

THE OPEN UNIVERSITY OF SRI LANKA
B.Sc. Degree Programme
Level 5 –CYU 5302–ANALYTICAL CHEMISTRY
FINAL EXAMINATION PAPER 2025/26



Date: 2025.11.29

Duration: Two hours

Time: 9.30 p.m.- 11.30 a.m.

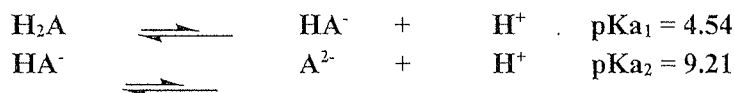
Instructions to students

Answer all four questions.

1.A) The purity of the salt $\text{-CuSO}_4 \cdot 5\text{H}_2\text{O}$, of a sample of 0.3695 g was tested by dissolving in 50 mL of water; added few drops of HCl followed by freshly prepared sulfurous acid; boiled and added slightly excess amount of freshly prepared thiocyanate solution. The precipitate formed, CuSCN was allowed to stand for 2 hrs.; filtered using a sintered glass crucible; washed with cold water followed by 10% thiocyanate mixed with a few drops of sulfurous acid; washed with 20% ethanol; dried at 120°C in an oven.

- (i) What may be the reasons for
 - (a) adding sulfurous acid?
 - (b) adding thiocyanate solution slightly in excess but not in excess?
 - (c) allowing to stand for 2 hrs.?
 - (d) washing with ethanol? (20 marks)
- (ii) How would you advice to add thiocyanate solution? Why? (20 marks)
- (iii) Comment on the following sentence.
 “Analysis of copper gravimetrically as CuSCN is very much suitable since unlike copper, thiocyanates of most metal ions are soluble.” (10 marks)
- (iv) The weight of the precipitate obtained was 0.1560 g. Calculate the percentage of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in the salt sample. ($\text{Cu} = 63.546 \text{ g}$, $\text{CuSCN} = 121.62 \text{ g}$, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O} = 249.68 \text{ g}$) (10 marks)

B) To prepare a buffer of pH 5.5 the weak acid H_2A was selected. H_2A dissociates as follows.



- (i) A 20.0 mL of H_2A was titrated with 0.1 M NaOH to find the concentration of H_2A . Sketch and label the titration curve. Explain the shape of the curve. (15 marks)
- (ii) When 5.0 ml of 0.1 M NaOH was added to the H_2A during the titration the pH was 4.54. Calculate the concentration of H_2A . (15 marks)
- (iii) Calculate the weight of the salt NaHA that should be added to 100.0 mL of above H_2A solution to make a buffer of pH 5.5. ($\text{NaHA} = 86.000 \text{ g mol}^{-1}$) (10 marks)

2. A) In order to determine the concentration of X^{2+} in a solution, to a sample solution (15.0 mL) Of the metal ion X^{2+} , 10.00 mL of 0.50 M EDTA (excess) was added and the pH was adjusted. The excess EDTA was titrated with 0.02 M Z^+ solution. (Equilibrium constants: $K_{XY} = 2 \times 10^{20}$, $K_{ZY} = 2 \times 10^{16}$)

- (i) Give possible reasons for not titrating X^{2+} directly with EDTA. (8 marks)
 (ii) Considering the information given below, what is the most suitable pH for the reaction between X^{2+} and EDTA? Justify your answer. (10 marks)

pH	$\alpha_{Y^{4-}}$	$\alpha_{M^{2+}}$
4.00	3×10^{-9}	1
1.00	5×10^{-18}	1

- (iii) If the end point was 20.00 mL, calculate the concentration of X^{2+} in the solution. (12 marks)
 (iv) Calculate the pZ^+ in equilibrium of the solution in the titration flask at the end point. (20 marks)
 (v) Comment on the following sentence.
 "Most metal-EDTA complexes becomes unstable at very high pH." (10 marks)

- B) To determine the sulfite content in a sample of 25.0 mL of fruit juice, KIO_3 and KI added and acidified the solution to generate 3×10^{-3} mols of I_2 which is in excess (KIO_3 reacts with KI in acid medium to produce I_2). The I_2 produced will be reacting with sulfite ions to produce sulfate ions and iodide ions. The excess I_2 was titrated with 0.2 M $Na_2S_2O_3$ solution and the end point was 25.0 mL.



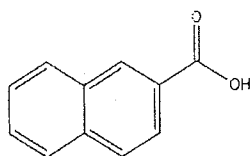
- (i) Write half-cell reactions and the balanced equation for the reaction of I_2 with sulfite ions. (12 marks)
 (ii) Calculate the concentration of sulfite ion in the sample solution. (10 marks)
 (iii) Comment on the following sentence.
 "Errors of iodometric titrations is reduced in the presence of excess I^- ions." (8 marks)
 (iv) Draw and label the expected titration curve for the titration. (10 marks)

3. A) The compound Y weighing 1.200 g is dissolved in 25.00 mL of water. The solution is extracted using a separatory funnel with 50.00 mL of diethyl ether. After separation and evaporation of the ether layer, 1.120 g of compound Y is recovered from the organic layer. The molecular weight of compound Y is 280 g mol^{-1} .

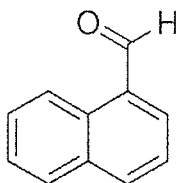
- (i) Calculate the distribution coefficient (K_d) of compound Y between ether and water. (10 marks)

- (ii) If the same extraction is repeated twice using two separate 25.00 mL portions of ether (instead of one 50.00 mL portion), calculate the total mass of compound Y that would be extracted into the ether layers. (14 marks)
- (iii) State two factors that affect the value of the distribution co-efficient. (6 marks)

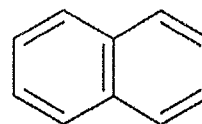
B) A mixture of three polycyclic aromatic carbons A, B, C is analyzed using silica gel thin layer chromatography (TLC). The solvent system used is hexane: chloroform in a 9:1 ratio.



A



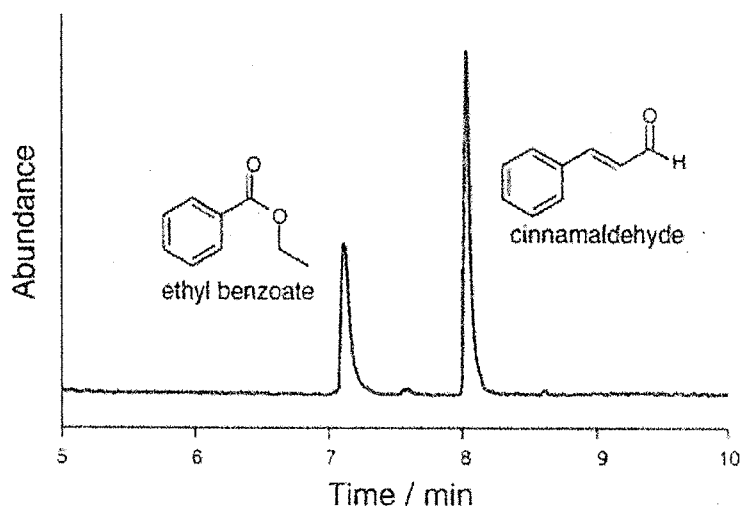
B



C

- (i) Predict the order of compounds A, B, and C on the TLC plate in terms of increasing R_f values. Justify your answer. (15 marks)
- (ii) It is observed that the distance travelled by the three compounds is too small, resulting in poor separation. Suggest one method to improve the separation. (10 marks)
- (iii) "TLC can be used to determine the purity of a compound", Justify this statement with appropriate reasoning. (10 marks)

C) An unknown sample containing two compounds, ethyl benzoate and cinnamaldehyde, was analyzed using high performance liquid chromatography (HPLC) with isocratic elution. The chromatogram showed two well-separated peaks as shown below:

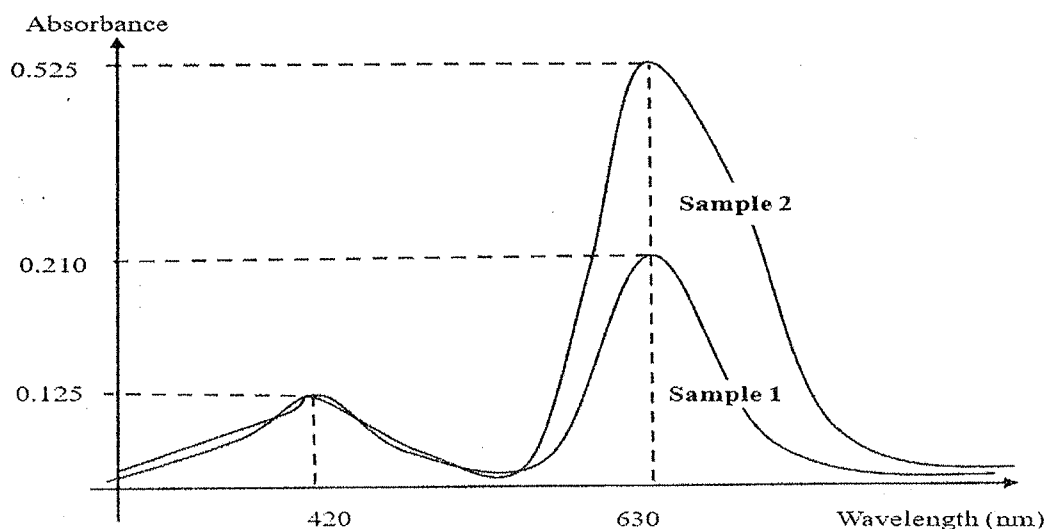


A second chromatogram was obtained from a 5.000 ppm standard solution of cinnamaldehyde, and the peak height was twice as large as that of cinnamaldehyde in the unknown sample, at the same retention time. The dead time (t_m) of the column is known to be 0.4 minutes.

- (i) The experiment was performed using normal phase chromatography. Justify this statement. (10 marks)
- (ii) Calculate the retention factor (k) for cinnamaldehyde. (08 marks)

- (iii) Determine the concentration of cinnamaldehyde in the unknown sample mixture. (07 marks)
- (iv) Briefly explain the following. (10 marks)
- (a) Isocratic elution (b) Chromatogram

- 4.A) Copper ion concentration in an effluent of a brass industry was determined using a double beam UV/visible Spectrophotometer. Two samples were prepared as follows:
 Sample 1: 10.00 mL of effluent diluted to 100.0 mL with distilled water.
 Sample 2: 10.00 mL of effluent mixed with 10.0 mL of 5.00 ppm Cu^{2+} standard solution, then diluted to 100.0 mL. An absorbance versus wavelength graph was recorded for both samples.



- (i) Identify the most suitable wavelength for measuring the absorbance of copper ions and justify your answer. (10 marks)
- (ii) Explain the purpose of preparing Sample 2 in this analysis. (10 marks)
- (iii) Calculate the concentration of copper ions in the effluent in ppm? (14 marks)
- (iv) Briefly explain the advantages of using a double- beam spectrophotometer. (06 marks)
- B) Another student proposed spectrophotometric titration to determine $[\text{Cu}^{2+}]$ in a solution. In this experiment, 10.00 mL of sample was diluted to 100.0 mL and titrated with 0.1 mM of EDTA. The equivalence point was reached at a burette reading of 4.00 mL, corresponding to an absorbance of 0.080.
- (i) Briefly describe the expected shape of the titration curve for this experiment. (10 marks)
- (ii) Calculate the concentration of Cu^{2+} in the original water sample in ppm. (15 marks)
- (iii) The addition of EDTA increases the total volume, which affects the absorbance reading. Suggest a method to correct this effect when plotting the titration curve. (5 marks)

- C) A 25.00 mL of 0.01 M NH_4OH was titrated with 0.01 M H_2SO_4 and conductivity reading were recorded after each 2.50 mL addition of H_2SO_4 up to total volume of 25.00 mL.
- (i) Sketch the titration curve for this titration. Clearly indicate the endpoint on your graph. (14 marks)
- (ii) Could this titration be performed same way using a pH meter instead of a conductivity meter? Justify your answer. (8 marks)
- (iii) Explain why the indicator method might be more suitable than the potentiometric method for this titration. (8 marks)