



UNIVERSITY OF RUHUNA – FACULTY OF ALLIED HEALTH SCIENCES

DEPARTMENT OF PHARMACY

FOURTH BPHARM PART I EXAMINATION – OCTOBER 2019

PH 4123 PHARMACEUTICAL ANALYSIS (SEQ)

TIME: TWO HOURS

INSTRUCTIONS

- There are four (04) questions in parts A,B,C,D,& E of SEQ paper.
- Answer each part in a separate booklet.
- No paper should be removed from the examination hall.
- Do not use any correction fluid.
- Use illustrations where necessary.

PART A

01. Answer **all** parts

1.1.

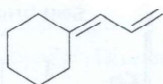
1.1.1. State the Beer-Lambert Law. (10 marks)

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

1.1.3. A  $4.50 \times 10^{-5}$  M solution of copper sulphate has a transmittance of 36.4% when measured in a 1.02 cm cell at a wavelength of 520 nm. Calculate the absorbance of this solution and the molar absorptivity ( $\epsilon$ ) of  $\text{CuSO}_4$ . (10 marks)

1.2. Using Woodward-Fieser rules, calculate the  $\lambda_{\text{max}}$  for the following compounds.

1.2.1.



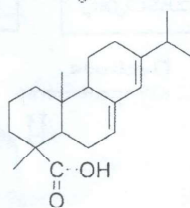
(10 marks)

1.2.2.



(10 marks)

1.2.3.



(10 marks)

1.3.

1.3.1. State the principle of atomic absorption spectroscopy (AAs). (05 marks)

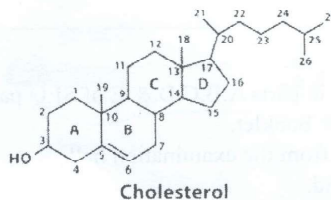
1.3.2. Describe briefly the function of each main part of the atomic absorption spectrophotometer. (30 marks)

1.3.3. What is meant by “atomization” in the atomic absorption spectrophotometry. (05 marks)

PART B

02. Answer all parts.

2.1 Polarimetry is a technique where a sample with optical activity rotates the plane of polarization of a polarized light beam passing through it. Chemical structure of the cholesterol molecule that possesses the same activity is given below.



2.1.1 Write down the numbers assigned to all chiral centers of the molecule. (15 marks)

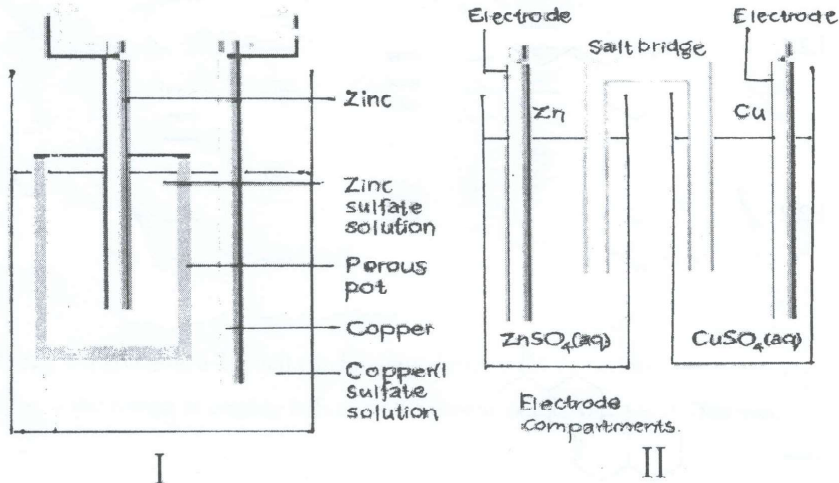
2.1.2 In a polarimetric experiment, it was found that for cholesterol,  $[\alpha]_D^{23} = -32.0^\circ$ . Define the notation. (15 marks)

2.1.3 A mixture of (+) and (-) cholesterol was analyzed by polarimetry and the observed rotation was  $16^\circ$ . What enantiomer is in excess in this mixture S or R? Calculate the percent composition of the (-) isomer in this mixture. (20 marks)

2.2

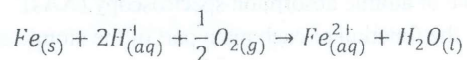
2.2.1 What is meant by liquid junction potential? (10 marks)

2.2.2 Give the cell notation of the following cells. (20 marks)

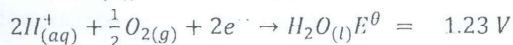
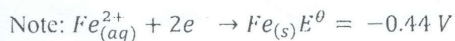


2.2.3 Write down the major difference exists between the above 2 cells shown in 2.2.2. (10 marks)

2.2.4 One of the reactions important for corrosion in an acidic environment is



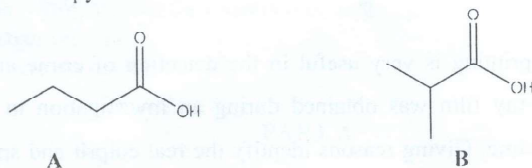
Show that the equilibrium constant of the above reaction favor formation of  $Fe^{2+}$ . (10 marks)



### PART C

03. Answer all parts.

- 3.1 Define the term chemical shift, and calculate the chemical shift for a  $^1\text{H}$  NMR signal appears at 514 Hz in a 300 Mz NMR spectrometer. (12 marks)
- 3.2 The chemical structures of *butyric acid* (A) and *isobutyric acid* (B), the isomers of  $\text{C}_4\text{H}_8\text{O}_2$  are shown below: Explain briefly how you would distinguish these isomers using only  $^1\text{H}$  NMR spectroscopy. (12 marks)



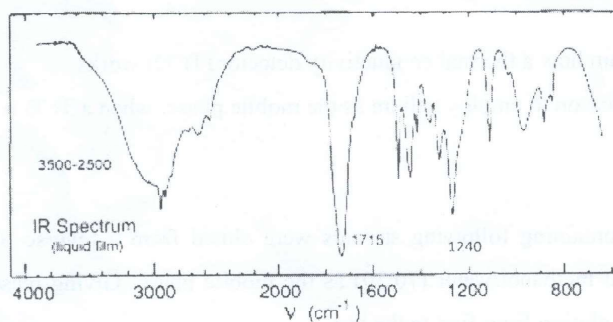
- 3.3 The  $^1\text{H}$  NMR spectrum of the compound X with the molecular formula of  $\text{C}_5\text{H}_{10}\text{O}$  shows NMR signals at:  $\delta$ 1: 0.95 (triplet, 3H),  $\delta$ 2: 1.62 (sextet, 2H),  $\delta$ 3: 2.13 (singlet, 3H) and  $\delta$ 4: 2.40 (triplet, 2H). The compound X gives an absorption band at  $1720\text{ cm}^{-1}$  in its IR spectrum. Giving reasons propose a plausible structure for compound X. (26 marks)

### Part D

- 3.4 List factors that determine the wavenumber of an infrared absorption and write down a mathematical expression for the Hook's law, which describes a vibrating frequency (wavenumber) of a covalent bond. (15 marks)
- 3.5 Which characteristic absorptions in IR spectra would allow you to distinguish between the following pair of alkynes? Give approximate frequencies for those absorptions. (10 marks)



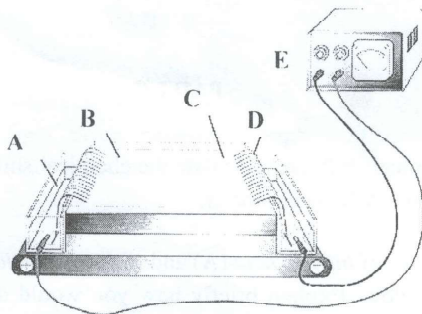
- 3.6 A molecule with molecular formula  $\text{C}_3\text{H}_6\text{O}_2$  gives the following IR spectrum. Propose a reasonable structure for the molecule. Assign indicated wavenumbers to the appropriate bonds. (25 marks)



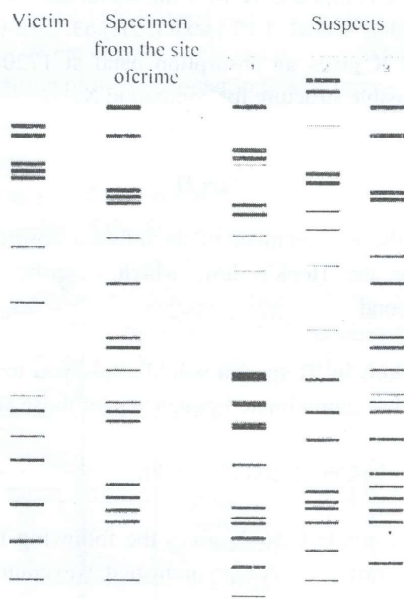
### Part E

04. Answer all parts.

- 4.1 Electrophoresis is a class of separation techniques in which analytes could be separated by the ability to move through a conductive medium.
- 4.1.1 Typical electrophoresis apparatus is shown bellow. Identify the parts labeled as A-E. (15 marks)

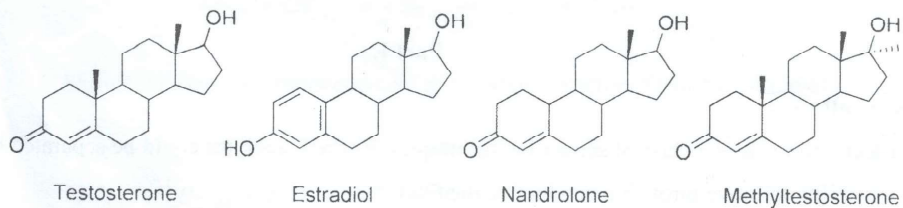


4.1.2 DNA finger printing is very useful in the detection of crime and legal pursuits. Following X-ray film was obtained during an investigation to identify the real culprit of a crime. Giving reasons identify the real culprit and specify the reasons to reject the other culprits. **(10 marks)**



4.2  
 4.2.1 Briefly explain how a thermal conductivity detector (TCD) works. **(30 marks)**  
 4.2.2 What is the reason to employ helium as the mobile phase, when a TCD is used? **(05 marks)**

4.3  
 4.3.1 A sample containing following steroids were eluted from a reverse phase HPLC column with methanol/water (70:30) as the mobile phase. Giving reasons, predict the order of elution from first to the last. **(30 marks)**



4.3.2 If the above four steroids were present in same concentration, predict the HPLC chromatogram. **(10 marks)**