



UNIVERSITY OF RUHUNA – FACULTY OF MEDICINE
ALLIED HEALTH SCIENCES DEGREE PROGRAMME
FOURTH BPHARM PART I EXAMINATION – DECEMBER 2016
PH 4123 PHARMACEUTICAL ANALYSIS (SEQ)

TIME: TWO HOURS

INSTRUCTIONS

- Answer **all** questions.
- Do not use any correction fluid.
- Answer questions in the booklet provided.
- Marks will be deducted for illegible hand writing.

01. Answer **all** parts

1.1 A liquid chromatographic column with a packing length of 24.7 cm was used to separate a four component (A-D) mixture. The chromatogram provided the following data:

	Retention time / min	Width of Peak Base (W), min
A	4.4	0.31
B	12.1	1.05
C	13.5	1.24
D	20.6	1.53

1.1.1 Calculate the number of plates from each peak.

1.1.2 Calculate an average plate height for the column.

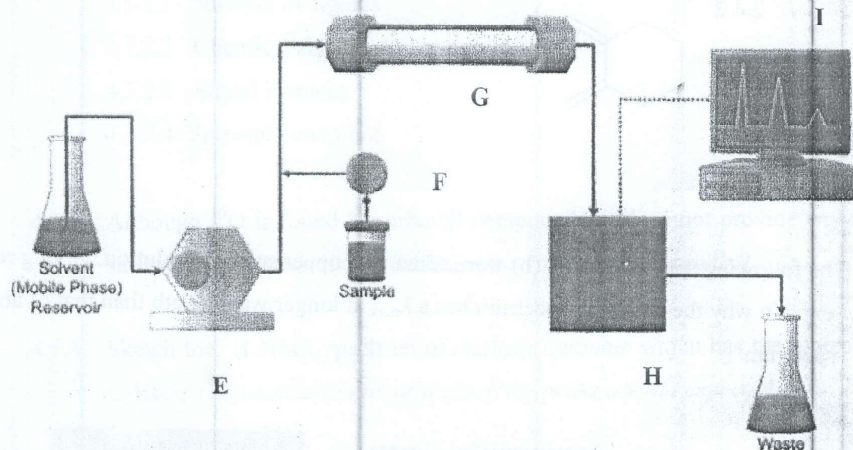
(30 marks)

1.2 Explain by giving reaction(s) how the strong and weak cation exchange resins are synthesized.

(25 marks)

1.3

1.3.1 Following diagram illustrates basic components of a high performance liquid chromatography (HPLC) system. Identify the components E-I.



1.3.2 What is the use of 'E' in the HPLC system.

(25 marks)

1.4

1.4.1 List three (03) major types of detectors used in gas chromatography.

1.4.2 Name a detector which is more sensitive to chlorinated pesticides.

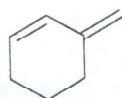
(20 marks)

2. Answer **all** parts2.1 State the Beer-Lambert Law. (05 marks)2.2 A 1.50×10^{-5} M solution of nickel sulphate has a transmittance of 16.2% when measured in a 1.01 cm cell at a wavelength of 644 nm. Calculate the absorbance of this solution and the molar absorptivity of nickel sulphate. (20 marks)2.3 A series of permanganate solutions given in the following table was used to prepare a calibration curve and determined the concentration of an unknown solution. Calculate the permanganate concentration of unknown solution. (Please note that the graphs are provided). (30 marks)

Concentration in μM	Absorbance measured
30.00	0.152
60.00	0.340
90.00	0.501
120.00	0.669
150.00	0.843
Unknown solution	0.528

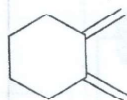
2.4 Using Woodward-Fieser rules, calculate the λ_{max} for the following compounds.

2.4.1

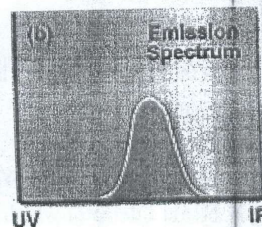
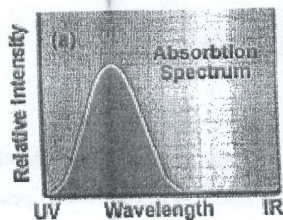


(15 marks)

2.4.2



(15 marks)

2.5 The following spectra (a) and (b) were taken for copper sulphate solution. Giving reasons, explain why the emission spectrum has a λ_{max} at longer wavelength than that of absorption spectrum. (15 marks)

3. Answer all parts

- 3.1 Whenever we use a conductivity meter, it is essential to calculate the cell constant.
- 3.1.1 Identifying terms, write down an expression for cell constant. (10 marks)
- 3.1.2 Resistance of a conductivity cell filled with 0.74 % KCl is 411.82 Ω . The conductivity of 0.74 % KCl is 1,286 S m^{-1} at 25 $^{\circ}\text{C}$. Calculate the cell constant of the conductivity cell. (10 marks)
- 3.1.3 Draw the conductance vs volume of base for the titration of acetic acid with ammonium hydroxide. Giving reasons, explain the shape of the graph and mark the end point. (20 marks)
- 3.2
- 3.2.1 Devise a cell in which the cell reaction is $\text{Mn}(s) + \text{Cl}_2(g) \rightarrow \text{MnCl}_2(aq)$. (10 marks)
- 3.2.2 Give the half reactions for the electrodes and from the standard cell potential of 2.54 V, deduce the standard potential of the Mn^{2+}/Mn couple. (20 marks)
- 3.3 Determination of water content in pharmaceutical products is essential.
- 3.3.1 Why do you need to determine water content in pharmaceutical products? (10 marks)
- 3.3.2 Explain two spectroscopic methods that can be used to determine water content in drugs. (20 marks)

4. Answer all parts.

- 4.1 Answer the following questions pertaining to Nuclear Magnetic Resonance (NMR) spectroscopy.
- 4.1.1 List five nuclei which are interested in NMR spectroscopy? (05 marks)
- 4.1.2 Indicate the basic structural information which could be derived from the following features of NMR spectra:
- 4.1.2.1 Number of signals
- 4.1.2.2 Chemical shift
- 4.1.2.3 Signal intensity
- 4.1.2.4 Spin-spin coupling (05 marks)
- 4.1.3 Although ^{16}O is found in carbonyl compounds, it does not provide any information for structure elucidation in ^1H NMR spectroscopy. Explain briefly? (10 marks)
- 4.1.4 Sketch the ^1H NMR spectrum of *methylpropionate* which has the molecular formula of $\text{C}_4\text{H}_8\text{O}_2$. (The actual chemical shifts of the peaks are not expected). (10 marks)
- 4.1.5 Derive the multiplicity and intensity ratio of the ^1H NMR signal of CH_3 group of the above molecule using a suitable stick diagram. (10 marks)
- 4.1.6 Suggest a method to distinguish *cyclopropane* and *chlorocyclopropane* using ^1H NMR spectroscopy. Giving reasons, justify your answer. (10 marks)

4.2

4.2.1 Arrange each set of bonds in order of increasing stretching frequency.

4.2.1.1 C-N, C=N, C≡N

4.2.1.2 sp^3 C-H, sp^3 C-N and sp^3 C-O4.2.1.3 The C-H, C-D and C-Cl bonds of $CHCl_3$ and $CDCl_3$

(10 marks)

4.2.2 Assign these stretching frequencies to the corresponding bonds in methanol: 3340, 2945, and 1030 cm^{-1} .

(10 marks)

4.2.3 The atmosphere of Titan was found to contain traces (1.5 ppb) of cyanoacetylene (C_3HN). IR spectrum of cyanoacetylene contains two distinct bands at 2250 and 2150 cm^{-1} and a sharp band above 3000 cm^{-1} .

4.2.3.1 Draw the structure of cyanoacetylene molecule.

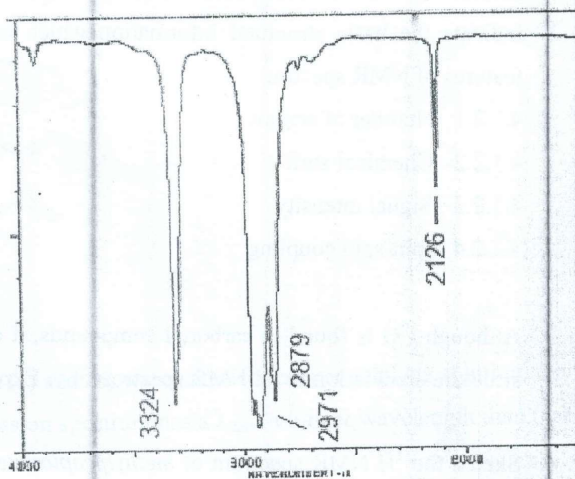
(10 marks)

4.2.3.2 Assign approximate peak values (wavenumbers) to the appropriate bonds.

(10 marks)

4.2.4 The molecule with molecular formula C_4H_6 gives following IR spectrum. Propose a reasonable structure for the molecule. Assign indicated wavenumbers to the appropriate bonds.

(10 marks)



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