



THE OPEN UNIVERSITY OF SRI LANKA  
B.Sc. Degree Programme / Stand alone courses in Chemistry  
Level 5 –Assignment Test II – 2011 / 2012

CMU 3128/CME 5128 – INSTRUMENTAL METHODS IN CHEMICAL ANALYSIS

Duration: One and half hours

Date and time: 29<sup>th</sup> March, 2012  
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. Briefly explain the following.

(i) Electrode polarization and over voltage.

(ii) Reverse phase Thin Layer Chromatography.

(iii) Although both gel electrophoresis and Ion exchange chromatography can be used to separate charged particles, the mechanism of separation of the two techniques is different.

(iv) When Polarography is carried out, the analyte solution should not be stirred.

(v) When Gas Chromatography is used for quantitative analysis, it is not the direct response that is taken into consideration but the relative response.

(30 marks)

2. (i) What is the principle behind Coulometric analysis?

(ii) Write two differences of **Potentiometric** titrations and **Amperometric** titrations.

(iii) A 500 mA current was passed through a 500.0 cm<sup>3</sup> solution of Cu<sup>2+</sup> ions for 10.00 minutes. Calculate the amount of moles of copper deposited. Give your answer to the correct number of significant figures. (Faraday's constant = 96500 C mol<sup>-1</sup>)

(iv) A sample solution having a mixture of X<sup>+</sup> and Y<sup>+</sup> (equal concentrations) was analyzed using normal voltametry. Nitrogen gas was bubbled through the solution before starting the analysis.

$E_{1/2}$  of X<sup>+</sup> = -0.35 V,  $E_{1/2}$  of Y<sup>+</sup> = -0.78 V.

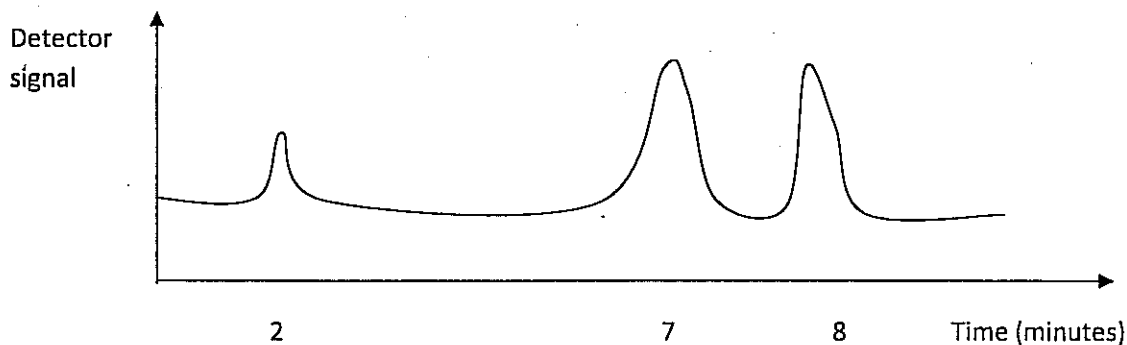
(a) What is the reason for carrying out the action underlined?

(b) Sketch the voltogram resulted if the above analysis was carried out using normal voltametry.

(c) Sketch the voltogram resulted if the above analysis was carried out using Anodic stripping voltametry.

(45marks)

3. The figure below is the chromatogram resulted when the compound A of a sample was separated from the compound B using a column of liquid – liquid chromatography. The time taken for the mobile phase, A and B for elution is 2 minutes, 7 minutes and 8 minutes respectively.



(i) What was dead time of the column?

(ii) If the column length is 20 cm, calculate the average linear rate of the compound A.

(iii) If this column was a normal phase column, what is more polar- A or B? Give reasons for your answer.

- (iv) Calculate the selectivity factor for the compounds A and B and the retention factor for the compound A.
  
- (v) Suggest one way of increasing the separation of the compounds A and B.
  
- (vi) The peak area for the compound A and B was 524 and 302 (arbitrary units) respectively. Calculate the percentage of B in the sample.
  
- (vii) What was the underline force for separation here?
  
- (viii) If this separation was done using HPLC (High Performance Liquid Chromatography) give a major difference that you are expecting in the chromatogram compared to the above chromatogram.

(25 marks)

Name:.....

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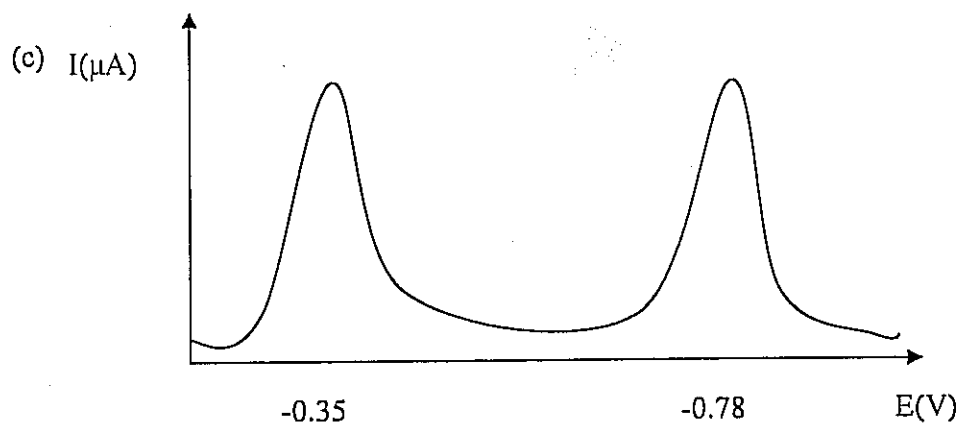
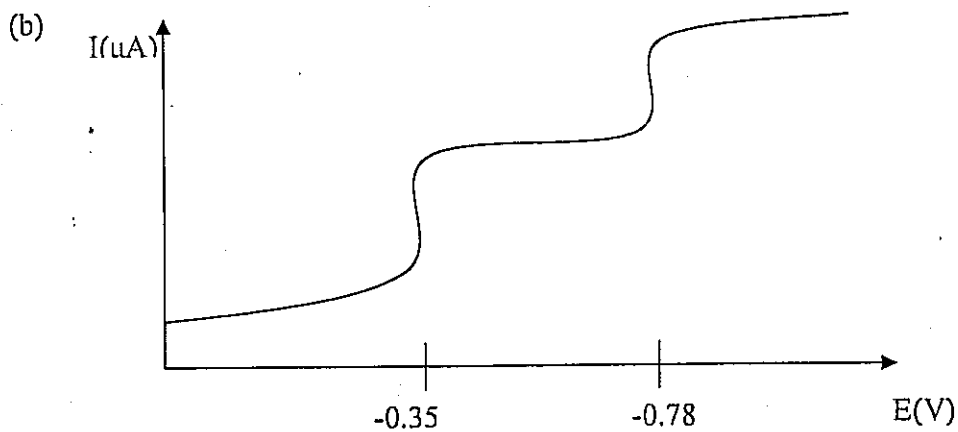
**CMU 3128/CME 5128 – INSTRUMENTAL METHODS IN CHEMICAL  
ANALYSIS  
ASSIGNMENT TEST II – 2011/2012 - ANSWER GUID**

1. (i) Electrode polarization - When external voltage increases, current doesn't increase due to various limiting factors.  
Over voltage – External voltage required to overcome electrode polarization
  - (ii) TLC – A planar chromatographic method; stationary phase may be solid or liquid and mobile phase is a liquid; principle of separation is either adsorption or partition. In reverse phase, stationary phase is non-polar compared to the mobile phase.
  - (iii) In electrophoresis ions migrate towards the opposite charge electrode according to their charge/size ratio; there is no mobile phase and no exchange of ions takes place here. In ion exchange chromatography, separation takes place because of exchange of ions between the two phases. The exchange of ions depends on the charge/size ratio.
  - (iv) In polarography, the diffusion current is measured and stirring will result a current due to migration and it should be avoided.
  - (v) Detectors don't respond in the same way to each and every compound. Therefore, we need to calculate a response relative to another compound which has been run in the same instrument.
2. (i) Coulometry is used for quantitative analysis. The concentration of the analyte is proportional to the charge (Q). Charge is proportional to current making the concentration proportional to the current. Therefore, here we measure the current which is proportional to the analyte concentration.
  - (ii)

Potentiometric titration	Amperometric titration
Measure the potential (voltage) Titration curve is of potential vs volume added from the burette and the curve is 'S' shaped. The end point is the mid point of the vertical portion in the graph. Require large number of readings. More time consuming.	Measure the current Titration curve is of current vs volume added from the burette and the graph is not 'S' shaped but two straight lines crossing each other. The end point lies at the intersection of two straight lines in the graph. Do not require large number of readings. Less time taken.

- (iii) 
$$N = \frac{Q}{nF} = \frac{It}{nF} = \frac{(500.0 \times 10^{-3} \text{ A}) \times (600.00 \text{ s})}{(2 \times 96500 \text{ C mol}^{-1})}$$

$$= 1.554 \times 10^{-3} \text{ mol (four significant figures)}$$
- (iv) (a) Nitrogen gas has to be bubbled to flush out O<sub>2</sub> gas.



2. (i) Dead time = 2 min.
- (ii)  $U = L/t_A = 20 \text{ cm} / 7 \text{ min} = \underline{2.86 \text{ cm min}^{-1}}$
- (iii) B is polar than A. In normal phase, stationary phase is polar and mobile phase is non-polar. The polar compound retains more in the stationary phase and therefore takes more time to elute.
- (iv) Selectivity factor ( $\alpha$ ) =  $(t_B - t_M) / (t_A - t_M) = (8 - 2) / (7 - 2) = \underline{1.2}$   
Retention factor ( $K_A'$ ) =  $(t_A - t_M) / t_M = 5 / 2 = \underline{2.5}$
- (v) Can do one of the following.  
1. Change the polarity of the mobile phase 2. Reduce the particle size of the stationary Phase 3. Increase the column length 4. Optimize the flow rate 5. Efficient packing .
- (vi)  $\% B = 302 / (524 + 302) \times 100 = \underline{36.56 \%}$
- (vii) Partition
- (viii) Well separated peaks (high resolution) with low band broadening will be resulted.