COMPREHENSIVE CHEMISTRY PRACTICALS

A STORY-DRIVEN, COMPETENCY-BASED GUIDE TO PRACTICAL MASTERY

With Real-Life Applications and Investigative Scenarios

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PREFACE

Chemistry is often called the central science — and for good reason. It bridges the physical and life sciences, explains the mysteries of everyday materials, and fuels the innovation behind everything from medicine to clean energy. But for many students, practical chemistry remains a blurry maze of lab coats, equations, and confusion. This book is here to change that.

What if every experiment was a story?

What if a titration wasn't just a reaction in a flask, but a thrilling encounter between a peacekeeping base and a fiery acid? What if you could walk into a lab and feel the tension of an ionic investigation, the drama of a disappearing mark, or the victory of finding the right concentration?

Welcome to ***Comprehensive Chemistry Practicals*** — where real scientific skills meet **engaging narratives**, **critical thinking**, and **competency-based learning**.

Inside, you'll meet recurring characters like **Kipute**, *a curious and courageous student with a knack for uncovering chemical truths*, and **Mr. Akilikubwa**, *the brilliant, slightly eccentric lab technician with wisdom (and warnings) to spare*. Through their stories, every experiment unfolds with purpose and imagination while reinforcing the skills and calculations needed to excel in exams and in real world applications.

This book explores a broad spectrum of essential chemistry practicals, categorized into three major sections: volumetric analysis, physical chemistry analysis, and qualitative analysis. The volumetric analysis section focuses on acid-base and redox titrations, while physical chemistry analysis is further divided into partition experiments, chemical kinetics, and thermochemistry.

Whether you're a form six student preparing for your final exams, a teacher looking to spice up your lab sessions, or just a chemistry lover who enjoys a good story, this book is your companion in mastering practical chemistry with confidence and joy.

So grab your goggles, steady your pipette, and dive in.

Let the experiments begin — and may the reactions always be balanced!

Acknowledgements

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Finally, I sincerely thank the dedicated **NEH team** for their tireless work in typing, editing, and designing the final version of this book. Their professionalism helped turn a vision into a reality.

Ngaiza Lusima.

PART ONE

VOLUMETRIC ANALYSIS

Chapter 1

INTRODUCTION TO VOLUMETRIC ANALYSIS

INTRODUCTION

Volumetric analysis is a part of quantitative analysis which depends on accurate measurements of volumes of solutions. This is commonly done by using titration method.

Titrations are done often to find out the concentration of one substance by reacting it with another substance of known concentration (**standard solution).** They are done with either neutralisation reactions in **acid-base titrations** or redox reactions in **redox titrations**.

Standard solution

Standard solution *is the solution of known concentration in the titration.* It is also known as **titrant** or **titrator**. During titration, the standard solution is usually kept in the burette.

Substance whose standard solution can be prepared directly (by either dissolving its definite mass in a specified volume of the solution or merely diluting its concentrated stock solution) is called **primary standard substance**. These substances are stable and they exist in pure form. Common examples of primary standard substances are:

- ✓ Anhydrous sodium carbonate
- ✓ Oxalic acid (ethanedioic acid)
- ✓ Sodium hydrogencarbonate
- ✓ Potassium dichromate
- ✓ Potassium iodate

A substance will be classified as a primary standard reagent, if it possesses the following characteristics:

1. It is easily available in state of high purity.
2. It is neither hygroscopic nor deliquescent.
3. It shows high solubility in water.
4. It does not dissociate or decompose during storage.
5. It does not get easily oxidised by air.
6. It should react instantaneously with another substance in stoichiometric proportion.
7. It has large molar mass (so as to reduce the effect of weighing error).

On contrast to standard solution, **Analyte** or **titrand** *is the solution of unknown concentration in the titration.* During the titration, the analyte is usually put in the conical flask.

Substances whose standard solutions cannot be prepared directly are called **secondary standard substances**. These substances lack one or more characteristics of primary standard substances (generally they are impure and unstable). Examples of primary standard substances are:

- ✓ Sodium hydroxide
- ✓ Potassium hydroxide
- ✓ Hydrochloric acid
- ✓ Sodium thiosulphate
- ✓ Potassium permanganate

Solutions of secondary standard reagents are usually not standard. So standard solutions of these substances are made indirectly by titrating their solutions against solution of some primary standard substance (followed by dilution to prepare the exact intended concentration).

HANDLING TITRATION EXPERIMENT

In the titration, titrant (known concentration but unknown reacting volume) is often put in the **burette** while analyte (known reacting volume but unknown concentration) is often put in the **conical flask**. The volume of the solution which is placed in the conical flask is measured by using a **pipette**. The standard phrase: titrate solution A with solution B means that A should be in the conical flask and B should be in the burette.

Getting correct volume in the titration is crucial because even small error may result into false conclusion. The following tips will help you to measure volume accurately and get correct result in the titration experiment.

Practical tip 1: *Pipette and burette are rinsed by their respective solutions which are going to be put in it while conical flask is rinsed by using distilled water.*

Practical tip 2: *In filling the pipette, make sure that the bottom of the meniscus sits on the line which shows the level to fill to.*

Practical tip 3: *Do not force out the small amount of the solution from the jet end of the pipette. The calibration of the pipette will take into account the effect of this small amount which is left in the pipette.*

Practical tip 4: *Care must be taken that no air bubbles remain in the narrow bottom tip of the burette. To remove this air, the stop-cock is opened and the liquid is allowed to run out rapidly into the beaker or flask.*

Practical tip 5: *Remove the funnel immediately after filling the burette.*

Practical tip 6: *In running the solution from burette into the titration flask make sure the solution falls directly into the solution of titration flask. It should not fall on the walls of the flask.*

Practical tip 7: *Use pilot experiment to obtain rough titre volume. Thereafter, in the next experiments add solution from burette while constantly swirling the mixture and near the end point ($1cm^3$ less than the rough titre volume of the pilot experiment) start to add the solution* ***drop-wise*** *(add a drop, close the pinch cock, shake and find out if the end point has been attained).*

Practical tip 8: *In reading volume of colourless solutions lower meniscus is read, while in case of coloured solutions, level is read from the upper meniscus. This is due to the reason that in case of coloured solutions lower meniscus is not visible clearly.*

Practical tip 8: *Let no drops of solution be hanging at the tip of the burette at the end point.*

Practical tip 9: *Only few drops (very small amount) of indicator should be added. Use a white tile underneath the flask to help observe the colour change.*

Practical tip 10*: Repeats titration until* ***three concordant results*** *(three readings within $0.10cm^3$ of each other.) are obtained.*

Practical tip 11: Titre volumes must be recorded to two decimal places and the mean titre volume is recorded to nearest $0.05cm^3$.

Interesting trick!

Check the correctness of the end point by adding one drop of analyte (with the help of a pipette) into the titration flask. Restoration of original colour confirms the correctness of the end point.

WAYS OF REPRESENTING CONCENTRATION OF SOLUTION

In doing experiments involving volumetric analysis and many other chemistry experiments, solutions are usually used. So understanding methods of representing their concentrations is very crucial in mastering not only volumetric analysis but quantitative analysis in general.

The concentration can be expressed either qualitatively which is the subjective measure or quantitatively which is objective measure. For example, qualitatively we can say that the solution is **dilute** (*contain very small amount of solute compared to the amount of solvent)* or it is **concentrated** (*contain very large amount of solute compared to that of solvent*). But in doing quantitative analysis (volumetric and physical chemistry analysis), these kinds of description are subjective and thus they can add to lot of confusion and hence the need for objective approach which is a quantitative description of the solution.

In this section, we are going to discuss few ways which are commonly employed to describe the concentration of the solution quantitatively in volumetric analysis experiments.

Mass concentration

This is the mass of the solute dissolved in a unit volume of solution.

That is: Mass concentration = $\frac{\text{Mass of solute}}{\text{Volume of solution}}$

Mass concentration is commonly given in g/dm^3,

Other units are g/cm^3, g/ml etc.

Molar concentration (molarity)

This is number of moles of solute dissolved in a litre of solution.

Thus Molarity $= \frac{\text{number of moles of solute}}{\text{volume of solution in litres (or dm}^3)}$

It is denoted by two square brackets, [ ]

Its unit is mol/L (or mol/dm^3) or M where $1\text{M} = 1\text{mol/dm}^3$

The reader should understand that: A molar solution is commonly used to mean that: One mole of solute is dissolved in a litre of solution.

Remember: 1dm^3 = 1Litre = 1000cm^3

Relationship between molarity and mass concentration

From Molarity $= \frac{\text{number of moles of solute}}{\text{volume of solution in dm}^3}$

But number of moles of solute = $\frac{\text{mass of solute}}{\text{molar mass of solute}}$

Then: Molarity $= \frac{\text{mass of solute}}{\text{volume of solution in dm}^3 \times \text{molar mass of solute}}$

$\frac{\text{mass of solute}}{\text{volume of solution in dm}^3}$ = Mass of concentration in g/dm^3

Hence: Molarity $= \frac{\text{mass concentration in g/dm}^3}{\text{molar mass of solute in g/mol}}$

Or mass concentration in g/dm^3 = molarity × molar mass of solute in g/mol.

Molar concentration is the most common method of representing concentration; so when it is stated just 'concentration', it always implies molar concentration.

Mass percentage $\left(\%\left(\frac{\mathbf{m}}{\mathbf{m}}\ \mathbf{or}\ \frac{\mathbf{w}}{\mathbf{w}}\right)\right)$

This is the mass of solute in grams dissolved in 100g of solution.

Thus $\%\left(\frac{\text{m}}{\text{m}}\right) = \frac{\text{mass of solute in g}}{\text{mass of solution in g}} \times 100\%$

The mass percentage is the most common method of representing concentration as percentage, so whenever we talk (or you asked to find) just percentage without any extra information, the implication will always be mass percentage.

Relationship between mass percentage and mass concentration

From $\%\left(\frac{\text{m}}{\text{m}}\right) = \frac{\text{mass of solute in g}}{\text{mass of solution in g}}$

Where $\%\left(\frac{\text{m}}{\text{m}}\right)$ has been converted to fraction by dividing the percentage by 100 so that there is no need of multiplying the right hand side of the equation by 100 as shown on original formula.

But mass of solution = Density of solution × volume of solution

(From Density = $\frac{\text{mass}}{\text{volume}}$; mass = Density × volume).

Then:

$\%\left(\frac{\text{m}}{\text{m}}\right) = \frac{\text{mass of solute in g}}{\text{volume of solution} \times \text{Density of solution}}$

But $\frac{\text{mass of solute in g}}{\text{volume of solution}}$ = mass concentration

Then $\%\frac{\text{m}}{\text{m}} = \frac{\text{mass concentration}}{\text{Density of solution}}$

Hence: Mass concentration = Density of solution $\times\%\left(\frac{\text{m}}{\text{m}}\right)$

Where mass percentage, $\%\left(\frac{\text{m}}{\text{m}}\right)$ must be converted to fraction by dividing the percentage by 100.

Normality

This is the number of equivalents of solute dissolved in one litre of the solution.

Thus: $\text{Normality} = \frac{\text{number of euivalents of solute}}{\text{volume of solution in litres (or dm}^3\text{)}}$

Its unit is N

Relationship between normality and molarity

From $\text{Normality} = \frac{\text{number of equivalents of solute}}{\text{volume of solution dm}^3}$

But number of equivalents of solute $= \frac{\text{mass of solute}}{\text{Equivalent weight of solute}}$

Then: $\text{Normality} = \frac{\text{mass of solute}}{\text{volume of solution in dm}^3 \times \text{equivalent weight of solute}}$

But $\frac{\text{mass of solute}}{\text{volume of solution in dm}^3} =$ mass concentration of solute in g/dm^3

Thus: $\text{Normality} = \frac{\text{mass concentration in g/dm}^3}{\text{equivalent weight of solute}}$

For acid-base reaction:

Equivalent weight = $\frac{\text{molar mass}}{\text{acidity or basicity}}$

Then: Normality = $\frac{\text{mass concentration in g/dm}^3 \times \text{Basicity or Acidity}}{\text{molar mas of solute}}$

But $\frac{\text{mas concentration in g/dm}^3}{\text{molar mass of solute}}$ = molarity

Hence for acid – base reaction

Normality = Molarity × Basicity (or Acidity)

For redox reactions:

Equivalent weight = $\frac{\text{molar mass}}{\text{number of electrons transfered (by one molecule of reductant or oxidant)}}$

Then from Normality = $\frac{\text{mass concentration in g/dm}^3}{\text{equivalent weight of solute}}$

It becomes:

Normality = $\frac{\text{mass concentration in g/dm}^3 \times \text{number of electrons transfered}}{\text{Molar mass of solute}}$

But $\frac{\text{mass concentration in g/dm}^3}{\text{molar mass of solute}}$ = Molarity

Hence for redox reactions: Normality = Molarity × Number of electrons transferred (**by one molecule of reductant or oxidant**)

The term **'A normal solution'** is used to express the solution of 1N

Relationship between normality of acid and base in acid – base reaction

For Acid – base reaction

$\frac{M_aV_a}{M_bV_b} = \frac{n_a}{n_b}$……………………………………(i)

Where;

M_a is the molarity of acid

M_b is the molarity of base

V_a is the volume of acid

V_b is the volume of base

n_a is the stochiometric coefficient of acid in a balanced chemical equation for the reaction

n_b is the stochiometric coefficient of base in a balanced chemical equation for the reaction

Then:

$M_an_bV_a = M_bn_aV_a$ (By rearranging equation (i) above)

But $n_b =$ Basicity of acid

And $n_{a=}$ Acidity of acid

So $M_an_b = N_a$ (Normality for acid)

And $M_bn_a = N_b$ (Normality for base)

Hence: $N_aV_a = N_bV_b$

Similarly, for redox reaction;

$N_oV_o = N_rV_r$

Where N_0 and N_r is the normality of oxidant and reductant respectively.

V_o and V_r is the volumes of oxidant and reductant respectively.

The final result is known as **law of equivalents** which may be stated as; *the number of equivalents of the titrand is equal to the number of equivalents of the titrant used.*

$$\mathbf{N_1V_1 = N_2V_2}$$

DILUTION PRINCIPLE

Dilution principle may be stated as:

Concentration of the solution containing fixed mass (or number of moles) of the solute varies inversely to its volume.

That is $M \propto \frac{1}{V}$ or MV = constant (n is constant)

From which $M_1V_1 = M_2V_2 = M_3V_3 = \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots = M_nV_n$

To have better understanding this, suppose we have the following:

M_c = Molar concentration of concentrated solution.

M_d = Molar concentration of dilute solution.

V_c = Volume of the concentrated solution used to prepare the diluted solution of concentration, M_d

V_d = Volume of dilute solution obtaining after mixing pure water and the concentrated solution $(V_d = V_c + V_{H_2O})$

Using $[\quad] = \frac{n}{V}$ or $n = V[\quad]$

Number of moles of the solute in the concentrated solution = M_cV_c

Number of moles of the solute in the diluted solution = M_dV_d

Since the dilution is done by adding pure water (which has no solute in it), number of moles of solute in the concentrated solution and diluted solution remains constant and hence;

$M_cV_c = M_dV_d$ (mathematical form of dilution principle)

So in other words, dilution principle may be stated as: *If the concentrated solution is diluted by adding more solvent, the number of moles of solute remains constant.*

Chapter 2

ACID-BASE TITRATIONS

INTRODUCTION

An **acid-base titration** *is the determination of the concentration of an acid or base by exactly neutralising the acid/base with a base or acid of known concentration.* It makes use of **neutralisation reaction** that occurs between acids and bases and the knowledge of how acids and bases will react if their formulas are known.

Acid-base titration may be **alkalimetry** or **acidimetry**. **Alkalimetry,** (sometimes spelled **alkimetry**), *is the specialized analytic use of acid – base titration to determine the concentration of a basic substance.* Thus in alkalimetry, the analyte is the basic substance. Whereas, a**cidimetry**, (sometimes spelled **acidometry**), *is the specialized analytic use of acid – base titration to determine the concentration of an acidic substance.* Thus in acidimetry, the analyte is the acidic substance.

End points of acid-base titrations are detected by pH indicators which are very weak organic acids. The most common indictors are **phenolphthalein (POP)** and **methyl orange (MO).**

Phenolphthalein is used in the titration which involves strong base. If an acid is added from the burette, its colour changes from pink to colourless; that is, at end point pink colour just disappears.

Methyl orange is used in the titration which involves strong acid. Again, if an acid is added from the burette, its colour changes from yellow to orange at the end point.

Acid-base titrations may involve single indicator or double indicators. In this book, we are going to focus more on titration by double indicators.

Titration by double indicators

Titration by using two different indicators can be used in:

- Estimation of a mixture of sodium hydroxide and sodium carbonate
- Estimation of a mixture of sodium carbonate and sodium hydrogencarbonate
- Estimation of a mixture of hydrochloric acid and ethanoic acid

1. Estimation of a mixture of sodium hydroxide and sodium carbonate by double indicators

This is done by titrating the solution mixture with solution of hydrochloric acid.

The reaction between sodium hydroxide and hydrochloric acid occurs in a single step according to the following equation:

$\mathrm{NaOH + HCl \longrightarrow NaCl + H_2O}$…………………………..… (i)

Where NaOH and HCl being strong base and strong acid respectively, any indicator (**methyl orange** or **phenolphthalein**) can be used to determine the end point of reaction (i).

The reaction between sodium carbonate and hydrochloric acid occurs in two steps, with the hydrogen carbonate ion as intermediate product.

$\mathrm{Na_2CO_3 + HCl \rightarrow NaCl + NaHCO_3}$ ………………….. (ii)

Then, $\mathrm{NaHCO_3 + HCl \rightarrow NaCl + H_2O + CO_2}$…………………. (iii)

Where:

- Na_2CO_3 and HCl being strong base and strong acid respectively, either methyl orange or phenolphthalein indicator can be used to determine the end point of reaction (ii).
- $NaHCO_3$ being weak base and HCl being strong acid, only methyl orange indicator is suitable for determination of end point of reaction (iii).

- Since mole ratio of Na_2CO_3 to HCl, $NaHCO_3$ to HCl as well as Na_2CO_3 to $NaHCO_3$ is 1:1, the amount (volume) of the acid (HCl) required for reaction (ii) and (iii) is the same.

Conclusion….!

- With **phenolphthalein** as indicator, the end point is registered at the completion of **reaction (i)** and **(ii)**.
- With **methyl orange** as an indicator, the end point is registered at the completion of **all three reactions**.
- Since the amount of acid required for reaction (ii) and (iii) is the same, and **the amount of acid used in the reaction (iii) is the difference between the titrations with methyl orange and phenolphthalein, it follows that twice this difference is the amount of acid which titrates the whole carbonate,** Na_2CO_3, according to equation,

$$Na_2CO_3 + 2HCl \rightarrow 2NaCl + H_2O + CO_2.$$

- The rest titrates the sodium hydroxide.

2. **Estimation of a mixture of sodium carbonate and sodium hydrogen carbonate by double indicators**

The same concept of the first case can be applied in this case as follows:

- With **phenolphthalein as indicator**, the end point is given when all the carbonate is converted to hydrogen carbonate according to the following equation.

$Na_2CO_3 + HCl \rightarrow NaHCO_3 + NaCl$ ……………(i)

- With **methyl orange as indicator**, the end point is given when the original hydrogen carbonate and that produced in (i) as an intermediate product are both converted to CO_2 and H_2O according to the following equation:

Intermediate $NaHCO_3$ from (i), $NaHCO_3 + HCl \rightarrow NaCl + H_2O + CO_2$ ….(ii)

Original $NaHCO_3$ in the mixture, $NaHCO_3 + HCl \rightarrow NaCl + H_2O + CO_2$.... (iii)

That is the end point with **methyl orange indicator is** obtained after completion of **all three reaction (i), (ii), (iii)** while that of **phenolphthalein indicator** is obtained after reaction (i) only.

- Since the amount of acid required for reactions (i) and (ii) is the same, **twice the phenolphthalein titration is the amount of which titrates the carbonate,** Na_2CO_3, according to equation: $Na_2CO_3 + 2HCl \rightarrow 2NaCl + H_2O + CO_2$

- **The rest titrates the original hydrogencarbonate** ($NaHCO_3$) which was originally present in the mixture with Na_2CO_3.

3. **Estimation of a mixture of hydrochloric acid and ethanoic acid by double indicators**

This is done by titrating the solution mixture with solution of sodium hydroxide.

The reaction between sodium hydroxide and hydrochloric acid occurs in a single step according to the following equation:

$$NaOH + HCl \longrightarrow NaCl + H_2O \text{………………………….. (i)}$$

Where NaOH and HCl being strong base and strong acid respectively, any indicator (**methyl orange** or **phenolphthalein**) can be used to determine the end point of reaction (i).

The reaction between sodium hydroxide and ethanoic acid also occurs in a single step according to the following equation:

$$NaOH + CH_3COOH \rightarrow CH_3COONa + H_2O \text{ ………………….. (ii)}$$

Where:

CH_3COOH being weak acid and NaOH being strong base, only **phenolphthalein** indicator is suitable for determination of end point of reaction (ii).

Conclusion….!

- With **methyl orange** as indicator, the end point is registered at the completion of **reaction (i)** only.
- With **phenolphthalein** as an indicator, the end point is registered at the completion of **all two reactions**.
- The difference of the two volumes, is the volume of NaOH used to titrates ethanoic acid (for reaction (ii) only).

EXPERIMENTS

Experiment 1

Kipute, a curious A-Level student and aspiring chemical analyst, was helping her uncle organize the family's small laboratory**.** Among the labeled chemicals, she discovered a small container labelled **"Unknown Carbonate Sample – M_2CO_3"**.

Eager to uncover the identity of the metal "M" in the compound, she decided to conduct a titration experiment.

She carefully weighed **2.12 grams** of the unknown **monovalent metal carbonate (M_2CO_3)** and dissolved it in exactly **250 cm^3** of distilled water to prepare **Solution A**. For the titration, she selected **0.25 M hydrochloric acid** (**Solution B**) and **methyl orange** as the indicator.

Titration procedure Kipute followed:

1. She pipetted **20 cm^3** of **Solution A** into a clean titration flask.
2. She added **2–3 drops** of **methyl orange (MO) indicator**.
3. She filled the **burette** with **solution**.
4. She titrated **Solution A** with **Solution B** until the colour change is observed.
5. She recorded the **titre volumes** carefully and repeated the titration for accuracy.

Fill the titration table below and answer the questions that follow:

Experiment	Pilot	1	2	3
Final reading (cm^3)				
Initial reading (cm^3)				
Titre value (cm^3)				

Questions

a) Calculate the number of moles of:

(i) Solution A used in the reaction

(ii) Solution B used in the reaction

b) Determine the relative atomic mass of the metal "M" in a metal carbonate
c) Identify the unknown metal 'M' in the compound M_2CO_3.
d) Explain why methyl orange is a suitable indicator for this titration.
e) Suggest three ways Kipute could improve the accuracy or reliability of her titration results.

Solution

The volume of the pipette used was 20cm^3

TABLE OF RESULTS

Experiment	Pilot	1	2	3
Final reading (cm^3)	13.00	12.90	12.80	25.60
Initial reading (cm^3)	0.00	0.00	0.00	12.80
Titre value (cm^3)	13.00	12.90	12.80	12.80

Average volume used $= \frac{V_1 + V_2 + V_3}{3} = \left(\frac{12.90 + 12.80 + 12.80}{3}\right) \text{cm}^3$

Average volume used $= 12.80\text{cm}^3$

Summary:

20cm^3 of A required **12.80cm^3** of B for complete neutralization.

For 25 mL pipette: Volume of B will be **16.00cm^3**.

Making Sense of the Experimental Data

Practical tip: *The monovalent metal carbonate which is commonly used in acid-base titration is only Na_2CO_3.*

The known value of atomic mass of Na is 23, making the molar mass of Na_2CO_3 to be 106g/mol.

It follows that: $[A] = \frac{m_A}{M_A \times V_{soln} \text{ in L}} = \frac{2.12g}{106g/mol \times 0.25L} = 0.08M$

Then from the reaction equation;

$$Na_2CO_3 + 2HCl \rightarrow 2NaCl + H_2O + CO_2;\ n_{acid} = 2 \text{ and } n_{base} = 1$$

Using; $\frac{M_{acid} \times V_{acid}}{M_{base} \times V_{base}} = \frac{n_{acid}}{n_{base}}$

Substituting; $\frac{0.25M \times V_{acid}}{0.08M \times 20cm^3} = \frac{2}{1}$; $\mathbf{V_{acid} = 12.8\ cm^3}$ = experimental data

To conclude: *The calculated volume of acid (HCl) agrees with the average volume of the acid obtained from the experiment.*

Response to Questions

a) A and B react according to the following equation:

$$Na_2CO_3 + 2HCl \rightarrow 2NaCl + H_2O + CO_2$$

Where: $n_{acid} = 2$ and $n_{base} = 1$

Using; $\frac{M_{acid} \times V_{acid}}{M_{base} \times V_{base}} = \frac{n_{acid}}{n_{base}}$

Where:

$$M_{acid} = 0.25M,\ V_{acid} = 12.8cm^3 \text{ and } V_{base} = 20cm^3$$

Substituting; $\frac{0.25\text{M}\times12.8\text{cm}^3}{\text{M}_\text{base}\times20\text{cm}^3}=\frac{2}{1}$; $\text{M}_\text{base}=0.08\text{M}$

Then, using: $\text{number of moles (n)} = \text{molarity (M)} \times \text{volume(V)}$

(i) $\text{Number of moles of A} = 0.08\text{mol/dm}^3 \times 0.02\text{dm}^3 = 1.6 \times 10^{-3}\text{mol}$
(ii) $\text{And number of moles of B} = 0.25\text{mol/dm}^3 \times 0.0128\text{dm}^3 = 3.2 \times 10^{-3}\text{mol}$

b) Using;

$$\text{Molarity (M)} = \frac{\text{Mass concentration}}{\text{Molar mass}} \text{ or Molar mass of } = \frac{\text{Mass concentration}}{\text{Molarity}}$$

Where: $\text{mass concentration of } M_2CO_3 = \frac{\text{mass (m)}}{\text{volume (V)}} = \frac{2.12\text{g}}{0.25\text{dm}^3} = 8.48\text{gdm}^{-3}$

It follows that: $\text{Molar mass of } M_2CO_3 = \frac{\text{mass concentration of} M_2CO_3}{\text{molarity of } M_2CO_3}$

$$= \frac{8.48\text{gdm}^{-3}}{0.08\text{moldm}^{-3}} = 106\text{g/mol}$$

But $\text{relative molecular mass of } M_2CO_3 = 2M + C + 3(O)$

$$106 = 2M + 12 + 3 \times 16;\ M = 23$$

Therefore; the relative atomic mass of the metal "M" of metal carbonate M_2CO_3 is 23

c) The monovalent metal with relative atomic mass of 23 is sodium.
Hence metal "M" in a metal carbonate, M_2CO_3 is **sodium**.

d) **Reason:**

This is titration which involve strong acid, hydrochloric acid (HCl).

Explanation:

The equivalence point of reaction between hydrochloric acid and sodium carbonate is obtained when the solution is acidic. Since methyl orange changes colour in the acidic solution too (pH range of 3.1–4.4), it is ideal for detecting the equivalence point of this reaction.

e) To improve accuracy and reliability, Kipute should:

1) Ensure that the burette and pipette are **rinsed properly** with the correct solutions before use.
2) Use a **white tile** beneath the flask to clearly observe the colour change.
3) **Swirl the flask consistently** during titration to mix the contents uniformly.

Experiment 2

One Tuesday morning, Mr. Akilikubwa received a sample of **sodium carbonate** labeled as **AB** which is **"Impure – 5.125 g dissolved in 250 cm³."** The school supplier claimed it was of acceptable purity, but Mr. Akilikubwa, ever the meticulous chemist, wasn't convinced.

"I'll let the titration decide," he said as he prepared the experiment.

To test the **percentage purity** of the sodium carbonate sample, he planned a titration against a known solution of sulphuric acid, prepared by dissolving **4.9 g of H_2SO_4** in **1 dm³ of distilled water** and labelled it as **AC**. Using **methyl orange** as an indicator, he would calculate just how much of the sample was truly sodium carbonate—and how much was useless impurity.

Titration procedure Mr. Akilikubwa followed

1. He pipetted **20 cm³** of **solution AB** into a clean titration flask.
2. He added **2–3 drops of methyl orange indicator**.
3. He filled the burette with **solution AC**.
4. He titrated **solution AB** against **solution AC** until a clear colour change was observed.
5. He repeated the titration to obtain three consistent readings.
6. He carefully recorded the results in the following table:

Experiment	Pilot	1	2	3
Final reading (cm^3)				
Initial reading (cm^3)				
Titre value (cm^3)				

Questions

a) Write a balanced chemical equation for the reaction between solution AB and solution AC
b) Calculate:
   i. Concentration of AC in gdm^{-3}
   ii. Concentration of AB in gdm^{-3}
c) Calculate the percentage purity of sodium carbonate sample.
d) Suggest possible reason why the sodium carbonate sample might be impure.

Solution

The volume of the pipette used was $20cm^3$

TABLE OF RESULTS

Experiment	Pilot	1	2	3
Final reading (cm^3)	20.30	40.20	20.00	40.10
Initial reading (cm^3)	0.00	20.30	0.00	20.00
Titre value (cm^3)	20.30	19.90	20.00	20.10

$$\text{Average volume used} = \frac{V_1 + V_2 + V_3}{3} = \left(\frac{19.90 + 20.00 + 20.10}{3}\right)cm^3$$

$$\text{Average volume used} = 20.00cm^3$$

Summary:

20cm³ of AB required **20.00cm³** of AC for complete neutralization.

For 25 mL pipette: The volume of AC will be **25.00cm³**.

Making Sense of the Experimental Data

All three titre values are within ±0.10 cm³ of each other, indicating high precision and hence the experiment was successful.

Response to Questions

a) $\mathrm{Na_2CO_3(aq) + H_2SO_4(aq) \longrightarrow Na_2SO_4(aq) + CO_2(g) + H_2O(aq)}$

b) Using; $\text{Mass concentration} = \frac{\text{Mass (m)}}{\text{Volume (V)}}$

Mass concentration of AC $= \frac{4.9\text{g}}{1\text{dm}^3} = 4.9\text{gdm}^{-3}$.

(i) Hence the concentration of AC in gdm^{-3} was 4.9gdm^{-3}.

Mass concentration of AB $= \frac{5.125\text{g}}{0.25\text{dm}^3} = 20.5\text{gdm}^{-3}$.

(ii) Hence the concentration of AB in gdm^{-3} was 20.5gdm^{-3}.

c) Using; $\frac{M_{acid} \times V_{acid}}{M_{base} \times V_{base}} = \frac{n_{acid}}{n_{base}}$

Where:

$M_{acid} = \frac{4.9\text{gdm}^{-3}}{98\text{gmol}^{-1}} = 0.05\text{M},\ V_{acid} = 20\text{cm}^3,\ V_{base} = 20\text{cm}^3$

And $n_{acid} = n_{base} = 1$ (From the balanced equation in (a))

Substituting; $\frac{0.05\text{M} \times 20\text{cm}^3}{M_{base} \times 20\text{cm}^3} = \frac{1}{1};\ M_{base} = 0.05\text{M}$

$\text{Mass concentration} = \text{Molarity(M)} \times \text{Molar mass}$

Where molar mass of $\mathrm{Na_2CO_3} = (2 \times 23 + 12 + 3 \times 16)\text{g/mol} = 106\text{g/mol}$

Thus; mass concentration of pure $\mathrm{Na_2CO_3} = 0.05\text{moldm}^{-3} \times 106\text{g/mol} = 5.3\text{gdm}^{-3}$

But from (b), mass concentration of impure $\mathrm{Na_2CO_3}$ (AB) was 20.5gdm^{-3}.

Hence the concentration of AB in gdm^{-3} was 5.3gdm^{-3}.

Then, percentage purity $= \frac{\text{mass concentration of pure sample}}{\text{mass concentration of impure sample}} \times 100\%$

$$= \frac{5.3\text{gdm}^{-3}}{20.5\text{gdm}^{-3}} \times 100\% = 25.85\%$$

Hence the percentage purity of sodium carbonate sample was 25.85%.

d) **Possible reason:** Presence of bicarbonates or hydrates.

The sample might have absorbed moisture or reacted with atmospheric carbon dioxide if it was not stored in a tightly sealed container, leading to the formation of impurities like bicarbonates or hydrates.

Experiment 3

During her school break, Kipute returned to her home village, only to find that the local youth science group was preparing for a regional inter-school chemistry fair. The topic? **Standardization of carbonate solutions.**

The group had two mysterious solutions:

- **Solution Q**: Made by dissolving **1.06 g of pure sodium carbonate** with 200cm^3 of excess hydrochloric acid and then diluting the solution to **1 dm³**.
- **Solution R**: A solution of sodium carbonate made by **diluting 225 cm³ of 0.4 M sodium carbonate** to **900 cm³** with water.

With her usual energy and leadership, Kipute stepped up to guide the team. She picked a **phenolphthalein (POP)** indicator for the titration.

Titration procedure Kipute led:

1. She pipetted **20 cm³** of **Solution R** into a clean titration flask.
2. She added **2–3 drops** of **phenolphthalein** (POP) indicator.
3. She filled the burette with **Solution Q**.
4. She titrated R against Q until the permanent colour change is observed.
5. She repeated the process until three consistent readings were recorded.

The young observers were amazed—not just by the colour change, but also by the methodical way Kipute conducted the titration. Now, it's your turn to complete her analysis.

Experiment	Pilot	1	2	3
Final reading (cm^3)				
Initial reading (cm^3)				
Titre volume (cm^3)				

Summary:

_________cm^3 of R required _________cm^3 of Q

Questions

a) Write a balanced chemical equation for the reaction which was taking place during:
  i. Preparation Q
  ii. Titration of R against Q
b) Calculate:
  i. The molarity of R
  ii. The molarity of excess hydrochloric acid in solution Q
  iii. The number of moles of excess hydrochloric acid
  iv. The number of moles of hydrochloric acid reacted with 1.06g of sodium carbonate in solution Q
  v. The total number of moles of hydrochloric acid originally present in 200cm^3 of acid.
c) Suggest a reason why the group used excess hydrochloric acid when preparing Solution Q.
d) Explain the importance of standardizing one solution before using it to analyse another.

Solution

The volume of the pipette used was 20cm^3

TABLE OF RESULTS

Experiment	Pilot	1	2	3
Final reading (cm^3)	21.00	41.10	20.00	39.90
Initial reading (cm^3)	0.00	21.00	0.00	19.90
Titre volume (cm^3)	21.00	20.10	20.00	20.00

$$\text{Average volume used} = \frac{V_1 + V_2 + V_3}{3} = \left(\frac{20.20 + 19.90 + 20.00}{3}\right)\text{cm}^3 = 20.00\text{cm}^3$$

$$\text{Average volume used} = 20.00\text{cm}^3$$

Summary:

20cm^3 of R required **20.00cm^3** of Q.

For 25 mL pipette: The volume of AC will be **25.00cm^3**.

Making Sense of the Experimental Data

All three titre values are within ±0.10 cm³, indicating high precision and hence the experiment was successful.

Response to Questions

a) Balanced chemical equations during:

(i) Preparation of Q

$$Na_2CO_3(aq) + 2HCl(aq) \rightarrow 2NaCl(aq) + CO_2(aq) + H_2O(l)$$

(ii) Titration of R against Q

$$Na_2CO_3(aq) + HCl(aq) \rightarrow NaHCO_3(aq) + NaCl(aq)$$ (With POP as indicator)

b) To calculate:

(i) Molarity of R

Using dilution formula principle;

$$M_cV_c = M_dV_d$$

Where:

M_c is the molarity before dilution $= 0.4M$

V_c is the volume of solution before dilution $= 225cm^3$

V_d is the volume of solution after dilution $= 900cm^3$

M_d is the molarity after dilution = unknown

Then from the above formula, $M_d = \frac{M_cV_c}{V_d} = \frac{0.4mol/dm^3 \times 225cm^3}{900cm^3} = 0.1mol/dm^3$

Hence the molarity of R was 0.1M.

(ii) Using; $\frac{M_{acid} \times V_{acid}}{M_{base} \times V_{base}} = \frac{n_{acid}}{n_{base}}$

Where:

$M_{base} = 0.1M, \ V_{acid} = 20cm^3, \ V_{base} = 20cm^3$

And $n_{acid} = n_{base} = 1$ (From the balanced equation in (a(i)))

Substituting; $\frac{M_{acid} \times 20cm^3}{0.1M \times 20cm^3} = \frac{1}{1}$; $M_{acid} = 0.1M$

The molarity of excess hydrochloric acid was 0.1M.

(iii) Using number of moles (n) = molarity(M) × Volume(V)

Where; Volume (V) of excess hydrochloric acid $= 200cm^3 = 0.2dm^3$

Thus; number of moles (n) $= 0.1mol/dm^3 \times 0.2dm^3 = 0.02mol$

The number of moles of excess hydrochloric acid was 0.02mol**.**

(iv) The number of moles of Na_2CO_3 reacted in $Q = \frac{\text{mass of } Na_2CO_3}{\text{molar mass of } Na_2CO_3}$

$$= \frac{1.06\text{g}}{106\text{g/mol}} = 0.01\text{mol}$$

But the carbonate reacted with hydrochloric acid to form solution Q according to the following equation:

$$Na_2CO_3(aq) + 2HCl(aq) \rightarrow 2NaCl(aq) + CO_2(aq) + H_2O(l)$$

From which mole ratio of Na_2CO_3 to HCl is 1:2.

Thus number of moles of HCl reacted $= 2 \times 0.01\text{mol} = 0.02\text{mol}$

The number of moles of hydrochloric acid reacted with 1.06g of sodium carbonate in solution Q was 0.02mol.

(v) Number of moles of HCl originally present $= n_{HCl}$ in excess $+ n_{HCl}$ reacted with Na_2CO_3

$$= 0.02\text{mol} + 0.02\text{mol} = 0.04\text{mol}$$

The total number of moles of hydrochloric acid originally present in 200cm^3 of acid was 0.04mol.

c) Excess HCl ensures that **all sodium carbonate completely reacts**, allowing the final solution (Q) to contain **unreacted acid** which is titrant in the experiment.

d) Standardization ensures that the **exact concentration** of the titrant is known, which is essential for **accurate and reliable** titration results.

Experiment 4

Mr. Akilikubwa was in his usual white coat early Monday morning, preparing solutions for the incoming Form Six practicals. As he glanced at the preparation log, he paused at a peculiar entry.

"Solution mixture of $Na_2CO_3.XH_2O$ *and* $NaHCO_3$ *made by dissolving 6.4125g of hydrated sodium carbonate contaminated with 1.05g of pure sodium hydrogen carbonate in distilled water and the volume was made up to 250cm*^{3}*."* This was labelled as **AA**.

"X again," he muttered. *"Every year, the mystery of the water of crystallization returns…"*

To solve it, he decided to conduct a titration using **hydrochloric acid (BB)** made by diluting **750 cm^3 of 0.25 M HCl to 937.5 cm^3**, and **methyl orange (MO)** as the indicator. He knew methyl orange would capture the complete neutralisation of both bases: **$Na_2CO_3 \cdot XH_2O$** and **$NaHCO_3$** and he was aware of the following **theory:**

> The reaction between the mixture of sodium carbonate and sodium hydrogen carbonate with dilute hydrochloric acid in the presence of methyl orange indicator is according to the following equations:
>
> $$Na_2CO_3 + 2HCl \rightarrow 2NaCl + CO_2 + H_2O$$
>
> $$NaHCO_3 + HCl \rightarrow NaCl + H_2O + CO_2$$
>
> The use of methyl orange as a suitable indicator for titration between strong acid against week base makes all the basic components of this reaction (Na_2CO_3 and $NaHCO_3$) to react completely and hence the average volume obtained during the titration will be of the two components.
>
> Thus, in this experiment

Average volume = volume of HCl reacted with $NaHCO_3$ + volume of HCl reacted with Na_2CO_3

Procedure Mr. Akilikubwa followed:

1. He pipetted **20 cm^3** of the mixed solution (**AA**) into a clean titration flask.
2. He added **2–3 drops** of **methyl orange** (MO).
3. He filled the burette with **solution BB**.

4. He titrated until the colour change is observed.
5. He carefully recorded the readings and ensured consistency across trials.

Experiment	Pilot	1	2	3
Final reading (cm^3)				
Initial reading (cm^3)				
Titre value (cm^3)				

Summary:

_________cm^3 of AA required _________cm^3 of BB

Questions

a) Calculate:
   i. The molarity of AA with respect to $NaHCO_3$
   ii. The molarity of AA with respect $Na_2CO_3.XH_2O$
b) Calculate the value of X in the compound $Na_2CO_3.XH_2O$
c) Suppose Mr. Akilikubwa mistakenly used 30.0 cm^3 of the mixture instead of 25.0 cm^3 for titration. Explain how would this affect the calculated value of X if not corrected?

Solution

The volume of the pipette used was $20cm^3$

TABLE OF RESULTS

Experiment	Pilot	1	2	3
Final reading (cm^3)	20.50	20.00	20.10	20.00
Initial reading (cm^3)	0.00	0.00	0.00	0.00
Titre value (cm^3)	20.50	20.00	20.10	20.00

$$\text{Average volume used} = \frac{V_1 + V_2 + V_3}{3}$$

$$\text{Average volume used} = \left(\frac{20.00 + 20.10 + 20.00}{3}\right) cm^3 = 20.00cm^3$$

$$\text{Average volume used} = 20.00cm^3$$

Summary:

$20cm^3$ of AA required **$20.00cm^3$** of BB for complete neutralisation.

For 25 mL pipette: The volume of BB will be **$25cm^3$**.

Making Sense of the Experimental Data

Practical tip: *For a given compound, number of moles of water of crystallization is constant and for sodium carbonate, that number is 10 ($Na_2CO_3.10H_2O$).*

So, molar mass of $Na_2CO_3.XH_2O$ is 286g/mol.

Since total mass of mixture of $Na_2CO_3.XH_2O$ and $NaHCO_3$ was 6.4125g, and mass of $NaHCO_3$ was 1.05g; mass of $Na_2CO_3.XH_2O$ will be $(6.4125 - 1.05)g = 5.3625g$.

Then $[Na_2CO_3.XH_2O] = \frac{5.3625g}{0.25L\times 286gmol^{-1}} = 0.075M$ and $[NaHCO_3] = \frac{1.05g}{0.25L\times 84gmol^{-1}} = 0.05M$

Using; $\frac{M_{acid}\times V_{acid}}{M_{base}\times V_{base}} = \frac{n_{acid}}{n_{base}}$

For the reaction between HCl and $Na_2CO_3.XH_2O$:

$M_{base} = 0.075M,\ V_{base} = 20cm^3,\ n_{acid} = 2,\ n_{base} = 1$

And $M_{acid} = \frac{M_cV_c}{V_d} = \frac{0.25M\times 750cm^3}{937.5cm^3} = 0.2M$

Substituting $\frac{0.2M\times V_{acid}}{0.075M\times 20cm^3} = \frac{2}{1};\ V_{acid} = 15cm^3$

For the reaction between HCl and $NaHCO_3$:

$M_{base} = 0.05M,\ V_{base} = 20cm^3,\ n_{acid} = 1,\ n_{base} = 1$ and $M_{acid} = 0.2M$

Substituting $\frac{0.2M\times V_{acid}}{0.05M\times 20cm^3} = \frac{1}{1};\ V_{acid} = 5cm^3$

Total volume of acid (HCl) required to react with both $Na_2CO_3.XH_2O$ and $NaHCO_3$ is $(15 + 5)cm^3$ or $20cm^3$ = experimental data

To conclude: *The calculated volume of acid (HCl) agrees with the average volume of the acid obtained from the experiment.*

Response to Questions

(a)

(i) Using molarity $= \frac{\text{number of moles(n)}}{\text{volume (V)}} = \frac{\text{mass(m)}}{\text{molar mass(M)}\times\text{volume (V)}}$

Where:

Molar mass of $NaHCO_3 = (23 + 1 + 12 + 3\times 16)g/mol = 84g/mol$

Mass of $NaHCO_3$ in the mixture was 1.05g

Volume (V) $= 250cm^3 = 0.25dm^3$

Molarity $= \frac{1.05g}{84g/mol\times 0.25dm^3} = 0.05moldm^{-3}$

Hence the molar concentration of AA with respect to $NaHCO_3$ was $0.05moldm^{-3}$.

(ii) Using the dilution principle to find molarity of acid (HCl) used in titration;

$$M_cV_c = M_dV_d$$

From which; $M_d = \frac{M_cV_c}{V_d} = \frac{0.25M\times 750cm^3}{937.5cm^3} = 0.2M = M_{acid}$

Using; $\frac{M_{acid}\times V_{acid}}{M_{base}\times V_{base}} = \frac{n_{acid}}{n_{base}}$

For the reaction between HCl and $NaHCO_3$:

$$NaHCO_3 + HCl \rightarrow NaCl + H_2O + CO_2,\ n_{acid} = 1,\ n_{base} = 1$$

$M_{base} = 0.05M,\ V_{base} = 20cm^3,\ M_{acid} = 0.2M$

Substituting $\frac{0.2M\times V_{acid}}{0.05M\times 20cm^3} = \frac{1}{1};\ V_{acid} = 5cm^3$

For the reaction between HCl and $Na_2CO_3.XH_2O$:

$$Na_2CO_3 + 2HCl \rightarrow 2NaCl + CO_2 + H_2O;\ n_{acid} = 2, \qquad n_{base} = 1$$

$V_{base} = 20cm^3$, $M_{acid} = 0.2M$ and $V_{acid} = (20 - 5)cm^3 = 15cm^3$

Substituting $\frac{0.2M \times 15cm^3}{M_{base} \times 20cm^3} = \frac{2}{1}$; $M_{base} = 0.075M$

Hence the molarity of AA with respect to $Na_2CO_3.XH_2O$ was $0.075moldm^{-3}$

(iii) The value of X in the compound $Na_2CO_3.XH_2O$

Using molar mass of hydrated Na_2CO_3

$$= \frac{\text{Mass concentration}}{\text{Molar concentration}} = \frac{\frac{(6.4125 - 1.05)g}{0.25L}}{0.075mol/L} = 286g/mol$$

But also;

Molar mass of hydrated Na_2CO_3

$$= \big((2 \times 23) + 12 + (3 \times 16) + 18X\big)g/mol = (106 + 18X)g/mol$$

Thus, $106 + 18X = 286;\ X = 10$

Therefore; the value of X in the compound $Na_2CO_3.XH_2O$ was 10.

(c) The calculated value of X would be smaller than 10.

Explanation

The greater volume of mixture means that greater volume of acid would be used too leading to greater calculated number of moles of $Na_2CO_3 \cdot XH_2O$. Consequently, the molar mass of the carbonate would appear smaller (since mass of $Na_2CO_3 \cdot XH_2O$ remains unchanged) leading to an underestimated value of X.

Experiment 5

Kipute had recently joined the National Chemistry Training Program for top-performing students. As part of the evaluation, she received a two-stage task involving unknowns and standardization—something she always enjoyed for the challenge.

On her lab bench were three labelled solutions:

- **Solution XX**: 0.05 M oxalic acid – a known standard.
- **Solution YY**: Sodium hydroxide – unknown concentration.
- **Solution ZZ**: A solution made by dissolving $6g/dm^3$ of an organic acid, HA.

Phenolphthalein indicator (POP)

The following **theory** was clear to her:

The concentration of sodium hydroxide is obtained by standardizing the solution with oxalic acid solution.

The standardized solution of sodium hydroxide is then titrated against the organic acid, HA and hence letter A can be determined as per its value.

Procedure Kipute followed:

Part A

1. She pipetted **20 cm³** of **Solution YY** into a clean titration flask.
2. She added **2–3 drops of phenolphthalein**, which turned the solution pink.
3. She filled the burette with **Solution XX**.
4. She titrated until there is a colour change.
5. She repeated the titration to obtain three concordant readings.

Experiment	Pilot	1	2	3
Final reading (cm^3)				
Initial reading (cm^3)				
Titre value (cm^3)				

Part B

1. She again pipetted **20 cm³** of **Solution YY** into a clean titration flask.
2. She added phenolphthalein as before.
3. This time, she filled the burette with **Solution ZZ**.
4. She titrated until there is a colour change.
5. Three consistent titrations were recorded to ensure accuracy.

Experiment	Pilot	1	2	3
Final reading (cm^3)				
Initial reading (cm^3)				
Titre value (cm^3)				

Now that the experimental phase was complete, Kipute proceeded with the calculations to solve the mystery of the unknown acid.

Summary:

________cm^3 of YY required _________cm^3 of ZZ

Questions

a) Write a balanced chemical equation for the reaction between YY and XX
b) Calculate:
   i. The concentration of YY in mol/dm^3
   ii. The molar concentration of ZZ
   iii. The radical mass of A in ZZ
c) Give the possible structure of the organic acid, HA.
d) Why must oxalic acid be used as a primary standard in Part A rather than sodium hydroxide?
e) Explain why is the volume of the pipetted solution (NaOH) always fixed at 20 cm³ (or 25 cm³) instead of varying with each trial?

Solution

PART A:

The volume of the pipette used was 20cm^3

TABLE OF RESULTS

Experiment	Pilot	1	2	3
Final reading (cm^3)	20.50	20.00	20.10	20.00
Initial reading (cm^3)	0.00	0.00	0.00	0.00
Titre value (cm^3)	20.50	20.00	20.10	20.00

$$\text{Average volume used} = \frac{V_1 + V_2 + V_3}{3} = \left(\frac{20.00 + 20.10 + 20.00}{3}\right)\text{cm}^3 = 20.00\text{cm}^3$$

$$\text{Average volume used} = 20.00\text{cm}^3$$

Summary:

20cm^3 of YY required **20.00cm^3** of XX for complete neutralisation

For 25 mL pipette: The volume of XX will be **25cm^3**.

Making Sense of the Experimental Data

All three titre values are within ±0.10 cm³, indicating high precision and hence the experiment was successful.

PART B

The volume of the pipette used was 20cm^3

TABLE OF RESULTS

Experiment	Pilot	1	2	3
Final reading (cm^3)	21.00	20.10	20.00	20.00
Initial reading (cm^3)	0.00	0.00	0.00	0.00
Titre value (cm^3)	21.00	20.10	20.00	20.00

$$\text{Average volume used} = \frac{V_1 + V_2 + V_3}{3} = \left(\frac{20.20 + 21.00 + 19.80}{3}\right)\text{cm}^3 = 20.00\text{cm}^3$$

$$\text{Average volume used} = 20.00\text{cm}^3$$

Summary:

20cm^3 of YY required **20.00cm^3** of ZZ for complete to complete the reaction

For 25 mL pipette: The volume of ZZ will be **25cm^3**.

Making Sense of the Experimental Data

Practical tip: *The only monobasic organic acid (HA) which is commonly used in acid-base titration is acetic acid* (CH_3COOH).

So, molar mass of HA (CH_3COOH) is 60g/mol.

And [HA] $= \frac{6g/dm^3}{60gmol^{-1}} = 0.1M$

So, number of moles of acetic acid reacted with NaOH $= 0.1V_{acetic}$ $(n = MV)$

The acetic acid reacts with sodium hydroxide according to the following equation:

$$CH_3COOH + NaOH \longrightarrow CH_3COONa + H_2O$$

From which; mole ratio of CH_3COOH to NaOH is 1:1.

Thus number of moles of NaOH reacted in part B was also $0.1V_{acetic}$.

In part A, oxalic acid reacted with sodium hydroxide according to the following equation;

$$H_2C_2O_4 + 2NaOH \rightarrow Na_2C_2O_4 + 2\,H_2O$$

From which; mole ratio of $H_2C_2O_4$ to NaOH is 1:2.

Since number of moles of oxalic acid reacted with NaOH $= 0.05V_{oxalic}$ (Again, $n = MV$)

Then number of moles of NaOH reacted in part A $= 2 \times 0.05V_{oxalic} = 0.1V_{oxalic}$

Since the same base (the same concentration) with the same pipette volume was used in part A and part B, it follows that:

Number of moles of NaOH reacted in part A = Number of moles of NaOH reacted in part B

Therefore, $0.1V_{acetic} = 0.1V_{oxalic}$ or $V_{acetic} = V_{oxalic} = \mathbf{20cm^3}$.

Hence titre volume of HA in part B ***must be equal*** *to the titre volume of oxalic acid in part A which agrees with the experimental data.*

Response to Questions

a) $H_2C_2O_4(aq) + 2NaOH(aq) \rightarrow Na_2C_2O_4(aq) + 2\,H_2O(l)$

b) Using; $\frac{M_{acid} \times V_{acid}}{M_{base} \times V_{base}} = \frac{n_{acid}}{n_{base}}$

Where:

$M_{acid} = 0.05M,\ V_{acid} = 20cm^3,\ V_{base} = 20cm^3$

And $n_{acid} = 1,\ n_{base} = 2$ (From the balanced equation in (a) above)

Substituting $\frac{0.05M \times 20cm^3}{M_{base} \times 20cm^3} = \frac{1}{2};\ M_{base} = 0.1M$

(i) The concentration of YY was $0.1mol/dm^3$.

(ii) ZZ (HA) reacts with NaOH according to the following equation:
$HA + NaOH \longrightarrow NaA + H_2O;\ n_{acid} = 1,\quad n_{base} = 1$

Then using $\frac{M_{acid} \times V_{acid}}{M_{base} \times V_{base}} = \frac{n_{acid}}{n_{base}}$

Where:

$M_{base} = 0.1M,\ V_{acid} = 20cm^3,\ V_{base} = 20cm^3$

Substituting $\frac{M_{acid} \times 20cm^3}{0.1 \times 20cm^3} = \frac{1}{1};\ M_{acid} = 0.1M$

The molar concentration of ZZ was $0.1mol/dm^3$.

(iii) Using;

$$\text{molarity} = \frac{\text{mass concentration}}{\text{molar mass}} \text{ or molar mass } = \frac{\text{mass concentration}}{\text{molarity}}$$

Then molar mass of ZZ (HA) $= \frac{6\text{g/dm}^3}{0.1\text{mol/dm}^3} = 60\text{g/mol}$

But molar mass of HA $=$ Radical mass of A $+$ Atomic mass of H

So, $60 =$ Radical mass of A $+ 1$; Radical mass of A $= 59$

The radical mass of A is 59.

c) The general structure of organic acid may be written as $C_nH_{2n+1}COOH$
Thus $12n + 2n + 1 + 12 + 16 + 16 + 1 = 60; 14n = 14$ or $n = 1$

Hence the possible structure of HA is CH_3COOH.

(d) Oxalic acid suits more as primary standard because it is more stable when exposed to atmosphere unlike sodium hydroxide which is hygroscopic and reacts with carbon dioxide in the atmosphere to form sodium carbonate.

(e) Varying the volume would introduce extra complexity, reduced accuracy, and make error detection more difficult while using a fixed volume ensures the following:

1) **Consistency**: Every titration trial is comparable
2) **Accuracy**: A pipette delivers very precise volumes
3) **Ease of calculation**: It simplifies mole calculations, as the same volume is used repeatedly
4) **Control of variables**: Only the volume of acid from the burette changes, making the endpoint easier to track

Experiment 6

It was a quiet Thursday morning at the National Water Standards Laboratory. Mr. Akilikubwa was inspecting a sample of a commercial cleaning product that had been causing quality concerns. The client, a regional soap manufacturer, claimed their formula used sodium carbonate with precise hydration—but the numbers weren't adding up.

Mr. Akilikubwa removed his lab coat from the hook and carefully reviewed the test plan. His goal: determine how much water of crystallization had been included in the sample of sodium carbonate. A routine titration would unveil the truth.

He prepared the following:

- **A_1:** 5.72 g of sodium carbonate hydrate ($Na_2CO_3 \cdot xH_2O$) dissolved in 250 cm³ of distilled water.
- **A_2:** 0.1 M hydrochloric acid (HCl).
- **A_3:** Phenolphthalein indicator.

Procedure Mr. Akilikubwa followed

1. He filled the burette with solution **A_2**.
2. Using a pipette, he measured either **20 cm³** of solution **A_1** into a clean titration flask.
3. He added two or three drops of **A**.
4. He titrated the mixture with **A_2** until the colour change is observed.
5. He repeated the process to obtain consistent readings and recorded results in a tabular form.

Experiment	Pilot	1	2	3
Final reading (cm^3)				
Initial reading (cm^3)				
Titre value (cm^3)				

Summary:

_________cm^3 of $\mathbf{A_1}$ required _________cm^3 of $\mathbf{A_2}$ for complete reaction.

Questions

(a) Write a balanced equation for the reaction taking place between $\mathbf{A_1}$ and $\mathbf{A_2}$.
(b) Calculate the concentration of $\mathbf{A_1}$ in $\mathrm{mol/dm^3}$ and $\mathrm{g/dm^3}$.
(c) What is the value of water of crystallization, x?
(d) How many moles of water of crystallization have been in the original 5.72g?
(e) From an industrial quality control perspective, why is determining the correct value of x important for manufacturing cleaning agents?

Solution

The volume of the pipette used was $20cm^3$

TABLE OF RESULTS

Experiment	Pilot	1	2	3
Final reading (cm^3)	16.70	16.00	16.00	16.00
Initial reading (cm^3)	0.00	0.00	0.00	0.00
Titre value (cm^3)	16.70	16.00	16.00	16.00

Titre volume used $= 20.00\mathrm{cm^3}$

Summary:

$20cm^3$ of $\mathbf{A_1}$ required **$16.00cm^3$** of $\mathbf{A_2}$ for complete reaction.

For 25 mL pipette: The volume of $\mathbf{A_2}$ will be **$20cm^3$**.

Making Sense of the Experimental Data

As mentioned before, the chemical formula of hydrated sodium carbonate is $\mathrm{Na_2CO_3.10H_2O}$.

So, molar mass of $Na_2CO_3.xH_2O$ is 286g/mol.

And $[\mathrm{Na_2CO_3.xH_2O}] = \frac{5.72\mathrm{g}}{0.25\mathrm{L}\times 286\mathrm{gmol^{-1}}} = 0.08\mathrm{M} = \mathrm{M_{base}}$

The reaction between sodium carbonate takes place in two stages as per equations:

$$\mathrm{Na_2CO_3} + \mathrm{HCl} \rightarrow \mathrm{NaCl} + \mathrm{NaHCO_3} \quad \ldots\ldots\ldots\ldots\ldots\text{(i)}$$

$$\mathrm{NaHCO_3} + \mathrm{HCl} \rightarrow \mathrm{NaCl} + \mathrm{H_2O} + \mathrm{CO_2} \quad \ldots\ldots\ldots\ldots\text{(ii)}$$

With POP indicator, the end point is registered after completion of reaction (i) only.

So the correct reaction equation for the titration is;

$$Na_2CO_3 + HCl \rightarrow NaCl + NaHCO_3;\ n_{acid} = n_{base} = 1$$

Using; $\frac{M_{acid} \times V_{acid}}{M_{base} \times V_{base}} = \frac{n_{acid}}{n_{base}}$

Where:

$M_{acid} = 0.1M,\ M_{base} = 0.08M,\ V_{base} = 20cm^3,\ n_{acid} = 1,\ n_{base} = 1$

Substituting $\frac{0.1M \times V_{acid}}{0.08M \times 20cm^3} = \frac{1}{1}$; $\mathbf{V_{acid} = 16cm^3}$ = experimental data

To conclude: *The calculated volume of acid (HCl) agrees with the volume of the acid obtained from the experiment.*

Response to Questions

(a) $Na_2CO_3 + HCl \rightarrow NaCl + NaHCO_3$

(b) Mass concentration $= \frac{\text{mass of solute}}{\text{volume of solution}} = \frac{5.72g}{0.25dm^3} = 22.88g/dm^3$

The concentration of A_1 in g/dm^3 is $22.88g/dm^3$.

Using; $\frac{M_{acid} \times V_{acid}}{M_{base} \times V_{base}} = \frac{n_{acid}}{n_{base}}$

Where:

$M_{acid} = 0.1M,\ V_{base} = 20cm^3,\ V_{acid} = 16cm^3,\ n_{acid} = 1,\ n_{base} = 1$

Substituting $\frac{0.1M \times 16cm^3}{M_{base} \times 20cm^3} = \frac{1}{1}$; $M_{base} = 0.08M$

The concentration of A_1 in mol/dm^3 is $0.08mol/dm^3$.

(c) Using molar mass of hydrated Na_2CO_3

$$= \frac{\text{Mass concentration}}{\text{Molar concentration}} = \frac{22.88g/dm^3}{0.08mol/dm^3} = 286g/mol$$

But also;

Molar mass of hydrated Na_2CO_3

$$= \left((2 \times 23) + 12 + (3 \times 16) + 18x\right)g/mol = (106 + 18x)g/mol$$

Thus, $106 + 18x = 286$; $X = 10$

Therefore; the value of x is 10.

(d) Number of water of crystallization 286g of the compound = 10mol

Thus, number of water of crystallization in 5.72g = $10mol \times \frac{5.72g}{286g} = 0.2mol.$

Hence, the number of water of crystallization in the original 5.72g is 0.2 mol.

(e) The effectiveness of a cleaning agent depends on the amount of sodium carbonate (Na_2CO_3) it contains. Determining the value of *x* in the hydrate form $Na_2CO_3 \cdot xH_2O$ allows us to calculate the actual amount of anhydrous sodium carbonate present in a given mass (or per gram) of the compound. This, in turn, enables accurate assessment of the cleaning agent's efficiency.

Experiment 7

It was the final round of preparations before the National School Chemistry Trials. Mr. Akilikubwa had been asked to prepare a particularly tricky test for the Form Six students—a titration involving a **mixture of two acids**: **acetic acid** and **hydrochloric acid**, both present in **Solution A**.

His goal? To use two different indicators— **C: methyl orange (MO)** and **D: phenolphthalein (POP)**—to determine how much of each acid was present in the mixture. With years of experience, he knew that:

- **Methyl orange** would register the end point for the **strong acid (HCl)**.
- **Phenolphthalein**, added **after** methyl orange, would reveal how much of the **weak acid (acetic acid)** remained unreacted.

Using a **0.1 M sodium hydroxide solution (Solution B)** in the burette, he proceeded to carry out a **two-stage titration**—carefully tracking how much base was needed to neutralize each component.

Procedure Mr. Akilikubwa followed:

1. He pipetted **20 cm³** of the acid mixture (Solution A) into a titration flask.
2. He added **2–3 drops of methyl orange (MO)**.
3. He titrated the solution against **Solution B** in the burette until colour change is observed.
4. He recorded the titre value under indicator C.
5. Without changing the solution, he then added **2–3 drops of D**.
6. He continued the titration until a second colour change occurred—this time.
7. He recorded the titre value under indicator D.
8. He recorded the results in a tabular form and repeated the process for accuracy.

Experiment	**PILOT**	**1**	**2**	**3**
Final reading (cm^3) under C				
Final reading (cm^3) under D				
Initial reading (cm^3)				
Titre value (cm^3) under C				
Titre value (cm^3) under D				

Summary:

_____cm^3 of A required _____cm^3 of B when C was used and ____cm^3 of B when D was used

Questions

a) Explain why is methyl orange used before phenolphthalein in this two-stage titration?

b) Write a balanced chemical equation to show the reaction:
   i. When C was used
   ii. When D was used

c) The colour change for the titration using C was from ____ to ____ and when D was added the calour changed from ____ to ______

d) Calculate the concentration of A in g/dm^3 when:

i. C was used

ii. D was used

e) What was the mass ratio of acetic acid to hydrochloric acid in the mixture?

Solution

The volume of the pipette used was 20cm^3

TABLE OF RESULTS

Experiment	**PILOT**	**1**	**2**	**3**
Final reading (cm^3) under C	4.40	4.10	4.00	4.20
Final reading (cm^3) under D	24.50	24.10	24.00	24.30
Initial reading (cm^3)	0.00	0.00	0.00	0.00
Titre value (cm^3) under C	4.40	4.10	4.00	4.20
Titre value (cm^3) under D	20.10	20.00	20.00	20.10

$$\text{Average volume used} = \frac{V_1 + V_2 + V_3}{3}$$

$$\text{Average volume used under C(MO)} = \left(\frac{4.10 + 4.00 + 4.20}{3}\right)\text{cm}^3 = 4.10\text{cm}^3$$

$$\text{Average volume used under D (POP)} = \left(\frac{20.00 + 20.00 + 20.10}{3}\right)\text{cm}^3 = 20.00\text{cm}^3$$

Summary:

20cm^3 of A required **4.10cm^3** of B when C (MO) was used and **20.00cm^3** of B when D (POP) was used.

For 25 mL pipette: With MO the volume will be **5.10cm^3** while with POP the volume will be **25cm^3**

Making Sense of the Experimental Data

All three titre values (for both indicators) are within ±0.10 cm³ of each other, indicating high precision and hence the experiment was successful.

Moreover, the fact that the titre value with phenolphthalein (POP) is significantly higher than that with methyl orange (MO) suggests that the molarity of acetic acid (CH_3COOH) is substantially greater than that of hydrochloric acid (HCl)—approximately five times greater. More precisely, it is about ***20 ÷ 4.1****, or* ***4.88 times*** *the molarity of HCl.*

Response to Questions

(a) Methyl orange shows the end point after complete neutralisation of hydrochloric acid only which is strong acid while with strong base (NaOH), phenolphthalein shows the end point after neutralisation of both strong acid (HCl) and weak acid (CH_3COOH). So by using methyl orange first, the titration stops at the strong acid's endpoint. Adding phenolphthalein later allows continuation to the second endpoint, targeting the weaker acid. This sequencing prevents overlap and ensures clear detection of each acid separately.

(b)

(i) With C (MO)

$$\text{HCl(aq)} + \text{NaOH(aq)} \longrightarrow \text{NaCl(aq)} + \text{H}_2\text{O(l)}$$

(ii) With D (POP)

$$CH_3COOH(aq) + NaOH(aq) \longrightarrow CH_3COONa(aq) + H_2O(l)$$

(c) The colour change for the titration using C(MO) was from **pink** to **orange** and when D(POP) added the colour changed from **orange** to **pink.**

(d)

(i) The reaction equation with C:

$$HCl(aq) + NaOH(aq) \longrightarrow NaCl(aq) + H_2O(l);\ n_{acid} = n_{base} = 1$$

Using; $\frac{M_{acid} \times V_{acid}}{M_{base} \times V_{base}} = \frac{n_{acid}}{n_{base}}$

Where:

$M_{base} = 0.1M,\ \ V_{acid} = 20cm^3\ , V_{base} = 4.1cm^3, n_{acid} = 1,\ \ n_{base} = 1$

Substituting $\frac{M_{acid} \times 20cm^3}{0.1M \times 4.1cm^3} = \frac{1}{1}; M_{acid} = 0.0205M$

Then using mass concentration $=$ molarity$(M) \times$ molar mass

Where molar mass of $HCl\ =\ (1 + 35.5)g/mol = 36.5g/mol$

It follows that mass concentration $= 0.0205mol/dm^3 \times 36.5g/mol$

$$= 0.74825g/dm^3$$

Therefore, the concentration of an acid (HCl) in g/dm^3 when C (MO) was used is $0.75g/dm^3$

(ii) The reaction equation with D:

$$CH_3COOH(aq) + NaOH(aq) \longrightarrow CH_3COONa(aq) + H_2O(l);\ n_{acid} = n_{base} = 1$$

Using; $\frac{M_{acid} \times V_{acid}}{M_{base} \times V_{base}} = \frac{n_{acid}}{n_{base}}$

Where:

$M_{base} = 0.1M,\ \ V_{acid} = 20cm^3\ , V_{base} = 4.1cm^3, n_{acid} = 1,\ \ n_{base} = 1$

Substituting $\frac{M_{acid} \times 20cm^3}{0.1M \times 20cm^3} = \frac{1}{1}; M_{acid} = 0.1M$

Then using mass concentration $=$ molarity$(M) \times$ molar mass

Where molar mass of $CH_3COOH = (2 \times 12 + 1 \times 4 + 2 \times 16)g/mol = 60g/mol$

It follows that mass concentration $= 0.1mol/dm^3 \times 60g/mol = 6g/dm^3$

Therefore, the concentration of an acid (HCl) in g/dm^3 when D (POP) was used is $6g/dm^3$.

(e) The mass ratio $= \frac{\text{mass concentration of acetic acid}}{\text{mass concentration of hydrochloric acid}} = \frac{6g/dm^3}{0.75g/dm^3} = 8$

Hence the mass ratio of acetic acid to hydrochloric acid in the mixture is 8 : 1

Experiment 8

It was Thursday afternoon, and Kipute was in the school lab reviewing a suspicious-looking mixture labelled:

"**AA: Sodium carbonate + Sodium hydroxide (4.65 g in 500 cm³)**"

She raised an eyebrow. *"Two bases in one solution? This smells like a classic dual-indicator case."*

The laboratory technician, Mr. Akilikubwa had provided a **solution of hydrochloric acid (BB)**—carefully prepared from **3.65 g of pure HCl in 500 cm³**—along with both **phenolphthalein** and **methyl orange** indicators.

Kipute understood the theory perfectly:

- **Phenolphthalein (POP)** would detect the point where **NaOH and part of Na_2CO_3** were neutralized.
- **Methyl orange (MO)**, added afterward, would detect when **the remaining $NaHCO_3$** (from Na_2CO_3) was neutralized.

This method would allow her to **separate the contribution** of each base to the total alkalinity.

Procedure Kipute followed:

1. She pipetted **20 cm^3** of **Solution AA** into a clean conical flask.
2. She added **2–3 drops of phenolphthalein (POP)**.
3. She titrated the solution against **Solution BB** until the colour change is observed.
4. She recorded the titre value under POP.
5. Without replacing the flask, she added **2–3 drops of methyl orange (MO)**.
6. She continued titrating until the colour change is observed.
7. She recorded the titre value under MO.
8. She repeated the entire sequence to ensure consistency.

Experiment	**PILOT**	**1**	**2**	**3**
Final reading (cm^3) under POP				
Initial reading (cm^3) under POP				
Volume used (cm^3) under POP				
Final reading (cm^3) under MO				
Initial reading (cm^3) under MO				
Volume used (cm^3) under MO				

Summary:

_____cm^3 of AA required _____cm^3 of BB when POP was used and ____cm^3 of BB when MO was used.

Questions

a) Calculate the volume of BB reacted completely with Na_2CO_3 in AA.
b) Calculate the volume of BB reacted completely with NaOH in AA.
c) Calculate the mass concentration of each component of the mixture.
d) Calculate mass percentage of Na_2CO_3 in AA.
e) Why is it important to titrate only 20cm^3 (or 25cm^3) of AA instead of the full 500 cm^3?

Solution

The volume of the pipette used was 20cm^3

TABLE OF RESULTS

Experiment	**PILOT**	**1**	**2**	**3**
Final reading (cm^3) under POP	14.80	14.50	14.60	14.40
Initial reading (cm^3) under POP	0.00	0.00	0.00	0.00
Volume used (cm^3) under POP	14.80	14.50	14.60	14.40
Final reading (cm^3) under MO	19.80	19.40	19.40	19.30
Initial reading (cm^3) under MO	14.80	14.50	14.60	14.40
Volume used (cm^3) under MO	5.00	4.90	4.80	4.90

$$\text{Average volume used} = \frac{V_1 + V_2 + V_3}{3}$$

$$\text{Average volume used under MO} = \left(\frac{4.90 + 4.80 + 4.90}{3}\right)\text{cm}^3 = 4.90\text{cm}^3$$

$$\text{Average volume used under POP} = \left(\frac{14.50 + 14.60 + 14.40}{3}\right)\text{cm}^3 = 14.50\text{cm}^3$$

Summary:

20cm^3 of AA required **14.50cm^3** of BB when POP was used and **4.90cm^3** of BB when MO was used.

For 25 mL pipette: With POP the volume will be **18.10cm^3** while with MO the volume will be **6.1cm^3**.

Making Sense of the Experimental Data

All three titre values (for both indicators) are within ±0.10 cm^3 of each other, indicating high precision and hence the experiment was successful.

Response to Questions

(a) The volume (V) of BB reacted completely with Na_2CO_3 in AA

$$= 2 \times V_{HCl} \text{ under MO} = 2 \times 4.90\text{cm}^3 = 9.80\text{cm}^3$$

Therefore, the volume of BB reacted completely with Na_2CO_3 in AA was 9.80cm^3.

(b) The volume of BB reacted completely with NaOH in AA

$$= V_{HCl} \text{ under POP} - V_{HCl} \text{ under MO} = 14.50\text{cm}^3 - 4.90\text{cm}^3 = 9.60\text{cm}^3$$

Therefore, the volume of BB reacted completely with NaOH in AA was 9.60cm^3

(c) Using; $\frac{M_{acid} \times V_{acid}}{M_{base} \times V_{base}} = \frac{n_{acid}}{n_{base}}$

For the reaction between HCl and NaOH:

$$\text{NaOH} + \text{HCl} \rightarrow \text{NaCl} + H_2O\ ;\ n_{acid} = 1,\ n_{base} = 1$$

$V_{base} = 20cm^3$,$V_{acid} = 9.6cm^3$ $M_{acid} = \frac{m}{M_r \times V} = \frac{3.65g}{36.5gmol^{-1} \times 0.5L} = 0.2M$

Substituting $\frac{0.2M \times 9.6cm^3}{M_{base} \times 20cm^3} = \frac{1}{1}$; $M_{base} = 0.096M$

Mass concentration of NaOH $=$ Molarity $\times$ Molar mass of NaOH

$$= 0.096mol/dm^3 \times 40g/mol = 3.84g/dm^3$$

The mass concentration of NaOH is $3.84g/dm^3$.

For the reaction between HCl and Na_2CO_3:

$$Na_2CO_3 + 2HCl \rightarrow 2NaCl + CO_2 + H_2O;\ n_{acid} = 2, n_{base} = 1$$

$V_{base} = 20cm^3$, $M_{acid} = 0.2M$ and $V_{acid} = 9.80cm^3$

Substituting $\frac{0.2M \times 9.80cm^3}{M_{base} \times 20cm^3} = \frac{2}{1}$; $M_{base} = 0.049M$

Mass concentration of Na_2CO_3 $=$ Molarity $\times$ Molar mass of Na_2CO_3

$$= 0.049mol/dm^3 \times 106g/mol = 5.194g/dm^3$$

The mass concentration of NaOH is $5.194g/dm^3$.

(d) And mass of Na_2CO_3 in $500cm^3(0.5dm^3)$ $=$ Mass concentration $\times$ Volume

$$= 5.194g/dm^3 \times 0.5dm^3 = 2.597g$$

Total mass of Na_2CO_3 and $NaHCO_3$ in $500cm^3$ of solution $= 4.65g$ (Given)

Then mass percentage of $Na_2CO_3 = \frac{\text{Mass of } Na_2CO_3}{\text{Total mass}} \times 100\% = \frac{2.597g}{4.65g} \times 100\% = 55.8\%$

Therefore, the percentage of Na_2CO_3 in in the mixture is 55.8%.

(e) This ensures the following:

1) **Practicality**: Titrating the full volume would require too much acid and would be inefficient.
2) **Precision**: Smaller, standard volume allow for accurate pipetting and multiple repeats for consistent results.
3) **Conservation of reagents**: It allows reuse of the mixture for multiple trials.

Experiment 9

In the chemistry prep room, Mr. Akilikubwa stood in front of a puzzling solution labelled:

"**A: 1.48 g mixture of sodium carbonate and sodium bicarbonate in 250 cm^3 solution**"

The goal was to determine the **exact composition** of the two carbonates in the mixture. The classic strategy: use **two indicators sequentially—phenolphthalein (POP)** followed by **methyl orange (MO)**—to separate their contributions during titration with **Solution B: Hydrochloric acid**, made from **1.46 g HCl in 400 cm^3 of water**.

He knew:

- **Phenolphthalein** would track the point where **carbonate was half-neutralized** (forming bicarbonate).
- **Methyl orange**, added next, would complete the titration by neutralizing all bicarbonate—both originally present and that formed during the first step.

This method would reveal how much acid was used **only for carbonate**, and how much for **bicarbonate**.

Procedure Mr. Akilikubwa Followed

1. He pipetted **20 cm³** of **Solution A** into a titration flask.
2. He added **2–3 drops of phenolphthalein (POP)**.
3. He titrated with **Solution B** until the colour change is observed.
4. He noted the first titre volume.
5. Without changing the contents, he added **2–3 drops of methyl orange (MO)**.
6. He continued titrating until another colour change is observed.
7. He noted the second titre volume.
8. He repeated the titration to confirm accuracy.

Summary:

_____cm^3 of solution A required _____cm^3 of solution B when POP was used and _____cm^3 of solution B when MO was used.

Questions

a) Basing on the indicators used, state the colour changes when:
   i. POP was used
   ii. MO was used
b) Calculate the molar concentration of solution B.
c) Calculate the concentration of solution A in moles per litre when:
   i. POP was used
   ii. MO was used
d) Calculate the percentage of sodium bicarbonate in the solution mixture.
e) Suppose **Mr. Akilikubwa** mistakenly added methyl orange first instead of phenolphthalein. What effect would this have on the experimental results and conclusions?

Solution

The volume of the pipette used was $20cm^3$

TABLE OF RESULTS

Experiment	**PILOT**	**1**	**2**	**3**
Final reading (cm^3) under MO	20.70	20.40	20.30	20.40
Final reading (cm^3) under POP	8.40	8.20	8.10	8.30
Initial reading (cm^3)	0.00	0.00	0.00	0.00
Volume used (cm^3) under POP	8.40	8.20	8.10	8.30
Volume used (cm^3) under MO	12.30	12.20	12.20	12.10

$$\text{Average volume used} = \frac{V_1 + V_2 + V_3}{3}$$

$$\text{Average volume used under POP} = \left(\frac{8.20 + 8.10 + 8.30}{3}\right)\text{cm}^3 = 8.20\text{cm}^3$$

Average volume used under MO $= \left(\frac{12.20 + 12.20 + 12.10}{3}\right)\text{cm}^3 = 12.20\text{cm}^3$

Summary:

20cm^3 of solution A required **8.20cm^3** of solution B when POP was used and **12.20cm^3** of solution B when MO was used.

For 25 mL pipette: With POP the volume will be **10.20cm^3** while with MO the volume will be **15.20cm^3**.

Making Sense of the Experimental Data

All three titre values (for both indicators) are within ±0.10 cm³ of each other, indicating high precision and hence the experiment was successful.

Response to Questions

(a) The colour changes:
  (i) With POP, the colour change was from **pink** to **colourless**.
  (ii) With MO, the colour change was from **yellow** to **orange.**

(b) Using molarity $= \frac{n}{V} = \frac{m}{M_r \times V}$

Molarity of B $= \frac{1.46\text{g}}{36.5\text{g/mol} \times 0.4\text{dm}^3} = 0.1\text{mol/dm}^3$

Hence the molar concentration of B was 0.1mol/dm^3

(c) Calculation for each case is as follows:
  (i) Reaction equation with POP:

$$Na_2CO_3 + HCl \rightarrow NaCl + NaHCO_3;\ n_{acid} = n_{base} = 1$$

Using; $\frac{M_{acid} \times V_{acid}}{M_{base} \times V_{base}} = \frac{n_{acid}}{n_{base}}$

Where:

$V_{base} = 20\text{cm}^3, V_{acid} = 8.2\text{cm}^3\ M_{acid} = 0.1\text{M}$

Substituting $\frac{0.1\text{M} \times 8.2\text{cm}^3}{M_{base} \times 20\text{cm}^3} = \frac{1}{1};\ M_{base} = 0.041\text{M}$

Hence the molarity of A under POP was 0.041mol/dm^3.

  (ii) Reaction equation with MO:

$$NaHCO_3 + HCl \rightarrow NaCl + H_2O + CO_2;\ n_{acid} = n_{base} = 1$$

Using; $\frac{M_{acid} \times V_{acid}}{M_{base} \times V_{base}} = \frac{n_{acid}}{n_{base}}$

Where:

$V_{base} = 20\text{cm}^3, V_{acid} = 12.2\text{cm}^3, M_{acid} = 0.1\text{M}$

Substituting $\frac{0.1\text{M} \times 12.2\text{cm}^3}{M_{base} \times 20\text{cm}^3} = \frac{1}{1};\ M_{base} = 0.061\text{M}$

Hence the molarity of A under MO was 0.061mol/dm^3.

(d) Reaction equation: $NaHCO_3 + HCl \rightarrow NaCl + H_2O + CO_2;\ n_{acid} = n_{base} = 1$
V_{HCl} reacted with original $NaHCO_3 = V_{HCl}$ under MO $- V_{HCl}$ under POP

$$= 12.20\text{cm}^3 - 8.20\text{cm}^3 = 4.00\text{cm}^3$$

Again using; $\frac{M_{acid} \times V_{acid}}{M_{base} \times V_{base}} = \frac{n_{acid}}{n_{base}}$

Where:

$V_{base} = 20cm^3$, $V_{acid} = 4cm^3$ $M_{acid} = 0.1M$

Substituting $\frac{0.1M \times 4cm^3}{M_{base} \times 20cm^3} = \frac{1}{1}$; $M_{base} = 0.02M$

$$\text{Mass concentration of } NaHCO_3 = \text{Molarity} \times \text{Molar mass of } NaHCO_3$$

$$= 0.02mol/dm^3 \times 84g/mol = 1.68g/dm^3$$

$$\text{And mass of } NaHCO_3 \text{ in } 250cm^3 (0.25dm^3) = \text{Mass concentration} \times \text{Volume}$$

$$= 1.68g/dm^3 \times 0.25dm^3 = 0.42g$$

Total mass of Na_2CO_3 and $NaHCO_3$ in $250cm^3$ of solution $= 1.48g$ (Given)

Then mass percentage of $NaHCO_3 = \frac{\text{Mass of } NaHCO_3}{\text{Total mass}} \times 100\% = \frac{0.42g}{1.48g} \times 100\% = 28.4\%$

Therefore, the percentage of $NaHCO_3$ in the mixture is 28.4%.

(e) With strong acid (HCl), methyl orange shows end point after complete neutralisation of both sodium carbonate and sodium bicarbonate, skipping the phenolphthalein (POP) stage, which only detects partial neutralisation of carbonate to bicarbonate.

Consequently, the ability to separate the reactions of Na_2CO_3 and $NaHCO_3$ will be lost and hence eliminating the sequential distinction, making it impossible to calculate the percentage composition of the mixture.

Experiment 10

After acing her school science fair, Kipute landed a short internship at **Kisasa Bakery Industries**, where her reputation as a sharp Chemistry mind had already spread. One sunny afternoon, the quality assurance manager handed her a sample labeled **M1**, saying it was a "special raising agent" made by blending **sodium carbonate** and **sodium hydrogencarbonate**.

But something was off—the batch wasn't reacting properly in test bakes. The manager looked at her seriously and said:
"Time to put your titration instincts to the test, Kipute. We need the exact percentage composition before this batch hits the supermarket shelves. The cake depends on you!"

Unshaken and curious, Kipute adjusted her lab coat and got to work.

Materials Provided

- **M1**: 1 dm^3 of a solution containing a mixture of sodium carbonate and sodium hydrogencarbonate.
- **M2:** 0.2 M hydrochloric acid solution.
- **MO:** methyl orange indicator:
- **POP:** phenolphthalein indicator.
- Distilled water.

Procedure followed by Kipute

1. Measured 75 cm^3 of M2 and put into a 250 cm^3 beaker. She then added 75 cm^3 of distilled water into the beaker containing M2 and stirred the mixture using a glass rod. She labelled the resulting solution as M5.
2. She put solution M5 into the burette.
3. She pipetted 20 or 25 cm^3 of M1 into a conical flask and added two or three drops of POP.
4. Titrated M5 against MI until the first colour change is observed. She recorded the first titre value.
5. Added two or three drops of MO to the same solution in the conical flask.
6. Titrated until the second colour change is observed and recorded the second titre value.
7. Repeated steps (1) to (6) three times and she recorded the titre values.

Questions

a) Record the results in a tabular form.
b) (i) What was the volume of the pipette used?
   (ii) Calculate the average titre values (cm^3) of M5 when MO and POP were used.
c) Write the balanced chemical equation for the reaction under POP and under MO.
d) Explain why POP was used first instead of MO in this experiment?
e) Why was the original HCl solution (M2) diluted to make M5 before titration?
f) Why does the reaction with phenolphthalein stop before all the acid has been added?
g) What practical importance does this analysis have for the bakery industry?
h) Calculate the percentage composition of sodium hydrogencarbonate and sodium carbonate in the product.

Solution

(a) **TABLE OF RESULTS**

Experiment	**PILOT**	**1**	**2**	**3**
Final reading (cm^3) under MO	20.90	20.00	40.10	20.10
Final reading (cm^3) under POP	9.40	9.00	29.10	9.00
Initial reading (cm^3)	0.00	0.00	20.00	0.00
Volume used (cm^3) under POP	9.40	9.00	9.10	9.00
Volume used (cm^3) under MO	11.50	11.00	11.00	11.10

Making Sense of the Experimental Data

All three titre values (for both indicators) are within ±0.10 cm^3 of each other, indicating high precision and hence the experiment was successful.

Response to the Remaining Questions

(b)
(i) The volume of the pipette used was 20cm^3
(ii) Average volume used $= \frac{V_1+V_2+V_3}{3}$

$$\text{Average volume used under POP} = \left(\frac{9.00 + 9.10 + 9.00}{3}\right) cm^3 = 9.00cm^3$$

$$\text{Average volume used under MO} = \left(\frac{11.00 + 11.00 + 11.10}{3}\right)\text{cm}^3 = 11.00\text{cm}^3$$

For 25 mL pipette: With POP the volume will be **11.25cm^3** while with MO the volume will be **13.75cm^3**.

(c) Reaction equation for each case is as follows:

With POP:

$$Na_2CO_3 + HCl \rightarrow NaCl + NaHCO_3$$

With MO:

$$NaHCO_3 + HCl \rightarrow NaCl + H_2O + CO_2$$

(d) Phenolphthalein only detects equivalence point of strong base and in this case it shows end point when the strong base, sodium carbonate is half neutralized whereas methyl orange responds to both sodium carbonate and sodium hydrogencarbonate which is weak base. Therefore, using POP first allows us to separately determine the volume of HCl used for the carbonate portion alone before methyl orange is added to account the volume of HCl for the hydrogencarbonate; providing a clear distinction between the two stages of neutralization.

(e) To reduce the concentration of hydrochloric acid, making the titration process more controlled and accurate.

(f) Phenolphthalein only changes colour when all the sodium carbonate (Na_2CO_3) has partially (half) reacted. It does not indicate the neutralization of sodium hydrogencarbonate ($NaHCO_3$), which continues reacting beyond this point.

(g) The composition of sodium carbonate and sodium hydrogen carbonate affects the pH, leavening strength, and taste of baked goods. Overuse of one could lead to off-flavours or ineffective rise. The analysis ensures quality control and product consistency.

(h) Reaction equation with POP:

$$Na_2CO_3 + HCl \rightarrow NaCl + NaHCO_3;\ n_{acid} = n_{base} = 1$$

Using; $\frac{M_{acid} \times V_{acid}}{M_{base} \times V_{base}} = \frac{n_{acid}}{n_{base}}$

Where:

$V_{base} = 20\text{cm}^3$, $V_{acid} = 9\text{cm}^3$ $M_{acid} = \frac{M_cV_c}{V_d} = \frac{0.2\text{M} \times 75\ \text{cm}^3}{(75+75)\ \text{cm}^3} = 0.1\text{M}$

Substituting $\frac{0.1\text{M} \times 9\text{cm}^3}{M_{base} \times 20\text{cm}^3} = \frac{1}{1}$; $M_{base} = 0.045\text{M}$

$\text{Mass concentration of } Na_2CO_3 = \text{Molarity} \times \text{Molar mass of } Na_2CO_3$

$$= 0.045\text{mol/dm}^3 \times 106\text{g/mol} = 4.77\text{g/dm}^3$$

Reaction equation with MO:

$$NaHCO_3 + HCl \rightarrow NaCl + H_2O + CO_2;\ n_{acid} = n_{base} = 1$$

V_{HCl} reacted with original $NaHCO_3 = V_{HCl}$ under MO $- V_{HCl}$ under POP

$$= 11\text{cm}^3 - 9\text{cm}^3 = 2\text{cm}^3$$

Again using; $\frac{M_{acid} \times V_{acid}}{M_{base} \times V_{base}} = \frac{n_{acid}}{n_{base}}$

Where:

$V_{base} = 20\text{cm}^3$, $V_{acid} = 2\text{cm}^3$, $M_{acid} = 0.1\text{M}$

Substituting $\frac{0.1\text{M} \times 2\text{cm}^3}{\text{M}_{\text{base}} \times 20\text{cm}^3} = \frac{1}{1}$; $\text{M}_{\text{base}} = 0.01\text{M}$

$$\text{Mass concentration of NaHCO}_3 = \text{Molarity} \times \text{Molar mass of NaHCO}_3$$

$$= 0.01\text{mol/dm}^3 \times 84\text{g/mol} = 0.84\text{g/dm}^3$$

Total mass of Na_2CO_3 and $NaHCO_3$ in 1dm^3 of solution $= 5.61\text{g}$

Then mass percentage of $NaHCO_3 = \frac{\text{Mass of NaHCO}_3}{\text{Total mass}} \times 100\% = \frac{0.84\text{g}}{5.61\text{g}} \times 100\% = 14.97\%$

Therefore, the percentage of $NaHCO_3$ in the product is 14.97%.

And the percentage of Na_2CO_3 in the product is $(100 - 14.97)\% = 85.03\%$

Experiment 11

Kipute had been invited to assist in a special research demonstration at a nearby university. Her challenge? To determine the individual concentrations of **sodium carbonate** and **sodium hydroxide** from a **5.00 g mixture** dissolved in **1 dm³** of solution (Solution **BB**).

She was provided with:

- **Solution AB**: 0.1 M hydrochloric acid
- **Solution BC**: Dilute barium chloride
- **Methyl orange** and **phenolphthalein** indicators

Kipute knew that:

When the mixture of sodium carbonate and sodium hydroxide solution is titrated against hydrochloric acid using methyl orange indicator, all the mixture of the sodium carbonate and sodium hydroxide reacts because methyl orange indicator favours the reaction between weak base against strong acid and that of strong base against strong acid as shown by the reaction equations below:

$$NaOH + HCl \longrightarrow NaCl + H_2O$$

$$Na_2CO_3 + 2HCl \rightarrow 2NaCl + CO_2 + H_2O$$

In the second portion, equal volume of the mixture solution of the alkalis and barium chloride are mixed and POP indicator is added. The mixture then is titrated against hydrochloric acid solution where all carbonates are precipitated as barium carbonate ($BaCO_3$) as shown by the reaction equation below:

$$Na_2CO_3 + BaCl_2 \rightarrow BaCO_3 + 2NaCl$$

This implies, in the presence of barium chloride; hydrochloric acid reacts with sodium hydroxide only.

Thus; the volume of acid required to react with sodium carbonate only will be obtained as the difference between the volume of acid reacted with all alkalis under MO indicator and the volume of acid reacted with sodium hydroxide only under POP indicator in the presence of $BaCl_2$.

It was a perfect example of smart experimental design—and she was ready to get to work.

Procedure Kipute followed:

Part 1

1. She pipetted **20 cm³** of **Solution BB** into a clean titration flask.
2. She added **2–3 drops of methyl orange (MO)**.
3. She titrated with **Solution AB** until the colour change is observed.
4. She recorded results in a tabular form as shown below.

Experiment	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)				
Initial reading (cm^3)				
Titre value (cm^3)				

Part 2

1. She pipetted another **20 cm^3** of **solution BB** into a clean titration flask.
2. She added an equal volume of **Solution BC**.
3. She added **2–3 drops of phenolphthalein (POP)**.
4. She titrated with **Solution AB** until the colour change is observed.
5. She recorded results in a tabular form as shown below.

Experiment	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)				
Initial reading (cm^3)				
Titre value (cm^3)				

Questions

a) What is the function of barium chloride in Part 2?

b) Calculate:
   i. The volume of acid required to react with sodium hydroxide
   ii. The volume of an acid required to react with sodium carbonate

c) Calculate:
   i. The molarity of sodium hydroxide
   ii. The molarity of sodium carbonate

d) Calculate the percentage by mass of:
   i. Sodium hydroxide
   ii. Sodium carbonate

Solution

PART 1

The volume of the pipette used was 20cm^3

TABLE OF RESULTS

Experiment	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)	20.50	20.00	20.00	20.10
Initial reading (cm^3)	0.00	0.00	0.00	0.00
Titre value (cm^3)	20.50	20.00	20.00	20.10

$$\text{Average volume used} = \frac{V_1 + V_2 + V_3}{3}$$

$$\text{Average volume used under MO} = \left(\frac{20.20 + 20.00 + 19.80}{3}\right)\text{cm}^3 = 20.00\text{cm}^3$$

For 25 mL pipette: The volume of acid will be **25cm^3**.

Making Sense of the Experimental Data

All three titre values are within ±0.10 cm³ of each other, indicating high precision and hence the experiment was successful.

PART 2

Table of results

the volume of the pipette used was 20cm^3

Experiment	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)	10.00	19.70	29.30	38.90
Initial reading (cm^3)	0.00	10.00	19.70	29.30
Titre value (cm^3)	10.00	9.70	9.60	9.60

$$\text{Average volume used} = \frac{V_1 + V_2 + V_3}{3}$$

$$\text{Average volume used under MO} = \left(\frac{9.70 + 9.60 + 9.60}{3}\right)\text{cm}^3 = 9.60\text{cm}^3$$

Making Sense of the Experimental Data

All three titre values are within ±0.10 cm³ of each other, indicating high precision and hence the experiment was successful.

For 25 mL pipette: The volume of acid will be **12cm^3**.

(a) Barium chloride reacts with sodium carbonate to form insoluble barium carbonate ($BaCO_3$), which precipitates out:

$$Na_2CO_3(aq) + BaCl_2(aq) \rightarrow BaCO_3(s) + 2NaCl(aq)$$

This removes carbonate ions from the solution, preventing them from reacting with HCl. This allows the titration to measure NaOH only, isolating its contribution to total alkalinity.

(b)

(i) The volume of acid required to react with sodium hydroxide

$$= \text{volume of acid obtained under POP in the presence of } BaCl_2 = 9.60\text{cm}^3$$

The volume of acid required to react with sodium hydroxide was 9.60cm^3

(ii) The volume of an acid required to react with sodium carbonate

$$= V_{HCl} \text{ reacted under MO} - V_{HCl} \text{ reacted under POP}$$
$$= 20\text{cm}^3 - 9.60\text{cm}^3 = 10.40\text{cm}^3$$

The volume of acid required to react with sodium carbonate was 10.40cm^3

(c)

(i) The reaction equation:

$$NaOH + HCl \rightarrow NaCl + H_2O;\ n_{acid} = n_{base} = 1$$

Using; $\frac{M_{acid} \times V_{acid}}{M_{base} \times V_{base}} = \frac{n_{acid}}{n_{base}}$

Where:

$V_{base} = 20cm^3$,$V_{acid} = 9.6cm^3$, $M_{acid} = 0.1M$

Substituting $\frac{0.1M \times 9.6cm^3}{M_{base} \times 20cm^3} = \frac{1}{1}$; $M_{base} = 0.048M$

The molarity of sodium hydroxide was $0.048mol/dm^3$

(ii) The reaction equation;

$$Na_2CO_3 + 2HCl \rightarrow 2NaCl + CO_2 + H_2O;\ n_{acid} = 2, n_{base} = 1$$

$V_{base} = 20cm^3$, $M_{acid} = 0.1M$ and $V_{acid} = 10.40cm^3$

Substituting $\frac{0.1M \times 10.4cm^3}{M_{base} \times 20cm^3} = \frac{2}{1}$; $M_{base} = 0.026M$

The molarity of sodium carbonate was $0.026mol/dm^3$.

(d) Using $m = nM_r$ = molarity × volume × molar mass

(i) For NaOH

Mass of NaOH in $1dm^3 = 0.048mol/dm^3 \times 1dm^3 \times 40g/mol = 1.92g$

Total mass of Na_2CO_3 and NaOH in $1dm^3$ of solution = 5g (Given)

Then mass percentage of NaOH = $\frac{\text{Mass of NaOH}}{\text{Total mass}} \times 100\% = \frac{1.92g}{5g} \times 100\% = 38.4\%$

Therefore, the percentage of NaOH in in the mixture is 38.4%.

(ii) For Na_2CO_3

Mass of Na_2CO_3 in $1dm^3 = 0.026mol/dm^3 \times 1dm^3 \times 106g/mol = 2.756g$

Total mass of Na_2CO_3 and NaOH in $1dm^3$ of solution = 5g (Given)

Then mass percentage of $Na_2CO_3 = \frac{\text{Mass of } Na_2CO_3}{\text{Total mass}} \times 100\% = \frac{2.756g}{5g} \times 100\% = 55.12\%$

The percentage of Na_2CO_3 in in the mixture is 55.12%.

Chapter 3

REDOX TITRATIONS

INTRODUCTION

Redox titrations employ redox reactions to determine the concentration of either oxidising agent or reducing agent. There must be a sufficiently large difference between oxidising and reducing capabilities of these agents for the reaction to undergo completion with sharp end point. In these kind of titrations, oxidising agent is often put in the burette while reducing agent is put in the conical flask (titration flask) by using pipette.

Indicators in redox titrations

Like in acid-base titrations, indicators are important component of redox titration for end point detection. There are three common types of redox indicator, namely:

(i) Self-indicators
(ii) Internal indicators
(iii) External indicators

Self-indicators

When the titrant itself is so **strongly coloured** that after equivalence point, a single drop of the titrant (of negligible volume) produces an intense colour in the reaction mixture thus acting as an indicator; such indicators are known as self-indicators. A good example of self-indicator is potassium permanganate ($KMnO_4$) which is deep purple coloured and it is reduced by reductant to form Mn^{2+} which is colourless. After complete reduction of the analyte, the solution mixture is colourless at the equivalence point; and since the permanganate is strongly coloured, its single drop of negligible volume can change the colour of solution into pink thus detecting end point. ***However, although $K_2Cr_2O_7$ solution is strongly coloured and its single drop change the colour of colourless solution into yellow, it is not used as self-indicator like $KMnO_4$, why?***

This is because its reduction product, Cr^{3+}, it is green in colour and not colourless like Mn^{2+} which is reduction product of acidified $KMnO_4$. This hinders visual detection of the end point by observing the dichromate colour. Hence indicator must be used in titrations of dichromate solutions.

Internal indictors

Such indicators are added to the reaction mixture and they show their action through redox reaction. The indicators are always weaker reducing agent than the analyte so that they react with titrant only when whole of the analyte has consumed, producing a readily detectable colour change. A good example of internal indicator (also can be termed as redox indicator) is diphenylamine which is used in titrations of potassium dichromate (VI).

External indicators

In case a suitable redox indicator is not available for given system; an indicator may be employed which will indicate the completion of reaction by physically or chemically reacting with analyte **(not through redox reaction).** Such indicators are called external indicators. A good example of external indicator is **starch indicator** which is commonly used in titrations which involve iodine. With iodine, starch gives blue colouration which disappears after complete reduction of the iodine i.e. when the iodine is absent. Thus iodine is used to detect presence of iodine. ***However, when iodine is an analyte, the starch must be added just close to the end point, why?***

This is because in large amount of iodine (at the beginning of titration), starch form a complex with the iodine. The iodine in the complex resists reduction of reducing agents like sodium thiosulphate and therefore blue colouration cannot disappear and hence end point can never be reached.

Classification of redox titrations

Redox titrations are classified into different types based on the identity of reagent that is used as an oxidising agent in the titration. Important types which we are going to discuss in this book are:

- Permanganometry
- Dichrometry
- Iodine titration

Permanganometry

This is the type of redox titration in which potassium permanganate is used as an oxidising agent. During redox titration, $KMnO_4$ is reduced according to the following ionic equation:

$$\underset{\textbf{Deep purple}}{MnO_4^-} + 8H^+ + 5e \rightarrow \underset{\textbf{Manganous ion (colourless)}}{Mn^{2+}} + 4H_2O$$

Presence of $8H^+$ in the above equation suggest that an acidic medium is needed to supply the $8H^+$ions. To achieve this, dilute sulphuric acid is commonly used. Phosphoric acid can also be used (especially in titrating Fe^{2+}). However, not all acids can suit doing this work due to the following reasons:

1) Some acids have reducing character because they contain anion which can be oxidised by $KMnO_4$ which is very powerful oxidising agent. For example, hydrochloric acid cannot be used due to this reason.
2) Some acids have oxidising character and thus they can oxidise the analyte in the titration flask. For example; concentrated sulphuric acid or nitric acid cannot be used due to this reason.
3) Some acids are weak and thus they are incapable of supplying enough concentration of H^+ (eight times the concentration of $KMnO_4$). For example, ethanoic acid cannot be used due to this reason.

Practical tip: *If you skip to add the acidic medium or you add insufficient volume of sulphuric acid,* MnO_2 *which is brown in colour will be produced instead of the colourless* Mn^{2+}. *The brown* MnO_2 *will mask the colour change and lead to a greater (inaccurate) volume of permanganate being used in the titration.*

$$MnO_4^- + 2H_2O + 3e \rightarrow \underset{\textbf{Brown precipitate}}{MnO_2} + 4OH^- \text{ (In absence of acidic medium)}$$

$$MnO_4^- + 4H^+ + 3e \rightarrow \underset{\textbf{Brown precipitate}}{MnO_2} \qquad \text{(Not acidic enough (}4H^+\text{ instead of }8H^+\text{) due to the insufficient volume)}$$

Dichrometry

This is the type of redox titration in which potassium dichromate (VI), $K_2Cr_2O_7$ is used as an oxidising agent. Potassium dichromate is strong oxidising agent. However, it is not strong oxidising agent as potassium permanganate, $KMnO_4$.

During redox titration, $K_2Cr_2O_7$is reduced according to the following ionic equation:

$$\underset{\textbf{Orange}}{Cr_2O_7^{2-}} + 14H^+ + 6e \rightarrow \underset{\textbf{Green}}{2Cr^{3+}} + 7H_2O$$

Again, presence of $14H^+$ in the above equation suggest that an acidic medium is needed to supply that large amount of H^+ions. Unlike $KMnO_4$ which may do oxidation even in absence of acidic medium (in neutral and basic medium), $K_2Cr_2O_7$ acts as an oxidising agent in acidic medium only.

It is also worth to note that: Unlike in $KMnO_4$, hydrochloric acid may be used as an acidic medium in titrations involving $K_2Cr_2O_7$ because the dichromate is incapable of oxidising Cl^- to Cl_2.

Iodine titration

This is the type of redox titration in which iodine is used as an oxidising agent. During redox titration, iodine is reduced according to the following ionic equation:

$$I_2 + 2e \longrightarrow 2I^-$$

The iodine must be present directly in the beginning of titration or may be produced by oxidation of excess potassium iodide by an oxidising agent. Thus, iodine titration may further be divided into **iodometry** and **iodimetry**.

- **Iodimetry** *is the redox titration whereby an analyte which is reducing agent is titrated directly with standard iodine solution.*
- **Iodometry** *is the redox titration whereby an analyte which is an oxidising agent is added to excess iodide solution to liberate iodine whose amount is determined by titration with sodium thiosulphate*.

EXPERIMENTS

Experiment 12

It was time for the redox practicals, and Mr. Akilikubwa stood quietly by the fume hood, inspecting two crystalline substances:

- **Potassium permanganate ($KMnO_4$)**.
- **Oxalic acid ($H_2C_2O_4 \cdot XH_2O$)**.

He had prepared two solutions:

- **Solution A**: 0.79 g of $KMnO_4$ dissolved in 250 cm³ of distilled water.
- **Solution B**: 3.15 g of oxalic acid crystals dissolved in 500 cm³ of distilled water
- **Solution C**: Dilute sulphuric acid.

Mr. Akilikubwa goal was not only to determine the molarity of both solutions, but also to uncover the **unknown value of X**, the number of water molecules of crystallization in the oxalic acid.

He knew that:

The reaction between potassium permanganate and oxalic acid in the presence of sulphuric acid is represented by the ionic equation as follows:

$$MnO_4^- + C_2O_4^{2-} \longrightarrow Mn^{2+} + CO_2$$

One of the substances loses its electrons to the other.

Procedure Mr. Akilikubwa followed:

1. He pipetted **20 cm³** of **Solution B** into a titration flask.
2. He added an **equal volume of dilute sulphuric acid (Solution C)**.
3. He heated the solution gently in a water bath to around **60–70°C**.
4. While still hot, he titrated it with **Solution A ($KMnO_4$)** from the burette until colour changes from colourless to pink.
5. He repeated the titration to ensure accuracy and recorded results in a tabular form.

Summary:

_____cm³ of solution B required ______cm³ of solution A in the presence of solution C for complete reaction.

Questions

a) Write the balanced half ionic equation for:
   i. Oxidation
   ii. Reduction
b) Calculate:
   i. The molarity of A
   ii. The molarity of B
   iii. The value of X in $H_2C_2O_4.XH_2O$
c) Why was an indicator not used during titration process?
d) Why was the end point pink in colour?
e) Why hydrochloric acid is not used to provide acidic medium in this reaction?

Solution

The volume of the pipette used was $20cm^3$

TABLE OF RESULTS

Experiment	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)	20.20	40.30	20.00	40.00
Initial reading (cm^3)	0.00	20.20	0.00	20.00
Volume used (cm^3)	20.20	20.10	20.00	20.00

$$\text{Average volume used} = \frac{V_1 + V_2 + V_3}{3}$$

$$\text{Average volume used } = \left(\frac{20.10 + 20.00 + 20.00}{3}\right) cm^3 = 20.00cm^3$$

Summary:

$20cm^3$ of solution B required **$20.00cm^3$** of solution A in the presence of solution C for complete reaction.

For 25 mL pipette: The volume of A will be **$25cm^3$**.

Making Sense of the Experimental Data

Practical tip: *The fixed number of moles of water of crystallization in oxalic acid is 2. So the chemical formula of oxalic acid crystals is* $H_2C_2O_4.2H_2O$.

Thus, molar mass of $H_2C_2O_4.XH_2O$ is 126g/mol.

And $[H_2C_2O_4.XH_2O] = \frac{3.15g}{0.5L\times126gmol^{-1}} = 0.05M = M_{reductant}$

Also $[KMnO_4] = \frac{0.79g}{0.25L\times158gmol^{-1}} = 0.02M = M_{oxidant}$

$KMnO_4$ reacts with $H_2C_2O_4.XH_2O$ according to the following ionic equation:

$$2MnO_4^- + 5C_2O_4^{2-} + 16H^+ \rightarrow 2Mn^{2+} + 10CO_2 + 8H_2O;\ n_{oxidant} = 2, n_{reductant} = 5$$

Using $\frac{M_{oxidant}\times V_{oxidant}}{M_{reductant}\times V_{reductant}} = \frac{n_{oxidant}}{n_{reductant}}$

Substituting $\frac{0.02M\times V_{oxidant}}{0.05M\times 20cm^3} = \frac{2}{5}$; $V_{oxidant} = \mathbf{20cm^3}$ = experimental data

To conclude: *The calculated volume of $KMnO_4$ agrees with the titre volume obtained from the experiment.*

Response to Questions

(a) The balanced half ionic equation for:

(i) Oxidation

$$C_2O_4^{2-} \rightarrow 2CO_2 + 2e$$

(ii) Reduction

$$MnO_4^- + 8H^+ + 5e \rightarrow Mn^{2+} + 4H_2O$$

(b) The calculation of each part is as follows:

(i) Using $[\ \ ] = \frac{n}{V} = \frac{m}{M_r \times V}$

Then $[A] = [KMnO_4] = \frac{0.79g}{0.25L \times 158gmol^{-1}} = 0.02M$

The molarity of A is $0.02mol/dm^3$.

(ii) The overall reaction equation:

$$2MnO_4^- + 5C_2O_4^{2-} + 16H^+ \rightarrow 2Mn^{2+} + 10CO_2 + 8H_2O;\ n_{oxidant} = 2, n_{reductant} = 5$$

Using $\frac{M_{oxidant} \times V_{oxidant}}{M_{reductant} \times V_{reductant}} = \frac{n_{oxidant}}{n_{reductant}}$

Where:

$$M_{oxidant} = 0.02M, V_{oxidant} = 20cm^3, V_{reductant} = 20cm^3$$

Substituting $\frac{0.02M \times 20cm^3}{M_{reductant} \times 20cm^3} = \frac{2}{5};\ M_{reductant} = 0.05M$

The molarity of B is $0.05mol/dm^3$.

(iii) Using molar mass of $H_2C_2O_4.XH_2O$

$$= \frac{\text{Mass concentration}}{\text{Molar concentration}} = \frac{\frac{3.15g}{0.5dm^3}}{0.05mol/dm^3} = 126g/mol$$

But also;

Molar mass of $H_2C_2O_4.XH_2O$

$$= \left((2 \times 1) + (2 \times 12) + (4 \times 16) + 18X\right)g/mol = (90 + 18X)g/mol$$

Thus, $90 + 18X = 126;\ X = 2$

Therefore; the value of X is 2.

(c) This is because $KMnO_4$ acts as a self-indicator and the end point is recognised when its last drop change the colourless solution to pink.

(d) This is due to presence of very small amount of unreacted $KMnO_4$ just after the equivalence point of the reaction.

(e) This is because potassium permanganate ($KMnO_4$) is so strong oxidising agent that can oxidise Cl^- from HCl thus interfering the measurement of volume of $KMnO_4$ which was exactly used to oxidise oxalic acid in the titration.

$$2MnO_4^- + 10Cl^- + 16H^+ \rightarrow 2Mn^{2+} + 5Cl_2 + 8H_2O$$

Experiment 13

When the district hospital's water lab reported inconsistencies in test results for oxidizing agents, they requested assistance from the school laboratory. Mr. Akilikubwa, with his experience in reagent quality verification, agreed to help.

He was handed a jar labelled:

"**Potassium Permanganate – for standard lab use, Net: 5.00 g**"

But something felt off. The crystals looked slightly dull, and the batch was from a new supplier. Before recommending its use in critical hospital analyses like **water treatment** and **disinfection checks**, Mr. Akilikubwa had to verify its **purity**.

He decided to analyze the sample by titrating it against **sodium oxalate**, a reliable reducing agent, under **acidic and heated conditions**, where permanganate undergoes a redox reaction.

Titration procedure Mr. Akilikubwa followed

1. He dissolved **5.00 g of impure potassium permanganate** in water and diluted it to **250 cm³** — this was **Solution G**.
2. He prepared **Solution H** by dissolving **4.00 g of sodium oxalate** in **250 cm³** of distilled water.
3. He pipetted **20 cm³ of Solution H** into a clean titration flask.
4. He added 15cm^3 of **solution of 1M sulphuric acid (Solution I)** to the flask.
5. He heated the mixture gently to about **50°C** to **60°C.**
6. He titrated with Solution G (2 to 5 drops of G were enough) until a **faint pink** colour persisted.
7. He repeated the titration to obtain consistent results and recorded the readings below.

Titration number	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)				
Final reading (cm^3)				
Volume used (cm^3)				

Summary:

________cm^3 of H required _______cm^3 of G in the presence of I for complete reaction.

Questions

a) Write a balanced half ionic equation for
   i. Oxidation
   ii. Reduction
b) Give explanation for the following:
   i. Why sulphuric acid was added initially in the sodium oxalate?
   ii. Why was the solution mixture warmed and titrated against solution H while still hot?
c) Calculate the following:
   i. Molarity of H
   ii. Molarity of G
   iii. Concentration of G in g/dm^3
   iv. Percentage purity of potassium permanganate

Solution

The volume of the pipette used was 20cm^3

TABLE OF RESULTS

Titration number	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)	13.80	27.50	41.10	13.60
Final reading (cm^3)	0.00	13.80	27.50	0.00
Volume used (cm^3)	13.80	13.70	13.60	13.60

$$\text{Average volume used} = \frac{V_1 + V_2 + V_3}{3}$$

$$\text{Average volume used } = \left(\frac{13.70 + 13.60 + 13.60}{3}\right)\text{cm}^3 = 13.60\text{cm}^3$$

Summary:

20cm^3 of solution H required $\mathbf{13.60cm^3}$ of solution G in the presence of solution I for complete reaction.

For 25 mL pipette: The volume of solution G will be **17cm^3**.

Making Sense of the Experimental Data

All three titre values are within ±0.10 cm³ of each other, indicating high precision and hence the experiment was successful.

Response to Questions

(a) The balanced half ionic equation for:

(i) Oxidation

$$C_2O_4^{2-} \rightarrow 2CO_2 + 2e$$

(ii) Reduction

$$MnO_4^- + 8\,H^+ + 5e \rightarrow Mn^{2+} + 4\,H_2O$$

(b)

(i) Sulphuric acid was added to provide an **acidic medium** for the redox reaction between potassium permanganate and sodium oxalate to occur.

(ii) The mixture was warmed to increase the rate of reaction between potassium permanganate and sodium oxalate.

(c) The calculation of each part is as follows:

(i) Using $[\ \ ] = \frac{n}{V} = \frac{m}{M_r \times V}$

Then $[H] = [Na_2C_2O_4] = \frac{4g}{0.25L \times 134gmol^{-1}} = 0.1194M$

The molarity of H is $0.1194mol/dm^3$.

(ii) The overall reaction equation:

$$2MnO_4^- + 5C_2O_4^{2-} + 16H^+ \rightarrow 2Mn^{2+} + 10CO_2 + 8H_2O;\ n_{oxidant} = 2, n_{reductant} = 5$$

Using $\frac{M_{oxidant} \times V_{oxidant}}{M_{reductant} \times V_{reductant}} = \frac{n_{oxidant}}{n_{reductant}}$

Where:

$$M_{reductant} = 0.1194M, V_{oxidant} = 13.6cm^3, V_{reductant} = 20cm^3$$

Substituting $\frac{M_{oxidant} \times 13.6cm^3}{0.1194M \times 20cm^3} = \frac{2}{5}$; $M_{oxidant} = 0.07M$

The molarity of G is $0.07mol/dm^3$.

(iii) $\text{Concentration of G in } g/dm^3 = \text{molarity} \times \text{molar mass}$

$$= 0.07mol/dm^3 \times 158g/mol = 11.06g/dm^3$$

The concentration of G in g/dm^3 was $11.06g/dm^3$.

(iv) $\text{Mass of pure } KMnO_4 \text{ in } 250cm^3 (0.25dm^3) = \text{Mass concentration} \times \text{Volume}$

$$= 11.06g/dm^3 \times 0.25dm^3 = 2.765g$$

$\text{Mass of impure } KMnO_4 \text{ in } 250cm^3 = 5g \text{ (Given)}$

Then mass percentage purity $= \frac{\text{Mass of pure}}{\text{Mass of impure}} \times 100\% = \frac{2.765g}{5g} \times 100\% = 55.3\%$

The percentage purity of potassium permanganate was 55.3%.

Experiment 14

During a chemistry research camp, Kipute volunteered to analyze samples collected from an old university storage shelf. One suspicious-looking label caught her attention:

"**Reducing Agent Mixture – Contains Oxalic Acid and Sodium Oxalate,**"

The research team needed to determine how much of each compound was present in the mixture. This was crucial because **oxalic acid and sodium oxalate behave differently** in redox reactions and have different reducing powers — especially in pharmaceutical and dye industries.

Kipute took the challenge. She planned to use **acidified potassium permanganate**, knowing that both components would react with it in a redox process. But unlike previous titrations, this sample was a **mixture**, and she would have to **calculate backwards** to separate the contributions of each component.

She prepared the experiment with full understanding of the following theory:

When the mixture of sodium oxalate and oxalic acid is titrated against sodium hydroxide, only oxalic acid in the mixture reacts with sodium hydroxide as per reaction equation below:

$$H_2C_2O_4(aq) + 2NaOH(aq) \rightarrow Na_2C_2O_4(aq) + H_2O(l)$$

When potassium permanganate is titrated against the solution mixture of sodium oxalate and oxalic acid in the presence of sulphuric acid, both oxalate ions from sodium oxalate and oxalic acid both react with potassium permanganate as per reaction equation below:

$$MnO_4^-(aq) + C_2O_4^{2-}(aq) \longrightarrow Mn^{2+}(aq) + CO_2(g)$$

Titration Procedure Kipute followed:

1. **She prepared the following:**

AB: A solution mixture made by dissolving anhydrous sodium oxalate ($Na_2C_2O_4$) and oxalic acid ($H_2C_2O_4.2H_2O$).

AC: A solution of potassium permanganate made by dissolving 3.16g in 1dm^3.

AD: 0.1M solution of sodium hydroxide.

AF: 1M sulphuric acid.

Phenolphthalein indicator and thermometer$(0 - 100°C)$

2. **PART A**
   - (i) She Pipetted 20 or 25cm^3 of solution of solution AB into a titration flask.
   - (ii) She added 2 to 3 drops of phenolphthalein indicator.
   - (iii) She titrated this solution against AD from the burette until colour change is observed.
   - (iv) She recorded the volume of AD used.
   - (v) She repeated procedure (i) to (ii) three times.
   - (vi) She recorded results in a tabular form as shown below:

Titration number	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)				
Initial reading (cm^3)				
Volume used (cm^3)				

Summary:

_______cm^3 of solution AB required _______cm^3 of solution AD for complete reaction

3. **PART B:**
   - (i) She pipetted 20 cm^3 of solution AB into a titration flask.
   - (ii) She measured equal amount of solution AF into a titration flask containing AB.
   - (iii) Heat the solution mixture in the titration flask to about $70°C$ in the water bath.
   - (iv) She titrated the hot solution mixture in the titration flask against AC from the burette until permanent colour change is observed.
   - (v) She recorded the volume of AC used.
   - (vi) She repeated procedure (i) to (v) three times
   - (vii) She recorded results in a tabular form as shown below:

Titration number	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)				
Initial reading (cm^3)				
Volume used (cm^3)				

Summary:

_______cm^3 of solution AB required _______cm^3 of solution AC for complete reaction.

Questions

a) Calculate the concentration of oxalic acid in $\mathrm{mol/dm^3}$
b) Calculate the concentration of sodium oxalate in $\mathrm{mol/dm^3}$
c) The redox reaction between permanganate ($\mathrm{MnO_4^-}$) and oxalate ions ($\mathrm{C_2O_4^{2-}}$) is kinetically slow at room temperature and thus it is undertaken at elevated temperatures (~70°C).
   - (i) Why the reaction is slow?
   - (ii) If potassium permanganate is added too quickly during the redox titration, how could this affect the titre volume?
   - (iii) What error would occur if oxalic acid is overheated during the experiment? Explain.
   - (iv) Why can both sodium oxalate and oxalic acid act as reducing agents in the same redox titration, even though one is a salt and the other an acid?

Solution

PART A

The volume of the pipette was 20cm^3

TABLE OF RESULTS

Titration number	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)	23.40	23.20	23.20	23.10
Initial reading (cm^3)	0.00	0.00	0.00	0.00
Volume used (cm^3)	23.40	23.20	23.20	23.10

$$\text{Average volume used} = \frac{V_1 + V_2 + V_3}{3}$$

$$\text{Average volume used } = \left(\frac{23.20 + 23.20 + 23.10}{3}\right)\text{cm}^3 = 23.20\text{cm}^3$$

Summary:

20cm^3 of solution AB required $\mathbf{23.20cm^3}$ of solution AD for complete reaction.

For 25 mL pipette: The volume of solution AD will be **29cm^3**.

Making Sense of the Experimental Data

All three titre values are within ±0.10 cm³ of each other, indicating high precision and hence the experiment was successful.

PART B

The volume of the pipette was 20cm^3

TABLE OF RESULTS

Titration number	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)	31.30	31.30	31.20	31.20
Initial reading (cm^3)	0.00	0.00	0.00	0.00
Volume used (cm^3)	31.30	31.30	31.20	31.20

$$\text{Average volume used} = \frac{V_1 + V_2 + V_3}{3}$$

$$\text{Average volume used } = \left(\frac{31.30 + 31.20 + 31.20}{3}\right)\text{cm}^3 = 31.20\text{cm}^3$$

Summary:

20cm^3 of solution AB required $\mathbf{31.20cm^3}$ of solution AC for complete reaction.

For 25 mL pipette: The volume of solution AC will be **39cm^3**.

Making Sense of the Experimental Data

All three titre values are within ±0.10 cm³ of each other, indicating high precision and hence the experiment was successful.

Response to Questions

(b) NaOH reacts with $H_2C_2O_4$. According to the following equation:

$H_2C_2O_4(aq) + 2NaOH(aq) \rightarrow Na_2C_2O_4(aq) + 2\,H_2O(l);\ n_{acid} = 1,\ n_{base} = 2$

Using; $\frac{M_{acid} \times V_{acid}}{M_{base} \times V_{base}} = \frac{n_{acid}}{n_{base}}$

Where:

$M_{base} = 0.1M,\ V_{acid} = 20cm^3,\ V_{base} = 20cm^3$

Substituting $\frac{M_{acid} \times 20cm^3}{0.1M \times 23.2cm^3} = \frac{1}{2};\ M_{acid} = 0.058M$

The concentration of oxalic acid in mol/dm^3 was $0.058mol/dm^3$.

(b) Permanganate ions and oxalate ions react according to the following equation:

$$2MnO_4^- + 5C_2O_4^{2-} + 16H^+ \rightarrow 2Mn^{2+} + 10CO_2 + 8H_2O;\ n_{oxidant} = 2, n_{reductant} = 5$$

The potassium permanganate reacts with both oxalic acid and sodium oxalate. But since the concentration of oxalic acid is already known, the volume of permanganate reacted with oxalic acid can be found by using:

$$\frac{M_{oxidant} \times V_{oxidant}}{M_{reductant} \times V_{reductant}} = \frac{n_{oxidant}}{n_{reductant}}$$

Where:

$$M_{reductant} = 0.058M\ \big(\text{from (a)}\big), V_{reductant} = 20cm^3, M_{oxidant} = \frac{3.16g}{1L \times 158gmol^{-1}} = 0.02M$$

Substituting $\frac{0.02M \times V_{oxidant}}{0.058M \times 20cm^3} = \frac{2}{5};\ V_{oxidant} = 23.2cm^3$

Thus $23.2cm^3$ of potassium permanganate reacted with the oxalic acid.

So volume of the permanganate reacted with sodium oxalate $(31.2 - 23.2)cm^3 = 8cm^3$.

Now, the concentration of sodium oxalate can be found by using the same formula;

$$\frac{M_{oxidant} \times V_{oxidant}}{M_{reductant} \times V_{reductant}} = \frac{n_{oxidant}}{n_{reductant}}$$

Where:

$$V_{oxidant} = 8cm^3, V_{reductant} = 20cm^3, M_{oxidant} = 0.02M$$

Substituting $\frac{0.02M \times 8cm^3}{M_{reductant} \times 20cm^3} = \frac{2}{5};\ M_{reductant} = 0.02M$

The concentration of sodium oxalate was $0.02mol/dm^3$.

(c)

(i) Both permanganate and oxalate ions being negative charge means that they repel each other leading to high activation energy for the reaction and hence the reaction is not kinetically favoured.

(ii) Since the reaction is slow, Adding $KMnO_4$ too quickly (especially early in the titration), prevents enough time to allow the redox reaction to take place completely. This leads to overshooting the endpoint, because excess unreacted $KMnO_4$ remains in solution and hence titre volume will be greater than $31.20cm^3$.

(iii) The titre volume of $KMnO_4$ would be smaller than the obtained one ($31.20cm^3$).

Explanation:

Oxalic acid is thermally unstable at high temperatures. If the solution is overheated (beyond 90°C), it can begin to decompose leading to decreased amount of oxalate ions which are responsible for reducing the permanganate.

$$H_2C_2O_4 \xrightarrow[90°C]{} CO + CO_2 + H_2O$$

(iv) Both oxalic acid and sodium oxalate contain oxalate ion. The reducing ability in the redox titration comes from this ion, which is oxidized to carbon dioxide according to the following equation:

$$C_2O_4^{2-} \rightarrow 2CO_2 + 2e$$

Experiment 15

While preparing for her NECTA chemistry practicals, Kipute was handed a suspicious sample of **potassium permanganate**. It looked slightly dull — not the vibrant purple she expected.

"This might be impure," said her lab instructor. *"You'll have to test it."*

To uncover the truth, Kipute was provided with the following:

OH: A solution containing 9.8g of pure ammonium iron (II) sulphate ($(NH_4)_2Fe(SO_4)_2.6H_2O$) in 250cm^3 of dilute sulphuric acid.

OI: A solution of impure potassium permanganate made by dissolving 2.1g in 500cm^3 of distilled water.

OJ: A dilute sulphuric acid.

Kipute was knowledgeable enough to understand that potassium permanganate reacts with iron (II) sulphate in the presence of sulphuric acid according to the reaction below:

$$10FeSO_4 + 2KMnO_4 + 8H_2SO_4 \longrightarrow 5F_2(SO_4)_3 + K_2SO_4 + 2MnSO_4 + 8H_2O$$

Titration procedure Kipute followed:

1. Pipetted 20 cm^3 of OH into a clean titration flask.
2. Added to it an equal volume of OJ.
3. Titrated this solution mixture against OI until pink colour is observed.
4. Repeated procedure (i) to (ii) three times.
5. Recorded her results in a tabular form as follows:

Titration number	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)				
Initial reading (cm^3)				
Volume used (cm^3)				

Summary:

_______cm^3 of solution OH required _______cm^3 of solution OI for complete reaction.

Questions

a) Write the balanced half ionic equation for:
   i. Reduction
   ii. Oxidation
b) Why ammonium iron (II) sulphate was used but not iron (II) sulphate?
c) Is it possible to use acetic acid as the acidic medium in this titration? Explain.
d) Calculate the molarity of:
   i. Iron (II) solution
   ii. Potassium permanganate
e) Calculate the percentage purity of potassium permanganate.

Solution

The volume of the pipette used was $20cm^3$

TABLE OF RESULTS

Titration number	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)	20.30	20.10	20.00	20.00
Initial reading (cm^3)	0.00	0.00	0.00	0.00
Volume used (cm^3)	20.30	20.10	20.00	20.00

$$\text{Average volume used} = \frac{V_1 + V_2 + V_3}{3}$$

$$\text{Average volume used } = \left(\frac{20.10 + 20.00 + 20.00}{3}\right)\text{cm}^3 = 20.00\text{cm}^3$$

Summary:

$20cm^3$ of solution OH required $\mathbf{20.00cm^3}$ of solution OI for complete reaction.

For 25 mL pipette: The volume of solution OI will be **$25cm^3$**.

Making Sense of the Experimental Data

Practical tip: *Pure potassium permanganate is commonly (not always) standardized to 0.02M.*

So in our formula, $\frac{M_{oxidant}\times V_{oxidant}}{M_{reductant}\times V_{reductant}} = \frac{n_{oxidant}}{n_{reductant}}$; $M_{oxidant} = 0.02M$.

With $V_{reductant} = 20cm^3$ (pipette volume),

And $[(NH_4)_2Fe(SO_4)_2.6H_2O] = \frac{9.8g}{0.25dm^3\times392g/mol} = 0.1M = M_{reductant}$; the volume of solution of potassium permanganate can be calculated as follows:

$\frac{0.02M\times V_{oxidant}}{0.1M\times20cm^3} = \frac{2}{10}$ ($n_{oxidant}$ and $n_{reductant}$ can be obtained from the given reaction equation).

From which; $V_{oxidant} = \mathbf{20cm^3}$ = exprimental data

To conclude: *The calculated volume of $KMnO_4$ solution agrees with the titre volume obtained from the experiment.*

Response to Questions

a) The balanced half ionic equation for:

(i) Reduction

$$MnO_4^- \quad + \ 8\,H^+ \quad + \ 5e \ \rightarrow \ Mn^{2+} \quad + \ 4\,H_2O$$

(ii) Oxidation

$$Fe^{2+} \ \rightarrow \ Fe^{3+} \ + e$$

b) Unlike iron(II) sulphate, ammonium iron(II) sulphate is **resistant to oxidation by moist air**, making it more stable during handling and storage. Additionally, its **relatively large molar mass** reduces weighing errors, ensuring greater accuracy in the preparation of standard solutions.

c) No, acetic acid is not suitable.

Explanation

Acetic acid is weak acid and thus it provides fewer H^+ ions, which are essential for ensuring strong oxidizing power of potassium permanganate in the redox reaction. In insufficient amount of H^+ ions, the reduction of the permanganate will be incomplete and hence the end point will be unclear.

d) The calculation of each part is as follows:

(i) Using $[\quad] = \frac{n}{V} = \frac{m}{M_r \times V}$

Then $[Fe^{2+}(aq)] = [(NH_4)_2Fe(SO_4)_2.6H_2O] = \frac{9.8g}{0.25dm^3 \times 392g/mol} = 0.1moldm^{-3}$

The concentration of iron (II) solution is $0.1moldm^{-3}$

(ii) The overall reaction equation:

$$MnO_4^- \ + \ 5Fe^{2+} \ + \ 8H^+ \ \rightarrow \ Mn^{2+} \ + \ 5Fe^{3+} \ + 4H_2O; \ n_{oxidant} = 1, n_{reductant} = 5$$

Using $\frac{M_{oxidant} \times V_{oxidant}}{M_{reductant} \times V_{reductant}} = \frac{n_{oxidant}}{n_{reductant}}$

Where:

$$M_{reductant} = 0.1M, V_{oxidant} = 20cm^3, V_{reductant} = 20cm^3$$

Substituting $\frac{M_{oxidant} \times 20cm^3}{0.1M \times 20cm^3} = \frac{1}{5}$; $M_{oxidant} = 0.02M$

The concentration of potassium permanganate is $0.02moldm^{-3}$.

(e) Mass of pure $KMnO_4$ in $500cm^3 (0.5dm^3) = \text{Molarity} \times \text{Volume} \times \text{Molar mass}$

$$= 0.02mol/dm^3 \times 0.5dm^3 \times 158g/mol = 1.58g$$

Mass of impure $KMnO_4$ in $500cm^3 = 2.1g$ (Given)

Then mass percentage purity $= \frac{\text{Mass of pure}}{\text{Mass of impure}} \times 100\% = \frac{1.58g}{2.1g} \times 100\% = 75.2\%$

The percentage purity of potassium permanganate is 75.2%.

Experiment 16

In the school laboratory, Mr. Akilikubwa came across a small amber bottle labelled only:

"Ammonium Iron(II) Sulphate"

No expiry date. No hydration value. Just the smell of uncertainty.

Hydrated salts like this are commonly used in **iron analysis**, but without knowing the value of **x** in its formula — $(NH_4)_2Fe(SO_4)_2 \cdot xH_2O$; preparing accurate solutions becomes guesswork. And in industrial labs, **precision is everything**.

He decided to solve the mystery by titrating the sample against **acidified potassium permanganate**, which reacts directly with Fe^{2+}.

To accomplish the analysis, he was provided with the following:

AM: A solution containing 4.91g of impure ammonium iron (II) sulphate $((NH_4)_2Fe(SO_4)_2.xH_2O)$ in 125cm^3 of distilled water.

AN: 0.005mol of $KMnO_4$ in 250cm^3 of distilled water.

AO: Dilute solution of sulphuric acid

As experienced lab technician, Mr. Akilikubwa is full aware of the fact that potassium permanganate reacts with ammonium iron (II) sulphate in acidic medium as shown by the ionic equation below:

$$MnO_4^- + Fe^{2+} \rightarrow Mn^{2+} + Fe^{3+}$$

The end point of the reaction is detected by the change in colour of the solution.

Titration Procedure Mr. Akilikubwa followed:

1. Pipetted 20 cm^3 of AM into a clean 250cm^3 conical flask.
2. Using the measuring cylinder, measured an equal volume of AO into the conical flask containing AM.
3. Titrated the solution mixture in the conical flask against AN from the burette until the colour change is observed.
4. Repeated procedure (i) to (iii) three times.
5. Recorded his results in tabular form as shown below:

Titration number	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)				
Initial reading (cm^3)				
Volume used (cm^3)				

Summary:

_______cm^3 of solution AM required _______cm^3 of solution AN for complete reaction.

Questions

a) Write the balanced half ionic equation for:
   i. Reduction of MnO_4^- to Mn^{2+}
   ii. Oxidation of Fe^{2+} to Fe^{3+}
b) Calculate the molar concentration of AM
c) Calculate the value of x in$(NH_4)_2Fe(SO_4)_2 .xH_2O$
d) Calculate the percentage by mass of Fe in $(NH_4)_2Fe(SO_4)_2.xH_2O$
e) What could happen if Mr. Akilikubwa forgot to add or just added $5cm^3$ (instead of $20cm^3$) of sulphuric acid before titrating?

Solution

The volume of the pipette used was 20cm^3

TABLE OF RESULTS

Titration number	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)	20.10	20.00	19.80	20.00
Initial reading (cm^3)	0.00	0.00	0.00	0.00
Volume used (cm^3)	20.10	20.00	19.90	20.00

$$\text{Average volume used} = \frac{V_1 + V_2 + V_3}{3}$$

$$\text{Average volume used } = \left(\frac{20.00 + 19.80 + 20.00}{3}\right)\text{cm}^3 = 20.00\text{cm}^3$$

Summary:

20cm^3 of solution AM required $\mathbf{19.90cm^3}$ of solution AN in the presence of solution AO for complete reaction.

For 25 mL pipette: The volume of AN will be **25cm^3**.

Making Sense of the Experimental Data

Practical tip: *The fixed number of moles of water of crystallization in ammonium iron (II) sulphate (Mohr’s’ salt) is 6. So the chemical formula of ammonium iron (II) sulphate is* $(NH_4)_2Fe(SO_4)_2.6H_2O$.

Thus, molar mass of $(NH_4)_2Fe(SO_4)_2.xH_2O$ is 392g/mol.

And $[(NH_4)_2Fe(SO_4)_2.xH_2O] = \frac{4.91g}{0.125L\times 392gmol^{-1}} = 0.1M = M_{reductant}$

Also $[KMnO_4] = \frac{0.005mol}{0.25L} = 0.02M = M_{oxidant}$

$KMnO_4$ reacts with $(NH_4)_2Fe(SO_4)_2.6H_2O$ according to the following ionic equation:

$$MnO_4^- + 5Fe^{2+} + 8H^+ \rightarrow Mn^{2+} + 5Fe^{3+} + 4H_2O;\ n_{oxidant} = 1, n_{reductant} = 5$$

Using $\frac{M_{oxidant}\times V_{oxidant}}{M_{reductant}\times V_{reductant}} = \frac{n_{oxidant}}{n_{reductant}}$

Substituting $\frac{0.02M\times V_{oxidant}}{0.1M\times 20cm^3} = \frac{1}{5}$; $V_{oxidant} = \mathbf{20cm^3}$ = experimental data

To conclude: *The calculated volume of* $KMnO_4$ *agrees with the titre volume obtained from the experiment.*

Response to Questions

a) The balanced half ionic equation for:

(i) Reduction

$$MnO_4^- + 8H^+ + 5e \rightarrow Mn^{2+} + 4H_2O$$

(ii) Oxidation

$$Fe^{2+} \rightarrow Fe^{3+} + e$$

b) The overall reaction equation:

$$\mathrm{MnO_4^-} + \mathrm{5Fe^{2+}} + \mathrm{8H^+} \rightarrow \mathrm{Mn^{2+}} + \mathrm{5Fe^{3+}} + \mathrm{4H_2O};\ \mathrm{n_{oxidant}} = 1, \mathrm{n_{reductant}} = 5$$

Using $\frac{\mathrm{M_{oxidant}} \times \mathrm{V_{oxidant}}}{\mathrm{M_{reductant}} \times \mathrm{V_{reductant}}} = \frac{\mathrm{n_{oxidant}}}{\mathrm{n_{reductant}}}$

Where:

$$\mathrm{V_{oxidant}} = 20\mathrm{cm^3}, \mathrm{V_{reductant}} = 20\mathrm{cm^3};\ \mathrm{M_{oxidant}} = \frac{\mathrm{n}}{\mathrm{V}} = \frac{0.005\mathrm{mol}}{0.25\mathrm{L}} = 0.02\mathrm{M}$$

Substituting $\frac{0.02\mathrm{M} \times 20\mathrm{cm^3}}{\mathrm{M_{reductant}} \times 20\mathrm{cm^3}} = \frac{1}{5};\ \mathrm{M_{reductant}} = 0.1\mathrm{M}$

The concentration of AM is $0.1\mathrm{moldm^{-3}}$.

(c) Using molar mass $= \frac{\text{Mass concentration}}{\text{Molar concentration}}$

Where mass concentration of $\mathrm{(NH_4)_2Fe(SO_4)_2.xH_2O} = \frac{4.91\mathrm{g}}{0.125\mathrm{L}} = 39.28\mathrm{g/L}$

So, molar mass of $\mathrm{(NH_4)_2Fe(SO_4)_2.xH_2O} = \frac{39.28\mathrm{g/L}}{0.1\mathrm{mol/L}} = 392.8\mathrm{g/mol}$

But also, molar mass of $\mathrm{(NH_4)_2Fe(SO_4)_2.xH_2O} = (284 + 18\mathrm{x})\mathrm{g/mol}$

Thus, $284 + 18\mathrm{x} = 392.8$ or $\mathrm{x} = 6$

The value of x in$\mathrm{(NH_4)_2Fe(SO_4)_2\ .xH_2O}$ is 6.

(d) Mass percentage of Fe $= \frac{\text{Atomic mass of Fe}}{\text{Molecular mass of } \mathrm{(NH_4)_2Fe(SO_4)_2\ .6H_2O}} \times 100\%$

$$= \frac{56}{392} \times 100\% = 14.3\%$$

The percentage by mass of Fe in $\mathrm{(NH_4)_2Fe(SO_4)_2\ .6H_2O}$ was 14.3%.

(e) In the absence of or insufficient amount of acidic medium, $\mathrm{KMnO_4}$ undergo undesired side reactions which produces brown precipitate instead of soluble $\mathrm{Mn^{2+}}$. Consequently, cloudy or muddy solution is formed making the endpoint hard to detect.

$$\mathrm{MnO_4^-} + \mathrm{2H_2O} + 3\mathrm{e} \rightarrow \underset{\textbf{Brown precipitate}}{\mathrm{MnO_2}} + \ \mathrm{4OH^-}$$ (In absence of acidic medium)

$$\mathrm{MnO_4^-} + \mathrm{4H^+} + 3\mathrm{e} \rightarrow \underset{\textbf{Brown precipitate}}{\mathrm{MnO_2}}$$ (Not acidic enough ($\mathrm{4H^+}$ instead of $\mathrm{8H^+}$) due to the insufficient volume)

Experiment 17

At the nearby health clinic, a technician approached Kipute with a request.

"We've received a batch of hydrogen peroxide, but the label is smudged. We need to know its ***volume strength*** *to determine if it's still fit for sterilizing instruments."*

Kipute, always eager to apply classroom chemistry to real-world use, agreed instantly. **Hydrogen peroxide** is commonly used in disinfecting surfaces, tools, and even in wound care but only if it's at the correct **oxidizing power**.

She decided to determine the **volume strength** of the solution through a **redox titration** with potassium permanganate in acidic medium.

She was provided with the following:

HA: A solution of hydrogen peroxide prepared by diluting $1\mathrm{cm^3}$ of concentrated hydrogen peroxide to $250\mathrm{cm^3}$.

HB: A solution of potassium permanganate made by dissolving 0.79g in $250\mathrm{cm^3}$ of solution.

HC: A dilute solution of sulphuric acid.

Titration Procedure Kipute followed:

1. Pipetted 20 or $25cm^3$ of HA into a clean $250cm^3$ conical flask.
2. Using the measuring cylinder, she measured an equal volume of HC into the conical flask containing HA.
3. Titrated the solution mixture in the conical flask against HB from the burette until the colour change is observed.
4. Repeated procedure (i) to (iii) three times.
5. Recorded his results in tabular form as shown below:

Titration number	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)				
Initial reading (cm^3)				
Volume used (cm^3)				

Summary:

_______cm^3 of solution HA required _______cm^3 of solution HB in the presence of HC for complete reaction.

Questions

a) Why must hydrogen peroxide be freshly diluted before titration?

b) Write down the half ionic equations for the reaction to show reduction and oxidation processes

c) Calculate the concentration of the original solution of hydrogen peroxide in g/dm^3.

d) To be suitable for disinfection in clinical settings, the volume strength must be at least 10. Was the hydrogen peroxide fit for the disinfection duty?

Solution

The volume of the pipette used was $20cm^3$

TABLE OF RESULTS

Titration number	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)	20.20	20.00	20.10	20.00
Initial reading (cm^3)	0.00	0.00	0.00	0.00
Volume used (cm^3)	20.20	20.00	20.10	20.00

$$\text{Average volume used} = \frac{V_1 + V_2 + V_3}{3}$$

$$\text{Average volume used } = \left(\frac{20.00 + 20.10 + 20.00}{3}\right) cm^3 = 20.00cm^3$$

Summary:

$20cm^3$ of solution HA required $\mathbf{20.00cm^3}$ of solution HB in the presence of HC for complete reaction.

For 25 mL pipette: The volume of solution HB will be **25cm^3**.

Making Sense of the Experimental Data

All three titre values are within ±0.10 cm³ of each other, indicating high precision and hence the experiment was successful.

Response to Questions

(a) Hydrogen peroxide is unstable and decomposes over time, especially in light according to the following equation:

$$2H_2O_2 \longrightarrow 2H_2O \ + \ O_2$$

So fresh dilution ensures that the concentration is accurate and correct titre values are obtained.

(b) Reduction process

$$MnO_4^- \ + \ 8\,H^+ \ + \ 5e \ \rightarrow \ Mn^{2+} \ + \ 4\,H_2O$$

Oxidation process

$$H_2O_2 \ \rightarrow \ O_2 \ + \ 2H^+ \ + 2e$$

(c) Overall reaction equation:

$$2MnO_4^- \ + \ 5H_2O_2 \ + \ 6H^+ \ \rightarrow \ 2Mn^{2+} \ + \ 5O_2 \ + \ 8H_2O; n_{oxidant} \ = 2, n_{reductant} = 5$$

Using $\frac{M_{oxidant} \times V_{oxidant}}{M_{reductant} \times V_{reductant}} = \frac{n_{oxidant}}{n_{reductant}}$

Where:

$$V_{oxidant} = 20cm^3, V_{reductant} = 20cm^3;\ M_{oxidant} = \frac{m}{M_r \times V} == \frac{0.79g}{158g/mol \times 0.25dm^3} = 0.02M$$

Substituting $\frac{0.02M \times 20cm^3}{M_{reductant} \times 20cm^3} = \frac{2}{5};\ M_{reductant} = 0.05M$

The molarity of hydrogen peroxide used in the titration is $0.05M$. But this peroxide was formed after the dilution.

From $M_cV_c = M_dV_d$

Where;

M_c = concentration of original (concentrated)hydrogen peroxide

V_c = volume of concentrated solution = $1cm^3$

V_d = volume of diluted dilution = $250cm^3$

M_d = concentration of diluted dilution = $0.05M$

Substituting $M_c \times 1cm^3 = 0.05M \times 250cm^3;\ M_c = 12.5M$.

But concentration of H_2O_2 in g/dm^3 = molarity of H_2O_2 × molar mass of H_2O_2

$$= 12.5mol/dm^3 \times 34g/mol = 425g/dm^3$$

The original concentration of H_2O_2 was $425g/dm^3$.

(d) Hydrogen peroxide decomposes to give oxygen gas according to the following equation:

$$2\,H_2O_2 \ \rightarrow \ 2\,H_2O + \ O_2$$

From which mole ratio of H_2O_2 to O_2 is $2:1$

Number of moles of H_2O_2 in $1cm^3$ of its solution

$= \frac{1}{1000}\text{dm}^3 \times 12.5\text{moldm}^{-3} = 0.0125\text{mol} \quad (\text{n} = \text{MV})$

Thus from the mole ratio 0.0125mol of H_2O_2 produces $\frac{0.0125}{2}\text{mol}$ or $6.25 \times 10^{-3}\text{mol}$ of oxygen.

But 0.0125mol of H_2O_2 is contained in 1cm^3 of its volume.

And 1mol of O_2 is contained in 22.4dm^3 or 22400cm^3 of its volume (at STP)

It follows that: 1cm^3 of H_2O_2 produces $6.25 \times 10^{-3}\text{mol} \times 22400\text{cm}^3/\text{mol}$ or 140cm^3 of O_2

Therefore, the volume strength of H_2O_2 solution is 140 which is far above the minimum value of 10 and hence the hydrogen peroxide was fit for the disinfection duty.

Experiment 18

One afternoon, Kipute visited a regional textile dyeing plant as part of her school's industrial chemistry field tour. During the visit, the factory chemist handed her a curious bottle labelled:

"Metal Peroxide – Reacted to form Hydrogen Peroxide"
"Source: MO_2"

The plant used this compound for **fabric bleaching**, but no one remembered which metal the peroxide was based on. Kipute immediately recognized an opportunity: she could identify the **metal "M"** by performing a titration with **potassium permanganate** in acidic conditions.

This classic redox setup would reveal not only the concentration of H_2O_2 — but also the **relative atomic mass of the mysterious metal M** in MO_2.

To perform the experiment, she was provided with the following:

XX: Acidified solution of hydrogen peroxide made by reacting 8.45g of the metal (M) peroxide (MO_2) with dilute sulphuric acid to make 1 litre of the solution using distilled water.

YY: A solution of potassium permanganate made by dissolving 3.16g of the permanganate to make 1 litre of the solution.

As a top student, Kipute knew that reaction between metal peroxide and dilute sulphuric acid is represented by the following reaction equation:

$$MO_2(s) + H_2SO_4(aq) \longrightarrow MSO_4(s) + H_2O_2(aq)$$

Titration Procedure Kipute followed:

1. Pipetted 20 cm^3 of XX into a clean 250cm^3 conical flask.
2. Put YY into the burette.
3. Titrated XX against YY from the burette until permanent faint pink colour is observed.
4. Repeated procedure (1) to (3) above for three more times.
5. Recorded her results in a tabular form as follows:

Titration number	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)				
Initial reading (cm^3)				
Volume used (cm^3)				

Summary:

_______cm^3 of solution XX required _______cm^3 of solution YY for complete reaction.

Questions

a) Why is potassium permanganate used as the titrant in this experiment rather than iodine or copper(II) ions?

b) Write the overall ionic equation for the reaction between hydrogen peroxide and potassium permanganate in acidic medium.

c) Calculate:

  i. The concentration of potassium permanganate in moldm^{-3}

  ii. The concentration of hydrogen peroxide in moldm^{-3}

  iii. The atomic mass of the metal M in MO_2 and hence name the metal, M.

d) Suggest two real-life industries (other than textile bleaching) where such a redox titration involving hydrogen peroxide would be important.

Solution

The volume of the pipette used was 20cm^3

TABLE OF RESULTS

Titration number	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)	20.10	20.00	20.10	20.00
Initial reading (cm^3)	0.00	0.00	0.00	0.00
Volume used (cm^3)	20.10	20.00	20.10	20.00

$$\text{Average volume used} = \frac{V_1 + V_2 + V_3}{3}$$

$$\text{Average volume used } = \left(\frac{20.00 + 20.10 + 20.00}{3}\right)\text{cm}^3 = 20.00\text{cm}^3$$

Summary:

20cm^3 of solution XX required **20.00cm^3** of solution YY for complete reaction.

For 25 mL pipette: The volume of solution YY will be **25cm^3**.

Making Sense of the Experimental Data

Practical Tip 1: *The given formula of the peroxide indicates that M is a divalent metal (Group II elements). Among group II elements, only strontium and barium form stable peroxides.*

Practical Tip 2: *In laboratory practice, barium compounds are more commonly used than those of strontium. Therefore, the metal peroxide in question is most likely barium peroxide (BaO_2).*

Molar mass of $BaO_2 = (137 + 2 \times 16) = 169\text{g/mol}$

Number of moles of $BaO_2 = \frac{8.45\text{g}}{169\text{g/mol}} = 0.05\text{mol}$

The H_2O_2 was produced according to the following reaction equation:

$$BaO_2(s) + H_2SO_4(aq) \longrightarrow BaSO_4(s) + H_2O_2(aq)$$

From which, mole ratio of BaO_2 to H_2O_2 is $1:1$.

Thus number of moles of H_2O_2 produced was also $0.05mol$.

Thus $[H_2O_2] = \frac{0.05mol}{1L} = 0.05M = M_{reductant}$

The H_2O_2 reacts with $KMnO_4$ according to the following reaction equation:

$$2MnO_4^- + 5H_2O_2 + 6H^+ \rightarrow 2Mn^{2+} + 5O_2 + 8H_2O; n_{oxidant} = 2, n_{reductant} = 5$$

Using $\frac{M_{oxidant} \times V_{oxidant}}{M_{reductant} \times V_{reductant}} = \frac{n_{oxidant}}{n_{reductant}}$

Where:

$$M_{reductant} = 0.05M, V_{reductant} = 20cm^3;\ M_{oxidant} = \frac{m}{M_r \times V} == \frac{3.16g}{158g/mol \times 1dm^3} = 0.02M$$

Substituting $\frac{0.02M \times V_{oxidant}}{0.05M \times 20cm^3} = \frac{2}{5}$; $V_{oxidant} = \mathbf{20cm^3}$ = experimental data

To conclude: *The calculated volume of $KMnO_4$ agrees with the titre volume obtained from the experiment.*

Response to Questions

(a) Iodine and copper (II) ions are not strong oxidizing agent enough to react significantly with hydrogen peroxide at reasonable rate.

(b) $2MnO_4^- + 5H_2O_2 + 6H^+ \rightarrow 2Mn^{2+} + 5O_2 + 8H_2O$

(c) Calculation of each part is as follows:

(i) $[KMnO_4] = \frac{m}{M_r \times V} = \frac{3.16g}{158g/mol \times 1dm^3} = 0.02M$

The concentration of $KMnO_4$ is $0.02moldm^{-3}$.

(ii) Reaction equation:

$$2MnO_4^- + 5H_2O_2 + 6H^+ \rightarrow 2Mn^{2+} + 5O_2 + 8H_2O; n_{oxidant} = 2, n_{reductant} = 5$$

Using $\frac{M_{oxidant} \times V_{oxidant}}{M_{reductant} \times V_{reductant}} = \frac{n_{oxidant}}{n_{reductant}}$

Where:

$$V_{oxidant} = 20cm^3, V_{reductant} = 20cm^3;\ M_{oxidant} = 0.02M$$

Substituting $\frac{0.02M \times 20cm^3}{M_{reductant} \times 20cm^3} = \frac{2}{5}$; $M_{reductant} = 0.05M$

The concentration of hydrogen peroxide is $0.05mol/dm^3$.

(iii) H_2O_2 was produced according to the following reaction equation:

$$MO_2(s) + H_2SO_4(aq) \longrightarrow BaSO_4(s) + H_2O_2(aq)$$

From which, mole ratio of MO_2 to H_2O_2 is $1:1$.

Thus number of moles of H_2O_2 produced was equal to the number of moles of MO_2 reacted.

But number of moles of H_2O_2 produced = $0.05mol/L \times 1L = 0.05mol$ (1L of XX was prepared).

Thus number of moles of MO_2 reacted was also $0.05mol$ (in 8.45g of its mass).

Then $M_r(MO_2) = \frac{m}{n} = \frac{8.45g}{0.05mol} = 169g/mol$

But $M_r(MO_2) = M + 32 = 169; M = 137$

The **atomic mass** of metal M in MO_2 is **137** which is the atomic mass of barium and hence M is **barium**.

(d) It may be important in the following:

1) Pharmaceutical industry

   H_2O_2 is used for sterilization of surgical equipment and packaging. So its concentration and stability must be monitored using redox titrations with $KMnO_4$.

2) Food processing and packaging

   H_2O_2 is used for disinfecting food containers and equipment. Regulatory bodies require regular quality control titrations to ensure correct concentration.

Experiment 19

At the local food inspection laboratory, Kipute joined a routine quality check on iodized salt samples. While assisting the chemists, she was introduced to a titration method used to determine iodine levels — a process vital in food regulation and public health.

She was handed four reagents: **potassium dichromate, potassium iodide, sulphuric acid**, and **sodium thiosulphate**. Her goal? Use these

To determine the concentration of thiosulphate via the **redox-based iodine liberation titration**, she was handed the following solutions:

ST: A solution made by diluting $10cm^3$ of 0.5M potassium dichromate to make exactly $250cm^3$ solution.

QP: A solution of sodium thiosulphate.

BA: A dilute sulphuric acid.

BC: Starch solution.

BD: A 10% potassium iodide solution.

She understood the principle well:

Potassium dichromate react quantitatively with acidified potassium iodide to liberate some amount of iodine as per equation below:

$$Cr_2O_7^{2-} + 6I^- + 14H^+ \longrightarrow 2Cr^{3+} + 3I_2 + 7H_2O$$

The liberated iodine is the titrated against thiosulphate and react as per reaction equation below:

$$2S_2O_3^{2-} + I_2 \longrightarrow S_4O_6^{2-} + 2I^-$$

Titration Procedure Kipute followed

1. Pipetted 20 cm^3 of ST into a clean 250 cm^3 conical flask.
2. Added $20cm^3$ of BD into the conical flask containing ST.
3. Added $20cm^3$ of BA into the conical flask containing the mixture of ST and BD.
4. Titrated the solution mixture in the flask against QP from the burette until pale yellow green colour occurs.
5. Added $2cm^3$ of BC and continue titrating the solution until blue colour disappears.
6. Recorded the volume of QP used.
7. Repeated procedure (i) to (vi) above for three more times.
8. Recorded her results in a tabular form as follows:

Titration number	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)				
Initial reading (cm^3)				
Volume used (cm^3)				

Summary:

_______cm^3 of solution ST required _______cm^3 of solution QP for complete reaction.

Questions

a) What was the function of BC in the experiment?
b) What was the purpose of adding BD into the conical flask containing acidified ST?
c) Why is it advisable to add BC just close to the end point in such this experiment?
d) Write a balanced half reaction equation to show oxidation-reduction processes
e) Write a balanced overall reaction equation for the whole experiment.
f) Calculate:
    i. The molarity of potassium dichromate ($K_2Cr_2O_7$)
    ii. The molarity of sodium thiosulphate ($Na_2S_2O_3$)
    iii. The Normality of sodium thiosulphate ($Na_2S_2O_3$)

Solution

The volume of the pipette used was $20cm^3$.

TABLE OF RESULTS

Titration number	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)	12.20	12.00	12.10	12.00
Initial reading (cm^3)	0.00	0.00	0.00	0.00
Volume used (cm^3)	12.20	12.00	12.10	12.00

$$\text{Average volume used} = \frac{V_1 + V_2 + V_3}{3}$$

$$\text{Average volume used } = \left(\frac{12.00 + 11.90 + 12.00}{3}\right) \text{cm}^3 = 12.00\text{cm}^3$$

Summary:

$20cm^3$ of solution ST required $\mathbf{12.00cm^3}$ of solution QP for complete reaction.

For 25 mL pipette: The volume of solution QP will be **$15cm^3$**.

Making Sense of the Experimental Data

All three titre values are within ±0.10 cm^3 of each other, indicating high precision and hence the experiment was successful.

Response to Questions

(a) BC was used as an external indicator to indicate the end point of titration between iodine and sodium thiosulphate.

(b) To liberate iodine (through its oxidation by acidified ST) which will be used in titration with sodium sulphate.

(c) This is done to prevent the formation of a very stable starch–iodine complex, which occurs when a large amount of iodine is present at the beginning of the titration. In such cases, the iodine bound within the complex becomes resistant to reduction**,** making the blue colour persist and causing the endpoint to be unclear or delayed**.**

(d) For the reaction between potassium iodide and acidified potassium dichromate (VI):

Reduction half reaction

$$Cr_2O_7^{2-} + 14H^+ + 6e \rightarrow 2Cr^{3+} + 7H_2O$$

Oxidation half reaction

$$2I^- \rightarrow I_2 + 2e$$

For the reaction between iodine and sodium thiosulphate:

Reduction half reaction

$$I_2 + 2e \rightarrow 2I^-$$

Oxidation half reaction

$$2S_2O_3^{2-} \rightarrow S_4O_6^{2-} + 2e$$

(e) Given that:

$Cr_2O_7^{2-} + 6I^- + 14H^+ \rightarrow 3I_2 + 2Cr^{3+} + 7H_2O$ --------- (i)
$I_2 + 2S_2O_3^{2-} \rightarrow S_4O_6^{2-} + 2I^-$ --------------------------------- (ii)

Eliminating I_2 in the two equations (i) and (ii) by taking (i) + 3× (ii) so as to write the overall reaction equation as follows:

$$Cr_2O_7^{2-} + 6I^- + 14H^+ + 3I_2 + 6S_2O_3^{2-} \rightarrow 3I_2 + 2Cr^{3+} + 7H_2O + 3S_4O_6^{2-} + 6I^-$$

Cancelling like terms which appears in both sides of the equation gives overall reaction equation for the whole experiment which is:

$$\mathbf{Cr_2O_7 + 6S_2O_3^{2-} + 14H^+ \rightarrow 2Cr^{3+} + 3S_4O_6^{2-} + 7H_2O}$$

(f) Calculation of each part is as follows:

(i) By using dilution principle;

$$M_cV_c = M_dV_d$$

Where:

M_c = Concentration before dilution = 0.5M
V_c = Volume before dilution = $10cm^3$
V_d = Volume after dilution = $250cm^3$
M_d = Concentration after dilution

Substituting $0.5M \times 10cm^3 = M_d \times 250cm^3, M_d = 0.02M$

The molarity of potassium dichromate is 0.02M.

(ii) From the equation of the whole experiment;

$$Cr_2O_7 + 6S_2O_3^{2-} + 14H^+ \rightarrow 2Cr^{3+} + 3S_4O_6^{2-} + 7H_2O;\ n_{oxidant} = 1, n_{reductant} = 6$$

Using $\frac{M_{\text{oxidant}} \times V_{\text{oxidant}}}{M_{\text{reductant}} \times V_{\text{reductant}}} = \frac{n_{\text{oxidant}}}{n_{\text{reductant}}}$

Where:

$$V_{\text{oxidant}} = 20\text{cm}^3, V_{\text{reductant}} = 12\text{cm}^3;\ M_{\text{oxidant}} = 0.02\text{M}$$

Substituting $\frac{0.02\text{M} \times 20\text{cm}^3}{M_{\text{reductant}} \times 12\text{cm}^3} = \frac{1}{6};\ M_{\text{reductant}} = 0.2\text{M}$

The molarity of sodium thiosulphate is 0.2M.

(iii) $Na_2S_2O_3$ donates electrons according to the following equation (oxidation half reaction):

$$2\,S_2O_3^{2-} \rightarrow S_4O_6^{2-} + 2e$$

Dividing by 2 throughout the above equation so as to get one molecule of $Na_2S_2O_3$ (or $S_4O_6^{2-}$)

$$S_2O_3^{2-} \rightarrow \frac{1}{2}\,S_4O_6^{2-} + e$$

From which one molecule of $S_2O_3^{2-}$ donates 1 electron.
And normality $Na_2S_2O_3 = [Na_2S_2O_3] \times$ number of electrons donated by one molecule.
$= 0.2 \times 1\text{N} = 0.2\text{N}$
The normality of $Na_2S_2O_3$ is 0.2N.

Experiment 20

Before dispatching a fresh batch of laboratory-grade reagents to schools in the district, Mr. Akilikubwa was assigned the task of verifying the **purity and concentration** of two key solutions: **potassium permanganate** and **hydrated sodium thiosulphate**.

"We can't afford imprecise labels," he reminded the lab assistants. *"These chemicals are used for exams — one error can ruin everything."*

Using a time-tested **iodometric titration**, he planned to react **acidified $KMnO_4$ with KI** to liberate iodine, then titrate that iodine using sodium thiosulphate according to the following equations:

$$MnO_4^- + I^- \longrightarrow Mn^{2+} + I_2$$

$$S_2O_3^{2-} + I_2 \longrightarrow S_4O_6^{2-} + I^-$$

His goal was to confirm the actual concentrations and determine the **hydration number (x)** in $Na_2S_2O_3 \cdot xH_2O$ — essential for correctly calculating molar mass.

To reach the goal, the following solutions were used:

LL: A solution made by dissolving 1.58g of potassium permanganate to make 500cm^3 solution.

MM: A solution made by dissolving 6.2g of hydrated sodium thiosulphate ($Na_2S_2O_3.xH_2O$) to make 250cm^3 solution.

NN: Starch solution.

PP: 10% potassium iodide solution.

QQ: Dilute sulphuric acid solution.

Titration procedure Mr. Akilikubwa followed

1. Pipetted 20cm^3 of LL into a clean 250cm^3 conical flask.
2. Added 20cm^3 of PP into the conical flask containing LL.
3. Added 20cm^3 of QQ into the conical flask containing the mixture of LL and PP.
4. Titrated the solution mixture in the flask against MM from the burette until colour change was observed.
5. Added 2cm^3 of NN and continue titrating the solution until blue colour disappears.
6. Recorded the volume of MM used.

7. Repeated procedure (1) to (6) above for three more times.
8. Recorded his results in a tabular form as follows:

Titration number	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)				
Initial reading (cm^3)				
Volume used (cm^3)				

Summary:

_______cm^3 of solution LL required _______cm^3 of solution MM for complete reaction.

Questions

a) Why must potassium iodide (KI) be in excess during this experiment?

b) Balance the reaction equation took place in the following:

   i. $MnO_4^- + I^- \rightarrow Mn^{2+} + I_2$
   ii. $I_2 + 2S_2O_3^{2-} \rightarrow SO_4O_6^{2-} + 2I^-$

c) Write the balanced overall ionic equation for the whole experiment.

d) Calculate the concentration of the following in mol/dm^3:
   i. Potassium permanganate
   ii. Sodium thiosulphate

e) Calculate the value of x in the formula $Na_2S_2O_3.xH_2O$.

Solution

The volume of the pipette used was 20cm^3

TABLE OF RESULTS

Titration number	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)	20.10	40.10	20.00	20.00
Initial reading (cm^3)	0.00	20.10	0.00	0.00
Volume used (cm^3)	20.10	20.00	20.00	20.00

Titre volume used was $\mathbf{20.00cm^3}$

Summary:

20cm^3 of solution LL required $\mathbf{20.00cm^3}$ of solution MM for complete reaction.

For 25 mL pipette: The volume of MM will be **25cm^3**.

Making Sense of the Experimental Data

Practical tip: *The fixed number of moles of water of crystallization in the hydrated sodium thiosulphate is 5* ($Na_2S_2O_3.5H_2O$).

Thus, molar mass of $Na_2S_2O_3.xH_2O$ is 248g/mol.

And $[Na_2S_2O_3.xH_2O] = \frac{6.2g}{0.25L\times248gmol^{-1}} = 0.1M = M_{reductant}$

Also $[KMnO_4] = \frac{1.58g}{0.5L\times158gmol^{-1}} = 0.02M = M_{oxidant}$

Balanced reaction equations:

$$2MnO_4^- + 10I^- + 16H^+ \rightarrow 2Mn^{2+} + 5I_2 + 8H_2O \ldots\ldots\ldots\ldots (i)$$

$$2\,S_2O_3^{2-} + I_2 \rightarrow S_4O_6^{2-} + 2I^- \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots. (ii)$$

Eliminating I_2 in the two equations (i) and (ii) by taking (i) + 5× (ii) so as to write the overall reaction equation (for whole experiment) as follows:

$$2MnO_4^- + 10I^- + 16H^+ + 5I_2 + 10S_2O_3^{2-} \rightarrow 5I_2 + 2Mn^{2+} + 8H_2O + 5S_4O_6^{2-} + 10I^-$$

Cancelling like terms which appears in both sides of the equation gives overall reaction equation for the whole experiment which is:

$$2MnO_4^- + 10S_2O_3^{2-} + 16H^+ \rightarrow 2Mn^{2+} + 5S_4O_6^{2-} + 8H_2O;\ n_{oxidant} = 2, n_{reductant} = 10$$

Using $\frac{M_{oxidant}\times V_{oxidant}}{M_{reductant}\times V_{reductant}} = \frac{n_{oxidant}}{n_{reductant}}$; where $V_{oxidant} = 20cm^3$(pipette volume)

Substituting $\frac{0.02M\times20cm^3}{0.1M\times V_{reductant}} = \frac{2}{10}$; $V_{reductant} = \mathbf{20cm^3}$ = experimental data

To conclude: *The calculated volume of $Na_2S_2O_3.xH_2O$ agrees with the titre volume obtained from the experiment.*

Response to Questions

(a) KI must be in excess to ensure complete reduction of all permanganate ions. Without excess KI, some $KMnO_4$ may remain unreacted, invalidating titration results because the unreacted permanganate would oxidize sodium thiosulphate as iodine does.

(b) The balanced equation for each part is as follows:

(i) $2MnO_4^- + 10I^- + 16H^+ \rightarrow 2Mn^{2+} + 5I_2 + 8H_2O$
(ii) $2\,S_2O_3^{2-} + I_2 \rightarrow S_4O_6^{2-} + 2I^-$

(c) From balanced reaction equations:

$$2MnO_4^- + 10I^- + 16H^+ \rightarrow 2Mn^{2+} + 5I_2 + 8H_2O \ldots\ldots\ldots\ldots (i)$$

$$2\,S_2O_3^{2-} + I_2 \rightarrow S_4O_6^{2-} + 2I^- \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots. (ii)$$

Eliminating I_2 in the two equations (i) and (ii) by taking (i) + 5× (ii) so as to write the overall reaction equation (for whole experiment) as follows:

$$2MnO_4^- + 10I^- + 16H^+ + 5I_2 + 10S_2O_3^{2-} \rightarrow 5I_2 + 2Mn^{2+} + 8H_2O + 5S_4O_6^{2-} + 10I^-$$

Cancelling like terms which appears in both sides of the equation gives overall reaction equation for the whole experiment which is:

$$\mathbf{2MnO_4^- + 10S_2O_3^{2-} + 16H^+ \rightarrow 2Mn^{2+} + 5S_4O_6^{2-} + 8H_2O}$$

(d) Calculation of each part is as follows:

(i) Using $[KMnO_4] = \frac{n}{V} = \frac{m}{M_r\times V} = \frac{1.58g}{0.5L\times158gmol^{-1}} = 0.02M$

The concentration of potassium permanganate is $0.02\ mol/dm^3$.

(ii) From the equation of the whole experiment;

$$2MnO_4^- + 10S_2O_3^{2-} + 16H^+ \rightarrow 2Mn^{2+} + 5S_4O_6^{2-} + 8H_2O; n_{oxidant} = 2, n_{reductant} = 10$$

Using $\frac{M_{oxidant} \times V_{oxidant}}{M_{reductant} \times V_{reductant}} = \frac{n_{oxidant}}{n_{reductant}}$

Where:

$$V_{oxidant} = 20cm^3, V_{reductant} = 20cm^3;\ M_{oxidant} = 0.02M$$

Substituting $\frac{0.02M \times 20cm^3}{M_{reductant} \times 20cm^3} = \frac{2}{10}; M_{reductant} = 0.1M$

The concentration of sodium thiosulphate is $0.1\ mol/dm^3$.

(e) Using molar mass $= \frac{\text{Mass concentration}}{\text{Molar concentration}}$

Where mass concentration of $Na_2S_2O_3.xH_2O = \frac{6.2g}{0.25L} = 24.8g/L$

So, molar mass of $Na_2S_2O_3.xH_2O = \frac{24.8g/L}{0.1mol/L} = 248g/mol$

But also, molar mass of $Na_2S_2O_3.xH_2O = (158 + 18x)g/mol$

Thus, $158 + 18x = 248$ or $x = 5$

The value of x in $Na_2S_2O_3.xH_2O$ is 5.

Experiment 21

Mr. Akilikubwa had just received a batch of **hydrated copper(II) sulphate** labelled only as "$CuSO_4 \cdot xH_2O$." The supplier hadn't indicated the number of water molecules — and knowing the exact value of **x** was essential before using the salt in classroom standard preparations.

Instead of guessing, Mr. Akilikubwa set up a **redox titration**. He reacted the copper salt with **potassium iodide**, liberating iodine, which he then titrated using **sodium thiosulphate**.

$$2Cu^{2+} + 4I^- \longrightarrow Cu_2I_2 + I_2$$

Then

$$2S_2O_3^{2-} + I_2 \longrightarrow S_4O_6^{2-} + 2I^-$$

To complete the analysis, he had the following:

VA: A solution made by dissolving 6.24g of hydrated copper (II) sulphate ($CuSO_4.xH_2O$) in 250cm^3 of aqueous solution.

VB: A containing 12.4g of pentahydrate sodium thiosulphate ($Na_2S_2O_3.5H_2O$) in 500cm^3 of aqueous solution.

VC: 10% of KI solution.

VD: Starch solution.

Titration procedure Mr. Akilikubwa followed

1. Pipetted 20 cm^3 of VA into a clean 250cm^3 conical flask.
2. Added 10cm^3 of VC into the conical flask containing VA and shake well the mixture.
3. Titrated the solution mixture in the flask against VB from the burette until a pale yellow colour appears.
4. Added 2cm^3 of VD and continued titrating the solution until pale blue colour disappears.
5. Recorded the volume of VB used.
6. Repeated procedure (1) to (6) above for three more times.
7. Recorded his results in a tabular form as follows:

Titration number	Pilot	1	2	3
Final reading (cm^3)				
Initial reading (cm^3)				
Volume used (cm^3)				

Summary:

_______cm^3 of solution VA required _______cm^3 of solution VB for complete reaction.

Questions

a) Calculate the concentration of VB in $\mathrm{mol/dm^3}$

b) Calculate:
  i. The Molarity of VA
  ii. The concentration of VA in $\mathrm{gdm^{-3}}$
  iii. The value of x in the formula $\mathrm{CuSO_4.xH_2O}$
  iv. The percentage of water in the salt $\mathrm{CuSO_4.xH_2O}$
  v. The number of electrons gained by VA in the experiment.

c) What role does shaking the flask after adding KI (VC) play in the titration accuracy?

d) During titration, the solution turns pale yellow before starch is added. What causes this colour? Explain.

Solution

The volume of the pipette used was 20cm^3

TABLE OF RESULTS

Titration number	Pilot	1	2	3
Final reading (cm^3)	20.60	40.70	20.00	20.00
Initial reading (cm^3)	0.00	20.60	0.00	0.00
Volume used (cm^3)	20.60	20.10	20.00	20.00

Average volume used $= \frac{\mathrm{V_1+V_2+V_3}}{3}$

Average volume used $= \left(\frac{20.10+20.00+20.00}{3}\right)\mathrm{cm^3} = 20.00\mathrm{cm^3}$

Summary:

20cm^3 of solution VA required $\mathbf{20.00cm^3}$ of solution VB for complete reaction.

For 25 mL pipette: The volume of VB will be **25cm^3**.

Making Sense of the Experimental Data

Practical tip: *The fixed number of moles of water of crystallization in the hydrated copper (II) sulphate is 5* $(CuSO_4.5H_2O)$.

Thus, molar mass of $CuSO_4.xH_2O$ is 249.6g/mol.

And $[CuSO_4.xH_2O\,] = \frac{6.24g}{0.25L\times249.6gmol^{-1}} = 0.1M = M_{oxidant}$

Also $[Na_2S_2O_3.5H_2O] = \frac{12.4g}{0.5L\times248gmol^{-1}} = 0.1M = M_{reductant}$

Equations for reactions:

$2Cu^{2+} + 4I^- \rightarrow Cu_2I_2 + I_2$ ……………………………….(i)

$2S_2O_3^{2-} + I_2 \rightarrow S_4O_6^{2-} + 2I^-$……………………….…..(ii)

(i)+(ii) gives the overall reaction equation which is:

$2Cu^{2+} + 2S_2O_3^{2-} + 2I^- \rightarrow Cu_2I_2 + S_4O_6^{2-};\ n_{oxidant} = 2, n_{reductant} = 2$

Using $\frac{M_{oxidant}\times V_{oxidant}}{M_{reductant}\times V_{reductant}} = \frac{n_{oxidant}}{n_{reductant}}$; where $V_{oxidant} = 20cm^3$(pipette volume)

Substituting $\frac{0.1M\times20cm^3}{0.1M\times V_{reductant}} = \frac{2}{2}$; $V_{reductant} = \mathbf{20cm^3}$ = experimental data

To conclude: *The calculated volume of* $Na_2S_2O_3.5H_2O$ *agrees with the titre volume obtained from the experiment.*

Response to Questions

(a) Using $[Na_2S_2O_3.5H_2O] = \frac{n}{V} = \frac{m}{M_r\times V} = \frac{12.4g}{0.5L\times248gmol^{-1}} = 0.1M$

The concentration of VB is $0.02\,mol/dm^3$.

(b) Calculation of each part is as follows:

(i) Equations for reactions:

$2Cu^{2+} + 4I^- \rightarrow Cu_2I_2 + I_2$ ……………………………….(i)

$2S_2O_3^{2-} + I_2 \rightarrow S_4O_6^{2-} + 2I^-$……………………….…..(ii)

(i)+(ii) gives the overall reaction equation which is:

$2Cu^{2+} + 2S_2O_3^{2-} + 2I^- \rightarrow Cu_2I_2 + S_4O_6^{2-};\ n_{oxidant} = 2, n_{reductant} = 2$

Using $\frac{M_{oxidant}\times V_{oxidant}}{M_{reductant}\times V_{reductant}} = \frac{n_{oxidant}}{n_{reductant}}$;

Where $V_{oxidant} = 20cm^3, V_{reductant} = 20cm^3, M_{reductant} = 0.1M$

Substituting $\frac{M_{oxidant}\times20cm^3}{0.1M\times20cm^3} = \frac{2}{2}$; $M_{oxidant} = 0.1M$

The molarity of VA is 0.1M.

(ii) Using mass concentration $= \frac{\text{mass of solute}}{\text{volume of solution}}$

The concentration of VA in $gdm^{-3} = \frac{6.4g}{0.25dm^3} = 24.96gdm^{-3}$

The concentration of VA in gdm^{-3} is $24.96gdm^{-3}$.

(iii) Using molar mass $= \frac{\text{Mass concentration}}{\text{Molar concentration}}$

So, molar mass of $CuSO_4.xH_2O = \frac{24.96gdm^{-3}}{0.1moldm^{-3}} = 249.6g/mol$

But also, molar mass of $CuSO_4.xH_2O = (159.6 + 18x)g/mol$

Thus, $159.6 + 18x = 249.6$ or $x = 5$

The value of x in $CuSO_4.xH_2O$ is 5.

(iv) The percentage of water in the salt $CuSO_4.xH_2O$

$$= \frac{\text{mass of water}}{\text{Total mass of the compound}} \times 100\% = \frac{5 \times 18g}{249.6g} \times 100\% = 36.06\%$$

The percentage of water in the salt $CuSO_4.xH_2O$ is 36.06%.

(v) VA gains electrons according to the following half reaction equation:

$$2Cu^{2+} + 2I^- + 2e \rightarrow Cu_2I_2$$

From which mole ratio of Cu^{2+} to electrons is 2: 2 or 1: 1. Thus number of moles of electrons gained is equal to the number of moles of VA reacted.

But number of moles of VA reacted $= \frac{20}{1000}dm^3 \times 0.1moldm^{-3} = 2 \times 10^{-3}mol$

$= \text{Number of moles of electrons gained}$

Using N = nN_A;

Number of electrons gained $= 2 \times 10^{-3} \times 6.02 \times 10^{23}$ electrons

$=1.204 \times 10^{21}$electrons

Hence number of electrons gained by VA in the experiment is 1.204×10^{21}electrons.

For 25 mL pipette: The answer will be 1.505×10^{21}electrons.

(c) Shaking the flask after adding potassium iodide (KI) ensures that the reaction between Cu^{2+} ions and iodide ions (I^-) proceeds quickly and uniformly as it increases collision frequency between Cu^{2+} ions and the I^- ions. If not shaken, the reaction may be incomplete or delayed, leading to a lower than actual titre value.

(d) The pale yellow colour is caused by the presence of small amount of liberated iodine dissolved in water.

Explanation:

When relatively large amount of iodine is dissolved in water, it forms a yellowish-brown solution. As titration proceeds, sodium thiosulphate reduces iodine (I_2) back to iodide (I^-), gradually decreasing iodine amount and hence gradual decolourisation of the solution, making the solution pale yellow when very small amount of iodine remains.

Experiment 22

At ANANGA Pharmaceuticals, one batch of a newly received chemical stock had a curious label: *Sodium thiosulphate ($Na_2S_2O_3{\cdot}XH_2O$), Mass: 12.4 g in 500 mL*. But here's the issue—it was unclear what **X** was. And that small number meant everything: from correct drug stability to the shelf life of antiseptic solutions.

So Mr. Akilikubwa, the lab technician, was asked to determine how many water molecules were attached to each formula unit of sodium thiosulphate. It was an iodometric analysis; one of his favourites.

He arranged the reagents neatly on the bench:

U1: solution of $0.04MKMnO_4$:
U2: solution of $Na_2S_2O_3.XH_2O$ made by dissolving 12.4 g in $0.5dm^3$ of distilled water;
U3: solution of 10% KI :
U4: starch solution:
U5: solution of 1 M sulphuric acid.

As well experienced lab technician, Mr. Akilikubwa knew that: A quantitative reaction between potassium permanganate and potassium iodide in acidic medium involves production of iodine. During the titration process, molecules of iodine produced react with sodium thiosulphate.

Procedure Mr. Akilikubwa Followed

1. Filled the burette with U2.
2. Pipetted $20\ \mathrm{cm^3}$ of U1 into a conical flask, Add $20\ \mathrm{cm^3}$ of distilled water. Swirled the mixture gently and then he added $20\ \mathrm{cm^3}$ of U3 into the flask followed by $20\ \mathrm{cm^3}$ of U5 into the same flask.
3. Titrated U2 with the solution mixture in the conical flask until a pale yellow colour is observed. He then added $2\ \mathrm{cm^3}$ of U4 and continue to titrate until the dark blue colour is discharged to colourless.
4. Recorded the first litre value.
5. Repeated steps (1) to (4) three times to obtain three titre values.

Questions

a) Tabulate the results.
b) Write the balanced chemical equation for the experiment which involved:
   (i) production of iodine.
   (ii) consumption of iodine.
   (iii) the whole experiment.
c) Calculate the actual molarity of solution U1 after dilution.
d) Compute the value of X in the formula $\mathrm{Na_2\ S_2O_3.XH_2O}$.
e) Explain what will happen when potassium permanganate is missing in this experiment.
f) If Mr. Akilikubwa did not dilute the potassium permanganate solution, how would this affect the titre volume obtained? Justify your answer.

Solution

(a)

TABLE OF RESULTS

Titration number	**Pilot**	**1**	**2**	**3**
Final reading ($\mathrm{cm^3}$)	40.20	40.00	40.00	40.00
Initial reading ($\mathrm{cm^3}$)	0.00	0.00	0.00	0.00
Volume used ($\mathrm{cm^3}$)	40.20	40.00	40.00	40.00

Making Sense of the Experimental Data

The fixed number of moles of water of crystallization in the hydrated sodium thiosulphate is 5 $(Na_2S_2O_3.5H_2O)$.

Thus, molar mass of $\mathrm{Na_2S_2O_3.xH_2O}$ is 248g/mol.

And $[\mathrm{Na_2S_2O_3.xH_2O}] = \frac{12.4\mathrm{g}}{0.5\mathrm{L}\times 248\mathrm{gmol^{-1}}} = 0.1\mathrm{M} = \mathrm{M_{reductant}}$

Also $[\mathrm{KMnO_4}] = \frac{\mathrm{M_cV_c}}{\mathrm{V_d}} = \frac{0.04\mathrm{M}\times 20\mathrm{cm^3}}{(20+20)\mathrm{cm^3}} = 0.02\mathrm{M} = \mathrm{M_{oxidant}}$

Balanced reaction equations:

$$2MnO_4^- + 10I^- + 16H^+ \rightarrow 2Mn^{2+} + 5I_2 + 8H_2O \ldots\ldots\ldots\ldots (i)$$

$$2\,S_2O_3^{2-} + I_2 \rightarrow S_4O_6^{2-} + 2I^- \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots.(ii)$$

Eliminating I_2 in the two equations (i) and (ii) by taking (i) + 5× (ii) so as to write the overall reaction equation (for whole experiment) as follows:

$$2MnO_4^- + 10I^- + 16H^+ + 5I_2 + 10S_2O_3^{2-} \rightarrow 5I_2 + 2Mn^{2+} + 8H_2O + 5S_4O_6^{2-} + 10I^-$$

Cancelling like terms which appears in both sides of the equation gives overall reaction equation for the whole experiment which is:

$$2MnO_4^- + 10S_2O_3^{2-} + 16H^+ \rightarrow 2Mn^{2+} + 5S_4O_6^{2-} + 8H_2O;\ n_{oxidant} = 2, n_{reductant} = 10$$

Using $\frac{M_{oxidant} \times V_{oxidant}}{M_{reductant} \times V_{reductant}} = \frac{n_{oxidant}}{n_{reductant}}$; where $V_{oxidant} = 40cm^3 (20 + 20)cm^3$

Substituting $\frac{0.02M \times 40cm^3}{0.1M \times V_{reductant}} = \frac{2}{10}$; $V_{reductant} = \mathbf{40cm^3}$ = experimental data

To conclude: *The calculated volume of* $Na_2S_2O_3.xH_2O$ *agrees with the titre volume obtained from the experiment.*

Response to the remaining Questions

(b) The balanced equation for each part is as follows:

(i) $2MnO_4^- + 10I^- + 16H^+ \rightarrow 2Mn^{2+} + 5I_2 + 8H_2O$

(ii) $2\,S_2O_3^{2-} + I_2 \rightarrow S_4O_6^{2-} + 2I^-$

(iii) From balanced reaction equations:

$$2MnO_4^- + 10I^- + 16H^+ \rightarrow 2Mn^{2+} + 5I_2 + 8H_2O \ldots\ldots\ldots\ldots (i)$$

$$2\,S_2O_3^{2-} + I_2 \rightarrow S_4O_6^{2-} + 2I^- \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots.(ii)$$

Eliminating I_2 in the two equations (i) and (ii) by taking (i) + 5× (ii) so as to write the overall reaction equation (for whole experiment) as follows:

$$2MnO_4^- + 10I^- + 16H^+ + 5I_2 + 10S_2O_3^{2-} \rightarrow 5I_2 + 2Mn^{2+} + 8H_2O + 5S_4O_6^{2-} + 10I^-$$

Cancelling like terms which appears in both sides of the equation gives overall reaction equation for the whole experiment which is:

$$\mathbf{2MnO_4^- + 10S_2O_3^{2-} + 16H^+ \rightarrow 2Mn^{2+} + 5S_4O_6^{2-} + 8H_2O}$$

(c) By using dilution formula; $M_cV_c = M_dV_d$

Where: $M_c = 0.04M, V_c = 20cm^3,\ V_d = (20 + 20)cm^3 = 40cm^3$

Substituting $0.04M \times 20cm^3 = M_d \times 40cm^3$

From which; $M_d = 0.02M$

The actual molarity of solution U1 after dilution is 0.02M.

(d) From the equation of the whole experiment;

$$2MnO_4^- + 10S_2O_3^{2-} + 16H^+ \rightarrow 2Mn^{2+} + 5S_4O_6^{2-} + 8H_2O;\ n_{oxidant} = 2, n_{reductant} = 10$$

Using $\frac{M_{oxidant} \times V_{oxidant}}{M_{reductant} \times V_{reductant}} = \frac{n_{oxidant}}{n_{reductant}}$

Where: $V_{oxidant} = 40cm^3,\ M_{oxidant} = 0.02M$

And from the table of results in (a), the titre volume used was 40.00cm^3 (if the titre values were different, use the average value).

Thus, $V_{\text{reductant}} = 40\text{cm}^3$

Substituting $\frac{0.02\text{M}\times 40\text{cm}^3}{\text{M}_{\text{reductant}}\times 40\text{cm}^3} = \frac{2}{10}$; $\text{M}_{\text{reductant}} = 0.1\text{M}$

The concentration of sodium thiosulphate is $0.1\ \text{mol/dm}^3$.

Using molar mass $= \frac{\text{Mass concentration}}{\text{Molar concentration}}$

Where mass concentration of $Na_2S_2O_3.xH_2O = \frac{12.4\text{g}}{0.5\text{L}} = 24.8\text{g/L}$

So, molar mass of $Na_2S_2O_3.xH_2O = \frac{24.8\text{g/L}}{0.1\text{mol/L}} = 248\text{g/mol}$

But also, molar mass of $Na_2S_2O_3.xH_2O = (158 + 18X)\text{g/mol}$

Thus, $158 + 18X = 248$ or $X = 5$

The value of X in $Na_2S_2O_3.XH_2O$ is 5.

(e) If potassium permanganate ($KMnO_4$) is missing, no iodine will be liberated from the mixture of potassium iodide (KI) and sulphuric acid. This is because $KMnO_4$ acts as the oxidizing agent that oxidizes iodide ions (I^-) into iodine molecules (I_2). Without this oxidation step, there will be no iodine available to react with sodium thiosulphate ($Na_2S_2O_3$) during the titration. Consequently, the characteristic blue starch-iodine complex will not form, and the titration cannot proceed—making the entire experiment invalid and inconclusive.

(f) The titre volume obtained would remain unchanged.

Justification:

Dilution does not alter number of moles of potassium permanganate which is already present in the conical flask. Consequently, the amount of sodium thiosulphate required from the burette to reach the end point remains the same, and the titre volume is unaffected.

Practical tip: *Dilution will affect the titre volume only if the solution being diluted is either placed in the burette or is the main stock solution from which a fixed volume is pipetted into the conical flask.*

PART TWO

PHYSICAL CHEMISTRY ANALYSIS

Chapter 4

PARTITION

INTRODUCTION

Distribution law or **partition law** suggests that when a non-volatile solute is **allowed to distribute itself** in immiscible solvents and then **allowed to attain an equilibrium**, the ratio of concentration of the solute in the two solvents will be constant. The constant is known as **distribution coefficient ($\mathbf{K_d}$).**

That is; $\mathbf{K_d} = \frac{\text{Concentration of the solute in the first solvent}}{\text{Concentration of the solute in the second solvent}}$

Now, from above the explanation, three important questions may come up in our mind:

1) *Which factor determines magnitude of the distribution coefficient?*
2) *How can we allow the solute to distribute itself in the two solvents?*
3) *How can we allow the solute to attain the equilibrium?*

Understanding answers of above questions is very crucial in mastering partition experiments. Now, let us answer the three questions, one after another!

The magnitude of $\mathbf{K_d}$

The magnitude of distribution coefficient depends on solubility of the solute in the given solvents. If $\mathbf{K_d}$ is large then the solute is more soluble in the first solvent and vice versa. In terms of mathematical equation, we can write this as follows;

$$\frac{\text{concetration of solute in first solvent}}{\text{concentration of solute in second solvent}} = \frac{\text{solubility of solute in first solvent}}{\text{solubility of solute in second solvent}} = K_d$$

Now we all understand that, solubility depend on nature of intermolecular forces. If the two solvents do not differ much in their intermolecular forces then their ability to dissolve the solute will not differ much too and hence the $\mathbf{K_d}$ value will be close to 1. But, if the two solvents differ much in their intermolecular forces then the $\mathbf{K_d}$ value will be very large (or very small depending on the type of solvent which is kept on numerator of the equation).

So knowing intermolecular forces of solvents is very crucial on understanding answers of next two questions.

Allowing solute to distribute itself in the solvents

This is done by shaking the solution mixture containing the immiscible solvents and the solute. The results of partition experiment depend on how much the shaking is done; more the shaking, better are the results.

If the two solvents differ much in their intermolecular forces ($\mathbf{K_d}$ value is very large) then stronger shaking is needed for longer time to enable them to mix each other.

Practical tip: *To ensure better results, shake the mixture vigorously for at least three minutes and alternates hand whenever you feel tired.*

Allowing establishment of equilibrium

This is done by leaving the solution mixture for enough time to allow it to 'settle' and form separating layer. Here, patience is a key, otherwise poor results will be obtained.

Solvents which differ much in their intermolecular forces they have no tendency to mix each other (they form separating layer easily). So, while stronger shaking is needed for them to mix, shorter time is enough for them to form separating layer.

On another hand, solvents which do not differ much in their intermolecular forces they have a greater ability to mix each other. So, in this case less shaking is needed for them to mix while longer time is required for them to form separating layer.

EXPERIMENTS

Experiment 23

Kipute was attending her weekend mentorship lab session at the regional science center. As part of her advanced chemistry project, she had taken interest in how pharmaceutical compounds behave in different solvents—something she imagined would be useful one day when formulating medicine.

That afternoon, she was tasked with analyzing **succinic acid**, a compound found in metabolism and also used in flavorings. The challenge? To investigate how it distributes between water and an organic solvent—**diethyl ether**—just like real chemists do when extracting drugs from natural sources.

She carefully set up her glassware. On the bench were three bottles labelled:

- **A1:** 0.1M sodium hydroxide solution
- **A2:** Unknown concentration of succinic acid
- **A3:** Diethyl ether
- Phenolphthalein indicator (P.O.P)

Kipute's mission was twofold: **First**, to determine the concentration of succinic acid in water. **Second**, to see how it split between water and ether when mixed—exactly how extraction works in pharmaceutical and environmental chemistry.

She was also aware with the fact that: Succinic acid dissolves in both water and diethyl ether at constant temperature while maintaining a constant ratio of concentrations in the solvents under consideration.

Procedure 1

1. She pipetted **20cm³ (or 25cm³)** of solution **A2** into a clean conical flask.
2. Added **2 to 3 drops** of **P.O.P indicator**.
3. Filled the burette with **A1 (NaOH)** and titrated **A2** against it until the colour change is observed.
4. She recorded the **volume of A1** used.
5. Finally, she noted the **room temperature** using a thermometer.

Results:

- Volume of pipette used: ____________ cm³
- Volume of **A1** used: ________________ cm³
- Room temperature: ________________ °C

Procedure 2

1. Kipute measured **75cm³ of A3 (ether)** into a separating funnel.
2. Added **75cm³ of A2**, shook vigorously, and let the system to stand for 2 minutes.
3. She drained the **aqueous layer**, took **20cm³ (or 25cm³)** of it (by using measuring cylinder), and titrated it with **A1** using P.O.P indicator.
4. She recorded the volume of **A1** used again.

Results:

1. The volume of the pipette used was _________ cm³

2. The volume of **A1** was __________ cm^3

Questions

a) Write a balanced chemical equation representing the reaction taking place in **procedure 1**(3)

b) Calculate:

   i. The initial concentration of **A2**
   ii. The concentration of **A2** in the aqueous layer
   iii. The concentration of **A2** in the organic layer

c) Calculate the distribution coefficient of **A2** between water and diethyl ether

d) In which solvent does A2 dissolve more between water and diethyl ether? Explain.

Solution

Results in procedure 1

1. The volume of the pipette used was **20 cm^3**
2. The volume of A_1 was **20.00 cm^3**
3. The room temperature was **27**°C

For 25 mL pipette: The volume of A1 will be **25cm^3**.

Results in procedure 2

1. The volume of the aqueous layer taken was **20 cm^3**
2. The volume of A_1 used was **16.80 cm^3**

For 25 mL pipette: The volume of A1 will be **21.00cm^3**.

Making Sense of the Experimental Data

The decrease in volume of NaOH used in the second titration clearly indicates that a portion of succinic acid was transferred into the ether layer.

However, since the drop in concentration is small, this confirms that succinic acid is much more soluble in water than in diethyl ether and hence the distribution coefficient of succinic acid between water and ether will be significantly greater than 1.

Response to Questions

(a) $\mathrm{HOOCCH_2CH_2COOH(aq) + 2NaOH(aq) \longrightarrow NaOOCCH_2CH_2COONa(aq) + 2H_2O(l)}$

(b) Calculation of each part is as follows:

(i) Using; $\frac{M_{acid} \times V_{acid}}{M_{base} \times V_{base}} = \frac{n_{acid}}{n_{base}}$

Where:

$V_{base} = 20cm^3$,$V_{acid} = 20cm^3$, $M_{base} = 0.1M$, $n_{acid} = 1$, $n_{base} = 2$

Substituting $\frac{M_{acid} \times 20cm^3}{0.1M \times 20cm^3} = \frac{1}{2}$; $M_{acid} = 0.05M$

The initial concentration of A2 was 0.05M.

(ii) Again using; $\frac{M_{acid} \times V_{acid}}{M_{base} \times V_{base}} = \frac{n_{acid}}{n_{base}}$

Where in this case, $V_{base} = 16.8cm^3$

Substituting $\frac{M_{acid} \times 20cm^3}{0.1M \times 16.8cm^3} = \frac{1}{2}$; $M_{acid} = 0.042M$

The concentration of A2 in aqueous layer was 0.042M.

(iii) Using $n = MV$;

Initial number of moles (before partition) $= 0.075L \times 0.05M$

Final number of moles (after partition) $= 0.075L \times 0.042M$

$$\text{Number of moles of A2 in organic layer} = \text{Initial number of moles} - \text{Final number of moles}$$

$$= 0.075L \times 0.05M - 0.075L \times 0.042M$$

$$= 0.075L\,(0.05M - 0.042M)$$

$$[A2] \text{ in organic layer} = \frac{n}{V} = \frac{0.075L\,(0.05M - 0.042M)}{0.075L}$$

The final equation simplifies to:

$$[A2] \text{ in organic layer} = 0.05M - 0.042M = 0.008M$$

The concentration of A2 in organic layer was $0.008M$.

You should be able to notice that: When volumes are the same, the concentration of A2 in organic layer becomes simply the difference between its initial concentration before partition and its concentration in aqueous layer after partition.

That is;

$[A2] \text{ in organic layer} = \text{Initial } [A2] - [A2] \text{ in aqueous layer} = (0.05 - 0.042)M = 0.008M$

Be careful!

This simplification is only valid when **equal volumes of both layers are used**. Refer to **Experiment 22** for a clearer understanding of how unequal phase volumes affect the validity of using direct concentration differences in partition coefficient calculations.

(c) $K_d = \frac{[A2] \text{ in aqueous layer}}{[A2] \text{ in organic layer}} = \frac{0.042M}{0.008M} = 5.25$

The distribution coefficient of A2 between water and diethyl ether is 5.25.

(d) A2 dissolves more in water.

Explanation:
Since the distribution coefficient is greater than 1**,** this indicates that A2 (succinic acid) dissolves more in water than in organic layer**.** This is because succinic acid can form strong intermolecular hydrogen bonds with water molecules, significantly enhancing its solubility in the aqueous phase.

Experiment 24

At the district education resource lab, **Mr. Akilikubwa**, the ever-dedicated laboratory technician, had received a request from the regional curriculum office. They needed validation of solvent extraction procedures for use in advanced practical sessions across schools.

One common experiment involved **succinic acid**, a dicarboxylic acid with pharmaceutical and industrial applications. The goal was to observe its behavior when distributed between **water and diethyl ether**—a model for drug extraction in biochemical industries.

To ensure the experiment was repeatable for school settings, Mr. Akilikubwa meticulously prepared three solutions:

- **TT**: 0.1M sodium hydroxide
- **PP**: Unknown concentration of succinic acid
- **RR**: Diethyl ether

- **Phenolphthalein indicator (P.O.P)**

He planned two procedures—first to determine the concentration of the succinic acid solution, and second to observe its distribution between the aqueous and organic layers.

Procedure 1: Direct Titration of Succinic Acid

1. Mr. Akilikubwa pipetted **$20cm^3$ (or $25cm^3$)** of **PP** into a clean conical flask.
2. Added **2–3 drops** of phenolphthalein (P.O.P).
3. Filled a burette with **TT (NaOH)**.
4. He titrated **PP** against **TT** until the colour change is observed.
5. Lastly, he noted the **room temperature** using a thermometer.

Results:

- Volume of pipette used: __________ cm^3
- Volume of TT used: _____________ cm^3
- Room temperature: _______________ ℃

Procedure 2: Extraction and Second Titration

1. Using a measuring cylinder, he measured **50 cm^3 of solution PP** and **diluted it with 50 cm^3 of distilled water** in a clean beaker to make a **total of 100 cm^3**.
2. Transferred the **diluted PP solution** into a **separating funnel**.
3. Using a clean measuring cylinder, he added **50 cm^3 of diethyl ether (RR)** into the separating funnel; shook the funnel vigorously, and allowed it to stand for 2 minutes.
4. After separation, he drained the **aqueous layer**, measured **$20cm^3$ (or $25cm^3$)** of it, and titrated it again against **TT** using P.O.P.
5. The volume of **TT** used was recorded carefully.

Results:

- Volume of aqueous layer taken: _____________ cm^3
- Volume of TT used: ________________________ cm^3

Questions

a) Why was it necessary to shake the separating funnel and then allow the system to stand before separating the layers?

b) Write a balanced chemical equation representing the reaction taking place in **procedure 1**(4)
c) Calculate:
   i. The original concentration of PP
   ii. The concentration of PP after dilution
   iii. The concentration of PP in the aqueous layer
   iv. The concentration of PP in the organic layer
d) Calculate the distribution coefficient of PP between water and diethyl ether

Solution

Procedure 1

Results:

1. The volume of the pipette used was **20 cm^3**.
2. The volume of TT was **28.00cm^3**.
3. The room temperature was **25**°C.

For 25 mL pipette: The volume of TT will be **35cm^3**.

Procedure 2

1. The volume of the aqueous layer taken was **20cm^3**.
2. The volume of TT used was **12.80cm^3**.

For 25 mL pipette: The volume of TT will be **16.00cm^3**.

Making Sense of the Experimental Data

The decrease in volume of NaOH used in the second titration clearly indicates that the acid was diluted first, then partially removed from water into ether.

Diluting 50cm^3 of the original solution with an equal volume of water (50cm^3) should halve the acid concentration—reducing the required NaOH volume from 28cm^3 to 14cm^3. However, since only 12.8 cm^3 of NaOH was used in the second titration, this indicates that some of the acid was moved into the ether phase.

Nevertheless, the small drop from 14cm^3 to 12.8cm^3 suggests that most of the succinic acid remained in the aqueous phase, confirming that succinic acid is much more soluble in water than in diethyl ether and hence the distribution coefficient of succinic acid between water and ether will be significantly greater than 1.

Response to Questions

(a) **Shaking the separating funnel** is essential to ensure maximum contact between the aqueous phase (water) and the organic phase (diethyl ether) so that the succinic acid can distribute (partition) itself according to its solubility in both solvents.

Allowing the system to stand undisturbed afterward serves two purposes: firstly, it permits clear separation of the two layers due to differences in density (ether floats on water). Also, it allows the distribution process to settle into equilibrium, making the aqueous layer analysis valid and reproducible.

(b) $\mathrm{HOOCCH_2CH_2COOH(aq) + 2NaOH(aq) \longrightarrow NaOOCCH_2CH_2COONa(aq) + 2H_2O(l)}$

(c) Calculation of each part is as follows:

(i) Using; $\frac{\mathrm{M_{acid} \times V_{acid}}}{\mathrm{M_{base} \times V_{base}}} = \frac{\mathrm{n_{acid}}}{\mathrm{n_{base}}}$

Where:

$\mathrm{V_{base}} = 28\mathrm{cm^3}\ ,\mathrm{V_{acid}} = 20\mathrm{cm^3}, \mathrm{M_{base}} = 0.1\mathrm{M}, \mathrm{n_{acid}} = 1,\ \mathrm{n_{base}} = 2$

Substituting $\frac{\mathrm{M_{acid}} \times 20\mathrm{cm^3}}{0.1\mathrm{M} \times 28\mathrm{cm^3}} = \frac{1}{2};\ \mathrm{M_{acid}} = 0.07\mathrm{M}$

The original concentration of PP was 0.05M.

(ii) Using $\mathrm{M_cV_c = M_dV_d}$

Where:

$\mathrm{M_c}$ = Concentration before dilution = 0.07M

$V_c = \text{Volume before dilution} = 50\text{cm}^3$

$V_d = \text{Volume after dilution} = 100\text{cm}^3$

$M_d = \text{Concentration after dilution}$

Substituting $0.07\text{M} \times 50\text{cm}^3 = M_d \times 100\text{cm}^3$; $M_d = 0.035\text{M}$

The concentration of PP after dilution was 0.035M.

(iii) Again using; $\frac{M_{acid} \times V_{acid}}{M_{base} \times V_{base}} = \frac{n_{acid}}{n_{base}}$

Where in this case, $V_{base} = 12.8\text{cm}^3$

Substituting $\frac{M_{acid} \times 20\text{cm}^3}{0.1\text{M} \times 12.8\text{cm}^3} = \frac{1}{2}$; $M_{acid} = 0.032\text{M}$

The concentration of PP in aqueous layer was 0.032M.

(iv) Using $n = MV$;

Initial number of moles (before partition) $= 0.05\text{L} \times 0.07\text{molL}^{-1} = 3.5 \times 10^{-3}\text{mol}$

Final number of moles (after partition) $= 0.1\text{L} \times 0.032\text{molL}^{-1} = 3.2 \times 10^{-3}\text{mol}$

Number of moles of PP in organic layer $= (3.5 \times 10^{-3} - 3.2 \times 10^{-3})\text{mol}$

$$= 3 \times 10^{-4}\text{mol}$$

$$[\text{PP}] \text{ in organic layer} = \frac{n}{V} = \frac{3 \times 10^{-4}\text{mol}}{0.05\text{L}} = 0.006\text{M}$$

The concentration of PP in organic layer was 0.006M.

(d) $K_d = \frac{[\text{PP}] \text{ in aqueous layer}}{[\text{PP}] \text{ in organic layer}} = \frac{0.032\text{M}}{0.006\text{M}} = 5.3$

The distribution coefficient of PP between water and diethyl ether is 5.3.

Experiment 25

It was a warm afternoon, and Kipute found herself in the senior laboratory with a curious task on her hands; **a double-solvent experiment** involving ammonia. Her chemistry instructor had mentioned this would be excellent preparation for real-life applications like **environmental cleanup** and **pharmaceutical extraction**, where understanding how chemicals behave between solvents was crucial.

On her bench were the following solutions:

- **A**: 0.5M hydrochloric acid
- **B**: 0.05M hydrochloric acid
- **C**: Ammonia solution of unknown concentration
- **D**: Chloroform
- **Indicator**: Methyl orange.

Kipute knew that ammonia solution dissolves in both water and chloroform at constant temperature while maintaining a constant ratio of concentrations in the solvents under consideration.

Procedure Kipute Followed

1. She measured **20cm³ (or 25cm³)** of **solution C** and poured it into a clean separating funnel.
2. Then, she added **75cm³ of D** into the same funnel.
3. She shook the mixture thoroughly and allowed the two layers to settle for about two minutes.

4. Using a pipette, she carefully transferred the **lower organic layer (chloroform)** into a clean conical flask.
5. Added **2–3 drops of methyl orange**.
6. Filled her burette with **solution B (0.05M HCl)** and titrated until a colour change indicated the endpoint.
7. She recorded the volume of **B** used.
8. She then **repeated** the titration (procedure 4 to 7) using the **upper aqueous layer**, titrating **20cm³ of it** against **solution A**.

Results

_______cm^3 of the aqueous layer required _____cm^3 of solution A for complete reaction.

_______cm^3 of the organic layer required ______cm^3 of solution B for complete reaction.

The room temperature was _________℃

Questions

a) Calculate the molarity of ammonia present in:
   i. The aqueous layer
   ii. The organic (chloroform) layer
b) Calculate the distribution coefficient of ammonia between aqueous layer and organic (chloroform) layer.
c) In reference to the distribution coefficient obtained in (b) above, in which layer ammonia dissolved more?
d) Explain why different concentrations of hydrochloric acid are used to titrate the two layers.

Solution

Results:

20cm^3 of the aqueous layer required **19.20cm^3** of solution A.

20cm^3 of the organic layer required **7.70cm^3** of solution B.

The room temperature was **28℃**.

For 25 mL pipette: The volume of solution A will be **24cm^3** while that of solution B will be **9.6cm^3.**

Making Sense of the Experimental Data

Both larger volume and higher concentration of acid was required to neutralize the aqueous layer compared to the organic layer. This indicates that the amount of ammonia in the organic (chloroform) layer is much lower than in the aqueous layer. As a result, the distribution coefficient of ammonia between water and chloroform is expected to be much greater than 1, confirming that ammonia is much more soluble in water than in chloroform.

Response to Questions

(a) Reaction equation:

$$NH_4OH + HCl \longrightarrow NH_4Cl + H_2O;\ n_{acid} = n_{base} = 1$$

Then from; $\frac{M_{acid} \times V_{acid}}{M_{base} \times V_{base}} = \frac{n_{acid}}{n_{base}}$

(i) For aqueous layer: $M_{acid} = 0.5M, V_{acid} = 19.2cm^3,\ V_{base} = 20cm^3$

Substituting $\frac{0.5\text{M}\times19.2\text{cm}^3}{\text{M}_{\text{base}}\times20\text{cm}^3}=\frac{1}{1}$; $\text{M}_{\text{base}}=0.48\text{M}$

The molarity of ammonia in aqueous layer was 0.48M.

(ii) For organic layer: $\text{M}_{\text{acid}}=0.05\text{M}, \text{V}_{\text{acid}}=7.7\text{cm}^3,\ \text{V}_{\text{base}}=\ 20\text{cm}^3$

Substituting $\frac{0.05\text{M}\times7.7\text{cm}^3}{\text{M}_{\text{base}}\times20\text{cm}^3}=\frac{1}{1}$; $\text{M}_{\text{base}}=0.02\text{M}$

The molarity of ammonia in organic layer was 0.02M.

(b) $\text{K}_{\text{d}}=\frac{[\text{NH}_3]\text{ in aqueous layer}}{[\text{NH}_3]\text{ in organic layer}}=\frac{0.48\text{M}}{0.02\text{M}}=24$

The distribution coefficient of ammonia between water and chloroform is 24.

(c) Ammonia is highly soluble in the aqueous layer compared to chloroform due to its ability to form intermolecular hydrogen bonds with water molecules, which significantly enhances its solubility in water.

(d) Different concentrations of HCl are used to match the expected ammonia concentration in each layer:

- The aqueous layer is expected to contain more ammonia because ammonia is more soluble in water (due to hydrogen bonding). Therefore, more concentrated acid (0.5 M HCl) is needed to titrate it efficiently.
- The organic layer (chloroform) contains less ammonia because ammonia is less soluble in chloroform which is less polar. So less concentrated acid (0.05 M HCl) is used to allow greater precision in measuring small amounts of ammonia without overshooting the endpoint.

Experiment 26

It was late in the afternoon at the district laboratory, and **Mr. Akilikubwa** was preparing a demonstration on solvent extraction for the upcoming NECTA practical exam. This time, he focused on an elegant but often misunderstood concept: **the distribution of a solute between two immiscible solvents.**

The solute? **Iodine.**
The solvents? **Water and iso-butyl alcohol.**
The goal? To calculate how iodine distributes between the two—just like in pharmaceutical purification or chemical waste separation.

Laid out on the lab bench were:

- **AA**: A 1.52g/L iodine solution in water
- **BB**: 0.001M sodium thiosulphate
- **CC**: iso-butyl alcohol
- **Starch solution**.

Titration procedure Mr. Akilikubwa followed

1. He measured **50cm³ of AA** and poured it into a clean **separating funnel**.
2. Added **50cm³ of CC**.
3. He capped the funnel, shook it thoroughly, and let it settle for **2 minutes**, forming **two distinct layers**.
4. The **lower aqueous layer** was collected into a beaker.
5. From this, he pipetted **20cm³** into a titration flask.

6. Added **2cm³ of starch solution**—the mixture turned a deep blue.
7. He titrated this solution with **BB** until the colour change is observed.
8. He repeated the process to obtain consistent readings and recorded results in tabular form.

Experiment	Pilot	1	2
Final reading (cm^3)			
Initial reading (cm^3)			
Titre reading (cm^3)			

Questions

a) What is the aim of using starch solution in this experiment?

b) Calculate:

  (i) The concentration of iodine in the aqueous layer

  (ii) The concentration of iodine in the organic layer

  (iii) The distribution constant, K_d of iodine between iso – butyl alcohol and water (iso butyl alcohol – water solvents system).

c) If Mr. Akilikubwa was tired and only shook the separating funnel gently for one minute, how might this affect the determination of the distribution coefficient?

d) Why was starch added at the beginning of the titration in this experiment, rather than near the end point as in iodometry?

Solution

The volume of the pipette used was $20cm^3$

TABLE OF RESULTS

Experiment	Pilot	1	2
Final reading (cm^3)	10.20	20.10	30.00
Initial reading (cm^3)	0.00	10.20	20.00
Titre reading (cm^3)	10.20	9.90	10.00

$$\text{Average volume} = \frac{V_1 + V_2}{2}$$

$$\text{Average volume} = \frac{(9.90+10.00)}{2}\text{cm}^3 = 9.95\text{cm}^3$$

$20cm^3$ of AA in the aqueous layer required **$9.95cm^3$** of BB for complete reaction.

For 25 mL pipette: The volume of BB will be **$12.50cm^3$.**

Making Sense of the Experimental Data

The use of a very low concentration of sodium thiosulphate, combined with the relatively small titre volume obtained, suggests that only a small amount of iodine remained in the aqueous layer; most of it was extracted into the iso-butyl alcohol.

Consequently, the distribution coefficient of iodine between iso-butyl alcohol and water is expected to be significantly greater than 1, confirming that iodine is far more soluble in iso-butyl alcohol than in water.

Response to Questions

(a) Starch solution was used to indicate the end point of the titration of iodine in aqueous layer against sodium sulphate solution (BB).

(b) Calculation of each part is as follows:

(i) Reaction equation:

$$2S_2O_3^{2-} + I_2 \longrightarrow S_4O_6^{2-} + 2I^-;\ n_{reductant} = 2, n_{oxidant} = 1$$

(i) From; $\frac{M_{oxidant} \times V_{oxidant}}{M_{reductant} \times V_{reductant}} = \frac{n_{oxidant}}{n_{reductant}}$

Where: $M_{reductant} = 0.001M, V_{oxidant} = 20cm^3,\ V_{reductant} = 9.95cm^3$

Substituting $\frac{M_{oxidant} \times 20cm^3}{0.001M \times 9.95cm^3} = \frac{1}{2};\ M_{reductant} = 2.4875 \times 10^{-4}M$

The concentration of iodine in aqueous layer was $2.4875 \times 10^{-4}M$.

(ii) Using Molar concentration $= \frac{\text{Mass concentration}}{\text{Molar mass}}$

$$\text{Initial } [I_2] = \frac{1.52g/L}{254g/mol} = 5.9843 \times 10^{-3}M$$

Since equal volumes of both layers were used;

$[I_2]$ in organic layer = Initial $[I_2]$ − $[I_2]$ in aqueous layer

$$= (5.9843 \times 10^{-3} - 2.4875 \times 10^{-4})M = 5.73555 \times 10^{-3}M$$

The concentration of iodine in organic layer was approximately $5.7356 \times 10^{-3}M$

(iii) $K_d = \frac{[I_2] \text{ in organic layer}}{[I_2] \text{ in aqueous layer}} = \frac{5.7356 \times 10^{-3}M}{2.4875 \times 10^{-4}M} = 23$

The distribution coefficient of iodine between iso-butyl alcohol and water is 23.

(c) Gentle shaking leads to incomplete transfer of iodine from the aqueous layer to the organic layer, resulting in a lower concentration of iodine in the organic phase. Consequently, the calculated distribution coefficient will be smaller than the true value.

(d) In this experiment, only a small amount of iodine was present in the aqueous layer from the beginning. Therefore, there was no risk of forming a very stable starch–iodine complex, which typically occurs when large amounts of iodine are present. This made it appropriate to add starch at the start of the titration.

Experiment 27

Kipute had reached the final phase of her weekend research assignment under the mentorship of the district laboratory team. This time, her task was a bit more advanced—**to investigate how acetic acid behaves when placed between two immiscible solvents: water and carbon tetrachloride.**

But this wasn't just about solubility. She'd been told that in **carbon tetrachloride**, **acetic acid tends to form dimers**. This unique association would impact the distribution and titration results, something she was eager to explore.

On her workbench were:

- **M**: 0.1M sodium hydroxide
- **N**: 0.5M sodium hydroxide
- **O**: Acetic acid

- **P**: Carbon tetrachloride
- **Phenolphthalein indicator (POP)**
- **Distilled water**

She was to perform two experiments, one using **1cm³** of acetic acid and another using **2cm³**, and observe how the acid distributed itself between the solvents.

Procedure Kipute followed

For Both Experiments (1 and 2):

1. She measured **20cm³ of distilled water** into a separating funnel.
2. Added **1cm³** of **acetic acid**.
3. Rinsed the measuring cylinder and measured **20cm³ of carbon tetrachloride**, adding it into the same funnel.
4. She stoppered, shook the funnel well and allowed the contents to settle for **3 minutes**.
5. The **lower organic layer** (CCl_4) was run off into a beaker, and any boundary layer was discarded.
6. From the **aqueous layer**, she pipetted **10cm³** into a titration flask, added a few drops of **POP**, and titrated it with **solution N** until the colour change is observed.
7. Separately, she measured **10cm³ of the organic layer**, added POP, and titrated with **solution M**.
8. She repeated this entire process using **2cm³ of acetic acid**, recording the volume of base used for both layers in a tabular form.

Experiment	Volume of acetic acid used (cm^3)	Vol of 0.5M NaOH reacted with the aqueous layer (cm^3)	Vol of 0.1M NaOH reacted with the organic layer (cm^3)
1.			
2.			

Questions

a) Calculate:
   i. The concentration of acetic acid in aqueous layer for experiment 1 and 2
   ii. The concentration of acetic acid in organic layer for experiment 1 and 2

b) Calculate the distribution coefficient, K_d of acetic acid between water and carbon tetrachloride in both experiments 1 and 2

c) In this experiment, the ratio; $\frac{\text{concentration of } CH_3COOH \text{ in } H_2O}{\text{concentration of } CH_3COOH \text{ in } CCl_4}$ is not constant. Why?

d) Explain the significance of using different concentrations of sodium hydroxide (0.1M and 0.5M) for the two layers.

e) Explain why must the boundary layer be discarded when separating the two solvents?

Solution

TABLE OF RESULTS

Experiment	Volume of acetic acid used (cm^3)	Vol of 0.5M NaOH reacted with the aqueous layer (cm^3)	Vol of 0.1M NaOH reacted with the organic layer (cm^3)
1.	1	17.20	1.40
2.	2	32.90	5.10

Making Sense of the Experimental Data

Both the larger volume and higher concentration of base (NaOH) were required to neutralize the aqueous layer compared to the organic layer. This indicates that the amount (or solubility) of acetic acid in the organic layer (CCl_4) is significantly lower than in the aqueous layer. The high solubility of acetic acid in water can be attributed to its ability to form intermolecular hydrogen bonds with water molecules.

Response to Questions

(a) Reaction equation:

$$\mathrm{CH_3COOH} + \mathrm{NaOH} \longrightarrow \mathrm{CH_3COONa} + \mathrm{H_2O};\ n_{acid} = n_{base} = 1$$

Then from; $\frac{M_{acid} \times V_{acid}}{M_{base} \times V_{base}} = \frac{n_{acid}}{n_{base}}$

(i) **For aqueous layer:**

First experiment: $M_{base} = 0.5M, V_{acid} = 10cm^3,\ V_{base} = 17.2cm^3$

Substituting $\frac{M_{acid} \times 10cm^3}{0.5M \times 17.2cm^3} = \frac{1}{1};\ M_{acid} = 0.86M$

The concentration of acetic acid in aqueous layer for experiment 1 is 0.86M.

Second experiment: $M_{base} = 0.5M, V_{acid} = 10cm^3,\ V_{base} = 32.9cm^3$

Substituting $\frac{M_{acid} \times 10cm^3}{0.5M \times 32.9cm^3} = \frac{1}{1};\ M_{acid} = 1.645M$

The concentration of acetic acid in aqueous layer for experiment 2 is 1.645M.

(ii) **For organic layer:**

First experiment: $M_{base} = 0.1M, V_{acid} = 10cm^3,\ V_{base} = 1.4cm^3$

Substituting $\frac{M_{acid} \times 10cm^3}{0.1M \times 1.4cm^3} = \frac{1}{1};\ M_{acid} = 0.014M$

The concentration of acetic acid in aqueous layer for experiment 1 is 0.014M.

Second experiment: $M_{base} = 0.5M, V_{acid} = 10cm^3,\ V_{base} = 5.1cm^3$

Substituting $\frac{M_{acid} \times 10cm^3}{0.1M \times 5.1cm^3} = \frac{1}{1};\ M_{acid} = 0.051M$

The concentration of acetic acid in aqueous layer for experiment 2 is 0.051M.

(b) Since acetic acid undergoes dimerization in the CCl_4 layer, the correct K_d expression is as follows:

$$K_d = \frac{\text{concentration of } CH_3COOH \text{ in water}}{\sqrt{\text{concentration of } CH_3COOH \text{ in } CCl_4}}$$

For experiment 1:

$$K_d = \frac{0.86M}{\sqrt{0.014M}} = 7.268$$

The distribution coefficient of acetic acid between water and carbon tetrachloride in experiment 1 is approximately 7.3.

For experiment 2:

$$K_d = \frac{1.645M}{\sqrt{0.051M}} = 7.284$$

The distribution coefficient of acetic acid between water and carbon tetrachloride in experiment 2 is approximately 7.3.

(c) Because molecular state of acetic acid is not the same in both solvents as it tends to undergo association into dimer in the carbon tetrachloride (CCl_4).

(d)

- The aqueous layer contains more acetic acid due to higher solubility of the acid in it. So more concentrated base (0.5M NaOH) is used to neutralize it efficiently in a manageable titre volume.
- The organic layer (CCl_4) contains less acetic acid due to smaller solubility and also due to dimer formation, so a weaker base (0.1M NaOH) is used to allow better precision in measurement of small acid quantities.

(e) The boundary layer is the thin zone where water and CCl_4 intermingle containing microscopic droplets of each solvent, along with a mixed concentration of acetic acid. So taking it, would lead to inaccurate titration readings and misleading results in calculating the amount of acid in either layer.

Chapter 5

CHEMICAL KINETICS

INTRODUCTION

Chemical kinetics is the branch of physical chemistry which is concerned about the study of determination of rates of chemical reactions and factors affecting it. In this book, we are going to study variation of reaction rate with respect to concentration and temperature.

In determining reaction rate, it is common to measure time for a certain amount of reactant to react or certain amount of product to be formed. Since the reaction rate varies inversely proportional to time for given concentration, the reciprocal of time $\left(\frac{1}{t}\right)$ is commonly treated as the reaction rate in these kind of experiments.

It is worth for a reader to understand that in chemical kinetics' experiments, we are more interested on accuracy of trend of values and not accuracy of individual values.

Experiments of chemical kinetics can be used to determine either **reaction order** (in studying effect of concentration on the reaction rate) or **activation energy** (in studying temperature effect on the reaction rate).

EXPERIMENTS

Experiment 28

It was a sunny Saturday morning, and Kipute ever eager to sharpen her experimental skills, decided to explore how the **rate of chemical reactions** changes with concentration. Her uncle, Mr. Akilikubwa, had left her alone in the school laboratory with a challenge: *"Figure out the relationship between concentration and reaction speed using sodium thiosulphate and hydrochloric acid. And observe carefully!"*

On the lab bench in front of her were:

- **A**: 0.5M Sodium thiosulphate solution
- **B**: 0.2M Hydrochloric acid
- **Distilled water**
- A **100 cm^3 beaker**, **50 mL measuring cylinder**, and a **stopwatch**

She had read about the experiment: *"A chemical reaction that produces sulphur causes the solution to turn milky, hiding a marked cross beneath the beaker. The faster the cross disappears, the faster the reaction."*

She was also told that sodium thiosulphate reacts with hydrochloric acid to give amorphous sulphur according to the following ionic equation:

$$S_2O_3^{2-}(aq) + 2H^+(aq) \longrightarrow SO_2(g) + S(g) + H_2O(l)$$

The disappearing cross procedure Kipute followed

1. She drew a **bold black cross** on white paper using her pen and placed a **100 cm^3 beaker** right over it, ensuring the cross was visible through the beaker's top.
2. Using a measuring cylinder, she poured **10 cm^3 of solution A** into the beaker.
3. After rinsing the cylinder, she measured **10 cm^3 of solution B**.
4. She added the acid into the beaker containing thiosulphate, quickly **started the stopwatch**, swirled the beaker once, and carefully watched.

5. As the solution turned cloudy, the black cross started to fade.
6. She **stopped the clock** at the exact moment the cross disappeared completely from view.
7. She was then repeated procedure (2) to (6) but this time by adding a given amount of distilled water to the given amount of sodium solution A as shown in the table below:

Volume of $S_2O_3^{2-}$ (cm^3)	Volume of H2O (cm3)	Volume of HCl (cm3)	$[S_2O_3^{2-}]$ (M)	Time, t(sec)	$[S_2O_3^{2-}] \times t$(MSec)	$\log[S_2O_3^{2-}]$	$\left(\frac{1}{t}\right) sec^{-1}$
10	0	10					
8	2	10					
6	4	10					
4	6	10					
2	8	10					

Questions

a) Comment on the product of concentration of thiosulphate and time.
b) Draw a graph of $\log[S_2O_3^{2-}]$ against time, $t(sec)$
c) Using the graph drawn in (b) above, comment on the order of the reaction with respect to $S_2O_3^{2-}$. Give reason(s)
d) What causes the cloudy milky colour in the reaction mixture?

Solution

TABLE OF RESULTS

Volume of $S_2O_3^{2-}$ (cm^3)	Volume of H_2O (cm^3)	Volume of HCl (cm^3)	$[S_2O_3^{2-}]$ (M)	Time, t(sec)	$[S_2O_3^{2-}] \times$ t(MSec)	$\log[S_2O_3^{2-}]$	$\left(\frac{1}{t}\right) sec^{-1}$
10	0	10	0.5	20	10	-0.30	0.05
8	2	10	0.4	24	9.6	-0.40	0.04
6	4	10	0.3	33	9.9	- 0.52	0.03
4	6	10	0.2	50	10	- 0.70	0.02
2	8	10	0.1	102	10.2	-1	0.0098

To fill the $[S_2O_3^{2-}]$ column, use dilution formula $(M_cV_c = M_dV_d)$.

Making Sense of the Experimental Data

Practical tip: *Reaction between sodium sulphate and hydrochloric acid to form sulphur is of the first order with respect to the thiosulphate.*

For first order chemical reaction; $\mathbf{Ct = constant}$.

Where C is the initial concentration of the reactant ($S_2O_3^{2-}$)

t is the time taken for the reaction to form given amount of product (for this case, time required to form certain amount of sulphur that will make the across to be disappeared completely from view).

But concentration of $S_2O_3^{2-}$ (reactant) varies directly proportional to its volume taken for dilution with water.

So, if V is the volume of $S_2O_3^{2-}$, then $Vt = \text{constant}$.

It follows that: $V_1t_1 = V_2t_2$ or $\mathbf{t_2} = \frac{\mathbf{V_1t_1}}{\mathbf{V_2}}$

Taking $V_1 = 10\text{cm}^3, t_1 = 20\text{sec}$

When:

$V_2 = 8\text{cm}^3, t_2 = \frac{10\text{cm}^3 \times 20\text{sec}}{8\text{cm}^3} = \mathbf{25sec} \approx$ experimental data (24sec)

$$V_2 = 6\text{cm}^3, t_2 = \frac{10\text{cm}^3 \times 20\text{sec}}{6\text{cm}^3} = \mathbf{33sec} = \text{experimental data}$$

$$V_2 = 4\text{cm}^3, t_2 = \frac{10\text{cm}^3 \times 20\text{sec}}{4\text{cm}^3} = \mathbf{50sec} = \text{experimental data}$$

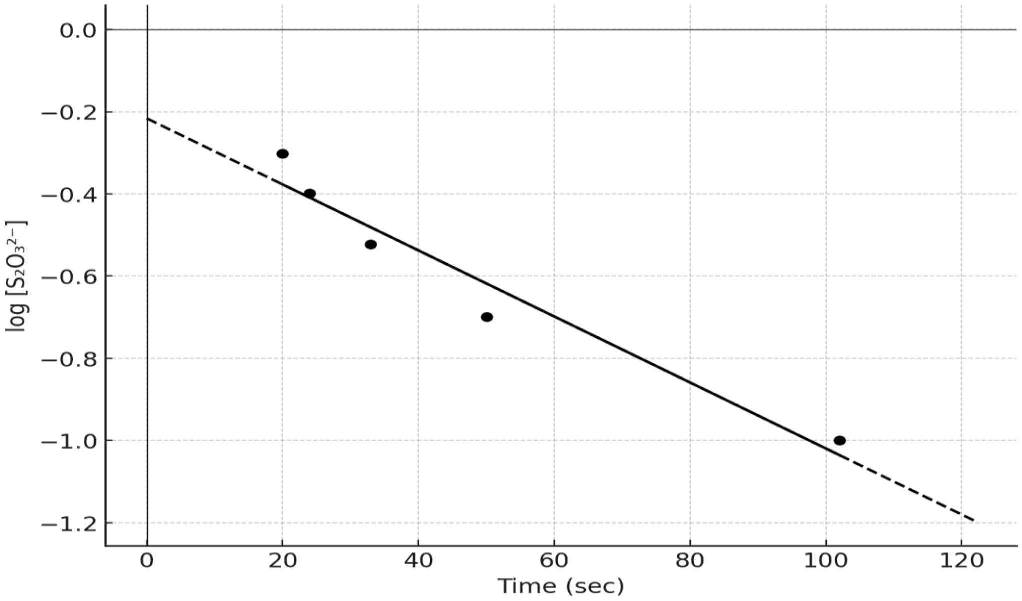
$$V_2 = 2\text{cm}^3, t_2 = \frac{10\text{cm}^3 \times 20\text{sec}}{2\text{cm}^3} = \mathbf{100sec} \approx \text{experimental data (102sec)}$$

To conclude: *The calculated time values closely match the experimentally observed times, indicating that the experiment was successful.*

Response to Questions

(a) The product of sodium thiosulphate concentration and time remains approximately constant across all experiments.

(b) **Graph of log $[S_2O_3^{2-}]$ vs Time**



(c) The order of the reaction with respect to $S_2O_3^{2-}$ is first order reaction since the graph drawn in (b) above is a straight line with negative slope implying that it agrees with the first order equation which is $\log(a-x) = \frac{-kt}{2.303} + \log a$

(d) The cloudy milky colour in the reaction mixture is caused by the formation of insoluble solid sulphur.

Experiment 29

It was a quiet afternoon in the school laboratory. With no students around, **Mr. Akilikubwa** decided to conduct one of his favorite classic experiments—not to teach, but to satisfy his own lingering curiosity: *Could he experimentally determine the orders of a reaction just by observing a vanishing cross?*

This time, he set out to investigate how the rate of a reaction depends on the **concentration of reactants** using the reaction between **sodium thiosulphate** and **hydrochloric acid**.

On the clean bench in front of him were:

- **X**: 0.2 M sodium thiosulphate
- **Y**: 0.1 M hydrochloric acid
- A bottle of **distilled water**
- A **stopwatch**, **50 mL beaker**, and a **measuring cylinder**
- A piece of paper with a bold black "+" marked in ink

Mr. Akilikubwa recalled that the reaction between $Na_2S_2O_3$ and an acid is governed by the rate expression as follows:

$$\text{Rate} = -\frac{d[S_2O_3^{2-}]}{dt} = k[S_2O_3^{2-}]^m[H^+]^n$$

Where m and n are the orders of the reaction with respect to sodium thiosulphate and the acid respectively.

The Disappearing "+" experiment procedure followed by Mr. Akilikubwa

1. He placed a **50 mL beaker** directly over the paper marked with a bold "+".
2. Using a measuring cylinder, he poured **10 cm³ of solution X** into the beaker.
3. After rinsing the cylinder, he measured **10 cm³ of solution Y** and quickly added it to the same beaker.
4. He **started the stopwatch immediately**, gave the beaker a gentle swirl, and watched closely.
5. As sulfur began to form, the "+" gradually faded until it could no longer be seen from above.
6. He **recorded the time** taken for the cross to disappear completely when viewed from the top of the beaker in which the reaction is taking place.
7. He repeated procedure (ii) to (vi) but this time by adding a given amount of distilled water to the given amount of sodium thiosulphate as shown in the table below:

Exp.	Volume of $Na_2S_2O_3$ (cm^3)	Volume of water (cm^3)	Volume of HCl (cm^3)	Time, t (sec)	$\left(\frac{1}{t}\right) sec^{-1}$
1.	10	0	10		
2.	8	2	10		
3.	6	4	10		
4.	4	6	10		
5.	2	8	10		

Questions

a) Write the ionic equation using the rate expression above.
b) What is the order of the reaction with respect to sodium thiosulphate?
c) Calculate the value of k if n = 2. Given that, the volume of individual component is direct proportional to the concentration.
d) Explain why is hydrochloric acid kept at a constant volume in all trials?

Solution

TABLE OF RESULTS

Exp.	Volume of $Na_2S_2O_3$ (cm^3)	Volume of water (cm^3)	Volume of HCl (cm^3)	Time, t (sec)	$\left(\frac{1}{t}\right)\text{sec}^{-1}$
1.	10	0	10	64	0.016
2.	8	2	10	80	0.0125
3.	6	4	10	108	0.0093
4.	4	6	10	162	0.0062
5.	2	8	10	321	0.0031

Making Sense of the Experimental Data

Again the reaction is of the first order with respect to the thiosulphate.

So using;

$$\mathbf{t_2 = \frac{V_1 t_1}{V_2}}$$

Where $V_1 = 10\text{cm}^3, t_1 = 64\text{sec}$

When:

$V_2 = 8\text{cm}^3, t_2 = \frac{10\text{cm}^3 \times 64\text{sec}}{8\text{cm}^3} = \mathbf{80sec} = \text{experimental data}$

$V_2 = 6\text{cm}^3, t_2 = \frac{10\text{cm}^3 \times 64\text{sec}}{6\text{cm}^3} = \mathbf{107sec} \approx \text{experimental data (108sec)}$

$V_2 = 4\text{cm}^3, t_2 = \frac{10\text{cm}^3 \times 64\text{sec}}{4\text{cm}^3} = \mathbf{160sec} \approx \text{experimental data (162sec)}$

$V_2 = 2\text{cm}^3, t_2 = \frac{10\text{cm}^3 \times 64\text{sec}}{2\text{cm}^3} = \mathbf{320sec} \approx \text{experimental data (321sec)}$

To conclude: *The calculated time values closely match the experimentally observed times, indicating that the experiment was successful.*

Response to Questions

(a) $S_2O_3^{2-}(aq) + 2H^+(aq) \longrightarrow SO_2(g) + S(g) + H_2O(l)$

(b) Since concentration of reactant varies directly proportional to its volume taken for dilution with water. Individual volume of reactants may be treated as concentration.

Also since the reaction rate varies inversely proportional to time for given concentration, the reciprocal of time $\left(\frac{1}{t}\right)$ is may be treated as the reaction rate (t is the time required to form fixed amount of sulphur which makes the cross to disappear).

Then from; $\text{Rate(R)} = k[S_2O_3^{2-}]^m \times [H^+]^n$

$$R_1 = k(10)^m \times (10)^n = 0.016\text{sec}^{-1} \ldots\ldots\ldots\ldots. \text{(i)},$$

$$R_2 = k(8)^m \times (10)^n = 0.0125\text{sec}^{-1} \ldots\ldots\ldots\ldots.(ii)$$

Taking (i) ÷ (ii); (You may take any two set of data)

$$\frac{R_1}{R_2} = \frac{k(10)^m \times (10)^n}{k(8)^m \times (10)^n}$$

$$\frac{0.016}{0.0125} = \left(\frac{10}{8}\right)^m \times \left(\frac{10}{10}\right)^n$$

$$1.28 = (1.25)^m$$

$$\log 1.28 = \log 1.25^m$$

$$\log 1.28 = m \log 1.25, \text{ or } m = \frac{\log 1.28}{\log 1.25} = 1$$

The reaction is of **the first order** with respect to sodium thiosulphate.

(c) From $R_1 = k(10)^m \times (10)^n = 0.016\text{sec}^{-1}$
Substituting $k(10\text{cm}^3)^1 \times (10\text{cm}^3)^2 = 0.016\text{sec}^{-1}$
From which; $k = \frac{0.016\text{sec}^{-1}}{1000\text{cm}^9} = 1.6 \times 10^{-5}\text{cm}^{-9}\text{sec}^{-1}$

The value of k is $1.6 \times 10^{-5}\text{cm}^{-9}\text{sec}^{-1}$.

(d) Hydrochloric acid is kept constant so that only one variable is changed which is the concentration of sodium thiosulphate. Thus any observed change in the reaction rate is due to the change in thiosulphate concentration only, not acid concentration. This allows for a controlled investigation of how thiosulphate concentration affects reaction rate.

Experiment 30

Kipute was in the school lab for her weekend research hour, a time she used to deepen her skills beyond classwork. Her latest interest? **Reaction kinetics**. Guided by her Chemistry teacher, she aimed to determine how **changing the concentration of sodium thiosulphate** affects the rate of its reaction with **hydrochloric acid**.

She wasn't just looking for a grade; she was exploring how these principles applied in fields like **pharmaceuticals**, where reaction time can be the difference between an effective treatment and a failed one.

To conduct her investigation, she carefully arranged:

- **L**: a solution containing 31.6 g/dm³ of sodium thiosulphate
- **M**: 0.1 M hydrochloric acid
- **D**: distilled water
- A **50 mL beaker**, **stopwatch**, **measuring cylinder**, and a **piece of paper marked "X"**

The disappearing cross procedure followed by Kipute

1. Kipute used a black pen to clearly mark a bold **"X"** on a sheet of white paper and placed a clean 50 mL beaker directly above it.
2. She measured **10 cm³** of solution L using a measuring cylinder and poured it into the beaker.
3. After rinsing the cylinder, she measured **10 cm³** of solution **M** and added it to the beaker. **She started the stopwatch immediately**.
4. She gave the beaker a gentle swirl and focused carefully on the **X**. As the **reaction progressed**, the solution turned **increasingly cloudy** due to sulphur formation.
5. When the X could no longer be seen through the top of the beaker, she **stopped the clock and recorded the time**.

6. She repeated the experiment, this time **diluting the sodium thiosulphate** with varying amounts of **distilled water** to observe how the reaction rate changed.

Exp.	**Volume of $Na_2S_2O_3$ (cm^3)**	**Volume of water (cm^3)**	**Volume of HCl (cm^3)**
1.	10	0	10
2.	8	2	10
3.	6	4	10
4.	4	6	10
5.	2	8	10

i. Record the findings in a tabular form as follows;

$[S_2O_3^{2-}]$(M)	**Time, t(sec)**	$[S_2O_3^{2-}] \times$ **t(MSec)**	$\left(\frac{1}{t}\right)$ sec^{-1}

Questions

a) Plot a graph of $[S_2O_3^{2-}]$(M) against t(sec)
b) Plot the graph of $(^1/_t)sec^{-1}$ against $[S_2O_3^{2-}]$(M)
c) Study your graphs and results then answer the following questions:
   i. What do you observe and notice about the values of $[S_2O_3^{2-}] \times t$
   ii. What is the effect of concentration of $Na_2S_2O_3$ on the rate of reaction? Support your answer with observations from the experiment.
   iii. What is the order of the reaction with respect to $Na_2S_2O_3$? Explain how you arrived at your conclusion.

d) Explain why it is better to use the same observer for all trials in this type of visual experiment?

Solution

TABLE OF RESULTS

$[S_2O_3^{2-}](M)$	Time, t(sec)	$[S_2O_3^{2-}] \times t(MSec)$	$\left(\frac{1}{t}\right) sec^{-1}$
0.2	64	12.8	0.0156
0.16	80	12.8	0.0125
0.12	108	12.96	0.0093
0.08	160	12.8	0.00625
0.04	321	12.84	0.0031

To fill the $[S_2O_3^{2-}]$ column, use dilution formula ($M_cV_c = M_dV_d$).

Making Sense of the Experimental Data

Again the reaction is of the first order with respect to the thiosulphate.

So using;

$$\mathbf{t_2 = \frac{C_1t_1}{C_2}}$$

Where $C_1 = 0.2M, t_1 = 64sec$

When:

$C_2 = 0.16M, t_2 = \frac{0.2M\times 64sec}{0.16M} = \mathbf{80sec} =$ experimental data

$C_2 = 0.12M, t_2 = \frac{0.2M \times 64sec}{0.12M} = \mathbf{107sec} \approx$ experimental data (108sec)

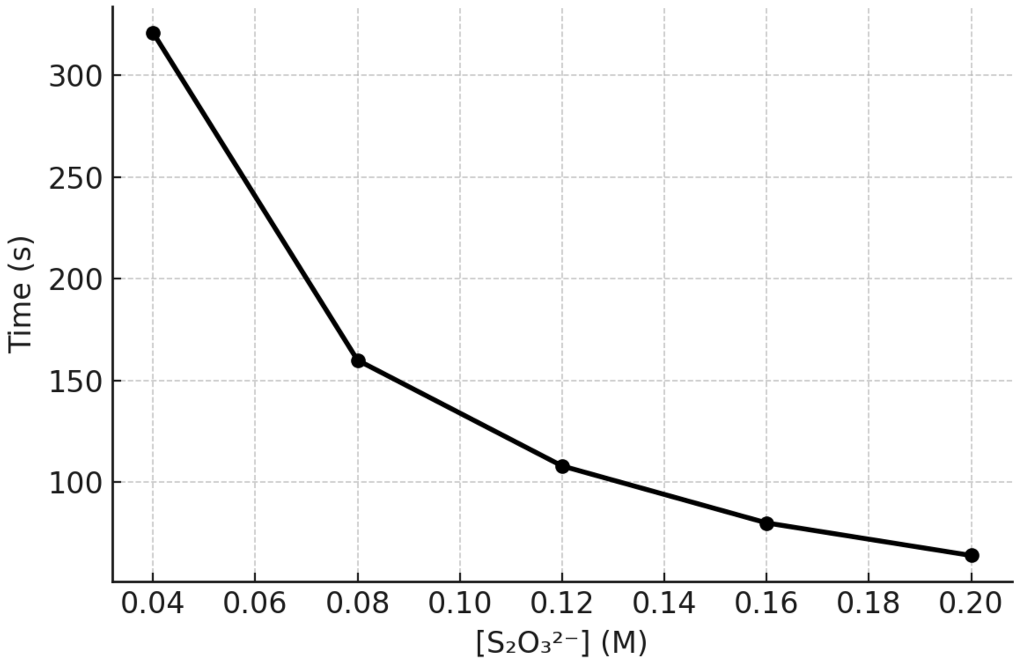
$C_2 = 0.08M, t_2 = \frac{0.2M \times 64sec}{0.08M} = \mathbf{160sec} =$ experimental data

$C_2 = 0.04M, t_2 = \frac{0.2M \times 64sec}{0.04M} = \mathbf{320sec} \approx$ experimental data (321sec)

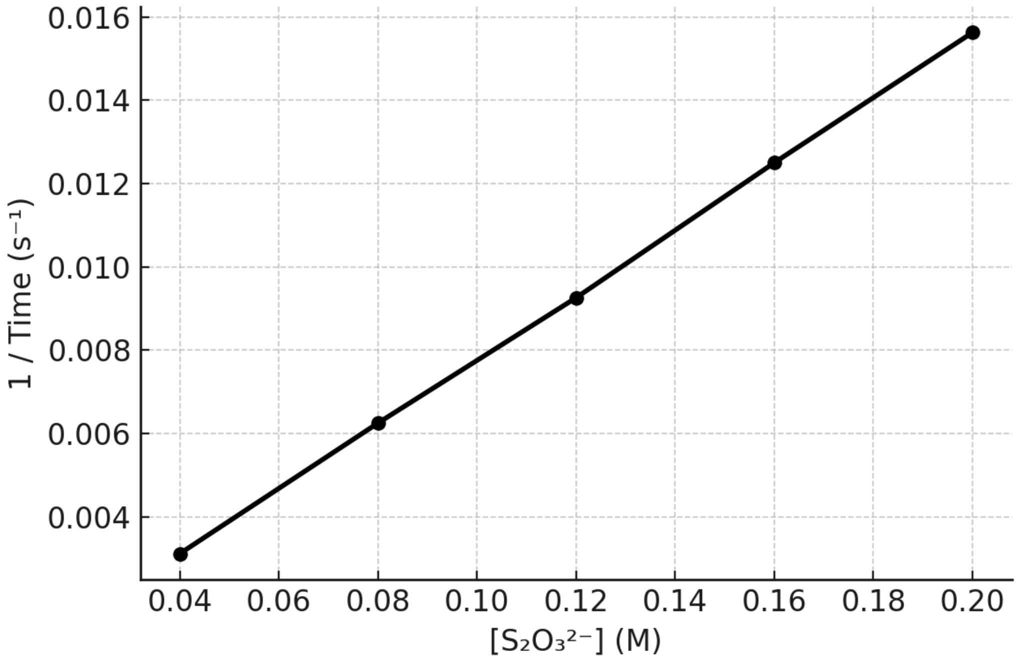
To conclude: *The calculated time values closely match the experimentally observed times, indicating that the experiment was successful.*

Response to Questions

(a) **$[S_2O_3^{2-}]$ vs Time**



(b) **(1/t) vs $[S_2O_3^{2-}]$**



(c) The answer for each part is as follows:

(i) The values of $[\mathbf{S_2O_3^{2-}}] \times \mathbf{t}$ remain approximately constant across all experiments.

(ii) As the concentration of $Na_2S_2O_3$ increases, the rate of reaction also increases. This is observed by the decrease in time taken for the cross to disappear as concentration increases, indicating a faster reaction.

(iii) The reaction is first order with respect to $Na_2S_2O_3$. This is due to the following facts:

- The value of $[\mathbf{S_2O_3^{2-}}] \times \mathbf{t}$ is approximately constant.
- The graph obtained when plotting $(\mathbf{1}/\mathbf{t})$ against $[\mathbf{S_2O_3^{2-}}]$ is straight line with positive slope.

(d) The decision of when the cross disappears is subjective and depends on the visual sensitivity and reaction speed of the observer. Using different observers could lead to inconsistent judgment, causing variation in timing that is not due to chemical differences. So using the same observer improves consistency and reliability of the measurements.

Experiment 31

It was a quiet Thursday afternoon in the school laboratory when **Mr. Akilikubwa**, the experienced laboratory technician, decided to test an experimental setup before handing it over to the form six students. The experiment was about **kinetics study** to determine how fast **hydrogen peroxide** reacts in the presence of **iodide ions and acid**, and what governs the speed of that reaction.

He was particularly interested in whether the reaction followed **first-order kinetics** with respect to hydrogen peroxide. To investigate, he designed a clever approach: monitor the **time taken for a fixed amount of iodine to form**, then react it with **sodium thiosulphate**, and repeat this at fixed intervals.

Mr. Akilikubwa gathered the following:

- **AB**: 0.2 M hydrogen peroxide
- **AC**: 0.2 M sodium thiosulphate solution
- **AD**: 10% potassium iodide
- **AE**: 1 M sulphuric acid
- **AF**: starch solution
- **AG**: distilled water
- **Burette**, **measuring cylinders**, **conical flask**, and a **stopwatch.**

As the experienced laboratory technician, Mr. Akilikubwa understood that in the presence of acid, hydrogen peroxide reacts with iodide ions as per equation below;

$$H_2O_2 + 2I^- + 2H^+ \rightarrow 2H_2O + I_2$$

The general expression of the equation is given by:

$$\text{Rate} = k[H_2O_2]^x[I^-]^y[H^+]^z$$

He also knew that if the concentrations of hydrogen ions and iodide ions are high, the order of the reaction with respect to hydrogen peroxide can be determined and hence the rate of the reaction. The rate of the reaction can be followed by noting the time taken by the reaction to produce amount of iodine enough to react with known volume of sodium thiosulphate of known concentration.

The reaction between iodine and sodium thiosulphate is represented as per reaction equation below:

$$I_2 + 2S_2O_3^{2-} \rightarrow SO_4O_6^{2-} + 2I^-$$

He carefully followed this procedure:

1. Measured **70 cm³** of distilled water (AG) into a 250 mL beaker or conical flask.
2. Added **10 cm³** of AD, **5 cm³** of AE, and **1 cm³** of AF.
3. Filled the burette with AC and added **2.5 cm³** into the reaction mixture. He swirled the flask.
4. Then, using a pipette, he added **10 cm³** of AB to the beaker.
5. As he poured about a half of the intended volume of AB, he started the **stopwatch**—timing how long it would take for the dark **blue-black colour** to appear. Swirled the mixture well to ensure complete mixing of the content.
6. Without stopping the stopwatch, he added **another 2.5 cm³** of AC and noted the next time when the colour appeared.
7. He continued this cycle of addition and timing, conducting a total of **six experiments**, adding 2.5 cm³ more AC each time.
8. He recorded the results in tabular form as follows:

Total vol. of AC added ($cm^{3)}$	Time,t (s) for colour to appear	Total calculated vol. of AC which reacted "x"(cm^3)	(a – x) where a is the initial vol. of AB in the mixture (cm^3)	Log(a – x)
2.5				
5.0				
7.5				
10.0				
12.5				
15.0				

Questions

a) Plot a graph of log (a – x)cm^3 against time, t(s).
b) Plot the graph of (a – x)cm^3 against time, t(s).
c) Using your two graphs in (a) and (c) above, what is the order of the reaction with respect to hydrogen peroxide? Explain your answer.
d) In this experiment, what is the significance of always adding the same volume of sodium thiosulphate at each cycle?
e) Why is starch solution added even though iodine is visible as a brown solution?

Solution

TABLE OF RESULTS

Total vol. of AC added ($cm^{3)}$	Time, t (s) for colour to appear	Total calculated vol. of AC which reacted "x"(cm^3)	(a – x) where a is the initial vol. of AB in the mixture (cm^3)	log(a – x)
2.50	140	1.25	8.75	0.942
5.00	300	2.50	7.50	0.875
7.50	646	3.75	6.25	0.796
10.00	877	5.00	5.00	0.699
12.50	1175	6.25	3.75	0.574
15.00	1594	7.50	2.50	0.398

How the value of x is obtained?

$H_2O_2 + 2I^- + 2H^+ \rightarrow 2H_2O + I_2$…………………………….(i)

$2S_2O_3^{2-} + I_2 \rightarrow S_4O_6^{2-} + 2I^-$…………………………...……....(ii)

Taking (i)+(ii) to eliminate iodine, gives the overall reaction equation which is:

$H_2O_2 + 2S_2O_3^{2-} + 2H^+ \rightarrow S_4O_6^{2-} + 2H_2O$;

From which, mole ratio of H_2O_2 to $S_2O_3^{2-}$ is $1:2$.

But since $n = MV$, number of moles reacted varies directly proportional to the individual volume of reactant reacted.

Thus mole ratio = volume ratio = $1:2$

Therefore, $\frac{\text{Volume of } H_2O_2 \text{ reacted}}{\text{Volume of } Na_2S_2O_3} = \frac{1}{2}$ or Volume of H_2O_2 reacted $= \frac{1}{2} \times$ Volume of $Na_2S_2O_3$

So for each of $\mathbf{2.5cm^3}$ of $Na_2S_2O_3$ added, volume of H_2O_2 reacted, $x = \frac{1}{2} \times \mathbf{2.5cm^3} = \mathbf{1.25cm^3}$

For example, when $2.5cm^3$ of AC was added from the burette for first time, $1.25cm^3$ of AB reacted, and thus, $a - x = 10cm^3 - 1.25cm^3 = \mathbf{8.75cm^3}$.

Making Sense of the Experimental Data

Practical tip: *Most chemical reactions involving hydrogen peroxide as a reactant, are first order with respect to the peroxide.*

For first order reaction; $\log(a - x) = \frac{-kt}{2.303} + \log a$

If concentration change from a to x_1 after t_1 and from a to x_2 after t_2, the above equation becomes;

$\log(a - x_1) = \frac{-kt_1}{2.303} + \log a$…………………(i)

$\log(a - x_2) = \frac{-kt_2}{2.303} + \log a$…………………(ii)

Taking (i) – (ii); $\log(a - x_1) - \log(a - x_2) = \frac{kt_2}{2.303} - \frac{kt_1}{2.303}$

From which; $\log\left(\frac{a-x_1}{a-x_2}\right) = \frac{k}{2.303}(t_2 - t_1)$ or $t_2 = \frac{2.303}{k}\log\left(\frac{a-x_1}{a-x_2}\right) + t_1$

But since number of moles (or concentration) reacted varies directly proportional to the individual volume of reactant reacted; $\frac{a-x_1}{a-x_2} = \frac{V_1}{V_2}$.

Then the above formula becomes, $\mathbf{t_2 = \frac{2.303}{k} \log\left(\frac{V_1}{V_2}\right) + t_1}$

Now, we can use the last formula to calculate time corresponding to each volume if the value of rate constant, k is known.

The value of k can be found by using any two pairs of values. So we may take the first two pairs as follows:

$$V_1 = 8.75\text{cm}^3, t_1 = 140s, V_2 = 7.5\text{cm}^3, t_2 = 300s$$

Substituting $300\text{s} = \frac{2.303}{k}\log\left(\frac{8.75}{7.5}\right) + 140\text{s}\,; k = 9.6361 \times 10^{-4}\text{s}^{-1}$

Thus the modified formula for calculating any time, t_2, becomes;

$$\mathbf{t_2 = \frac{2.303}{9.6361 \times 10^{-4}s^{-1}} \log\left(\frac{8.75cm^3}{V_2}\right) + 300s}$$

When:

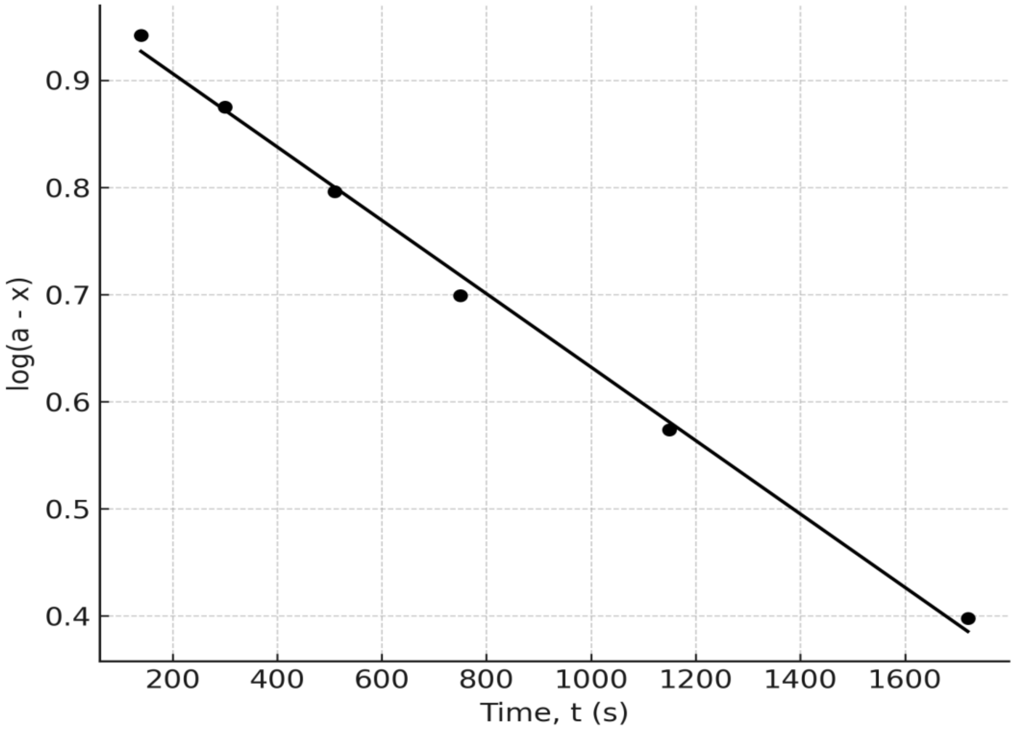
$V_2 = 6.25\text{cm}^3$; $t_2 = \frac{2.303}{9.6361\times10^{-4}\text{s}^{-1}}\log\left(\frac{8.75\text{cm}^3}{6.25\text{cm}^3}\right) + 300\text{s} = 649\text{s}$ (experimental data = 646s)

$V_2 = 5\text{cm}^3$; $t_2 = \frac{2.303}{9.6361\times10^{-4}\text{s}^{-1}}\log\left(\frac{8.75\text{cm}^3}{5\text{cm}^3}\right) + 300\text{s} = 881\text{s}$ (experimental data = 877s)

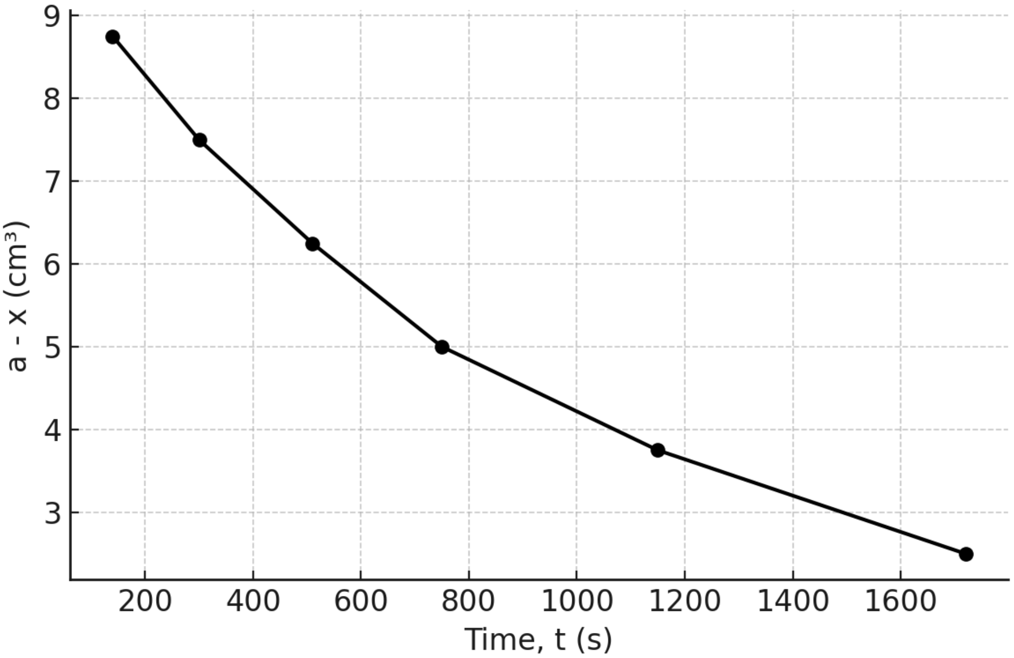
$V_2 = 3.75\text{cm}^3$; $t_2 = \frac{2.303}{9.6361\times10^{-4}\text{s}^{-1}}\log\left(\frac{8.75\text{cm}^3}{3.75\text{cm}^3}\right) + 300\text{s} = 1179\text{s}$ (experimental data = 1175s)

$V_2 = 2.5\text{cm}^3$; $t_2 = \frac{2.303}{9.6361\times10^{-4}\text{s}^{-1}}\log\left(\frac{8.75\text{cm}^3}{2.5\text{cm}^3}\right) + 300\text{s} = 1600\text{s}$ (experimental data = 1594s)

(a) **Graph of log(a - x) vs Time (t)**



(b) **Graph of (a - x) vs Time (t)**



(c) The order of the reaction with respect to hydrogen peroxide is first order because the graph of log (a – x) against time, t is a straight line with negative slope.

(d) Adding the same fixed volume of sodium sulphate ensures that the amount of reactant available for each time interval is constant. This consistency is crucial because it allows for a fair comparison of the time taken to produce the same amount of iodine in each cycle. Any change in the time recorded must therefore reflect a change in the rate, not a change in quantity of reactant.

(e) While iodine is visible as a brown solution, its colour may be too faint to detect low concentrations accurately. Starch acts as a sensitive indicator, forming a deep blue-black complex with even small amounts of iodine. This makes it easier to detect the exact moment when free iodine appears (just after equivalence point), improving accuracy and precision in time recording.

Experiment 32

Kipute had always been fascinated by how **temperature** affects the speed of reactions. One afternoon in the school laboratory, she decided to test a reaction that could actually be "seen happening" — the reaction between **sodium thiosulphate** and **hydrochloric acid**, which produces a fine **sulphur precipitate** that turns the solution cloudy according to the following equation:

$$S_2O_3^{2-}(aq) + 2H^+(aq) \longrightarrow SO_2(g) + S(g) + H_2O(l)$$

"It's like watching fog build up in a clear sky," she said as she placed a beaker on a piece of paper marked **X**. The time it took for the mark to disappear would tell her how fast the reaction occurred and how temperature changed that speed.

Her goal? To **calculate the activation energy** for the reaction, a critical factor in chemical kinetics — the kind of concept used in **industrial processes**, such as making photographic films or controlling pollution where sulphur compounds are involved.

Turbidity timing procedure Kipute followed

She gathered the following reagents and apparatus:

- **L**: 0.5 M sodium thiosulphate solution
- **M**: 0.1 M hydrochloric acid
- **Stopwatch**, **thermometer (0–100°C)**, **50 mL beaker**, **white paper with "X"**, **250 mL beaker (for water bath)**

Step-by-Step Procedure

1. Placed a **100 mL beaker** on a white paper marked with an **X**, so the mark was visible through the clear solution.
2. Poured about **200 cm³ of distilled water** into a 250 mL beaker to serve as a **water bath**.
3. Measured **10 cm³** of **solution L (sodium thiosulphate)** into one test tube and **10 cm³** of **solution M (hydrochloric acid)** into another.
4. Placed both test tubes into the water bath and allowed them to **warm to 40°C** (monitored with the thermometer).
5. Once they reached the desired temperature, quickly poured both solutions into the small beaker over the mark "X" and started the stopwatch.
6. Swirled the mixture and **recorded the time taken** for the **"X" mark to completely disappear** due to cloudiness from precipitated sulphur.
7. Repeated steps 3 to 6 for the temperatures: **40°C, 50°C, 60°C, 70°C, and 80°C**.
8. Recorded and analyzed her data as follows:

Temperature (T)		Time, t(sec)	$\left(\frac{1}{T}\right) K^{-1}$	$\left(\frac{1}{t}\right) sec^{-1}$	$\log\left(\frac{1}{t}\right)$
T(°C)	T(K)				
40	313				
50	323				
60	333				
70	343				
80	353				

Questions

a) Plot the graph of $\log\left(\frac{1}{t}\right) s^{-1}$ against $\left(\frac{1}{T}\right) K^{-1}$

b) Plot the graph of $\log t(s)$ against $\left(\frac{1}{T}\right) K^{-1}$

c) If $\log t = \frac{Ea}{2.303R}\left(\frac{1}{T}\right) - \log A$ and $\log\left(\frac{1}{t}\right) = \frac{-Ea}{2.303R}\left(\frac{1}{T}\right) + \log A$

Calculate the activation energy using both of the above formula and comment whether the values are similar or not.

d) Explain why equal volumes of sodium thiosulphate and hydrochloric acid was used in each trial?

e) List down at least two industrial or environmental processes that could be modelled using this experiment.

Solution

TABLE OF RESULTS

Temperature (T)		Time, t(sec)	$\left(\frac{1}{T}\right)K^{-1}$	$\left(\frac{1}{t}\right)\sec^{-1}$	$\log\left(\frac{1}{t}\right)$
$T(°C)$	$T(K)$				
40	313	61	3.19×10^{-3}	0.02	-1.78
50	323	38	3.10×10^{-3}	0.03	-1.50
60	333	20	3.00×10^{-3}	0.05	-1.30
70	343	12	2.92×10^{-3}	0.08	-1.10
80	353	6	2.83×10^{-3}	0.17	-0.78

Making Sense of the Experimental Data

Practical Tip: *For a given chemical reaction within a moderate temperature range, the temperature coefficient, Q_{10} (the factor by which the reaction rate increases with a 10°C rise in temperature) remains fairly consistent.*

$$Q_{10} = \frac{\text{Rate at }(T+10)°C}{\text{Rate at }T°C} = \frac{\text{Time taken for the cross to disappear at }T°C}{\text{Time taken for the cross to disappear at }(T+10)°C}$$

For this experiment:

If $T°C = 40$, then $(T + 10)°C = 50$ and their respective time are 61sec and 38sec.

Thus $Q_{10} = \frac{61\text{sec}}{38\text{sec}} = 1.61$

Then $t_3 = \frac{t_2}{1.61} = \frac{38\text{sec}}{1.61} = 24\text{sec}$ (experimental data $= 20$sec)

$$t_4 = \frac{t_3}{1.61} = \frac{24\text{sec}}{1.61} = 15\text{sec} \text{ (experimental data} = 12\text{sec)}$$

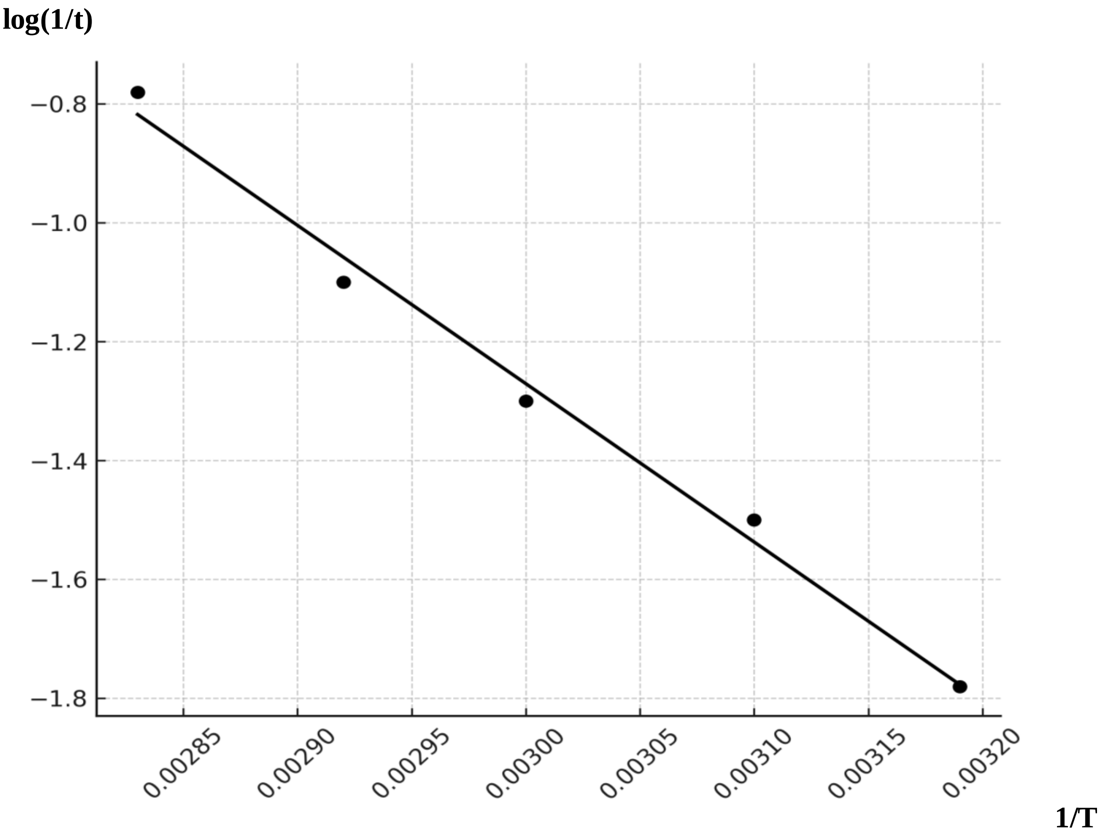
$$t_5 = \frac{t_4}{1.61} = \frac{12\text{sec}}{1.61} = 15\text{sec} \text{ (experimental data} = 7\text{sec)}$$

Conclusion: *The actual reaction proceeded slightly faster than the rate predicted by the temperature coefficient. This can be attributed to the fact that, as temperature increases, the reaction rate increases exponentially, rather than uniformly — as described by the Arrhenius equation.*

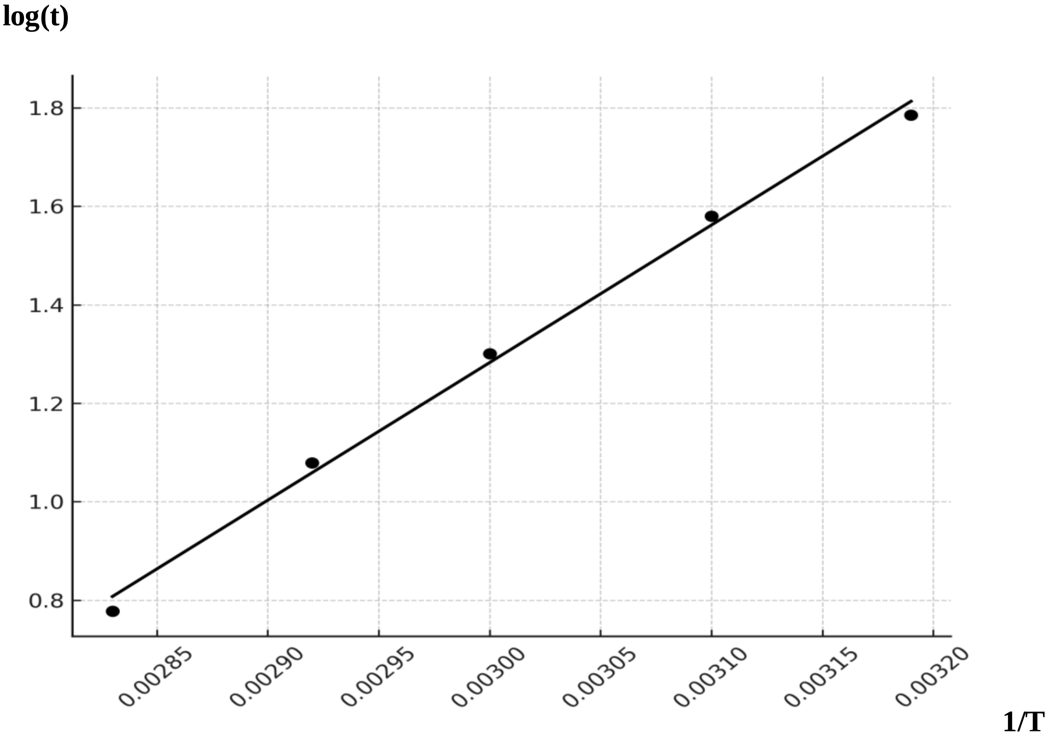
Nevertheless, the close agreement between the predicted values and the experimentally recorded times indicates that the Q_{10} value remained relatively consistent throughout the experiment, as the theory suggests. This consistency supports the validity of the experimental approach and confirms that the practical was successfully conducted.

Response to Questions

(a) **Graph: log(1/t) vs 1/T**



(b) **Graph: log(t) vs 1/T**



(c) From $\log\left(\frac{1}{t}\right) = \frac{-Ea}{2.303R}\left(\frac{1}{T}\right) + \log A$ (used to plot graph in (a))

The equation is in the form of $y = mx + c$ with $y = \log\left(\frac{1}{t}\right)$ and $x = 1/T$

From which; the slope of the graph, $m = \frac{-E_a}{2.303R}$

Thus $E_a = -2.303mR$ where $R = 8.314Jmol^{-1}K^{-1}$

Using any two points from the graph, the calculated slope is as follows:

$$m = -2662.34$$

So $E_a = -2.303mR = -2.303 \times -2662.34 \times 8.314Jmol^{-1} = 50976Jmol^{-1} = 50.976kJmol^{-1}$

The activation energy by using the graph in (a) is $\mathbf{50.976kJmol^{-1}}$.

Also from $\log t = \frac{Ea}{2.303R}\left(\frac{1}{T}\right) - \log A$ (used to plot graph in (b))

The equation is in the form of $y = mx + c$ with $y = \log(t)$ and $x = 1/T$

From which; the slope of the graph, $m = \frac{E_a}{2.303R}$

Thus $E_a = 2.303mR$ where $R = 8.314Jmol^{-1}K^{-1}$

Using any two points from the graph, the calculated slope is as follows:

$$m = 2793.74$$

So $E_a = 2.303mR = 2.303 \times 2793.74 \times 8.314Jmol^{-1} = 53492Jmol^{-1} = 53.492kJmol^{-1}$

The activation energy by using the graph in (b) is $\mathbf{53.492kJmol^{-1}}$.

Comment: The two values of activation energy are similar within the limits of experimental error**.**

(d) **Reason:**

To ensure that the change the change in the reaction rate is accounted by temperature change only.

Explanation:

To ensure a fair comparison between trials, the total amount of reacting substances must be constant. If the volume of either reactant changes, it would alter the number of moles available, which directly affects the rate of reaction. By keeping the volumes constant, the only variable affecting the rate becomes temperature, allowing a clear relationship to be observed.

(e) This experiment models how temperature affects reaction rates, applicable in:

1) **Air pollution control** where sulphur compounds are converted or removed.
2) **Industrial chemical synthesis** where precise temperature control is needed to optimize speed and cost.
3) **Food processing or sterilization** where temperature must be optimized to trigger or halt chemical changes.

Experiment 33

On a quiet Friday morning, Mr. Akilikubwa stood by the school laboratory's window, watching the morning mist rise. He wasn't just a lab technician; he was a detective of molecules. That day, he was intrigued by a special reaction, one that changed colour before your eyes: the **reaction between potassium permanganate and oxalic acid**.

The purple permanganate was known for its **intense hue**, but in the presence of oxalic acid and heat, it vanished; either brown colour precipitate at the bottom of the experimental container or the reaction colour may change directly from purple to colourless without passing to brown colour, depending on how fast the reaction took place.

He recorded the time taken for disappearance of purple colour of potassium permanganate after reaction with oxalic acid. However, even if the brown colour appear he correctly stopped the stopwatch at this point and record the time taken without waiting for appearance of colourless.

"Understanding this rate," he thought, *"can help us model processes like* ***industrial bleaching****,* ***wastewater treatment****, or* ***oxidation in biological systems****."*

The colour-change kinetics procedure Mr. Akilikubwa followed

He carefully assembled the required reagents and tools:

- **AA**: Potassium permanganate solution (3.16 g/dm³)
- **BB**: 0.05 M oxalic acid in 0.5 M sulphuric acid
- Thermometer (0°C – 100°C)
- Water bath
- Stopwatch
- Measuring cylinders
- Boiling tubes
- A small beaker

Step-by-Step colour-change investigation

1. Using a **measuring cylinder**, he measured **10 cm³ of AA** into a boiling tube.
2. In another boiling tube, he measured **10 cm³ of BB**.
3. He placed both tubes into a **hot water bath** and warmed the solutions to approximately **50°C**.
4. Once the temperature stabilized, he quickly **poured both solutions into a small beaker** and **started his stopwatch**.
5. He watched carefully: sometimes the solution turned **brown**, sometimes it went directly from **purple to colourless**.
6. He **stopped the timer the moment the purple colour of AA disappeared** completely.
7. He repeated the procedure at higher temperatures: **60°C, 70°C, 80°C, and 90°C**.
8. He recorded results in a tabular form as shown below

Experiment	Temperature, T		$\left(\frac{1}{T}\right) K^{-1}$	Time, t(sec)	$\left(\frac{1}{t}\right) sec^{-1}$	$\log t$	$\log\left(\frac{1}{t}\right)$
	(T)°C	(T)K					
1.	50						
2.	60						
3.	70						
4.	80						

Questions

a) Plot the graph of $\log t$ against $\left(\frac{1}{T}\right)$

b) Given the Arrhenius equation $k = Ae^{-Ea/RT}$

Where:

k = rate constant

A = Arrhenius constant (frequency factor)

Ea = activation energy

R = universal gas constant

T = absolute temperature.

Calculate the activation energy, E_a.

c) Why does the colour sometimes change directly from purple to colourless, while at other times it passes through a brown stage?

d) It is not advisable for this experiment, the temperature to exceed 100°C. Explain.

Solution

TABLE OF RESULTS

Exp.	Temperature, T		$\left(\frac{1}{T}\right)K^{-1}$ $(\times 10^{-3})$	Time, t(sec)	$\left(\frac{1}{t}\right)\sec^{-1}$ $(\times 10^{-3})$	$\log t$	$\log\left(\frac{1}{t}\right)$
	(T)°C	(T)K					
1.	50	323	3.096	311	3.215	2.493	−2.493
2.	60	333	3.003	204	4.902	2.400	−2.400
3.	70	343	2.915	130	7.692	2.114	−2.114
4.	80	353	2.833	80	12.5	1.903	−1.903

Making Sense of the Experimental Data

Like in the previous experiment;

$$Q_{10} = \frac{\text{Rate at } (T+10)°C}{\text{Rate at } T°C} = \frac{\text{Time taken for the cross to disappear at } T°C}{\text{Time taken for the cross to disappear at } (T+10)°C}$$

For this experiment:

If $T°C = 50$, then $(T+10)°C = 60$ and their respective time are 311sec and 204sec.

Thus $Q_{10} = \frac{311\text{sec}}{204\text{sec}} = 1.52$

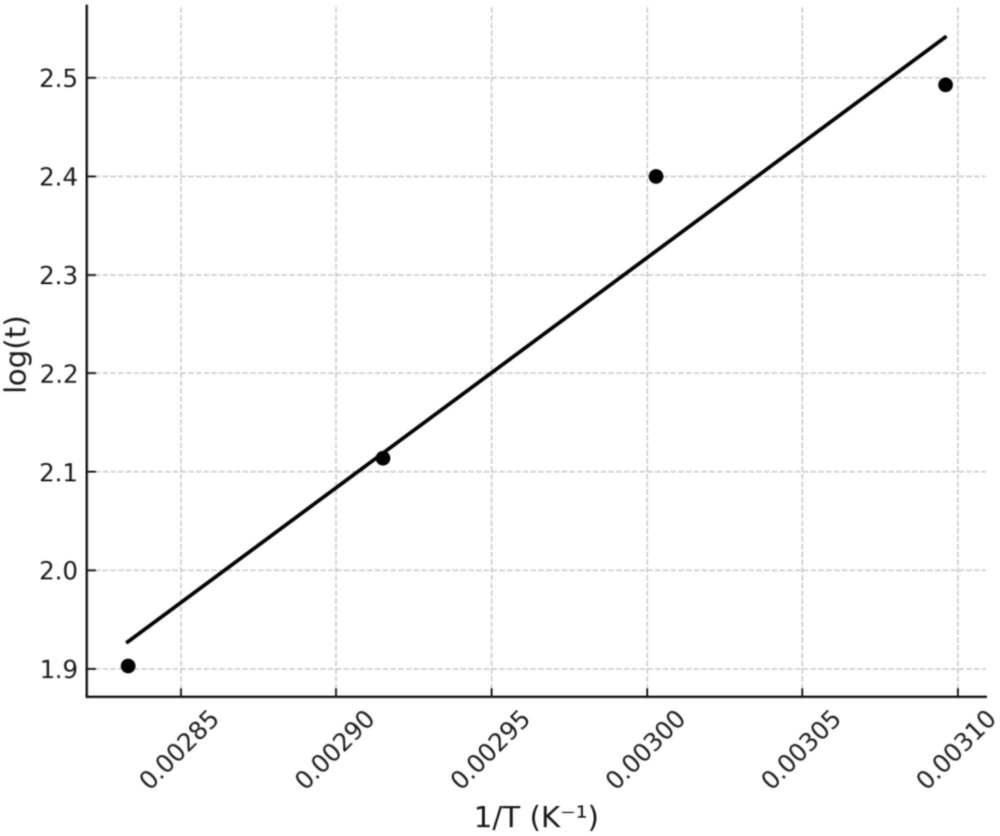
Then $t_3 = \frac{t_2}{1.52} = \frac{204\text{sec}}{1.52} = 134\text{sec}$ (experimental data = 130sec)

$$t_4 = \frac{t_3}{1.52} = \frac{134\text{sec}}{1.52} = 88\text{sec} \text{ (experimental data} = 80\text{sec)}$$

Conclusion: *The close agreement between the predicted values and the experimentally recorded times indicates that the Q_{10} value remained relatively consistent throughout the experiment, as the theory suggests. This consistency supports the validity of the experimental approach and confirms that the practical was successfully conducted.*

Response to Questions

(a) **Graph: log(t) vs 1/T**



(b) From the Arrhenius equation: $k = Ae^{-E_a/RT}$

Introducing natural logarithm both sides: $\ln k = \ln Ae^{-E_a/RT}$

$\ln k = \ln A + \ln e^{-E_a/RT}$;

$$\ln k = \ln A + \frac{-E_a}{RT}\ln e$$

But $\ln e = 1$

$$\text{Then } \ln k = \ln A + \frac{-E_a}{RT}$$

Changing natural logarithms to common logarithm:

Using $\ln x = 2.303\log x$

Then the above equation become $2.303\log k = \frac{-E_a}{RT} + 2.303\log A$

Hence, $\log k = \frac{-E_a}{2.303RT} + \log A$

But the rate constant, k varies inversely to time required for given amount of reactant to react, t

So $\log\left(\frac{1}{t}\right) = \frac{-Ea}{2.303R}\left(\frac{1}{T}\right) + \log A$ or $\log t^{-1} = \frac{-Ea}{2.303R}\left(\frac{1}{T}\right) + \log A$ or $-\log t = \frac{-Ea}{2.303R}\left(\frac{1}{T}\right) + \log A$

From which; $\log t = \frac{Ea}{2.303R}\left(\frac{1}{T}\right) - \log A$

The final equation corresponds to the plotted graph in (a) with:

$y = mx + c$, where $y = \log(t)$ and $x = 1/T$

And the slope of the graph, $m = \frac{E_a}{2.303R}$

Thus $E_a = 2.303mR$ where $R = 8.314Jmol^{-1}K^{-1}$

Using any two points from the graph, the calculated slope is as follows:

$$m = 2339$$

So $E_a = 2.303mR = 2.303 \times 2339 \times 8.314Jmol^{-1} = 44785Jmol^{-1} = 44.785kJmol^{-1}$

The activation energy of the reaction is $44.785kJmol^{-1}$.

(c) The brown colour indicates MnO_2 precipitate which is an intermediate in the reduction of MnO_4^- to Mn^{2+}. At lower temperatures, the reaction proceeds slower, allowing intermediate MnO_2 to accumulate visibly. At higher temperatures, the reaction proceeds faster, and MnO_2 is rapidly reduced to Mn^{2+}, causing the solution to go directly from purple to colourless.

(d) Exceeding 100°C may cause the solutions to boil, leading to loss of volume due to evaporation, and unsafe conditions due to splashing. More importantly, the reaction could occur too rapidly to time accurately, leading to loss of precision in measuring the rate.

Chapter 6

THERMOCHEMISTRY

INTRODUCTION

Thermochemistry is the branch of physical chemistry which deals with the study of measurement of heat change in various chemical reactions. In the chemical reactions heat may be either evolved to the surroundings in exothermic reactions or absorbed from the surroundings in endothermic reactions.

So, in thermochemistry experiments, we are determining amount of heat evolved (it is indicated by negative sign) or amount of heat absorbed (it is indicated by positive sign) in chemical reactions. This is simply done by measuring temperature (s), before and after reaction.

One type of experiment is one in which substances are mixed in an insulated container and the temperature change is measured. This could be a solid dissolving or reacting in a solution or it could be two solutions reacting together. We can use these kind of experiments to determine several heat changes including:

- ✓ Heat of reactions
- ✓ Heat of neutralisation
- ✓ Heat of solution

EXPERIMENTS

Experiment 34

Kipute stood in the school laboratory with a puzzled look on her face, clutching a thermometer like a sword. Today's mission was quite different—it wasn't about bubbling purple liquids or vanishing crosses. No, today she had to deal with something far sneakier: **hidden energy changes**!

Mr. Akilikubwa had left a mysterious note on the lab bench that read:

> *"Dear Kipute, in your hands lies the power to reveal what your eyes cannot see—**enthalpy change**! Use the materials wisely, and record everything. The Ever-Curious Akilikubwa"*

Kipute grinned. It was time to solve another thermal puzzle.

She was provided with the following:

- **AB**: 2g of anhydrous copper(II) sulphate.
- **AC**: 4g of hydrated copper(II) sulphate.
- **AD**: Distilled water.
- A thermometer, a 250 cm³ beaker, and a measuring cylinder.

Kipute's thermochemical procedure

1. She measured 50 cm³ of distilled water and poured it into a clean dry beaker, recording its temperature.
2. Then she added the **dry AB sample**, stirred with the thermometer, and noted the **highest or lowest temperature** it reached.
3. She repeated the same with **AC**.
4. She recorded the results in a tabular form as follows:

Experiment	Volume of water (cm^3)	Mass of salt (g)	Initial temp. (°C)	Final temp. (°C)	Change in temp. (°C)	Molecular mass of salt (g)
$CuSO_4$						
$CuSO_4.5H_2O$						

Questions

a) What is the specific term used to describe the **hidden enthalpy change** determined in this experiment?

b) Calculate the enthalpy of each experiment.
Given that; specific heat capacity of water = 4.2J/g°C and density of water = 1g/cm^3.

c) The accepted values for the two experiments are:
$\Delta H(CuSO_4) = -66.1kJ/mol$ and $\Delta H(CuSO_4.5H_2O) = +11.3KJ/mol$.
How do your experimental values compare with the accepted values? Explain your answer.

d) Why it is more acceptable to use a plastic beaker instead of a glass beaker for this type of experiment?

Solution

TABLE RESULTS

Experiment	Volume of water (cm^3)	Mass of salt (g)	Initial temp. (°C)	Final temp. (°C)	Change in temp. (°C)	Molecular mass of salt (g)
$CuSO_4$	50	2	16	18	+2	159.5
$CuSO_4.5H_2O$	50	4	16	15.5	− 0.5	249.5

Making Sense of the Experimental Data

Using enthalpy of solution, $\Delta H_{soln} = \frac{\text{Total enthalpy change, } \Delta H}{\text{Number of moles of solute, } n_{su}}$;

From which; $\Delta H = \Delta H_{soln} \times n_{su}$

For $CuSO_4$:

Standard $\Delta H_{soln} = -66.1kJ/mol = -66100Jmol^{-1}$and $n_{su} = \frac{2g}{159.5g/mol} = \frac{2}{159.5}mol$

Then $\Delta H = -66100Jmol^{-1} \times \frac{2}{159.5}mol = -828.84J$

But also $\Delta H = -m_{soln}C_{soln}\Delta T = -\rho_{soln}V_{soln}C_{soln}\Delta T$

You have to understand this: *Negative sign is included in the above formula because the sign the enthalpy (heat) change is always opposite to the sign of temperature change. Negative temperature change (decrease in temperature) implies that heat is absorbed from surrounding as result of endothermic process (positive heat change) while positive temperature change (increase in temperature) implies that heat is evolved to surrounding as result of exothermic process (negative heat change).*

Substituting $-828.84J = -1gcm^{-3} \times 50cm^3 \times 4.2Jg^{-1}°C^{-1} \times \Delta T$

From which; $\Delta T = +\mathbf{3.9}°C$ (experimental data $= +\mathbf{2}°C$).

For $\mathbf{CuSO_4.5H_2O}$:

Standard $\Delta H_{soln} = +11.3kJ/mol = +11300Jmol^{-1}$and $n_{su} = \frac{4g}{249.5g/mol} = \frac{4}{249.5}mol$

Then $\Delta H = +11300Jmol^{-1} \times \frac{4}{249.5}mol = +181.16J$

But also $\Delta H = -m_{soln}C_{soln}\Delta T = -\rho_{soln}V_{soln}C_{soln}\Delta T$

Substituting $+181.16J = -1gcm^{-3} \times 50cm^3 \times 4.2Jg^{-1}°C^{-1} \times \Delta T$

From which; $\Delta T = \mathbf{-0.9}°C$ (experimental data $= \mathbf{-0.5}°C$).

***Conclusion:** In both cases, the experimental results do not deviate significantly from the calculated values, which suggests that the results are plausible.*

However, slight differences arise due to the following factors:

1. **Concentration Factor:**

   The temperature change in the experiment was compared with that calculated from the standard enthalpy of solution, which is defined under the condition of a **very dilute solution**. This condition may not have been strictly maintained during the experiment. A highly dilute solution is essential to eliminate additional heat changes from solute-solute and solute-solvent interactions. If the solution is not sufficiently dilute, the dissolution process may be incomplete. As a result:

   - In **exothermic dissolutions**, less heat may be evolved, leading to a **smaller rise in temperature**.
   - In **endothermic dissolutions**, less heat may be absorbed, resulting in a **smaller temperature drop**.

2. **Heat transfer factor:**

   Standard enthalpy values assume **no heat exchange with the surroundings**. In reality:

   - For **exothermic processes**, some heat is inevitably lost to the surroundings, reducing the observed temperature rise.
   - For **endothermic processes**, the system may absorb heat from the surroundings, resulting in a smaller recorded temperature decrease.

3. **Conditions factor:**

   Standard enthalpy changes are measured under **standard conditions** (298K and 1atm pressure). Since the experiment was carried out under **non-standard laboratory conditions**, the enthalpy change (and hence the temperature change) observed may not exactly match the standard value due to variations in temperature and pressure.

Response to Questions

(a) The specific term is **enthalpy of solution**.

(b) Using enthalpy of solution, $\Delta H_{soln} = \frac{\text{Total enthalpy change, } \Delta H}{\text{Number of moles of solute, } n_{su}}$;

Where; $\Delta H = -m_{soln}C_{soln}\Delta T = -\rho_{soln}V_{soln}C_{soln}\Delta T$ and $n_{su} = \frac{m_{su}}{M_{su}}$

For $CuSO_4$:

$$\Delta H_{soln} = \frac{-\rho_{soln}V_{soln}C_{soln}\Delta T \times M_{su}}{m_{su}} = \frac{-1gcm^{-3} \times 50cm^3 \times 4.2Jg^{-1}°C^{-1} \times 2°C \times 159.5gmol^{-1}}{2g}$$

$$= -33495Jmol^{-1} = -33.495kJmol^{-1}$$

The enthalpy of solution of $CuSO_4$ is $-33.5kJmol^{-1}$.

For $CuSO_4.5H_2O$:

$$\Delta H_{soln} = \frac{-\rho_{soln}V_{soln}C_{soln}\Delta T \times M_{su}}{m_{su}} = \frac{-1gcm^{-3} \times 50cm^3 \times 4.2Jg^{-1}°C^{-1} \times -0.5°C \times 249.5gmol^{-1}}{4g}$$

$$= +6549Jmol^{-1} = +6.549kJmol^{-1}$$

The enthalpy of solution of $CuSO_4.5H_2O$ is $+6.5kJmol^{-1}$.

(c) In both cases, the magnitude of experimental values is smaller than accepted standard values due to the following reasons:
  - Incomplete dissolution as the solutions are not diluted to infinite as done in the determination of the accepted values.
  - Heat loss to surroundings (in dissolution of $CuSO_4$) or heat gain from surroundings (in dissolution of $CuSO_4.5H_2O$).
  - Non-standard experimental conditions (accepted values are determined under standard conditions of 25°C and 1atm).

(d) Plastic has better insulation properties than glass, which means that it minimizes heat loss to the surroundings and therefore improves the accuracy of temperature readings and the calculated enthalpy change.

Experiment 35

It was a slow, sunny afternoon at Kipute's school. The class had just finished a long theory session, and everyone looked as though they'd melted into their lab coats.

Just as the bell rang, Kipute found a mysterious note slipped inside her lab manual:

*"Detective Kipute, we suspect some common salts have been hiding thermal secrets. Your mission is to discover whether they **heat things up** or **chill them out** when dissolving.*

Lab Technician, Akilikubwa.*"*

Kipute's eyes lit up. *"A thermal mystery? Count me in!"* she whispered.

She was handed three suspects with 2g for each:

- **J1**: Sodium oxalate ($Na_2C_2O_4$) – the quiet one with a shady past in organic circles.
- **J2**: Sodium hydroxide (NaOH) – known to be reactive and a little too ***hot-headed***.
- **J3**: Sodium chloride (NaCl) – classic, always in the middle of every kitchen brawl.

Equipped with:

- **A thermometer**,
- **A 250 cm^3 beaker**,
- **Distilled water**, and
- **A measuring cylinder**,

Kipute stepped into her role as the *thermochemical detective*.

Kipute's procedure for extracting the truth

1. She measured **50 cm^3 of distilled water** into a clean 250 cm^3 beaker by using a measuring cylinder.
2. She checked the **initial temperature** with her trusty thermometer.
3. Then, she added **2 g of one of the salty suspects** (J1, J2, or J3).
4. She carefully stirred and recorded the **maximum or minimum temperature** reached.
5. She repeated the entire procedure for each of the other salts, keeping her lab coat as white as her record sheet.
6. She recorded the results in tabular form.

Questions

a) State whether each salt ***heats things up*** or ***chills things out*** during the dissolving process.
b) Calculate the heat change of the solution for each of the salts.
c) Calculate the molar enthalpy for each of the salts.
d) In the introduction of this experiment, **J2** (NaOH) was portrayed as '***hot-headed.***' Justify this characterization using the experimental results.
e) In real-life applications, why might an exothermic dissolving substances like NaOH be used with caution in industrial or household settings?

Solution

Temperature of water = T_1

Temperature change, $\Delta T = T_2 - T_1$

TABLE OF RESULTS

SALT	Initial temperature, T_1 (°C)	Final temperature T_2 (°C)	Temperature change,ΔT(°C)
J1	23	21.5	-1
J2	23	26	$+6.5$
J3	23	22	-0.4

Making Sense of the Experimental Data

Using enthalpy of solution, $\Delta H_{soln} = \frac{\text{Total enthalpy change, } \Delta H}{\text{Number of moles of solute, } n_{su}}$;

From which; $\Delta H = \Delta H_{soln} \times n_{su}$

For $Na_2C_2O_4$:

Standard $\Delta H_{soln} = +16.6kJ/mol = +16600Jmol^{-1}$and $n_{su} = \frac{2g}{134g/mol} = \frac{2}{134}mol$

Then $\Delta H = +16600Jmol^{-1} \times \frac{2}{134}mol = +247.76J$

But also $\Delta H = -m_{soln}C_{soln}\Delta T = -\rho_{soln}V_{soln}C_{soln}\Delta T$

Substituting $+247.76J = -1gcm^{-3} \times 50cm^3 \times 4.2Jg^{-1}°C^{-1} \times \Delta T$

From which; $\Delta T = \mathbf{-1.2}$°C (experimental data $= \mathbf{-1}$°C).

For NaOH:

Standard $\Delta H_{soln} = -44.5kJmol^{-1} = -44500Jmol^{-1}$and $n_{su} = \frac{2g}{40g/mol} = \frac{2}{40}mol$

Then $\Delta H = -44500Jmol^{-1} \times \frac{2}{40}mol = -2225J$

But also $\Delta H = -m_{soln}C_{soln}\Delta T = -\rho_{soln}V_{soln}C_{soln}\Delta T$

Substituting $-2225J = -1gcm^{-3} \times 50cm^3 \times 4.2Jg^{-1}{}^\circ C^{-1} \times \Delta T$

From which; $\Delta T = \mathbf{+10.6}$°C (experimental data $= \mathbf{+6.5}$°C).

For NaCl:

Standard $\Delta H_{soln} = +3.9kJmol^{-1} = +3900Jmol^{-1}$and $n_{su} = \frac{2g}{58.5g/mol} = \frac{2}{58.5}mol$

Then $\Delta H = +3900Jmol^{-1} \times \frac{2}{58.5}mol = +133.33J$

But also $\Delta H = -m_{soln}C_{soln}\Delta T = -\rho_{soln}V_{soln}C_{soln}\Delta T$

Substituting $= +133.33J = -1gcm^{-3} \times 50cm^3 \times 4.2Jg^{-1}{}^\circ C^{-1} \times \Delta T$

From which; $\Delta T = \mathbf{-0.6}$°C (experimental data $= \mathbf{-0.4}$°C).

Conclusion: *In all cases, the experimental results are plausible and hence the experiment was successful.*

Response to Questions

(a) **J2** heat things up while **J1** and **J3** chills things out.

(b) Using $\Delta H = -m_{soln}C_{soln}\Delta T = -\rho_{soln}V_{soln}C_{soln}\Delta T$

For $Na_2C_2O_4$:

$$\Delta H = -1gcm^{-3} \times 50cm^3 \times 4.2Jg^{-1}{}^\circ C^{-1} \times -1^\circ C = +210J = +0.21kJ$$

The enthalpy change for $Na_2C_2O_4$ is $+210J$ or $+0.21kJ$.

For NaOH:

$$\Delta H = -1gcm^{-3} \times 50cm^3 \times 4.2Jg^{-1}{}^\circ C^{-1} \times 6.5^\circ C = -1365J = -1.365kJ$$

The enthalpy change for NaOH is $-1.365kJ$.

For NaCl:

$$\Delta H = -1gcm^{-3} \times 50cm^3 \times 4.2Jg^{-1}{}^\circ C^{-1} \times -0.4^\circ C = +84J$$

The enthalpy change for NaCl is $+84J$.

(c) Using molar enthalpy of solution, $\Delta H_{soln} = \frac{\text{Total enthalpy change, } \Delta H}{\text{Number of moles of solute, } n_{su}}$;

Where; $n_{su} = \frac{m_{su}}{M_{su}}$

For $Na_2C_2O_4$:

$$\Delta H_{soln} = \frac{\Delta H \times M_{su}}{m_{su}} = \frac{+0.21kJ \times 134gmol^{-1}}{2g} = +14.07kJmol^{-1}$$

The molar enthalpy of solution of $Na_2C_2O_4$ is $+14.07kJmol^{-1}$.

For NaOH:

$$\Delta H_{soln} = \frac{\Delta H \times M_{su}}{m_{su}} = \frac{-1.365kJ \times 40gmol^{-1}}{2g} = -27.3kJmol^{-1}$$

The molar enthalpy of solution of NaOH is $-27.3kJmol^{-1}$

For NaCl:

$$\Delta H_{soln} = \frac{\Delta H \times M_{su}}{m_{su}} = \frac{+84J \times 58.5gmol^{-1}}{2g} = +2457Jmol^{-1} = +2.457kJmol^{-1}$$

The molar enthalpy of solution of NaCl is $+2.457kJmol^{-1}$

(d) NaOH was portrayed as hot-headed because it **released the most heat upon dissolving in water, causing a rapid rise in temperature** which is a clear sign of its exothermic and energetically reactive nature

(e) Exothermic dissolving substances releases a significant amount of heat when dissolved in water. If added too quickly or in large quantities, the temperature can rise rapidly, potentially causing:

- Boiling and splashing of the solution,
- Cracking of glassware due to sudden heating,
- Chemical burns on skin or eyes.

Experiment 36

"Have you ever wondered," Mr. Akilikubwa asked as Kipute walked into the lab, *"why indigestion tablets fizz in your mouth and metals corrode slowly in acid rain?"*

Kipute, always curious, replied, *"Isn't that all about chemical reactions?"*

"Exactly. And today," he said, handing her a thermometer, *"you'll measure the* actual heat *involved when acids meet magnesium carbonate and magnesium ribbon."*

The lab was quiet, only the sound of swirling solutions and soft bubbling filled the air. Kipute methodically worked with **hydrochloric acid**, **magnesium carbonate**, and **magnesium ribbon**—tracking temperature changes and calculating the energy stored or released in these reactions and **hidden** enthalpy of formation of $MgCO_3$.

By the end, she had not only gathered data but learned that every quiet fizz or gentle temperature shift told a deeper story about matter and energy.

She was handed the following materials:

- **A**: 1M Hydrochloric acid
- **B**: 1g Magnesium carbonate
- **C**: 0.2g Magnesium ribbon
- Thermometer, 100 cm^3 beaker or conical flask, distilled water.

Her procedure was as follows:

Procedure A

1. Measured 50cm^3 of solution A into a clean 100cm^3 beaker or conical flask.
2. Determined the initial temperature T_1.
3. Added 1g of B, swirl the mixture and record the maximum or minimum temperature reached as T_2.

Procedure B:

1. Measured out 50cm^3 of solution A into a clean 100cm^3 beaker or conical flask.
2. Determined the initial temperature T_3.
3. Added 0.2g of C, swirl the mixture and record the maximum or minimum temperature reached T_4.

Questions

a) Calculate the heat change per mole during the reaction in procedure A and B

b) Calculate the enthalpy of formation of magnesium carbonate ($MgCO_3$)

Given that:

The enthalpy of formation of $CO_2 = -394\text{kJ/mol}$
The enthalpy of formation of $H_2O = -286\text{kJ/mol}$

c) Suggest one way in which heat loss to the environment might affect your calculated enthalpy change. Would it lead to an underestimation or overestimation? Explain.

d) In real life, reactions between magnesium carbonates and acids are common in antacid tablets. How does this experiment simulate such a reaction, and what conclusions can be drawn about the heat change involved?

Solution

TABLE OF RESULTS

Experiment	**Volume of HCl (cm^3)**	**Initial temp.** (°C)	**Final temp.** (°C)	**Temp. change,**ΔT(°C)
$MgCO_3$	50	T_1= 26	T_2=29	+3
Mg ribbon ($Mg_{(s)}$)	50	T_3=26	T_4=41	+16

Making Sense of the Experimental Data

Since the reaction between magnesium and hydrochloric acid is a redox reaction involving the transfer of electrons, it is expected to release a greater amount of energy compared to the reaction between magnesium carbonate and hydrochloric acid, which is simply an acid-base neutralization.

The larger amount of heat evolved in the redox reaction leads to a higher temperature rise, which aligns well with the experimental observations.

Therefore, the results are consistent with theoretical expectations, indicating that the experiment was successful.

Response to Questions

(a) Using molar heat of reaction, $\Delta H_r = \frac{\text{Total heat change, } \Delta H}{\text{Number of moles of reactant, } n_r}$;

Where; $\Delta H = -m_{soln}C_{soln}\Delta T = -\rho_{soln}V_{soln}C_{soln}\Delta T$ and $n_r = \frac{m_r}{M_r}$

For procedure A:

$$\Delta H_r = \frac{-\rho_{soln}V_{soln}C_{soln}\Delta T \times M_r}{m_r} = \frac{-1\text{gcm}^{-3} \times 50\text{cm}^3 \times 4.2\text{Jg}^{-1}°\text{C}^{-1} \times 3°\text{C} \times 84\text{gmol}^{-1}}{1\text{g}}$$

$$= -52920\text{Jmol}^{-1} = -52.92\text{kJmol}^{-1}$$

The molar heat change for procedure A is -52.92kJmol^{-1}

For procedure B:

$$\Delta H_r = \frac{-\rho_{soln}V_{soln}C_{soln}\Delta T \times M_r}{m_r} = \frac{-1\text{gcm}^{-3} \times 50\text{cm}^3 \times 4.2\text{Jg}^{-1}{}^\circ\text{C}^{-1} \times 16^\circ\text{C} \times 24\text{gmol}^{-1}}{0.2\text{g}}$$

$$= -403200\text{Jmol}^{-1} = -403.2\text{kJmol}^{-1}$$

The molar heat change for procedure B is -403.2kJmol^{-1}

(b) Reaction equation for each procedure is as follows:

Procedure A: $MgCO_3(aq) + 2HCl(aq) \longrightarrow MgCl_2(aq) + CO_2(g) + H_2O(l); -52.92\text{kJmol}^{-1}$

Procedure B: $Mg(s) + 2HCl(aq) \longrightarrow MgCl_2(aq) + H_2(g); -403.2\text{kJmol}^{-1}$

Using $\Delta H_r = \sum \Delta H_f(\text{Products}) - \sum \Delta H_f(\text{Reactants})$

From procedure A;

$\Delta H_r = -52.92\text{kJmol}^{-1} = \Delta H_f(MgCl_2) + \Delta H_f(CO_2) + \Delta H_f(H_2O) - \Delta H_f(MgCO_3) - 2\Delta H_f(HCl) \ldots$ (i)

From procedure B;

$$\Delta H_r = -403.2\text{kJmol}^{-1} = \Delta H_f(MgCl_2) + \Delta H_f(H_2) - \Delta H_f(Mg) - 2\Delta H_f(HCl) \ldots\ldots\ldots \text{(ii)}$$

Then taking (ii) − (i) gives;

$(-403.2 - (-52.92))\text{kJmol}^{-1} = \Delta H_f(H_2) - \Delta H_f(Mg) - \Delta H_f(CO_2) - \Delta H_f(H_2O) + \Delta H_f(MgCO_3)$

Where:

$$\Delta H_f(H_2) = \Delta H_f(Mg) = 0, \Delta H_f(CO_2) = -394\text{kJmol}^{-1}, \Delta H_f(H_2O) = -286\text{kJmol}^{-1}$$

Substituting $-350.28\text{kJmol}^{-1} = 0 - 0 - (-394\text{kJmol}^{-1}) - (-286\text{kJmol}^{-1}) + \Delta H_f(MgCO_3)$

From which; $\Delta H_f(MgCO_3) = 1030.28\text{kJmol}^{-1}$

The enthalpy of formation of magnesium carbonate is 1030.28kJmol^{-1}.

(c) If heat is lost to the environment during the reaction, the thermometer will record a lower temperature change than what actually occurred. Since enthalpy change (ΔH) is directly calculated from the temperature difference, this heat loss will cause the measured temperature rise to be smaller. As a result, the calculated enthalpy change will be less negative (smaller in magnitude) than the true value. Therefore, heat loss to the surroundings leads to an **underestimation** of the enthalpy change.

(d) This experiment simulates the neutralization reaction that occurs when antacid tablets like magnesium carbonate react with stomach acid (hydrochloric acid). The fizzing observed during the experiment is due to the release of carbon dioxide (CO_2) which is the same gas released in the stomach when antacids are taken.

From the experiment, we observe a temperature increase, indicating that the reaction is exothermic. This means that when an antacid reacts with stomach acid, heat is released, which may help soothe the discomfort by neutralizing the acid and slightly warming the surrounding stomach fluid.

Experiment 37

It was a hot Tuesday afternoon and Kipute, armed with her lab coat and a quiet curiosity, was determined to uncover the secrets of salts—yes, plain old salts! They were mysterious substances that could either be hot like school gossip or cold like a surprise test in math you didn't study for!

Mr. Akilikubwa, always a step ahead, had left her a sticky note on the lab bench:

"Kipute, ever wonder why your ORS packet cools your water while Eno bubbles heat it up? Well… now is your time to find out. These salts may look innocent, but they're thermochemically dramatic. Good luck!"

She was provided with the following:

- **W:** 2g of anhydrous sodium thiosulphate ($Na_2S_2O_3$)
- **X:** 2g of sodium oxalate ($Na_2C_2O_4$)
- **Y:** 2g of sodium chloride (NaCl)
- **Z:** 2g of ammonium chloride (NH_4Cl)
- **Distilled water**
- **100 mL beaker**
- **Thermometer (0–100°C)**
- **50 mL measuring cylinder**
- **Stirring rod**
- **Stopwatch or clock**

Kipute's thermochemical procedure

1. She measured exactly **50 cm³ of distilled water** into a clean 100 mL beaker using the measuring cylinder.
2. Using a thermometer, she recorded the **initial temperature (T_1)** of the water.
3. She then added **2g of salt W** into the beaker, started her stopwatch, and stirred the mixture with care.
4. She recorded the **temperature every 30 seconds for 4 minutes**, watching the thermometer like a hawk.
5. She repeated steps (1) to (4) for each of the other salts: **X**, **Y**, and **Z**.
6. All results were entered neatly into a table like this:

SALT	Initial temperatureT_1 (°C)	Temperature, T_2 (°C)at time, t(sec)								Temperature change. $\Delta T = T_2 - T_1$
		30	60	90	120	150	180	210	240	
W										
X										
Y										
Z										

Questions

a) For each of the salts, determine whether it behaves like an Oral Rehydration Solution (ORS), which cools the solution, or like Eno (an effervescent antacid), which warms it up. Justify your answer based on the results obtained in the experiment.
b) Calculate the heat change of solution for each of the salt.
c) Calculate the molar enthalpy of solution for each of the salt.
d) How does the dissolution of ammonium chloride relate to its use in cold packs? Justify with data from your experiment.

Solution

Practical tip: *The final temperature (T_2) is the maximum or minimum temperature recorded.*

TABLE OF RESULTS

SALT	Initial temp, T_1 (°C)	Temperature, T_2 (°C) at time, t(sec)								Temperature change. $\Delta T = T_2 - T_1$ (°C)
		30	60	90	120	150	180	210	240	
W	25	25.8	24.6	24.5	24.4	24.9	25	25	25	$24.4 - 25 = -0.6$
X	25	25	25	25	24.4	24.2	24	23.8	23.5	$23.5 - 25 = -1.5$
Y	25	24.8	24.5	24.1	24.1	24.1	24	24.1	24.5	$24 - 25 = -1$
Z	25	23	22.5	22.5	22	22.4	22.9	23	24	$22 - 25 = -3$

Making Sense of the Experimental Data

None of the salts increased the temperature implying that all processes are endothermic to varying degrees.

Z *(Ammonium chloride) shows the largest drop in temperature* ($\boldsymbol{-3\,°C}$)*, indicating that it absorbs the greatest amount of heat from water.*

Response to Questions

(a) All four salts behave like ORS.

Justification:

Based on the experimental data, all salts caused a decrease in temperature upon dissolving, indicating endothermic behaviour. This means they absorb heat from the surrounding water like ORS.

(b) Using $\Delta H = -m_{soln}C_{soln}\Delta T = -\rho_{soln}V_{soln}C_{soln}\Delta T$

For $Na_2S_2O_3$:

$$\Delta H = -1\text{gcm}^{-3} \times 50\text{cm}^3 \times 4.2\text{Jg}^{-1}{}°\text{C}^{-1} \times -0.6°\text{C} = +126\text{J}$$

The heat change for $Na_2S_2O_3$ is $+126$J.

For $Na_2C_2O_4$:

$$\Delta H = -1\text{gcm}^{-3} \times 50\text{cm}^3 \times 4.2\text{Jg}^{-1}{}°\text{C}^{-1} \times -1.5°\text{C} = +315\text{J}$$

The heat change for $Na_2C_2O_4$ is $+315$J.

For NaCl:

$$\Delta H = -1\text{gcm}^{-3} \times 50\text{cm}^3 \times 4.2\text{Jg}^{-1}{}^\circ\text{C}^{-1} \times -1^\circ\text{C} = +210\text{J}$$

The heat change for NaCl is $+210\text{J}$.

For NH_4Cl:

$$\Delta H = -1\text{gcm}^{-3} \times 50\text{cm}^3 \times 4.2\text{Jg}^{-1}{}^\circ\text{C}^{-1} \times -3^\circ\text{C} = +630\text{J}$$

The heat change for NH_4Cl is $+630\text{J}$.

(c) Using molar enthalpy of solution, $\Delta H_{\text{soln}} = \frac{\text{Total enthalpy change, } \Delta H}{\text{Number of moles of solute, } n_{su}}$;

Where; $n_{su} = \frac{m_{su}}{M_{su}}$

For $Na_2S_2O_3$:

$$\Delta H_{\text{soln}} = \frac{\Delta H \times M_{su}}{m_{su}} = \frac{+126\text{J} \times 158\text{gmol}^{-1}}{2\text{g}} = +9954\text{Jmol}^{-1} = +9.954\text{kJmol}^{-1}$$

The molar enthalpy of solution of $Na_2S_2O_3$ is $+9.954\text{kJmol}^{-1}$.

For $Na_2C_2O_4$:

$$\Delta H_{\text{soln}} = \frac{\Delta H \times M_{su}}{m_{su}} = \frac{+315\text{J} \times 134\text{gmol}^{-1}}{2\text{g}} = +21105\text{Jmol}^{-1} = +21.105\text{kJmol}^{-1}$$

The molar enthalpy of solution of $Na_2C_2O_4$ is $+21.105\text{kJmol}^{-1}$.

For NaCl:

$$\Delta H_{\text{soln}} = \frac{\Delta H \times M_{su}}{m_{su}} = \frac{+210\text{J} \times 58.5\text{gmol}^{-1}}{2\text{g}} = +6142.5\text{Jmol}^{-1} = +6.1425\text{kJmol}^{-1}$$

The molar enthalpy of solution of NaCl is $+6.1425\text{kJmol}^{-1}$.

For NH_4Cl:

$$\Delta H_{\text{soln}} = \frac{\Delta H \times M_{su}}{m_{su}} = \frac{+630\text{J} \times 53.5\text{gmol}^{-1}}{2\text{g}} = +16852.5\text{Jmol}^{-1} = +16.8525\text{kJmol}^{-1}$$

The molar enthalpy of solution of NaCl is $+16.8525\text{kJmol}^{-1}$.

(d) Among given salts, ammonium chloride caused highest drop in temperature when dissolved in water which implies that the process is most endothermic. This property makes it suitable for use in instant cold packs, where a quick drop in temperature per given mass is desired.

Experiment 38

It was one of those unusually quiet afternoons in the school lab until Mr. Akilikubwa, the ever-curious lab technician, burst in like a chemical equation waiting to happen. He was muttering, *"Today... we uncover the* heat *of* ***love****, or at least, the heat of neutralisation!"*

With his lab coat slightly wrinkled and his goggles perched askew, he laid out the tools of his experiment like a chef prepping for a spicy dish. But instead of spices, he had:

- **A**: 2.0 M Sodium Hydroxide solution (NaOH).
- **B**: 2.0 M Hydrochloric acid (HCl).
- **C**: 2.0 M Acetic acid (CH_3COOH).

- A thermometer, a plastic beaker, and a measuring cylinder.

"Time to see who gets hotter*—HCl or CH_3COOH,"* he chuckled. *"Let's see which one makes NaOH sweat more!"*

Akilikubwa's procedure

1. He measured **100 cm³** of solution **A** (NaOH) into a clean plastic beaker and recorded the temperature. Let's call this $\mathbf{T_A}$.
2. He rinsed the measuring cylinder and carefully measured **100 cm³** of **solution B**, recording its temperature too as $\mathbf{T_B}$.
3. He poured the acid into the beaker with the base, stirred gently, and recorded the maximum or minimum temperature reached as $\mathbf{T_f}$,.
4. Then, after rinsing everything squeaky clean, he repeated the same steps—but this time using **solution C** instead of B.

The **initial temperature ($\mathbf{T_i}$)** for each experiment was found by averaging the temperatures of the two separate solutions before mixing $\left(T_i = \frac{T_A+T_B}{2}\right)$.

Experiment	Initial temp, T_i(°C)	Final temp, T_f(°C)	Temperature change,ΔT(°C)
1.			
2.			

Questions

a) Calculate the molar enthalpy of neutralisation for the reaction of:
   - (i) $NaOH(aq) + HCl(aq) \longrightarrow NaCl(aq) + H_2O(l)$
   - (ii) $NaOH(aq) + CH_3COOH(aq) \longrightarrow CH_3COONa(aq) + H_2O(l)$

b) Compare the two values and explain about the molar enthalpies of the two reactions as per comparison.

c) Why might the measured ΔT be less than expected in either experiment? Give at least three possible reasons.

d) Explain why does the temperature rise when NaOH reacts with HCl or CH_3COOH?

Solution

TABLE OF RESULTS

Experiment	Initial temp, T_i(°C)	Final temp, T_f(°C)	Temperature change,ΔT(°C)
1.	31	43.9	+12.9
2.	30.5	42.7	+12.2

Making Sense of the Experimental Data

Practical tip 1: *Under given conditions, enthalpy of neutralization between any strong acid and any strong base is approximately the same and it is typically around −57.3 kJ/mol at standard conditions.*

Practical tip 2: *Under given conditions, enthalpy of neutralization between acetic acid (weak acid) and any strong base is approximately the same and it is typically around −55.2 kJ/mol at standard conditions.*

Using enthalpy of neutralization, $\Delta H_n = \frac{\text{Total enthalpy change, } \Delta H}{\text{Number of moles of water formed, } n_w}$;

From which; $\Delta H = \Delta H_n \times n_w$

For $\mathrm{HCl - NaOH}$ reaction:

Standard $\Delta H_n = -57.3\mathrm{kJ/mol} = -57300\mathrm{Jmol^{-1}}$

And $n_w = n_{acid} = n_{base} = \frac{100}{1000}\mathrm{L} \times 2\mathrm{molL^{-1}} = 0.2\mathrm{mol}$

Then $\Delta H = -57300\mathrm{Jmol^{-1}} \times 0.2\mathrm{mol} = -11460\mathrm{J}$

But also $\Delta H = -m_{soln}C_{soln}\Delta T = -\rho_{soln}V_{soln}C_{soln}\Delta T$

Substituting $-11460\mathrm{J} = -1\mathrm{gcm^{-3}} \times (100 + 100)\mathrm{cm^3} \times 4.2\mathrm{Jg^{-1}°C^{-1}} \times \Delta T$

From which; $\Delta T = \mathbf{+13.6}$°C (experimental data = $\mathbf{+12.9}$°C).

For $\mathrm{CH_3COOH - NaOH}$ reaction:

Standard $\Delta H_n = -55.2\mathrm{kJ/mol} = -55200\mathrm{Jmol^{-1}}$

And $n_w = n_{acid} = n_{base} = \frac{100}{1000}\mathrm{L} \times 2\mathrm{molL^{-1}} = 0.2\mathrm{mol}$

Then $\Delta H = -55200\mathrm{Jmol^{-1}} \times 0.2\mathrm{mol} = -11040\mathrm{J}$

But also $\Delta H = -m_{soln}C_{soln}\Delta T = -\rho_{soln}V_{soln}C_{soln}\Delta T$

Substituting $-11040\mathrm{J} = -1\mathrm{gcm^{-3}} \times (100 + 100)\mathrm{cm^3} \times 4.2\mathrm{Jg^{-1}°C^{-1}} \times \Delta T$

From which; $\Delta T = \mathbf{+13.1}$°C (experimental data = $\mathbf{+12.2}$°C).

Conclusion: *In both cases, the experimental results do not deviate significantly from the calculated values, which suggests that the results are plausible and hence the experiment was successful.*

It's kind of funny when you think about this:

*Since the reaction with **HCl** release more heat, it clearly **wins the thermochemical showdown** — it's the strong acid that makes **NaOH hotter...** and **sweat more** than the weak acid, **CH_3COOH** ever could!*

Now, take a deep breath. Pause. Ask yourself:

*Why did Mr. Akilikubwa called the enthalpy of neutralization the **heat of love**?*

*Well, maybe because it's the perfect chemistry: two opposites (acid and base) coming together, releasing heat, and forming something stable (water) — **a bond made in thermodynamic heaven**!*

Feeling a positive vibe? A little smile? Great! Now you've got just the right energy to dive into the **analysis questions** in the next section. Let's keep the chemistry alive!

Response to Questions

(a) Using enthalpy of neutralization, $\Delta H_n = \frac{\text{Total enthalpy change, } \Delta H}{\text{Number of moles of water formed, } n_w}$

Where; $\Delta H = -m_{soln}C_{soln}\Delta T = -\rho_{soln}V_{soln}C_{soln}\Delta T$

It follows that; , $\Delta H_n = \frac{-\rho_{soln}V_{soln}C_{soln}\Delta T}{n_w}$

(i) For $HCl - NaOH$ reaction:

$$NaOH(aq) + HCl(aq) \longrightarrow NaCl(aq) + H_2O(l)$$

From which; mole ratio of $NaOH$ (or HCl) to H_2O is $1:1$

Thus $n_w = n_{HCl}(\text{or } n_{NaOH}) = 0.1dm^3 \times 2moldm^{-3} = 0.2mol$

Then;

$$\Delta H_n = \frac{-\rho_{soln}V_{soln}C_{soln}\Delta T}{n_w} = \frac{-1gcm^{-3} \times (100+100)cm^3 \times 4.2Jg^{-1}°C^{-1} \times 12.9°C}{0.2mol}$$

$$= -54180Jmol^{-1} = -54.18kJmol^{-1}$$

The enthalpy of neutralization for this reaction is $-54.18kJmol^{-1}$.

(ii) For $CH_3COOH - NaOH$ reaction:

$$NaOH(aq) + CH_3COOH(aq) \longrightarrow CH_3COONa(aq) + H_2O(l)$$

From which; mole ratio of $NaOH$ (or CH_3COOH) to H_2O is $1:1$

Thus $n_w = n_{NaOH} = 0.1dm^3 \times 2moldm^{-3} = 0.2mol$

Then;

$$\Delta H_n = \frac{-\rho_{soln}V_{soln}C_{soln}\Delta T}{n_w} = \frac{-1gcm^{-3} \times (100+100)cm^3 \times 4.2Jg^{-1}°C^{-1} \times 12.2°C}{0.2mol}$$

$$= -51240Jmol^{-1} = -51.24kJmol^{-1}$$

The enthalpy of neutralization for this reaction is $-51.24kJmol^{-1}$.

(b) The molar enthalpy of neutralization in the first experiment was higher than that of the second experiment.

This is because it involved the reaction between a strong acid and a strong base, both of which are fully ionized in solution, allowing the reaction to release more energy.

In contrast, the second experiment involved a weak acid and a strong base. Since weak acids only partially dissociate in solution, additional energy is required to further ionize the acid and produce enough hydrogen ions to neutralize all the hydroxide ions from sodium hydroxide. This energy absorbed reduces the net heat released, resulting in a lower overall enthalpy of neutralization.

(c) Possible reasons include the following:
1) Heat loss to the surroundings,
2) Incomplete mixing,
3) Temperature measurement delay,
4) Poor thermal insulation.

(d) This is because the neutralisation is an exothermic reaction as it involves attraction between H^+ from acid and OH^- from base to form water. Thus energy is released into the solution, causing the temperature to increase, which is what we observe during the experiment.

$$H^+(aq) + OH^-(aq) \longrightarrow H_2O(l) + \text{Heat}$$

Experiment 39

Kipute wasn't exactly thrilled about staying behind after school especially when her friends were out watching Star Wars movie series. But Mr. Akilikubwa had asked her for a special task. And Kipute could never resist a chemistry mystery… especially if it involved acids and bases plotting behind her back.

As she stepped into the lab, she found a sticky note from Mr. Akilikubwa taped dramatically to a plastic beaker:

> *"Dear Kipute, Today, you'll witness a war between acids—one strong, one weak—and the same old base trying to cool them both down. Stir well, observe wisely, and remember: not all neutralisations are born equal.* Sir Reagent of Heat."

Kipute rolled her eyes and chuckled. She was ready.

Materials she was provided with:

- **J1**: 0.25 M Hydrochloric acid (the strong one with serious attitude)
- **J2**: 0.5 M Acetic acid (a weaker acid that smells like vinegar and humility)
- **J3**: 0.4 M Sodium hydroxide (the **peacekeeper** base, always cool-headed)
- **Also**: Plastic beaker, thermometer, measuring cylinder, and the ever-reliable tap water for rinsing.

Kipute's neutralisation procedure

1. She measured 100 mL of **J3** using a cylinder and poured it into a clean plastic beaker. She noted its temperature as $\mathbf{T_{J3}}$.
2. Then, she rinsed the cylinder, filled it with 100 mL of **J1**, and recorded its temperature as $\mathbf{T_{J1}}$.
3. With a twinkle of suspense, she poured **J1** into the beaker containing **J3**. As she stirred with her thermometer, she watched the temperature change and recorded the maximum and minimum temperature reached.
4. She threw away the content of the beaker and wash it as well as the measuring cylinder and thermometer.
5. Next round, same steps—but this time, she used **J2** instead of **J1**.
6. And as always, she recorded results in a tabular form.

Experiment	Initial temp, T_i (°C)	Final temp, T_f (°C)	Temperature change, ΔT (°C)
1.			
2.			

Questions

a) Why in this experiment, sodium hydroxide was introduced as peace keeper?

b) Calculate the number of mole of water formed between the reaction of:

   (i) $NaOH(aq) + HCl(aq) \longrightarrow NaCl(aq) + H_2O(l)$

   (ii) $NaOH(aq) + CH_3COOH(aq) \longrightarrow CH_3COONa(aq) + H_2O(l)$

c) Calculate the molar enthalpy of neutralisation in each of the experiment.

d) What does this experiment reveal about how the strength of an acid affects the amount of heat released during neutralisation? Explain.

e) Why must the thermometer remain in the reaction mixture throughout stirring and temperature recording?

Solution

TABLE OF RESULTS

Experiment	Initial temp, T_i(°C)	Final temp, T_f(°C)	Temperature change,ΔT(°C)
1.	27	30	+3
2.	25.5	28	+2.5

Making Sense of the Experimental Data

Using enthalpy of neutralization, $\Delta H_n = \frac{\text{Total enthalpy change, } \Delta H}{\text{Number of moles of water formed, } n_w}$;

From which; $\Delta H = \Delta H_n \times n_w$

For $\mathbf{HCl - NaOH}$ reaction:

Standard $\Delta H_n = -57.3\text{kJ/mol} = -57300\text{Jmol}^{-1}$

But in this case, molar concentrations of HCl and NaOH are not equal like in the previous experiment. Sine equal volumes were taken for both acid and base and $n = MV$; for this case $n_{acid} \neq n_{base}$.

However, the stoichiometric of the reaction requires equal number of moles of the two. Unequal number of moles means that one of the reactant (acid or base) was present in excess and thus to know number of moles of water formed we have to firstly identify the amount of limited reactant.

$$n_{acid} = \frac{100}{1000}\text{L} \times 0.25\text{molL}^{-1} = 0.025\text{mol}$$

And $n_{base} = \frac{100}{1000}\text{L} \times 0.4\text{molL}^{-1} = 0.04\text{mol}$

Since $n_{acid} < n_{base}$, HCl is the limited reactant and is the one which will determine the amount of water formed.

Thus $n_{acid} = n_w = 0.025\text{mol}$

Now $\Delta H = -57300\text{Jmol}^{-1} \times 0.025\text{mol} = -1432.5\text{J}$

But also $\Delta H = -m_{soln}C_{soln}\Delta T = -\rho_{soln}V_{soln}C_{soln}\Delta T$

Substituting $-1432.5\text{J} = -1\text{gcm}^{-3} \times (100 + 100)\text{cm}^3 \times 4.2\text{Jg}^{-1}{}^\circ\text{C}^{-1} \times \Delta T$

From which; $\Delta T = \mathbf{+1.7}$°C (experimental data = $\mathbf{+3}$°C).

For $\mathbf{CH_3COOH - NaOH}$ reaction:

Standard $\Delta H_n = -55.2\text{kJ/mol} = -55200\text{Jmol}^{-1}$

In this case, $n_{acid} = \frac{100}{1000}\text{L} \times 0.5\text{molL}^{-1} = 0.05\text{mol}$

And n_{base} is still 0.04mol

Now, $\mathrm{n_{base}} < \mathrm{n_{acid}}$ and for similar reasoning as in the previous reaction, NaOH is the limited reactant and is the one which will determine the amount of water formed.

Thus $\mathrm{n_{base}} = \mathrm{n_w} = 0.04\mathrm{mol}$

Now $\Delta\mathrm{H} = -55200\mathrm{Jmol^{-1}} \times 0.04\mathrm{mol} = -2208\mathrm{J}$

But also $\Delta\mathrm{H} = -\mathrm{m_{soln}C_{soln}}\Delta\mathrm{T} = -\rho_{\mathrm{soln}}\mathrm{V_{soln}C_{soln}}\Delta\mathrm{T}$

Substituting $-2208\mathrm{J} = -1\mathrm{gcm^{-3}} \times (100 + 100)\mathrm{cm^3} \times 4.2\mathrm{Jg^{-1}{}^{\circ}C^{-1}} \times \Delta\mathrm{T}$

From which; $\Delta\mathrm{T} = \mathbf{+2.6}{}^{\circ}\mathrm{C}$ (experimental data $= \mathbf{+2.5}{}^{\circ}\mathrm{C}$).

Conclusion……but wait! Before we get to the conclusion, let's quickly go through a few practical tips that will help make everything clearer.

Practical tip 1: *Accurate experimental results of enthalpy of neutralization are obtained when the acid and base are mixed in their correct stoichiometric proportions.*

Clarification:

Any unreacted acid or base remaining in solution may undergo its own enthalpy of solution (heat of dissolution), which can add to the total heat change measured in the experiment.

*This interferes with the accurate determination of the enthalpy of neutralization, because the heat measured will not come **solely** from the neutralization reaction; it will be a **mixture of both enthalpy of solution and enthalpy of neutralization**.*

Practical tip 2: *In most cases, systematic errors in thermochemical experiments lead to an underestimation of the heat change, usually because some heat is lost to the surroundings or absorbed by the container instead of being measured.*

That is all about practical tips, at least for now! Now, let us return to our conclusion!

That wraps up our practical tips—at least for now! Armed with those insights, it's time to shift gears and bring everything together **in our conclusion.**

Conclusion:

The results of the first experiment showed a greater deviation from the calculated enthalpy value compared to the second experiment. Notably, the deviation in the first experiment was unusual, as the measured temperature rise exceeded the calculated value, indicating an overestimation of the heat change. This unexpected result can be explained by the following factors:

- *In the first experiment, the excess reagent was sodium hydroxide (NaOH), which has a highly exothermic enthalpy of solution of approximately –44.5 kJ/mol under standard conditions. The unreacted NaOH continued to dissolve in water, releasing additional heat and resulting in a significantly higher temperature rise than expected.*
- *In the second experiment, the excess reagent was acetic acid (CH_3COOH), which has a much lower enthalpy of solution, around –1.7 kJ/mol. The minor heat released from its dissolution was largely offset by heat loss to the surroundings, making the experimental temperature rise slightly lower (and much closer) to the calculated value.*

Armed with this understanding, let us now proceed to the analysis questions that follow.

Response to Questions

(a) Because it neutralizes both acids, regardless of their strength to form neutral water.

(b)

(i) For $\mathrm{HCl - NaOH}$ experiment:

$$\mathrm{NaOH(aq) + HCl(aq) \longrightarrow NaCl(aq) + H_2O(l)}$$

From which; mole ratio of NaOH to HCl is 1: 1 (Equal number of moles for acid and base).

Using $\mathrm{n = MV}$;

$$\mathrm{n_{acid} = \frac{100}{1000} L \times 0.25molL^{-1} = 0.025mol}$$

And $\mathrm{n_{base} = \frac{100}{1000} L \times 0.4molL^{-1} = 0.04mol}$

Since $\mathrm{n_{acid} < n_{base}}$, HCl is the limited reactant and is the one which will determine the amount of water formed.

Thus $\mathrm{n_{acid} = n_w = 0.025mol}$ (From the mole ratio between acid and water).

The number of moles of water is 0.025mol.

(ii) For $\mathrm{CH_3COOH - NaOH}$ experiment:

$$\mathrm{NaOH(aq) + CH_3COOH(aq) \longrightarrow CH_3COONa(aq) + H_2O(l)}$$

Again, the mole ratio of NaOH to $\mathrm{CH_3COOH}$ is 1: 1.

Using $\mathrm{n = MV}$;

In this case, $\mathrm{n_{acid} = \frac{100}{1000} L \times 0.5molL^{-1} = 0.05mol}$

And $\mathrm{n_{base} = \frac{100}{1000} L \times 0.4molL^{-1} = 0.04mol}$

Since $\mathrm{n_{base} < n_{acid}}$, NaOH is the limited reactant and is the one which will determine the amount of water formed.

Thus $\mathrm{n_{base} = n_w = 0.04mol}$ (from the stoichiometric ratio)

The number of moles of water is 0.04mol.

(c) Using enthalpy of neutralization, $\Delta\mathrm{H_n} = \frac{\text{Total enthalpy change, } \Delta\mathrm{H}}{\text{Number of moles of water formed, } \mathrm{n_w}}$

Where; $\Delta\mathrm{H} = -\mathrm{m_{soln}C_{soln}}\Delta\mathrm{T} = -\rho_{\mathrm{soln}}\mathrm{V_{soln}C_{soln}}\Delta\mathrm{T}$

It follows that; , $\Delta\mathrm{H_n} = \frac{-\rho_{\mathrm{soln}}\mathrm{V_{soln}C_{soln}}\Delta\mathrm{T}}{\mathrm{n_w}}$

For $\mathrm{HCl - NaOH}$ experiment:

$$\Delta\mathrm{H_n} = \frac{-\rho_{\mathrm{soln}}\mathrm{V_{soln}C_{soln}}\Delta\mathrm{T}}{\mathrm{n_w}} = \frac{-1\mathrm{gcm^{-3}} \times (100+100)\mathrm{cm^3} \times 4.2\mathrm{Jg^{-1}{}^{\circ}C^{-1}} \times 3^{\circ}\mathrm{C}}{0.025\mathrm{mol}}$$

$$= -100800\mathrm{Jmol^{-1}} = -100.8\mathrm{kJmol^{-1}}$$

The enthalpy of neutralization for this experiment is $-100.8\mathrm{kJmol^{-1}}$.

For $\mathrm{CH_3COOH - NaOH}$ experiment:

$$\Delta\mathrm{H_n} = \frac{-\rho_{\mathrm{soln}}\mathrm{V_{soln}C_{soln}}\Delta\mathrm{T}}{\mathrm{n_w}} = \frac{-1\mathrm{gcm^{-3}} \times (100+100)\mathrm{cm^3} \times 4.2\mathrm{Jg^{-1}{}^{\circ}C^{-1}} \times 2.5^{\circ}\mathrm{C}}{0.04\mathrm{mol}}$$

$$= -52500\text{Jmol}^{-1} = -52.5\text{kJmol}^{-1}$$

The enthalpy of neutralization for this experiment is -52.5kJmol^{-1}.

(d) The experiment reveals that strong acids like HCl typically release more heat upon neutralisation than weak acids like acetic acid. This is because strong acids fully ionise in solution, allowing for a more complete and rapid reaction with OH^- ions from NaOH. Weak acids, on the other hand, only partially ionise, so some energy is absorbed to enable their ionization and therefore less heat is released.

(e) Keeping the thermometer in the mixture ensures that continuous and accurate temperature readings are taken, especially at the moment when the maximum temperature is reached. Removing and reinserting the thermometer can lead to heat loss and inaccurate readings.

Experiment 40

It was a steamy Thursday afternoon when Kipute found herself alone in the chemistry lab again. Mr. Akilikubwa had mysteriously vanished, but not before leaving his signature behind, a post; it stuck on the fume cupboard, complete with doodles of molecules holding hands.

"Kipute, sometimes in life, relationships aren't always 50/50. Today, you'll witness two very different acid-base pairings. One gets 80 mL of love, the other only 25 mL. Your job: figure out which one is more ***exothermantic****.* Akilikubwa, the Curious*"*

Kipute laughed out loud. Only Mr. Akilikubwa could make thermochemistry funny and exciting!

Reagents and tools at Kipute's disposal

- **J**: 1M Hydrochloric acid
- **K**: 1M Acetic acid
- **L**: 1M Sodium hydroxide
- Plastic beaker, thermometer, measuring cylinder, and distilled water.

Kipute's procedure

1. She measured **100 mL** of **solution L** and poured it into a clean plastic beaker. Measured its temperature and noted as $\mathbf{T_L}$.
2. Rinsed her cylinder and then measured **80 mL** of **J**. Its temperature was measured and recorded as $\mathbf{T_J}$.
3. She poured **J** into **L** and stirred the solution gently using a thermometer and record the maximum or minimum temperature reached as $\mathbf{T_f}$.
4. She threw away the content of the beaker and washed it as well as the measuring cylinder and thermometer.
5. Repeated the procedures (1) to (4) but this time using 25mL of solution K instead of J and 50mL of solution L
6. She recorded the results in tabular form.

Questions

a) Calculate the number of moles of water formed in each experiment.
b) Calculate the molar enthalpy in each experiment.
c) Explain why must the initial temperature of the acid and base be measured separately before mixing?
d) If the plastic beaker was replaced with a metallic one, how might the observed temperature change be affected and why?

Solution

TABLE OF RESULTS

Experiment	Initial temp, T_i(°C)	Final temp, T_f(°C)	Temperature change, ΔT(°C)
1.	29	34	+5
2.	28	32	+4

Making Sense of the Experimental Data

To a large extent, this experiment closely resembles **Experiment 39**. The key difference lies in the fact that, in this case, the unequal number of moles of acid and base results from different volumes being used, while their molar concentrations remain constant**.**

Response to Questions

(a)

(i) For HCl − NaOH experiment:

$$\mathrm{NaOH(aq) + HCl(aq) \longrightarrow NaCl(aq) + H_2O(l)}$$

From which; mole ratio of NaOH to HCl is 1: 1 (Equal number of moles for acid and base).

Using $n = MV$;

$$n_{acid} = \frac{80}{1000}\mathrm{L} \times 1\mathrm{molL^{-1}} = 0.08\mathrm{mol}$$

And $n_{base} = \frac{100}{1000}\mathrm{L} \times 1\mathrm{molL^{-1}} = 0.1\mathrm{mol}$

Since $n_{acid} < n_{base}$, HCl is the limited reactant and is the one which will determine the amount of water formed.

Thus $n_{acid} = n_w = 0.08\mathrm{mol}$ (From the stoichiometric ratio).

The number of moles of water is 0.08mol.

(ii) For $\mathbf{CH_3COOH}$ − NaOH experiment:

$$\mathrm{NaOH(aq) + CH_3COOH(aq) \longrightarrow CH_3COONa(aq) + H_2O(l)}$$

Again, the mole ratio of NaOH to CH_3COOH is 1: 1.

Using $n = MV$;

In this case, $n_{acid} = \frac{25}{1000}\mathrm{L} \times 1\mathrm{molL^{-1}} = 0.025\mathrm{mol}$

And $n_{base} = \frac{50}{1000}\mathrm{L} \times 1\mathrm{molL^{-1}} = 0.05\mathrm{mol}$

Since $n_{acid} < n_{base}$, CH_3COOH is the limited reactant and is the one which will determine the amount of water formed.

Thus $n_{acid} = n_w = 0.025\mathrm{mol}$ (from the stoichiometric ratio)

The number of moles of water is 0.025mol.

(b) Using enthalpy of neutralization, $\Delta H_n = \frac{\text{Total enthalpy change, } \Delta H}{\text{Number of moles of water formed, } n_w}$

Where; $\Delta H = -m_{soln}C_{soln}\Delta T = -\rho_{soln}V_{soln}C_{soln}\Delta T$

It follows that; , $\Delta H_n = \frac{-\rho_{soln}V_{soln}C_{soln}\Delta T}{n_w}$

For $HCl - NaOH$ experiment:

$$\Delta H_n = \frac{-\rho_{soln}V_{soln}C_{soln}\Delta T}{n_w} = \frac{-1gcm^{-3} \times (100 + 80)cm^3 \times 4.2Jg^{-1}°C^{-1} \times 5°C}{0.08mol}$$

$$= -47250Jmol^{-1} = -47.25kJmol^{-1}$$

The enthalpy of neutralization for this experiment is $-47.25kJmol^{-1}$.

For $CH_3COOH - NaOH$ experiment:

$$\Delta H_n = \frac{-\rho_{soln}V_{soln}C_{soln}\Delta T}{n_w} = \frac{-1gcm^{-3} \times (50 + 25)cm^3 \times 4.2Jg^{-1}°C^{-1} \times 4°C}{0.025mol}$$

$$= -50400Jmol^{-1} = -50.4kJmol^{-1}$$

The enthalpy of neutralization for this experiment is $-50.4kJmol^{-1}$.

(c) The initial temperature of both the acid and the base must be measured separately to calculate an accurate average initial temperature before the neutralisation reaction begins. This average value is used to determine the temperature change caused solely by the chemical reaction.

If only one initial temperature is taken, it may not reflect the true thermal condition of the mixed solution (especially if the acid and base were at different temperatures before mixing). This would lead to inaccurate enthalpy calculations, as part of the observed temperature change could be due to thermal exchange between the solutions rather than the neutralisation process itself.

(d) If a metallic beaker were used instead of a plastic one, the observed temperature change (ΔT) would likely be lower.

This is because metals are excellent conductors of heat, so they would absorb and lose more heat to the surroundings quickly, reducing the amount of heat retained in the solution.

PART THREE

QUALITATIVE ANALYSIS

Chapter 7

QUALITATIVE ANALYSIS

INTRODUCTION

Analytical chemistry deals with qualitative and quantitative analysis of the substances. All practicals we have dealt with so belong to quantitative analysis. In qualitative analysis, the given compound is analyzed for the ions, *i.e.*, cation and the anion, that it contains. Physical procedures like noting the colour, smell or taste of the substance have very limited scope because of the corrosive, poisonous nature of the chemical compounds. Therefore, what one has to resort to is the chemical analysis of the substance that has to be carried out along with the physical examination of the compound under consideration.

The common procedure for testing any unknown sample is to make its solution and then test this solution for the ions present in it. There are separate procedures for detecting cations and anions, therefore qualitative analysis is studied under cation analysis and anion analysis. The systematic procedure for qualitative analysis of an inorganic salt involves the following steps:

1. **Preliminary tests**
    (i) Physical appearance.
    (ii) Action of heat (dry heating test).
    (iii) Flame test.
    (iv) Solubility.
    (v) Acid test.
2. **Test in aqueous solutions**
3. **Confirmatory tests.**

The preliminary tests are generally for solid samples as follows:

1. Appearance: this involves
    i. Colour of the sample
    ii. Texture of the sample
    iii. Deliquescence
    iv. Odour

PHYSICAL APPEARANCE

The physical examination of the unknown salt involves the study of **colour, smell** and **texture.** The test is not much reliable, but is certainly helpful in identifying some coloured cations especially those of transition elements. Ionic compounds of non-transition elements are generally colourless (white). Characteristic smell helps to identify some ions such as ammonium, acetate and sulphide.

Physical Examination

Experiment	**Observations**	**Inference**
1. Colour	Blue or Bluish green	Cu^{2+} or Ni^{2+} may be present.
	Greenish	Ni^{2+} may be present.
	Light green	Fe^{2+} may be present.
	Dark brown	Fe^{3+} may be present.
	Pink	Co^{2+} may be present.
	Light pink, flesh colour or earthy colour	Mn^{2+} may be present.
	White	$NH_4^+, Na^+, Ca^{2+}, Zn^{2+}, Pb^{2+}$ may be present OR transition metals, $Cu^{2+}, Ni^{2+}, Fe^{2+}, Fe^{3+}$, Mn^{2+}, Co^{2+} may be absent.
2. Smell Take a pinch of the salt between your fingers and rub with a drop of water	Ammoniacal smell Vinegar like smell Smell like that of rotten eggs	$NH_4{}^+$ may be present. CH_3COO^- may be present. S^{2-} may be present.
3. Texture	(i) Crystalline form (ii) Powder form	NO_3^-, SO_4^{2-}, Cl^- may be present. $NO_3^-, HCO_3^-, CO_3^{2-}$ may be present
4. Deliquescence	Salt absorbs moisture and becomes paste like	(i) If coloured, $Cu(NO_3)_2, FeCl_3$ may be present (ii) If colourless, $Zn(NO_3)_2$, chlorides of Zn^{2+}, Mg^{2+} etc. may be present.

DRY HEATING TEST

This test is performed by heating a small amount of salt in a dry test tube. Quite valuable information can be gathered by carefully performing and noting the observations here. On heating, some salts undergo decomposition, thus, evolving the gases or may undergo characteristic changes in the colour of residue. On another hand, if a compound is heated and a colourless liquid condenses on the cooler inner walls of the test tube, the compound is likely a hydrate, and the condensed liquid is most probably water. These observations are tabulated in the following table along with the inferences that you can draw.

Experiments	**Observations**	**Inferences**
Action of heat on the sold sample Transfer a small amount of the solid sample in a dry test tube. Heat gently and then strongly until no further change is observed. Test for any gas evolved and observe the appearance of the residues.	White sublime and colourless gas with a chocking smell which turns moist red litmus paper blue and forms dense white fumes with HCl	NH_4^+ may be present.
	Reddish – brown fumes evolve which turns moist blue litmus paper red and a gas which relights a glowing splint.	NO_3^- may be present, except that of Na^+
	Colorless gas evolves which relights a glowing splint	NO_3^- of Na^+ may be present.
	Colourless gas evolves which turns limewater milky and moist blue litmus paper to red	CO_3^{2-}, HCO_3^- may be present
	Colourless gas with pungent smell evolves which turns moist blue litmus paper red or reduces acidified potassium dichromate solution from orange to green or decolorizes the solution of potassium permanganate	SO_4^{2-} may be present except that of Na^+
	Colourless gas with a pungent smell evolves which gives dense white fumes with ammonia gas and turns moist blue litmus paper red	Cl^- of hydrated $Ca^{2+}, Zn^{2+}, Fe^{2+}, Fe^{3+}, Cu^{2+}$ May be present.
	No gas evolves	SO_4^{2-} of Na^+, Ca^{2+}, Pb^{2+} may be present
		Cl^- of Na^+, Pb^{2+} may be present
		CO_3^{2-} of Na^+ may be present
	Colourless droplets forming on the cooler parts of the test tube that turns anhydrous $CuSO_4$ blue or $CoCl_2$ pink	Hydrated salts, HCO_3^- may be present
	Cracking sound and brown gas	NO_3^- of Pb^{2+} may be present
	White sublimate and colourless gas with chocking smell which turns moist red litmus paper blue	NH_4^+ may be present.
	Cracking sound with no evolution of gas	Cl^- of Na^+ may be present

	Yellow residue when hot and white when cold	Zn^{2+} may be present.
	Reddish – brown residue when hot and yellow when cold	Pb^{2+} may be present
	Black residue	Cu^{2+}, may be present
	Reddish – brown residue	Fe^{2+}, Fe^{3+}, may be present.
	White residue	Na^+, Ca^{2+} may be present.
	Blue crystals which turns white even without warming	SO_4^{2-} of hydrated Cu^{2+} may be present.

Practical tips:

1. *Use a perfectly dry test-tube for performing this test. While drying a test-tube, keep it in slanting position with its mouth slightly downwards so that the drops of water which condense on the upper cooler parts, do not fall back on the hot bottom, as this may break the tube.*
2. *Do not heat the tube strongly at one point as it may break.*

FLAME TEST

Certain salts on reacting with concentrated hydrochloric acid from their chlorides, that are volatile in non-luminous flame. Their vapours impart characteristic colour to the flame. This colour can give reliable information of the presence of certain cations. For proceeding to this test, the paste of the mixture with conc. hydrochloric acid is introduced into the flame with the help of platinum wire.

Flame Test

Experiments	Observations	Inferences
Dip a clean nichrome wire (or clean glass rod or base of the test tube) in concentrated HCl to allow a small sample to adhere on it, then pick up small amount of the sample using the wet wire (or glass rod or the base of the test tube) and heat it on a flame	Golden yellow flame	Na^+ may be present.
	Brick red flame	Ca^{2+} may be present.
	Bluish – green flame	Cu^{2+} may be present.
	Blue – white (pale – blue) flame	Pb^{2+} may be present.
	Yellow (orange) sparks	Fe^{2+}, Fe^{3+} may be present.
	No definite flame colour	NH_4^+ , Zn^{2+} may be present.

ACID TEST

The identification of anions is first done on the basis of preliminary tests. Dry heating test is one of the preliminary tests performed earlier which may give some important information about the anion present. Identification of anions is usually further done by acid test by using either:

- Dilute acids (dilute H_2SO_4 or dilute HCl is used) or
- Concentrated acid (usually concentrated H_2SO_4 is used)

The use of these preliminary tests (to identify anions) are based upon the fact that:

1. $CO_3^{2-}, S^{2-}, NO_2^-$ and SO_3^{2-} react with dil. H_2SO_4 to give out CO_2, H_2S, NO_2 and SO_2 gases respectively which can be identified by certain tests.

2. $Cl^-, Br^-, I^-, NO_3^-, C_2O_4^{2-}$ and CH_3COO^- react with conc. H_2SO_4 but not with dil. H_2SO_4 to produce characteristic gases.

3. SO_4^{2-} and PO_4^{3-} react neither with dil. H_2SO_4 nor with conc. H_2SO_4. These are therefore, identified by individual tests.

4. Thus, the anions may be identified by performing the following tests in the order given below:
   (i) **Dil. H_2SO_4 test:** Treat a pinch of the salt with dil. H_2SO_4 and identify the gas evolved.
   (ii) **Conc. H_2SO_4 test:** If no action takes place with dil. H_2SO_4, warm a pinch of the salt with conc. H_2SO_4 and identify the gas evolved.
   (iii) **Independent Group (SO_4^{2-} and PO_4^{3-}):** If the salt does not react with dil. H_2SO_4 as well as with conc. H_2SO_4, test for SO_4^{2-} and PO_4^{3-} by performing their individual tests.

Let us now discuss these tests in detail one by one.

Dilute sulphuric acid test

Take a small quantity of the salt in a test tube and add 1–2 mL of dilute sulphuric acid (or dilute HCl). Identify the gas and draw inferences from table below.

Dilute Sulphuric Acid Test

Observations	**Inference**	
	Gas evolved	***Radical***
1. Colourless, odourless gas with brisk effervescence, which turns lime water milky.	CO_2	CO_3^{2-} may be present.
2. Colourless gas with the smell like that of burning sulphur which turns acidified potassium dichromate paper or solution green.	SO_2	SO_3^{2-} may be present.
3. Colourless gas with smell like that of rotten eggs which turns lead acetate paper black.	H_2S	S^{2-} may be present.
4. Reddish brown gas with the pungent smell. The gas turns ferrous sulphate solution black.	NO_2	NO_2^- may be present.
5. No gas evolved.	-	$CO_3^{2-}, S^{2-}, NO_2^-, SO_3^{2-}$ may be absent

Practical tips:

1. *Do not treat the salt with a large quantity of dilute acid.*
2. *Do not heat the salt with dilute acid.*
3. *Some acetates may react with dilute sulphuric acid and produce vapours of acetic acid having vinegar-like smell.*

Chemical reactions involved in Dil. H_2SO_4 test

Dilute H_2SO_4 (or dilute HCl) decomposes carbonates, sulphides and nitrites in cold to give gases. These gases on identification indicate the nature of the acid radical present in the salt.

1. **Carbonates:** On treating the solid carbonate, carbon dioxide gas is given off in the cold with brisk effervescence, which turns lime water milky.

$$CaCO_3 + H_2SO_4 \longrightarrow CaSO_4 + H_2O + CO_2(g)$$
$$Ca(OH)_2 + CO_2 \longrightarrow \underset{\text{Milkiness}}{CaCO_3\ (s)} + H_2O$$

2. **Sulphides:** Sulphides when treated with dil. H_2SO_4 give H_2S gas, which turns lead acetate paper black due to the formation of lead sulphide.

$$ZnS + H_2SO_4 \longrightarrow ZnSO_4 + H_2\ S(g)$$

3. **Sulphites:** On treating solid sulphite with dil. H_2SO_4, SO_2 gas is evolved, which turns potassium dichromate solution green.

$$Na_2SO_3 + H_2SO_4 \longrightarrow Na_2SO_4 + SO_2(g) + H_2O$$
$$K_2Cr_2O_7 + H_2SO_4 + 3SO_2 \longrightarrow K_2SO_4 + \underset{\substack{\text{Chromium sulphate}\\\text{(Green)}}}{Cr_2(SO_4)_3} + H_2O$$

4. **Nitrites:** On treating the solid nitrite with dil. H_2SO_4, nitric oxide (NO) gas is evolved which readily gives dense brown fumes of NO_2 with oxygen of the air.

$$3KNO_2 + 3H_2SO_4 \longrightarrow 3KHSO_4 + HNO_8 + H_2O + 2NO$$
$$\underset{\text{Colourless}}{2NO + O_2} \longrightarrow \underset{\substack{\text{Brown}\\\text{fumes}}}{2NO_2}$$

Concentrated sulphuric acid test

This test is performed by treating small quantity of salt with conc. sulphuric acid (2–3 mL) in a test tube. Identify the gas evolved in cold and then on heating. Observe the changes and draw inferences as given in the following table.

Conc. Sulphuric Acid Test

Observations	Inference	
	Gas evolved	***Radical***
1. Colourless gas with pungent smell, white fumes with aqueous ammonia (NH_4OH), white ppt. with $AgNO_3$ solution.	HCl	Cl^- may be present.
2. Reddish brown vapours with pungent smell, turns starch paper yellow. It does not turn $FeSO_4$ solution black.	Br_2	Br^- may be present.
3. Deep violet vapours with pungent smell, turns starch paper blue. A sublimate is formed on the sides of the tube.	I_2 vapours	I^- may be present.
4. Reddish brown gas with pungent smell, turns $FeSO_4$ solution black.	NO_2	NO_3^- may be present.
5. Colourless vapours with smell of vinegar, turns blue litmus red.	CH_3COOH vapours	CH_3COO^- may be present.
6. A colourless gas which turns lime water milky and also a gas which burns with pale-bluish flame.	$CO_2 + CO$	$C_2O_4^{2-}$ may be present.
7. No gas/vapours evolve.	-	$Cl^-, Br-, I^-, NO_3^-$, CH_3COO^- may be absent.

Practical tips:

1. *If some gas evolves with dilute sulphuric acid, then there is no need for performing conc. sulphuric acid test.*
2. *Some acetates may react with dilute sulphuric acid and give out vapours of acetic acid in dilute sulphuric acid test.*
3. *Do not boil the salt with conc. sulphuric acid. On boiling, the acid may decompose to give SO_2 gas.*
4. *Nitrates give vapours of nitric acid (colourless) when heated with conc. sulphuric acid. When a paper pellet or copper chips is added, dense brown fumes evolve. Paper pellet acts as a reducing agent and reduces nitric acid to NO_2 (Reddish brown gas).*

$$NaNO_3 + H_2SO_4 \longrightarrow NaHSO_4 + HNO_3$$

$$4HNO_3 + C\text{ (from paper pellet)} \longrightarrow 2H_2O + 4NO_2(g) + CO_2(g)$$

Chemical reactions involved in conc. H_2SO_4 test

1. Chlorides

$$NaCl + H_2SO_4 \longrightarrow NaHSO_4 + HCl$$

2. Bromides

$$NaBr + H_2SO_4 \longrightarrow NaHSO_4 + HBr$$

$$H_2SO_4 + 2HBr \longrightarrow SO_2 + Br_2 + 2H_2O$$

3. Iodides

$$KI + H_2SO_4 \longrightarrow KHSO_4 + HI$$
$$H_2SO_4 + 2HI \longrightarrow SO_2 + I_2 + 2H_2O$$

4. Nitrates

$$KNO_3 + H_2SO_4 \longrightarrow KHSO_4 + HNO_3$$
$$HNO_3 + C\ \text{(paper pellet)} \longrightarrow 4NO_2 + CO_2 + 2H_2O$$

5. Acetates

$$CH_3COONa + H_2SO_4 \longrightarrow NaHSO_4 + \underset{\substack{\text{Acetic acid} \\ \text{(Vinegar smell)}}}{CH_3COOH}$$

6. Oxalates

$$Na_2C_2O_4 + H_2SO_4 \longrightarrow Na_2SO_4 + CO_2 + CO + H_2O$$

TESTS FOR INDEPENDENT RADICALS (SO_4^{2-} AND PO_4^{3-})

As already discussed these radicals are not detected by dilute or concentrated H_2SO_4. They are tested individually.

1. Sulphate (SO_4^{2-})

Boil a small amount of salt with dilute HCl in a test tube. Filter the contents, and to the filtrate add few drops of $BaCl_2$ solution. A **white ppt.** insoluble in conc. HCl indicates presence of sulphate.

2. Phosphate (PO_4^{3-})

Add conc. HNO_3 to the salt in a test tube. Boil the contents and add excess of ammonium molybdate solution. **A yellow precipitate** indicates presence of phosphate.

SOLUBILITY OF SOLID SAMPLES

The solubility of different substances in water serves as a useful criterion for their identification. To test solubility, a small amount of the solid sample is placed in a test tube, followed by the addition of distilled water and gentle stirring.

- **If the substance is soluble**, it dissolves completely, forming a clear, homogeneous solution.
- **If the substance is slightly soluble**, only a portion dissolves, leaving some residue behind.
- **If the substance is insoluble**, it remains undissolved, and no noticeable change occurs in the mixture.

General trends for solubility of some compounds in water

Understanding solubility rules is essential for identifying ionic compounds during qualitative analysis. The following general trends summarize the solubility behaviour of common salts in water:

1. All nitrates are soluble
2. All salts of sodium, potassium and ammonium are soluble
3. All chlorides, bromides and iodides are soluble except those of silver, mercury (I) and lead (II)
4. All sulphates are soluble except those of barium and lead (II). Sulphates of calcium and silver are sparingly soluble.
5. All carbonates, sulphites and phosphates are insoluble except those of sodium, potassium and ammonium
6. All sulphites are insoluble except those of alkali metals, the alkaline earth metals and ammonium.
7. All hydroxides are insoluble except those of the alkali metals. The hydroxides of calcium and barium are sparingly soluble. Ammonium hydroxide exists as aqueous ammonia.

The test for solubility of the solid samples is summarized in the table below:

Experiments	**Observations**	**Inferences**
Solubility of a solid sample Transfer about 0.5g of the solid sample into the test tube and add enough cold distilled water to dissolve the sample. If the sample does not dissolve, warm it	Soluble and forming a colourless solution	NO_3^- of Na^+, NH_4^+ may be present
		CO_3^{2-}, HCO_3^-, of Na^+, NH_4^+ may be present.
		Cl^- of Zn^{2+}, Ca^{2+} may be present.
		SO_4^{2-} of Zn^{2+} may be present.
	Soluble forming a blue or green solution	Cu^{2+} may be present
	Soluble and forming a yellowish – brown solution	Fe^{3+} may be present.
	Soluble and forming pale green solution	Fe^{2+} may be present.
	White crystals formed, insoluble in cold water but soluble in hot water and the crystals reappear on cooling	Cl^- of Pb^{2+} may be present.
	Insoluble	CO_3^{2-} of Ca^{2+}, Zn^{2+}, Fe^{2+}, $Fe^{3+}, Cu^{2+}, Pb^{2+}$ may be present
		SO_4^{2-} of Ca^{2+}, Pb^{2+} may be present.

TEST IN SOLUTIONS

To identify the cations, present in a given sample, specific reagents are added to its solution. Two common tests include:

1. Action of sodium hydroxide solution
2. Action of ammonia solution

1. Action of sodium hydroxide solution on sample solutions

This test involves the stepwise addition of dilute sodium hydroxide solution (NaOH) to the sample solution. The procedure is as follows:

Step 1: Add dilute NaOH solution dropwise to the sample solution.

Step 2: Stir or shake the mixture and observe for any precipitate.

Step 3:

- If a precipitate forms, continue adding NaOH in excess and observe any changes.

- If no precipitate forms, gently warm the mixture and observe.

The observations help to identify the cation based on the colour and solubility of the precipitate formed.

The summary of sodium hydroxide's action on different sample solutions is shown in the table below:

Experiments	**Observations**	**Inferences**
Action of NaOH solution on the sample solutions Add sodium hydroxide solution to about 1cm3 of the original sample solution dropwise until excess	White precipitate is formed soluble in excess NaOH	$Zn^{2+}, Pb^{2+}, Al^{3+}$ may be present
	White precipitate is formed insoluble in excess NaOH	Ca^{2+} may be present.
	Blue precipitate is formed insoluble in excess NaOH	Cu^{2+} may be present.
	Green precipitate is formed insoluble in excess NaOH and turns brown on standing	Fe^{2+} may be present.
	Reddish – brown precipitate is formed insoluble in excess NaOH	Fe^{3+} may be present.
	No precipitate is formed. On warming; a colourless gas with a choking smell which turns moist red litmus paper blue evolves	NH_4^+ may be present.
	No precipitate is formed even on warming	Na^+ may be present.

Action of NH_3 solution on sample solutions

To test the effect of ammonia solution on a given sample:

Step 1: Add dilute ammonia solution (NH_3) dropwise to the sample solution.

Step 2: Shake or stir the mixture gently and observe any visible changes.

Step 3: If a precipitate forms, continue adding ammonia solution drop by drop to see whether the precipitate dissolves or remains unchanged.

These observations can help identify specific metal ions based on the nature of the precipitate and its solubility in excess ammonia.

The following table summarizes the action of NH_3 solution on the sample solution.

Experiments	**Observations**	**Inferences**
Action of NH_3 solution on the sample solutions	White precipitate is formed insoluble in excess NH_3	Pb^{2+}, Sn^{2+} may be present.

Add ammonia solution to about 1cm3 of the original sample solution until excess	White gelatinous precipitate is formed soluble in excess NH_3	Zn^{2+} may be present.
	No precipitate is formed	$Ca^{2+}, Na^{+}, NH_4^{+}$ may be present
	Pale blue precipitate is formed soluble in excess NH_3 forming deep blue solution	Cu^{2+} may be present.
	Green precipitate is formed, insoluble in excess and turns brown on exposure of air	Fe^{2+} may be present.
	Reddish – brown precipitate is formed insoluble in excess	Fe^{3+} may be present.

CONFIRMATORY TESTS

Confirmatory tests are performed to verify the presence of specific ions in a sample after preliminary analysis. These tests are crucial for ensuring that the inferences made from earlier observations are accurate and conclusive.

Confirmatory tests are generally grouped into two main categories:

- Confirmatory tests for cations
- Confirmatory tests for anions

Each test uses characteristic reactions that produce distinct changes, such as precipitate formation or colour changes; that help confirm the identity of the ion in question.

Confirmatory tests of selected cations

These tests are carried out to identify specific cations present in a given sample. The procedure involves adding reagents that produce characteristic reactions such as precipitate formation, color changes, or gas evolution. The results confirm the identity of the suspected cation.

A summary of common confirmatory tests for selected cations is shown in the following table:

Experiments	**Observations**	**Inferences**
Confirmatory test for Na^{+} Perform the flame test	Golden yellow flame	Na^{+} confirmed
Confirmatory test for NH_4^{+} Transfer about 0.2g of the original solid sample into a test	Colourless gas evolves which has urine smell and turns moist red litmus paper blue and forms	NH_4^{+} confirmed

tube, add sodium hydroxide solution just to cover the whole solid, then warm gently. Dip a glass rod in concentrated HCl and pass it to the mouth of the test tube containing the mixture. Test the gas evolved with moist red litmus paper.	white fumes with concentrated HCl	
Confirmatory tests for Ca^{2+} i. To about $1cm^3$ of the original sample solution, add ammonia solution followed by ammonia oxalate solution	White precipitate is formed	Ca^{2+} confirmed
ii. Dip a nichrome wire or glass road or base of attest tube into a concentrated hydrochloric acid then to the solid sample. Heat the wire (glass road or base of a test tube) over a non – luminous flame	Burns with a brick red flame	Ca^{2+} confirmed
Confirmatory tests for Pb^{2+} i. Add potassium dichromate solution to about $1cm^3$ of the sample solution	Yellow precipitate is formed	Pb^{2+} confirmed
ii. Add potassium iodide solution to about $1cm^3$ of the sample solution. Warm and cool the mixture	Yellow precipitate disappears on warming but reappear on cooling	Pb^{2+} confirmed
Confirmatory tests for Zn^{2+} i. Add potassium hexacyanoferrate (II) solution to about $1cm^3$ of the sample solution followed by few drops of dilute HCl	Whitish – blue precipitate insoluble in dilute HCl formed	Zn^{2+} confirmed

ii. To about 1cm^3 of the sample solution, add ammonia solution until excess	White gelatinous precipitate soluble in excess ammonia solution formed	Zn^{2+} confirmed
Confirmatory tests for Mg^{2+} To the solution sample add aqueous ammonium oxalate, filter and discard any precipitate i. To the prepared sample solution, add dilute ammonia solution, solid ammonium chloride and disodium hydrogen phosphate.	White crystalline precipitate is formed	Mg^{2+} confirmed
ii. To the prepared sample solution add few drops of Magneson I reagent followed by excess NaOH solution	Sky – blue precipitate is formed	Mg^{2+} confirmed
Confirmatory tests for K^+ i. Perform a flame test	Lilac/light purple flame observed	K^+ confirmed
ii. To the solution of the sample, add sodium hexanitritocobaltate (III) solution followed by ethanoic acid	Yellow precipitate is formed	K^+ confirmed
Confirmatory tests for Cu^{2+} i. To the solution sample, add ammonium solution until excess	Pale blue precipitate formed soluble in excess ammonia solution	Cu^{2+} confirmed
ii. Add few drops of potassium hexacyanoferrate (II) to the solution sample	Reddish – brown precipitate formed	Cu^{2+} confirmed

Confirmatory tests for Fe^{2+}, Fe^{3+} i. Add few drops of potassium hexacyanoferrate (III) to the sample solution ii. Add few drops of hexacyanoferrate (II) to the solution sample iii. Add few drops of potassium thiocyanate or ammonium thiocyanate solution.	Deep blue precipitate formed	Fe^{2+} confirmed
	Light blue precipitate is formed	Fe^{2+} confirmed
	Deep blue precipitate formed	Fe^{3+} confirmed
	Deep blood –red solution is formed	Fe^{3+} confirmed
Confirmatory tests for Al^{3+} Acidify the solution sample with dilute HCl and add few drops of litmus solution followed by ammonia solution	Blue lake precipitate is formed	Al^{3+} confirmed

Confirmatory tests for selected anions

These tests are carried out to identify the anions present in a sample. The procedure involves specific chemical reactions that produce distinctive observations such as precipitates, colour changes, or gas evolution. The results of these confirmatory tests are summarized in the following table:

Experiments	Observations	Inferences
Confirmatory tests for SO_4^{2-} i. To about 1cm^3 of the original sample solution, add barium chloride followed by dilute HCl or barium nitrate followed by dilute HNO_3	White precipitate insoluble in dilute HCl or HNO_3	SO_4^{2-} confirmed
ii. To about 1cm^3 of the sample solution, add ethanoic acid followed by lead ethanoate. Divide the resulting solution mixture into two portions. In one portion, add dilute HCl and in another portion add ammonium ethanoate solution	White precipitate insoluble in dilute HCl but soluble in ammonium ethanoate solution formed	SO_4^{2-} confirmed
Confirmatory tests for NO_3^- i. Transfer a small amount of the original solid sample into a test tube. Add copper turnings followed by concentrated H_2SO_4 along the side of the test tube	Brown fumes evolves	NO_3^- confirmed
ii. Transfer small volume of the sample solution into the test tube. Add freshly prepared $FeSO_4$ solution followed by slowly addition of concentrated H_2SO_4 along the side of the test tube	A brown ring is formed at the junction of the liquid	NO_3^- confirmed
iii. Transfer a small amount of the original solid sample into a test tube. Add Al or Zn metal		

followed by NaOH solution then heat.	A colourless gas with a choking smell which turns moist red litmus paper to blue	NO_3^- confirmed
Confirmatory tests for Cl^- i. Transfer a small volume of the extract into the test – tube. Add dilute HNO_3 followed by $AgNO_3$ solution then ammonia solution	White precipitate soluble in dilute ammonia solution is formed	Cl^- confirmed
ii. Transfer small amount of the original solid sample into the test tube. Add potassium dichromate solution followed by few drops of concentrated H_2SO_4	Red – orange vapour is observed	Cl^- confirmed
iii. Transfer small amount of the original solid sample into the test tube. Add equal amount of solid MnO_2 followed by few drops of concentrated H_2SO_4	Greenish – yellow gas evolves which bleaches mois red litmus paper	Cl^- confirmed
Confirmatory tests for NO_2^- i. Transfer a small volume of the extract into the test tube. Add dilute H_2SO_4 followed by freshly prepared $FeSO_4$ solution	Brown solution is formed	NO_2^- confirmed
ii. Transfer a small volume of the extract into the test tube. Add KI solution followed by concentrated HCl	Dark brown precipitate is formed	NO_2^- confirmed

Confirmatory tests for $C_2O_4^{2-}$ i. Transfer a small volume of the extract into the test tube. Add a small volume of $CaCl_2$ solution dropwise. Divide the resulting mixture into two portions. In one portion add dilute ethanoic acid. In another portion add dilute HCl or dilute HNO_3	White precipitate forms insoluble in dilute ethanoic acid but soluble in dilute HNO_3	$C_2O_4^{2-}$ confirmed
ii. Transfer a small volume of the extract into the test tube. Add dilute H_2SO_4 followed by one drop of potassium permanganate solution and warm	Acidified permanganate is decolorized	$C_2O_4^{2-}$ confirmed
iii. Transfer a small volume of the extract into the test tube. Add $AgNO_3$ solution. If precipitate is formed, add dilute ammonia solution	White precipitate soluble in dilute ammonia solution is formed	$C_2O_4^{2-}$ confirmed.
iv. Transfer a small volume of the extract into the test tube. Add concentrated H_2SO_4	Colourless gas which turns limewater milky and burns with blue flame evolves.	$C_2O_4^{2-}$ confirmed.
v. Transfer a small volume of the extract into the test tube. Add $BaCl_2$ or $Ba(NO_3)_2$ solution. If precipitate is formed, add dilute ammonium chloride or HCl.	White precipitate soluble in dilute ammonia chloride or dilute HCl is formed.	$C_2O_4^{2-}$ confirmed.

Confirmatory tests for $CrO_4^{2-}, Cr_2O_7^{2-}$ Transfer a small volume of the extract into the test tube. Add dilute HNO_3 followed ammonia solution until the solution becomes neutral then boil. i. To neutral solution, add $BaCl_2$ solution followed dilute HCl	Yellow precipitate soluble in dilute HCl.	CrO_4^{2-} confirmed.
ii. To the neutral solution, add $Pb(CH_3COO)_2$ or $Pb(NO_3)_2$ solution.	Yellow precipitate is formed.	CrO_4^{2-}confirmed.
iii. Transfer a small volume of the extract into the test tube. Add few drops of dilute NaOH solution.	The solution changes from orange to yellow.	$Cr_2O_7^{2-}$ confirmed.
iv. To the neural solution, add $Pb(CH_3COO)_2$ or $Pb(NO_3)_2$ solution.	Orange precipitate is formed.	$Cr_2O_7^{2-}$ confirmed.
Confirmatory tests for CH_3COO^- Transfer a small volume of the extract into the test tube. Add dilute HNO_3 followed by ammonia solution until the solution becomes neutral then boil. i. To the neutral solution, add $FeCl_3$ solution.	Deep red colour is observed.	CH_3COO^- confirmed.
ii. To the neutral solution, add concentrated H_2SO_4 followed by ethanol solution	Smell vinegar.	CH_3COO^- confirmed.
Confirmatory tests for CO_3^{2-} and HCO_3^- i. Transfer a small volume of the original sample	White precipitate is formed before warming the contents.	CO_3^{2-} confirmed.

solution into the test tube. Add few drops of $MgSO_4$ solution. If no precipitate is formed, warm the contents		
ii. Transfer a small volume of the original sample solution into the test tube. Add few drops of $CaCl_2$ solution. If no precipitate formed, warm the contents.	White precipitate is formed after warming the contents.	HCO_3^- confirmed.
iii. Transfer a small volume of the original sample solution into the test tube. Add $BaCl_2$ solution followed dilute HCl (or barium nitrate followed by dilute nitric acid)	White precipitate is formed before warming the contents.	CO_3^{2-} confirmed.
iv. Transfer small amount of the original solid sample into the test tube. Add small volume of dilute HNO_3	White precipitate is formed after warming the contents.	HCO_3^- confirmed.
	White precipitate soluble in dilute HCl (or dilute HNO_3) is formed.	CO_3^{2-} confirmed
	Effervescence of colourless gas which turns limewater milky formed.	CO_3^{2-} confirmed

GROUP SEPARATION OF CATIONS IN QUALITATIVE ANALYSIS

Group separation in qualitative analysis is a systematic approach used to identify metal cations in a mixture by classifying them into distinct groups based on similar chemical behaviours—particularly their selective reactions with specific group reagents. This classical method is based on the principles of solubility product equilibria and complex ion formation, which guide the stepwise separation and identification of ions.

By exploiting differences in solubility under controlled conditions, cations are selectively precipitated in groups, making the identification process more efficient, logical, and accurate. Each group is defined by the ease with which its cations precipitate in the presence of a particular reagent. In the following sections, we will examine each cation group individually, along with the specific reagents used for their separation.

Group I: Ag^+, Hg^+ and Pb^{2+}

Chlorides of mentioned ions have very small solubility product, K_{sp}.

So the mentioned ions need very small concentration of chloride Cl^- ions to precipitate their respective chlorides.

The common useful reagent with ensures that small concentration of Cl^- ion for causing precipitation is **dilute hydrochloric acid, HCl.**

(i) $Ag^+(aq) + Cl^-(aq) \rightleftharpoons AgCl(s)$

(ii) $Hg^+(aq) + Cl^-(aq) \rightleftharpoons HgCl(s)$

(iii) $Pb^{2+}(aq) + 2Cl^-(aq) \rightleftharpoons PbCl_2(s)$

The colour of the precipitates (AgCl, HgCl and $PbCl_2$) is white.

The use of dilute hydrochloric acid ensures small production of chloride, Cl^- ions. This has two advantages:

1. It differentiates cations of first group from other cations which need relatively greater concentration of chloride, Cl^- ions to form their respective precipitates.
2. It avoids formation of soluble complex, $[PbCl_4]^{2-}$which is formed in presence of greater concentration of chloride, Cl^- ions produced by concentrated hydrochloric acid (or excess HCl).

Silver chloride, AgCl can be distinguished from other chlorides by the following ways:

1. Adding excess of ammonia, NH_3 solution, it forms the soluble complex, $[Ag[NH_3]_2]^+$ while others chloride do not (the precipitate do not dissolve).
2. Adding solution of potassium cyanide, KCN it forms the soluble complex, $[Ag(CN)_2]^-$ while other chlorides do not.

Group II: $Cu^{2+}, Cd^{2+}, Sn^{2+}, Sn^{4+}, As^{3+}, Sb^{3+}, Bi^{3+}$, and Hg^{2+}

Sulphides of mentioned ions have very small solubility product, K_{sp}.

So the mentioned cations need very small concentration of sulphide, S^{2-} ions to precipitate their respective sulphides.

How the production of the small concentration of sulphide, S^{2-} ions is ensured?

This is done by applying **hydrogen sulphide in the presence of dilute hydrochloric acid.**

The two acids ionise in solution according to the following equations:

(i) $H_2S \rightleftharpoons 2H^+ + S^{2-}$

(ii) $HCl \longrightarrow H^+ + Cl^-$

The presence of strong acid, HCl which ionises completely in the solution, increase the concentration of H^+ shifting the equilibrium (i) to the left hand side, thereby decreasing concentration of sulphide S^{2-}. Hence only those sulphides (sulphides of second group cations) needing low concentration of sulphide, S^{2-} ions will precipitate.

Cu^{2+} and Cd^{2+} can be distinguished from the rest by using the solution of potassium cyanide, KCN whereby the cations are capable of forming their respective soluble complexes and hence their precipitates appear to dissolve.

$$Cu^{2+} + 4CN^- \longrightarrow [Cu(CN)_4]^{2-}$$

$$Cd^{2+} + 4CN^- \longrightarrow [Cd(CN)_4]^{2-}$$

Therefore, CuS and CdS will not precipitate in presence of KCN(aq) while sulphide of other cations of second group precipitate.

Cu^{2+} can be distinguished from the rest by adding excess of ammonia, NH_3 solution whereby it forms the soluble complex, $[Cu(NH_3)_4]^{2+}$ which appears as deep blue solution.

Also in small addition of concentrated hydrochloric acid, CuS does not precipitate due to the formation of $[CuCl_4]^{2-}$.

Group III: $Al^{3+}, Fe^{3+}, Fe^{2+}, Cr^{3+}$

Hydroxides of the mentioned cations have very small solubility product, K_{sp}.

So the cations need very small concentration of hydroxide, OH^- ions to precipitate their respective hydroxides: ***How this is achieved?***

Low concentration of hydroxide, OH^- ions is obtained by using the reagent which is **the mixture of ammonium chloride, NH_4Cl and ammonium hydroxide, NH_4OH solutions.**

NH_4Cl solution being strong salt undergoes complete ionisation while NH_4OH solution being weak base undergoes partial ionisation.

$NH_4OH \rightleftharpoons NH_4^+ + OH^-$ ....(i)

$NH_4Cl \longrightarrow NH_4^+ + Cl^-$ ....(ii)

$NH_4^+ +$ ions from (ii) disturb the position of equilibrium in (i) by common ion effect, shifting the position of equilibrium to the left hand side, thus decreasing concentration of OH^- ions. Hence only those hydroxides (of third group) needing low concentration of hydroxide, OH^- ion will precipitate

- Al^{3+} and Cr^{3+} can be distinguished from the rest by adding excess solution of sodium hydroxide, NaOH.

- **White precipitate** of $Al(OH)_3$ dissolve to gives clear solution of $[Al(OH)_4]^-$ with NaOH(aq)
- **Green precipitate** of $Cr(OH)_3$ dissolve to give green solution of $[Cr(OH)_6]^{3-}$

- **Cr^{3+} can be also be distinguished from the rest** by adding excess solution of ammonia, $NH_3(aq)$

- Only $Cr(OH)_3$ dissolve to give green solution of $[Cr(NH_3)_6]^{3+}$

Tests of Fe^{2+} and Fe^{3+} (from $Fe(OH)_2$ and $Fe(OH)_3$

To test Fe^{2+}, the precipitate ($Fe(OH)_2$) is dissolved in dilute hydrochloric acid followed by addition of potassium hexacyanoferrate (III), $K_3[Fe(CN)_6]$

With $K_3[Fe(CN)_6]$, Fe^{2+} gives deep blue solution. The colour is commonly known as **Turn bull's blue** . The colour is the result of formation of **$K[Fe(Fe(CN)_6)]$**

That is: $Fe(OH)_2(s) + 2HCl(aq) \longrightarrow FeCl_2(aq) + 2H_2O(l)$

Then $Fe^{2+} + [Fe(CN)_6]^{3-} \longrightarrow [Fe(Fe(CN)_6)]^-$

(From $FeCl_2$) (from $K_3[Fe(CN)_6]$,) Turn bull's blue (deep blue)

Fe^{3+} can be tested by firstly dissolving the precipitate ($Fe(OH)_3$), then followed by addition of potassium hexacyanoferrate (II), $K_4[Fe(CN)_6]$ or ammonium thiocyanate, NH_4SCN

- **With $K_4[Fe(CN)_6]$,** Fe^{3+} gives deep blue solution of $K[Fe(Fe(CN)_6)]$. In this case the colour is commonly known as **Prussian blue** (to differentiate to the deep blue colouration formed in the case of Fe^{2+}).
- With NH_4SCN, Fe^{3+} gives bloody red colouration of $[Fe(H_2O)_5SCN]^{2+}$

That is $Fe(OH)_3 + 3HCl \longrightarrow FeCl_3 + 3H_2O$

Then $Fe^{3+}(aq) + [Fe(CN)_6]^{4-} \longrightarrow \left[Fe\left(Fe(CN)\right)_6\right]^-$

(From $FeCl_3$) (From K_4 $[Fe(CN)_6]$ Prussian blue (deep blue)

And $[Fe(H_2O)_6]^{3+} + SCN^- \longrightarrow [Fe(H_2O)_5(SCN)]^{2+}$

(From $Fe^{3+}(aq)$) Bloody red colour

Group IV: $Ni^{2+}, Co^{2+}, Mn^{2+}, Zn^{2+}$

Compared to cations of second group, the above mentioned **cations need relatively greater concentration of sulphides, S^{2-} ions to precipitate their respective sulphides.**

How the greater concentration of sulphide ions is ensured?

Greater concentration of sulphide, S^{2-} ions is obtained by using **ammonium sulphide, $(NH_4)_2S$ only or hydrogen sulphide, H_2S in the presence of ammonium hydroxide, NH_4OH solution.**

(i) By using $(NH_4)_2S$

Ammonium sulphide, $(NH_4)_2S$ being strong salt ionises completely to give large concentration of S^{2-} ions according to the following equation:

$$(NH_4)_2S \longrightarrow 2NH_4^+ + S^{2-} \ldots\ldots\ldots\ldots\ldots\ldots\text{(i)}$$

Sulphide of the fourth group cations, being slightly soluble, ionises according to the following equation:

$$M(s) \rightleftharpoons M^{2+}(aq) + S^{2-}(aq) \ldots\ldots\ldots\ldots\ldots\ldots\text{(ii)}$$

Where M may be Ni, Co, Mn, or Zn

Ammonium sulphide, $(NH_4)_2S$ increases concentration of sulphide, S^{2-} ions, shifting the position of equilibrium in (ii) towards the left hand side and therefore forming precipitation of the sulphide. Hence **sulphides (of fourth group cations) needing large concentration of S^{2-} ions will precipitate**

(ii) By using H_2S in presence of NH_4OH

Both H_2S and NH_4OH being weak acid and weak base respectively, undergo partial ionisation in solution according to the following equation:

$$H_2S \rightleftharpoons 2H^+ + S^{2-} \ldots\ldots\ldots\ldots\ldots\ldots\text{(i)}$$

$$NH_4OH \rightleftharpoons NH_4^+ + OH^- \ldots\ldots\ldots\ldots\ldots\ldots\text{(ii)}$$

Hydrogen protons, H^+ from (i) combine with hydroxide, OH^- ions from (ii) to form water, H_2O according to the following equation:

$$H^+ + OH^- \longrightarrow H_2O$$

This decreases concentration of sulphide, S^{2-} ions, shifting the position of equilibrium in (i) towards the right had side by producing more sulphide, S^{2-} ions and **cations (of fourth group) needing large concentration of the sulphide, S^{2-} ions will precipitate**

*To distinguished the sulphides, the precipitates are firstly dissolved in mineral acids, (**to produce large concentration of $Ni^{2+}, Co^{2+}, Mn^{2+}$, or Zn^{2+} in the solution**) followed by:*

(i) Addition of NaOH(aq)

Zn^{2+} firstly give **white precipitate of** $Zn(OH)_2$ which dissolve in excess NaOH(aq) to give **clear solution** due to formation of $[Zn(OH)_4]^{2-}$

(ii) Addition of $NH_3(aq)$

- Zn^{2+} gives **clear solution** of $[Zn(NH_3)_4]^{2+}$
- Ni^{2+} gives **deep blue solution** of $[Ni(NH_3)_6]^{2+}$

(iii) Addition of concentrated HCl

Co^{2+} gives **blue solution** of $[CoCl_4]^{2-}$

Group V: $Ca^{2+}, Ba^{2+}, Sr^{2+}$

This is the only case where complex formation has no application on the **cationic test** (**Note:** All Ca^{2+}, Ba^{2+} and Sr^{2+} are incapable of making complexes).

Reagent used for test: Ammonium carbonate, $(NH_4)_2CO_3$

$$(NH_4)_2CO_3 \longrightarrow 2NH_4^+ + CO_3^{2-}$$

With the carbonate, all Ca^{2+}, Ba^{2+} and Sr^{2+} gives white precipitates of $CaCO_3, BaCO_3$ and $SrCO_3$ respectively.

Cation groups and their separation

Group	**Group Reagent**	**Ions Separated**	**Type of Precipitate**
Group I (Silver Group)	Dilute HCl (cold)	Ag^+, Pb^{2+}, Hg_2^{2+}	Chlorides
Group II (Copper-Arsenic Group)	H_2S in acidic medium	Cu^{2+}, Pb^{2+}, Bi^{3+}, Cd^{2+}, Hg^{2+}, As^{3+}, Sb^{3+}, Sn^{4+}	Sulphides in acidic medium
Group III (Iron Group)	$NH_4OH + NH_4Cl$ (buffered alkaline medium)	Fe^{2+}, Fe^{3+}, Cr^{3+}, Al^{3+}	Hydroxides
Group IV (Zinc Group)	H_2S in alkaline medium or $(NH_4)_2S$	Zn^{2+}, Mn^{2+}, Ni^{2+}, Co^{2+}	Sulphides in basic medium
Group V (Alkaline Earth Group)	$(NH_4)_2CO_3$	Ba^{2+}, Sr^{2+}, Ca^{2+}	Carbonates
Group VI (Alkali Metals)	No precipitate with above reagents	Na^+, K^+, NH_4^+	Identified by flame or specific tests

EXPERIMENTS

Experiment 41

As part of your project at **HOPEGEN Chemical Industries**, you were assigned a simple but crucial task: verify the ionic content of a sample from the company's production line.

The supervisor slid a container across the bench and said:

"This ***sample Q*** *is a mixed product from the coating department. If it's what we think, it's a mix of anti-rust compound and a pH buffering agent. Confirm its composition—don't let the factory pack the wrong formula."*

Your task is to use the given information to complete the observations and inferences column, hence identify the two anions and the anion present in **Q**.

S/N	Experiments	Observations	Inferences
a)	Observe the appearance of the sample		
b)	Put a spatula full of sample Q in a dry test tube and heat		
c)	Dissolve a little amount of sample Q in distilled water and divide the resulting solution into four portions		
	i. To the first portion, add $AgNO_3$ followed by HNO_3		
	ii. To the second portion add $MgSO_4$		
	iii. To the third portion add $K_4[Fe(CN)_6]$		
	iv. To the forth portion add dilute NaOH		
d)	Perform confirmatory test for the cation		

Conclusion

i. The anions present in sample Q are __________ and ____________
ii. The common cation present was ____________
iii. The two salts mixed were __________ and ___________

Solution

S/N	Experiments	Observations	Inferences
a)	Appearance of sample i. Colour	White colour observed	Non – transition elements may be present
	ii. Texture	Powder form was observe	CO_3^{2-}, HCO_3^- may be present except that of NH_4^+
b)	A small amount of sample Q was transferred into a dry test tube then heated	Colourless gas evolved which turns lime water milky	CO_3^{2-}, HCO_3^- may be present
c)	A small amount of sample Q was put into the test tube then distilled water was added. The resulting solution was divided into four.	Insoluble in water.	CO_3^{2-} may be present except those of Na^+, K^+, NH_4^+
	i. To the first portion, $AgNO_3$ was added followed by HNO_3	White precipitate formed.	Cl^- confirmed
	ii. To the second portion, $MgSO_4$ was added	White precipitate formed before warming the contents	CO_3^{2-} confirmed
	iii. To the third portion, $K_4[Fe(CN)_6]$ was added	Bluish – white precipitate formed.	Zn^{2+} may be present
	iv. To the forth portion dilute NaOH was added.	White precipitate soluble in excess was formed	Zn^{2+} may be present
d)	Confirmatory test To the solution sample Q, dilute NaOH was added until excess	White precipitate soluble in excess was formed	Zn^{2+} confirmed

Conclusion

i. The anions present in sample Q are Cl^- and CO_3^{2-}
ii. The common cation present was Zn^{2+}
iii. The two salts mixed were $ZnCl_2$ and $ZnCO_3$

Experiment 42

As the lab intern at **IZANGA Fertilizers Ltd.**, you were wrapping up a quiet afternoon when the Quality Control Manager rushed into the lab with a worried look.

"Someone mislabeled one of our storage containers. It could be an essential dual-nutrient compound or a costly error waiting to happen. We suspect it contains a blend of supplements, but we can't risk packaging it without verification."

She handed you a powdered **Sample J** and added, *"All we know is that it contains one anion and two different cations. Use your qualitative analysis skills to confirm if this is our standard nutrient salt or a mistake from storage."*

Armed with reagents and your record sheet, **your mission is to complete the investigation**, fill in the observation and inference columns, and **identify** ions present in **J**.

S/N	**Experiments**	**observations**	**Inferences**
a)	Appearance of Sample J		
b)	Heat a small portion of sample J in a dry test tube		
c)	Prepare a solution of sample J and divide the resulting solution into four portions i. To the first portion, add NaOH and warm gently.		
	ii. To the second portion, add $AgNO_3$ solution.		
	iii. To the third portion, add few drops of potassium hexacyanoferrate (III)		
	iv. To the forth portion, add ethanoic acid followed by lead ethanoate		
d)	To a little amount of sample J in a dry test tube, add dilute sodium hydroxide		

Conclusion

i. The cations present in the sample J were ________ and _________
ii. The anion present in sample J was ________
iii. The molecular formula of sample J was
iv. Write down the reaction equation taking place in (d)

Solution

S/N	**Experiments**	**observations**	**Inferences**
a)	Appearance of Sample J	The sample was pale green crystal	Transition metal ions $Fe^{2+}, Fe^{3+}, Cu^{2+}$ may be present
b)	On heating a small portion of sample J in a dry test tube	A colourless gas which turned a moist red litmus paper blue and white dense fumes formed when a glass rod dipped in concentrated hydrochloric acid and heated was observed.	Compounds of NH_4^+ may be present.
c)	On dissolving sample J in water.	The sample was clearly soluble in water	SO_4^{2-}, Cl^-, NO_3^- of NH_4^+, Cu^{2+} may be present
	i. To the first portion, NaOH was added and warmed gently.	A colourless gas with urine smell was evolved which turned a red litmus paper blue	NH_4^+ confirmed
	ii. To the second portion, $AgNO_3$ solution was added.	No precipitate was observed	Cl^- was absent
	iii. To the third portion, few drops of potassium hexacyanoferrate (III) solution was added	Deep blue precipitate was observed	Fe^{2+} confirmed
	iv. To the forth portion, ethanoic acid was added followed by lead ethanoate	White precipitate insoluble upon addition of lead ethanoate was observed	SO_4^{2-} confirmed
d)	To a little amount of sample J in a dry test tube, dilute sodium hydroxide was added	Greenish precipitate was observed which turned to brown on standing	Fe^{3+} present upon oxidation.

Conclusion

i. The cations present in the sample J were NH_4^+ and Fe^{2+}
ii. The anion present in sample J was SO_4^{2-}
iii. The molecular formula of sample J was $(NH_4)_2Fe(SO_4)_2$
iv. The reaction equation taking place in (d)

$$Fe^{2+}(aq) + 2OH^-(aq) \longrightarrow Fe(OH)_2(s)$$

Experiment 43

As part of your internship in the Quality Control Division at SALAMA Detergents Ltd., you were handed a powder labelled **Sample H** for component verification. This sample had been flagged during batch testing due to inconsistencies in pH buffering and foaming properties.

Your task was clear: identify the two anions and the common cation present in Sample H. The factory suspected the mixture might be a blend of compounds responsible for water softening and pH regulation—key ingredients in powdered detergents. Any error in composition could lead to customer complaints or machine damage, so your analysis had to be sharp and accurate.

You proceeded to perform the qualitative analysis outlined in the table, carefully recording observations and inferences to deduce the identities of the ions involved.

S/N	Experiments	Observations	inferences
1.	Appearance of sample H		
2.	To a little solid sample H in dry test tube, add dilute HCl. Warm gently and observe any gas evolves		
3.	Heat a little amount of a solid sample H in a dry test tube, place a wet litmus paper on the mouth of the test tube		
4.	Dissolve sample H in distilled water and divide the resulting solution into two portions		
	i. To the first portion, add NaOH solution until excess		
	ii. To the second portion, add $BaCl_2$ followed by dilute HCl		
5.	Perform a confirmatory test for cation		

Conclusion

a) In the sample H:
   i. Anions are __________ and _________
   ii. The cation is _________
b) Write the molecular formula for sample H
c) Write the chemical equation for the reaction taking place in experiment (3)

Solution

S/N	Experiments	Observations	inferences
1.	Appearance of sample H	White crystals observed	Transition metals may be absent
2.	To a little solid sample H in dry test tube, dilute HCl was added and warmed.	Effervescence observed and the gas evolved which turned lime water milky	CO_3^{2-} confirmed
3.	A little amount of a solid sample H was heated in a dry test tube, and a wet litmus paper was placed on the mouth of the test tube	A colourless gas with pungent smell that turned blue litmus paper red	SO_4^{2-}, SO_3^{2-} may be present
4.	Sample H was dissolved in distilled water.	Sample H was soluble in water.	SO_4^{2-} of Na^+, K^+ may be present
	i. To the first portion, add NaOH solution until excess	No precipitate was formed	Na^+, K^+ may be present
	ii. To the second portion, $BaCl_2$ was added followed by dilute HCl	White precipitate insoluble in dilute HCl observed	SO_4^{2-} confirmed
5.	A nichrome wire was dipped in concentrated HCl then in the solid sample H and heated in non – luminous flame	A golden yellow flame was observed	Na^+ confirmed

Conclusion

a) In the sample H:
   i. Anions are SO_4^{2-} and CO_3^{2-}

   ii. The cation is Na^+

b) The molecular formula for sample H Na_2SO_4 and Na_2CO_3

c) The chemical equation for the reaction taking place in experiment (3)

$$Na_2SO_4(s) \underset{\Delta}{\rightarrow} Na_2O(s) + SO_3(g)$$

Experiment 44

As part of a regulatory compliance audit at MALUCHEM Fertilizer Plant, you—an analytical chemist trainee, were assigned to assess a critical raw material labeled **Sample T**. This sample was suspected to be a blend of compounds used both for nitrogen enrichment and as stabilizers in the plant's granulated fertilizer line.

Although it appeared like a standard intermediate product, concerns were raised about possible chloride contamination, which could corrode processing equipment if left undetected.

Your objective was to carry out qualitative analysis, following the procedures provided in the table below.

S/N	Experiments	Observations	Inferences
1)	Observe the appearance of sample T		
2)	Heat small amount of sample T in a dry test tube.		
3)	Place a spatula full of sample T in a test tube, add concentrated sulphuric acid.		
4)	Place a spatula full of sample T in a test tube, add sodium hydroxide solution until excess		
5)	Prepare a solution of sample T and divide it into two portions		
	i. To the first portion add barium chloride solution		
	ii. To the second portion, add silver nitrate solution.		

Conclusion

i. Write down the ionic equations which take place in stage 5(i) and 5(ii) above.
ii. Identify the cation present in the sample
iii. Identify the anions present in the sample

Solution.

S/N	Experiments	Observations	Inferences
1)	The appearance of sample T	White crystals observed	Transition metals may be present
2)	A little amount of sample T in a dry test tube was heated.	Colourless gas with urine smell which turned red litmus paper blue was evolved.	NH_4^+ Confirmed.
3)	A concentrated sulphuric acid was added to a spatula full of sample T in a test tube	Colourless gas with irritating smell evolved which turned a moist blue litmus paper red and forms dense fumes with ammonia gas.	Cl^-, SO_4^{2-} may be present.
4)	To a spatula full of sample T in a test tube, sodium hydroxide solution was added until excess	No precipitate formed	Cl^-, SO_4^{2-} of NH_4^+ may be present
5)	A small amount of sample T was dissolved in water.	Soluble in water	Cl^-, SO_4^{2-} of NH_4^+ may be present
	i. To the first portion, barium chloride solution was added.	White precipitate was formed	SO_4^{2-} confirmed
	ii. To the second portion, silver nitrate was added.	White precipitate was formed.	Cl^- confirmed

Conclusion

i. Ionic equation in:
Stage 5(i)

$$Ba^{2+}(aq) + SO_4^{2-}(aq) \longrightarrow BaSO_4(s)$$

Stage 5(ii)

$$Ag^+(aq) + Cl^-(aq) \longrightarrow AgCl(s)$$

ii. The cation present in the sample was NH_4^+

iii. The anions present in sample T were SO_4^{2-} and Cl^-

Experiment 45

As part of your routine check in the quality control lab of UHAKIKA Agrochemicals Ltd, you received **Sample X**, a fine powder used in the formulation of foliar feeds. Recent complaints from farmers claimed that the product caused a sharp smell when mixed with water and left faint white residues on spray tanks.

Your task was simple but vital: determine the identity of the ions present in **Sample X** to verify its integrity before it could be cleared for packaging.

You carried out a series of qualitative tests using the standard laboratory procedures outlined below.

a) Appearance of the sample
b) Action of heat on the sample
c) Solubility of the sample on water
d) Action of aqueous sodium hydroxide on the solution of sample X
e) Action of freshly prepared iron (II) sulphate solution on the solution of sample X followed by addition of concentrated sulphuric acid through the side of the test tube.
f) Action of lead ethanoate followed by boiling
g) Perform the confirmatory test for the cation and anion.

Questions

i. Prepare a relevant table showing the qualitative analysis results.
ii. Write a balanced chemical equation for the reaction in experiment (b)
iii. Write the chemical formula of the sample

Solution

S/N	Experiments	Observations	Inferences
a)	Appearance of sample X	White crystals are observed	Non – transition elements may be present
			NO_3^-, SO_4^{2-}, Cl^- may be present
b)	A small amount of solid sample X was placed on a dry test tube and heated	White sublime and colourless gas with choking smell which turned moist red litmus paper blue observed	NH_4^+ may be present
		Colourless gas which turned moist blue litmus paper red evolved.	Cl^- may be present
c)	A small amount of sample X was dissolved.	The sample was soluble in cold water.	Cl^- may be present except those of $Ag^+, Pb^{2+}, Na^+, K^+, NH_4^+$
d)	To the little amount of solution sample X, NaOH solution was added until excess	No precipitate formed.	NH_4^+ may be present.

e)	To the little amount of solution sample X, freshly prepared $FeSO_4$ was added followed by concentrated sulphuric acid.	No brown ring is observed, but a gas which turned moist blue litmus paper red and forms white fumes on addition of concentrated sulphuric acid	NO_3^- is absent
f)	To the little amount of solution sample X, lead ethanoate then the mixture was boiled	White precipitate was formed which dissolved slowly on boiling.	Cl^- of Pb^{2+} may be present.
g)	i. To a small of sample X, dilute NaOH was added and the moist litmus paper is passed to the mouth of the test tube containing the mixture, then the glass rod was dipped in concentrated hydrochloric acid and passed on the mouth of the test tube containing the mixture.	Colourless gas which turned red litmus paper blue and white fumes with concentrated HCl evolved	NH_4^+ confirmed.
	ii. To a little volume of solution sample X, silver nitrate was added followed by dilute nitric acid	White precipitate soluble in dilute nitric acid	Cl^- confirmed

i. A balanced chemical equation in (b)

$$NH_4Cl(s) \xrightarrow[\Delta]{} NH_3(g) + HCl(g)$$

ii. The chemical formula for the sample X was NH_4Cl.

Experiment 46

You were contracted as a freelance laboratory analyst by BYAKATONDA Industrial Coatings Ltd. A shipment labelled **"Substance Z"** had just arrived from a new supplier. This batch was intended for the formulation of a dual-purpose primer known to resist both corrosion and chemical attack. However, before the mixing tanks could be engaged, quality assurance requested urgent verification of its chemical identity.

The technical team suspected the mixture contained metal salts for anti-rust properties and possible pH-sensitive components. You were handed the investigation sheet and asked to identify all ions present using classical qualitative analysis.

S/N	Experiments	Observations	Inferences
1.	Put a spatula full of sample Z into a boiling test tube and add distilled water. Boil the mixture for about 1 minute then filter or centrifuge the mixture to obtain the residue and clear solution. Divide the resulting clear solution into two portions.		
	i. To the first portion, add sodium hydroxide solution till excess.		
	ii. To the second portion, add dilute nitric acid followed by silver nitrate		
2.	To a little amount of the residue in step 1, add hydrochloric acid		
1.	Dilute the resulting solution in step 2 with distilled water and divide the resulting solution into two portions		
	i. To the first portion, add dilute sodium hydroxide solution		
	ii. To the second portion, add dilute ammonia solution		
2.	Perform one confirmatory test for each ion.		

Conclusion

The two cations in sample Z are _____ and ______

The two anions are _______ and _________

Solution

S/N	Experiments	Observations	Inference
.	A spatula full of sample Z was put into a boiling test tube then distilled water was added. The mixture was boiled for about 1 minute then filtered to obtain the residue and clear solution. The resulting clear solution was divided into two portions.	Insoluble in cold water	CO_3^{2-} may be present except those of Na^+, K^+, NH_4^+
	i. To the first portion, sodium hydroxide solution was added till excess.	White precipitate soluble in excess NaOH was formed.	$Zn^{2+}, Pb^{2+}, Sn^{2+}, Al^{3+}$ May be present
	ii. To the second portion, dilute nitric acid was added followed by silver nitrate	White precipitate was observed	Cl^- confirmed
.	To a little amount of the residue in step 1 hydrochloric acid was added	Effervescence of a colorless gas was observed	CO_3^{2-}, HCO_3^- may be present
.	The resulting solution in step 2 was diluted with distilled water and divided into two portions To the first portion, dilute sodium hydroxide solution was added	Reddish brown precipitate insoluble in excess was observed.	Fe^{3+} may be present
	To the second portion, dilute ammonia solution was added	Reddish brown precipitate insoluble in excess was observed	Fe^{3+} may be present
.	**Confirmatory tests** To a small amount of sample solution in a test tube, potassium ferrocyanide were added	Bluish white precipitate was formed.	Zn^{2+} confirmed
	To a small amount of sample solution in a test tube, few drops of potassium ferrocyanide were added	Dark blue precipitate was formed.	Fe^{3+} confirmed.
	To a small volume of the original sample, $BaCl_2$ was added followed by dil. HCl	White precipitate soluble in dilute HCl was formed.	CO_3^{2-} confirmed

Conclusion

The two cations in the sample are Zn^{2+} and Fe^{3+}

The anions are Cl^- and CO_3^{2-}

Experiment 47

As part of your internship at **MUSHU Pharmaceuticals Ltd**, you were called into the quality control lab to verify the identity of a white crystalline substance labelled **Sample M**. This salt was extracted from a purification line used in the production of antacid tablets.

The production engineer suspected it was a salt, commonly used to stabilize active ingredients and regulate moisture in formulations. However, the shipment lacked proper labelling, and regulatory compliance demanded clear identification before packaging.

Your task is to carry out the qualitative analysis procedures described in the table. Carefully record your **observations** and **inferences** and hence identify the cation and anion present in the sample.

S/N	Experiments	Observations	Inferences
1.	Appearance of sample M		
2.	To a small amount of the solid sample M in a dry test tube, add few drops of conc. H_2SO_4 .		
3.	Perform the flame test.		
4.	Dissolve the spatula full sample in $50cm^3$ of distilled water		
5.	To a little volume of solution sample, add three drops of dilute NaOH then add until excess and warm the mixture.		
6.	To a little volume of solution sample, add dil. HNO_3 followed by $AgNO_3$ solution.		
7.	To a little volume of solution sample, add ammonia oxalate solution.		
8.	To a little volume of solution sample, add $Ba(NO_3)_2$ solution followed by dil. HNO_3		
9.	To a little volume of solution sample, add potassium iodide solution.		

Conclusion

i. The cation present is _________
ii. The anion present is _________
iii. Write an ionic equation for the first observation of the reactions in experiment (7).

Solution

S/N	Experiments	Observations	Inferences
1.	Appearance of sample M	White crystals with no choking smell.	$NO_3^-, Cl^-, SO_4^{2-}, C_2O_4^{2-}$ of Na^+, Pb^{2+}, Ca^{2+} may be present
2.	To a small amount of the solid sample M in a dry test tube, few drops of conc. H_2SO_4 was added.	Colourless gas with irritating smell evolved which turned moist blue litmus paper red.	Cl^- may be present
3.	A small amount of a solid sample was heated in a dry test tube.	Brick red flame observed	Ca^{2+} confirmed.
4.	The spatula full sample was dissolved in $50cm^3$ of distilled water	The sample was soluble in cold water	NO_3^-, Cl^-, SO_4^{2-} may be present.
5.	To a little volume of solution sample, three drops of dil. NaOH was added then until excess and the mixture was warmed.	White precipitate which are insoluble in excess NaOH was observed.	Sr^{2+}, Ba^{2+} may be present.
6.	To a little volume of solution sample, $AgNO_3$ solution was added followed by dil. HNO_3 .	White precipitate insoluble in dil. HNO_3	Cl^- confirmed.
7.	To a little volume of solution sample, ammonia oxalate solution was added.	White precipitate observed	Ca^{2+} is present.
8.	To a little volume of solution sample, $Ba(NO_3)_2$ solution was added followed by dil. HNO_3	No white precipitate formed.	SO_4^{2-} absent
9.	To a little volume of solution sample, potassium iodide solution was added.	No yellow precipitate formed.	Pb^{2+} absent

Conclusion

i. The cation present was Ca^{2+}
ii. The anion present was Cl^-
iii. The ionic equation in step (7)

$$Ca^{2+}(aq) + C_2O_4^{2-}(aq) \longrightarrow CaC_2O_4(s)$$

APPENDIX

A: ATOMIC MASSES OF SELECTED ELEMENTS

Element name	Symbol	Atomic mass (g/mol)
Hydrogen	H	1
Carbon	C	12
Nitrogen	N	14.
Oxygen	O	16
Sodium	Na	23
Magnesium	Mg	24
Aluminum	Al	27
Sulphur	S	32
Chlorine	Cl	35
Potassium	K	39
Calcium	Ca	40
Iron	Fe	56
Copper	Cu	63.6
Barium	Ba	137
Iodine	I	127

B: COMMON HYDRATED COMPOUNDS

Compound name	Chemical formula
Copper(II) sulfate pentahydrate	$CuSO_4.5H_2O$
Iron(II) sulfate heptahydrate	$FeSO_4.7H_2O$
Sodium carbonate decahydrate	$Na_2CO_3.10H_2O$
Oxalic acid dihydrate	$H_2C_2O_4.2H_2O$
Ammonium iron(II) hexahydrate (Mohr's salt)	$(NH_4)_2Fe(SO_4)_2.6H_2O$
Sodium thiosulphate pentahydrate	$Na_2S_2O_3.5H_2O$