CHUKA   **UNIVERSITY**

UNIVERSITY EXAMINATIONS

FOURTH YEAR EXAMINATION FOR THE AWARD OF DEGREE OF BACHELOR OF

CHIN 101: QUANTITATIVE CHEMICAL ANALYSIS

STREAMS: **TIME: 2 HOURS**

DAY/DATE: MONDAY 08/4/2019   **11.30 A.M. – 1.30 P.M.**

INSTRUCTIONS: Answer question ONE and any other TWO questions

QUESTION ONE (30 MARKS)

(a)   (i)   List four conditions which a reaction must fulfill for it to be used in titrimetric analysis   [4½ marks]

        (ii)   State six requirements which a primary standard should satisfy   [3 marks]

        (iii)   Briefly explain how organic nitrogen can be determined using Kjeldahl procedure.   [1½ marks]

        (iv)   The The Kjeldahl procedure was used to analyze 500 μ 1 of a solution containing 50.0 mg protein/ml solution. The liberated NH_3 was collected in 5.00 mL of 0.0300M Hcl. The remaining acid required 10.00 mL of 0.010M NaoH for complete titration. Calculate the weight percent of nitrogen in the protein.   [7 marks]

(b)   (i)   Sketch the titration curve that would be obtained during the titration of Na2CO3 versus HCl showing the pH range of phenolphthalein, methyl red, methyl orange indicators and also the effect of boiling the solution near the end point.   [3 marks]

         (ii)   Discuss the titration of sodium carbonate (a diprotic base) using phenolphthalein and methyl orange indicator.   [5 marks]

         (iii)   25ml from a stock solution containing $NaHCO_3$ and Na2 CO_3 was diluted to 250ml with CO_2 free distilled water, 25ml of the diluted solution when titrated with 0.12 M Hcl required 8ml when phenolphthalein was used as an indicator.   When 20ml of diluted solution was titrated with same acid it required 18ml when methyl orange was used as an indicator. Calculate concentration of $NaHCO_3$ in the stock solution in gm/litre and in mole/litre.
                 [3 marks]

(c)   (i)   Briefly explain how Mg, Zn and Cu can be determined in the mixture using complexion titration.   [2 marks]

         (ii)   A solution containing 25.00 ml of Ni^{2+} in dilute Hcl was treated with 25.00 ml of 0.05283 M Na_2EDTA. The solution was neutralized with NaoH and the pH was adjusted to 5.5 with acetate buffer. The solution turned yellow when a few drops of xylenol orange indicator were added. It was then titrated with 17.61ml of 0.002299m Zn^{2+} to reach the red end point. Determine the molarity of Ni^{2+} in the unknown solution.   [3 marks]

QUESTION TWO (20 MARKS)

(a)   (i)   Explain why iodimetric titrations must be performed in neutral or mildly alkaline (Ph 8) to weakly acidic solutions.   [3 marks]

(ii) The purity of a hydrazine (N_2H_4) sample is determined by titration with iodine. A sample of the oily liquid weighing 1.4286g is dissolved in water and diluted to IL in a volumetric flask. A 50.00 Ml aliquot is taken with a pipette and titrated with standard iodine solution, requiring 42.41ml. The iodine was standardized against 0.4123g primary standard AS_2O_3 by dissolving the AS2O3 in a small amount of NaOH solution adjusting the pH to 8 and titrating requiring 40.28ml iodine solution. Calculate the percent purity by weight of the hydrazine. [4 marks]

$$[As=74.9216, 0=15.9994, h=1.00794, i=126.9045, n=14.0067]$$

(b) (i) Give an explanation for the error encountered when using Volhard's method for the titration of silver in the presence of free nitric acid with either standard potassium thiocyanate or using iron (III) ammonium sulphate as indicator. [2 marks]

(ii) Suggest three ways of overcoming the problem in (a b (i) above [3 marks]

(iii) The dissociation constant Kd for $CaSO_4$ in aqueous solution is $5.2 x 10^{-3}$. Calculate the solubility of calcium sulphate in water and the percent of the dissolved solute that is present as the undissociated compound.

$$[solubili\,ty\ product\ of\ CaSO_4=2.6x10^{-5}]$$

[3 marks]

(c) (i) Outline the general procedures for testing hypotheses [2½ marks]

(ii) Discuss/describe ways of characterizing analytical procedures during chemical analysis. [2½ marks]

QUESTION THREE (20 MARKS)

(a) Explain why a calibration program should be in place in any analytical chemistry which deals with quality assurance of instrumentation. [1 mark]

(b)   For a particular analysis, it has been determined that the sampling error is 6%

$\left(S_s=0.06\right)$. A particular analytical technique can give a precision of 1%. What is the

overall precision and it is worth considering a slower technique which can give a

precision of 0.2%?   [2½ marks]

(c)   (i)   Explain ways of overcoming problems associated with gravimetric analysis.

[5½

marks]

(ii)   A mixture containing only $FeCl_3$ and $AlCl_3$ weighs 5.95g. The chlorides

are   converted to the hydrous oxides and ignited to Fe_2O_3 and Al_2O_3 .

The resulting   oxide mixture weighs 2.62. Calculate the percent Fe and Al in the

original   mixture.

[4 marks]

(iii)   A 0.2795 g sample of an insecticide containing only lindane

$\left(C_6H_6Cl_6;fw=290.8\right)$ and DDT $\left(C_{14}H_9Cl_5;fw=354.5\right)$ was burned in a stream of oxygen

in a   quartz tube. The products $\left(CO_2H_2O\wedge HCl\right)$ were passed through a

solution of   NaHCO₃. After acidification, the chloride in this solution yielded

0.7161g of   AgCl. Calculate the percentage Lindane and DDT in the sample.

[3 marks]

(d)   (i)   State the various physical properties of electrolytic precipitates   [1½ marks]

(ii)   List the major factors that influence the physical characteristics of deposits.

[1½

marks]

(iii)   Briefly explain the analytical basis of electrogravimetry   [1 mark]
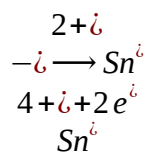

QUESTION FOUR (20 MARKS)

(a)   (i)   With the help of a diagram, describe experimental set up for electrogravimetry.

[5 marks]

   (ii)   A sample of stannic chloride was reduced completely to stannous chloride
          according to the reaction

$$Sn^{4+} + 2e^- \longrightarrow Sn^{2+}$$

   The applied current was 9.65A and the time taken for reduction was 16.0 min 40s.
   Calculate the initial weight of stannic ion present   [2½ marks]

   (iii)   Discuss the problem with simple electrogravimetry system during analysis.

[1½ marks]

   (iv)   State and explain two advantages of a three electrode system in electrogravimetry
          technique.   [1½ marks]

   (v)   Write short notes on the application of electrogravimetry under the following
         heading:
         (I)   Quantitative analysis   [1½ marks]
         (II)   Separations   [1 mark]
         (III)   Preconcentrations   [1 mark]

(b)   (i)   A sample consisting entirely of pure Li_2CO_3 and pure $BaCO_3$ weighs 1.000 g and
            requires 30.00 ml of 0.500M HCl for neutralization. Calculate the number
            of grams of Li_2CO_3 in the sample. $[Li=6.9, Ba=137.3, c=12, 0=16]$ [3 marks]

   (ii)   A 0.500g sample of pure CaCO3 is dissolved in water to which 50.00ml of HCl
          solution have been added. The solution then requires 6.20ml of NaOH
          solution of which 1.000ml $\equiv$ 1.010ml of the Hcl. Calculate the normality of

each of the two   solutions $[Ca=40.1, C=12, 0=16]$

[3 marks]

$[Ca=40.1, C=12, 0=16]$