THE OPEN UNIVERSITY OF SRI LANKA
B.Sc. Degree Programme / Stand alone courses in Chemistry
Level 5 –Assignment Test 1 – 2011 / 2012

CMU 3128/CME 5128 – INSTRUMENTAL METHODS IN CHEMICAL ANALYSIS

Duration: One and half hours
Date and time: 27th February, 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. (i) Briefly explain the following.

(a) Phosphorescence

(b) Quantum efficiency in Fluorescence

(c) ~~A third electrode is used in constant potential coulometry~~

Rayleigh scattering in Raman spectroscopy.

(15 marks)

(ii) A 10.0 cm^3 of an aqueous sample solution having quinine was diluted to 50.0 cm^3 and found to have an absorbance of 0.300 at 348 nm when measured in a 2.00 cm cell. Another 10.0 cm^3 of the sample solution of quinine was added to a flask having 10.0 cm^3 of 5 ppm standard quinine solution and was diluted to 50.0 cm^3. The absorbance of this solution was 0.600 at 348 nm when measured in the same 2.00 cm cell. Calculate the concentration of quinine in the original sample solution showing all the steps.

(2.5 marks)

2. (i) Identify whether the following methods are used either for qualitative or quantitative analysis and state the principle behind the analysis.

(a) Mass spectroscopy

(b) Polarography IR - spectroscopy

(c) Goulometry photometric titration

(30 marks)

(ii) Draw and label a schematic diagram of a Mass spectrophotometer.

(10 arks)

3. (i) Give two major differences of the following methods:

(a) Atomic Absorption Spectroscopy and Atomic Florescence Spectroscopy

Photometric

(b) Classical titrations and ~~Amperometric~~ titrations

20

(~~16~~ marks)

(ii) A 500 mA current was passed through a 500.0 cm^3 solution of Cu^{2+} 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 $C\ mol^{-1}$)

(14 marks)

Name:.....

Address:.....

.....

.....

THE OPEN UNIVERSITY OF SRI LANKA
B.Sc. Degree Programme / Stand alone courses in Chemistry
CMU 3128/CME 5128 – INSTRUMENTAL METHODS IN CHEMICAL ANALYSIS — 2011 / 2012

Assignment Test 1 - Answer Guide

1. (i)

(a) Relaxation of energy (emission) with change of spin- singlet to triplet or triplet to singlet.

(b) Quantum efficiency = $\frac{\text{No of fluorescing molecules}}{\text{Total no of exited molecules}}$

$= \frac{\text{No of photons emitted by fluorescence}}{\text{Total no of photons absorbed}}$

(c) When molecules are irradiated by a beam of monochromatic light radiation, scattering of light is occurred. Scattered radiation having the same frequency as the incident light is called the Rayleigh scattering. Rayleigh scattering results a more intense peak compared to other peaks appeared in the Raman spectroscopy.

(ii) If A = Absorbance, C_u = Concentration of the unknown, l = path length, C_u = Concentration of the standard

$A_1 = C_u \varepsilon l \longrightarrow (1)$

$A_2 = (C_u + C_s) \varepsilon l \longrightarrow (2)$

(1) / (2) $\frac{A_1}{A_2} = \frac{C_u}{(C_u + C_s)}$

$\frac{0.300}{0.600} = \frac{C_u}{(C_u + 0.500)}$

$C_u = 5.00$ ppm

Concentration of quinine in the original sample = 5.00 ppm x 5 = 25.0 ppm

2. (i) (a) Mass spectroscopy

Qualitative : Fragmentation pattern is characteristic .
Here kinetic / electrical energy interact with matter to undergo fragmentation. Fragmentation patterns result a mass spectrum which is a graph of mass *vs* relative abundance.

Quantitative : Draw a calibration curve suing standard samples and interpolate the measurement of the unknown using the calibration curve to get the concentration/amount of the unknown.

(b) IR spectroscopy

Qualitative: Non linear molecules absorb IR radiation and move to a higher vibration state. IR spectrum (Transmittance *vs* frequency) is characteristic.

Quantitative : not much. Use only for CO, SO_2 - environmental analysis.
Draw a calibration curve suing standard samples and interpolate the measurement of the unknown using the calibration curve to get the concentration/amount of the unknown.

(c) Photometric titrations

Quantitative: A titration of which the end point is detected by the drastic change in absorbance. Either titrant, titrand or the product should absorb light.

(ii) Refer the diagram given in the text book (unit I). The main parts should be labelled – sample inlet, ionizing chamber, mass analyser, vacuum, detector.

3. (i) (a)

AAS	AFS
Absorbance is measured	Emission is measured
Source and the detector in the same line	They are parallel to each other
Can be applied to most of the elements	Only for fluorescing compounds
Less sensitive	Highly sensitive

(b)

Classical titrations	Photometric titrations
Either pH/E_{cell} / pM changes with the volume of titrant added.	Absorbance is changing with the volume of titrant added.
Needs an indicator	No need
Not necessary	Either titrant/titrand/ product should absorb light.
Less accurate	More accurate
Titration curve is S shape with a vertical portion resulting at the end point.	Intersection of two straight lines which meets at the end point.