THE OPEN UNIVERSITY OF SRI LANKA
B.Sc. Degree Programme / Stand alone courses in Chemistry
Level 5 –Assignment Test 1 – 2014 / 2015

CMU 3128/CME 5128 – INSTRUMENTAL METHODS IN CHEMICAL ANALYSIS

Duration: One hour
Date and time: 01st August, 2015
2.30 p.m. to 3.30 p.m.

Reg. No.....

Question number	marks
1	
2	
Total	

Instructions to students

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

1. (i) Explain the principles behind Raman spectroscopy as a qualitative analytical method. (12 marks)

(ii) State two main differences in the instrumentation of a fluorimeter compared to UV/Visible spectrophotometer. (10 marks)

(iii) Sketch and label the expected photometric titration curve of a titration in which only the product absorbs light (assume that a micro burette is used). (10 marks)

(iv) Explain the following in brief. (18 marks)

(a) Singlet excited state.

(b) Molecular ion peak in a Mass spectrum.

(c) Disadvantages of using flame as the energy source for atomization compared to plasmas in Emission Spectroscopy.

2. (i) What is the principle behind Coulometry as a quantitative method of analysis?

(06 marks)

(ii) State two important practical aspects that you should consider in order to get a smooth and dense deposit in Electrogravimetry. (06 marks)

(iii) A student carried out voltammetry to determine the concentration of a metal ion (A^{n+}) in an unknown sample present in the form of a solution. A 25.00 cm^3 of 0.5 M KCl (as an inert supporting electrolyte) was added to 10.00 cm^3 of the sample and the total volume made up to 100.0 cm^3 with distilled water. The diffusion current (after correcting for residual current) was found to be 0.15 A. The analysis was then repeated using the same amount of sample and KCl and also including 10.00 cm^3 of a 5.0 ppm standard solution of A ion before diluting to a final volume of 100.0 cm^3 ; the diffusion current now was found to be 0.90 A. Determine the concentration of A in the original sample in ppm. (12 marks)

(iv) Suppose the above analysis was carried out selectively in the presence of another ion B^{2+}, what can you say about the half wave potentials of $A^{n+}$and B^{2+}? (06 marks)

(v) Sketch and label the expected Amperometric titration curve for the titration between 10.0 $cm^3$of X^+ and 0.01 mol dm^{-3} Y. Only Y undergoes reduction.

$X^+_{(aq)} + Y_{(aq)} \rightarrow XY_{(s)}$ (10 marks)

(vi) State two advantages of Dropping Mercury Electrode in polarography.
(10 marks)

Name:.....

Address:.....

.....

.....

THE OPEN UNIVERSITY OF SRI LANKA

B.Sc Degree Programme 2014/2015

CMU 3128 – Instrumental Chemistry

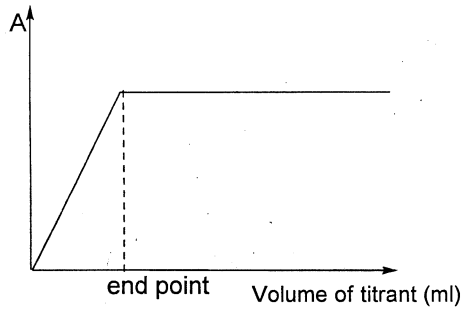
Continuous Assessment Test I –Answer Guide

[1]. (i) The scattered light resulted when a monochromatic light is shone on a sample, will have frequencies different to the incident light. This difference is related to the vibrational transitions in a molecule which are characteristic.

(ii) In Fluorimeter

1. Light source is situated at right angle position to the transducer (or detector)
2. In fluorimeter there is an emission monochromator.
3. Simple filters are used as monochromators.

(iii)



(iv) (a) Singlet excited state

When an 'e' of a filled orbital of a molecule in the ground state absorbs $\overline{E}$ and occupies an empty orbital having a higher $\overline{E}$ without changing its spin, it is called singlet excited state.

(b) Molecular ion peak

Usually it is the peak with the highest mass (if there are no isotopes) which correspond to the molecular weight.

(c) Disadvantages of flame as the Energy source

1. Temperatures are lower thus less sensitive.
2. Limited to few elements (i.e. Less applicable).
3. Nebulizers tend to waste sample resulting lower sensitivity.

[2]. (i) Principle behind coulometry

Total charge passed through the cell is directly proportional to the concentration of the analyte.

(ii) Factors affecting the deposit

1. Current density 2. stirring 3. Addition of complexing agents 4. Temparature

(iii) $i_D = kC_A$

i_D = Limiting diffusion current

C_A = Bulk analyte concentration

K = Constant

$0.15 = kC_A$..... (1)

$0.90 = k(C_A+5)$..... (2)

(1) ÷(2), $0.15 / 0.90 = kC_A / k(C_A+5)$

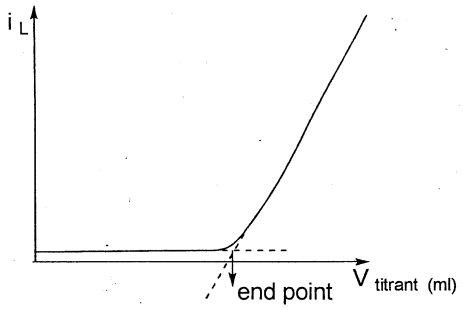
$C_A + 5 = 6\ C_A$

C_A = 1ppm

Dilution factor is 10: $C_{\text{original sample}}$ = 10ppm

(iv) Half wave potentials of A^{n+} & B^{2+} is greater than 0.2V .

(v)



(vi) Advantages of DME

1) Each drop that fells to the solution is having a fresh, uncontaminated surface. Hence give reproducible results.

2) The overpotential for H^+ reduction is very large, even metal ions which are less favorable in reduction than that of H^+ can be determind.

(Results are not interfering with H^+)