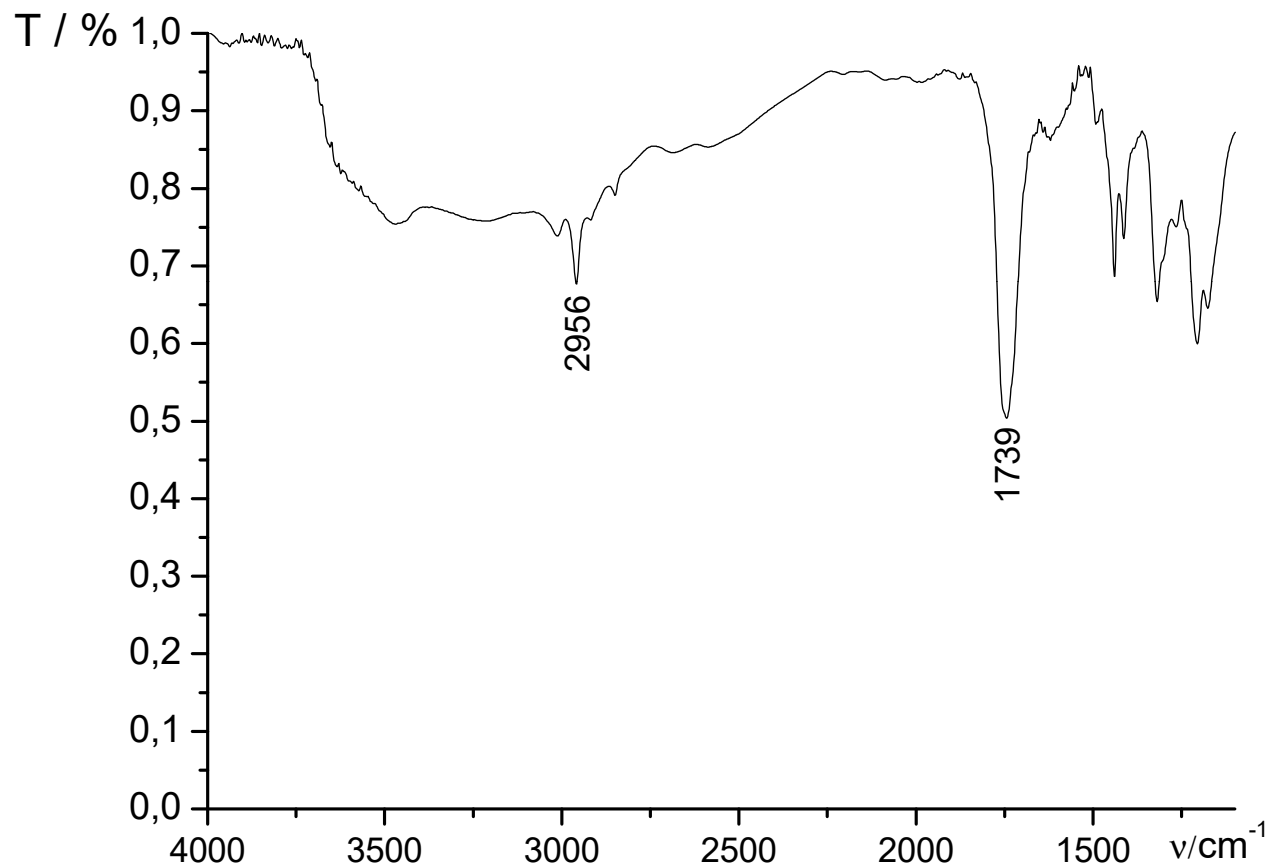


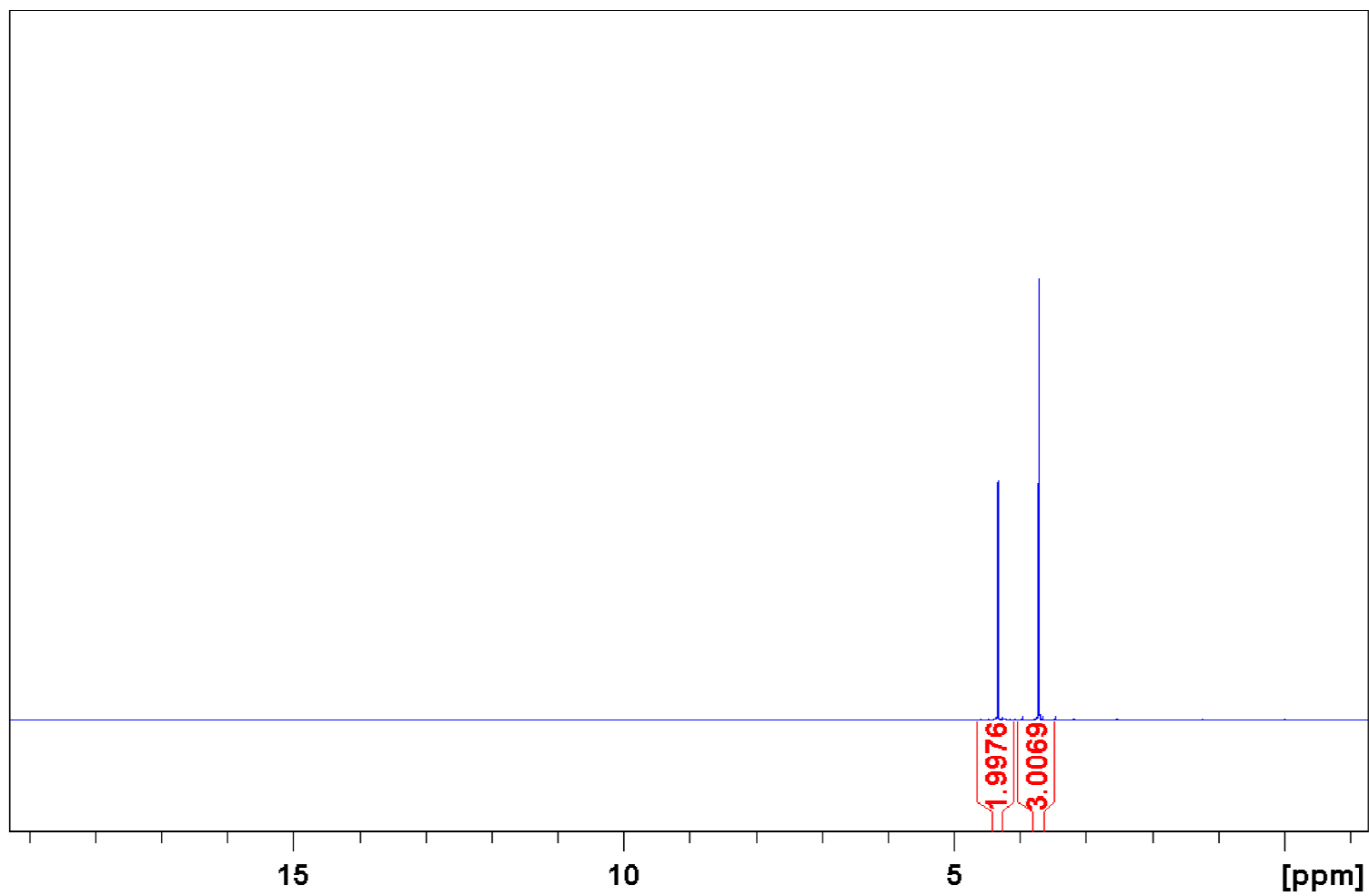
# Spektroskopska strukturna analiza

prof.dr. sc. Predrag Novak  
doc. dr. sc. Tomislav Jednačak; ak. god. 2022./23.

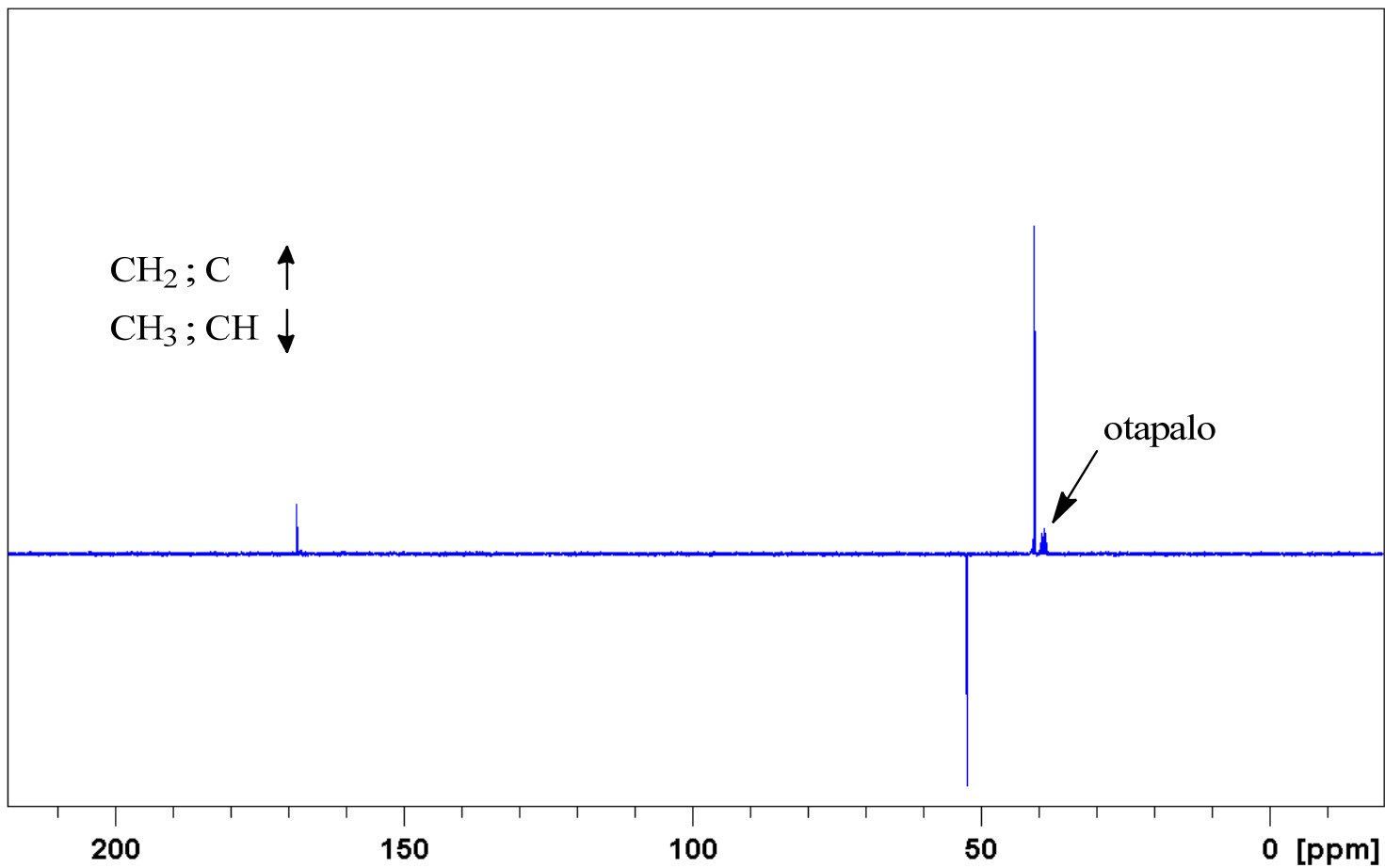
1. Odredite strukturu spoja molekulske mase 108 g/mol na temelju njegovog infracrvenog,  $^1\text{H}$  NMR i APT spektra



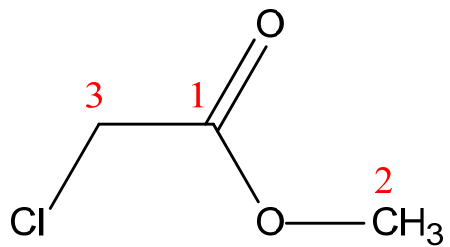
Infracrveni spektar spoja snimljen u tekućinskoj kiveti od  $\text{CaF}_2$ .



$^1\text{H}$  NMR spektar spoja snimljen u DMSO- $d_6$ .



APT spektar spoja snimljen u DMSO-d<sub>6</sub>.



Struktura spoja i brojanje atoma.

### Karakteristične vibracijske vrpce u infracrvenom spektru spoja

$\nu / \text{cm}^{-1}$	Način vibracije
2956	$\nu_{\text{alifatsko}} (\text{C-H})$
1739	$\nu (\text{C=O})$

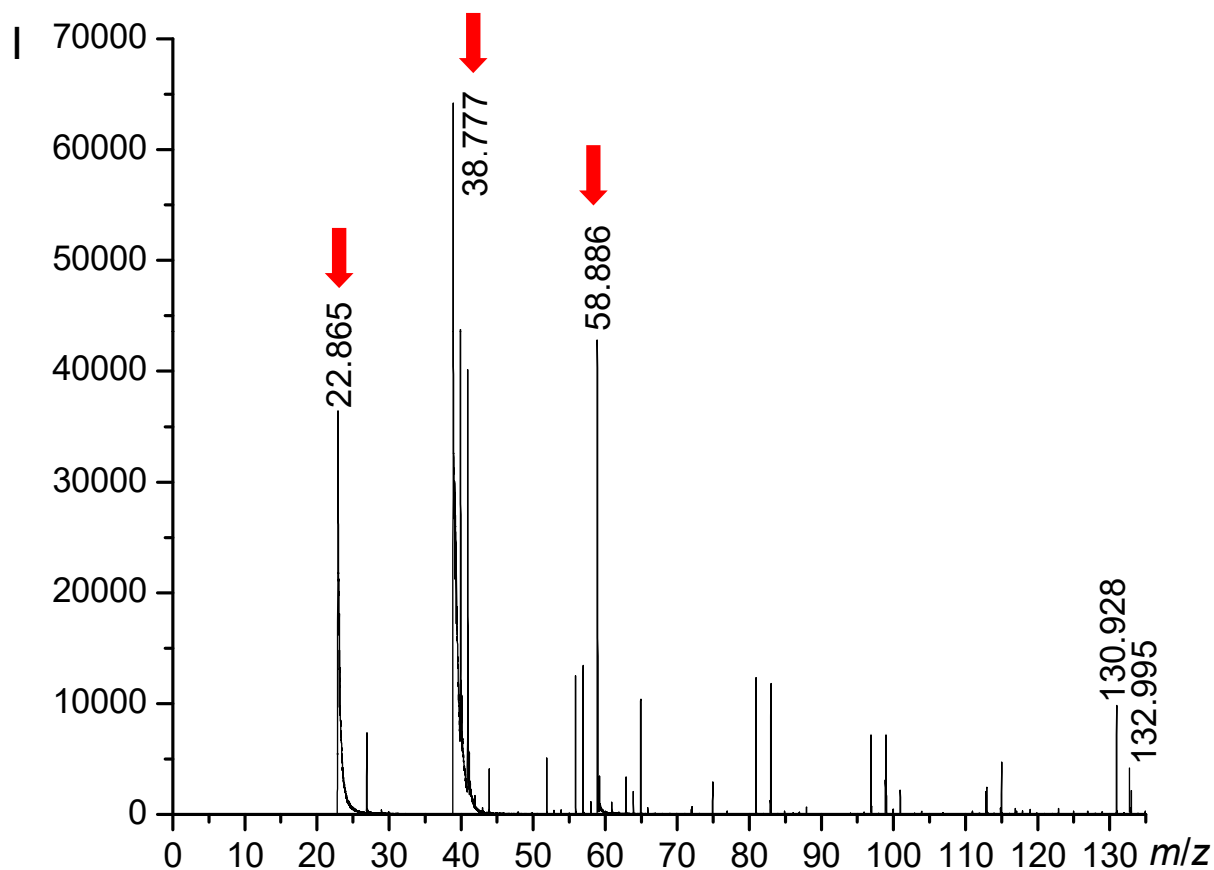
### Asignacija $^1\text{H}$ NMR kemijskih pomaka spoja

H-atom	$\delta / \text{ppm}$	Intenzitet	Multipletnost
1			
2	3,7	3,01	s
3	4,3	1,99	s

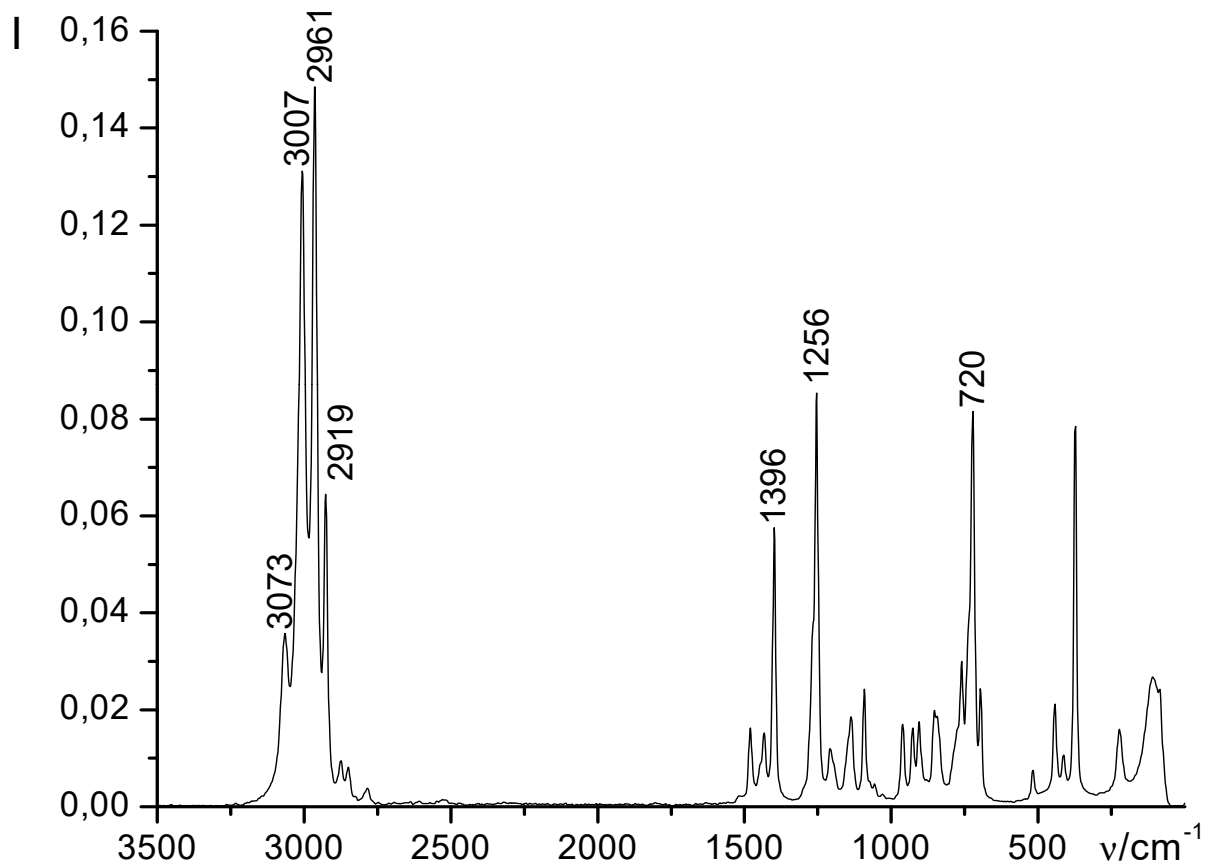
### Asignacija $^{13}\text{C}$ NMR kemijskih pomaka spoja

C-atom	$\delta / \text{ppm}$
1	170
2	53
3	40

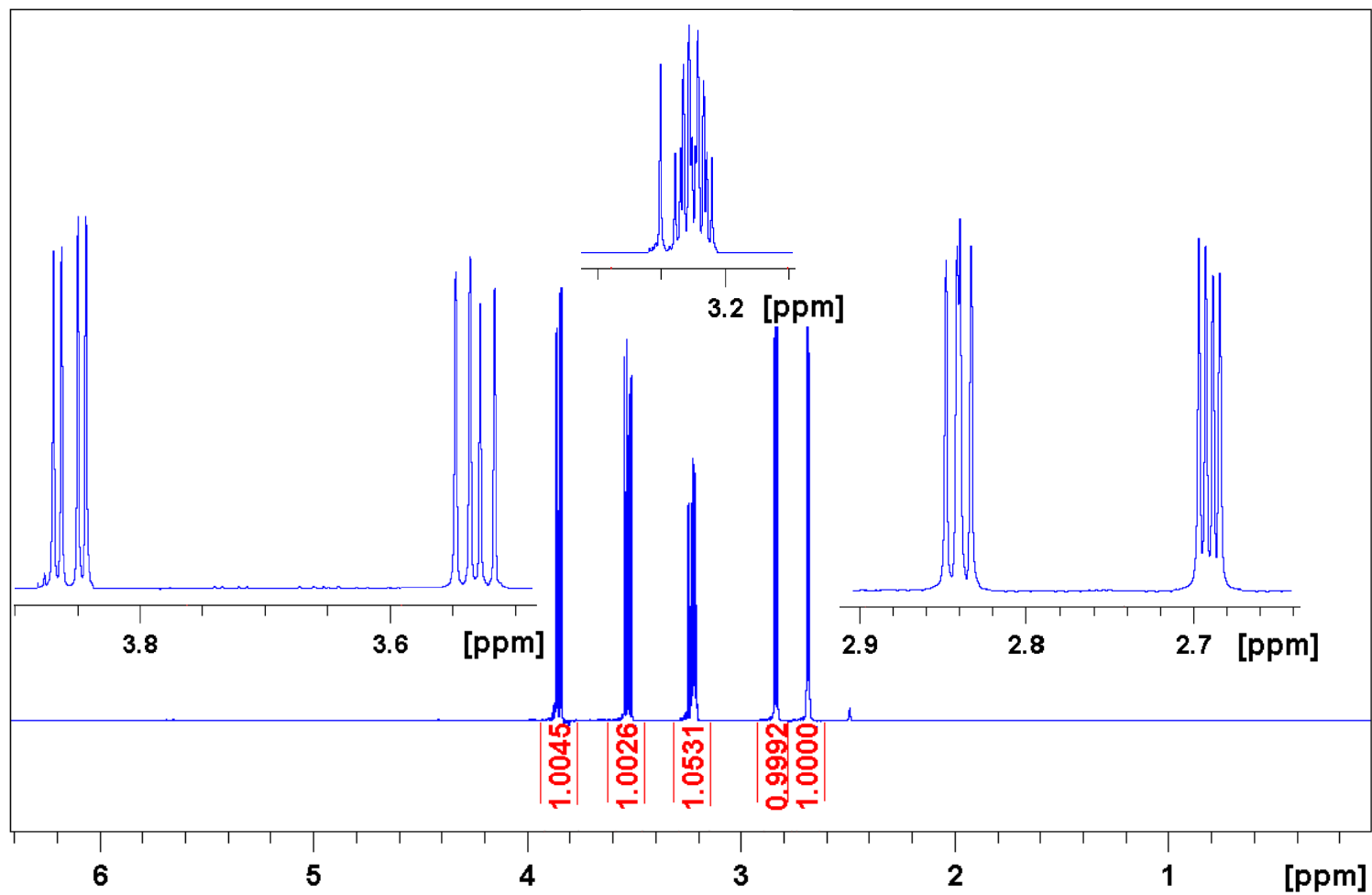
2. Odredite strukturu spoja na temelju njegovog MS, Ramanovog,  $^1\text{H}$  NMR, APT, COSY i HSQC spektra.



Spektar masa spoja dobiven tehnikom MALDI-TOF. Uzorak je otopljen u smjesi acetona i vode (v/v=1:1). Signali označeni crvenim strelicama odgovaraju ionima  $\text{Na}^+$ ,  $\text{K}^+$  i  $\text{Cl}^-$  koji su prisutni u matrici uzorka te njihovim aduktima.

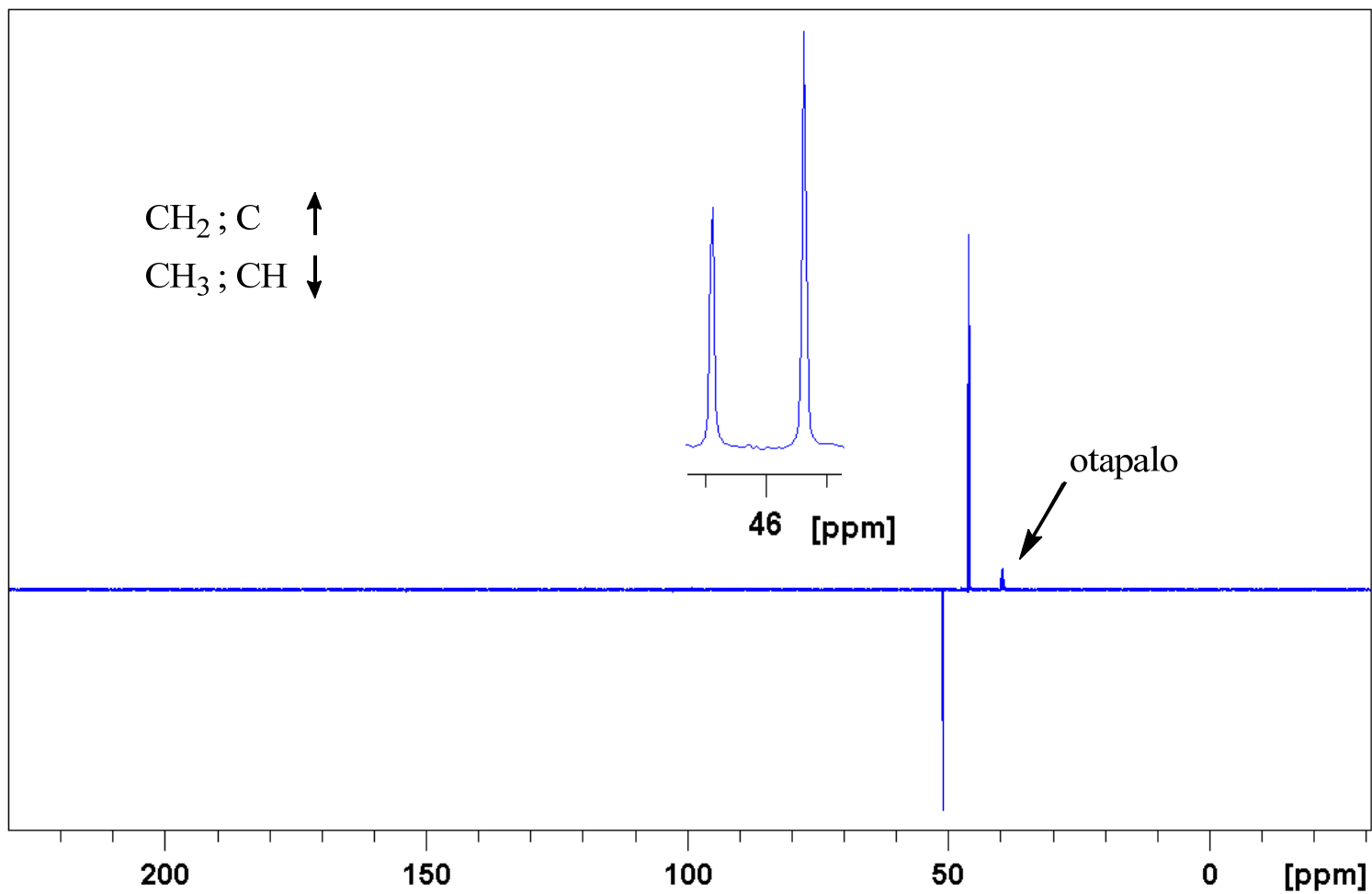


Ramanov spektar spoja.

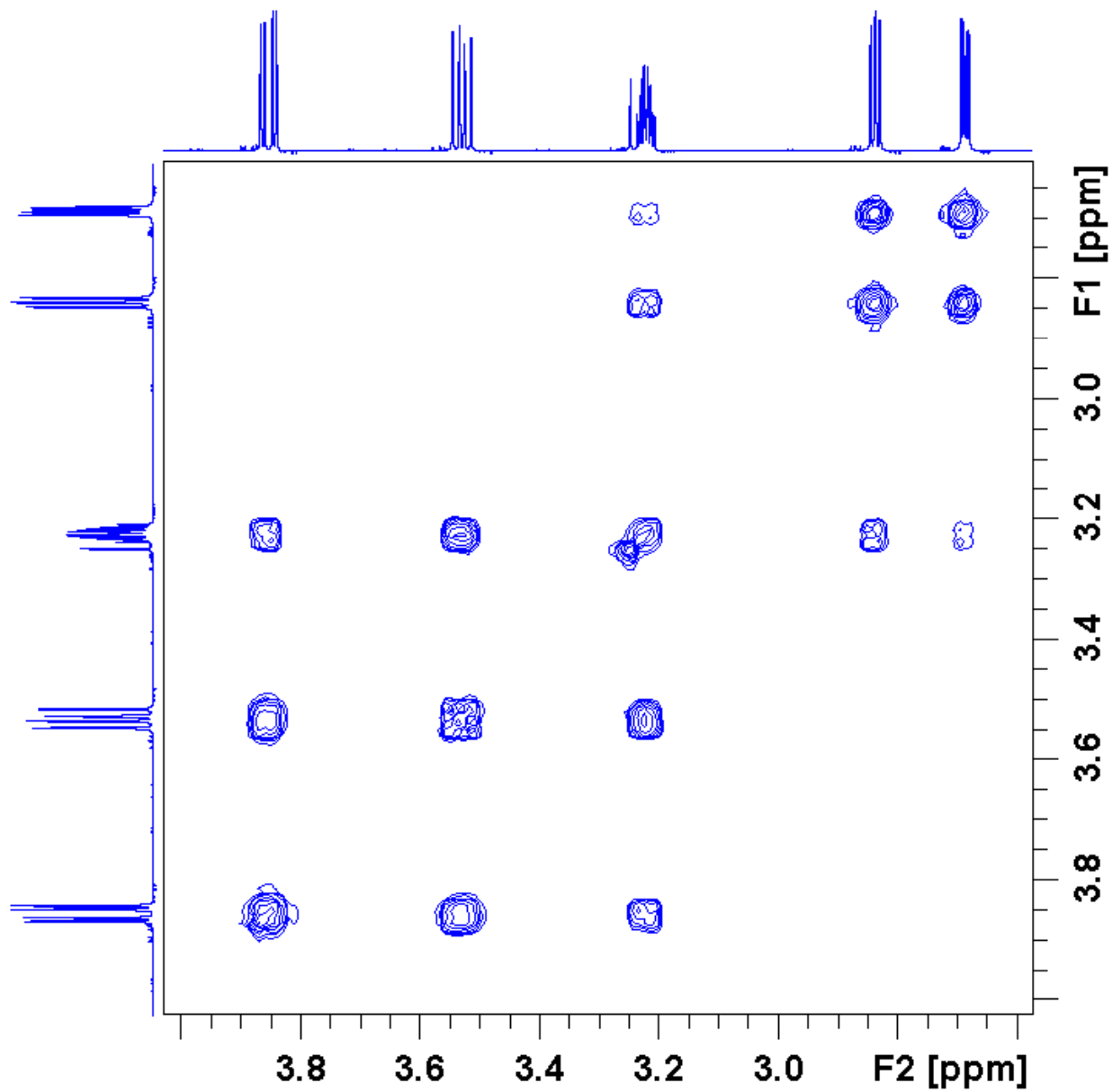


$^1\text{H}$  NMR spektar spoja snimljen u  $\text{DMSO-d}_6$  uz uvećani prikaz područja koja odgovaraju pojedinim signalima.

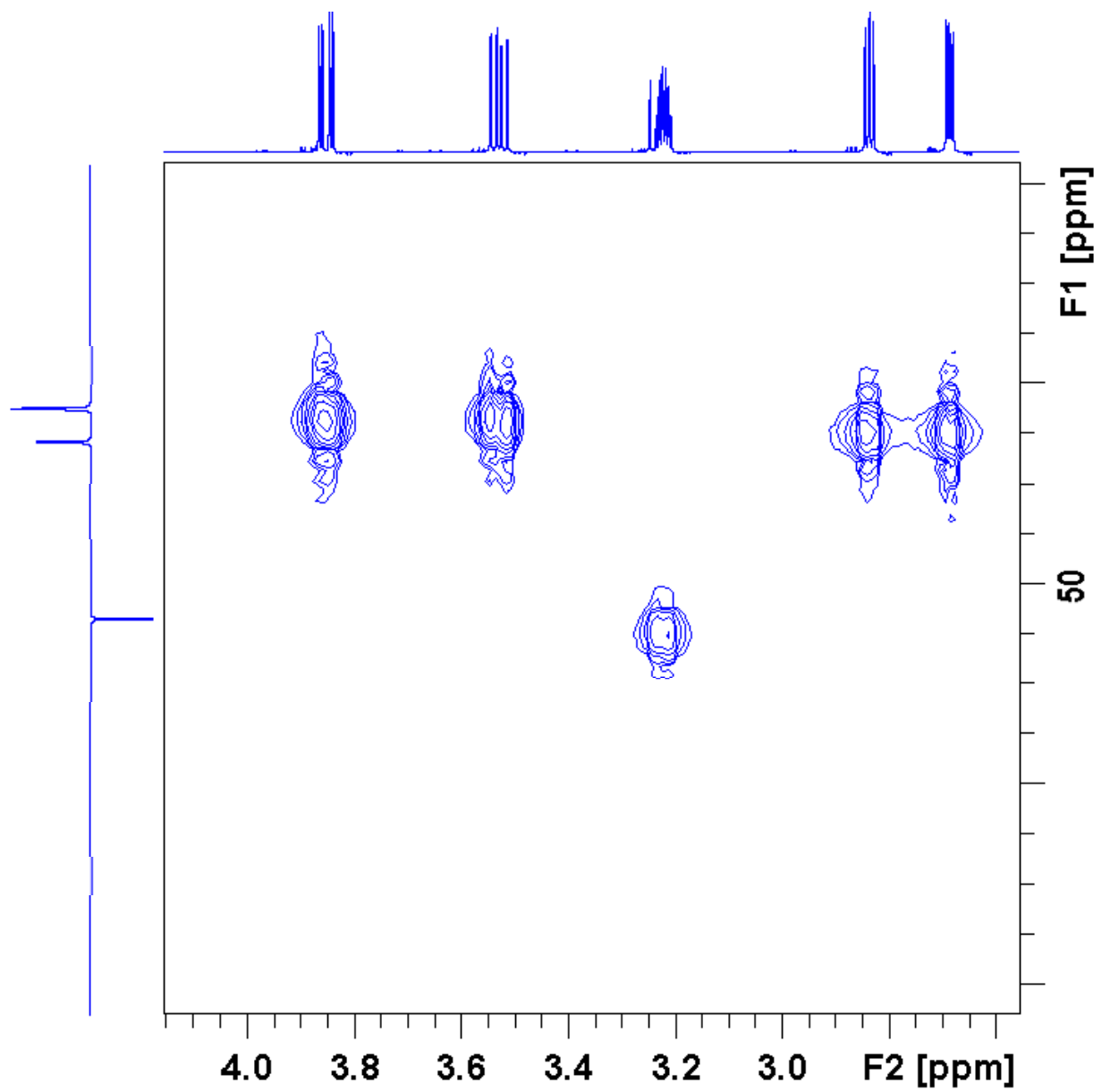




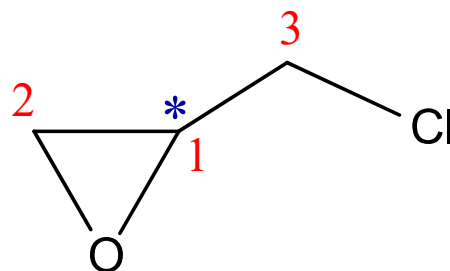
APT spektar spoja snimljen u DMSO-d<sub>6</sub> uz uvećani prikaz područja koja odgovaraju pojedinim signalima.



COSY spektar spoja snimljen u DMSO-d<sub>6</sub>.



HSQC spektar spoja snimljen u DMSO-d<sub>6</sub>.



Struktura spoja i brojanje atoma. Simbolom \* označen je kiralni centar.

Asignacija karakterističnih fragmenata u spektru masa spoja

Signal	$m/z$
bazni signal	38,777
Na <sup>+</sup>	22,885
K <sup>+</sup>	38,777
NaCl <sup>+</sup>	58,886
(M+K) <sup>+</sup>	130,928
(M+2+K) <sup>+</sup>	132,995

### Karakteristične vibracijske vrpce u Ramanovom spektru spoja

$\nu / \text{cm}^{-1}$	Način vibracije
3073; 3007; 2961; 2919	$\nu$ (C–H)
1396	$\delta$ (CH <sub>2</sub> )
1256	$\nu_s$ (C–O)
720	$\nu_{as}$ (C–O)

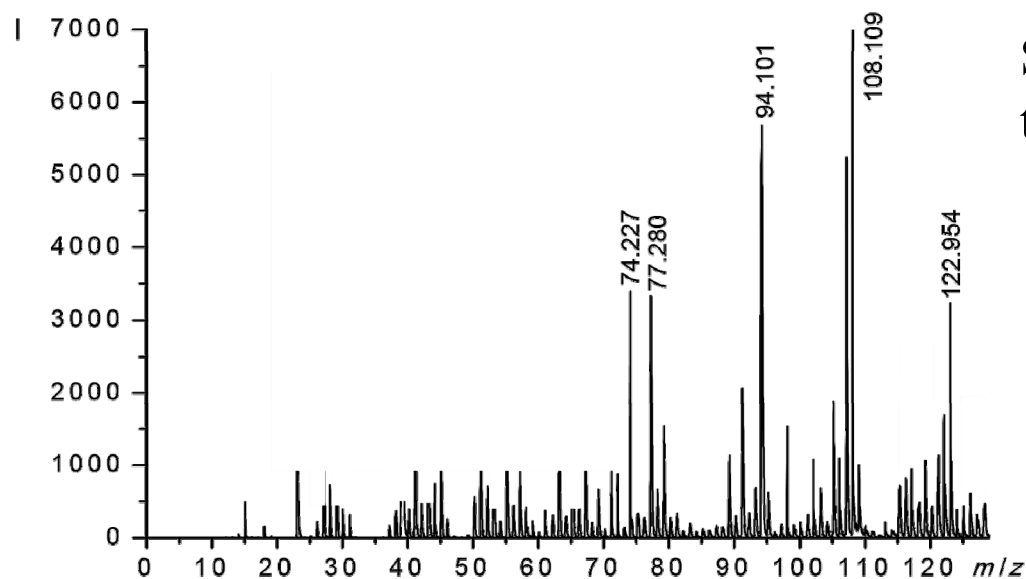
### Asignacija <sup>1</sup>H NMR kemijskih pomaka spoja

H-atom	$\delta / \text{ppm}$	Intenzitet	Multipletnost
1	3,12	1,0	m
2	2,69; 2,84	2,0	dd
3	3,83; 3,56	2,0	dd

### Asignacija <sup>13</sup>C NMR kemijskih pomaka spoja

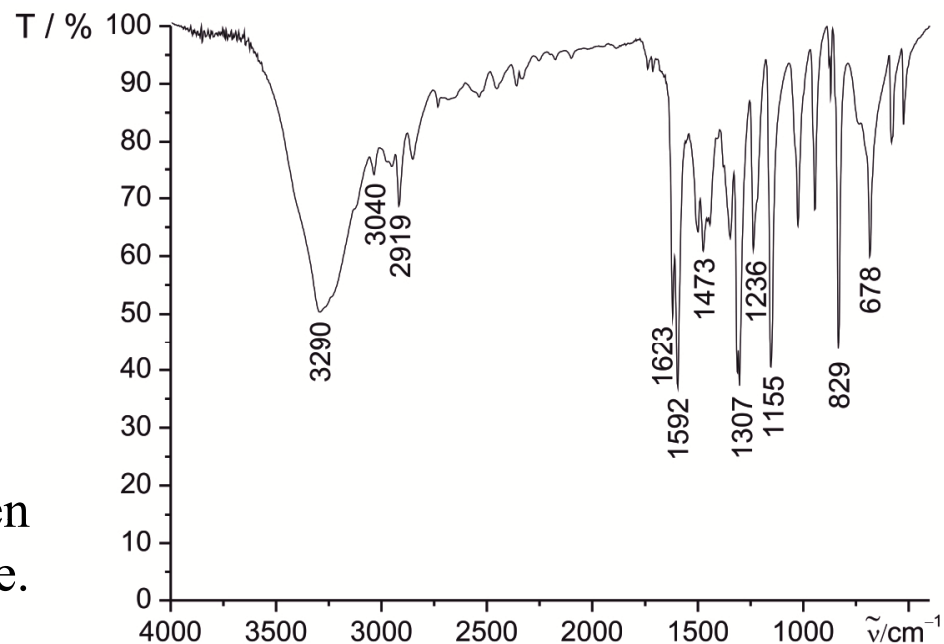
C-atom	$\delta / \text{ppm}$
1	52
2	46,2
3	45,8

### 3. Odredite strukturu spoja na temelju njegovog MS, IR, $^1\text{H}$ NMR, APT, HSQC i HMBC spektra

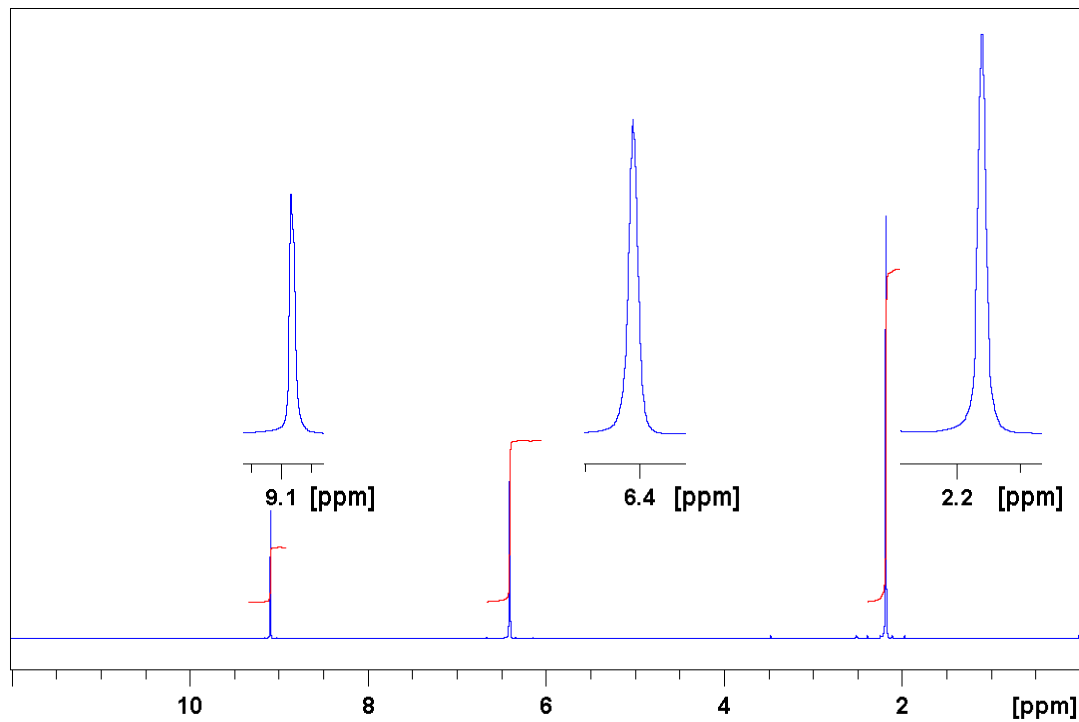


Spektar masa spoja dobiven tehnikom MALDI-TOF.

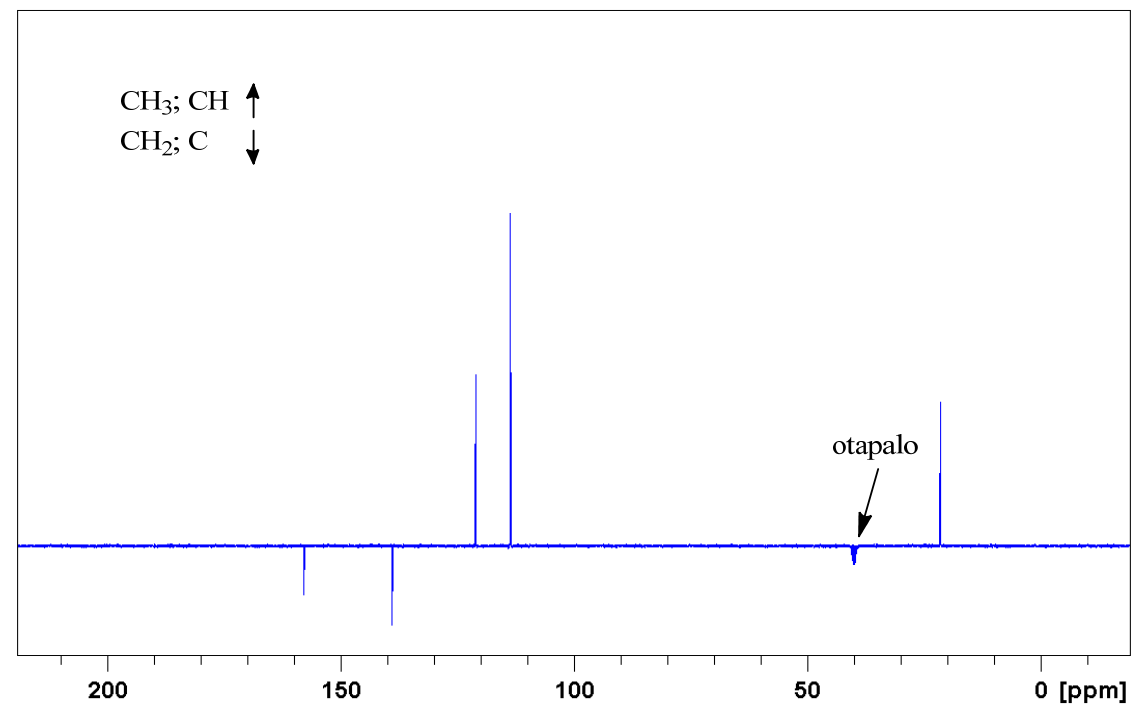
IR spektar spoja snimljen tehnikom KBr pastile.

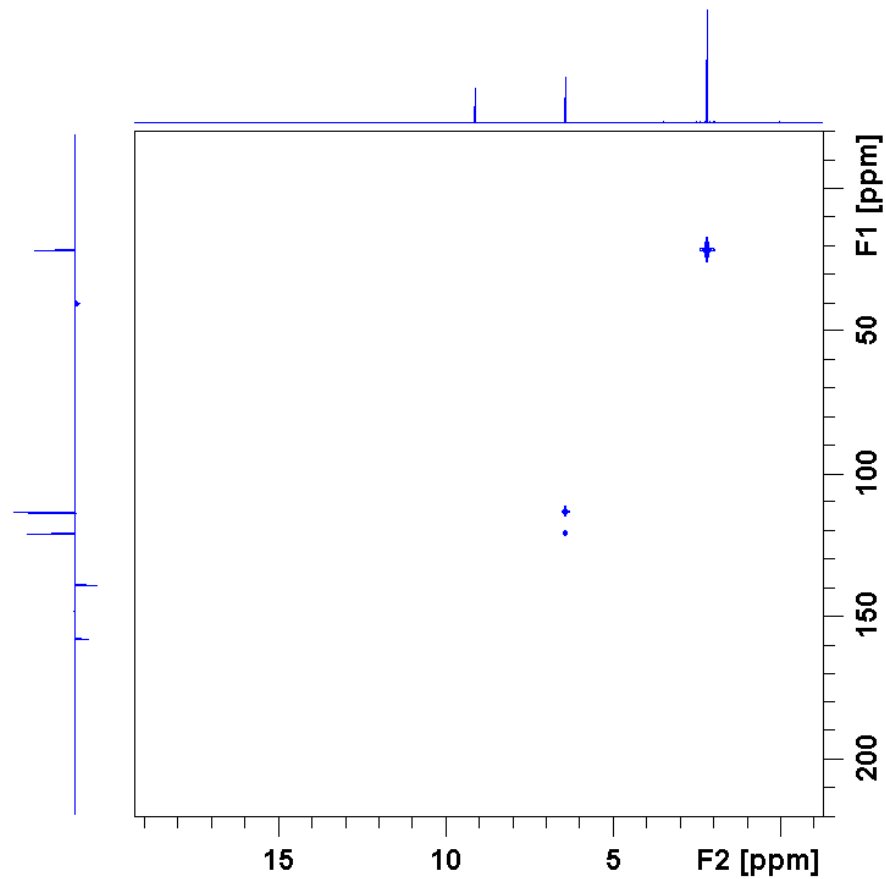


$^1\text{H}$  NMR spektar spoja  
snimljen u  $\text{DMSO-d}_6$ .

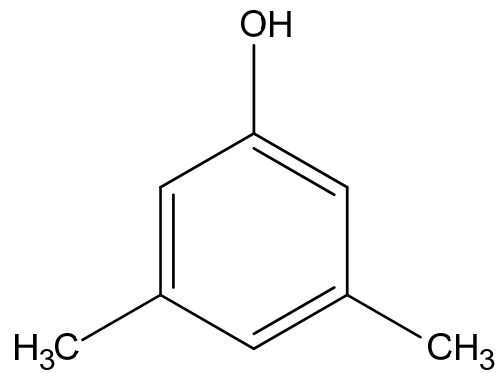


APT spektar spoja  
snimljen u  $\text{DMSO-d}_6$ .

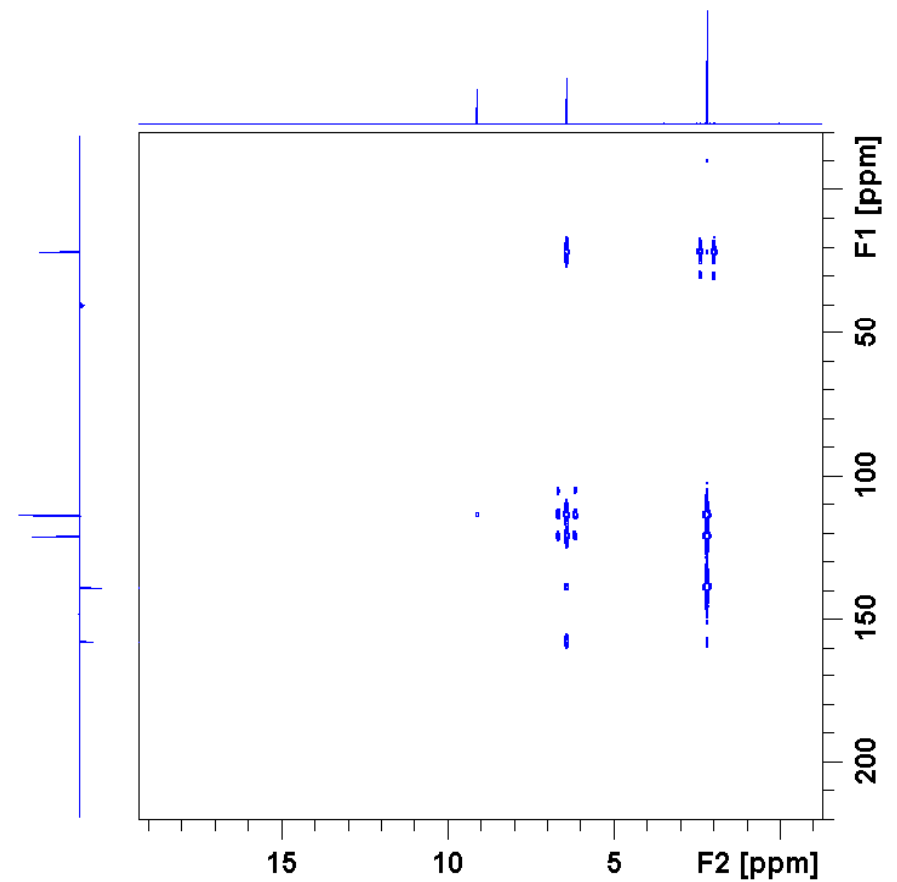




HSQC spektar spoja snimljen u DMSO-d<sub>6</sub>.



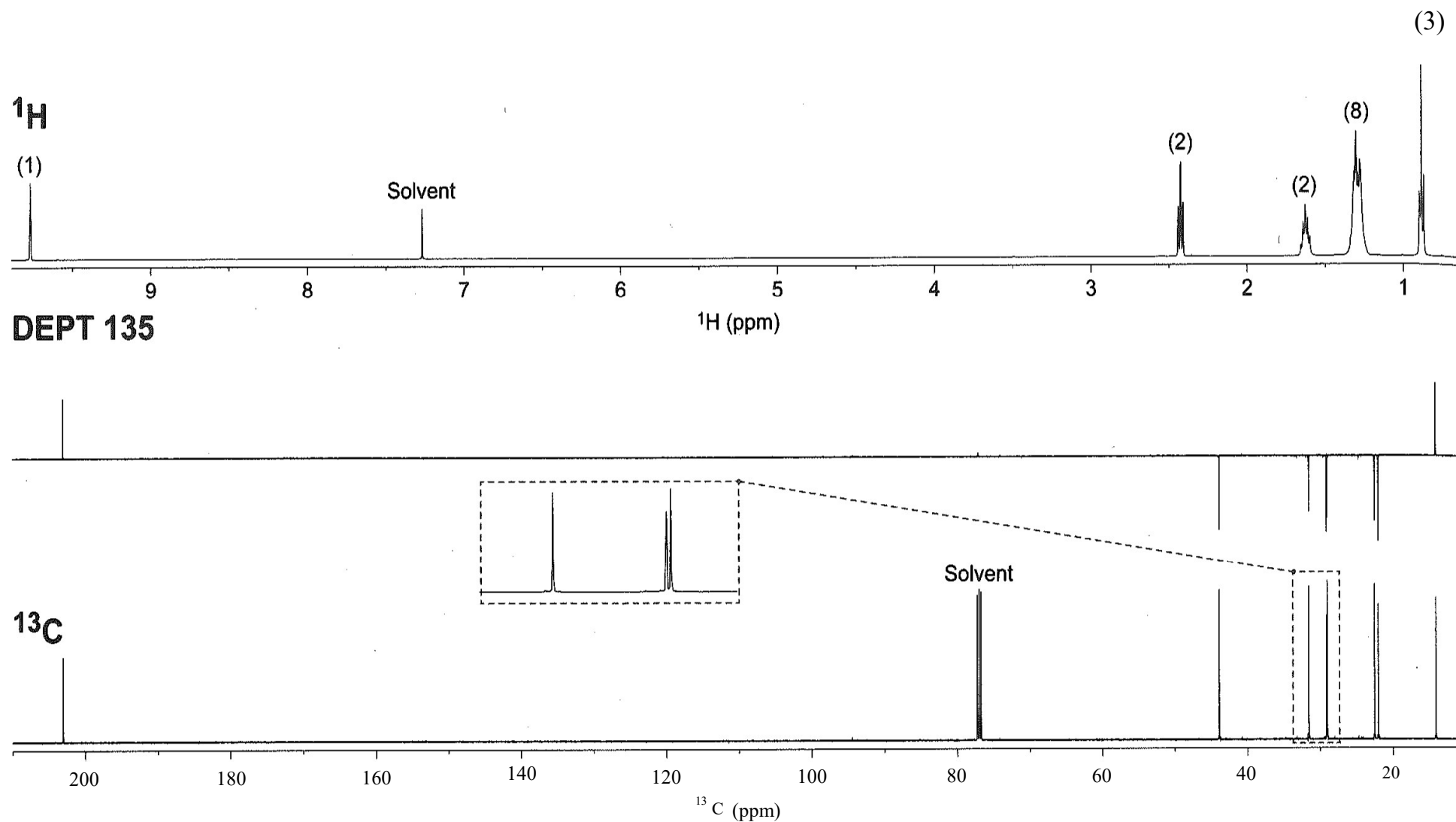
3,5-dimetilfenol



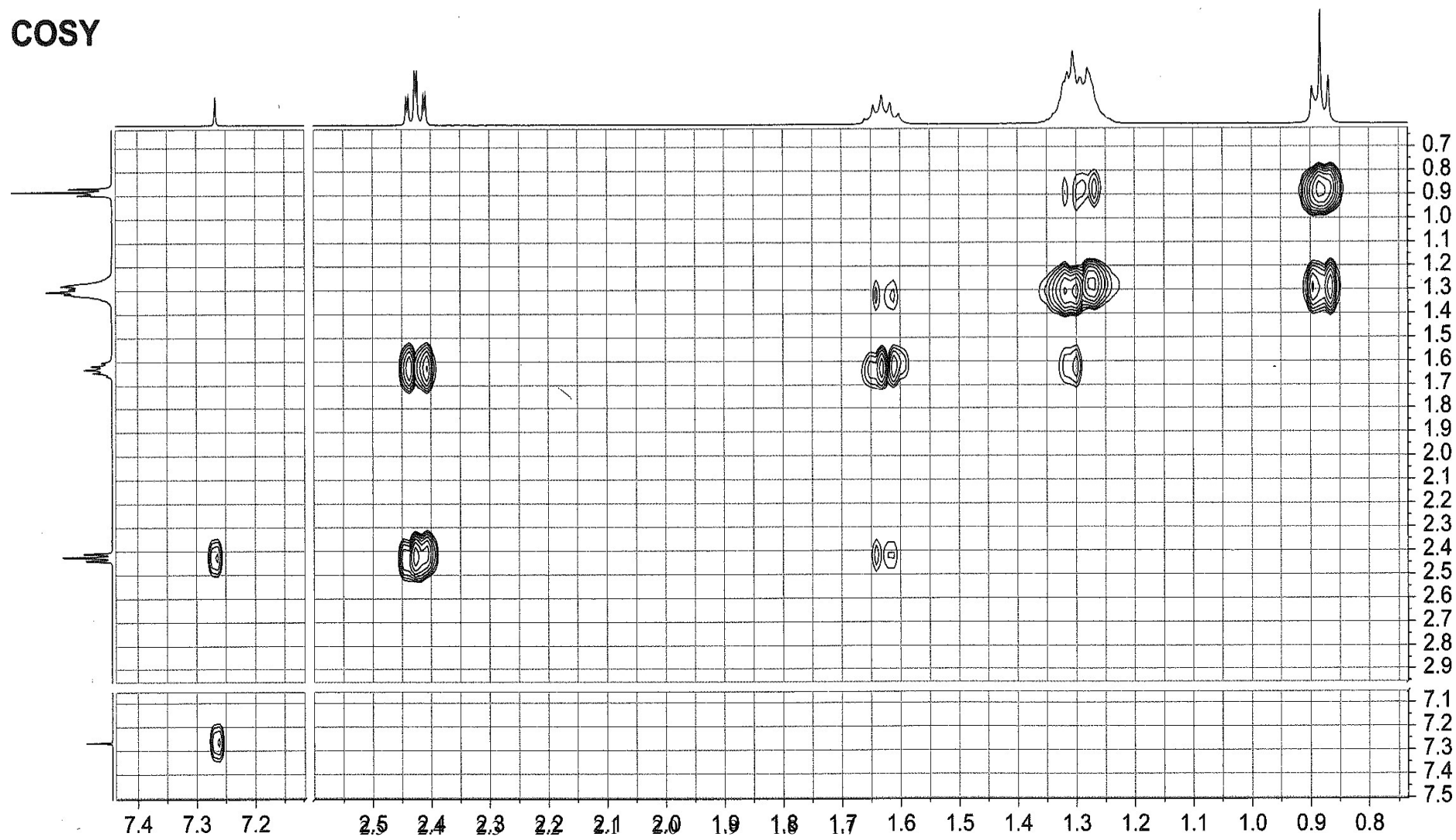
HMBC spektar spoja snimljen u DMSO-d<sub>6</sub>.



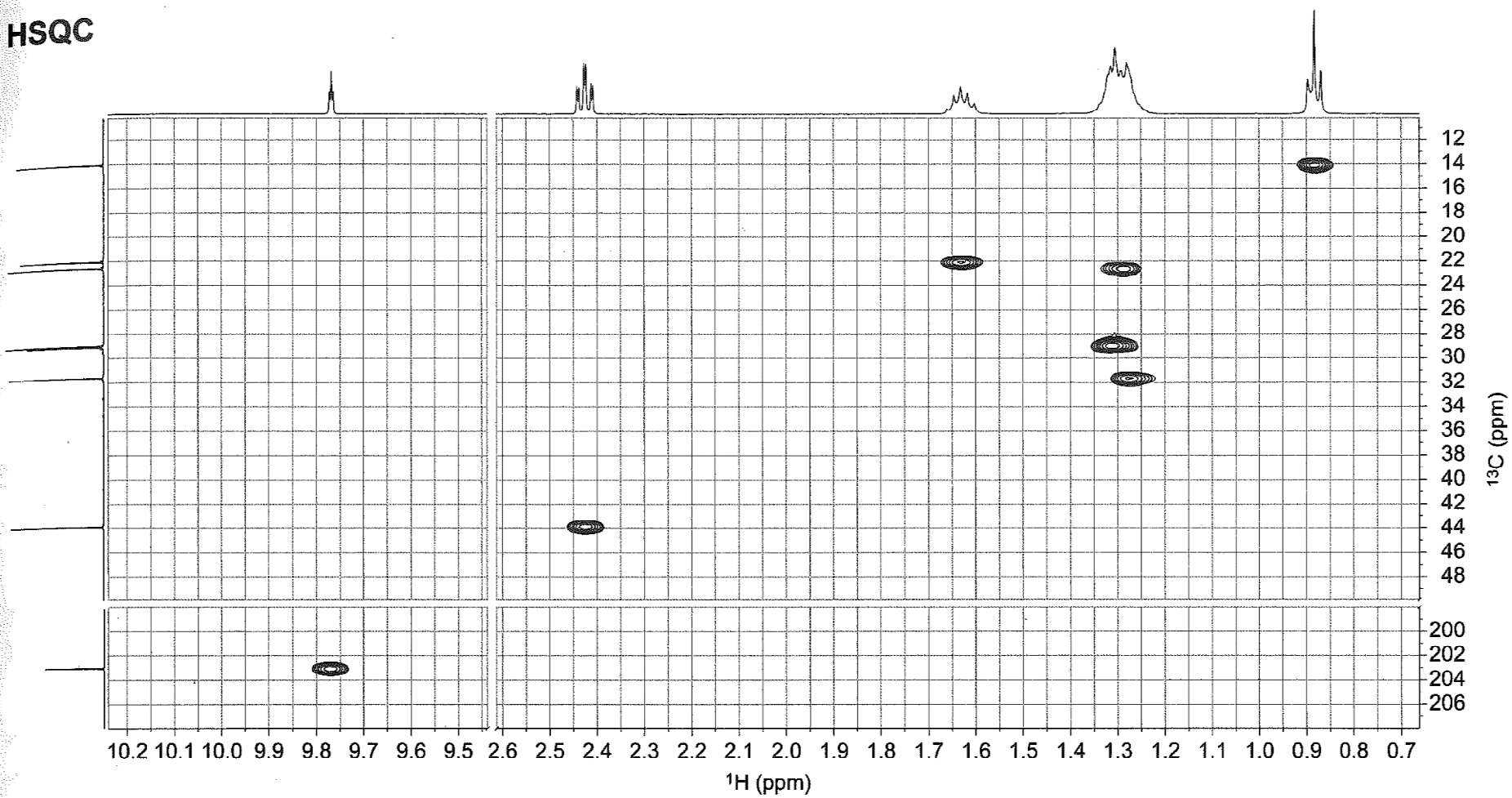
4. Odredite strukturu spoja molekulske formule  $C_8H_{16}O$  na temelju njegovog  $^1H$  NMR,  $^{13}C$  NMR, COSY, HSQC i HMBC spektra



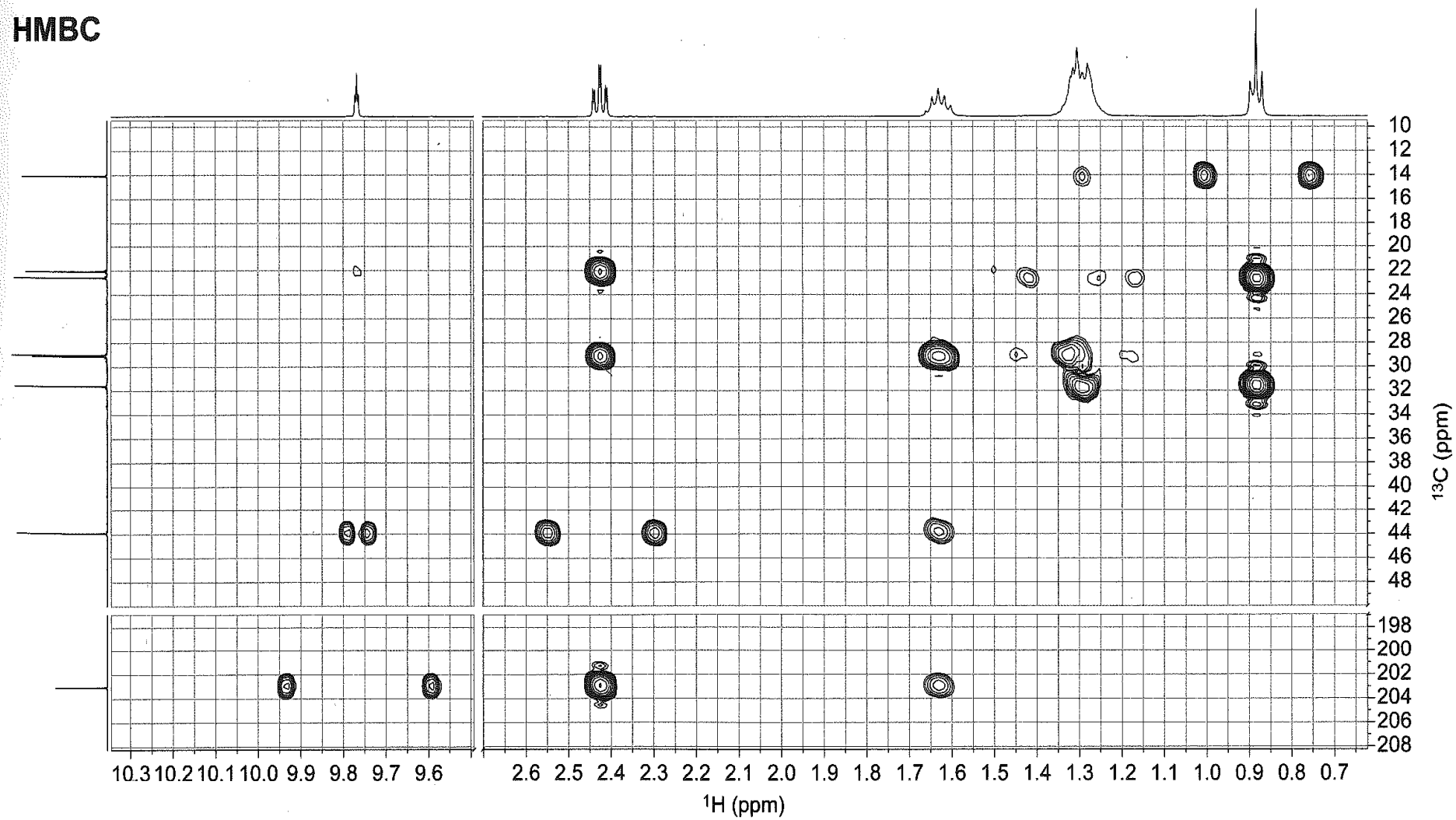
# COSY



HSQC



# HMBC

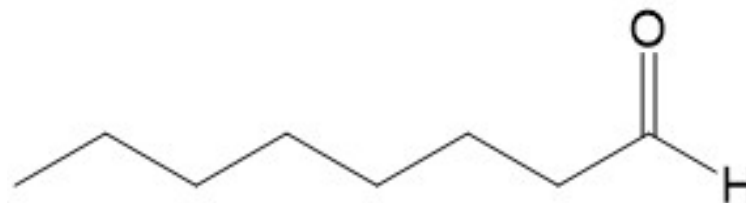


- informacije iz  $^1\text{H}$  NMR:

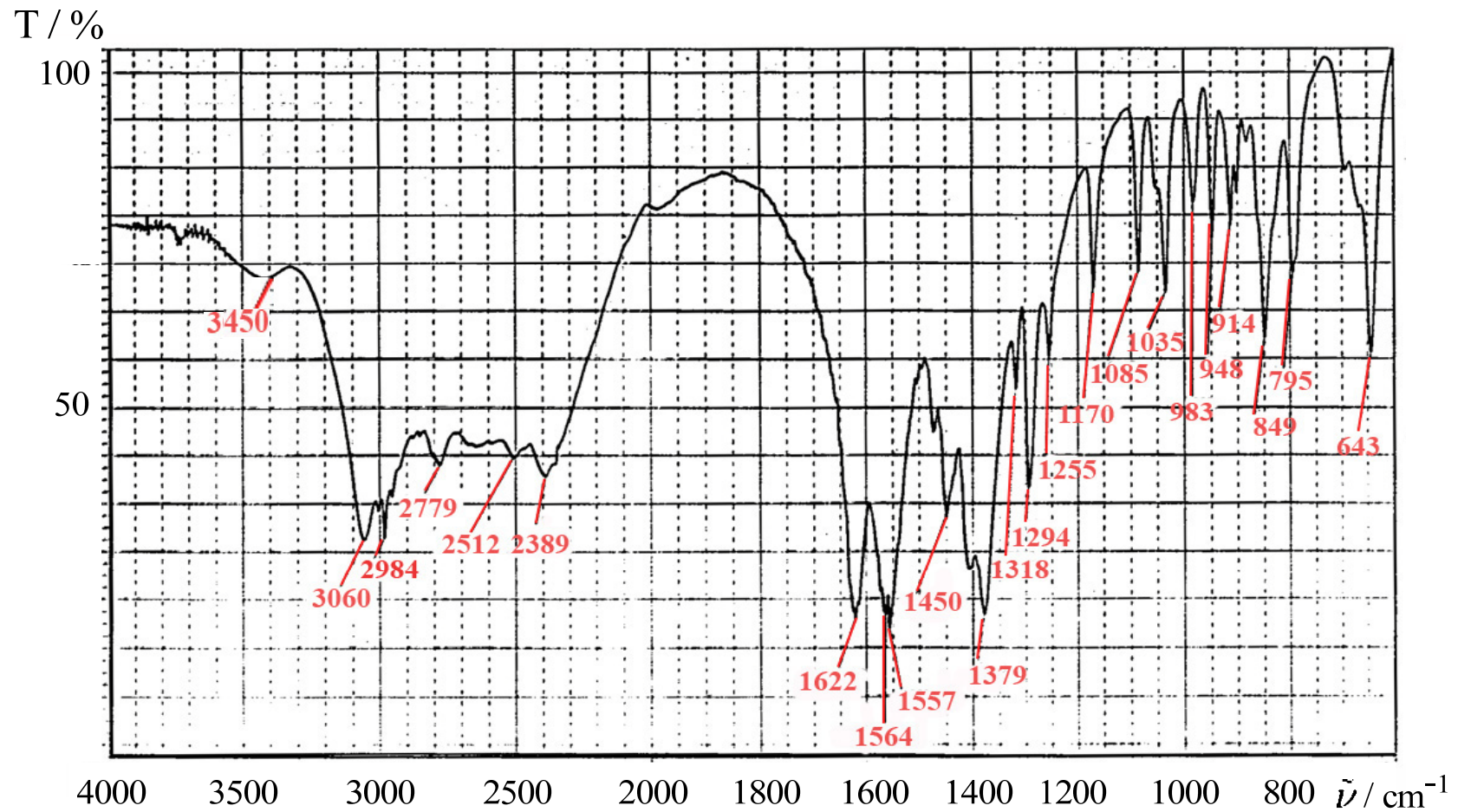
$\delta(^1\text{H})/\text{ppm}$	multiplet	H-atom
0,88	t	$-\text{CH}_3$
1,28	m	$-\text{CH}_2-$
1,29	m	$-\text{CH}_2-$
1,31	m	$-\text{CH}_2-$
1,31	m	$-\text{CH}_2-$
1,62	m	$-\text{CH}_2-$
2,42	dt	$-\text{CH}_2-$
9.77	t	$=\text{CH}-$

- informacije iz  $^{13}\text{C}$  NMR:

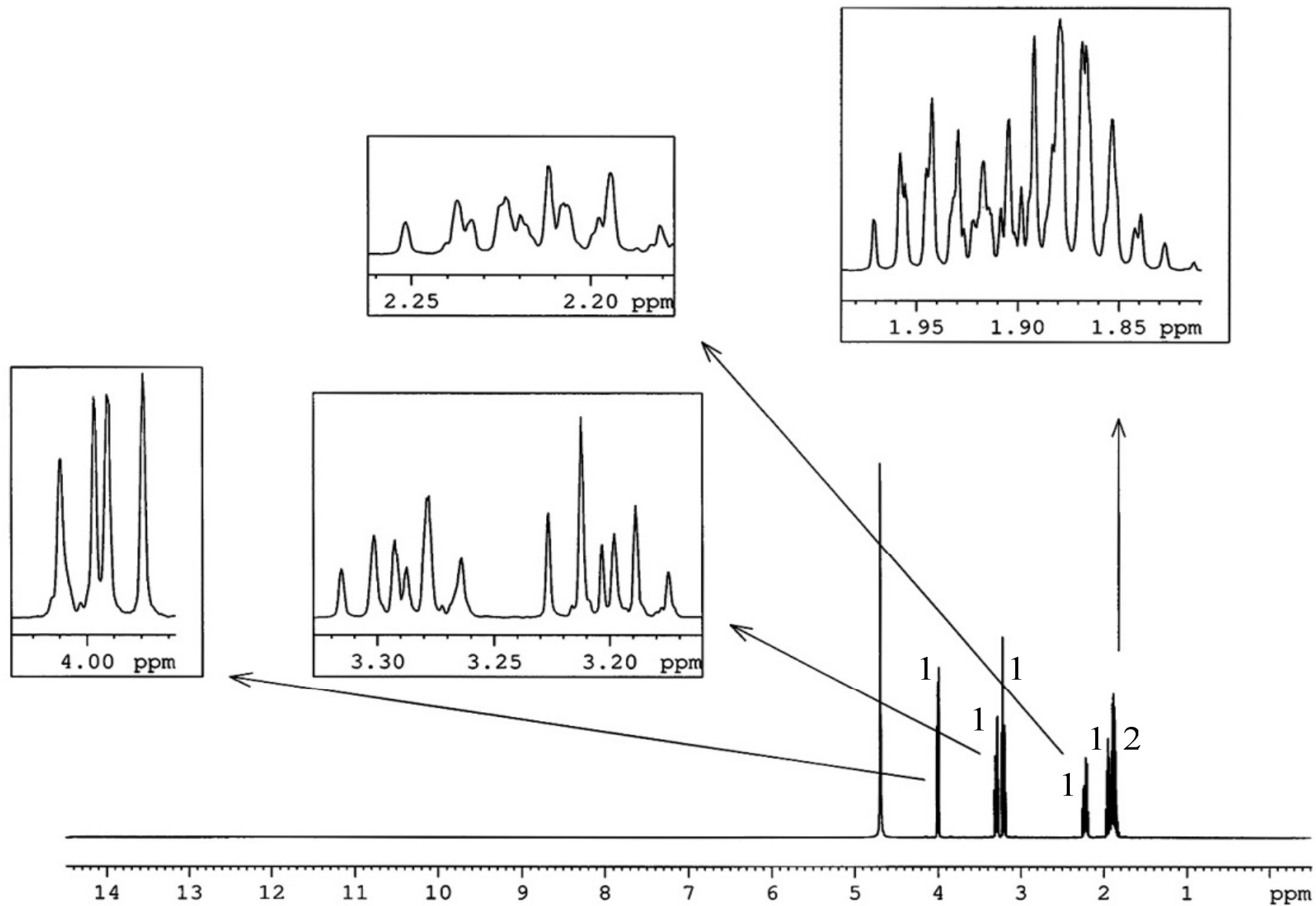
$\delta(^{13}\text{C})/\text{ppm}$	C-atom
14,1	$-\text{CH}_3$
22,2	$-\text{CH}_2-$
22,6	$-\text{CH}_2-$
29,1	$-\text{CH}_2-$
29,1	$-\text{CH}_2-$
31,7	$-\text{CH}_2-$
44,0	$-\text{CH}_2-$
203,1	$=\text{CH}-$



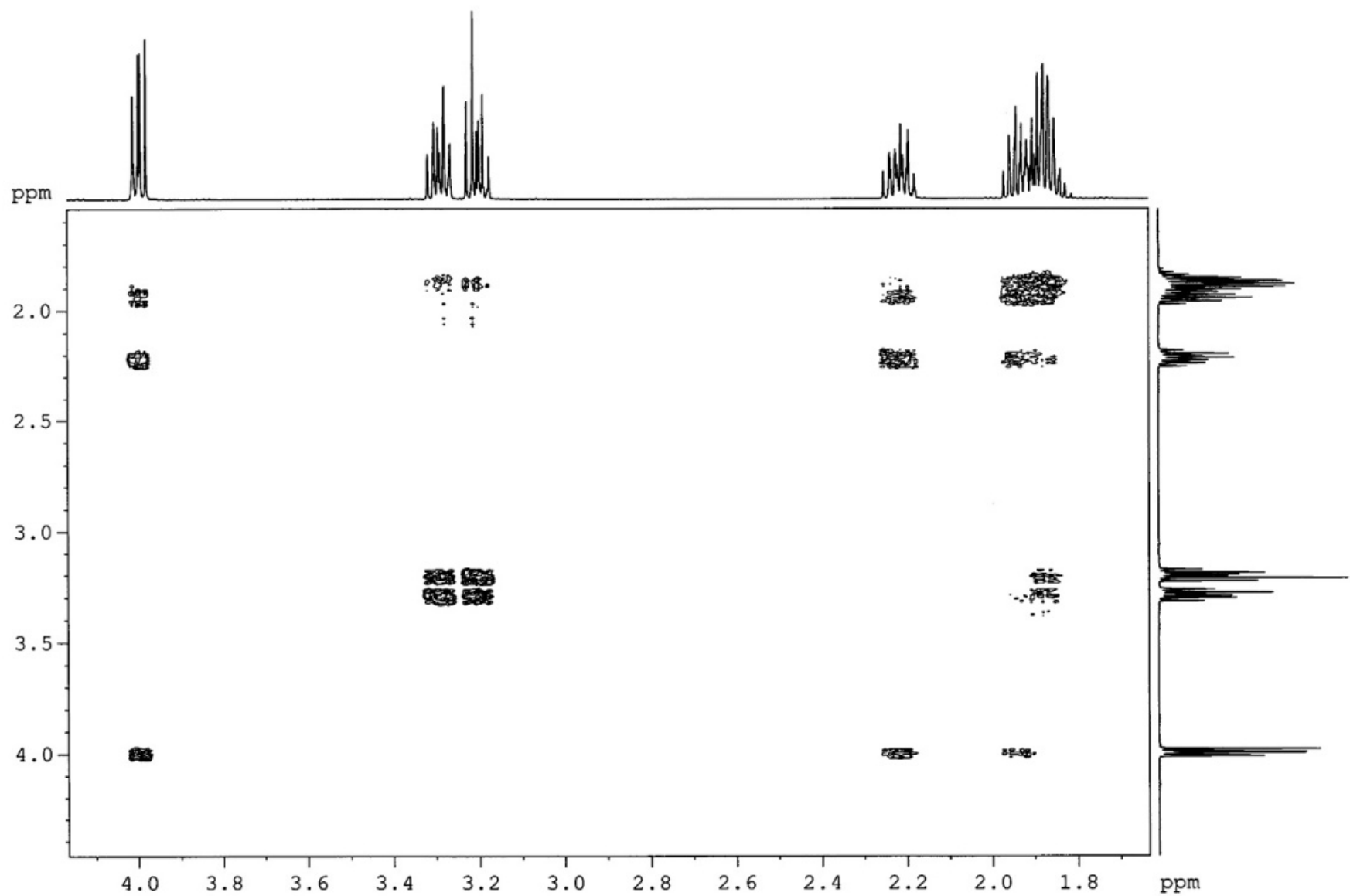
5. Odredite strukturu spoja molekulske mase 115 Da na temelju njegovog IR,  $^1\text{H}$  NMR, COSY, HMQC i HMBC spektra



IR spektar spoja snimljen tehnikom KBr pastile.

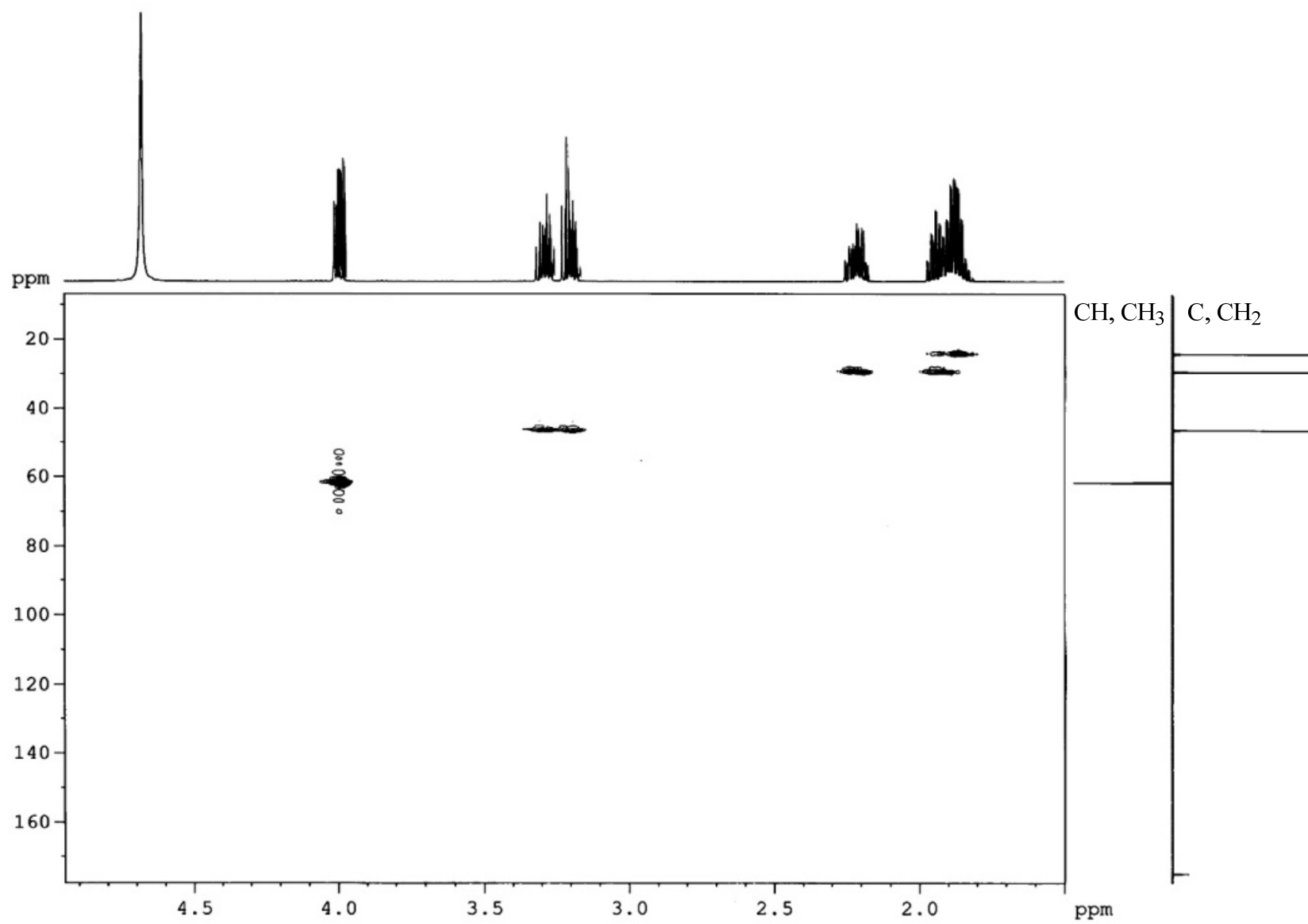


$^1\text{H}$  NMR spektar spoja snimljen u  $\text{D}_2\text{O}$  uz uvećani prikaz područja koja odgovaraju pojedinim signalima.

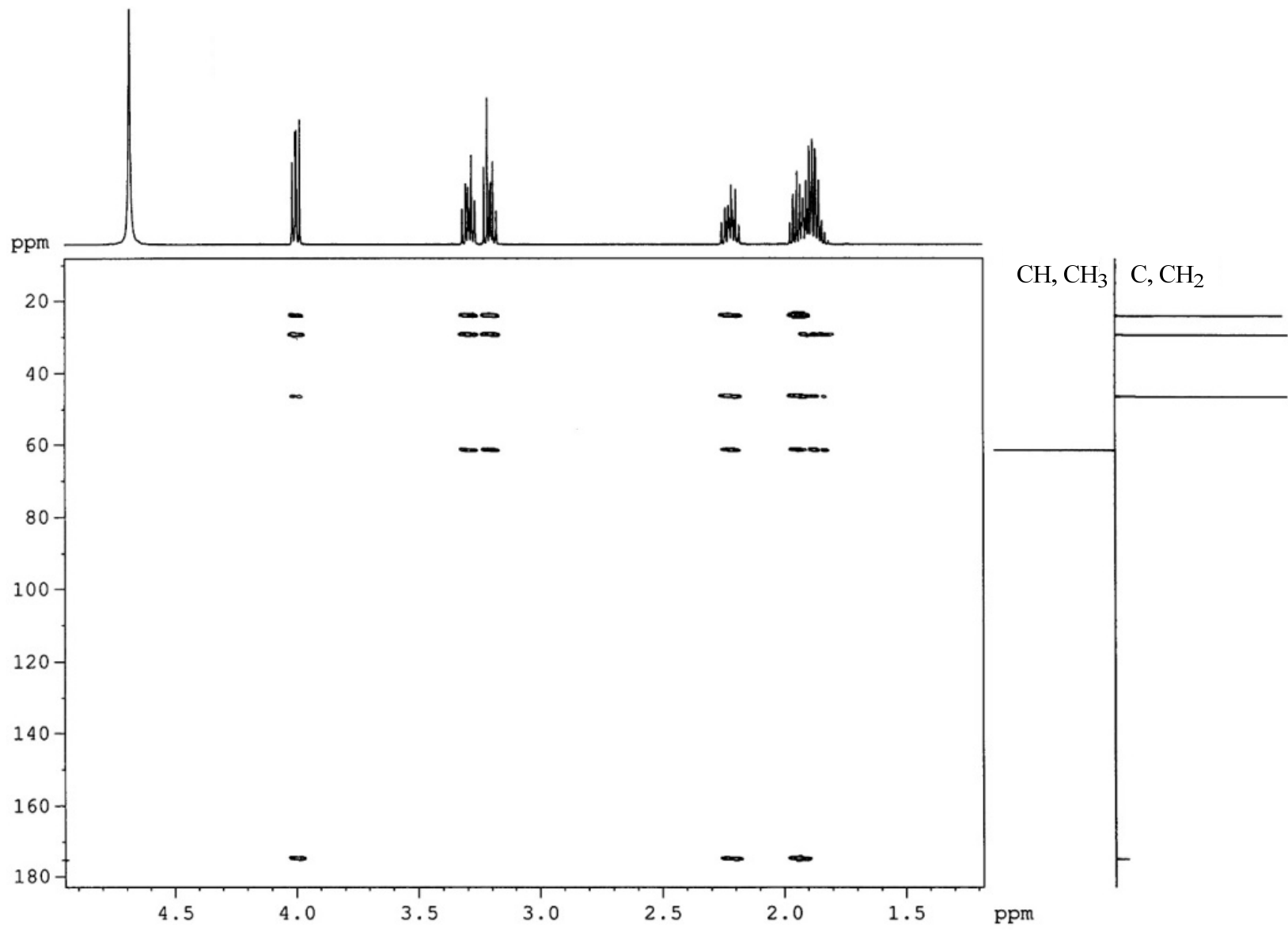


COSY spektar spoja snimljen u D<sub>2</sub>O.





HMQC spektar spoja snimljen u D<sub>2</sub>O.



HMBC spektar spoja snimljen u  $\text{D}_2\text{O}$ .

### Karakteristične vibracijske vrpce u IR spektru

$\nu / \text{cm}^{-1}$	Način vibracije
3200–2000	$\nu (\text{NH}_2^+)$
3060, 2984	$\nu (\text{C-H})$
1622	$\delta (\text{NH}_2^+)$
1564	$\nu_{\text{as}} (\text{COO}^-)^{\text{a}}$
1450	$\nu_{\text{s}} (\text{COO}^-)^{\text{b}}$

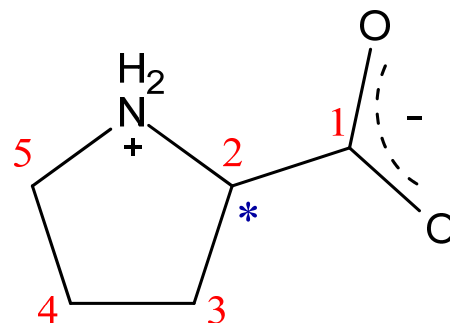
<sup>a</sup> – asimetrično istežanje; <sup>b</sup> – simetrično istežanje

### Asignacija $^1\text{H}$ NMR kemijskih pomaka

H-atom	$\delta / \text{ppm}$	Intenzitet	Multipletnost
1			
2	4,00	1	dd
3	1,95	1	m
3	2,22	1	m
4	1,87	2	m
5	3,20	1	m
5	3,30	1	m

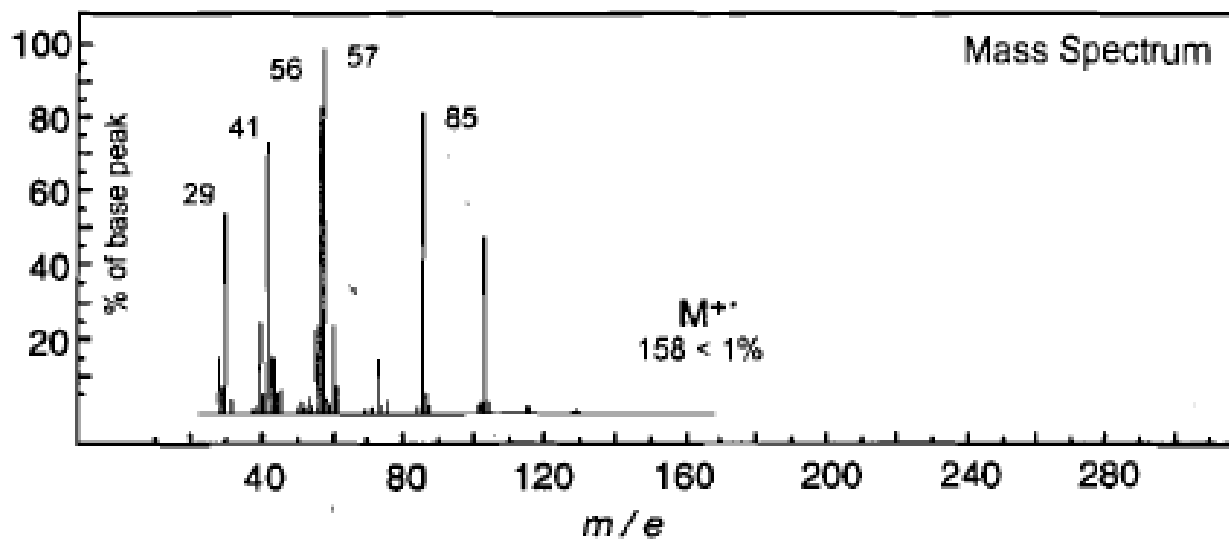
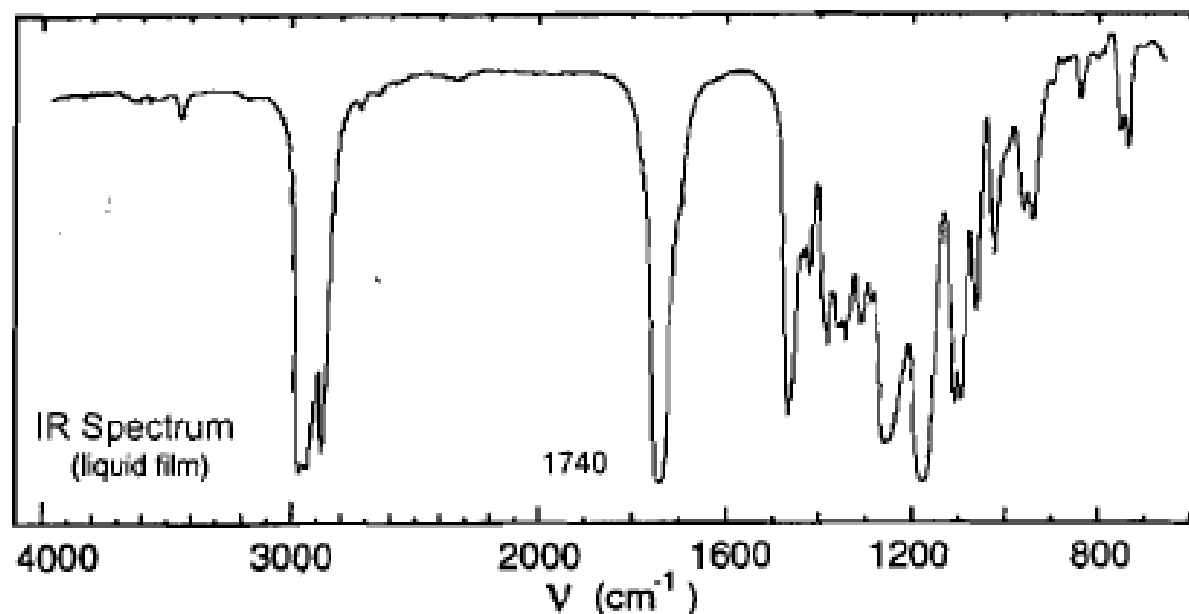
### Asignacija $^{13}\text{C}$ NMR kemijskih pomaka

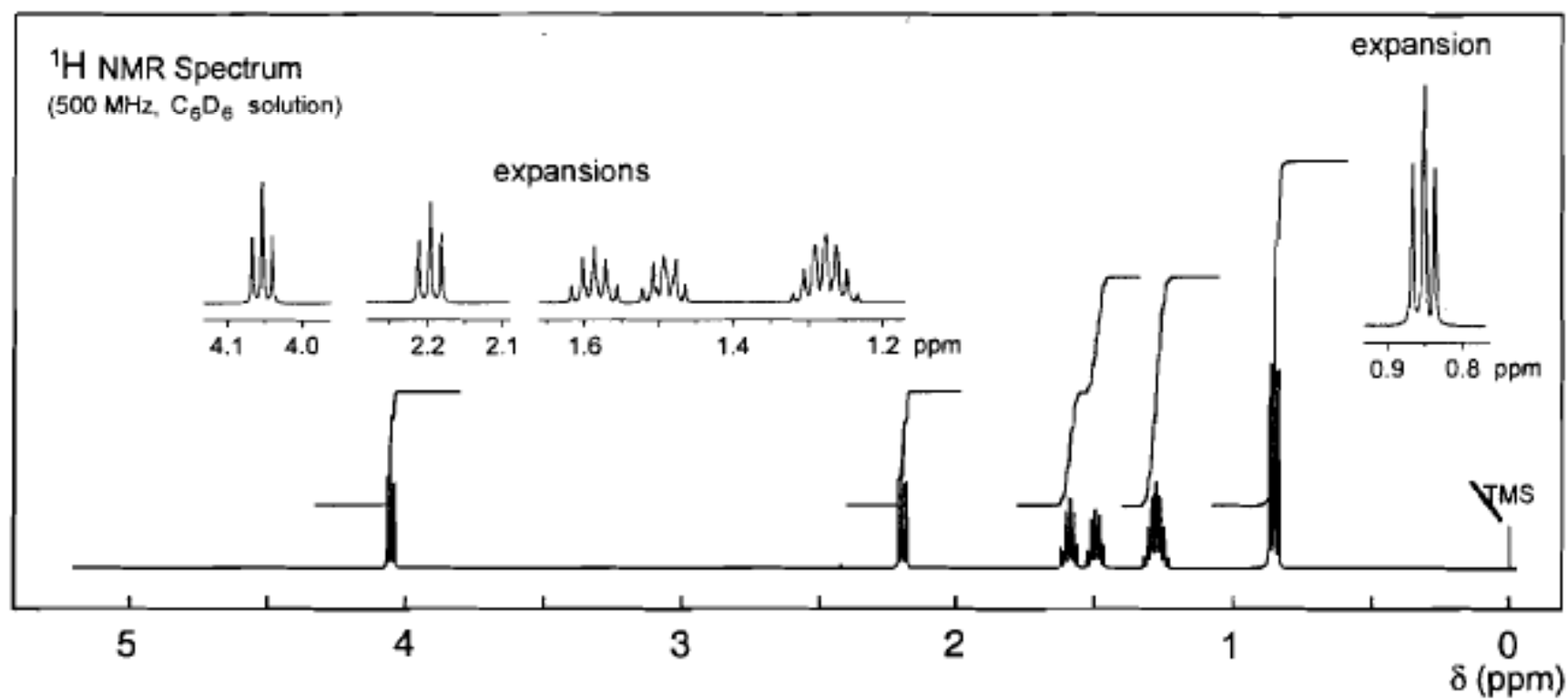
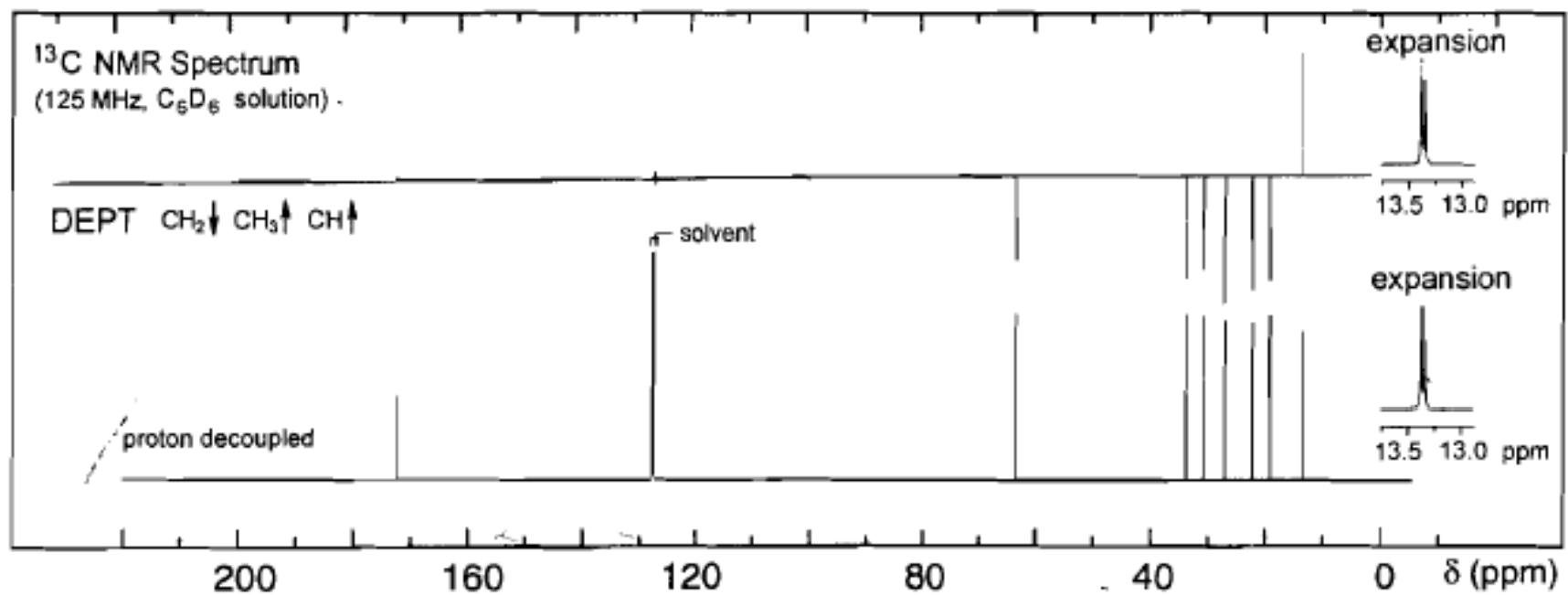
C-atom	$\delta$ / ppm
1	175
2	63
3	30
4	25
5	43

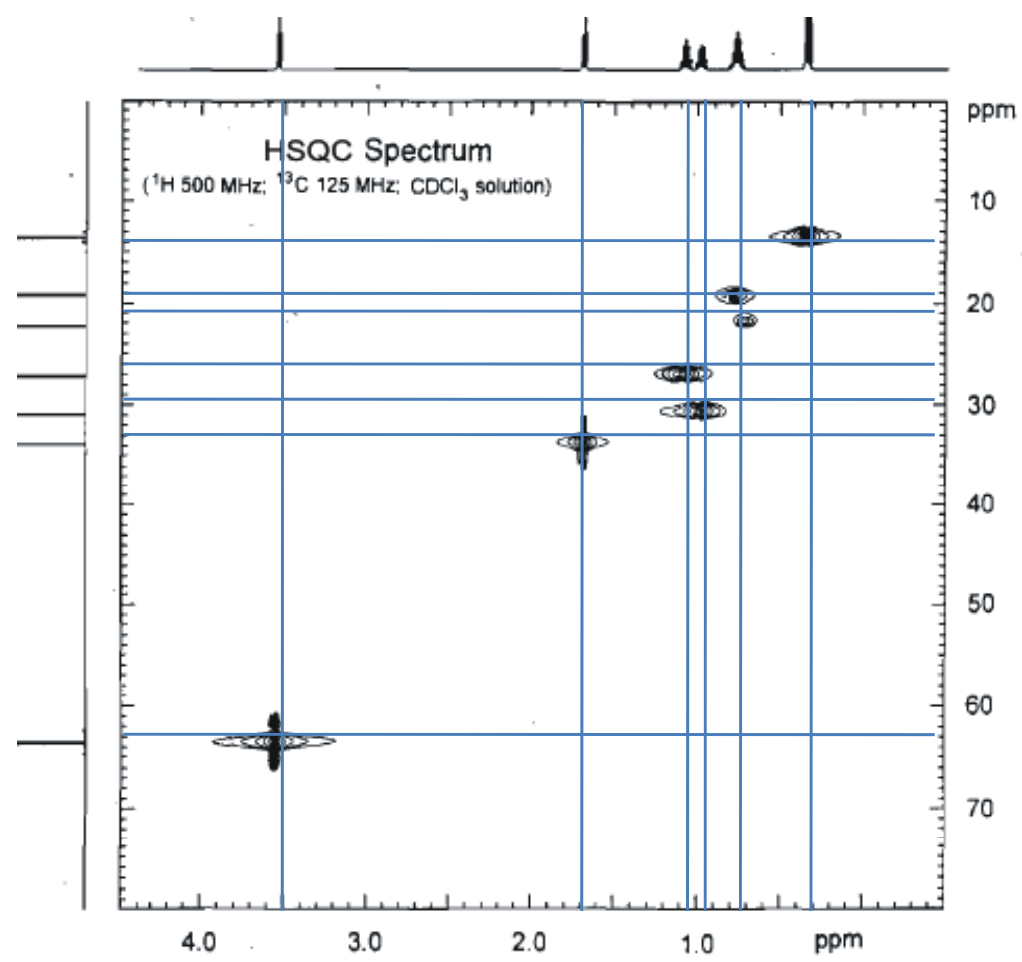
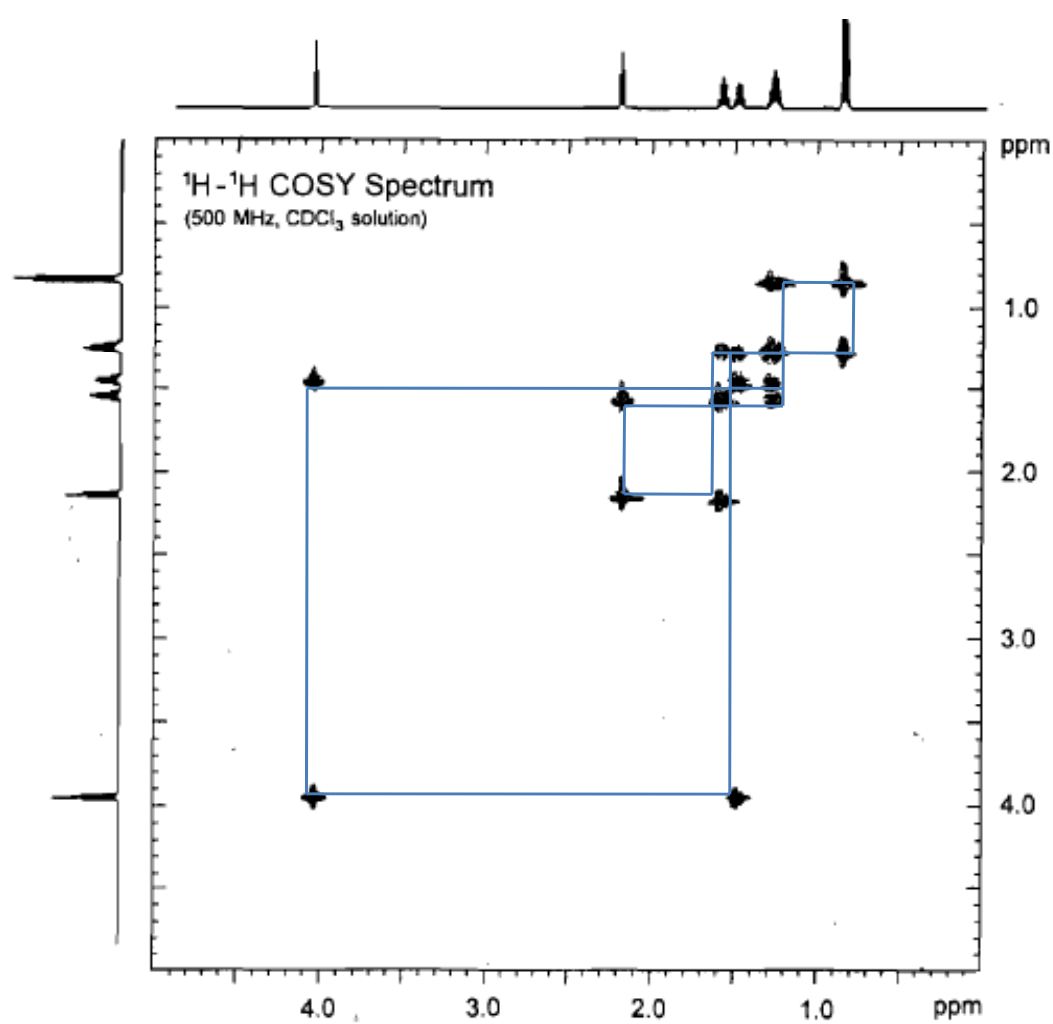


Struktura spoja i brojanje atoma. Simbolom \* označen je kiralni centar.

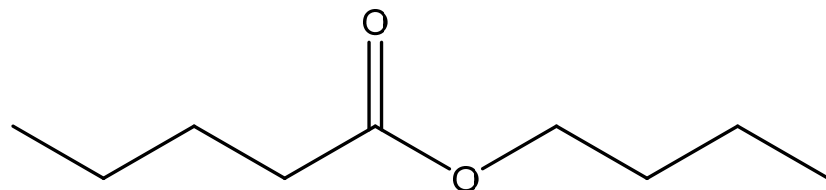
6. Odredite strukturu spoja na temelju njegovog IR, MS,  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR, COSY i HSQC spektra.







- informacije iz MS:  
bazni pik: 57  
 $M^+ = 158$



- informacije iz  $^1\text{H}$  NMR:

$\delta(^1\text{H})/\text{ppm}$	multiplet	H-atom
0,85	t	$-\text{CH}_3$
1,27	m (septet)	$-\text{CH}_2-$
1,49	m (kvintet)	$-\text{CH}_2-$
1,59	m (kvintet)	$-\text{CH}_2-$
2,20	t	$-\text{CH}_2-$
4,05	t	$-\text{CH}_2-$

- informacije iz IR:  
 $2950\text{ cm}^{-1}$  C–H alifatsko istežanje  
 $1740\text{ cm}^{-1}$  C=O istežanje

- informacije iz  $^{13}\text{C}$  NMR:

$\delta(^{13}\text{C})/\text{ppm}$	C-atom
13,3	$-\text{CH}_3$
13,4	$-\text{CH}_3$
19	$-\text{CH}_2-$
22	$-\text{CH}_2-$
27	$-\text{CH}_2-$
31	$-\text{CH}_2-$
34	$-\text{CH}_2-$
63	$-\text{CH}_2-$
173	$\begin{array}{c} \diagup \\ \text{C} \\ \diagdown \end{array}$