THE OPEN UNIVERSITY OF SRI LANKA
B.Sc. Degree Programme / Stand alone courses in Chemistry
Level 5 –Assignment Test 1 – 2015 / 2016

CMU 3128/CME 5128 – INSTRUMENTAL METHODS IN CHEMICAL ANALYSIS

Duration: One hour
Date and time: 15th October, 2016
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) What is the principle behind IR spectroscopy with respect to qualitative analysis?

(10 marks)

(ii) Draw and label a schematic diagram of a mass spectrum showing the base peak and the molecular ion peak.

(10 marks)

(iii) The complex X^+ is colored. It reacts with fluoride ions in aqueous medium giving a colourless compound XF.

$$X^+ + F^- \rightarrow XF$$

The absorbance of a 750.0 mL of X^+ solution was 0.4000. When a 250.0 mL of a F^- solution was added to it, the absorbance was 0.100 given that the path length is equal to 1 cm. and the molar absorptivity coefficient of X^+ was 3.00×10^4 $Lcm^{-1}mol^{-1}$. Calculate the concentration of the added F^- solution.

(16 marks)

(iv) State two advantages of Raman spectroscopy over IR spectroscopy.

(04 marks)

(v) Sketch and label the expected photometric titration curve where only the reactant added by the micro burette absorbs light while the reactant in the flask and the product do not absorb light.

(10 marks)

(vi) State **one** important **difference in the spectrum** corresponding to Atomic Fluorescence spectroscopy and Molecular fluorescence spectroscopy.

(04 marks)

2. (i) An amperometric titration was carried out to determine the concentration of B^{2+} (20.0 mL) with A^- (0.100 M) and the end point reading obtained was 25.00 mL. Only B^{2+} is reducible while A^- does not undergo any reduction or oxidation.

$$A^-_{(aq)} + B^{2+}_{(aq)} \rightarrow A_2B_{(s)}$$

(a) Sketch and label the amperometric titration curve.

(10 marks)

(b) The measurements taken in the above titration have to be corrected. Why? Suggest how you would overcome this error.

(08 marks)

(ii) What is the principle behind **Voltametry as a quantitative** analytical method?

(10 marks)

(iii) Briefly explain the following statement.
" Electrogravimetry does not require calibration standards."

(06 marks)

(vi) A coulometric titration was carried out with Y^{2+} to determine the concentration of Z^-. Y^{2+} is generated electrochemically from Y. The titration of 20.0 mL of Y^{2+} required 400 seconds at 20 mA to reach the end point. Calculate the concentration of Z^-. (Faraday's constant = 96,500 $Cmol^{-1}$)

$$Y^{2+}{}_{(aq)} + Z^-{}_{(aq)} \rightarrow Y Z_2{}_{(aq)}$$

(12 marks)

Name: .....

Address: .....

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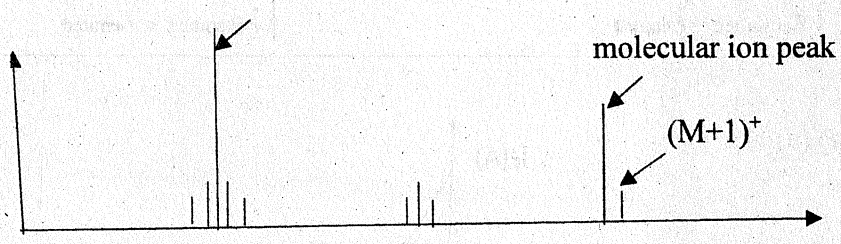
Answer Guide (Assignment Test 1-2015/2016)

1 (i) When IR radiation is absorbed by a molecule, it moves to a higher vibrational state and the radiation absorbed (IR spectrum) is characteristic. In addition, specific functional groups give IR spectrum in specific regions with characteristic shape and size thus can be identified.

(ii)



(iii) $[X^{2+}]_{initial} = c_1 \qquad \varepsilon = 3 \times 10^4\ L\ cm^{-1}\ mol^{-1} \qquad A = 0.400 \qquad l = 1\ cm$

$$A = \varepsilon c_1 l$$

$$c_1 = \frac{A}{\varepsilon l} = \frac{0.400}{3 \times 10^4\ L\ cm^{-1}\ mol^{-1} \times 1cm} = {}^4/_3 \times 10^{-5} mol\ L^{-1}$$

$$Number\ of\ moles\ of\ X^{2+}\ in\ 750\ mL = \frac{{}^4/_3 \times 10^{-5}\ mol}{1000\ mL} \times 750\ mL = 10^{-5}\ mol$$

$[X^{2+}]_{remainng}$ *in the final solution* $= c_2$ (total volume = 1000. mL)

$$c_2 = \frac{A}{\varepsilon l} = \frac{0.100}{3 \times 10^4\ L\ cm^{-1}\ mol^{-1} \times 1cm} = {}^1/_3 \times 10^{-5} mol\ L^{-1}$$

$$Number\ of\ moles\ of\ X^{2+} remaing\ in\ 1000\ mL = {}^1/_3 \times 10^{-5} mol$$

$$Number\ of\ moles\ X^{2+} reacted = 10^{-5}\ mol - {}^1/_3 \times 10^{-5} mol = {}^2/_3 \times 10^{-5} mol$$

$$Number\ of\ moles\ F^{2-} reacted = {}^2/_3 \times 10^{-5} mol$$

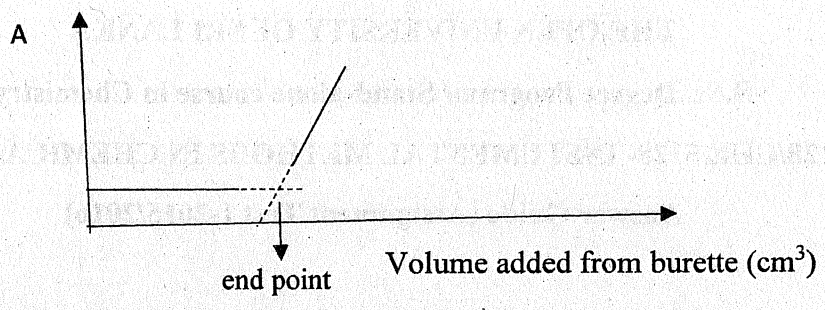
$$Concentration\ of\ F^{2-} = \frac{{}^2/_3 \times 10^{-5} mol}{250\ mL} \times 1000\ mL = \mathbf{2.67\ mol\ L^{-1}}$$

(volume of F^- =250.0 mL)

(iv) Raman Spectroscopy is having the following advantages compared to IR Spectroscopy:

1. Vibration modes of symmetric molecules can be identified.
2. Samples with water also can be analyzed.
3. Special cells (made out of salts) are not necessary.

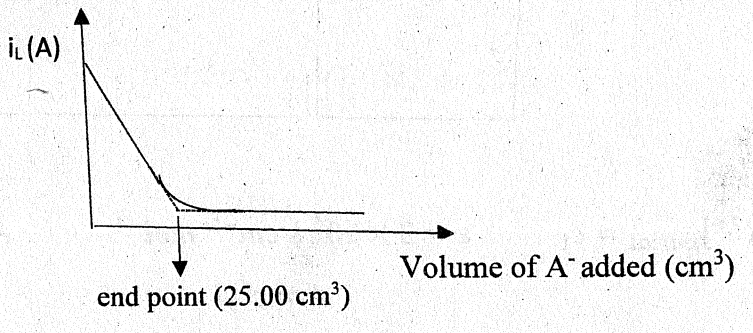
(v)



(vi)

Atomic fluorescence spectroscopy	Molecular fluorescence spectroscopy
Line spectrum $\lambda_{absorbed} = \lambda_{emited}$	Band spectrum $\lambda_{absorbed} < \lambda_{emited}$

2 (i) (a)



(b) Reason: with the addition of solution from the burette, dilution takes place decreasing concentration.

This can be overcome by using a micro burette with a highly concentrated solution in the burette or calculating the corrected limiting current ($i_{corrected}$) using the formula given below.

$$i_{corrected} = i_{measured} \times \frac{Volume_{(total)}}{Volume_{(initial)}}$$

(ii) Voltametry: when the transport of analyte ions due to convention and migration are minimized,

$Diffution\ current(i_D) \propto Concentration\ of\ analyte(C_A)$.

(iii) Electrogravimetry: The weight difference of the electrode before and after the deposition of salt is measured followed by calculation using the molecular formula of the salt.

(iv) $i = 20\ mA = 20 \times 10^{-3}\ A$ $\qquad t = 400\ s$

$Q = it = 20 \times 10^{-3}\ A \times 400\ s = 8\ C$

$$Number\ of\ moles\ of\ Y^{2+}\ genetated = \frac{Q}{nF} = \frac{8\ C}{2 \times 96{,}500\ Cmol^{-1}}$$

$$Number\ of\ moles\ of\ Z^{-}\ reacted = \left(\frac{8\ C}{2 \times 96{,}500\ Cmol^{-1}}\right) \times 2$$

$(Y^{2+}: Z^{-} = 1:2)$

$$Concentration\ of\ Z^{-} = \left(\frac{8\ C}{2 \times 96{,}500\ Cmol^{-1}}\right) \times 2 \times \frac{10^3}{20.00\ cm^3}$$

$$= \mathbf{0.00415\ mol\ dm^{-3}}$$