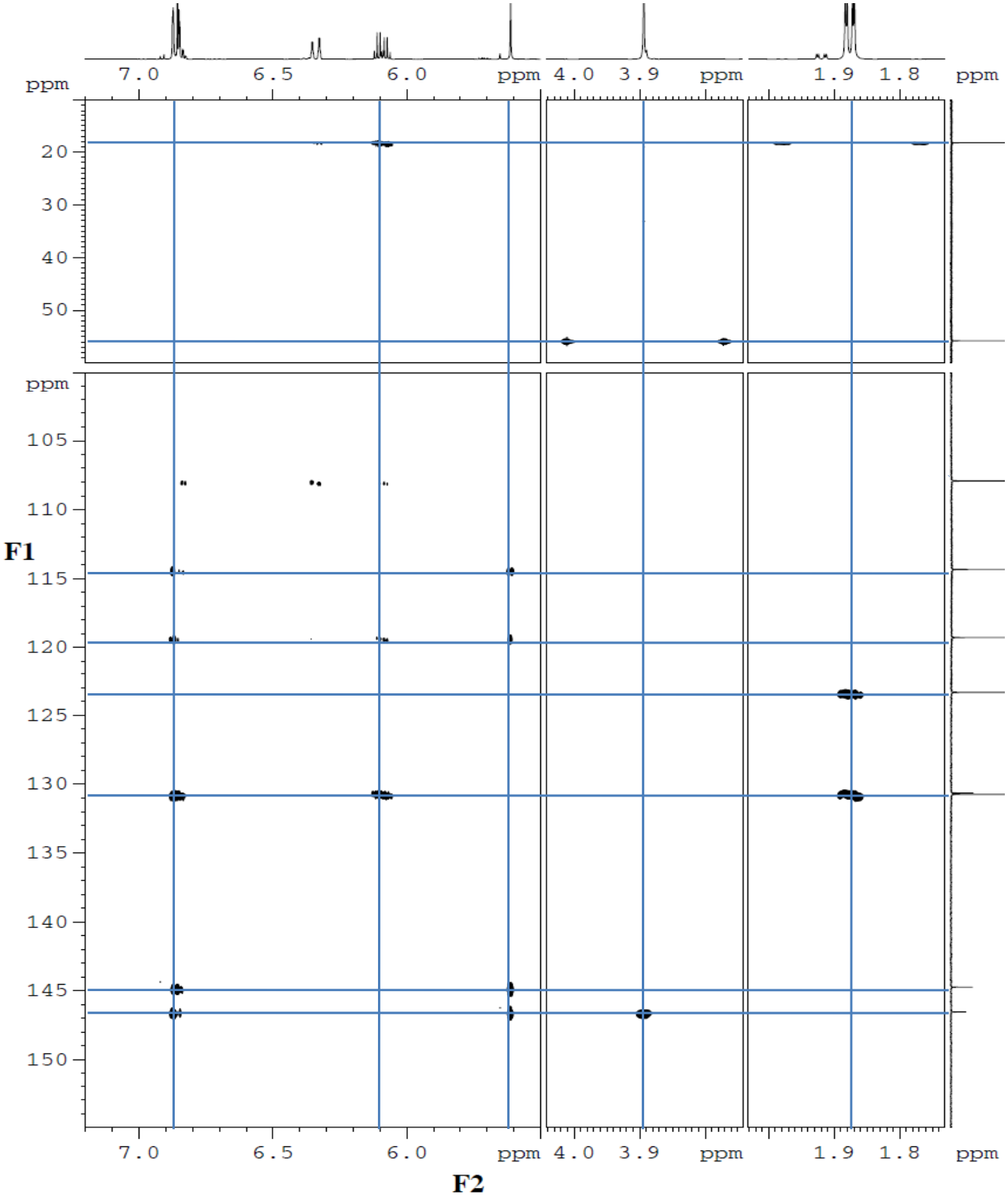


COSY spektar



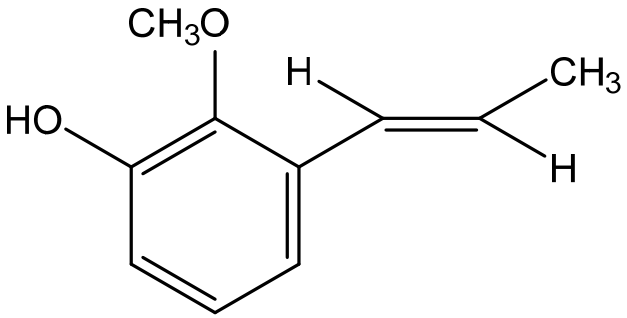
HMQC spektar





HMBC spektar

J ≈ 20 Hz
trans



- informacije iz IR:
  3514 cm^{-1} O–H istezanje
  3016 cm^{-1} C–H aromatsko istezanje
  2935 i 2850 cm^{-1} C–H alifatsko istezanje

- informacije iz ^{1}H NMR:

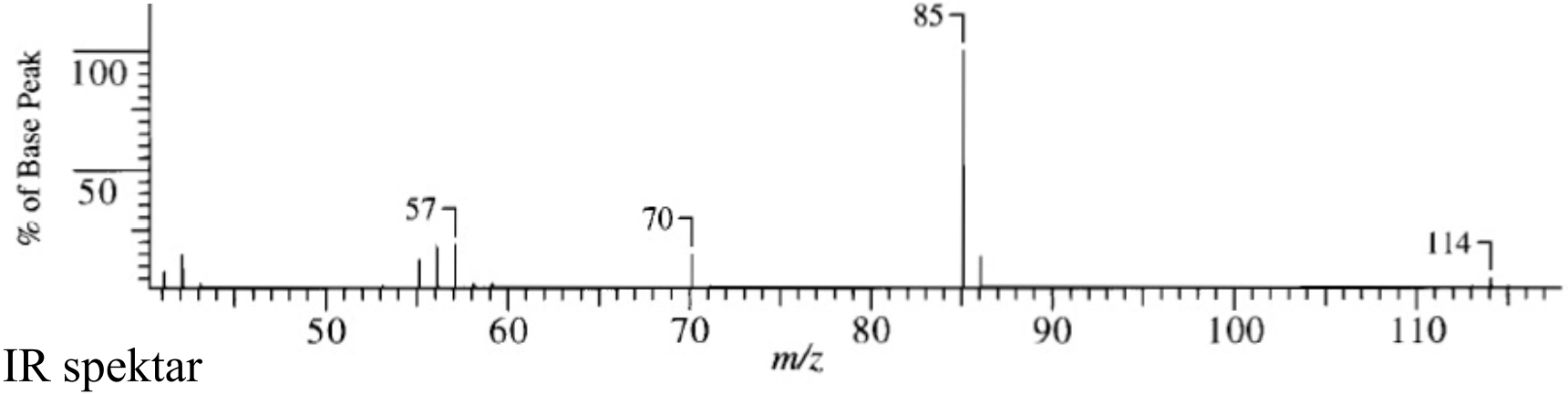
$\delta(^{1}H)$ / ppm	**multiplet**	**H-atom**
1,9	dd	$–CH_3$
3,9	s	$–CH_3$
5,6	s	–OH
6,1	qd	=CH–
6,3	qd	=CH–
6,9	m	=CH–, =CH–

- informacije iz ^{13}C NMR:

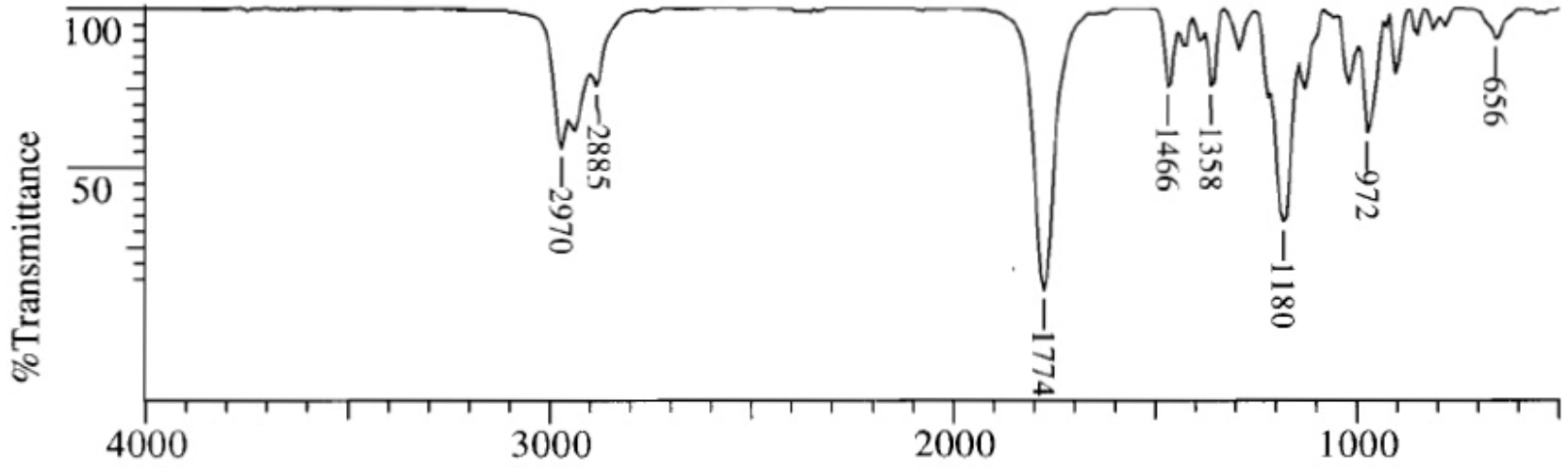
$\delta(^{13}C)$ / ppm	**C-atom**
18	$–CH_3$
56	$–CH_3$
108	=CH–
115	=CH–
120	=CH–
124	=CH–
131	=CH–
145	C
147	C

7. Identificirajte spoj na temelju njegovih MS, IR, ^{1}H NMR, ^{13}C NMR, COSY i HMQC spektara.

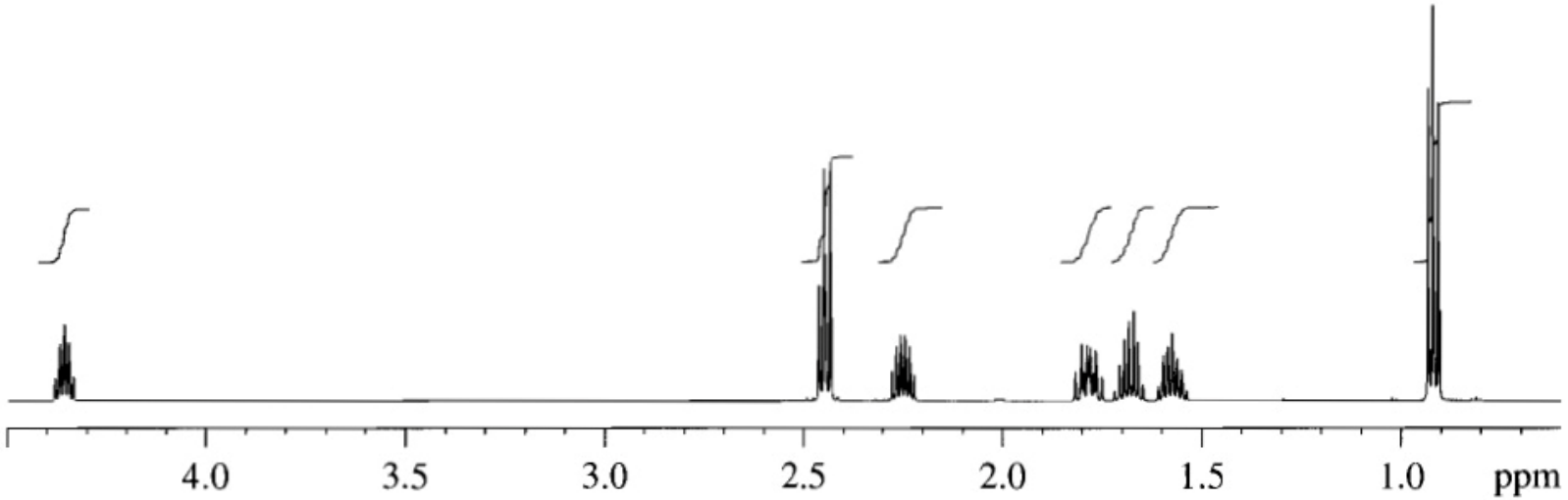
Spektar MS



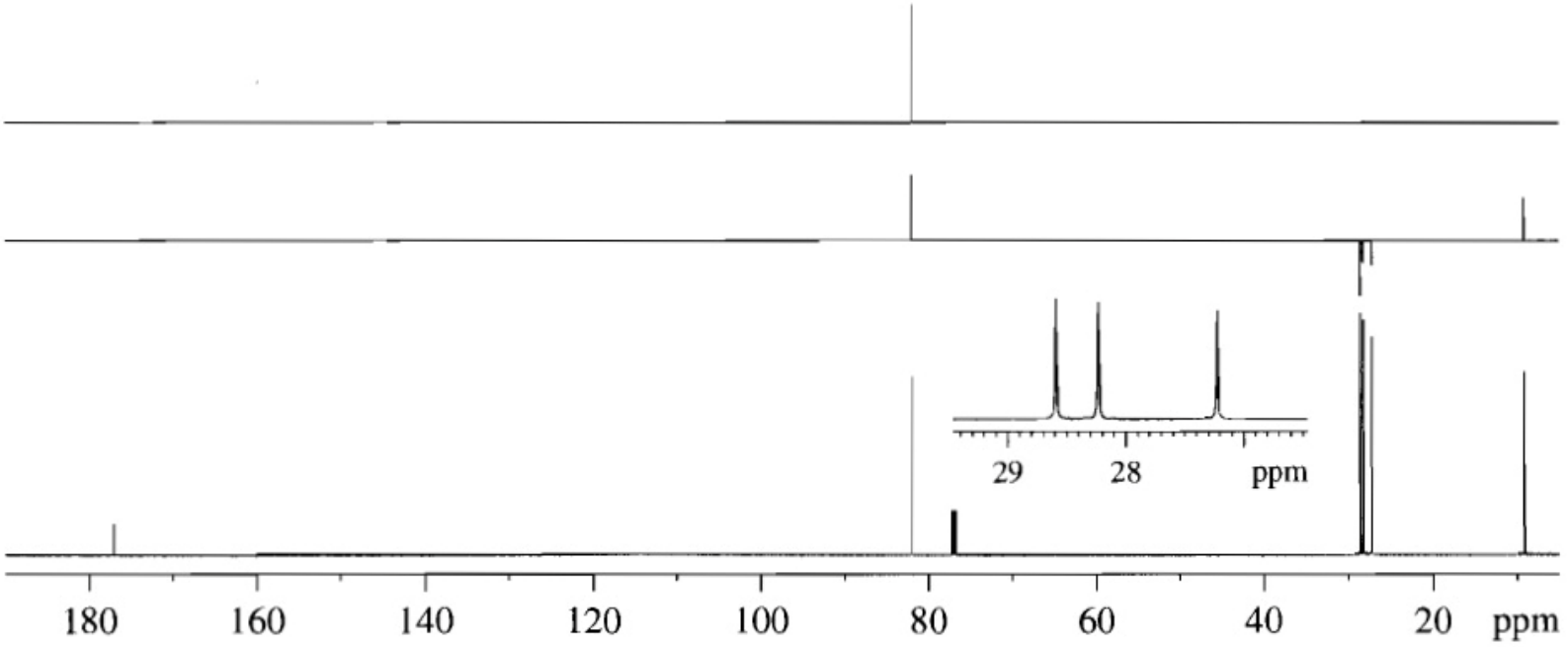
IR spektar

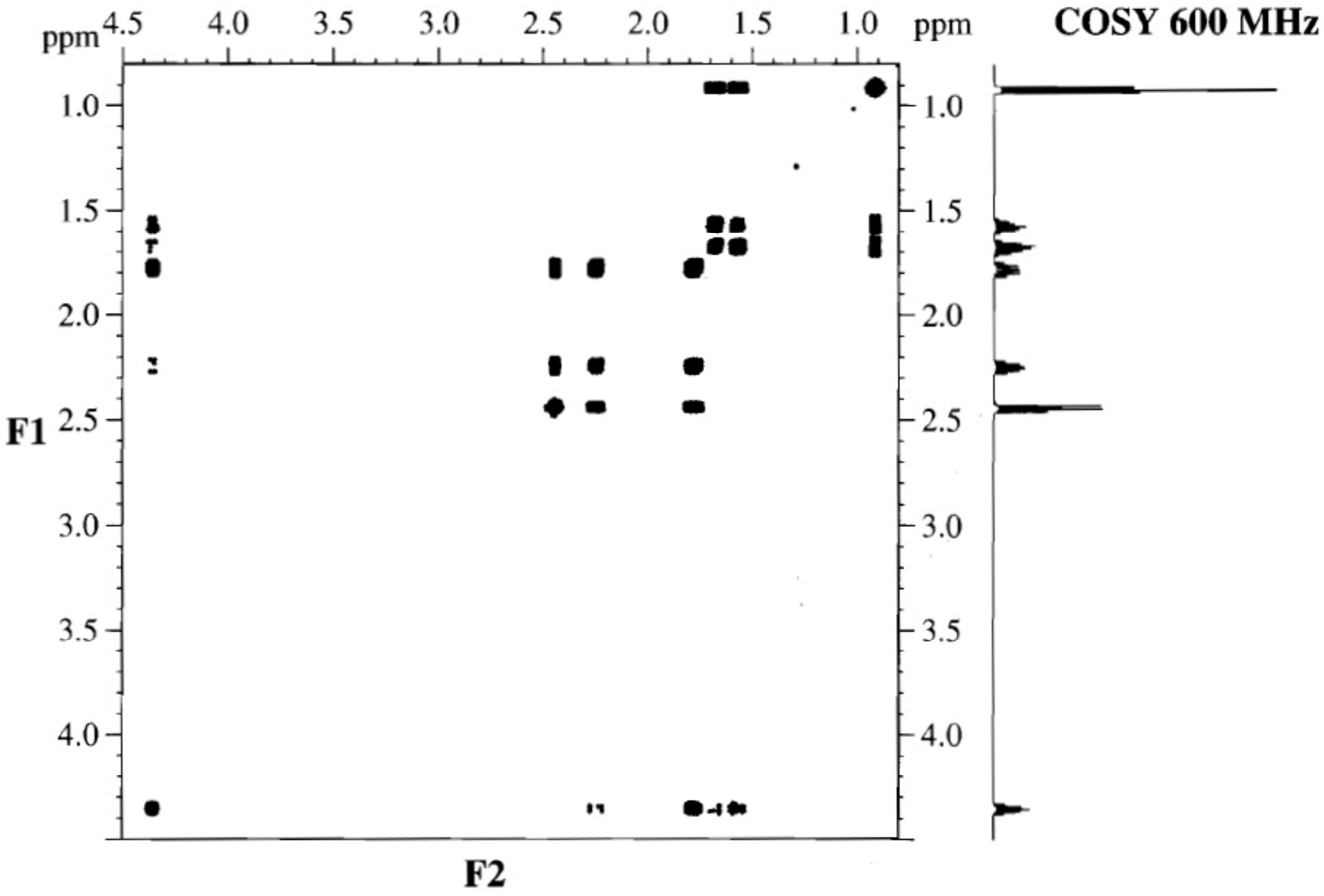


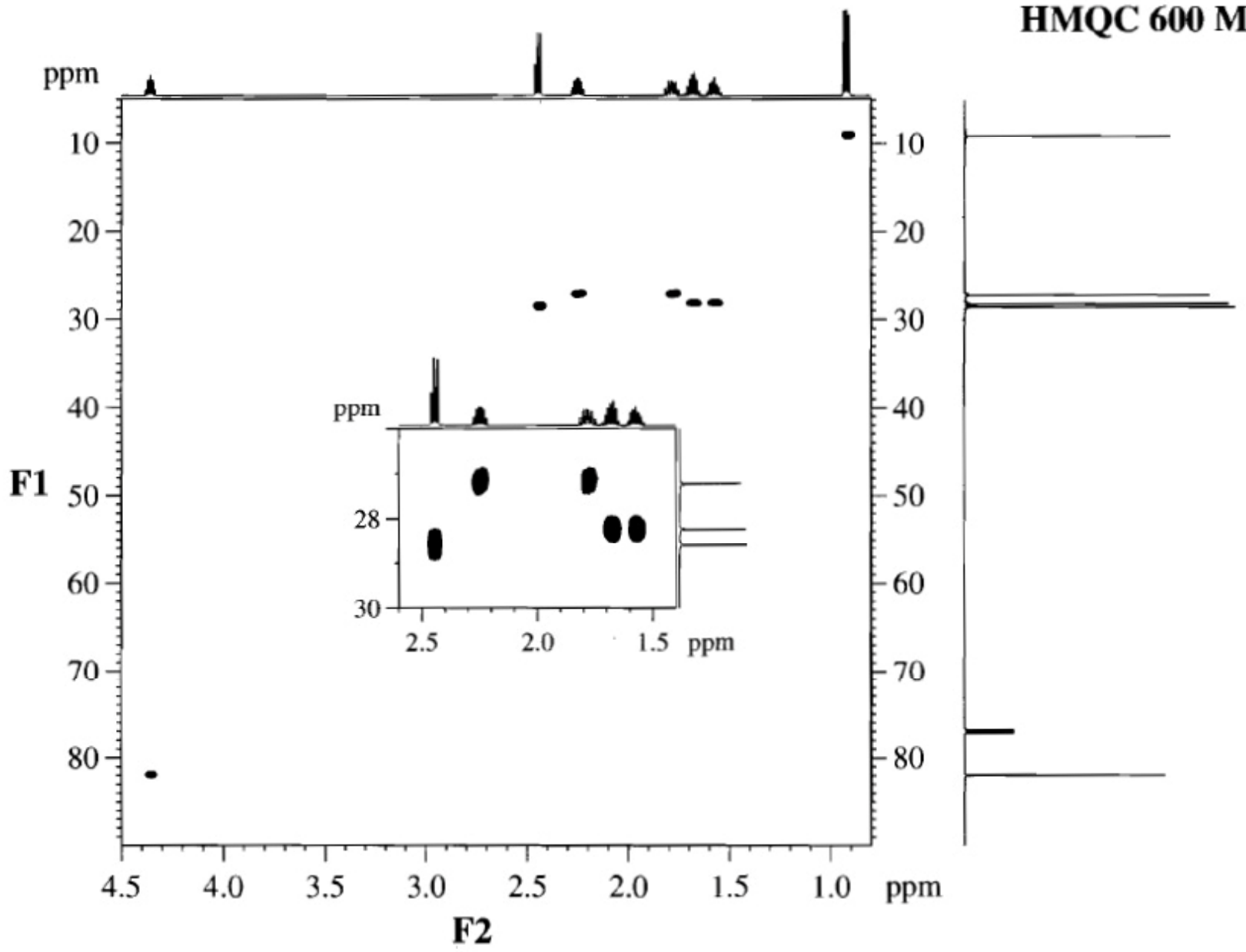
^{1}H NMR 600 MHz

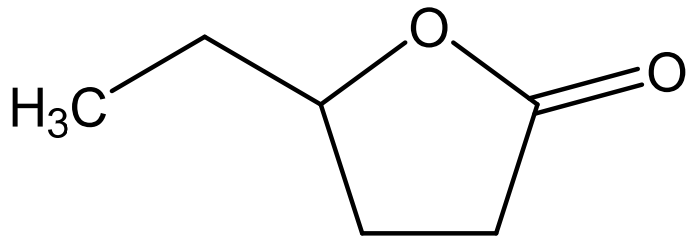


^{13}C/DEPT NMR 150.9 MHz




COSY 600 MHz
ppm
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
F1
F2


HMQC 600 MHz
ppm
10
20
30
40
50
60
70
80
F1
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
F2
ppm
28
30
2.5
2.0
1.5
ppm



- informacije iz IR:
  2970 i 2885 cm^{-1} C–H alifatsko istezanje
  1774 cm^{-1} C=O istezanje lakton
  1180 cm^{-1} C–O asimetrično istezanje

- informacije iz MS:
  bazni pik: 85
  $M^+ = 114$

- informacije iz ^1H NMR:

$\delta(^1H)$ / ppm	multiplet	H-atom
0,90	t	$–CH_3$
1,57; 1,67	m	$–CH_2–$
1,80; 2,25	m	$–CH_2–$
2,45	t	$–CH_2–$
4,35	m	CH

- informacije iz ^{13}C NMR:

$\delta(^1H)$ / ppm	C-atom
9	$–CH_3$
27,2	$–CH_2–$
28,2	$–CH_2–$
28,6	$–CH_2–$
83	CH
178	C=O