THE OPEN UNIVERSITY OF SRI LANKA
B.Sc. Degree Programme / Stand alone courses in Chemistry
Level 5 –Assignment Test 1 – 2013 / 2014

CMU 3128– INSTRUMENTAL METHODS IN CHEMICAL ANALYSIS

Duration: One hour
Date and time: 22nd August, 2014 from 9.30 a.m. to 10.30 a.m.

Reg. No.......................................

Question number	marks
1	
2	
Total	
%	

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

1. (i) Comment briefly on the following statements.

(a) Organic compounds having only single bonds do not absorb UV or visible radiation.

(b) Fluorescent molecules often absorb UV light and emit visible light.

(20 marks)

(ii) (a) What is the necessary property in a molecule to absorb IR radiation?

(b) What is the principle behind Raman Spectroscopy?

(20 marks)

(iii) State how the light being measured is resulted in each of the following methods.
(a) Atomic Emission Spectroscopy.

(b) Atomic Absorption Spectroscopy.

(c) Atomic Fluorescence Spectroscopy.

(b) phosphorescence

(24 marks)

(iii) Draw and label a schematic diagram of a mass spectrophotometer.

(12 marks)

(iv) A plant leaf sample was digested (5.034 g in 10.0 mL) and analyzed for potassium by atomic emission spectroscopy using the method of standard addition. Two 1.00 mL portions of the sample solution were diluted to 10.0 mL with distilled water in separate flasks. To one flask, 5.00 mL of 100 ppm K^+ solution was added before diluting. The emissions of the two solutions were recorded as 15 and 90. What is the weight of potassium in the plant leaf?

(24 marks)

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

(12 marks)

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

(12 marks)

(iii) The concentration of an unknown solution of Cu^{2+}(100 cm^3) was determined using polarography. The maximum limiting current was 455 mA while the residual current was 5 mA. A 50 ppm Cu^{2+}(100 cm^3) solution gave a limiting current of 405 mA and the residual current remained the same. What is the concentration of Cu^{2+} in the sample?

(20 marks)

(iv) (a) State the measurements taken/recorded in amperometric titration and photometric titrations.

(b) State **one major similarity** in amperometric titration and photometric titrations.

(12marks)

(v) Assume that both A^{2+} and B^{2+} ions are present in a solution.

$A^{2+}(aq) + 2e \rightarrow A(s) \qquad E° = 0.54\ V$

$B^{2+}(aq) + 2e \rightarrow B(s) \qquad E° = 0.40\ V$

Can you determine the concentration of both A^{2+} and B^{2+} using electrogravimetry?
Explain your answer in brief.

(14 marks)

(vi) Briefly explain what is meant by the following terms.

(i) Overpotential

(ii) Ohmic drop

(iii) Electrode polarisation

(30 marks)

THE OPEN UNIVERSITY OF SRI LANKA
B.Sc. Degree Programme / Stand alone courses in Chemistry
Level 5 – Assignment Test 1 – 2013/2014

CMU 3128 – INSTRUMENTAL METHODS IN CHEMICAL ANALYSIS

ANSWER GUIDE

1. (i)

a.

In single bonds, $\sigma \longrightarrow \sigma^*$ transition which needs high energy will result breaking of the bond. It is hard to generate high energy. The high energy can be absorbed by other atmospheric compounds.

b.

Due to absorption of light, molecules travel from the lowest vibrational energy level of the ground state to any vibrational energy level of the exited state. Then they lose some of this energy and relax to the lowest vibrational energy level of the excited state and finally to any vibrational energy level of the ground state emitting radiation. Therefore, energy absorbed is more than emitted thus λ absorbed is lower than λ emitted.

(ii)

a. A Vibrational dipole or a dipole moment resulted in an asymmetric molecule is required for IR spectroscopic determination.

b. The difference in frequencies of the scattered light & the incident light correspond to vibrational transitions which are characteristic to a molecule. Therefore, with this information it is possible to do qualitative analysis.

(iii)

a. Exited state $\longrightarrow$ Ground state

b. Ground state $\longrightarrow$ Exited state

c. Exited state $\longrightarrow$ Ground state

d. Triplet exited state $\longrightarrow$ Singlet ground state

(iv) Refer Unit I, page no.59, Figure 5.3

(v) Concentration of added amount in the flask now $= \frac{100}{1000} X \frac{5}{10} = 50$ ppm

Emission (E) α Concentration (C)

E = KC Where K is Constant

When we apply the information given,

$15 = KC \longrightarrow ①$

$90 = K(50 + C) \longrightarrow ②$

$$\frac{①}{②} \quad \frac{15}{90} = \frac{C}{50+C}$$

$50 + C = 6C$

$50 = 5C$

C = 10 ppm This is the concentration of the diluted sar

(10 ppm = 10 mg in 1000ml)

Weight in 10ml of the diluted sample (which is obtained by 1ml of the original sample) $\Big\} = \frac{10\ \text{mg}}{1000\ \text{ml}} \times 10\text{ml} = 0.1\ \text{mg}$

$$\left.\begin{array}{l}\text{The amount in 10ml of the original leaf sample}\\ \text{(It's 10 times diluted)}\end{array}\right\} = 0.1\ \text{mg} \times 10 = \underline{\underline{\mathbf{1\ mg}}}$$

2. (i) Refer Unit II, page no.20, Figure 2.2

(ii) Diffusion current (i_D) α Amount of analyte (C) (i.e. Concentration)

(iii) $i_d = i_L - I_R$

For unknown sample, $i_{d(1)} = 455\text{mA} - 5\text{mA} = 450\ \text{mA}$

For 50 ppm sample, $i_{d(2)} = 405\text{mA} - 5\text{mA} = 400\ \text{mA}$

$i_d = KC$ where K is constant & C is concentration of the sample

For unknown sample, 450 mA = KC ——— ① For 50 ppm, 400 mA = K 50 ppm ——— ②

$$\frac{①}{②} \qquad \frac{450}{400} = \frac{C}{50} = \frac{225}{4}$$

$$C = \underline{\underline{56.25\ \text{ppm}}}$$

(iv)

(a) Amperometric titration = Current & volume of the burette.
Photometric titration = Absorbance & Volume of the burette.

(b) Higher accuracy; higher sensitivity; higher precision

(v) No. The concentrations of A^{2+} & B^{2+} cannot be determined separately because the difference in reduction potentials = 0.54 V- 0.4 V = 0.14 V. In the electrogravaimetry, selectivity can be achieved (i.e. precipitation of two ions separately), if only the difference in the reduction potentials is equal or larger than 0.2V. In this case 0.14 V < 0.2 V.

(vi)

(i) The difference between the theoretical & actual voltage required to carry out electrolysis.

(ii) When a current flows through a cell, the voltage across the electrolysis is dropped. This voltage drop is equal to IR where; I = current & R= Internal resistance.

$$E_{app} = E_{cell} - IR$$

(iii) Number of processes takes place in the surface of the electrode by the exchange of electrons within the surface. As the external voltage increases, one of these processes becomes limited with no increase of current accordingly. Then the electrode is said to be polarized.

$$E_{app} = E_{cell} - IR - \eta \quad \text{where } \eta \text{ is the over potential}$$