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

CMU 3123/CME 5123 – Analytical Chemistry

Duration: One hour
Date and time: 11th April, 2014 from 8.45 p.m. to 9.45 p.m.

Reg. No.....

Question number	Max. marks	marks
1	40	
2	36	
3	24	
Total	100	

Instructions to students

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

(1). (i) What is the **difference** between the

(a) distribution coefficient and distribution ratio?

(b) reverse phase and normal phase chromatography?

(10 x 2 marks)

(ii) The distribution coefficient for the compound "Y" between diethyl ether and water is 60. With a single extraction, how much of ether should be used to extract 90% of "Y" from 100 cm^3 of an aqueous of "Y"?

(06 marks)

(iii) Describe **the principles** behind Thermo Gravimetry and Differential Thermal Analysis in brief.

(14 marks)

2. (i) Draw and label a schematic diagram of a UV/ Visible spectrophotometer.

(06 marks)

(ii) An effluent of a factory was analyzed for Cu^{2+} using Atomic Absorption Spectrophotometer. A 25.0 cm^3 of a sample was diluted to 250.0 cm^3 and the absorption obtained was 0.380. A standard solution of 5 ppm Cu^{2+} gave an absorbance of 0.190. Calculate the concentration of Cu^{2+} in the effluent.

(10 marks)

(iii) Comment on the following statements.

(a) Molecular spectra are band spectra but atomic spectra are line spectra.

(b) UV/Visible spectroscopy is not a good method to analyze compounds with single bonds.

(10 x 2 marks)

3. (i) Give two major advantages of potentiometric titrations over classical redox titrations.

(06 marks)

(ii) **Sketch and explain** the conductometric titration curve of a titration between 25.0 cm^3 of 0.01 M HCl and 0.01 M NH_4OH.

(18 marks)

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CMU3123 CAT II – Answers Guide

(1)

(i) (a) Distribution coefficient (K_d)

One species (form) is considered. $K_d = \frac{\text{molar concentration of the species in organic phase}}{\text{molar concentration of the species in aqueous phase}}$

Distribution ratio (D)

The total/analytical concentration of all existing forms of the component is considered. It includes ionized, complexed and also undissociated forms.

$$D = \frac{\textit{Analytical concentration of the species in the organic phase}}{\textit{Analytical concentration of the species in the aqeous phase}}$$

(b) Reverse phase: mobile phase – polar; stationary phase – non polar
Normal phase: mobile phase – non polar; stationary phase – polar

(ii) $K_d = 60\%$, % extracted $= 90\%$, $V_{aq} = 100cm^3$
If we assume that a total of 100 moles of Y is distributed,
No. of moles in the ether layer = 90
No. of moles in the aqueous layer = 10

$$K_d = \frac{[y]_{org}}{[y]_{aq}} \qquad 60 = \frac{90/V_{org}}{10/100cm^3} \qquad V_{aq} = 15cm^3$$

(iii) Thermo Gravimetry (TG)

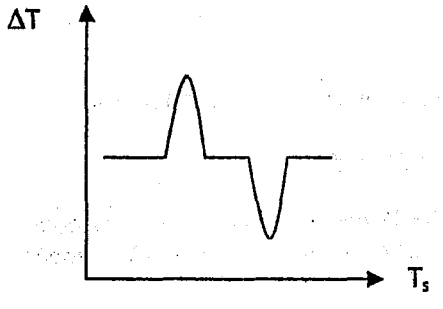
In TG, mass of sample is measured as a function of temperature. Change of mass is due to formation and evaluation of a gas. From the loss of weight we can calculate or quantify the analyte amount. From the TG curve the temperature range in which the compound is stable can be found.

Differential Thermal Analysis (DTA)

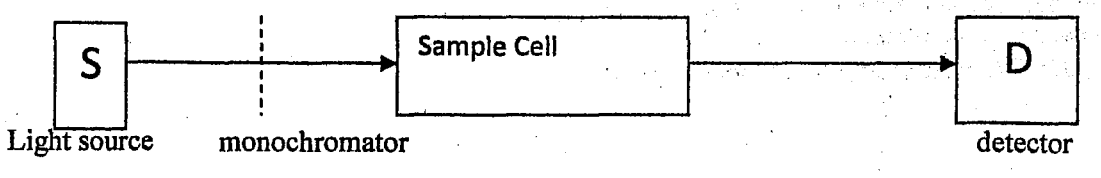
The temperature difference (ΔT) between the sample & the reference is measured with the increasing sample temperature (T_s). Any physical or chemical change which either absorbs (endothermic) or release (exothermic) energy will change the temperature of the sample.

In the curve of ΔT *Vs* T_s, peak area (A) is proportional to the mass of the sample and thereby can do quantitative analysis.

$A = -kGm\Delta H$



(2). (i)UV/Visible Spectrophotometer



(ii) $A = \varepsilon C l$

$0.380 = \varepsilon C l$ ——————(1)

$0.190 = \varepsilon . 5ppm . l$ ——————(2)

(1) / (2), $2 = C / 5ppm$ $C = 10$ ppm

dilution factor = 10 (25 cm^3 to 250 cm^3) ; Concentration of Cu^{2+} in the effluent = 100 ppm

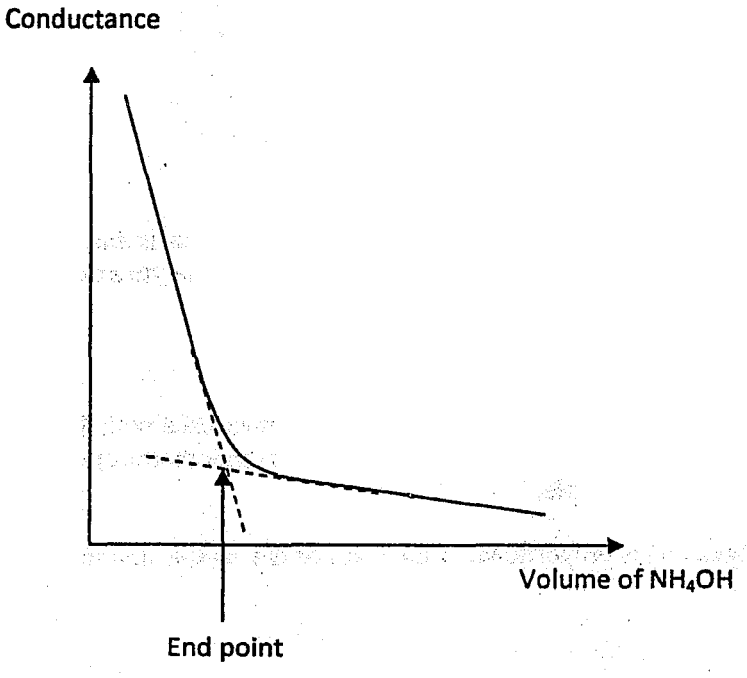
(iii)(a) Molecular Spectra : Molecules have vibrational & rotational energy levels in an electronic energy state. So, light absorbed or emitted can have slightly different energies & therefore, wavelengths. This results a band spectrum.

Atomic Spectra : Atoms absorb or emit light of only one fixed energy (therefore, one wavelength or can say a narrow range of wavelengths). This results a line spectrum.

(b) Single bonds
They have only σ bonds thus absorb high energy which is difficult to generate by a UV light source.
Such high energy can be absorbed by atmospheric components resulting inaccurate values and also might break the bond.

(3). Conductometric titration curve between 25.0 cm^3 of 0.01 M HCl & 0.01 M NH_4OH

(Strong Acid *Vs* Weak Base)



Explanation

Conductance of HCl will decrease with the addition of NH_4OH due to the decrease of H^+ amount.

$HCl + NH_4OH \longrightarrow NH_4Cl + H_2O$

NH_4^+ ions have a lower conductivity than H^+. After the end point, with the addition of more NH_4OH, (weak base), $[NH_4^+]$ will be less due low rate of dissociation since NH_4^+ is already present and also due to dilution.

(iii) Advantages of potentiometric titrations compared to classical redox titrimetry(Any two of the following)
1).More accurate.
2).Can carryout titrations up to where $\Delta E^0 = 0.2$ V (In classical titrimetry it is only up to 0.4 V)
3).Can apply for non aqueous solutions.
4).No need to have indicators.
5).Can use for turbid solutions or colored solutions.