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UNIVERSITY OF RUHUNA – FACULTY OF ALLIED HEALTH SCIENCES

DEPARTMENT OF PHARMACY

FOURTH BPHARM PART I EXAMINATION – DECEMBER 2017

PH 4123 PHARMACEUTICAL ANALYSIS (SEQ)

TIME: TWO HOURS

INSTRUCTIONS

- There are **four (04)** questions.
- Answer each question in a separate booklet provided.
- Do not use any correction fluid.
- Marks will be deducted for illegible hand writing.

01. Answer **all** parts.

1.1

1.1.1 An open tubular column used for gas chromatography had an inside diameter of 0.25 mm. A volumetric flow rate of 0.95 mL/min was used. Find the linear flow velocity in cm/s at the column outlet.

1.1.2 A packed column in gas chromatography had an inside diameter of 5.0 mm. The measured volumetric flow rate at the column outlet was 48.0 mL/min. If the column porosity was 0.43, what was the linear flow velocity in cm/s?

(20 marks)

1.2 Calculate an average number of plates required for a packed column with height, 40-cm in a gas-liquid chromatography by using the data given in the table below.

(20 marks)

Compound	Retention time (t_R, min)	Width (W, min)
Air	1.9	—
Methylcyclohexane	10.0	0.76
Methylcyclohexene	10.9	0.82
Toluene	13.4	1.06

1.3 Open tubular columns are widely used in gas chromatographic separations.

1.3.1 What are the major types of open tubular columns? (Illustrations are necessary). ***(25 marks)***

1.3.2 Name the type of the column stated in question 1.3.1, which can be used for larger samples? ***(05 marks)***

1.4 Using a suitable diagram, briefly explain the "eddy diffusion" in chromatography. ***(15 marks)***

1.5 Briefly describe how you would improve the resolution of a mixture of amino acids those have similar R_f values, using a chromatographic development method. ***(15 marks)***

02. Answer **all** parts

2.1

2.1.1 Light consists of photons. Write down the relationship between energy of a photon and the wavelength of the light. ***(10 marks)***

2.1.2 Derive the relationship between transmittance (T) and the absorbance (A). ***(10 marks)***

2.2 Explain briefly how a UV- visible spectrophotometer and a flame atomic absorption spectrophotometer work by using schematic diagrams. ***(25 marks)***

2.3

2.3.1 Indicate different types of possible electronic transitions of an organic molecule using an energy level diagram. ***(10 marks)***

2.3.2 Explain non radiative processes of an excited state molecule by using the Jablonski diagram. ***(10 marks)***

2.4 Write short accounts on

2.4.1 Photoluminescence

2.4.2 Chemiluminescence

(35 marks)

03. Answer **all** parts.

3.1 Electrochemical methods are selective, sensitive, rapid, and provide easy techniques in the pharmaceutical field.

3.1.1 Sketch a diagram of a galvanic cell where the following reaction takes place.

$2\ Al(s) + 3\ ZnSO_4(aq) \rightarrow Al_2(SO_4)_3\ (aq) + 3\ Zn(s)$

3.1.2 Label the electrodes and indicate the flow of electrons in the cell.

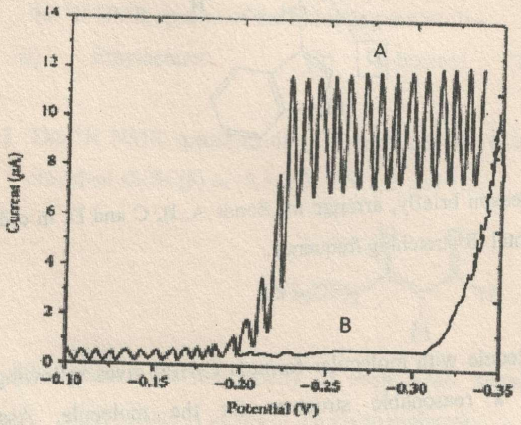
3.1.3 Write down the half-cell reactions of the anode and the cathode.

3.1.4 Assuming that the concentrations of $Al_2(SO_4)_3$ and $ZnSO_4$ are C_1 ($moldm^{-3}$) and C_2 ($moldm^{-3}$) respectively, write down the standard cell notation for the above mentioned galvanic cell.

(10 x4 marks)

3.2 In polarography, the current flowing through the cell is computed as a function of the potential of the working electrode and this current is proportional to the concentration of the analyte.

3.2.1 A polarogram for a 1 mol L^{-1} HCl with 10^{-4} Cu^{2+} (A) and (B) a 1 mol L^{-1} solution of HCl is given below. Answer the following questions using the given polarogram.



Write down the approximate values of

(i). maximum current, $(i_d)_{max}$ (ii) diffusion current, (i_d)

(iii) half- wave potential.

(15 marks)

3.2.2 A polarogram of 3.00 mL of solution containing the antibiotic tetracycline in 0.1 M acetate at pH 4, gives a maximum current of 152 nA at a potential of –1.05 V (versus Standard Calomel Electrode- S.C.E.). When 0.50 mL containing 2.65 mg/mL of tetracycline was added into the mixture, the current increased to 206 nA. Calculate the concentration of tetracycline in the original solution.

(25 marks)

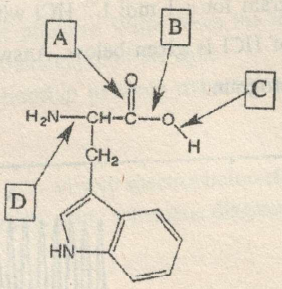
3.3. Compare and contrast nephelometry and turbidimetry used in pharmaceutical analysis.

(20 marks)

04. Answer **all** parts

4.1

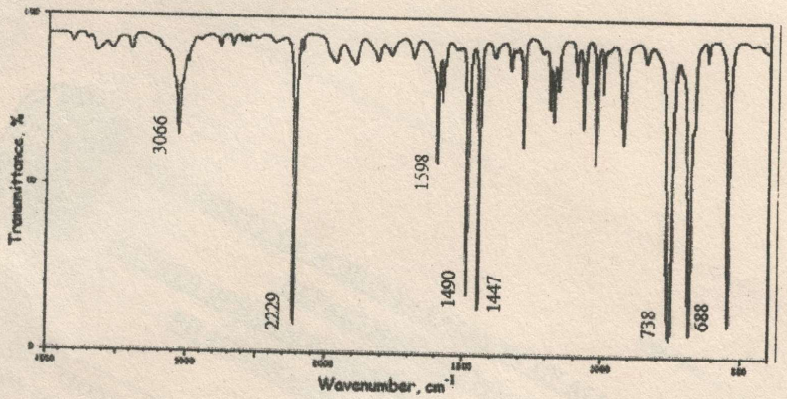
4.1.1 Tryptophan is an essential amino acid that is required for the production of serotonin. Consider the four bonds indicated by arrows labeled A, B, C, and D.



Giving reason briefly, arrange the bonds A, B, C and D, in order of increasing fundamental IR stretching frequency.

(20 marks)

4.1.2 The molecule with molecular formula C_7H_5N gives following IR spectrum. Propose a reasonable structure for the molecule. Assign indicated wavenumbers to the appropriate bonds.



(30 marks)

4.2 Answer all parts.

4.2.1 Define the following terms used in NMR spectroscopy.

(i) Chemical shift

(ii) Signal intensity

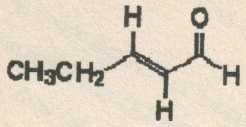
(10 marks)

4.2.2 Indicate the number of peaks and their multiplicity that you would expect in the ^{1}H NMR spectra of the following molecules.

(i) Ethylacetate (ii) 2-butanol (iii) 4-hydroxyaniline

(10 marks)

4.2.3. The ^{1}H NMR spectrum of (E)-2-pentenal has five distinct NMR signals at chemical shifts (δ) of: 9.5, 6.9, 6.0, 2.2, and 1.0.



(i) Derive the multiplicity of each NMR signal, and assign them into appropriate H atoms in the given molecule. ***(20 marks)***

(ii) Draw a suitable stick diagram to illustrate the splitting pattern and the intensity ratio of the ^{1}H NMR signal of the above molecule appears at 1.0 ppm. ***(10 marks)***

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