

BACHELOR OF PHARMACY HONOURS
FMU4307 – PHARMACEUTICAL ANALYSIS I – LEVEL 4
FINAL EXAMINATION
DURATION: THREE (03) HOURS

DATE: 05.10.2022

TIME: 1.30 P.M. - 4.30 P.M.

Part B- Short Answer Questions (20 Marks)

01.

1.1 The Absorbance of a colored complex that is measured in 1 cm path length and at 530 nm is found to be 0.26

(a) Calculate the Transmittance (T). (02 marks)

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1.2 If the absorbance of sample is measured in a 2 cm path length (l) cell at the same wavelength, calculate the expected absorbance. (04 marks)

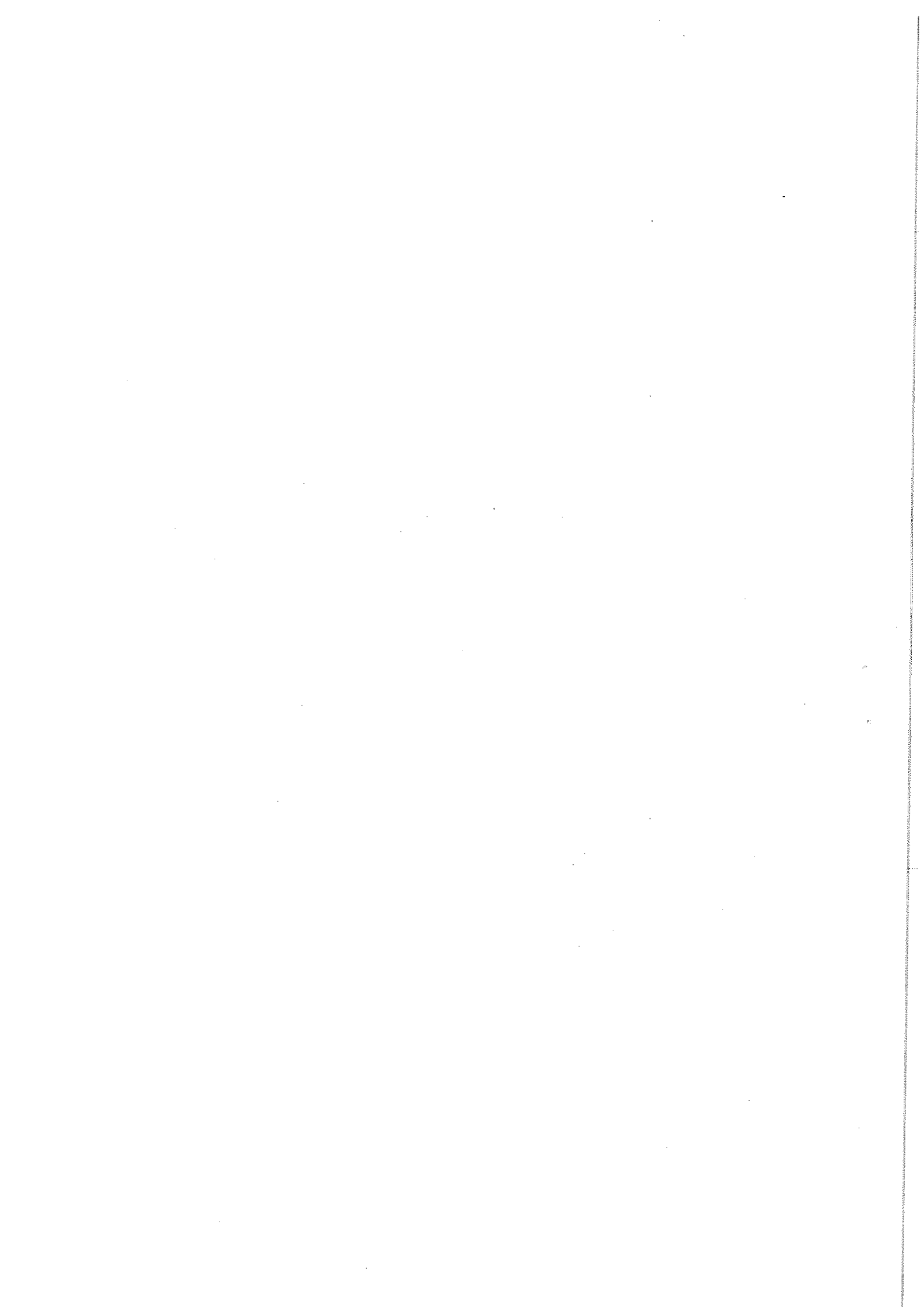
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1.3 Would the absorbance value change when the absorbance of the sample (in 1.1) measured at a different wavelength? State the reason. (02 marks)

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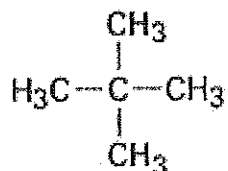
1.4 State two (02) reasons why Beers law is only applicable for dilute solutions. (02 marks)

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02.

2.1 State the m/z value of the most abundance peak of the following molecule that appears in the mass spectrum. (02 marks)

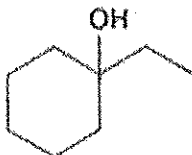


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2.2 Draw the structure of charged mass fragment when 2-pentanone ($\text{CH}_3\text{COCH}_2\text{CH}_2\text{CH}_3$) undergoes alpha cleavage in fragmentation. (02 marks)

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2.3 Give the structure of the mass fragment of the following molecule that resulted due to dehydration in fragmentation. (03 marks)



.....

2.4 Suggest another abundant mass fragment (give structure) that could occur during fragmentation in the above (2.3) molecule. (03 marks)

.....

Part C- Structured Essay Question (60 Marks)**(C=12, H=1, O=16)**

01.

1.1 State two (02) radiation sources used in atomic absorption spectrometry. (04 marks)

1.2 State why a color development reaction is required for spectrochemical analysis. (04 marks)

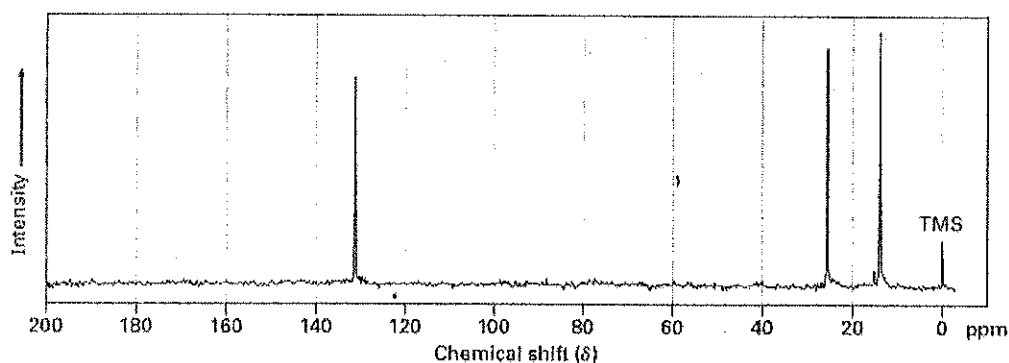
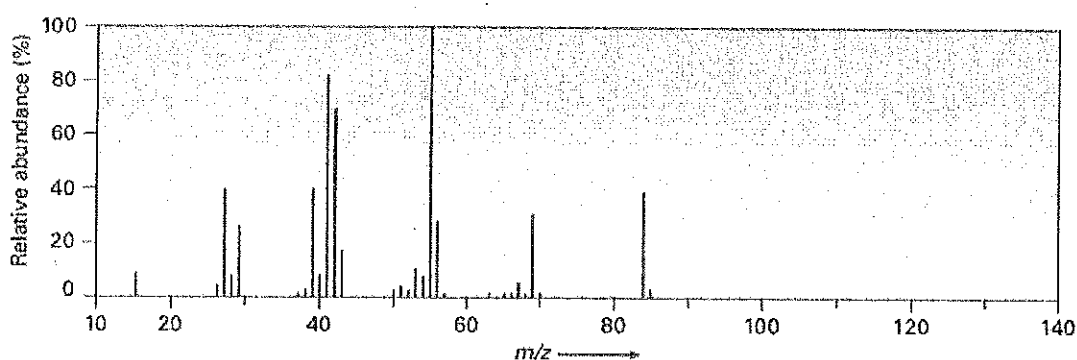
1.3 State why mercury does not require an atomization step by heating in AAS? (04 marks)

1.4 State two (02) background correction methods used in AAS. (04 marks)

1.5 State two (02) advantages of graphite furnace atomization over flame atomization in AAS. (04 marks)

02.

Given below are the mass spectrum and ^{13}C NMR spectrum of a non-aromatic hydrocarbon compound.



2.1 What is the molar mass of the compound? (1.5 marks)

2.2 What is the mass of the most abundance peak. (1.5 marks)

2.3 Draw the structure of the fragment **that lost from the molecule** to give the most abundance peak. (02 marks)

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2.4 What is this hydrocarbon (Alkane, Alkene or alkyne)? Explain your answer. (03 marks)

2.5 Draw the structure of the hydrocarbon compound that mostly fit with these spectroscopic data. (05 marks)

2.6 Where (give $\bar{\nu}$ value) the functional group of this molecule appears in the IR spectrum. (02 marks)

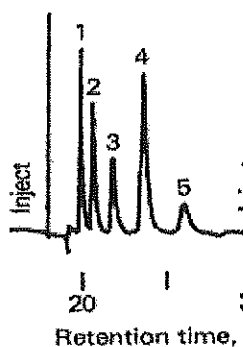
03.

3.1 Name three (03) chromatographic techniques based on its nature of the mobile phase. (03 marks)

3.2 Name three (03) information that can be obtained from a chromatograph. (03 marks)

3.3 Briefly explain how Elution chromatography is performed. (03 marks)

3.4 Given below is a model chromatograph taken with a sample containing 6 compounds



Given below are some chromatographic data for compounds 4 and 5.

Compound	Retention time	Width at peak base
4	23.2	0.27
5	24.2	0.32

(a) State why the chromatogram shows only 5 peaks though sample contains six compounds. (02 marks)

3.5 Calculate the resolution (R_s) for compound 5 and 6 and comment on the resolution status of the two compounds. Resolution of two compounds A and B is given by, (04 marks)

$$R_s = \frac{2[(t_R)_B - (t_R)_A]}{W_A + W_B}$$

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04.

- 4.1 State the role of following parts of gas chromatography (03 marks)
- (a) Column oven –
 - (b) ^{63}Ni isotope in electron capture detector –
 - (c) Inlet system –
- 4.2 Name two (02) injectors used in gas chromatography. (02 marks)
- 4.3 State three (03) important features required for an ideal detector used in GC (03 marks)
- 4.4 Briefly explain the operating principles of the flame ionization detector used in GC. (04 marks)
- 4.5 State three (03) operating conditions required for liquid chromatography (03 marks)

Data sheet

Type of hydrogen	Chemical shift (δ)	Type of hydrogen	Chemical shift (δ)		
Reference	$\text{Si}(\text{CH}_3)_4$	0			
Alkyl (primary)	$-\text{CH}_3$	0.7-1.3	Alcohol		
Alkyl (secondary)	$-\text{CH}_2-$	1.2-1.6	$\begin{array}{c} \\ -\text{C}-\text{O}-\text{H} \\ \end{array}$	2.5-5.0	
Alkyl (tertiary)	$\begin{array}{c} \\ -\text{CH}- \\ \end{array}$	1.4-1.8	Alcohol, ether	$\begin{array}{c} \text{H} \\ \\ -\text{C}-\text{O}- \\ \end{array}$	3.3-4.5
Allylic	$\begin{array}{c} \text{H} \\ \\ \text{C}=\text{C}-\text{C}- \\ \end{array}$	1.6-2.2	Vinyllic	$\begin{array}{c} \text{H} \\ \diagup \\ \text{C}=\text{C} \\ \diagdown \end{array}$	4.5-6.5
Methyl ketone	$\begin{array}{c} \text{O} \\ \\ -\text{C}-\text{CH}_3 \end{array}$	2.0-2.4	Aryl	$\text{Ar}-\text{H}$	6.5-8.0
Aromatic methyl	$\text{Ar}-\text{CH}_3$	2.4-2.7	Aldehyde	$\begin{array}{c} \text{O} \\ \\ -\text{C}-\text{H} \end{array}$	9.7-10.0
Alkynyl	$-\text{C}\equiv\text{C}-\text{H}$	2.5-3.0	Carboxylic acid	$\begin{array}{c} \text{O} \\ \\ -\text{C}-\text{O}-\text{H} \end{array}$	11.0-12.0
Alkyl halide	$\begin{array}{c} \text{H} \\ \\ -\text{C}-\text{Hal} \\ \end{array}$	2.5-4.0			

Functional Group	Absorption (cm^{-1})	Intensity	Functional Group	Absorption (cm^{-1})	Intensity
Alkane			Amine		
C-H	2850-2960	Medium	N-H	3300-3500	Medium
Alkene			C-N	1030-1230	Medium
=C-H	3020-3100	Medium	Carbonyl compound		
C=C	1640-1680	Medium	C=O	1670-1780	Strong
Alkyne			Aldehyde	1730	Strong
=C-H	3300	Strong	Ketone	1715	Strong
C=C	2100-2260	Medium	Ester	1735	Strong
Alkyl halide			Amide	1690	Strong
C-Cl	600-800	Strong	Carboxylic acid	1710	Strong
C-Br	500-600	Strong	Carboxylic acid		
Alcohol			O-H	2500-3100	Strong, broad
O-H	3400-3650	Strong, broad	Nitrile		
C-O	1050-1150	Strong	C=N	2210-2260	Medium
Arene			Nitro		
C-H	3030	Weak	NO ₂	1540	Strong
Aromatic ring	1660-2000	Weak			
	1450-1600	Medium			

