Spektroskopska strukturna analiza
Dodatni zadaci

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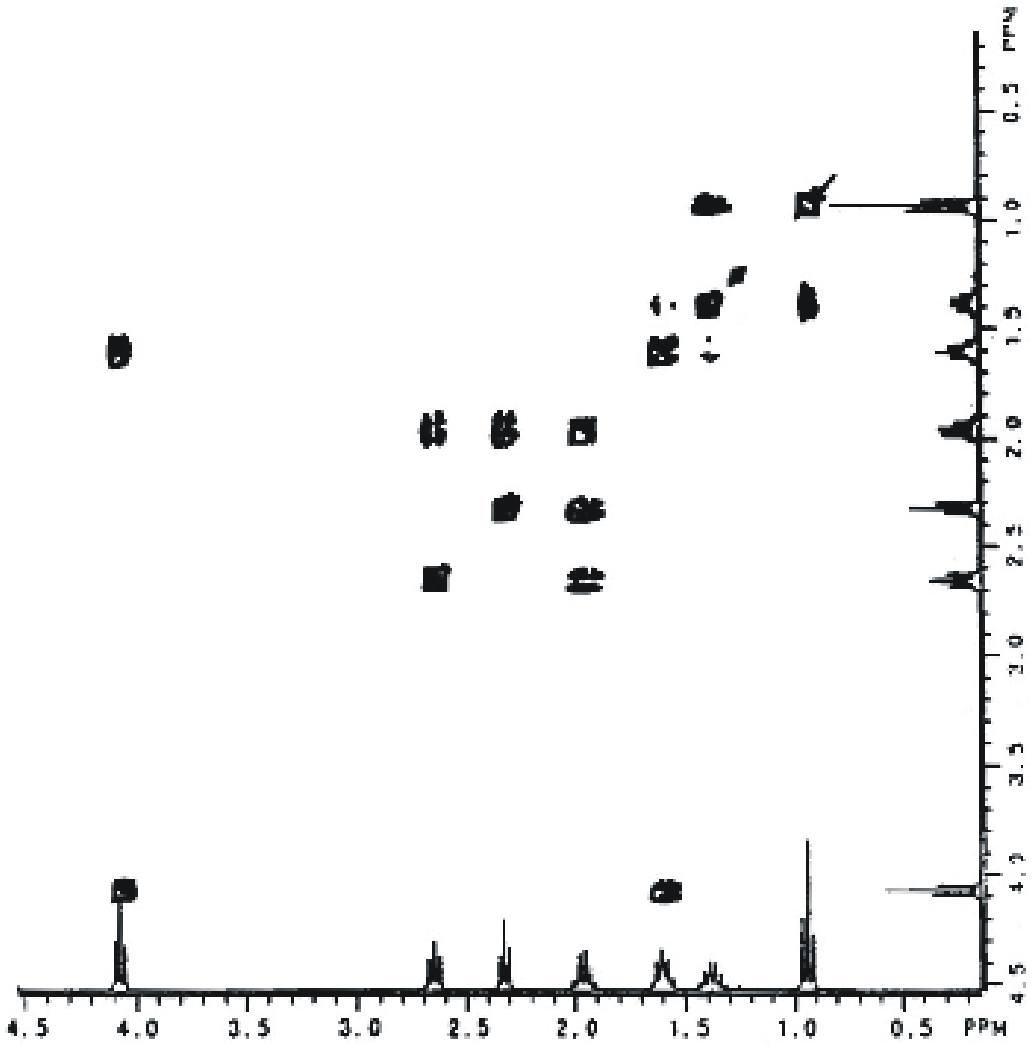
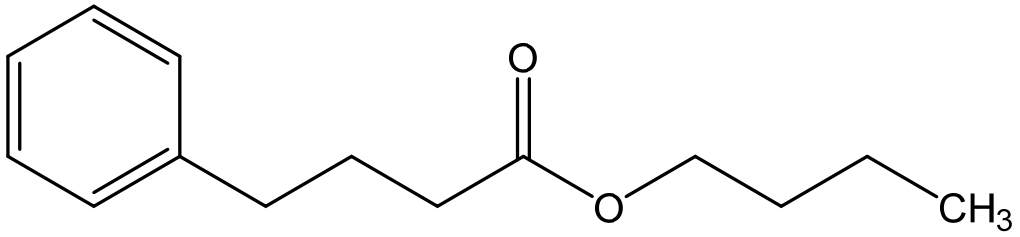
1. Odredite strukturu spoja molekulske formule $C_{14}H_{20}O_2$ na temelju njegovog COSY spektra. ^1H NMR spektar sadrži još široki singlet s integralom 5.



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

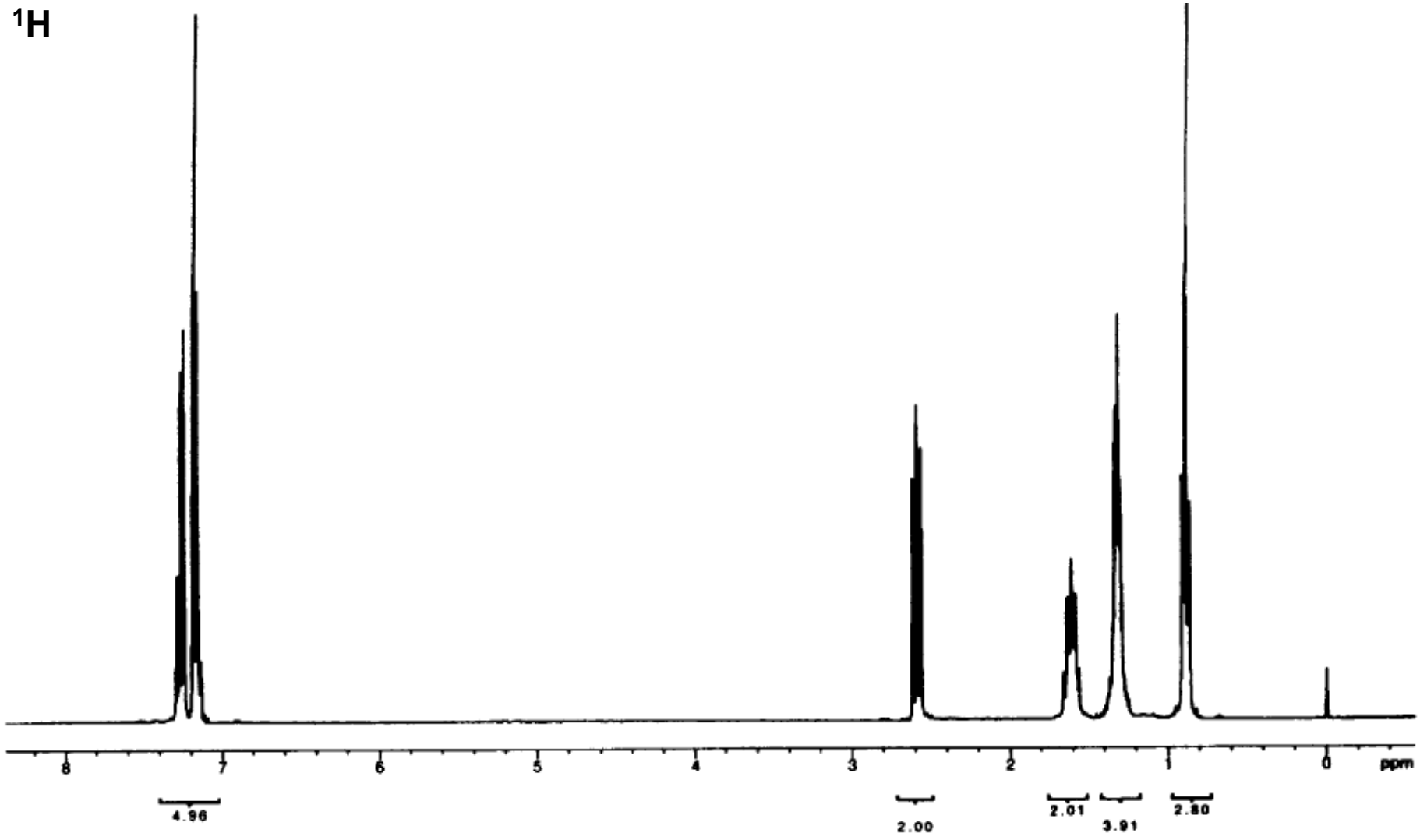
$$\mathrm{IHD} = \frac{1}{2}[2\mathrm{C} + 2 + \mathrm{N} - (\mathrm{H} + \mathrm{X})]$$

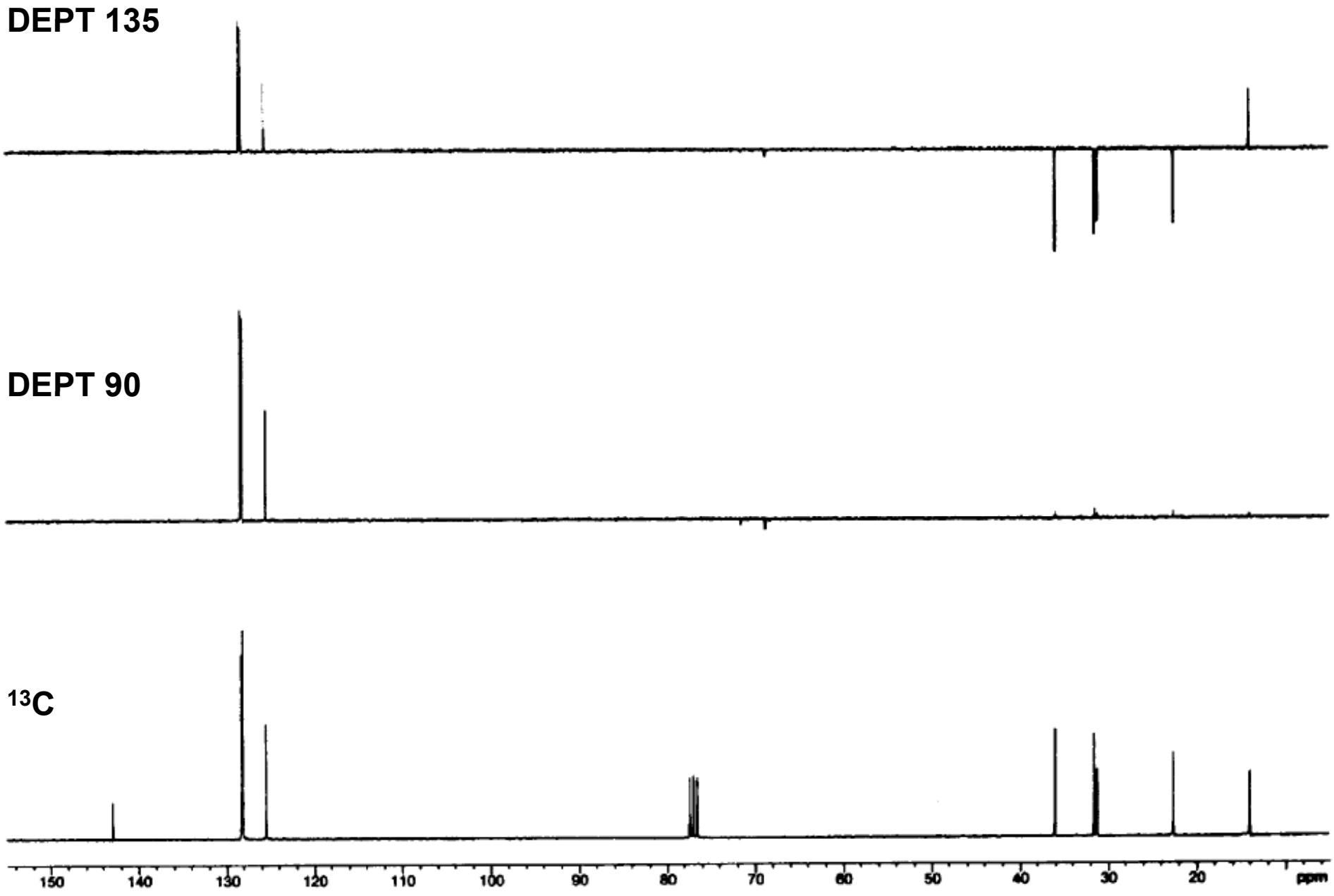
- za ovaj spoj: IHD = [2·14+2–20]/2 = 5

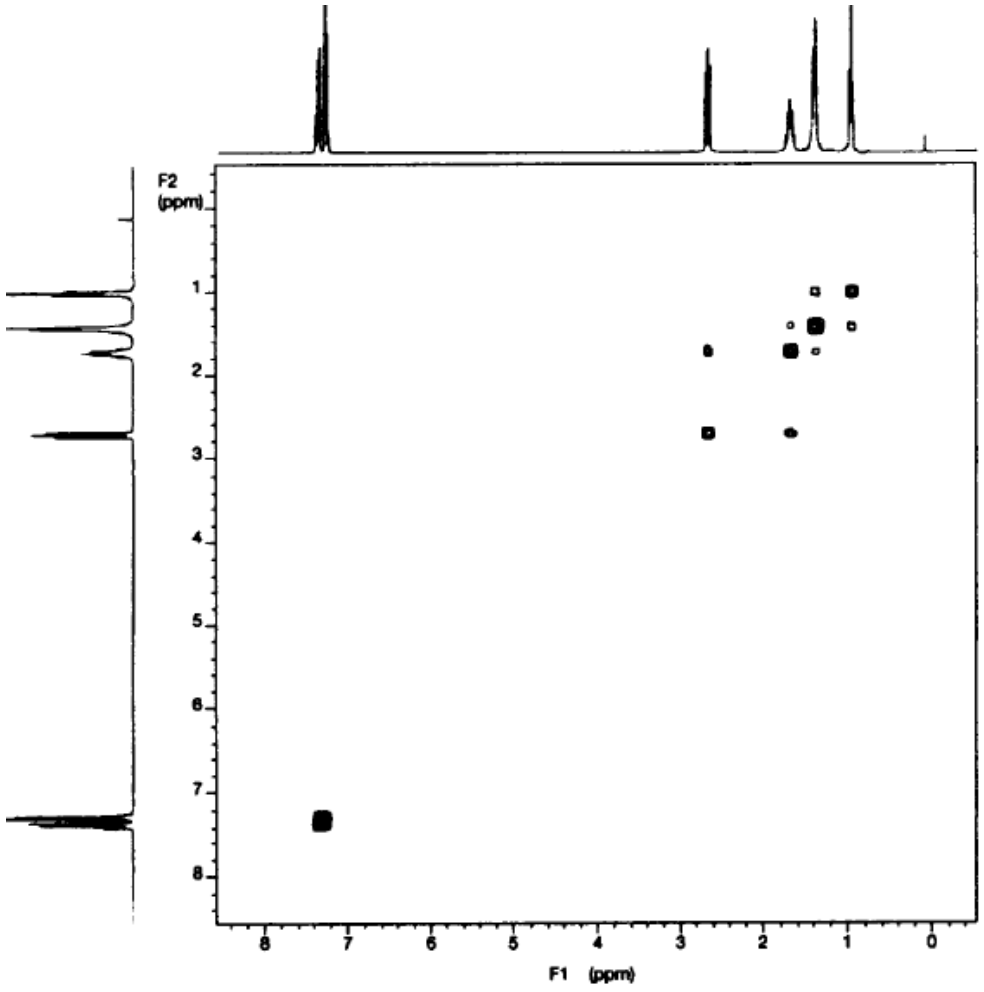


$\delta(^1\mathrm{H})$ / ppm	multiplet	H-atom
0,95	t	$-CH_3$
1,40	m (heptet)	$-CH_2-$
1,55	m (kvintet)	$-CH_2-$
1,95	m (kvintet)	$-CH_2-$
2,35	t	$-CH_2-$
2,65	t	$-CH_2-$
4,10	t	$-CH_2-$
	s	$=CH-$

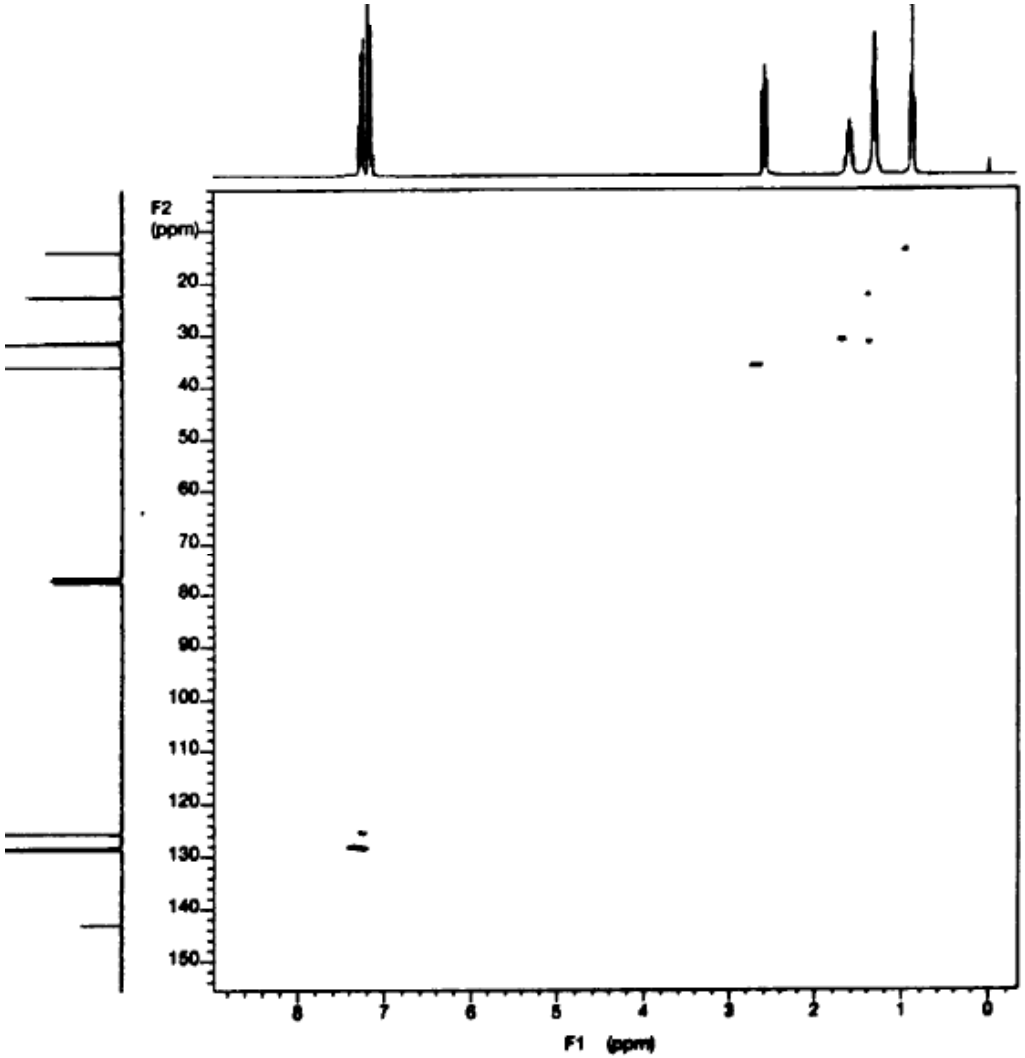
2. Odredite strukturu spoja molekulske formule $C_{11}H_{16}$ na temelju njegovih ^{1}H NMR, ^{13}C NMR, COSY i HETCOR spektara.




DEPT 135
DEPT 90
^{13}C
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

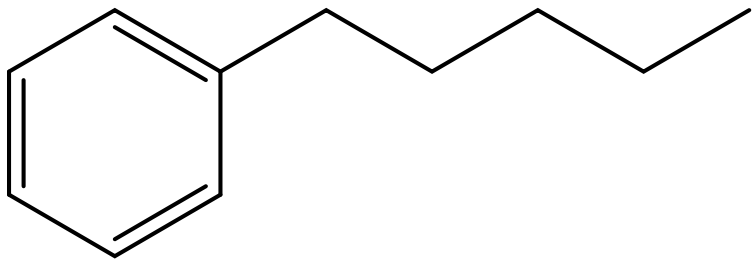

F2
(ppm)
1
2
3
4
5
6
7
8
8
7
6
5
4
3
2
1
0
F1 (ppm)

COSY


F2
(ppm)
20
30
40
50
60
70
80
90
100
110
120
130
140
150
8
7
6
5
4
3
2
1
0
F1 (ppm)

HETCOR

IHD = [2·11+2–16]/2 = 4



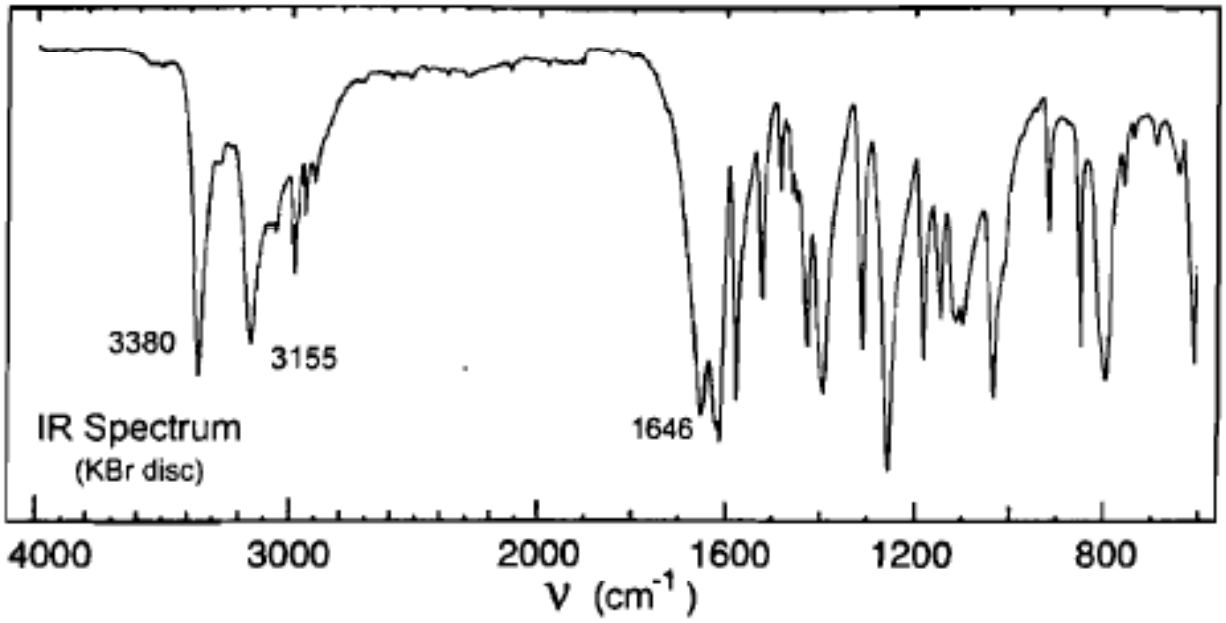
- informacije iz ^{1}H NMR:

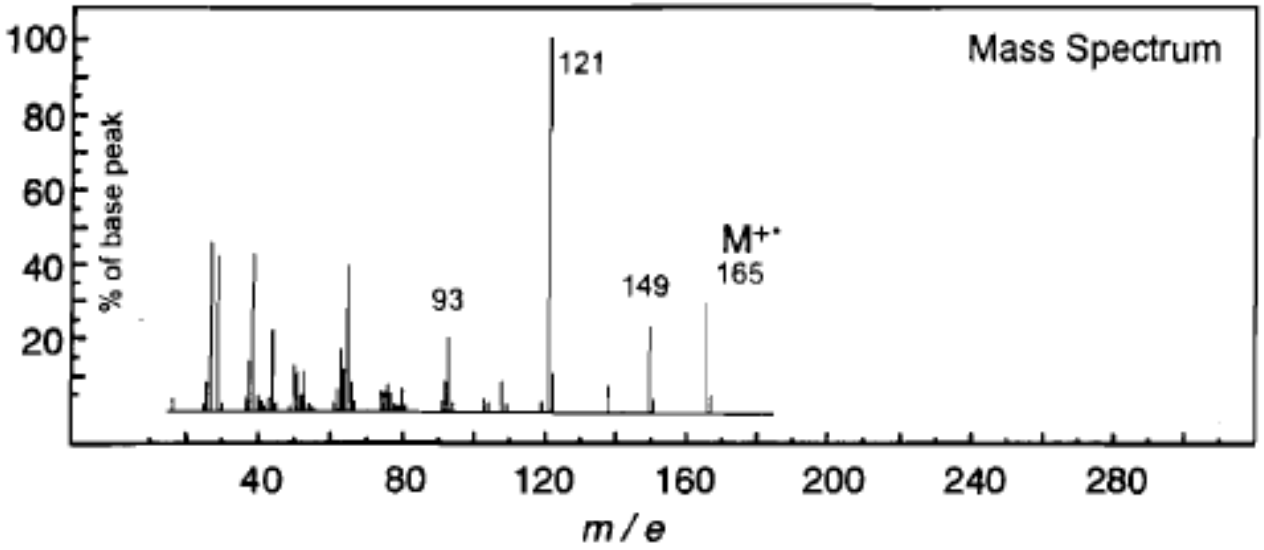
$\delta(^{1}H)$ / ppm	multiplet	H-atom
0,9	t	$–CH_3$
1,3	m	$–CH_2–$, $–CH_2–$
1,6	m(kvintet)	$–CH_2–$
2,6	t	$–CH_2–$
7,2	m	=CH–
7,3	m	=CH–

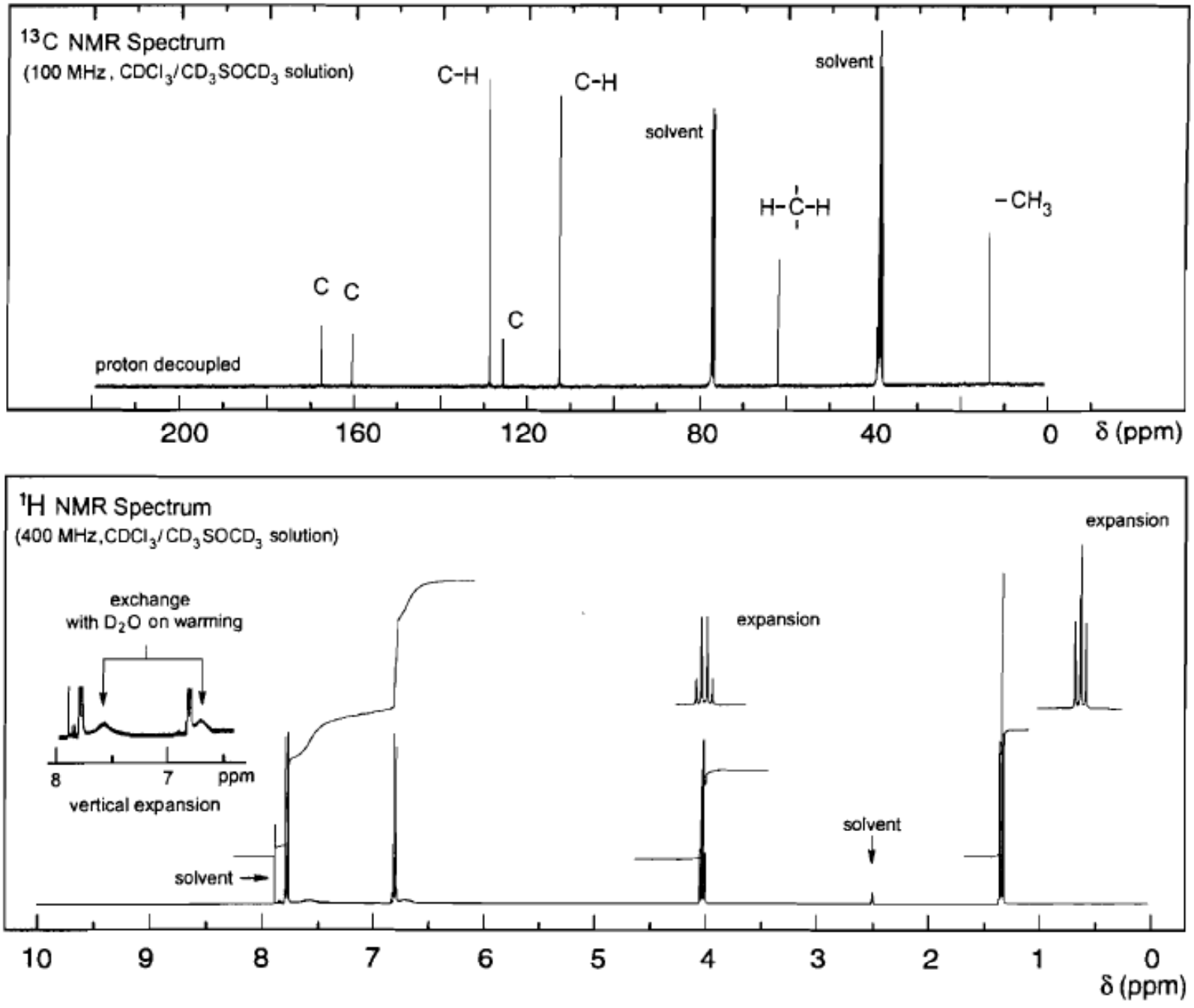
- informacije iz ^{13}C NMR:

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

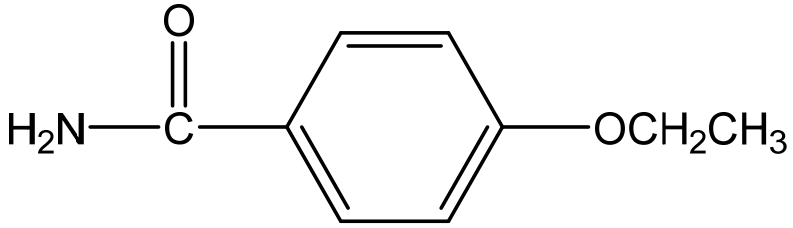
3. Odredite strukturu spoja na temelju njegovog IR, MS, ^{1}H NMR i ^{13}C NMR spektra.






^{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

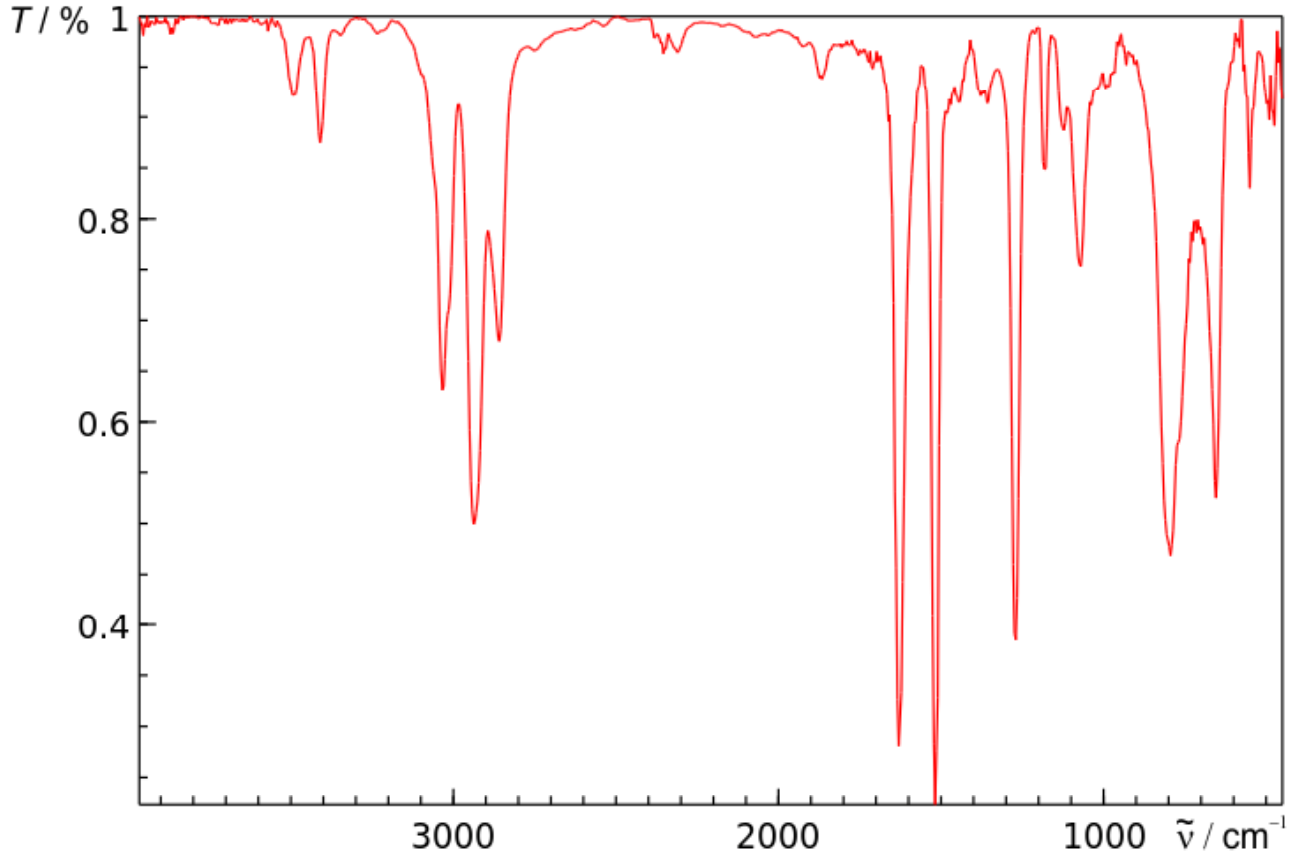
- informacije iz ^{1}H NMR:

$\delta(^1H)$ / 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–

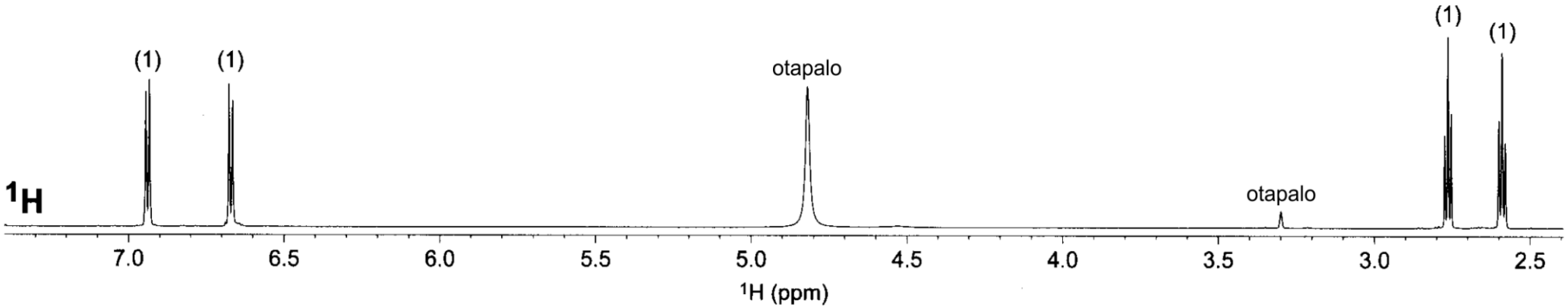
- informacije iz ^{13}C NMR:

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

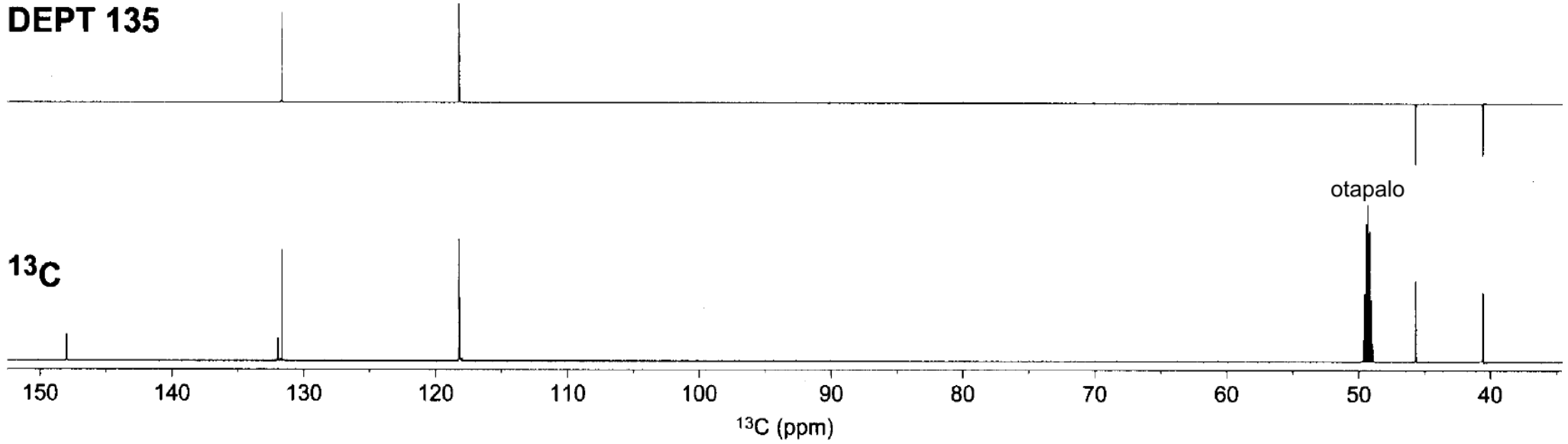
4. Odredite strukturu spoja molarne mase 136 g mol^{-1} na temelju njegovih IR, ^{1}H NMR, ^{13}C NMR, COSY, HSQC i HMBC spektara.



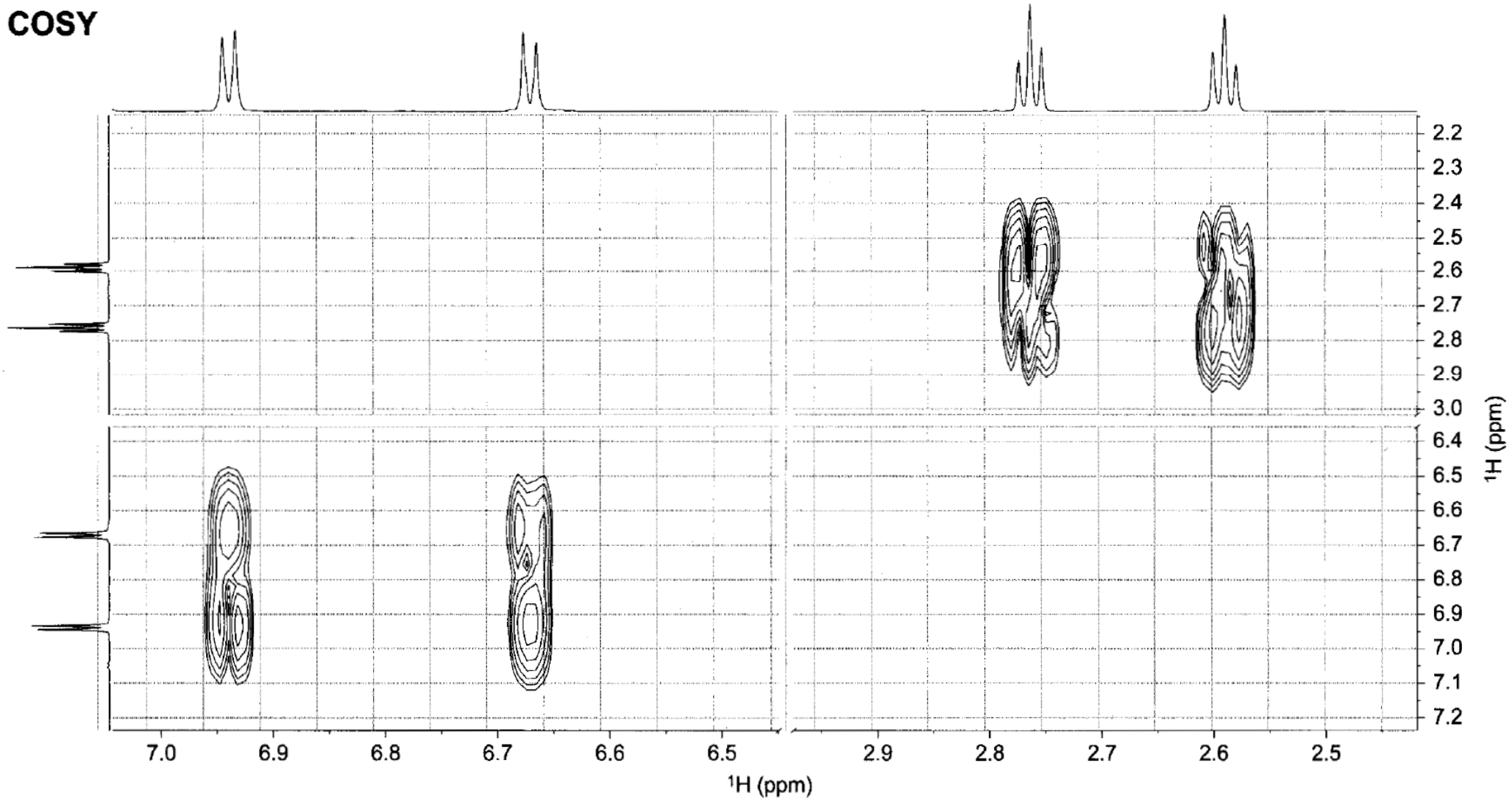
IR spektar spoja snimljen tehnikom KBr pastile.



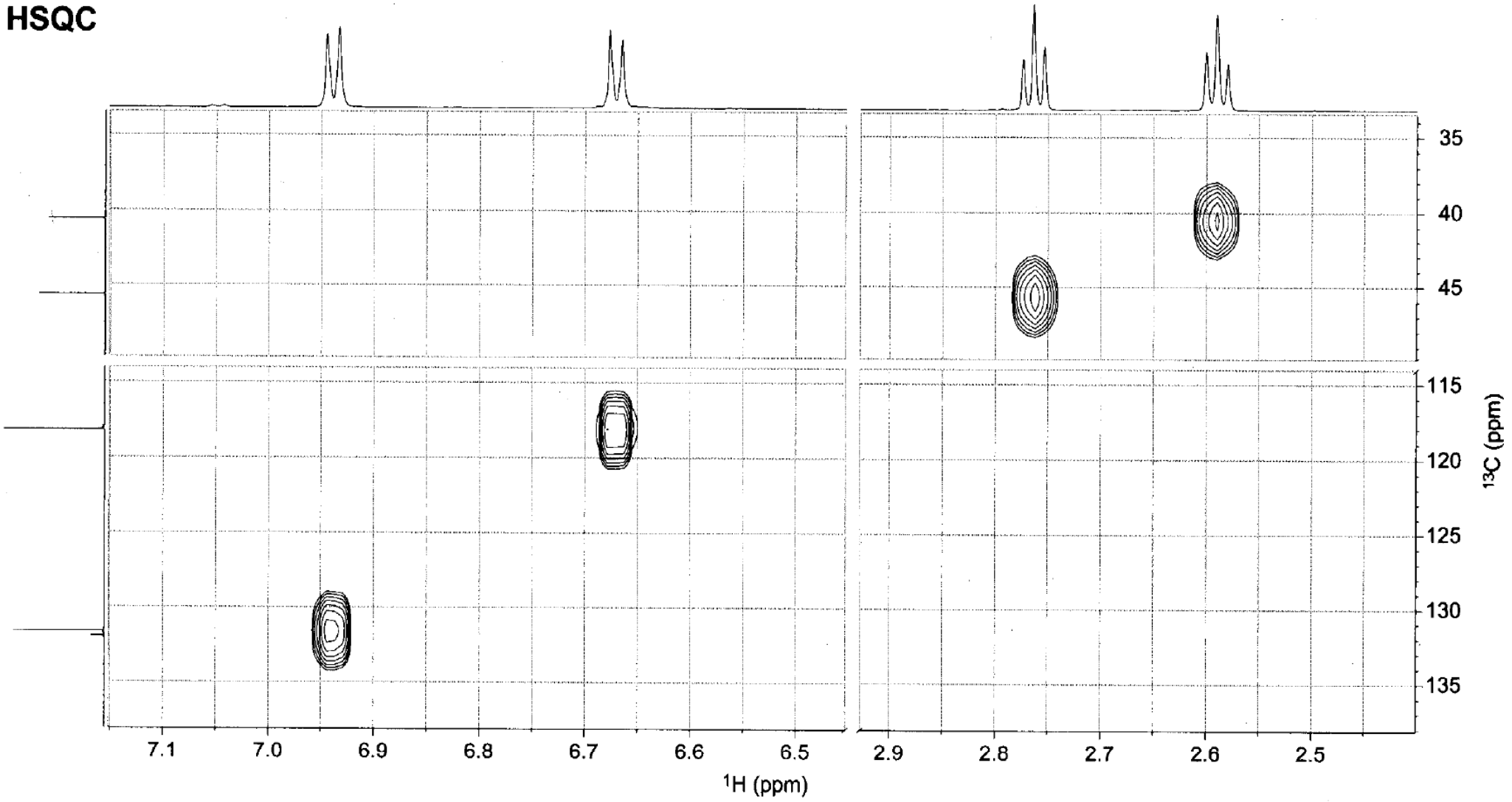
^{1}H NMR spektar spoja snimljen u CD_3OD. Spoj sadrži i protone koji se izmijene u CD_3OD.



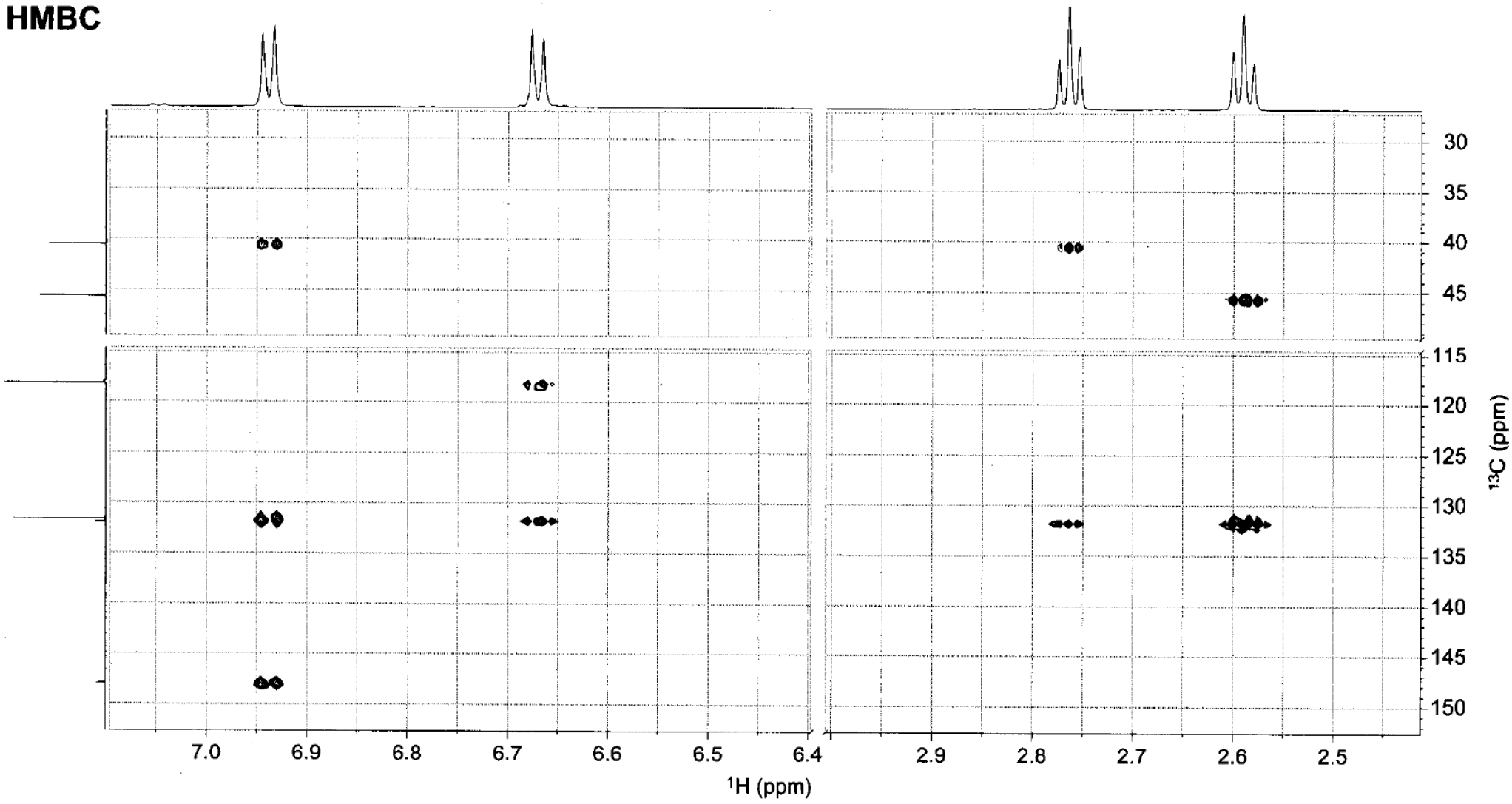
^{13}C NMR spektri spoja snimljeni u CD_3OD.



COSY spektar spoja snimljen u CD_3OD.

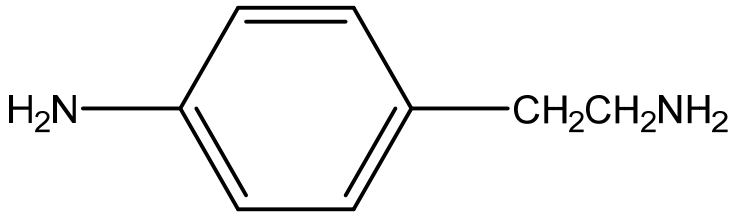


HSQC spektar spoja snimljen u CD_3OD.



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

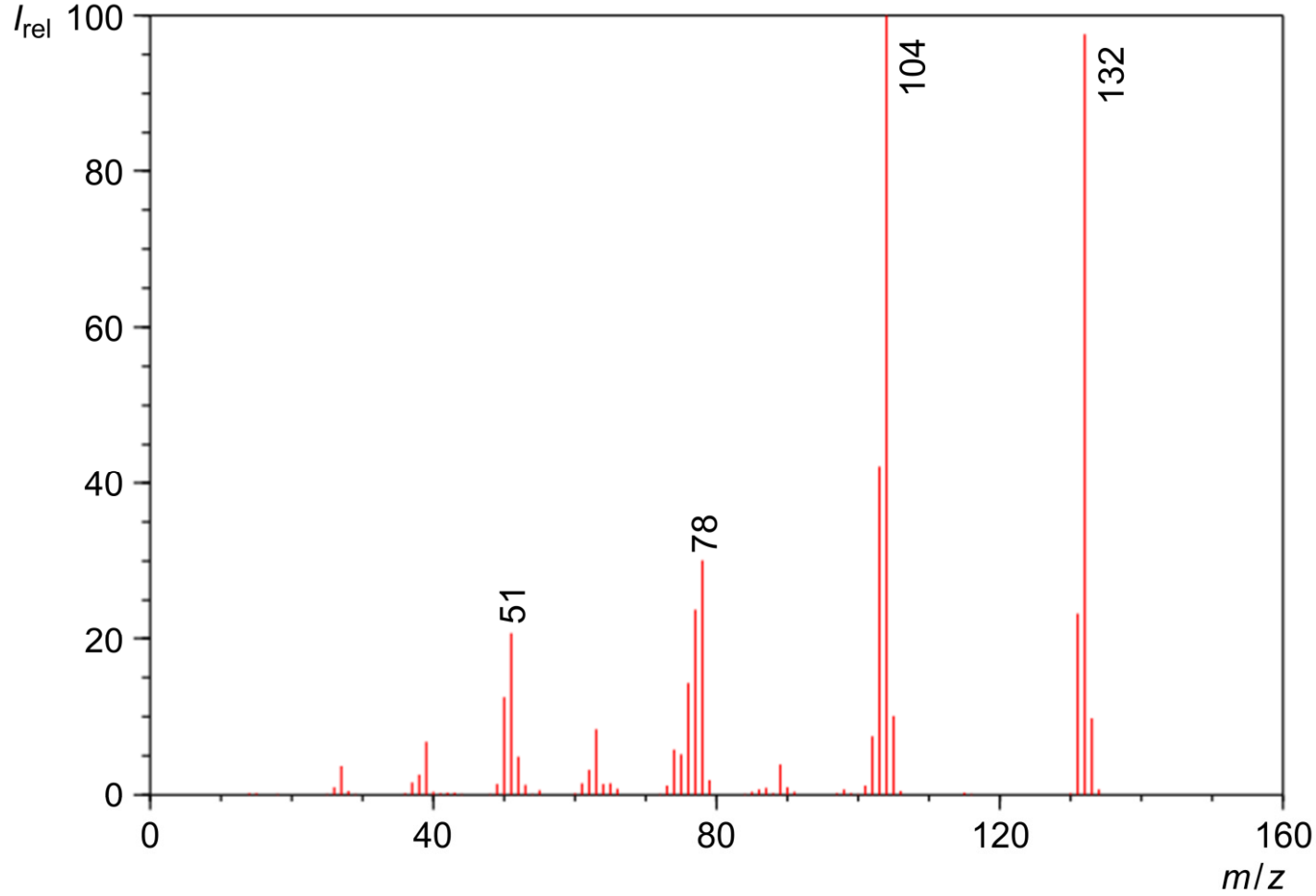
- informacije iz ^{1}H NMR:

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

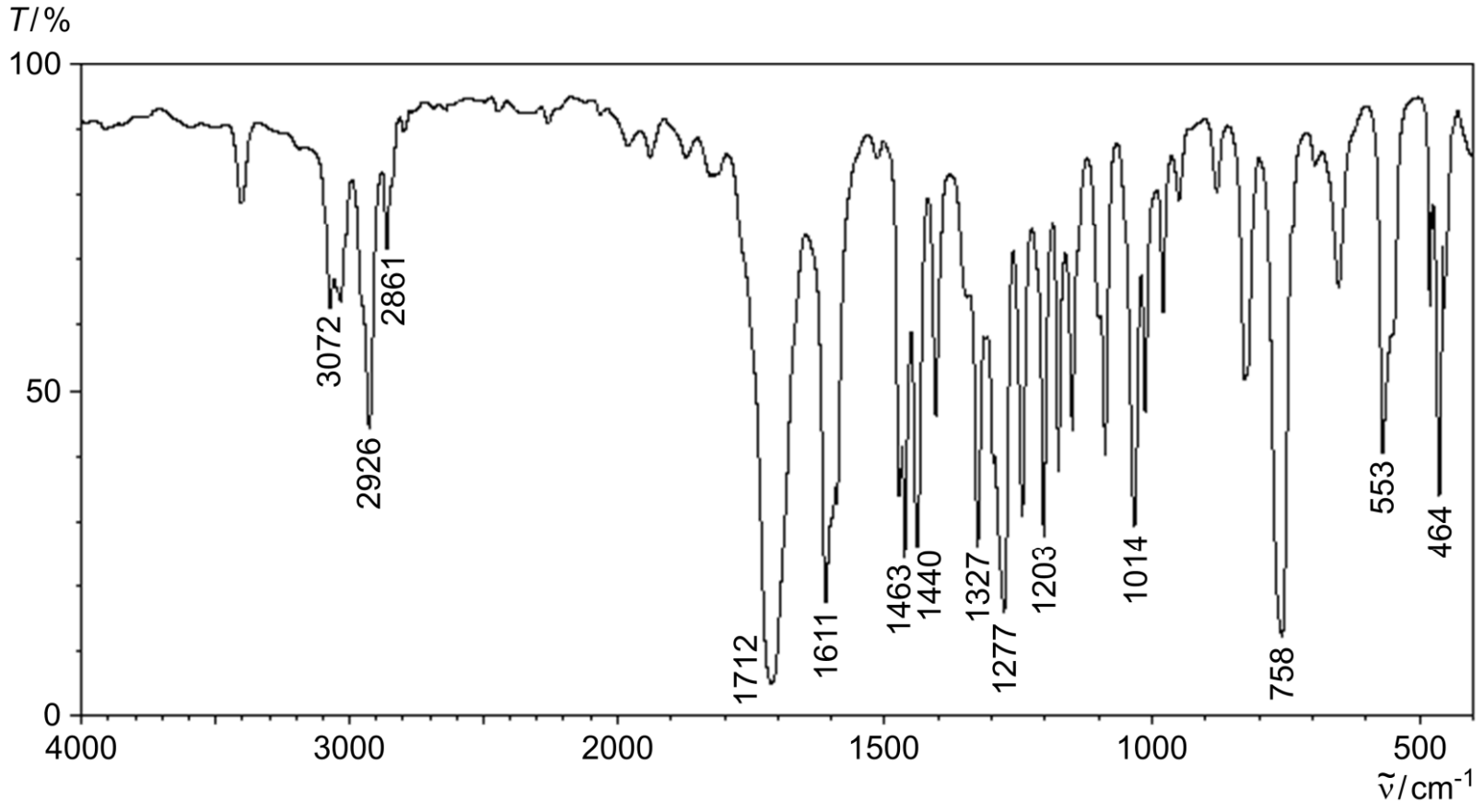
- informacije iz ^{13}C NMR:

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

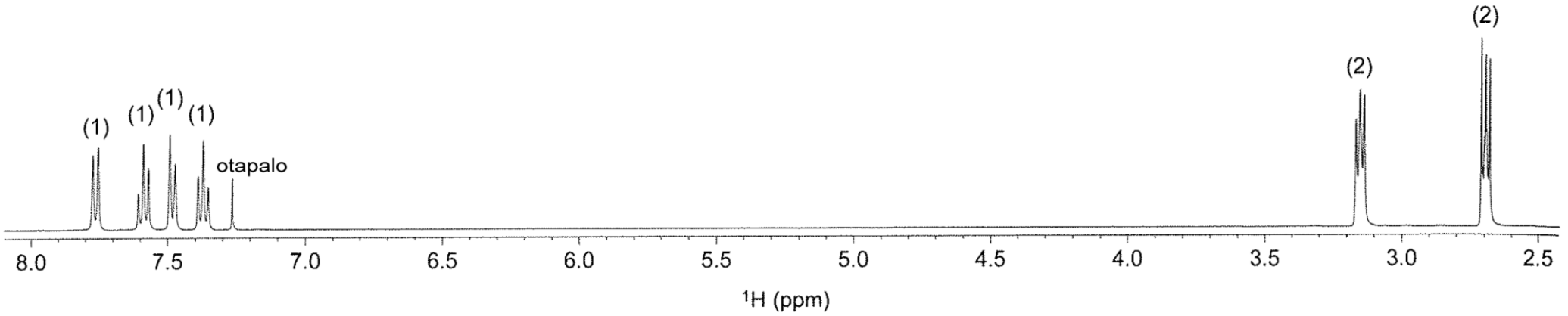
5. Odredite strukturu spoja na temelju njegovih MS, IR, ^{1}H NMR, ^{13}C NMR, COSY, HSQC i HMBC spektara.



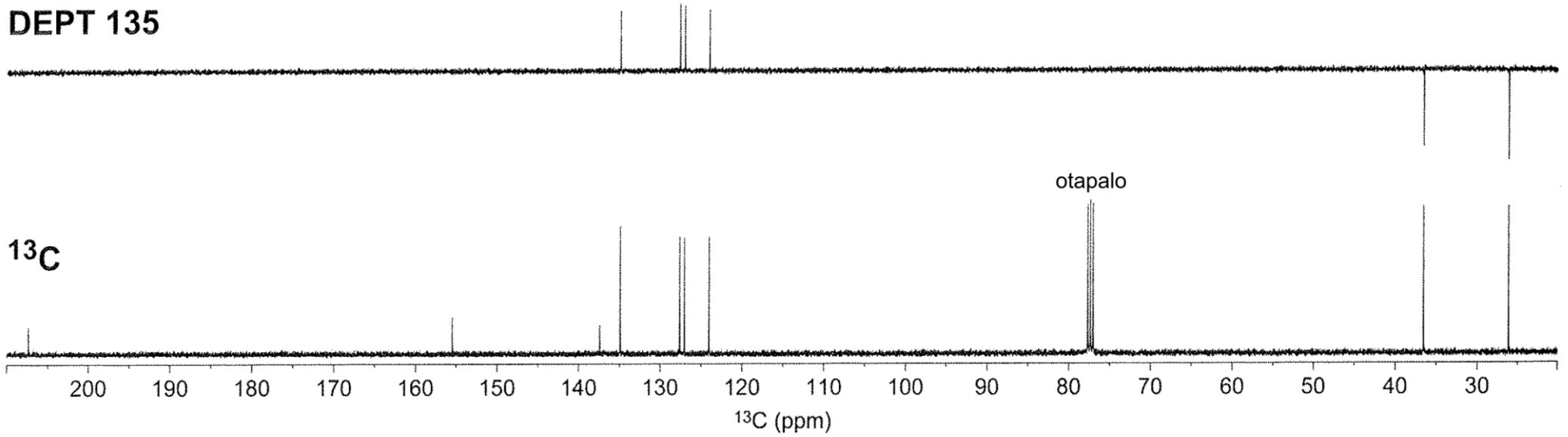
MS spektar spoja dobiven tehnikom ionizacije brzim elektronima (EI).



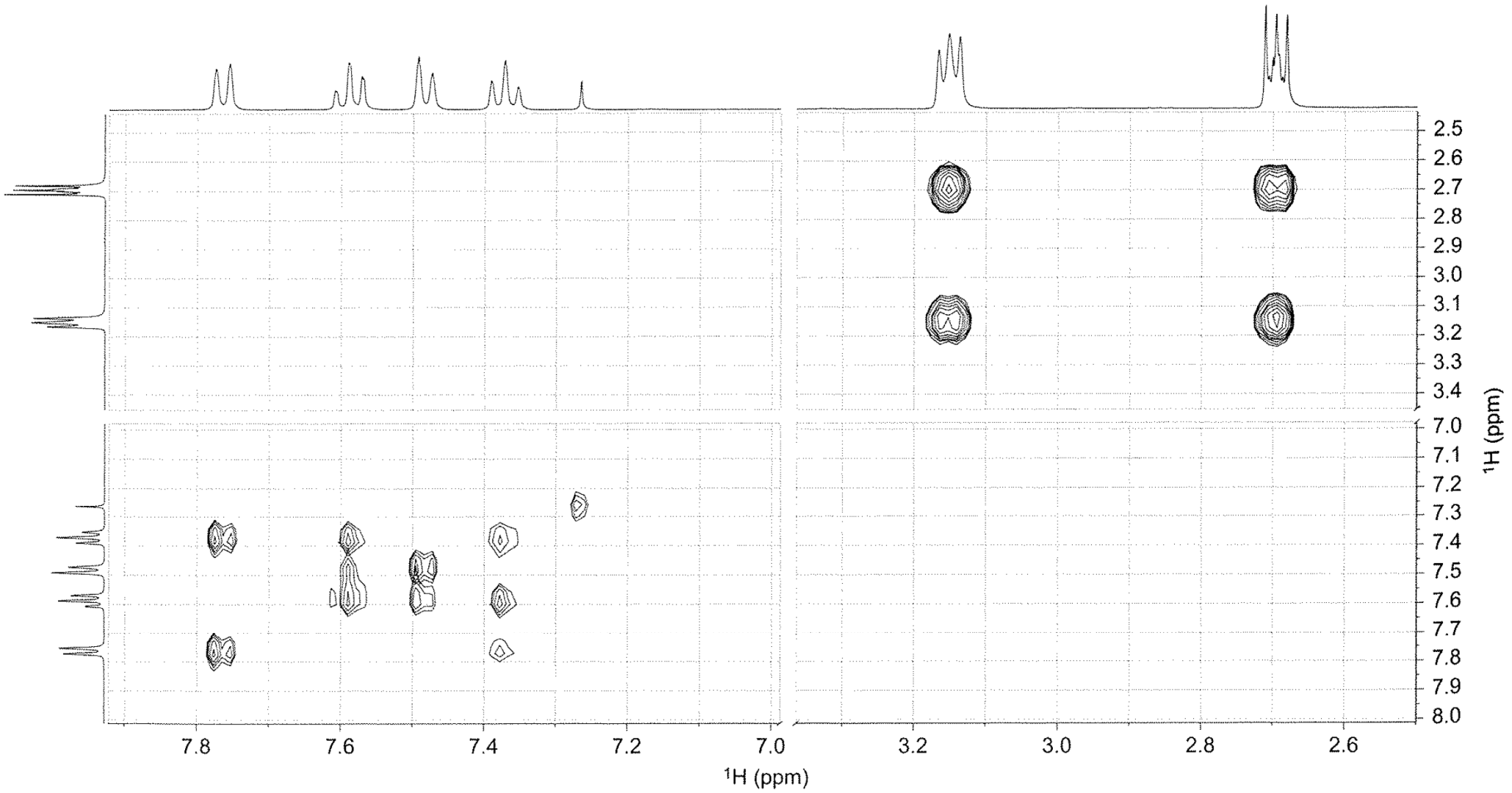
IR spektar spoja snimljen tehnikom KBr pastile.



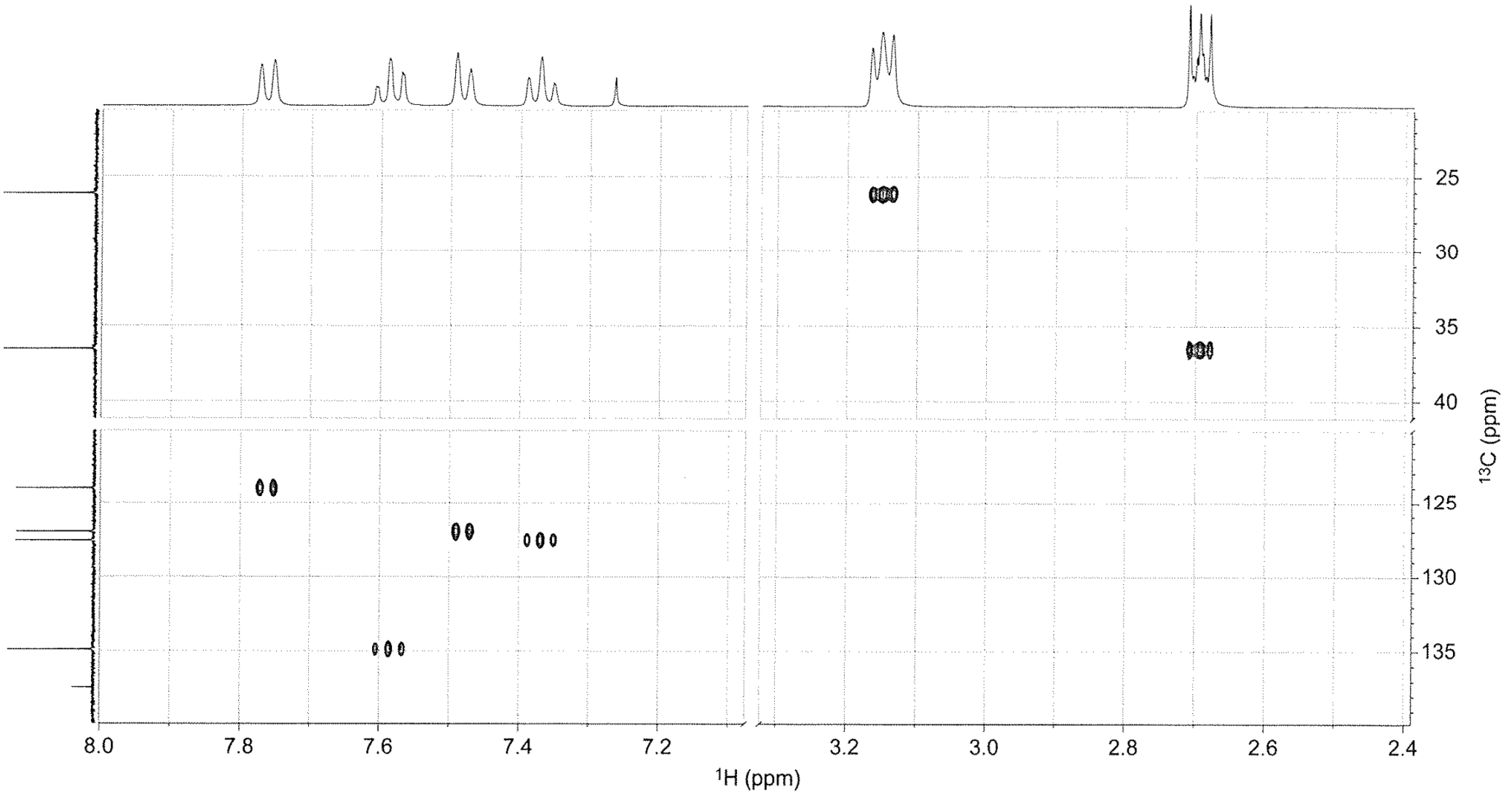
^{1}H NMR spektar spoja snimljen u $CDCl_3$.



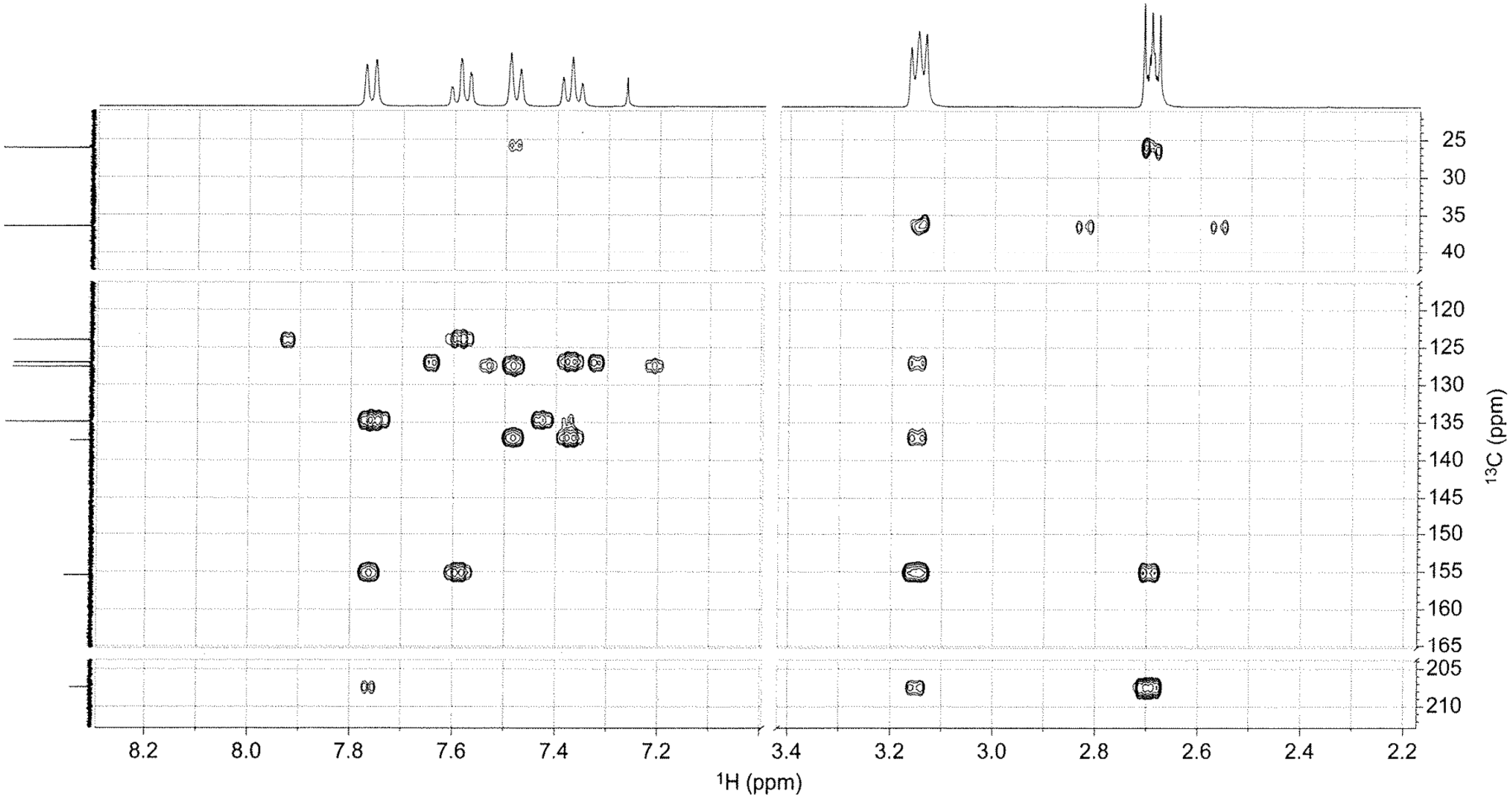
^{13}C NMR spektri spoja snimljeni u $CDCl_3$.



COSY spektar spoja snimljen u $CDCl_3$.

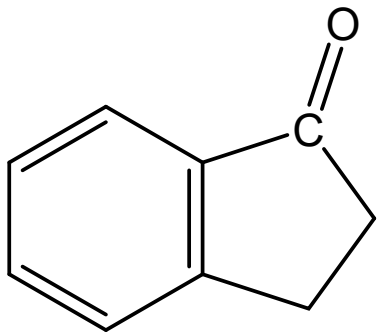


HSQC spektar spoja snimljen u $CDCl_3$.



HMBC spektar spoja snimljen u $CDCl_3$.

- informacije iz MS:
  bazni pik: 104
  $M^+ = 132$



- informacije iz IR:
  3072 cm^{-1} aromatsko C–H istezanje
  2926 i 2861 cm^{-1} alifatsko C–H istezanje
  1712 cm^{-1} C=O istezanje
  1611, 1463 i 1440 cm^{-1} aromatsko C=C istezanje
  758, 553 i 464 cm^{-1} aromatsko C–H svijanje

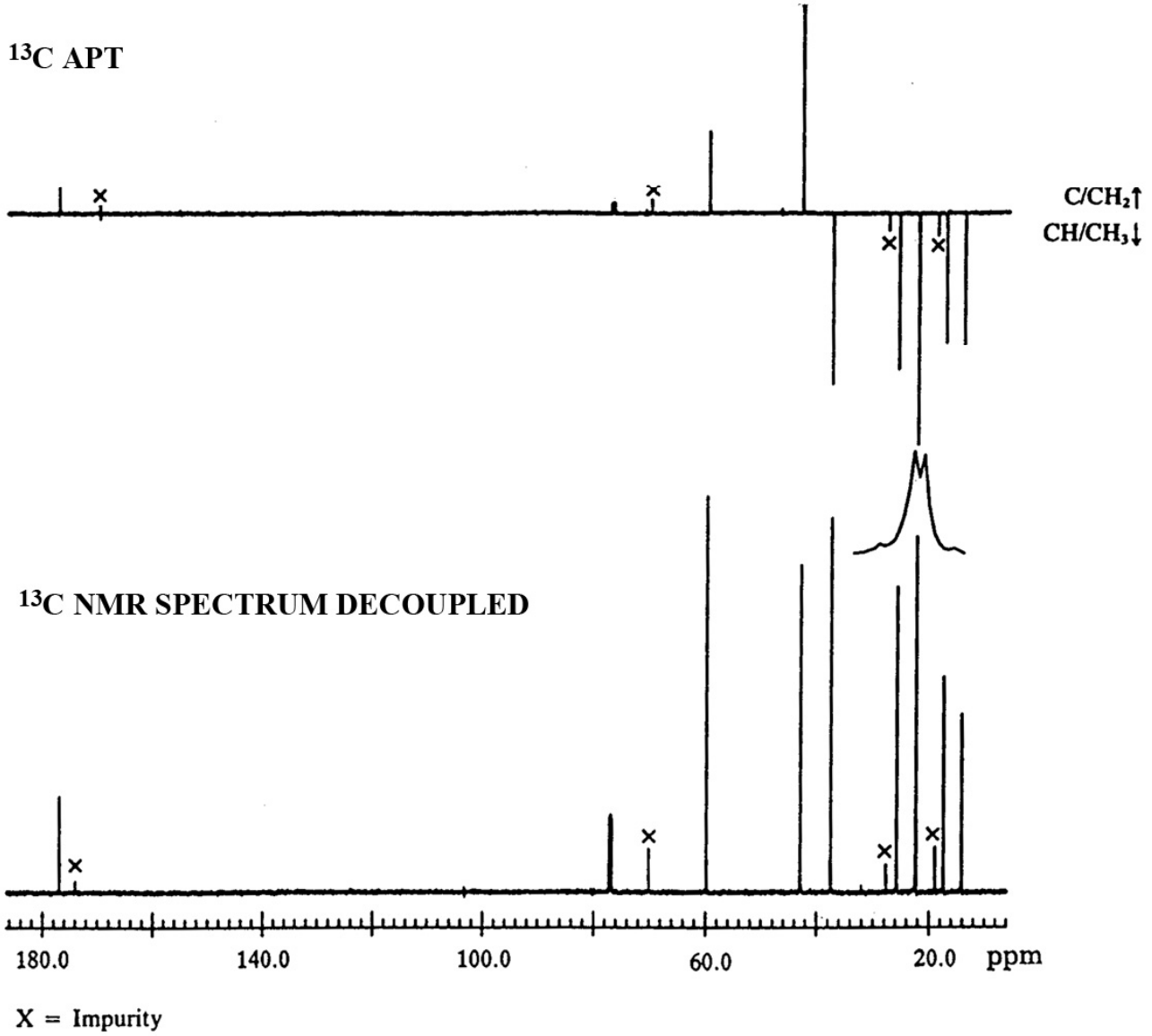
- informacije iz ^{1}H NMR:

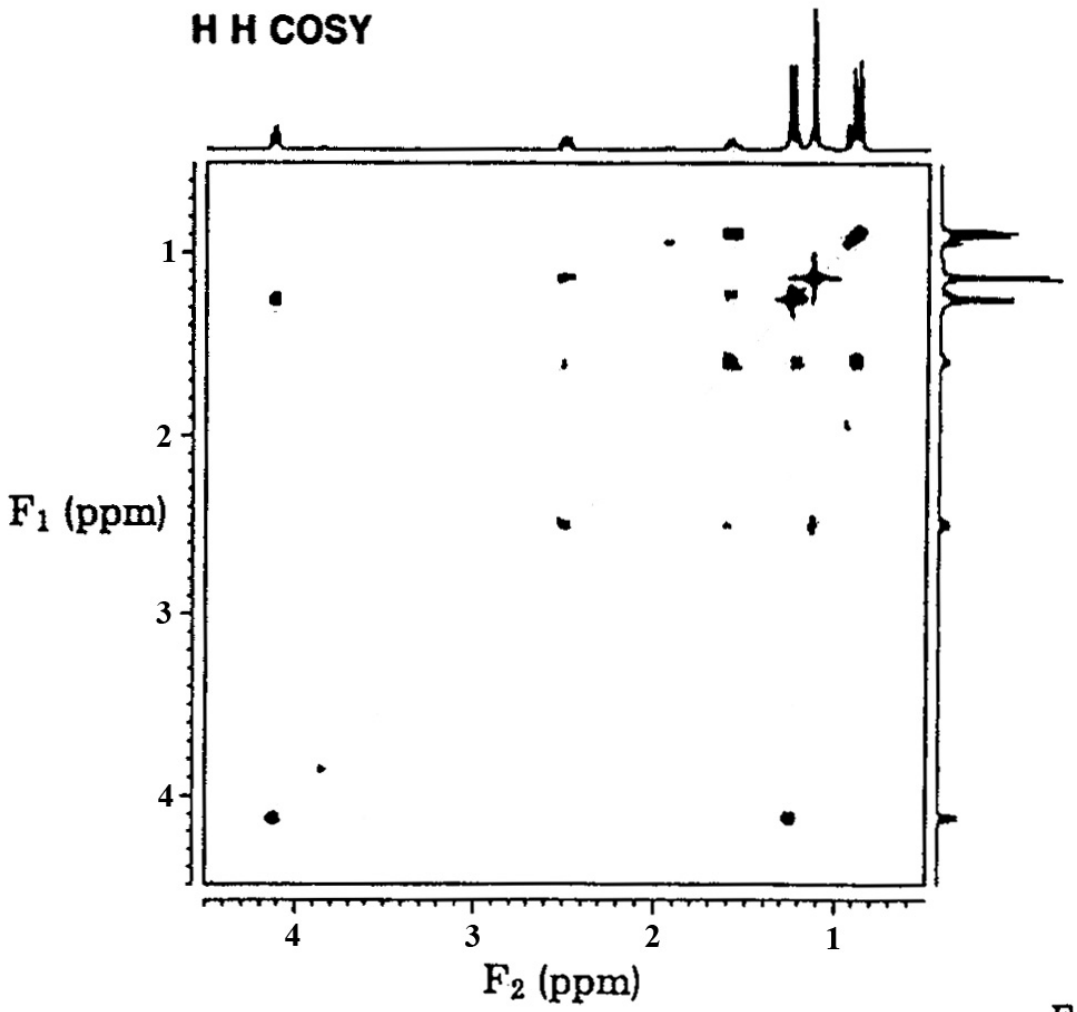
$\delta(^1H)$ / ppm	multiplet	H-atom
2,70	t	$–CH_2–$
3,15	t	$–CH_2–$
7,37	t	=CH–
7,48	d	=CH–
7,59	t	=CH–
7,77	d	=CH–

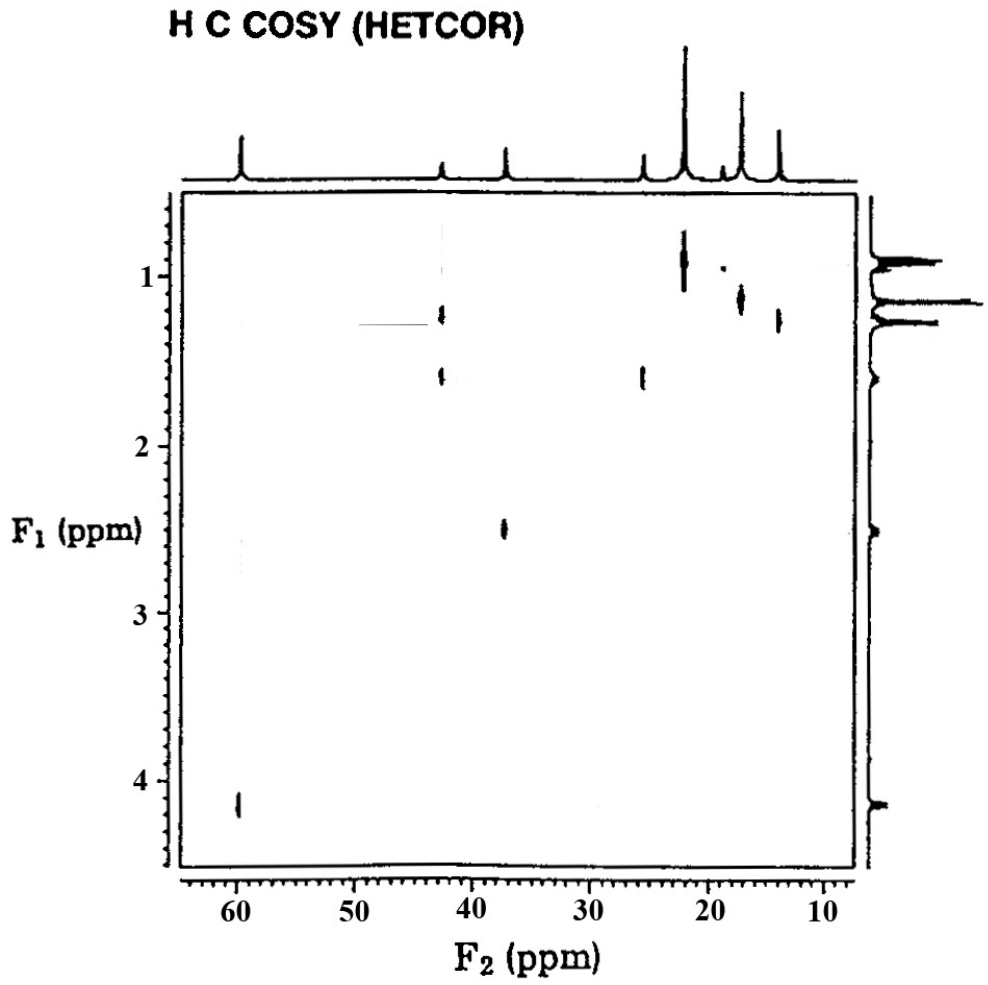
- informacije iz ^{13}C NMR:

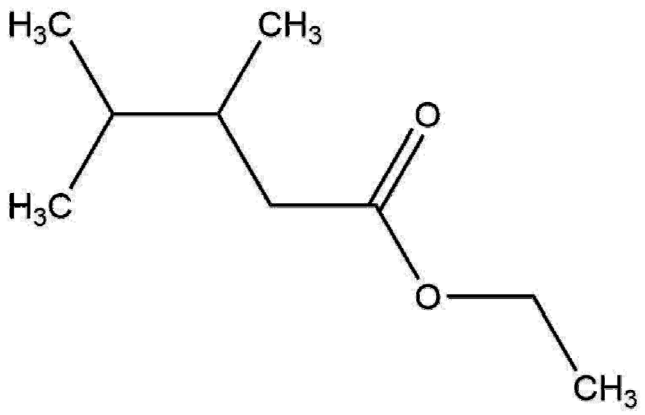
$\delta(^{13}C)$ / ppm	C-atom
26	$–CH_2–$
36	$–CH_2–$
124	=CH–
127	=CH–
128	=CH–
135	=CH–
137	C
156	C
207	C=O

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




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


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



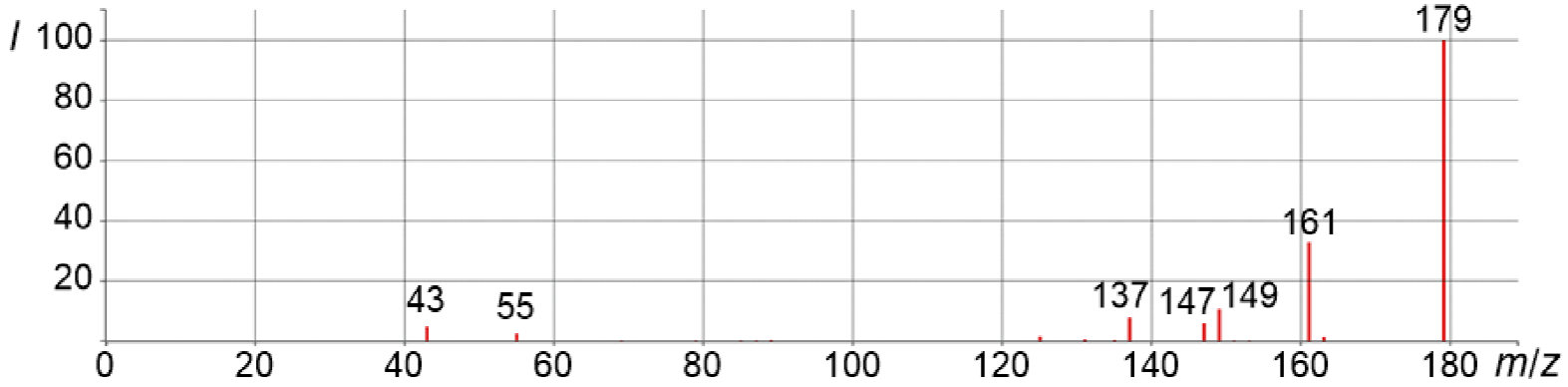
- informacije iz ^{1}H NMR:

$\delta(^{1}H)$ / 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	$-CH_2-$

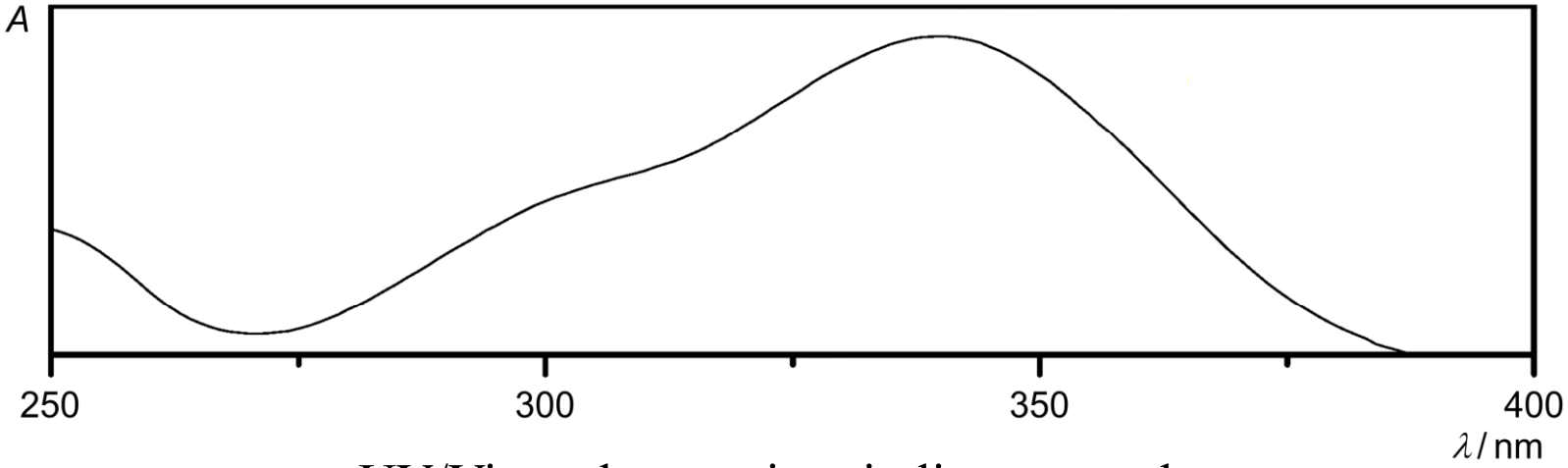
- informacije iz ^{13}C NMR:

$\delta(^{13}C)$ / 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

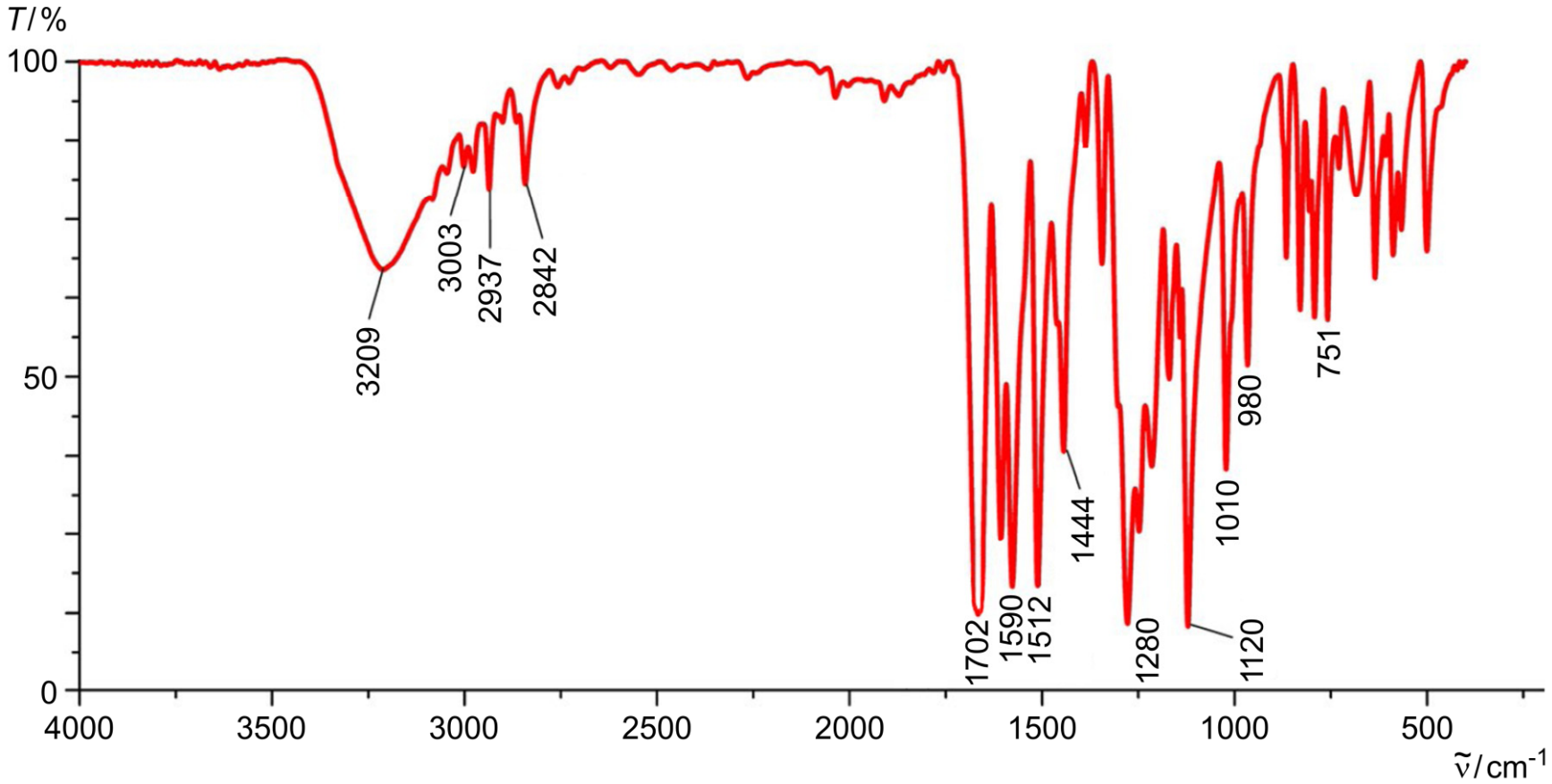
7. Identificirajte spoj na temelju njegovih MS, UV/Vis, IR, ^{1}H NMR, ^{13}C NMR, COSY, HMQC i HMBC spektara.



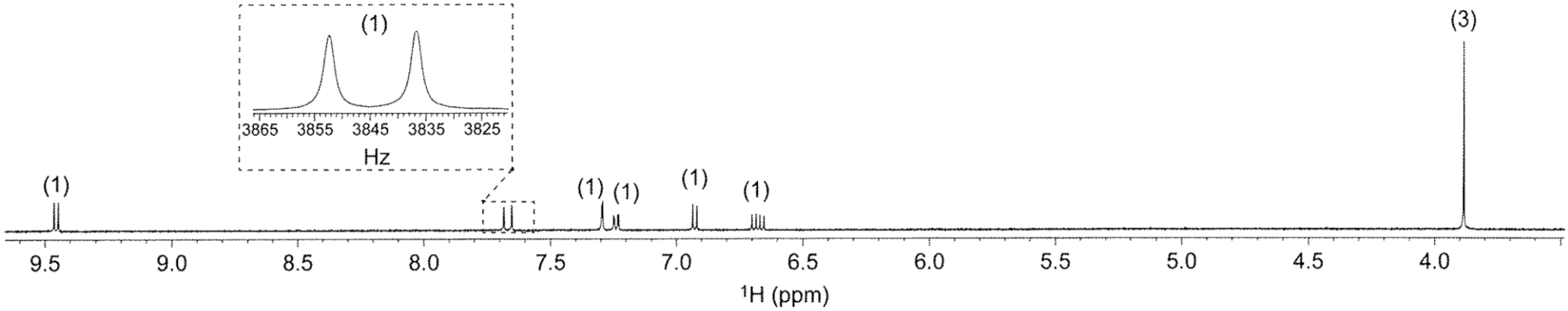
MS spektar spoja dobiven kemijskom ionizacijom u pozitivnom modu.



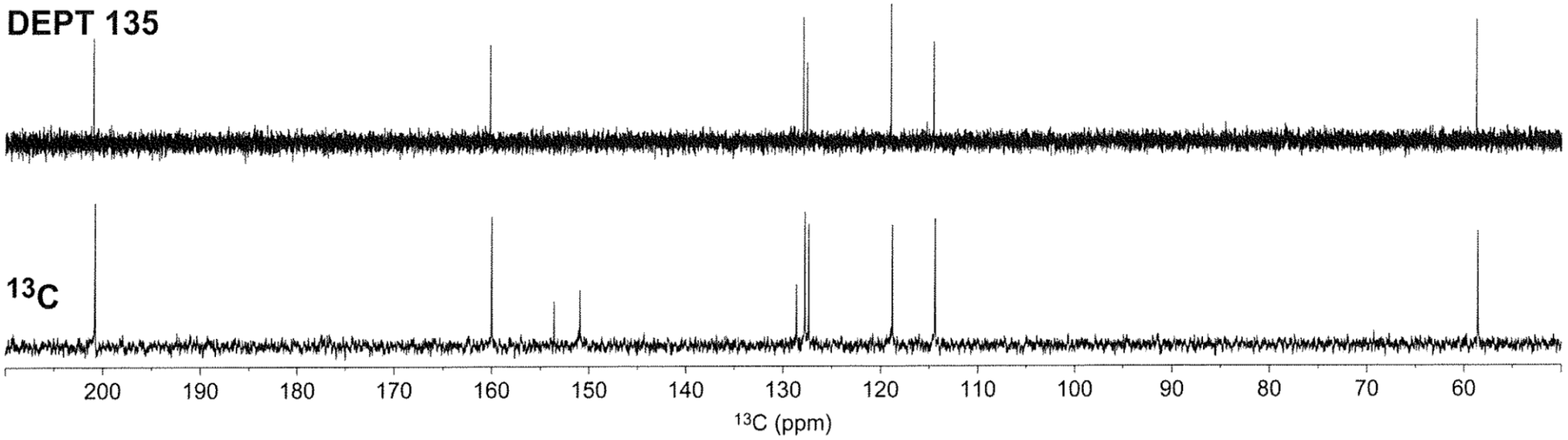
UV/Vis spektar spoja snimljen u etanolu.



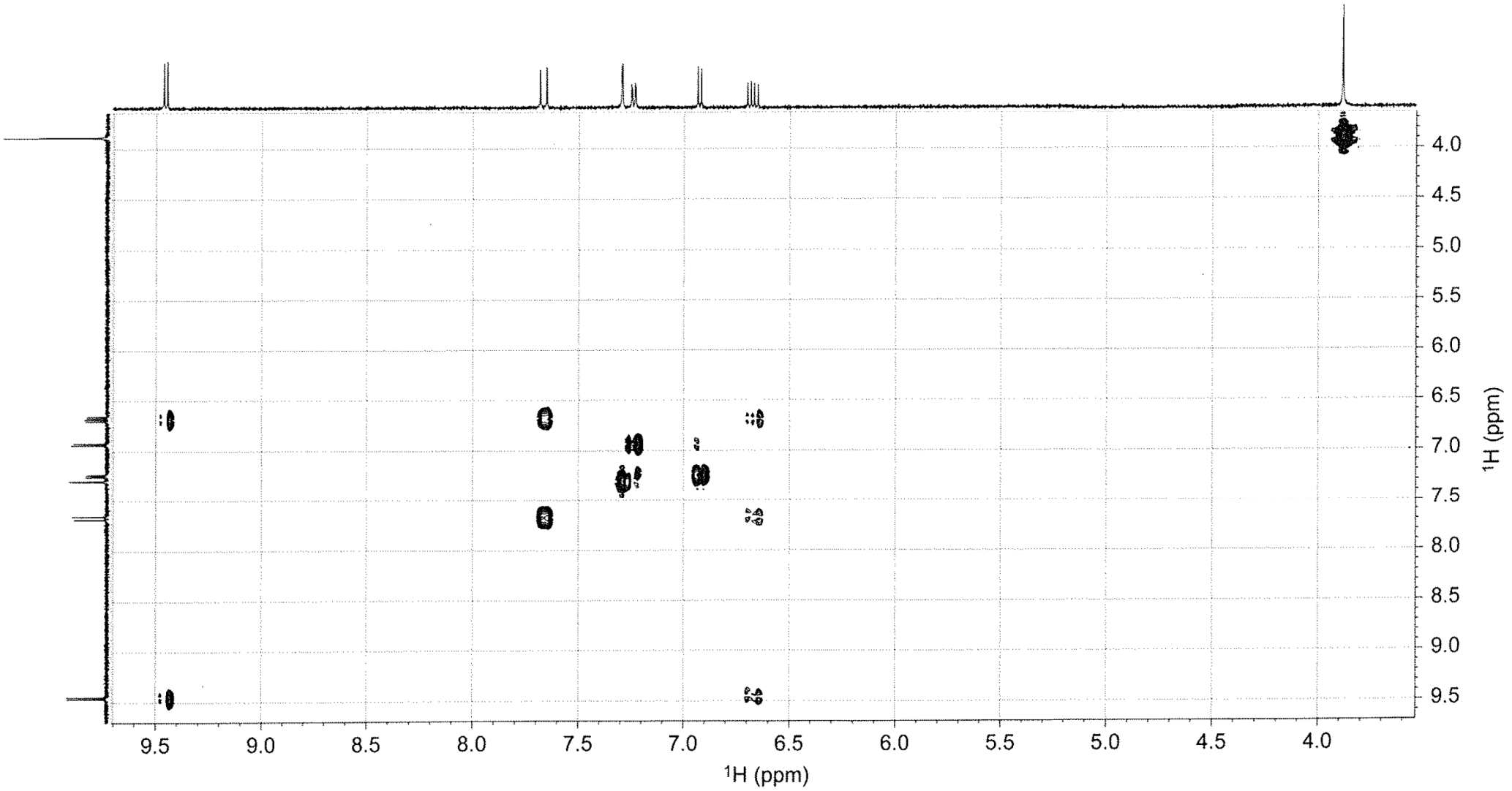
IR spektar spoja snimljen tehnikom KBr pastile.



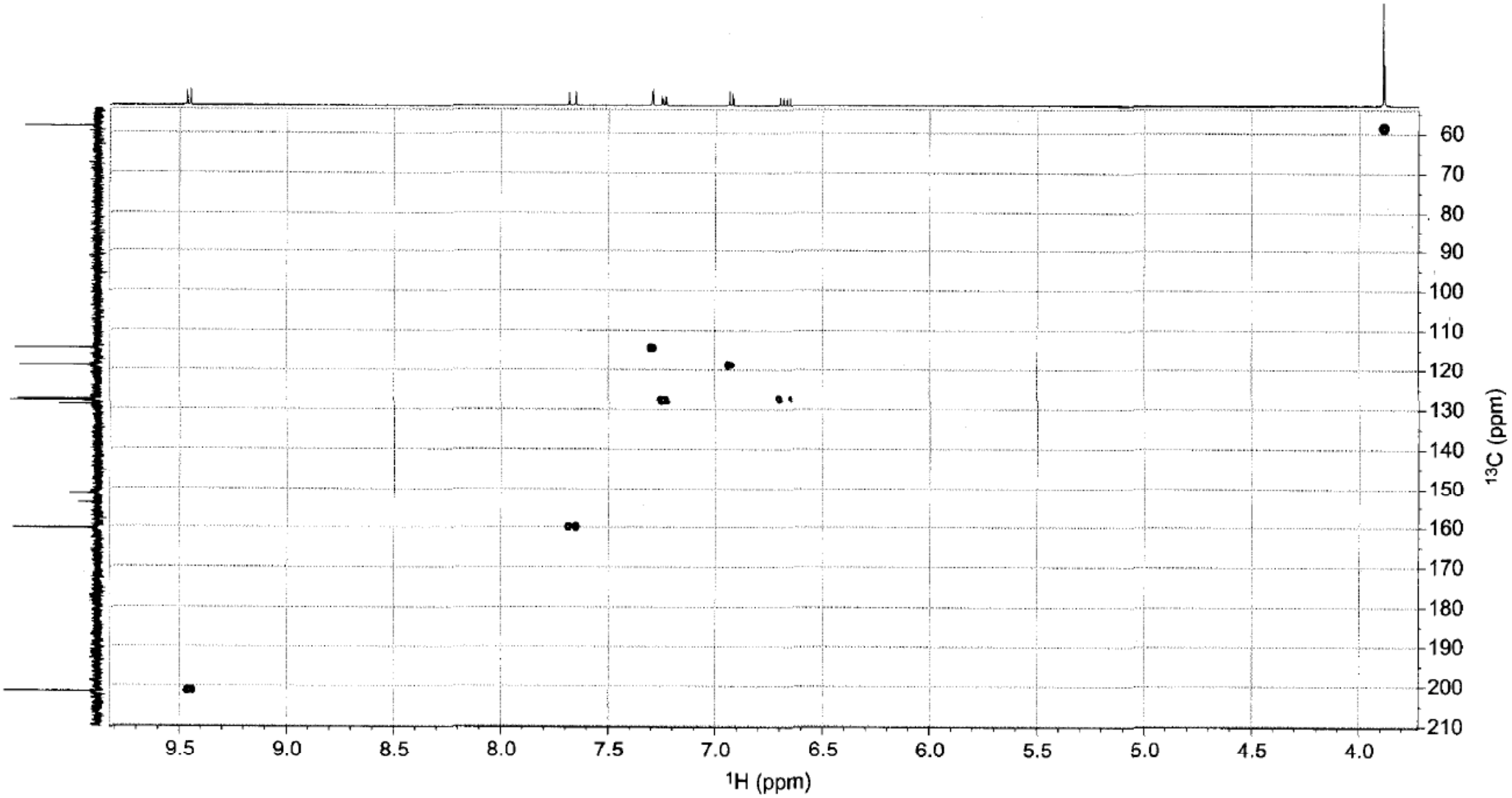
^{1}H NMR spektar spoja snimljen u D_2O. Spoj sadrži i protone koji se izmijene u D_2O.



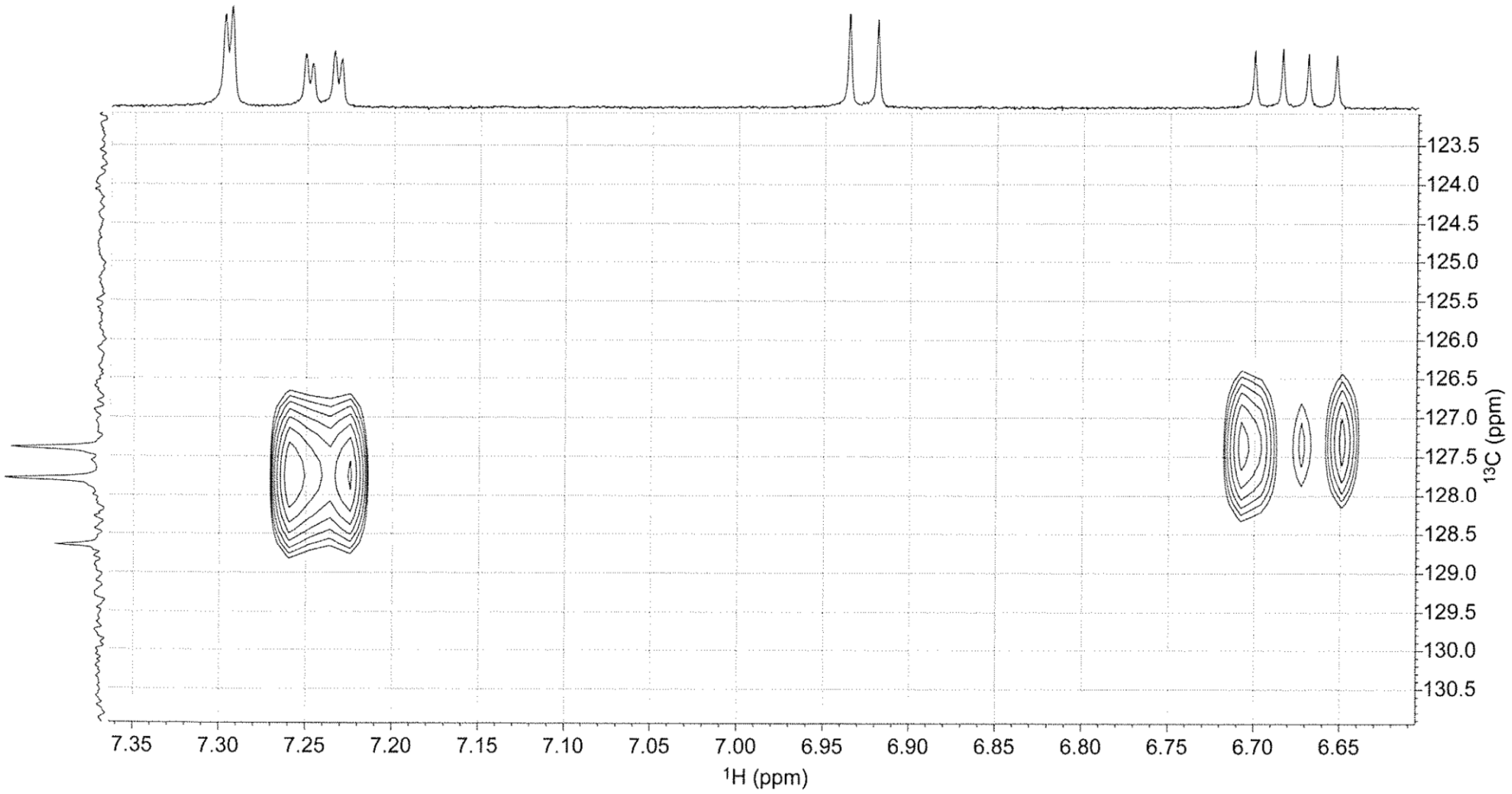
^{13}C NMR spektri spoja snimljeni u D_2O.



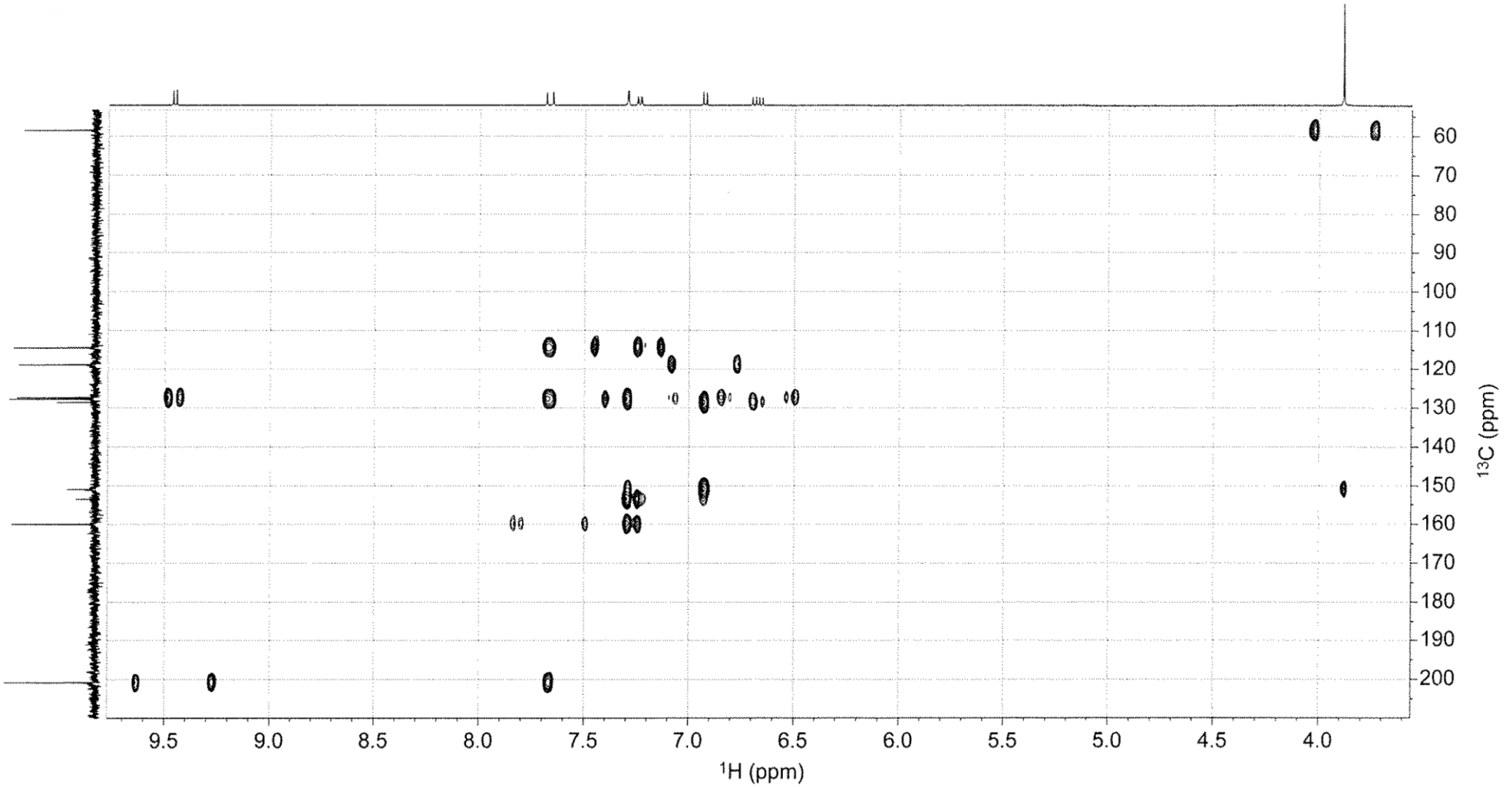
COSY spektar spoja snimljen u D_2O.



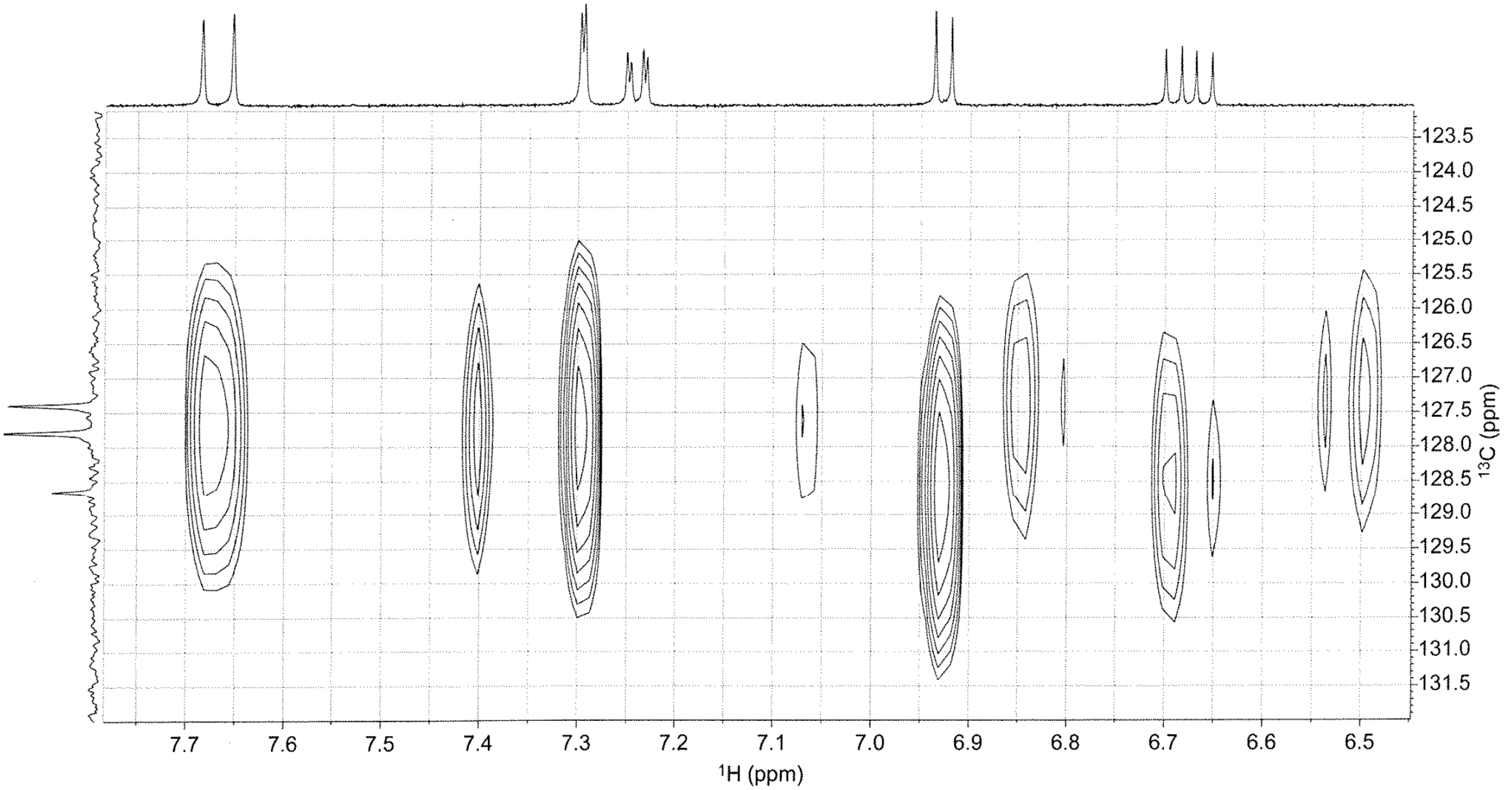
HSQC spektar spoja snimljen u D_2O.



Uvećani dio HSQC spektra spoja snimljenog u D_2O.

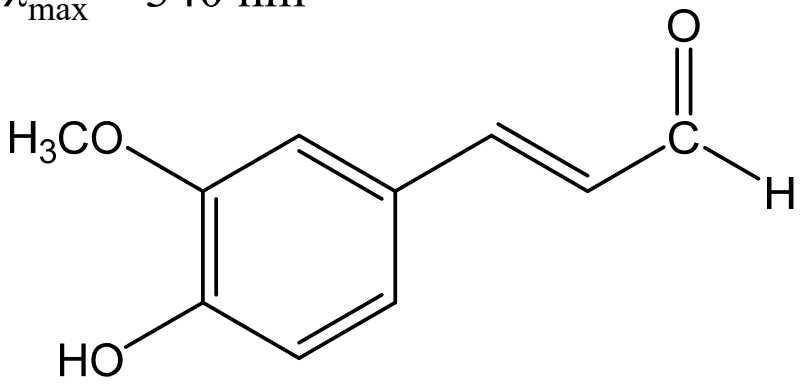


HMBC spektar spoja snimljen u D_2O.



Uvećani dio HMBC spektra spoja snimljenog u D_2O.

- informacije iz MS:
  $(M+H)^+ = 179$
- informacije iz UV/Vis:
  $\lambda_{max} = 340$ nm



- informacije iz IR:
  3209 cm^{-1} OH istezanje
  3003, 2937 i 2842 cm^{-1} C–H istezanje
  1702 cm^{-1} C=O istezanje
  1590, 1512 i 1444 cm^{-1} C=C istezanje
  980 i 751 cm^{-1} aromatsko C–H svijanje

- informacije iz ^{1}H NMR:

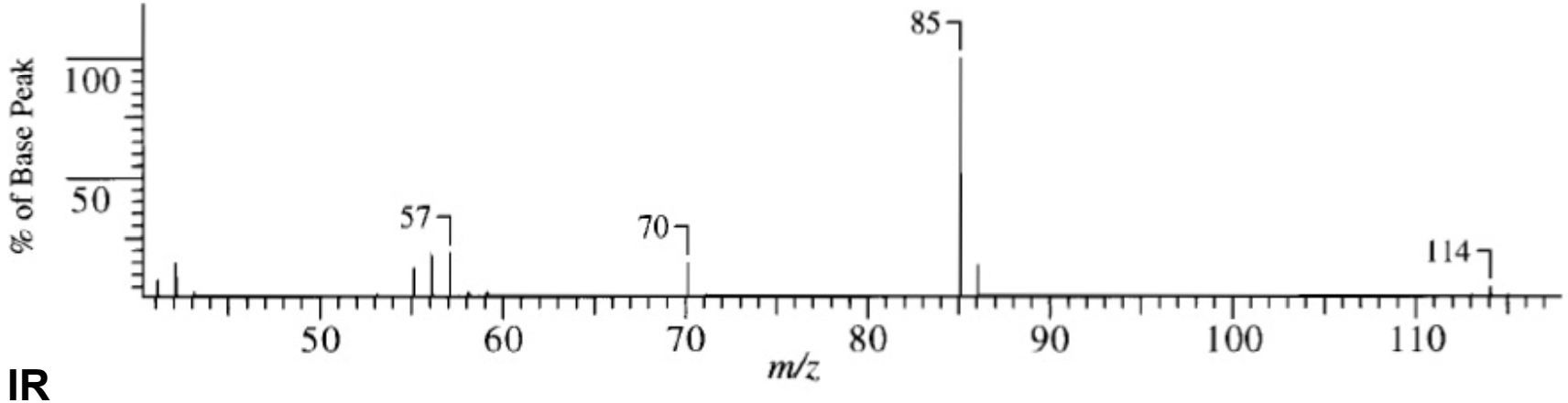
$\delta(^1H)$ / ppm	multiplet	H-atom
3,89	s	$-CH_3$
6,67	dd	=CH–
6,92	d	=CH–
7,24	dd	=CH–
7,30	d	=CH–
7,69	d	=CH–
9,45	d	HC=O

- informacije iz ^{13}C NMR:

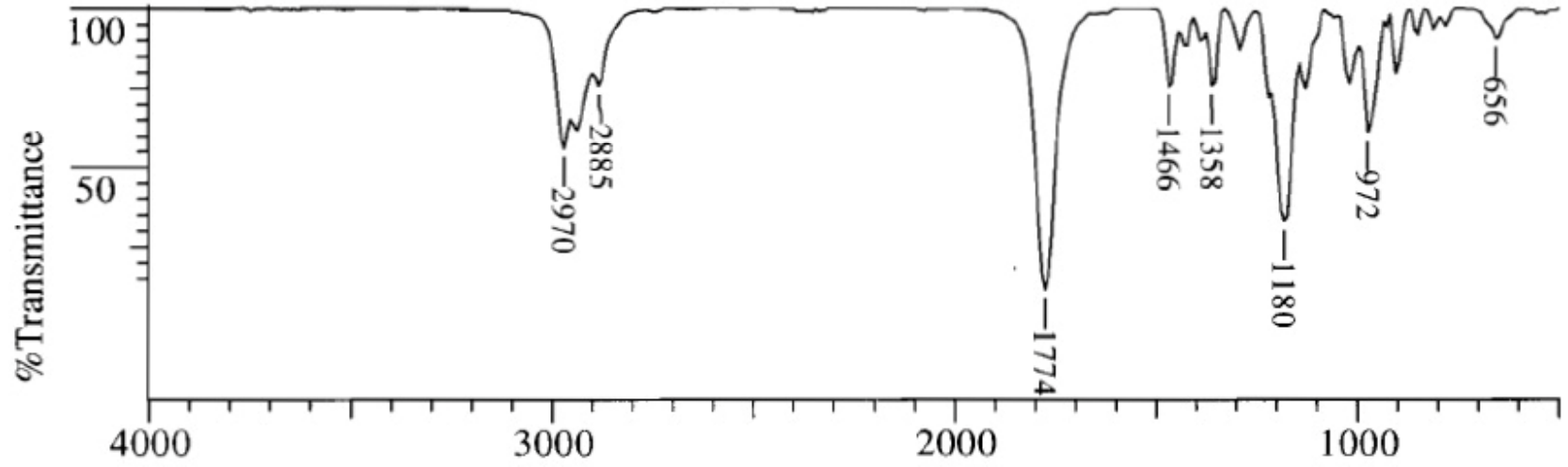
$\delta(^{13}C)$ / ppm	C-atom
58	$-CH_3$
114	=CH–
118	=CH–
127	=CH–
127,5	=CH–
128	C
151	C
153,5	C
160	=CH–
201	C=O

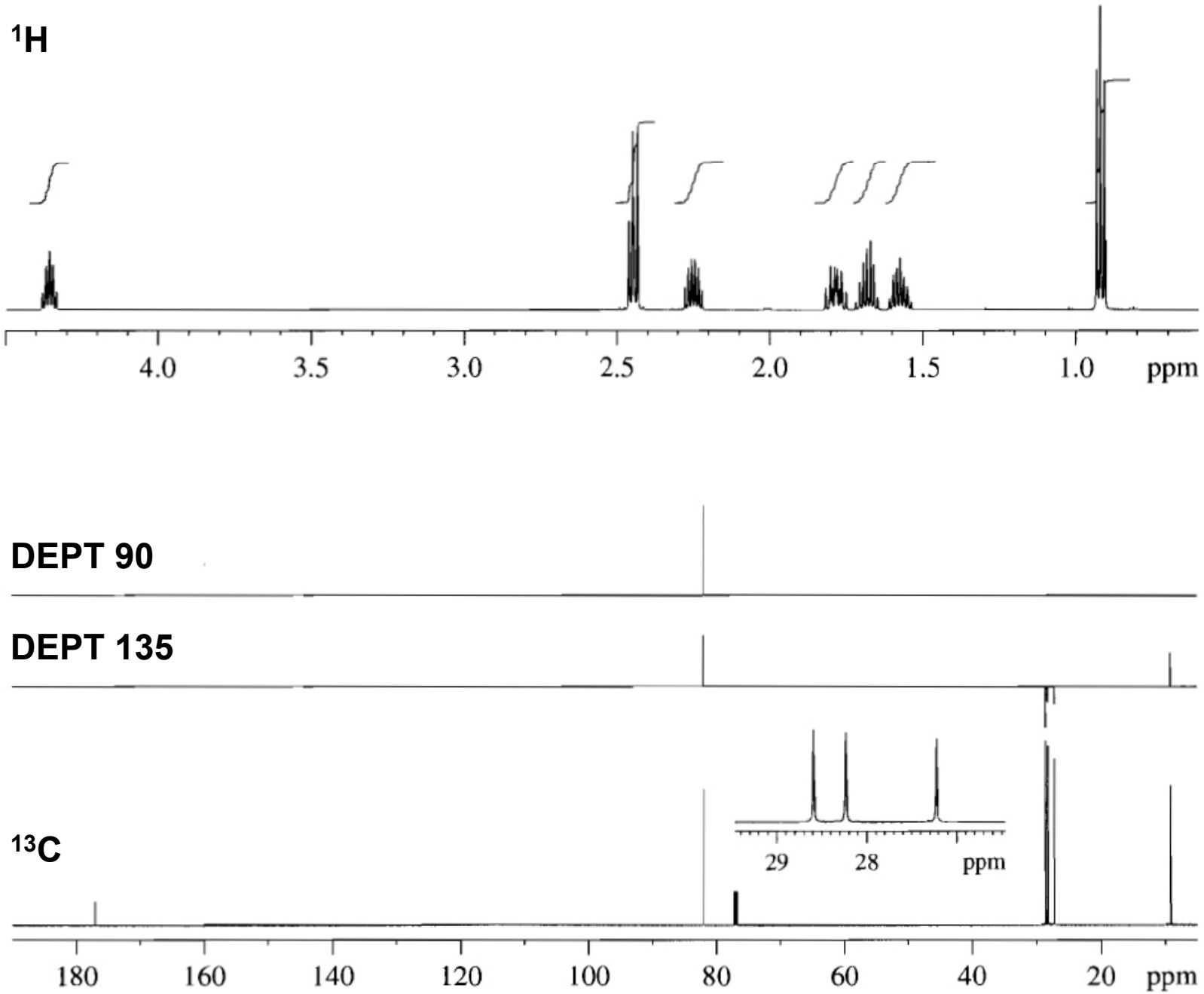
8. Identificirajte spoj na temelju njegovih MS, IR, ^{1}H NMR, ^{13}C NMR, COSY i HMQC spektara.

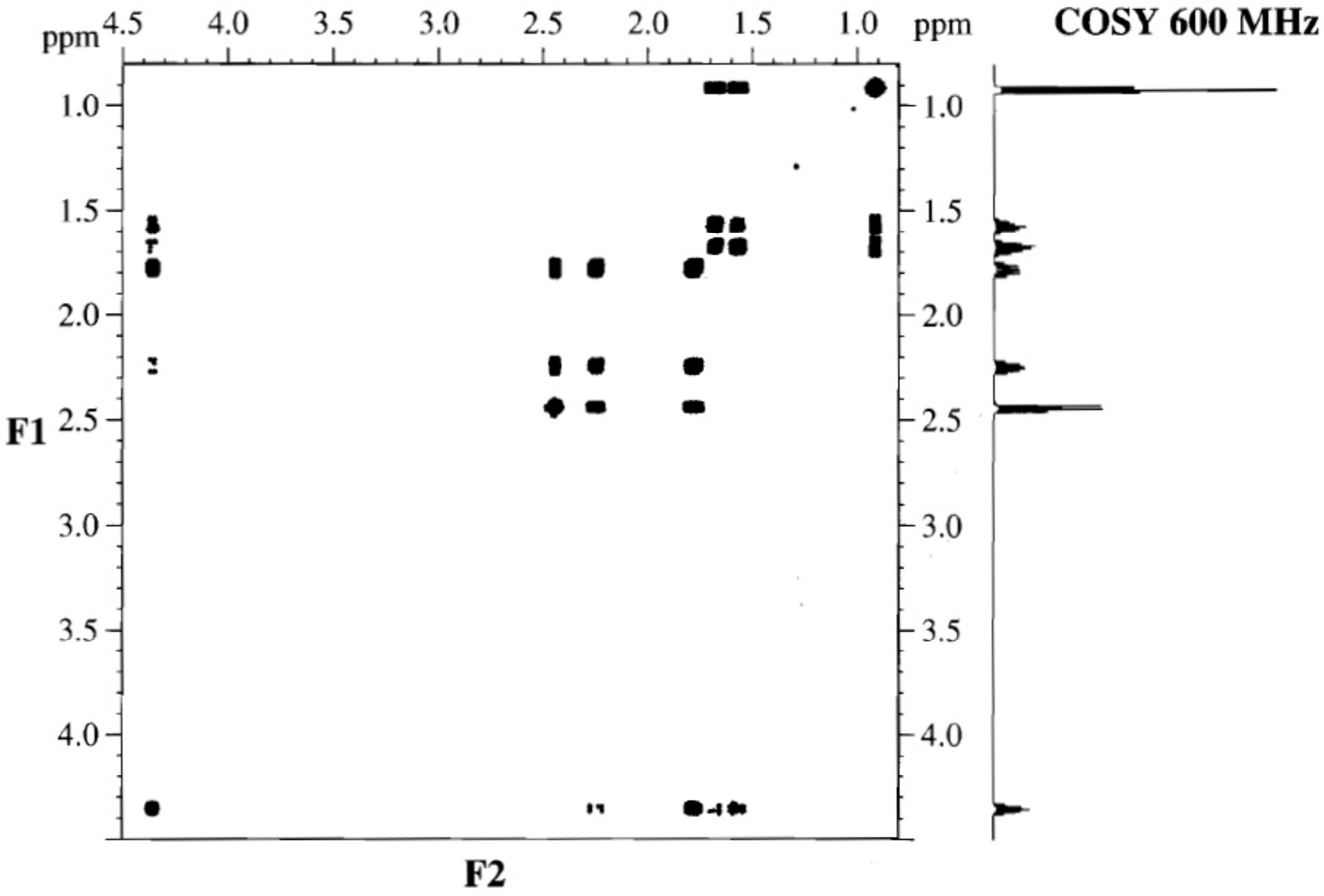
MS

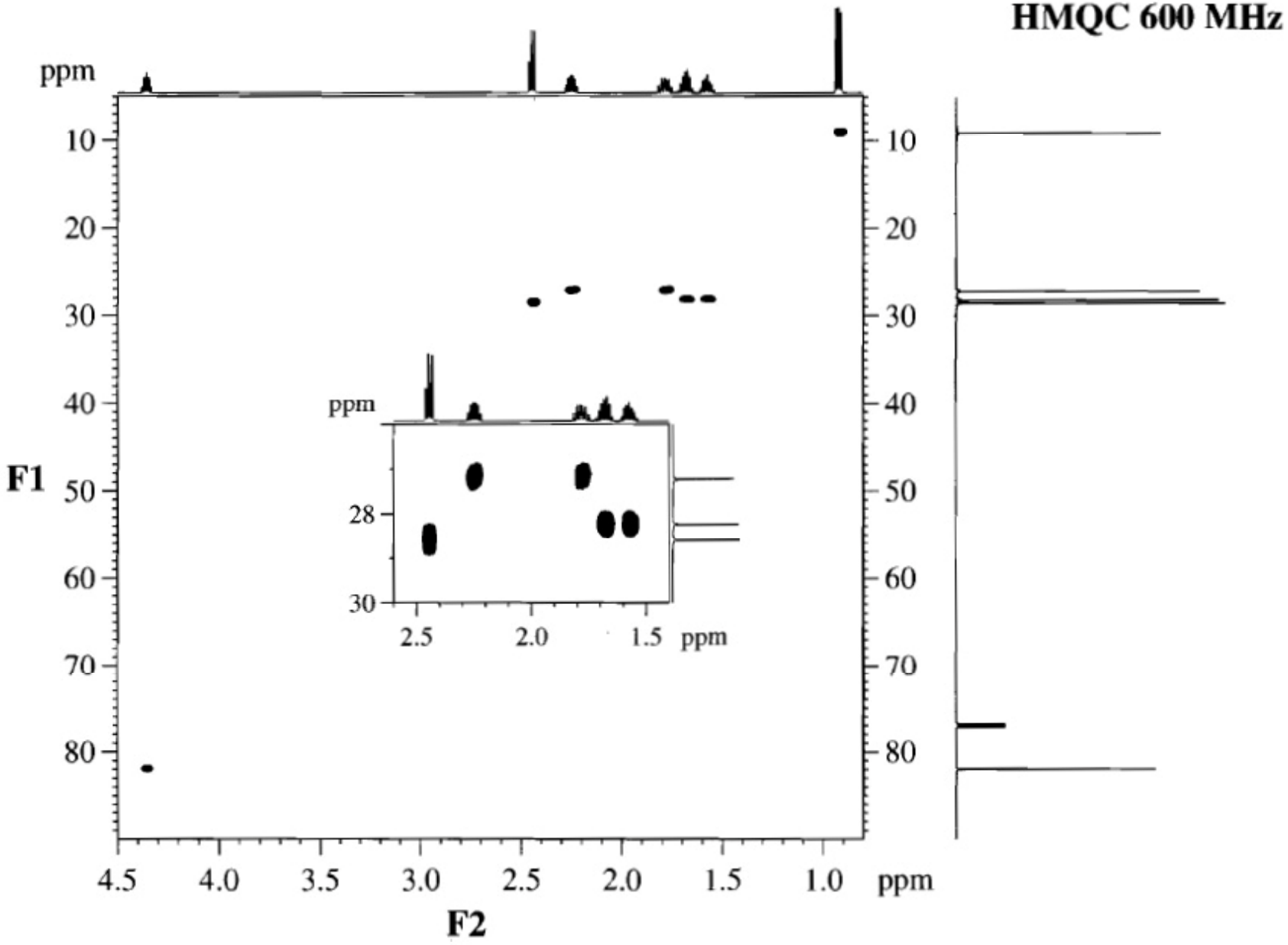


IR

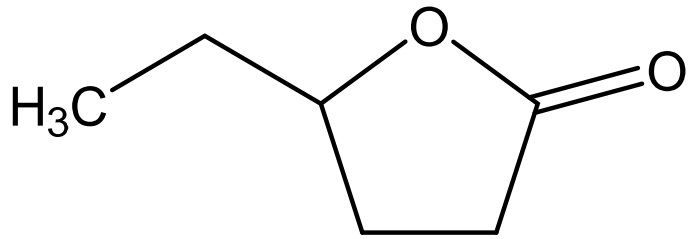



1H
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
DEPT 90
DEPT 135
13C
29
28
ppm
180
160
140
120
100
80
60
40
20
ppm


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 MS:
  bazni pik: 85
  $M^+ = 114$



- 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 ^{1}H 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