université PARIS-SACLAY | GRADUATE SCHOOL Health and Drug Sciences

MASTER
Sciences du Médicament et des Produits de Santé

université PARIS-SACLAY

OTU 06

Basic structural elucidation

Practical work

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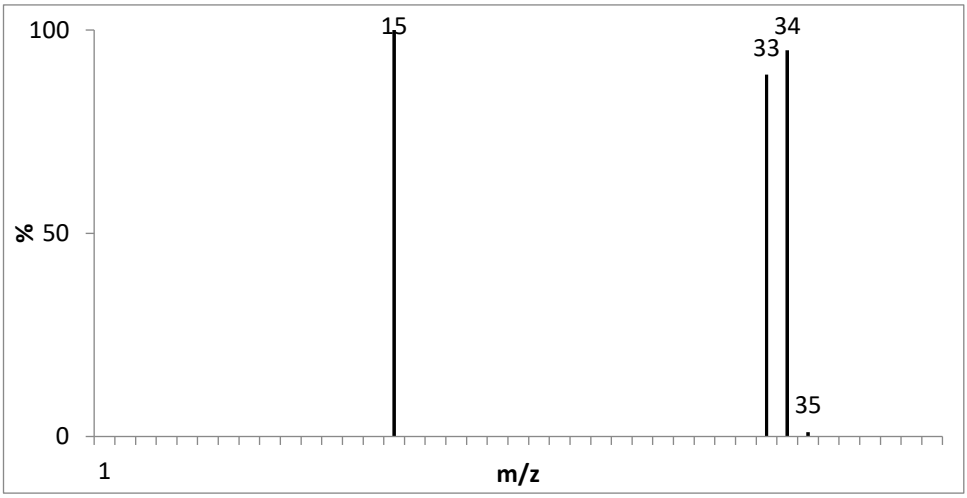
2024-2025

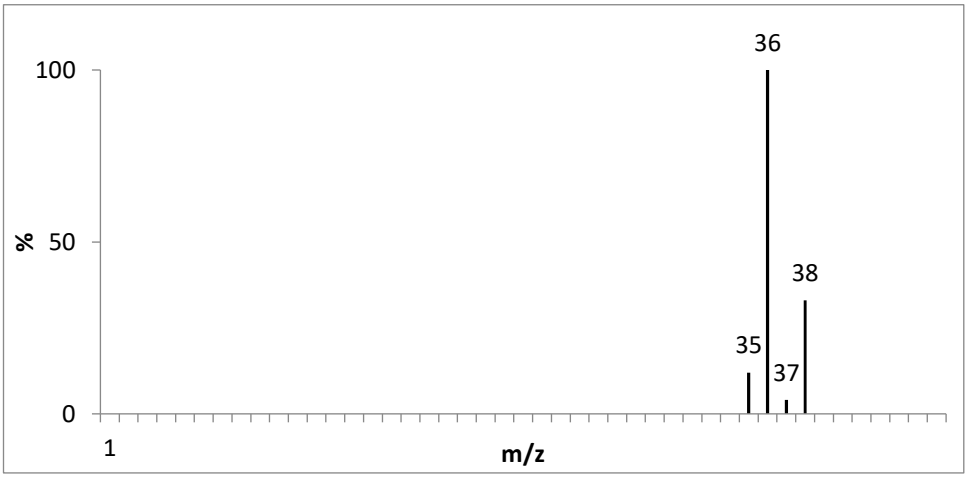
Mass spectrometry

Exercise 1:



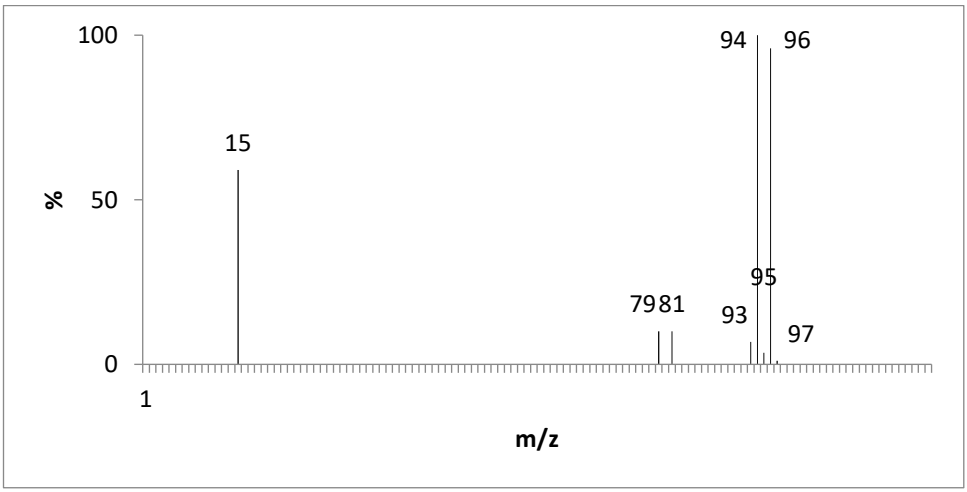
m/z	%
15	100
33	89
34	95
35	1,1

Exercise 2:



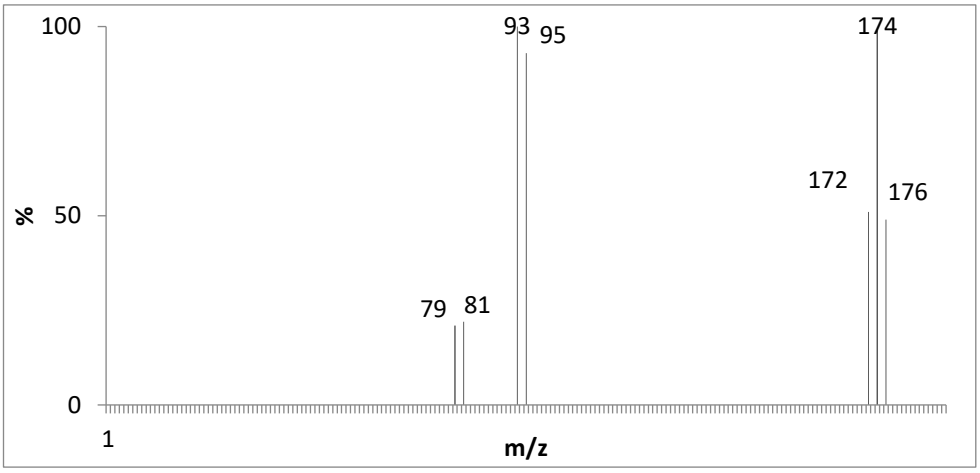
m/z	%
35	12
36	100
37	4,1
38	33

Exercise 3:



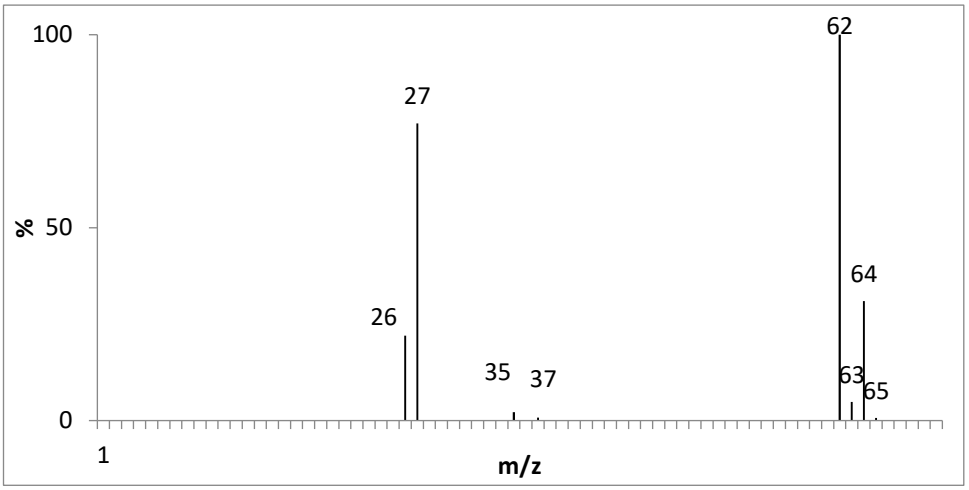
m/z	%
15	59
79	10
81	10
93	6,8
94	100
95	3,5
96	96
97	1,1

Exercise 4:



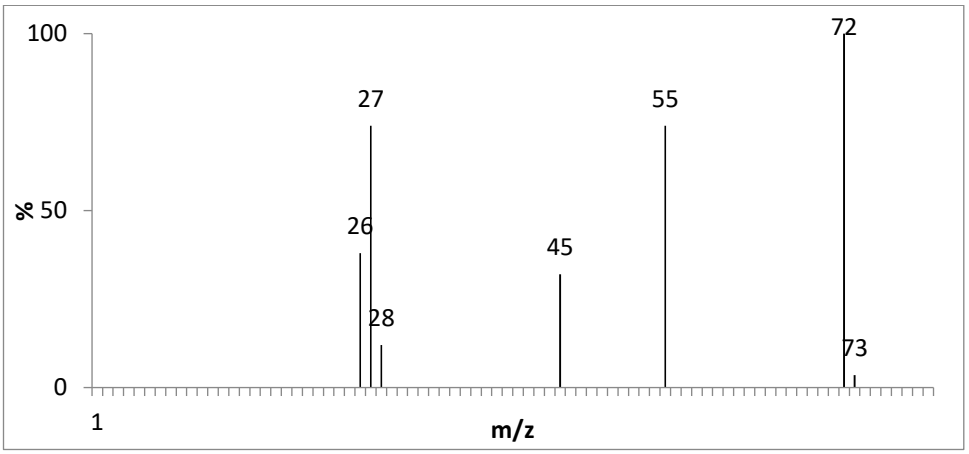
m/z	%
79	21
81	22
93	100
95	93
172	51
174	99
176	49

Exercise 5:



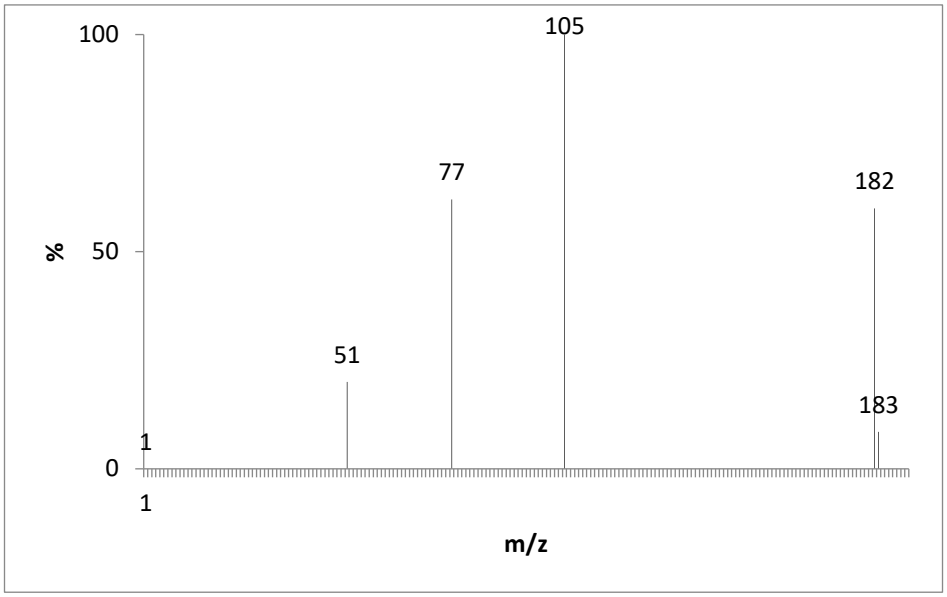
m/z	%
26	22
27	77
35	2,2
37	0,8
62	100
63	4,8
64	31
65	0,7

Exercise 6: $C_3H_4O_2$



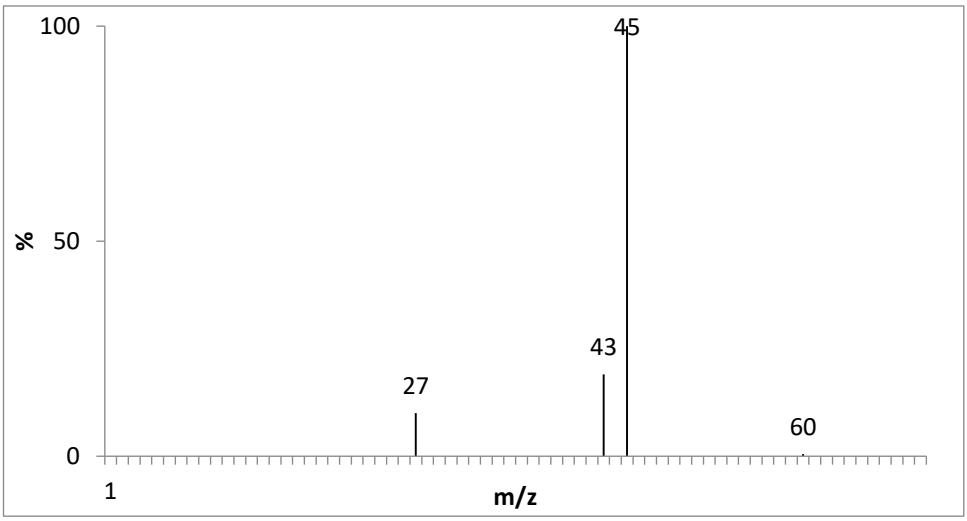
m/z	%
26	38
27	74
28	12
45	32
55	74
72	100
73	3,5

Exercise 7:



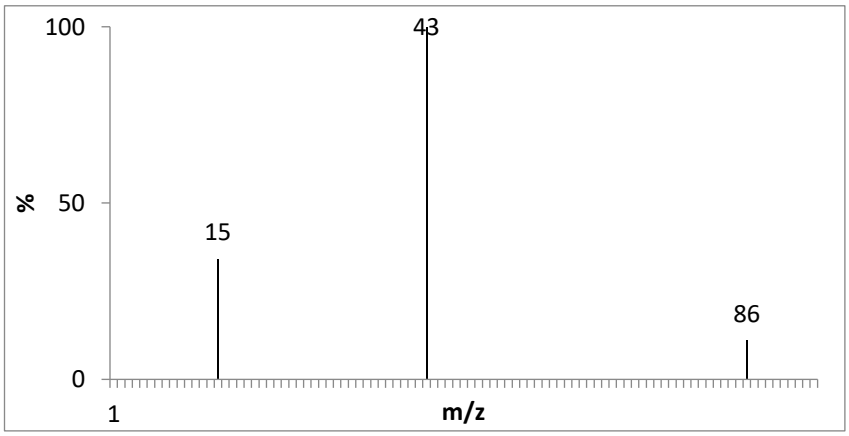
m/z	%
51	20
77	62
105	100
182	60

Exercise 8: C_3H_8O



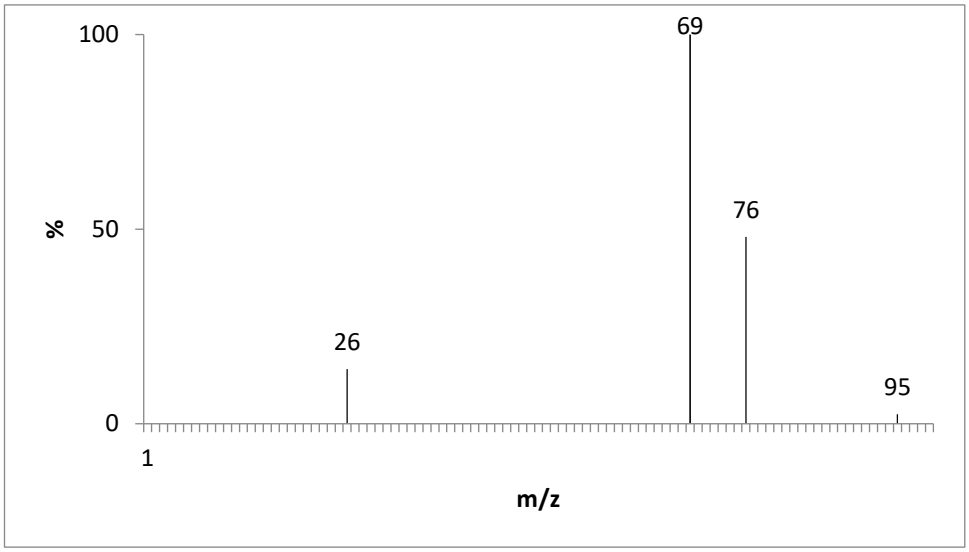
m/z	%
27	10
43	19
45	100
60	0,51

Exercise 9: $C_4H_6O_2$



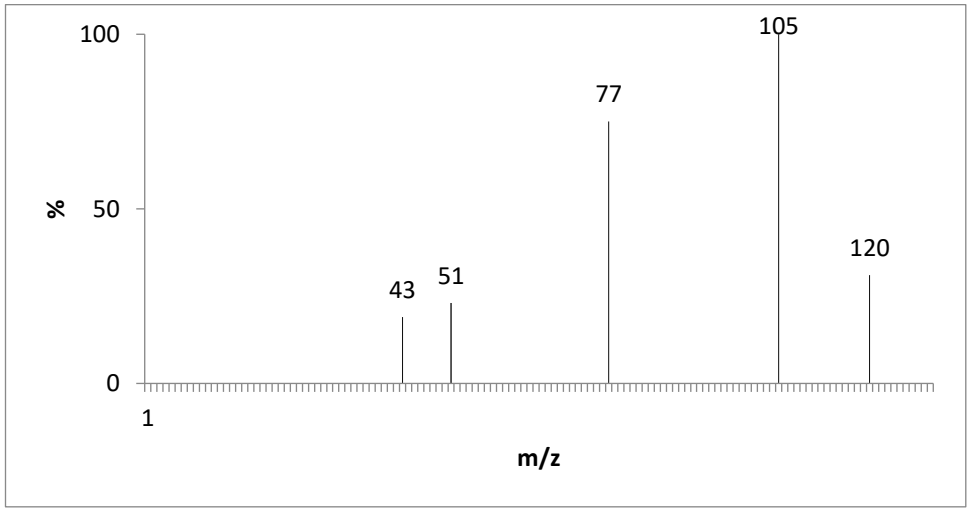
m/z	%
15	34
43	100
86	11

Exercise 10:



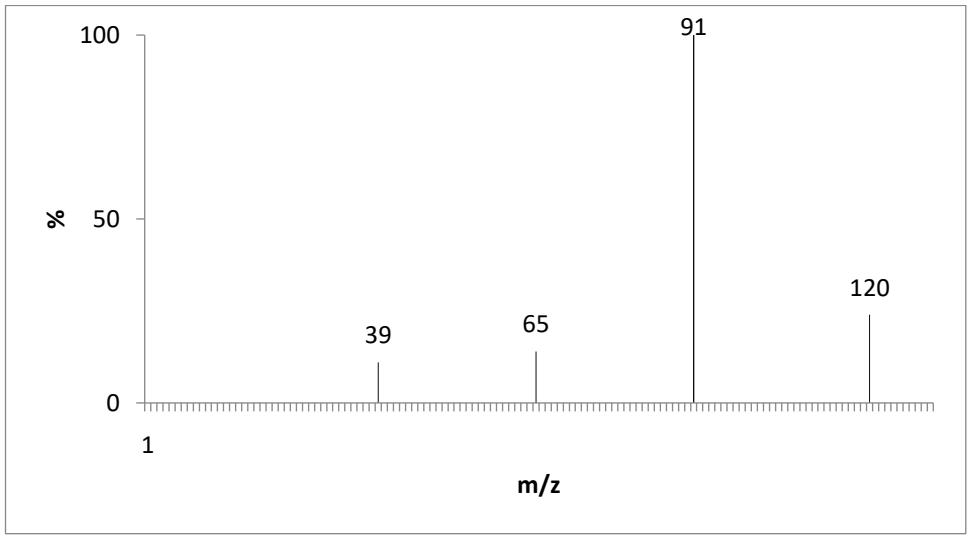
m/z	%
26	14
31	22
69	100
76	48
95	2,4

Exercise 11: C_8H_8O



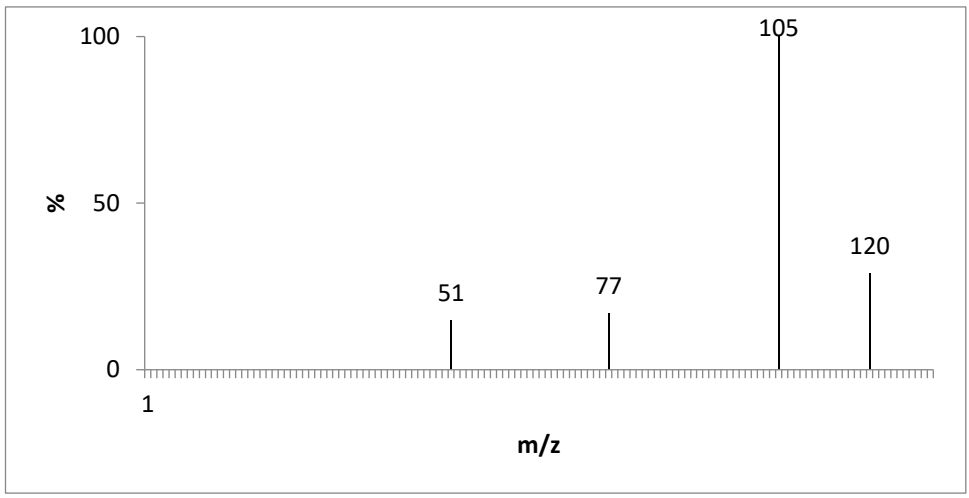
m/z	%
43	19
51	23
77	75
105	100
120	31

Exercise 12: C_9H_{12}



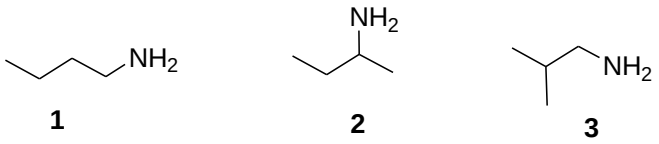
m/z	%
39	11
65	14
91	100
120	24

Exercise 13: C_9H_{12}

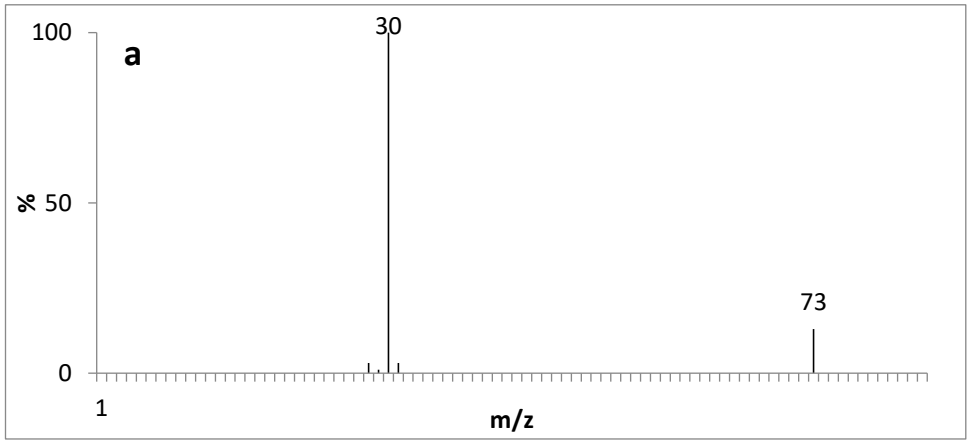


m/z	%
51	15
77	17
105	100
120	29

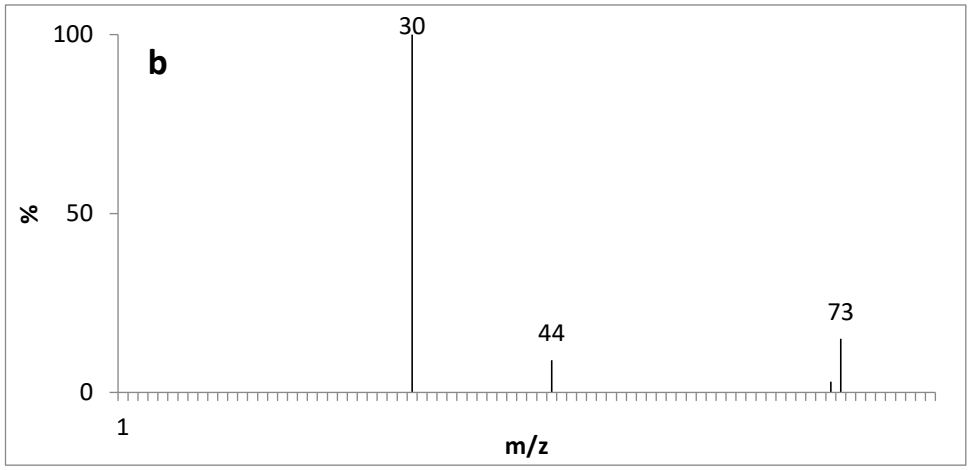
Exercise 14: Assign the corresponding MS spectrum (a, b, and c) to right amine compound having the molecular formula $C_4H_{11}N$



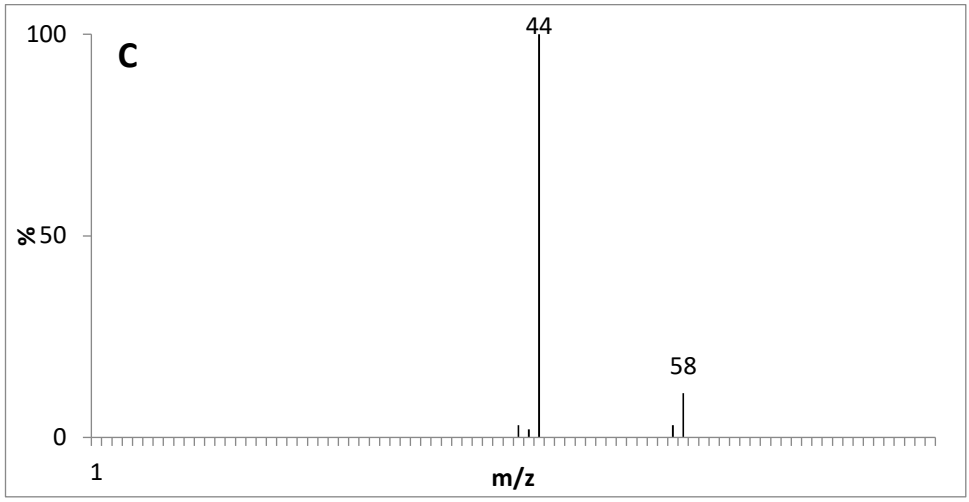
1 2 3



m/z	%
30	100
73	13

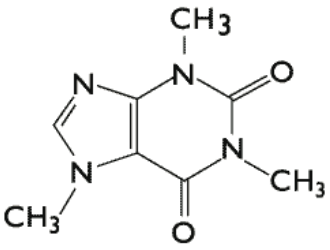


m/z	%
30	100
44	9
73	15

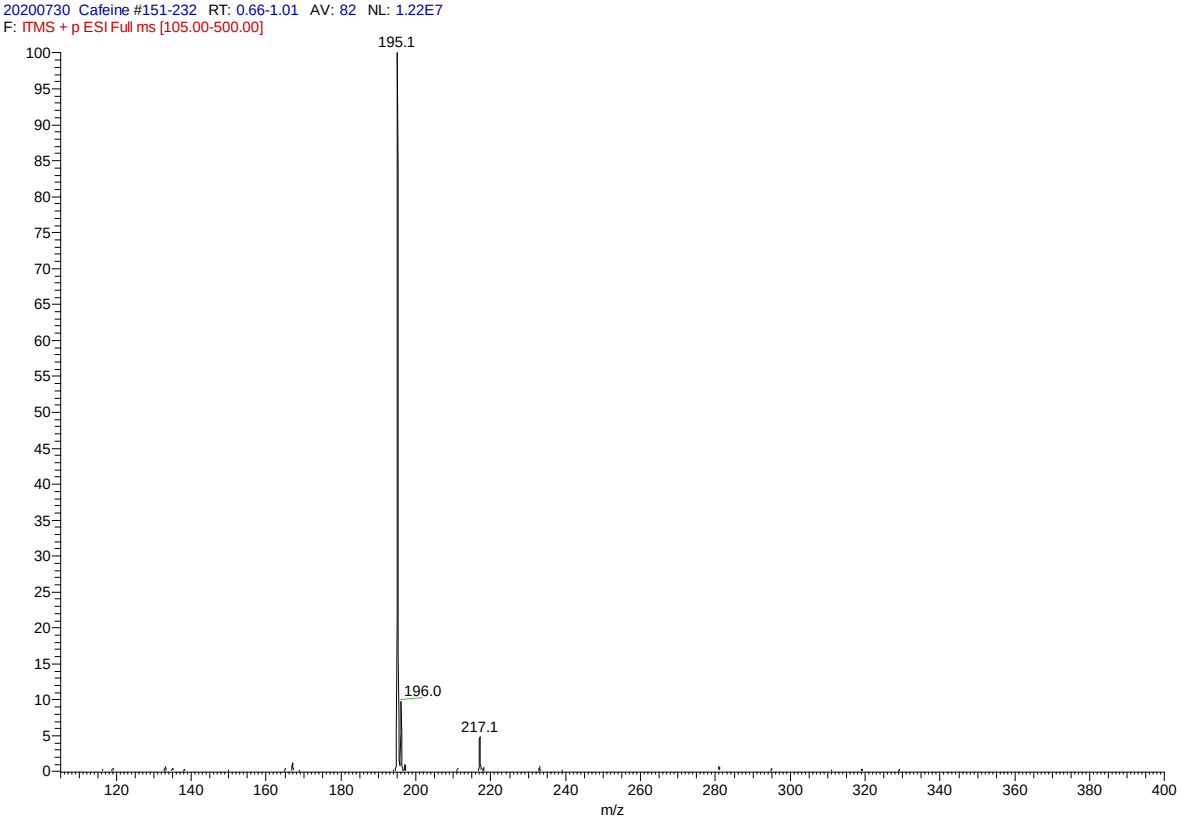


m/z	%
44	100
58	11

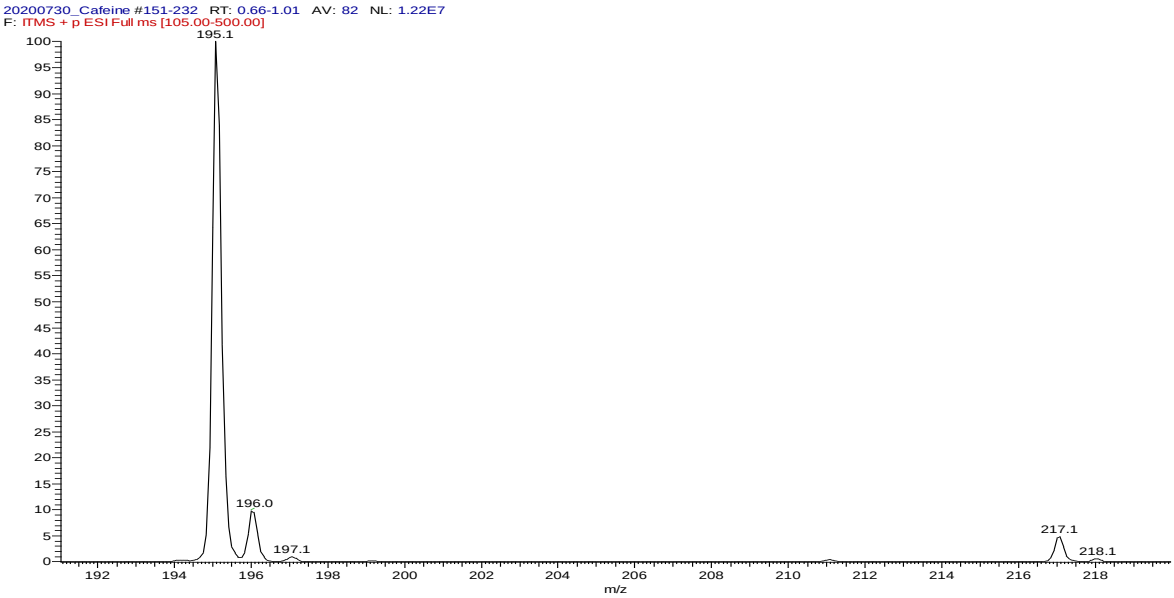
Caffeine: in methanol/water ESI (+) low resolution



$C_8H_{10}N_4O_2$ M = 194

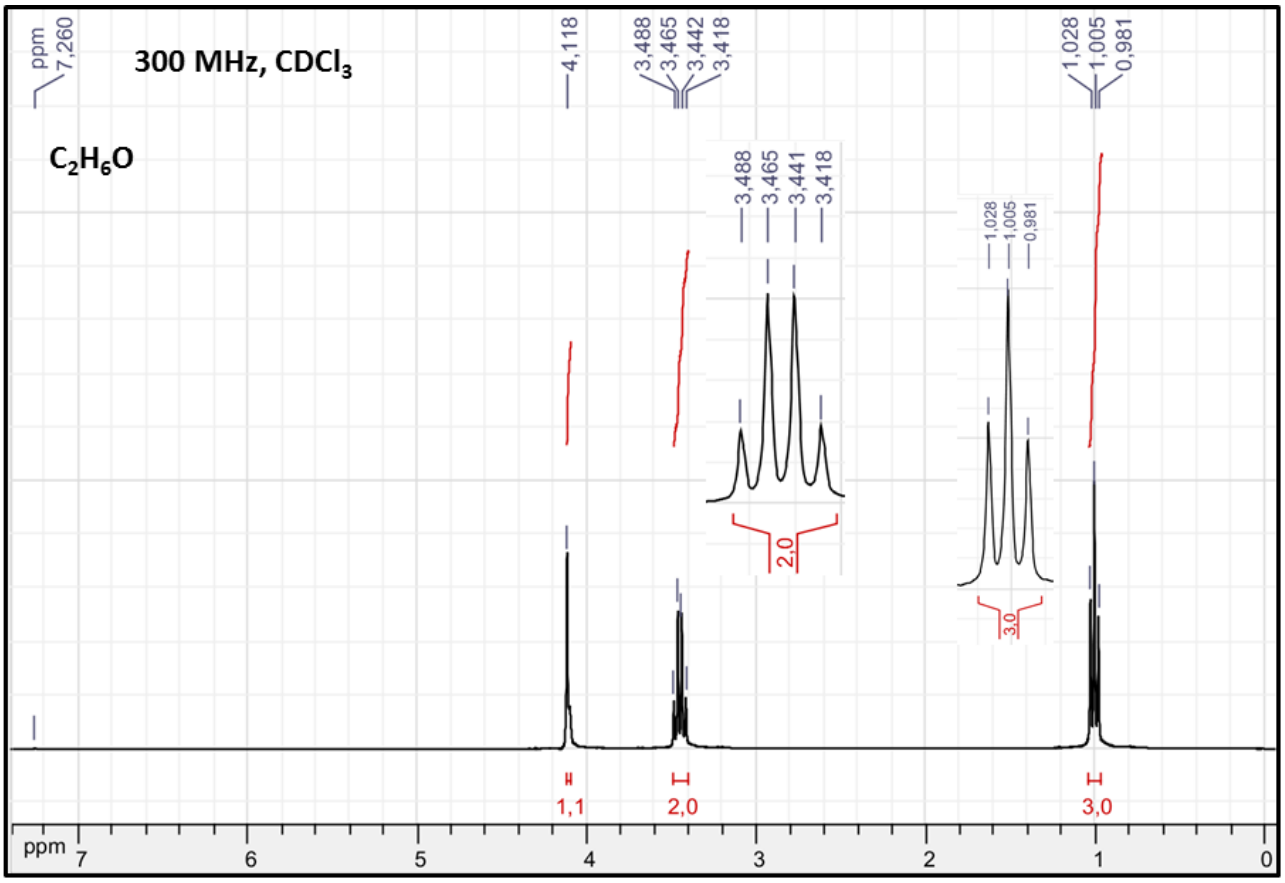


Zoom :

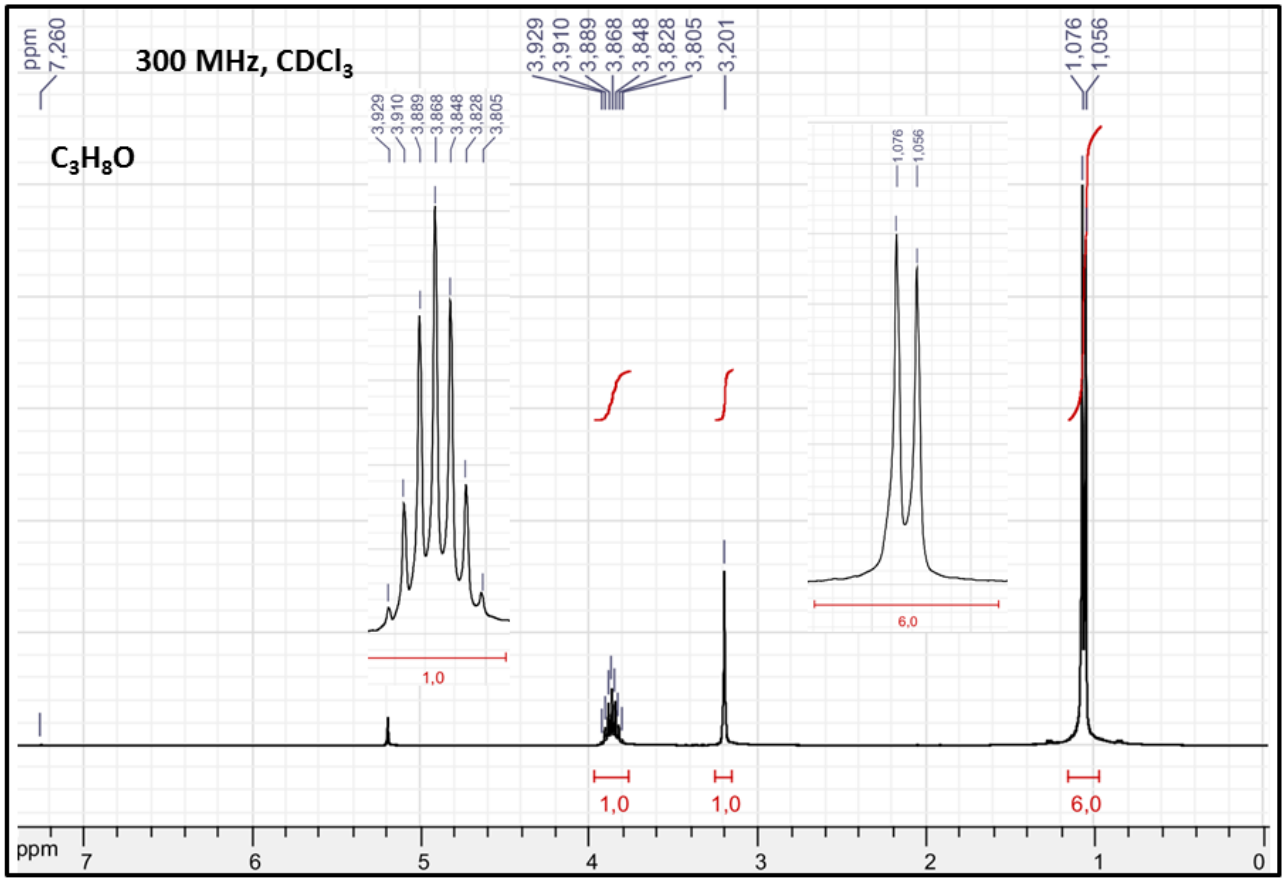


^{1}H NMR

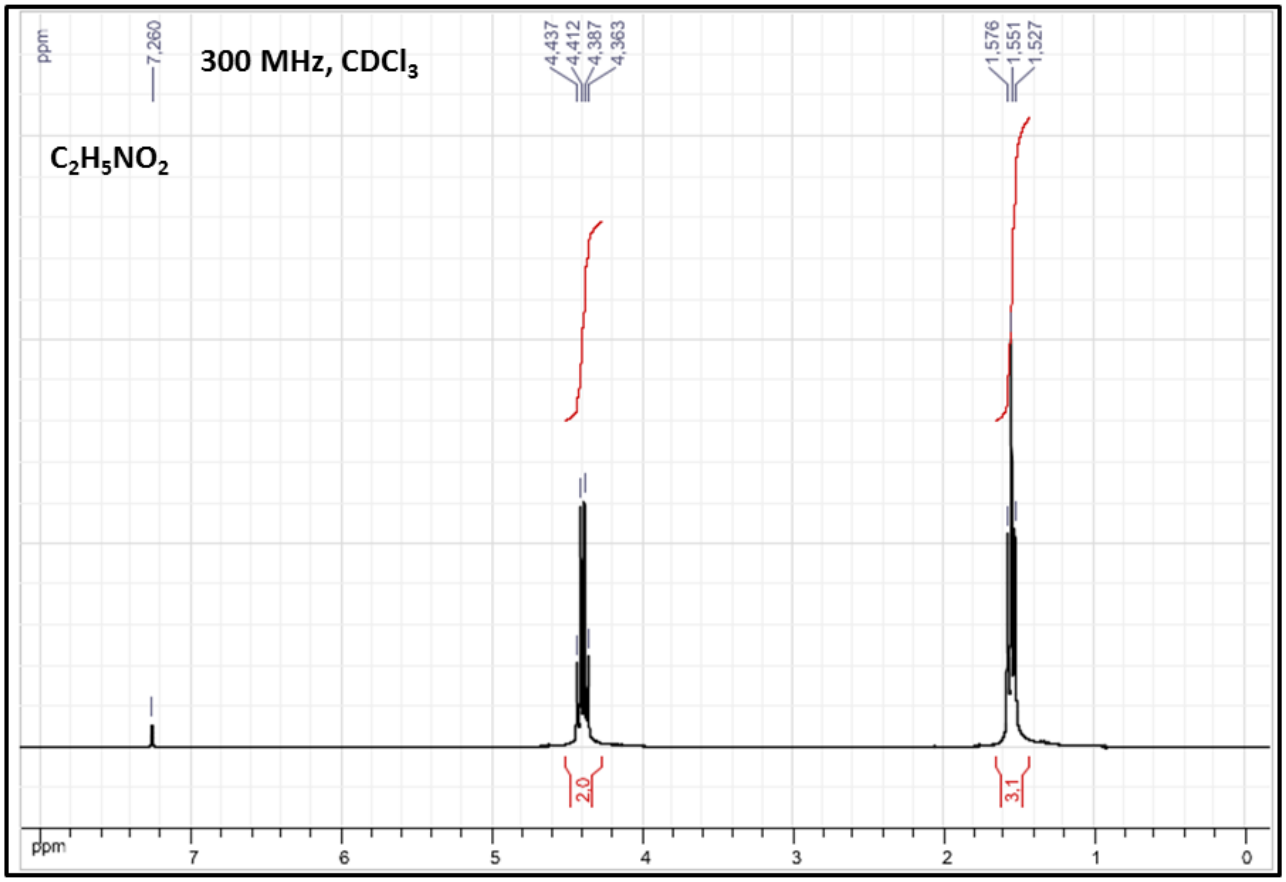
Exercise 1:



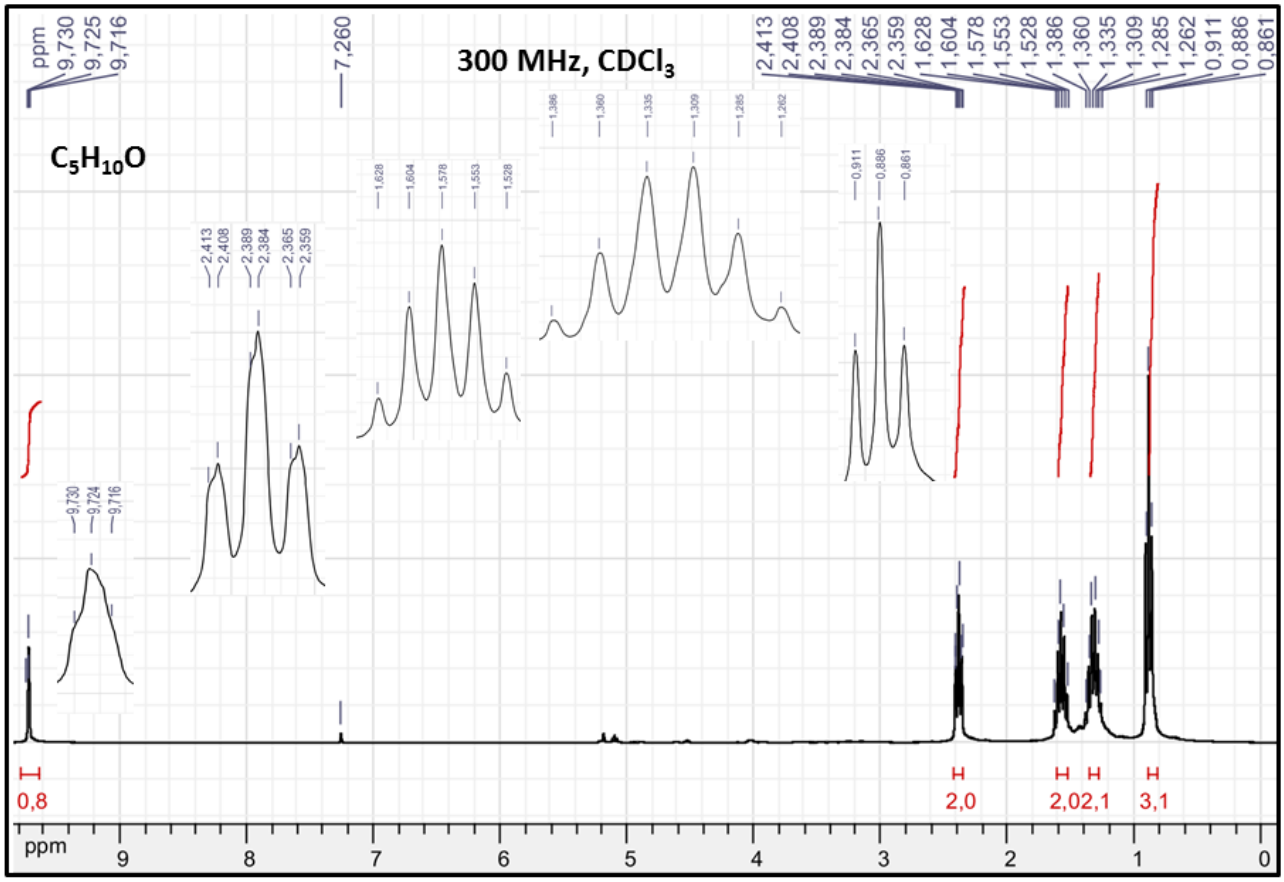
Exercise 2:



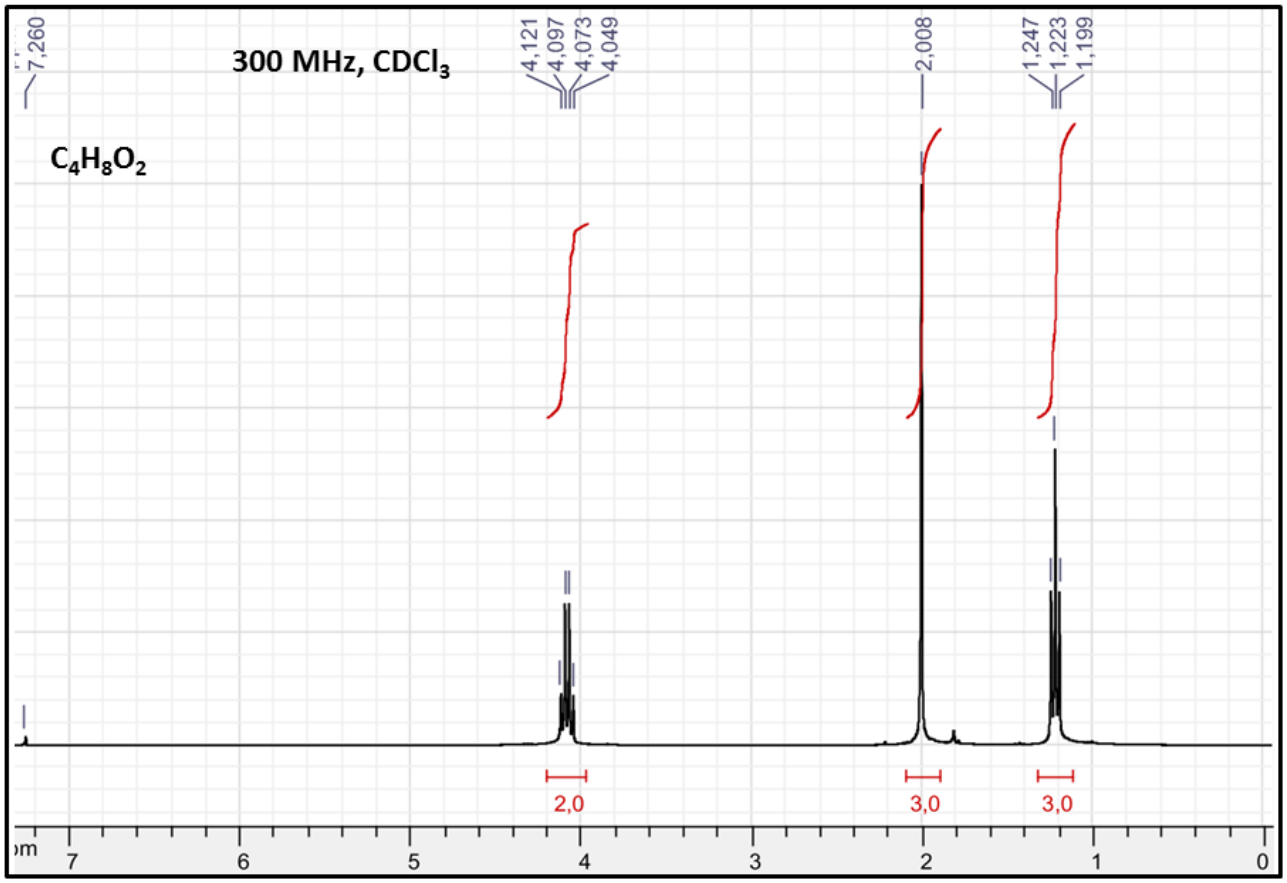
Exercise 3:



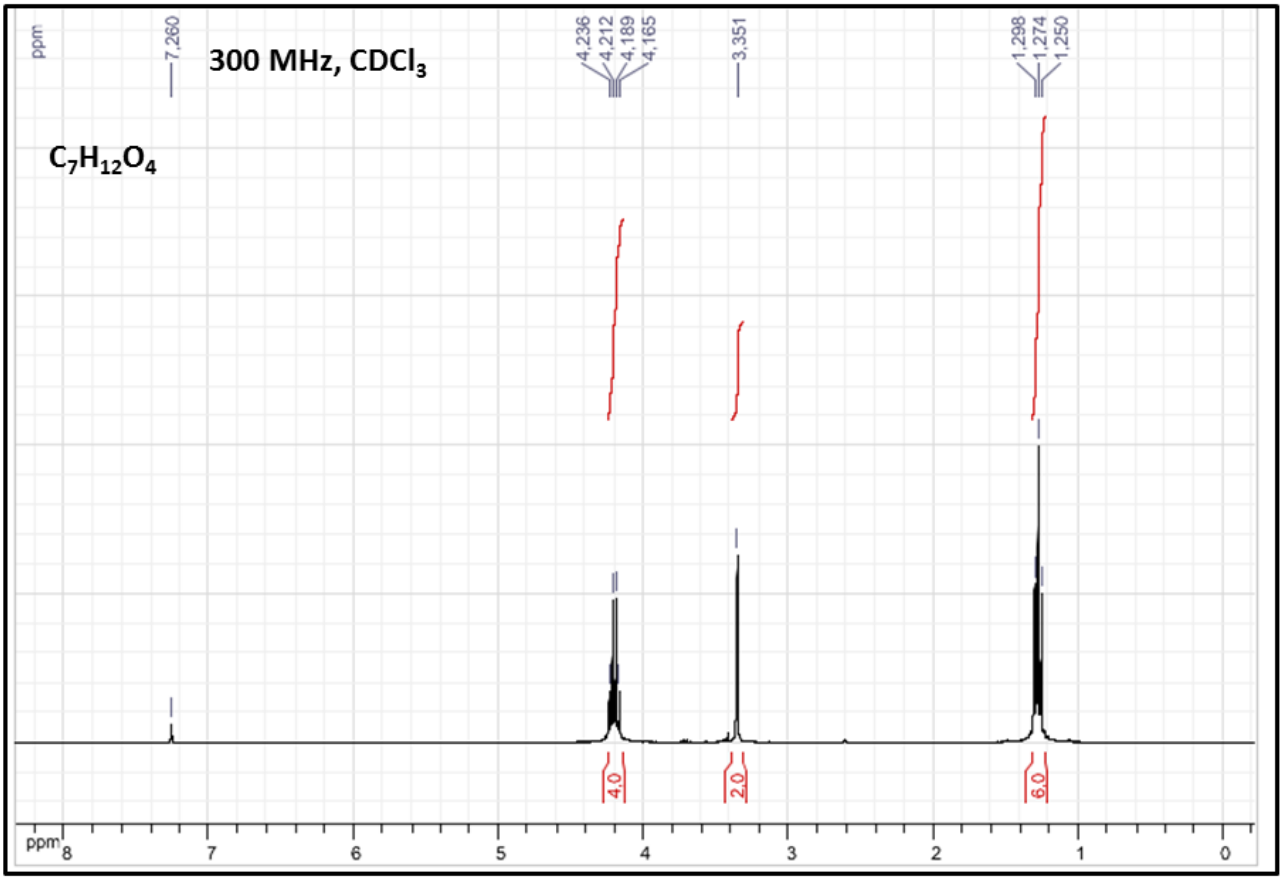
Exercise 4:



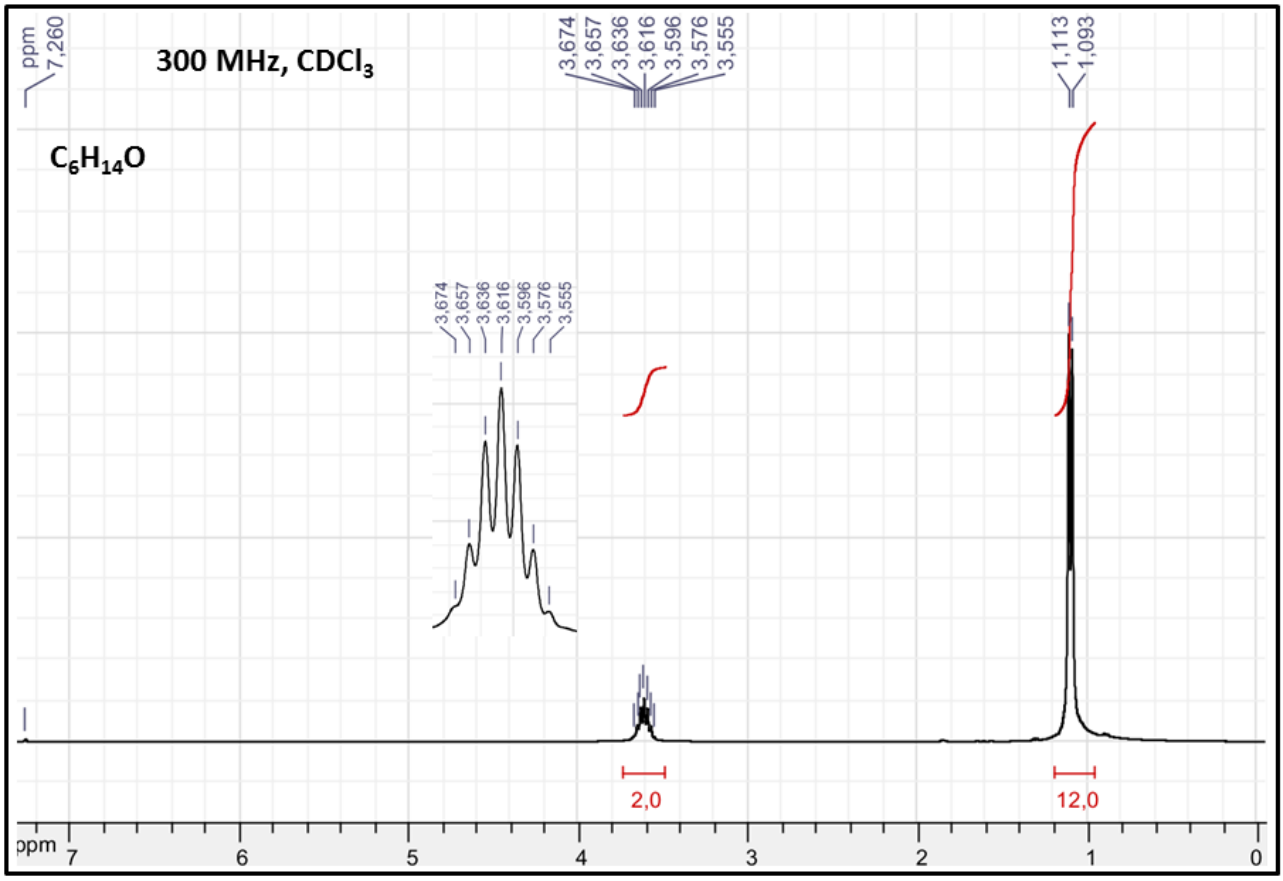
Exercise 5:



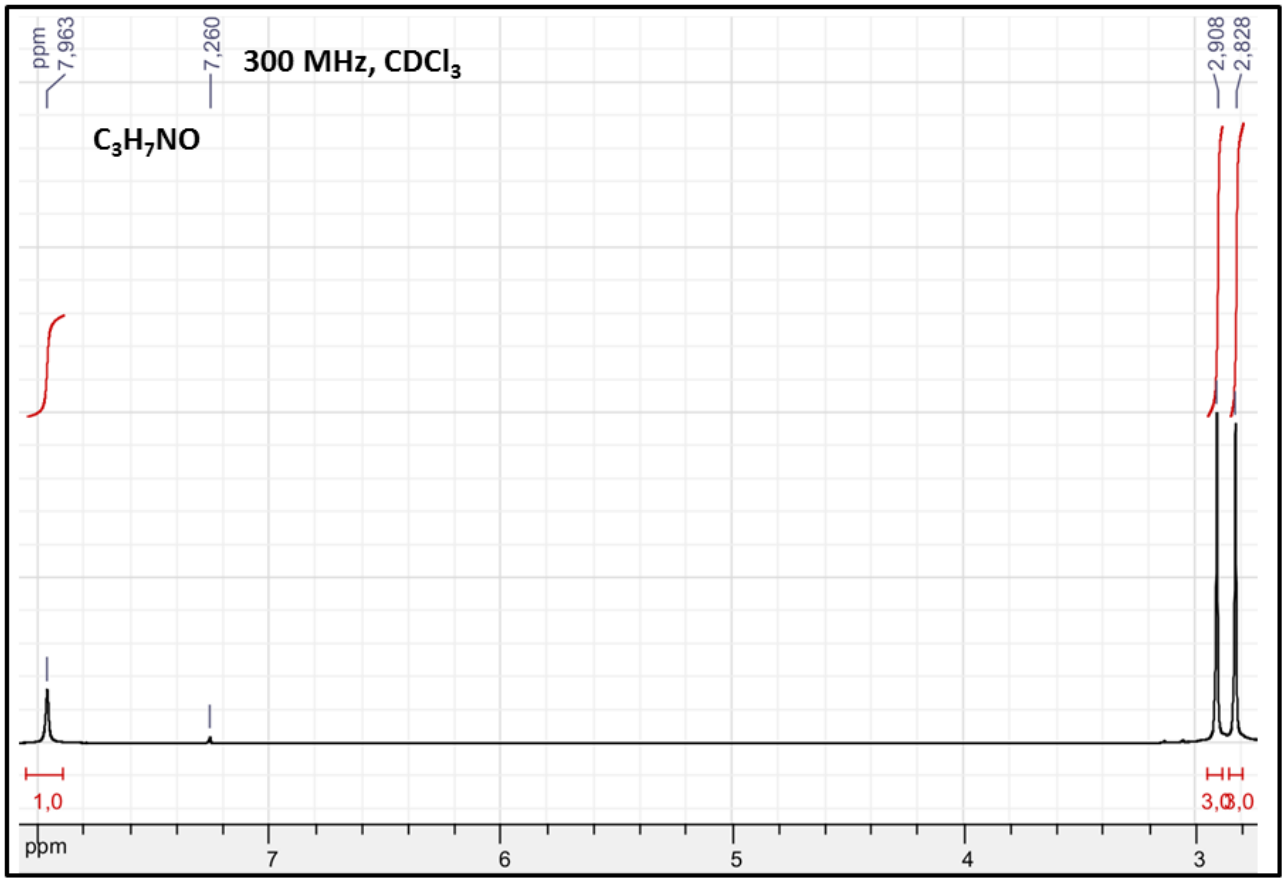
Exercise 6:



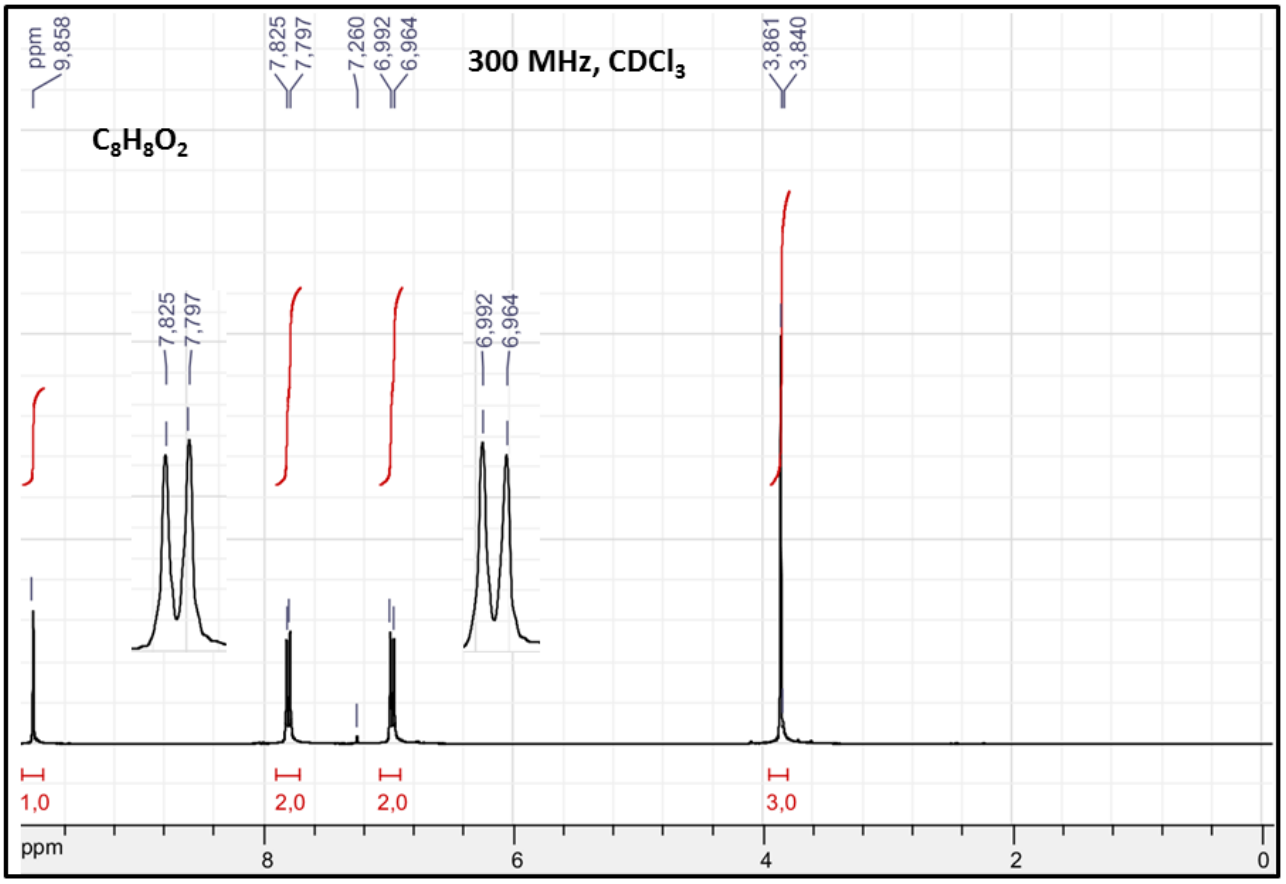
Exercise 7:



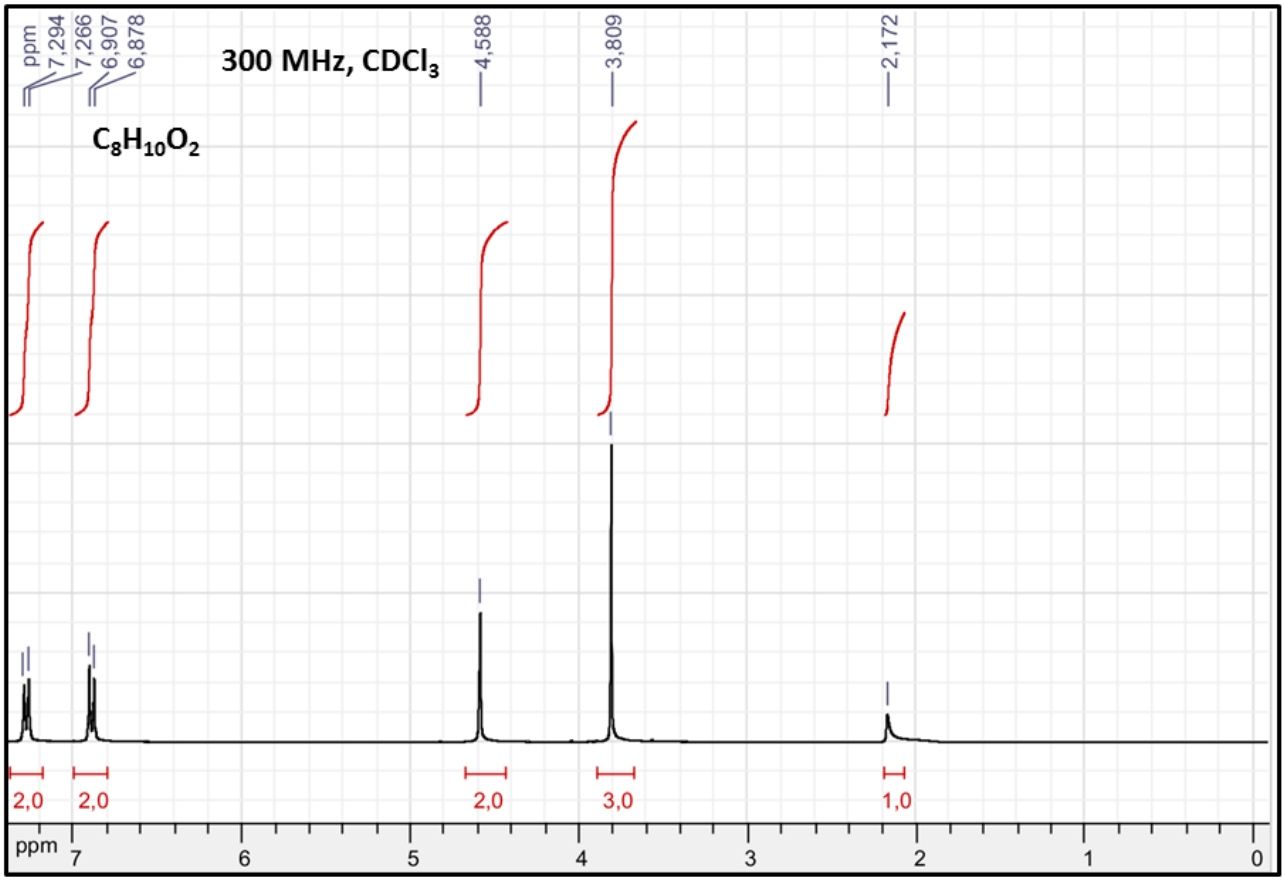
Exercise 8:



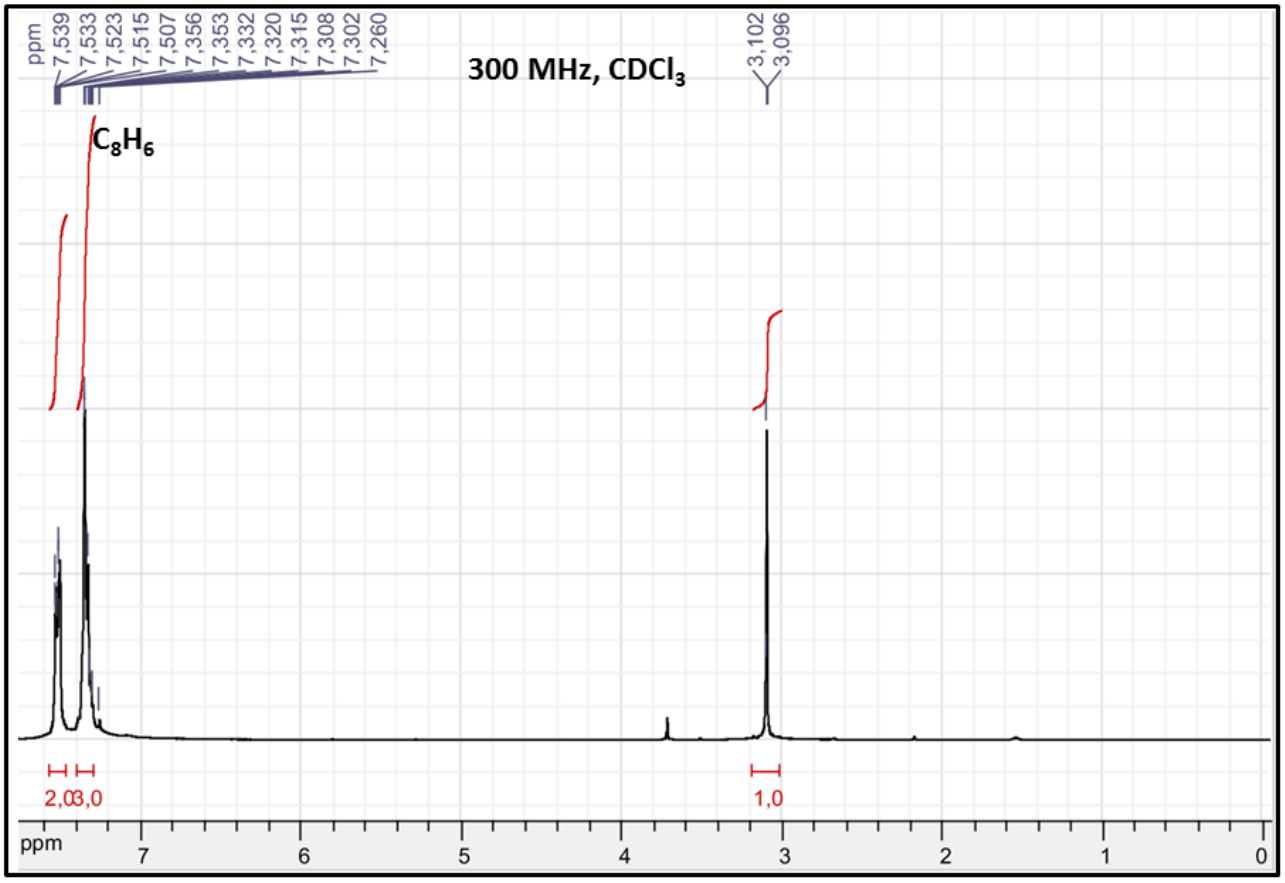
Exercise 9:



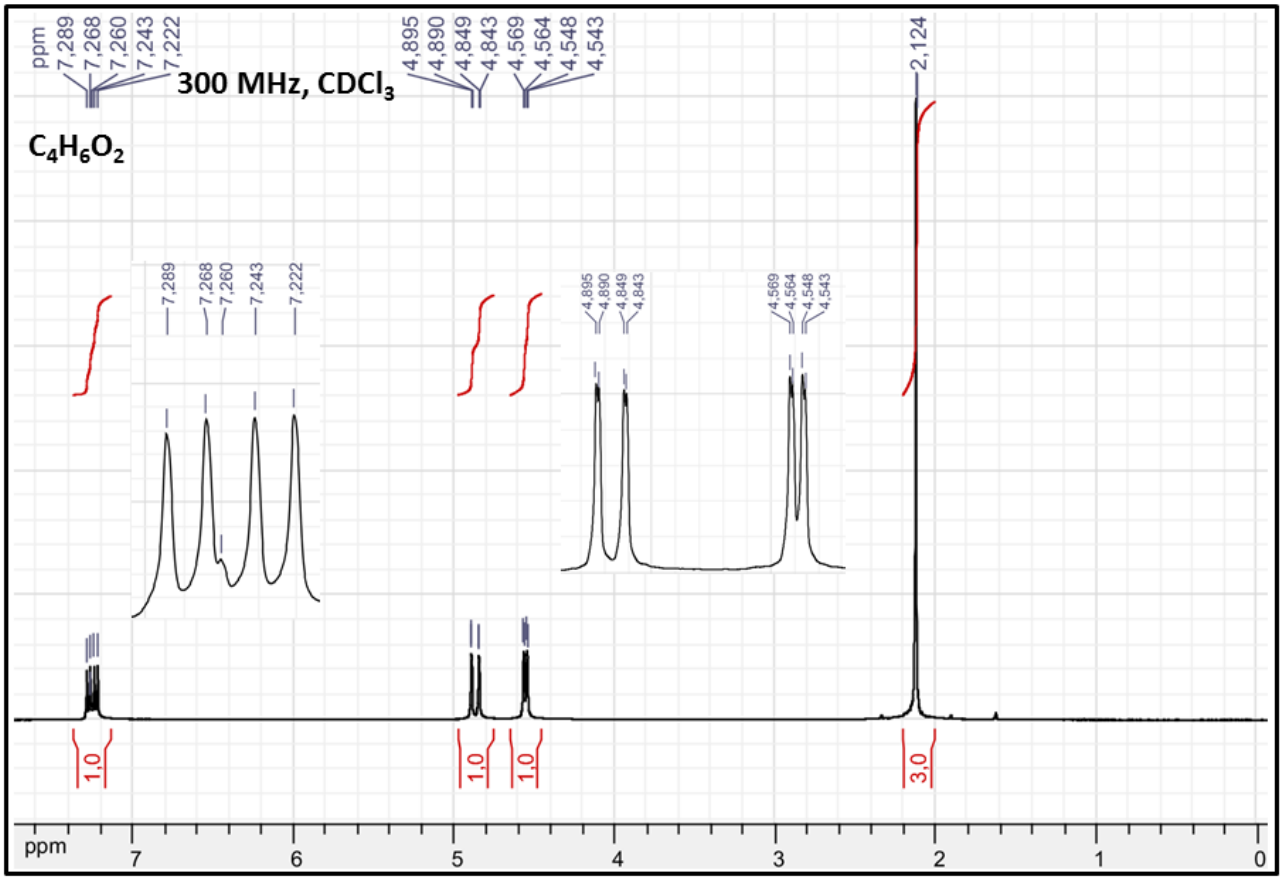
Exercise 10:



Exercise 11:



Exercise 12:

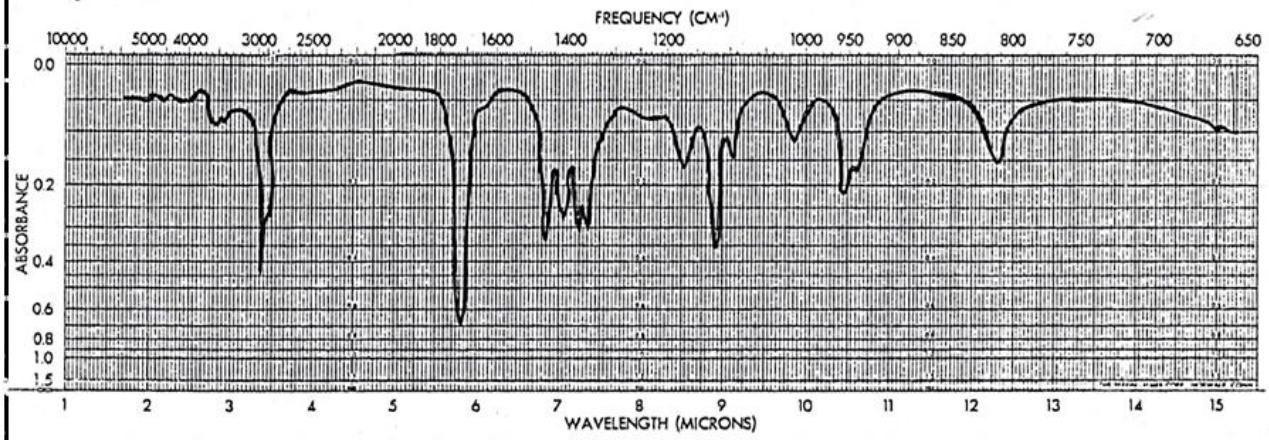


^{1}H NMR/UV/MS/IR

Exercise 1:

Spectre I. R.

Composé n° 6-2



Spectre de masse (% du pic de base)

m/e	%
26	11.
27	39.
29	92.
39	4.
41	3.
42	5.
43	3.
56	5.
57	100.
58	4.
86	18.5
87	1.15
88	0.074

Abondance isotopique

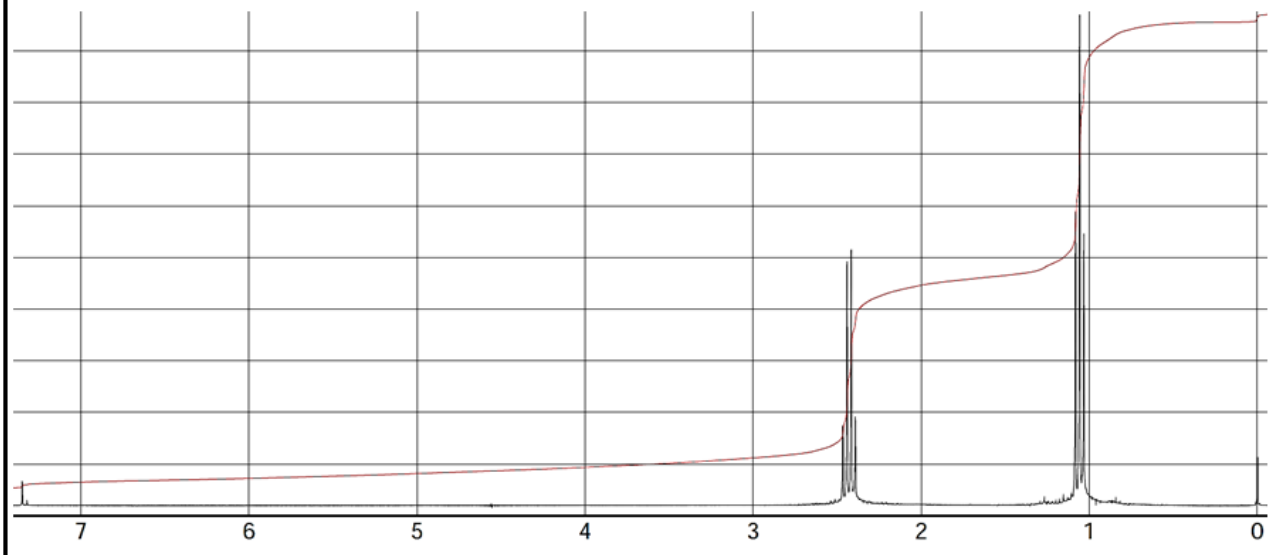
m/e	% de *P*
86 (*P*)	100.
87 (*P* + 1)	6.2
88 (*P* + 2)	0.4

Spectre U. V.

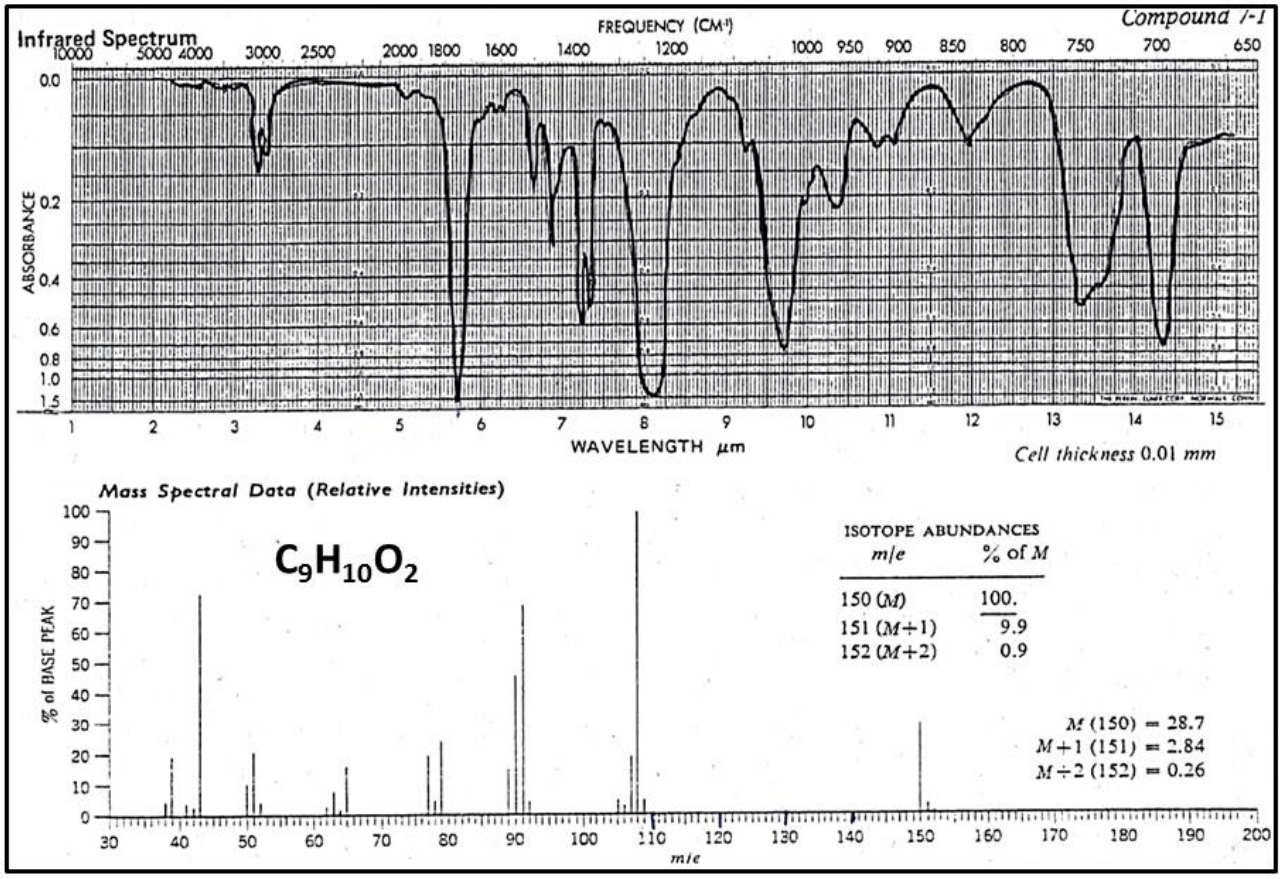
λ^{EtOH}_{max}	log ϵ_{max}
273	1.31

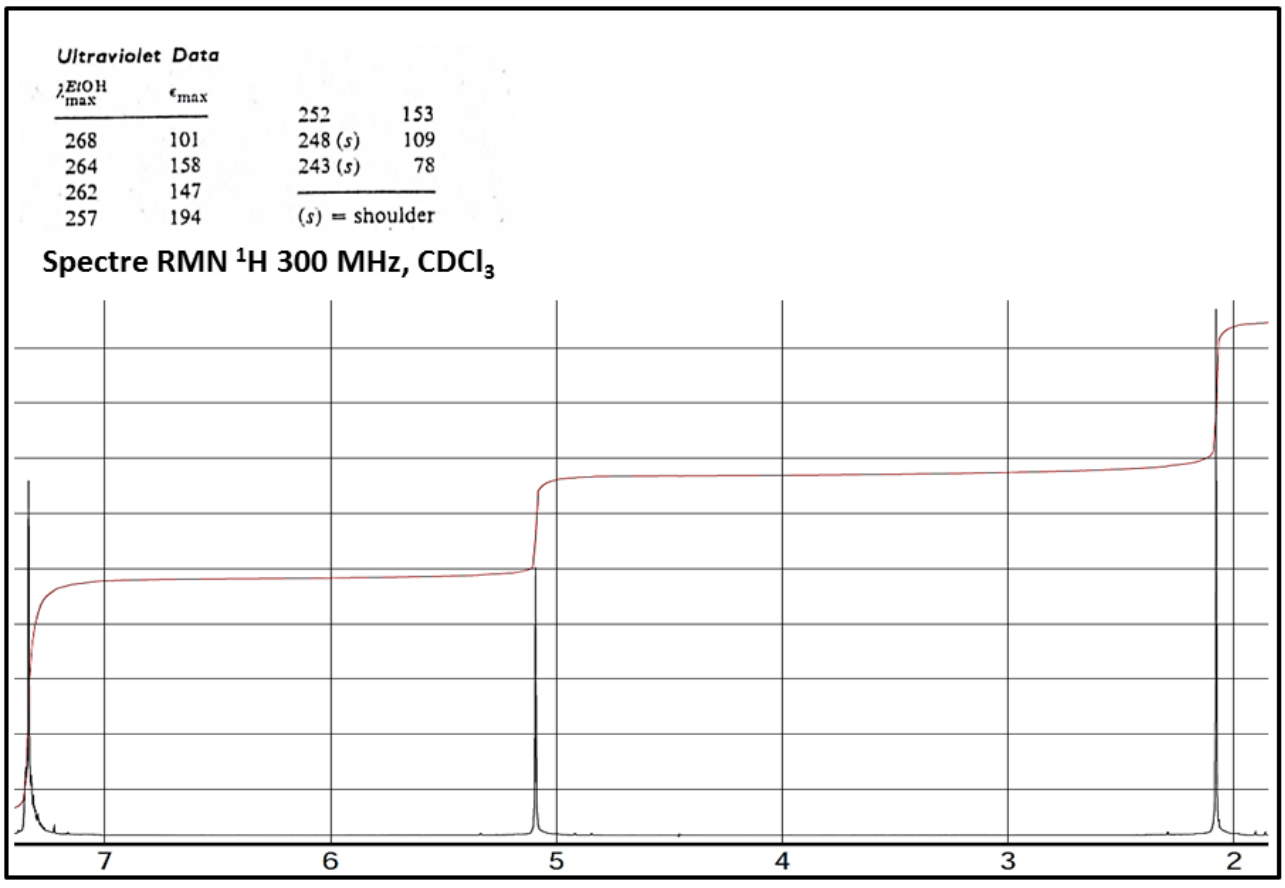
$C_5H_{10}O$

Spectre RMN ^1H 300 MHz, $CDCl_3$

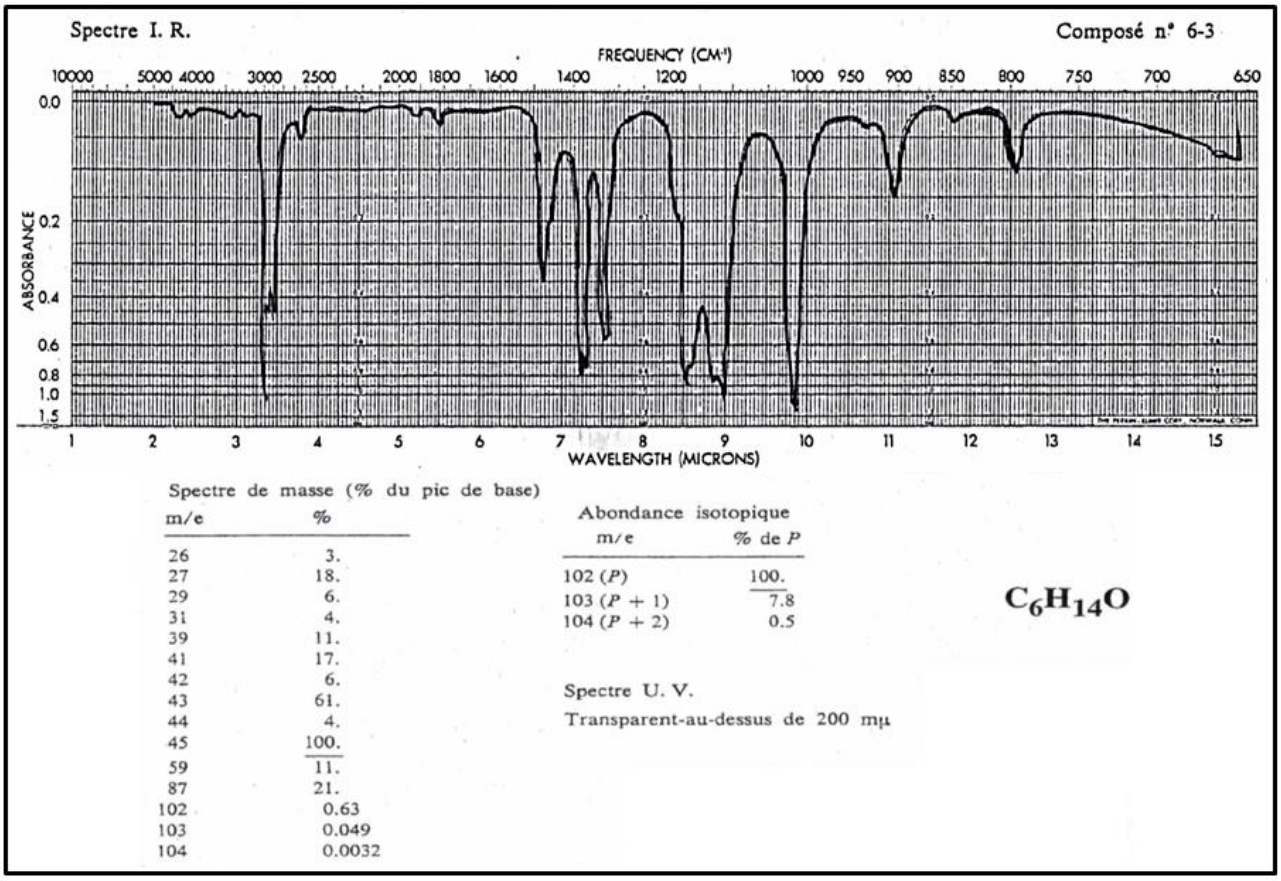


Exercise 2:


Infrared Spectrum
Compound 7-1
FREQUENCY (CM⁻¹)
ABSORBANCE
WAVELENGTH μm
Cell thickness 0.01 mm
Mass Spectral Data (Relative Intensities)
% of BASE PEAK
m/e
C9H10O2
ISOTOPE ABUNDANCES
m/e % of M
150 (M) 100.
151 (M+1) 9.9
152 (M+2) 0.9
M (150) = 28.7
M+1 (151) = 2.84
M+2 (152) = 0.26


Ultraviolet Data
λmax EtOH
εmax
268 101
264 158
262 147
257 194
252 153
248 (s) 109
243 (s) 78
(s) = shoulder
Spectre RMN ¹H 300 MHz, CDCl3

Exercise 3:



Spectre de masse (% du pic de base)

m/e	%
26	3.
27	18.
29	6.
31	4.
39	11.
41	17.
42	6.
43	61.
44	4.
45	100.
59	11.
87	21.
102	0.63
103	0.049
104	0.0032

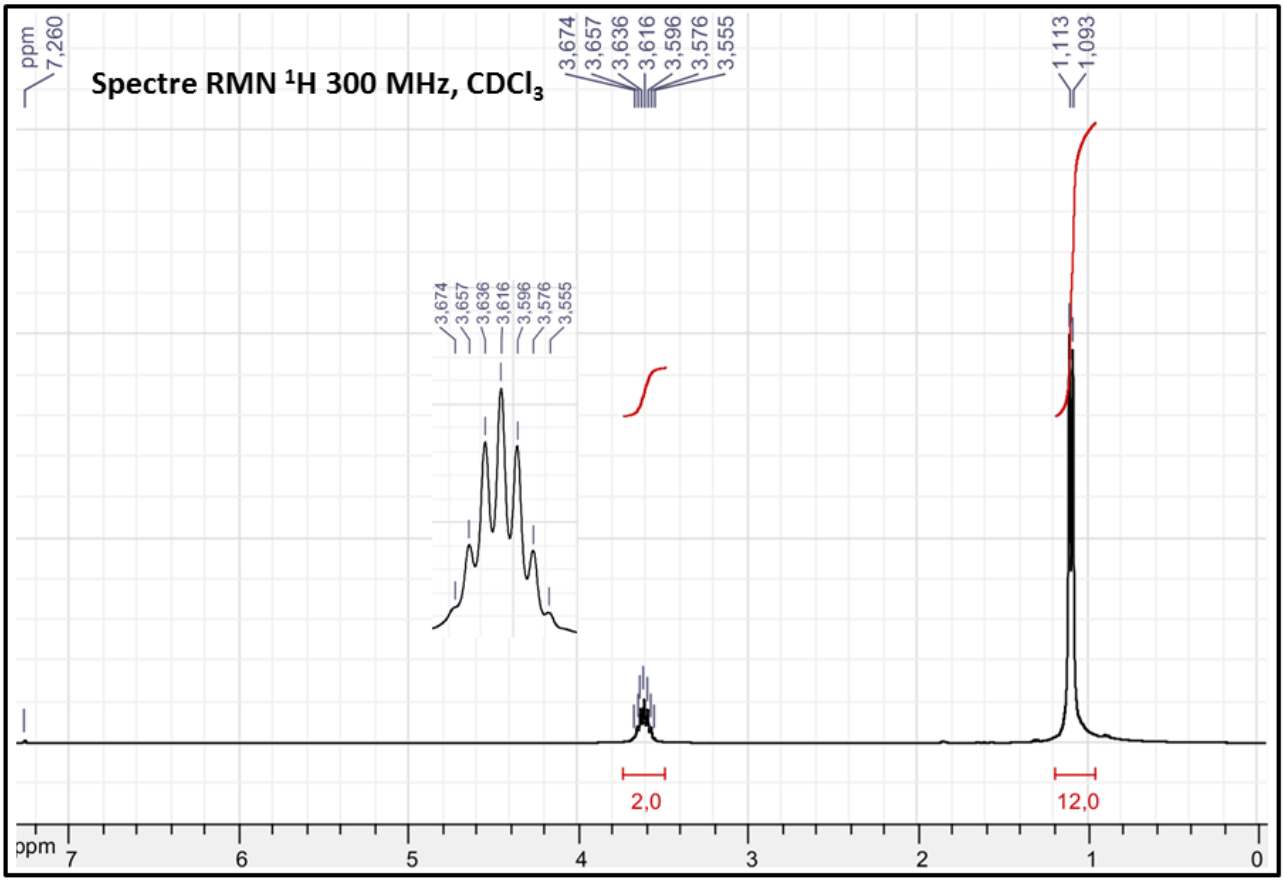
Abondance isotopique

m/e	% de *P*
102 (*P*)	100.
103 (*P* + 1)	7.8
104 (*P* + 2)	0.5

$C_6H_{14}O$

Spectre U. V.

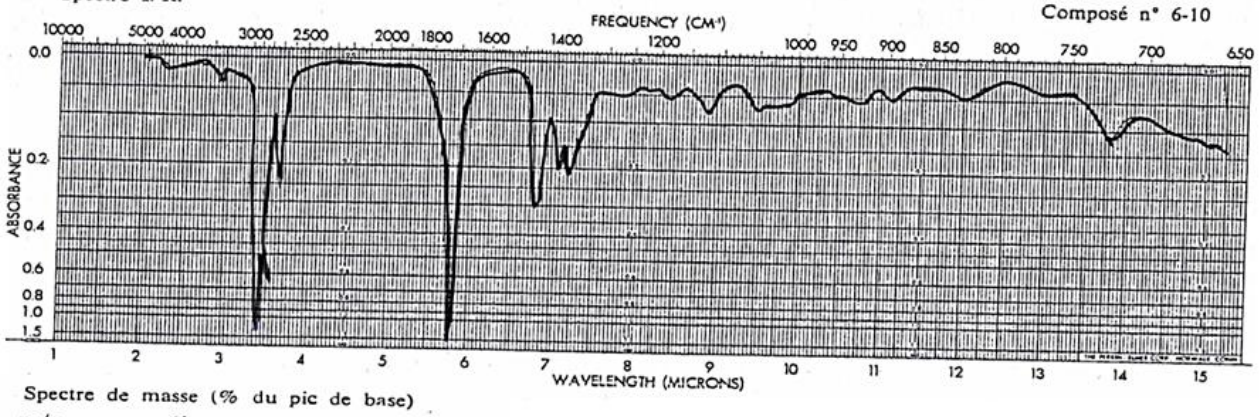
Transparent-au-dessus de 200 mμ



Exercise 4:

Spectre I. R.

Composé n° 6-10



Spectre de masse (% du pic de base)

m/e	%	m/e	%
26	10.	55	55.
27	86.	56	7.
29	97.	57	50.
31	4.	58	5.
38	4.	67	7.
39	45.	68	14.
41	91.	69	5.
42	57.	70	73.
43	89.	71	23.
44	100.	72	7.
45	22.	81	14.
53	6.	85	3.
54	7.	86	12.
		96	6.
		113	0.2
		114	1.2
		115	0.13
		116	0.012

Abondance isotopique

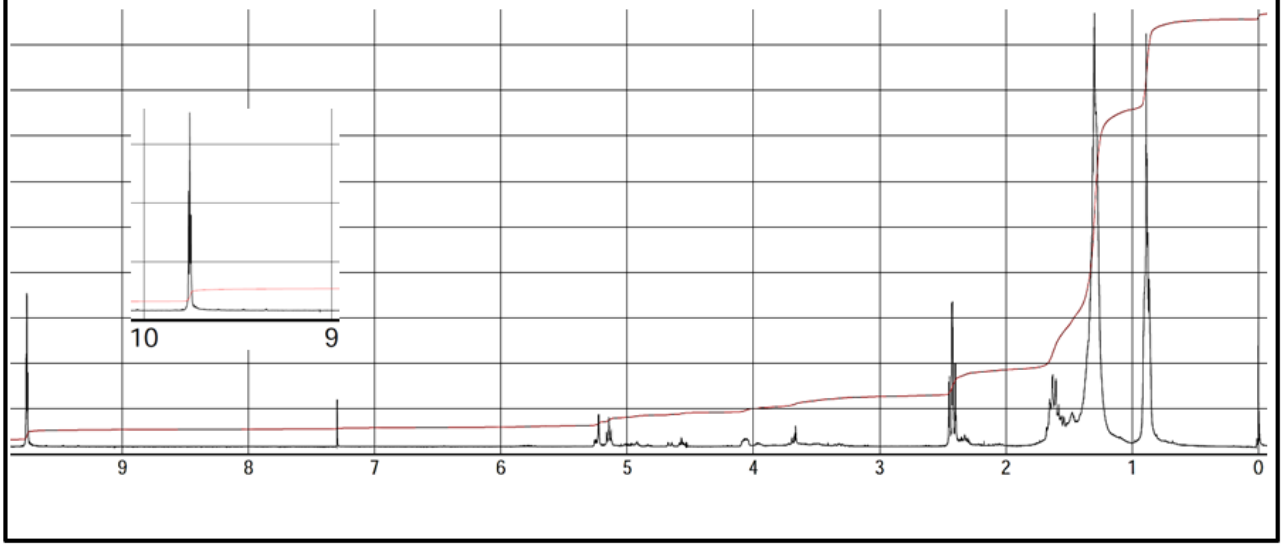
m/e	% de P
114 (P)	100.
115 (P + 1)	11.0
116 (P + 2)	0.97

$C_7H_{14}O$

Spectre U. V.

$\lambda^{Cyclohexane}_{max}$	ϵ_{max}
292	23.2

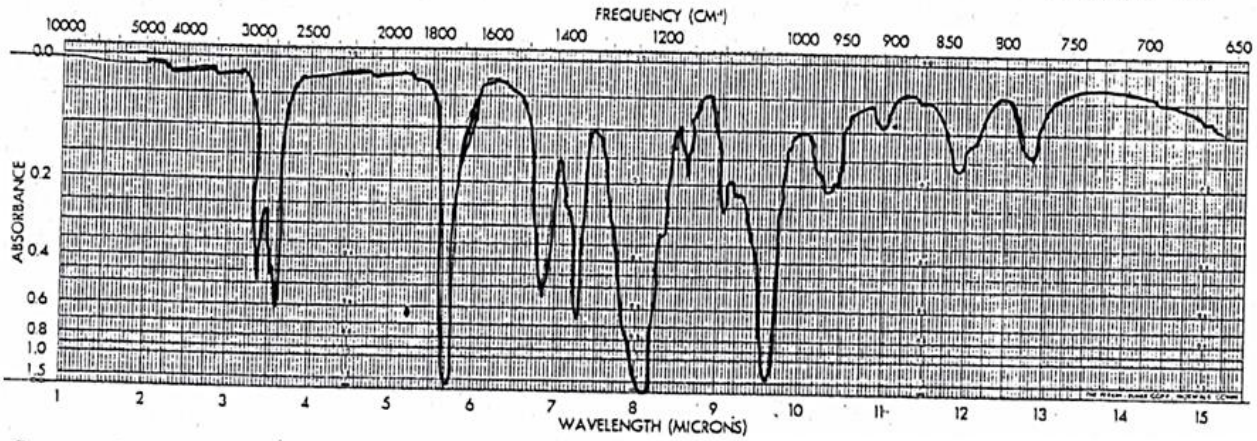
Spectre RMN ^1H 300 MHz, $CDCl_3$



Exercise 5:

Spectre I. R.

Composé n° 6-16



Spectre de masse (% du pic de base)

m/e	%	m/e	%
26	3.	55	3.
27	6.	56	7.
28	9.	57	3.
30	6.	58	100.
41	4.	59	4.
42	23.	71	11.
43	17.	131 (*P*)	0.346
44	7.		

Abondance isotopique

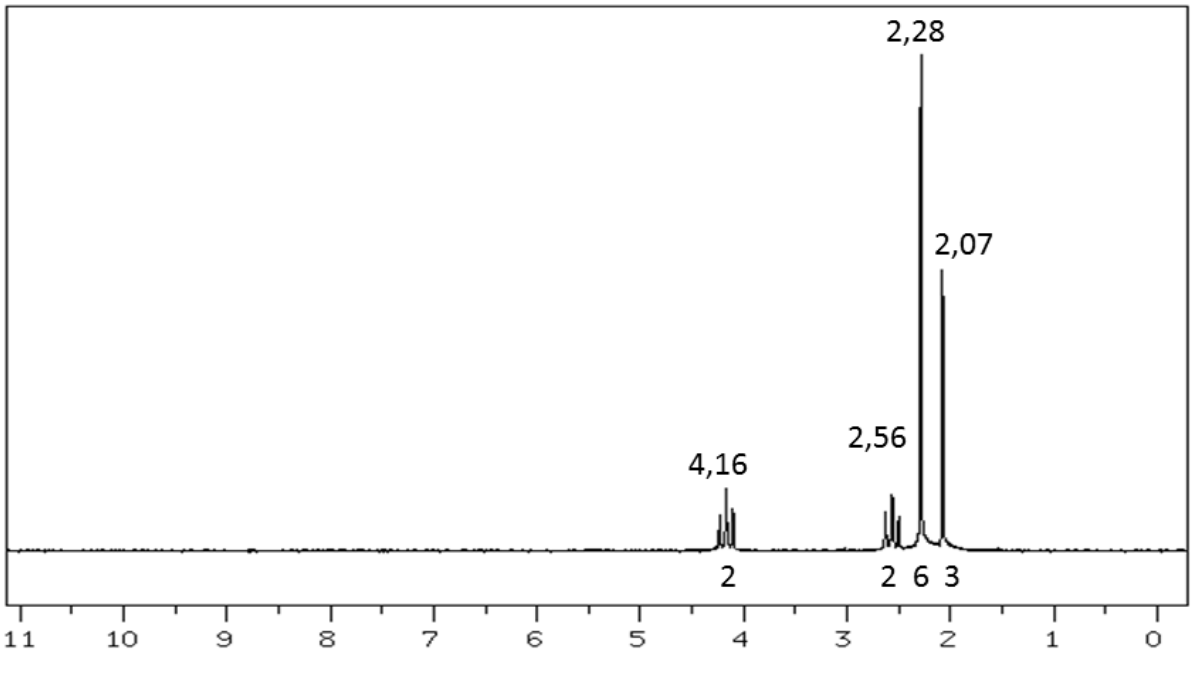
P + 1)
P + 2)

$C_6H_{13}NO_2$

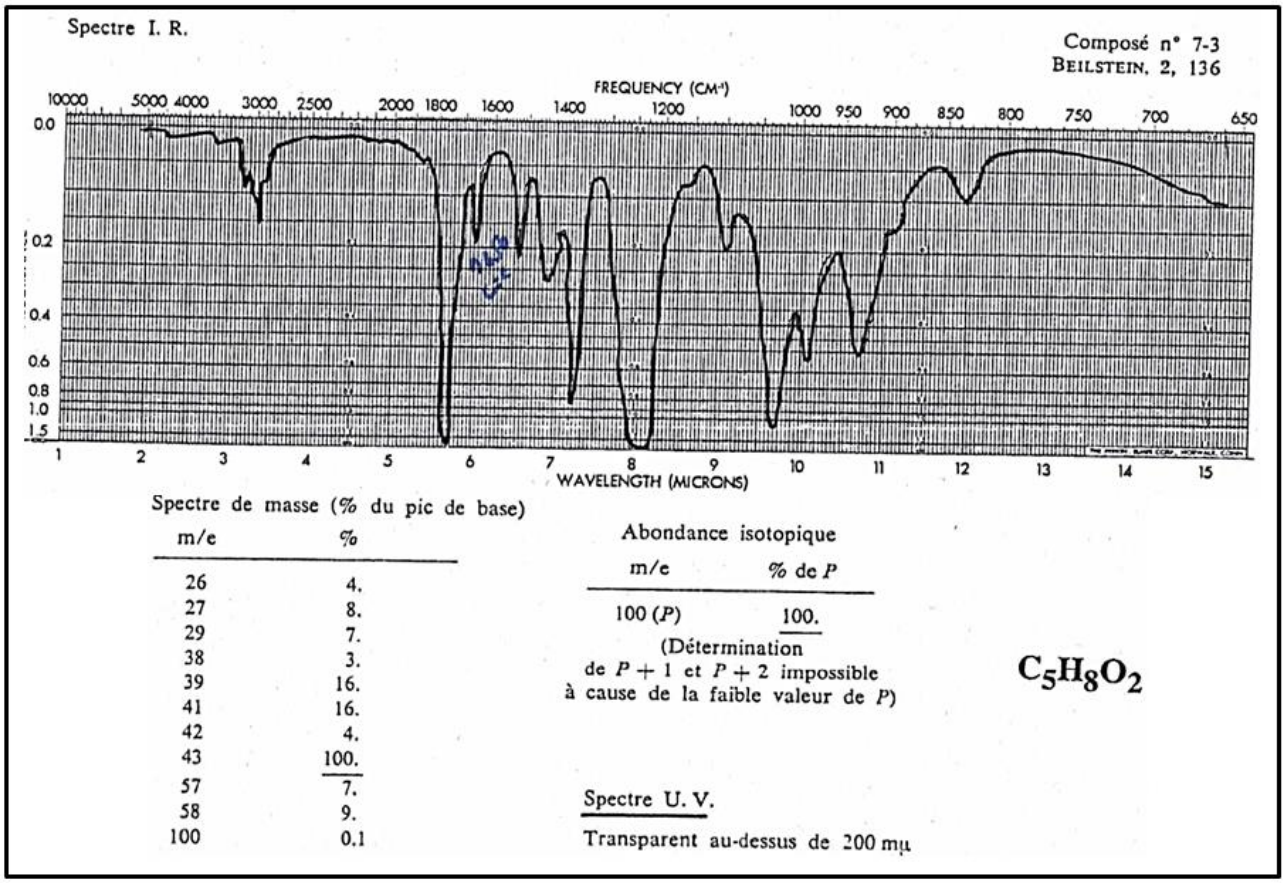
Spectre U. V.

Transparent au-dessus de 210 mμ

Spectre RMN ^{1}H 90 MHz, $CDCl_3$



Exercise 6:



Spectre de masse (% du pic de base)

m/e	%
26	4.
27	8.
29	7.
38	3.
39	16.
41	16.
42	4.
43	100.
57	7.
58	9.
100	0.1

Abondance isotopique

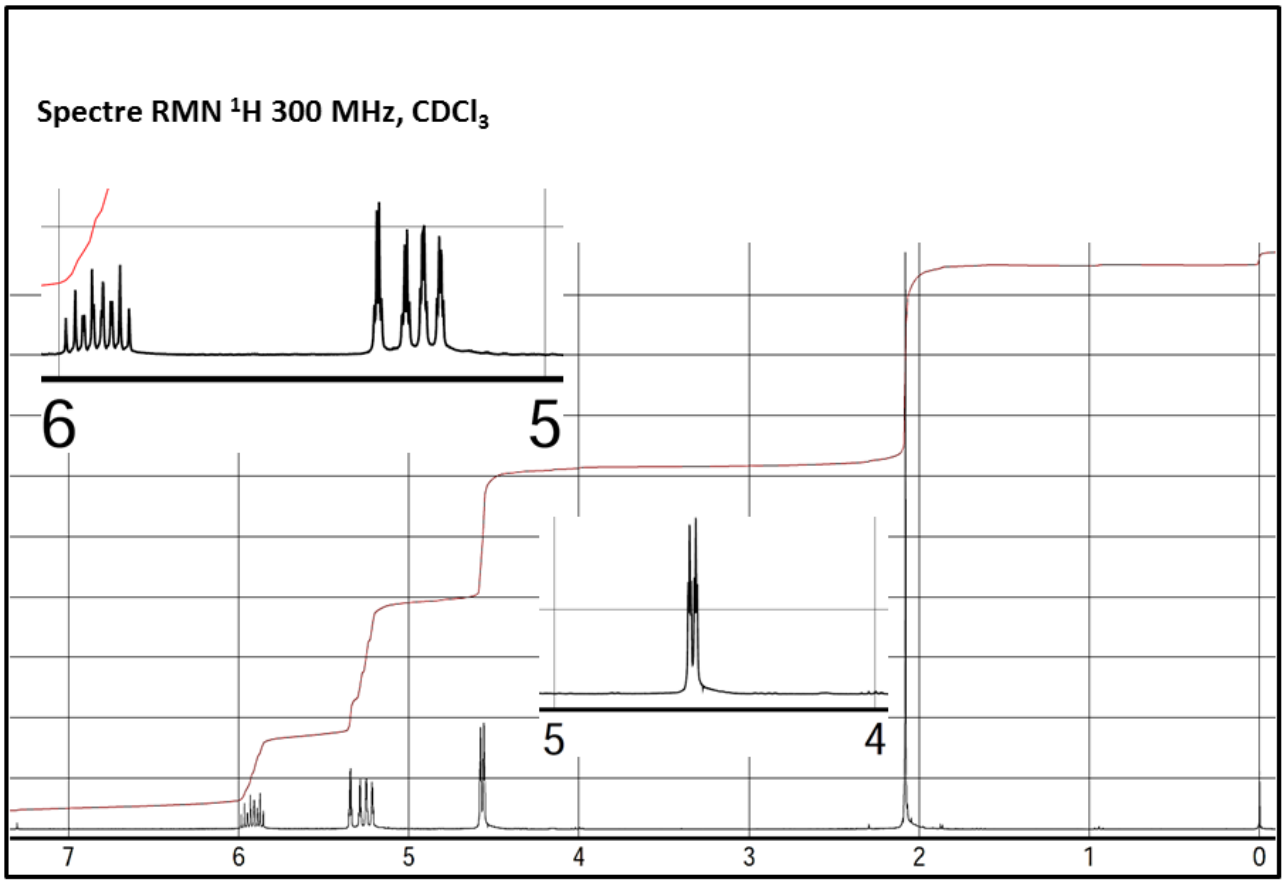
m/e	% de *P*
100 (*P*)	100.

(Détermination de $P+1$ et $P+2$ impossible à cause de la faible valeur de *P*)

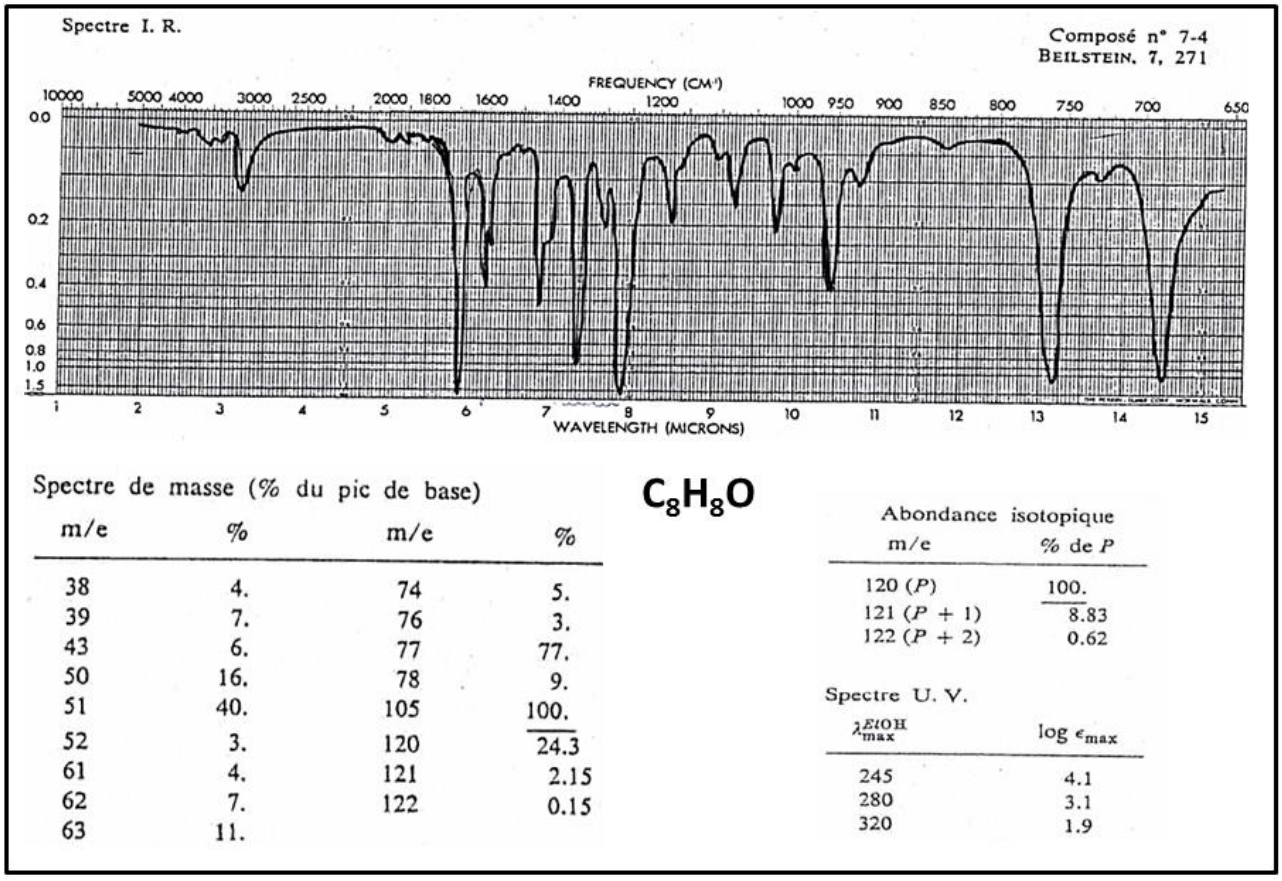
$C_5H_8O_2$

Spectre U. V.

Transparent au-dessus de 200 mμ



Exercise 7:



Spectre de masse (% du pic de base)

m/e	%	m/e	%
38	4.	74	5.
39	7.	76	3.
43	6.	77	77.
50	16.	78	9.
51	40.	105	100.
52	3.	120	24.3
61	4.	121	2.15
62	7.	122	0.15
63	11.		

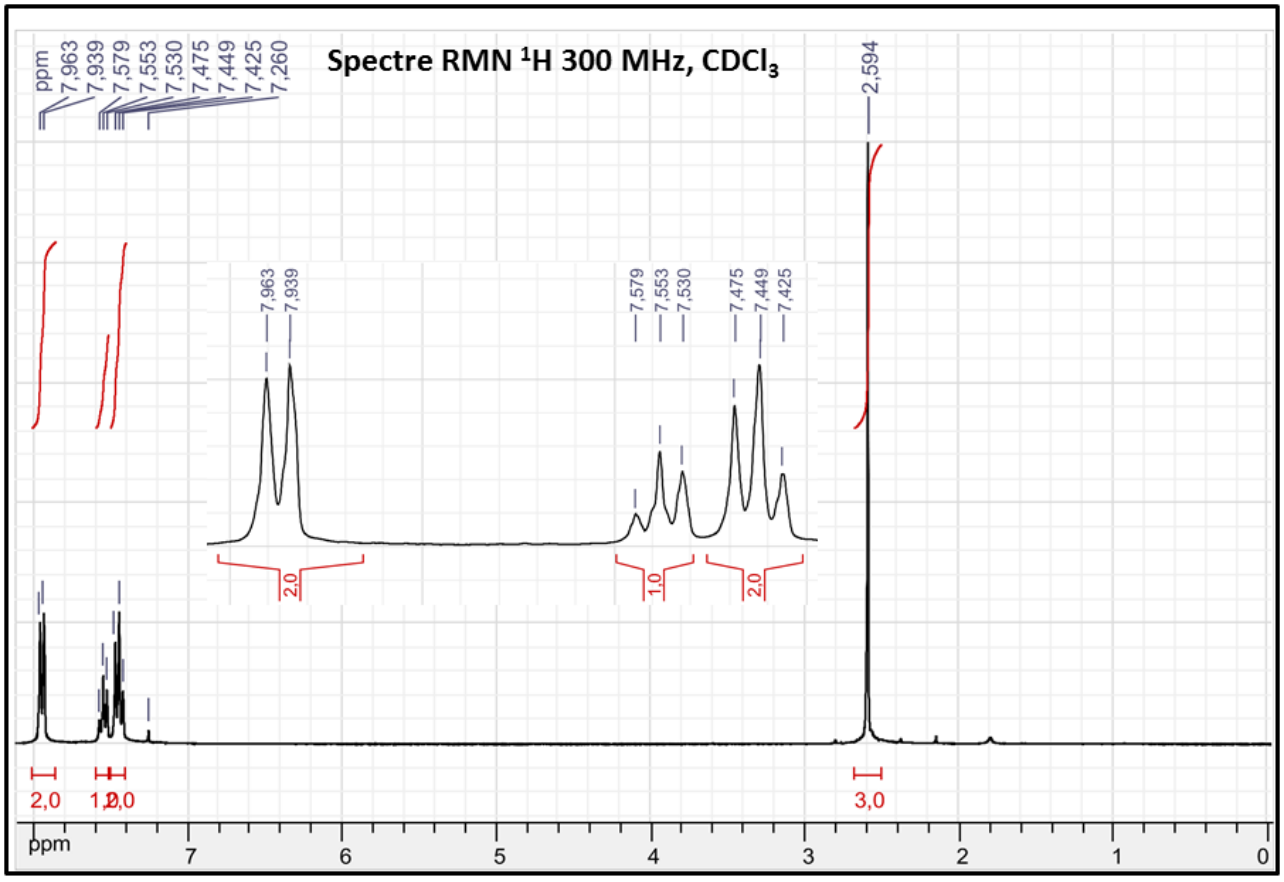
C_8H_8O

Abondance isotopique

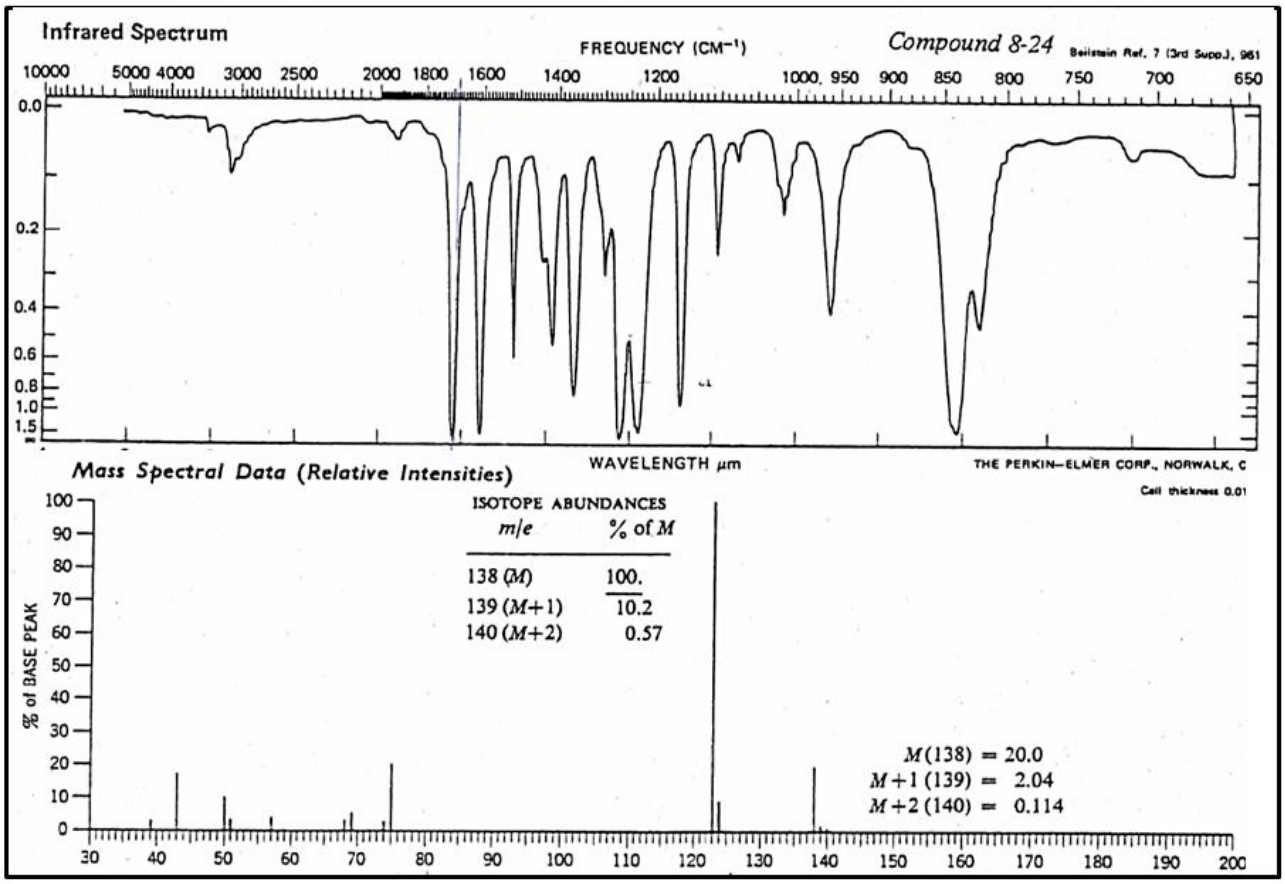
m/e	% de P
120 (P)	100.
121 (P + 1)	8.83
122 (P + 2)	0.62

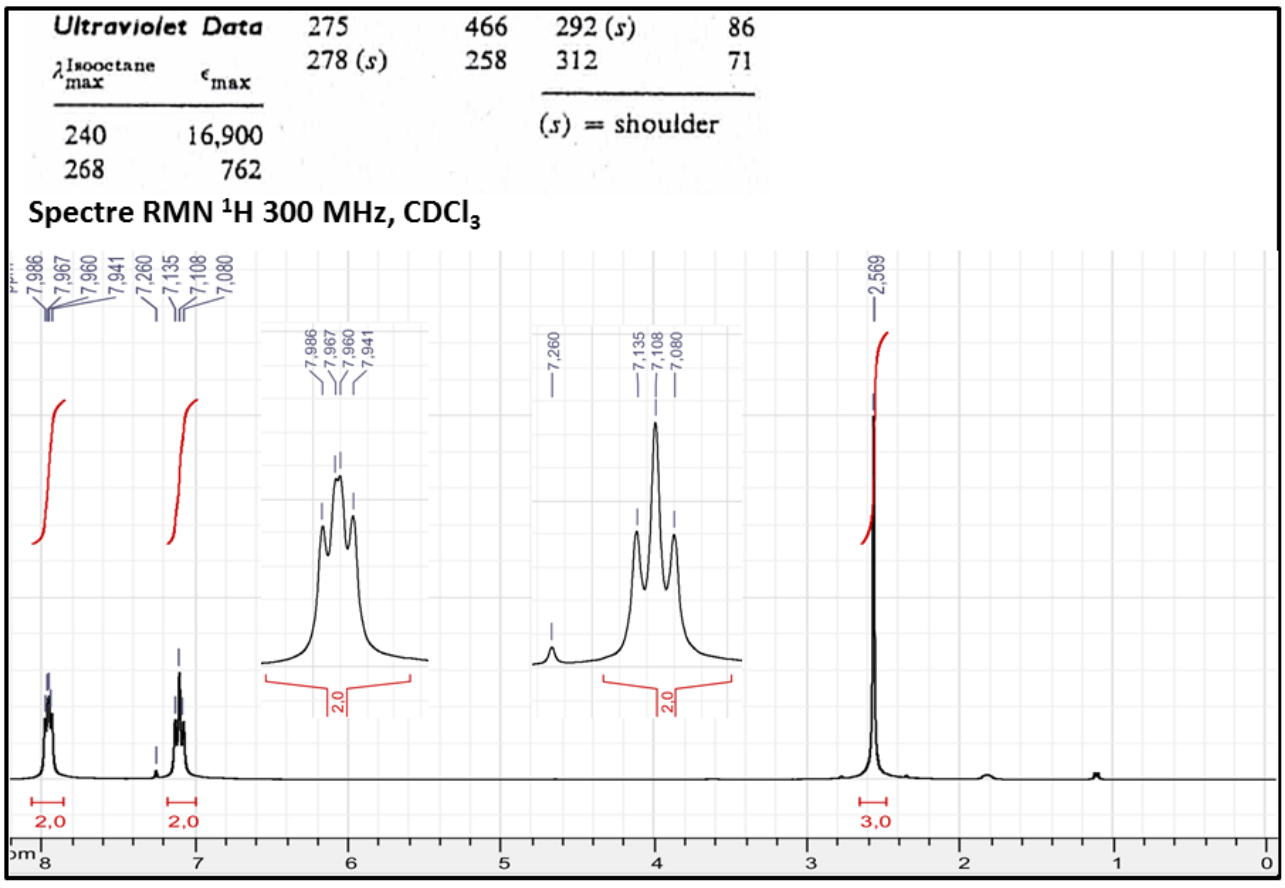
Spectre U. V.

λ_{max}^{EtOH}	log ϵ_{max}
245	4.1
280	3.1
320	1.9

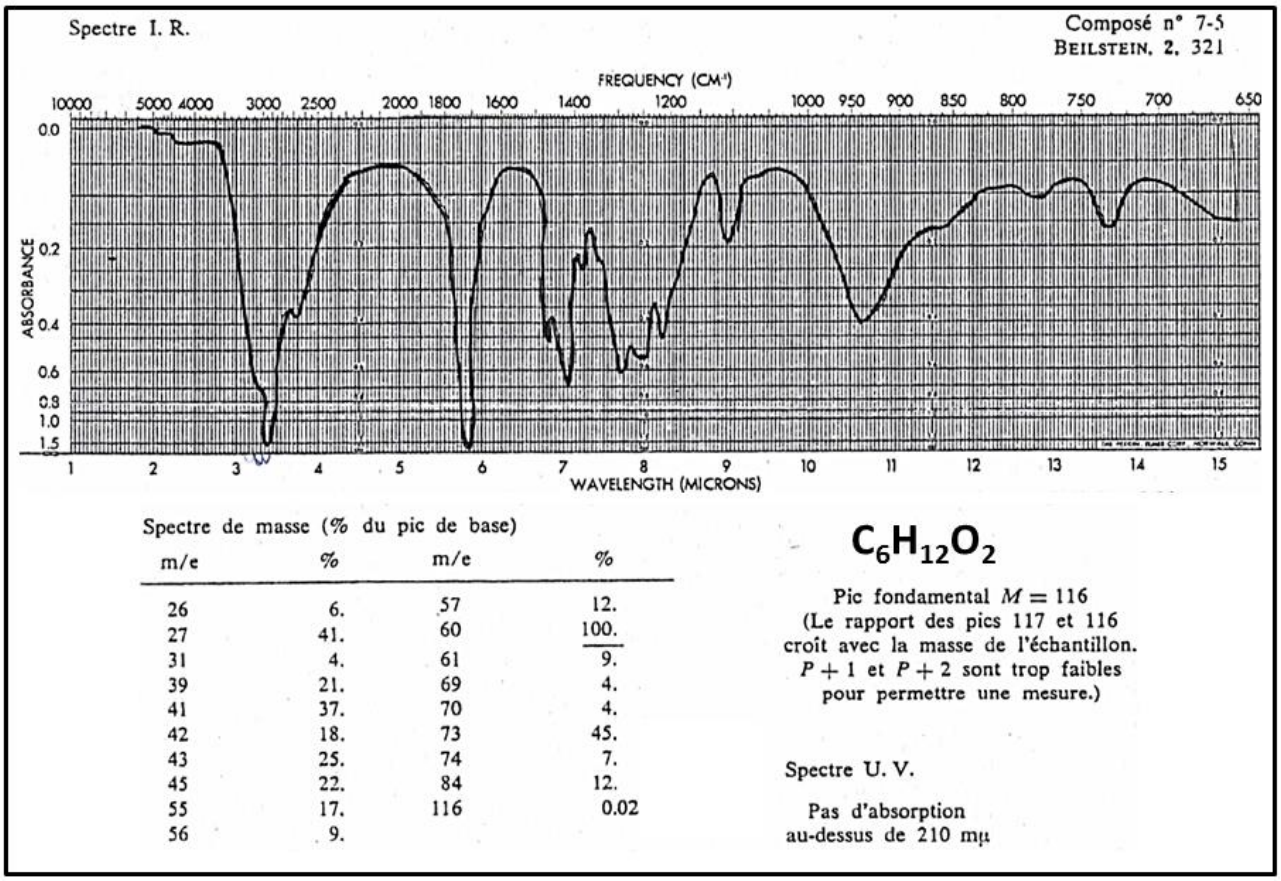


Exercise 8:


Infrared Spectrum
FREQUENCY (CM⁻¹)
Compound 8-24
WAVELENGTH μm
THE PERKIN–ELMER CORP., NORWALK, C
Mass Spectral Data (Relative Intensities)
% of BASE PEAK
ISOTOPE ABUNDANCES
m/e % of M
138 (M) 100.
139 (M+1) 10.2
140 (M+2) 0.57
M(138) = 20.0
M+1 (139) = 2.04
M+2 (140) = 0.114


Ultraviolet Data
λ Isooctane max
ε max
240 16,900
268 762
275 466
278 (s) 258
292 (s) 86
312 71
(s) = shoulder
Spectre RMN ¹H 300 MHz, CDCl₃
7,986
7,967
7,960
7,941
7,260
7,135
7,108
7,080
2,569
2,0
2,0
2,0
2,0
3,0
ppm

Exercise 9:



Spectre de masse (% du pic de base)

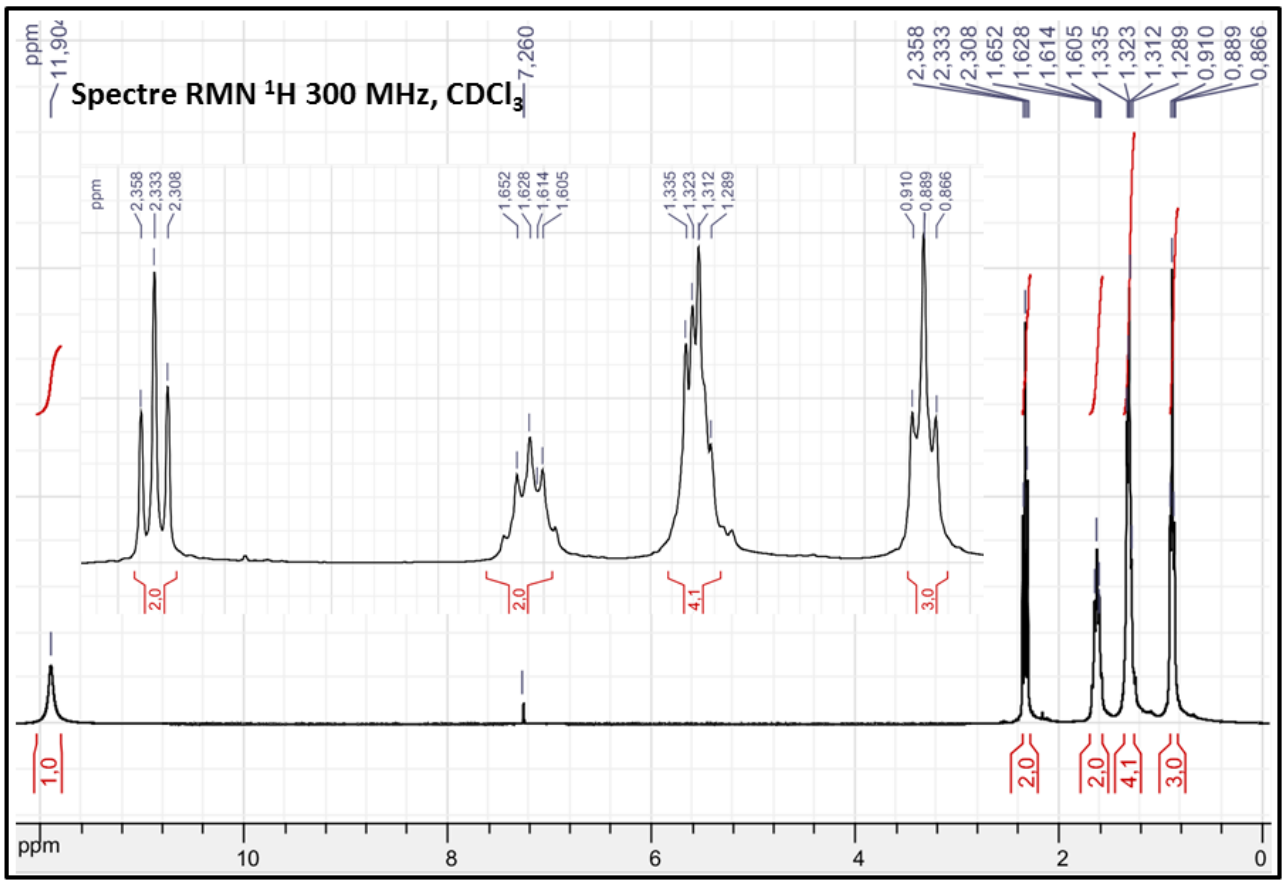
m/e	%	m/e	%
26	6.	57	12.
27	41.	60	100.
31	4.	61	9.
39	21.	69	4.
41	37.	70	4.
42	18.	73	45.
43	25.	74	7.
45	22.	84	12.
55	17.	116	0.02
56	9.		

$C_6H_{12}O_2$

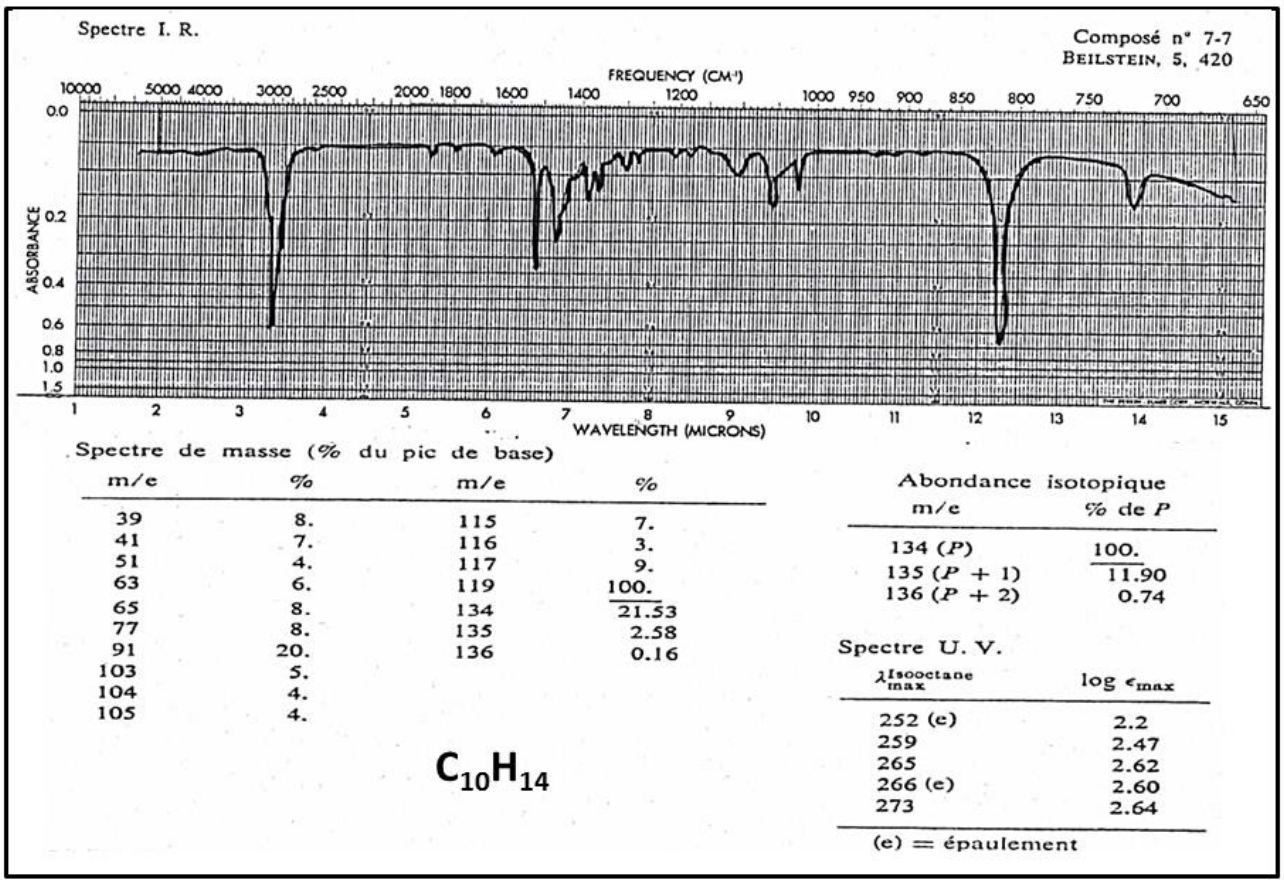
Pic fondamental $M = 116$
(Le rapport des pics 117 et 116 croît avec la masse de l'échantillon. $P + 1$ et $P + 2$ sont trop faibles pour permettre une mesure.)

Spectre U. V.

Pas d'absorption au-dessus de 210 mμ



Exercise 10:



Spectre de masse (% du pic de base)

m/e	%	m/e	%
39	8.	115	7.
41	7.	116	3.
51	4.	117	9.
63	6.	119	100.
65	8.	134	21.53
77	8.	135	2.58
91	20.	136	0.16
103	5.		
104	4.		
105	4.		

Abondance isotopique

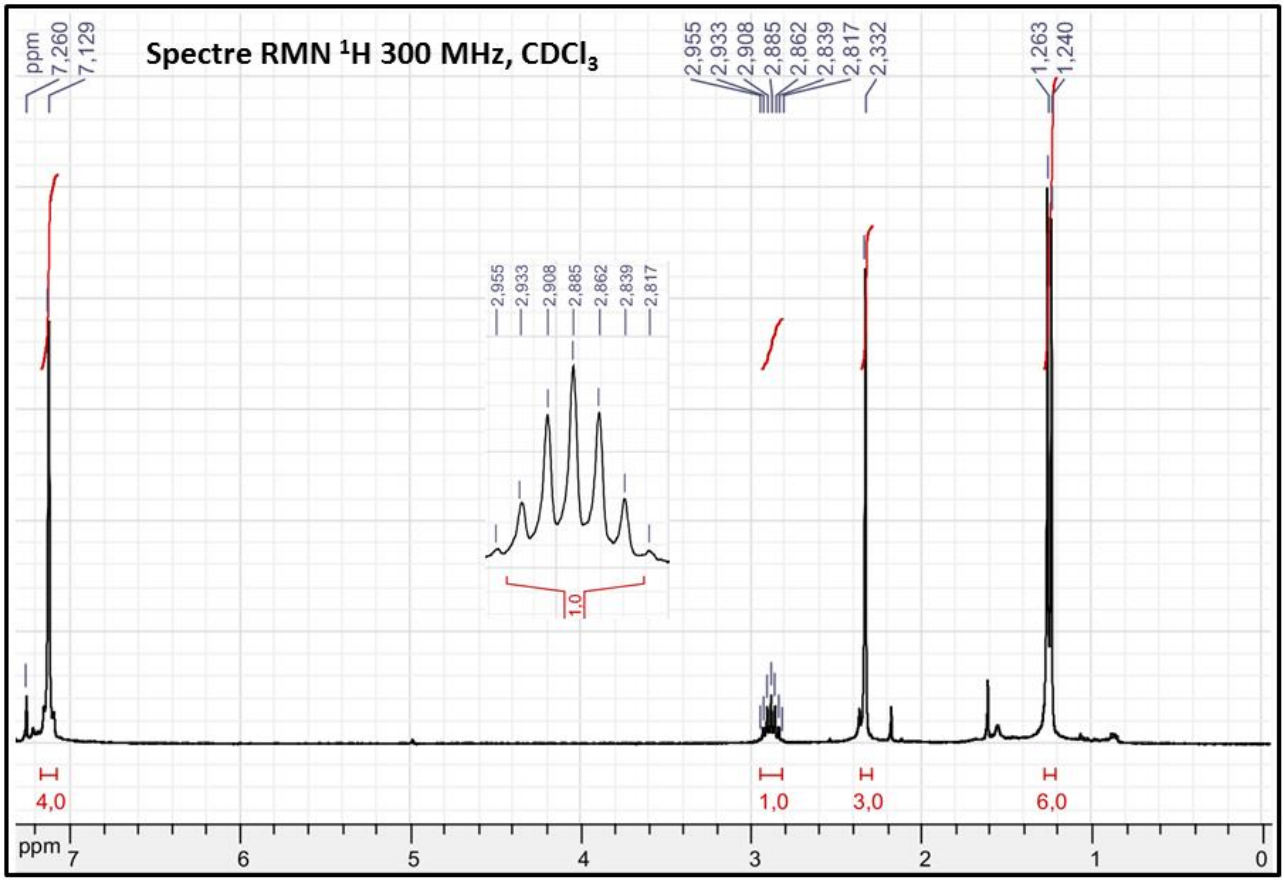
m/e	% de P
134 (P)	100.
135 (P + 1)	11.90
136 (P + 2)	0.74

Spectre U. V.

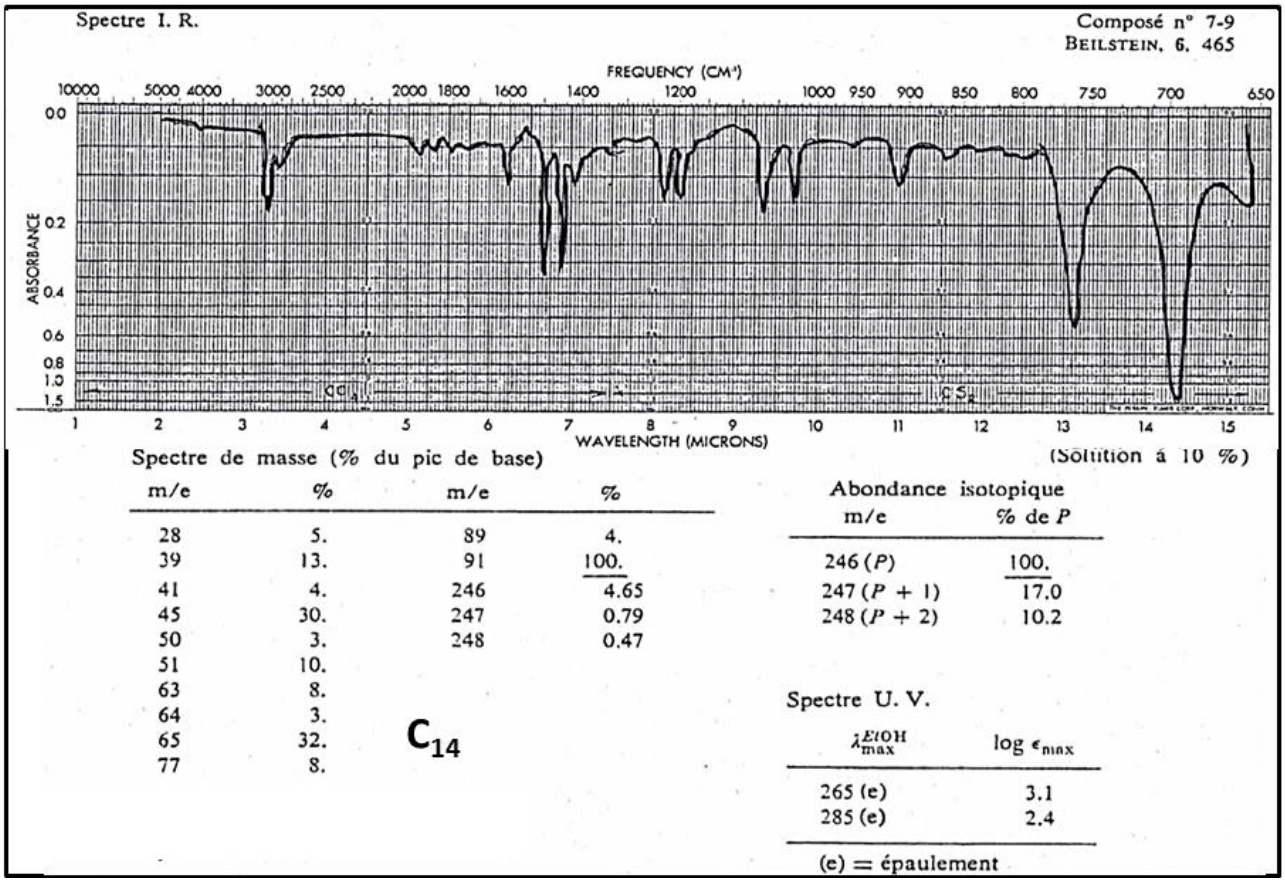
$\lambda_{max}^{Isooctane}$	log ϵ_{max}
252 (e)	2.2
259	2.47
265	2.62
266 (e)	2.60
273	2.64

(e) = épaulement

$C_{10}H_{14}$



Exercise 11:



Spectre de masse (% du pic de base)

m/e	%	m/e	%
28	5.	89	4.
39	13.	91	100.
41	4.	246	4.65
45	30.	247	0.79
50	3.	248	0.47
51	10.		
63	8.		
64	3.		
65	32.		
77	8.		

C_{14}

(Solution à 10 %)

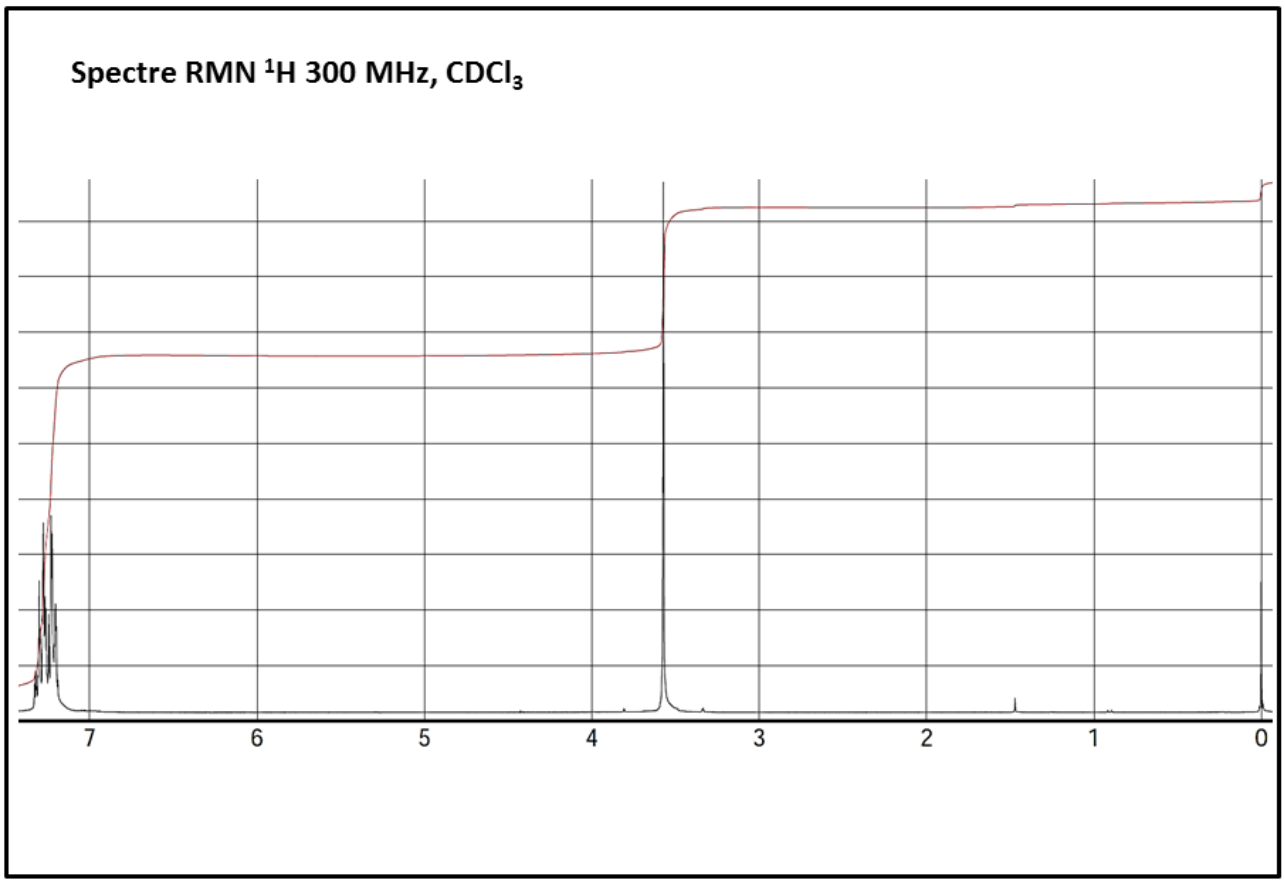
Abondance isotopique

m/e	% de *P*
246 (*P*)	100.
247 (*P* + 1)	17.0
248 (*P* + 2)	10.2

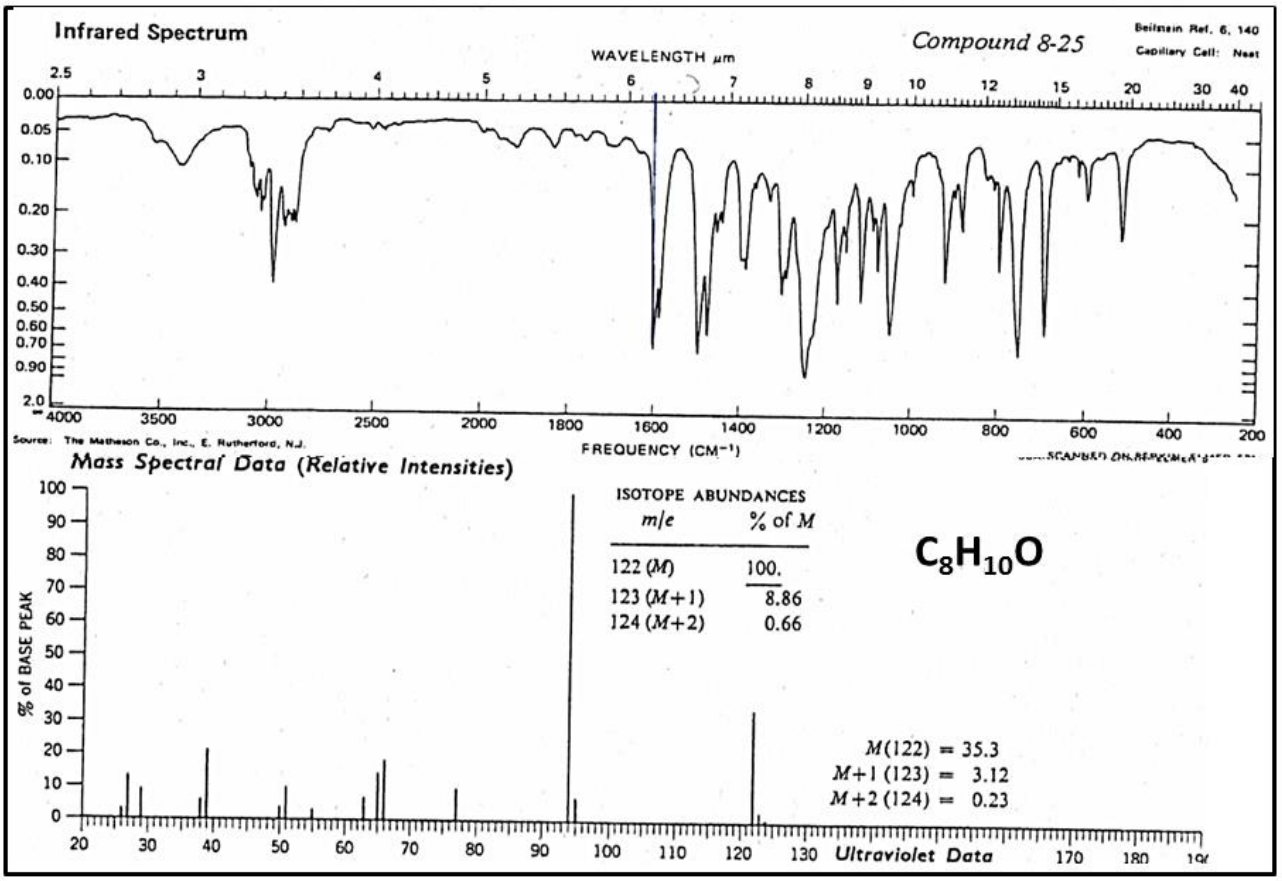
Spectre U. V.

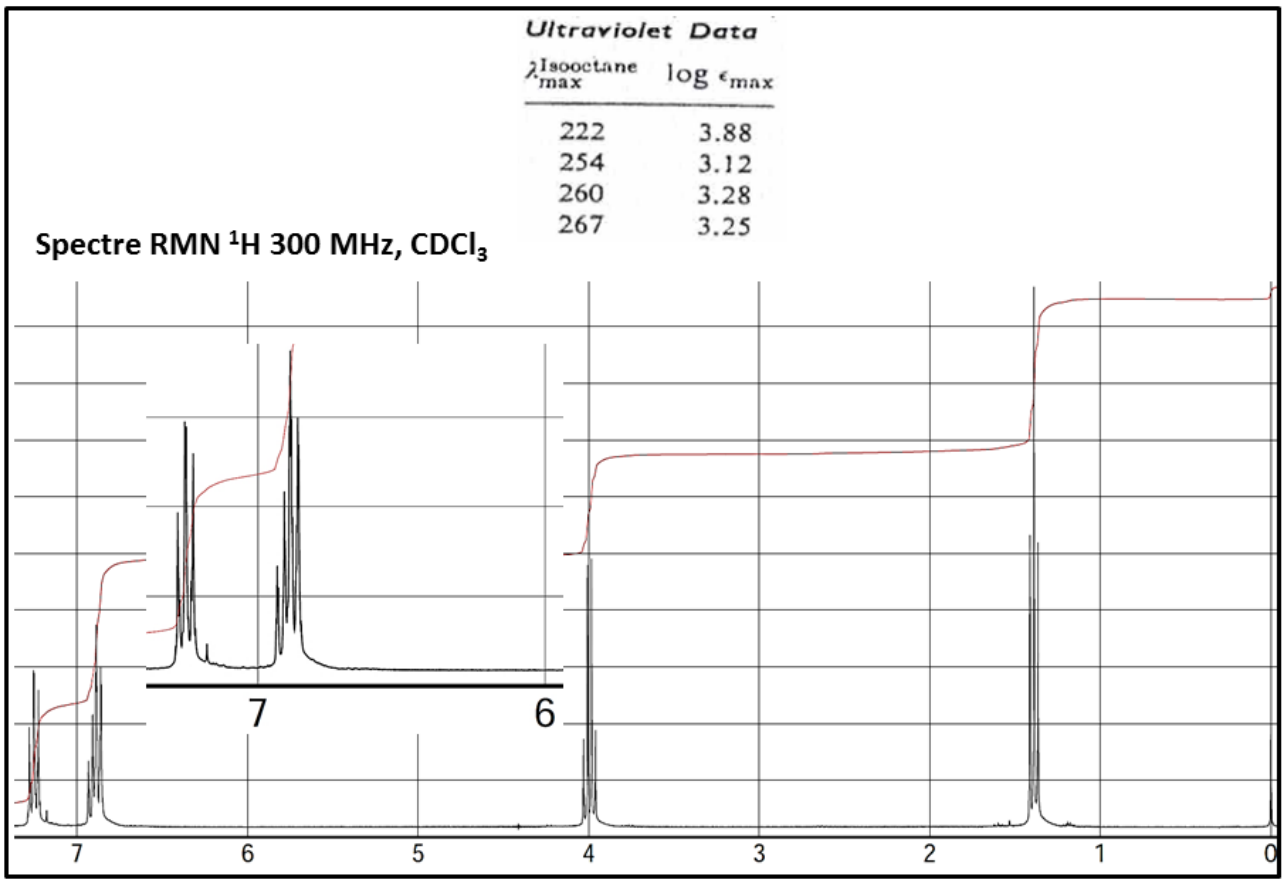
λ_{max}^{EtOH}	log ϵ_{max}
265 (e)	3.1
285 (e)	2.4

(e) = épaulement

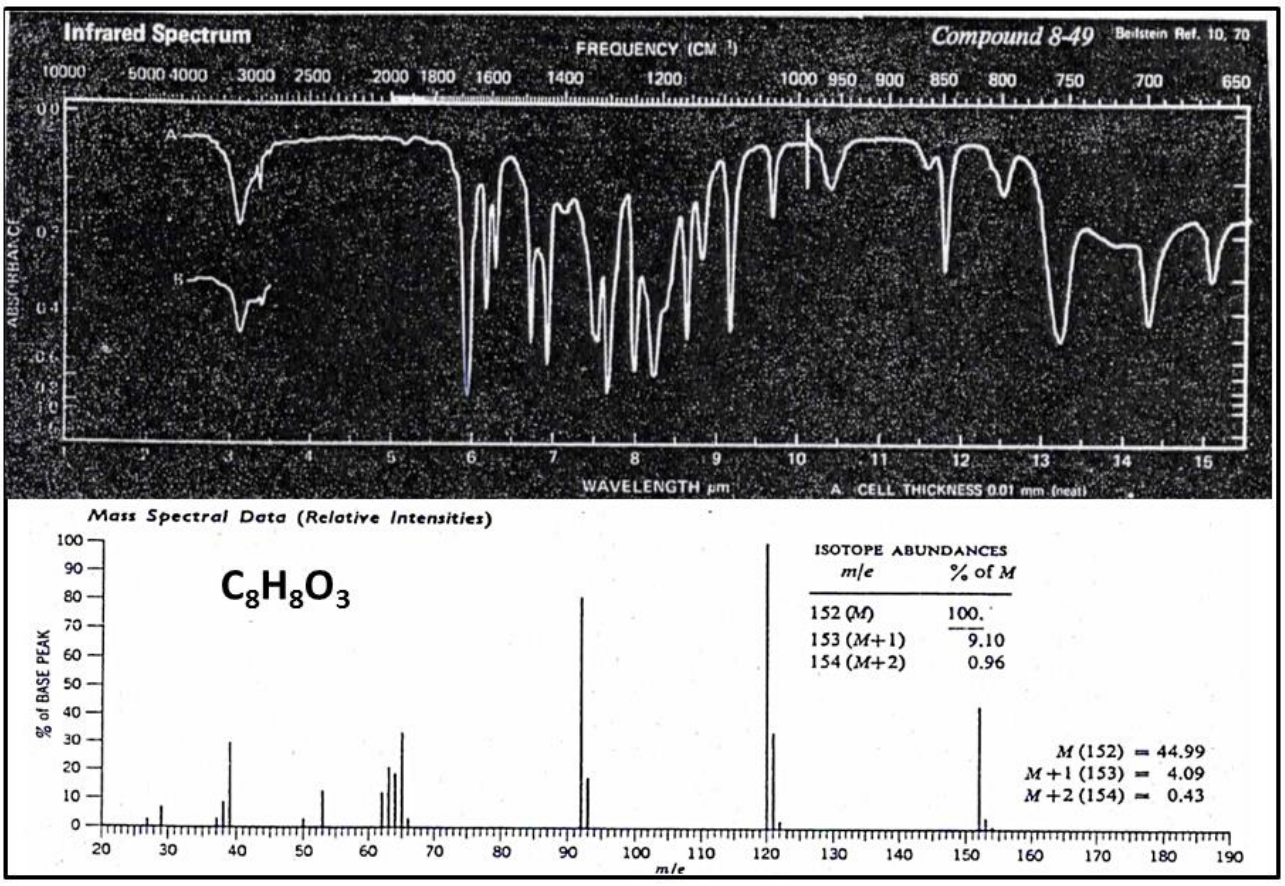


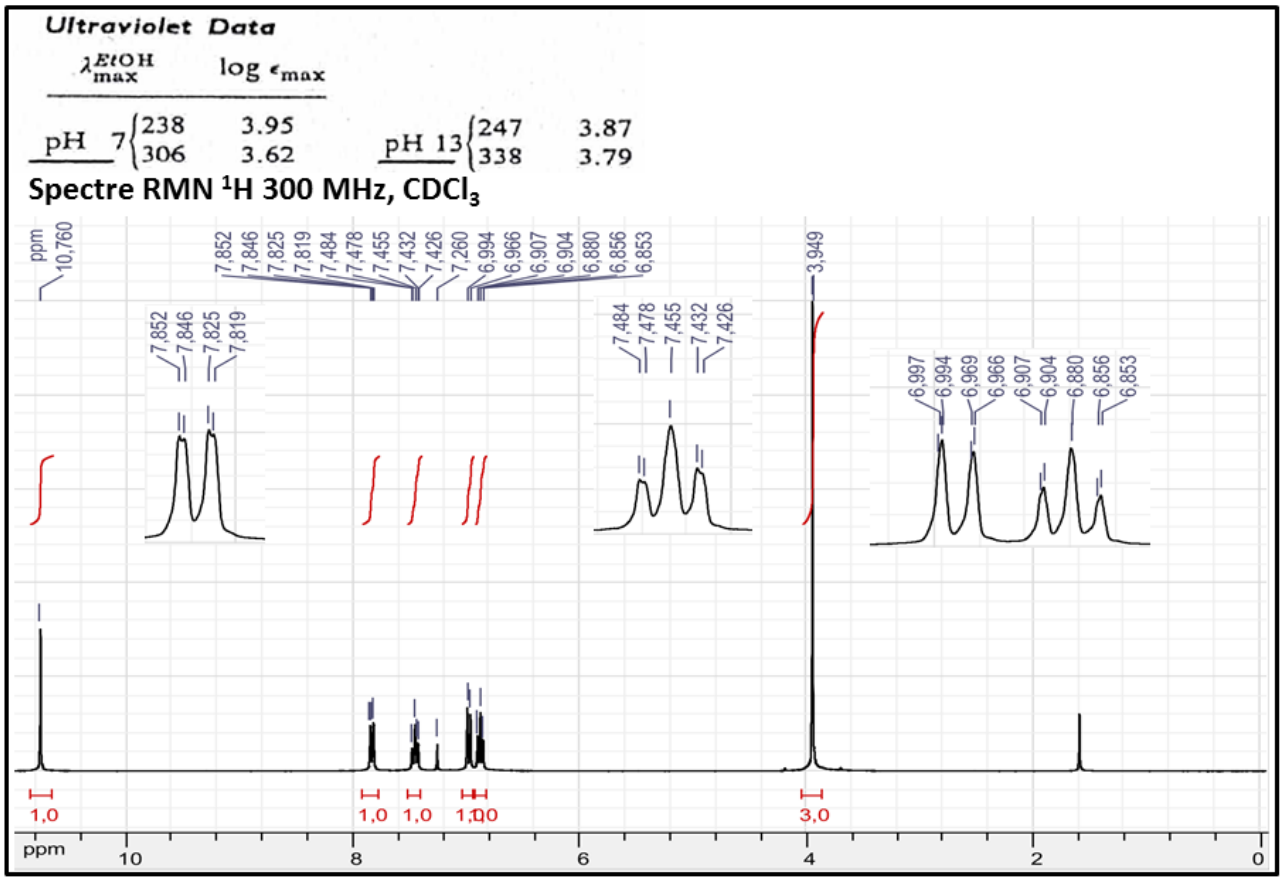
Exercise 12:


Infrared Spectrum
Compound 8-25
WAVELENGTH μm
FREQUENCY (CM⁻¹)
Mass Spectral Data (Relative Intensities)
% of BASE PEAK
ISOTOPE ABUNDANCES
m/e % of M
122 (M) 100.
123 (M+1) 8.86
124 (M+2) 0.66
C8H10O
M(122) = 35.3
M+1 (123) = 3.12
M+2 (124) = 0.23
Ultraviolet Data


Ultraviolet Data
λmax Isooctane log εmax
222 3.88
254 3.12
260 3.28
267 3.25
Spectre RMN ¹H 300 MHz, CDCl3
7
6

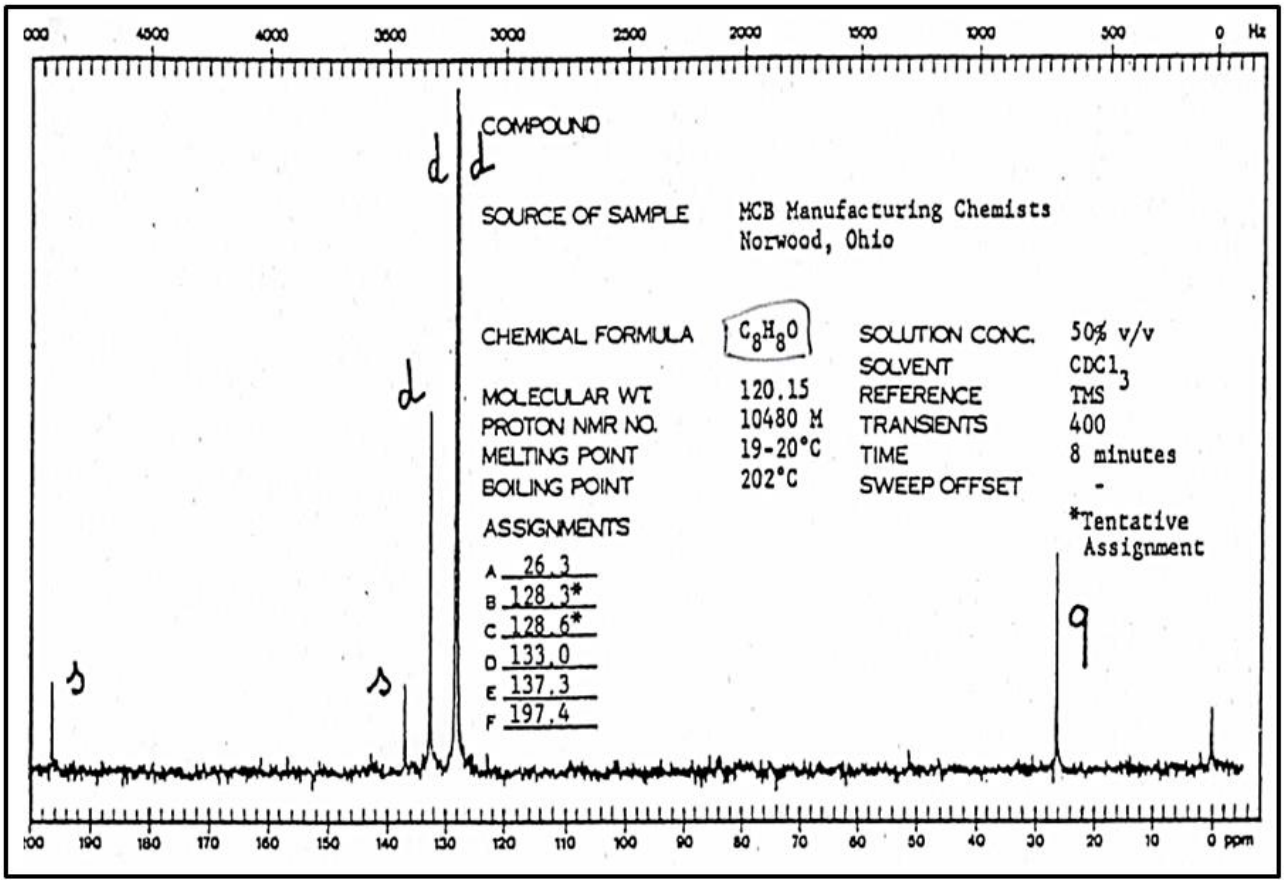
Exercise 13:


Infrared Spectrum
Compound 8-49
Beilstein Ref. 10, 70
FREQUENCY (CM⁻¹)
WAVELENGTH μm
A CELL THICKNESS 0.01 mm (neat)
Mass Spectral Data (Relative Intensities)
C8H8O3
% of BASE PEAK
m/e
ISOTOPE ABUNDANCES
m/e % of M
152 (M) 100.
153 (M+1) 9.10
154 (M+2) 0.96
M (152) = 44.99
M +1 (153) = 4.09
M +2 (154) = 0.43

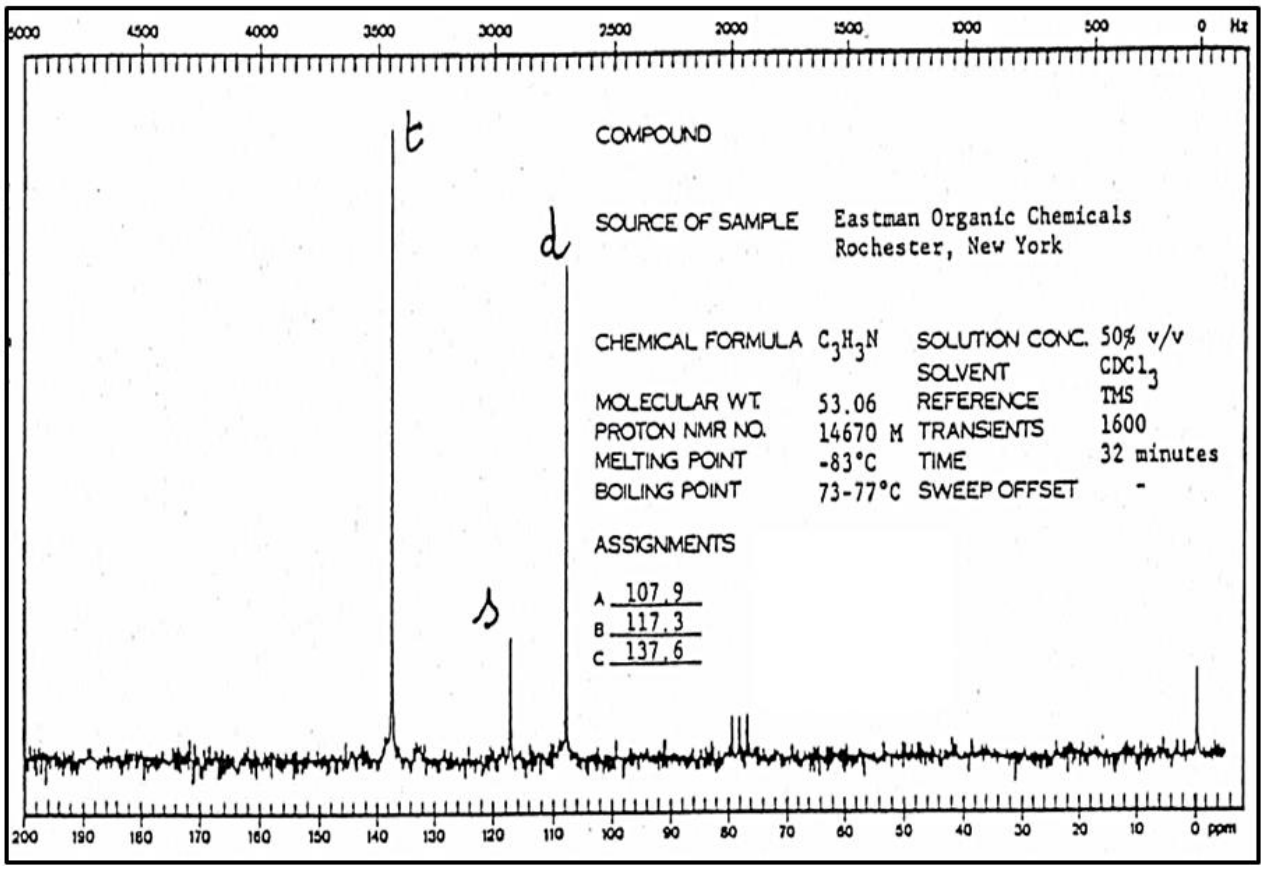

Ultraviolet Data
λmax EtOH
log εmax
pH 7 238 3.95
306 3.62
pH 13 247 3.87
338 3.79
Spectre RMN ¹H 300 MHz, CDCl3
ppm
10,760
7,852
7,846
7,825
7,819
7,484
7,478
7,455
7,432
7,426
7,260
6,994
6,966
6,907
6,904
6,880
6,856
6,853
3,949
6,997
6,969
1,0
1,0
1,0
1,00
3,0

^{13}C NMR

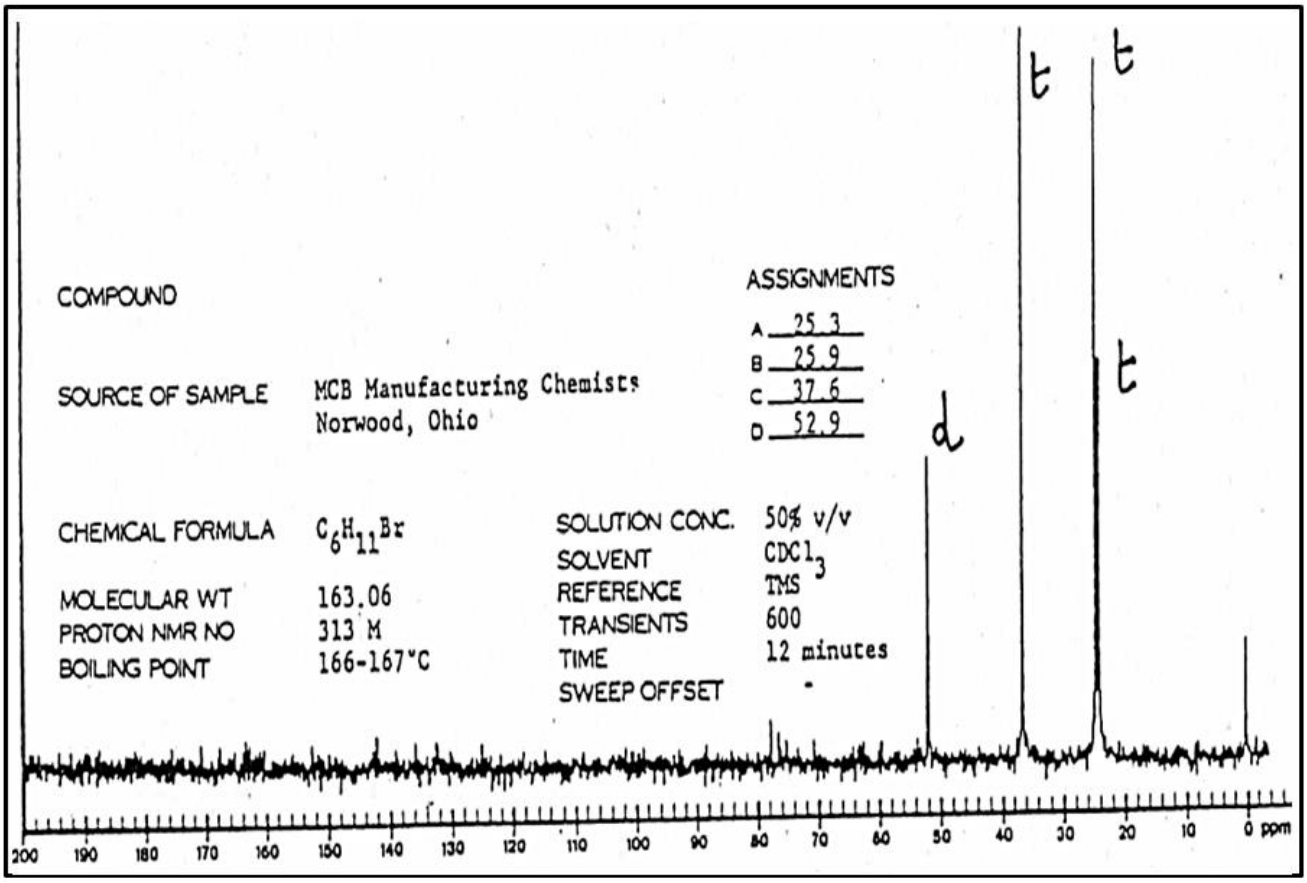
Exercise 1:



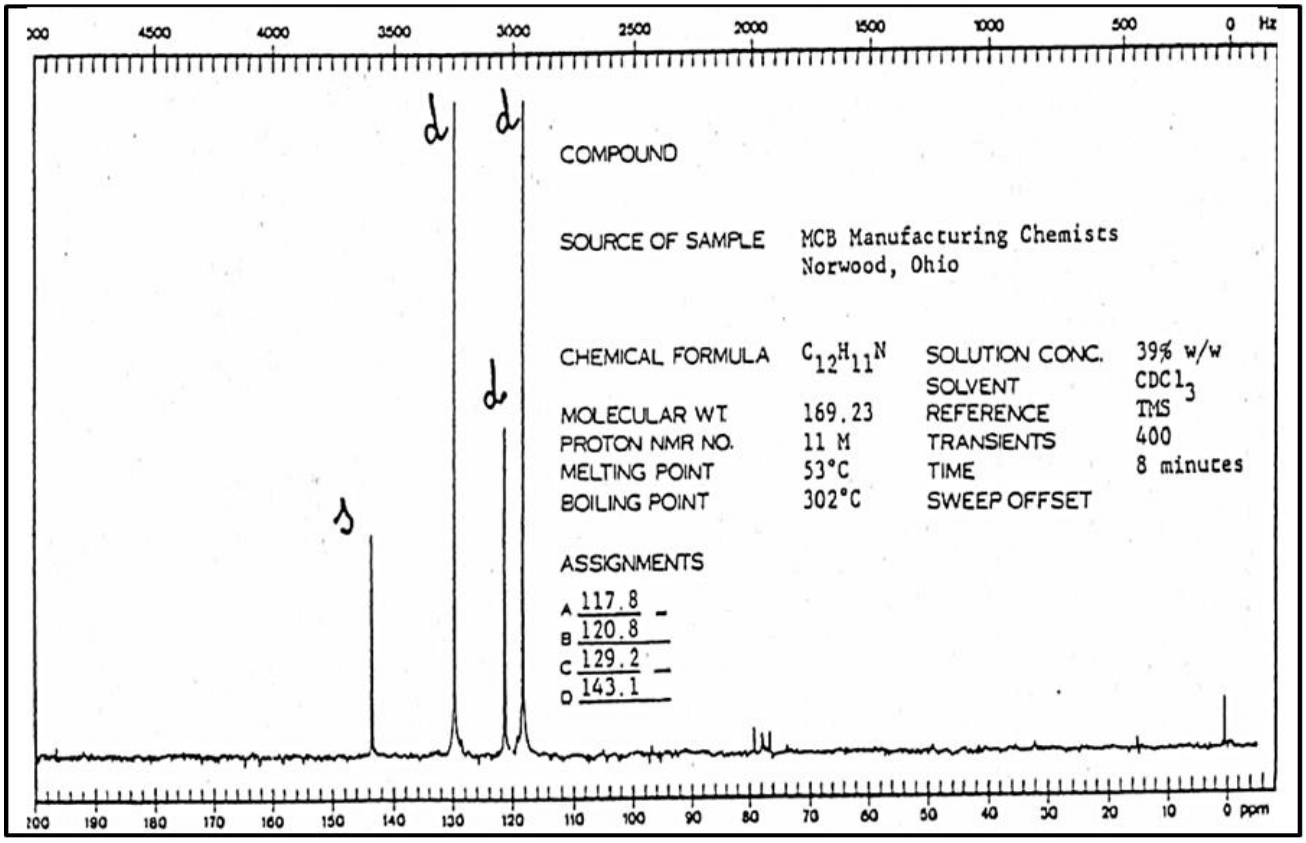
Exercise 2:



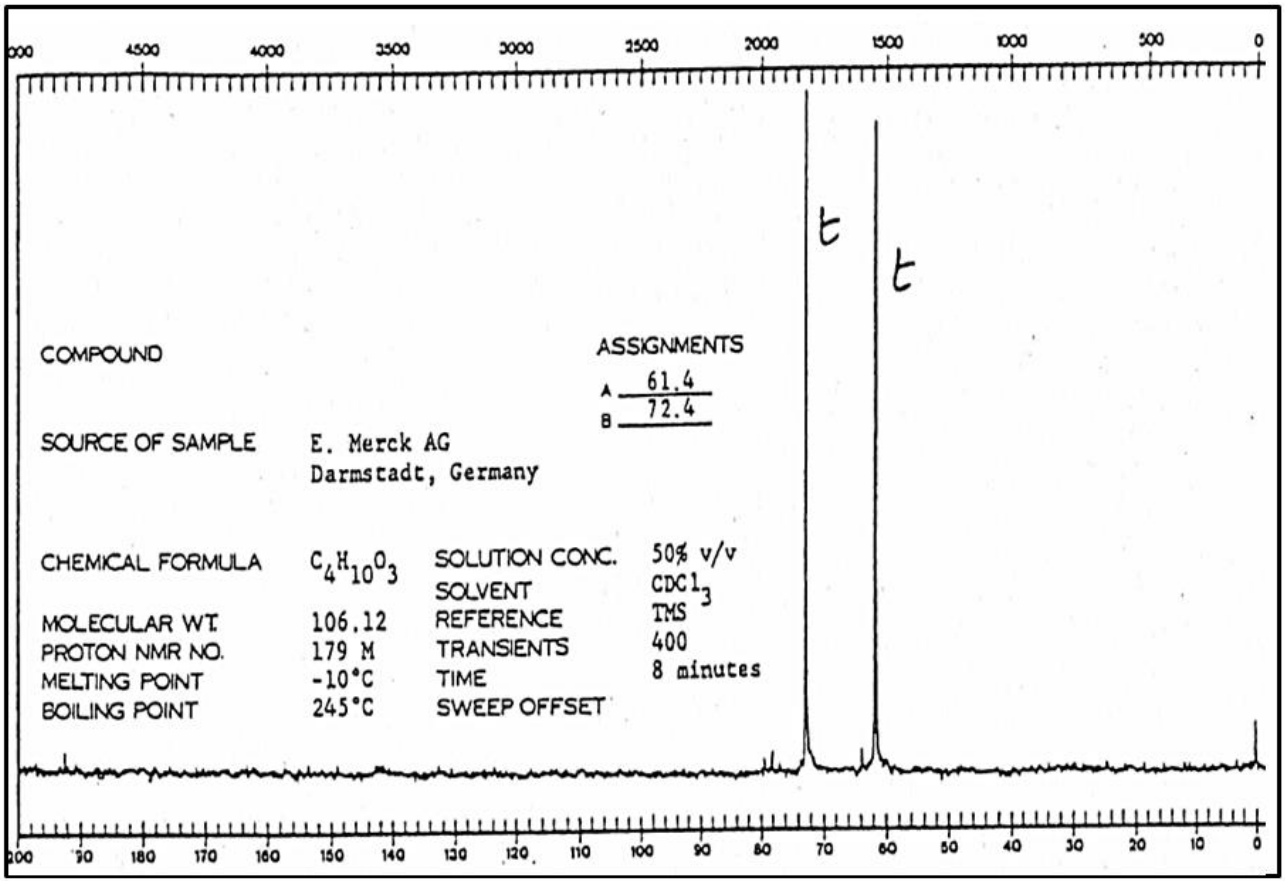
Exercise 3:



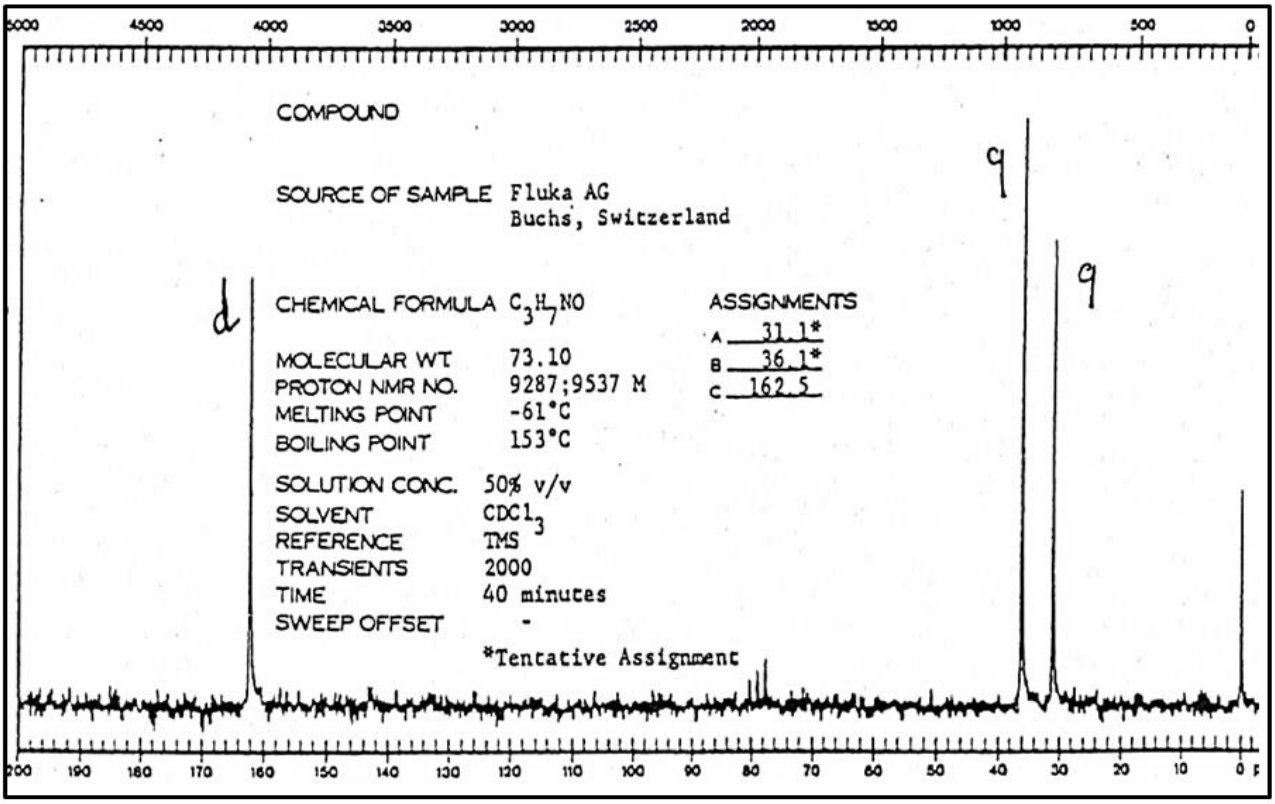
Exercise 4:



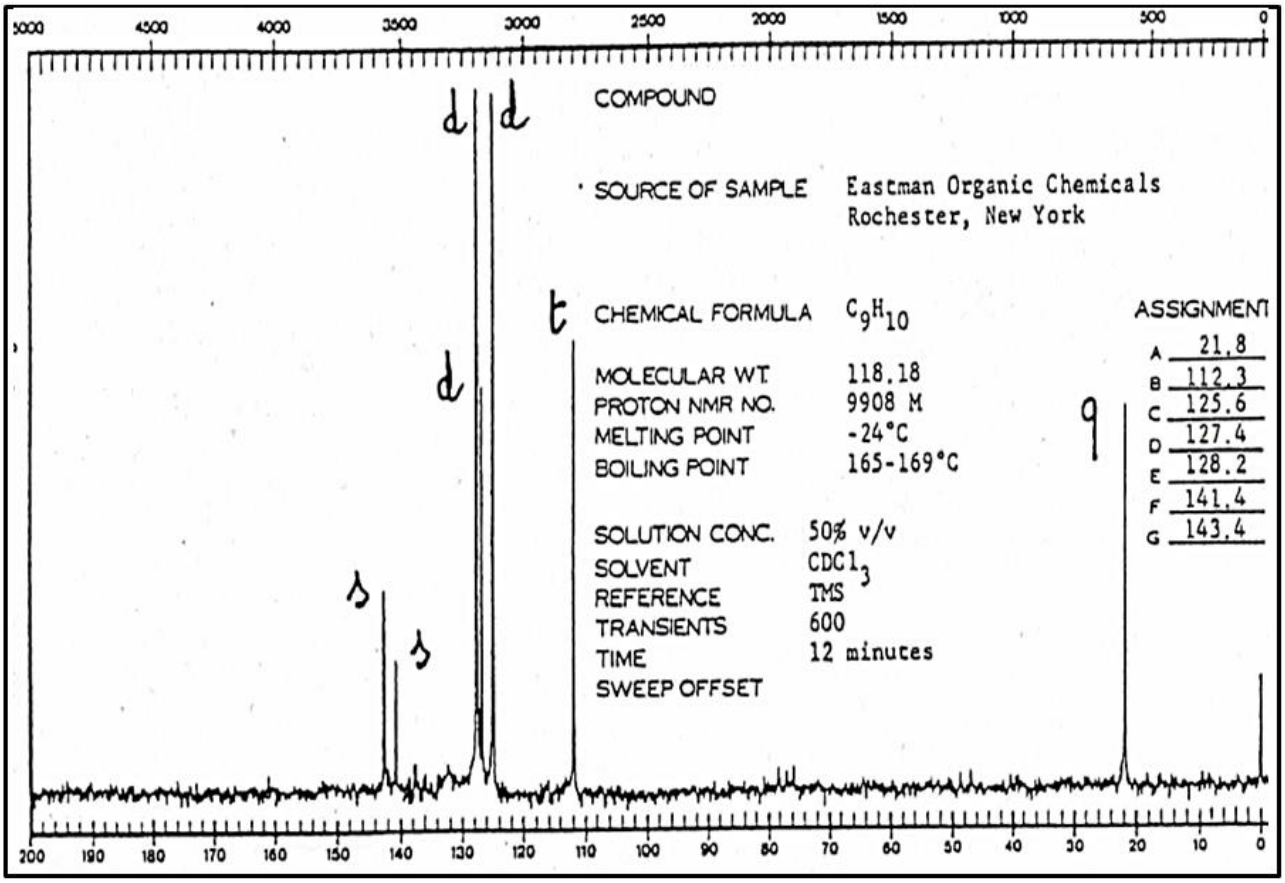
Exercise 5:



Exercise 6:



Exercise 7:



Exercise 8: