2528/304
2922/304
ENVIRONMENTAL ANALYTICAL TECHNIQUES
June/July 2020
Time: 3 hours

THE KENYA NATIONAL EXAMINATIONS COUNCIL

DIPLOMA IN ENVIRONMENTAL SCIENCE AND TECHNOLOGY

MODULE III

ENVIRONMENTAL ANALYTICAL TECHNIQUES

3 hours

INSTRUCTIONS TO CANDIDATES

You should have the following for this examination:
answer booklet;
non - programmable scientific calculator.
*This paper consists of **TWO** sections; **A** and **B**.*
*Answer **ALL** the questions in section A and any **THREE** questions from section B in the answer booklet provided.*
*Each question in section **A** carries **4** marks while each question in section **B** carries **20** marks.*
Maximum marks for each part of a question are as shown.
Candidates should answer the questions in English.

This paper consists of 6 printed pages.

Candidates should check the question paper to ascertain that all the pages are printed as indicated and that no questions are missing.

SECTION A (40 marks)

Answer ALL the questions in this section.

1. Define each of the following as used in redox titrations:

   (a) Reducing agent; (2 marks)

   (b) Oxidation number. (2 marks)

2. (a) Write a balanced redox equation for the reaction between copper (II) sulphate and iron (II) nitrate solution. (2 marks)

   (b) Identify giving a reason the oxidizing agent in (a) above. (2 marks)

3. Outline the process of preparing 250 mL of 0.085 M potassium dichromate solution. ($K_2Cr_2O_7 = 294.2$ g/mol). (4 marks)

4. (a) Write the mathematical expression of the formation constant, k_f, for the reaction between Cd^{2+} and fully deprotonated ethylenediamine tetra acetic acid, EDTA ($k_f = 2.9 \times 10^{16}$). (2 marks)

   (b) Explain the significance of the formation constant, k_f, in relation to equilibrium position of the reaction. (2 marks)

5. Match the gravimetric method with the appropriate example as shown in table 1. (4 marks)

Table 1

Gravimetric method	Example
• Precipitation gravimetry	$10\,g\ H_2O \xrightarrow[2.\ Drying]{1.\ Filtration} 2\,g$ increase in mass of filter paper.
• Volatilization gravimetry	$Zn(OH)_4 + 2e^- \longrightarrow Zn_{(s)} + 4OH^-$
• Electrogravimetry	$Ag^+_{(aq)} + Cl^-_{(aq)} \longrightarrow AgCl_{(s)}$
• Particulate gravimetry	$SiO_{2(s)} + 4HF_{(aq)} \longrightarrow SiF_{4(g)} + 2H_2O_{(l)}$

6. Name the predominant phosphate form in the segments labelled **A, B, C** and **D** of the solubility curve of $Ca_3(PO_4)_2$ shown in figure 1. (4 marks)

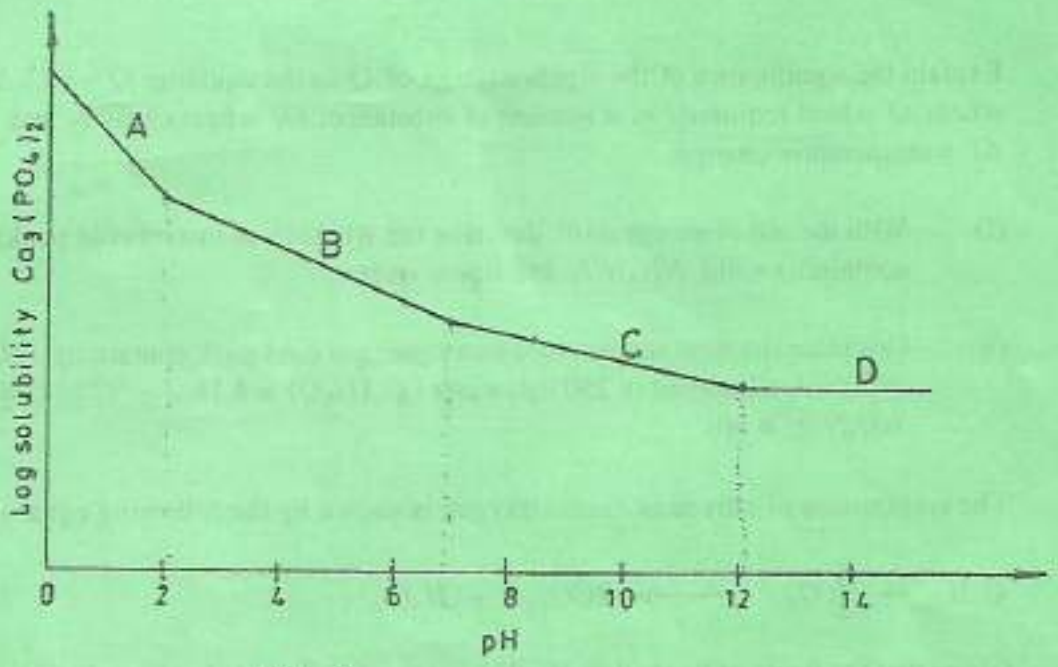

Fig. 1

7. Draw a labelled experimental set-up describing the transfer of supernatant to the filter paper cone during filtration of precipitation. (4 marks)

8. (a) Define peptization. (2 marks)

(b) Describe the method used to minimize peptization of precipitate in gravitmetry. (2 marks)

9. A 0.4960 g sample of $CaCO_3$ was dissolved in an acidic solution and the calcium precipitated as $CaC_2O_4.H_2O$. Determine the percentage of CaO in the sample given that 0.618 g of the precipitate was obtained after drying ($CaO = 56.08$, $CaC_2O_4.H_2O = 146.2$). (4 marks)

10. Compare the molar heat capacities of gold and iron given that the specific heat capacities of gold and iron are 0.128 $JK^{-1}g^{-1}$ and 0.451 $JK^{-1}g^{-1}$, respectively.
($Fe = 55.85$, $Au = 196.97$). (4 marks)

SECTION B (60 marks)

*Answer any **THREE** questions from this section.*

11. (a) Explain the significance of the algebraic sign of Q in the equation $Q = mC_p\Delta T$, where Q = heat required, m = amount of substance, C_p = heat capacity and ΔT = temperature change. (4 marks)

(b) (i) With the aid of an equation, describe the working of instant cold packs containing solid NH_4NO_3 and liquid water. (5 marks)

(ii) Calculate the final temperature in a squeezed cold pack containing 100 g of NH_4NO_3 dissolved in 250 mL water ($C_p(H_2O) = 4.18\ J\,g^{-1}C^{-1}$, $T_i = 25\ °C$, $NH_4NO_3 = 80$). (7 marks)

(c) The combustion of ethyne in excess oxygen is shown by the following equation:

$$C_2H_{2(g)} + \frac{5}{2}O_{2(g)} \longrightarrow 2CO_{2(g)} + H_2O_{(l)}$$

Given that the heats of formation of ethyne gas, carbon dioxide gas and water liquid are 277 kJ/mol, -393.5 kJ/mol and -285.8 kJ/mol, respectively, determine the heat of combustion of ethyne. (4 marks)

12. (a) (i) With the aid of a mathematical expression, describe the supersaturation as used in gravimetry. (4 marks)

(ii) Explain why homogeneous precipitation is a preferred method in gravimetry. (4 marks)

(b) Draw a labelled model of the solid-solution interface of a AgCl particle in a solution containing excess silver nitrate. (7 marks)

(c) Determine the solubility of silver chloride dissolved in 200 mL water yielding an initial concentration of 0.006 M solution. (5 marks)
($K_{sp} = 1.8 \times 10^{-10}$)

13. (a) (i) Name the parts labelled **K, L, M, N** and **P** in the filtration set-up shown in figure 2. (5 marks)

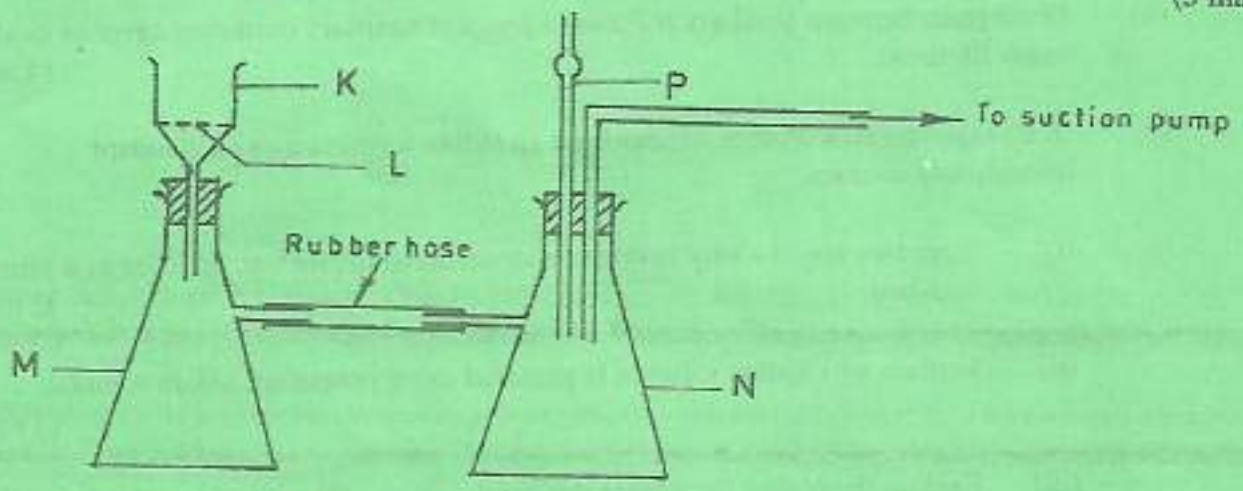

Fig. 2

(ii) State the importance of the vessel labelled **N**. (2 marks)

(b) (i) Describe occluded impurity as used in precipitation gravimetry. (4 marks)

(ii) State the process used in removing occluded impurity. (2 marks)

(c) A 0.2650 g sample of hydrated barium halide salt $BaX_2.2H_2O$ was dissolved in 200 mL water and excess sulphuric acid added to yield $BaSO_4$ precipitate. Determine the identity of X given that 0.2533 g of the substance was obtained after the gravimetric process (Ba = 137.34, S = 32.06, O = 16) (7 marks)

14. (a) State two conditions required for a complexometric reaction to be used as a volumetric technique. (2 marks)

(b) Outline the process of preparing a 0.25 M sodium thiosulphate solution in a 500 mL volumetric flask (Na = 22.99, S = 32.06, O = 16.00). (6 marks)

(c) (i) Draw the structure of a fully deprotonated EDTA molecule. (2 marks)

(ii) Use asterix (*) to show all the coordinate points in the structure in c(i) above. (3 marks)

(d) A 5.613 g sample of dried egg shell was dissolved in 25.0 mL of 6 M HCL solution. After filtration, the solution was transferred to a 250 mL volumetric flask and topped up to the mark. Given that a 10.0 mL aliquot required exactly 44.11 mL of 0.04988 M EDTA to reach end point, determine the percentage (w/w) of Ca in the egg shell. (7 marks)

15. (a) Define formal potential as used in redox titrations. (2 marks)

(b) Distinguish between auxiliary reducing agent and auxiliary oxidizing agent as used in redox titrations. (4 marks)

(c) In an experiment, a student standardized an iodine solution using a standard thiosulphate solution.

(i) State two reasons why hydrated sodium thiosulphate was not used as a primary standard. (2 marks)

(ii) Explain why iodine solution is prepared using potassium iodide solution. (3 marks)

(iii) Explain the reason for adding the starch indicator close to the end point. (3 marks)

(d) Consider the following two half reactions:

$$S_4O_6^{2-}{}_{(aq)} + 2e^- \longrightarrow 2S_2O_3^{2-}{}_{(aq)}$$

$$I_{2(aq)} + 2e^- \longrightarrow 2I^-{}_{(aq)}$$

(i) Construct the full ionic redox equation for the reaction. (2 marks)

(ii) 25.0 cm³ of an iodine solution in potassium iodide required 26.5 cm³ of 0.0950 M sodium thiosulphate solution to reach end point. Determine the molarity of the iodine solution. (4 marks)

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