THE OPEN UNIVERSITY OF SRI LANKA
B.Sc. Degree Programme / Stand alone courses in Chemistry
Level 5 –Assignment Test 11 – 2012 / 2013

CMU 3128/CME 5128 – INSTRUMENTAL METHODS IN CHEMICAL ANALYSIS

Duration: One and half hours
Date and time: 09th September, 2013
4.00 p.m. to 5.30 p.m.

Reg. No.....

Question number	marks
1	
2	
3	
4	
Total	

Instructions to students

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

1. A) Explain the principle behind the following methods with respect to **quantitative analysis.**
   (i) Voltametry

   (ii) Coulometry

   B) Give **one advantages** and **one disadvantage** of Electrogravimetry compared to other electro analytical methods.

(20 marks)

2. A) Explain the **principle** behind the following methods with respect to **separation.**
   (i) Gel permeation chromatography

   (ii) Gas Liquid chromatography

B) (i) Give **two differences** of Thin Layer Chromatography and Paper chromatography.

(ii) Give **two major differences in the instrumentation** of High Performance Liquid Chromatography (HPLC) compared to simple liquid chromatographic techniques.

(20 marks)

3. The following data were obtained by gas chromatography in which the length of the column, the volume of the stationary phase and the volume of the mobile phase were 45 cm, 20.0 cm^3 and 60 cm^3 respectively. Dead time of the column was 1.2 minutes.

Compound	Retention time (minutes)
A	5.2
B	9.2
C	9.8

(i) What do you mean by the "**dead time**" of the column?

(ii) Calculate the **selectivity factor** for the two compounds, A and B.

(iii) If the width at the base of all the peaks is the same, what can you say about the **resolution** between the peaks? Suggest a method to increase the resolution between B and C using the same column.

(iv) Calculate the **distribution coefficient** for B.

(v) What would have been the **linear flow rate** of this column?

(35 marks)

4. A) **Draw and label** the following.

(i) A cell with three electrodes used in constant potential coulometry.

(ii) The titration curve of the amperometric titration of 25.0 cm^3 Pb^{2+} with a standard sulphate ion solution.

B) List **two advantages** of using dropping mercury electrode in polarography?

C) How do you identify the end point in potentiometric titrations?

(25 marks)

Name:.....

Address:.....

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CMU3128 – Instrumental Methods of Chemical Analysis
ANSWER GUIDE - CAT II

01. A) (i) $i_d \alpha C_A$
Limiting diffusion current (i_d) is proportional to the concentration f the analyte in the bulk (C_A)
(ii) Charge is proportional to the concentration of the analyte passed through a cell.

B) Advantage
No calibration is required.

Disadvantages
Low sensitivity, Low speed,Have to carry out processes like drying, purifying etc.....

02. A) (i) Size of the molecule
(ii) Partition

B) (i)

TLC	Paper chromatography
Principle – adsorption	Principle – partition
Stationary phase – solid	Stationary phase - liquid

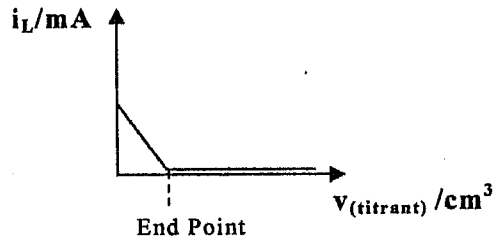
(ii) In HPLC
- Mobile phase (liquid) is delivered with pressure.
- Sample is introduced by a special valve.
- Small, pre-packed & re-usable column.
- The element is passed through a detector.
- High performance, better separation, higher reproducibility.

03. (i) The time taken for the unretained components/solvent to reach the detector.
(ii) Selectivity factor (α) = $t_b - t_m / t_a - t_m$ = $(9.2 - 1.2) / (5.2 - 1.2) = 2$
(iii) A & B – high resolution B & C – poor resolution
Suggestion - Change the mobile phase / solvent
Change the temperature

(iv) $k'_B = (K_B V_S) / V_M = (t_B - t_M) / t_M$
K_B ($20cm^3/60cm^3$) = (9.2 – 1.2)min / 1.2 min
$K_B = 20$

(v) $u = L / t_M$ = 45cm / 1.2 min = $37.5 cm min^{-1}$

04. A)(i) Page 20 Figure 2.2
(ii) Only the analyte is reduced.



B) Advantages of D.M.P –Pg 35
C) It is the volume at which the drastic change (or average of the drastic change) of potential takes place.