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

CMU 3123/CME 5123 – Analytical Chemistry

Duration: One hour
Date and time: 01st March, 2015 from 9.00 a.m. to 10.00 a.m.

Reg. No...

Question number	Max. marks	marks
1	56	
2	44	
Total		

Instructions to students

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

1. A soil sample (5.1420 g) was digested, filtered and the volume was made up to 1.0 L. A 25.0 cm^3 aliquot of this solution was diluted to 100.0 ml and the amount of Ca^{2+} was determined using Atomic Absorption Spectroscopy. A 8 ppm standard solution gave an absorbance of 0.672 at λ_{max} at a path length of 1 cm. The diluted sample gave an absorbance of 0.424 (using the same cell, λ_{max} and the spectrophotometer).

   (i) Calculate the % of Ca in the soil sample assuming that there is no interference. (Ca = 40.0 g).

(12 marks)

(ii) Give one possible chemical interference that could have taken place in the above analysis and suggest a way of overcoming that interference.

(10 marks)

(iii) When this soil sample was subjected to Thermogravimetry, the weight was reduced to 4.564 g at 100 °C.

(a) Draw and label the Differential Thermogravimetric curve (DTG).

(b) Give a reason for the weight loss.

(c) Give one major difference between thermogravimetry and Differential Thermal analysis.

(12 marks)

(iv) Give two differences between the UV- Visible spectrophotometer and Atomic absorption Spectrophotometer.

(08 marks)

(v) A conductometric titration was carried out between 25.0 ml of 0.1 M HCl and 0.1 M NH_4OH. Draw and explain the titration curve.

(14 marks)

2. (i) Calculate the minimum distribution ratio of X between, which enables to extract at least 95% of the solute X from 100 ml of water with 50 ml of diethyl ether.

(06 marks)

(ii) Assuming that the distribution ratio does not change, calculate the percentage remaining in the aqueous layer if we do the extraction with two portions of 25ml of diethyl ether?

(10 marks)

(iii) Give two major differences between electrophoresis and paper chromatography.

(08 marks)

(iv) Polarity of the compounds N,P and Q decreases in the following manner.

$N > Q > P$

The R_f values of N, P and Q on a Thin Layer Chromatographic (TLC) plate are 0.9, 0.7 and 0.5 respectively.

Was this TLC run in the normal phase or reverse phase? Justify your answer.

(10 marks)

(vi) Comment briefly on the following statement.
"Selective extraction of metal-ligand complexes can be done by varying pH"

(10 marks)

Name ....
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CMU 3123 – ANALYTICAL CHEMISTRY - ANSWER GUIDE

1. (i) $A = \epsilon c l$

$0.672 = \epsilon.8\,\text{ppm}.l \longrightarrow$ ①

$0.424 = \epsilon.c\,\text{ppm}.l \longrightarrow$ ②

① / ② C = 5.04 ppm of the diluted sample.

Dilution Factor = 4 (25 ml of the original sample is diluted to 100 ml)

Therefore, concentration of the analytical solution = 5.04 x 4 ppm = 20.16 mgl^{-1} = $20.16 \times 10^{-3}\ gl^{-1}$

Percentage of Ca = $\frac{20.16 \times 10^{-3}}{5.142} \times 100\,\%$ = <u>0.36%</u>

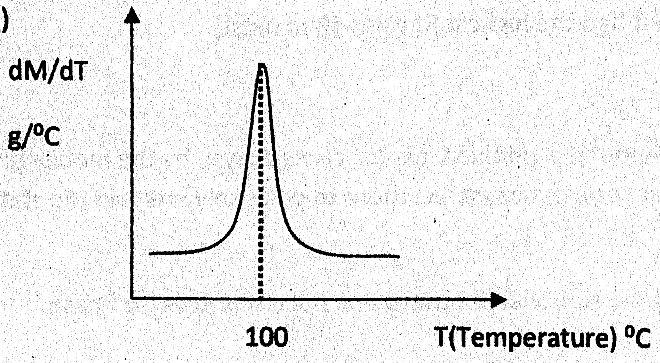
(ii) Differences in instruments:

UV/ Visible Spectroscopy	Atomic Absorption Spectroscopy
1. Source – Tungsten Lamp 2. No Modulator 3. Two monochromators	1. HCL 2. There is a modulator. 3. Only one monochromators

(iii)

Interference	Over coming
1. Stable compound formation	1. Use of releasing agent/Higher fuel temperature/Pre- flame shielding agent.
2. Ionization	2. Add an ionization suppressant

(iv) (a)

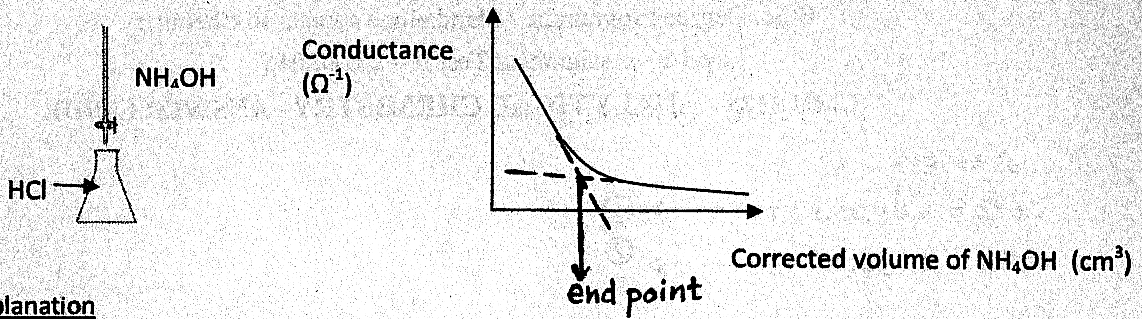


(b) Loss of moisture / evaporation of a gas

(c)

Thermogravimetry	Differential Thermal Analysis
Mass of the sample is continuously monitored with varying temperature.	Temperature difference between the sample and the reference is continuously monitored with varying sample temperature.

(v)



Explanation

HCl is a strong acid which dissociates fully. Therefore the initial conductance is high due to H^+. When adding NH_4OH, the following reaction takes place. $HCl + NH_4OH \longrightarrow NH_4Cl + H_2O$

NH_4Cl dissociates fully, but conductivity of NH_4^+ is much lower than H^+ thus conductance in the solution reduces drastically. After the end point when excess NH_4OH is added. Since NH_4OH is a weak base, it is not fully dissociated and dissociation will be further reduced due to the presence of NH_4^+ in the solution. Therefore, only very little amount of OH^- be existing in the solution thus the conductance will not be increased. The concentration of these ions (NH_4^+ and OH^-) also decreases due to dilution resulting a gradual decrease in conductance.

02. (i) $D = \frac{[X]_{org}}{[X]_{aq}} = \frac{95/50}{5/100} = 38$

(ii) $C_2 = \left[\frac{V_{(aq)}}{DV_{(org)} + V_{(aq)}}\right]^n . C_0$ $C_2 = Concentration\ remaining\ in\ the\ aqueous\ layer$

$V_{(aq)} = 100\,ml$ $D = 38$ $V_{(org)} = 25\,ml$ n = No. of extractions = 2

$C_2 = \left[\frac{100}{(38 \times 25) + 100}\right]^2 . C_0 . 100\% \longrightarrow 100\% \times \frac{C_2}{C_0} = 0.9\,\%$

(iii) Most polar compound is N and it had the highest Rf value (Run most).

Explanation:

The results show that the polar compound is retained less (or carried away by the mobile phase). Therefore, the mobile phase is polar since polar compounds attract more to polar solvents and the stationary phase has to be non polar.

Since the mobile phase is polar and the stationary phase is non polar it is Reverse Phase.

(iv)

Electrophoresis	Paper chromatography
1. No mobile Phase 2. Separation is due to the charge difference.	1. There is a mobile phase. 2. Separation is due to partition

(v) If the metal ligand complex is ML and the Ligand is HL,

$HL \longrightarrow H^+ + L^-$

At low pH (or High $[H^+]$) HL is stable. Therefore, only the complexes with high formation constant will be formed thus extracted. Therefore, selective extraction is possible with varying pH.