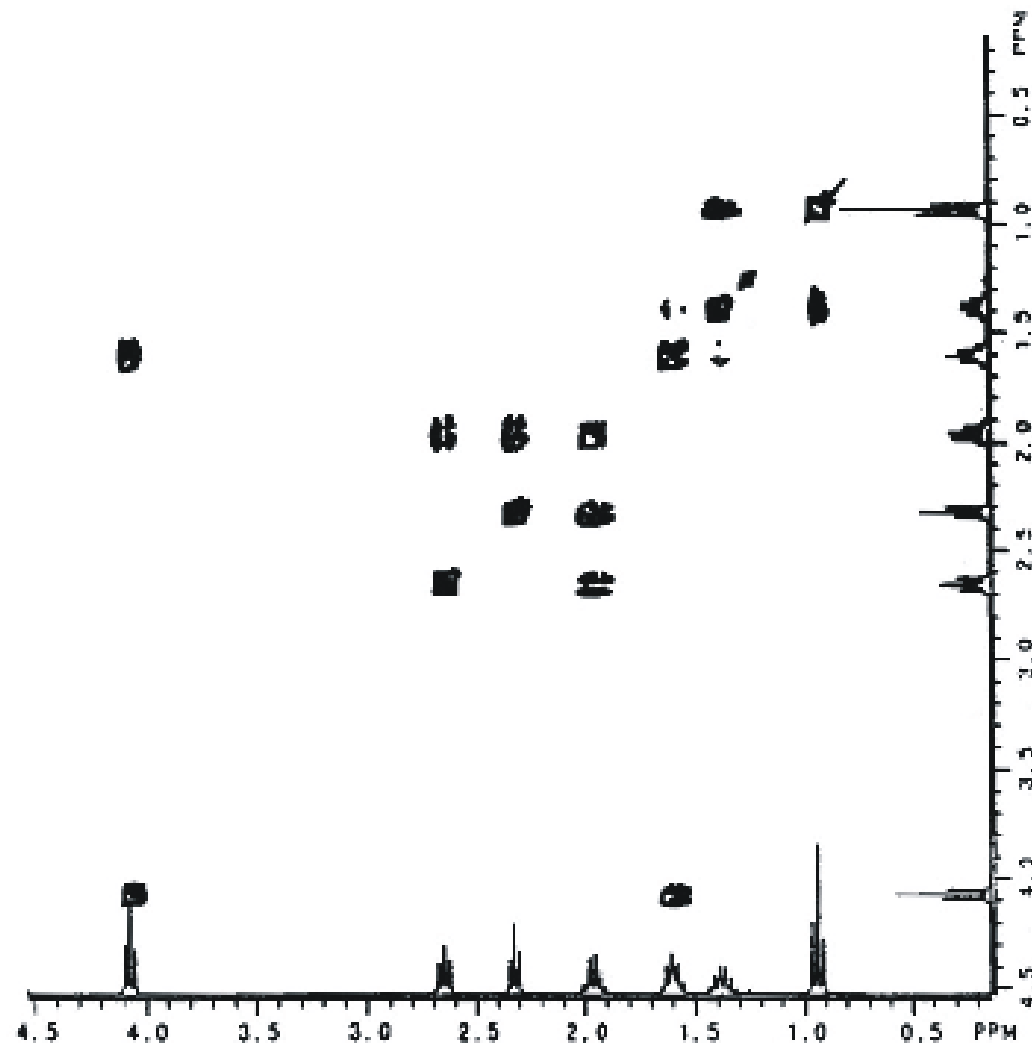


Spektroskopska strukturna analiza

Dodatni zadaci

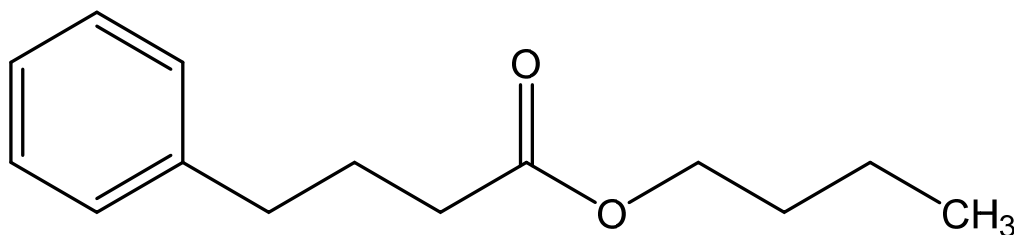
1. Odredite strukturu spoja molekulske formule $C_{14}H_{20}O_2$ na temelju njegovog COSY spektra. 1H NMR spektar sadrži još široki singlet s integralom 5.



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

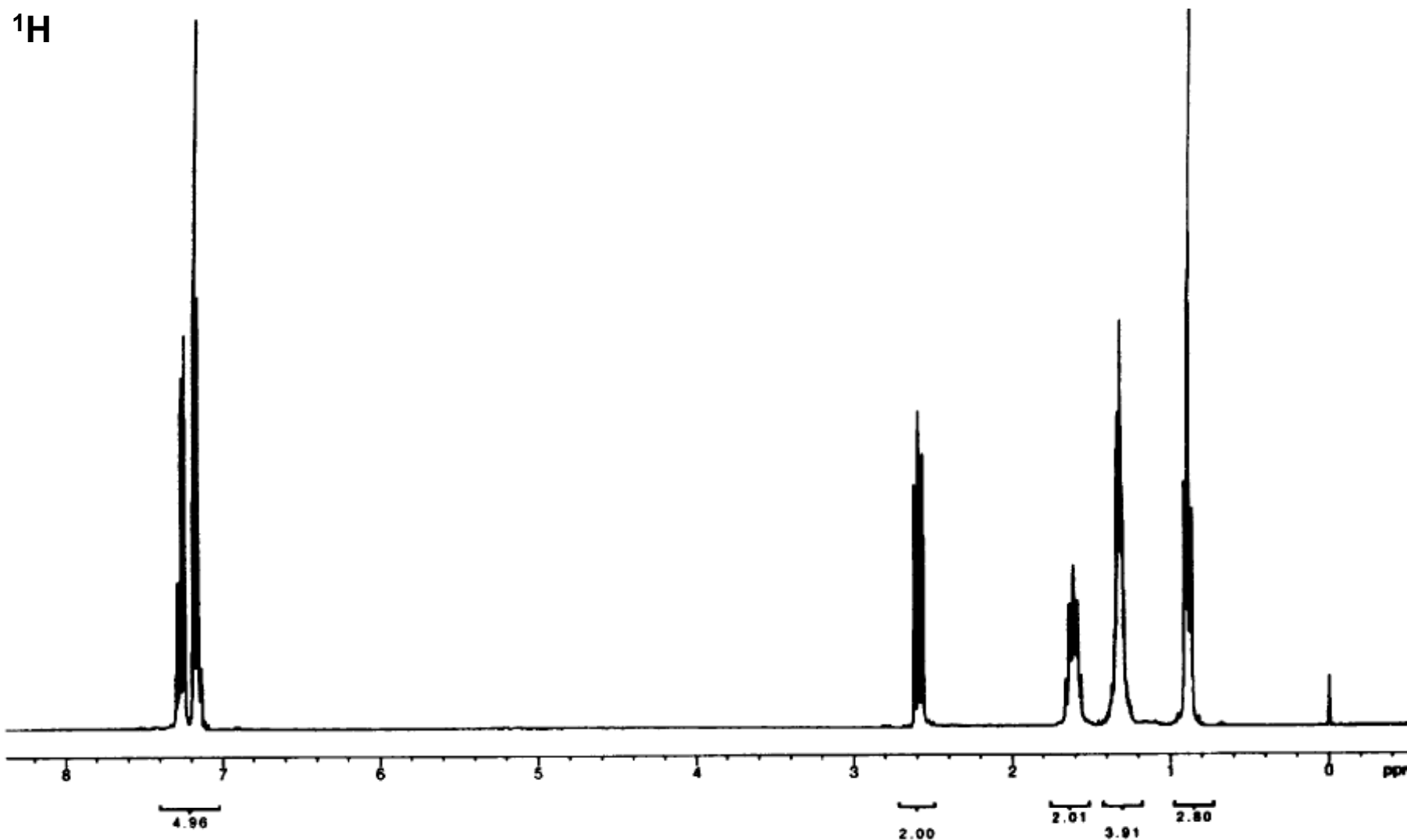
- za ovaj spoj: $\text{IHD} = [2 \cdot 14 + 2 - 20] / 2 = 5$

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

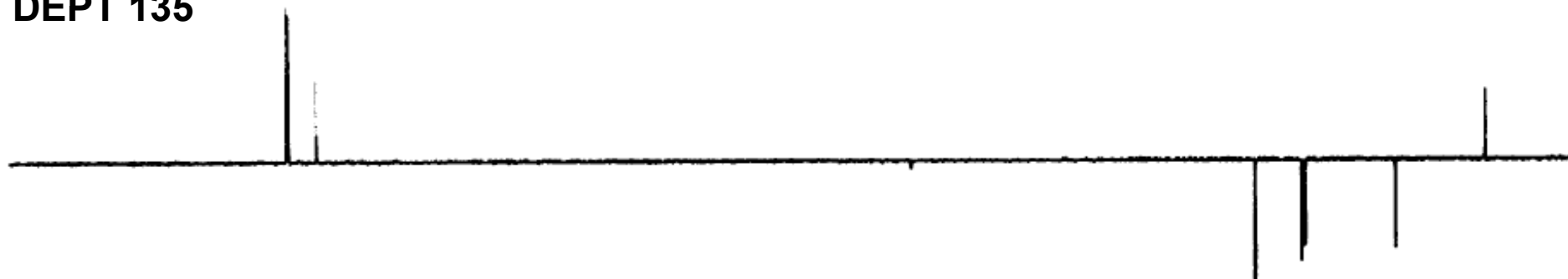


$\delta(^1\text{H})$ / ppm	multiplet	H-atom
0,95	t	–CH ₃
1,40	m (heptet)	–CH ₂ –
1,55	m (kvintet)	–CH ₂ –
1,95	m (kvintet)	–CH ₂ –
2,35	t	–CH ₂ –
2,65	t	–CH ₂ –
4,10	t	–CH ₂ –
	s	=CH–

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



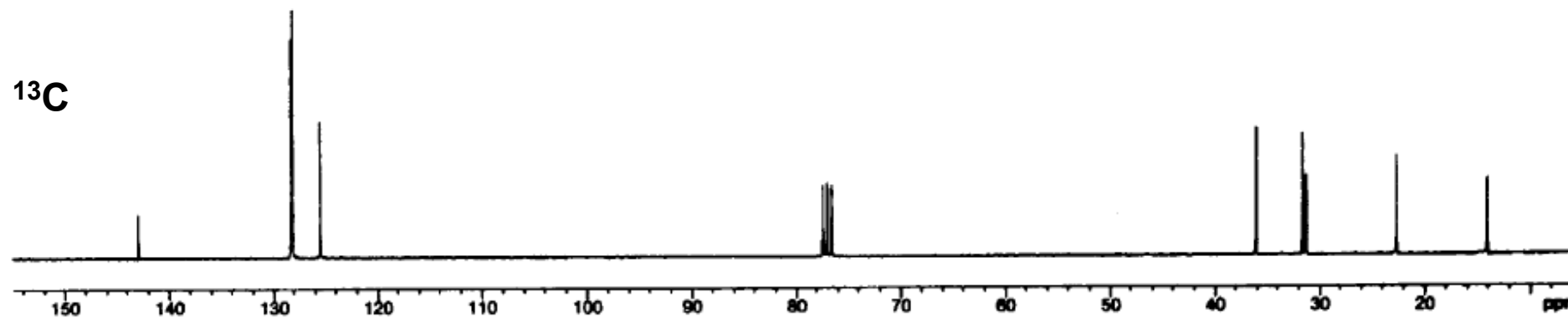
DEPT 135

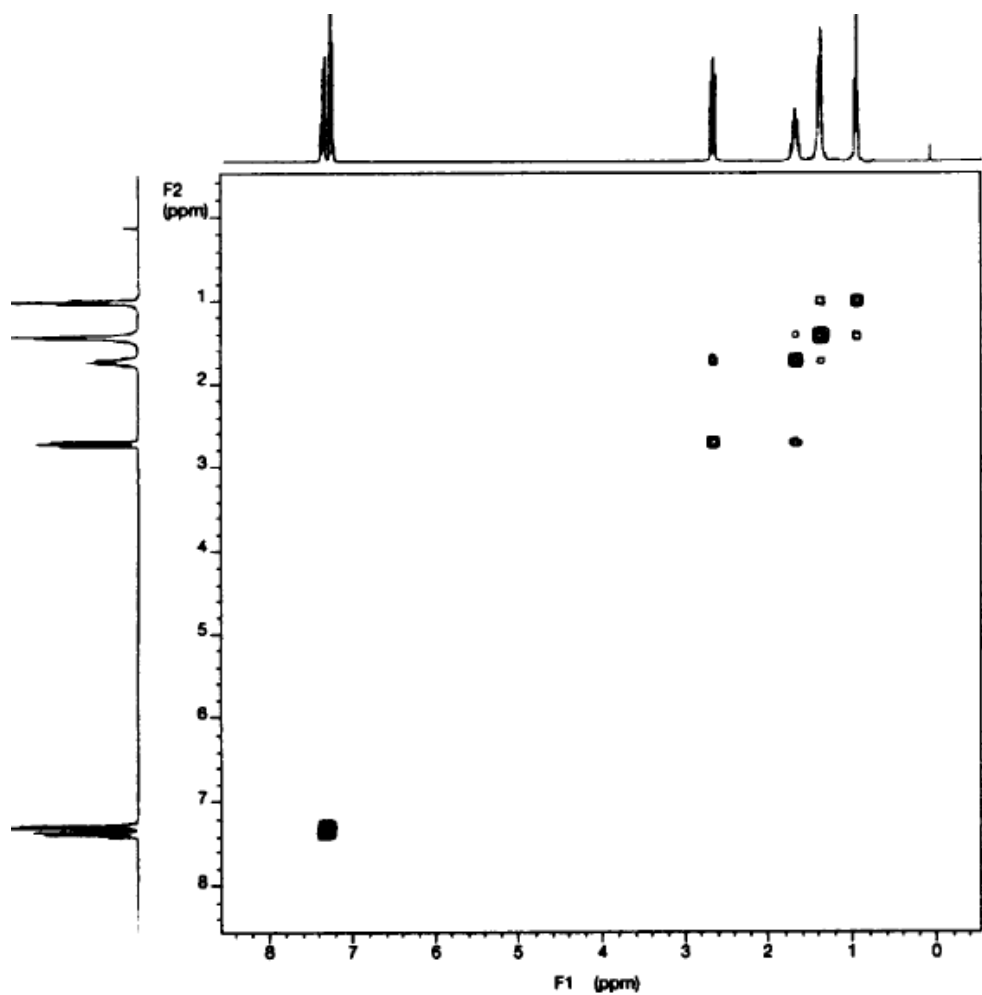


DEPT 90



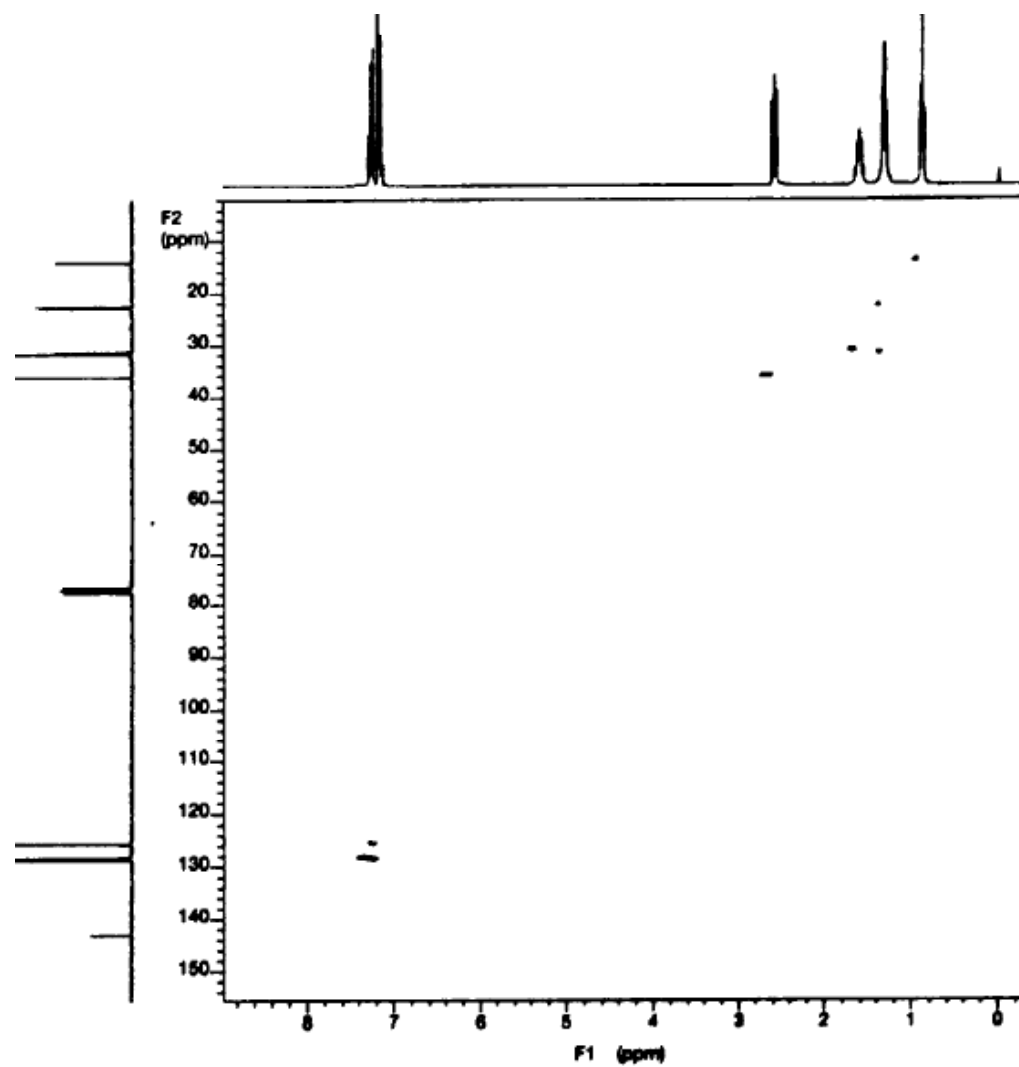
^{13}C



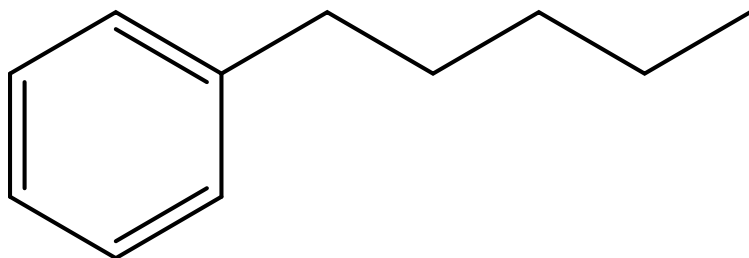


COSY

HETCOR



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



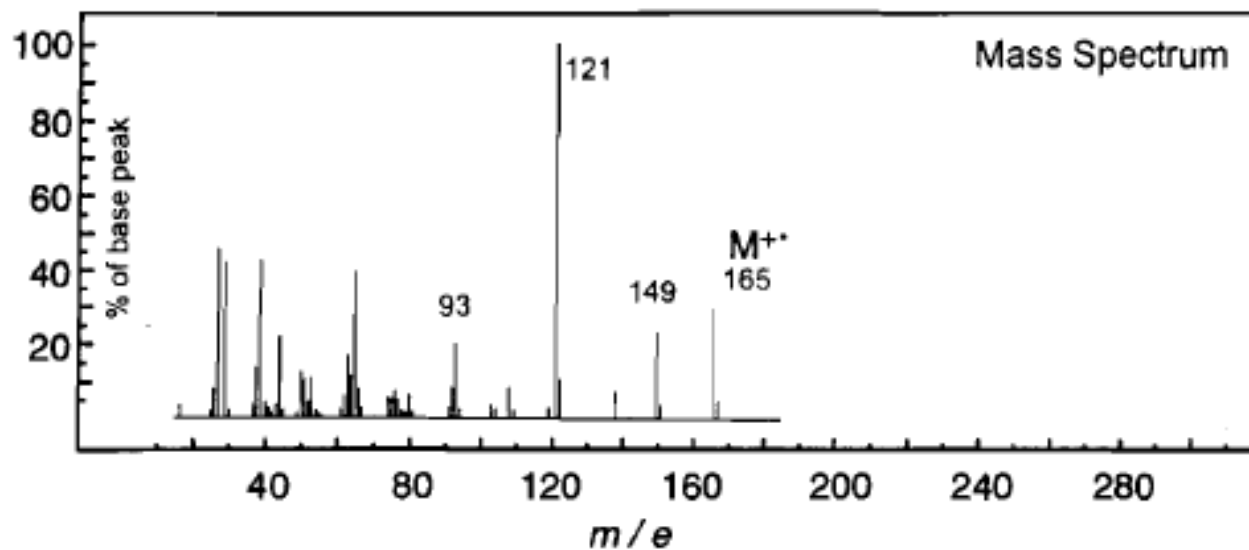
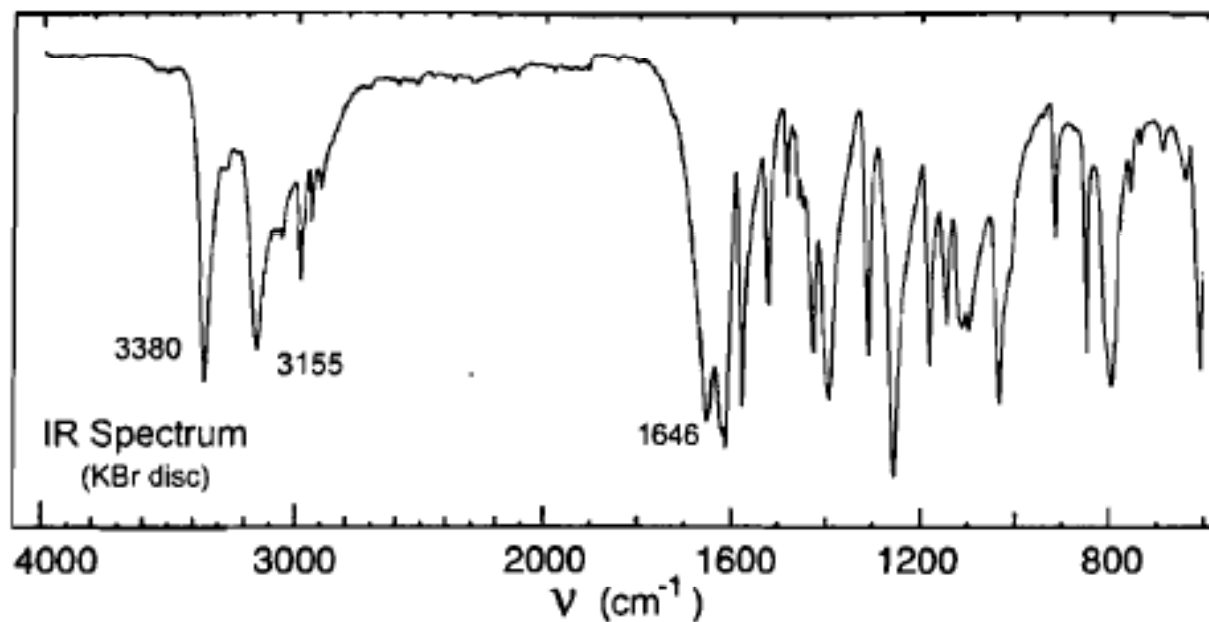
- informacije iz ^1H NMR:

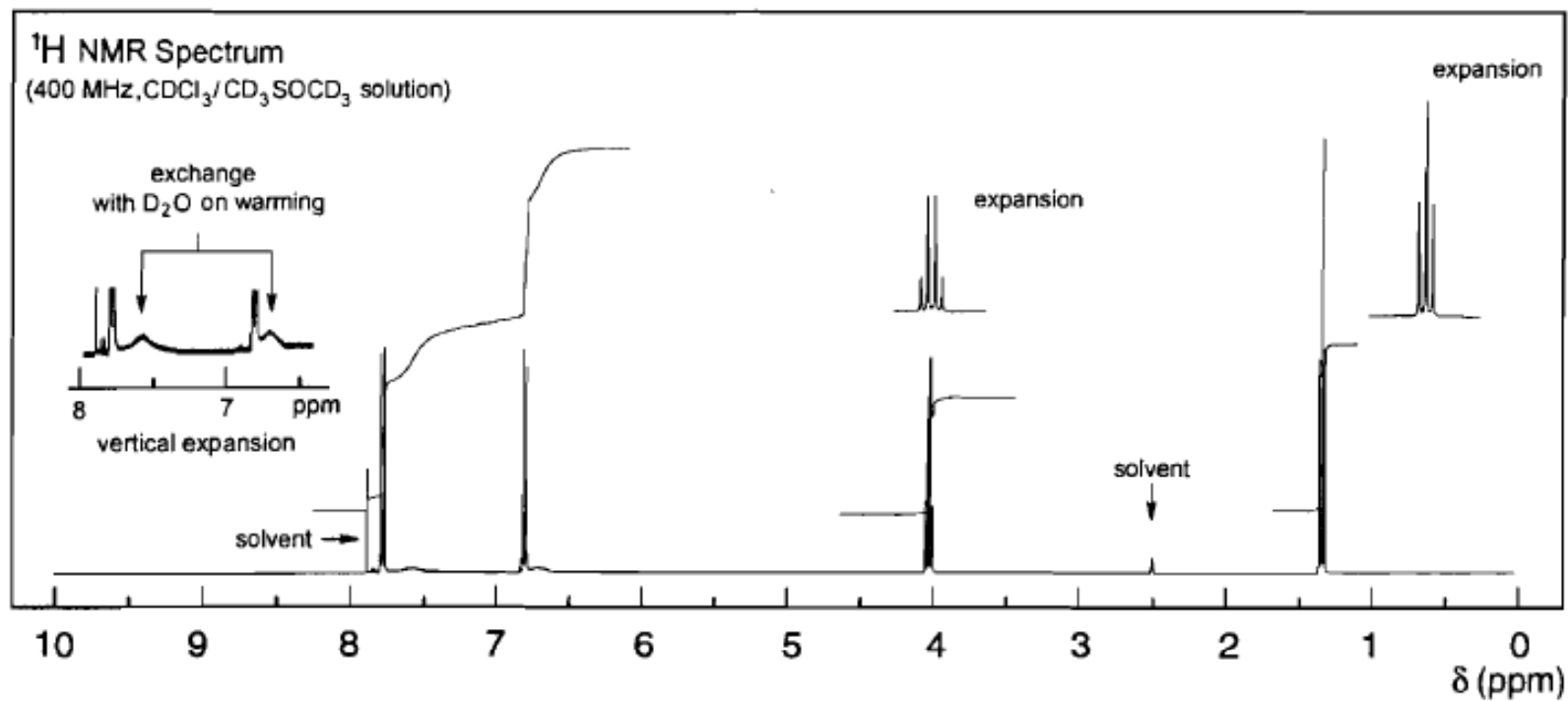
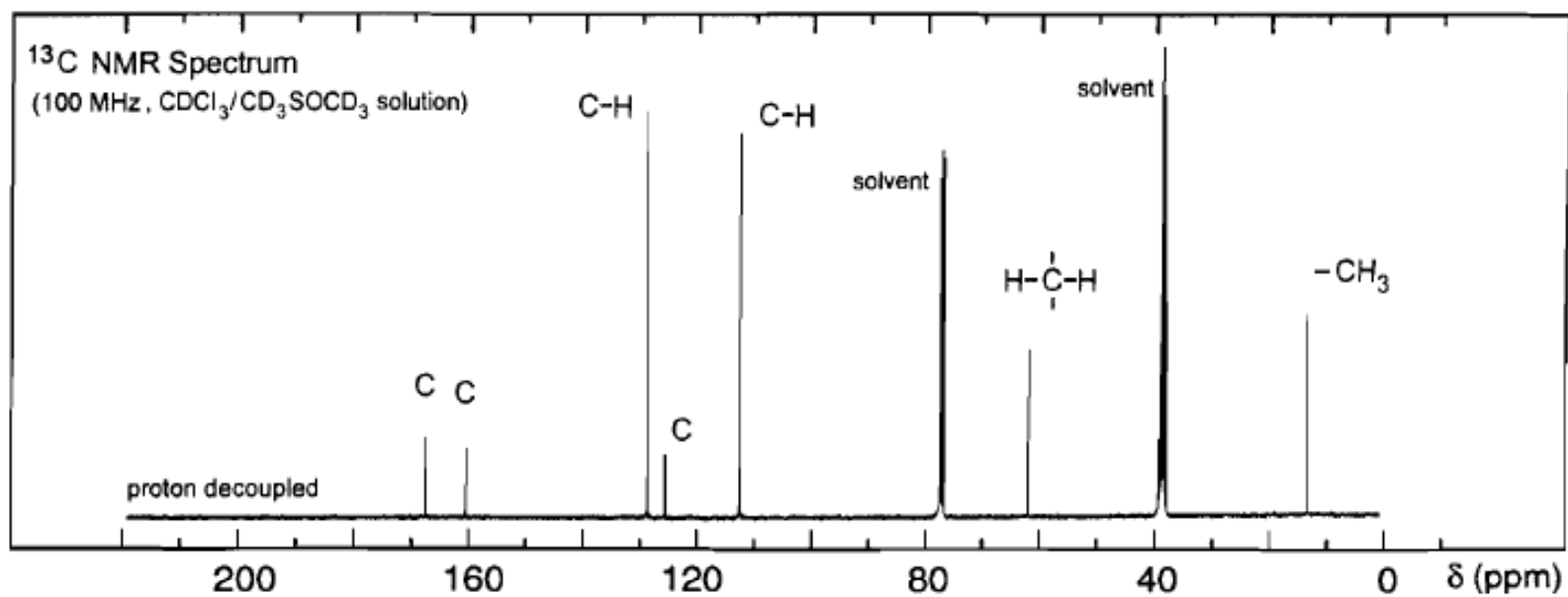
$\delta(^1\text{H}) / \text{ppm}$	multiplet	H-atom
0,9	t	$-\text{CH}_3$
1,3	m	$-\text{CH}_2-$, $-\text{CH}_2-$
1,6	m(kvintet)	$-\text{CH}_2-$
2,6	t	$-\text{CH}_2-$
7,2	m	$=\text{CH}-$
7,3	m	$=\text{CH}-$

- informacije iz ^{13}C NMR:

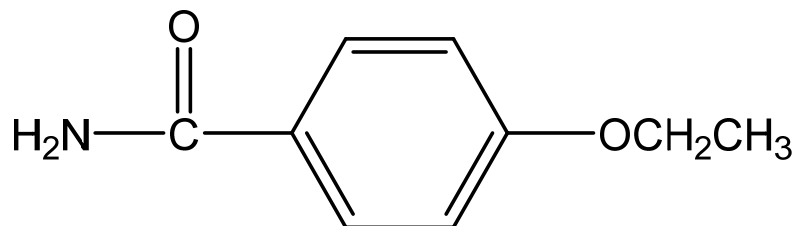
$\delta(^{13}\text{C}) / \text{ppm}$	C-atom
14	$-\text{CH}_3$
23	$-\text{CH}_2-$
31	$-\text{CH}_2-$
32	$-\text{CH}_2-$
36	$-\text{CH}_2-$
125	$=\text{CH}-$
128	$=\text{CH}-$
143	C

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

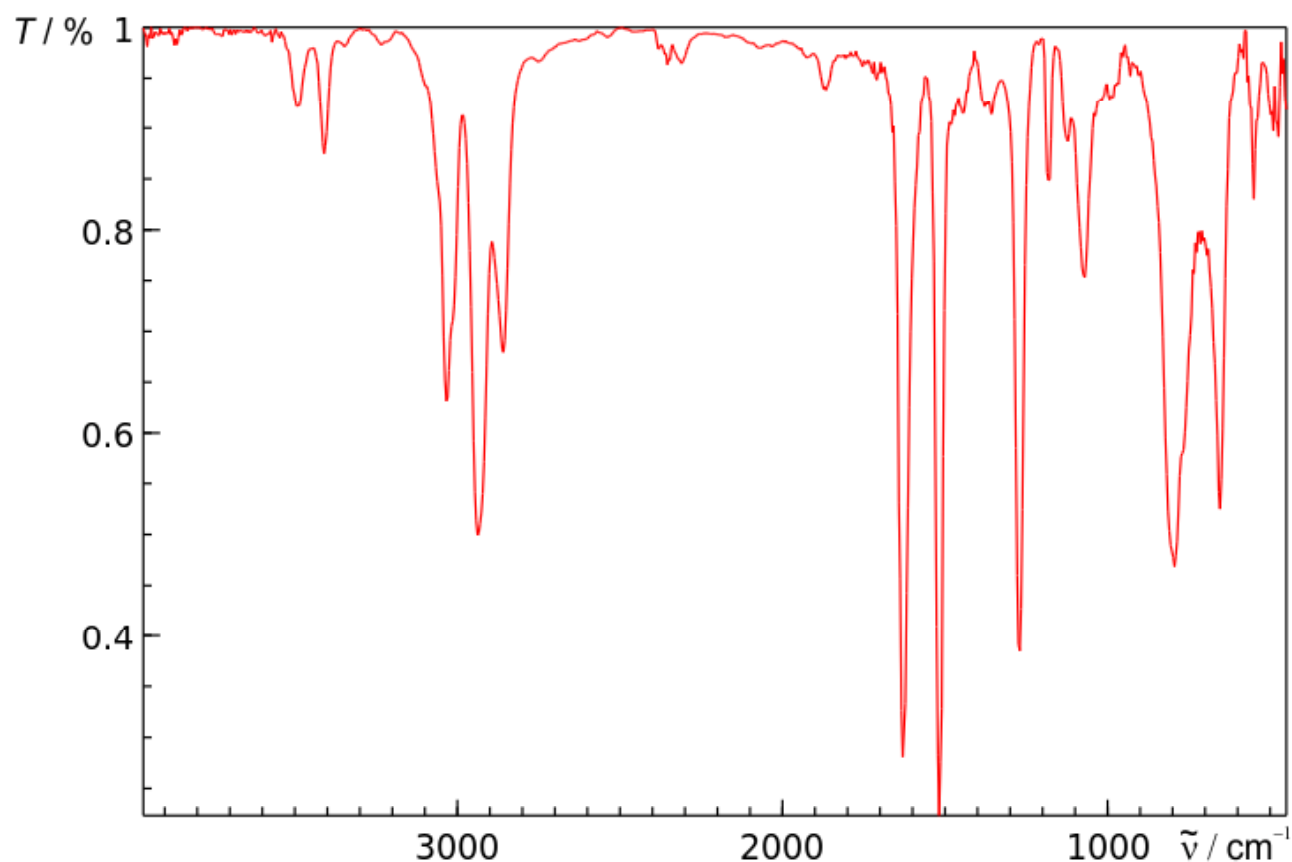
- informacije iz ^1H NMR:

$\delta(^1\text{H}) / \text{ppm}$	multiplet	H-atom
1,4	t	–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–

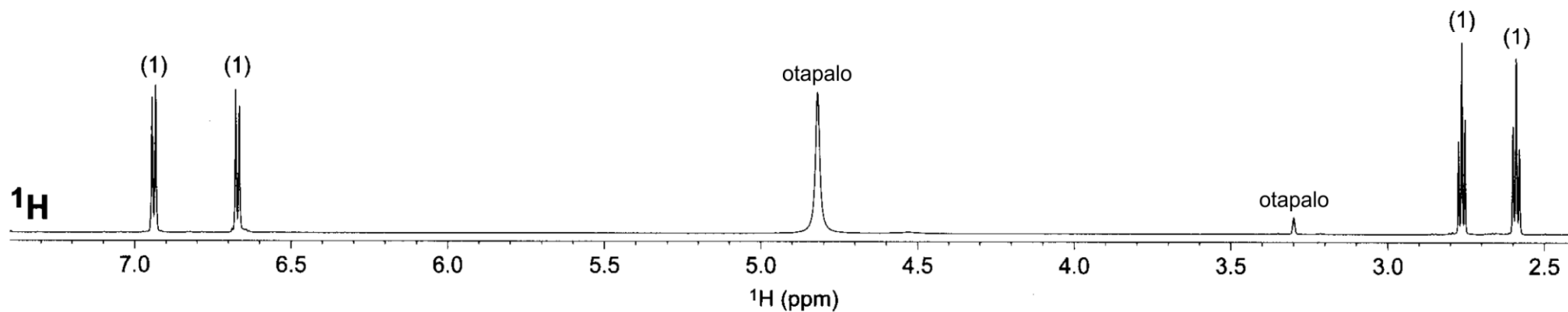
- informacije iz ^{13}C NMR:

$\delta(^{13}\text{C}) / \text{ppm}$	C-atom
12	–CH ₃
62	–CH ₂ –
110	=CH–
126	C
129	=CH–
160	C
168	C=O

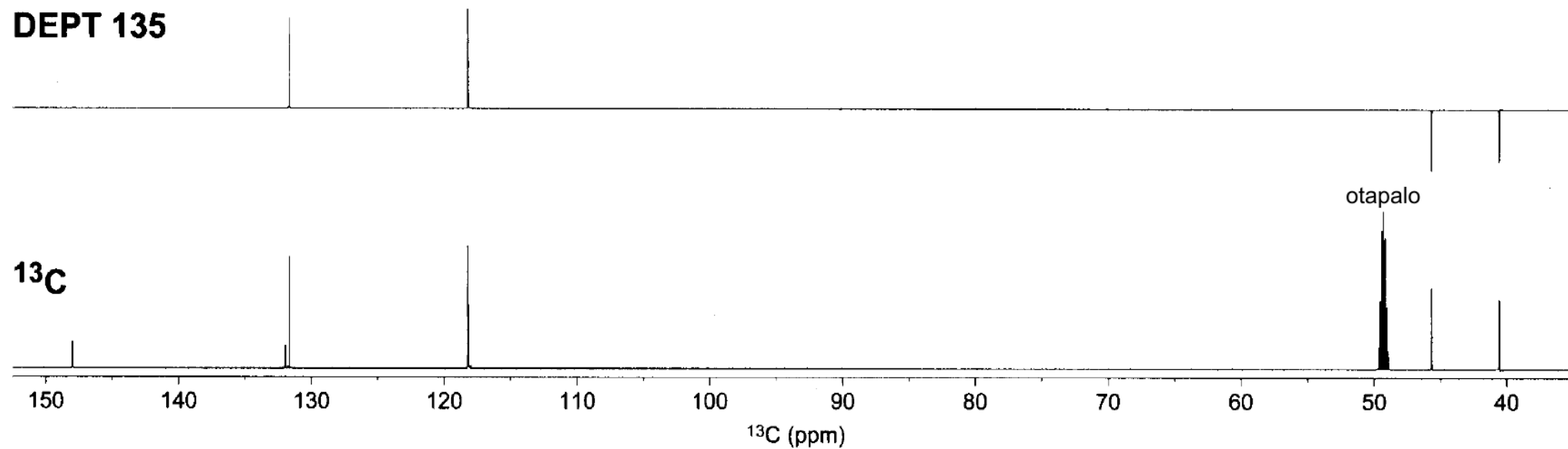
4. Odredite strukturu spoja molarne mase 136 g mol^{-1} na temelju njegovih IR, ^1H NMR ^{13}C NMR, COSY, HSQC i HMBC spektara.



IR spektar spoja snimljen tehnikom KBr pastile.

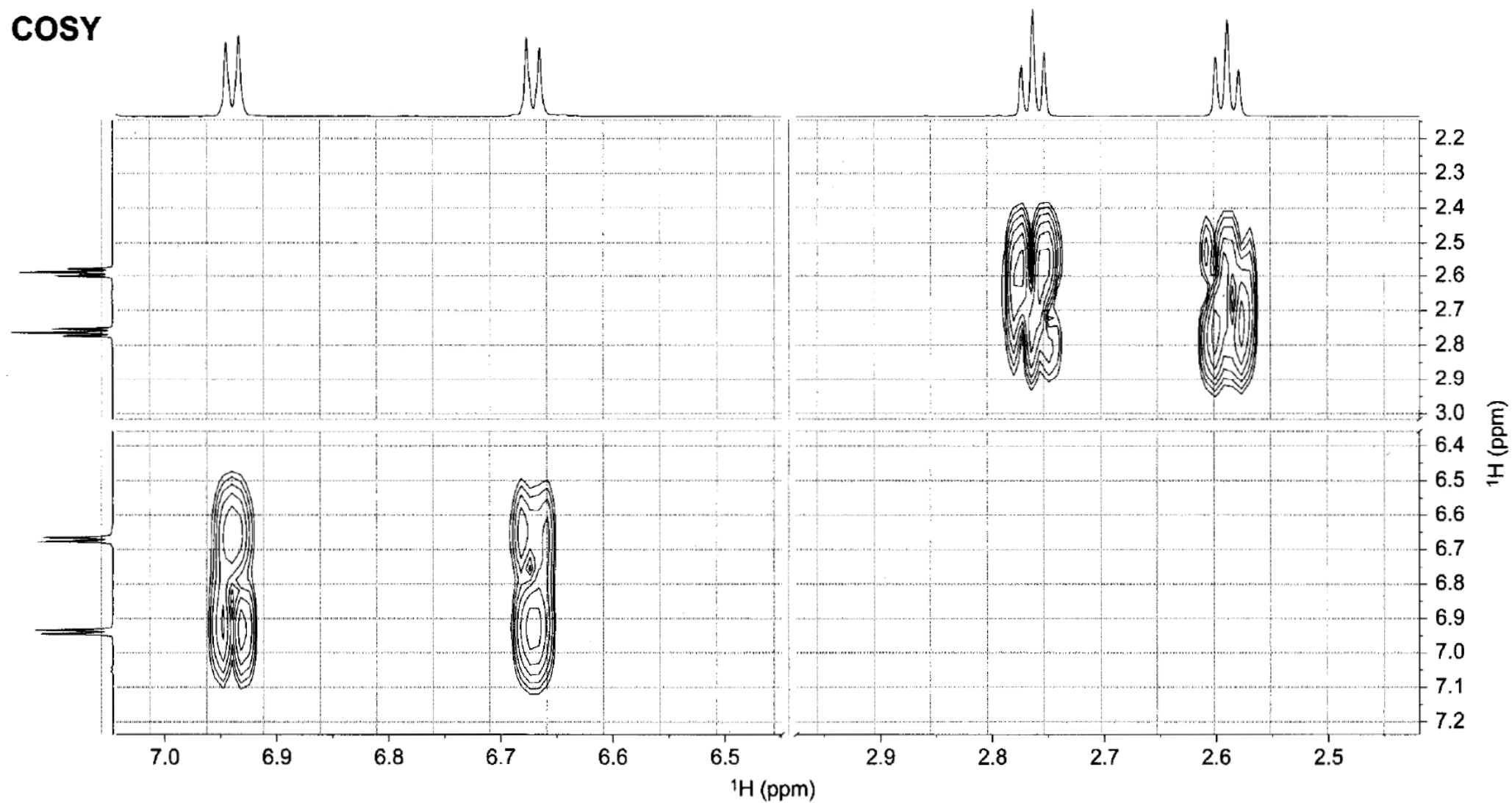


^1H NMR spektar spoja snimljen u CD_3OD . Spoj sadrži i protone koji se izmijene u CD_3OD .



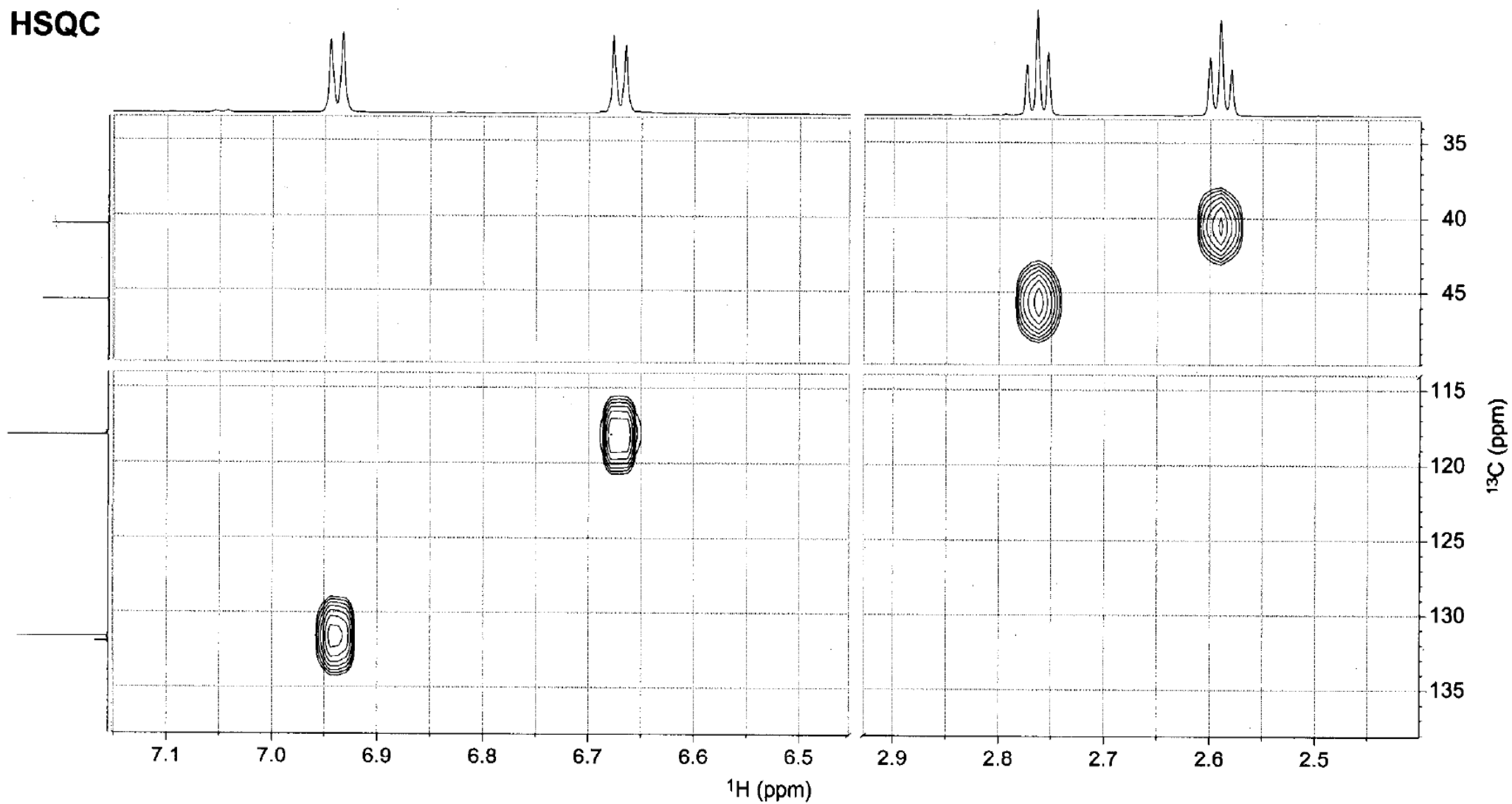
^{13}C NMR spektri spoja snimljeni u CD_3OD .

COSY



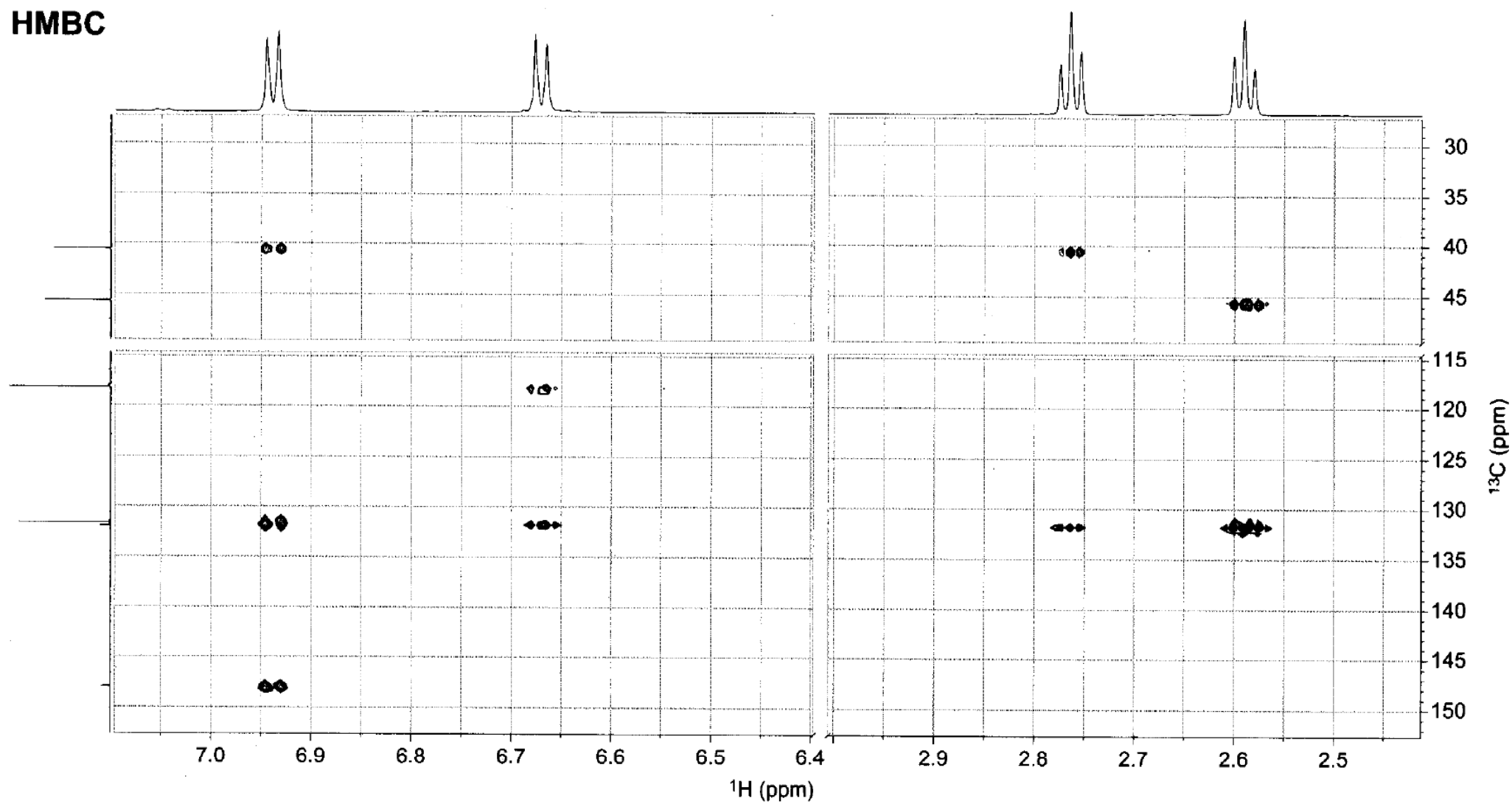
COSY spektar spoja snimljen u CD_3OD .

HSQC



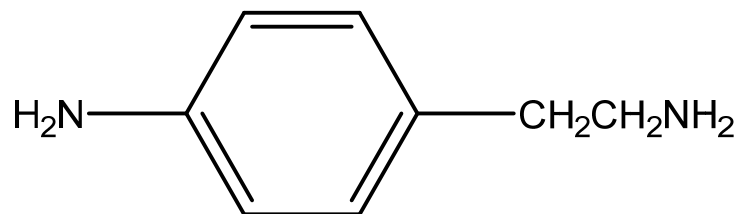
HSQC spektar spoja snimljen u CD_3OD .

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	$=\text{CH}-$
6,95	d	$=\text{CH}-$

- 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

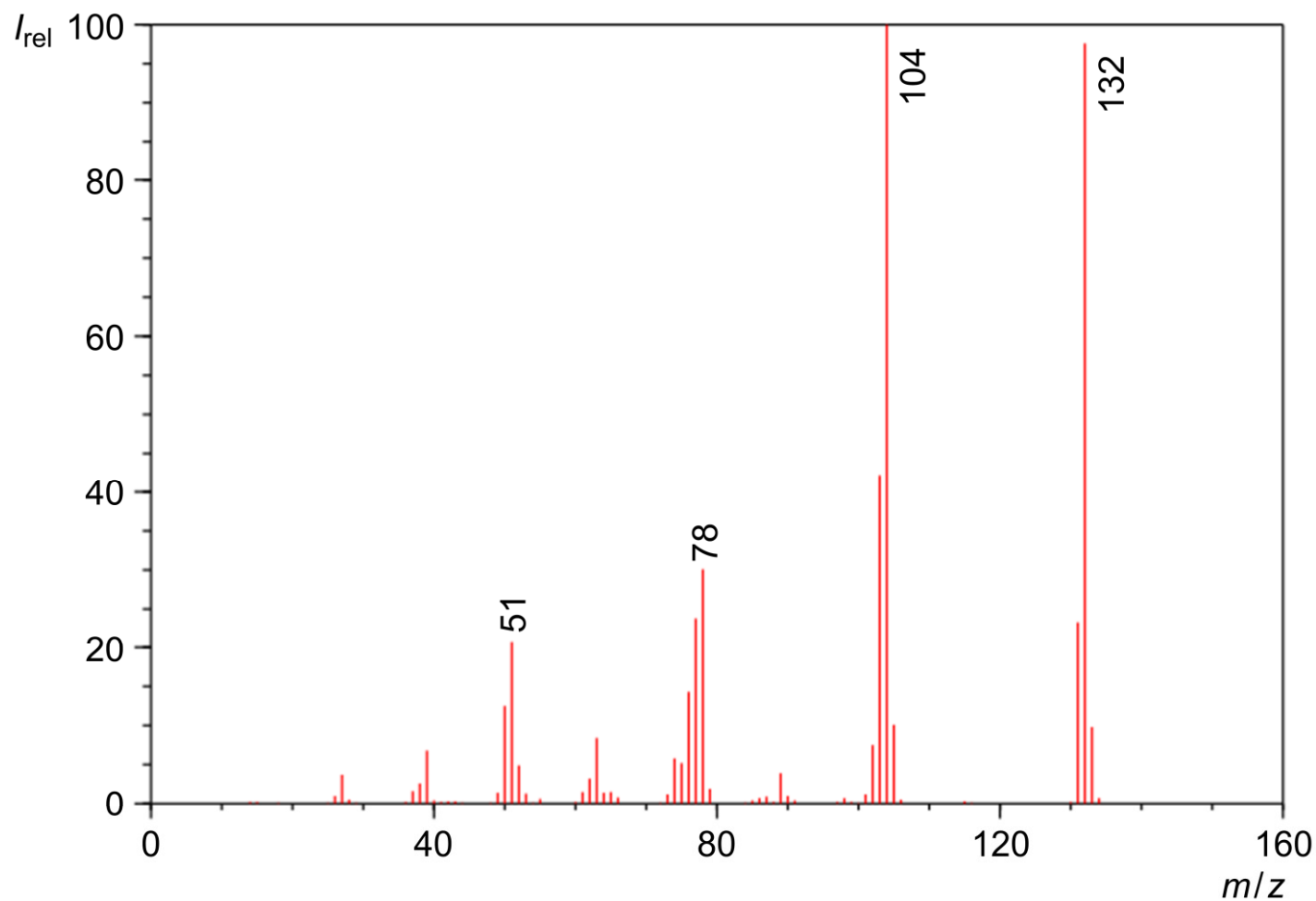
1500 i 1400 cm^{-1} C–C istežanja

800 cm^{-1} C–H aromatsko svijanje

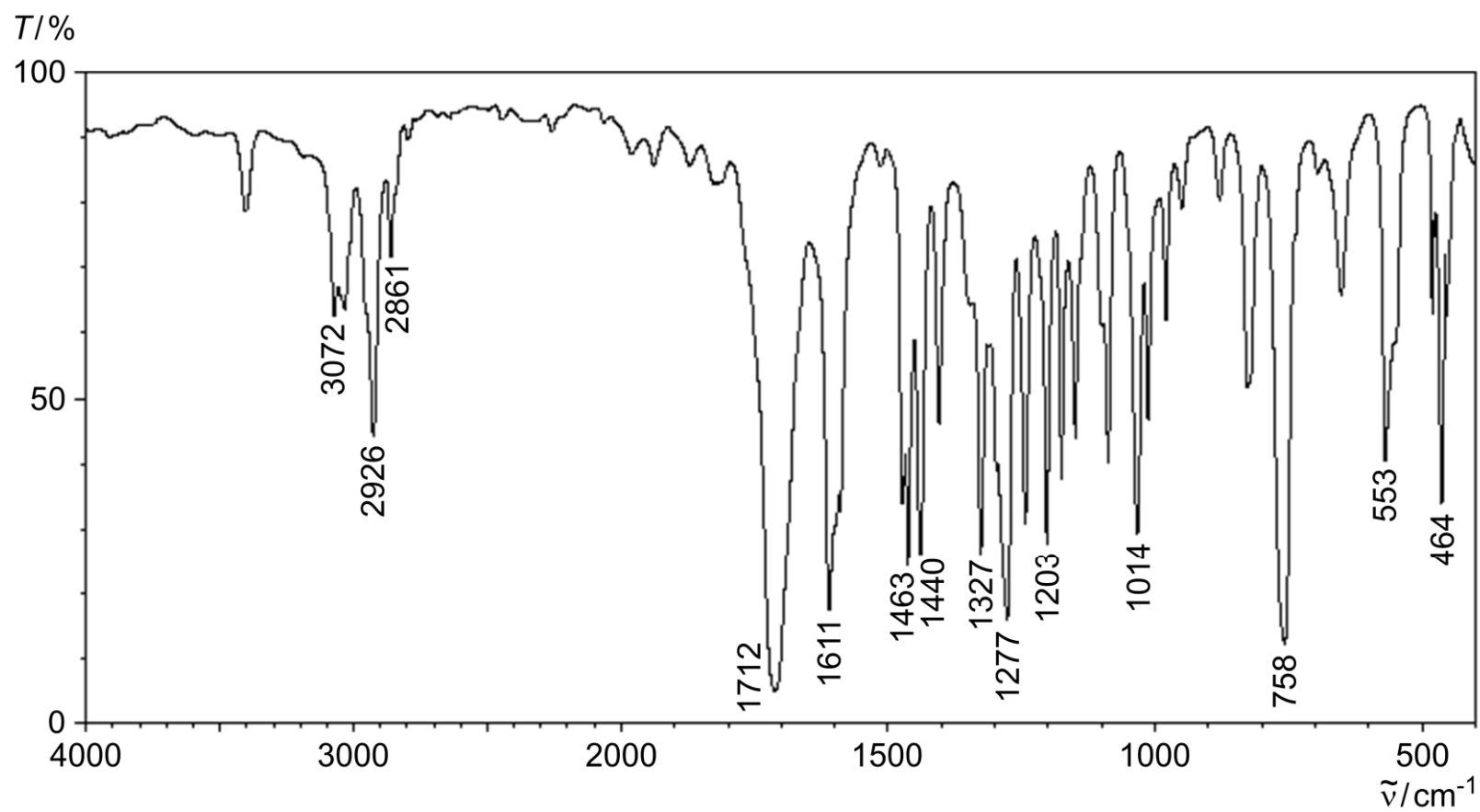
- informacije iz ^{13}C NMR:

$\delta(^{13}\text{C}) / \text{ppm}$	C-atom
41	$-\text{CH}_2-$
46	$-\text{CH}_2-$
118	$=\text{CH}-$
131	$=\text{CH}-$
131,5	C
148	C

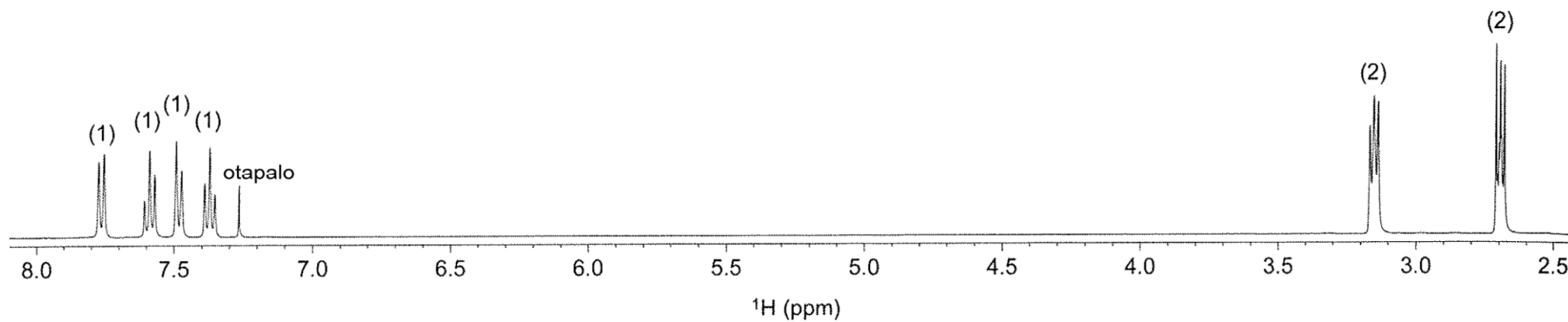
5. Odredite strukturu spoja na temelju njegovih MS, IR, ^1H NMR, ^{13}C NMR, COSY, HSQC i HMBC spektara.



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

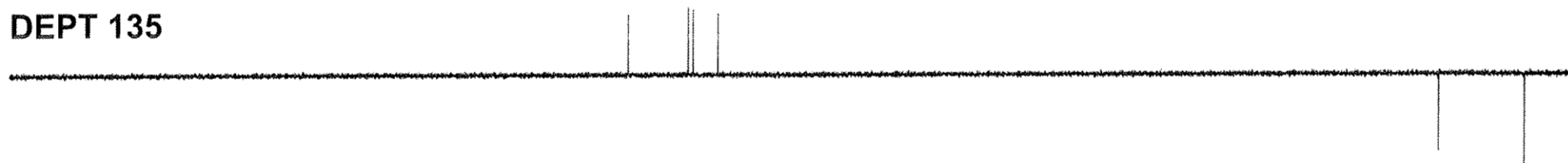


IR spektar spoja snimljen tehnikom KBr pastile.

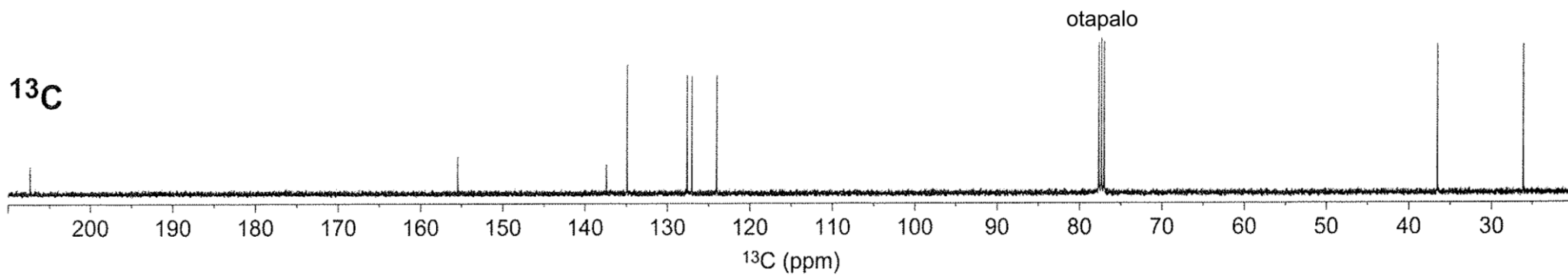


^1H NMR spektar spoja snimljen u CDCl_3 .

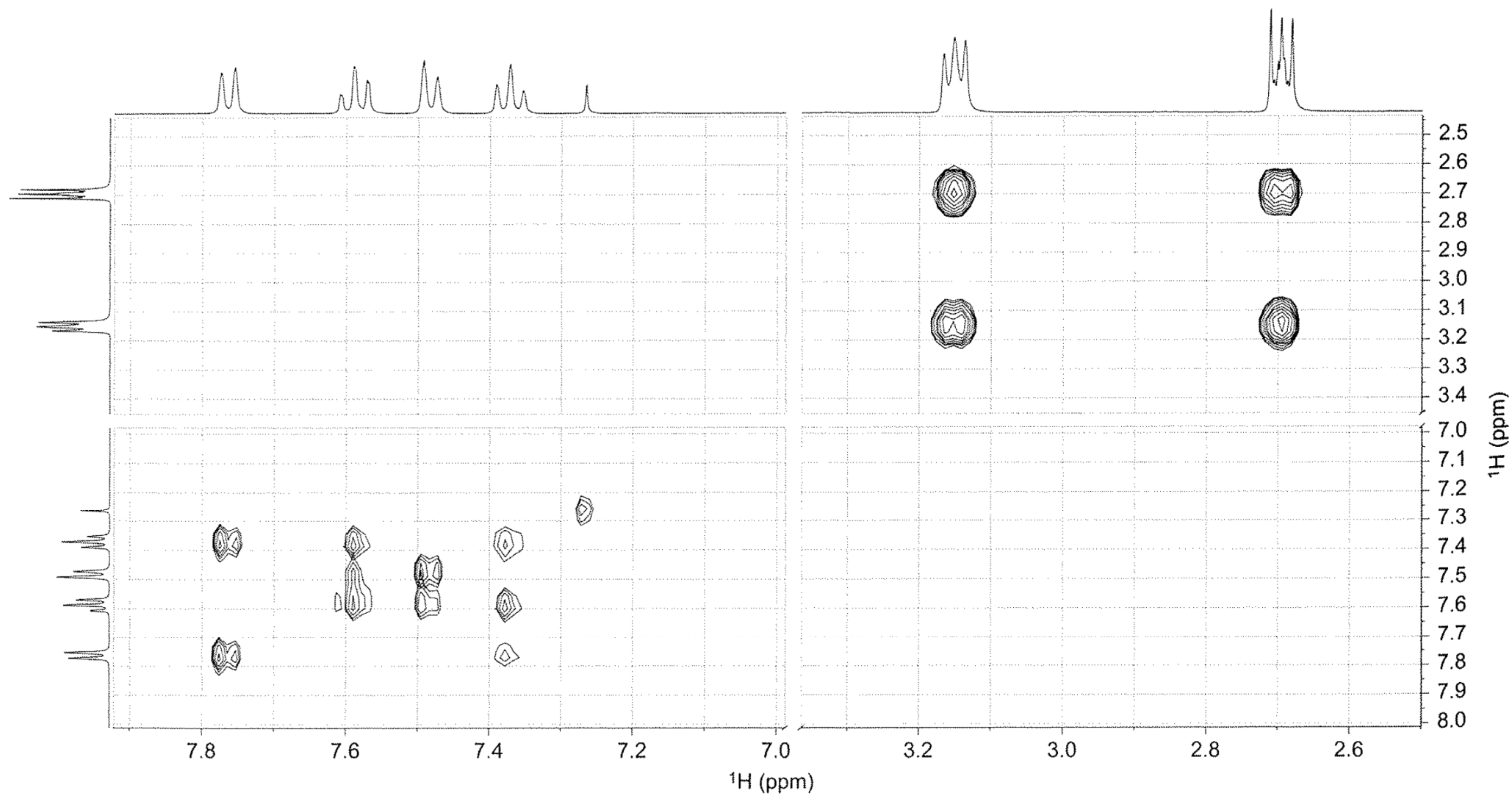
DEPT 135



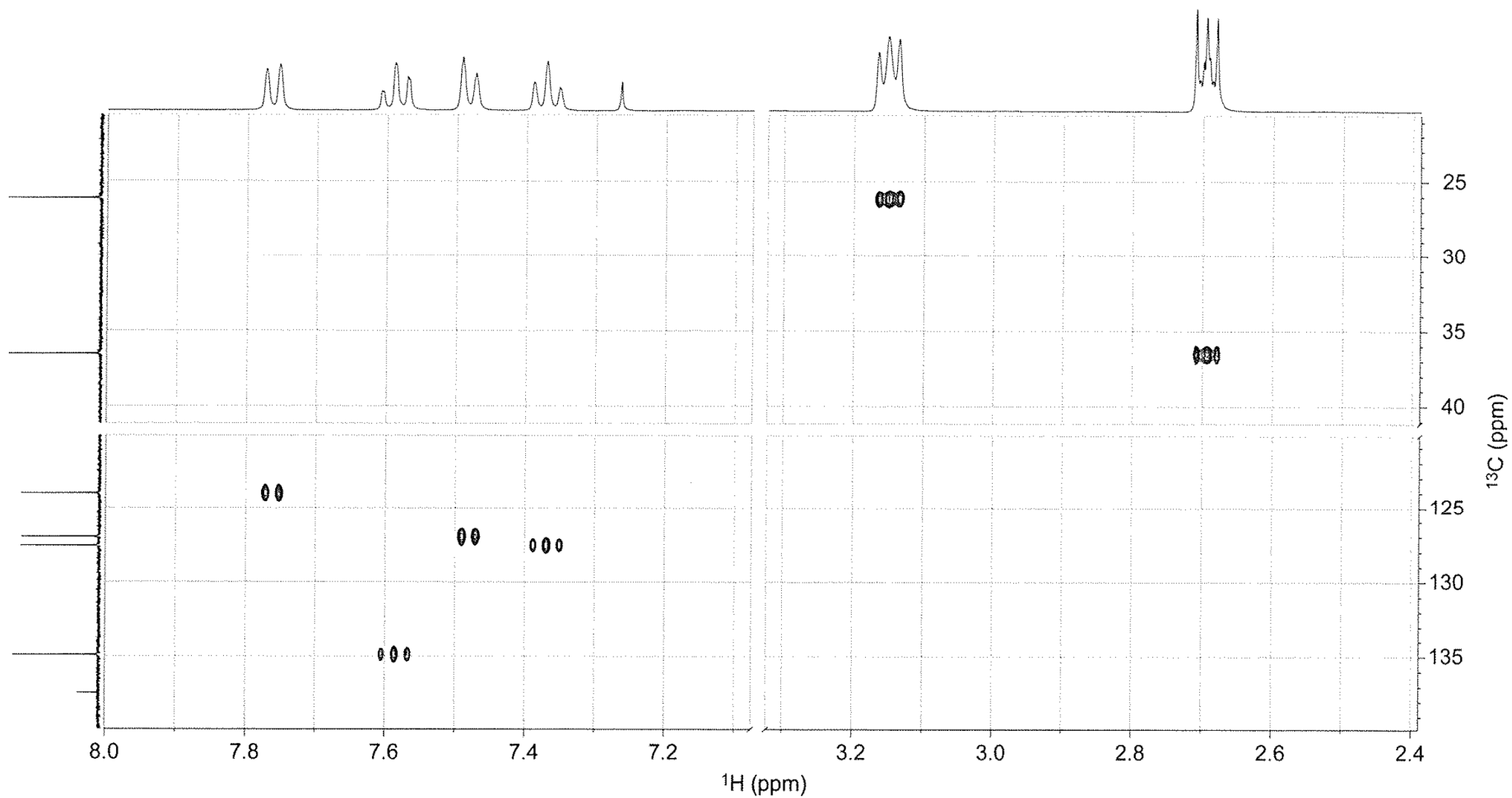
^{13}C



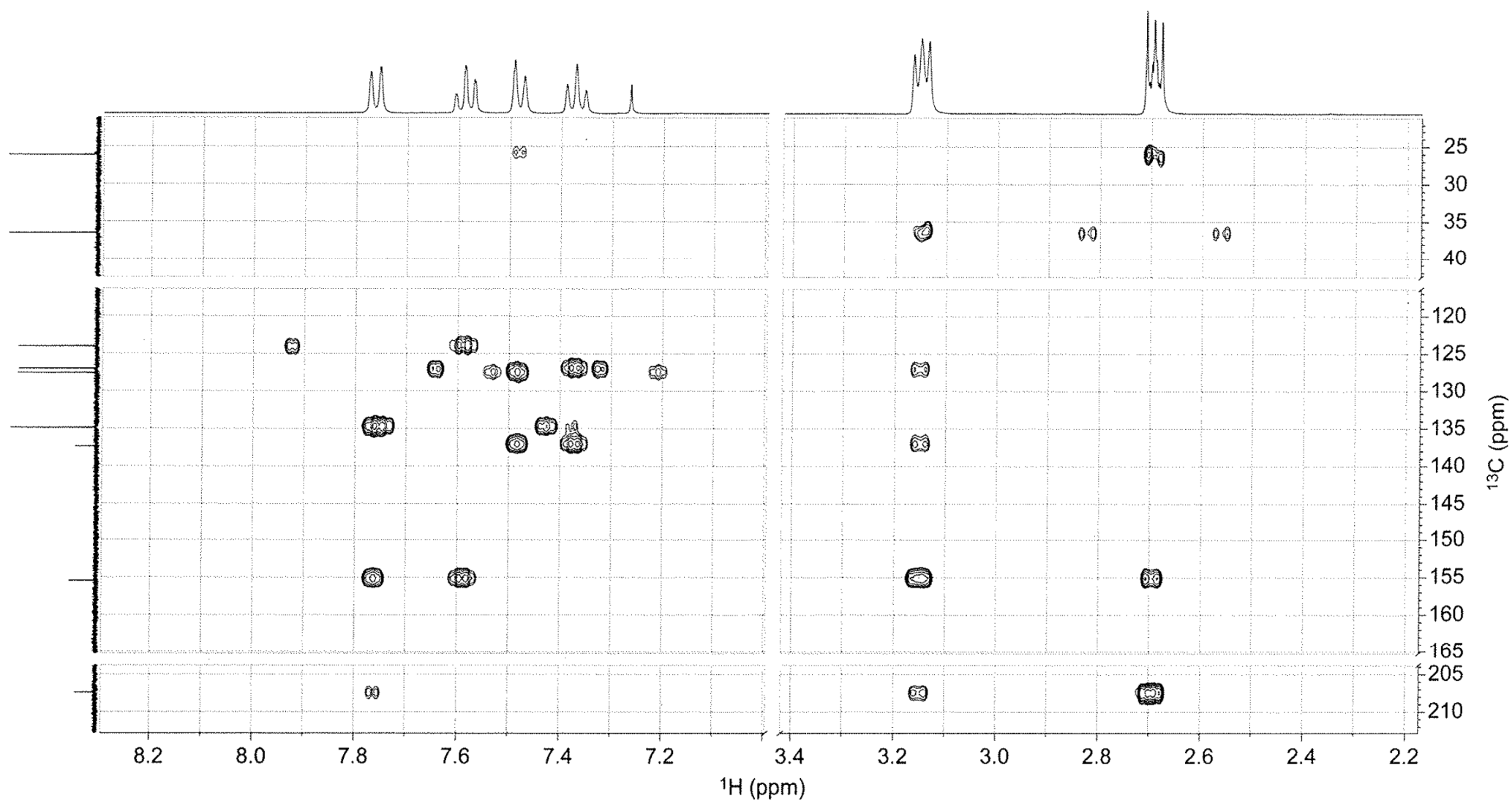
^{13}C NMR spektri spoja snimljeni u CDCl_3 .



COSY spektar spoja snimljen u CDCl_3 .



HSQC spektar spoja snimljen u CDCl_3 .

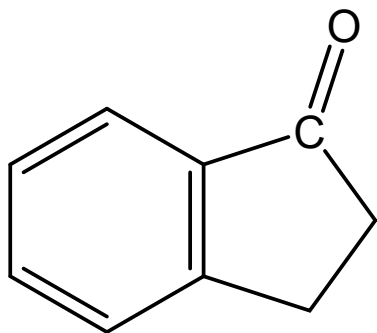


HMBC spektar spoja snimljen u CDCl_3 .

- informacije iz MS:

bazni pik: 104

$M^+ = 132$



- informacije iz ^1H NMR:

$\delta(^1\text{H}) / \text{ppm}$	multiplet	H-atom
2,70	t	$-\text{CH}_2-$
3,15	t	$-\text{CH}_2-$
7,37	t	$=\text{CH}-$
7,48	d	$=\text{CH}-$
7,59	t	$=\text{CH}-$
7,77	d	$=\text{CH}-$

- informacije iz IR:

3072 cm^{-1} aromatsko C–H istežanje

2926 i 2861 cm^{-1} alifatsko C–H istežanje

1712 cm^{-1} C=O istežanje

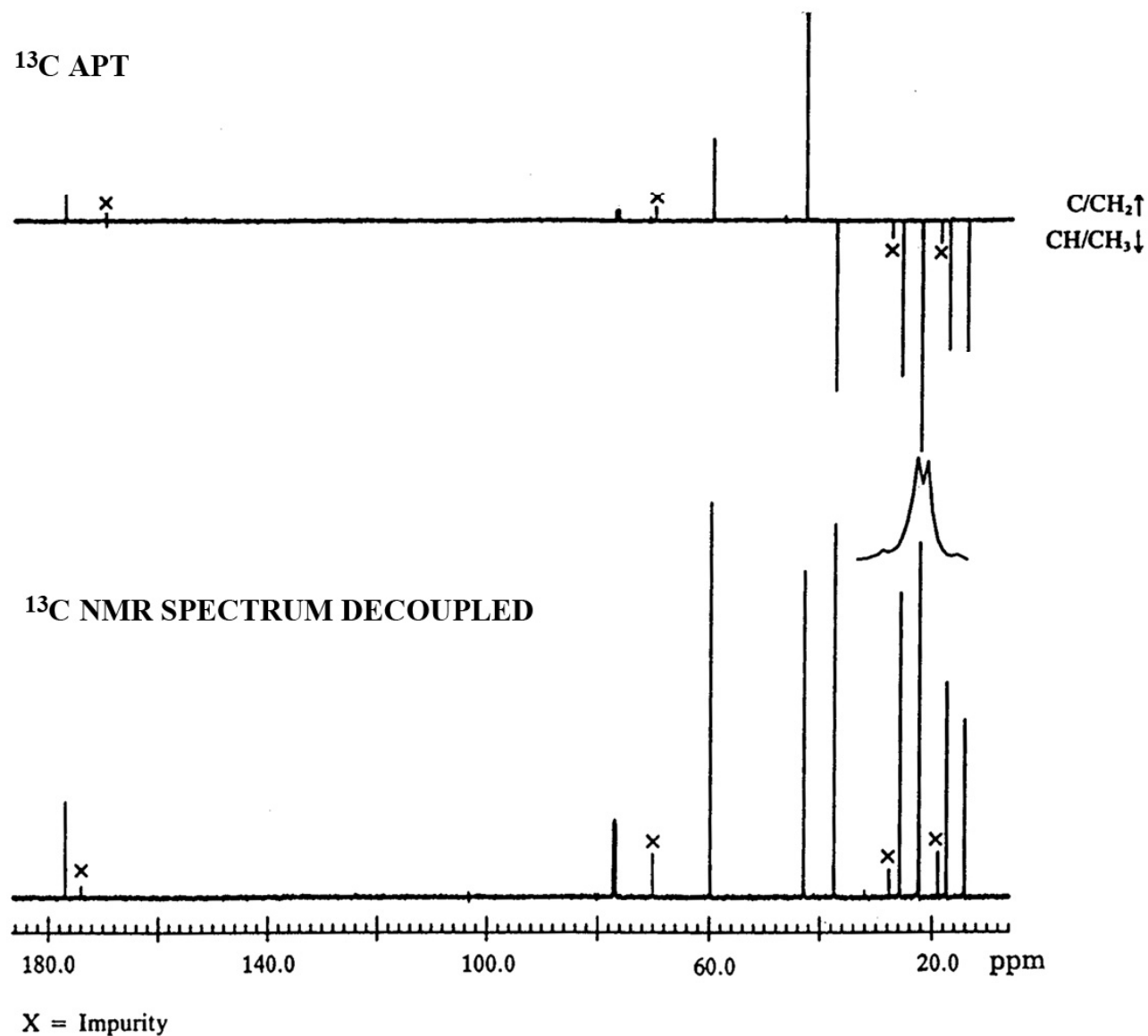
1611 , 1463 i 1440 cm^{-1} aromatsko C=C istežanje

758 , 553 i 464 cm^{-1} aromatsko C–H svijanje

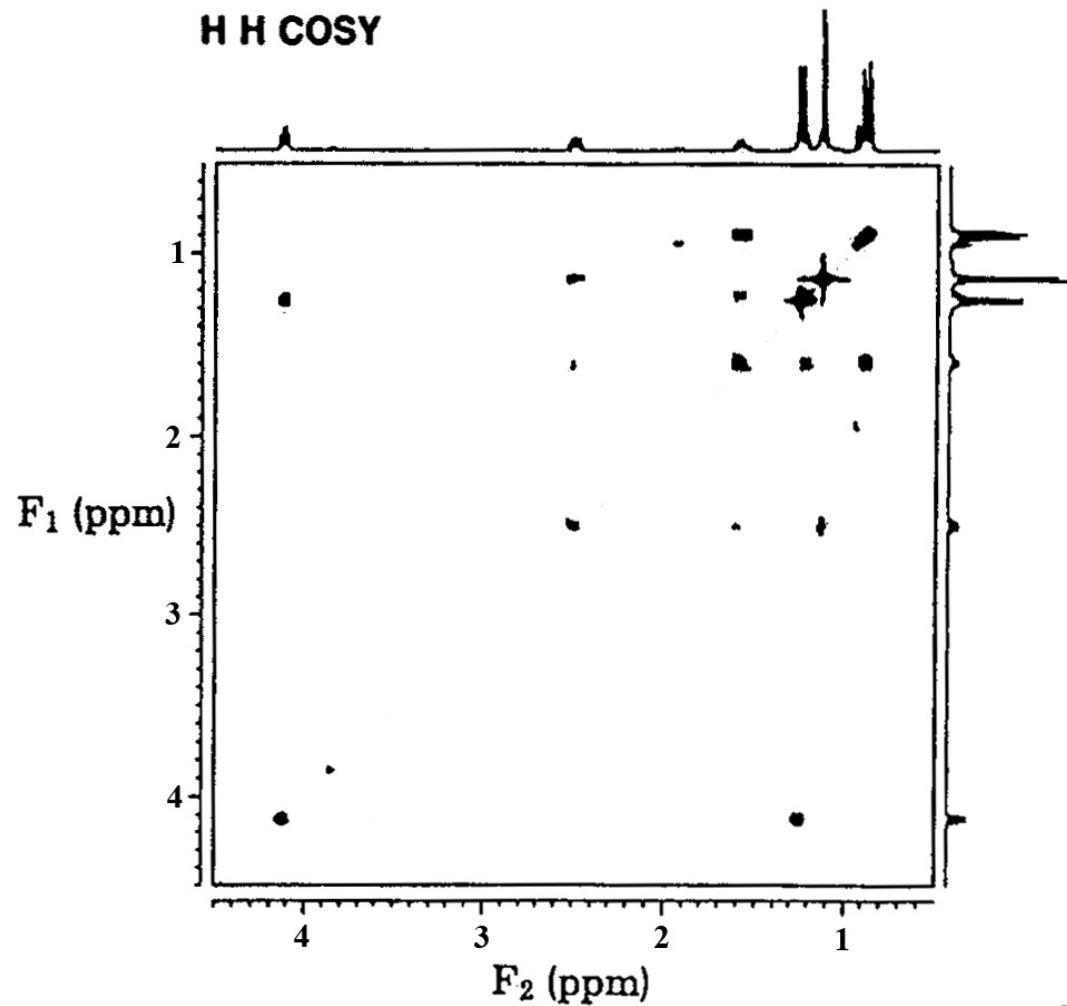
- informacije iz ^{13}C NMR:

$\delta(^{13}\text{C}) / \text{ppm}$	C-atom
26	$-\text{CH}_2-$
36	$-\text{CH}_2-$
124	$=\text{CH}-$
127	$=\text{CH}-$
128	$=\text{CH}-$
135	$=\text{CH}-$
137	C
156	C
207	C=O

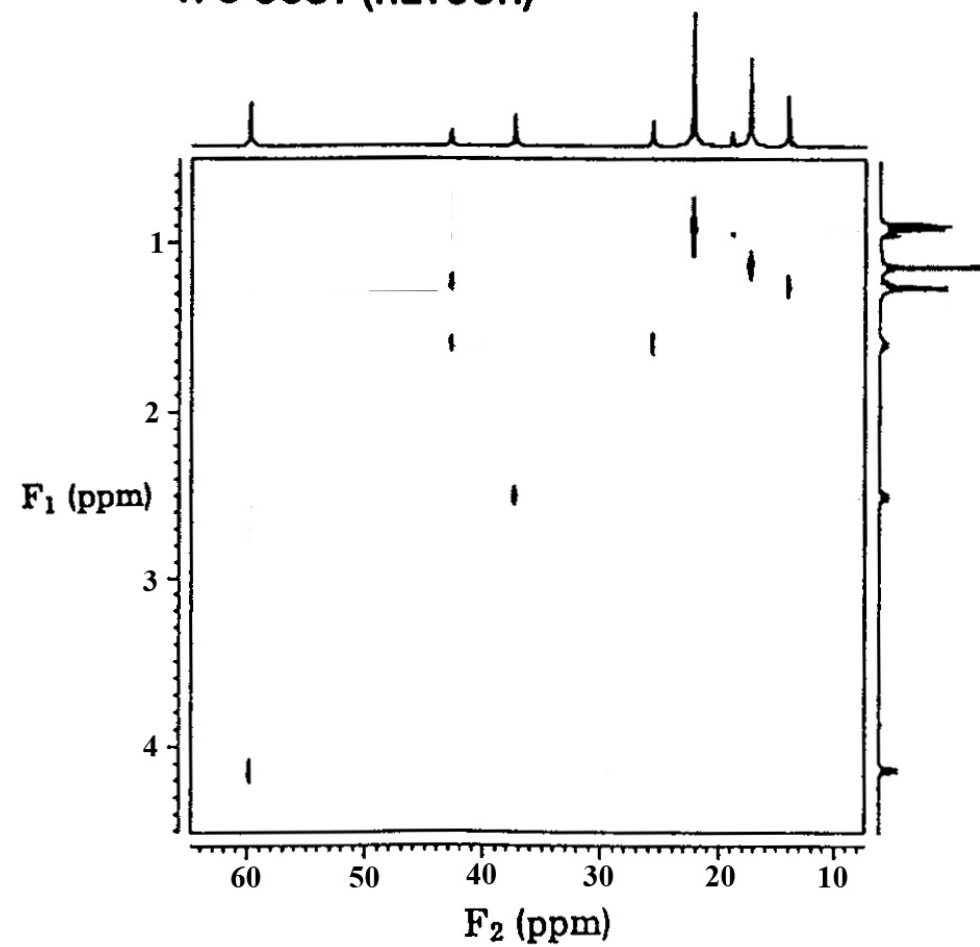
6. Odredite strukturu spoja molekulske formule $C_9H_{18}O_2$ na temelju njegovih ^{13}C NMR, COSY i HETCOR spektara.

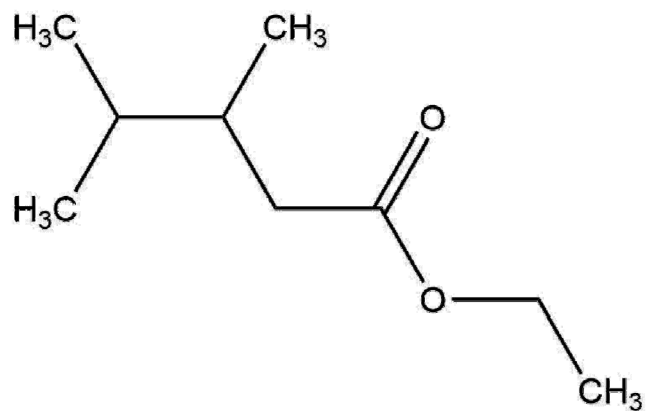


H H COSY



H C COSY (HETCOR)





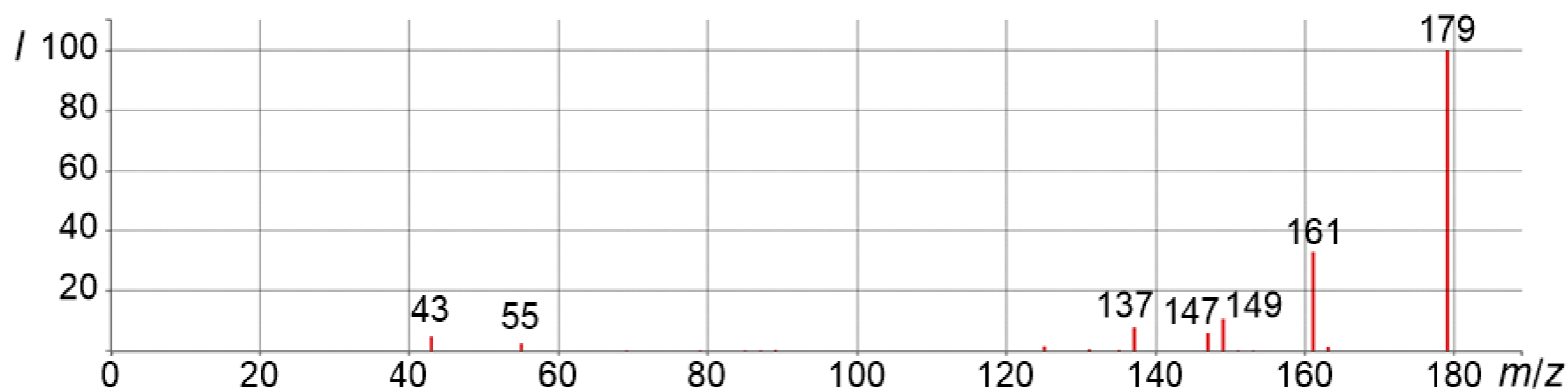
- informacije iz ^1H NMR:

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

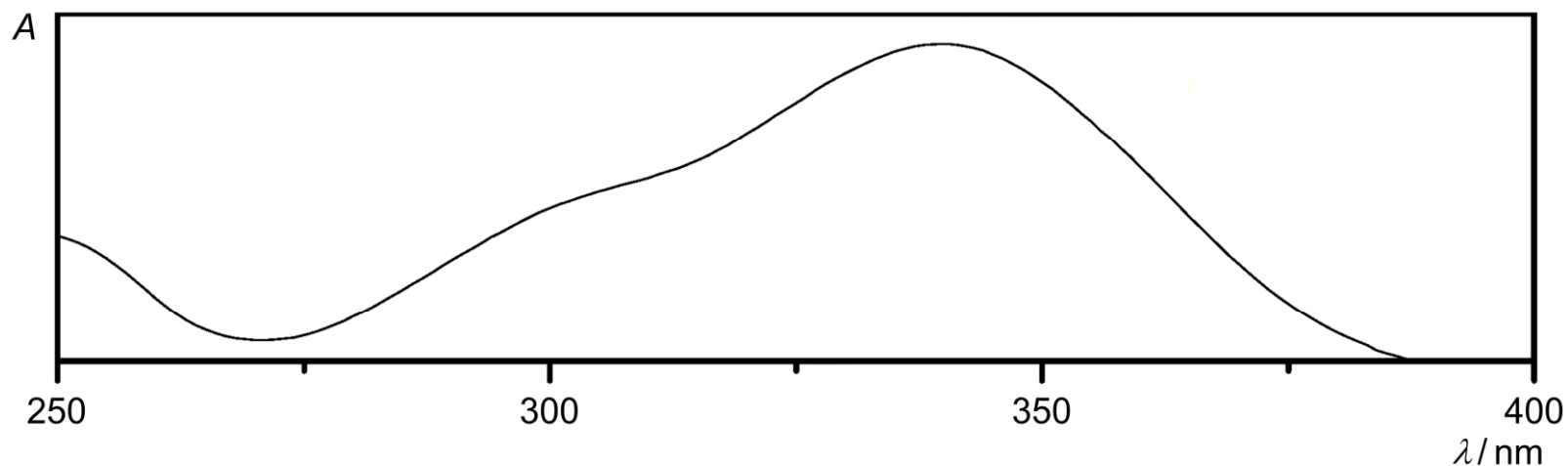
- informacije iz ^{13}C NMR:

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

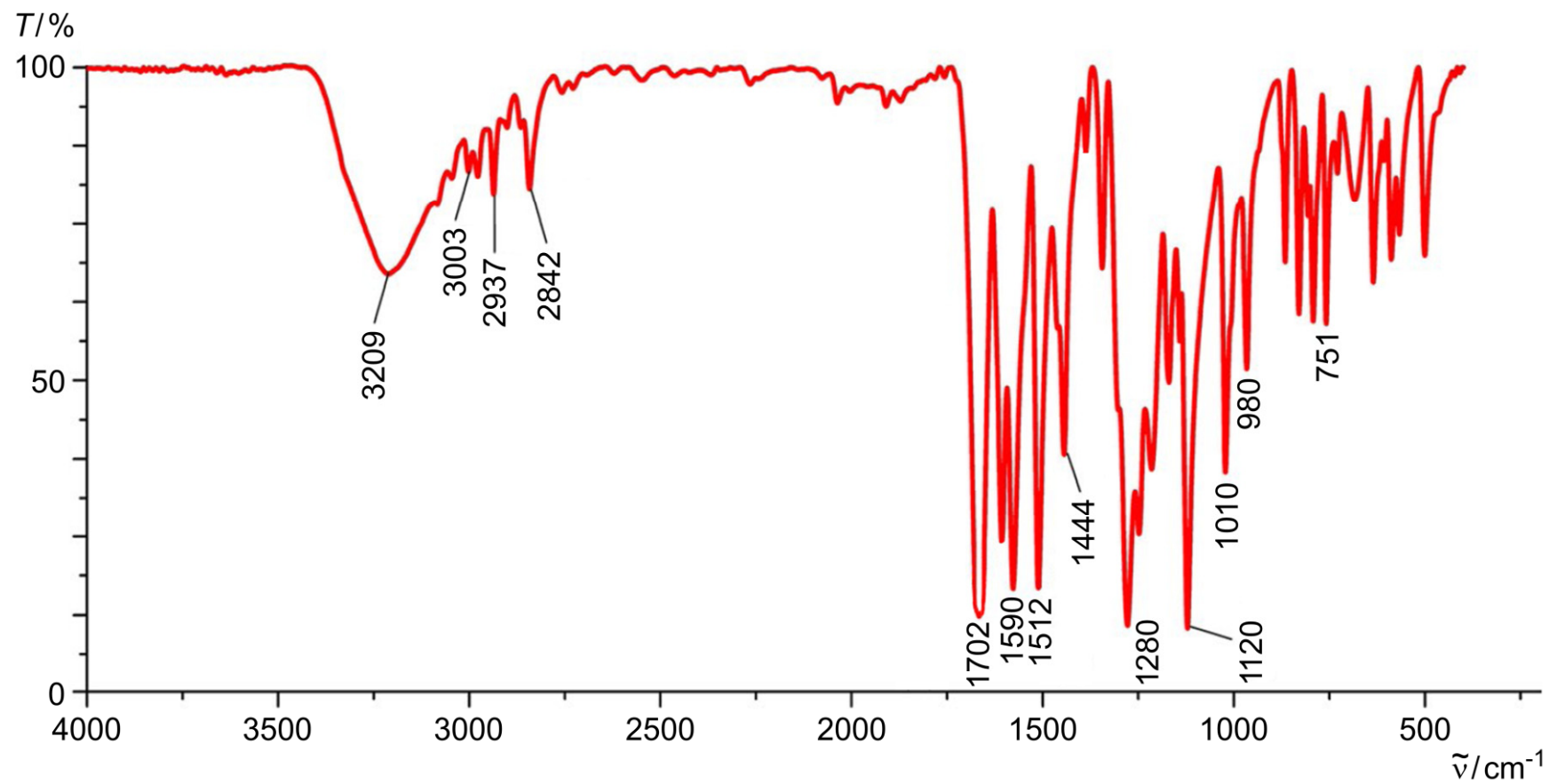
7. Identificirajte spoj na temelju njegovih MS, UV/Vis, IR, ^1H NMR, ^{13}C NMR, COSY, HMQC i HMBC spektara.



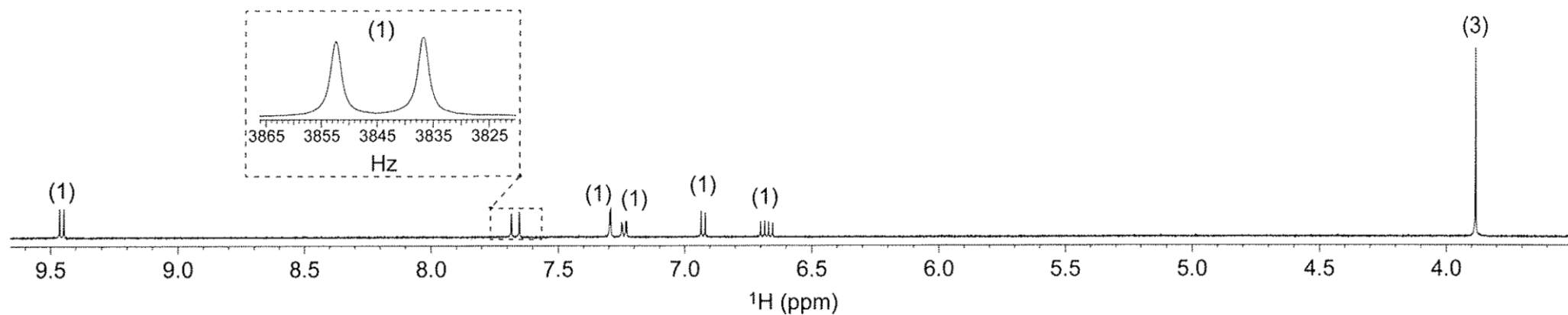
MS spektar spoja dobiven kemijskom ionizacijom u pozitivnom modu.



UV/Vis spektar spoja snimljen u etanolu.

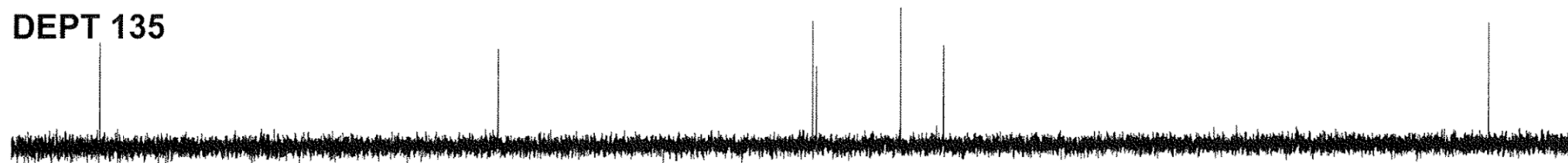


IR spektar spoja snimljen tehnikom KBr pastile.

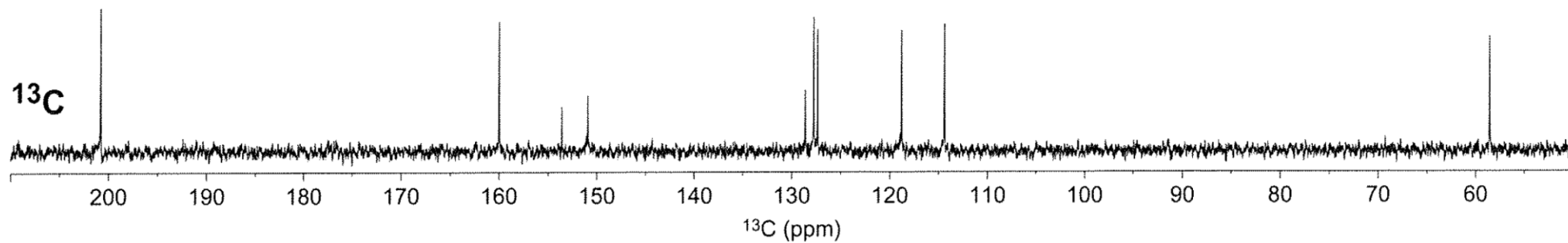


^1H NMR spektar spoja snimljen u D_2O . Spoj sadži i protone koji se izmijene u D_2O .

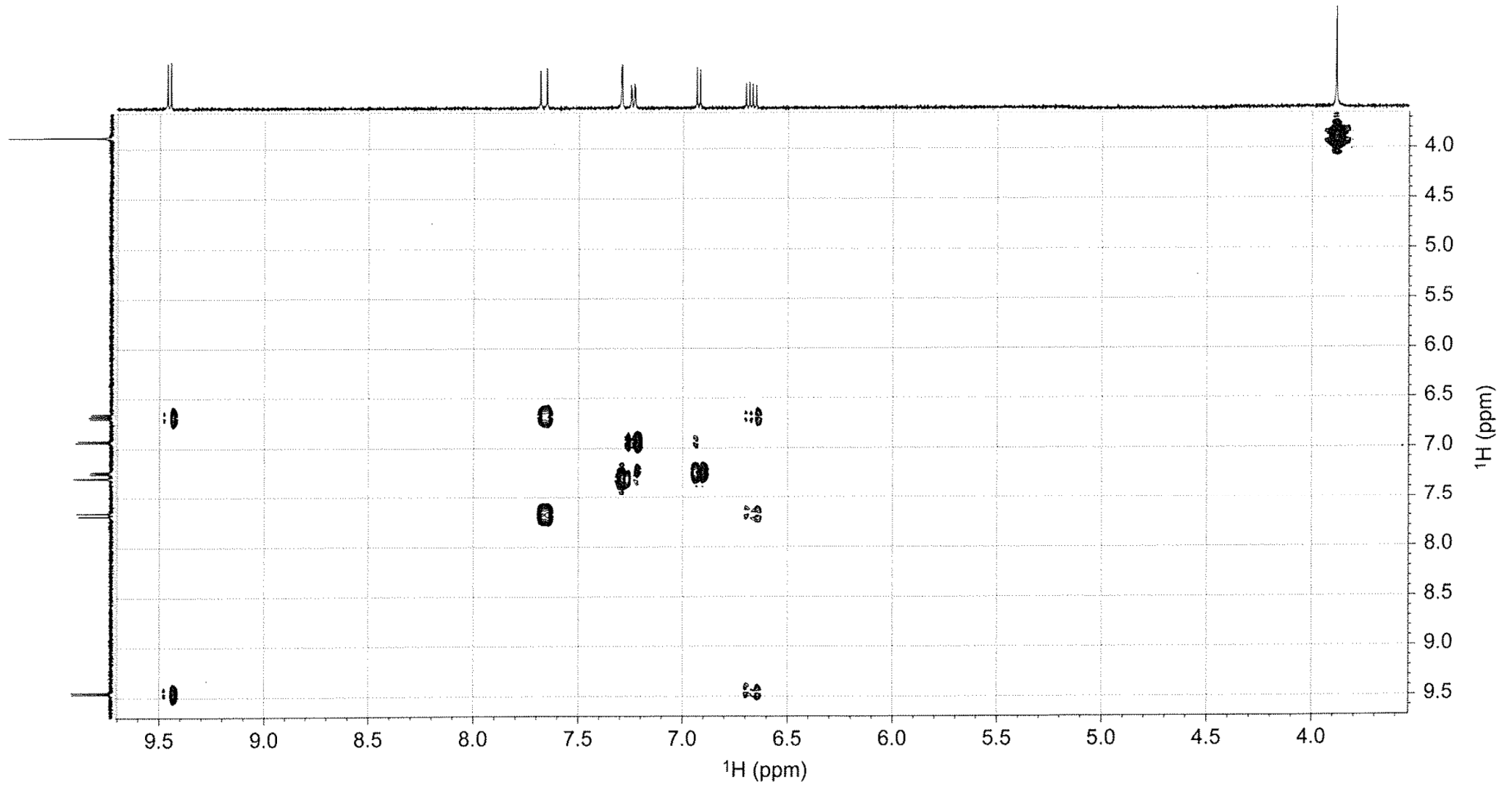
DEPT 135



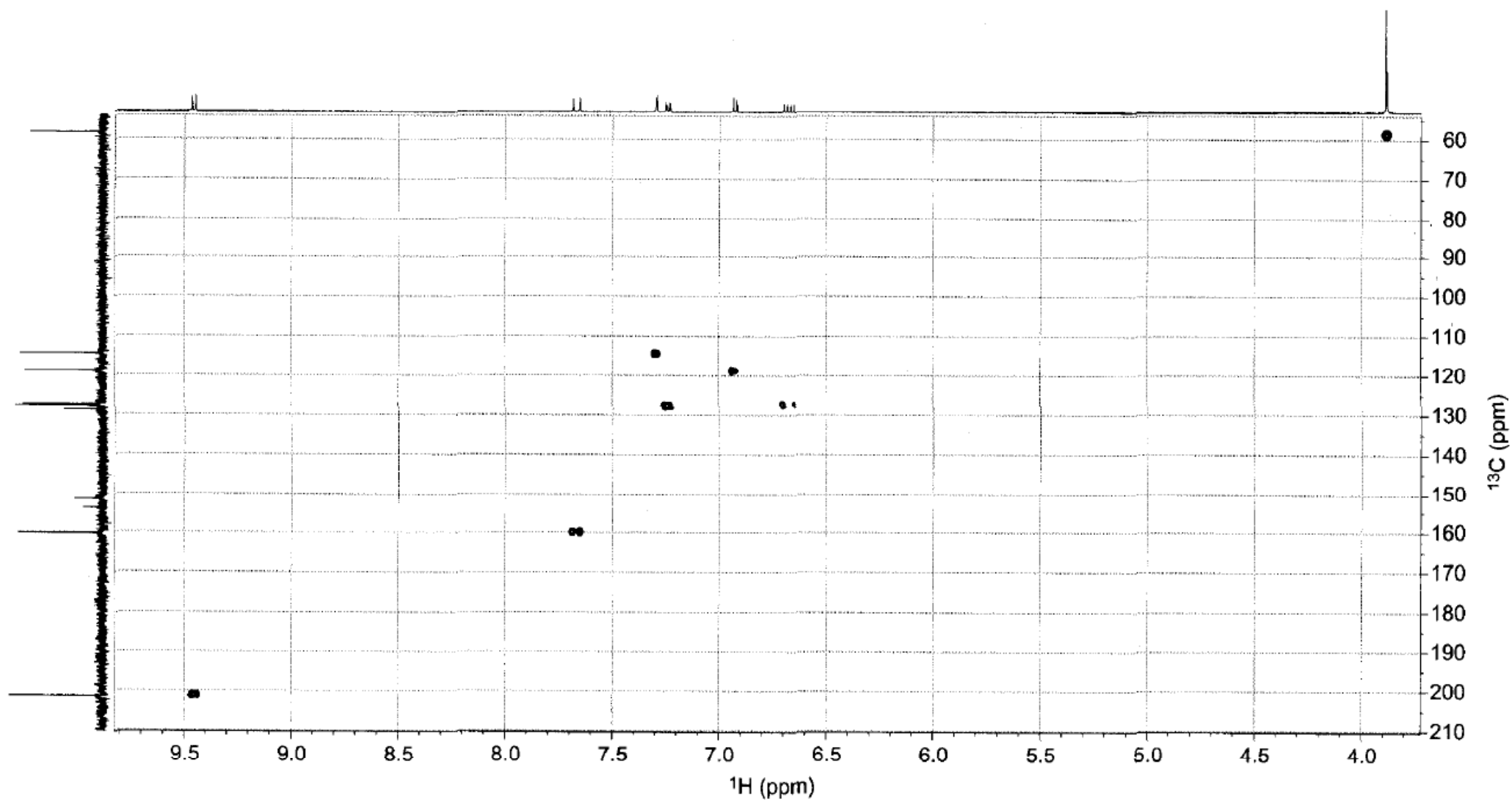
^{13}C



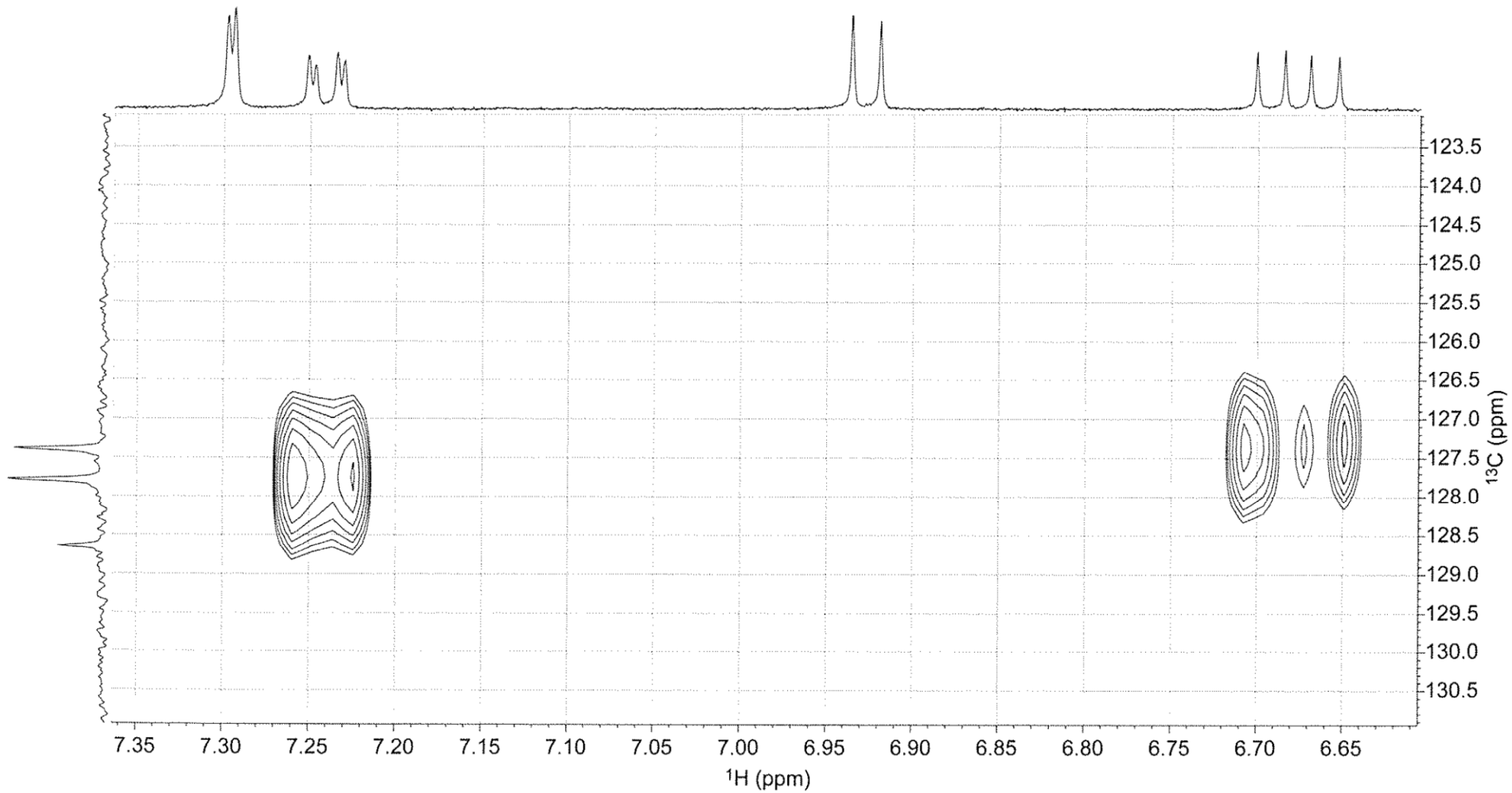
^{13}C NMR spektri spoja snimljeni u D_2O .



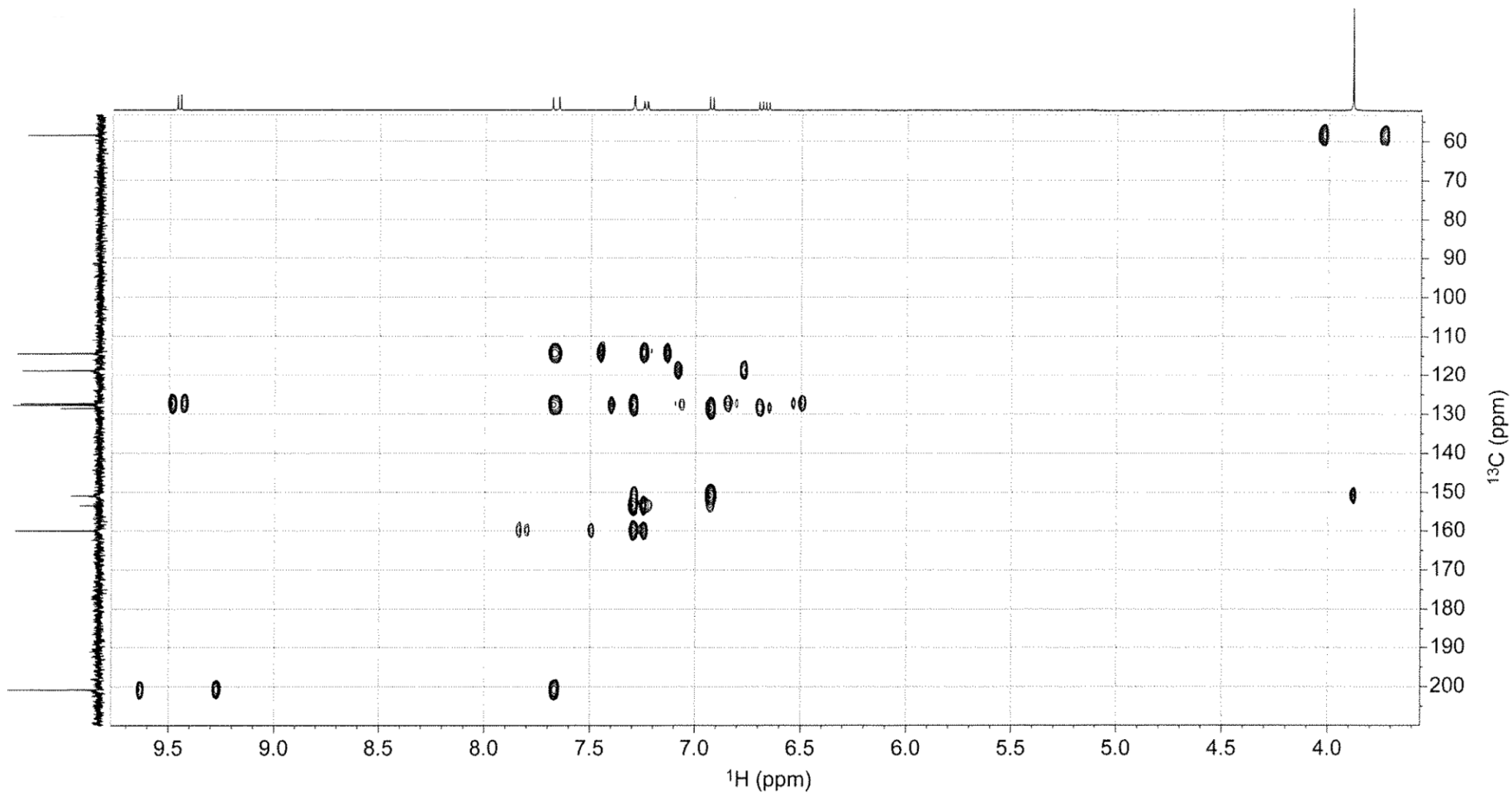
COSY spektar spoja snimljen u D_2O .



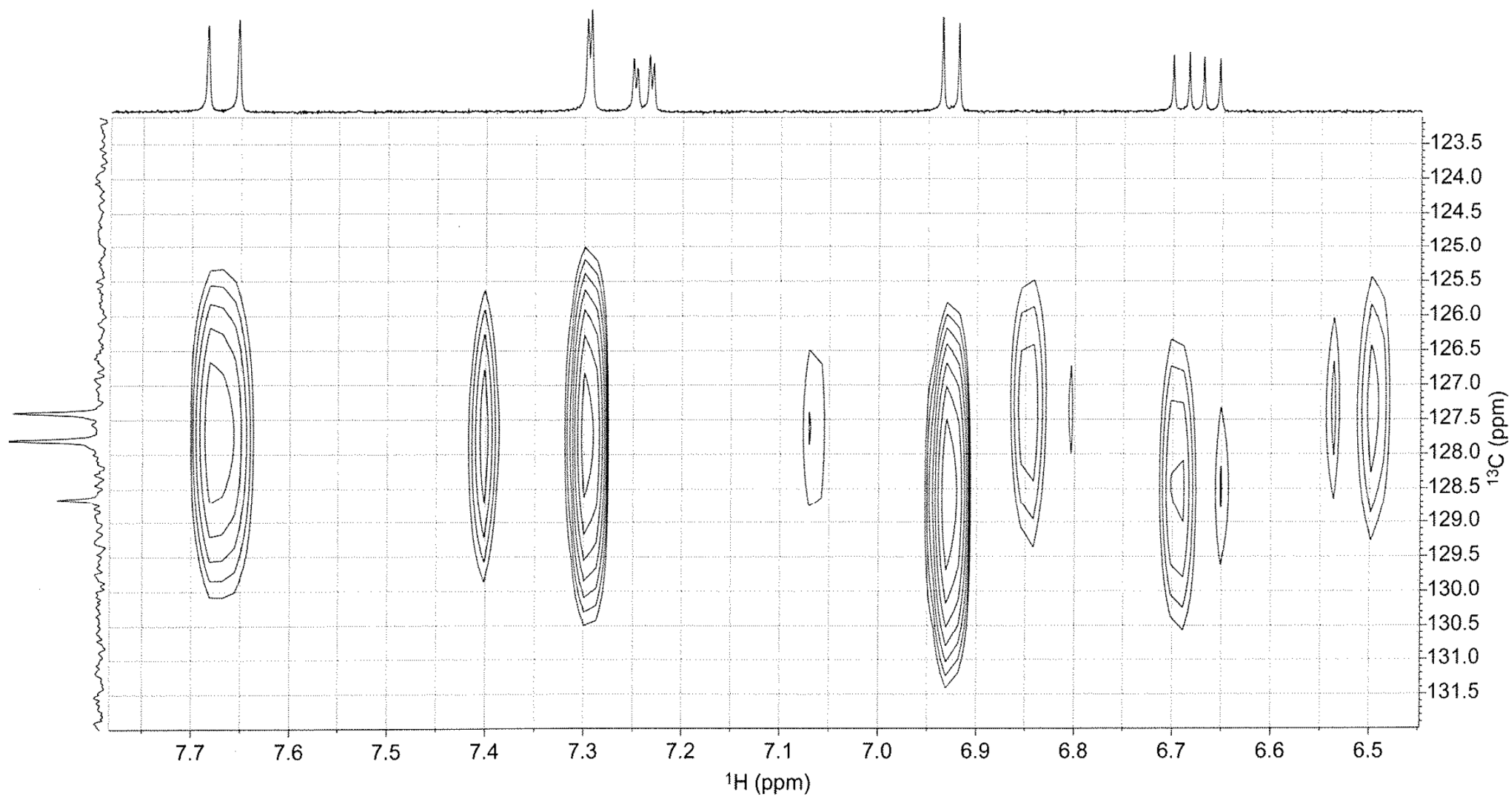
HSQC spektar spoja snimljen u D_2O .



Uvećani dio HSQC spektra spoja snimljenog u D_2O .



HMBC spektar spoja snimljen u D_2O .



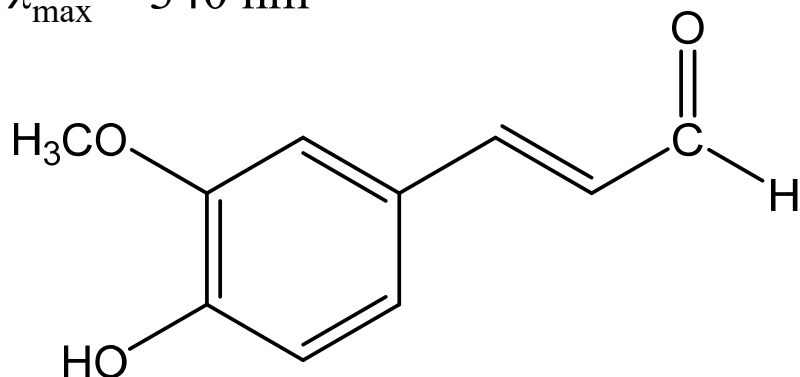
Uvećani dio HMBC spektra spoja snimljenog u D_2O .

- informacije iz MS:

$$(M+H)^+ = 179$$

- informacije iz UV/Vis:

$$\lambda_{\max} = 340 \text{ nm}$$



- informacije iz ^1H NMR:

$\delta(^1\text{H}) / \text{ppm}$	multiplet	H-atom
3,89	s	$-\text{CH}_3$
6,67	dd	$=\text{CH}-$
6,92	d	$=\text{CH}-$
7,24	dd	$=\text{CH}-$
7,30	d	$=\text{CH}-$
7,69	d	$=\text{CH}-$
9,45	d	$\text{HC}=\text{O}$

- informacije iz IR:

3209 cm^{-1} OH istežanje

$3003, 2937$ i 2842 cm^{-1} C–H istežanje

1702 cm^{-1} C=O istežanje

$1590, 1512$ i 1444 cm^{-1} C=C istežanje

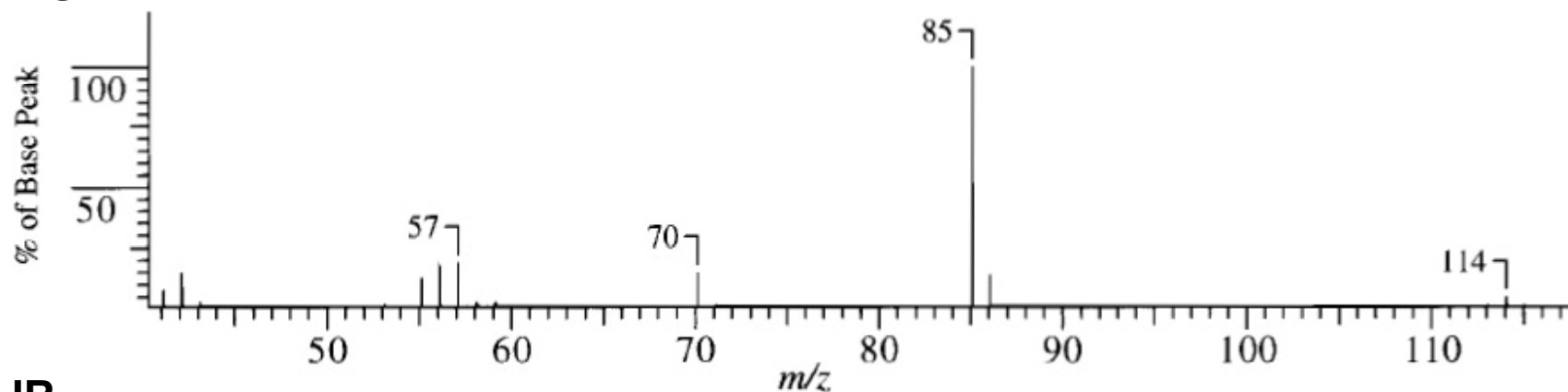
980 i 751 cm^{-1} aromatsko C–H svijanje

- informacije iz ^{13}C NMR:

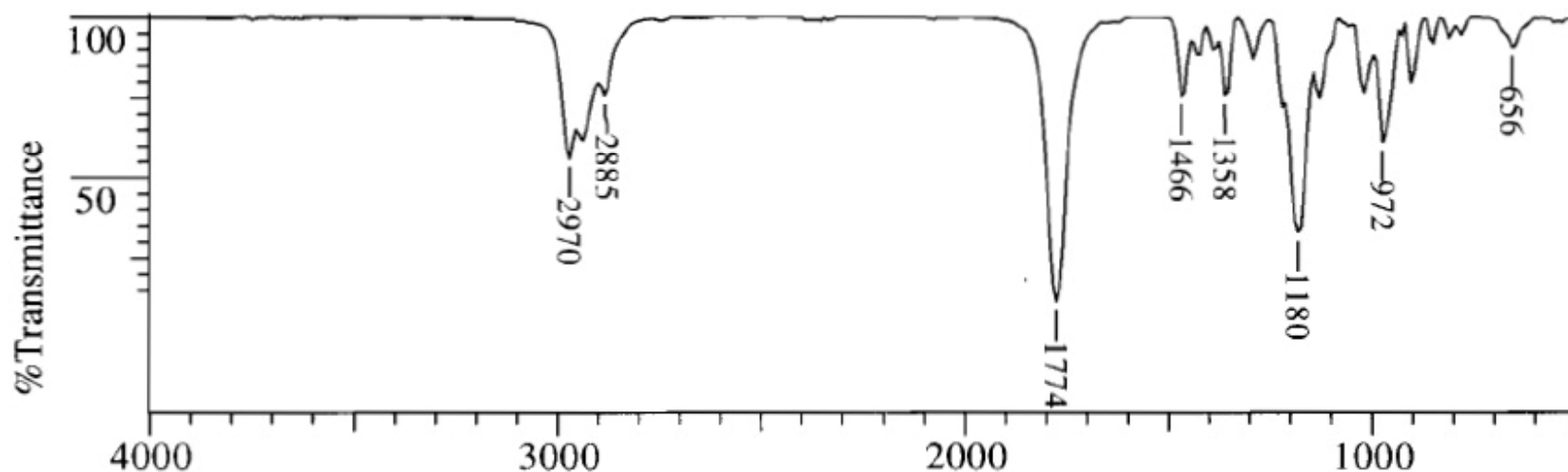
$\delta(^{13}\text{C}) / \text{ppm}$	C-atom
58	$-\text{CH}_3$
114	$=\text{CH}-$
118	$=\text{CH}-$
127	$=\text{CH}-$
127,5	$=\text{CH}-$
128	C
151	C
153,5	C
160	$=\text{CH}-$
201	C=O

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

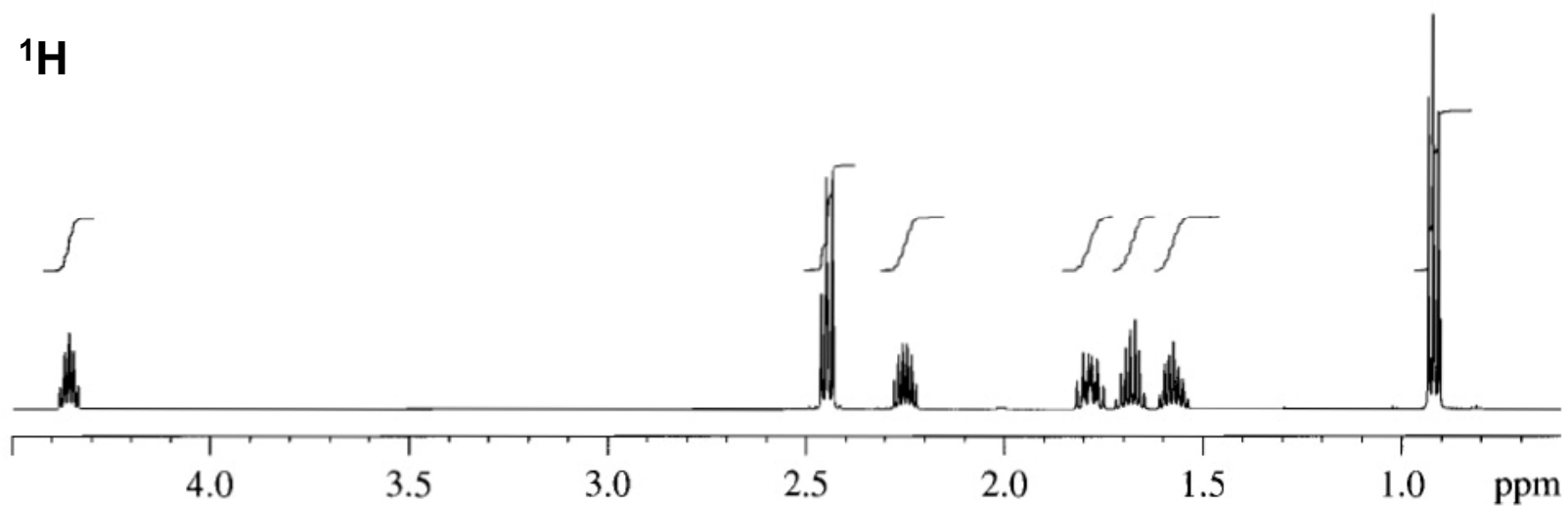
MS



IR



^1H



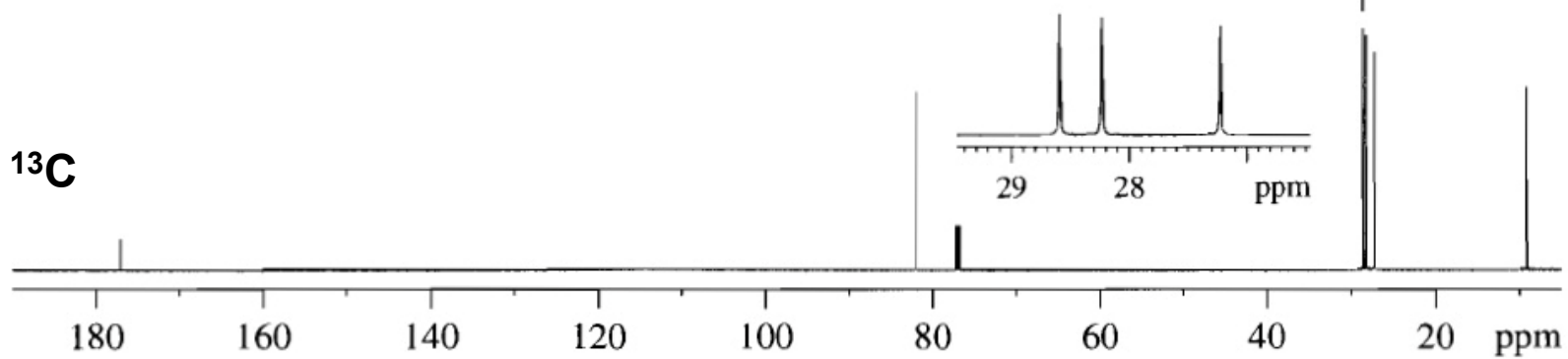
DEPT 90

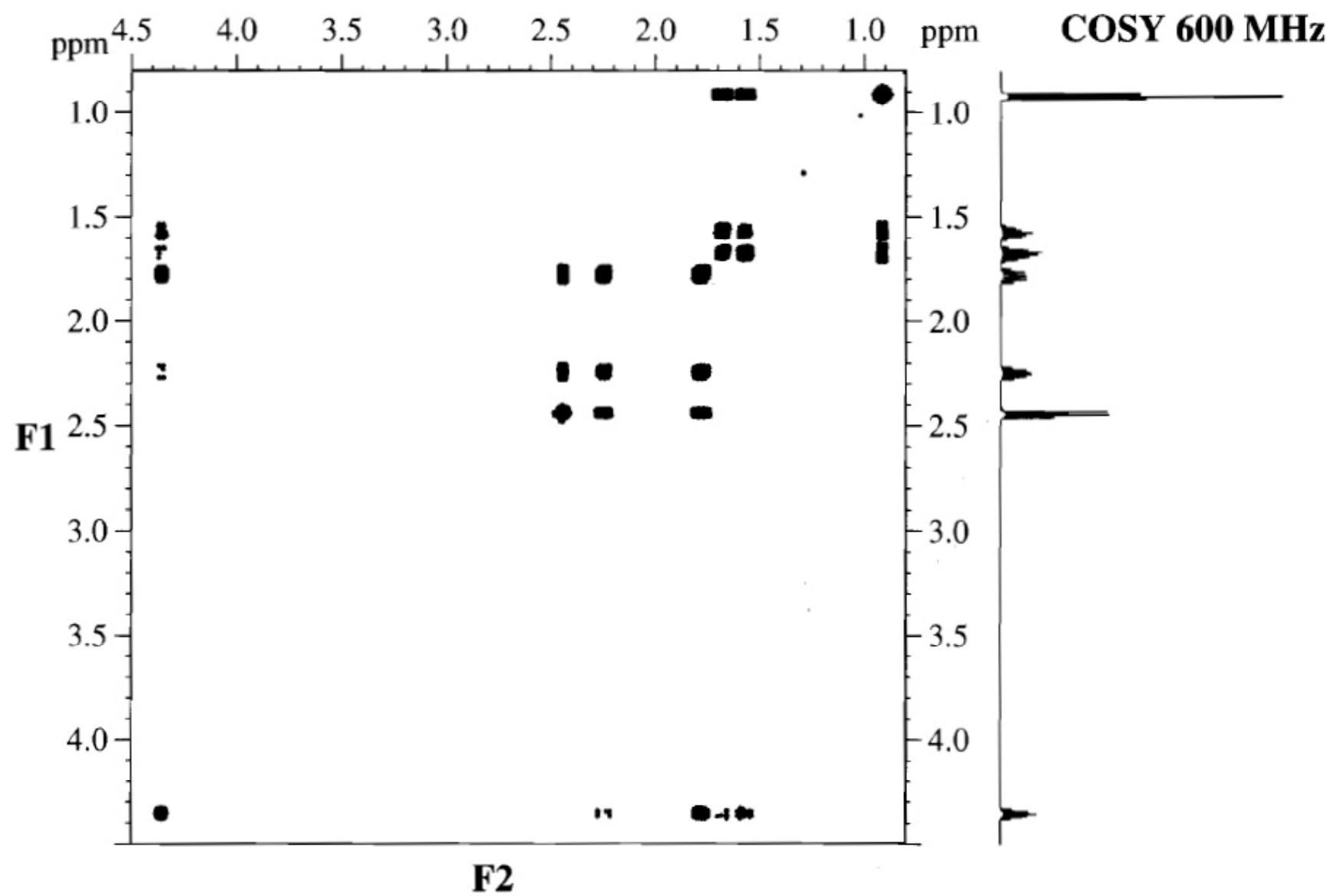


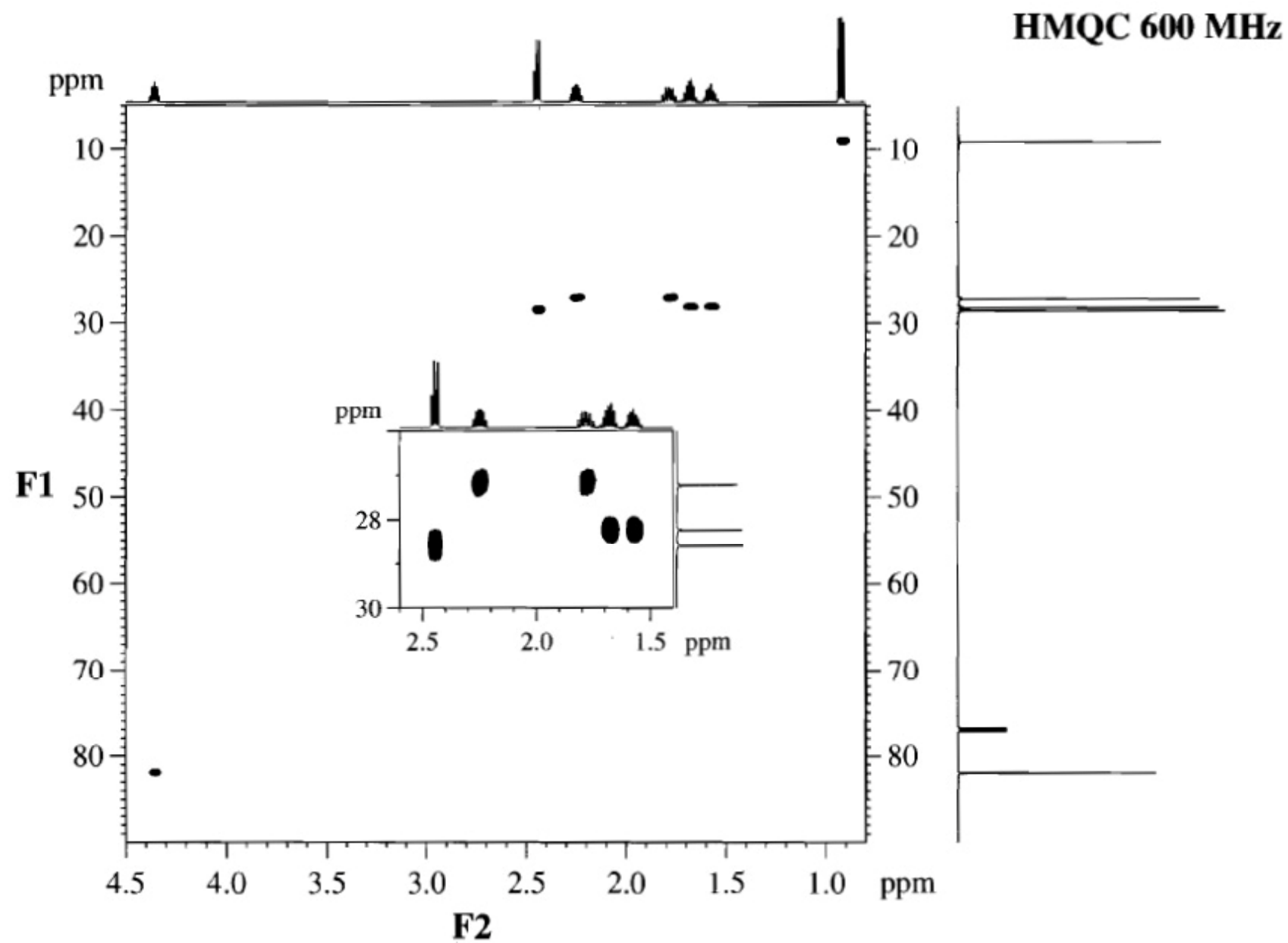
DEPT 135



^{13}C



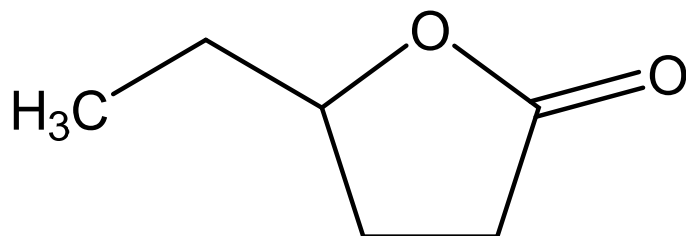




- informacije iz MS:

bazni pik: 85

$M^+ = 114$



- 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 ^1H NMR:

$\delta(^1\text{H}) / \text{ppm}$	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:

$\delta(^1\text{H}) / \text{ppm}$	C-atom
9	–CH ₃
27,2	–CH ₂ –
28,2	–CH ₂ –
28,6	–CH ₂ –
83	CH
178	C=O