098

THE OPEN UNIVERSITY OF SRI LANKA
FACULTY OF HEALTH SCIENCES
DEPARTMENT OF PHARMACY
ACADEMIC YEAR 2019/2020 – SEMSETER II

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

DATE: 06TH MAY 2021 TIME: 09.30 A.M. – 12.30 P.M.

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Part B – SAQs (20 marks)

01.

The % transmittance (%T) of an aqueous sample of a colored compound with a concentration of 0.015 mol dm^{-3} that is subjected to spectrochemical analysis is found to be 65.9% at a particular wavelength.

1.1 Calculate the absorbance of this solution. (02 marks)

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1.2 If this ample is run in 1 cm path length (l) cell, calculate the molar absorptivity coefficient(ε) (03 marks)

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1.3 Would the absorbance value change when this sample tested at a different wavelength? State the reason. (02 marks)

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1.4 State three chemical deviations that limit the use of Beer's law. (03 marks)

I. .....

II. .....

III. .....

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02.

The retention times (t_R) of compounds A and B are 2.34 min and 3.15 min respectively. The holdup time (t_M) for this column is 0.72 min.

2.1 Calculate the time that compound A spent in the stationary phase. (02 marks)

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2.2 Determine time that compound A spent in mobile phase. (01 mark)

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2.3 Do both A and B compounds spent the same time in the mobile phase? (01 mark)

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2.4 Calculate the capacity factor (k′) for compound B. (03 marks)

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2.5 Based on the capacity factor (k′) value you obtained in 2.4, comment on the chromatographic efficiency of compound B. (03 marks)

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Part C – SEQs (60 marks)

(Each SEQ has 35 marks and total marks for Part C will be converted into 60 marks)

01.

You are given unlabeled samples of acetone (CH_3COCH_3) and propan-1-ol ($H_3CCH_2CH_2OH$). (C =12, H=1, O =16)

1.1 Identify the functional group/s appear in IR spectrum for acetone and give the approximate wave number of functional group/s. (04 marks)

1.2 Identify the functional group/s that appear in the IR spectrum for and propan-1-ol and give the approximate wave number of functional group/s. (06 marks)

1.3 State how could you use IR spectroscopy to distinguish these two compounds?(07 marks)

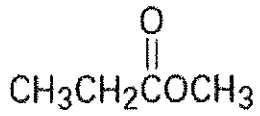
1.4 Give the **m/z value**, **the structure**, and the **name of the cleavage path** of the most dominant mass fragment of acetone produced in the mass spectrum. (06 marks)

1.5 Give the **m/z value**, the **structure**, and the **name of the cleavage path** of the most dominant mass fragment of propan-1-ol produced in mass spectrum. (06 marks)

1.6 How could you use mass spectroscopy to distinguish these two compounds? (06 marks)

02.

Given below is the structure of methyl propionate



2.1 Which Spectrum (FTIR or ^1H NMR) is the best one for the identification of structural information (03 marks)

2.2 What is the main functional group in this molecule? (03 marks)

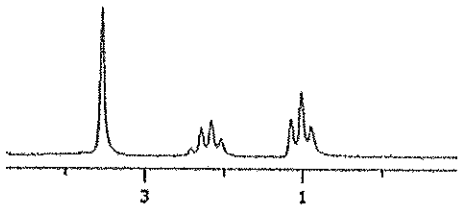
2.3 Can you identify the main functional group you selected in this molecule using ^1H NMR spectrum? Justify your answer. (06 marks)

2.4 Explain how do you identify the functional group/s of methyl propionate this using IR spectroscopy. (05 marks)

2.5 How many peaks you expect in ^{13}C NMR spectrum of methyl propionate. (03 marks)

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2.6 Sketch the ^1H NMR spectrum of methyl propionate. Show the splitting patterns. (09 marks)



2.7 Explain how the splitting patterns occur in your ^1H NMR sketch. (06 marks)

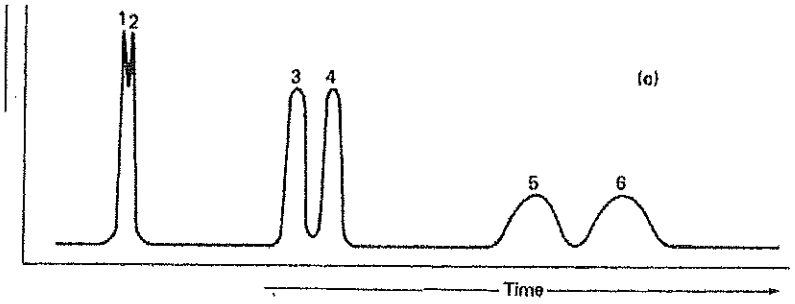
03.

3.1 Name three (03) chromatographic techniques based on their nature of the mobile phase. (06 marks)

3.2 Name three (03) information that can be obtained from a chromatograph. (06 marks)

3.3 Briefly explain how the separation (of two compounds) takes place in chromatography. (08 marks)

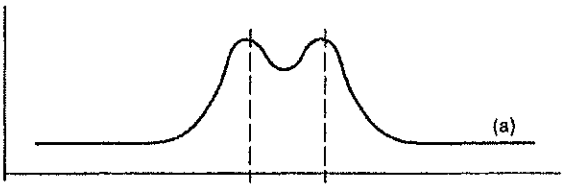
3.4 Given below is a model chromatograph



3.4.1 Briefly explain the separation status of compounds 1 and 2, 3 and 4, and 5 and 6. (06 marks)

3.4.2 What can you do to improve the separation of compound 1 and 2. (04 marks)

3.5 Given below is the chromatograph of two unresolved peaks.



Briefly explain two (02) conceptual methods to improve the separation of these two compounds. (05 marks)

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04.

4.1 State role of following parts of gas chromatography

4.1.1 Inlet system (03 marks)

4.1.2 Split point in inlet system (03 marks)

4.1.3 Septum (03 marks)

4.2 Name three (03) ionization detectors used in Gas chromatography . (06 marks)

4.3

4.3.1 State why regular pump is not suitable for liquid chromatography to run mobile phase. (06 marks)

4.3.2 State how to overcome this problem. (04 marks)

4.4

4.4.1 State three (03) interferences occur in atomic spectroscopy. (06 marks)

4.4.2 State two (02) methods that can be used to correct the background in atomic spectroscopy. (04 marks)

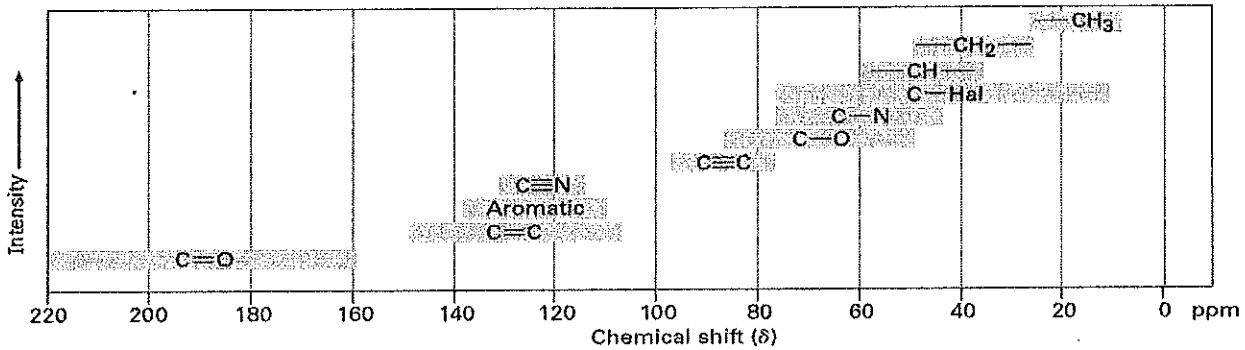


Figure 01 - ^{13}C NMR Chemical shifts

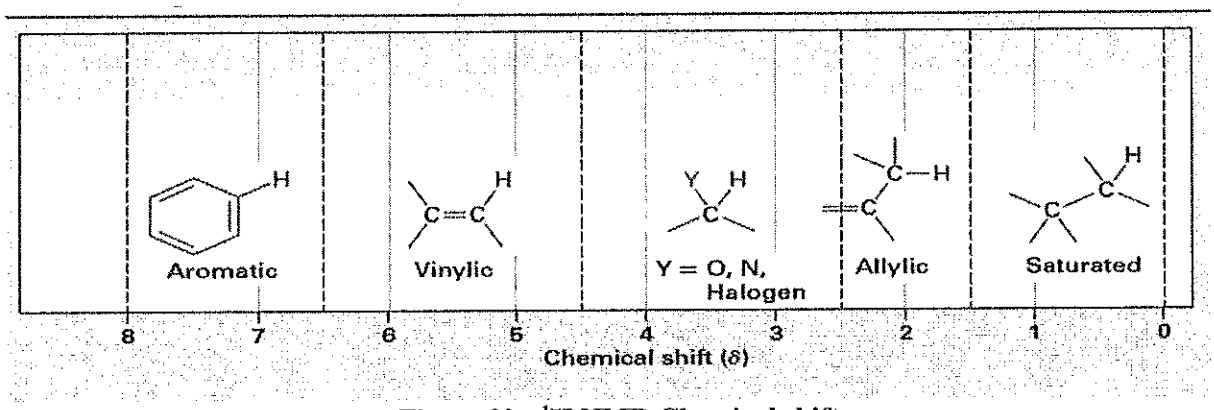


Figure 02 - ^{1}H NMR Chemical shifts

Table 01 - Characteristic IR bands of functional groups.

Functional Group	Absorption (cm^{-1})	Intensity
Alkane		
C–H	2850–2960	Medium
Alkene		
=C–H	3020–3100	Medium
C=C	1640–1680	Medium
Alkyne		
≡C–H	3300	Strong
C≡C	2100–2260	Medium
Alkyl halide		
C–Cl	600–800	Strong
C–Br	500–600	Strong
Alcohol		
O–H	3400–3650	Strong, broad
C–O	1050–1150	Strong
Arene		
C–H	3030	Weak
Aromatic ring	1660–2000	Weak
	1450–1600	Medium

Functional Group	Absorption (cm^{-1})	Intensity
Amine		
N–H	3300–3500	Medium
C–N	1030–1230	Medium
Carbonyl compound		
C=O	1670–1780	Strong
Aldehyde	1730	Strong
Ketone	1715	Strong
Ester	1735	Strong
Amide	1690	Strong
Carboxylic acid	1710	Strong
Carboxylic acid		
O–H	2500–3100	Strong, broad
Nitrile		
C≡N	2210–2260	Medium
Nitro		
NO_2	1540	Strong