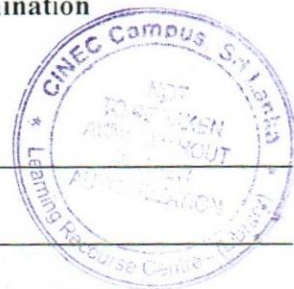


Faculty of Health Sciences
Bachelor of Science Honours in Industrial Pharmaceutical Science
IPS 3153 – Advanced Analytical Techniques
Batch – 05-3rd year 1st semester-End Semester SEQ Examination

INDEX NUMBER:

Date : 22nd November 2023
Time : 09.00 a.m. – 12.00 p.m. (Three hours)


INSTRUCTIONS TO CANDIDATES

- This question paper consists of **SIX** questions.
- Answer **ALL** questions.
- You should write legibly in black or blue ink.
- You are allowed to use non-programmable calculators.

Question 01 **(100 marks)**

- 1.1. What is the principle of gravimetric analysis? (10 marks)
- 1.2. What are the requirements for accurate gravimetric analysis? (20 marks)
- 1.3. Define volatilization gravimetric analysis? (10 marks)
- 1.4. An excess of aqueous sodium phosphate is added to a 50 mL sample of copper (ii) chloride, with unknown concentration. A precipitate form, as shown in the molecular equation below.



A student purifies, dries, and weighs the solid precipitate. She weighs the solids and finds she has collected 18 g of $\text{Cu}_3(\text{PO}_4)_2$. Based on this data, determine the molarity of CuCl_2 solution. (30 marks)

(Cu = 63.546 g/mol, Na= 23 g/mol, P =30.9 g/mol, O = 16 g/mol, cl= 35.5 g/mol)

- 1.5. Determine the percentages of Na and K in a 0.6128 g sample containing NaCl and KCl treated with AgNO_3 yielded 1.039 g of dried AgCl. (30 marks)

(The molecular weight of AgCl, NaCl and KCl are 143.32 g/mol, 58.44 g/mol and 74.55 g/mol, respectively.)

Question 02 **(100 marks)**

- 2.1. State **04** characteristic properties in an ideal standard solution. (20 marks)
- 2.2. Briefly describe the **02** types of standard solutions used for analytical standardization. (20 marks)
- 2.3. Classify the non-aqueous solutions and provide one example per each. (20 marks)
- 2.4. In precipitation titrations, Mohr method determines the chloride ion concentration of a solution by titration with silver nitrate solution.
- 2.4.1. State the chemical indicator used in this titration method. (05 marks)
- 2.4.2. Briefly describe the method of determining the end point of this titration method by indicating the indicator reaction at the end point. (20 marks)

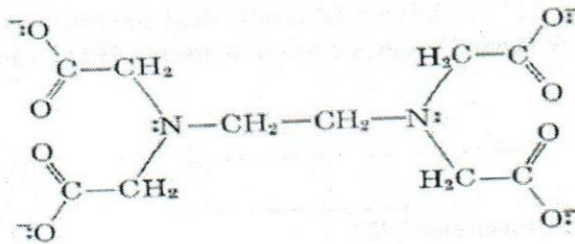
2.4.3. The pH of the sample solutions should be between 6.5 and 10 during this titration method. Briefly explain the reason. (15 marks)

Question 03

(100 marks)

3.1. Briefly describe the formation of coordination compounds in analytical chemistry. (20 marks)

3.2.

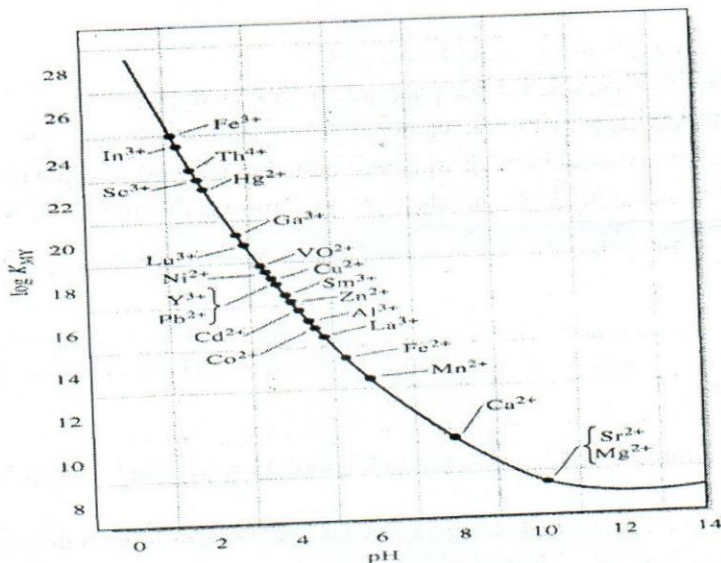


3.2.1. Identify the above-mentioned ligand. (05 marks)

3.2.2. State the number of donor groups provided by the ligand mentioned in 3.2.1. (05 marks)

3.2.3. Briefly explain how to consider the titration between Fe^{3+} ion only with the above-mentioned ligand when there is a mixture of ions including Fe^{3+} , Mg^{2+} , Al^{3+} and Ca^{2+} . (20 marks)

(Note: Consider the below mentioned pH vs cation concentration graph to answer the question).



3.3. Calculate pCa ($-\log[\text{Ca}^{2+}]$) in 100 mL of a solution of 0.100 M Ca^{2+} at pH 10 after addition of 0 mL, 50 mL, 100 mL, and 150 mL of 0.100 M of the ligand Y^{4-} . (50 marks)

(Note: The formation constant (K_f) for CaY^{2-} is 5.0×10^{10} and $\alpha_{\text{Y}^{4-}}$ at pH 10 is 0.35).

Question 04

(100 marks)

4.1. List 03 differences between Column chromatography and Thin Layer Chromatography (TLC). (20 marks)

4.2. Describe the main steps of TLC technique. (30 marks)

4.3. Write short notes on Sodium Dodecyl Sulphate Polyacrylamide gel electrophoresis (SDS-PAGE). (20 marks)

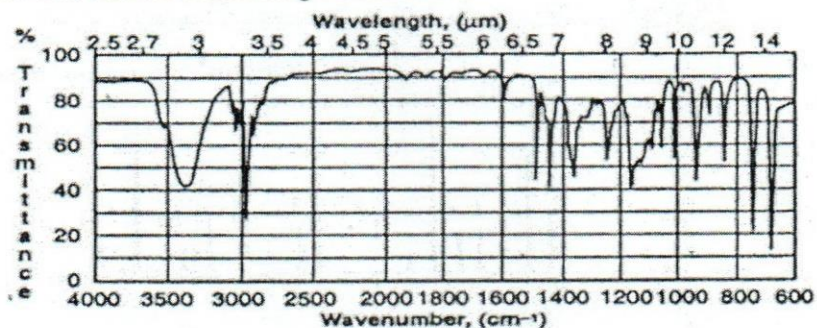
4.4. Briefly describe factors affecting gel electrophoresis. (30 marks)

Question 05**(100 marks)**

5.1. State 04 methods of instrumental methods of structure determination of molecules.

(10 marks)

5.2. You have provided an IR spectrum of molecule ($C_8H_{10}O$). It was reported that the molecule can't be oxidized using chromic acid.



5.2.1. Find the degree of unsaturation of the molecule.

(20 marks)

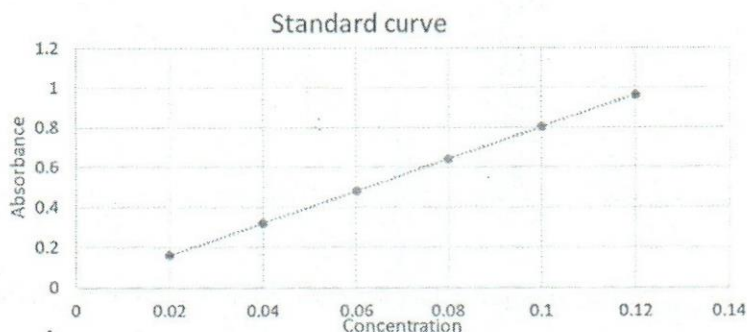
5.2.2. Which functional group do you assume for the peak between 3500 cm^{-1} and 3000 cm^{-1} ?

(10 marks)

5.2.3. According HNMR and C^{13} NMR spectrum obtained, it was determined that the molecule is mono substituted product of benzene. R- C_2H_7O . (R is the attached benzene group). Suggest the arrangement of molecule, C_2H_7O .

(20 marks)

5.3. You have provided a standard curve plot of concentration (mg/mL) vs. UV/visible absorbance of metformin hydrochloride. The average weight of a metformin 500 mg tablet are 650 mg. 20 tablets were crushed, and 100 mg of equivalent metformin was dissolved in 100 mL. The 10 mL of the solution was taken from that solution and diluted up to 100 mL. The absorption was then taken.



5.3.1. Predict the concentration of the solution when the absorption is 0.7.

(10 marks)

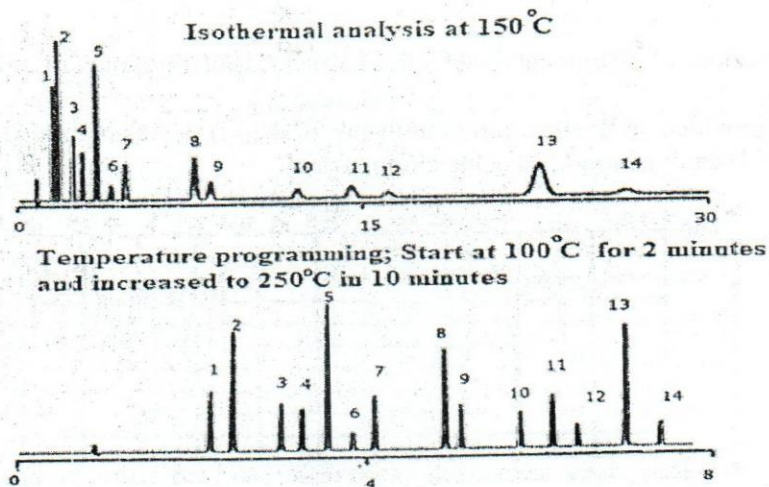
5.3.2. Calculate molar extinction coefficient of metformin chloride solution.

(10 marks)

5.3.3. Calculate the assay percentage of metformin tablet.

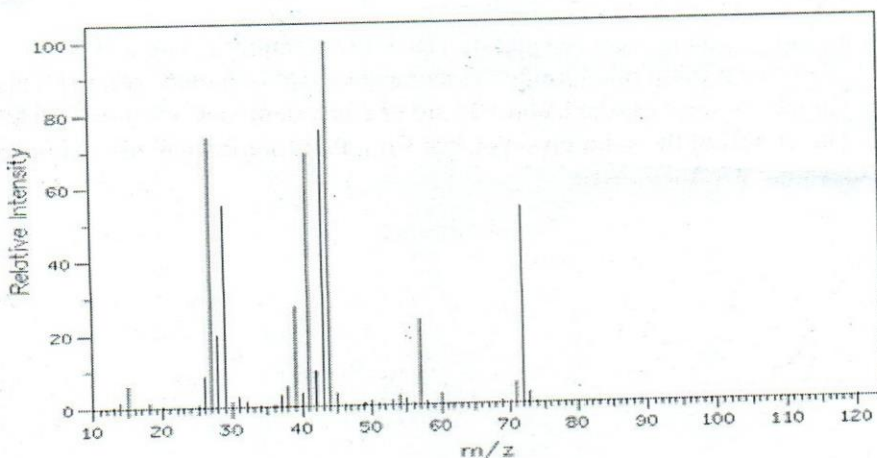
(20 marks)**Question 06****(100 marks)**

6.1. The following two chromatographs are for the GC separation of a mixture of compounds under isothermal conditions and under temperature programming using He as the carrier gas at its optimum flow rate on a capillary column. The GC operating temperatures and the identity of the individual signals are labelled in each chromatogram.



- 6.1.1. Briefly explain the role of carrier gas and makeup gas in GC separation and account for the suitability of He as a carrier gas. (15 marks)
- 6.1.2. Giving reasons explain the differences in the retention times and resolution for the first two and the last two signals in the two chromatograms given above. (40 marks)

6.2. The mass spectrum of butanal is given below.



- 6.2.1. Provide the structure for the molecular ion that would result in the m/z of 72 peak. (10 marks)
- 6.2.2. Based on fragmentation pattern, predict the mode of ionization that has been used. (10 marks)

6.3. Karl Fisher volumetric titration was carried out to determine the water content in Lidocaine HCL.

- 6.3.1. Briefly explain the difference between Volumetric Karl Fisher and Coulometric Karl Fisher titration. (10 marks)
- 6.3.2. Given that the 0.0244 g of water is equivalent to KFR 5.249 mL and 0.1564 g of Lidocaine HCL is equivalent to KFR 2.094 ml. Find the water percentage of Lidocaine HCL. (15 marks)