MANICALAND STATE UNIVERSITY OF APPLIED SCIENCES

FACULTY OF ENGINEERING

DEPARTMENT: CHEMICAL AND PROCESSING ENGINEERING

MODULE: ANALYTICAL CHEMISTRY

CODE: CHEP 226

SESSIONAL EXAMINATIONS
DECEMBER 2022

DURATION: 3 HOURS

EXAMINER: MR M MAPOSA

INSTRUCTIONS

1. *Answer **Any four** questions in this question paper*
2. *All questions carry the same number of marks.*
3. *Start **each** question on a fresh page*
4. *Total marks 100*

Additional material: *Data booklet*
Statistics tables

QUESTION 1

a) Write brief notes to distinguish between the following analytical chemistry terms

i) *Error and bias*

ii) *Precision and accuracy*

iii) *Signal and noise* [6]

b) Describe fully the circumstances which may compel an analyst to

i) *calibrate the instrument of measurement*

ii) *employ internal calibration method*

iii) validate an analytical method [6]

c) The following sets of results were obtained by two analysts **A** and **B** on water hardness of a sample of borehole water

B [Ca^{+2}] /ppm	10.2	10.4	10.3	10.3	10.5	10.4	10.0	
A [Ca^{+2}]/ppm	10.1	10.0	10.1	10.3	10.3	10.2	10.1	10.4

i) Find the mean, median, mode, range, standard deviation, relative standard deviation and variance for each set of data.

ii) Identify the analyst who produced more precise results. Give reasons for your answer

iii) Using an appropriate test determine if there any significant difference between the means of the two sets of data [13]

QUESTION 2

a) Describe precisely, the basic principle of gravimetric analysis. [3]

b) Give any **two** advantages and **one** disadvantage of gravimetric analysis. [3]

c) Outline a flame based experimental procedure which can be used to approximate the amount of calcium carbonate ($CaCO_3$) in a mixture of calcium carbonate ($CaCO_3$) and sodium chloride ($NaCl$). Your answer should include the materials used, reactions taking place and the measurements you would take. [6]

d) A sample of manganese ore weighing 1.35 grams was heated in an excess solution of nitric acid to dissolve manganese ion producing manganese (III) nitrate ($Mn(NO_3)_3$). After adjusting the acidity of the solution, hydrogen sulfide (H_2S) gas was bubbled into the solution producing 0.32 grams of manganese (III) sulfide (Mn_2S_3). Given that manganese exists mainly as manganese (III) oxide, (Mn_2O_3) in the ore.

i) Explain why manganese ore dissolves in dilute nitric acid

ii) By means of an equation, show the main reaction in the solubility of manganese ore in dilute nitric acid.

iii) Explain the need for excess nitric acid solution during the heating stage

iv) Determine the percentage of manganese in the ore sample?

v) Given that the actual percentage of manganese in the ore was 16 %, calculate percentage yield and percentage error of this analysis

vi) Give any **two** sources of error in this analysis [13]

QUESTION 3

(a) A metal **X** forms a hydroxide, **X**$(OH)_2$ and a carbonate **X**CO_3.

i) Give the charge on the **X** ion in the ionic compounds above

ii) At 25 °C, a saturated solution of **X**$(OH)_2$ has a pH of 10.2, calculate the molar concentration of $OH^-(aq)$ in the saturated solution.

iii) Calculate the value of the solubility product, K*sp,* for $X(OH)_2$ at 25 °C [8]

b) The metal carbonate, XCO_3 has a solubility product, K_{sp}, of 8.35×10^{-16} mol^2dm^{-6} at 25°C.

i) Find a value for the molar concentration of the saturated solution of XCO_3

ii) Using your answer to b(i) and a(ii) select between $X(OH)_2$ and XCO_3, the compound with greater molar solubility in water at 25 °C. Justify your answer

iii) Calculate the solubility of XCO_3 in 0.1 mol dm^{-3} sodium carbonate at 25 °C [9]

c) Nernst equation is an important tool in potentiometry

i) Give the Nernst equation for the determination of cell potential at 298 K

ii) Suggest the significance of Nernst equation in electrochemistry

iii) A zinc-lead cell has an overall equation: $Zn_{(s)} + Pb^{2+}_{(aq)} \rightarrow Zn^{2+}_{(aq)} + Pb_{(s)}$

Calculate the standard cell potential of the system

iv) Calculate the cell potential when 0.1 $moldm^{-3}$ Zn^{2+} and 0,15 $moldm^{-3}$ Pb^{2+} are the electrolytes at 30 °C [8]

QUESTION 4

a) Epsom salt is hydrated magnesium sulphate (MgSO4.nH_2O). The percentage by mass of water of crystallisation and the value of *n* were determined using gravimetric methods. 200 g sample of Epsom salt was heated in an oven at a constant temperature and its mass decreased to a constant mass of 97.8 g. Calculate

i) the percentage by mass of water of crystallisation in Epsom salt

ii) the value of *n*, the number of moles of water of crystallisation per mole of Epsom salt. [7]

b) EDTA titration is a method which can be used to measure the quantify of magnesium in a pack of Epsom. A sample of Epsom Salt of mass 0.85 g was measured and dissolved uniformly in distilled water to make 250 cm^3 of solution. 25 cm^3 portions of the resulting solution were titrated using a 0.010 $moldm^{-3}$ solution of EDTA using eriochrome black as an indicator. It was found that 30.50 cm^3 (0.0305 dm^3) of 0.01000 $moldm^{-3}$ EDTA were required to reach end point. Calculate

i) the number of moles of magnesium ions in the 25 ml volume of solution

ii) concentration of magnesium ions in the solution in ppm

iii) percentage by mass (% w / w) of the magnesium in the pack of Epsom salt. [12]

c) i) Give any two disadvantages associated with the use of hard water

ii) Outline the methods used to reduce temporary and permanent water hardness [6]

QUESTION 5

(a) Volhard and Mohr methods are titration techniques for the determination of chloride ion concentration in an unknown. Give a description of each of the techniques, highlighting the reagents used, procedure, equations of reactions occurring during each analysis and indicators used. [8]

(b) Mohr method is a direct titration method while Volhard method is a back titration method.

i) Distinguish clearly between direct and back titration.

ii) Under what circumstances would back titration be preferred instead of direct titration? [5]

c) Two samples from the same crude industrial effluent were known each to contain 30 g of a phenol per 100 cm^3 of aqueous solution. During phenol recovery experiments, the samples were treated with ether in two different ways to extract the phenol, sample one: 100 cm^3 of ether once and sample two; 50 cm^3 of ether twice at room temperature. Given that the partition coefficient is 40,

i) Calculate the mass of phenol extracted in each case

ii) Calculate the percentage recovery in each case [6]

d) Give any two applications of

i) *GLC*

ii) *HPLC*

iii) *TLC* [6]

END OF EXAMINATION