2915/106
ANALYTICAL CHEMISTRY PRACTICAL I
June/July 2019
Time: 4 hours

THE KENYA NATIONAL EXAMINATIONS COUNCIL

DIPLOMA IN ANALYTICAL CHEMISTRY

MODULE I

ANALYTICAL CHEMISTRY PRACTICAL I

4 hours

INSTRUCTIONS TO CANDIDATES

You should have a battery operated scientific calculator for this examination.
*Answer **ALL** the questions in the answer booklet provided.*
Maximum marks for each part of a question are indicated.
Candidates should answer the questions in English.

This paper consists of 7 printed pages.

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

1. A. You are provided with:

- 0.25 M standard solution of oxalic acid.
- Acidified $KMnO_4$ solution.
- Iron-steel wool solution.
- $2MH_2SO_4$.
- Filter papers, labels.
- Filter funnel.
- Stand, Bunsen burner.
- 100 g zinc dust AR.
- 2 burettes (0 - 50 cm^3).
- 2 pipettes (25 cm^3).
- 1,000 cm^3 of de-ionized water.
- 5 conical flasks - pyrex glass.
- Pipette fillers (2).

B. You are required to:

- standardise the acidified $KMnO_4$;
- determine the percentage purity w/w of the kitchen iron - wool (or steel wool).

C. **PROCEED AS FOLLOWS:**

PART I

(i) Fill the burette with the acidified $KMn0_4$ solution.

(ii) Pipette 25 cm^3 of the oxalic acid solution and heat gently in a pyrex glass conical flask until it boils, titrate while hot unit a pinkish colouration permanently appears.

Repeat the experiment three more times and tabulate the results. (8 marks)

(iii) I. Write an ionic equation of the reaction taking place during the titration. (1 mark)

$2MnO_4^{2-} + 5C_2O_4^{2-} + 16H^+ \longrightarrow 2Mn^{2+} + 8H_2O + 10CO_2$

II. Calculate the molarity of the $KMn0_4$ solution. (3 marks)

PART II

(i) Fill the burette with the standardised $KMn0_4$ solution.

(ii) Pipette 25 cm^3 of the freshly digested iron-wool solution into a clean conical flask.

23.7, 23.8 23.7

(iii) Titrate the iron wool solution with the $KMn0_4$ until a permanent colour change appears. Repeat the experiment three more times and tabulate the results. (8 marks)

(iv) Write an ionic equation of the reaction taking place during the titration. (2 marks)

(v) Calculate the percentage purity of the kitchen - iron-wool sample w/w. (5 marks)

(vi) With the aid of equations, explain why it is necessary to have the iron-wool solution freshly prepared. (3 marks)

(vii) Explain why the purity of the iron-wool is not 100% w/w. (3 marks)

2. A. You are provided with:

- A mixture of Na_2CO_3 and $NaHCO_3$ labelled as "Aqueous mixture".
- A solution of dilute HCl.
- A standard solution of 0.05 M Na_2CO_3.
- Methyl orange indicator.
- Phenolphthalein indicator.
- Burette (0 - 50 cm^3)
- Pipette (25 cm^3)
- Labels.
- 6 conical flasks.
- Stand.

B. You are required to:

- standardise the dilute HCl solution;
- determine the molarity of Na_2CO_3 and $NaHCO_3$ in the mixture.

C. **PROCEED AS FOLLOWS:**

PART I

(i) Fill the burette with the dilute HCl solution.

(ii) Pipette 25 cm^3 of the standard Na_2CO_3 solution into a clean conical flask and add 3 drops of methyl orange indicator and titrate until a permanent colour change appears. Repeat the experiment three more times and tabulate the results. (8 marks)

(iii) Calculate the molarity of the HCl solution. (2 marks)

PART II

(i) Pipette 25 cm^3 of the aqueous mixture solution into a clean conical flask and add 3 drops of phenolphthalein indicator.

(ii) Fill the burette with the standardised solution of HCl. Titrate the mixture until a permanent colour change appears. Repeat the experiment three more times and tabulate the results. (8 marks)

(iii) Write an equation of the reaction taking place during the titration. (1 mark)

(iv) Calculate the molarity of the substance reacting with the HCl during the titration. (2 marks)

(v) Fill the burette with the HCl solution.

(vi) Pipette 25 cm^3 of the aqueous mixture solution into a clean conical flask and add 3 drops of methyl orange indicator. Titrate the mixture with the acid until a permanent colour change occurs. Repeat the experiment three more times and tabulate the results. (8 marks)

(vii) Write equations for the reactions taking place during the titration. (2 marks)

(viii) Calculate the molarity of the second component of the mixture of $NaCO_3/NaHCO_3$. (3 marks)

3. **PART A**

You are provided with the following:

- Solutions **A**, **B** and **C** containing cations of group II metals.
- 1 M sodium hydroxide.
- 1 M ammonia solution.

You are required to use $NaOH$ and NH_4OH solutions to test and identify the group 2 ions contained in the solutions **A**, **B** and **C**.

PROCEDURE I

(i) Pour half of solution **A** into an empty test tube.

(ii) To the first half, add a few drops of the 1 M $NaOH$ solution. Record your observations in table I.

(iii) Add an excess of the 1 M $NaOH$ solution (about 20 drops) to the test tube. Record your observations in the table I.

(iv) Repeat procedures (i) to (iii) for solutions **B** and **C** and record observations in the table I.

Table I

Unknown solution	OBSERVATIONS	
	Addition of a few drops of *NaOH*	Addition of excess *NaOH*
A	(1 mark)	(1 mark)
B	(1 mark)	(1 mark)
C	(1 mark)	(1 mark)

(v) To the second half of solution **A**, add a few drops of 1 M NH_4OH solution. Record your observations in table II.

(vi) Add an excess of NH_4OH solution (About 20 drops) to the test tube. Record your observations in table II.

(vii) Repeat procedures (v) and (vi) for unknown solutions **B** and **C** and record your observations in table II.

Table II

Unknown solution	OBSERVATIONS	
	Addition of a few drops of *NaOH*	Addition of excess *NaOH*
A	(1 mark)	(1 mark)
B	(1 mark)	(1 mark)
C	(1 mark)	(1 mark)

PART B

You are provided with the following:

- Unknown solids **A**, **B** and **C**.
- Platinum wire/nicrome wire.
- Concentrated HCl.

PROCEDURE II

(i) Clean the platinum wire by dipping it in concentrated HCl in a test tube.

(ii) Heat the platinum wire strongly in the flame until the wire imparts no colour to the flame.

(iii) Dip the wire into concentrated HCl in a test tube/ watch glass and then into a little solid **A**, so that the some solid **A** sticks to the wire.

(iv) Introduce the wire to the flame and note the colour imparted to the flame.

(v) Record the observation in table III provided.

(vi) Repeat procedures / steps (i) to (v) for solids **B** and **C**.

Table III

Unknown solid	Observation	Inference
A	(1 mark)	(1 mark)
B	(1 mark)	(1 mark)
C	(1 mark)	(1 mark)

(vii) Using the observations in procedure I and II, identify the group II ions in the solutions **A**, **B** and **C**. (5 marks)

PART C

You are provided with organic compounds labelled **X** and **Y**. You are required to carry out the following tests and complete table IV:

Table IV

		PROCEDURE	OBSERVATION	INFERENCE
A	(i)	Place a drop of compound **X** on moist blue litmus paper	(1 mark)	(1 mark)
	(ii)	Take about 1 ml of compound **X** in a test tube and add a few drops of sodium hydrogen carbonate	(1 mark)	(1 mark)
	(iii)	Place about 1 ml of compound **X** into a test tube, add 1 ml of ethyl alcohol and 1 - 2 drops of concentrated sulphuric acid. Heat the mixture on a water bath for about 5 minutes. Pour the mixture into a beaker containing cold water.	(1 mark)	(1 mark)
B	(i)	Add a few drops of bromine water to **Y** and shake well.	(1 mark)	(1 mark)
	(ii)	Dissolve a little of compound **Y** in about 2 ml of distilled water in a test tube and add a few drops of alkaline solution of $KMnO_4$ to it and shake well.	(1 mark)	(1 mark)

THIS IS THE LAST PRINTED PAGE.