THE OPEN UNIVERSITY OF SRI LANKA
B.Sc. Degree Programme
Level 5 –CYU 5302–ANALYTICAL CHEMISTRY
FINAL EXAMINATION PAPER 2021/22

Date: 2022.10.29 Duration: Two hours
Time: .1.30 p.m. – 3.30p.m.

Instructions to students

This question paper consists of five pages having four questions. Answer all four questions.

1. The effluent of a manufacturing company contains the cation X^+ which was decided to be analyzed using Gravimetry. Both precipitates – $X_2Y_4O_2$ and $X_2Y_2O_7$ satisfies the requirements of Gravimetric analysis. (X= 43.00g, Y= 96.50g, O= 16.00g)

   (i) Out of the two precipitates, formation of $X_2Y_4O_2$ has been selected. Was it a good decision? Give reasons for your answer with proper calculations. (15 marks)

   (ii) Briefly explain the meaning of the underlined sentence above. (10 marks)

   (iii) In the process of gravimetric analysis, 25.0 mL of the effluent sample was treated with excess of Y^{2-} solution and the resultant precipitate was ignited to get only the final product $X_2Y_4O_2$ selectively, of which the weight was 0.4350g. What is the $[X^+]$ in the effluent? (10 marks)

   (iv) As an alternative method, to a 10.0 mL sample solution, 50.0 mL of 0.02 M EDTA solution was added and the excess EDTA was determined by titrating with the metal ion A^{2+} (0.02M) at pH=9 (K_{AY}= 8.4 x 10^{22}, α_Y^{4-} = 0.70, K_{AY}= 4.2 x 10^{22}). The end point obtained was 12.50 mL with Eriochrome Black T as the indicator.

   (a) Were there any side reactions of the metal ion A^{2+}? Justify your answer with proper calculations. (15 marks)

   (b) Calculate the $[X^+]$ in the sample using the titration result. (15 marks)

   (c) Comment of the stability of the two EDTA complexes resulted. (05 marks)

   (d) The end point was not sharp. Suggest a method to improve the sharpness of the end point. Draw the titration curve to show the difference before and after improving. (20 marks)

   (e) Are you happy with the methods selected Titrimetry and Gravimetry for the objective stated? Give reasons for your answer. (10 marks)

2. (i) The following procedure was carried out for the analysis of total alkali (carbonate and hydroxide) in caustic soda.

Titration I-A 25.0 mL of commercial sodium hydroxide solution was titrated with 0.10 M HCl using Methyl Orange as the indicator. The end point obtained was 20.00 mL.

Titration II-Another 25.0 mL of the solution was warmed and 1% $BaCl_2$ was added slowly until no further precipitate was formed. The solution was cooled and titrated with 0.1 M HCl using phenolphthalein as the indicator. The end point obtained was 15.00 mL.

a) Calculate the concentration of sodium carbonate in the sample. (20 marks)

b) Sketch and label both titration curves in one graph. (15 marks)

c) To get a more accurate end point, the titration was modified and repeated. Here, after the precipitate was formed, a sufficient amount of 0.1 M HCl was added from the burette and the solution was heated again; cooled and continued the titration. Justify the modification. (15 marks)

(ii) A sample solution (25.0 mL) having equal amounts of D^+ and Q^{2+} was titrated with 0.01 M Y^- solution and the potential of the solution in the flask was 0.640 V after adding 30.00 mL from the burette.

$E^0 (D^{2+}/D^+) = 0.112$ V $\quad E^0 (Q^{3+}/Q^{2+}) = 0.640$ V $\quad E^0 (Y^-/Y^{2-}) = 1.556$ V

a) Draw and label the titration curve. Describe feasibility and the selectivity of the titration. (20 marks)

b) Calculate the concentrations of D^+ and Q^{3+}. (20 marks)

c) Explain the following action in brief.

"Powdered limestone is added to the surface water of lakes to increase the buffer capacity in water so that the effect of acid rain on the living species in lakes will be reduced." (10 marks)

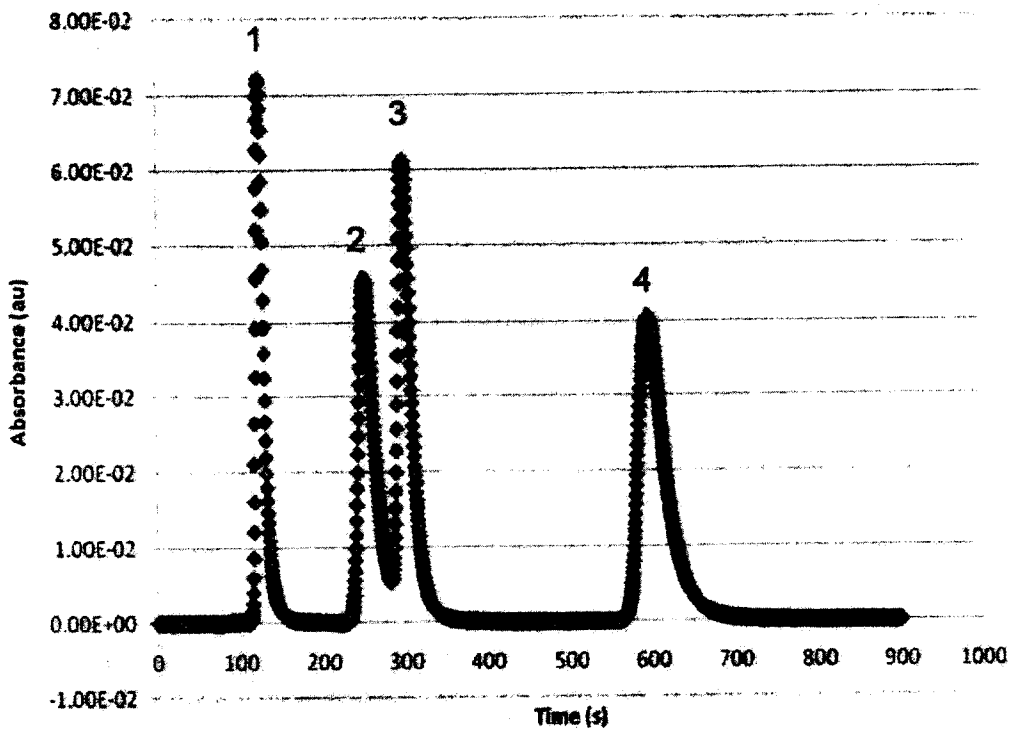
3. Use the following information to answer the questions (question number). The figure is a liquid chromatogram of the four deoxy mononucleotides.

Column length: 25.0 cm $\quad$ Column diameter: 0.46 cm

Stationary phase: C-18 $\quad$ Mobile phase: aqueous buffered at **pH** = 7.2

Flow rate: 1.2 mL/min

Dead time: 16.7 s



Physical Quantity	Component			
	1	2	3	4
Peak Height	0.072	0.046	0.061	0.041
Elution time (s)	120	255	310	608
Peak width at base	28	70	55	98

(i) What is the retention factor for component 4? (20 marks)

(ii) What is the efficiency of the column for component 4? (20 marks)

(iii) Assuming a value of H = 0.08 cm for each component, calculate the number of theoretical plates (N) of column. (20 marks)

(iv) Calculate the resolution between peaks 3 and 2? (20 marks)

(v) How can you increase the peak separation in HPLC? (20 marks)

4. During the Cu mining, diluted H_2SO_4 is used to separate the Cu from its ore. One student needed to determine the amount of Cu in a Cupper ore and optimize the amount of H_2SO_4. For this purpose, first he digested 0.2000 g of sample of Cu orc with 10.00 mL of 4 M H_2SO_4 and filtered the solution in to 100.0 mL of a volumetric flask and diluted up to the mark using distilled water. Thereafter he used two methods (A and B) to find out the purity of Cu orc and the minimum amount of H_2SO_4 required to digest the Cupper ore.

Method A: He transferred two 10.00 mL aliquots of prepared solution into two 50.0 mL volumetric flasks and added 0.01 $\mathbf{moldm^{-3}}$of 10.00 mL of standard $CuSO_4$ solution into one flask and diluted up to the mark using distilled water. The absorbance at 635 nm was 0.356 and 0.487 for the solutions without and with standard respectively. The absorption coefficient of $CuSO_4$ at 635 nm is 2.82 $\mathbf{dm^3mol^{-1}cm^{-1}}$. (Cu-63.5, S-32, O-16)

Method B: He transferred 10.00 mL of the previously prepared solution and carried out a conductometric titration using 4 $\mathbf{moldm^{-3}}$ of NaOH. He plotted a graph (conductance vs volume of 4 $\mathbf{moldm^{-3}}$ of NaOH) and marked the end point reading as 12.00 mL.

i) Find the moles of $CuSO_4$ present in the original solution after the separation of copper from its ore. (30 marks)

ii) Hence, determine the purity of Cu in its ore. (15 marks)

iii) What is the reason to add standard $CuSO_4$ solution to one flask in **method A**? (06 marks)

iv) Suggest a suitable method to improve the accuracy of the results obtained by the **method A**. (06 marks)

v) Determine the minimum amount of 4 M H_2SO_4 for the digestion of Cu ore. (20 marks)

vi) Are you satisfied with the accuracy obtained by the **Method B**? If not, how do you suggest to improve the accuracy? (08 marks)

vii) Sketch the titration curve related to the **method B**. (10 marks)

viii) Another student said "potentiometric titration can be used instead of the method B". Justify. (05 marks)