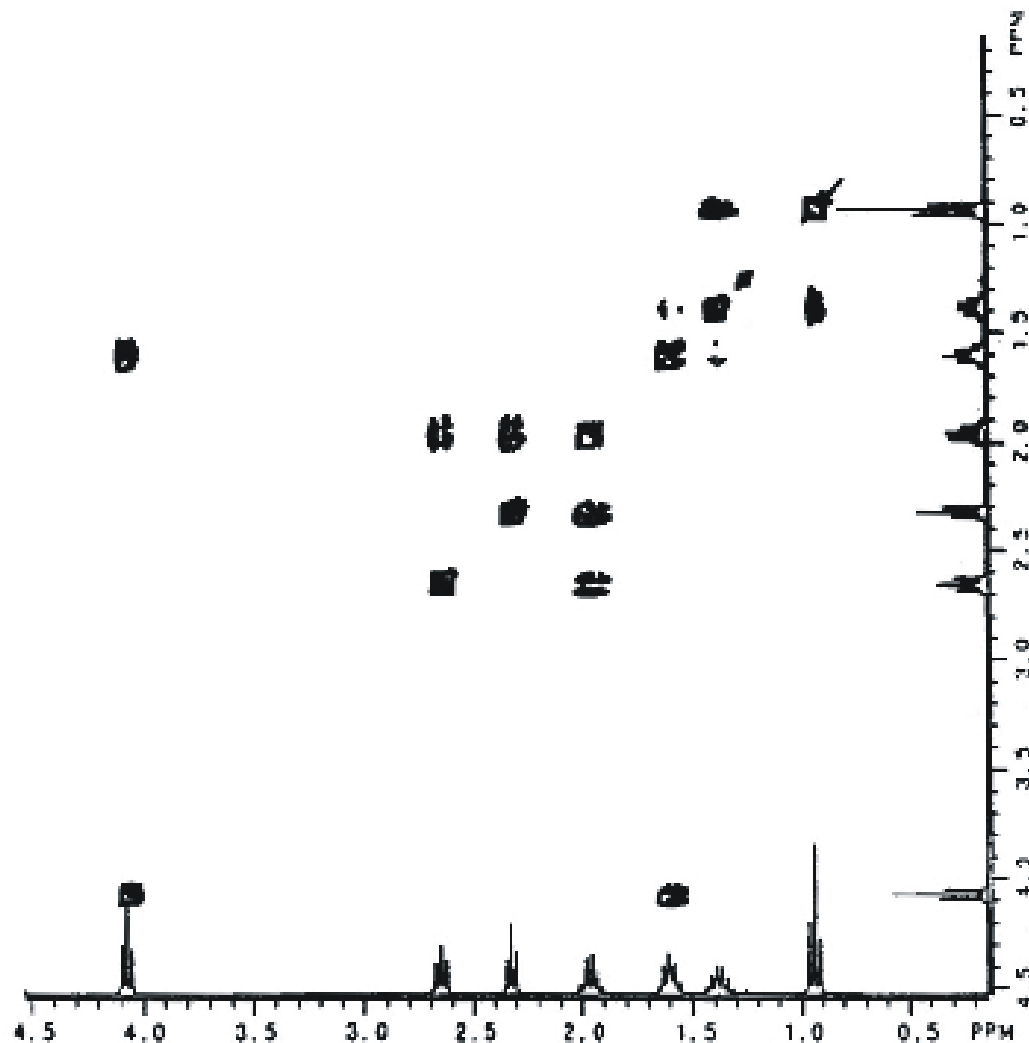


Spektroskopska strukturna analiza

Dodatni zadaci

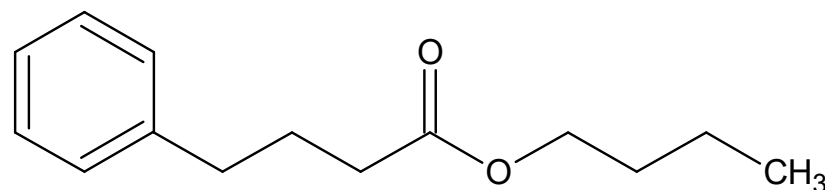
1. Odredite strukturu spoja molekulske formule $C_{14}H_{20}O_2$ na temelju njegovog COSY spektra. 1H NMR spektar sadrži još široki singlet s integralom 5.



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

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

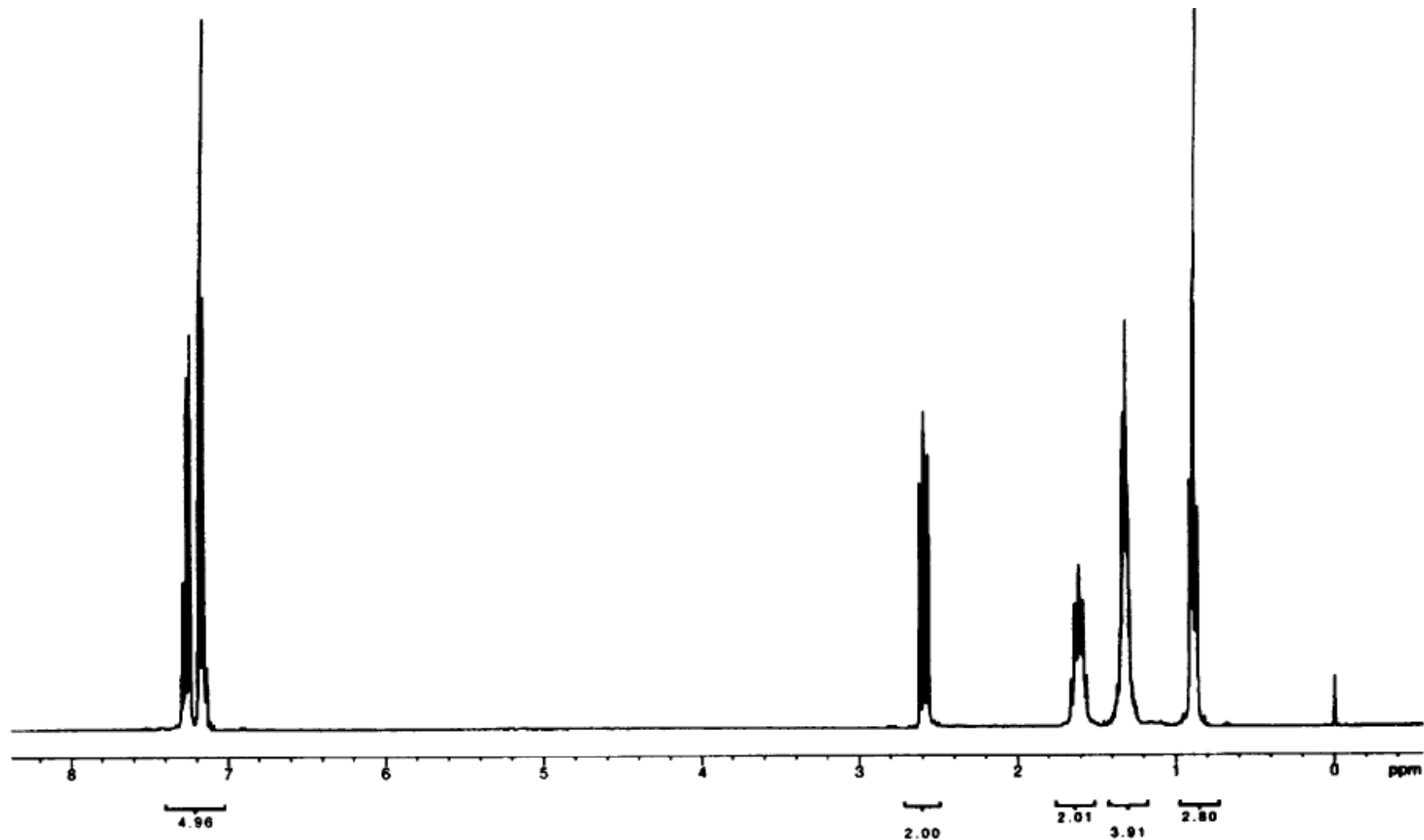
- za ovaj spoj: $IHD = [2 \cdot 14 + 2 - 20] / 2 = 5$



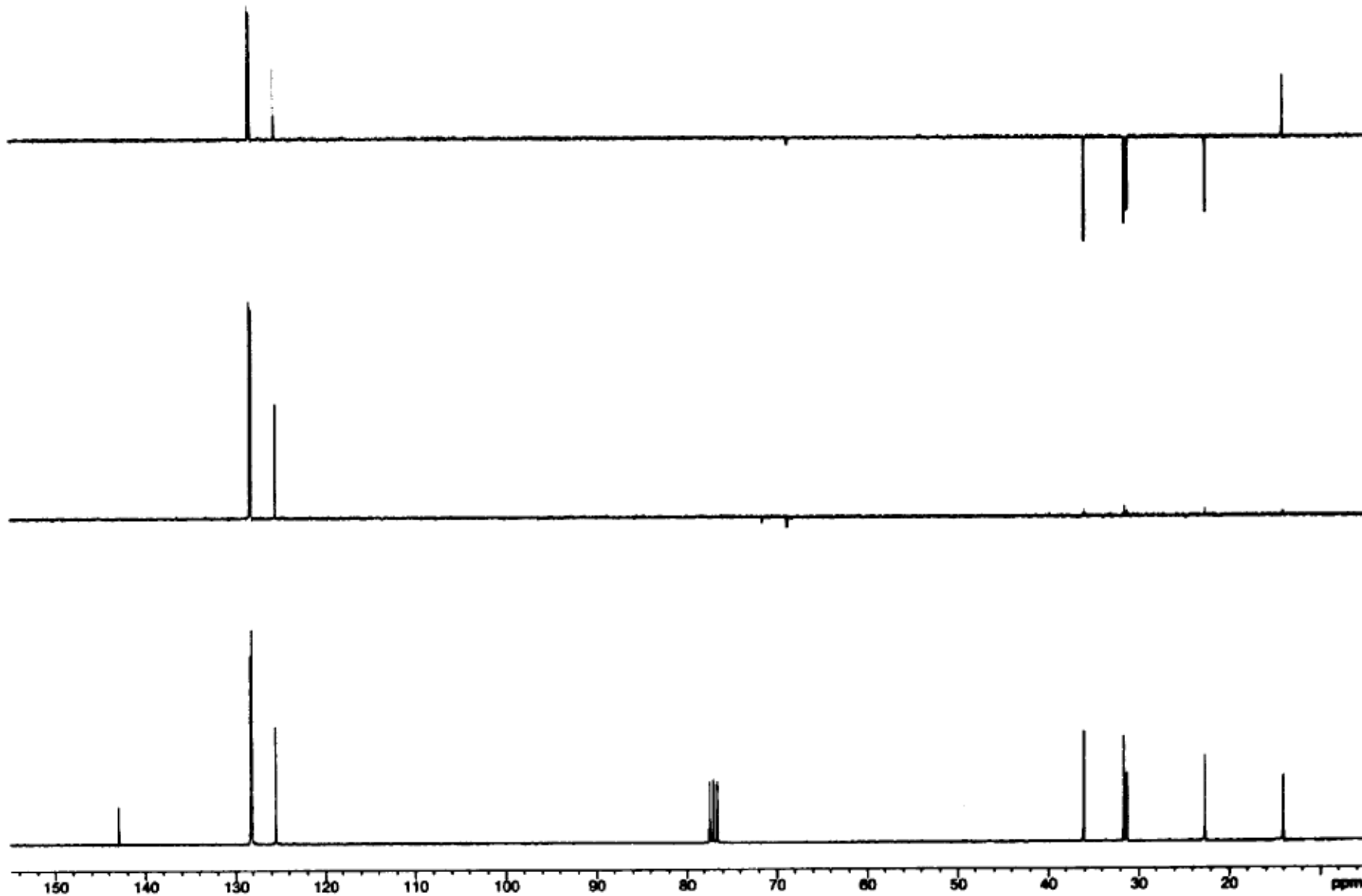
$\delta(^1\text{H})/\text{ppm}$	multiplet	H-atom
0,95	t	-CH ₃
1,40	m(heptet)	-CH ₂ -
1,55	m(kvintet)	-CH ₂ -
1,95	m(kvintet)	-CH ₂ -
2,35	t	-CH ₂ -
2,65	t	-CH ₂ -
4,10	t	-CH ₂ -
7,30	s	>CH

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

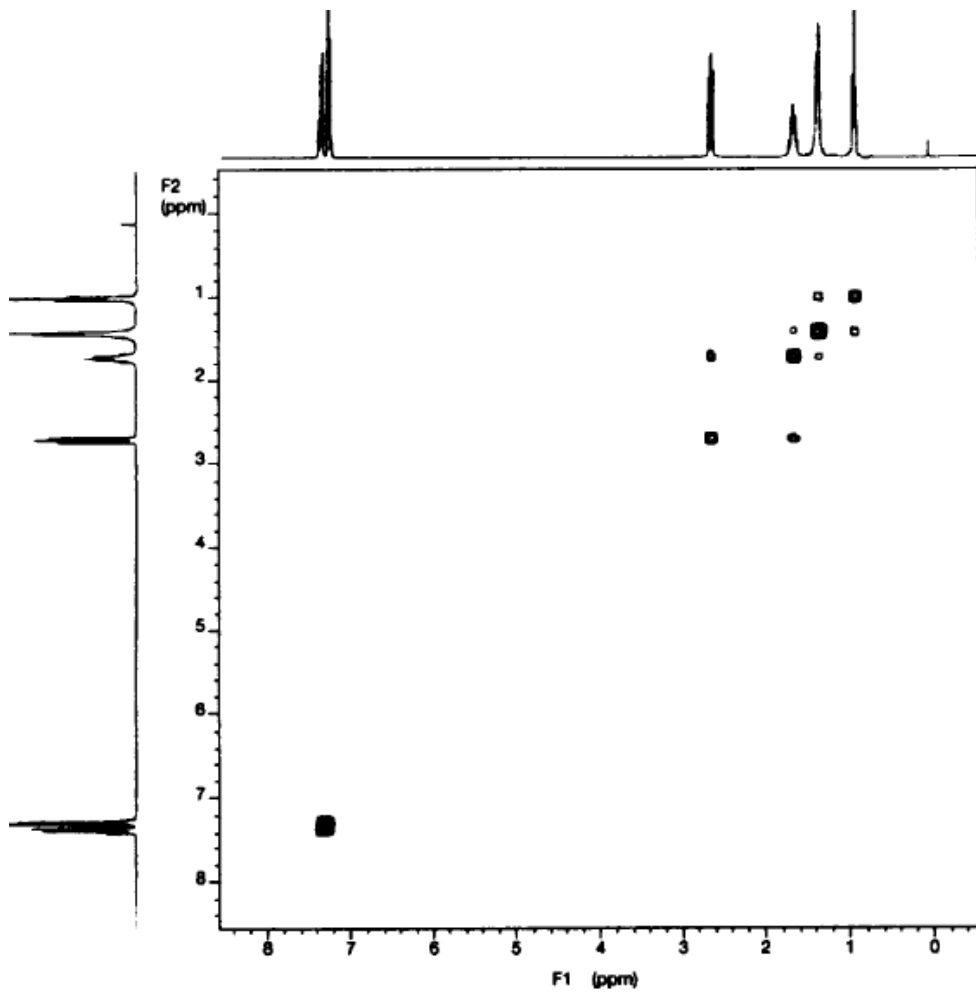
1H NMR spektar



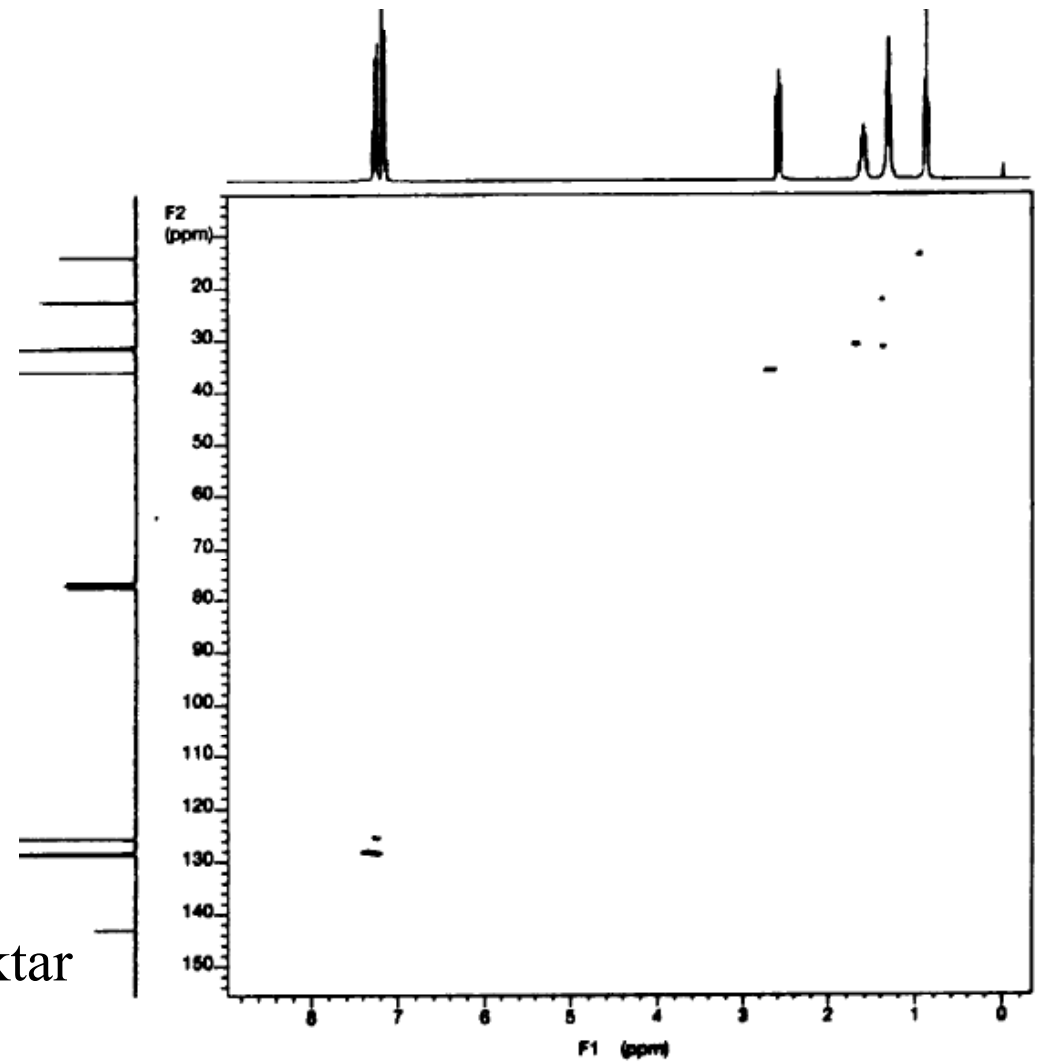
^{13}C NMR/DEPT spektr



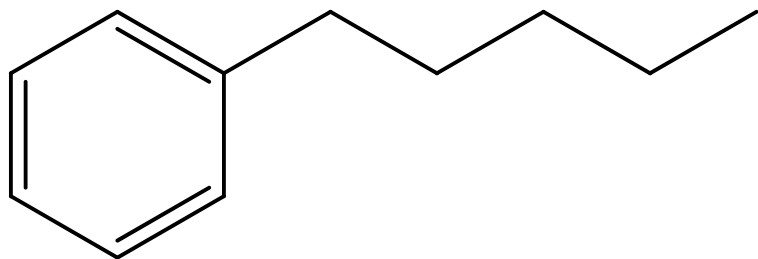
COSY spektr



HETCOR spektr



$$\text{IHD} = [2 \cdot 11 + 2 - 16] / 2 = 4$$



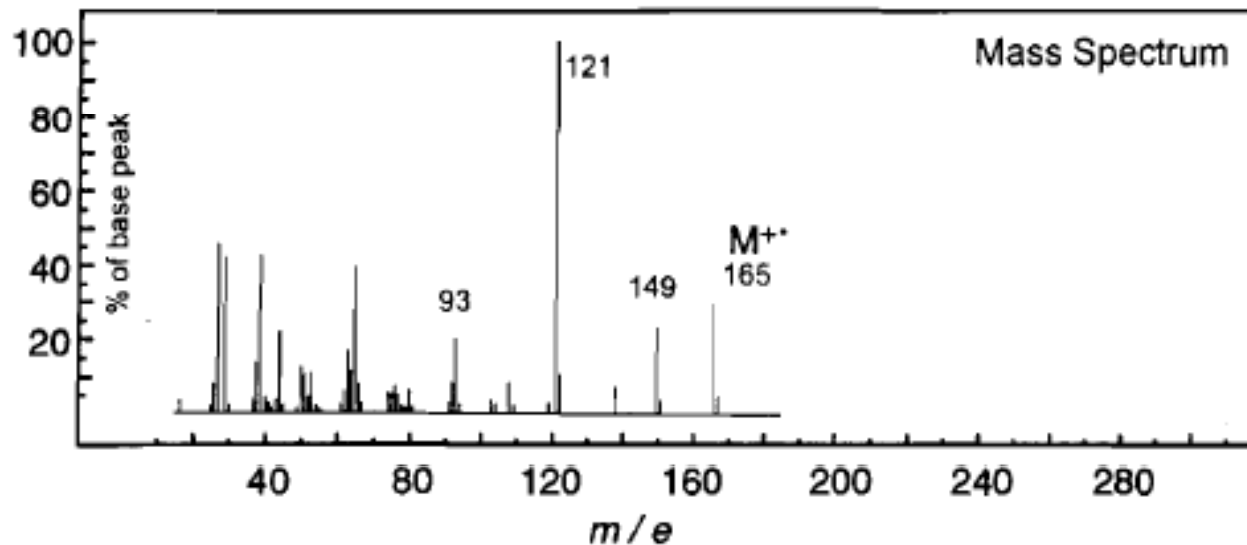
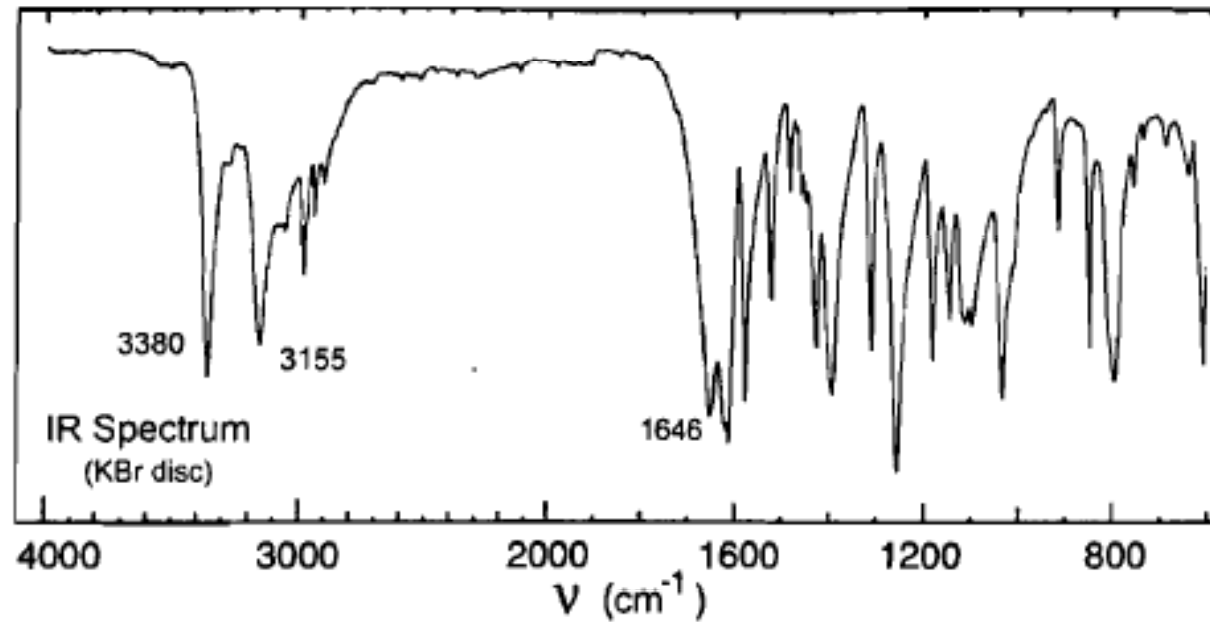
- informacije iz ^1H NMR:

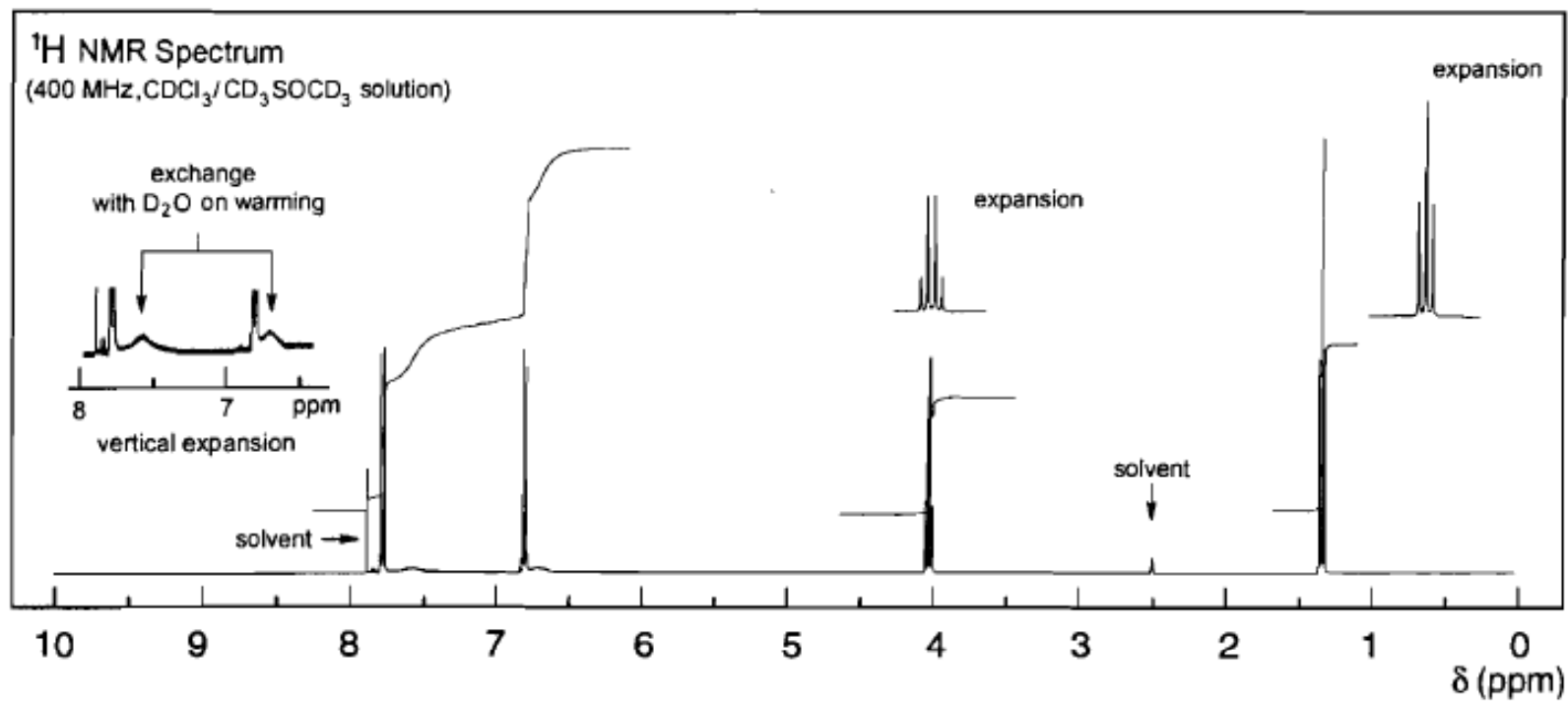
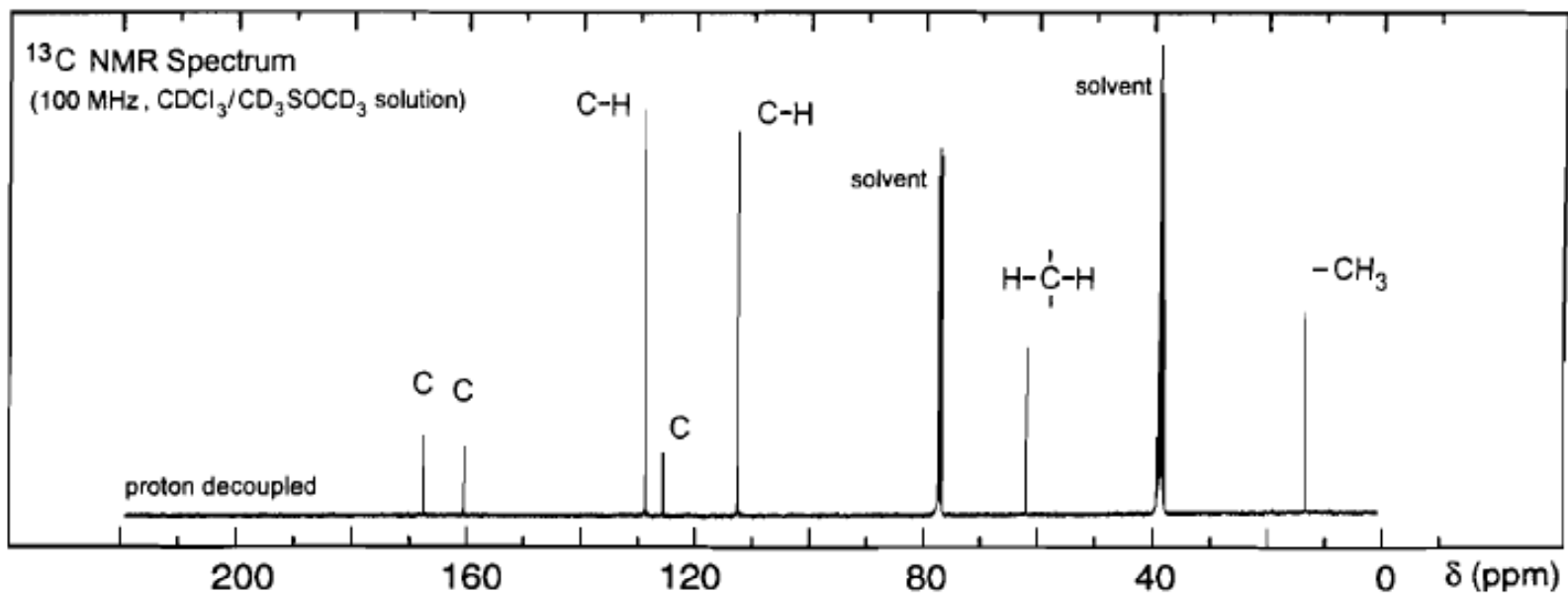
$\delta(^1\text{H})/\text{ppm}$	multiplet	H-atom
0,9	t	$-\text{CH}_3$
1,3	m	$-\text{CH}_2-$ $-\text{CH}_2-$
1,6	m(kvintet)	$-\text{CH}_2-$
2,6	t	$-\text{CH}_2-$
7,2	m	>CH
7,3	m	>CH

- informacije iz ^{13}C NMR:

$\delta(^{13}\text{C})/\text{ppm}$	C-atom
14	$-\text{CH}_3$
23	$-\text{CH}_2-$
31	$-\text{CH}_2-$
32	$-\text{CH}_2-$
36	$-\text{CH}_2-$
125	>CH
128	>CH
143	>C

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

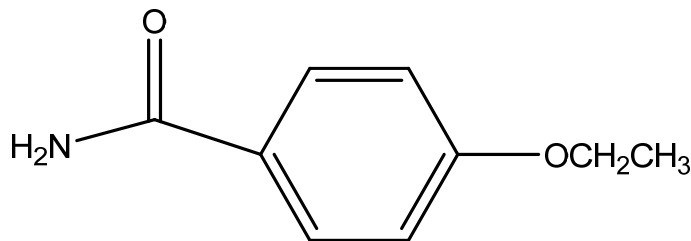




- informacije iz MS:

bazni pik: 121

$M^+ = 165$



- informacije iz IR:

3380 i 3155 cm^{-1} N–H istežanje

1646 cm^{-1} C=O istežanje

1250 cm^{-1} C–O istežanje

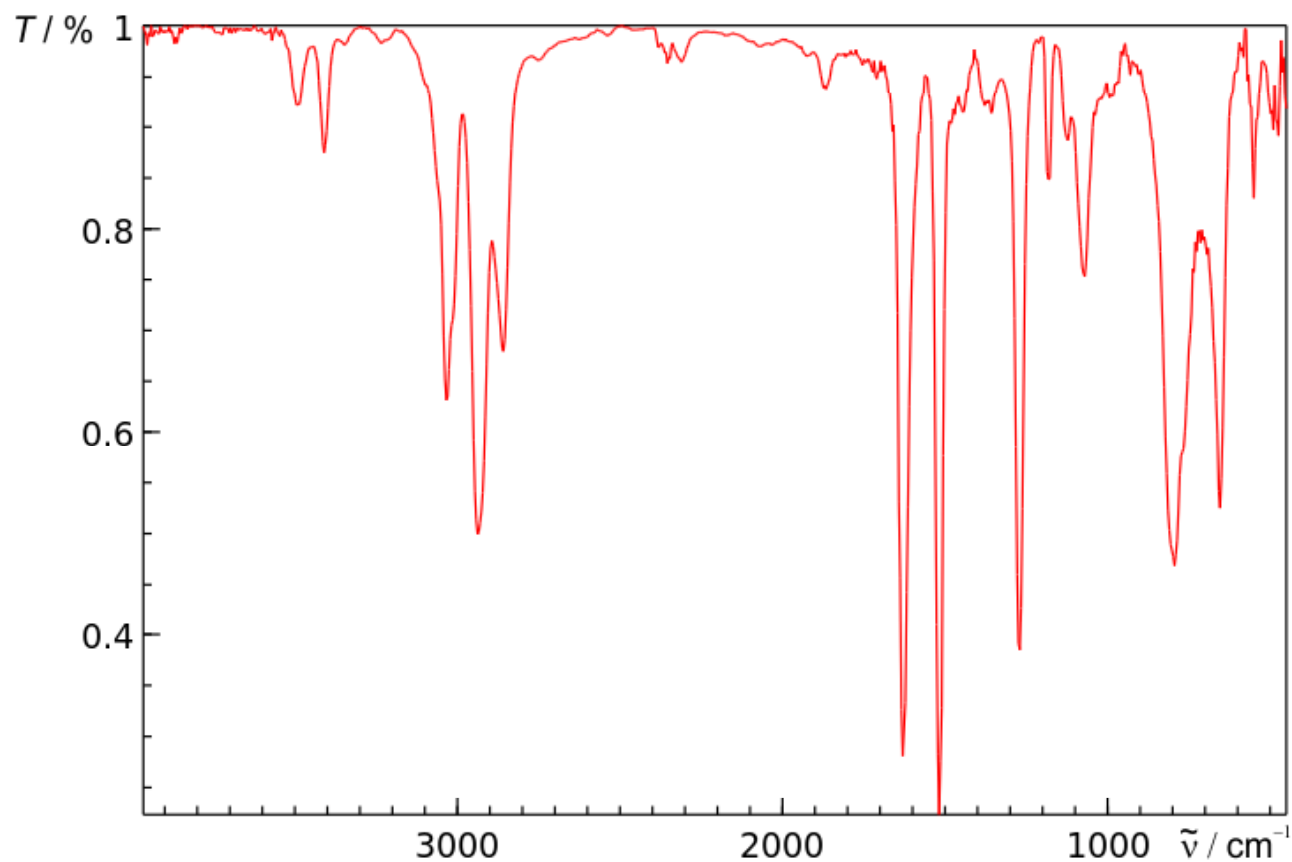
- informacije iz ^1H NMR:

$\delta(^1\text{H})/\text{ppm}$	multiplet	H-atom
1,4	t	$-\text{CH}_3$
4,0	q	$-\text{CH}_2-$
6,7	s	$-\text{NH}_2$
6,8	d	>CH
7,6	s	$-\text{NH}_2$
7,8	d	>CH

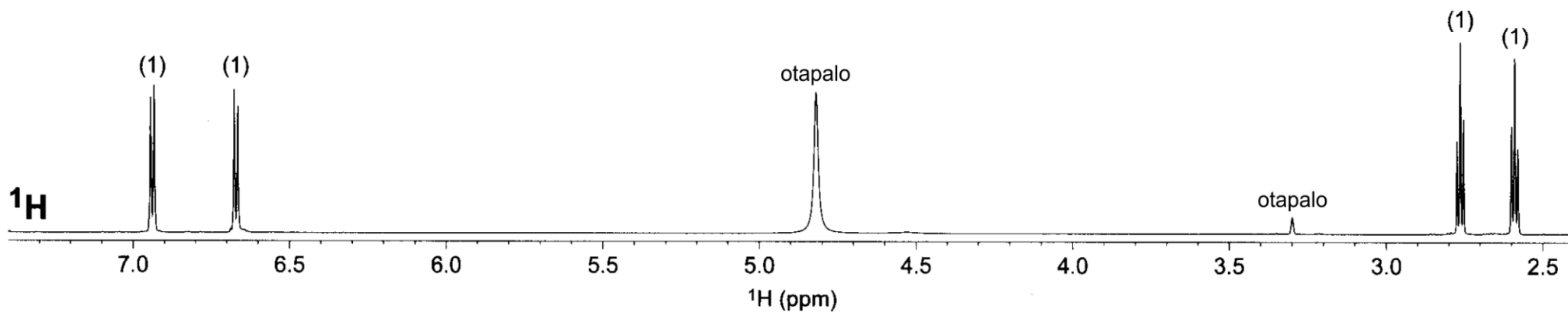
- informacije iz ^{13}C NMR:

$\delta(^{13}\text{C})/\text{ppm}$	C-atom
12	$-\text{CH}_3$
62	$-\text{CH}_2-$
110	>CH
126	>C
129	>CH
160	>C
168	>C

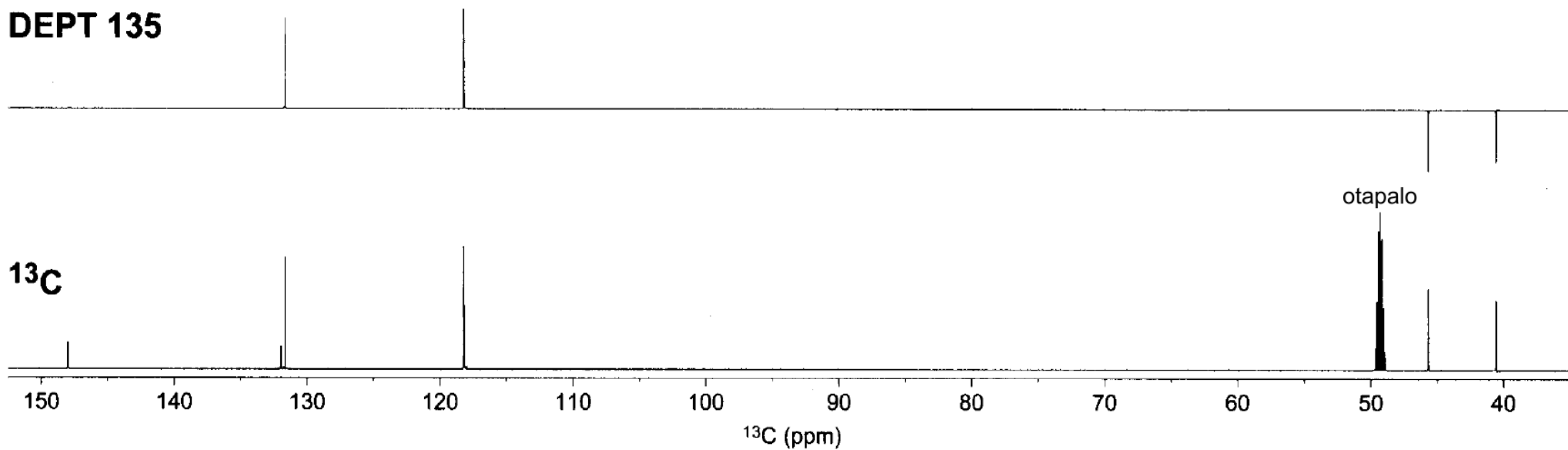
4. Odredite strukturu spoja molarne mase 136 g mol^{-1} na temelju njegovih IR, $^1\text{H NMR}$ $^{13}\text{C NMR}$, COSY, HSQC i HMBC spektara.



IR spektar spoja snimljen tehnikom KBr pastile.

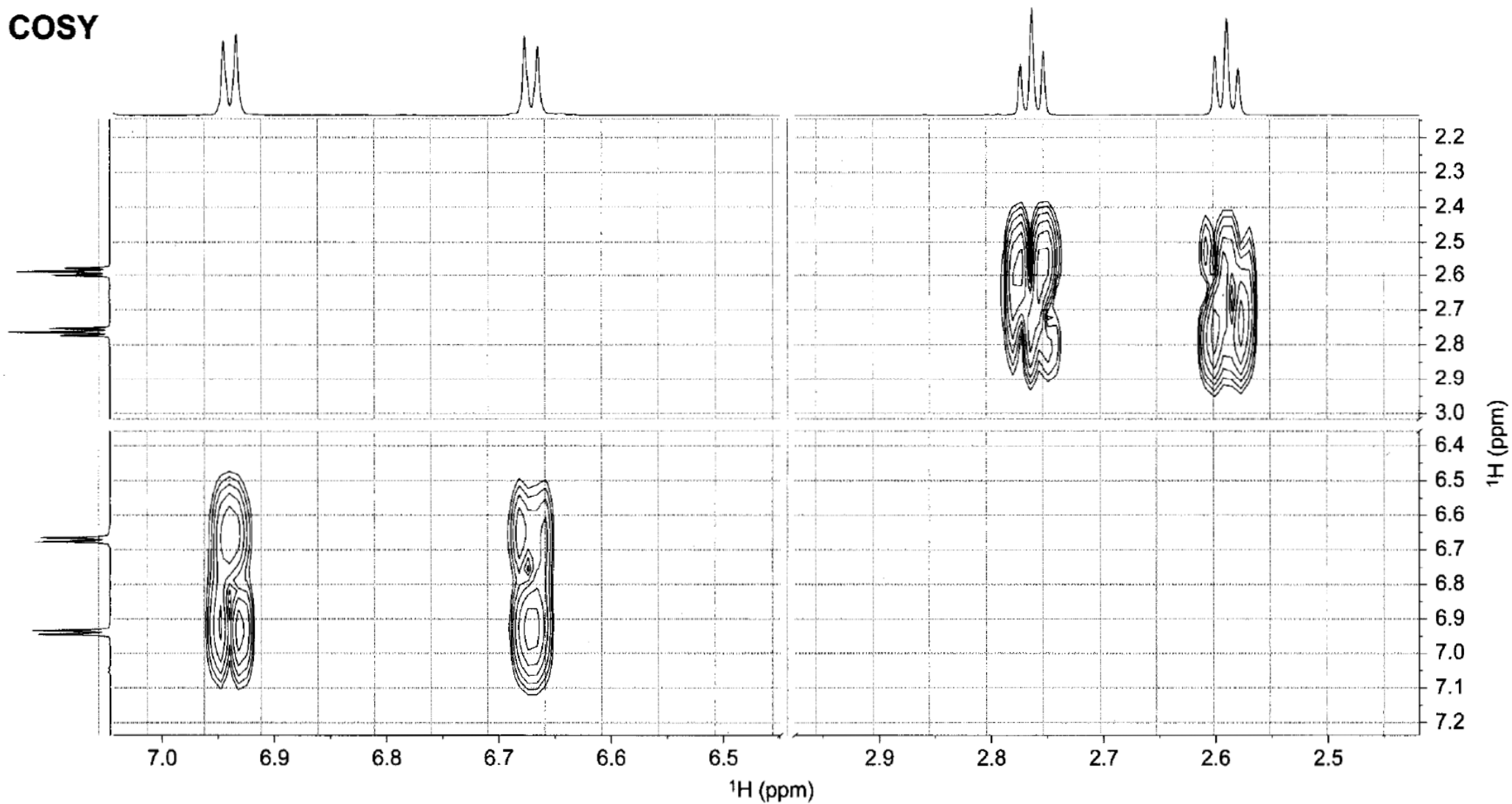


^1H NMR spektar spoja snimljen u CD_3OD . Spoj sadrži i protone koji se izmijene u CD_3OD .



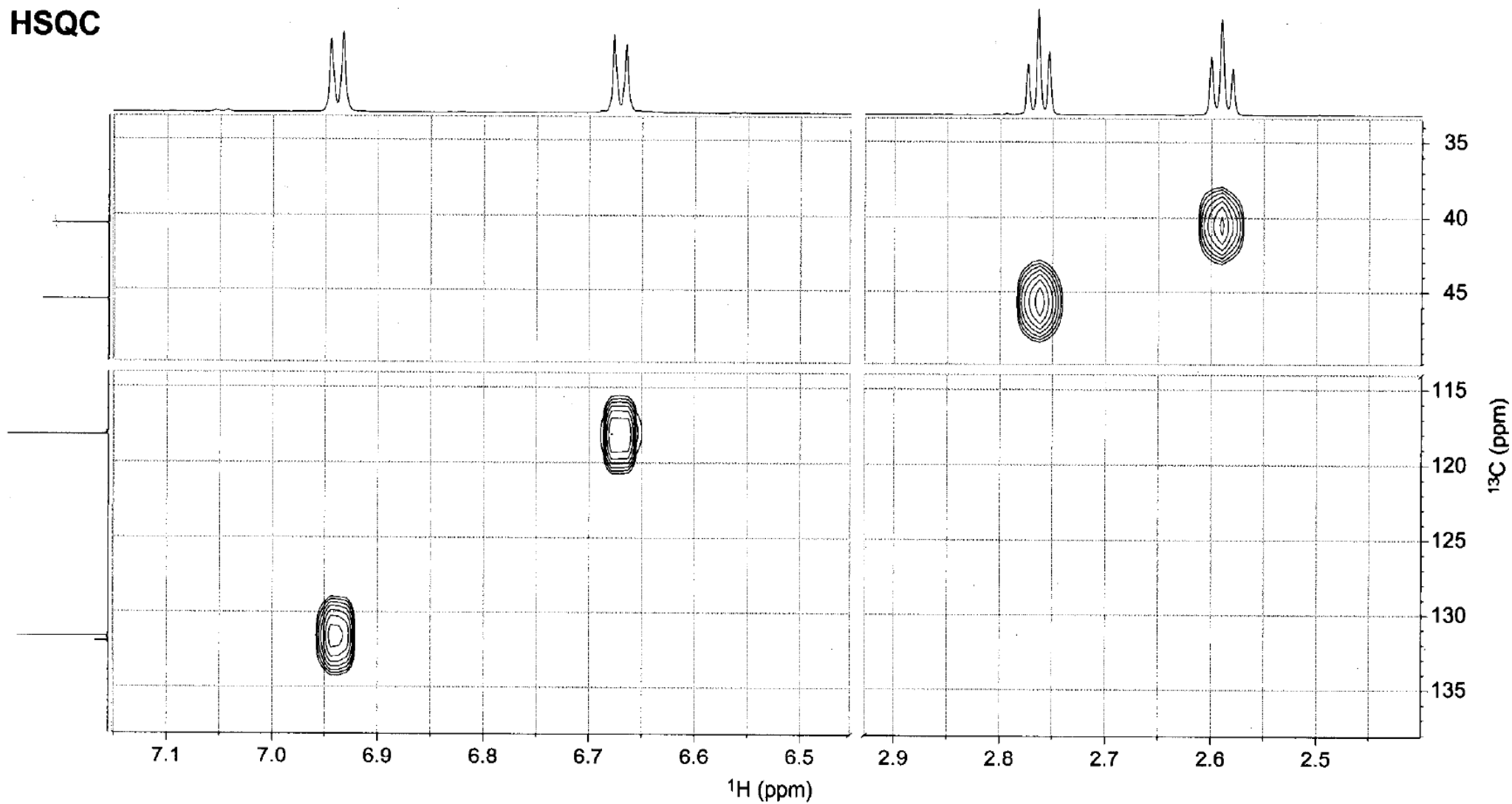
^{13}C NMR spektri spoja snimljeni u CD_3OD .

COSY



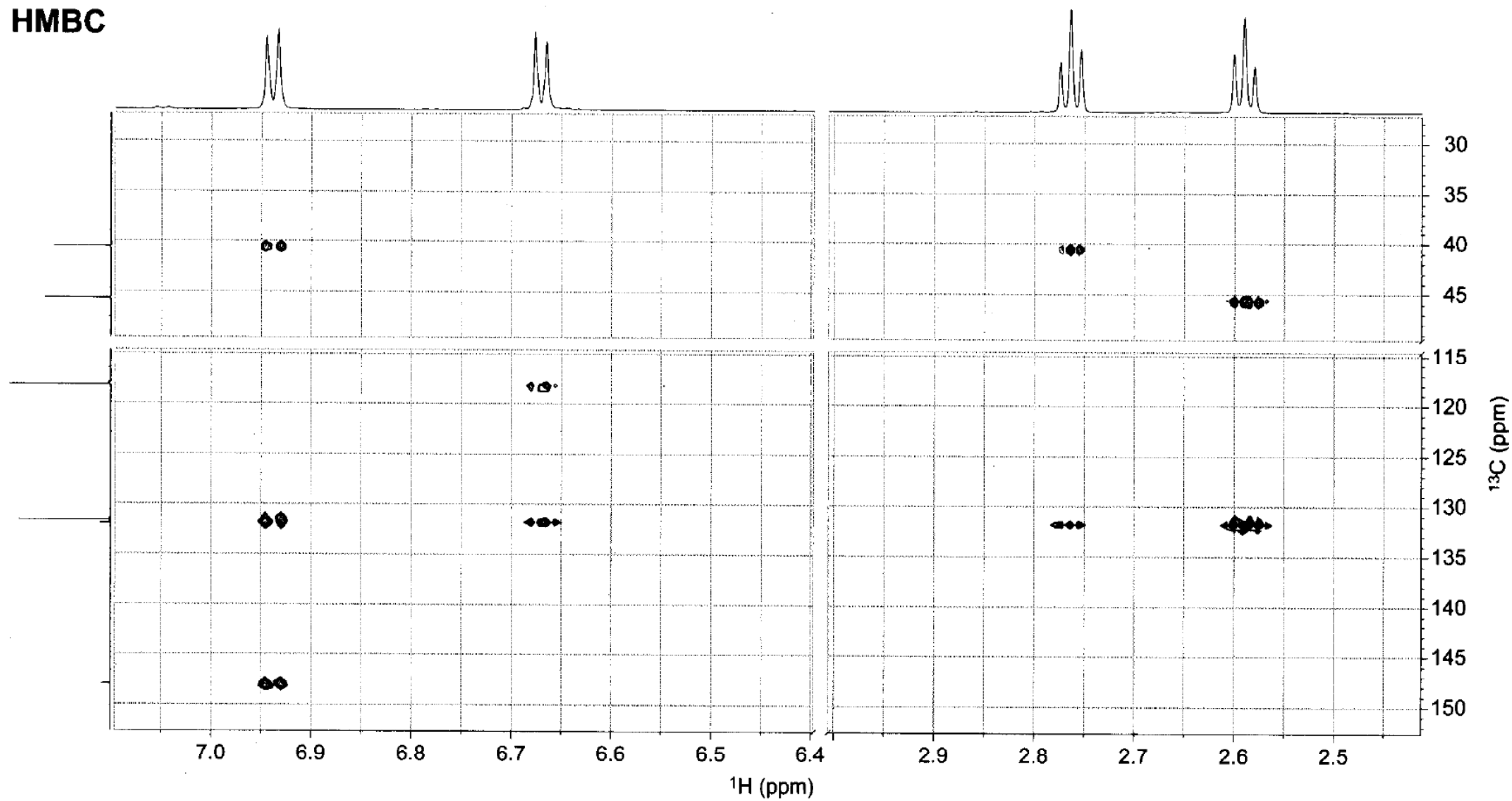
COSY spektar spoja snimljen u CD_3OD .

HSQC



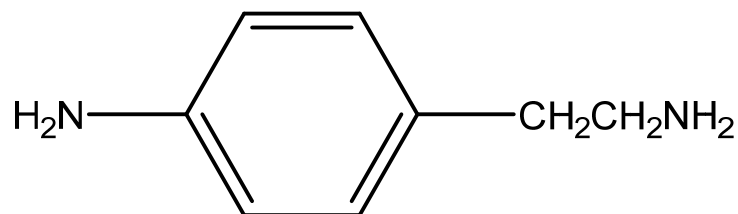
HSQC spektar spoja snimljen u CD_3OD .

HMBC



HMBC spektar spoja snimljen u CD_3OD .

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



- informacije iz ^1H NMR:

$\delta(^1\text{H})/\text{ppm}$	multiplet	H-atom
2,59	t	$-\text{CH}_2-$
2,75	t	$-\text{CH}_2-$
6,68	d	>CH
6,95	d	>CH

- informacije iz IR:

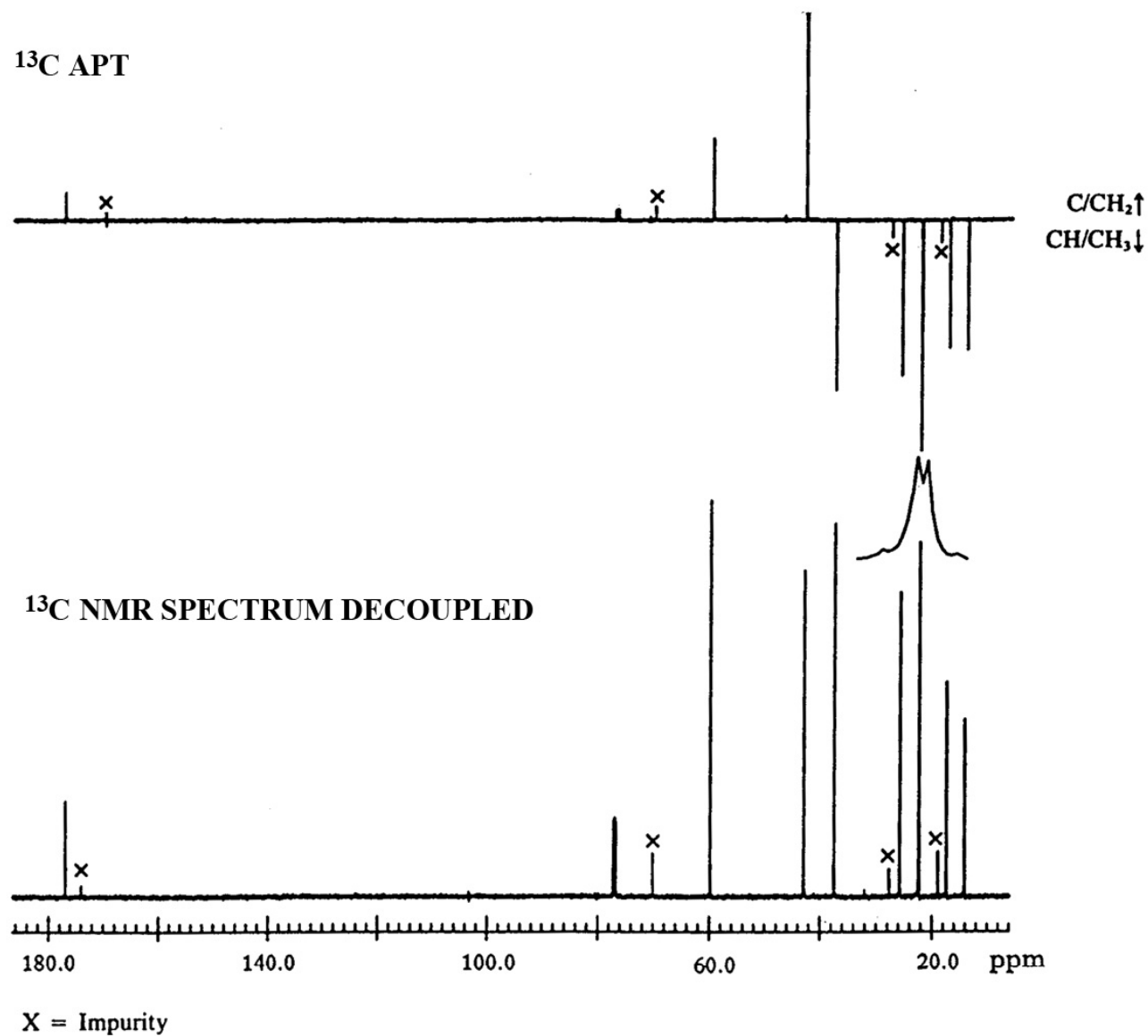
3400 i 3550 cm^{-1} N–H istežanje
 2900 i 2950 cm^{-1} C–H alifatsko istežanje

3050 cm^{-1} C–H aromatsko istežanje
 1500 i 1400 cm^{-1} C–C istežanja
 800 cm^{-1} C–H aromatsko svijanje

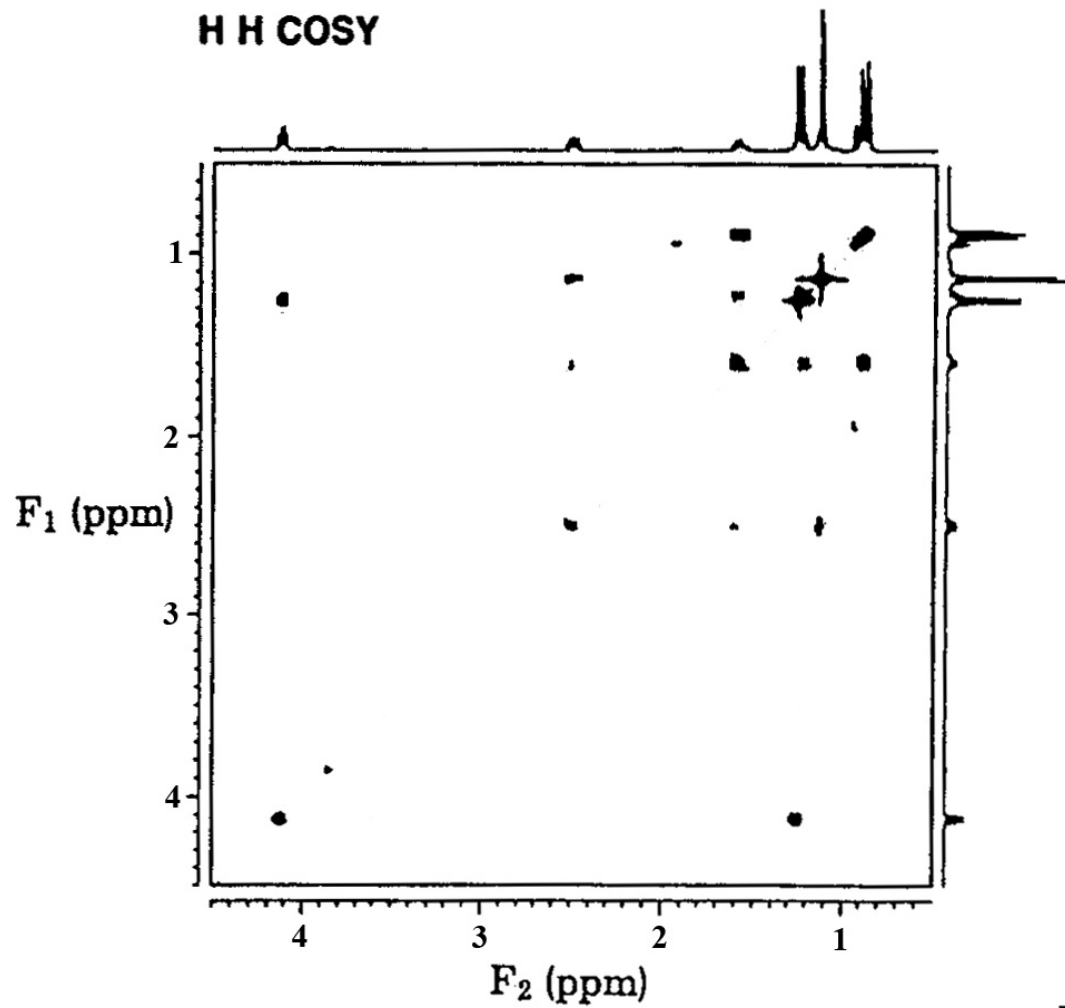
- informacije iz ^{13}C NMR:

$\delta(^{13}\text{C})/\text{ppm}$	C-atom
41	$-\text{CH}_2-$
46	$-\text{CH}_2-$
118	>CH
132	>CH
148	>C

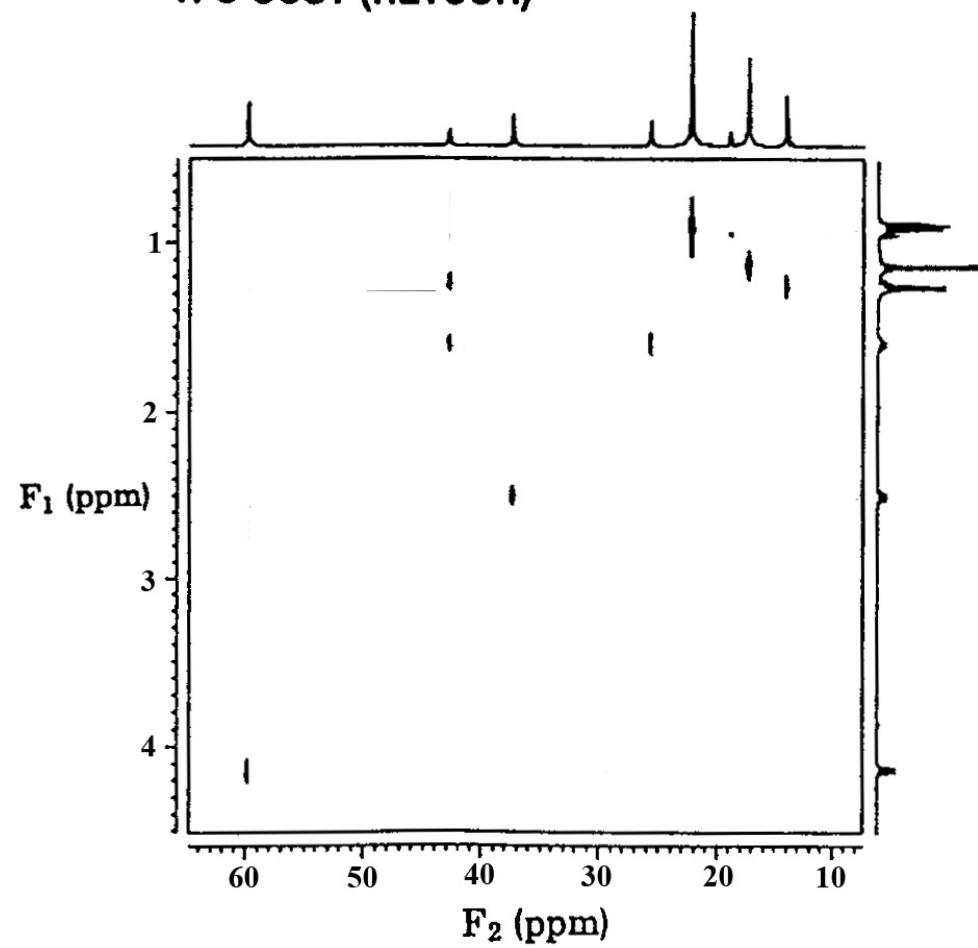
5. Odredite strukturu spoja molekulske formule $C_9H_{18}O_2$ na temelju njegovih ^{13}C NMR, COSY i HETCOR spektara.

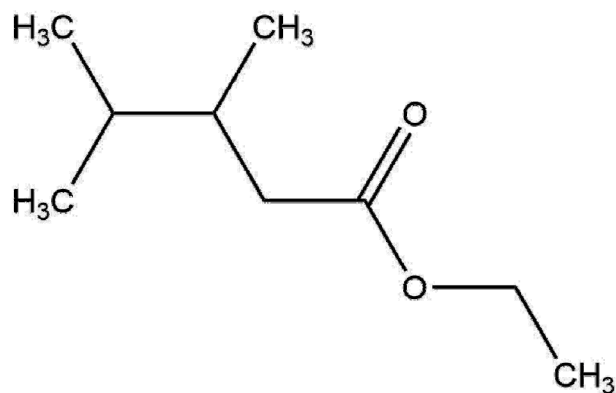


H H COSY



H C COSY (HETCOR)





- informacije iz ^1H NMR:

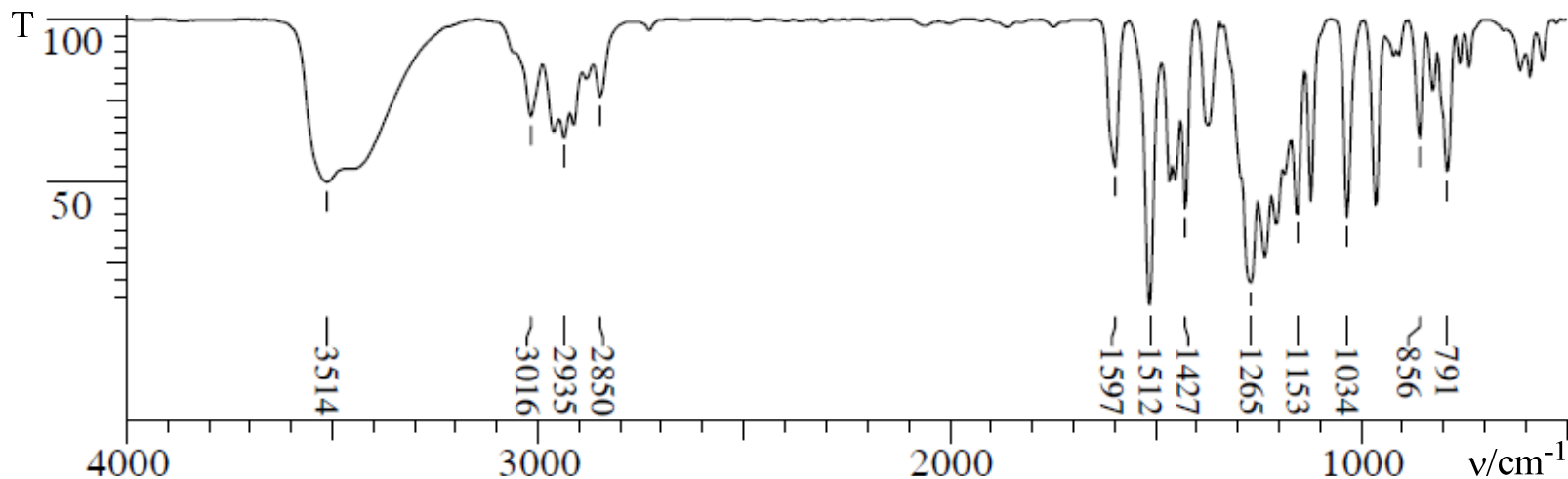
$\delta(^1\text{H})/\text{ppm}$	H-atom
0,9	$-\text{CH}_3-\text{CH}_3$
1,2	$-\text{CH}_3$
1,3	$-\text{CH}_3$
1,7	$-\text{CH}_2-$ >CH
2,5	>CH
4,1	$-\text{CH}_2-$

- informacije iz ^{13}C NMR:

$\delta(^{13}\text{C})/\text{ppm}$	C-atom
17	$-\text{CH}_3$
19	$-\text{CH}_3$
20,9	$-\text{CH}_3$
21,1	$-\text{CH}_3$
27	>CH
38	>CH
43	$-\text{CH}_2-$
60	$-\text{CH}_2-$
178	>C

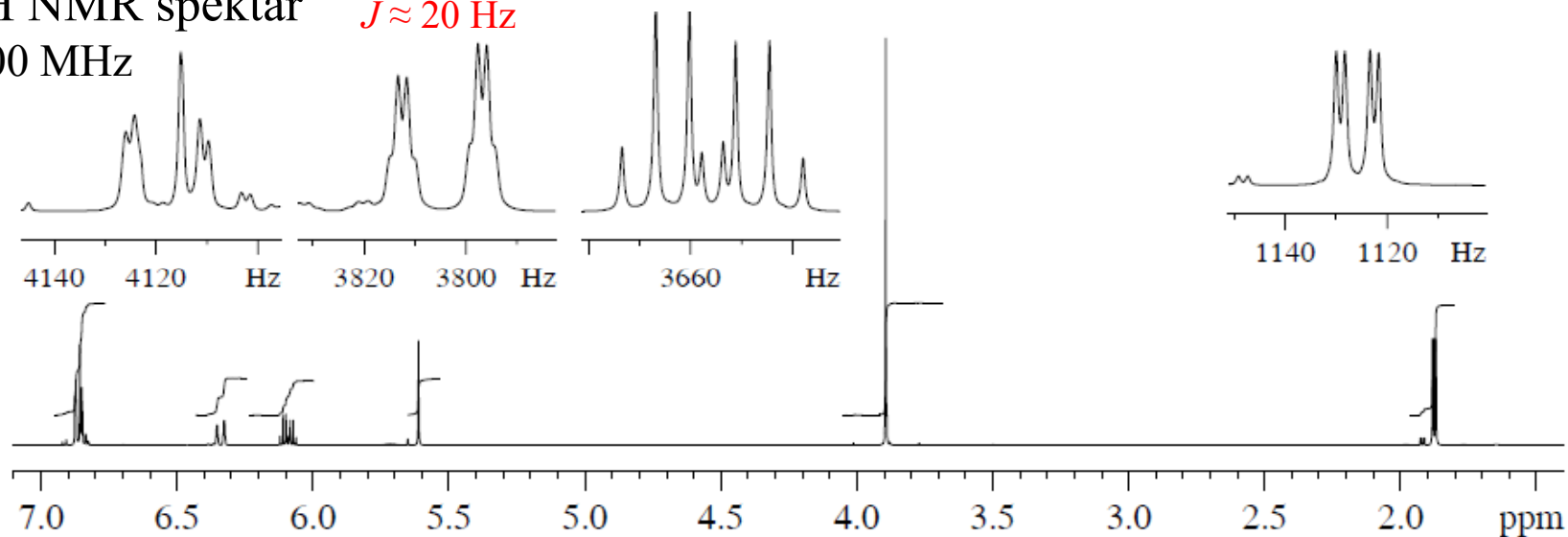
6. Identificirajte spoj molarne mase 164 na temelju njegovih IR, ^1H NMR, ^{13}C NMR, COSY, HMQC i HMBC spektara.

IR spektar

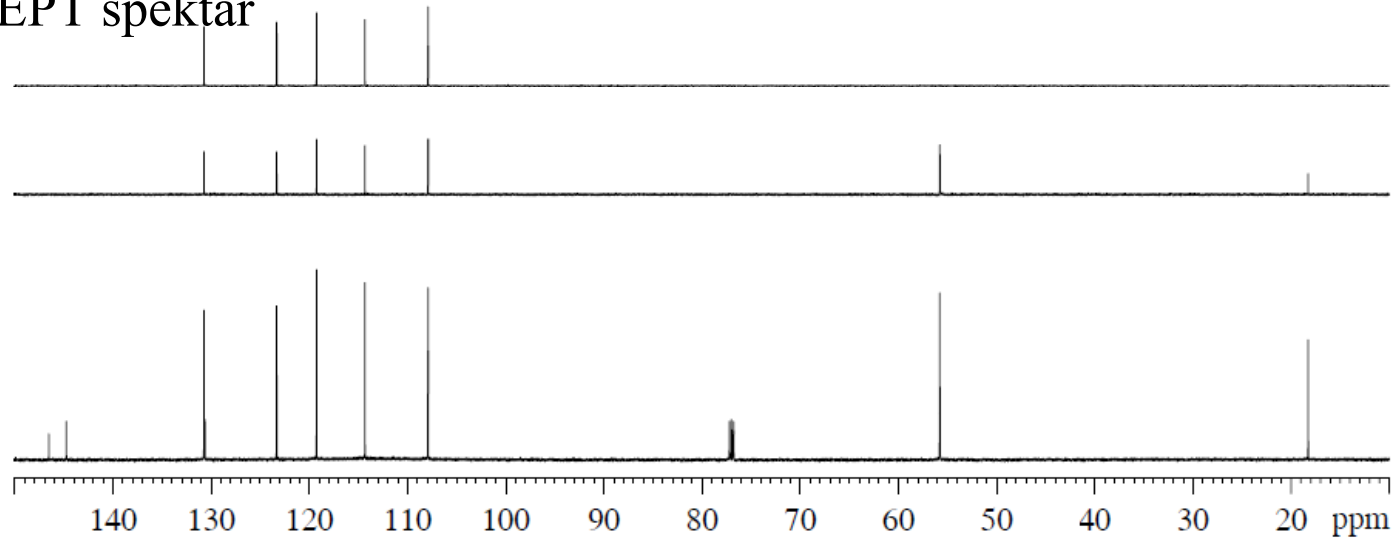


^1H NMR spektar $J \approx 20 \text{ Hz}$

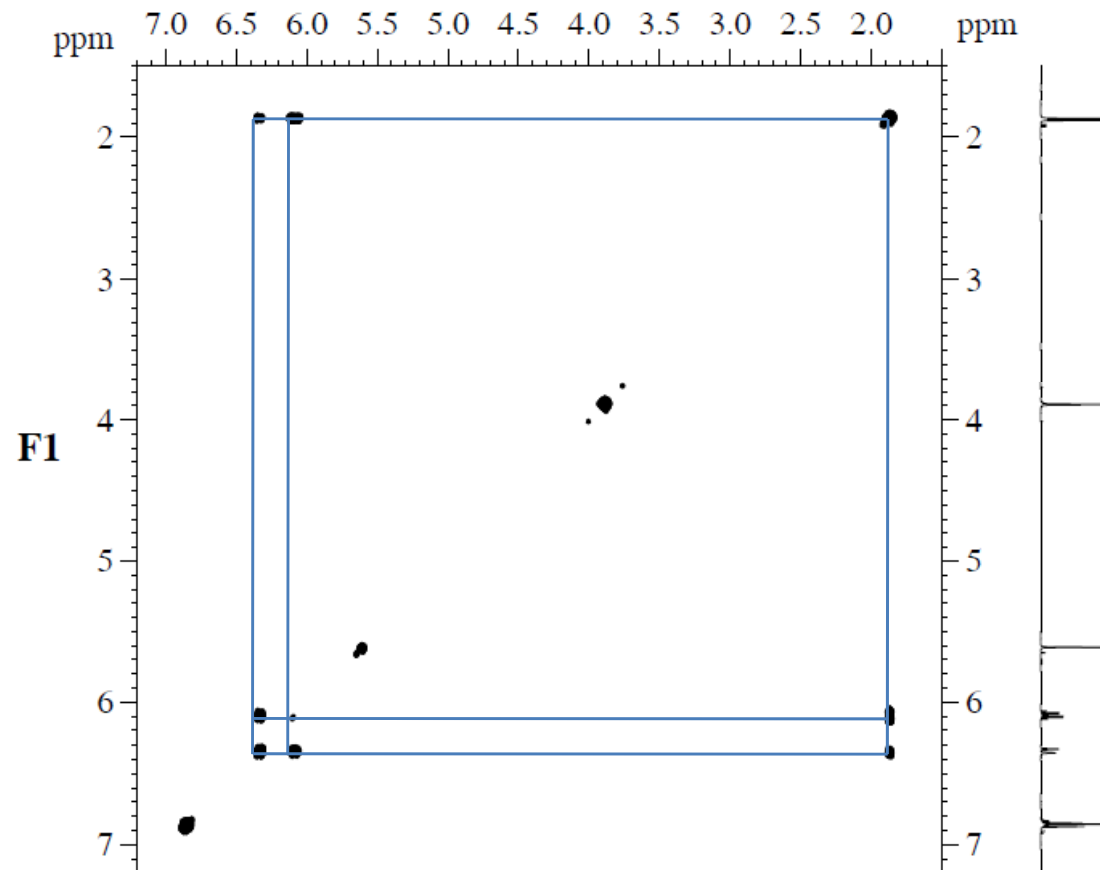
600 MHz



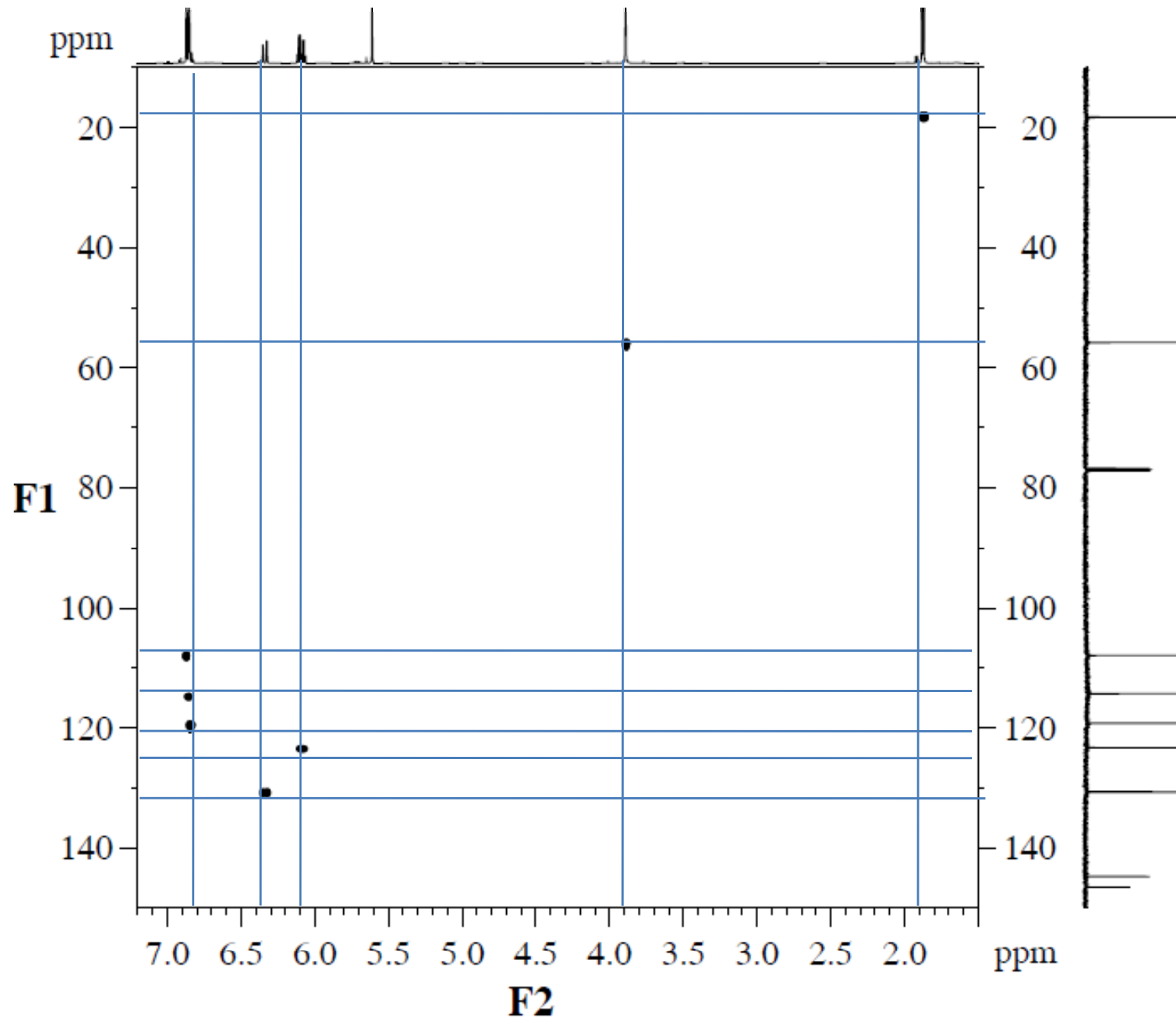
^{13}C NMR/DEPT spektr

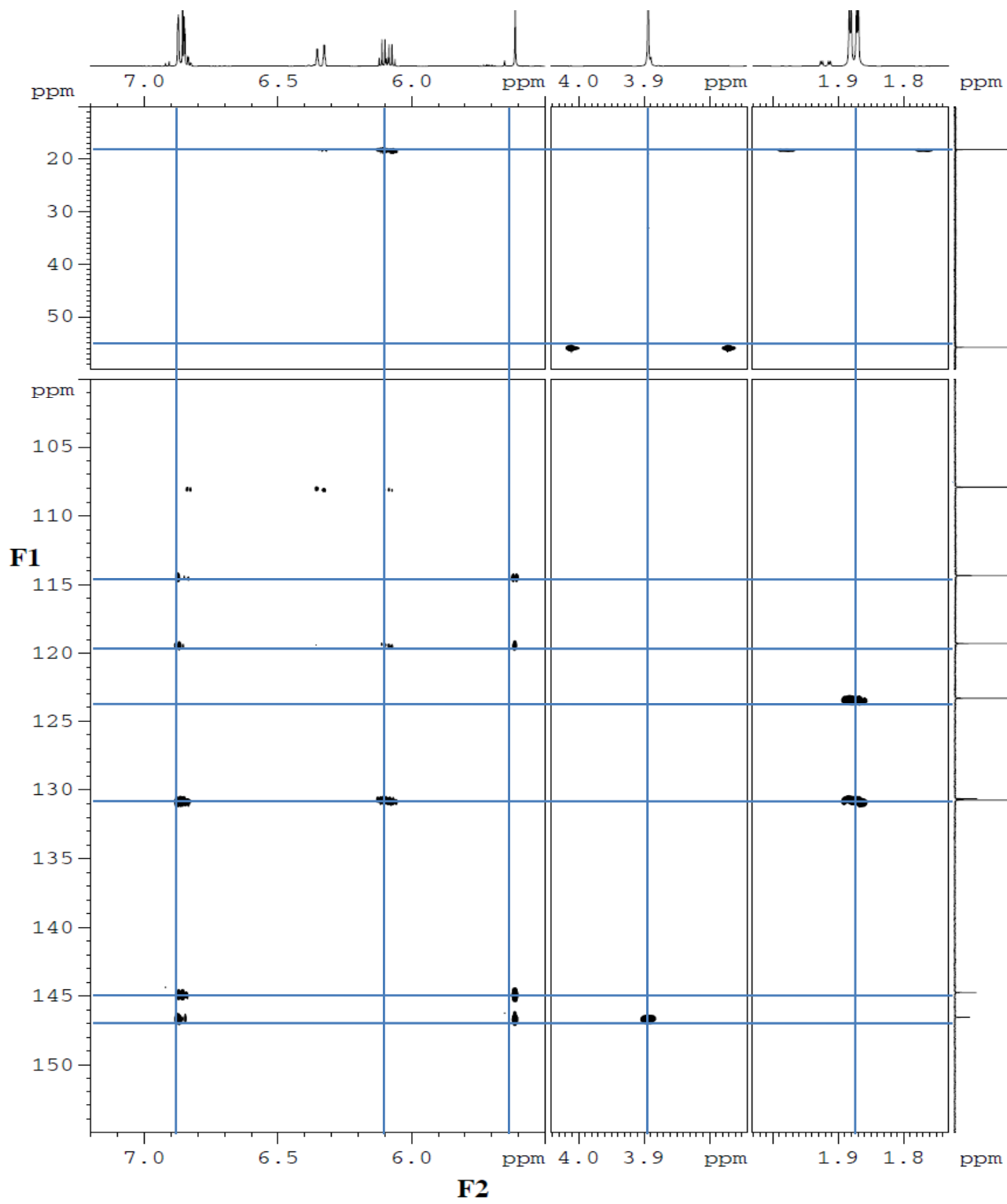


COSY spektr



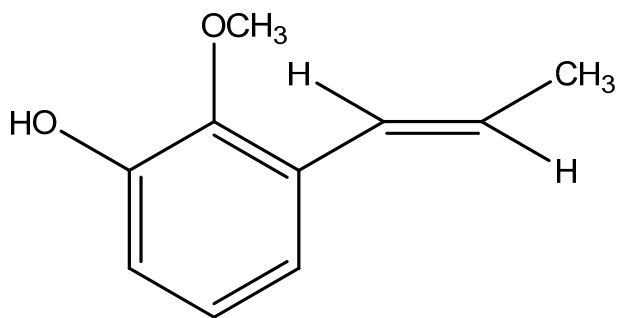
HMQC spektar





HMBC spektr

$J \approx 20 \text{ Hz}$
trans



- informacije iz ^1H NMR:

$\delta(^1\text{H})/\text{ppm}$	multiplet	H-atom
1,9	dd	$-\text{CH}_3$
3,9	s	$-\text{CH}_3$
5,6	s	$-\text{OH}$
6,1	qd	—CH
6,3	qd	—CH
6,9	m	$\text{—CH} \quad \text{—CH}$

- informacije iz IR:

3514 cm^{-1} O–H istežanje

3016 cm^{-1} C–H aromatsko istežanje

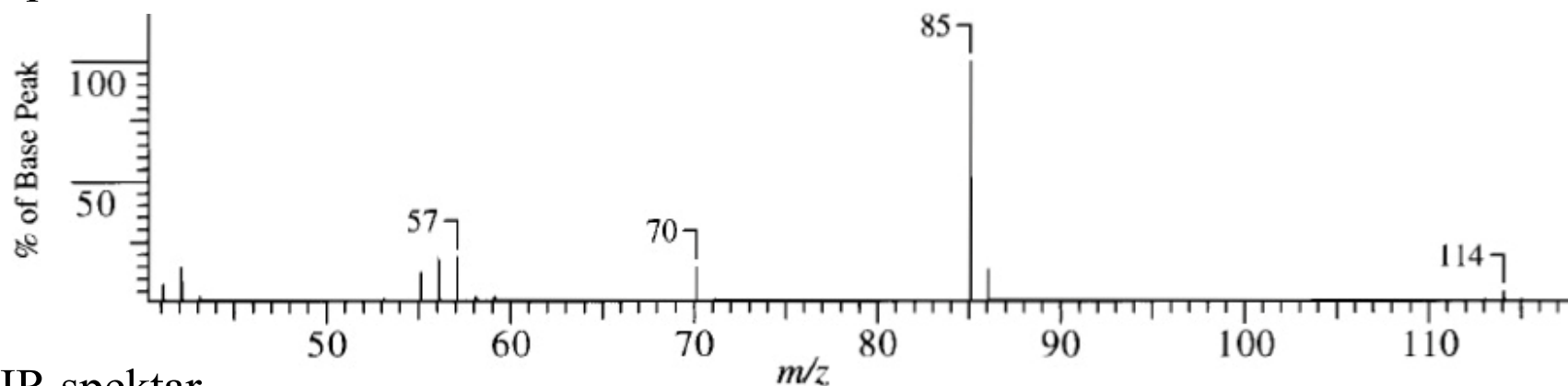
2935 i 2850 cm^{-1} C–H alifatsko istežanje

- informacije iz ^{13}C NMR:

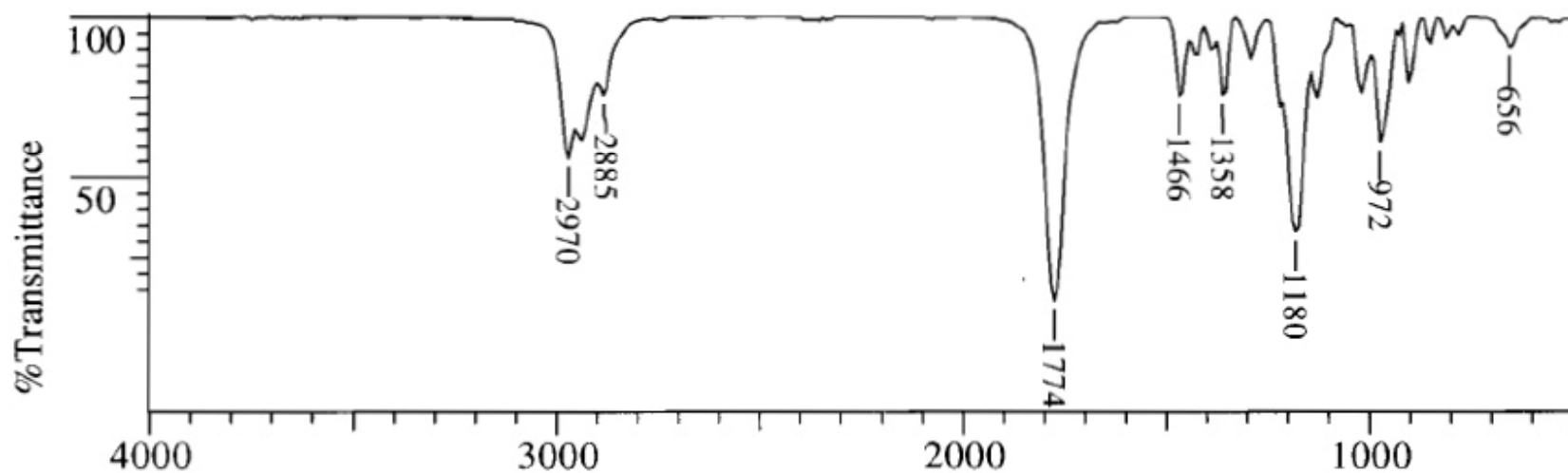
$\delta(^{13}\text{C})/\text{ppm}$	C-atom
18	$-\text{CH}_3$
56	$-\text{CH}_3$
108	—CH
115	—CH
120	—CH
124	—CH
131	—CH
145	—C—
147	—C—

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

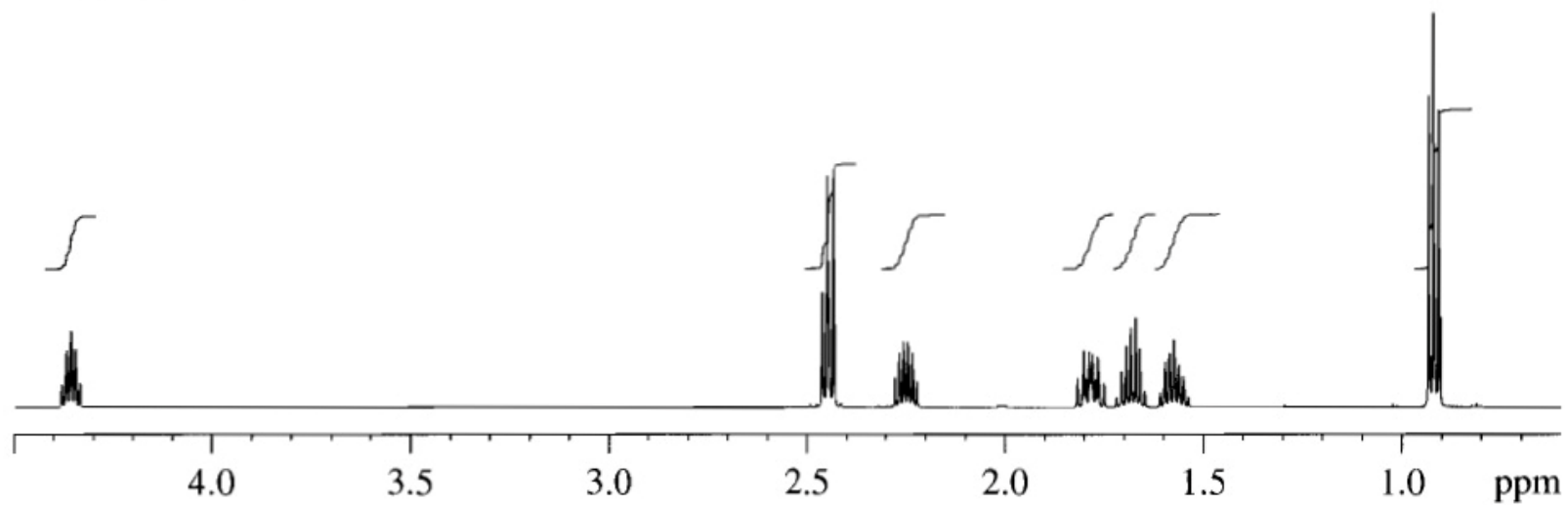
Spektar MS



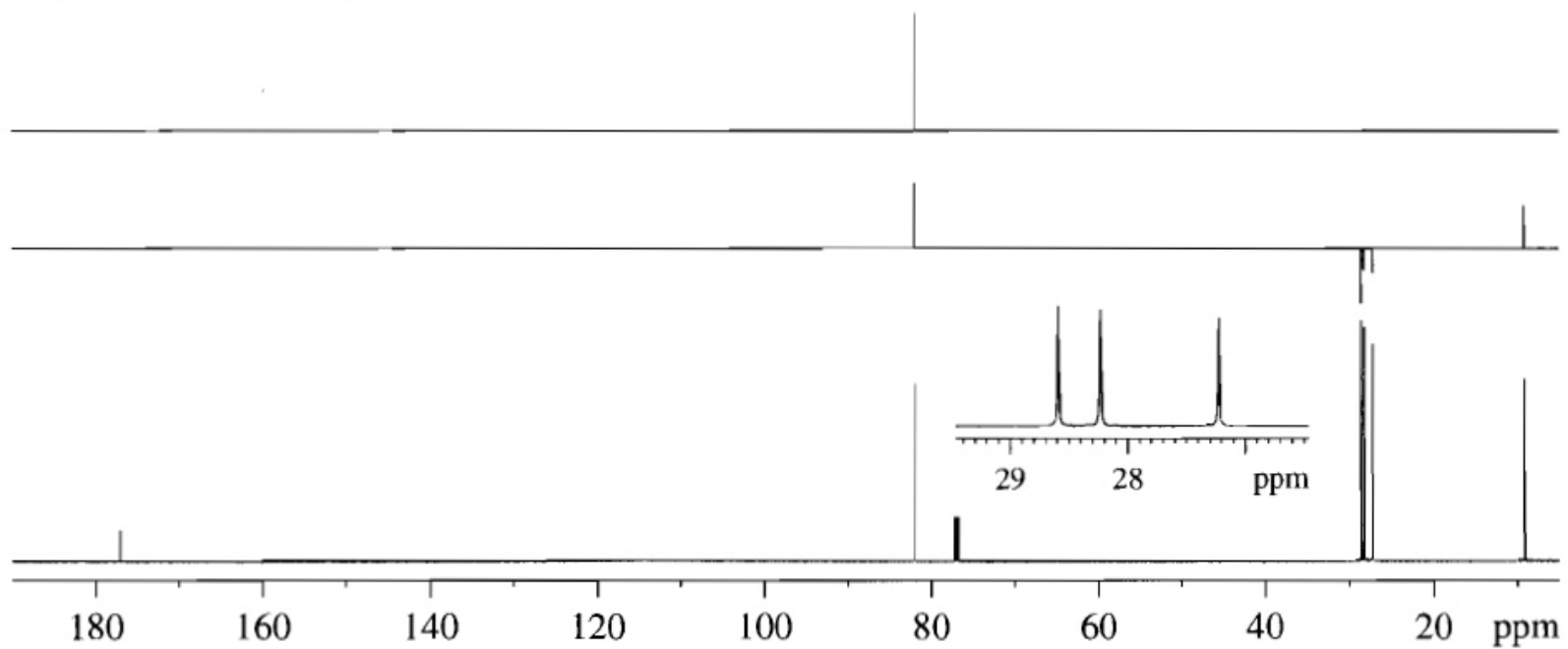
IR spektar

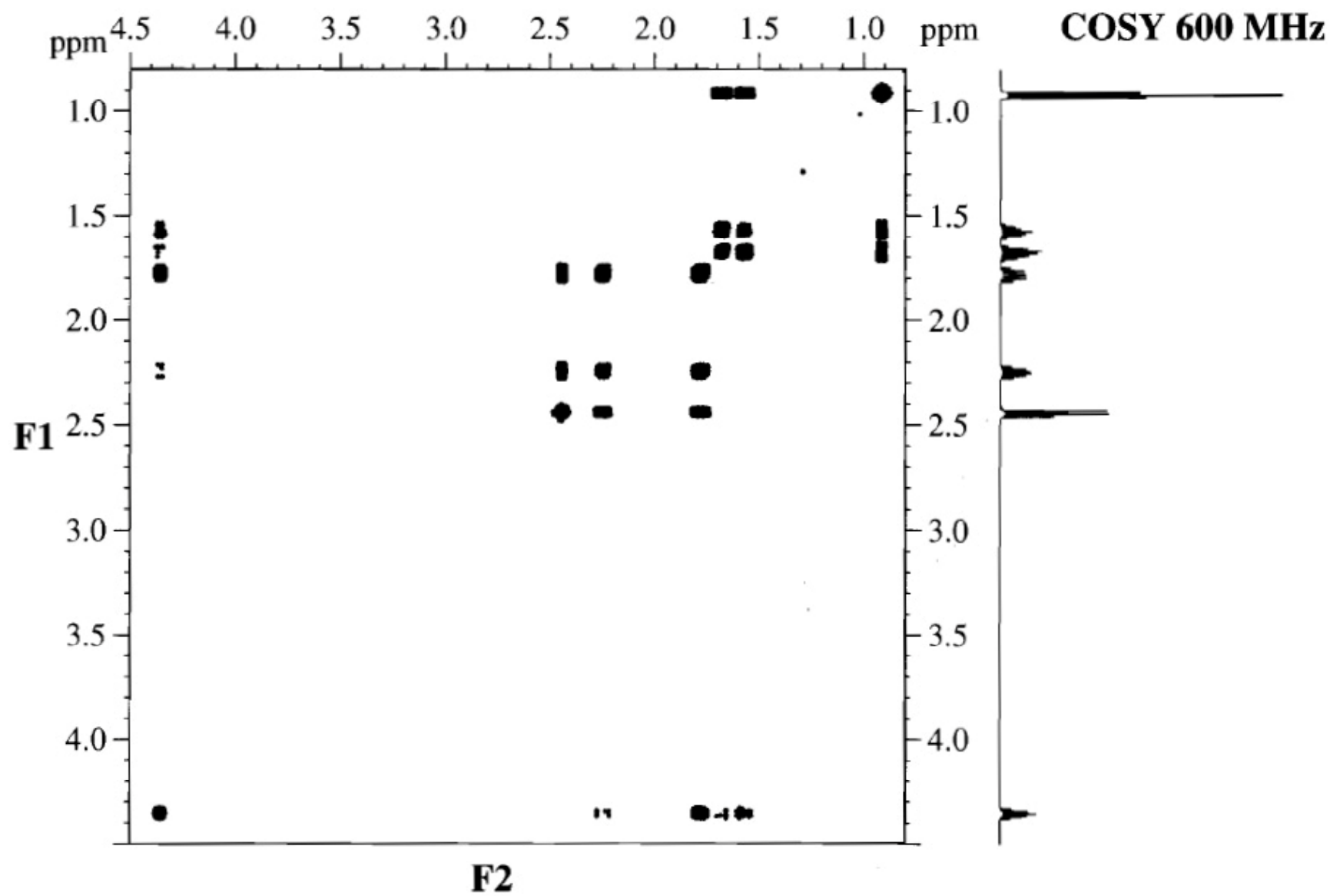


^1H NMR 600 MHz

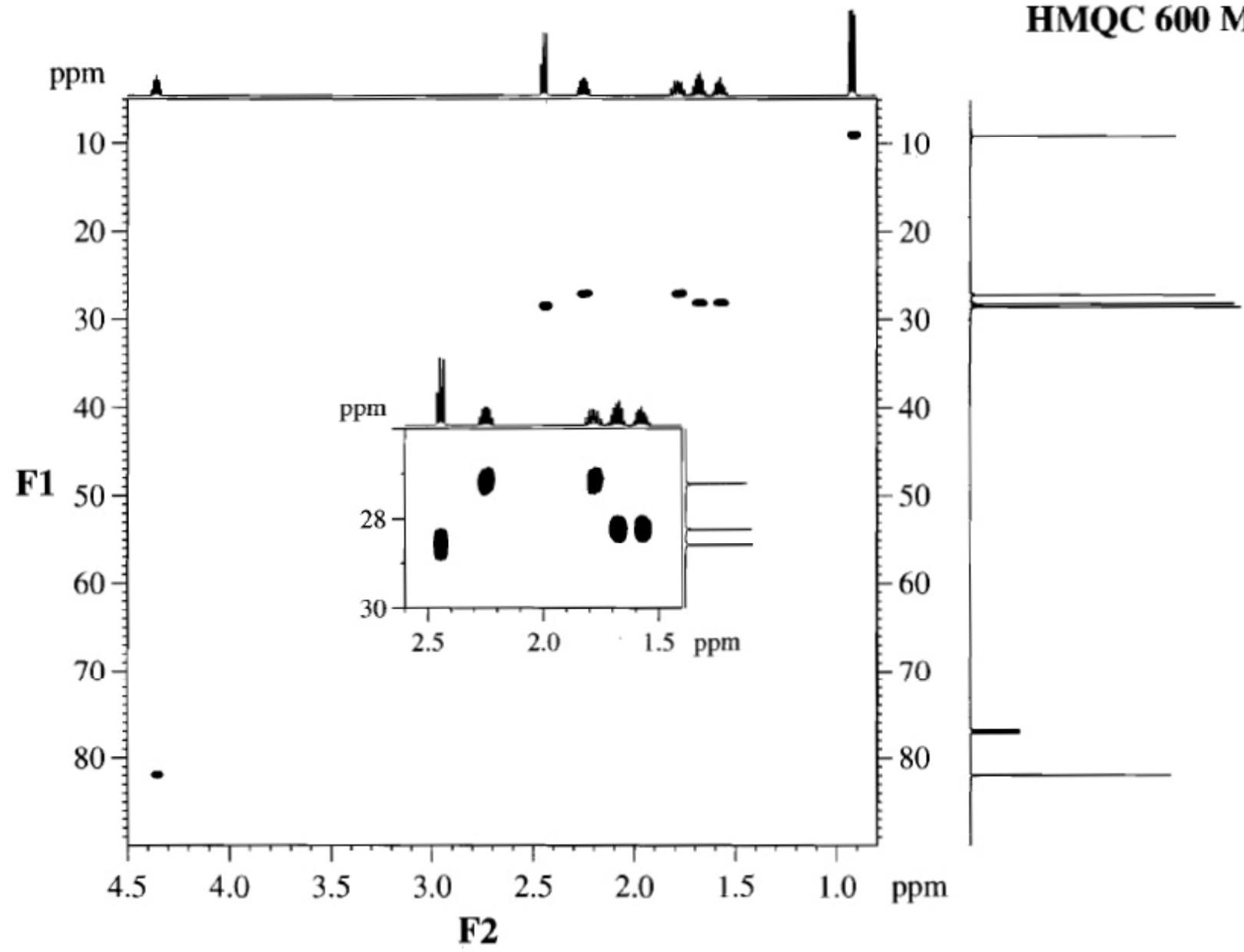


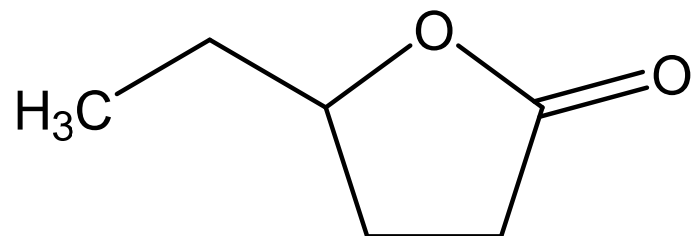
$^{13}\text{C}/\text{DEPT}$ NMR 150.9 MHz





HMQC 600 MHz





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

- informacije iz ^1H NMR:

$\delta(^1\text{H})/\text{ppm}$	multiplet	H-atom
0,90	t	$-\text{CH}_3$
1,57; 1,67	m	$-\text{CH}_2-$
1,80; 2,25	m	$-\text{CH}_2-$
2,45	t	$-\text{CH}_2-$
4,35	m	>CH

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

- informacije iz ^{13}C NMR:

$\delta(^1\text{H})/\text{ppm}$	C-atom
9	$-\text{CH}_3$
27,2	$-\text{CH}_2-$
28,2	$-\text{CH}_2-$
28,6	$-\text{CH}_2-$
83	>CH
178	>C