THE OPEN UNIVERSITY OF SRI LANKA
B.Sc. Degree Programme / Stand alone courses in Chemistry
Level 5 –Assignment Test 2 – 2010 / 2011

CHU 3129/CHE 5129 – INSTRUMENTAL METHODS IN CHEMICAL ANALYSIS

Duration: One and half hours
Date and time: 19th April, 2011 from 4.00 p.m. to 5.30 p.m.

Reg. No...

Question number	marks
1	
2	
3	
Total	

Instructions to students

Answer all questions in the spaces given. Additional sheets will not be marked.

1. (i) Give reasons for the following statements.

(a) In gas chromatography, the solvent in which the liquid sample is dissolved should not have a high retention time in the column.

(b) The affinity of Ca^{2+} is greater than that of Mg^{2+} for a cation exchanger column.

(c) Samples cannot be directly injected to the column in HPLC.

(ii) Give two major differences and similarities in Thin Layer Chromatography and Paper Chromatography.

Differences.

1.

2.

Similarities

1.

2.

(iii) Write a short account on "Eddy diffusion" in column chromatography.

(iv) Write equations using retention time for the terms "retention factor" and "selectivity factor" in chromatography (the symbols should be defined).

(40 marks)

2. A student was asked to determine the concentration of the metal ion M^{2+} in an aqueous solution using Coulometry.

$M^{2+}(aq) \quad + \quad 2e \quad \rightarrow \quad M(s) \qquad (0.39\ V)$

(i) What is the principle behind coulometry as a quantitative analytical method?

(ii) Draw and label a schematic diagram of the instrumentation of constant Potential coulometry.

(iii) What is the function of the reference electrode?

(iv) The student was advised to use a potential more than the theoretical cell potential. Why?

(v) What is the principle behind the voltametric methods as a quantitative analytical method?

(vi) Give one specific feature in the instrumentation of voltametric methods giving reasons.

(35 marks)

3. (i) The concentration of an unknown solution of Cu^{2+}(100 cm^3) was determined using polarography. The maximum limiting current was 855 mA while the residual current was 5 mA. A 50 ppm Cu^{2+}(100 cm^3) solution gave a limiting current of 805 mA and the residual current remained the same. What is the concentration of Cu^{2+} in the sample?

(ii) The same solution was subjected to electrogravimetry. Give three factors affecting the deposit.

(iii) Assume that Cd^{2+} ions are also in the solution.

$Cu^{2+}(aq) + 2e \rightarrow Cu(s) \qquad E^\circ = 0.34\ V$

$Cd^{2+}(aq) + 2e \rightarrow Cd(s) \qquad E^\circ = -0.40\ V$

(a) Can you determine the concentration of both Cu^{2+} and Cd^{2+} using electrogravimetry? Explain your answer.

(b) Write down the major steps in the analysis of two metal ions of a mixture using electrogravimetry.

(iv) Give **one major difference** and **one major similarity** in amperometric titrations and photometric titrations.

(25 marks)

Name ....
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Answer guide to Assignment test II

1.(i) (a) If the solvent does not have a high retention time the solvent peaks will not interfere with the sample peaks.

(b) Since charges are the same, the affinity depends on the size of the hydrated ion. Smaller the ion larger will be the size of the hydrated ion thus decreasing the affinity. Ca^{+2} is larger than Mg^{+2}. Hydrated ion of Ca^{+2} is smaller than Mg^{+2}. Therefore, the affinity of Ca^{+2} is greater than that of Mg^{+2}.

(c) Samples might contain particles which might damage the column. So the samples are passed through the guard column first. The column is run with a high pressure which should not be altered.

(ii) **Differences**

Thin layer Chromatography	Paper Chromatography
Principle behind the separation is Adsorption	Principle behind separation is partition
Stationary phase is silica	Stationary phase is water

Similarities

Both are used in qualitative analysis
Fast
Simple and inexpensive
A visualization technique is required

(iii) Eddy diffusion (A) is the difference in the rate of diffusion of the mobile phase resulted due to molecules having different pathways in the stationary phase.
Eddy diffusion α Diameter of particles
Eddy diffusion depends on the uniformity of packing of the stationary phase and the column geometry. When eddy diffusion increases height of the theoretical plates (H) also increases decreasing the efficiency of separation. Therefore, to reduce eddy diffusion (A), particles should be small.

(iv) Retention factor $(K'_A) = \dfrac{t_A - t_M}{t_M}$ Selectivity factor $(\alpha) = \dfrac{t_B - t_M}{t_A - t_M}$

t_A = Retention time of A
t_B = Retention time of B
t_M = Retention time of mobile phase

2. (i) The total charge α amount of analyte reacted or produced
(ii) Unit II Page 34 Figure 11.2
(iii) To keep the potential of the working electrode a constant.
(iv) To overcome the internal resistance and electrode polarization.

(v) Diffusion current α amount of analyte reduced

(vi) The working electrode has a very small surface area exposed to the solution (Micro electrode)

Reason :

The current flowing through the electrode is very small. Therefore, the analyte concentration will not change during the experiment.

3. (i) $i_D = i_L - i_R$

$i_{D1} = 855\text{-}5 = 850$ mA

$i_{D2} = 805\text{-}5 = 800$ mA

$i_D = k\ C$

$850 = k\ C$

$800 = k\ 50$

$C/\ 50 = 850\ /\ 800$

$C = 53.125$ ppm

(ii) Current density

Stirring

Temperature

Addion of complexing agents

(iii) Can.

Difference of the reduction potentials should be greater than 0.2 V

(iv) Keep the cathode potential constant at value needed to reduce one metal ion; dry and weigh the electrode after deposition; replace the cathode and keep the potential constant at value needed to reduce the other metal ion. Dry and weigh again. Take the difference in weight for the calculations.

(v) <u>Differences</u>

Amperometric titration	Photometric Titration
Measurement is current	Measurement is absorbance
Analyte (or related substance) should be oxidizing or reducing	Analyte,reagent or product should be able to absorb light

<u>Similarity</u>

Both are accurate, reproducible and sensitive