A Simplified Approach to A' Level

CHEMISTRY PRACTICALS

P525/3

(Chemistry Practical)

With Guiding Notes, Worked Out Examples and a Variety of Systematic Practical Exercises for Senior Five and Six on:

- *Preparation of standard solutions*
- *Standardization of solutions*
- *Double Indicator Titrations*
- *Redox Titrations*
- *Back Titration*
- *Solubility Product*
- *Partition Coefficient*
- *Inorganic Qualitative Analysis*
- *Organic Qualitative Analysis*
- *Chemical Energetics*
- *Chemical Kinetics*
- *Colligative Properties*

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Fourth Edition

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DEDICATION

I dedicate this book to my Beloved Biological Brothers and Sisters who include: Merry Baruuli Akiiki, Esther K. Yalibanda Abooki, Elijah Baruuli Amooti, Enoch Baruuli Adyeri, Stephen Magezi Ateenyi, Joseph Kirungi Abooki, Ellen Asiimwe Amooti and Joy Basemera Akiiki.

I also dedicate this book to my Beloved Paternal Uncle Paul Ngonzi Atwooki and my Brother-in-law John MwavuYalibanda Atwooki.

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PREFACE TO FOURTH EDITION

A Simplified Approach to A' Level Chemistry Practicalshas been specially designed to meet the requirements of students being prepared for the A' Level Chemistry Practical Examination. The book will also be extremely relevant to Chemistry teachers whose dream is to acquire experience in teaching the Chemistry Practical Paper at A' Level.

The publication is composed of guiding notes at the beginning of every chapter, worked out examples and practical exercises which have been arranged systematically from simpler to more complex ones, all of which are precisely in line with the most current UNEB Syllabus for the A' Level Chemistry Practical Paper (P525/3). The book covers areas of: Preparation of Standard solutions, Standardization of solutions, Double Indicator titrations, Redox Titrations, Back Titration, Solubility Product, Partition Coefficient, Inorganic Qualitative Analysis, Organic Qualitative Analysis, Chemical Energetics, Chemical Kinetics and Colligative Properties.
The author has particularly prepared the practical exercises taking into account, the most recent techniques in setting the A' Level Chemistry Practical Questions, making it **theBest Chemistry Practical Workbook that each Secondary School should acquire for use by Senior Five and Senior Six students.**

This book will also be useful in teaching A' Level Chemistry Practicals throughout East Africa.

The approach used by the author makes the book much more user friendly to both students and teachers than any other previous publication. The language and arrangement of content in the notes, worked out examples and particularly in the procedures of the practical exercises/experiments is the kind that is easily interpreted and understood by students.

Another unique property of this publication is the **Teacher'sPreparation Manual** that will be availed to every institution that will choose this book for its A' Level students. The Teacher's preparation manual contains the confidential information for all the experiments in the twelve chapters of the book. This will simplify the work of the Chemistry Teachers which will ultimately save on the time that would be wasted in preparing for the practical lessons.

This book is therefore intended for any School and Chemistry Teacher whose aim is to have excellent students in the A' Level Chemistry Practical Paper **after all success comes to those who utilize all the opportunities available (such as the opportunity to utilize this book), so as to reach their full potential.**

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TABLE OF CONTENTS

CHAPTER ONE
1.0 INTRODUCTION TO VOLUMETRIC ANALYSIS (QUANTITATIVE ANALYSIS)

1.1 Definition of Volumetric Analysis

Volumetric analysis is an experimental method of determining the concentration of a substance in a given solution by use of another substance in another solution whose concentration is accurately known. In other words, volumetric analysis involves two solutions, one is a standard solution (solution of accurately known concentration) and the other is of unknown concentration. The number of moles in the volume of the standard solution used are calculated and then by use of an appropriate stoichiometry (reaction mole ratio), basing on a balanced equation, the concentration of the substance under analysis can be calculated.

1.2 The Titration Process

Volumetric analysis is carried out through a process known as titration which involves running one solution from a burette into a known volume of the other solution in a conical flask until the two solutions have just reacted completely. The point at which the two solutions have just reacted completely is known as the end point. In case it is an acid-base titration, the end point is detected using an indicator such as phenolphthalein indicator and/or methyl orange indicator. During analysis of aqueous iodine, starch solution is used as an indicator yet analysis of some reactants such as acidified potassium manganate(VII) does not require use of indicators; such reactants are said to be self indicators.

1.3 Applications of Volumetric Analysis

Volumetric analysis is applied in the:

- Standardization of solutions.
- Determination of relative formula masses and relative atomic masses.
- Determination of number of molecules of water of crystallisation for hydrated compounds.
- Determination of percentage purity and percentage of the impurity in various compounds.
- Determination of the stoichiometry (reaction mole ratio) of reactants.
- Determination of the Basicity of an acid.
- Determination of the partition coefficient, K_D.
- Determination of the solubility product, K_{sp}.

Volumetric analysis includes.the following categories of reactions:

- Acid-base titrations (neutralisation reactions).
- Redox titrations.
- Reactions that involve formation of complex ions.
- Reactions that involve formation of precipitates.

1.4 Terms commonly used in Volumetic Analysis

a) The Mole
The mole is the amount of a substance which contains as many elementary units as there are atoms in 12 grams of the carbon-12 isotope. It is abbreviated as **mol.**

b) Molar mass
Molar mass is the mass of one mole of a substance. It is calculated by summing up the atomic masses of the constituent elements in the chemical formula of the compound. Its units are grams (g) or grams per mole ($gmol^{-1}$).
For example: Calculate the molar mass of:

i) Sodium hydroxide, NaOH. (Na=23, O=16, H=1)
= [(23x1) + (16x1) + (1x1)] g
= (23 + 16 + 1) g
= 40g

ii) Copper(II) sulphate pentahydrate, $CuSO_4.5H_2O$. (Cu=64, S=32, O=16, H=1)
= [(64x1) + (32x1) + (16x4) + (1x5x2) + (16x5)] g
= (64 + 32 + 64 + 10 + 80) g
= 250g

c) Relative Formula Mass
It is determined in the same way as for molar mass except that the answer is recorded with no units.

d) Concentration
Concentration is the number of grams of a solute dissolved in a solvent to make one litre (one cubic decimeter) of solution **OR:**
The number of moles of a substance dissolved in a solvent to make one litre (one cubic decimetre) of solution.

e) **Molar solution**
It is a solution which contains one mole of a substance in one litre (one cubic decimetre) of solution.

f) Molarity
Molarity is the number of moles of a substance in one litre (one cubic decimetre) of solution.

g) Titrant
The reagent used in volumetric analysis, whose concentration is accurately known.

h) Titrand
The reagent used in volumetric analysis, whose concentration is to be determined.

i) End point
It is a point reached during titration when the substances in the two solutions provided have just reacted completely. For acid-base titrations, the end point is detected using an indicator.

j) Indicators
An indicator is a substance whose colour changes according to the hydrogen ion concentration of the solution to which it has been added. The table below shows the commonly used indicators, their colour in neutral, alkaline and acidic medium together with their pH ranges.

Indicator	Neutral medium	Alkaline medium	Acidic medium	pH range
Litmus solution	Purple	blue	Red	5.0 - 8.0
Phenolphthalein	Colourless	Pink/Purple	Colourless	8.3 - 10.0
Methyl orange	Orange	Yellow	Pink/Red	2.9 – 4.6

d) Standardization
It is the process of determining the concentration of a substance. Standardization is done through reacting a known volume of a substance with a known volume of a standard solution.

e) Standard solution
A standard solution is a solution whose concentration is accurately known.

f) Primary standard
A primary standard is a substance which is chemically stable in aqueous solution whereby its concentration remains constant with change in time and can be used to standardize other solutions.

Examples of primary standards

- Anhydrous sodium carbonate
- Sodium hydrogencarbonate
- Sodium tetraborate (Borax)
- Benzoic acid
- Potassium hydrogenphthalate
- Constant boiling point hydrochloric acid or sulphuric acid
- Potassium iodate
- Potassium dichromate
- Sodium oxalate
- Sodium ethanoate
- Potassium ethanoate
- Iodine
- Silver nitrate
- Sodium chloride
- Potassium bromideand others.

Characteristics of a primary standard

i) It should be readily available with the highest degree of purity.
ii) It should readily dissolve in water under the conditions in which it is employed.
iii) It should have a high relative molecular mass so that the effect of weighing errors is negligible.
iv) It should be stable at ordinary temperature so that it can be stored indefinitely without change in composition or without contamination.
v) It should remain unaltered in air during weighing. In otherwards, it should not get oxidized by air or react with carbon dioxide and it should not be hygroscopic, deliquescent or efflorescent.
vi) Its reaction should be stoichiometric and practically instantaneous.

1.5 Preparation of standard solutions

Standard solutions may be prepared either by weighing solid compounds or by dilution of more concentrated solutions.

1.5.1 Preparation of standard solutions by weighing solids

A known mass of a solid is weighed accurately using a digital weighing balance which determines the mass of the solid to 1 or 2 decimal places or by a triple beam balance (mechanical weighing balance) which determines the mass of the solid to 1 decimal place.

a) Preparation of 250cm^3 of 0.1M Sodium hydrogencarbonate solution

The following procedure is followed:
i)The mass of Sodium hydrogencarbonate required to be dissolved in 250cm^{3}of solutionis calculated. This is because the volumetric flask normally used in preparation of standard solutions from solids has a capacity of 250cm^3.However, before the mass required is calculated, the molar mass of the solid is calculated as follows:

Molar Mass of $NaHCO_3$ = [(23x1) + (1x1)+(12x1)+(16x3)]= 84g

The calculation is then done as follows:

I mole of $NaHCO_3$ weighs 84g
0.1 moles of $NaHCO_3$ weighs (84x0.1)g
=8.4g

1000cm^3 of solution contains 8.4g

250cm^3 of solution contain $\left(\frac{8.4}{1000} \text{x } 250\right)g$

=2.1g

ii) A clean container e.g. beaker or petri-dish is weighed and its mass recorded, say 40.0g. Using a clean spatula, the solid Sodium hydrogencarbonate is scooped and added into the beaker such that the total mass determined by the weighing balancecorresponds to the sum of the mass of the container and the Sodium hydrogencarbonate required i.e. 42.1g.

iii) The weighed Sodium hydrogencarbonate is then transferred into a clean beaker and a minimum amount of distilled water, say 100cm^3 is added to the solid in the beaker and the mixture stirred with a glass rod thoroughly to form a solution.

iv)The solution in the clean beaker is then carefully transferred into a clean 250cm^3 volumetric flask using a clean filter funnel. Care is taken that all the solution on the walls of the beaker and filter funnel is completely transferred into the volumetric flask.

v) More distilled water is then carefully added to the solution in the volumetric flask until its level is about 3cm to the calibration mark of the volumetric flask. More distilled water is then added drop by drop until the lower meniscus of the solution coincides with the calibration mark. The lower meniscus of the solution is viewed while the eye is at the same level with the calibration mark of the volumetric flask.

b) Preparation of 10 litres of 0.05M Sodium hydroxide solution

The following procedure is followed:

i)The mass of sodium hydroxide required to be dissolved in 10litres (10,000cm^3) of solutionis calculated. However, before the mass required is calculated, the molar mass of sodium hydroxide is calculated as follows:

*Molar Mass of NaOH= [(23x1) + (16x1)+(1x1)]***= 40g**

The calculation is then done as follows:

1 mol of NaOH weighs 40g

0.05mol of NaOHweighs (40 x0.05)g

= 2.0g

1000cm^3 of solution contains 2.0g

10,000cm^3 of solution contain $\left(\frac{2.0}{1000} \text{x } 10{,}000\right)g$

=20g

ii) A clean container e.g. beaker or petri-dish is weighed and its mass recorded, say 38.0g. Using a clean spatula, the solid sodium hydroxide is scooped and added into the beaker such that the total mass determined by the weighing balancecorresponds to the sum of the mass of the container and the Sodium hydroxide required i.e. 58.0g.

iii) The weighed sodium hydroxide is then transferred into a clean beaker and a minimum amount of distilled water, say 200cm^3 is added to the solid in the beaker and the mixture stirred with a glass rod thoroughly to form a solution.

iv)The solution in the clean beaker is then carefully transferred into a clean 1000cm^{3}or 2000cm^{3}volumetric flask/measuring cylinder using a clean filter funnel.Care is taken that all the solution on the walls of the beaker and filter funnel is completely transferred into the volumetric flask/measuring cylinder.

v) More distilled water is then carefully added to the solution in the volumetric flask/measuring cylinder, little at a time until the lower meniscus of the solution coincides with the 1,000m^3 or 2,000cm^3 calibration mark. The lower meniscus of the solution is viewed while the eye is at the same level with the calibration mark of the volumetric flask/measuring cylinder.
vi) The **1000cm^3or 2000cm^3solution**is then poured into a **larger vessel, say a 10 litre jerrican** and more **distilled water**added using the **1000cm^3 or 2000cm^3 volumetric flask/measuring cylinder until the total volume is 10,000cm^3.**

c) Preparation of 12litres of 10% potassium iodide solution
The following procedure is followed:
i)The mass of potassium iodide required to be dissolved in 12litres (12,000cm^3) of solutionis calculated.
*{**Note:** 10% potassium iodide is prepared by dissolving 10g of potassium iodide in water to make 100$cm^3$of solution}*

100$cm^3$of solution contain 10g of potassium iodide
12,000$cm^3$of solution contain $\left(\frac{10}{100}\text{x } 12000\right)$*g of potassium iodide*
= 1200g of potassium iodide

ii) A clean container e.g. beaker is weighed and its mass recorded, say 39.2g. Using a clean spatula, the solid potassium iodide is scooped and added into the beaker such that the total mass determined by the weighing balancecorresponds to the sum of the mass of the container and the potassium iodide required i.e. 1239.2g.
iii) To the weighed potassiumiodide in the clean beaker, a minimum amount of distilled water, say 500cm^3 is added and the mixture stirred with a glass rod thoroughly to form a solution.
iv)The solution in the clean beaker is then carefully transferred into a clean 1000cm^3or 2000cm^3volumetric flask/measuring cylinder using a clean filter funnel. Care is taken that all the solution on the walls of the beaker and filter funnel is completely transferred into the volumetric flask/measuring cylinder.
v) More distilled water is then carefully added to the solution in the volumetric flask/measuring cylinder, little at a time until the lower meniscus of the solution coincides with the 1000m^3 or 2000cm^3 calibration mark. The lower meniscus of the solution is viewed while the eye is at the same level with the calibration mark of the volumetric flask/measuring cylinder.
vi) The **1000cm^3or 2000cm^3solution**is then poured into a **larger vessel, say a 20 litre jerrican** and more **distilled water**added using the **1000cm^3 or 2000cm^3volumetric flask/measuring cylinder until the total volume is 12,000cm^3.**

1.5.2 Preparation of standard solutions by dilution of more concentrated solutions

a) Preparation of 1 litre of 0.1M sodium hydroxide from a stock solution of 2M sodium hydroxide
The following calculation is done first:
2moles of sodium hydroxide are contained in 1000cm^3 of the stock solution.
0.1moles of sodium hydroxide are contained in $\left(\frac{1000}{2}\text{x } 0.1\right)$ *$cm^3$of the stock solution*
*= **50cm^3** of the stock solution*

Procedure of preparation:
Withdraw ***50cm^3*** *of the stock solution using a* ***suitable measuring cylinder*** *and transfer it to a* ***1,volumetric flask or1,000cm^3 measuring cylinder*** *and make up to the mark with distilled water.*

Note: If a1,000cm^3 volumetric flask is not available, then a 250cm^3 volumetric flask can be used but the following calculation has to be done first:
1000cm^3 of the dilute solution contain 50cm^3 of the stock solution
250cm^3 of the dilute solution contains $\left(\frac{50}{1000}\text{x } 250\right)$*cm^3 of the stock solution.*
= 12.5cm^3*of the stock solution.*

Procedure of preparation:
Withdraw 12.5cm^3 of the stock solution using a suitable measuring cylinder and transfer it to a 250cm^3 volumetric flask or 250cm^3 measuring cylinder and make up to the mark with distilled water.

b) Preparation of 5 litres of 0.1M sulphuric acid from a bottle of concentrated sulphuric acid given the following specifications on the bottle:
Minimum assay/percentage purity = 98%
Specific gravity/weight per millilitre/density = 1.84
Molecular weight = 98g
*{**Note:** 1ml = 1cm^3}*

1cm^3 of the 98% concentrated solution contains $\left(\frac{\mathbf{1.84}}{\mathbf{100}}\mathbf{x\ 98}\right)$*g of sulphuric acid*
= 1.8032g of sulphuric acid
Therefore,1cm^3 of the 98% concentrated solution contains$\left(\frac{1.8032}{98}\right)$*moles of sulphuric acid*
= 0.0184*moles of sulphuric acid*
1 cm^3 of the 98% concentrated solution contains 0.0184 moles of sulphuric acid
1000cm^3 of the 98% concentrated solution contain (0.0184x1000) moles of sulphuric acid
= 18.4 M
18.4 mol of sulphuric acid are contained in 1000 cm^3 of the 98% concentrated solution.
0.1mol of sulphuric acid are contained in$\left(\frac{1000}{18.4}\text{x } 0.1\right)$ *cm^3 of the 98%concentrated solution.*
=5.43cm^3*of the 98%concentrated solution.*
1000cm^{3}of solution require 5.43cm^{3}of the 98%concentrated solution
5000cm^{3}of solution require $\left(\frac{5.43}{1000}\text{x } 5000\right)$*cm^{3}of the 98%concentrated solution*
=27.15cm^3*of the 98%concentrated solution.*

Procedure of preparation:
i)*Withdraw* ***27.15cm^3****of the 98%concentrated solution using a* ***suitable measuring cylinder*** *and transfer it to a* ***1000cm^3 volumetric flaskor1000cm^3 measuring cylinder****already containing water, say about* ***200cm^3 of distilled water****and then make up to the mark with distilled water.*
ii) *Pour the* ***1000cm^3 solution*** *into a* ***larger vessel, say a 5litre jerrican*** *and add* ***extra 4000cm^{3}of distilled water*** *using the* ***1000cm^3 volumetric flaskor1000cm^3 measuring cylinder.***
Note: Whenever you are preparing a dilute solution of an acid from a bottle of the concentrated acid, especially for sulphuric acid, never add water to the measured concentrated acid, instead always add the measured concentrated acid to water in the volumetric flask or measuring cylinder before making up to the mark with more distilled water. Doing otherwise will result in the upward movement of

concentrated sulphuric acid which will cause severe burns to the individual from the acid since concentrated sulphuric acid is extremely water loving.

c) Preparation of 650cm^3 of 0.2M hydrochloric acid from a bottle of concentrated hydrochloric acid given the following specifications on the bottle:
Minimum assay = 35-38%
Specific gravity/weight per millilitre/density = 1.18
Molecular Weight = 36.5g

Average minimum assay $= \left(\frac{35+38}{2}\right) = 36.5\%$

1cm^3 of the 36.5% concentrated solution contains $\left(1.18 \times \frac{36.5}{100}\right)$*g of hydrochloric acid*

$= 0.4307$*g of hydrochloric acid*

Thus, 1cm^3 of the 36.5% concentrated solution contains $\left(\frac{0.4307}{36.5}\right)$*moles of hydrochloricacid*

$= 0.0118$*moles of hydrochloric acid*

1 cm^3 of the 36.5% concentrated solution contains 0.0118moles of hydrochloric acid

1000cm^3 of the 36.5% concentrated solution contain(0.0118x1000)moles of hydrochloric acid

$= 11.8$ *M*

11.8mol of hydrochloric acid are contained in 1000cm^3 of the 36.5% concentrated solution.

0.2mol of HCl are contained in $\left(\frac{1000}{11.8} \times 0.2\right)$*cm^3 of the 36.5%concentrated solution*

$=16.95$*cm^3 of the 36.5%concentrated solution.*

1000cm^3 of solution require 16.95cm^3 of the 36.5%concentrated solution.

650cm^3 of solution require $\left(\frac{16.95}{1000} \times 650\right)$*cm^3 of the 36.5%concentrated solution*

$=11.02$*cm^3 of the 36.5%concentrated solution*

$\simeq 11$*cm^3 of the 36.5%concentrated solution*

Procedure of preparation:
Withdraw ***11cm^3*** *of the 36.5%concentrated solution using a* ***suitable measuring cylinder*** *and transfer it to a* ***1000cm^3 measuring cylinder*** *containing a good amount of water, say about* ***400cm^3 of distilled water*** *and then make up to* ***650cm^3*** *with* ***distilled water.***

1.5.3 Trial Exercises on Preparation of Standard Solutions

a)Calculate the mass of sodium hydroxide required to prepare 1 litre of 0.1M sodium hydroxide solution.(Na=23, O=16, H=1)

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b) Determine the mass of anhydrous sodium carbonate required to prepare 250cm^3 of 0.06M sodium carbonate solution. (Na=23, C=12, O=16)

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c) Calculate the mass of potassium hydroxide required to prepare 20 litres of 0.2M potassium hydroxide solution. (K=39, O=16, H=1)

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d) Determine the mass of:
(i) potassium iodide required to prepared 6 litres of 10% potassium iodide solution.

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(ii) starch required to prepare 2 litres of 1% starch solution.

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e) Calculate the volume of a:

(i) 2M stock solution of sodium hydroxide required to prepare 1 litre of 0.08M sodium hydroxide solution.

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(ii) 4M stock solution of hydrochloric acid required to prepare 15 litres of 0.2M hydrochloric acid solution.

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f) Determine the molarity of a solution of concentrated sulphuric acid in a bottle with the following specifications:

Minimum assay = 98%
Specific gravity (density) = 1.84
Molecular Weight = 98g

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g)A bottle of concentrated hydrochloric acid has got the following specifications:
Minimum assay = 35-38%
Wt per ml (density)=1.18
Molecular weight = 36.5g
Calculate the volume of the concentrated hydrochloric acid required to prepare 1 litre of 2M hydrochloric acid.

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h)A bottle of concentrated nitric acid has got the following specifications:
Minimum assay = 69-72%
Wt per ml (density)=1.41-1.43
Molecular weight = 63g
Calculate the volume of the concentrated nitric acid required to prepare 40 litres of 2M nitric acid.

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i) A bottle of concentrated ammonia solution has got the following specifications:
Minimum assay =30%
Wt per ml (density) = 0.89
Molecular weight =17g
Calculate the volume of the concentratedammonia solution required to prepare 2 litres of 2M ammonia solution.

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1.6 Rules followed in presentation of work in Volumetric Analysis

(The Dos and Don'ts of Volumetric Analysis)

- In case the procedure involves weighing, the values of mass should be recorded to the same number of decimal placese.g. one decimal place or two decimal places depending on the weighing scale used. Some weighing scales determine mass to one decimal place while others determine the mass to two decimal places. In the same way, the mass of the beaker should have been correctly subtracted from the mass of the beaker plus the solid sample to give the mass of the solid sample.
- The capacity/volume of the pipette used is recorded to zero decimal places, one decimal place or two decimal places (However, recording the capacity/volume of the pipette to one decimal place is preferred).

- Each value in the table of results is recorded to two decimal places and the second decimal place must be a zero. For instance, 24.20 and not 24.02. However, the second decimal place can be five ifthe reading is exactly between two values, say between 22.20 and 22.30 in which case it can be recorded as 22.25.
- The initial burette readings may be different.
- To get the titre value of each experiment, each initial burette reading should have been correctly subtracted from the final burette reading.
- The three titre values are not expected to be exactly the same since it is not practically possible.
- There should be consistency in atleast two titre values,which are supposed to be used in calculating the averagetitre value. These two titre values should not deviate beyond $\pm 0.10 cm^3$.
- The method/working used to calculate the average titre value should be clearly shown and the answer obtained should be preferably recorded to two decimal places.
- All calculations must be based on the mole concept and should be done from first principles without direct application of mathematical formulae.

CHAPTER TWO
2.0 STANDARDIZATION OF SOLUTIONS

2.1Introduction

Standardization of a solution refers to the process of determining the concentration of a substance in a solution. Standardization of a solution is done by reacting a known volume of the solution of the substance with a known volume of a standard solution. At this level, the standard solution is some times prepared by the student basing on the procedures given in the experiment.

2.2Worked out examples on Standardization of solutions

Worked out example 2.2.1

You are provided with the following:
FA1 which is a solution of approximately 0.1M hydrochloric acid
Substance **H** which is anhydrous sodium carbonate.
You are required to determine the actual concentration of the hydrochloric acid in FA1 in moldm^{-3} and also ing dm^{-3} (Na=23, C=12, O=16, H=1, Cl=35.5)

Procedure

i)Weigh accurately 1.3g of solid H into a clean beaker.
ii)To the solid in the beaker, add 100cm^3 of distilled water and stir well to dissolve.
iii)Transfer the solution in the beaker carefully into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label this solution FA2.
iv)Pipette 25 or 20cm^3 of FA2 into a clean conical flask. Add 2-3 drops of methyl orange indicator and titrate with FA1 from the burette until the end point is reached.
v)Repeat the titration until you obtain consistent results.
vi) Record your results in the table below.

Results

Mass of beaker + H ……*41.5*……..g
Mass of beaker alone……*40.2*……..g
Mass of H alone ……*1.3*……..g
Capacity of pipette used……*25.0*……cm^3

Final burette reading (cm^3)	*25.30*	*25.10*	*35.00*
Initial burette reading (cm^3)	*0.00*	*0.00*	*10.00*
Volume of FA1 used (cm^3)	*25.30*	*25.10*	*25.00*

Values used to calculate average volume of FA1……*25.10, 25.00*……*cm^3*

Average volume of FA1 used……$\left(\frac{25.10+25.00}{2}\right)$ = ***25.05***……*cm^3*

a) Determine the concentration of the sodium carbonate in FA2 in moldm^{-3}

Molar mass of Na_2CO_3= [(23x2) + (12x1) + (16x3)]= 106g
106g of sodium carbonate contain 1 mole

1.3g of sodium carbonate contain $\left(\frac{1}{106} \times 1.3\right)$ *moles*

= 0.01226 moles

250cm^3 of FA2 contain 0.01226 moles of sodium carbonate

1000 cm^3 of FA2 contain $\left(\frac{0.01226}{250} \times 1000\right)$ *moles of sodium carbonate per litre*

=0.049 mol dm^{-3}

b) Write the equation of the reaction

Na_2CO_3 (aq) + 2HCl (aq) → 2NaCl(aq) + H_2O(l) + CO_2(g)

.....

c) Calculate the number of moles of the hydrochloric acid in FA1 that reacted with the sodium carbonate in FA2**.**

1000 cm^3 of FA2 contain 0.049 moles of sodium carbonate

25 cm^3 of FA2 contains $\left(\frac{0.049}{1000} \times 25\right)$ *moles of sodium carbonate*

***= 0.001225** moles of sodium carbonate*

Moles of hydrochloric acid = (2xmoles of sodium carbonate)

= (2x0.001225)

=0.00245

d) Determine the concentration of the hydrochloric acid in FA1 in:

i) moles per dm^3

25.05 cm^3 of FA1 contain 0.00245 moles of hydrochloric acid

1000 cm^3 of FA1 contains $\left(\frac{0.00245}{25.05} \times 1000\right)$ *moles of a hydrochloric acid*

= 0.0978 mol dm^{-3}

ii) grams per dm^3.

Molar mass of HCl =[(1x1) + (35.5x1)]= 36.5g

1 mole of hydrochloric acid weighs 36.5g

0.0978 moles of hydrochloric acid weighs (36.5x0.0978)g

= 3.57g dm^{-3}

2.3 Practical Exercises on Standardization of solutions

Experiment 2.3.1

You are provided with the following:

GA1 which is a solution of approximately 0.1M sulphuric acid

Substance **J** which is sodium hydrogencarbonate

You are required to determine the concentration of the sulphuric acid in GA1 in grams per dm^3.

(Na=23, C=12, =16, H=1, S=32)

Procedure

i) Weigh accurately 3.1g of solid J into a clean beaker. To the solid in the beaker, add 100cm^3 of distilled water and stir well with a glass rod to dissolve.

ii) Transfer the solution in the beaker carefully into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label this solution GA2.

iii) Pipette 25 or 20cm^3 of GA2 into a clean conical flask. Add 2-3 drops of methyl orange indicator and titrate with GA1 from the burette until the end point is reached.

iv)Repeat the titration until you obtain consistent results.
v) Record your results in the table below.

Mass of beaker + J ……..……..g

Mass of beaker ……..…….g

Mass of J...……..…….....…….....g

Capacity of pipette used……cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of GA1 used (cm^3)			

Values used to calculate average volume of GA1…….cm^3

Average volume of GA1 used…….....cm^3

…….

a) Write the equation of the reaction that occurrs.

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b) Determine the number of moles of the sodium hydrogencarbonate in 1 dm^3 of GA2.

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c) Calculate the number of moles of the sulphuric acid in GA1 that reacted with the sodium hydrogencarbonate in GA2.

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d) Determine the concentration of the sulphuric acid inGA1 ingdm^{-3}.

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Experiment 2.3.2

You are provided with the following:
FA1 which is a solution of 0.1M potassium hydroxide
FA2 which is approximately 1.0 M sulphuric acid
You are required to determine the molar concentration of the sulphuric acid in FA2.

Procedure

i)Using a suitable measuring cylinder, measure and transfer 15cm^3 of FA2 into a 250cm^3 volumetric flask. Add about 100cm^3 of distilled water and shake well to mix, then make up to the mark with distilled water. Label the solution FA3.
ii)Pipette 25 or 20cm^3 of FA1 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with FA3 from the burette until the end point is reached.
iii)Repeat the titration until you obtain consistent results. Record your results in the table below.

Capacity of pipette used……cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA3 used (cm^3)			

Values used to calculate average volume of FA3…….cm^3

Average volume of FA3 used…….....cm^3

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a) Calculate the number of moles of sulphuric acid:

i) in FA3 that reacted with the potassium hydroxide in FA1.

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ii) in 250cm^3 of FA3.

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b) Determine the molar concentration of the sulphuric acid in FA2.

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Experiment 2.3.3

You are provided with the following:
FA1 which is a solution of approximately 0.2M hydrochloric acid
FA2 which is sodium hydroxide solution
Substance **K** which is anhydrous sodium carbonate
You are required to standardise the hydrochloric acid in FA1.
(Na=23, C=12, O=16, H=1, Cl=35.5)

Procedure I

i) Weigh accurately 1.4 g of solid K into a clean beaker. To the solid in the beaker, add 100cm^3 of distilled water and stir well with a glass rod to dissolve.
ii) Transfer the solution in the beaker carefully into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label this solution FA3.
iii) Pipette 25 or 20cm^3 of FA3 into a clean conical flask. Add 2-3 drops of methyl orange indicator and titrate with FA1 from the burette until the end point is reached.
v) Repeat the titration until you obtain consistent results.
vi) Record your results in the table below.

Mass of beaker + K ……..……..g

Mass of beaker ……..……..g

Mass of K ...……..…….....…….....g

Capacity of pipette used……..cm^3

Table I

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA1 used (cm^3)			

Values used to calculate average volume of FA1……cm^3

Average volume of FA1 used…….....cm^3

……..

a) Determine the number of moles of Sodium carbonate in 1 dm^3 of FA3.

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b) Calculate the number of moles of the hydrochloric acid in FA1 that reacted with the sodium carbonate in FA3.

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c) Determine the concentration of the hydrochloric acid in FA1 in:

i) moles per dm^3.

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ii) grams per dm^3.

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Procedure II

i) Using a measuring cylinder, measure and transfer 50cm^3 of FA1 into a clean beaker. Add 100cm^3 of distilled water and label the solution FA4.

ii) Pipette 20 or 25cm^3 of FA2 into a clean conical flask. Add 2-3 drops of methyl orange indicator and titrate with FA4 from the burette until the end point is reached.

iii) Repeat the titration until you obtain consistent tresults. Record your results in the table below.

Table II

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA4 used (cm^3)			

Values used to calculate average volume of FA4……cm^3

Average volume of FA4 used…….....cm^3

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d) Determine the concentration of:
i) hydrochloric acid in FA4 in mol dm^{-3}.

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ii) sodium hydroxide in FA2 in mol dm^{-3}.

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Experiment 2.3.4

You are provided with the following:

Solid T which is Borax (sodium tetraborate, $Na_2B_4O_7.10H_2O$)

GA1 which is a solution of hydrochloric acid.

GA2 which is a solution prepared by dissolving 11.0g of impure sodium carbonate in water to form one litre of solution.

Methyl orange indicator

You are required to determine the:

i) concentration of hydrochloric acid in mol dm^{-3} using borax.

ii) percentage purity of the sodium carbonate used in the preparation of GA2.

Theory

Sodium tetraborate reacts with hydrochloric acid according to the equation below:

Molecular equation

$Na_2B_4O_7.10H_2O(aq) + 2HCl(aq) \longrightarrow 2NaCl(aq) + 4H_3BO_3(aq) + 5H_2O(l)$

Ionic equation

$B_4O_7^{2-}(aq) + 2H^+(aq) + 5H_2O(l) \longrightarrow 4H_3BO_3(aq)$

Since the boric acid formed (H_3BO_3) is a weak acid and therefore, it does not affect the titration as long as methyl orange (or methyl red) is the indicator used.

Procedure I

(i) Weigh accurately, 4.5g of solid T into a beaker. Using a measuring cylinder, add 100cm^3 of distilled water and stir well to dissolve. Transfer the resultant solution into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label the solution GA3.

(ii) Pipette 25.0 or 20.0 cm^3 of GA3 into a conical flask. Add 2-3 drops of methyl orange indicator and titrate with GA1 from the burette until the end point is reached.

(iii) Repeat the titration until you obtain consistent results.

(iv) Record your results in **table I** below.

Mass of beaker + T ……..……..g

Mass of beaker ……..……..g

Mass of T ...……..……....…….....g

Capacity of pipette used……..cm^3

Table I

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of GA1 used (cm^3)			

Values used to calculate average volume of GA1……cm^3

Average volume of GA1 used…….....cm^3

……

a) Determine the concentration of:

(i) borax in GA3 in mol dm^{-3}. (Na=23, B=11, O=16, H=1)

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(ii) hydrochloric acid in GA1 in mol dm^{-3}.

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Procedure II

(i) Pipette 25.0 or 20.0 cm^3 of GA2 into a conical flask. Add 2-3 drops of methyl orange indicator and titrate with GA1 from the burette until the end point is reached.
(ii) Repeat the titration until you obtain consistent results.
(iii) Record your results in **table II** below.

Table II

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of GA1used (cm^3)			

Values used to calculate average volume of GA1……cm^3

Average volume of FA4 used…….....cm^3

b) Calculate the number of moles of sodium carbonate in GA2 which reacted with hydrochloric acid in GA1.

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c) Determine the:

(i) concentration of sodium carbonate in GA2 in grams per litre. (Na = 23, C=12, O=16)

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ii) percentage purity of the sodium carbonate sample used in the preparation of GA2.

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CHAPTER THREE
3.0SOLUTIONMIXTURES

Solution mixtures are categorised as either simple or complex mixtures.

3.1 Simple Mixtures

Simple mixtures are those which when titrated against a standard solution, only one of the components of the mixture reacts with the standard solution while the other component of the mixture does not. Therefore, the number of moles and consequently, the concentration of the component that reacts can be calculated basing on the reaction stoichiometry (reaction mole ratio) while the number of moles and consequently, the concentration of the component that does not react is calculated by difference.
Examples of simple mixtures include the following:

- Potassium hydroxide and potassium chloride titrated against hydrochloric acid.
- Sodium hydroxide and sodium chloride titrated against hydrochloric acid.
- Potassium hydroxide and potassium sulphate titrated against sulphuric acid.
- Sodium hydroxide and sodium sulphate titrated against sulphuric acid.
- Potassium carbonate and potassium chloride titrated against hydrochloric acid.
- Sodium carbonate and sodium chloride titrated against hydrochloric acid.
- Oxalic acid, $H_2C_2O_4.2H_2O$ and sodium oxalate, $Na_2C_2O_4$ titrated against sodium hydroxide or potassium hydroxide.
- Iron(II) ions, Fe^{2+} and iron(III) ions, Fe^{3+} titrated against manganate(VII) ions, MnO_4^- in the presence of an acid.

3.1.1 Worked out Examples on Simple Mixtures

Worked out example 3.1.1.1

You are provided with the following:
FA1 which is1M nitric acid.
FA2 which is a mixture of potassium carbonate and potassium nitrate12.8g per litre.
You are required to determine the percentage of potassium carbonate in the FA2 mixture.

Procedure
Using a measuring cylinder, measure and transfer 50cm^3 of FA1 into a 250cm^3 volumetric flask. Add 100cm^3 of distilled water and shake well to mix and then make up to the mark with more distilled water. Label the solution FA3.

Pipette 20 or 25cm^3 of FA2 into a clean conical flask. Add 2-3 drops of methyl orange indicator and titrate with FA3 from the burette until the end point is reached. Repeat the titration until the end point is reached. Repeat the titration untilyou opbtain consistent results. Record your results in the table below.

Capacity of pipette used……*20*……cm^3(½ mark)

Final burette reading (cm^3)	*12.80*	*12.60*	*20.50*
Initial burette reading (cm^3)	*0.00*	*0.00*	*8.00*
Volume of FA3 used (cm^3)	*12.80*	*12.60*	*12.50*

(4½ marks)

Values used to calculate average volume of FA3……*12.60, 12.50*……cm^3 (½ mark)

Average volume of FA3 used……$\left(\frac{12.60 + 12.50}{2}\right)$= ***12.5***……$cm^3$ (2½ marks)

a) Calculate the:

i) molarity of nitric acid in FA3. (1½ marks)

$1000cm^3$ of FA1contain 1 mole of nitricacid

$50cm^3$ of FA1 contain $\left(\frac{1}{1000} \times 50\right)$ *moles of nitric acid*

= 0.05moles of nitric acid

$250cm^3$ of FA3 contain 0.05 moles of nitric acid

$1000cm^3$ of FA3 contain $\left(\frac{0.05}{250} \times 1000\right)$ *moles of nitric acid*

= 0.2 moles per litre

i) number of moles of potassium carbonate in FA2 that reacted with the nitric acid in FA3.(3 marks)

$2HNO_3(aq) + K_2CO_3(aq) \rightarrow 2KNO_3(aq) + H_2O(l) + CO_2(g)$

$1000cm^3$ of FA3 contain 0.2 moles of nitric acid

$12.55cm^3$ of FA3 contain $\left(\frac{0.2}{1000} \times 12.55\right)$ *moles of nitric acid*

= 0.00251 moles of nitric acid

Moles of potassium carbonate = $\frac{1}{2}$ *x moles of nitric acid*

$= \left(\frac{1}{2} \times 0.00251\right)$

= 0.001255

b) Determine the:

i) concentration of potassium carbonate in FA2 in gdm^{-3}.(3 marks)

$20cm^3$ of FA2 contain 0.001255 moles of potassium carbonate

$1000cm^3$ of FA2 contain $\left(\frac{0.001255}{20} \times 1000\right)$ *moles of potassium carbonate*

= 0.0628 $moldm^{-3}$

Molar mass of K_2CO_3 = (39x2) + (12x1) + (16x3)=138g

1 mole of potassium carbonate weighs 138g

0.0628 moles of potassium carbonate weigh (138 x 0.0628)g

=8.67gdm^{-3}

ii) percentage of potassium carbonate in the FA2 mixture.

Percentage of potassium carbonate $= \left(\frac{8.67}{12.8} \text{x } 100\right)\%$

$= 67.7\%$

Worked out example 3.1.1.2

You are provided with the following:
FA1 which is 0.1M sodium hydroxide.
FA2 which is a mixture of oxalic acid ($H_2C_2O_4.2H_2O$) and sodium oxalate ($Na_2C_2O_4$)containing $12.4gdm^{-3}$.
You are required to determine the percentage of sodium oxalate in FA2.

Theory

In the process of titration, the sodium hydroxide in FA1 reacts with only oxalic acid in the FA2 mixture according to the reaction below.

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

Therefore, the number of moles of oxalic acid can be determined basing on the mole ratio between sodium hydroxide and oxalic acid in the reaction above. Sodium oxalate does not take part in the reaction.

Procedure:

Pipette $25cm^3$ or $20cm^3$of FA1 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with FA2 from the burette. Repeat the titration until you obtain consistent results. Record your results in the table below.

Capacity of pipette used……*25.0*……cm^3

Final burette reading (cm^3)	*20.50*	*20.30*	*24.30*
Initial burette reading (cm^3)	*0.00*	*0.00*	*4.00*
Volume of FA2 used (cm^3)	*20.50*	*20.30*	*20.30*

Values used to calculate average volume of FA2……..*20.30, 20.30* ……cm^3

Average volume of FA2 used……$\left(\frac{\mathbf{20.30+20.30}}{\mathbf{2}}\right)$=***20.30***……$cm^3$

a) Calculate the number of moles of oxalic acid that reacted with the sodium hydroxide.

$1000cm^3$ of FA1 contain 0.1 moles of sodium hydroxide

$25cm^3$of FA1 contain $\left(\frac{0.1}{1000} \text{x } 25\right)$ *moles of sodium hydroxide*

$= 0.0025$*moles of sodium hydroxide*

Moles of oxalic acid $= \frac{1}{2}$ x *moles of sodium hydroxide*

$= \left(\frac{1}{2} \text{x } 0.0025\right)$

$= 0.00125$

b) Determine the:
   i) concentration of sodium oxalate in FA2 in gdm^{-3}.(Na=23, C=12, O=16, H=1)

20.30 cm^3 of FA2 contain 0.00125 moles of oxalic acid

1000 cm^3 of FA2 contain $\left(\frac{0.00125}{20.30} \times 1000\right)$ *moles of oxalic acid*

$= 0.0616\ mol\ dm^{-3}$

Molar mass of $H_2C_2O_4.2H_2O = [(1x2) + (12x2) + (16x4) + (2x18)] = 126g$

1 mole of oxalic acid weighs 126g

0.0619 moles of oxalic acid weighs (126x0.0616)g

$= 7.76g\ dm^{-3}$

Concentration of sodium oxalate in FA2= (12.4—7.76)gdm^{-3}

$= 4.64\ g\ dm^{-3}$

   ii) percentage of sodium oxalate in FA2

Percentage ofsodium oxalate = $\left(\frac{4.64}{12.4} \times 100\right)\%$

$= 37.42\%$

3.1.2 Experiments on Simple Mixtures

Experiment 3.1.2.1

You are provided with the following:
FA1 which is 0.1M sulphuric acid.
FA2 which is a mixture of sodium hydroxide and sodium sulphate containing 32.8g per litre.
You are required to determine the percentage of sodium hydroxide in the FA2 mixture.

Procedure

Using a measuring cylinder, measure and transfer 120cm^3 of FA2 into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label the solution FA3.

Pipette 20 or 25cm^3 of FA3 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with FA1 from the burette until the end point is reached. Repeat the titration until the end point is reached. Repeat the titration untilyou obtain consistent results. Record your results in the table below.

Capacity of pipette used……...……cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA1 used (cm^3)			

Values used to calculate average volume of FA1……..……....cm^3

Average volume of FA1 used……cm^3

……

a) Calculate the number of moles of:

i) sulphuric acid in FA1 that reacted.

……

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ii) sodium hydroxide in FA3 that reacted with the acid in FA1.

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b) Determine the:

i) concentration of sodium hydroxide in FA2 in gdm^{-3}.

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ii) percentage of sodium hydroxide in the FA2 mixture.

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Experiment 3.1.2.2

You are provided with the following**:**
FA1which is 0.018M potassium manganate(VII) solution
FA2 which is 0.1M sodium hydroxide solution
FA3 which is a mixture of sodium oxalate,$Na_2C_2O_4$ and oxalic acid, $H_2C_2O_4.2H_2O$.
FA4 which is 2M sulphuric acid
You are required to determine the percentage of sodium oxalate in the FA3 mixture.

Theory

In the process of titration, the sodium hydroxide in FA2 reacts with only oxalic acid in the FA3 mixture according to the reaction below.

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

Therefore, the number of moles of oxalic acid can be determined basing on the reaction mole ratio between sodium hydroxide and oxalic acid in the reaction above. Sodium oxalate does not react with sodium hydroxide.
However, the acidified potassium manganate(VII) in FA1 reacts with both oxalic acid and sodium oxalate in FA3. In other wards, the acidified manganate(VII) ions in FA1 react with oxalate ions from both Oxalic acid and Sodium oxalate in FA3 according to the following equation:

$$2MnO_4^-(aq) + 16H^+(aq) + 5C_2O_4^{2-}(aq) \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) + 10CO_2(g)$$

Therefore, the total number of oxalate ions in the reaction mixture can be determined basing on the reactionmole ratio betweenacidified manganate(VII) ions and oxalate ions. The number of moles of oxalate ions from sodium oxalate can then be calculated by difference.
Note: Acidifiedmanganate(VII) ions react with oxalate ions only when the solution containing oxalate ions is heated to a temperature of about 60^0C.

Procedure A

i) Pipette 25 or 20cm^3 of FA3 into a clean conical flask and titrate with FA2 from the burette using phenolphthalein indicator.
ii) Repeat the titration until you obtain consistent results.
iii) Record your results in the table below.

Capacity of pipette used……...……cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA2 used (cm^3)			

Values used to calculate average volume of FA2……..……cm^3

Average volume of FA2 used……cm^3

……

a) Determine the concentration of oxalic acid in FA3 in:
i)moles per dm^3

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ii) grams per dm^3 (C=12, O=16, H=1)

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Procedure B

i) Rinse the burette thoroughly with water and then fill it with FA1.

ii) Pipette 25 or 20cm^3 of FA3 into a clean conical flask. Add an equal volume of FA4 using a measuring cylinder and warm the mixture to about 60^{0}C and titrate the hot mixture with FA1 from the burette until the solution just turns permanently pink.

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA1 used (cm^3)			

Values used to calculate average volume of FA1……..…….cm^3

Average volume of FA1 used……..cm^3

……

b) Determine the:

i)total concentration of oxalate ions in FA3 in moles per dm^3.

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ii) concentration of sodium oxalate in FA3 in grams per dm^3.(Na=23, C=12, O=16)

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c) Calculatethe percentage of sodium oxalate in the FA3 mixture.

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3.2Complex Mixtures

Complex mixtures are those which when titrated against a standard solution, both of the components of the mixture react with the standard solution. Therefore, the number of moles and consequently, the concentration of both components of the mixture can be calculated basing on the reaction stoichiometry (reaction mole ratio) of each of the components with the reactant in the standard solution.
Examples of complex mixtures include the following:

- Sodium hydroxide and sodium carbonate titrated against hydrochloric acid.
- Sodium hydroxide and sodium carbonate titrated against nitric acid.
- Sodium hydroxide and sodium carbonate titrated against sulphuric acid.
- Potassium hydroxide and potassium carbonate titrated against hydrochloric acid.
- Potassium hydroxide and potassium carbonate titrated against nitric acid.
- Potassium hydroxide and potassium carbonate titrated against sulphuric acid.
- Sodium hydroxide and sodium hydrogencarbonate titrated against hydrochloric acid.
- Sodium hydroxide and sodium hydrogencarbonate titrated against nitric acid.
- Sodium hydroxide and sodium hydrogencarbonate titrated against sulphuric acid.
- Potassium hydroxide and potassium hydrogencarbonate titrated against hydrochloric acid
- Potassium hydroxide and potassium hydrogencarbonate titrated against nitric acid
- Potassium hydroxide and potassium hydrogencarbonate titrated against sulphuric acid
- Sodium carbonate and sodium hydrogencarbonate titrated against hydrochloric acid.
- Sodium carbonate and sodium hydrogencarbonate titrated against nitric acid.
- Sodium carbonate and sodium hydrogencarbonate titrated against sulphuric acid.
- Potassium carbonate and potassium hydrogencarbonate titrated against hydrochloric acid.
- Potassium carbonate and potassium hydrogencarbonate titrated against hydrochloric acid.
- Potassium carbonate and potassium hydrogencarbonate titrated against nitric acid.
- Potassium carbonate and potassium hydrogencarbonate titrated against sulphuric acid, **e.t.c.**

Note: Complex mixtures introduce us to the concept of ***Double Indicator Titrations.***

3.3Double Indicator Titrations

In double indicator titrations, a mineral acid such as hydrochloric acid, nitric acid or sulphuric acid is reacted with a mixture containing a base such as sodium hydroxide or potassium hydroxide and a carbonate or hydrogen carbonate of sodium or potassium.Alternatively, the mineral acid is reacted with a mixture of sodium carbonate or potassium carbonate and sodium hydrogencarbonate or potassium hydrogencarbonate.The titration is carried out using two indicators, that is, phenolphthalein indicator and methyl orange indicator hence double indicator titrations.

Note:Students should never attempt questions of double indicator titrations basing on cram work, but should instead attempt those questions by recalling the following facts as summarized below:

a) With Phenolphthalein Indicator

When a base such as sodium hydroxide or potassium hydroxide is titrated against a mineral acid, such as hydrochloric acid,nitric acid or sulphuric acid using phenolphthalein indicator, the base undergoes complete neutralization to form the corresponding salt plus water.

$NaOH(aq) + HCl(aq) \rightarrow NaCl(aq) + H_2O(l)$

$NaOH(aq) + HNO_3(aq) \rightarrow NaNO_3(aq) + H_2O(l)$
$2NaOH(aq) + H_2SO_4(aq) \rightarrow Na_2SO_4(aq) + 2H_2O(l)$
$KOH(aq) + HCl(aq) \rightarrow KCl(aq) + H_2O(l)$
$KOH(aq) + HNO_3(aq) \rightarrow KNO_3(aq) + H_2O(l)$
$2KOH(aq) + H_2SO_4(aq) \rightarrow K_2SO_4(aq) + 2H_2O(l)$

The general ionic equation for the above reactions is:

$OH^-(aq) + H^+(aq) \rightarrow H_2O(l)$

When sodium carbonate or potassium carbonate is titrated against a mineral acid such as hydrochloric acid, nitric acid or sulphuric acid using phenolphthalein indicator, the metal carbonate undergoes half neutralization to form sodium hydrogencarbonate or potassium hydrogencarbonate plus the corresponding salt.

$Na_2CO_3(aq) + HCl(aq) \rightarrow NaHCO_3(aq) + NaCl(aq)$
$Na_2CO_3(aq) + 2HNO_3(aq) \rightarrow NaHCO_3(aq) + NaNO_3(aq)$
$2Na_2CO_3(aq) + H_2SO_4(aq) \rightarrow 2NaHCO_3(aq) + Na_2SO_4(aq)$
$K_2CO_3(aq) + HCl(aq) \rightarrow KHCO_3(aq) + KCl(aq)$
$K_2CO_3(aq) + HNO_3(aq) \rightarrow KHCO_3(aq) + KNO_3(aq)$
$2K_2CO_3(aq) + H_2SO_4(aq) \rightarrow 2KHCO_3(aq) + K_2SO_4(aq)$

The general ionic equation for the above reactions is:

$CO_3^{2-}(aq) + H^+(aq) \rightarrow HCO_3^-(aq)$

When sodium hydrogencarbonate or potassium hydrogencarbonate is titrated against a mineral acid such as hydrochloric acid,nitric acid or sulphuric acid using phenolphthalein indicator, **no reaction occurs.**

$NaHCO_3(aq) + HCl(aq) \rightarrow$ No reaction
$NaHCO_3(aq) + HNO_3(aq) \rightarrow$ No reaction
$NaHCO_3(aq) + H_2SO_4(aq) \rightarrow$ No reaction
$KHCO_3(aq) + HCl(aq) \rightarrow$ No reaction
$KHCO_3(aq) + HNO_3(aq) \rightarrow$ No reaction
$KHCO_3(aq) + H_2SO_4(aq) \rightarrow$ No reaction

b) With Methyl orange indicator

Whensodium hydroxide or potassium hydroxideis titrated against a mineral acid such as hydrochloric acid, nitric acid or sulphuric acid using methyl orange indicator,the metal hydroxide undergoes complete neutralization to form the corresponding salt plus water.

$NaOH(aq) + HCl(aq) \rightarrow NaCl(aq) + H_2O(l)$
$NaOH(aq) + HNO_3(aq) \rightarrow NaNO_3(aq) + H_2O(l)$
$2NaOH(aq) + H_2SO_4(aq) \rightarrow Na_2SO_4(aq) + 2H_2O(l)$
$KOH(aq) + HCl(aq) \rightarrow KCl(aq) + H_2O(l)$
$KOH(aq) + HNO_3(aq) \rightarrow KNO_3(aq) + H_2O(l)$
$2KOH(aq) + H_2SO_4(aq) \rightarrow K_2SO_4(aq) + 2H_2O(l)$

The general ionic equation for the above reactions is:

$OH^-(aq) + H^+(aq) \rightarrow H_2O(l)$

When sodium carbonate orpotassium carbonate is titrated against a mineral acid such as hydrochloric acid, nitric acid or sulphuric acid usingmethyl orange indicator, the metal carbonate undergoes complete neutralisation to form the corresponding salt, water plus carbon dioxide gas.

$Na_2CO_3(aq) + 2HCl(aq) \rightarrow 2NaCl(aq) + H_2O(l) + CO_2(g)$
$Na_2CO_3(aq) + 2HNO_3(aq) \rightarrow 2NaNO_3(aq) + H_2O(l) + CO_2(g)$
$Na_2CO_3(aq) + H_2SO_4(aq) \rightarrow Na_2SO_4(aq) + H_2O(l) + CO_2(g)$
$K_2CO_3(aq) + 2HCl(aq) \rightarrow 2KCl(aq) + H_2O(l) + CO_2(g)$
$K_2CO_3(aq) + 2HNO_3(aq) \rightarrow 2KNO_3(aq) + H_2O(l) + CO_2(g)$
$K_2CO_3(aq) + H_2SO_4(aq) \rightarrow K_2SO_4(aq) + H_2O(l) + CO_2(g)$

The general ionic equation for the above reactions is:

$CO_3^{2-}(aq) + 2H^+(aq) \rightarrow H_2O(l) + CO_2(g)$

In a similar way, when sodium hydrogencarbonate or potassium hydrogencarbonate is titrated against a mineral acid such as hydrochloric acid,nitric acid or sulphuric acid using methyl orange indicator, the metal hydrogencarbonate undergoes complete neutralisation to form the corresponding salt, water plus carbon dioxide gas.

$NaHCO_3(aq) + HCl(aq) \rightarrow NaCl(aq) + H_2O(l) + CO_2(g)$
$NaHCO_3(aq) + HNO_3(aq) \rightarrow NaNO_3(aq) + H_2O(l) + CO_2(g)$
$2NaHCO_3(aq) + H_2SO_4(aq) \rightarrow Na_2SO_4(aq) + 2H_2O(l) + 2CO_2(g)$
$KHCO_3(aq) + HCl(aq) \rightarrow KCl(aq) + H_2O(l) + CO_2(g)$
$KHCO_3(aq) + HNO_3(aq) \rightarrow KNO_3(aq) + H_2O(l) + CO_2(g)$
$2KHCO_3(aq) + H_2SO_4(aq) \rightarrow K_2SO_4(aq) + 2H_2O(l) + 2CO_2(g)$

The general ionic equation for the above reactions is:

$HCO_3^-(aq) + H^+(aq) \rightarrow H_2O(l) + CO_2(g)$

Complex mixtures in which sodium carbonate, potassium carbonate, sodium hydrogencarbonate or Potassium hydrogencarbonate is one of the components of the mixture, are analyzed by titrating them against a standard solution of a mineral acid using both phenolphthalein and methyl orange indicator using two methods. The two methods are:**The Continuous Method** where the two different indicators are used concurrently and **The Two Step Method** where the two different indicators are used separately.
Note: *A lot of careshould be taken while dealing with calculations for double indicator titration experiments in which the procedures involve dilution of one of the solutions to prepare another solution which is actually used in the titration. (For instance in worked out examples 3.3.1.2,3.3.1.3, 3.3.3.2and 3.3.3.3 plus practicle exercises 3.3.2.3,3.3.2.4, 3.3.4.5and 3.3.4.6).*

A) The ContinuousMethod (Using the Two Indicators Concurrently)

In the continuous method, an aliquot (a portion) of the mixture is pipetted and titrated against a standard mineral acid using phenolphthalein indicator and the volume, $V_1$of the mineral acid required to reach the end point is noted.
Without pouring the solution in the conical flask, 2-3 drops of methyl orange indicator are added and the titration continued using the same standard mineral acidand the volume, $V_2$of the mineral acid required to reach the end point is noted.

3.3.1Worked out Examples on Double Indicator Titrations using the Continuous Method (Using the Two Indicators Concurrently)

Worked out example 3.3.1.1

You are provided with the following:
FA1 which is a mixture of sodium hydroxide and sodium carbonate
FA2 which is a solution of 0.2M hydrochloric acid
You are required to determine the concentrations of sodium hydroxide and sodium carbonate in grams per litre and hence the percentage of sodium hydroxide in the mixture.

Procedure

Pipette 25 or 20cm^3 of FA1 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with FA2 from the burette until the end point is reached. Record your results in table I below and then add 2-3 drops of methyl orange indicator to the resultant solution and continue the titration with FA2until the end point is reached.Record your results in table II below. Repeat the titrations until you obtain consistent results.

Volume of pipette used……*25.0*……cm^3

Burette readings	**Table I** (With Phenolphthalein indicator)			**Table II** (With Methyl orange indicator)		
Final burette reading (cm^3)	*32.70*	*32.50*	*34.40*	*42.90*	*42.50*	*44.30*
Initial burette reading(cm^3)	*0.00*	*0.00*	*2.00*	*32.70*	*32.50*	*34.40*
Volume of FA2(cm^3)	*32.70*	*32.50*	*32.40*	*10.20*	*10.00*	*9.90*

Average volume of FA2 used for table I
……...…$\left(\frac{32.50 + 32.40}{2}\right)$= ***32.45***……*$cm^3$*

Average volume of FA2 used for table II
……..…$\left(\frac{10.00 + 9.90}{2}\right)$=***9.95***……*$cm^3$*

Note: 1) With phenolphthalein indicator

$NaOH(aq) + HCl(aq) \rightarrow NaCl(aq) + H_2O(l)$
$Na_2CO_3(aq) + HCl(aq) \rightarrow NaHCO_3(aq) + NaCl(aq)$

2) With methyl orange indicator

$NaHCO_3(aq) + HCl(aq) \rightarrow NaCl(aq) + H_2O(l) + CO_2(g)$
(from Na_2CO_3)

a) Determine the volume of hydrochloric acid in FA2 required for the neutralization of:
i) Sodium carbonate ……=*(2x9.95)****cm^3***……
……. ……...***= 19.90cm^3***……
i) Sodium hydroxide
……= *(32.45-9.95)cm^3*……
…….***=22.50cm^3***……..…..

b) Calculate the concentration of sodium carbonate in FA1 in grams per litre.

1000cm^3 of FA2 contain 0.2moles of hydrochloricacid

19.90cm^3 of FA2 contain $\left(\frac{0.2}{1000} \times 19.90\right)$ *moles of hydrochloricacid*

= 0.00398 moles of hydrochloricacid

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

Moles of sodium carbonate = $\left(\frac{1}{2} \times 0.00398\right)$

= 0.00199

25cm^{3}of FA1 contain 0.00199 moles of sodium carbonate

1000 cm^3 of FA1 contain $\left(\frac{0.00199}{25} \times 1000\right)$ *moles of sodium carbonate*

= 0.0796 mol l^{-1}

≃ 0.08 mol l^{-1}

Molar Mass of Na_2CO_3= [(23x2)+(12x1)+(16x3)]= 106g

1 mole of sodium carbonate weighs 106g

0.08 moles of sodium carbonate weigh (106x0.08)g

= 8.48g

c) Determinethe concentration of sodium hydroxide in FA1 in grams per litre.

1000cm^3 of FA2 contain 0.2moles of hydrochloric acid

22.50cm^3 of FA2 contain $\left(\frac{0.2}{1000} \times 22.50\right)$ *moles of hydrochloric acid*

= 0.0045 moles of hydrochloric acid

$NaOH(aq) + HCl(aq) \rightarrow NaCl(aq) + H_2O(l)$

Moles of sodium hydroxide = (1xmoles of hydrochloric acid)

= (1x0.0045)

= 0.0045

25cm^3 of FA1 contain 0.0045 moles of sodium hydroxide

1000cm^3 of FA1 contain $\left(\frac{0.0045}{25} \times 1000\right)$ *moles of sodium hydroxide*

= 0.18 mol l^{-1}

Molar Mass of NaOH= [(23x) +(16x1) + (1x1)]

1 mole of sodium hydroxide weighs 40g

0.18 moles of sodium hydroxide weigh (40x0.18) ***= 7.2gl^{-1}***

d) Determine the percentage of sodium hydroxide in the FA1 mixture.

Total mass of FA1 mixture per litre = (8.48 + 7.2) ***=15.68g***

Percentage of sodium hydroxide = $\left(\frac{\mathbf{7.2}}{\mathbf{15.68}} \mathbf{\times 100}\right)$*%*

=45.9%

Note: *For mixtures containing either* ***sodium carbonate or potassium carbonate,*** *while establishing their mole ratio of reaction with a particular acid, we write a balanced equation for their* ***reaction for complete neutralization*** *by that particular acid after which we proceed with the calculation of the moles just as shown in part (b) above.*

Worked out example 3.3.1.2

You are provided with the following:
FA1 which is a mixture of sodium hydroxide and sodium carbonate
FA2 which is a solution of **0.1M**hydrochloric acid
You are required to determine the percentage of sodium carbonate in the FA1 mixture.
(Na=23, C=12, O=16, H=1)

Procedure:

i) By use of a measuring cylinder, measure and transfer 100cm^3 of FA1 into a 250cm^{3}volumetric flask and make up to the mark with distilled water. Label the resultant solution FA3.
ii) Pipette 25 or 20cm^3 of FA3 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with FA2 from the burette until the end point is reached. Record your results in table I below and then add 2-3 drops of methyl orange indicator to the resultant solution and continue the titration with FA2 until the end point is reached. Record your results in table II below. Repeat the titration until you obtain consistent results.

Volume of pipette used……*25.0*……cm^3

Burette readings	**Table I** (With Phenolphthalein indicator)			**Table II** (With Methyl orange indicator)		
Final burette reading (cm^3)	*27.90*	*28.80*	*27.80*	*40.50*	*41.30*	*40.30*
Initial burette reading(cm^3)	*0.00*	*1.00*	*0.00*	*27.90*	*28.80*	*27.80*
Volume of FA2(cm^3)	*27.90*	*27.80*	*27.80*	*12.60*	*12.50*	*12.50*

Average volume of FA2 used for table I

……$\left(\frac{27.80 + 27.80}{2}\right)$…=…*27.80*……cm^3

……

Average volume of FA2 used for table II …….$\left(\frac{12.50 + 12.50}{2}\right)$…=…*12.50*……cm^3

a) Determine the volume of hydrochloric acid in FA2 required for the neutralisation of:

i) sodium carbonate

…… (*2x12.50) =25.00*……cm^3

ii) sodium hydroxide

……(*27.80—12.50)=15.30*……cm^3

b) Calculate the concentration of:

(i)sodium carbonate in FA1 in gdm^{-3}.

1000cm^3 of FA2 contain 0.1moles of hydrochloric acid

25.00cm^3 of FA2 contain $\left(\frac{0.1}{1000} \times 25.00\right)$ *moles of hydrochloric acid*
= 0.0025 moles of hydrochloric acid

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

Moles of Sodium carbonate = (½ xmoles of hydrochloric acid)

= (½ x 0.0025)

= 0.00125

25cm^3 of FA3 contain 0.00125 moles of sodium carbonate

250cm^3 of FA3 contain $\left(\frac{0.00125}{25} \text{ x } 250\right)$ *moles of sodium carbonate*

= 0.0125 moles of sodium carbonate

100cm^3 of FA1 contain 0.0125 moles of sodium carbonate

1000cm^3 of FA1 contain $\left(\frac{0.0125}{100} \text{ x } 1000\right)$ *moles of sodium carbonate*

= 0.125 mol l^{-1}

Molar Mass of Na_2CO_3= [(23x2) +(12x1) + (16x3)] = 106g

1 mole of sodium carbonate weighs 106g

0.125 moles of sodium hydroxide weigh (106 x0.125)g

= 13.25 gl^{-1}

(ii) sodium hydroxide in FA1 in gdm^{-3}.

1000cm^3 of FA2 contain 0.1moles of hydrochloric acid

15.30cm^3 of FA2 contain $\left(\frac{0.1}{1000} \text{ x } 15.30\right)$ *moles of hydrochloric acid*

=0.00153 moles of hydrochloric acid

$NaOH(aq) + HCl(aq) \rightarrow NaCl(aq) + H_2O(l)$

Moles of sodium hydroxide = (1xmoles of hydrochloric acid)

= (1x0.00153)

= 0.00153

25cm^3 of FA3 contain 0.00153 moles of sodium hydroxide

250cm^3 of FA3 contain $\left(\frac{0.00153}{25} \text{ x } 250\right)$ *moles of sodium hydroxide*

= 0.0153 moles of sodium hydroxide

100cm^3 of FA1 contain 0.0153 moles of sodium hydroxide

1000cm^3 of FA1 contain $\left(\frac{0.0153}{100} \text{ x } 1000\right)$ *moles of sodium hydroxide*

= 0.153 mol l^{-1}

Molar Mass of NaOH= [(23x) +(16x1) + (1x1)] ***= 40g***

1 mole of sodium hydroxide weighs 40g

0. 153 moles of sodium hydroxide weigh (40x0.153)g

= 6.12gl^{-1}

c) Determine the percentage of sodium carbonate in the FA1 mixture.

Total mass of FA1 mixture per litre = (13.25+ 6.12) =19.37g

Percentage of sodium carbonate= $\left(\frac{\mathbf{13.25}}{\mathbf{19.37}} \mathbf{\text{ x } 100}\right)\%$

= 68.40%

Worked out example 3.3.1.3

GA1 which is a mixture of sodium hydrogencarbonate and sodium carbonate
GA2 which is a solution of **0.1M** sulphuric acid
You are required to determine the concentration of sodium hydrogencarbonate and sodium carbonate in GA1 in g l^{-1}.(Na=23, C=12, O=16, H=1)

Procedure:

i) Using a measuring cylinder, measure and transfer 80cm^3 of GA1 into a clean beaker. To the solution in the beaker, add 120cm^3 of distilled water. Label the resultant solution GA3
ii) Pipette 25 or 20cm^3 of GA3 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with GA2 from the burette until the end point is reached. Record your results in table I below and then add 2-3 drops of methyl orange indicator to the resultant solution and continue the titration with GA2 until the end point is reached. Record your results in table II below. Repeat the titration until you obtain consistent results.

Volume of pipette used……*25.0*……cm^3

Burette readings	Table I (With Phenolphthalein indicator)			Table II (With Methyl orange indicator)		
Final burette reading (cm^3)	*12.30*	*14.20*	*12.20*	*42.30*	*44.30*	*42.20*
Initial burette reading(cm^3)	*0.00*	*2.00*	*0.00*	*12.30*	*14.20*	*12.20*
Volume of GA2(cm^3)	*12.30*	*12.20*	*12.20*	*30.00*	*30.10*	*30.00*

Average volume of GA2 used for table I

……$\left(\frac{12.20 + 12.20}{2}\right)$…= …***12.20***……..$cm^3$

Average volume of GA2 used for table II

……..……..…$\left(\frac{30.00 + 30.00}{2}\right)$…= …***30.00***……..$cm^3$

a) Determine the volume of sulphuric acid in GA2 required for the neutralisation of:

i) sodium carbonate.

……..……*(2x12.20)*…=…***24.40***……...cm^3

ii) sodium hydrogencarbonate.

……*(30.00—12.20)*…=…***17.80.***……...cm^3

b) Calculate the concentration of:

(i) sodium carbonate in GA1 in g l^{-1}.

1000cm^3 of GA2 contain 0.1moles of sulphuric acid

24.40cm^3 of GA2 contain $\left(\frac{0.1}{1000} \times 24.40\right)$ *moles of sulphuric acid*

= 0.00244moles of sulphuric acid

$Na_2CO_3(aq) + H_2SO_4(aq) \rightarrow Na_2SO_4(aq) + H_2O(l) + CO_2(g)$

Moles of sodium carbonate = (1 xmoles of sulphuric acid)

= (1x 0.00244)

= 0.00244

25.0cm^3 of GA3 contain 0.00244 moles of sodium carbonate

200cm^3 of GA3 contain $\left(\frac{0.00244}{25.0} \times 200\right)$ *moles of sodium carbonate*

= 0.01952moles of sodium carbonate

80cm^3 of GA1 contain 0.01952moles of sodium carbonate

1000cm^3 of GA1 contain $\left(\frac{0.01952}{80} \times 1000\right)$ *moles of Sodium carbonate*

= 0.244 mol l^{-1}

Molar Mass of Na_2CO_3= [(23x2) +(12x1) + (16x3)]= 106g

1 mole of sodium carbonate weighs 106g

0.244 moles of sodium hydroxide weigh (106 x0.244)g

= 25.864gl^{-1}

(ii) sodium hydrogencarbonate in GA1 in g l^{-1}.

1000cm^3 of GA2 contain 0.1moles of sulphuric acid

17.80cm^3 of GA2 contain $\left(\frac{0.1}{1000} \times 17.80\right)$ *moles of sulphuric acid*

= 0.00178moles of sulphuric acid

$2NaHCO_3(aq) + H_2SO_4(aq) \rightarrow Na_2SO_4(aq) + 2H_2O(l) + 2CO_2(g)$

Moles of Sodium hydrogencarbonate = (2 xmoles of sulphuric acid)

= (2x 0.00178)

= 0.00356

25.0cm^3 of GA3 contain 0.00356 moles of sodium carbonate

200cm^3 of GA3 contain $\left(\frac{0.00356}{25.0} \times 200\right)$ *moles of sodium carbonate*

= 0.0285moles of sodium carbonate

80cm^3 of GA1 contain 0.0285moles of sodium carbonate

1000cm^3 of GA1 contain $\left(\frac{0.0285}{80} \times 1000\right)$ *moles of sodium carbonate*

= 0.356 mol l^{-1}

Molar Mass of $NaHCO_3$= [(23x1) +(1x1)+(12x1) + (16x3)]= ***84g***

1 mole of sodium hydrogencarbonate weighs 84g

0.356 moles of sodium hydrogencarbonate weigh (84 x0.356)g

= 29.904gl^{-1}

3.3.2 Practical Exercises on Double Indicator Titrations using the Continuous Method(Using the Two Indicators Concurrently)

Experiment 3.3.2.1

You are provided with the following:
FA1 which is a solution of **0.3M**hydrochloric acid
FA2 which is a mixture of potassium hydroxide and potassium carbonate
You are required to determine the concentrations of potassium carbonate in gdm^{-3} and hence the percentage of potassium hydroxide in the FA2 mixture.
(K=39, C=12, O=16, H=1)

Procedure:

Pipette 25 or 20cm^3 of FA2 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with FA1 from the burette until the end point is reached. Record your results in table I below and then add 2-3 drops of methyl orange indicator to the resultant solution and continue the titration with FA1 until the end point is reached. Record your results in table II below. Repeat the titration until you obtain consistent results.

Volume of pipette used……..……cm^3

Burette readings	**Table I** (With Phenolphthalein indicator)			**Table II** (With Methyl orange indicator)		
Final burette reading (cm^3)						
Initial burette reading(cm^3)						
Volume of FA1(cm^3)						

Average volume of FA1 used for table I

……cm^3

Average volume of FA1used for table II

……cm^3

Note: 1) With phenolphthalein indicator

$KOH(aq) + HCl(aq) \rightarrow KCl(aq) + H_2O(l)$

$K_2CO_3(aq) + HCl(aq) \rightarrow KHCO_3(aq) + KCl(aq)$

2) With methyl orange indicator

$KHCO_3(aq) + HCl(aq) \rightarrow KCl(aq) + H_2O(l) + CO_2(g)$

(from K_2CO_3)

a) Determine the volume of hydrochloric acid in FA1required for the neutralization of:
   i) potassium carbonate.

……

……. …… ……

ii) potassium hydroxide.

……

……

b)Calculate the concentration of potassium carbonate in FA2 in gdm^{-3}

……

……

……

……

……

c)Determine the percentage of potassium hydroxide in the FA2 mixture.

……

……

……

……

……

……

Experiment 3.3.2.2

You are provided with the following:

FA1 which is a solution of **0.2M**nitric acid

FA2 which is a mixture of sodium carbonate and sodium hydrogencarbonate

You are required to determine the concentrations of sodium carbonate and sodium hydrogencarbonate in gdm^{-3} and hence the percentage of sodium hydrogencarbonatein the FA2 mixture.

(Na=23, C=12, O=16, H=1)

Procedure

Pipette 25 or $20cm^3$ of FA2 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with FA1 from the burette until the end point is reached. Record your results in table I below and then add 2-3 drops of methyl orange indicator to the resultant solution and continue the titration with FA1until the end point is reached. Record your results in table II below. Repeat the titrations until you obtain consistent results.

Volume of pipette used……..……cm^3

Burette readings	**Table I** (With phenolphthalein indicator)			**Table II** (With methyl orange indicator)		
Final burette reading (cm^3)						
Initial burette reading(cm^3)						
Volume of FA1(cm^3)						

Average volume of FA1 used for table I:

……cm^3

Average volume of FA1used for table II

……cm^3

Note: 1) With phenolphthalein indicator

$Na_2CO_3(aq) + HNO_3(aq) \rightarrow NaHCO_3(aq) + NaNO_3(aq)$

$NaHCO_3(aq) + HNO_3(aq) \rightarrow$ *No reaction occurs*

2) With methyl orange indicator

$NaHCO_3(aq) + HNO_3(aq) \rightarrow NaNO_3(aq) + H_2O(l) + CO_2(g)$

(Initially present in FA2)

$NaHCO_3(aq) + HNO_3(aq) \rightarrow NaNO_3(aq) + H_2O(l) + CO_2(g)$

(from Na_2CO_3)

a) Determine the volume of nitric acid in FA1required for the neutralisation of:

i) sodium carbonate

…… ……

……. ……

ii) sodium hydrogencarbonate

……

……

b)Calculate the concentration of sodium carbonate in FA2 in gdm^{-3}

……

……

……

……

……

……

(c) Determine the concentration of sodium hydrogencarbonate inthe FA2 mixture in gdm^{-3}.

……

……

……

……

……

……

(d) Calculate the percentage of sodium hydrogencarbonate in the FA2 mixture.

……

……

……

……

Experiment 3.3.2.3

You are provided with the following:
FA1 which is a mixture of sodium hydroxide and sodium carbonate
FA2 which is a solution of **0.15M** hydrochloric acid
You are required to determine the percentage of sodium carbonate in the FA1 mixture.
(Na=23, C=12, O=16, H=1)

Procedure:

i) By use of a measuring cylinder, measure and transfer $100cm^3$ of FA1 into a $250cm^3$volumetric flask and make up to the mark with distilled water. Label the resultant solution FA3.

ii) Pipette 25 or $20cm^3$ of FA3 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with FA2 from the burette until the end point is reached. Record your results in table I below and then add 2-3 drops of methyl orange indicator to the resultant solution and continue the titration with FA2 until the end point is reached. Record your results in table II below. Repeat the titration until you obtain consistent results.

Volume of pipette used……...……cm^3

Burette readings	**Table I** (With phenolphthalein indicator)			**Table II** (With methyl orange indicator)		
Final burette reading (cm^3)						
Initial burette reading(cm^3)						
Volume of FA2 (cm^3)						

Average volume of FA2 used for table I

……cm^3

Average volume of FA2used for table II

……cm^3

a) Determine the volume of hydrochloric acid in FA2 required for the neutralisation of:

i) sodium carbonate

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ii) sodium hydroxide

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d) Calculate theconcentration of:

(i)sodium carbonate in FA1 in gdm^{-3}.

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(ii) sodium hydroxide in FA1 in gdm^{-3}.

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e) Determine the percentage of sodium carbonate in the FA1 mixture.

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Experiment 3.3.2.4

You are provided with the following:
GA1 which is a mixture of sodium carbonate and sodiumhydrogencarbonate
GA2 which is a solution of **0.1M**nitric acid
You are required to determine the percentage of sodium hydrogencarbonate in GA1.
(Na=23, C=12, O=16, H=1)

Procedure:

i) Using a measuring cylinder, measure and transfer 100cm^3 of GA1 into a clean beaker. To the solution in the beaker, add 100cm^3 of distilled water. Label the resultant solution GA3.

ii) Pipette 25 or 20cm^3 of GA3 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with GA2 from the burette until the end point is reached. Record your results in table I below and then add 2-3 drops of methyl orange indicator to the resultant solution and continue the titration with GA2 until the end point is reached. Record your results in table II below. Repeat the titration until you obtain consistent results.

Volume of pipette used……..……cm^3

Burette readings	**Table I** (With phenolphthalein indicator)			**Table II** (With methyl orange indicator)		
Final burette reading (cm^3)						
Initial burette reading(cm^3)						
Volume of GA2(cm^3)						

Average volume of GA2 used for table I

……cm^3

Average volume of GA2 used for table II

……cm^3

a) Determine the volume of nitric acid in GA2 required for the neutralisation of:

i) sodium carbonate

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ii) sodium hydrogencarbonate

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b) Calculate theconcentration of:

(i)sodium carbonate in GA1 in gl^{-1}.

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(ii) sodium hydrogencarbonate in GA1 in gl^{-1}.

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c) Determine the percentage of sodium hydrogencarbonate in GA1.

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Experiment 3.3.2.5

You are provided with the following:
HA1 which is a mixture of sodium hydrogencarbonate and sodiumhydroxide
HA2 which is a solution of **0.1M** sulphuric acid
You are required to determine the concentration of sodium hydrogencarbonate and sodium hydroxide in HA1 in gdm^{-3}.
(Na=23, C=12, O=16, H=1)

Procedure:

i) Using a measuring cylinder, measure and transfer 125cm^3 of HA1 into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label the resultant solution HA3.
ii) Pipette 25 or 20cm^3 of HA3 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with HA2 from the burette until the end point is reached. Record your results in table I below and then add 2-3 drops of methyl orange indicator to the resultant solution and continue the titration with HA2 until the end point is reached. Record your results in table II below. Repeat the titration until you obtain consistent results.

Volume of pipette used……...……cm^3

Burette readings	**Table I** (With phenolphthalein indicator)			**Table II** (With methyl orange indicator)		
Final burette reading (cm^3)						
Initial burette reading(cm^3)						
Volume of HA2(cm^3)						

Average volume of HA2 used for table I

……..cm^3

Average volume of HA2 used for table II

……..cm^3

a) Determine the volume of sulphuric acid in HA2 required for the neutralisation of:

i) sodium hydroxide

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……. …… ……

ii) sodium hydrogencarbonate

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b) Calculate theconcentration of:

(i)sodium hydroxide in HA1 in g dm^{-3}.

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(ii) sodium hydrogencarbonate in HA1 in g dm^{-3}.

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B) The Two Step Method(Using the Two Indicators Separately)

In the **Two Step method**, an aliquot (a portion) of the mixture is pipetted and titrated against a standard mineral acid using phenolphthalein indicator and the volume, $V_3$of the mineral acid required to reach the end point is noted.

The solution in the conical flask is then poured awayand a fresh aliquot (a fresh portion) of the mixture is pipetted into the clean conical flask, 2-3 drops of Methyl orange indicator are added and the aliquot titratedagainst the same standard mineral acid, and the volume, V_4 of the mineral acid required to reach the end point is noted.

3.3.3Worked out Examples on Double Indicator Titrations using the Two Step Method(Using the Two Indicators Separately)

Worked out example 3.3.3.1

You are provided with the following:

FA1 which is a mixture of sodium hydroxide and sodium carbonate

FA2 which is a solution of **0.2M**hydrochloric acid

You are required to determine the concentrations of sodium hydroxide and sodium carbonate in grams per litre. (Na=23, C=12, O=16, H=1)

Procedure I

Pipette 25 or 20cm^3 of FA1 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with FA2 from the burette until the end point is reached. Repeat the titration until you obtain consistent results.

Record your results in table I below.

Volume of pipette used……*25.0*……cm^3

Table I (With phenolphthalein indicator)

Final burette reading (cm^3)	*22.40*	*22.20*	*32.20*
Initial burette reading(cm^3)	*0.00*	*0.00*	*10.00*
Volume of FA2(cm^3)	*22.40*	*22.20*	*22.20*

Average volume of FA2 used for table I ……$\left(\frac{22.20 + 22.20}{2}\right)$=***22.20***……cm^3

Procedure II

Pipette 25 or20cm^3 of FA1 into a clean conical flask. Add 2-3 drops of methyl orange indicator and titrate with FA2 from the burette until the end point is reached. Repeat the titration until you obtain consistent results.Record your results in table II below.

Volume of pipette used……*25.0*……cm^3

Table II (With methyl orange indicator)

Final burette reading (cm^3)	*31.40*	*31.20*	*39.10*
Initial burette reading(cm^3)	*0.00*	*0.00*	*8.00*
Volume of FA2(cm^3)	*31.40*	*31.20*	*31.10*

Average volume of FA2 used for table II

……$\left(\frac{31.20 + 31.10}{2}\right)$…= ***31.15***……cm^3

Note: 1) With phenolphthalein indicator

$NaOH(aq) + HCl(aq) \rightarrow NaCl(aq) + H_2O(l)$

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

2) With methyl orange indicator

$NaOH(aq) + HCl(aq) \rightarrow NaCl(aq) + H_2O(l)$

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

a)Determine the volume of hydrochloric acid in FA2 required for the complete neutralization of:

i) sodium carbonate ……*=2(31.15-22.20)cm^3*……
…… ……*= (2x8.95)cm^3*……
……***=17.90cm^3***……
ii) sodium hydroxide……*=(31.15-17.90)cm^3*……
……***=13.25cm^3***……

b) Calculate the concentration of sodium carbonate in FA1 in grams per litre.

1000cm^3 of FA2 contain 0.2moles of hydrochloric acid

17.90cm^3 of FA2 contain $\left(\frac{0.2}{1000} \times 17.90\right)$ *moles of hydrochloric acid*

= 0.00358 moles of hydrochloric acid

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

Moles of sodium carbonate = $\frac{1}{2}$*x moles of hydrochloric acid*

Moles of sodium carbonate = $\frac{1}{2}$ $\boldsymbol{x\ 0.00358}$

= 0.00179 moles of sodium carbonate

25cm^3 of FA1 contain 0.00179 moles of sodium carbonate

1000 cm^3 of FA1 contain $\left(\frac{0.00179}{25} \times 1000\right)$ *moles of sodium carbonate*

= 0.0716 mol l^{-1}

Molar mass of Na_2CO_3 = [(23x2)+(12x1)+(16x3)] ***= 106g***

1 mole of sodium carbonate weighs 106g

0.08 moles of sodium carbonate weigh (106x0.0716)g

= 7.59gl^{-1}

c) Determine concentration of sodium hydroxide in FA1 in grams per litre.

1000cm^3 of FA2 contain 0.2moles of hydrochloric acid

13.25cm^3 of FA2 contain $\left(\frac{0.2}{1000} \times 13.25\right)$ *moles of hydrochloric acid*

***=0.00265** moles of hydrochloric acid*

NaOH(aq) + HCl(aq) → NaCl(aq) + H_2O(l)

Moles of sodium hydroxide = (1xmoles of hydrochloric acid)

= (1x0.00265)

= 0.00265

25cm^3 of FA1 contain 0.00265 moles of sodium hydroxide

1000cm^3 of FA1 contain $\left(\frac{0.00265}{25} \times 1000\right)$ *moles of sodium hydroxide per litre*

= 0.106 mol l^{-1}

*Molar Mass of NaOH= [(23x) + (16x1) + (1x1)]= **40g***

1 mole of sodium hydroxide weighs 40g

0.18 moles of sodium hydroxide weigh (40x0.106)g

= 4.24gl^{-1}

Worked out example 3.3.3.2

You are provided with the following:

HA1 which is a solution of **0.1M**hydrochloric acid

HA2 which is a mixture of sodium hydroxide and sodium carbonate

You are required to determine the percentage of sodium carbonate in the HA1 mixture.

(Na=23, C=12, O=16, H=1)

Procedure I

i) By use of a measuring cylinder, measure and transfer 100cm^3 of HA2 into a 250cm^{3}volumetric flask and make up to the mark with distilled water. Label the resultant solution HA3.

ii) Pipette 25 or 20cm^3 of HA3 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with HA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results.

Record your results in table I below.

Volume of pipette used……*25.0*……cm^3

Table I (With Phenolphthalein indicator)

Final burette reading (cm^3)	*20.10*	*22.00*	*24.00*
Initial burette reading(cm^3)	*0.00*	*2.00*	*4.00*
Volume of HA1(cm^3)	*20.10*	*20.00*	*20.00*

Average volume of HA1 used for table I

……$\left(\frac{20.00 + 20.00}{2}\right)$…= *20.00*…….. *cm*^{3}

Procedure II

Pipette 25 or20cm^3 of HA3 into a clean conical flask. Add 2-3 drops of methyl orange indicator and titrate with HA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results.Record your results in table II below.

Volume of pipette used……*25.0*……..cm^3

Table II (With Methyl orange indicator)

Final burette reading (cm^3)	*30.40*	*36.20*	*30.10*
Initial burette reading(cm^3)	*0.00*	*6.00*	*0.00*
Volume of HA1(cm^3)	*30.40*	*30.20*	*30.10*

Average volume of HA1 used for table II

……$\left(\frac{30.20 + 30.10}{2}\right)$…=…*30.15*……cm^3

a) Determine the volume of hydrochloric acid in HA1 required for the complete neutralisation of:

i) sodium carbonate

…….*2(30.15—20.00)...=...(2x10.15)...=...**20.30.***.....cm^3

ii) sodium hydroxide

..... 30.15—*2(30.15—20.00)..=..(30.15—20.30)..= **9.85.***.....cm^3

b) Calculate the concentration of:

i) sodium carbonate in HA2 in grams per litre.

1000cm^3 of HA1 contain 0.1moles of hydrochloric acid

20.30cm^3 of HA1 contain $\frac{0.1}{1000}$ *x 20.30moles of hydrochloric acid*

= 0.00203 moles of hydrochloric acid

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

Moles of sodium carbonate = (½ xmoles of hydrochloric acid)

= (½ x 0.00203)

= 0.001015

25cm^3 of HA3 contain 0.001015 moles of sodium carbonate

250cm^3 of HA3 contain $\left(\frac{0.001015}{25} \text{ x } 250\right)$*moles of sodium carbonate*

= 0.01015moles of sodium carbonate

100cm^3 of HA2 contain 0.01015 moles of sodium carbonate

1000cm^3 of HA2 contain $\left(\frac{0.01015}{100} \text{ x } 1000\right)$*moles of sodium carbonate*

= 0.1015 mol l^{-1}

Molar Mass of Na_2CO_3= [(23x2) +(12x1) + (16x3)]= ***106g***

1 mole of sodium carbonate weighs 106g
0.1015 moles of sodium hydroxide weigh (106 x0.1015)g
= 10.76 gl^{-1}

ii) sodium hydroxide in HA2 in grams per litre.

1000cm^3 of HA1 contain 0.1moles of hydrochloric acid
9.85cm^3 of HA1 contain $\left(\frac{0.1}{1000} \times 9.85\right)$ *moles of hydrochloric acid*
= 0.000985 moles of hydrochloric acid
NaOH(aq) + HCl(aq) → NaCl(aq) + H_2O(l)
Moles of sodium hydroxide = (1xmoles of hydrochloric acid)
= (1x0.000985)
= 0.000985
25cm^3 of HA3 contain 0.000985 moles of sodium hydroxide
250cm^3 of HA3 contain $\left(\frac{0.000985}{25} \times 250\right)$ *moles of sodium hydroxide*
= 0.00985moles of sodium hydroxide
100cm^3 of HA2 contain 0.00985moles of sodium hydroxide
1000cm^3 of HA2 contain $\frac{0.00985 \times 1000}{100}$ *moles of sodium hydroxide*
= 0.0985 mol l^{-1}
Molar Mass of NaOH= [(23x) +(16x1) + (1x1)]= ***40g***
1 mole of sodium hydroxide weighs 40g
0.0985 moles of sodium hydroxide weigh (40x0.0985)g
= 3.94 gl^{-1}

c) Determine the percentage of sodium carbonate in the HA2 mixture.

Total mass of FA1 mixture per litre = (10.76+ 3.94)g
=14.70g
Percentage of sodium carbonate = $\left(\frac{\mathbf{10.76}}{\mathbf{14.70}} \mathbf{\times 100}\right)\%$
= 73.20%

Worked out example 3.3.3.3

FA1 which is a mixture of sodium carbonate and sodium hydrogencarbonate
FA2 which is a solution of **0.1M**nitric acid
You are required to determine the percentage of sodium hydrogencarbonate in FA1.
(Na=23, C=12, O=16, H=1)

Procedure I

i) By use of a measuring cylinder, measure and transfer 125cm^3 of FA1 into a clean beaker. Add 75cm^3 of distilled water and label the resultant solution FA3.
ii) Pipette 25 or 20cm^3 of FA3 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with FA2 from the burette until the end point is reached. Repeat the titration until you obtain consistent results.Record your results in table I below.

Volume of pipette used……*25.0*……cm^3

Table I (With phenolphthalein indicator)

Final burette reading (cm^3)	*12.20*	*14.00*	*16. 00*
Initial burette reading(cm^3)	*0.00*	*2.00*	*4.00*
Volume of FA2(cm^3)	*12.20*	*12.00*	*12.00*

Average volume of FA2 used for table I

……$\left(\frac{12.00 + 12.00}{2}\right)$= *12.00*……..cm^3

Procedure II

Pipette 25 or20cm^3 of FA3 into a clean conical flask. Add 2-3 drops of Methyl orange indicator and titrate with FA2 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in table II below.

Volume of pipette used……*25.0*……cm^3

Table II (With methyl orange indicator)

Final burette reading (cm^3)	*36.60*	*38.70*	*36.60*
Initial burette reading(cm^3)	*0.00*	*2.00*	*0.00*
Volume of FA2(cm^3)	*36.60*	*36.70*	*36.60*

Average volume of FA2 used for table II

……$\left(\frac{36.60 + 36.60}{2}\right)$…=……*36.60*……..cm^3

a) Determine the volume of nitric acid in FA2 required for the complete neutralisation of:

i) sodium carbonate

…….…….*(2x12.00) =* ***24.00***……cm^3

ii) sodium hydrogecarbonate

……..……..... *36.60—(2x12.00) =(* 36.60*—24.00) =****12.60***……cm^3

b) Calculate the concentration of:

i) sodium carbonate in FA1 in grams per litre.

1000cm^3 of FA2 contain 0.1moles nitric acid

24.00cm^3 of FA2 contain $\left(\frac{0.1}{1000} \times 24.00\right)$ *moles of nitric acid*

= 0.0024 moles of nitric acid

$Na_2CO_3(aq) + 2HNO_3(aq) \rightarrow 2NaNO_3(aq) + H_2O(l) + CO_2(g)$

Moles of sodium carbonate = (½ xmoles of nitric acid)
= (½ x 0.0024)
= 0.0012
25.0cm³ of FA3 contain 0.0012 moles of sodium carbonate
200cm³ of GA3 contain $\left(\frac{0.0012}{25.0} \times 200\right)$ *moles of sodium carbonate*
= 0.0096moles of sodium carbonate
125cm³ of FA1 contain 0.0096moles of sodium carbonate
1000cm³ of FA1 contain $\left(\frac{0.0096}{125} \times 1000\right)$ *moles of sodium carbonate*
= 0.0768 mol l⁻¹
Molar Mass of Na_2CO_3= [(23x2) +(12x1) + (16x3)]= 106g

1 mole of sodium carbonate weighs 106g
0.0768 moles of sodium carbonate weigh (106 x0.0768)g
= 8.14gl⁻¹

ii) sodium hydrogencarbonate in FA1 in grams per litre.

1000cm³ of FA2 contain 0.1moles of nitric acid
12.60cm³ of FA2 contain $\left(\frac{0.1}{1000} \times 12.60\right)$ *moles of nitric acid*
= 0.00126moles of nitric acid
$NaHCO_3(aq) + HNO_3(aq) \rightarrow NaNO_3(aq) + H_2O(l) + CO_2(g)$
Moles of sodium hydrogencarbonate = (1 xmoles of nitric acid)
= (1x 0.00126)
= 0.00126
25.0cm³ of FA3 contain 0.00126 moles of sodium hydrogencarbonate
200cm³ of FA3 contain $\left(\frac{0.00126}{25.0} \times 200\right)$ *moles of sodium hydrogencarbonate*
= 0.01moles of sodium hydrogencarbonate
125cm³ of FA1 contain 0.01moles of sodium hydrogencarbonate
1000cm³ of FA1 contain $\left(\frac{0.01}{125} \times 1000\right)$ *moles of sodium hydrogencarbonate*
= 0.08 mol l⁻¹
Molar Mass of $NaHCO_3$= [(23x1) +(1x1)+(12x1) + (16x3)]= 84g

1 mole of sodium hydrogencarbonate weighs 84g
0.08 moles of sodium hydroxide weigh (84 x0.08)g
= 6.72 gl⁻¹

c) Determine the percentage of sodium hydrogencarbonate in the FA1 mixture.

Total mass of FA1 mixture per litre = (8.14+ 6.72) =14.86g
Percentage of sodium hydrogencarbonate = $\left(\frac{\mathbf{6.72}}{\mathbf{14.86}} \times \mathbf{100}\right)\%$
= 45.22%

3.3.4 Practical Exercises on Double Indicator Titrations using the Two Step Method(Using the Two Indicators Separately)

Experiment 3.3.4.1
You are provided with the following:
FA1 which is 0.1M sulphuric acid
FA2 which is a mixture of sodium hydroxide and sodium carbonate
You are required to determine the percentage ofsodium carbonate inFA2.
(Na=23, C=12, O=16, H=1)

Procedure I
Pipette 25or 20cm^3 of FA2 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with FA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results.
Record your results in table I below.

Volume of pipette used……cm^3

Table I (With phenolphthalein indicator)

Final burette reading (cm^3)			
Initial burette reading(cm^3)			
Volume of FA1(cm^3)			

Average volume of FA1 used for table I
…….....cm^3

Procedure II
Pipette 25or 20cm^3 of FA2 into a clean conical flask. Add 2-3 drops of methyl orange indicator and titrate with FA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results.Record your results in table II below.

Volume of pipette used……cm^3

Table II (With methyl orange indicator)

Final burette reading (cm^3)			
Initial burette reading(cm^3)			
Volume of FA1(cm^3)			

Average volume of FA1 used for table II
…….....cm^3

Note: 1) With phenolphthalein indicator

$2NaOH(aq) + H_2SO_4(aq) \rightarrow Na_2SO_4(aq) + 2H_2O(l)$

$2Na_2CO_3(aq) + H_2SO_4(aq) \rightarrow 2NaHCO_3(aq) + Na_2SO_4(aq)$

2) With methyl orange indicator

$2NaOH(aq) + H_2SO_4(aq) \rightarrow Na_2SO_4(aq) + 2H_2O(l)$

$Na_2CO_3(aq) + H_2SO_4(aq) \rightarrow Na_2SO_4(aq) + H_2O(l) + CO_2(g)$

a)Determine the volume of sulphuric acid in FA1 required for the complete neutralisation of:

i) sodium carbonate.

……

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ii) sodium hydroxide.

……

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b)Calculate the concentration of sodium carbonate in FA2 in gdm^{-3}.

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c)Determine the concentration of sodium hydroxide in FA2 in gdm^{-3}.

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d) Calculate the percentage of sodium carbonate in FA2.

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Experiment 3.3.4.2
You are provided with the following:
FA1 which is **0.2M**nitric acid
FA2which is a mixture of sodium hydroxide and sodium hydrogencarbonate
You are required to determine the percentage of sodium hydrogencarbonate in FA2.
(Na=23, C=12, O=16, H=1)

Procedure

Pipette 25cm^3 of FA2 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with FA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in table I below.

Volume of pipette used……..……cm^3

Table I (With phenolphthalein indicator)

Final burette reading (cm^3)			
Initial burette reading(cm^3)			
Volume of FA1(cm^3)			

Average volume of FA1 used for table I

……cm^3

Volume of pipette used……cm^3

Table II (With methyl orange indicator)

Final burette reading (cm^3)			
Initial burette reading(cm^3)			
Volume of FA1(cm^3)			

Average volume of FA1 used for table II

……cm^3

Note: 1) With phenolphthalein indicator

$NaOH(aq) + HNO_3(aq) \rightarrow NaNO_3(aq) + H_2O(l)$

$NaHCO_3(aq) + HNO_3(aq) \rightarrow$ *No reaction occurs*

2) With methyl orange indicator

$NaOH(aq) + HNO_3(aq) \rightarrow NaNO_3(aq) + H_2O(l)$

$NaHCO_3(aq) + HNO_3(aq) \rightarrow NaNO_3(aq) + H_2O(l) + CO_2(g)$

e)Determine the volume of nitric acid in FA1 required for the complete neutralisation of:

i) sodium hydroxide.

……..…….…….....cm^3

……...…….....

ii) sodium hydrogencarbonate

……cm^3

……..……..…….

f)Calculate the concentration of sodium hydroxide in FA2 in gdm^{-3}.

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g)Determine the:

i) concentration of sodium hydrogencarbonate in FA2 in gdm^{-3}.

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ii) percentage of sodium hydrogencarbonate in FA2.

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Experiment 3.3.4.3
You are provided with the following:
HA1 which is **0.1M**sulphuric acid
HA2 which is a mixture of sodium carbonate and sodium hydrogencarbonate.
You are required to determine the percentage of sodium carbonate in HA2.
(Na=23, C=12, O=16, H=1)

Procedure I
Pipette 25or 20cm^3 of HA2 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with HA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in Table I below.

Volume of pipette used……..……cm^3

Table I (With phenolphthalein indicator)

Final burette reading (cm^3)			
Initial burette reading(cm^3)			
Volume of HA1(cm^3)			

Average volume of HA1 used for table I

……cm^3

Procedure II
Pipette 25or 20cm^3 of HA2 into a clean conical flask. Add 2-3 drops of methyl orange indicator and titrate with HA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results.Record your results in Table II below.

Volume of pipette used……cm^3

Table II (With methyl orange indicator)

Final burette reading (cm^3)			
Initial burette reading(cm^3)			
Volume of HA1(cm^3)			

Average volume of HA1 used for table II

……cm^3

Note:

1) With phenolphthalein indicator

$2Na_2CO_3(aq) + H_2SO_4(aq) \rightarrow 2NaHCO_3(aq) + Na_2SO_4(aq)$

$NaHCO_3(aq) + H_2SO_4(aq) \rightarrow$ *No reaction*

2) With methyl orange indicator

$Na_2CO_3(aq) + H_2SO_4(aq) \rightarrow Na_2SO_4(aq) + H_2O(l) + CO_2(g)$

$2NaHCO_3(aq) + H_2SO_4(aq) \rightarrow Na_2SO_4(aq) + 2H_2O(l) + 2CO_2(g)$

a)Determine the volume of sulphuric acid in HA1 required for the complete neutralisation of:

i) sodium carbonate

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ii) sodium hydrogencarbonate

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b)Calculate the concentration of sodium carbonate in HA2 in gdm^{-3}.

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c) Determine the concentration of sodium hydrogencarbonate in HA2 in gdm^{-3}.

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d) Calculate the percentage of sodium carbonate in HA2.

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Experiment 3.3.4.4
You are provided with the following:
FA1 which is a mixture of sodium hydroxide and sodium carbonate
FA2 which is a solution of **0.1M**hydrochloric acid
You are required to determine the percentage of sodium hydroxide in the FA1 mixture.
(Na=23, C=12, O=16, H=1)

Procedure I

i) By use of a measuring cylinder, measure and transfer $100cm^3$ of FA1 into a $250cm^3$volumetric flask and make up to the mark with distilled water. Label the resultant solution FA3.
ii) Pipette 25 or $20cm^3$ of FA3 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with FA2 from the burette until the end point is reached. Repeat the titration until you obtain consistent results.
Record your results in table I below.

Volume of pipette used……..……cm^3

Table I (With phenolphthalein indicator)

Final burette reading (cm^3)			
Initial burette reading(cm^3)			
Volume of FA2(cm^3)			

Average volume of FA2 used for table I

……......cm^3

Procedure II

Pipette 25 or20cm^3 of FA3 into a clean conical flask. Add 2-3 drops of methyl orange indicator and titrate with FA2 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in table II below.

Volume of pipette used……...……cm^3

Table II (With methyl orange indicator)

Final burette reading (cm^3)			
Initial burette reading(cm^3)			
Volume of FA2(cm^3)			

Average volume of FA2 used for table II

……cm^3

a) Determine the volume of hydrochloric acid in FA2 required for the complete neutralisation of:

i) sodium carbonate

.....….....cm^3

.....

ii) sodium hydroxide

.....…….....….cm^3

…….....

b) Calculate the concentration of:

i) sodium carbonate in FA1 in grams per litre.

……

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ii) sodium hydroxide in FA1 in grams per litre.

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c) Determine the percentage of sodium hydroxide in the FA1 mixture.

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Experiment 3.3.4.5

You are provided with the following:

FA1 which is a mixture of sodium carbonate and sodium hydrogencarbonate

FA2 which is a solution of **0.1M** nitric acid

You are required to determine the percentage of sodium carbonate in FA1.

(Na=23, C=12, O=16, H=1)

Procedure I

i) By use of a measuring cylinder, measure and transfer 120cm^3 of FA1 into a clean beaker. Add 80cm^3 of distilled water and label the resultant solution FA3.

ii) Pipette 25 or 20cm^3 of FA3 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with FA2 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in table I below.

Volume of pipette used……..……cm^3

Table I (With Phenolphthalein indicator)

Final burette reading (cm^3)			
Initial burette reading(cm^3)			
Volume of FA2(cm^3)			

Average volume of FA2 used for table I

……cm^3

Procedure II

Pipette 25 or20cm^3 of FA3 into a clean conical flask. Add 2-3 drops of methyl orange indicator and titrate with FA2 from the burette until the end point is reached. Repeat the titration until you obtain consistent results.Record your results in table II below.

Volume of pipette used……..……cm^3

Table II (With methyl orange indicator)

Final burette reading (cm^3)			
Initial burette reading(cm^3)			
Volume of FA2(cm^3)			

Average volume of FA2 used for table II

…….cm^3

a) Determine the volume of nitric acid in FA2 required for the complete neutralisation of:
   i) sodium carbonate

   ……

   .....

   ii) sodium hydrogecarbonate

   .....

   .....

b) Calculate the concentration of:
   i) sodium carbonate in FA1 in grams per litre.

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ii) sodium hydrogencarbonate in FA1 in grams per litre.

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c) Determine the percentage of sodium carbonate in the FA1 mixture.

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Experiment 3.3.4.6
You are provided with the following:
FA1 which is **0.1M** sulphuric acid
FA2which is a mixture of sodium hydroxide and sodium hydrogencarbonate
You are required to determine the percentage of sodium hydroxide in FA2.
(Na=23, C=12, O=16, H=1)

Procedure

i) By use of a measuring cylinder, measure and transfer 100cm^3 of FA2 into a 250cm^3volumetric flask and make up to the mark with distilled water. Label the resultant solution FA3.

ii) Pipette 25cm^3 of FA3 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with FA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results.
Record your results in table I below.

Volume of pipette used……..……cm^3

Table I (With phenolphthalein indicator)

Final burette reading (cm^3)			
Initial burette reading(cm^3)			
Volume of FA1(cm^3)			

Average volume of FA1 used for table I

……cm^3

Volume of pipette used……cm^3

Table II (With methyl orange indicator)

Final burette reading (cm^3)			
Initial burette reading(cm^3)			
Volume of FA1(cm^3)			

Average volume of FA1 used for table II

……cm^3

a) Determine the volume of sulphuric acid in FA1 required for the complete neutralisation of:
i) sodium hydroxide

……..……..……..……

……..……..……

ii) sodium hydrogencarbonate

……

……

b) Calculate the concentration of sodium hydroxide in FA2 in gdm^{-3}.

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c) Determine the:

i) concentration of sodium hydrogencarbonate in FA2 in gdm^{-3}.

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ii) percentage of sodium hydrogencarbonate in FA2.

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CHAPTER FOUR
4.0REDOX TITRATIONS

4.1Introduction

Redox titrations are based on redox reactions. Redox reactions are reactions in which both reduction and oxidation occur simultaneously.

Oxidation can be defined in three different ways as either:

- The addition of oxygen to a substance OR
- The removal of electrons from a substance OR
- The removal of hydrogen from a substance.

Reduction can be defined in three different ways as either:

- The removal of oxygen from a substance OR
- The addition of electrons to a substance OR
- The addition of hydrogen to a substance.

In redox titrations, however, we shall look at oxidation and reduction in terms of transfer of electrons. The substance that loses or gains electrons may be an element, compound or ion.

4.1.1 Terms commonly used in Redox Reactions

a) Oxidation number

The Oxidation number of an atomis the real or hypothetical charge that the atom bearsin its pure state or in its compound basing on specific rules.

When the oxidation number of a substance increases, the substance is said to be oxidized and when the oxidation number of a substance decreases, the substance is said to be reduced.

b) Oxidizing agent

An oxidizing agent is a substance which removes electrons from other substances. In the process of removing electrons from other substances,the oxidizing agent gains those electrons and as a result, its oxidation number decreases. Therefore, an oxidizing agent is always reduced at the end of a redox reaction.

c) Reducing agent

A reducing agent is a substance which donates electrons to other substances. In the process of donating the electrons, it loses them, and the loss of the negative charges (in form of electrons),results in increase of the positive charge, which corresponds to an increase in the oxidation number. Therefore, a reducing agent is always oxidized at the end of a redox reaction.

d) Half equations

Half equations are separate equations that specify which substances gain or lose electrons in a redox reaction.Zinc powder, when added to a blue solution of copper(II) sulphate, and the mixture warmed gently, it reduces the copper(II) ions in the blue solution to copper metal which appears as a brown solid and itself oxidized to zinc ions which appear as a colourless solution.Equations (1) and (2) below are half equations.

$Zn(s) \rightarrow Zn^{2+}(aq) + 2e-$ …….....(1)

$Cu^{2+}(aq) + 2e- \rightarrow Cu(s)$ …….....(2)

e) OverallRedox Equation

An overall redox equation is an equation that is obtained after combining two half equations where by one half equation represents an oxidation process and the other represents a reduction process.When the two half equations are combined, the two electrons on each side cansel to give equation (3) below which is the overall redox equation for the two half equations above.

$Zn(s) + Cu^{2+}(aq) \rightarrow Zn^{2+}(aq) + Cu(s)$ …..……(3)

4.1.2Commonoxidizing agents

The most commonly used oxidizing agents include the following chemical species:

- Acidified manganate(VII) ions,MnO_4^-/ H^+
- Acidified dichromate(VI) ions,$Cr_2O_7^{2-}/ H^+$
- Acidified chromate(VI) ions, CrO_4^{2-}/ H^+
- Acidified chlorate ions, ClO_3^-/ H^+
- Acidified oxochlorate ions,ClO^-/ H^+
- Acidified iodate ions, IO_3^-/ H^+
- Persulphate ions, $S_2O_8^{2-}$
- Acidified manganese(IV) oxide, MnO_2/H^+
- Concentratednitric acid, HNO_3
- Concentratedsulphuric acid, H_2SO_4
- Acidified manganate(VI) ions, MnO_4^{2-}/ H^+
- Acidified hydrogen peroxide, H_2O_2/ H^+
- Oxygen,O_2, e.t.c.

4.1.3Half equations of common oxidizing agents

$Cu^{2+}(aq) + 2e- \rightarrow Cu(s)$

$Fe^{2+}(aq) + 2e^- \rightarrow Fe(s)$

$Ag^+(aq) + e^- \rightarrow Ag(s)$

$I_2(aq) + 2e^- \rightarrow 2I^-(aq)$

$Cl_2(aq) + 2e^- \rightarrow 2Cl^-(aq)$

$Br_2(aq) + 2e^- \rightarrow 2Br^-(aq)$

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

$Cr_2O_7^{2-}(aq) + 14H^+(aq) + 6e^- \rightarrow Cr^{3+}(aq) + 7H_2O(l)$

$ClO_3^-(aq) + 6H^+(aq) + 4e^- \rightarrow Cl^-(aq) + 3H_2O(l)$

$ClO^-(aq) + 2H^+(aq) + 2e- \rightarrow Cl^-(aq) + H_2O(l)$

$IO_3^-(aq) + 6H^+(aq) + 6e^- \rightarrow I^-(aq) + 3H_2O(l)$ {when acidified with concentrated acid}

$2IO_3^-(aq) + 12H^+(aq) + 10e^- \rightarrow I_2(aq) + 6H_2O(l)$ {when acidified with diluteacid}

$H_2O_2(aq) + 2H^+(aq) + 2e^- \rightarrow 2H_2O(l)$

$MnO_2(aq) + 4H^+(aq) + 3e^- \rightarrow Mn^{2+}(aq) + 2H_2O(l)$

$2O_2(g) + 4e^- \rightarrow 2O_2^{2-}(aq)$

$CrO_4^{2-}(aq) + 8H^+(aq) + 3e^- \rightarrow Cr^{3+}(aq) + 4H_2O(l)$

$S_2O_8^{2-}(aq) + 2e^- \rightarrow 2SO_4^{2-}(aq)$

4.1.4Common Reducing Agents

The most commonly used reducing agents include the following Chemical species:

- Magnesium metal, Mg
- Zinc metal, Zn
- Iron(II) ions, Fe^{2+}
- Oxalate ions, $C_2O_4^{2-}$
- Thiosulphate ions, $S_2O_3^{2-}$
- Sulphite ions, SO_3^{2-}
- Nitrite ions, NO_2^-
- Iodide ions, I^-
- Chloride ions, Cl^-
- Tin(II) ion, Sn^{2+}
- Hydrogen peroxide, H_2O_2
- Hydrogen, H_2
- Carbon, C
- Amino group, $-NH_2$, e.t.c.

4.1.5Half equations of common Reducing Agents

$Mg(s) \rightarrow Mg^{2+}(aq) + 2e^-$
$Zn(s) \rightarrow Zn^{2+}(aq) + 2e^-$
$Fe^{2+}(aq) \rightarrow Fe^{3+}(aq) + e^-$
$C_2O_4^{2-}(aq) \rightarrow 2CO_2(g) + 2e^-$
$2S_2O_3^{2-}(aq) \rightarrow S_4O_6^{2-}(aq) + 2e^-$
$SO_3^{2-}(aq) + H_2O(l) \rightarrow SO_4^{2-}(aq) + 2H^+(aq) + 2e^-$
$NO_2^-(aq) + H_2O(l) \rightarrow NO_3^-(aq) + 2H^+(aq) + 2e^-$
$H_2O_2(aq) \rightarrow O_2(g) + 2H^+(aq) + 2e^-$
$2I^-(aq) \rightarrow I_2(aq) + 2e^-$
$Sn^{2+}(aq) \rightarrow Sn^{4+}(aq) + 2e^-$
$2C(s) + O_2^{2-}(aq) \rightarrow 2CO(g) + 2e^-$

4.2Major categories of Redox Reactions

4.2.1Redox reactions involving acidified potassium manganate(VII) solution

In acidic medium, the manganese is reduced from the oxidation state of +7 in the manganate(VII) ion, MnO_4^-which appears as a purple solution, to the oxidation state of +2 in form of manganese(II) ions, Mn^{2+}which are very faint pink in solution hence appear as a colourless solution.

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

The manganate(VII) ion, MnO_4^- is acidified using only sulphuric acid and not hydrochloric acid. This is because MnO_4^- is a very strong oxidizing agent and oxidizes the chloride ions, Cl^- in the hydrochloric acid solution to chlorine gas.The half equationsfor this redox reaction are shown below.

$$MnO_4^-(aq) + 8H^+(aq) + 5e^- \rightarrow Mn^{2+}(aq) + 4H_2O(l) \quad \text{.....(i)}$$
$$2Cl^-(aq) \rightarrow Cl_2(g) + 2e^- \quad \text{.....(ii)}$$

To come up with the overall redox equation, we aim at ensuring that the number of electrons on the left side is equal to the number of electrons on the right side. This is done by multiplying equation (i) by 2 and multiplying equation (ii) by 5 such that the number of electrons on the left and on the right is 10.
Equation (i) now becomes:

$$2MnO_4^-(aq) + 16H^+(aq) + 10e^- \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) \quad \text{.....(iii)}$$

Equation (ii) now becomes:

$$10Cl^-(aq) \rightarrow 5Cl_2(g) + 10e^- \quad \text{.....(iv)}$$

When the number of electrons are 10 on both the left and right, they counsel and the overall redox equation becomes:

$$2MnO_4^-(aq) + 16H^+(aq) + 10Cl^-(aq) \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) + 5Cl_2(g)$$

Nitric acid is also not used to acidify MnO_4^- because the nitric acid itself is an oxidizing agent which would compete with the MnO_4^-.

Note: Acidified potassium manganate(VII) is commonly used in volumetric analysis because of the following reasons:

1) It is the most powerful oxidising agent and is stable in water.

2) It is a self indicator (no indicator is required since at the end point, the purple solution turns faint pink).

Potassium manganate(VII)can be used in redox titrations in the oxidation of reducing agents such as:

a) Iron(II) salts

Half equations

$Fe^{2+}(aq) \rightarrow Fe^{3+}(aq) + e^-$ ……...……...(i)

$MnO_4^-(aq) + 8H^+(aq) + 5e^- \rightarrow Mn^{2+}(aq) + 4H_2O(l)$……(ii)

Multiplying equation (i) by 5, equation (i) becomes:

$5Fe^{2+}(aq) \rightarrow 5Fe^{3+}(aq) + 5e^-$...……..(iii)

Combining equation (ii) and (iii) gives us the overall redox equation below:

$MnO_4^-(aq) + 8H^+(aq) + 5Fe^{2+}(aq) \rightarrow Mn^{2+}(aq) + 4H_2O(l) + 5Fe^{3+}(aq)$

b) Oxalate ions

Half equations

$C_2O_4^{2-}(aq) \rightarrow 2CO_2(g) + 2e^-$ …….…….....(i)

$MnO_4^-(aq) + 8H^+(aq) + 5e^- \rightarrow Mn^{2+}(aq) + 4H_2O(l)$…….…….....(ii)

Multiplying equation (i) by 5, equation (i) becomes:

$5C_2O_4^{2-}(aq) \rightarrow 10CO_2(g) + 10e^-$…....…….…….....(iii)

Multiplying equation (ii) by 2, equation (ii) becomes:

$2MnO_4^-(aq) + 16H^+(aq) + 10e^- \rightarrow 2Mn^{2+}(aq) + 8H_2O(l)$…….....(iv)

Combining equation (iii) and (iv) gives us the overall redox equation below:

$5C_2O_4^{2-}(aq) + 2MnO_4^-(aq) + 16H^+(aq) \rightarrow 10CO_2(g) + 2Mn^{2+}(aq) + 8H_2O(l)$

c) Sulphite ions

Half equations

$SO_3^{2-}(aq) + H_2O(l) \rightarrow SO_4^{2-}(aq) + 2H^+(aq) + 2e^-$…….....(i)

$MnO_4^-(aq) + 8H^+(aq) + 5e^- \rightarrow Mn^{2+}(aq) + 4H_2O(l)$…….....(ii)

Multiplying equation (i) by 5, equation (i) becomes:

$5SO_3^{2-}(aq) + 5H_2O(l) \rightarrow 5SO_4^{2-}(aq) + 10H^+(aq) + 10e^-$…….....(iii)

Multiplying equation (ii) by 2, equation (ii) becomes:

$2MnO_4^-(aq) + 16H^+(aq) + 10e^- 2Mn^{2+}(aq) + 8H_2O(l)$…….....(iv)

Combining equation (iii) and (iv) gives us the overall redox equation below:

$5SO_3^{2-}(aq) + 2MnO_4^-(aq) + 6H^+(aq) \rightarrow 5SO_4^{2-}(aq) + 2Mn^{2+}(aq) + 3H_2O(l)$

d) Nitrite ions

Half equations

$NO_2^-(aq) + H_2O(l) \rightarrow NO_3^-(aq) + 2H^+(aq) + 2e^-$…….....(i)

$MnO_4^-(aq) + 8H^+(aq) + 5e^- \rightarrow Mn^{2+}(aq) + 4H_2O(l)$…….....(ii)

Multiplying equation (i) by 5, equation (i) becomes:

$5NO_2^-(aq) + 5H_2O(l) \rightarrow 5NO_3^-(aq) + 10H^+(aq) + 10e^-$…….....(iii)

Multiplying equation (ii) by 2, equation (ii) becomes:

$2MnO_4^-(aq) + 16H^+(aq) + 10e^- \rightarrow 2Mn^{2+}(aq) + 8H_2O(l)$…….....(iv)

Combining equation (iii) and (iv) gives us the overall redox equation below:

$5NO_2^-(aq) + 2MnO_4^-(aq) + 6H^+(aq) \rightarrow 5NO_3^-(aq) + 2Mn^{2+}(aq) + 3H_2O(l)$

e) Hydrogen peroxide

Half equations

$H_2O_2(aq) \rightarrow O_2(g) + 2H^+(aq) + 2e^-$.....(i)

$MnO_4^-(aq) + 8H^+(aq) + 5e^- \rightarrow Mn^{2+}(aq) + 4H_2O(l)$…….....(ii)

Multiplying equation (i) by 5, equation (i) becomes:

$5H_2O_2(aq) \rightarrow 5O_2(g) + 10H^+(aq) + 10e^-$.....(iii)

Multiplying equation (ii) by 2, equation (ii) becomes:

$2MnO_4^-(aq) + 16H^+(aq) + 10e^- \rightarrow 2Mn^{2+}(aq) + 8H_2O(l)$..…….....(iv)

Combining equation (iii) and (iv) gives us the overall redox equation below:

$5H_2O_2(aq) + 2MnO_4^-(aq) + 6H^+(aq) \rightarrow 5O_2(g) + 2Mn^{2+}(aq) + 8H_2O(l)$

Apart from acting as a reducing agent where it is oxidized to oxygen, hydrogen peroxide can also act as an oxidizing agent where it is reduced to water as shown in **section 4.1.3** above.

Volume strength of hydrogen peroxide

Hydrogen peroxide is obtainable from suppliers in form of an aqueous solution whose concentration is expressed as 6%, 12% or very commonly 30% hydrogen peroxide in which cases it is simply referred to as "20 volume", "40 volume" or "100 volume" hydrogen peroxide respectively. In other words, many times, the concentration of hydrogen peroxide is looked at in terms of how much volume (usually in cm^3) of oxygen gas is evolved by a fixed amount or mass of hydrogen peroxide but usually by $1cm^3$ of hydrogen peroxide solution at standard temperature and pressure (s.t.p). This unit of concentration of hydrogen peroxide is referred to as volume strength.

The volume strength of hydrogen peroxide is the volume of oxygen available from a unit volume of hydrogen peroxide solution measured at s.t.p.

A volume solution of hydrogen peroxide, therefore, yields one volume of oxygen for each volume of the hydrogen peroxide solution decomposed at standard temperature and prerssure (s.t.p.).

For example:

i) $1cm^3$ of "100 volume" hydrogen peroxide decomposes to yield $100cm^3$ of oxygen gas measured at s.t.p.

ii) $1cm^3$ of "30 volume" hydrogen peroxide decomposes to yield $30cm^3$ of oxygen gas measured at s.t.p.

iii) $1cm^3$ of "20 volume hydrogen peroxide decomposes to yield $20cm^3$ of oxygen gas measured at s.t.p.

Hydrogen peroxide decomposes according to the following equation:

$2H_2O_2(aq) \rightarrow O_2(g) + 2H_2O(l)$

Moles of hydrogen peroxide decomposing = 2

Mass of hydrogen peroxide decomposing = [(1x4) + (16x4)]g = **68g**

Moles of oxygen evolved at s.t.p = 1

Volume of oxygen evolved at s.t.p. = $22.4dm^3$ (i.e. $22400cm^3$)

It follows that:

i) 2moles of hydrogen peroxide produce 1 mole of oxygen ($22.4dm^3$ i.e. $22400cm^3$ of oxygen at s.t.p.). Thus 2 moles of hydrogen peroxide have a volume strength of $22.4dm^3$ ($22400cm^3$) at s.t.p.

ii) Also 68g of hydrogen peroxide produce 1 mole of oxygen ($22.4dm^3$ i.e. $22400cm^3$ of oxygen at s.t.p.). Thus a "22400 volume" solution of hydrogen peroxide contains 68g of hydrogen peroxide.

Note: Since we usually carry out experiments in the chemistry laboratory at **room temperature,** then in such situations, we carry out our calculations for determination of volume strength of hydrogen peroxide solutions basing on the fact that:***2 moles of hydrogen peroxide decompose to yield 24dm^3 (i.e. 24000cm^3) of oxygen gas at room temperature*** rather than 22.4 dm^3 ($22400cm^3$) at s.t.p.

4.2.2Redox reactions involving acidified potassium dichromate(VI) solution

In acidic medium, the chromium is reduced from the oxidation state of +6 in the dichromate(VI) ion, $Cr_2O_7^{2-}$which appears orange in solution, to the oxidation state of +3 in form of chromium(III) ions, Cr^{3+}which appear green in solution.

$Cr_2O_7^{2-}(aq) + 14H^+(aq) + 6e^- \rightarrow 2Cr^{3+}(aq) + 7H_2O(l)$

Potassium dichromate(VI), when pure can remain stable in aqueous state for a long period of time and can be used as a primary standard.

Potassium dichromate(VI) can be used in redox titrations in the oxidation of reducing agents such as:

a) Iron(II) salts

Half equations

$Fe^{2+}(aq) \rightarrow Fe^{3+}(aq) + e^-$ ……..……(i)

$Cr_2O_7^{2-}(aq) + 14H^+(aq) + 6e^- \rightarrow 2Cr^{3+}(aq) + 7H_2O(l)$.....……..(ii)

Multiplying equation (i) by 6, equation (i) becomes:

$6Fe^{2+}(aq) \rightarrow 6Fe^{3+}(aq) + 6e^-$…….....(iii)

Combining equation (ii) and (iii) gives us the **overall redox equation** below:

$Cr_2O_7^{2-}(aq) + 14H^+(aq) + 6Fe^{2+}(aq) \rightarrow 2Cr^{3+}(aq) + 7H_2O(l) + 6Fe^{3+}(aq)$

b) Sulphite ions

Half equations

$SO_3^{2-}(aq) + H_2O(l) \rightarrow SO_4^{2-}(aq) + 2H^+(aq) + 2e^-$…….....(i)

$Cr_2O_7^{2-}(aq) + 14H^+(aq) + 6e^- \rightarrow 2Cr^{3+}(aq) + 7H_2O(l)$…….....(ii)

Multiplying equation (i) by 3, equation (i) becomes:

$3SO_3^{2-}(aq) + 3H_2O(l) \rightarrow 3SO_4^{2-}(aq) + 6H^+(aq) + 6e^-$…….....(iii)

Combining equation (ii) and (iii) gives us the **overall redox equation** below:

$Cr_2O_7^{2-}(aq) + 8H^+(aq) + 3SO_3^{2-}(aq) \rightarrow 2Cr^{3+}(aq) + 4H_2O(l) + 3SO_4^{2-}(aq)$

c)Oxalate ions

Half equations

$C_2O_4^{2-}(aq) \rightarrow 2CO_2(g) + 2e^-$……...(i)

$Cr_2O_7^{2-}(aq) + 14H^+(aq) + 6e^- \rightarrow 2Cr^{3+}(aq) + 7H_2O(l)$ ……..….....(ii)

Multiplying equation (i) by 3, equation (i) becomes:

$3C_2O_4^{2-}(aq) \rightarrow 6CO_2(g) + 6e^-$…….....(iii)

Combining equation (ii) and (iii) gives us the **overall redox equation** below:

$Cr_2O_7^{2-}(aq) + 14H^+(aq) + 3C_2O_4^{2-}(aq) \rightarrow 2Cr^{3+}(aq) + 7H_2O(l) + 6CO_2(g)$

d)Iodide ions

Half equations

$2I^-(aq) \rightarrow I_2(aq) + 2e^-$……...(i)

$Cr_2O_7^{2-}(aq) + 14H^+(aq) + 6e^- \rightarrow 2Cr^{3+}(aq) + 7H_2O(l)$ …….....(ii)

Multiplying equation (i) by 3, equation (i) becomes:

$6I^-(aq) \rightarrow 3I_2(aq) + 6e^-$.....…...……..…….....(iii)

Combining equation (ii) and (iii) gives us the **overall redox equation** below:

$Cr_2O_7^{2-}(aq) + 14H^+(aq) + 6I^-(aq) \rightarrow 2Cr^{3+}(aq) + 7H_2O(l) + 3I_2(aq)$

4.3 Titrations involving acidified potassium manganate(VII)solution

4.3.1Worked out examples on titrations involving acidified potassium manganate(VII) solution

Worked out example 4.3.1.1

You are provided with the following:
FA1 which is potassium manganate(VII) solution.
FA2which was prepared by dissolving 6.8g of impure sodium sulphite in one litre of solution.
FA3 which is 2M sulphuric acid.
Solid B which are crystals of iron(II) sulphate, $FeSO_4.7H_2O$
You are required to determine the percentage purity of the sodium sulphite in FA2.

PROCEDURE A

Weigh accurately 3.8g of B into a clean beaker. Add 100$cm^3$of FA3 and stir well to dissolve. Transfer the resultant solution into a 250cm^3volumetric flask and make up to the mark with distilled water. Label the solution FA4.
Pipette 20 or 25cm^3 of FA4 into a clean conical flask and titrate with FA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below.

Mass of beaker + B……*45.0*……g
Mass of beaker……*41.2*……g
Mass of B……*3.8*……g
Capacity of pipette used……*25.0*……cm^3

Final burette reading (cm^3)	*17.30*	*17.10*	*23.00*
Initial burette reading (cm^3)	*0.00*	*0.00*	*6.00*
volume of FA1 (cm^3)	*17.30*	*17.10*	*17.00*

Values used to calculate average……*17.10, 17.00*……cm^3
Average volume of FA1 used……$\left(\frac{\mathbf{17.10 + 17.00}}{\mathbf{2}}\right)$ = ***17.05***……cm^3

Questions

a) Write the equation of reaction that occurs between Fe^{2+} and MnO_4^-.

$MnO_4^-(aq) + 8H^+(aq) + 5Fe^{2+}(aq) \rightarrow Mn^{2+}(aq) + 4H_2O(l) + 5Fe^{3+}(aq)$

b) Calculate the number of moles of Fe^{2+}in FA4 that reacted with MnO_4^- in FA1.
(Fe=56, S=32,O=16, H=1)

Molar mass of $FeSO_4.7H_2O$ = [(56x1) + (32x1) + (16x4) + (7x18)] = ***278****g*
278g of $FeSO_4.7H_2O$ contain 1 mole
3.8g of $FeSO_4.7H_2O$ contain $\left(\frac{1}{278} \text{ x } 3.8\right)$ *moles*

$= 0.0137$ moles

$250cm^3$ of FA4 contain 0.0137 moles of Fe^{2+}

$25cm^3$ of FA4 contain $\left(\frac{0.0137}{250} \times 25\right)$ moles of Fe^{2+}

$= 0.00137$ moles of Fe^{2+}

c) Determine the MnO_4^- in FA1 in $moldm^{-3}$

Moles of $MnO_4^- = \frac{1}{5} \times$ moles of Fe^{2+}

$= \left(\frac{1}{5} \times 0.00137\right)$

$= \mathbf{0.000273}$

$17.05cm^3$ of FA1 contain 0.000273 moles of MnO_4^-

$1000\ cm^3$ of FA1 contain $\left(\frac{0.000273}{17.05} \times 1000\right)$ moles of MnO_4^-

Concentration of $MnO_4^- = \mathbf{0.016\ moldm^{-3}}$

PROCEDURE B

Using a measuring cylinder, measure accurately $25cm^3$ of FA2 into a clean conical flask. Add $10cm^3$ of FA3 and titrate the mixture with FA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below.

Final burette reading (cm^3)	*25.30*	*25.10*	*29.10*
Initial burette reading (cm^3)	*0.00*	*0.00*	*4.00*
volume of FA1 (cm^3)	*25.30*	*25.10*	*25.10*

Values used to calculate average……*25.10, 25.10*……cm^3

Average volume of FA1 used …….. $\left(\frac{\mathbf{25.10 + 25.10}}{\mathbf{2}}\right) = \mathbf{25.10}$ ……cm^3

Questions

c) Calculate the moles of SO_3^{2-} in FA2 that reacted with MnO_4^- in FA1.

$1000cm^3$ of FA1 contain 0.016 moles of MnO_4^-

$25.10cm^3$ of FA1 contain $\left(\frac{0.016}{1000} \times 25.10\right)$ moles of MnO_4^-

$= 0.0004$ moles of MnO_4^-

$2MnO_4^-(aq) + 6H^+(aq) + 5SO_3^{2-}(aq) \rightarrow 2Mn^{2+}(aq) + 3H_2O(l) + 5SO_4^{2-}(aq)$

2moles of MnO_4^- react with 5moles of SO_3^{2-}

0.0004 moles of MnO_4^- react with $\left(\frac{5}{2} \times 0.0004\right)$ moles of SO_3^{2-}

$= \mathbf{0.001\ moles\ of\ SO_3^{2-}}$

d) Determine the:

i) concentration of SO_3^{2-} in FA2 in $moldm^{-3}$.

$25cm^3$ of FA2 contain 0.001 moles of SO_3^{2-}

$1000cm^3$ of FA2 contain $\left(\frac{0.001}{25} \times 1000\right)$ moles of SO_3^{2-}

$= \mathbf{0.04\ moldm^{-3}}$

ii) Percentage purity of the sodium sulphite sample used in the preparation of FA2.
(Na=23, S=32, O=16)

Molar mass of Na_2SO_3 = [(23x2)+(32x1)+(16x3)]g
=(46+32+48)g
=126g

1 mole of Na_2SO_3 weighs 126g
0.04 moles of Na_2SO_3 weigh (126 x 0.04)g
*=**5.04** gdm^{-3}*

Percentage purity of the sodium sulphite sample $= \left(\frac{5.04}{6.8} \times 100\right)\%$
= 74.12%

Worked out example 4.3.1.2

You are provided with the following:
FA1 which contains 1.35g of potassium manganate(VII) in 500cm^3 of solution.
FA2 which is hydrogen peroxide solution
FA3 which is 2.0 M sulphuric acid
You are required to determine the volume strength of the hydrogen peroxide solution provided.

Theory

Hydrogen peroxide undergoes oxidation as shown by the equation below.

$H_2O_2(aq) \rightarrow O_2(g) + 2H^+(aq) + 2e^-$

Procedure

Using a measuring cylinder, measure and transfer 25cm^3 of FA2 into a 250cm^3 volumetric flask. Add about 50cm^3 of distilled water and shake well to mix and then make up to the mark by adding more distilled water. Label the solution FA4.
Pipette 20 or 25cm^3 of FA4 into a clean conical flask. Add an equal volume of FA3 using a measuring cylinder and titrate with FA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below.

Capacity of pipette used……*25.0*……cm^3

Final burette reading (cm^3)	*29.40*	*39.60*	*29.40*
Initial burette reading (cm^3)	*0.00*	*10.00*	*0.00*
e of FA1 used(cm^3)	*29.40*	*29.60*	*29.40*

Values used to calculate average……*29.40, 29.40*……cm^3
Average volume of FA1 used……$\left(\frac{29.40 + 29.40}{2}\right)$= ***29.40***……$cm^3$

a) Write the equation of reaction between hydrogen peroxide and acidified manganate(VII) ions.

$2MnO_4^-(aq) + 6H^+(aq) + 5H_2O_2(aq) \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) + 5O_2(g)$

b) Determine the molar concentration of potassium manganate(VII) in FA1.

Molar mass of $KMnO_4$ = (39x1) + (55x1) + (16x4) = 158g
158g of $KMnO_4$ contain 1 mole.
1.35g of $KMnO_4$ contain $\left(\frac{1}{158} \times 1.35\right)$ *moles*
= 0.008544 moles
500cm^3 of FA1 contain 0.008544 moles of potassium manganate(VII)
1000cm^3 of FA1 contain $\left(\frac{0.008544}{500} \times 1000\right)$ *moles of potassium manganate(VII)*
= 0.017mol dm^{-3}

c) Calculate the:
i) number of moles of hydrogen peroxide in 250cm^3 of FA4.

1000cm^3 of FA1 contain 0.017moles of potassium manganate(VII)
29.40cm^3 of FA1 contain $\left(\frac{0.017}{1000} \times 29.40\right)$ *moles of potassium manganate(VII)*
= 0.0004998 moles of potassium manganate(VII)
Moles of hydrogen peroxide = $\frac{5}{2}$ *x* 0.0004998
= 0.00125 moles
25cm^3 of FA4 contain 0.00125 moles of hydrogen peroxide.
250cm^3 of *FA4 contain* $\left(\frac{0.0125}{25} \times 250\right)$ *moles of hydrogen peroxide*
= 0.0125moles of hydrogen peroxide

ii) molar concentration of hydrogen peroxide in the FA2 solution.
25cm^3 of FA2 contain 0.0125moles of hydrogen peroxide
1000cm^3 of FA2 contain $\left(\frac{0.0125}{25} \times 1000\right)$ *moles of hydrogen peroxide*
= 0.5mol dm^{-3}

iii) volume strength of hydrogen peroxide in the original FA2 solution.
(**NB:***Volume strength is the volume of oxygen gas liberated by 1cm^3 of hydrogen peroxide solution; 1 mole of a gas occupies 24dm^3 at room temperature).*

1000cm^3 of FA2 contain 0.5 moles of H_2O_2
1cm^{3}of FA2 contains $\left(\frac{0.5}{1000}\right)$ *moles of H_2O_2*
= 0.0005 moles of H_2O_2

$2H_2O_2(aq) \rightarrow 2H_2O(l) + O_2(g)$

2moles of hydrogen peroxide evolve 1 mole of oxygen gas
0.0005 moles of hydrogen peroxide evolve $\left(\frac{1}{2} \times 0.0005\right)$ *moles of oxygen gas*
=0.00025moles of oxygen gas
1 mole of oxygen gas occupies 24dm^3 at room temperature
0.00025moles of oxygen gas occupy (24x0.00025) dm^3 at room temperature
Volume strength of hydrogen peroxide in FA2 = 0.006 dm^3

Worked out example 4.3.1.3
You are provided with the following:
FA1 which is a solution that contains 4.66g of anhydrous sodium sulphite, Na_2SO_3 per ltre.
FA2 which is potassium manganate(VII) solution.
2.0M sulphuric acid solution.
Solid D which is impure ferrous oxalate, FeC_2O_4.
You are required to determine the:
(i) molar concentration of the potassium mangante(VII) in FA2.
(ii) percentage purity of the ferrousoxalate sample.

Theory
Acidified manganate(VII) ions react with sulphite ions, iron(II) ions and oxalate ions according to the following equations.

$2MnO_4^-(aq) + 6H^+(aq) + 5SO_3^{2-}(aq) \rightarrow 5SO_4^{2-}(aq) + 2Mn^{2+}(aq) + 3H_2O(l)$

$MnO_4^-(aq) + 8H^+(aq) + 5Fe^{2+}(aq) \rightarrow Mn^{2+}(aq) + 4H_2O(l) + 5Fe^{3+}(aq)$

$2MnO_4^-(aq) + 16H^+(aq) + 5C_2O_4^{2-}(aq) \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) + 10CO_2(g)$

Procedure I
Using a measuring cylinder, transfer 25cm^3 of FA1 into a conical flask. Add 10cm^3 of 2.0M sulphuric acid and titrate the solution with FA2 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in table I below.

Table I

Final burette reading (cm^3)	*18.60*	*20.50*	*20.50*
Initial burette reading (cm^3)	*0.00*	*2.00*	*2.00*
Volume of FA2 used(cm^3)	*18.60*	*18.50*	*18.50*

Titre values used to calculate verage ……*18.50,18.50*……cm^3

Average volume of FA2 used……$\left(\frac{18.50 + 18.50}{2}\right)$= ***18.50***……cm^3

Procedure II
Weigh accurately1.4g of D into a clean beaker. Add 100cm^3 of 2.0M sulphuric acid and stir well to dissolve. Transfer the resultant solution into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label the solution FA3.

Pipette 25cm^3 (or 20cm^3) of FA3 into a conical flask and heat the solution to 70^{0}C. Titrate the hot solution with FA2 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in table II below.

Mass of beaker + D……*39.6*……g
Mass of beaker……*38.2*……g
Mass of D……*1.4*……g
Capacity of pipette used……*25.0*……cm^3

Table II

Final burette reading (cm^3)	*22.90*	*27.10*	*22.90*
Initial burette reading (cm^3)	*0.00*	*4.00*	*0.00*
Volume of FA2 used(cm^3)	*22.90*	*23.10*	*22.90*

Titre values used to calculate average ……*22.90, 22.90*……cm^3

Average volume of FA2 used ……$\left(\frac{22.90 + 22.90}{2}\right)$***=22.90***……$cm^3$

(a) Calculate the:

(i) molarity of potassium manganate(VII) in FA2. (Na=39, S=32, O=16)

Molar mass of Na_2SO_3= [(23x2) + (32x1) + (16x3)] ***= 126g***

126g of Na_2SO_3 contain 1 mole

4.66g of Na_2SO_3 contains $\left(\frac{4.66}{126} \times 4.66\right)$ *moles*

$= 0.037M$

$1000cm^3$ of FA1 contain 0.037 moles of SO_3^{2-}

$25.0cm^3$ of FA1 contain $\left(\frac{0.037}{1000} \times 25.0\right)$ *moles of SO_3^{2-}*

$= 9.25x10^{-4}$ *moles of SO_3^{2-}*

Moles of MnO_4^- $= \left(\frac{2}{5}\right)$ *x moles of SO_3^{2-}*

$= (\frac{2}{5} \times 9.25x10^{-4}) = 3.7x10^{-4}$ *moles*

$18.50cm^3$ of FA2 contain $3.7x10^{-4}$ moles of MnO_4^-

$1000cm^3$ of FA2 contain $\left(\frac{3.7x10-4}{18.50} \times 1000\right)$ *moles of MnO_4^-*

= 0.02M

(ii) moles of manganate(VII) ions that reacted with $25cm^3$ (or $20cm^3$) of FA3.

$1000cm^3$ of FA2 contain 0.02 moles of MnO_4^-

$22.90cm^3$ of FA2 contain $\left(\frac{0.02}{1000} \times 22.90\right)$ *moles of MnO_4^-*

$= 4.58x10^{-4}$ *moles of MnO_4^-*

(iii) moles of iron(II) ions in $25cm^3$ (or $20cm^3$) of FA3.

Combining the two equations of reaction between MnO_4^- and both Fe^{2+} and $C_2O_4^{2-}$, we have:

$3MnO_4^-(aq)+24H^+(aq) +5Fe^{2+}(aq) +5C_2O_4^{2-}(aq) \rightarrow 3Mn^{2+}(aq) +12H_2O(l) +5Fe^{3+}(aq)+10CO_2(g)$

Moles of $Fe^{2+} = \frac{5}{3}$ *x moles of* MnO_4^-

$= \left(\frac{5}{3} \text{ x } 4.58x10-4\right) =$ ***7.63x10⁻⁴ molesof*** Fe^{2+}

(b) Determine the:
(i) mass of ferrous oxalate, FeC_2O_4 in $250cm^3$ of FA3. (Fe = 56, C=12, O=16)
25cm³of FA3 contain 7.63x10⁻⁴moles of FeC_2O_4
250cm³of FA3 contain $\left(\frac{7.63x10-4}{25} \text{ x } 250\right)$ *moles of* FeC_2O_4
$= 7.63x10^{-3}$ *moles of* FeC_2O_4

Molar mass of FeC_2O_4 *= (56x1)+(12x2)+(16x4) =* ***144g***
1mole of FeC_2O_4 *weighs 144g*
7.63x10⁻³moles of FeC_2O_4 *weighs (144 x 7.63x10⁻³)*
= 1.099g

(ii) percentage purity of the ferrous oxalate sample.
$Percentage\ purity = \left(\frac{Mass\ of\ the\ ferrous\ oxalate}{Total\ mass\ of\ impure\ sample} \text{ x } 100\right)\ \%$
$= \left(\frac{1.099}{1.4} \text{ x } 100\right)\ \%$ ***= 78.5%***

Worked out example 4.3.1.4

You are provided with the following:
HA1 which contains 2.8g of sodium hydroxide per litre.
HA2 which is 0.02Mpotassium manganate(VII).
HA4 is 1.5M sulphuric acid.
Solid J which is an acidic compound of the formula $H_a(C_2O_4)_b.cH_2O$.
You are required to determine the value of a, b and c in $H_a(C_2O_4)_b.cH_2O$.

Theory

Solid E dissolves readily in water. In aqueous state, the acid salt ionizes according to the equation below.

$$H_a(C_2O_4)_b.cH_2O(aq) \rightarrow aH^+(aq) + bC_2O_4^{2-}(aq) + cH_2O(l)$$

The Hydrogen ions from the acid salt react with hydroxyl ions from potassium hydroxide according to the equation below.

$$H^+(aq) + OH^-(aq) \rightarrow H_2O(l)$$

Acidified manganate(VII) ions from potassium manganate(VII) react with ethanedioate ions according to the equation below:

$$2MnO_4^-(aq) + 16H^+(aq) + 5C_2O_4^{2-}(aq) \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) + 10CO_2(g)$$

Procedure I

Weigh accurately, 1.0g of solid J into a clean beaker. Add $100cm^3$ of distilled water using a measuring cylinder. Transfer the solution into a $250cm^3$ volumetric flask and make up to the mark with distilled water. Label the solution HA3.

Pipette 20 or 25cm^3 of HA3 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with HA1 from the burette until the end point is reached.
Repeat the titration until you obtain consistent results. Record your results in table I below.

Results

Mass of beaker + J……*41.1*……..…..g
Mass of beaker alone……*40.1*……...g
Mass of J alone……*1.0*……...g
Capacity of pipette used……*25.0*……cm^3

Table I

Final burette reading (cm^3)	22.90	24.70	22.60
Initial burette reading (cm^3)	*0.00*	*2.00*	*0.00*
Volume of HA1 used(cm^3)	22.90	22.70	22.60

Values used to calculate average……*22.70, 22.60*…….....cm^3

Average volume of HA1 used……$\left(\frac{22.70 + 22.60}{2}\right)$ …=…***22.65***……cm^3

Procedure II

Pipette 20 or 25cm^3 of HA3 into a conical flask. Add an equal volume of HA4 and heat the mixture to 70^{0}C. Titrate the hot solution immediately with HA2 from the burette. Repeat the titration until you obtain consistent results. Record your results in table II
below.

Table II

Final burette reading (cm^3)	*16.20*	*15.90*	*21.90*
Initial burette reading (cm^3)	*0.00*	*0.00*	*6.00*
Volume of HA2 used(cm^3)	*16.20*	*15.90*	*15.90*

Values used to calculateaverage……*15.90, 15.90*……cm^3

Average volume of HA2 used……$\left(\frac{15.90 + 15.90}{2}\right)$…=…***15.90***……cm^3

Questions

a) calculate the concentration of:
i) H^+ in HA3 in moles per litre.
Molar mass of NaOH = (23x1)+(16x1)+(1x1) = 40g
40g of sodium hydroxide contain 1 mole.
2.8g of sodium hydroxide contain $\left(\frac{1}{40} \text{ x } 2.8\right)$ *moles*
= 0.07M

1000cm^3 of HA1 contain 0.07 moles of sodium hydroxide.

22.65cm^3 of HA1 contain $\left(\frac{0.07}{1000} \times 22.65\right)$ *moles of sodium hydroxide.*

= 0.00159 moles of sodium hydroxide.

Moles of H^+ = (1xmoles of OH^-)

=(1x0.00159)

=0.00159

25.0cm^3 of HA3 contain 0.00159moles of H^+.

1000cm^3 of HA3 contain $\left(\frac{0.00159}{25.0} \times 1000\right)$ *moles of H^+.*

= 0.0636 moles per litre

ii) $C_2O_4^{2-}$ in HA3 in moldm^{-3}.

1000cm^3 of HA2 contain 0.02 moles of MnO_4^-.

15.90cm^3 of HA1 contain $\left(\frac{0.02}{1000} \times 15.90\right)$ *moles of MnO_4^-.*

= 0.00318 moles of MnO_4^-.

$$2MnO_4^-(aq) + 16H^+(aq) + 5C_2O_4^{2-}(aq) \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) + 10CO_2(g)$$

Moles of $C_2O_4^{2-}$ = ($\frac{5}{2}$ x moles of MnO_4^-)

$= \frac{5}{2} x\ 0.00318$

= 0.000795

25.0cm^3 of HA3 contain 0.000795 moles of $C_2O_4^{2-}$.

1000cm^3 of HA3 contain $\left(\frac{0.00795}{25.0} \times 1000\right)$ *moles of $C_2O_4^{2-}$.*

= 0.0318 moles per litre

b) Determine the:

i) ratio of *a* to *b*

Reactants:	H^+	$C_2O_4^{2-}$
Molar concentration:	*0.0636*	*0.0318*
Ratio:	$\frac{0.0636}{0.0318}$	$\frac{0.0318}{0.0318}$
	2	***1***

∴Ratio of a to b is ***2:1***

ii) value of *c* in $H_a(C_2O_4)_b.\ cH_2O$.

250cm^3 of HA3 contain 1 g of $H_2C_2O_4.\ cH_2O$.

1000cm^3 of HA3 contain $\left(\frac{1}{250} \times 1000\right)$ *g of $H_2C_2O_4.\ cH_2O$.*

= 4g of $H_2C_2O_4.\ cH_2O$ per litre.

$$H_2C_2O_4.cH_2O(aq) \rightarrow 2H^+(aq) + C_2O_4^{2-}(aq) + cH_2O(l)$$

∴Molar concentration of the acidic compound, $H_2C_2O_4.cH_2O$ = (1xmolar concentration of $C_2O_4^{2-}$)

= (1 x 0.0318)

= 0.0318

0.0318 moles of $H_2C_2O_4.cH_2O$ weigh 4g
1 mole of $H_2C_2O_4.cH_2O$ weigh $\left(\frac{4}{0.0318}\right)$ *g*
$= 125.8 \simeq 126g$
$H_2C_2O_4.cH_2O = 126$
$(1x2)+(12x2)+(16x4)+c[(1x2)+(16x1)] = 126$
$90 + 18c = 126$
$18c = 126-90$
$c = \frac{36}{18}$; $\boldsymbol{c = 2}$

4.3.2Practical exercises involving acidified potassium manganate(VII)

Experiment 4.3.2.1

You are provided with the following:
FA1 which is asolution of potassium manganate(VII), $KMnO_4$
FA2 which is 2.0M sulphuric acid
Solid H which are crystals of sodium oxalate, $Na_2C_2O_4$
You are required to determine theconcentration of:
i) sodium oxalate in FA3 in mol l^{-1}.
ii) potassium manganate(VII) in FA1 in mol l^{-1}.
(Na=23, C=12, O=16)

Theory

Manganate(VII) ions are reduced in acididic medium according to the equation below:

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

Oxalate ions, $C_2O_4^{2-}$undergo oxidationaccording to the equation below:

$$C_2O_4^{2-}(aq) \rightarrow 2CO_2(g) + 2e^-$$

Procedure

Weigh accurately 1.6g of H into a clean beaker. Add about $100cm^3$ of distilled water and stir well to dissolve. Transfer the solution into a $250cm^3$ volumetric flask and make up to the mark withdistilled water. Label this solution FA3.

Pipette 20 or $25cm^3$ of FA3 into a clean conical flask. Using a measuring cylinder, measure and add an equal volume of FA2 to the solution in the conical flask. Heat the mixture to about 60^0C and immediately titrate the hot solution with FA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below.

Mass of beaker + H…….……...g

Mass of beaker……..……..g

Mass of H……..…….g

Capacity of pipette used……cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA1 used(cm^3)			

Values used to calculate average volume of FA1 ……..cm^3

Average volume of FA1 used……cm^3

Questions

a) Write the overall redox equation between acidified manganate(VII) ions and oxalate ions.

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b) Calculate theconcentration of:

i) sodium oxalate in FA3 in moles per litre.

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ii) potassium manganate(VII) in FA1 in moles per litre.

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Experiment 4.3.2.2

You are provided with the following:
HA1 which is potassium manganate(VII) solution
HA2 which contains 11.5g of $FeSO_4.xH_2O$ in $500cm^3$ of solution.
HA3 which is 2.0M sulphuric acid
Solid W which are crystals of sodium oxalate, $Na_2C_2O_4$
You are required to determine the:
i) concentration of potassium manganate(VII) in HA1 in mol l^{-1}.
ii) value of x in $FeSO_4.xH_2O$.
(Na=23, C=12, H=1, Fe=56, S=32)

Theory

Manganate(VII) ions react with $Fe^{2+}$and $C_2O_4^{2-}$ according to the equations below:

$$MnO_4^-(aq) + 8H^+(aq) + 5Fe^{2+}(aq) \rightarrow Mn^{2+}(aq) + 4H_2O(l) + 5Fe^{3+}(aq)$$

$$2MnO_4^-(aq) + 16H^+(aq) + 5C_2O_4^{2-}(aq) \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) + 10CO_2(g)$$

Procedure I

Weigh accurately 1.4g of W into a clean beaker. Add about $100cm^3$ of distilled water and stir well to dissolve. Transfer the solution into a $250cm^3$ volumetric flask and make up to the mark withdistilled water. Label this solution HA4.
Pipette 20 or $25cm^3$ of HA4 into a clean conical flask. Using a measuring cylinder, measure and add $25cm^3$ of HA3 to the solution in the conical flask. Heat the mixture to about 60^0C and immediately titrate the hot solution with HA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below.

Mass of beaker + W……..……...g

Mass of beaker……..……..g

Mass of W……..…….g

Capacity of pipette used…….……cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of HA1 used(cm^3)			

Values used to calculate average……cm^3

Average volume of HA1 used……cm^3

Procedure II

Pipette 20 or 25cm^3 of HA2 into a clean conical flask. Using a measuring cylinder, measure and add 25cm^3 of HA3 to the solution in the conical flask and titrate with HA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below.

Capacity of pipette used……cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of HA1 used(cm^3)			

Values used to calculate average……cm^3

Average volume of HA1 used……cm^3

Questions

c) Calculate the concentration of:

i) sodium oxalate in HA4 in moles per litre.

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ii) potassium manganate(VII) in HA1 in moles per litre.

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iii) $FeSO_4.xH_2O$ in HA2 in moles per litre.

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b) Determine the value of x in $FeSO_4.xH_2O$.

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Experiment 4.3.2.3

You are provided with the following:
DA1 which contains 0.675g of potassium manganate(VII) in 250cm^3 of solution.
DA2 which is a solution containing a mixture of iron(II) sulphate heptahydrate, $FeSO_4.7H_2O$ and ammoniumiron(III) sulphate-12-water, $(NH_4)_2SO_4Fe(SO_4)_3.12H_2O$.
DA3 which is 1.5M sulphuric acid
Solid Q which is magnesium metal powder.
You are required to determine the percentage of the iron(II) salt in the DA2 mixture.
(K=39, Mn=55, O=16, N=14, S=32, H=1)

Theory

The MnO_4^- ions from potassium manganate(VII) react with only Fe^{2+} in the DA2 mixture according to the equation below:

$$MnO_4^-(aq) + 8H^+(aq) + 5Fe^{2+}(aq) \rightarrow Mn^{2+}(aq) + 4H_2O(l) + 5Fe^{3+}(aq)$$

On the other hand, themagnesium powder, when added to the DA2 mixture, in the presence of an acid and the mixture warmed gently, the magnesium reduces Fe^{3+} to Fe^{2+} while the magnesium is itself oxidized to Mg^{2+} according to the equation below:

$$2Fe^{3+}(aq) + Mg(s) \rightarrow 2Fe^{2+}(aq) + Mg^{2+}(aq)$$

Procedure A

Pipette 20 or 25cm^3 of DA2 into a clean conical flask. Add 25cm^3 of DA3 and titrate with DA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below.

Capacity of pipette used……cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of DA1 used(cm^3)			

Values used to calculate average……cm^3

Average volume of DA1 used…….....cm^3

a) Determine the:

i) concentration of potassium manganate(VII) in DA1 $moldm^{-3}$.

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ii) concentration of Fe^{2+} in DA2 in $moldm^{-3}$.

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b) Calculatethe mass of iron(II) sulphate heptahydrate, $FeSO_4.7H_2O$ in $1dm^3$ of the DA2 mixture.

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Procedure B

By use of a measuring cylinder, transfer $120cm^3$ of DA2 into a clean conical flask and to the solution in the conical flask, add 2g of Magnesium powder followed by $40cm^3$ of DA3. Warm the mixture until the solution obtained is almost colourless. Allow the solution to stand and to cool. Label this solution DA4. Pipette 20 or $25cm^3$ of the clear part of DA4 into a clean conical flask. Add $25cm^3$ of DA3 and titrate with DA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below.

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of DA1 used(cm^3)			

Values used to calculate average……cm^3

Average volume of DA1 used……cm^3

c) Calculate the:

i) total number of moles of Fe^{2+} in the $160cm^3$ of DA4.

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ii) total concentration of Fe^{2+} in the DA2 solution used in ***procedure B***in mol dm^{-3}.

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ii) concentration of Fe^{3+} in the DA2 solutionin mol dm^{-3}.

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d) Determine the:

i) total mass of the iron(II) and iron(III) salt in 1 dm^3 of DA2.

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ii) percentage by mass of the iron(II) salt in the DA2 mixture.

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Experiment 4.3.2.4

You are provided with the following:
FA1 which contains 3.2g of potassium manganate(VII) per litre.
FA2 which contains 4.48g of potassium hydroxide per litre.
FA4 is 2M sulphuric acid
Solid E which is an acid salt of the formula $H_w(C_2O_4)_x.yH_2O$.
You are required to determine the value of w, x and y in $H_w(C_2O_4)_x.yH_2O$.

Theory

Solid E dissolves readily in water. In aqueous state, the acid salt ionizes according to the equation below.

$H_w(C_2O_4)_x.yH_2O(aq) \rightarrow wH^+(aq) + xC_2O_4^{2-}(aq) + yH_2O(l)$

The Hydrogen ions from the acid salt react with hydroxyl ions from potassium hydroxide according to the equation below.

$H^+(aq) + OH^-(aq) \rightarrow H_2O(l)$

Acidified manganate(VII) ions from potassium manganate(VII) react with oxalate ions/ethanedioate ions according to the equation below:

$2MnO_4^-(aq) + 16H^+(aq) + 5C_2O_4^{2-}(aq) \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) + 10CO_2(g)$

Procedure I

Weigh accurately, 1.2g of solid E into a clean beaker. Add 100cm^3 of distilled water using a measuring cylinder. Transfer the solution into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label the solution FA3.
Pipette 20 or 25cm^3 of FA3 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with FA2 from the burette until the end point is reached.
Repeat the titration until you obtain consistent results. Record your results in table I below.

Results

Mass of beaker + E……..g

Mass of beaker alone……...g

Mass of E alone……...g

Capacity of pipette used……..cm^3

Table I

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA2 used(cm^3)			

Values used to calculate average…….....cm^3

Average volume of FA2 used…….cm^3

Procedure II

Pipette 20 or 25cm^3 of FA3 into a conical flask. Add an equal volume of FA4 and heat the mixture to 70^0C. Titrate the hot solution immediately with FA1 from the burette. Repeat the titration until you obtain consistent results. Record your results in table II below.

Table II

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA1 used(cm^3)			

Values used to calculateaverage……..…cm^3

Average volume of FA2 used……..cm^3

Questions

b) calculate the concentration of:

i) H^+ in FA3 in moles per litre.

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ii) $C_2O_4^{2-}$ in FA3 in $moldm^{-3}$.

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b) Determine the:

i) ratio of *w* to *x*

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ii) value of *y* in $H_w(C_2O_4)_x \cdot yH_2O$.

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Experiment 4.3.2.5

You are provided with the following:
FA1 which contains 2.38g of manganate(VII) ions, MnO_4^- in one litre of solution.
FA2which is 1.5M sulphuric acid
Solid **J**are4tablets of ferrous sulphate [iron(II) sulphate].
You are required to determine the percentage of the active constituent, that is, iron in a single tablet of iron(II) sulphate (Ferrous sulphate).
(Mn=55, O=16, Fe=56, S=32)

Procedure

i)Using a measuring cylinder, measure and transfer 190cm^3 of FA1 into a 250cm^3 of volumetric flask and make up to the mark with distilled water. Label the solution FA3.
ii)Weigh accurately, substance J (the 4 tablets of Ferrous sulphate provided) and record your findings in the spaces below.

Mass of beaker + J……...g

Mass of beaker alone……...g

Mass of J alone……....g

iii)Crush the entire substance J you have weighed using a mortar and pestle and transfer the crushed substance into a clean conical flask. Add about 60cm^3 of FA2and warm the mixture to about 70^{0}C as you gently stir the content of the conical flask to ensure that all the powder dissolves.

iv)Carefully filter the mixture into a clean beaker. Pour the solution in the beaker into a 100cm^3 measuring cylinder and make up to the mark with distilled water. Transfer the solution into a clean beaker and label this solution FA4.

v) Pipette 20 or 25cm^3 of FA4 into a clean conical flaskand titrate with FA3from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below.

Capacity of pipette used……..……...cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA3 used(cm^3)			

Values used to calculate average……cm^3

Average volume of FA3 used……cm^3

a) Determine the concentration of MnO_4^- in FA3 in $moldm^{-3}$

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b) Calculate the number of moles iron(II) sulphate present in the entire mass of J measured.

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c)Calculate the

i)mass of iron(II) sulphate, $FeSO_4$in the entire substance J measured.

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ii) mass of iron(II) sulphate, $FeSO_4$ in a single tablet.

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d)Determine the:

i) mass of one tablet.

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ii) percentage of iron(II) sulphate in one tablet.

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iii) percentage of the active ingredient, iron in one tablet of ferrous sulphate.

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Experiment 4.3.2.6

You are provided with the following:
FA1 which contains 1.35g of potassium manganate(VII) in 500cm^3 of solution.
FA2 which ishydrogen peroxide solution
FA3 which is 1.5M sulphuric acid
You are required to determine the volume strength of the hydrogen peroxidesolution provided.

Theory

Hydrogen peroxide undergoes oxidation as shown by the equation below.
$H_2O_2(aq) \rightarrow O_2(g) + 2H^+(aq) + 2e^-$

Procedure

Using a measuring cylinder, measure and transfer 25cm^3 of FA2 into a 250cm^3 volumetric flask. Add about 50cm^3 of distilled water and shake well to mix and then make up to the mark by adding more distilled water. Label the solution FA4.
Pipette 20 or 25cm^3 of FA4into a clean conical flask. Add an equal volume of FA3 using a measuring cylinder and titrate with FA1 from the buretteuntil the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below.

Capacity of pipette used……..cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA1 used(cm^3)			

Values used to calculate average……..cm^3

Average volume of FA1 used

……...cm^3

a) Write the equation of reaction between hydrogen peroxide and acidified manganate(VII) ions.

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b)Determine the molar concentration of potassium manganate(VII) in FA1.

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c)Calculate the:

i)number of moles of hydrogen peroxide in 250cm^3 of FA4.

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ii)molar concentration of hydrogen peroxide in the FA2 solution.

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iii)volume strength of hydrogen peroxide in the original FA2 solution.

(**NB:** *Volume strength is the volume of oxygen gas liberated by 1cm^3 of hydrogen peroxide solution; 1 mole of a gas occupies 24dm^3 at room temperature).*

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Experiment 4.3.2.7

You are provided with the following:

FA1 which is a solution that contains 1.26g of anhydrous sodium sulphite, Na_2SO_3in 200cm^3 of solution.

FA2 which is potassium manganate(VII) solution.

2.0 M sulphuric acid solution.

Solid F which is impure ferrous ethanedioate, FeC_2O_4.

You are required to determine the:

(i) molar concentration of the potassium mangante(VII) in FA2.

(ii) percentage impurity in the ferrous ethanedioate sample.

Theory

Acidified manganate(VII) ions react with sulphite ions, iron(II) ions and ethanedioateions according to the following equations.

$2MnO_4^-(aq) + 6H^+(aq) + 5SO_3^{2-}(aq) \rightarrow 5SO_4^{2-}(aq) + 2Mn^{2+}(aq) + 3H_2O(l)$

$MnO_4^-(aq) + 8H^+(aq) + 5Fe^{2+}(aq) \rightarrow Mn^{2+}(aq) + 4H_2O(l) + 5Fe^{3+}(aq)$

$2MnO_4^-(aq) + 16H^+(aq) + 5C_2O_4^{2-}(aq) \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) + 10CO_2(g)$

Procedure I

Using a measuring cylinder, transfer 20cm^3 of FA1 into a conical flask. Add 10cm^3 of 2.0M sulphuric acid and titrate the solution with FA2 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in table I below.

Table I

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA2 used(cm^3)			

Titre values used to calculateaverage ……..……..cm^3

Average volume of FA2 used…….…...…….……cm^3

Procedure II

Weigh accurately1.5g of F into a clean beaker. Add 100cm^3 of 2.0M sulphuric acid and stir well to dissolve. Transfer the resultant solution into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label the solution FA3.

Pipette 25cm^3 (or 20cm^3) of FA3 into a conical flask and heat the solution to 70^{0}C. Titrate the hot solution with FA2 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in table II below.

Mass of beaker + F…….……..……..g

Mass of beaker…….……..……g

Mass of F……..……..g

Capacity of pipette used…….....……..cm^3

Table II

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA2 used(cm^3)			

Titre values used to calculate verage ……..……..cm^3

Average volume of FA2 used ……..…….cm^3

(a) Calculate the:

(i) molarity of potassium manganate(VII) in FA2. (Na=39, S=32, O=16)

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(ii) moles of manganate(VII) ions that reacted with $25cm^3$ (or $20cm^3$) of FA3.

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(iii) moles of iron(II) ions in $25cm^3$ (or $20cm^3$) of FA3.

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(b) Determine the:

(i) mass of ferrous ethanedioate, FeC_2O_4 in $250cm^3$ of FA3. (Fe = 56, C=12, O=16)

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(ii) percentage impurityin the ferrous oxalate sample.

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4.4 Titrations involving iodine solution and sodium thiosulphate solution(Iodimetry and Iodometry Titrations)

a) Iodimetry titrations

These are titrations in which a standard solution of iodine is used directly in volumetric analysis. The standard iodine solution is used in oxidizing chemical species such as:

i)Thiosulphate ions ($S_2O_3^{2-}$) to Tetrathionate ions ($S_4O_6^{2-}$)

$$I_2(aq) + 2S_2O_3^{2-}(aq)2I^-(aq) + S_4O_6^{2-}(aq)$$

ii)Sulphite ions (SO_3^{2-}) to Sulphate ions(SO_4^{2-})

Sulphite ions are oxidized by iodineto sulphate ions according to the equation below:

$$SO_3^{2-}(aq) + H_2O(l) + I_2(aq) \rightarrow SO_4^{2-}(aq) + 2H^+(aq) + 2I^-(aq) \text{ .....(i)}$$

Note: This reaction is used in iodimetry particularly when the iodine is present is in excess and some remains unreacted. The unreacted iodine is titrated against standard sodium thiosulphate solution. Since the hydrogen ions produced by the reaction above would react with thiosulphate ions resulting in precipitation of sulphur, before titration with standard sodium thiosulphate, about 2g of sodium hydrogencarbonate are added to the solution in the conical flask so as to react with all the hydrogen ions (to remove **all** the hydrogen ions from the solution) as shown below.

$2HCO_3^-(aq) + 2H^+(aq) \rightarrow 2CO_2(aq) + 2H_2O(l)$ .....(ii)

When equation (i) and (ii) are combined, then we can agree that sulphite ions are oxidised by iodine in the presence of hydrogencarbonate ions to sulphate ions according to the equation below:

$SO_3^{2-}(aq) + I_2(aq) + 2HCO_3^-(aq) \rightarrow SO_4^{2-}(aq) + 2I^-(aq) + 2CO_2(aq) + H_2O(l)$

iii)Tin(II) ions (Sn^{2+}) to Tin(IV) ions(Sn^{4+}), e.t.c.

$Sn^{2+}(aq) + I_2(aq) Sn^{4+}(aq) + 2I^-(aq)$

Note: In these titrations, starch indicator is used.Standard iodine solution is titrated against a suitable solution e.g. sodium thiosulphate solution until the iodine solution turns from intense brown to pale yellow. At this point, starch indicator is added whereby a blue-black/dark blue solution of the starch-iodine complex is formed. Titration is continued and the end point is indicated by the sudden change from a blue-black/dark blue solution to a colourless solution.

b) Iodometry titrations

These are titrations in which involve the titration of iodine liberated by a given chemical reaction. In order to liberate the iodine, a known amount of a suitable inorganic oxidizing agent in acidic medium, is reacted witha solution containing excess iodide ions (e.g a solution of excess potassium iodide or excess sodium iodide). The excess iodide ions are oxidized to form a reasonable amount of aqueous iodine which can then be titrated withstandard sodium thiosulphate.

Reactions that can liberate aqueous iodine include some of the following:

$ClO_3^-(aq) + 6I^-(aq) + 6H^+(aq) \rightarrow Cl^-(aq) + H_2O(l) + 3I_2(aq)$

$IO_3^-(aq) + 5I^-(aq) + 6H^+(aq) \rightarrow H_2O(l) + 3I_2(aq)$

$OCl^-(aq) + 2I^-(aq) + 2H^+(aq) \rightarrow Cl^-(aq) + H_2O(l) + 3I_2(aq)$

$Cr_2O_7^{2-}(aq) + 14H^+(aq) + 6I^-(aq) \rightarrow 2Cr^{3+}(aq) + 7H_2O(l) + 3I_2(aq)$

$2MnO_4^-(aq) + 16H^+(aq) + 6I^-(aq) \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) + 5I_2(aq)$

$H_2O_2(aq) + 2H^+(aq) + 2I^-(aq) \rightarrow 2H_2O(l) + I_2(aq)$

Some reactions may not require an acidic medium e.g:

$2Cu^{2+}(aq) + 4I^-(aq) \rightarrow Cu_2I_2(s) + I_2(aq)$

$Cl_2(aq) + 2I^-(aq) \rightarrow 2Cl^-(aq) + I_2(aq)$

Note:

1)The aqueous iodine liberated in all the situations above is titrated against standard sodium thiosulphate solution and the reaction that occurs is shown by the following equation.

$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$

2)Some times, to simplify the application of the mole ratio, the equation of the reaction leading to the liberation of iodine may be combined with the one for the reaction of the liberated iodine with thiosulphate ions so that the iodine is eradicated from the combined overall equation.

For instance, combining the following equations:

$Cr_2O_7^{2-}(aq) + 14H^+(aq) + 6I^-(aq) \rightarrow 2Cr^{3+}(aq) + 7H_2O(l) + 3I_2(aq)$….....(i)

$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$ ……..….....(ii)

To eradicate the iodine from both equations, equation (ii) is multiplied by 3 to give us the following combined overall equation:

$3I_2(aq) + 6S_2O_3^{2-}(aq) \rightarrow 6I^-(aq) + 3S_4O_6^{2-}(aq)$ ……..…….....(iii)

Combining equation (i) and (iii) gives us the following equation:

$Cr_2O_7^{2-}(aq) + 14H^+(aq) + 6S_2O_3^{2-}(aq) \rightarrow 2Cr^{3+}(aq) + 7H_2O(l) + 3S_4O_6^{2-}(aq)$

3) Acidifiedpotassium dichromate(VI) solution is commonly associated with iodometry titrations.

4.4.1Worked out examples on Iodimetry and Iodometry Titrations

Worked out example4.4.1.1

You are provided with the following:
FA1 which is iodine solution
Substance L which is sodium thiosulphate pentahydrate, $Na_2S_2O_3.5H_2O$**.**
You are required to determine the molar concentration of iodine solution in FA1.

Theory

Aqueousiodine reacts with thiosulphate ions according to the following equation.

$$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$$

Procedure

i) Using a measuring cylinder, measure and transfer 150cm^3 of FA1 into a 250cm^3 volumetric flask and make up to the mark with distilled water. Transfer the solution into a clean beaker and label it FA2. Rinse the volumetric flask properly for use in procedure (ii) below.

ii)Weigh accurately, 4.6g of substance Linto a clean beaker. Add about 100cm^3 of water and stir well to dissolve. Transfer the solution into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label the solution FA3.

iii) Pipette 20 or 25cm^3 of FA2 into a clean conical flask and titrate with FA3 from the burette until the solution turns pale yellow. Add 1cm^3 of starch solutionand continue the titration until the solution just turns colourless. Repeat the titration until you obtain consistent results. Record your results in the table below.

Results

Mass of beaker + L……*44.80*……g
Mass of beaker alone……*40.20*……g
Mass of L alone……*4.60*……g
Capacity of pipette used……*25.0*……cm^3

Final burette reading (cm^3)	*18.00*	*17.80*	*23.70*
Initial burette reading (cm^3)	*0.00*	*0.00*	*6.00*
Volume of FA3 used(cm^3)	*18.00*	*17.80*	*17.70*

Values used to calculate average……*17.80, 17.70*……*cm*^{3}

Average volume of FA3 used……$\left(\frac{\mathbf{17.80 + 17.70}}{\mathbf{2}}\right)$***= 17.75****……cm*^{3}

……

a) Determine the molar concentration sodium thiosulphate in FA3.
(Na=23, S=32, O=16, H=1)

*Molar mass of $Na_2S_2O_3.5H_2O$ = [(23x2)+(32x2)+(16x3)+(5x18)]****=248g***

248g of sodium thiosulphate contain 1 mole
4.6 moles of sodium thiosulphate contain: $\left(\frac{1}{248} \text{ x } 4.6\right)$ *moles*
= 0.0185 moles

250cm³ of FA3 contain0.0185 moles of sodium thiosulphate

1000cm³ of FA3 contain $\left(\frac{0.0185}{250} \times 1000\right)$ *moles of sodium thiosulphate per litre*

= 0.0742 M

b) Calculate the number of moles of aqueous iodine in FA2 that reacted with thiosulphate ions in FA3.

1000cm³ of FA3 contain 0.0742moles of sodium thiosulphate

17.75cm³ of FA3 contain $\left(\frac{0.0742}{1000} \times 17.75\right)$ *moles of sodium thiosulphate*

=0.00132*moles of sodium thiosulphate*

Moles of I_2 $= \frac{1}{2}$*x moles of* $S_2O_3^{2-}$

$= \left(\frac{1}{2} \times 0.00132\right) =$ ***0.000658*** *moles of* I_2

c) Determine themolar concentration of aqueous iodine in the original FA1 solution.

25cm³ of FA2 contain 0.000658 moles of I_2

250cm³ of FA2 contain $\left(\frac{0.00658}{25} \times 250\right)$ *moles of* I_2

= 0.00658 *moles of* I_2

150cm³ of FA1 contain 0.00658 moles of I_2

1000cm³ of FA1 contain $\left(\frac{0.00658}{150} \times 1000\right)$ *moles of* I_2

=0.0439M

Worked out example 4.4.1.2

You are provided with the following:
FA1 which is 10% potassium iodide solution
FA2 which is sodium thiosulphate solution
FA3 which is potassium dichromate(VI) solution
1M sulphuric acid
Solid W which is potassium iodate, KIO_3

You are required to determine the concentration of:

i) sodium thiosulphate in FA2 in moldm^{-3}.

ii) potassium dichromate(VI) In FA3 in gdm^{-3}.

Theory

Iodate ions in acidic medium, react with iodide ions according to the equation below:

$IO_3^-(aq) + 6H^+(aq) + 5I^-(aq) \rightarrow 3I_2(aq) + 3H_2O(l)$.....*(*)*

Iodide ions are oxidized toaqueous iodine according to the following equation:

$2I^-(aq) \rightarrow I_2(aq) + 2e^-$

Note: *Equation (*) above is arrived at through the following series of reactions.*

$2IO_3^-(aq) + 12H^+(aq) + 10e^- \rightarrow I_2(aq) + 6H_2O(l)$.....*(i)*

$2I^-(aq) \rightarrow I_2(aq) + 2e^-$.....*(ii)*

Multiplying equation (ii) by 5 gives:

$10I^-(aq) \rightarrow 5I_2(aq) + 10e^-$.....*(iii)*

Adding equation (i) to (iii) gives:

$2IO_3^-(aq) + 12H^+(aq) + 10I^-(aq) \rightarrow I_2(aq) + 6H_2O(l) + 5I_2(aq)$ .....*(iv)*

Dividing equation (iv) by 2 gives:

$\boldsymbol{IO_3^-(aq) + 6H^+(aq) + 5I^-(aq) \rightarrow 3I_2(aq) + 3H_2O(l)}$

PART I

Procedure

Weigh accurately, 2.7g of W into a clean beaker. Add about 150cm^{3}of distilled water and stir well to dissolve. Transfer the solution into a 250cm^{3}volumetric flask and make up to the mark with distilled water. Label the resultant solution FA4.

Pipette 10cm^3 of FA4 into a conical flask. Using a measuring cylinder, add 10cm^3 of FA1 followed by 10cm^3 of 1M Sulphuric acid and shake well to mix and titrate with FA2 from the buretteuntil the solution turns pale yellow/pale brown.Then add 2cm^3 of starch indicator and continue titratingwith FA2 until the blue-black starch-iodine complex is just discharged. Record your results in the spaces below.

Mass of container + W……*45.1*……..g

Mass of container alone……..*42.4*……..g

Mass of solid W……*2.7*……...g

Capacity of pipette used……..…*25.0*……..cm^3

Table I

Final burette reading (cm^3)	*09.70*	*09.50*	*15.50*
Initial burette reading (cm^3)	*0.00*	*0.00*	*6.00*
Volume of FA2 used(cm^3)	*09.70*	*09.50*	*09.50*

Values used to calculate average……*09.50, 09.50*...……..cm^3

Average volume of FA3 used……...…….$\left(\frac{\mathbf{09.50 + 09.50}}{\mathbf{2}}\right)$=***09.50***…….. *cm^3*

Questions

a) Calculate the concentration of the potassium iodate in FA4 in mol dm^{-3}.

*Molar mass of KIO_3 = [(39x1) + (127x1) + (16x3)]=**214g***

214g of potassium iodate contain 1 mole

2.7g of potassium iodate contain $\left(\frac{1}{214} \text{ x } 2.7\right)$ *moles*

250cm^3 of FA4 contain $\left(\frac{2.7}{214} \text{ x } 1000\right)$ *moles*

1000cm^3 of contain $\left(\frac{2.7}{214\ x\ 250} \text{ x } 1000\right)$ *moles per dm^3*

$= \boldsymbol{0.05\ moldm^{-3}}$

b) Determine the concentration of the sodium thiosulphate in FA2 in moldm^{-3}.

1000cm^3 of FA4 contain 0.05 moles of IO_3^-

10cm^3 of FA4 contain $\left(\frac{0.05}{1000} \text{ x } 10\right)$ *moles of IO_3^-*

=0.0005 moles of IO_3^-

Moles of I_2 = 3xmoles of IO_3^-
= (3x0.0005)
= 0.0015
$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$
Moles of $S_2O_3^{2-}$= (2xmoles of I_2)
= (2x0.0015)
= 0.003
9.50cm^3 of FA2 contain 0.003 moles of $S_2O_3^{2-}$
1000cm^3 of FA2 contain $\left(\frac{0.003}{9.50} \text{x } 1000\right)$ moles of $S_2O_3^{2-}$
= 0.316 moldm^{-3}

PART II

Procedure

Pipette 10cm^3 of FA3 into a conical flask. Using a measuring cylinder, add 20$cm^3$of 1M sulphuric acid followed by 10$cm^3$of FA1 and shake well to mix and titrate with FA2 from the buretteuntil the solution is pale yellow. Then add 2cm^3 of Starch indicator and titrate continue the titration with FA2 until the solution turns pale blue. Repeat the titration until you obtain consistent results. Record your results in the table below.Record your results in the table below.

Table II

Final burette reading (cm^3)	*45.60*	*45.40*	*46.40*
Initial burette reading (cm^3)	*0.00*	*0.00*	*1.00*
Volume of FA2 used(cm^3)	*45.60*	*45.40*	*45.40*

Values used to calculate average……*45.40, 45.40*…… ……*cm^3*
Average volume of FA2 used……$\left(\frac{\mathbf{45.40 + 45.40}}{\mathbf{2}}\right)$=***45.40***…… *$cm^3$*

c) Calculate the number of moles of the liberated iodine that reacted with thiosulphate ions in FA2.
1000cm^3 of FA2 contain 0.316 moles of $S_2O_3^{2-}$
45.40cm^3 of FA2 contain $\left(\frac{0.316}{1000} \text{x } 45.40\right)$moles of $S_2O_3^{2-}$

$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$
Moles of I_2=$\frac{1}{2}$x moles of$S_2O_3^{2-}$
=$\frac{1}{2}$ x $\left(\frac{0.316}{1000} \text{x } 45.40\right)$
***= 0.00717** moles of I_2*

d) Determine the concentration of potassium dichromate in FA3 in gdm^{-3}.(K=39, Cr=52, O=16)

$Cr_2O_7^{2-}(aq) + 14H^+(aq) + 6I^-(aq) \rightarrow 2Cr^{3+}(aq) + 7H_2O(l) + 3I_2(aq)$

Moles of $Cr_2O_7^{2-}$= $\frac{1}{3}$x moles of I
=$\frac{1}{3}$ x 0.00717
= 0.00239 moles of $Cr_2O_7^{2-}$

$10cm^3$ of FA3 contain 0.00239 moles of $Cr_2O_7^{2-}$
$1000\ cm^3$ of FA3 contain $\left(\frac{0.00239}{10} \times 1000\right)$ moles of $Cr_2O_7^{2-}$
***= 0.239** mol dm^{-3}*
*Molar mass of $K_2Cr_2O_7$ = [(39x2) + (52x2) + (16x7)]= **294**g*
1 mole of $K_2Cr_2O_7$ weighs 294g
0.239 moles of $K_2Cr_2O_7$ weigh (294 x 0.239)g
= 70.266 gdm^{-3}

Worked out example 4.4.1.3

You are provided with the following:
GA1 which contains 3.6g of potassium dichromate(VI) per litre.
GA2 which is hydrogen peroxide solution
GA3 which is sodium thiosulphate pentahydrate
GA4 which is 10% potassium iodide solution
1.5M sulphuric acid
You are required to determine the:
i) concentration of sodium thiosulphate in grams per litre in GA3.
ii) volume strength of the hydrogen peroxide solution in GA2.
(K=39, Cr =52, O=16)

Theory

In acidic medium, dichromate(VI) ions and Hydrogen peroxide react with iodide ions as shown by the equations below.

$$Cr_2O_7^{2-}(aq) + 14H^+(aq) + 6I^-(aq) \rightarrow 2Cr^{3+}(aq) + 7H_2O(l) + 3I_2(aq)$$
$$H_2O_2(aq) + 2H^+(aq) + 2I^-(aq) \rightarrow I_2(aq) + 2H_2O(l)$$

Procedure I

Pipette 20 or 25cm^3 of GA1 into a clean conical flask. Add an equal volume of GA4 followed by 30cm^3 of 1.5M sulphuric acid using a measuring cylinder and titrate with GA3 from the burette until the solution turns pale yellow; add 1cm^3 of starch indicator and continue the titration until the blue-black starch-iodine complex turns pale blue. Record your results in table I below.

Capacity of pipette used……*25.0*……..cm^3

Table I

Final burette reading (cm^3)	*23.10*	*34.90*	*22.90*
Initial burette reading (cm^3)	*0.00*	*12.00*	*0.00*
Volume of GA3 used(cm^3)	*23.10*	*22.90*	*22.90*

Values used to calculate average……*22.90, 22.90*……cm^3

Average volume of FA3 used……$\left(\frac{22.90 + 22.90}{2}\right)$… = ***22.90***……..$cm^3$

……

a) Determine the molar concentration of:

i) potassium dichromate(VI) in GA1.

Molar Mass of $K_2Cr_2O_7$= [(39x2)+(52x2)+(16x7)]= ***294g***

294g of $K_2Cr_2O_7$ contain 1 mole

3.6g of $K_2Cr_2O_7$ contain $\left(\frac{1}{294} \text{x } 3.6\right)$ *moles*

= 0.012M

ii) sodium thiosulphate in GA3.

1000cm^3 of GA1 contain 0.012 moles of $Cr_2O_7^{2-}$

25cm^3 of GA1 contain $\left(\frac{0.012}{1000} \text{x } 25\right)$ *moles of $Cr_2O_7^{2-}$*

= 0.0003 moles of $Cr_2O_7^{2-}$

Moles of I_2 formed = (3xmoles of $Cr_2O_7^{2-}$)

= (3x0.0003)

= 0.0009

$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$

Moles of $S_2O_3^{2-}$= (2xmoles of I_2)

= (2x0.0009)

= 0.0018

22.90cm^3 of GA3 contain 0.0018 moles of $S_2O_3^{2-}$

1000cm^3 of GA3 contain $\left(\frac{0.00182}{22.90} \text{x } 1000\right)$ *moles of $S_2O_3^{2-}$*

= 0.0786M

Procedure II

Using a measuring cylinder, measure and transfer 75cm^3 of GA3 into a clean beaker. Add 75cm^3 of distilled water, shake well to mix and label the solution GA5.

Pipette 20 or 25cm^3 of GA2 into a clean conical flask. Add an equal volume of GA4 followed by 30cm^3 of 1.5M Sulphuric acid using a measuring cylinder. Leave the mixture to settle for 10 minutes and then titrate with GA5 from the burette as you shake the conical flask and its contents vigorously until the solution turns pale yellow; add 1cm^3 of starch indicator and continue the titration until the blue-black starch-iodine complex turns colourless. Record your results in table II below.

Capacity of pipette used……*25.0*……..cm^3

Table II

Final burette reading (cm^3)	*31.40*	*39.20*	*31.10*
Initial burette reading (cm^3)	*0.00*	*8.00*	*0.00*
Volume of GA5 used(cm^3)	*31.40*	*31.20*	*31.10*

Values used to calculate average……*31.20, 31.10*…… cm^3

Average volume of GA5 used……$\left(\frac{31.20 + 31.10}{2}\right)$……= ***31.15***……cm^3

b) Calculate the:

i) molar concentration of sodium thiosulphate in GA5.

1000cm^3 of GA3 contain 0.0786moles of $S_2O_3^{2-}$

75cm^3 of GA3 contain $\left(\frac{0.0786}{150} x\ 75\right)$ *moles of $S_2O_3^{2-}$*

= 0.005895 moles of $S_2O_3^{2-}$

Total volume of GA5= (75+75) = 150cm^3

150cm^3 of GA5 contain 0.005895moles of $S_2O_3^{2-}$

1000cm^3 of GA5 contain $\left(\frac{0.005895}{150} x\ 1000\right)$ *moles of $S_2O_3^{2-}$*

=0.0393M

ii) molar concentarion of hydrogen peroxide in the GA2 solution.

1000cm^3 of GA5 contain 0.0393 moles of $S_2O_3^{2-}$

31.15cm^3 of GA5 contain $\left(\frac{0.0393}{1000} x\ 31.15\right)$ *moles of $S_2O_3^{2-}$*

= 0.00122moles of $S_2O_3^{2-}$

$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$

Moles of I_2 that reacted = $\left(\frac{1}{2} x\ moles\ of\ S_2O_3^{2-}\right)$

$= \left(\frac{1}{2} x\ 0.00122\right)$

=0.000612moles of I_2

Moles of H_2O_2 that reacted =(1xmoles of I_2 formed)

=(1 x 0.000612)

=0.000612

25cm^3 of GA2 contain 0.000612moles of H_2O_2

1000cm^3 of GA2 contain $\left(\frac{0.000612}{25} x\ 1000\right)$ *moles of H_2O_2*

=0.0245M

iii) volume strength of hydrogen peroxide in the GA2 solution.

(**NB:** Volume strength is the volume of oxygen gas liberated by 1cm^3 of hydrogen peroxide solution; 1 mole of a gas occupies 24dm^3 at room temperature)

1000cm^3 of GA2 contain 0.0245moles of H_2O_2

1cm^3 of GA2 contains $\left(\frac{0.0245}{1000}\right)$ *moles of H_2O_2*

= 0.0000245moles of H_2O_2

$2H_2O_2(aq) \rightarrow 2H_2O(l) + O_2(g)$

2moles of hydrogen peroxide evolve 1 mole of oxygen gas

0.0000245 moles of hydrogen peroxide evolve $\left(\frac{1}{2} x\ 0.0000245\right)$ *moles of oxygen gas*

= 0.00001225moles of oxygen gas

1 mole of oxygen gas occupies 24dm^3 at room temperature

0.00001225 moles of oxygen gas occupy (24x0.00001225) dm^3 at room temperature

Volume strength of hydrogen peroxide in GA2 = 0.000294dm^3

Worked out example 4.4.1.4

You are provided with the following:
FA1 which is a solution that contains 11.16g of sodium thiosulphate pentahydrate, $Na_2S_2O_3.5H_2O$ in $500cm^3$ of solution.
FA2 which is potassium manganate(VII) solution.
0.6M potassium iodide solution.
2.0M sulphuric acid solution.
Solid V which is impure ferrous ethanedioate, FeC_2O_4.
You are required to determine the:
(i) molarity of the potassium mangante(VII) solution.
(ii) percentage impurity in the ferrous ethanedioate sample.

Theory

Acidified manganate(VII) ions react with thiosulphate ions, iron(II) ions and ethanedioate ions according to the following equations.

$$2MnO_4^-(aq) + 16H^+(aq) + 10S_2O_3^{2-}(aq) \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) + 5S_4O_6^{2-}(aq)$$

$$MnO_4^-(aq) + 8H^+(aq) + 5Fe^{2+}(aq) \rightarrow Mn^{2+}(aq) + 4H_2O(l) + 5Fe^{3+}(aq)$$

$$2MnO_4^-(aq) + 16H^+(aq) + 5C_2O_4^{2-}(aq) \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) + 10CO_2(g)$$

Procedure I

Pipette $25cm^3$ (or $20cm^3$) of FA2 into a conical flask. Add $15cm^3$ of 2.0M sulphuric acid followed by $15cm^3$ of 0.6M potassium iodide solution and titrate the solution with FA1 from the burette until the solution becomes pale yellow. Add $1cm^3$ of starch indicator and continue the titration until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in table I below.

Capacity of pipette used……*25.0*……cm^3

Table I

Final burette reading (cm^3)	*27.80*	*33.50*	*29.50*
Initial burette reading (cm^3)	*0.00*	*6.00*	*2.00*
Volume of FA1 used(cm^3)	*27.80*	*27.50*	*27.50*

Titre values used to calculate verage ……*27.50, 27.50*……..cm^3

Average volume of FA1 used……$\left(\frac{27.00 + 27.00}{2}\right)$……= ***27.50***…….……$cm^3$

Procedure II

Weigh accurately1.5g of V into a clean beaker. Add $100cm^3$ of 2.0M sulphuric acid and stir well to dissolve. Transfer the resultant solution into a $250cm^3$ volumetric flask and make up to the mark by addition of more distilled water. Label the solution FA3.

Pipette 25cm^3 (or 20cm^3) of FA3 into a conical flask and heat the solution to 70^{0}C. Titrate the hot solution with FA2 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in table II below.

Mass of beaker + V……*38.7*……g
Mass of beaker……*37.2*……g
Mass of V……*1.5*……g
Capacity of pipette used……*25.0*……cm^3

Table II

Final burette reading (cm^3)	*27.00*	*31.10*	*27.00*
Initial burette reading (cm^3)	*0.00*	*4.00*	*0.00*
Volume of FA2 used(cm^3)	*27.00*	*27.10*	*27.00*

Titre values used to calculate verage ……*27.00, 27.00*……cm^3

Average volume of FA2 used ……$\left(\frac{27.00+27.00}{2}\right)$……=***27.00***……cm^3

(a) Calculate the:

(i) molarity of potassium manganate(VII) in FA2. (Na=39, S=32, O=16)

Molar mass of $Na_2S_2O_3.5H_2O$= [(23x2) + (32x2) + (16x3)+(5x1x2)+(16x5)]= ***248****g*

248g of $Na_2S_2O_3.5H_2O$contains 1 mole

11.16g of $Na_2S_2O_3.5H_2O$contains $\left(\frac{1}{248}\text{x } 11.16\right)$*moles = 0.045moles*

500cm^3 of FA1 contain 0.045molesof $S_2O_3^{2-}$

1000cm^{3}of FA1 contain $\left(\frac{0.045}{500}\text{x } 1000\right)$ *molesof $S_2O_3^{2-}$*

= 0.09M

1000cm^{3}of FA1 contain0.09moles of $S_2O_3^{2-}$

27.50cm^{3}of FA1 contain $\left(\frac{0.09}{1000}\text{x } 27.50\right)$ *molesof $S_2O_3^{2-}$*

= 2.475x10^{-3}molesof $S_2O_3^{2-}$

Moles of MnO_4^- = $\frac{2}{10}$*x moles of $S_2O_3^{2-}$*

$= \frac{2}{10} x\ 2.475x10^{-3} = 4.95x10^{-4}$*moles*

25cm^{3}of FA2 contain 4.95x10^{-4}moles of MnO_4^-

1000cm^{3}of FA2 contain $\left(\frac{4.95x10-4}{25}\text{x } 1000\right)$ *moles moles of MnO_4^-*

= 0.0198M

(ii) moles of manganate(VII) ions that reacted with 25cm^3 (or 20cm^3) of FA3.

1000cm^{3}of FA2 contain 0.0198 moles of MnO_4^-

27.00cm^{3}of FA2 contain $\left(\frac{0.0198}{1000}\text{x } 27.00\right)$ *moles of MnO_4^-*

= 5.346x10^{-4} moles of MnO_4^-

(iii) moles of iron(II) ions in 25cm^3 (or 20cm^3) of FA3.

Combining the two equations of reaction between $MnO_4^-$and both $Fe^{2+}$and $C_2O_4^{2-}$, we have:

$$3MnO_4^-(aq)+24H^+(aq)+5Fe^{2+}(aq)+5C_2O_4^{2-}(aq)\rightarrow 3Mn^{2+}(aq)+12H_2O(l)+5Fe^{3+}(aq)+10CO_2(g)$$

Moles of $Fe^{2+}=\frac{5}{3}x$ *moles of* MnO_4^-

$=\left(\frac{5}{3}x\ 5.346x10-4\right)=$ ***8.91x10⁻⁴ moles***

(b) Determine the:

(i) mass of ferrous ethanedioate, FeC_2O_4 in 250cm^3 of FA3. (Fe = 56, C=12, O=16)

25cm³ of FA3 contain 8.91x10⁻⁴ moles of FeC_2O_4

250cm³ of FA3 contain $\left(\frac{8.91x10-4}{25}x\ 250\right)$ *moles of* FeC_2O_4

$= 8.91x10^{-3}$ *moles of* FeC_2O_4

Molar mass of FeC_2O_4 *= (56x1)+(12x2)+(16x4) =* ***144g***

1mole of FeC_2O_4 *weighs 144g*

8.91x10⁻³ moles of FeC_2O_4 *weighs (144 x 8.91x10⁻³)g*

= 1.283g

(ii) percentage impurity in the ferrous ethanedioate sample.

Mass of the impurity in the sample = (1.5—1.283) = 0.217g

Percentage impurity $=\left(\frac{mass\ of\ the\ impurity}{total\ mass\ of\ impure\ sample}x\ 100\right)\%$

$\left(\frac{0.217}{1.5}x\ 100\right)\%$ ***= 14.47%***

4.4.2Practical Exercises on Iodimetry and IodometryTitrations

Experiment 4.4.2.1

You are provided with the following:

FA1 which is 0.06M iodine solution

FA2 which is a solution containing 17.4g of themetal thiosulphate, $XS_2O_3.5H_2O$ in one litre of solution.

You are required to determine the molarity of the metal thiosulphate in FA2 and hence the value of X in $XS_2O_3.5H_2O$***.***

Theory

Aqueous iodine reacts with thiosulphate ions according to the following equation.

$$I_2(aq)\ +\ 2S_2O_3^{2-}(aq)\ \rightarrow 2I^-(aq)\ +\ S_4O_6^{2-}(aq)$$

Procedure

i) Using a measuring cylinder, measure and transfer 100cm^3 of FA1 into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label the solution FA3.

ii) Pipette 20 or 25cm^3 of FA3 into a clean conical flask and titrate with FA2 from the burette until the solution turns pale yellow. Add 1cm^3 of starch solution and continue the titration until the solution just turns colourless. Repeat the titration until you obtain consistent results. Record your results in the table below.

Capacity of pipette used……cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA2used(cm^3)			

Values used to calculate average…….....cm^3

Average volume of FA2 used…….……cm^3

a) Calculate the molarity of iodine in FA3.

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b) Determine the:

i) number of moles of thiosulphate ions in FA2 that reacted with the iodine in FA3

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ii) molarity of the thiosulphate ions in FA2 and hence the value of X in $XS_2O_3.5H_2O$.
(S=32, O=16, H=1)

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Experiment 4.4.2.2

You are provided with the following:
FA1 which is potassium manganate(VII) solution
FA2 which issodium thiosulphate solution
FA3 which is 10% potassium iodide solution
FA4 which is 2.0M sulphuric acid
Solid M which is potassium dichromate(VI)
You are required to determine the molar concentration of FA2 and then use it to determine the molar concentration of FA1.

Theory

Dichromate(VI) ions and manganate(VII) ions in an acidic medium, react with iodide ionsaccording to the following equations:

$$Cr_2O_7^{2-}(aq) + 14H^+(aq) + 6I^-(aq) \rightarrow 2Cr^{3+}(aq) + 7H_2O(l) + 3I_2(aq)$$
$$2MnO_4^-(aq) + 16H^+(aq) + 6I^-(aq) \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) + 5I_2(aq)$$

Aqueous iodine reacts with thiosulphate ions according to the following equation.

$$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$$

Procedure I

Weigh accurately 1.4g of solid M into a clean beaker. Measureand transfer about 60cm^{3}of FA4 into the beaker containing solid M and stir well to dissolve. Transfer the solution into a 250cm^3 volumetric flaskand make up to the mark with distilled water. Label the solution FA5.
Pipette 20 or 25cm^3 of FA5 into a conical flask. Add 15cm^3 of FA3 followed by 15cm^3 of FA4. Titrate the resultant mixture with FA2 from the burette until the solution turns pale yellow. Add 1cm^3 of Starch solution and continue the titration until the solution turns from dark blue to a pale blue solution. Repeat the titration until you obtain consistent results. Record your results in table I below.

Mass of container + M…….g
Mass of container alone…….....g
Mass of solid M……g
Capacity of pipette used……...cm^3

Table I

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA2 used(cm^3)			

Values used to calculate average…….....cm^3

Average volume of FA2 used…….....cm^3

Questions

a) Calculate the:

i) number of moles of $Cr_2O_7^{2-}$ ions in the 20 or 25cm^3 of FA5 pipetted.
(K=39, Cr=52, O=16).

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ii) molar concentration of FA2

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Procedure II

Pipette 20 or 25cm^3 of FA1 into a conical flask. Add 15cm^3 of FA3 followed by 15cm^3 of FA4. Titrate the resultant mixture with FA2 from the burette until the solution turns pale yellow. Add 1cm^3 of Starch solution and continue the titration until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in table II below.

Table II

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA2 used(cm^3)			

Values used to calculate average……cm^3

Average volume of FA2 used……cm^3

b) Determine the molar concentration of FA1.

……

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……

Experiment 4.4.2.3

You are provided with the following:
HA1 which is a solution containing 1.12g of potassium chromate in 200cm^3 of solution.
HA2which is a solution made by dissolving 25.0g of a hydrated metal thiosulphate, $MS_2O_3.xH_2O$ in one litre of solution.
HA3 is 10% potassium iodide solution
HA4 is 2M sulphuric acid
Solid T which is impure potassium iodate, KIO_3.
You are required to determine the:

i) molarity of the hydrated metal thiosulphate in HA2 and the percentage of water of crystallization in the metal thiosulphate, $MS_2O_3.xH_2O$.

ii) percentage purity of the potassium iodate used in the preparation of HA1.

Theory

In acidic medium, CrO_4^{2-} ions are converted to dichromate ions according to the following equation:

$$2CrO_4^{2-}(aq) + 2H^+(aq) \rightarrow Cr_2O_7^{2-}(aq) + H_2O(aq)$$

Still, in acidic medium, IO_3^- and $Cr_2O_7^{2-}$ react with iodide ions according to the equations below:

$$IO_3^-(aq) + 6H^+(aq) + 5I^-(aq) \rightarrow 3I_2(aq) + 3H_2O(l)$$

$$Cr_2O_7^{2-}(aq) + 14H^+(aq) + 6I^-(aq) \rightarrow 2Cr^{3+}(aq) + 7H_2O(l) + 3I_2(aq)$$

PART I

Procedure

Pipette 20 or 25cm^3 of HA1 into a clean conical flask. Add 30cm^3 of the 2M sulphuric acid followed by 10cm^3 of the 10% potassium iodide solution and titrate with HA2 from the burette until the solution becomes pale yellow; then add 1cm^3 of starch indicator and continue the titration until the dark blue solution just turns to a pale bluesolution. Repeat the titration until you obtain consistent results. Record your results in table I below.

Capacity of pipette used……...cm^3

Table I

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of HA2 used(cm^3)			

Values used to calculate average…….....cm^3

Average volume of HA2 used……..…….....cm^3

Questions

a) Calculate the number of moles of CrO_4^{2-} ions in the 20 or 25cm^3 of HA1 pipetted.

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b) Determine the:

i) molarity of the $S_2O_3^{2-}$ ions in HA2.

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ii)value of x in $MS_2O_3.xH_2O$.(M=46, S=32, O=16, H=1)

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iii) percentage by mass of water of crystallisation in the hydrated metal thiosulphate,$MS_2O_3.xH_2O$.

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PART II

Procedure

i) Weigh accurately, 1.0g of solid T into a clean beaker. Add about 150cm^3 of water and stir well to dissolve. Transfer the solution to a 250cm^3 volumetric flask and make up to the mark with distilled water. Label the solution HA5.

ii) Using a measuring cylinder, measure and transfer 25cm^{3}of HA5 into a clean conical flask and add 10cm^3 of the 10% potassium iodide solution followed by 10cm^3 of the 2M sulphuric acid and then titrate the liberated Iodine with HA2 from the burette until the solution is pale yellow; then add 1cm^3 of starch indicator and continue the titration until the solution just turns colourless.Repeat the titration until you obtain consistent results. Record your results in table II below.

Mass of container + T……...g

Mass of container alone…….....g

Mass of solid T…….g

Table II

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of HA2 used(cm^3)			

Values used to calculate average……cm^3

Average volume of HA2 used…….cm^3

Questions

c) Determine the:

i) number of moles of IO_3^- ions in the 250cm^3 of HA5.

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ii)mass of pure potassium iodate in the 250cm^3 of HA5and hence the percentage purity of the potassium iodate sample used in the preparation of HA5.(K=39, I=127, O=16)

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Experiment 4.4.2.4

You are provided with the following:
FA1 which contains 1.8g of potassium dichromate(VI) in 500cm^3 of solution.
FA2 which ishydrogen peroxide solution
FA3 which issodium thiosulphate-5-water
FA4 which is 10% potassium iodide solution
1.5M sulphuric acid
You are required to determine the:
i) concentration of sodium thiosulphate in grams per litre in FA3.
ii)volume strength of the hydrogen peroxide solution in FA2.

Theory

In acidic medium, dichromate(VI) ions and hydrogen peroxide react with iodide ions as shown by the equations below.

$$Cr_2O_7^{2-}(aq) + 14H^+(aq) + 6I^-(aq) \rightarrow 2Cr^{3+}(aq) + 7H_2O(l) + 3I_2(aq)$$
$$H_2O_2(aq) + 2H^+(aq) + 2I^-(aq) \rightarrow I_2(aq) + 2H_2O(l)$$

Procedure I

Pipette 20 or 25cm^3 of FA1 into a clean conical flask. Add an equal volume of FA4 followed by 30cm^3 of 1.5M sulphuric acid using a measuring cylinder and titrate with FA3 from the burette until the solution turns pale yellow; add 1cm^3 of starch indicator and continue the titration until the blue-black starch-iodine complex turns pale blue.

Capacity of pipette used……..cm^3

Table I

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA3 used(cm^3)			

Values used to calculate average……cm^3

Average volume of FA3 used……cm^3

a) Determine the molar concentration of:
i) potassium dichromate(VI) in FA1.

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ii) sodium thiosulphate in FA3.

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Procedure II

Using a measuring cylinder, measure and transfer 100cm^3 of FA3 into a clean beaker. Add 100cm^3 of distilled water, shake well to mix and label the solution FA5.

Pipette 20 or 25cm^3 of FA2 into a clean conical flask. Add an equal volume of FA4 followed by 30cm^3 of 1.5M Sulphuric acid using a measuring cylinder. Leave the mixture to settle for 12 minutes and then titrate with FA5 from the burette as you shake the conical flask and its contents vigorously until the solution turns pale yellow; add 1cm^3 of starch indicator and continue the titration until the blue-black starch-iodine complex turns colourless.

Capacity of pipette used……..cm^3

Table II

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA5 used(cm^3)			

Values used to calculate average……cm^3

Average volume of FA5 used…….....cm^3

b) Calculate the:

i) molar concentration of sodium thiosulphate in FA5.

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ii) molar concentarion of hydrogen peroxide in the FA2 solution.

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iii) volume strength of hydrogen peroxide in the FA2 solution.

(NB: Volume strength is the volume of oxygen gas liberated by 1cm^3 of hydrogen peroxide solution; 1 mole of a gas occupies 24dm^3 at room temperature)

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Experiment 4.4.2.5

You are provided with the following:
FA1 which contains 19.84g of sodium thiosulphate-5-water in one litre of solution.
FA2 which is 10% potassium iodide solution
FA3 which is 2M sulphuric acid
Solution Bwhich is Jik solution [a solution of a bleaching agent that contains hypochlorite/chloric(I) ions]
You are required to determinethe:

i) concentration of sodium thiosulphate in FA1in moldm^{-3}.
ii) percentage by mass of aqueous chlorine in solution B.

Theory

Solutions of bleaching agents such as Jik are prepared by bubbling chlorine gas through a cold dilute solution of sodium hydroxide. Sodium chloride, sodium hypochlorite and water are formed according to the following equation.

$$Cl_2(g) + 2NaOH(aq) \rightarrow NaCl(aq) + NaOCl(aq) + H_2O(l)$$

Addition of a dilute acid to a solution of such a bleaching agent liberates aqueous chlorine according to the equation below.

$$Cl^-(aq) + OCl^-(aq) + 2H^+(aq) \rightarrow Cl_2(aq) + H_2O(l)$$

Since chlorine is more reactive than iodine, the aqueous chlorine has the ability to displace iodide ions from the salt potassium iodide, forming aqueous iodine as shown below.

$$Cl_2(aq) + 2I^-(aq) \rightarrow 2Cl^- + I_2(aq)$$

The aqueous iodine formed can thus be titrated against standard sodium thiosulphate solution in accordance with the equation below.

$$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$$

Procedure

i) Using a suitable measuring cylinder, measure 20cm^3 of solution B into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label the resultant solution FA4.

ii) Using a measuring cylinder, measure and transfer 25cm^3 of FA4 into a clean conical flask. Using a measuring cylinder, add about 10cm^3 of FA3 followed by 10cm^3 of FA2. Titratethe liberated iodine with FA1 from the burette until the solution turns pale yellow; add 1cm^3 of starch indicator and continue the titration until the blue-black starch-iodine complex just turns colourless. Repeat the titration until you obtain consistent results. Record your results in the table below.

Capacity of pipette used……..cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA1 used(cm^3)			

Values used to calculate average…….cm^3

Average volume of FA1 used…….....cm^3

a) Determine the concentration of sodium thiosulphate in FA1 in moldm^{-3}.
(Na=23, S=32, O=16, H=1)

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b)Calculate the:

i) number of moles of aqueous iodine liberated by 25cm^3 of FA4.

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ii) massof aqueous chlorine in the $20cm^3$ of solution B.(Cl=35.5)

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iii) concentration of aqueous chlorine in solution B in gdm^{-3}.

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iv) percentage by mass of aqueous chlorine in solution B. (Density of solution B=$1gcm^{-3}$)

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Experiment 4.4.2.6

You are provided with the following:
GA1 which is 10% potassium iodide solution.
GA2 which contains 8.68gof sodium thiosulphate in 500cm^3 of solution.
2M sulphuric acid
Solid M which is bleaching powder ($CaOCl_2$).
You are required to determinethe:
i) molarity of sodium thiosulphate in GA1.
ii) percentage by mass of available chlorine in the bleaching powder sample.

Theory

The bleaching owder can be dissolved in a specific volume of water and the mixture stirred to make a dilute solution of the calcium hypochlorite.

On addition of a dilute acid to thecalcium hypochlorite solution, aqueous chlorine is liberated from the calcium hypochlorite, $CaOCl_2$according to the equation below.

$$CaOCl_2(aq) + 2H^+(aq) \rightarrow Ca^{2+}(aq) + Cl_2(aq) + H_2O(l)$$

Since chlorine is more reactive than iodine, the aqueous chlorine has the ability adisplace iodide ions from its salt, forming aqueous iodine as shown below.

$$Cl_2(aq) + 2I^-(aq) \rightarrow 2Cl^- + I_2(aq)$$

The aqueous iodine formed can then be titrated against standard sodium thiosulphate solution in according to the equation below.

$$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$$

Procedure

Weigh accurately, 1.1g of solid M into a beaker. Add 100cm^3 of water and transfer the resultant solution into a 250cm^3 volumetric flask. Label the solution GA3.

Pipette 25.0 or 20.0cm^3 of GA3 into a conical flask. Using a measuring cylinder, add 10cm^3 of 2M sulphuric acid followed by 25cm^3 of GA1and titrate the liberated iodine with GA2 from the burette until the solution turns pale yellow. Add1cm^3 of starch indicator and continue the titration until the blue-black starch-iodine complex just turns colourless. Repeat the titration until you obtain consistent results. Record your results in the table below.

Capacity of pipette used……..cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of GA2 used(cm^3)			

Values used to calculate average……cm^3

Average volume of GA2 used…….....cm^3

b) Calculate the molarity of sodium thiosulphate in GA2.
(Na=23, S=32, O=16, H=1)

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b)Calculate the:

i) number of moles of aqueous iodine liberated by 25.0 or 20.0cm^3 of GA3.

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ii) mass ofchlorine in the 250cm^3 of GA3.(Cl=35.5)

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iii) percentage by mass of available chlorine in solid M.

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Experiment4.4.2.7

You are provided with the following:
FA1 which is sodium thiosulphate solution.
FA2 which is a solution containing 20.5g of a hydrated copper(II) salt, $CuY.nH_2O$ per litre.
FA3 which is 10% potassium iodide solution
FA4 which is 1M sulphuric acid
Solid U which is potassium iodate
You are required to determine the:
i) concentration of sodium thiosulphate in FA1in moldm^{-3}.
ii) value of n in $CuY.nH_2O$ and hence the percentage by mass, of water of crystallisation in the hydrated copper(II) salt.

Theory

In acidic medium, iodate ions react with iodide ions according to the following equation.

$$IO_3^-(aq) + 5I^-(aq) + 6H^+(aq) \rightarrow H_2O(l) + 3I_2(aq)$$

Copper(II) ions react with iodide ions to form both copper(I) iodide and aqueous Iodine as shown by the equation below.

$$2Cu^{2+}(aq) + 4I^-(aq) \rightarrow Cu_2I_2(s) + I_2(aq)$$

The liberated Iodine can then be titrated with sodium thiosulphate as shown by the equation below.

$$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$$

Procedure I

(i) Weigh accurately, 1.0g of solid U into a clean beaker. Using a measuring cylinder, add about 100cm^{3}of water and stir well to dissolve. Transfer into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label the solution FA5.

Mass of container + U……..g
Mass of container alone…….....g
Mass of solid U…….g

(ii) Pipette 20 or 25cm^3 of FA5 into a clean conical flask. Using a measuring cylinder, add about 10cm^3 of FA4 followed by 10cm^3 of FA3.
(iii) Titrate the liberated Iodine with FA1 from the burette until the solution turns pale yellow; add 1cm^3 of starch indicator and continue the titration until the blue-black starch-iodine complex just turns colourless.
(iv) Repeat the titration until you obtain consistent results. Record your results in the table below.

Capacity of pipette used……..cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA1 used(cm^3)			

Values used to calculate average……cm^3

Average volume of FA1 used……cm^3

a) Determine the concentration of potassium iodate in FA5 in $moldm^{-3}$.*(K=39, I=127, O=16)*

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b) Calculate the concentration of the sodium thiosulphate in FA1in $moldm^{-3}$

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Procedure II

(i) Pipette 20 or 25cm^3 of FA2 into a clean conical flask. Using a measuring cylinder, add about 10cm^3 of FA3.

(ii) Titrate the liberated Iodine with FA1 from the burette until the solution turns pale yellow; add 1cm^3 of starch indicator and continue the titration until the blue-black starch-iodine complex just turns colourless.

(iii)Repeat the titration until you obtain consistent results. Record your results in the table below.

Capacity of pipette used……..cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA1 used(cm^3)			

Values used to calculate average……cm^3

Average volume of FA1 used……cm^3

c) Calculate the:

i) number of moles of copper(II)ions in FA2 which reacted with the iodide ions in FA3.

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ii) concentration of copper(II) ions in FA2 in $moldm^{-3}$.

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d) Determine the value of n in $CuY.nH_2O$ and hence the percentage by mass, of water of crystallization in the hydrated copper(II) salt. (Cu=64, Y=96, H=1, O=16)

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Experiment 4.4.2.8

GA1 which is a solution that contains 5.58g of hydrated sodium thiosulphate, $Na_2S_2O_3.5H_2O$ in 250cm^3 of solution.
GA2 which is potassium manganate(VII) solution.
10% potassium iodide solution.
2M sulphuric acid.
Solid Z which is impure iron(II) oxalate, $Fe(CO_2)_2$.
You are required to determine the:
(i) concentration of the potassium manganate(VII) solution in mol dm^{-3}.
(ii) percentage purity of theiron(II) oxalate sample.

Theory
Acidified manganate(VII) ions react with thiosulphate ions, iron(II) ions and oxalate ions according to the following equations.

$2MnO_4^-(aq) + 16H^+(aq) + 10S_2O_3^{2-}(aq) \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) + 5S_4O_6^{2-}(aq)$

$MnO_4^-(aq) + 8H^+(aq) + 5Fe^{2+}(aq) \rightarrow Mn^{2+}(aq) + 4H_2O(l) + 5Fe^{3+}(aq)$

$2MnO_4^-(aq) + 16H^+(aq) + 5C_2O_4^{2-}(aq) \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) + 10CO_2(g)$

Procedure A
Pipette 25cm^3 (or 20cm^3) of GA2 into a conical flask. Add 15cm^3 of 2.0M sulphuric acid followed by 15cm^3 of 10% potassium iodide solution and titrate the solution with GA1 from the burette until the solution becomes pale yellow. Add 1cm^3 of starch indicator and continue the titration until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in table I below.

Capacity of pipette used……cm^3

Table I

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of GA1 used(cm^3)			

Titre values used to calculate verage ……cm^3

Average volume of GA1 used……cm^3

Procedure B
i) Weigh accurately1.5g of Z into a clean beaker. Add 100cm^3 of 2.0M sulphuric acid and stir well to dissolve. Transfer the resultant solution into a 250cm^3 volumetric flask and make up to the mark by addition of more distilled water. Label the solution GA3.

ii) Pipette 25cm^3 (or 20cm^3) of GA3 into a conical flask and heat the solution to 70^{0}C. Titrate the hot solution with GA2 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in table II below.

Mass of beaker + Z……g
Mass of beaker……g
Mass of Z……g
Capacity of pipette used……cm^3

Table II

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of GA2 used(cm^3)			

Titre values used to calculate verage ……cm^3

Average volume of GA2 used ……cm^3

(a) Calculate the:
(i) molarity of potassium manganate(VII) in GA2. (Na=39, S=32, O=16)

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(ii) moles of manganate(VII) ions that reacted with $25cm^3$ (or $20cm^3$) of GA3.

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(iii) moles of iron(II) ions in $25cm^3$ (or $20cm^3$) of GA3.

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(b) Determine the:

(i) mass of iron(II) oxalate, FeC_2O_4 in $250cm^3$ of GA3. (Fe = 56, C=12, O=16)

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(ii) percentage purity of the iron(II) oxalate sample.

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CHAPTER FIVE
5.0BACK TITRATION

5.1Introduction

Back titration is a technique in volumetric analysis in which the amount of a given substance is not obtained by direct titration.In back titration, a given substance (J), which is a standard solution that is in excess, is reacted with a small amount of another substance(K) to give a product(L) mixed with the unreacted substance (J).The unreacted substance J is then reacted with a standard solution of another substance (M) to give another product (N).A summary of the reactions above is shown below.

J + K L + J ⟶ (Unreacted)
(Standard solution used in excess)

J + M N ⟶
(Unreacted) (Standard solution)

One of the reasons why this technique is referredto as back titration is because the calculations involved start with the number of moles of the reactants used in the final stages of the experiment followed by those of the reactants used in the initial stages of the experiment.

ILLUSTRATION

Experiment Procedure
INITIAL STAGES OF EXPERIMENTFINAL STAGES OF EXPERIMENT
Calculations

5.1.1 Stages followed in calculations of Back Titration

The stages will be explained basing on the summary of reactions shown above.
The calculations in back titration follow the following stages.

- The number of moles of the standard solutionof substance Mare calculated basing on the volume of substance M used in the final stage of the titration.
- By use of the mole ratio of reaction between M and Unreacted J, the moles of unreacted J are calculated.
- The total number of moles available in the total volume of the standard solution of substance J used initially is then calculated basing on the volume of the standard solution of substance J used initially.
- The number of moles of substance J that reacted with substance K can then be calculated by subtracting the moles of unreacted J (which reacted with M) from the total number of moles in the total volume of the standard solution of substance J initially used in the reaction with substance K.

5.1.2Applications of Back Titration

Back titration can be applied in the following ways:

- Determination of the percentage purity of electropositive metals such as magnesium, zinc, iron e.t.c. basing on their reaction with a solution of a suitable standard acid used in excess.
- Analysis of materials containing calcium carbonate, for instance egg shells of different animals, limestone, e.t.c., plus analysis of other metal carbonates.
- Determination of concentrations of oxalates, iodides, chromates and alike, in a series of redox reactions plus so many other applications.

5.2 Worked out examples on Back Titration

Worked out example 5.2.1

You are provided with the following:
FA1 which is 0.4M hydrochloric acid
FA2 which is 0.05M sodium hydroxide
Solid P which is impure barium carbonate
You are required to determine the percentage purity of the barium carbonate sample used.

Procedure

Weigh accurately, 1.4g of P into a clean beaker. Using a measuring cylinder, add 50cm^3 of water and stir to form a paste, followed by 100cm^3 of FA1 and continue to stir until effervescence stops. Transfer the resultant solution into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label the solution FA3.
Pipette 20 or 25cm^3 of FA2 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with FA3 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below.

Mass of container + P……*42.90*……..g
Mass of container alone……..*41.50*……..g
Mass of solid P……*1.40*……..g
Capacity of pipette used……*25*……cm^3

Final burette reading (cm^3)	*12.30*	*12.10*	*16.10*
Initial burette reading (cm^3)	*0.00*	*0.00*	*4.00*
Volume of FA3 used(cm^3)	*12.30*	*12.10*	*12.10*

Values used to calculate average……*12.10, 12.10*……*cm^3*
Average volume of FA3 used……..…$\left(\frac{\mathbf{12.10 + 12.10}}{\mathbf{2}}\right)$= ***12.10***……*cm^3*

Questions

a) Calculate the:

i) moles of sodium hydroxide in FA2 that reacted withhydrochloric acid in FA3

1000cm^3 of FA2 contain 0.05moles of sodium hydroxide

25cm^3 of FA2 contain $\left(\frac{0.05}{1000} \text{ x } 25\right)$ *moles of sodium hydroxide*

=0.00125 moles of sodium hydroxide

ii) moles of excess hydrochloric acid in FA3 that reacted with sodium hydroxide in FA2.

$$NaOH(aq) + HCl(aq) \rightarrow NaCl(aq) + H_2O(l)$$

Moles of HCl = (1xmoles of sodium hydroxide)

= (1 x 0.00125)
= 0.00125

iii)number of moles of excess hydrochloric acid in the 250cm^3 of FA3.

12.10cm^3 of FA3 contain 0.00125 moles of hydrochloric acid

250cm^{3}of FA3 contain $\left(\frac{0.000125}{12.10} \text{x } 250\right)$ *moles of hydrochloric acid*

$= \mathbf{0.0258}$ *moles of hydrochloric acid*

iv) number of moles of hydrochloric acid that reacted with barium carbonate.

1000cm^3 of FA1 contain 0.4 moles of hydrochloric acid

100cm^3 of FA1 contain $\left(\frac{0.4}{1000} \text{x } 100\right)$ *moles of hydrochloric acid*

$= 0.04$ *moles of hydrochloric acid*

Moles of hydrochloric acid that reacted with barium carbonate = (0.04-0.0258)

$= \mathbf{0.0142}$ ***moles of hydrochloric acid***

b) Determine the:

i) mass of pure barium carbonate that reacted with hydrochloric acid.

(Ba=137, C=12, O=16)

$BaCO_3(aq) + 2HCl(aq) \rightarrow BaCl_2(aq) + H_2O(l) + CO_2(g)$

Moles of pure $BaCO_3$ = $(\frac{1}{2}$ *x moles of HCl)*

$= \left(\frac{1}{2} \text{x } 0.0142\right)$

$= 0.0071$

Molar mass of $BaCO_3$ = [(137x1) + (12x1) +(16x3)]g

=(137 + 12 + 48) g

=197 g

1 mole of barium carbonate weighs 197g

0.0071 moles of barium carbonate weigh (197x0.0071)g

=1.3987g

ii) percentage purity of the barium carbonate sample.

Percentage purity = $\left(\frac{\mathbf{1.3987}}{\mathbf{1.4}} \boldsymbol{x100}\right)\%$

$= \mathbf{99.9}$ **%**

Worked out example 5.2.2

You are provided with the following:

GA1 which was made by dissolving 11.2g of potassium hydroxide in 200cm^3 of solution.

GA3 which is a solution of hydrochloric acid

Solid Z which is impure zinc powder.

You are required to determine the molar concentration of hydrochloric acid in GA3 and the percentage of the impurity in the zinc powder provided.

Theory

Zinc powder reacts with excess hydrochloric acid according to the following equation.

$Zn(s) + 2HCl(aq) \rightarrow ZnCl_2(aq) + H_2(g)$

PART I

Procedure

i) Using a measuring cylinder, transfer 100cm^3 of GA1 into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label the solution GA2.

ii) Pipette 20 or 25cm^{3}of GA2 into a clean conical flask. Add 2-3 drops of Methyl orange indicator and titrate with GA3 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below.

Capacity of pipette used……*25*……..cm^3

Final burette reading (cm^3)	*10.40*	*10.10*	*20.00*
Initial burette reading (cm^3)	*0.00*	*0.00*	*10.00*
Volume of GA3 used (cm^3)	*10.40*	*11.10*	*10.00*

Values used to calculate average……..….*10.10, 10.00*……..cm^3

Average volume of GA3 used……$\left(\frac{\mathbf{10.10 + 10.00}}{\mathbf{2}}\right)$= ***10.05***…….cm^3

Questions:

a) Determine the concentration of:

i) potassium hydroxide in GA2 in moldm^{-3}

Molar mass of KOH = [(39x1) + (16x1) + (1x1)]= ***56g***

56g of potassium hydroxide contain 1 mole

11.2g of potassium hydroxide contain: $\left(\frac{1}{56}\text{x } 11.2\right)$*moles*

200cm^3 of GA1 contain $\left(\frac{11.2}{56}\right)$*moles of potassium hydroxide*

100cm^3 of GA1 contain $\left(\frac{11.2\ x\ 100}{56x200}\right)$*moles of potassium hydroxide*

= 0.1 moles of potassium hydroxide

250cm^3 of GA2 contain 0.1 moles of potassium hydroxide

1000cm^3 of GA2 contain $\left(\frac{0.1\ x\ 1000}{250}\right)$*moles of potassium hydroxide*

= 0.4moldm^{-3}

ii) hydrochloric acid in GA3 in moldm^{-3}.

1000cm^3 of GA2 contain 0.4 moles of potassium hydroxide

25cm^3 of GA2 contain $\left(\frac{0.4\ x\ 25}{1000}\right)$*moles of potassium hydroxide*

KOH(aq) + HCl(aq)→ KCl(aq) + H_2O(l)

Moles of hydrochloric acid = (1xmoles of potassium hydroxide)

$=\left(\frac{1\ x\ 0.4\ x\ 25}{1000}\right)$

$= \left(\frac{0.4\ x\ 25}{1000}\right)$

10.05cm^3 of GA3 contain $\left(\frac{0.4\ x\ 25}{1000}\right)$*moles of hydrochloric acid*

1000cm^3 of GA3 contain $\left(\frac{0.4\ x\ 25}{1000\ \text{x}\ 10.05}\ \text{x}\ 1000\right)$ *moles of hydrochloric acid*

$= 0.995\ moldm^{-3}$

PART II

Procedure

Weigh accurately 1.2 of solid Z and place it in a clean plastic beaker. Add 100cm^3 of GA3 to solid Z and warm gently until the solid dissolves (until effervescence is over). Transfer the resultant solution into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label the solution GA4.
Pipette 20 or 25cm^3 of GA2 into a clean conical flask. Add 2-3 drops of methyl orange indicator and titrate with GA4 from the burette until the end point is reached. Record your results in the table below. Repeat the titration until you obtain consistent results.

Mass of container + Z……..…….21.40……..……..g
Mass of container alone……....20.20……..……..g
Mass of solid Z…….……1.20……..……..g
Capacity of pipette used……..……25…..……..cm^3

Final burette reading (cm^3)	*35.90*	*35.70*	*41.60*
Initial burette reading (cm^3)	*0.00*	*0.00*	*6.00*
Volume of GA4 used (cm^3)	*35.90*	*35.70*	*35.60*

Values used to calculate average……*35.70, 35.60*……cm^3

Average volume of GA4 used……$\left(\frac{\mathbf{35.70 + 35.60}}{\mathbf{2}}\right)$*=35.65*……cm^3

Questions

a) Calculate the:

i)moles of hydrochloric acid in GA4 that reacted with the Potassium hydroxide in GA2.

1000cm^3 of GA2 contain 0.4 moles of potassium hydroxide

25cm^3 of GA2 contain $\left(\frac{0.4}{1000}\text{x}\ 25\right)$ *moles of potassium hydroxide*

$KOH(aq) + HCl(aq) \rightarrow KCl(aq) + H_2O(l)$

Moles of hydrochloric acid = (1xmoles of potassium hydroxide)

$= \left(\frac{1x\ 0.4\ x\ 25}{1000}\right)$

$= \left(\frac{0.4\ x\ 25}{1000}\right)$

ii)moles of hydrochloric acid in GA3 that did not react with the zincmetal.

36.65cm^3 of GA4 contain $\left(\frac{0.4\ x\ 25}{1000}\right)$ *moles of hydrochloric acid*

250cm^3 of GA4 contain $\left(\frac{0.4\ x\ 25}{1000\ \text{x}\ 36.65}\ \text{x}\ 250\right)$ *moles of hydrochloric acid*

$=0.07$ *moles of hydrochloric acid*

iii)moles of hydrochloric acid that reacted with zinc metal.

1000cm^3 of GA3 contain 0.995 moles of hydrochloric acid

100cm^3 of GA3 contain $\left(\frac{0.995}{1000}\ \text{x}\ 100\right)$ *moles of hydrochloric acid*

=0.0995 moles of hydrochloric acid

Moles of hydrochloric acid that reacted with zinc metal = (0.0995-0.07)
= 0.0295moles

b) Determine the mass of zinc that reacted with Hydrochloric acid and hence the percentage of the impurity in the zinc sample provided.(Zn=65)

From the balanced equation,

Moles of zinc $= \frac{1}{2}$ *x moles of hydrochloric acid*

$= \left(\frac{1}{2} \text{ x } 0.0295\right)$

$= 0.01475$

1 mole of Zinc metal weighs 65g

0.01475 moles of Zinc metal weigh: (65x0.01475)g

= 0.959g

≃ 0.96g

Mass of the impurity = (1.20 – 0.96)g

= 0.24g

Percentage of the impurity $= \left(\frac{0.24}{1.20} \text{ x } 100\right)\%$

= 20%

Worked out example 5.2.3

You are provided with the following:

FA1 which is potassium manganate(VII) solution of unknown concentration.

FA2 which isa solution prepared by dissolving 5.4g of a metal persulphate, $X_2S_2O_8$ in $500cm^3$ of solution.

FA3 which is 2M sulphuric acid.

Solid Hwhich is iron(II) sulphateheptahydrate, $FeSO_4.7H_2O$.

You are required to determine the:

(i) molarity of manganate(VII) in FA1.

(ii) relative atomic mass of X in $X_2S_2O_8$.

Theory:

Persulphate ions react with excess iron(II) ions in accordance with the equation below:

$$S_2O_8^{2-}(aq) + 2Fe^{2+}(aq) \rightarrow 2SO_4^{2-}(aq) + 2Fe^{3+}(aq)$$

The unreacted iron(II) ions are titrated with acidified manganate(VII) ions in accordance with the equation below:

$$MnO_4^-(aq) + 5Fe^{2+}(aq) + 8H^+(aq) \rightarrow Mn^{2+}(aq) + 5Fe^{3+}(aq) + 4H_2O(l)$$

Procedure I

Weigh accurately 4.2g of solid H and dissolve it in about $100cm^3$ of distilled water; then transfer the solution into a $250cm^3$volumetric flask and make up to the mark with distilled water. Label the solution FA4.

RESULTS

Mass of container + H……*40.6*……g
Mass of container alone……*36.4*……g
Mass of solid H……*4.2*……g

Pipette 25 (or 20cm^3) of FA4 into a conical flask. Add an equal volume of FA3 and titrate the mixture with FA1 from the burette. Repeat the titration until you obtain consistent results. Record your results in the table below.

Volume of pipette used ……*25.0*……..cm^3

Final burette reading (cm^3)	*15.70*	*17.60*	*15.60*
Initial burette raeding (cm^3)	*0.00*	*2.00*	*0.00*
Volume of FA1 used (cm^3)	*15.70*	*15.60*	*15.60*

Titre value to be used to calculate average volume of FA1
……*15.60, 15.60*……..cm^3
Average volume of FA1
……$\left(\frac{15.60 + 15.60}{2}\right)$=*15.60* ……..cm^3

Questions

Determine the:
(i) number of moles of manganate(VII) ions that reacted with iron(II) ions.

Molar mass of $FeSO_4.7H_2O$ = (56x1)+(32x1)+(16x4)+(18x7) = ***278g***
278g of $FeSO_4.7H_2O$ contain 1 mole
4.2g of $FeSO_4.7H_2O$contain $\left(\frac{1}{278} \text{x } 4.2\right)$*moles*
250cm^{3}of FA4 contain$\left(\frac{4.2}{278}\right)$*moles of$Fe^{2+}$*

25cm^3 of FA4 contain $\left(\frac{4.2 \ x \ 25}{278 \text{ x } 250}\right)$*moles of$Fe^{2+}$*

Moles of MnO_4^- = $\frac{1}{5}$ x $\left(\frac{4.2x25}{278 \text{ x } 250}\right)$ = ***3.02x10^{-4}***

(ii) concentration of manganate(VII) ions in FA1 in mol dm^{-3}.
25.60cm^3 of FA1 contain 3.02x10^{-4}moles of MnO_4^-.
1000cm^3 of FA1 contain $\left(\frac{3.02x10-4}{25.60} \text{ x } 1000\right)$*moles of MnO_4^-.*
= ***0.0118 mol dm^{-3}***

PROCEDURE II

By use of a measuring cylinder, measure and transfer 25cm^3 of FA2 into a 250cm^3 beaker followed by 75cm^3 of FA4. Shake well and label the solution FA5.

Pipette 25 (or 20cm^3) of FA5 into a conical flask. Add an equal volume of FA3. Titrate the mixure with FA1 from the burette. Repeat the titration until you obtain consistent results. Record your results in the table below.

Volume of pipette used……*25.0*……...cm^3

Final burette raeding (cm^3)	*10.60*	*12.50*	*10.50*
Initial burette raeding (cm^3)	*0.00*	*2.00*	*0.00*
Volume of FA1 used (cm^3)	*10.60*	*10.50*	*10.50*

Titre value to be used to calculate average volume of FA1
……*10.50, 10.50* ……...…….cm^3

Average volume of FA1
……$\left(\frac{10.50 + 10.50}{2}\right)$= *10.50*……$cm^3$

Questions
(b) Calculate the number of moles of:
(i) manganate(VII) ions that reacted with the excess Fe^{2+}.

1000cm^3 of FA1 contain 0.0118 moles of MnO_4^-.
10.50 cm^3 of FA1 contain $\left(\frac{0.0118}{1000} \text{ x } 10.50\right)$ *moles of MnO_4^-.*
=1.239x10^{-4} moles of MnO_4^-

(ii) unreacted Fe^{2+} ions in 100cm^3 of FA5.
Moles of Fe^{2+} that reacted with MnO_4^-= (5xmoles of MnO_4^-)
= (5x1.239x10^{-4}) = 6.195 x10^{-4}
25.0cm^3 of FA5 contain 6.195 x10^{-4}moles of Fe^{2+}
100cm^3 of FA5 contain $\left(\frac{6.195\ x10-4}{25.0} \text{ x } 100\right)$ *moles of Fe^{2+}*
= 2.478x10^{-3} moles of Fe^{2+}

(iii) Fe^{2+} ions in FA4 that reacted with persulphate ions in FA2.
250$cm^3$of FA4 contain $\left(\frac{4.2}{278}\right)$ *moles ofFe^{2+}*
75cm^3 of FA4 contain $\left(\frac{4.2}{278 \text{ x } 250} \text{ x } 75\right)$ *moles ofFe^{2+}*
= 4.53x10^{-3}moles ofFe^{2+}
Moles of Fe^{2+}ions in FA4 that reacted with persulphate ions in FA2 = (4.53x10^{-3}—2.478x10^{-3})
= 2.054 x10^{-3} moles of Fe^{2+}ions

(iv) moles of persulphate in 500cm^3 of FA2.
Moles of $S_2O_8^{2-}$ that reactedwith Fe^{2+}= (½ x moles of Fe^{2+}) = (½ x 2.054 x10^{-3})= 1.027x10^{-3}
25cm^3 of FA2 contain 1.027x10^{-3} moles of $S_2O_8^{2-}$.
500cm^3 of FA2 contain $\left(\frac{1.027x10-3}{25} \text{ x } 500\right)$ *moles of $S_2O_8^{2-}$.*
= 0.02054 ≃0.02moles of $S_2O_8^{2-}$

(c) Determine the formula mass of the metal persulphate, $X_2S_2O_8$ and hence determine the relative atomic mass of X.

0.02moles of $X_2S_2O_8$ weigh 5.4g.

1 mole of $X_2S_2O_8$ weighs $\left(\frac{5.4}{0.02}\right)$*g*

= 270g

2x + (32x2) + (16x8) = 270

2x +64+128 = 270

2x = 270—192

2x = 78; $x = \frac{78}{2}$; ***x = 39***

5.3Practical Exercises on Back Titration

Experiment 5.3.1

You are provided with the following:
FA1 which is 0.1Msodium hydroxide
FA2 which is 1M nitric acid
Solid C which is a metal carbonate, PCO_3
You are required to determine the relative atomic mass of the metal P in the metal carbonate, PCO_3.

Procedure

Weigh accurately, 2.6g of C into a clean beaker. Using a measuring cylinder, add 50cm^3 of water and stir to form a paste, followed by 100cm^3 of FA2 and continue to stir until effervescence stops. Transfer the resultant solution into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label the solution FA3.
Pipette 20 or 25cm^3 of FA1 into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with FA3 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below.

Mass of container + C……..……..……..……..g

Mass of container …….…….....g

Mass of solid C……..……..……..g

Capacity of pipette used……...cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA3 used (cm^3)			

Values used to calculate average……..……cm^3

Average volume of FA3 used……..…..……cm^3

Questions

a) Calculate the:

i) number of moles of excess nitric acid in FA3 that reacted with thesodium hydroxide in FA1.

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ii) moles of nitric acid that did not react with PCO_3.

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iv) number of moles of nitric acid that reacted with PCO_3.

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b) Write the equation of reaction between the metal carbonate, PCO_3 and nitric acid.

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c) Determine the:

i)mass of one mole of the metal carbonate, PCO_3.

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ii) relative atomic mass of the metal P in PCO_3.

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Experiment 5.3.2

You are provided with the following:
GA1 which is 0.1M potassium carbonate solution
GA2 which is 0.1M potassium hydroxide solution
GA3 which is approximately 1M sulphuric acid
Solid D which is a metal oxide, XO
You are required to determine the concentration of sulphuric acid in FA3 in moles per litre and the value of X in XO.

Theory

The metal oxide XO reacts with sulphuric acid which is in excess in accordance with the following equation.

$$XO(s) + H_2SO_4(aq) \rightarrow XSO_4(aq) + H_2O(l)$$

The excess, unreacted Sulphuric acid reacts with Potassium hydroxide in accordance with the following equation.

$$2KOH(aq) + H_2SO_4(aq) \rightarrow K_2SO_4(aq) + 2H_2O(aq)$$

PART I

Procedure

(i) By use of a measuring cylinder, measure and transfer 20cm^3 of GA3 into a clean beaker. To the solution in the beaker, add 100cm^3 of distilled water. Label the solutionGA4.
(ii) Pipette 20 or 25cm^3 of GA1 into a clean conical flask. Add 2-3 drops of methyl orange indicator and titrate with GA4 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below.

Capacity of pipette used……..cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of GA4 used (cm^3)			

Values used to calculate average……..……..cm^3

Average volume of GA4 used…….cm^3

Questions

a) Calculate the:

i) number of moles ofsulphuric acid in the 120cm^3 of GA4.

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ii) concentration of sulphuric acid in GA3 in moles per litre.

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PART II

Procedure

(i) Weigh accurately 3.0g of solid D and place it in a clean glass plastic beaker. Add 60cm^3 of GA3 and stir well to dissolve (you may warm gently as you stir to dissolve if necessary).

(ii) Cooland transfer the resultant solution into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label the solution GA5.

Mass of container + D……..…….……..……..g

Mass of container …….…….....g

Mass of solid D……..…….……..g

(iii)Pipette 20 or 25cm^3 of GA2 into a clean conical flask. Add 2-3 drops of methyl orange indicator and titrate with GA5 from the burette until the end point is reached.

(iv)Repeat the titration until you obtain consistent results. Record your results in the table below.

Capacity of pipette used……..cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of GA5 used (cm^3)			

Values used to calculate average……..……cm^3

Average volume of GA5 used……..cm^3

b) Calculate the number of moles of sulphuric acid which:

i) did not react with the metal oxide, XO.

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ii) reacted with the metal oxide, XO.

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c) Determine the:

i) number of moles of the metal oxide, XO that reacted with thesulphuric acid in GA3.

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ii) molar mass of the metal oxide and hence the value of X in XO. (O=16)

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Experiment 5.3.3

You are provided with the following:
HA1 which is a solution of hydrochloric acid whose concentration is approximately 0.5M.
HA2 which was made by dissolving 18.63g of potassium carbonate in water to make 500cm^3 of solution.
HA3 which is 0.1 M potassium hydroxide solution.
Substance Q which is a magnesium ribbon.
You are required to determine the:
i) concentration of hydrochloric acid in HA1 in moldm^{-3}.
ii) percentage purity of the magnesium ribbon provided.
iii) mass of pure magnesium per unit length of the ribbon.
(K=39, C=12, O=16, Mg=24)

Theory

The Magnesium ribbon provided reacts with excess hydrochloric acid according to the following equation.
$Mg(s) + 2HCl(aq) \rightarrow MgCl_2(aq) + H_2(g)$
Potassium reacts with the excess unreacted hydrochloric acid according to the equation below.
$KOH(aq) + HCl(aq) \rightarrow KCl(aq) + H_2O(l)$

Procedure I

Pipette 20 or 25cm^{3}of HA2 into a clean conical flask. Add 2-3 drops of methyl orange indicator and titrate with HA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below.

Capacity of pipette used……..……..cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of HA1 used (cm^3)			

Values used to calculate average……..……cm^3

Average volume of HA1 used……..cm^3

Questions

a) Calculate the number of moles of potassium carbonate in HA2 that reacted with the hydrochloric acid in HA1.

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c) Determine the concentration ofhydrochloric acid in HA1 in moldm^{-3}.

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Procedure II

i) Measure and break off a portion of substance Q which is exactly 10cm long.
ii) Determine the mass of the ten- centimetre-long substance Q.
iii) Break the ten- centimetre-long substance Q into 4 small parts and place them in a clean plastic beaker; then add 100cm^3 of HA1 and stir until the pieces of Q completely dissolve. Transfer the resultant solution into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label this solution HA4.
iv) Pipette 20 or 25cm^3 of HA3 into a clean conical flask. Add 2-3 drops of Methyl orangeindicator and titrate with HA4 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below.

Mass of container + Q…….…….…….……g

Mass of container alone…….....g

Mass of solid Q…….……..….g

Capacity of pipette used……...…….cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of HA4 used (cm^3)			

Values used to calculate average……..…….cm^3

Average volume of HA4 used……cm^3

Questions

c) Calculate the moles of hydrochloric acid:
i) in HA4 thatreacted with the potassium hydroxide in HA3.

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ii) in HA1 that did not react with the magnesium.

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iii) in HA1 that reacted with the magnesium.

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d) Determine the:

i) mass of pure magnesiumthat reacted with the hydrochloric acid in HA1.

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ii) percentage purity of the magnesium ribbon provided.

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iii) mass of pure magnesium per unit length of the ribbon provided

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Experiment 5.3.4

You are provided with the following:
FA1 which is a solution containing 1.18g of manganate(VII) ions in 500cm^3 of solution
FA2 which is a solution of oxalic acid.
FA3 which is 1M sulphuric acid
Solid E which is impure manganese(IV) oxide, MnO_2 referred to as *pyrolusite.*

You are required to determine the:

i) molarity of manganate(VII) ions in FA1
ii) percentage of the impurity in the manganese(IV) oxide sample used.

(Mn=55, O=16)

Theory

The oxalate ions from Oxalic acid react separately with manganate(VII) ions and manganese(IV) oxide in the pyrolusite according to the following equations:

$$2MnO_4^-(aq) + 16H^+(aq) + 5C_2O_4^{2-}(aq) \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) + 10CO_2(g)$$
$$MnO_2(s) + 4H^+(aq) + C_2O_4^{2-}(aq) \rightarrow Mn^{2+}(aq) + 2H_2O(l) + 2CO_2(g)$$

Procedure I

By use of a measuring cylinder, measure and transfer 50cm^3 of FA2 into a clean beaker. Add 75cm^3 of distilled water and label the solution FA4

Pipette 20 or 25cm^3 of FA4 into a clean conical flask. Add an equal volume of FA3 and heat the mixture to a temperature of about 70^{0}C and immediately titrate the hot solution with FA1 from the burette. Repeat the titration until you obtain consistent results. Record your results in the table below.

Capacity of pipette used……cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA1 used(cm^3)			

Values used to calculate average……cm^3

Average volume of FA1used…….....cm^3

a) Calculate the molarity of MnO_4^- in FA1.

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b) Determine the molarity of $C_2O_4^{2-}$ in FA2.

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Procedure II

Weigh accurately, 1.0g of solid E and transfer it into a conical flask. By use of a measuring cylinder, measure and add 150cm^3 of FA2 followed by 50cm^3 of FA3 to the solid in the conical flask.

Boil the mixture gently for about 5 to 6 minutes (until the remaining solid particles turn brown). Cool the mixture and transfer it into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label the solution FA5.

Measure and transfer 50cm^3 of FA1 and transfer into a clean beaker. Add 50cm^3 of distilled water and label this solution FA6.
Pipette 20 or 25cm^3 of FA5 into a clean conical flask. Add an equal volume of FA3 by use of a measuring cylinder and heat the mixture to about 70^{0}C and immediately titrate the hot solution with FA6 from the burette. Repeat the titration until you obtain consistent results. Record your results in the table below.

Mass of container + E……..……..……..……..g
Mass of container alone…….....g
Mass of solid E……..……..g

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA6 used(cm^3)			

Values used to calculate average……cm^3

Average volume of FA6 used……cm^3

c) Calculate the number of moles of:

i) MnO_4^- in FA6 that reacted with$C_2O_4^{2-}$ in FA5.

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ii) $C_2O_4^{2-}$ in FA5 that reacted with MnO_4^- in FA6.

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iii) $C_2O_4^{2-}$ in FA2 that reacted with the MnO_2.

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iv) MnO_2 that reacted with the $C_2O_4^{2-}$ in FA2.

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v) Determine the percentage of the impurity in the sample of manganese(IV) oxide, MnO_2 used.

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Experiment5.3.5

You are provided with the following:
GA1 which is iodine solution
GA2 which is a solution containing 9.3g of hydratedsodium thiosulphate, $Na_2S_2O_3.5H_2O$ in $500cm^3$.
Solid P isanhydrous sodium sulphite, Na_2SO_3.

You are required to determine the:

i) molarity of iodine in GA1.

ii) percentage impurity of the sodium sulphite sample.

Theory

Sulphite ions are oxidised by iodine to sulphate ions according to the equation below:

$$SO_3^{2-}(aq) + H_2O(l) + I_2(aq) \rightarrow SO_4^{2-}(aq) + 2H^+(aq) + 2I^-(aq) \text{ .....(i)}$$

Since the hydrogen ions produced by the reaction above are capable of reacting with thiosulphate ions resulting in precipitation of sulphur, before titration with standard sodium thiosulphate, some sodium hydrogencarbonate(about 2g) is added to the solution in the conical flask so as to **remove all the hydrogen ions from the solution** as shown by the reaction below.

$$2HCO_3^-(aq) + 2H^+(aq) \rightarrow 2CO_2(aq) + 2H_2O(l) \text{ .....(ii)}$$

When equations (i) and (ii) above are combined, the overall equation below is obtained.

$$SO_3^{2-}(aq) + I_2(aq) + 2HCO_3^-(aq) \rightarrow SO_4^{2-}(aq) + 2I^-(aq) + 2CO_2(aq) + H_2O(l)$$

Hence sulphite ions are oxidized by aqueous Iodine to sulphate ions in the presence of hydrogencarbonate ions according to the aboveoverall equation.

Procedure I

Pipette $10cm^3$ of GA1 into a clean conical flask and titrate the solution with GA2 until the solution becomes pale yellow; then add 5 drops of Starch indicator and continue the titration until the blue-black starch-iodine complex just turns colourless. Repeat the titration until you obtain consistent results. Record your results in table I below.

Capacity of pipette used……cm^3

Table I

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of GA2 used(cm^3)			

Values used to calculate average…….....cm^3

Average volume of GA2 used…….....cm^3

Questions

a) Write the equation of reaction between iodine and thiosulphate ions.

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b) Calculate the molarity of the:

i) sodium thiosulphate in GA2

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ii) iodine in GA1

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Procedure II

i) Weigh accurately, 1.0g of P into a clean beaker. Add about 100cm^3 of water and stir well to dissolve. Transfer the solution into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label the resultant solution GA3.

ii) Using a glass measuring cylinder, measure and transfer 70cm^3 of GA1 into a clean conical flask. While shaking the conical flask and its contents, by use of another measuring cylinder add slowly, 30cm^3 of GA3 followed by 2.0g of sodium hydrogencarbonate and shake well to dissolve. Label the resultant solution GA4.

iii) Pipette 20 or 25cm^3 of GA4 into a clean conical flask and titrate with GA2 until the solution turns pale yellow, then add 1cm^3 of Starch indicator and continue the titration until the blue-black complex just turns colourless. Repeat the titration until you obtain consistent results. Record your results in table II below.

Mass of container + P……..……..……..……..g

Mass of container…….....g

Mass of solid P……..……..g

Table II

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of GA2 used(cm^3)			

Values used to calculate average…….....cm^3

Average volume of GA2 used…….....cm^3

b) Calculate the number of moles of iodine in GA1 that reacted with sulphite ions in GA3.

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c) Determine the mass of pure sodium sulphite in the:

i) 30cm^3 of GA3 which reacted with iodine in GA1.(Na=23, S=32, O=16)

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ii) 250cm^3 of GA3 and hence the percentage impurity of the sodium sulphite sample used in the preparation of GA3.

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Experiment 5.3.6

You are provided with the following:
FA1 which is a solution of iodine.
FA2 which is a solution containing19.84g of hydrated sodium thiosulphate, $Na_2S_2O_3.5H_2O$ in one litre.
Solid R which is a reducing agent.
Solid S which is sodium hydrogencarbonate.
You are required to determine the:
i) molarity of iodine in FA1
ii) mole ratio for the reaction between iodine and solid R.

Theory

Iodine reacts with thiosulphate ions according to the equation below:

$$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow S_4O_6^{2-}(aq) + 2I^-(aq)$$

Procedure I

Pipette 10cm^3 of FA1 into a clean conical flask and titrate the solution with FA2 until the solution becomes pale yellow; then add 5 drops of starch indicator and continue the titration until the blue-black starch-iodine complex just turns colourless. Repeat the titration until you obtain consistent results. Record your results in table I below.

Capacity of pipette used……...cm^3

Table I

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA2 used(cm^3)			

Values used to calculate average……cm^3

Average volume of FA2 used……cm^3

Questions

a) Calculate the number of moles of thiosulphate ions in FA2 that reacted with iodine in FA1.

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b) Determine the concentration of iodine in FA1 in $moldm^{-3}$.

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Procedure II

i) Weigh accurately, 1.5g of R into a clean beaker. Add about $100cm^3$ of water and stir well to dissolve. Transfer the solution into a $250cm^3$ volumetric flask and make up to the mark with distilled water. Label the resultant solution FA3.

ii) Using a measuring cylinder, measure and transfer $10cm^3$ of FA3 into a clean conical flask. Add $25cm^3$ of FA1 followed by 2.0g of solid S and shake to mix well. Titrate the excess iodine in the mixture with FA2 until the solution turns pale yellow; then add $1cm^3$ of starch indicator and continue the titration until the blue-black starch-iodinecomplex just turns colourless. Repeat the titration until you obtain consistent results. Record your results in table II below.

Mass of container + R……..……..……..……..g

Mass of container…….…….....g

Mass of solid R……..……..g

Table II

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA2 used(cm^3)			

Values used to calculate average…….....cm^3

Average volume of FA2 used…….cm^3

c) Calculate the number of moles of:

i)excess iodine from FA1 that reacted with thiosulphate ions from FA2.

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ii) iodine that reacted with solid R.

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d) Determine the:

i)number of moles of R that reacted with iodine (molar mass of R is 126g).

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ii) mole ratio of reaction between iodine and R.

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Experiment 5.3.7

You are provided with the following:
GA1 which isa solution containing manganate(VII) ions.
GA2 which isa solution containing 2.64g of an impure metal persulphate, $M_2S_2O_8$ in 200cm^3 of solution.
GA3 which is 2.0M sulphuric acid.
Solid Pwhich isdiammoniumiron(II) sulphate hexahydrate, $(NH_4)_2SO_4FeSO_4.6H_2O$.
You are required to determine the:
(i) molar concentration of manganate(VII) ions in GA1.
(ii) percentage purity of the sample of the metal persulphate, $M_2S_2O_8$.

Theory:
Persulphate ions react with excess iron(II) ions according to the equation below:

$$S_2O_8^{2-}(aq) + 2Fe^{2+}(aq) \rightarrow 2SO_4^{2-}(aq) + 2Fe^{3+}(aq)$$

The unreacted iron(II) ions are titrated with acidified manganate(VII) ions according to the equation below:

$$MnO_4^-(aq) + 5Fe^{2+}(aq) + 8H^+(aq) \rightarrow Mn^{2+}(aq) + 5Fe^{3+}(aq) + 4H_2O(l)$$

Procedure I
Weigh accurately 6.3g of solid P and dissolve it in about 100cm^3 of distilled water. Transfer the solution into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label the solution GA4.

RESULTS
Mass of container + P……..……g
Mass of container alone……..……g
Mass of solid P……g

Pipette 25 (or 20cm^3) of GA4 into a conical flask. Add an equal volume of GA3 and titrate the mixture with GA1 from the burette. Repeat the titration until you obtain consistent results. Record your results in the table below.

Volume of pipette used ……..cm^3

Final burette reading (cm^3)			
Initial burette raeding (cm^3)			
Volume of GA1 used (cm^3)			

Titre value to be used to calculate average volume of GA1……..……..cm^3

Average volume of GA1
…….……..cm^3

Questions

Determine the molar concentration of:

(i) Fe^{2+}ions in GA4.

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(ii) manganate(VII) ions in GA1.

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PROCEDURE II

i) Using a measuring cylinder, measure and transfer 15cm^3 of GA2 into a 250cm^3 beaker followed by 85cm^3 of GA4. Shake well and label the solution GA5.

ii) Pipette 25 (or 20cm^3) of GA5 into a conical flask. Add an equal volume of GA3. Titrate the mixure with GA1 from the burette. Repeat the titration until you obtain consistent results. Record your results in the table below.

Volume of pipette used……..……...cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of GA1 used (cm^3)			

Titre value to be used to calculate average volume of GA1…….……cm^3

Average volume of GA1
……..……...cm^3

Questions

(b) Calculate the number of moles of:

(i) manganate(VII) ions that reacted with the excess Fe^{2+} in GA5.

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(ii) Fe^{2+} ions which did not react with the persulphate ions.

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(iii) Fe^{2+} ions in FA4 that reacted with persulphate ions in GA2.

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(iv) moles of the persulphate ions in $200cm^3$ of GA2.

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(c) Determine the mass of the metal persulphate, $M_2S_2O_8$in $200cm^3$ of GA2and hence calculate the percentage purity of the metal persulphate. (M=39, S=32, O=16)

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CHAPTER SIX
6.0 SOLUBILITY PRODUCT

6.1 Introduction

Solubility product is the product of the molar concentrations of the constituent ions of a sparingly soluble salt or electrolyte that existin its saturated solution raised to their respective powers at a fixed temperature.

A sparingly soluble salt is one which dissolves up to a certain extent beyond which it cannot dissolve. At this point, equilibrium is established between the undissolved salt and the ions formed by the portion of the salt that dissolved.

In general, for any sparingly soluble ionic salt expressed as M_aX_b, its equation of solubility is expressed as shown below:

$$M_aX_b(s)+(aq) \rightleftharpoons aM^{b+}(aq) + bX^{a-}(aq)$$

OR: $M_aX_b(s) \rightleftharpoons aM^{b+}(aq) + bX^{a-}(aq)$

The expression for the solubility product is:

$$K_{sp} = [M^{b+}]^a[X^{a-}]^b$$

For silver chloride, its equation of solubility is:

$$AgCl(s) + (aq) \rightleftharpoons Ag^+(aq) + Cl^-(aq)$$

Its expression for the solubility productis:

$$K_{sp} = [Ag^+][Cl^-]$$

For lead(II) chloride, its equation of solubility is:

$$PbCl_2(s) + (aq) \rightleftharpoons Pb^{2+}(aq) + 2Cl^-(aq)$$

Its expression for the solubility productis:

$$K_{sp} = [Pb^{2+}][Cl^-]^2$$

6.2 Worked out examples on the Solubility Product

Workedout example 6.2.1

You are provided with the following:

FA1 which is 0.08M hydrochloric acid

Solid M which is calcium hydroxide

You are required to determine the solubility product, K_{sp} of calcium hydroxide at room temperature.

Theory

Calcim hydroxide is sparingly soluble in water as shown by the following equation:

$$Ca(OH)_2(s) \rightleftharpoons Ca^{2+}(aq) + 2OH^-(aq)$$

But Solubility of $Ca(OH)_2 = [Ca^{2+}] = ½ [OH^-]$

$K_{sp} = [Ca^{2+}][OH^-]^2$

When the mixture is titrated with an appropriate acid such as hydrochloric acid, the concentration of hydroxyl ions, OH^- can be determined basing on the following equation.

$OH^-(aq) + HCl(aq) \rightarrow H_2O(l) + Cl^-(aq)$

Ionic equation

$OH^-(aq) + H^+(aq) \rightarrow H_2O(l)$

Procedure

i) Weigh accurately 2.3g of solid M from a piece of paper and transfer it carefully into a 250cm^3 volumetric flask. Add 170cm^3 of distilled water. Stopper/cork the flask immediately and shake vigorously for 8-10 minutes and then filter the mixture into a clean conical flask.

ii) Pipette 20 or 25cm^3 of the filtrate into another clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with FA1 until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below.

Capacity of pipette used……25……..cm^3

Final burette reading (cm^3)	*13.90*	*13.70*	*21.60*
Initial burette reading (cm^3)	*0.00*	*0.00*	*8.00*
Volume of FA1 used (cm^3)	*13.90*	*13.70*	*13.60*

Values used to calculate average……..... *13.70, 13.60.. ……cm^3*

Average volume of FA1 used……...$\left(\frac{13.70 + 13.60}{2}\right)$= ***13.65***…..……..*cm^3*

Questions

a) Calculate the:

i) number of moles of hydrochloric acid in FA1 that reacted.

1000cm^{3}of FA1 contain 0.08 moles of hydrochloric acid

13.65cm^{3}of FA1 contain $\left(\frac{0.08}{1000} \times 13.65\right)$ *moles of hydrochloric acid*

=0.001092 moles of hydrochloric acid

ii) concentration of hydroxyl ions in moldm^{-3}.

Moles of hydroxyl ions =(1xmoles of the acid)

= (1x0.001092)

= 0.001092

25cm^{3}of the filtrate contained 0.001092 moles of hydroxyl ions

1000cm^{3}of the filtrate contain $\left(\frac{0.001092}{25} \times 1000\right)$ *moles of hydroxyl ions.*

=***0.0437moldm^{-3}***

b) Determine the:

i) solubility of calcium hydroxide in moldm^{-3} (Ca = 40,O=16, H=1).

Solubility of $Ca(OH)_2 = [Ca^{2+}] = ½ [OH^-]$

*= (½ x 0.0437)***= 0.022moldm^{-3}**

ii) solubility product, K_{sp} of calcium hydroxide

$K_{sp} = [Ca^{2+}][OH^-]^2$

$= [0.022 moldm^{-3} x (0.0437 moldm^{-3})^2] = \mathbf{4.2x10^{-5} mol^3 dm^{-9}}$

c) Determine the percentage of calcium hydroxide that dissolved in water.

(Ca=40, O=16, H=1)

Molar Mass of $Ca(OH)_2 = [(40x1)+(16x2)+(1x2)]g$

$= (40+32+2)g$

$= \mathbf{74g}$

1 mole of $Ca(OH)_2$ *weighs 74g*

0.022 moles of $Ca(OH)_2$ *weighs* $(74x0.022)g$

$= 1.628g$

Percentage of $Ca(OH)_2$ *that dissolved* $\left(\frac{1.628}{2.3} \text{ x } 100\right)\%$

$= \mathbf{70.78\%}$

6.3 Practical Exercises on the Solubility Product

Experiment 6.3.1

You are provided with the following:

FA1 which is 0.05M nitric acid

Solid H which is a metal hydroxide, $M(OH)_2$

You are required to determine the percentage of the metal hydroxide that dissolved in water and its solubility product, K_{sp} at room temperature.

Theory

The metal hydroxide is sparingly soluble in water as shown by the following equation:

$M(OH)_2(s) \rightleftharpoons M^{2+}(aq) + 2OH^-(aq)$

But solubility of $M(OH)_2 = [M^{2+}] = \frac{1}{2}[OH^-]$

$\therefore Ksp = [M^{2+}][OH^-]^2$

When the mixture is titrated with an appropriate acid such as nitric acid, the concentration of hydroxyl ions, OH^- can be determined basing on the following equation.

$OH^-(aq) + HNO_3(aq) \rightarrow H_2O(l) + NO_3^-(aq)$

Procedure

i) Weigh accurately 2.5g of solid H and transfer it carefully into a 250cm^3 volumetric flask. Add 170cm^3 of distilled water. Stopper/cork the flask immediately and shake vigorously for 8—10 minutes and then filter the mixture into a clean conical flask.

ii) Pipette 20 or 25cm^3 of the filtrate into another clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with FA1 until the end point is reached. Repeat the titration until you obtain consistent tresults. Record your results in the table below.

Capacity of pipette used……..cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA1 used (cm^3)			

Values used to calculate average……..……...cm^3

Average volume of FA1 used……..…….……cm^3

Questions

a) Write an ionic equation for the reaction between nitric acid and the hydroxyl ions in the pipetted filtrate.

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b) Calculate the:

i) number of moles of nitric acid in FA1 that reacted.

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ii) concentration of hydroxyl ions in $moldm^{-3}$.

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c) Determine the:

i) solubility of the metal hydroxide in $moldm^{-3}$

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ii) percentage of the metal hydroxide that dissolved in water (M=40, O=16, H=1).

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iii) Solubility product, K_{sp}ofthe metal hydroxide, $M(OH)_2$. (Indicate its units).

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Experiment 6.3.2
You are provided with the following:
GA1 is 0.4M potassium iodide solution
GA2 which is a solution containing 14.9g of sodium thiosulphate pentahydrate, $Na_2S_2O_3.5H_2O$ dissolved in water to make $500cm^3$ of solution.
GA3 which is 2M sulphuric acid
Solid W which is calcium iodate, $Ca(IO_3)_2$.
You are required to determine the percentage of calcium iodate that dissolved in water and the solubility of the salt at room temperature.

Theory
Calcim iodate is sparingly soluble in water as shown by the following equation:

$$Ca(IO_3)_2(s) \rightleftharpoons Ca^{2+}(aq) + 2IO_3^-(aq)$$

But Solubility of $Ca(IO_3)_2 = [Ca^{2+}] = \frac{1}{2}[IO_3^-]$

$K_{sp} = [Ca^{2+}][IO_3^-]^2$
$K_{sp} = \frac{1}{2}[IO_3^-][IO_3^-]^2$
The iodate ions in the presence of an acid, oxidize iodide ions to aqueous iodine according to the equation below.

$$IO_3^-(aq) + 6H^+(aq) + 5I^-(aq) \rightarrow 3I_2(aq) + 3H_2O(l)$$

The aqueous iodine formed is then titrated with a standard solution of thiosulphate ions.

$$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$$

Procedure

i) Weigh accurately 1.2g of solid W and transfer it carefully into a 250cm^3 volumetric flask. Add 150cm^3 of distilled water and shake well to mix; then add more distilled water and make up to the mark. Label the solution GA4.

ii) Using a measuring cylinder, measure and transfer 15cm^3 of GA4 into a clean conical flask. Add 15cm^3 of GA1 followed by 15cm^{3}of GA3 and shake well to mix and titrate the mixture with GA2 until the solution turns pale yellow. Add 1cm^3 of starch indicator and continue the titration until the blue-black starch-iodine complex just turns colourless. Repeat the titration until you obtain consistent results. Record your results in the table below.

Mass of beaker + solid W…….g

Mass of beaker alone…….g

Mass of solid W…….g

Volume of pipette used…….cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of GA2 used (cm^3)			

Values used to calculate average……..……cm^3

Average volume of GA2 used……..…….cm^3

Questions

a) Calculate the molar concentration of:

i) $S_2O_3^{2-}$ in GA2 (Na=23, S=32, O=16, H=1).

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ii) IO_3^- in GA4.

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b) Determine the:

i) solubility of calcium iodate in moldm^{-3}.

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ii) percentage of calcium iodate that dissolved in water.(Ca= 40, I=127, O=16).

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CHAPTER SEVEN
7.0 THE PARTITION COEFFICIENT (DISTRIBUTION CONSTANT)

7.1 Introduction

The partition coefficientis the ratio of the equilibrium concentration of a solute in one solvent to the equilibrium concentration of the same solute in another solvent provided the two solvents are immiscible and the solute remains in the same molecular state in both solvents at a particular temperature.

Partition coefficient, $K_D = \frac{[\text{solute}]_{\text{solvent 1}}}{[\text{solute}]_{\text{solvent 2}}}$

Note: The partition coefficient, K_D derives its name from the Partition Law (Distribution Law).
The Partition Law (Distribution Law) states that:
At constant temperature, a solute distributes itself between two immiscible liquids which are in contact in such a way that the ratio of the concentration of the solute in the two liquids is constant at equilibrium provided the solute remains in the same molecular state.

The Partition Law has got some limitations and they include the following:

- The temperature must be kept constant.
- The solute must be miscible with both solvents.
- The solute must not associate or dissociate in the two solvents.
- The solute should not react with either solvent.
- The two solvents must be immiscible.
- The solutions used should be fairly dilute (the solute should not saturate either solvent).

7.2 Worked out examples on the Partition Coefficient

Worked out example 7.2.1
You are provided with the following:
GA1 which is trichloromethane
GA2 which is ammonia solution
GA3 which is 0.5M hydrochloric acid
GA4 which is 0.05M hydrochloric acid
Distilled water
You are required to determine the partition coefficient of ammonia between water and trichloromethane.

Procedure

i) Using a thermometer, measure and record the room temperature.
ii) Using a measuring cylinder, measure and transfer $60cm^3$ of GA1 into a clean conical flask. Add $15cm^3$ of GA2. To this mixture, add $100cm^3$ of distilled water and shake vigorously for 4 minutes. Allow the mixture to stand for about 7 minutes.
iii) Decant the aqueous layer (upper layer) carefully into a small beaker (or boiling tube) and then cover.

iv) Transfer the organic layer (lower layer) carefully into another small beaker (or boiling tube) and then cover.

v) Pipette 25cm^3 of the aqueous layer using a **pipette filleror suction pump** into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with GA3. Repeat the titration until you obtain consistent results. Record your results in **Table 1** below.

Room temperature……...*24.0*……..^{0}C

Volume of pipette used for aqueous layer……*25.0*……cm^3

Table 1

Final burette reading (cm^3)	*23.00*	*22.80*	*32.70*
Initial burette reading (cm^3)	*0.00*	*0.00*	*10.00*
Volume of GA3 used (cm^3)	*23.00*	*22.80*	*22.70*

Values used to calculate average……*22.80, 22.70*…..……cm^3

Average volume of GA3 used……$\left(\frac{\mathbf{22.80 + 22.70}}{\mathbf{2}}\right)$= ***22.75***……cm^3

vi) Pipette 25cm^3 of the organic layer using a **pipette filleror suction pump** into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with GA4. Repeat the titration until you obtain consistent results. Record your results in **Table 2** below.

Volume of pipette used for organic layer……*25*……cm^3

Table 2

Final burette reading (cm^3)	*9.50*	*9.30*	*15.20*
Initial burette reading (cm^3)	*0.00*	*0.00*	*6.00*
Volume of GA4 used (cm^3)	*9.50*	*9.30*	*9.20*

Values used to calculate average……*9.30, 9.20*……..cm^3

Average volume of GA4 used……$\left(\frac{\mathbf{9.30 + 9.20}}{\mathbf{2}}\right)$= ***9.25..***……cm^3

Questions

a) Calculate concentration of ammonia in the:

i) aqueous layer in moldm^{-3}.

1000cm^3 of GA3 contain 0.5 moles of hydrochloric acid.

22.75cm^{3}of GA3 contain $\left(\frac{0.5}{1000} \times 22.75\right)$ *moles of hydrochloric acid*

= 0.011375moles of hydrochloric acid

$HCl(aq) + NH_3(aq) \rightarrow NH_4Cl(aq)$

OR $HCl(aq) + NH_4OH(aq) \rightarrow NH_4Cl(aq) + H_2O(l)$

Moles of ammonia = (1xmoles of hydrochloric acid)

= (1x0.011375)

= 0.011375molesof ammonia

25cm^{3}of the aqueous layer contain 0.011375 moles of ammonia

1000cm^3 of the aqueous layer contain $\left(\frac{0.011375}{25} \times 1000\right)$ *moles of ammonia*

= 0.455moldm^{-3}

ii) organic layer in moldm^{-3}.

1000cm^3 of GA4 contain 0.05 moles of hydrochloric acid.

9.25cm^{3}of GA4 contain $\left(\frac{0.05}{1000} \text{ x } 9.25\right)$ *moles of hydrochloric acid*

= 0.0004625moles of hydrochloric acid

Moles of ammonia = (1xmoles of hydrochloric acid)

= (1x0.0004625)

= 0.0004625

25cm^{3}of the organic layer contain 0.0004625moles of ammonia

1000cm^3 of the organic layer contain $\left(\frac{0.0004625}{25} \text{ x } 1000\right)$ *moles of ammonia*

= 0.0185moldm^{-3}

b) Determine the value of the partition coefficient between water and trichloromethane at the specified temperature.

$$K_D = \frac{[NH_3]_{Water}}{[NH_3]_{Trichloromethane}}$$

$$= \frac{0.455 \text{ mol dm}^{-3}}{0.0185 \text{ mol dm}^{-3}}$$

= 24.59

7.3 Practical Exercises on the Partition Coefficient

Experiment 7.3.1

You are provided with the following:
HA1 which is trichloromethane
HA2 which is ammonia solution
HA3 which is 0.5M hydrochloric acid
HA4 which is 0.05M hydrochloric acid
Distilled water

You are required to determine the partition coefficient of ammonia between trichloromethane and water.

Procedure

i) Using a thermometer, measure and record the room temperature in the space provided below.
ii)Using a measuring cylinder, measure and transfer 50cm^3 of HA1 into a clean conical flask. Add 25cm^3 of HA2. To this mixture, add 100cm^3 of distilled water and shake vigorously for 4 minutes. Allow the mixture to stand for 7 minutes.
iii) Decant the aqueous layer (upper layer) carefully into a small beaker (or boiling tube) and cover.
iii) Transfer the organic layer (lower layer) carefully into another small beaker (or boiling tube) and cover.
iv) Pipette 25cm^3 of the aqueous layer using a **pipette filleror suction pump** into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with HA3. Repeat the titration until you obtain consistent results. Record your results in **Table 1** below.

Room temperature……..…….……...^{0}C

Volume of pipette used for the aqueous layer…..……..……..cm^3

Table 1

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of HA3 used (cm^3)			

Values used to calculate average……..……cm^3

Average volume of HA3 used……..cm^3

v) Pipette 20 or 25cm^3 of the organic layer using a **pipette filleror suction pump** into a clean conical flask. Add 2-3 drops of phenolphthalein indicator and titrate with HA4. Repeat the titration until you obtain consistent results. Record your results in **Table 2** below.

Volume of pipette usedfor organic layer……cm^3

Table 2

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of HA4 used (cm^3)			

Values used to calculate average……..……cm^3

Average volume of HA4 used……cm^3

Questions

a) Calculate concentration of ammonia in the:

i) aqueous layer in moldm^{-3}.

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ii) organic layer in moldm^{-3}.

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c) Determine the value of the partition coefficient between trichloromethane and water at the specified temperature.

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Experiment 7.3.2

You are provided with the following:

GA1 which is aqueous iodine.

GA2 which is a solution containing 6.2g of sodium thiosulphate pentahydrate, $Na_2S_2O_3.5H_2O$ per litre of solution.

GA3 which is Trichloromethane

You are required to determine the distribution coefficient of iodine between trichloromethaneand water.

Theory

Iodine dissolves in both the aqueous solution of GA1 and in Trichloromethane in GA3. Consequently, when GA1 is shaken with a given volume of GA3, the iodine distributes itself between the two solutions. When the mixture is allowed to stand, two distinct layers are formed whereby the upper layer is a solution of iodine in tetrachloromethane while the lower layer is a solution of iodine in the aqueous layer of GA1.

The concentration of iodine solution in each of the layers is determined by titrating each of the two layers against the standard solution of thiosulphate ions.

Procedure

i) Using a measuring cylinder, measureand carefully transfer $40cm^3$ of GA1 into a clean conical flask. Using another measuring cylinder, measure $40cm^3$ of GA3 and add it to the 40 cm^3 of GA1 in the conical flask. Label this as **flask 1** and stopper it immediately. Shake the mixture and leave it to settle for 5 minutes.

ii) By use of a measuring cylinder, still, measure carefully and transfer $20cm^3$ of GA1 into anotherconical flask. Again, by use of another measuring cylinder, add $40cm^3$ of GA3 to the 20 cm^3 of GA1 in the conical flask. Label this as **flask 2** and stopper it immediately. Shake the mixture and leave it to settle for 5 minutes.

iii) From **flask 1,** carefully pipette 10cm^3 of the upper layer **using a suction pump or pipette filler** into a clean conical flaskand titrate this portion against GA2 fromthe burette using **starch indicator** while continuously shaking the flask in the process of titration. Record the volume of GA2 used in the table below.

iv)Repeat procedure (iii) above, but this time while dealing with the lower layer of **flask 1.**

v) From **flask 2,** using a different pipette, carefully pipette 10cm^3 of the upper layer **using a suction pump** or **pipette filler** into a clean conical flaskand titrate this portion against GA2 fromthe burette using **starch indicator** while continuously shaking the flask in the process of titration. Record the volume of GA2 used in the table below.

vi) Repeat procedure (v) above, but this time while dealing with the lower layer of **flask 2.**

Note: *If you have no suction pump or pipette filler, you can carefully pour off the lower aqueous layer into a measuring cylinder to measure the required volume. Theupper organic layer (purple in colour) will remain floating within the conical flask and can also be carefully poured off as well into a measuring cylinder in order to measure off the required volume. However, thisapproach is inaccurate.*

Flask Number	**Flask 1**		**Flask 2**	
Layer	Upper layer	Lower layer	Upper layer	Lower layer
Final burette reading (cm^3)				
Initial burette reading (cm^3)				
Volume of GA2 used (cm^3)				

Questions

1) (a) Determine the concentration of $S_2O_3^{2-}$ in GA2 in moldm^{-3}.(Na=23, S=32, O=16, H=1)

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b)Write an ionic equation for the reaction between iodine and thiosulphate ions.

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2) (a) Flask1

Calculate the:

i) concentration of iodine in the upper layer (trichloromethane layer).

……

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……

……

ii) concentration of iodine in the lower layer (aqueous layer).

……

……

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……

iii) distribution coefficient, K_D of iodine between trichloromethane and water for **flask1.**

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(b) Flask2

Calculate the:

i) concentration of iodine in the upper layer (trichloromethane layer)

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ii) concentration of iodine in the lower layer (aqueous layer)

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……

……

iii) distribution coefficient, K_D of iodine between trichloromethane and water for **flask 2.**

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3) Basing on the values of $K_D$obtained for both 2(a) (iii) and 2(b) (iii) above, what conclusion do you make?

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CHAPTER EIGHT

8.0 INORGANIC QUALITATIVE ANALYSIS

8.1INTRODUCTION

Inorganic Qualitative Analysis is a branch of practical chemistry which deals with the identification of cations and anions present in a variety of inorganic compounds. At Advanced Level, Inorganic Qualitative Analysis deals with quite a large number of cations and anions.

8.1.1Cations

Cations are positively charged ions. They fall in two major categories: Non-transition metal ions (non-coloured cations) and Transition metal ions (coloured cations). Non-transition metal ions form white compounds and colourless solutions while Transition metal ions form coloured compounds and coloured solutions.

a) Non-transition metal ions (Non-coloured cations)

At this Level, the non-coloured cations dealt with include the following:
Zinc ions, Zn^{2+}
Lead(II) ions, Pb^{2+}
Aluminium ions, Al^{3+}
Ammonium ions, NH_4^+
Calcium ions, Ca^{2+}
Magnesium ions, Mg^{2+}
Barium ions, Ba^{2+}
Note: Others may include:
Tin(II) ions, Sn^{2+}
Tin(IV) ions, Sn^{4+} and Silver ions, Ag^+.

b) Transition metal ions (Coloured Cations)

At this level, the coloured cations dealt with include the following:
Copper(II) ions, Cu^{2+}
Iron(II) ions, Fe^{2+}
Nickel(II) ions, Ni^{2+}
Chromium(III) ions, Cr^{3+}
Iron(III) ions, Fe^{3+}
Manganese(II) ions, Mn^{2+}
Cobalt(II) ions, Co^{2+}

8.1.2Anions

Anions are negatively charged ions. The Anions dealt with at this level include:
Carbonate ions, CO_3^{2-} and Hydrogencarbonate ions, HCO_3^-
Sulphate ions, SO_4^{2-}
Sulphite ions,SO_3^{2-}
Thiosulphate ions, $S_2O_3^{2-}$
Oxalate ions, $C_2O_4^{2-}$
Chloride ions, Cl^-
Iodide ions, I^-

Bromide ions, Br^-
Acetate ions/ethanoate ions, CH_3COO^-
Nitrate ions, NO_3^-
Nitrite ions, NO_2^-
Phosphate ions, PO_4^{3-}
Chromate ions, CrO_4^{2-}
Note: Others include:
Chloric(I) ions/Hypochlorite ions, ClO^-/OCl^-
Dichromate ions, $Cr_2O_7^{2-}$

8.2PRELIMINARY AND CONFIRMATORY TESTS

Preliminary tests are tests that give us a clue/hint on the nature of the solid and on which ions are possibly present (suspected to be present) in the sample under analysis.
Confirmatory tests are tests that prove to us beyond reasonable doubt that a given ion is actually present in the sample under analysis.
Note: Inorganic Qualitative Analysis is carried out basing on preliminary and confirmatory tests of both cations and anions.

8.2.1Physical appearance of solids

The physical appearance of a solid concerns the colour and texture of the solid. The texture of a solid concerns whether the solid is crystalline or powdered/powdery. In terms of colour, a solid may appearwhite, green, blue, pink/red, brown, yellow or orange, purple, violet, e.t.c. If the solid is white, then it contains non-transition metal ions. However, if a solid is coloured, then it contains transition metal ions whereby the specific colour of the solid points to which specific transition metal ion is possibly present. The colour of a solid, therefore, gives an overview of which cations are suspected to be present in that solid. The table below is a summary of cations that should be suspected for specific colours of solids.

Colour of solid	Deduction
White	Zn^{2+}, Pb^{2+}, Al^{3+}, Ca^{2+}, Mg^{2+}, Ba^{2+}, NH_4^+, Sn^{2+}, Sn^{4+}
Green	Fe^{2+}, Cu^{2+}, Ni^{2+}, Cr^{3+}
Blue	Cu^{2+}
Brown	Fe^{2+}, Fe^{3+}
Yellow or Orange	CrO_4^{2-} or $Cr_2O_7^{2-}$ i.e. Cr^{6+} **or** PbO(s) hence Pb^{2+}
Deep pink or Red	Co^{2+}
Very pale pink	Mn^{2+}
Purple	MnO_4^- i.e. Mn^{7+}
Violet	Cr^{3+}
Black	CuO(s), FeO, NiO(s) **or** CoO(s) hence Cu^{2+}, Fe^{2+}, Ni^{2+} **or** Co^{2+}

Note: 1) In case a solid has got a crystalline texture, the expectation is that it has got water of crystallisation and therefore when such a solid is heated, water is given off.
2)While making the deduction, in relation to the physical appearance of a solid, thededuction should be based on mainly the colour of the solid and therefore the main point is the type of cations present which actually influence the colour of the solid.

8.2.2Soluble and Insoluble Salts

Solubility of salts in inorganic qualitative analysis is looked at in relation to water as the solvent.

a) Soluble Salts

Soluble salts are those that dissolve in water to form aqueous solutions. They tend to have a crystalline texture. Soluble salts include the following:

- All potassium, sodium and ammonium salts
- All nitrates
- All Ethanoates/Acetates
- All hydrogencarbonates
- All nitrites except except silver nitrite which is sparingly soluble.
- All common Halides (Chlorides, Bromides and Iodides) except those of Ag^+, $Pb^{2+}$and Cu^+.

Note: Halides of Pb^{2+} are insoluble in cold water but sparingly soluble in warm water/hot water.

- All common sulphates except those of $Pb^{2+}$and Ba^{2+}.

Note: Calcium sulphate and silver sulphate are sparingly soluble.

- All sulphites are soluble except those of Pb^{2+}, $Ba^{2+}$and Ca^{2+}.
- All hypochlorites are soluble except those of Pb^{2+} and Ag^+.
- All chromates are soluble except those of Pb^{2+}, Ba^{2+} and Ag^+.

Note: Calcium chromate is sparingly soluble.

b) Insoluble salts

Insoluble salts are those that do not dissolve in water. However, they can dissolve in dilute acids. The dilute acid most commonly used in dissolving insoluble salts is dilute nitric acid; dilute hydrochloric acid and dilute sulphuric acid may also be used in dissolving specific insoluble salts.

Insoluble salts tend to have a powdered/powdery texture. Insoluble salts include the following:

- All Carbonates except those of K^+, Na^+ and NH_4^+.
- All Oxalates except those of K^+, Na^+ and NH_4^+.
- All Phosphates except those of K^+, Na^+ and NH_4^+.
- Halides of Pb^{2+}, Ag^+, Cu^+.

Note: Halides of Pb^{2+} are insoluble in cold water but sparingly soluble in warm water/hot water.

- Sulphates of $Pb^{2+}$and Ba^{2+}.

Note: Calcium sulphate and Silver sulphate are sparingly soluble.

- Sulphites of Pb^{2+}, $Ba^{2+}$and Ca^{2+}.
- Hypochlorites of Pb^{2+} and Ag^+.
- Chromates of Pb^{2+}, Ba^{2+} and Ag^+.

Note: Calcium chromate is sparingly soluble.

8.2.3Solutions formed by soluble and insoluble salts

In dissolving a salt in water or an acid, to a spatula endful or two spatula endfuls of the solid, in a clean test tube, 4 to 7cm^3 of water or the acid is added. This is usually followed by shaking of the test tube and its contents in order to dissolve effectively. While dissolving an insoluble salt in an acid, it is important to note whether the solid dissolves with effervescence or not; and in case the insoluble salt does not dissolve effectively in the acid, some gentle warming may be required.

Soluble salts dissolve in water and insoluble salts dissolve in acids to form solutions which may becolourless for non-coloured cations or coloured for coloured cations.
Note: Dilute nitric acid can dissolve insoluble salts of all cations while dilute hydrochloric and sulphuric acids dissolve insoluble salts of all cations except lead(II) ions.

Colour of solution	Deduction
Colourless	Zn^{2+}, Pb^{2+}, Al^{3+}, Ca^{2+}, Mg^{2+}, Ba^{2+}, NH_4^+, Sn^{2+}
Green	Fe^{2+}, Cu^{2+}, Ni^{2+}, Cr^{3+}
Blue	Cu^{2+}
Brown	Fe^{3+}
Yellow or Orange	Cr^{6+}(in form of CrO_4^{2-} or $Cr_2O_7^{2-}$)
Deep pink or Red	Co^{2+}
Very pale pink (basically appearing colourless in dilute solution)	Mn^{2+}
Purple	Mn^{7+} (in form of MnO_4^-)

8.2.4Action of heat on unknown solids

In this case, one or two spatula end fulls of the solid are heated gently and then strongly in a dry test tube until there is no further change. In most cases salts decompose on heating to form metal oxides as residues; gaseous non-metal oxides are also evolved in the process; for instance,carbon dioxide gas in case the solid contains a carbonate, hydrogencarbonate,oxalateor ethanoate/acetate;plussulphur dioxide and sulphur trioxide in case the solid contains a sulphate or sulphite. The gases evolved depend on the anions present in the solid while the metal oxide formed as residue depends on the cations present in the solid sample.
Note: The ammonium ion is the only cation which influences the gas evolved on heating a solid (Ammonia gas is evolved when a solid containing the ammonium ion is heated).
The following should be noted in the process of heating:

a) The physical appearance of the solid before and after heating (residue).The colour of the residue should be noted when hot and on cooling (some metal oxides have different colours when hot and when cold).
b) The colour of the sublimate, if at all the solid sublimes on heating (ammonium compounds form a white sublimate on heating).
c) Any vapour or gas evolved should be identified using an appropriate chemical test.

i) Colour changes that occur to solids on heating and their implication

Appearance of solid before heating	Appearance of the residue	Deduction
White crystalline/powdered solid	Yellow residue when hot, turns white on cooling.	ZnO(s) formed hence Zn^{2+}
White crystalline/powdered solid	Reddish-brown residue when hot, turns yellow/orange on cooling.	PbO(s) formed hence Pb^{2+}
White crystalline/powdered solid.	White residue	Al_2O_3(s) formed hence Al^{3+} **or** MgO(s) formed hence Mg^{2+}

		orCaO(s) formed hence Ca^{2+} **or**BaO(s) formed hence Ba^{2+}
Blue crystalline solid.	Black residue.	CuO(s) formed hence Cu^{2+}
Blue crystalline solid.	Whiteresidue.	Hydrated Cu^{2+} turns to anhydrous Cu^{2+}
Green crystalline/powdered solid	Black residue	CuO(s) formed hence Cu^{2+} **Or**NiO(s) formed hence Ni^{2+}
Pale green crystalline solid	Reddish-brown residue	Fe_2O_3(s) formed Fe^{2+} oxidized to Fe^{3+}
Brown	Reddish-brown residue.	Fe_2O_3(s) formed hence Fe^{3+}
Deep pink	Blue and then black on very strong heating.	Hydrated Co^{2+} turns to anhydrous Co^{2+} and then to CoO(s)
Very pale pink crystalline/powdered solid	Black residue	MnO_2(s) formed Mn^{2+} oxidized to Mn^{4+}.

ii) Gases and vapours given off during heating of a solid

Observation	Deduction
A colourless condensate(**or** a colourless liquid) which turns anhydrous copper(II) sulphate from white to blue (**or** turns cobalt(II) chloride paper from blue to pink).	Water of crystallization (**or** H_2O given off from a hydrated salt)
A colourless gas which turns moist blue litmus paper red/pink and limewater milky.	CO_2(g) evolved CO_3^{2-}, HCO_3^-, $C_2O_4^{2-}$, CH_3COO^-suspected
A colourless, pungent, chocking gas, turns moist red litmus paper blue and forms dense white fumes with concentrated hydrochloric acid.	NH_3(g) evolved NH_4^+ suspected
A colourless, pungent gas, turns moist blue litmus paper red and bleaches it, and turns acidifie potassium dichromate solution from orange to green/turns acidified potassium permanganate solution from purple to colourless.	SO_2(g) evolved SO_4^{2-},SO_3^{2-}suspected
White fumes (smoky white fumes) which turn moist blue litmus paper red and form a white precipitate with barium nitrate solution.	SO_3(g) evolved SO_4^{2-} suspected
Misty fumes with a chocking smell turn moist blue litmus paper red and form dense white fumes with concentrated ammonia.	HCl(g) evolved Cl^- suspected
Brown fumes with a pungent smell and turn moist blue litmus paper red anda colourless gas which relights a glowing splint. A cracking sound is heared.	NO_2(g) evolved O_2(g) evolved NO_3^- suspected
A colourless vapour with a sweet odour; forms a yellow precipitate with 2,4-dinitrophenylhydrazine solution (Brady's reagent).	CH_3COCH_3(g) evolved CH_3COO^- suspected
Brown vapour which turns moist blue litmus paper red and bleaches it.	Br_2(g) evolved Br^-suspected
Purple vapour which turns moist blue litmus paper red; sublimes to form a black/purple/purplish-black solid.	I_2(g) evolved I^- suspected

Note: When nitrates are decomposed by heat, there are some differences in the products formed depending on the position of the metal in the electrochemical series.
Nitrates of potassium and sodium decompose to form the metal nitrites and oxygen gas. For instance:

$$2KNO_3(s) \rightarrow 2KNO_2(s) + O_2(g)$$
$$2NaNO_3(s) \rightarrow 2NaNO_2(s) + O_2(g)$$

Nitrates of calcium, magnesium, aluminum, zinc, iron, lead and copper decompose to form the metal oxides, nitrogen dioxide gas and oxygen gas. For instance:

$$2Pb(NO_3)_2(s) \rightarrow 2PbO(s) + 4NO_2(g) + O_2(g)$$

Nitrates of mercury and silver decompose to form the metal (since the metal oxides are themselves decomposed by heat), nitrogen dioxide gas and oxygen gas.

$$Hg(NO_3)_2(s) \rightarrow Hg(s) + 2NO_2(g) + O_2(g)$$
$$2AgNO_3(s) \rightarrow Ag(s) + 2NO_2(g) + O_2(g)$$

8.3 DETECTION OF CATIONS AND ANIONS IN SOLIDS AND SOLUTIONS

8.3.1 Reagents used in the identification of cations in solids and solutions

The major reagents used include dilute sodium hydroxide solution, dilute ammonium hydroxide solution (dilute ammonia solution), sodium carbonate solution, potassium iodide solution, potassium chromate(VI) solution, disodium hydrogenphosphate solution, ammonium oxalate solution, litmus solution, dimethyl glyoxime solution, potassium hexacyanoferrate(II) solution, potassium hexacyanoferrate(III) solution, ammonium thiocyanate solution/potassium thiocyanate solution plus many other reagents.

a) Action of dilute sodium hydroxide solution

To the test solution, dilute sodium hydroxide solution is added drop wise until in excess. In case a precipitate is formed on addition of a few drops of dilute sodium hydroxide solution, the precipitate's colour is noted and the sodium hydroxide solution is added in excess as the student notes whether the precipitate dissolves in excess or not.
Incase no precipitate is formed, the solution is warmed gently and the gas evolved is tested with litmus paper and concentrated HCl (this is done in case the ammonium ion is suspected to be present).
Zn^{2+}, Pb^{2+} and Al^{3+} not only form white precipitates of their hydroxides on addition of a few drops of sodium hydroxide solution, but their precipitates also dissolve in excess sodium hydroxide solution to form colourless complexes which appear as colourless solutions. This is so because their hydroxides are amphoteric (have both basic and acidic properties).

i) For Zn^{2+},

In a little dilute Sodium hydroxide solution,

$$Zn^{2+}(aq) + 2OH^-(aq) \rightarrow Zn(OH)_2(s)$$

White precipitate of zinc hydroxide

In excess dilute Sodium hydroxide solution,

$$Zn(OH)_2(s) + 2OH^-(aq) \rightarrow [Zn(OH)_4]^{2-}(aq)$$

Colourless solution of the tetrahydroxozincate(II) ion

ii) For Pb^{2+},

In a little dilute sodium hydroxide solution,

$$Pb^{2+}(aq) + 2OH^-(aq) \rightarrow Pb(OH)_2(s)$$

White precipitate of lead(II) hydroxide

In excess dilute sodium hydroxide solution,

$Pb(OH)_2(s) + 2OH^-(aq) \rightarrow [Pb(OH)_4]^{2-}(aq)$

Colourless solution of the tetrahydroxoplumbate(II) ion

iii)For Al^{3+},

In a little dilute sodium hydroxide solution,

$Al^{3+}(aq) + 3OH^-(aq) \rightarrow Al(OH)_3(s)$

White precipitate of aluminium hydroxide

In excess dilute sodium hydroxide solution,

$Al(OH)_3(s) + OH^-(aq) \rightarrow [Al(OH)_4]^-(aq)$

Colourless solution of the tetrahydroxoaluminate(III) ion

iv) For Sn^{2+},

In a little dilute sodium hydroxide solution,

$Sn^{2+}(aq) + 2OH^-(aq) \rightarrow Sn(OH)_2(s)$

White precipitate of tin(II) hydroxide

In excess dilute sodium hydroxide solution,

$Sn(OH)_2(s) + 2OH^-(aq) \rightarrow [Sn(OH)_4]^{2-}(aq)$

Colourless solution of the tetrahydroxostannate(II) ion

Other cations such as **Ca^{2+}, $Mg^{2+}$and Ba^{2+}**form white precipitates of their hydroxides in a little sodium hydroxide solution which are insoluble in excess.

$Ca^{2+}(aq) + 2OH^-(aq) \rightarrow Ca(OH)_2(s)$

White precipitate of calcium hydroxide

$Mg^{2+}(aq) + 2OH^-(aq) \rightarrow Mg(OH)_2(s)$

White precipitate of magnesium hydroxide

$Ba^{2+}(aq) + 2OH^-(aq) \rightarrow Ba(OH)_2(s)$

White precipitate of barium hydroxide

Note:1) With NH_4^+, there is no observable change with dilute sodium hydroxide solution. This is because the ammonium hydroxide formed is a soluble salt and therefore the solution remains colourless.

2) However, when dilute sodium hydroxide solution is added to a solution of NH_4^+ and the solution warmed, acolourless, pungent, chocking gas which turns moist red litmus paper blue and forms dense white fumes with concentrated hydrochloric acid, is evolved. The gas evolved is ammonia.

$NH_4^+(aq) + OH^-(aq) \rightarrow NH_3(g) + H_2O(l)$

Cu^{2+} form a pale blue precipitate of copper(II) hydroxide in a little sodium hydroxide solution, which is insoluble in excess.

$Cu^{2+}(aq) + 2OH^-(aq) \rightarrow Cu(OH)_2(s)$

Pale blue precipitate of copper(II) hydroxide

In case addition of excess sodium hydroxide solution is followed by heating, the pale blue precipitate turns black. This is due to the formation of copper(II) oxide.

$Cu(OH)_2(s) \rightarrow CuO(s) + H_2O(l)$

Black solid

Fe^{2+} form a dirty green precipitate of iron(II) hydroxide in a little sodium hydroxide solution which is insoluble in excess.

$Fe^{2+}(aq) + 2OH^-(aq) \rightarrow Fe(OH)_2(s)$

Dirty green precipitateof iron(II) hydroxide

The dirty green precipitateslowly turns brown on standing due to aerial oxidation of Fe^{2+} to Fe^{3+}.

i.e. $4Fe(OH)_2(s) + O_2(g) + 2H_2O(l) \rightarrow 4Fe(OH)_3(s)$

Brown precipitate of iron(III) hydroxide

Fe^{3+} form a brown precipitate (reddish-brown/rusty-brown precipitate) in a little sodium hydroxide solution which is insolublein excess.

$Fe^{3+}(aq) + 3OH^-(aq) \rightarrow Fe(OH)_3(s)$

Brown precipitate of iron(III) hydroxide

Ni^{2+} form a pale green precipitatae insoluble in excess

$Ni^{2+}(aq) + 2OH^-(aq) \rightarrow Ni(OH)_2(s)$

Pale green precipitate of nickel(II) hydroxide

Cr^{3+} form a green precipitate (grey-green precipitate) soluble in excess to form a green solution.

$Cr^{3+}(aq) + 3OH^-(aq) \rightarrow Cr(OH)_3(s)$

Green precipitate of chromium(III) hydroxide

In excess dilute sodium hydroxide solution,

$Cr(OH)_3(s) + OH^-(aq) \rightarrow [Cr(OH)_4]^-(aq)$

Green solution of the tetrahydroxochromate(III) ion

OR $Cr(OH)_3(s) + 3OH^-(aq) \rightarrow [Cr(OH)_6]^{3-}(aq)$

Green solution of the hexahydroxochromate(III) ion

Mn^{2+} form a dirty white precipitate insoluble in excess.

$Mn^{2+}(aq) + 2OH^-(aq) \rightarrow Mn(OH)_2(s)$

Dirty white precipitate of manganese(II) hydroxide

The dirty white precipitate, however, turns brown on standing in air (due to aerial oxidation of Mn^{2+} to Mn^{3+}).

$2Mn(OH)_2(s) + \frac{1}{2}O_2(g) \rightarrow Mn_2O_3.2H_2O(s)$

Brown precipitate of hydrated manganese(III) oxide

Co^{2+} form a blue precipitate insoluble in excess.

$Co^{2+}(aq) + 2OH^-(aq) \rightarrow Co(OH)_2(s)$

Blue precipitate of cobalt(II) hydroxide

The blue precipitate, however, turns pink on standing due to aerial oxidation of Co^{2+} to Co^{3+}.

$2Co(OH)_2(s) + \frac{1}{2}O_2(g) \rightarrow Co_2O_3.2H_2O(s)$

Pink precipitate of hydratedcobalt(III) oxide

With Cr^{6+} in the form of dichromate(VI) ions, $Cr_2O_7^{2-}$, the orange solution turns yellow. This is due to formation of chromate(VI) ions, CrO_4^{2-} as shown below:

$Cr_2O_7^{2-}(aq) + 2OH^-(aq) \rightarrow 2CrO_4^{2-}(aq) + H_2O(l)$

Orange solution *Yellow solution*

Table showing summary of action of dilute sodium hydroxide solution on solutions of various cations

Observation	Deduction
A white precipitate soluble in excess to form a colourless solution.	Zn^{2+}, Pb^{2+}, Al^{3+} Sn^{2+}
A white precipitate insoluble in excess.	Ca^{2+}, Mg^{2+}, Ba^{2+}
A pale blue precipitate insoluble in excess.	Cu^{2+}
A dirty green precipitate insoluble in excess.	Fe^{2+}

A brown precipitate (reddish-brown precipitate/rusty-brown precipitate) insoluble in excess.	Fe^{3+}
No observable change but on warming, a colourless, pungent, chocking gas that turns moist red litmus paper blue and forms dense white fumes with concentrated hydrochloric acid.	$NH_3(g)$ evolved NH_4^+ confirmed present
A pale green precipitate insoluble in excess.	Ni^{2+}
A green precipitate (grey-green precipitate) soluble in excess to form a green solution.	Cr^{3+}
A dirty white precipitate insoluble in excess, turns brown on standing in air.	Mn^{2+}
A blue precipitate insoluble in excess, turns pink on standing, turns brown on further standing.	Co^{2+}
Orange solution turns yellow.	$Cr_2O_7^{2-}$ turns to CrO_4^{2-}

b) Action of dilute ammonium hydroxide solution (dilute ammonia solution)

To the test solution, dilute ammonium hydroxide solution is added drop wise until in excess. Incase a precipitate is formed on addition of a few drops of dilute ammonium hydroxide solution, the precipitate's colour is noted and the ammonium hydroxide solution is added in excess as the student notes whether the precipitate dissolves in excess or not.

It is only Zn^{2+} ions that form a white precipitate that is soluble in excess ammonia solution to form a colourless solution. This is due to formation of a soluble complex called the tetraamminezinc(II) ion in excess ammonia solution.

With a little dilute ammonium hydroxide solution,

$$Zn^{2+}(aq) + 2OH^-(aq) \rightarrow Zn(OH)_2(s)$$

White precipitate of zinc hydroxide

With excess dilute ammonia solution,

$$Zn(OH)_2(s) + 4NH_3(aq) \rightarrow [Zn(NH_3)_4]^{2+}(aq) + 2OH^-(aq)$$

Tetraamminezinc(II) ion (Colourless solution)

Pb^{2+}, $Al^{3+}$and Sn^{2+}form white precipitates with a little dilute ammonia solution, which are insoluble in excess. The white precipitates are of lead(II) hydroxide aluminium hydroxide and tin(II) hydroxide respectively.

$$Pb^{2+}(aq) + 2OH^-(aq) \rightarrow Pb(OH)_2(s)$$

White precipitate of lead(II) hydroxide

$$Al^{3+}(aq) + 3OH^-(aq) \rightarrow Al(OH)_3(s)$$

White precipitate of aluminium hydroxide

$$Sn^{2+}(aq) + 2OH^-(aq) \rightarrow Sn(OH)_2(s)$$

White precipitate of tin(II) hydroxide

Since the three cations, Pb^{2+}, Al^{3+} and Sn^{2+}behave in a similar way with both dilute sodium hydroxide and dilute ammonia solution, hence there is need for another reagent to differentiate between the two; the reagent is potassium iodide solution. Pb^{2+} ions form a yellow precipitate of lead(II) iodide while with Al^{3+} and Sn^{2+} ions, there is no observable change.

$$Pb^{2+}(aq) + 2I^-(aq) \rightarrow PbI_2(s)$$

Yellow precipitate

In case the potassium iodide solution used is sufficiently concentrated (e.g. 50—60% potassium iodide solution), and is used in excess, the yellow precipitate of lead(II) iodide dissolves in excess to form a colourless solution. This is due to formation a soluble complex referred to as the tetraiodoplumbate(II) ion.

$$PbI_2(s) + 2I^-(aq) \rightarrow PbI_4^{2-}(aq)$$

Colourless solution of the tetraiodoplumbate(II) ion

Note: This can also be used as a confirmatory test for Pb^{2+}.

With Mg^{2+} and Ba^{2+}, on addition of a little dilute ammonia solution, a white precipitate is formed, which is insoluble in excess.

$$Mg^{2+}(aq) + 2OH^-(aq) \rightarrow Mg(OH)_2(s)$$

White precipitate of magnesium hydroxide

$$Ba^{2+}(aq) + 2OH^-(aq) \rightarrow Ba(OH)_2(s)$$

White precipitate of barium hydroxide

With Ca^{2+}, on addition of a little dilute ammonia solution until in excess, there is no observable change. This is because the concentration of hydroxyl ions, OH^- formed by partial ionization of the ammonia solution is low and hence the K_{sp} is not sufficient to exceed the ionic product.

Similarly, when ammonia solution is added to a solution containing NH_4^+, there is no observable change.

Cu^{2+} form a pale blue precipitate of copper(II) hydroxide with a little ammonia solution, which dissolves in excess to form a deep blue solution. The deep blue solution is due to formation of a soluble complexreferred to as the tetraamminecopper(II) ion.

With a little dilute ammonia solution,

$$Cu^{2+}(aq) + 2OH^-(aq) \rightarrow Cu(OH)_2(s)$$

Pale blue precipitate

With excess dilute ammonia solution,

$$Cu(OH)_2(s) + 4NH_3(aq) \rightarrow [Cu(NH_3)_4]^{2+}(aq) + 2OH^-(aq)$$

Tetraamminecopper(II) ion (Deep blue solution)

Note: Thisis one of the confirmatory tests for Cu^{2+}ions.

Fe^{2+} form a dirty green precipitate of iron(II) hydroxide with a little dilute ammonia solution, which is insoluble in excess.

$$Fe^{2+}(aq) + 2OH^-(aq) \rightarrow Fe(OH)_2(s)$$

Dirty green precipitate

The dirty green precipitate, however, slowly turns brown on standing due to aerial oxidation of Fe^{2+} to Fe^{3+}.

$$\text{i.e. } 4Fe(OH)_2(s) + O_2(g) + 2H_2O(l) \rightarrow 4Fe(OH)_3(s)$$

Brown precipitate of iron(III) hydroxide

Fe^{3+} form a brown precipitate (reddish-brown/rusty-brown precipitate) of iron(III) hydroxide in a little dilute ammonia solution, which is insoluble in excess.

$$Fe^{3+}(aq) + 3OH^-(aq) \rightarrow Fe(OH)_3(s)$$

Brown precipitate

Ni^{2+} form a pale green precipitate soluble in excess to form a blue solution. The blue solution is due to formation of the complex ion called the hexaamminenickel(II) ion.

$$Ni^{2+}(aq) + 2OH^-(aq) \rightarrow Ni(OH)_2(s)$$

Pale green precipitate of nickel(II) hydroxide

$$Ni(OH)_2(s) + 6NH_3(aq) \rightarrow [Ni(NH_3)_6]^{2+}(aq) + 2OH^-(aq)$$

Blue solution of the hexaamminenickel(II) ion

Cr^{3+} form a green precipitate(grey-green precipitate) insoluble in excess.

$$Cr^{3+}(aq) + 3OH^-(aq) \rightarrow Cr(OH)_3(s)$$

Green precipitate of chromium(III) hydroxide

Mn^{2+} form a dirty white precipitate insoluble in excess, turns brown on standing in air (due to aerial oxidation of Mn^{2+} to Mn^{3+}).

$$Mn^{2+}(aq) + 2OH^-(aq) \rightarrow Mn(OH)_2(s)$$

Dirty white precipitate of manganese(II) hydroxide

Co^{2+} form a blue precipitate insoluble in excess.

$$Co^{2+}(aq) + 2OH^-(aq) \rightarrow Co(OH)_2(s)$$

Blue precipitate of cobalt(II) hydroxide

However, if concentrated ammonia solution is added drop wise until in excess to an aqueous solution of cobalt(II) ions, Co^{2+}, a blue precipitate is formed which is soluble in excess to form a pale yellow solution, which turns brown on standing in air.

$$Co^{2+}(aq) + 2OH^-(aq) \rightarrow Co(OH)_2(s)$$

Blue precipitate of cobalt(II) hydroxide

$$Co(OH)_2(s) + 6NH_3(aq) \rightarrow [Co(NH_3)_6]^{2+}(aq) + 2OH^-(aq)$$

Hexaamminecobalt(II) ion (Pale yellow solution)

$$[Co(NH_3)_6]^{2+}(aq) \rightarrow [Co(NH_3)_6]^{3+}(aq) + e^-$$

Hexaamminecobalt(III) ion (Brown solution)

Table showing summary of action of dilute ammonia solution (dilute ammonium hydroxide solution) on solutions of various cations

Observation	Deduction
A white precipitate soluble in excess to form a colourless solution.	Zn^{2+}
A white precipitate insoluble in excess.	Pb^{2+}, Al^{3+}, Sn^{2+}, Mg^{2+}, Ba^{2+}
No observable change.	Ca^{2+}, NH_4^+
A pale blue precipitate soluble in excess to form a deep blue solution.	Cu^{2+}
A dirty green precipitate insoluble in excess.	Fe^{2+}
A brown precipitate(reddish-brown precipitate/rusty-brown precipitate) insoluble in excess.	Fe^{3+}
A pale green precipitate soluble in excess to form a blue solution.	Ni^{2+}
A green precipitate(grey-green precipitate) insoluble in excess.	Cr^{3+}
A blue precipitate insoluble in excess.	Co^{2+}

c) Action of sodium carbonate solution

To the test solution, sodium carbonate solution is added drop wise until in excess. All cations whose carbonates are stable, e.g. Zn^{2+}, Pb^{2+}, Mg^{2+}, Ca^{2+}, Cu^{2+} and Fe^{2+} form precipitates that are insoluble in excess. . For Zn^{2+}, Pb^{2+}, Mg^{2+}, Ca^{2+}, Ba^{2+}, a white precipitate insoluble in excess is observed; for Cu^{2+}, a green precipitate insoluble in excess (or a blue precipitate insoluble in excess) is observed while for Fe^{2+} a dirty green precipitate insoluble in excess is observed. However, for the aluminium ion, $Al^{3+}$the iron(III) ion, Fe^{3+} and and chromium(III) ions, Cr^{3+}whose carbonatesare very unstable, as soon as they are formed, they decompose to form carbon dioxide gas and meanwhile the corresponding metal hydroxides are formed. Consequently, the precipitates of the metal hydroxide are formed which are insoluble in excess sodium carbonate solution with effervescence of a colourless gas which turns moist blue litmus paper red and limewater milky.

(a) $Zn^{2+}(aq) + CO_3^{2-}(aq) \rightarrow ZnCO_3(s)$

(b) $Pb^{2+}(aq) + CO_3^{2-}(aq) \rightarrow PbCO_3(s)$

(c) $Mg^{2+}(aq) + CO_3^{2-}(aq) \rightarrow MgCO_3(s)$

(d) $Ca^{2+}(aq) + CO_3^{2-}(aq) \rightarrow CaCO_3(s)$

(e) $Ba^{2+}(aq) + CO_3^{2-}(aq) \rightarrow BaCO_3(s)$

(f) $Cu^{2+}(aq) + CO_3^{2-}(aq) \rightarrow CuCO_3(s)$

OR:

$CO_3^{2-}(aq) + 2H_2O(l) \longrightarrow 2OH^-(aq) + H_2CO_3(aq)$

$Cu^{2+}(aq) + 2OH^-(aq) \longrightarrow Cu(OH)_2(s)$

(g) $Fe^{2+}(aq) + CO_3^{2-}(aq) \rightarrow FeCO_3(s)$

(h) $2Al^{3+}(aq) + 3CO_3^{2-}(aq) + 3H_2O(l) \rightarrow 2Al(OH)_3(s) + 3CO_2(g)$

(i) $2Fe^{3+}(aq) + 3CO_3^{2-}(aq) + 3H_2O(l) \rightarrow 2Fe(OH)_3(s) + 3CO_2(g)$

(j) $2Cr^{3+}(aq) + 3CO_3^{2-}(aq) + 3H_2O(l) \rightarrow 2Cr(OH)_3(s) + 3CO_2(g)$

Observation	Cation
A white precipitate insoluble in excess.	Zn^{2+}
White precipitate insoluble in excess with ffervescence of a colourless gas that turns limewater milky.	Al^{3+}
A white precipitate insoluble in excess.	Zn^{2+}, Pb^{2+}, Mg^{2+}, Ca^{2+}, Ba^{2+}, Sn^{2+}
A dirty white precipitate insoluble in excess, turns brown on standing in air.	Mn^{2+}
A green precipitate (or grey-green or grey-blue precipitate)insoluble in excesswith effervescence of a colourless gas that turns limewater milky.	Cr^{3+}
A green precipitate insoluble in excess (**or** blue precipitate insoluble in excess).	Cu^{2+}
Adirty green precipitate insoluble in excess.	Fe^{2+}
A brown/reddish-brown /rusty brown precipitate insoluble in excess	Fe^{3+}

with effervescence of a colourless gas which turns limewater milky.	
Pale green precipitate insoluble in excess.	Ni^{2+}
A blue-green precipitate soluble in excess.	Cr^{3+}
A pink-violet precipitate insoluble in excess.	Co^{2+}
No observable change.	NH_4^+

Note: When sodium carbonate solution is added to a solution containing an acid (a solution containing Hydrogen ions), there is effervescence of a colourless gas which turns moist blue litmus paper red and limewater milky. The gas evolved is carbon dioxide due to the following reaction.

$$2H^+(aq) + CO_3^{2-}(aq) \rightarrow H_2O(l) + CO_2(g)$$

d) Action of potassium iodide solution

Potassium iodide solution is used to test for the presence of Pb^{2+}. About 2-3 drops of potassium iodide solution are added to the test solution. Formation of a yellow precipitate indicates the presence ofPb^{2+}. lead(II) ions react with Iodide ions in potassium iodide to form lead(II) iodide which is an insoluble salt and appears as a yellow precipitate.

$$Pb^{2+}(aq) + 2I^-(aq) \rightarrow PbI_2(s)$$

Yellow precipitate

Test	Observation	Deduction
To the test solution, add 2-3 drops of otassium iodide solution.	A yellow precipitate is formed.	Pb^{2+}present

In case the Potassium iodide solution used is sufficiently concentrated and is used drop wise until in excess, the yellow precipitate of lead(II) iodide formed on addition of a few drops of potassium iodide solution dissolves in excess to form a colourless solution. This is due to formation of the tetraiodoplumbate(II) ion. When used in such a way, potassium iodide solution is used to confirm the presence of Pb^{2+}.

$$PbI_2(s) + 2I^-(aq) \rightarrow PbI_4^{2-}(aq)$$

Colourless solution of the tetraiodoplumbate(II) ion

Test	Observation	Deduction
To the test solution, add potassium iodide solution dropwise until in excess.	A yellow precipitate soluble in excess forming a colourless solution.	Pb^{2+}confirmed present

Note: Potassium iodide solution can also be used to confirm the presence of copper(II) ions, Cu^{2+}. The presence of Cu^{2+} is shown by formation of a white precipitate in a brown solution. The white precipitate is due to formation of copper(I) iodide, Cu_2I_2. The brown solution is due to formation of Iodine solution.

$$2Cu^{2+}(aq) + 4I^-(aq) \rightarrow Cu_2I_2(s) + I_2(aq)$$

However, if this is followed by addition of Sodium thiosulphate solution, the brown solution turns colourless, leaving behind the white precipitate. This is due to the reduction of aqueous Iodine by thiosulphate ions to iodide ions.

$$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$$

e) Action of Litmus solution

Litmus solution is used in confirming the presence of Al^{3+}. To the test solution, about 2 drops of litmus solution, followed by about $1cm^3$ of dilute hydrochloric acid and then dilute ammonia solution untilthe solution is just alkaline and then Alizarin Reagent. Formation of a pink colouration confirms the presence of Al^{3+}.

OR To the test solution 2-3 drops of dilute nitric acid are added followed by 3-4 drops of litmus solution and then dilute ammonia solution drop wise until in excess.Formation of a blue lake solution confirms the presence of Al^{3+}.

Note: Litmus blue solution should be preferably used for this test.

f) Action of dilute hydrochloric acid

Dilute hydrochloric acid is also used to test for the presence of Pb^{2+}. To the test solution, a few drops (1-2drops) of dilute hydrochloric acid are added and the resultant solution warmed/heated. This may be followed by cooling the warmed/heated solution in the test tube under running tap water. Presence of Pb^{2+} is shownby formation of a white precipitate which dissolves on warming/heating. The precipitate reappears if the warmed/heated solution is allowed to cool.

Note: Sodium chloride solution can also be used for the same purpose in place of dilute hydrochloric acid.

Observation	Deduction
A white precipitate which dissolves on warming/boiling, precipitate reappears on cooling.	Pb^{2+}present

The above test is based on the fact that Pb^{2+} ions react with Cl^- ions in the dilute hydrochloric acid to form lead(II) chloride which is insoluble in cold water and soluble in warm/hot water.

$$Pb^{2+}(aq) + 2Cl^-(aq) \rightarrow \underset{\textit{White precipitate}}{PbCl_2(s)}$$

g) Action of dilute sulphuric acid

Dilutesulphuric acid is used to test for the presence of Pb^{2+}, $Ba^{2+}$and Ca^{2+}. When any of the cations stated above is present, a white precipitate is formed. This is because the sulphates of the cations stated above are insoluble and appear as white precipitates.However, calcium sulphate is sparingly soluble. For example:

$$Pb^{2+} + SO_4^{2-}(aq) \rightarrow \underset{\textit{White precipitate}}{PbSO_4(s)}$$

Test	Observation	Deduction
To the test solution, add 2-3 drops of dilute sulphuric acid.	A white precipitate is formed.	Pb^{2+}, Ba^{2+}, Ca^{2+}

Note: Sodium sulphate solution or potassium sulphate solution may also be used for the same purpose in place of dilute sulphuric acid.

h) Action of disodium hydrogenphosphate solution

Disodium hydrogen phosphate solution is used in conjunction with solid ammonium chloride and excess ammonia solution to confirm the presence of zinc ions or magnesium ions.

Test	Observation	Deduction

To the test solution, add solid ammonium chloride, followed by 3-4 drops of disodium hydrogen phosphate solution and then dilute ammonia solution drop wise until in excess.	A white precipitate soluble in excess dilute ammonia solution.	Zn^{2+}confirmed present.
	A white precipitate insoluble in excess dilute ammonia solution.	Mg^{2+} confirmed present.

i) Action of potassium chromate solution

Potassium chromate solution is used in conjunction with excess dilute sodium hydroxide solution to confirm the presence of both Pb^{2+} and Ba^{2+} ions.

Test	Observation	Deduction
To the test solution, add 2-3 drops of potassium chromate solution followed by dilute sodium hydroxide solution until in excess.	A yellow precipitate soluble in excess dilute sodium hydroxide solution forming a yellow solution.	Pb^{2+} confirmed present.
	A yellow precipitate insoluble in excess dilute sodium hydroxide solution.	Ba^{2+} confirmed present.

Note:1) The yellow precipitates are due to the formation of the insoluble chromates of Pb^{2+} and Ba^{2+}which are yellow in colour.

$Pb^{2+}(aq) + CrO_4^{2-}(aq) \rightarrow PbCrO_4(s)$

$Ba^{2+}(aq) + CrO_4^{2-}(aq) \rightarrow BaCrO_4(s)$

2)The sequence of observations for confirming the Pb^{2+} are due to the following other reaction:

$PbCrO_4(s) + 4OH^-(aq) \rightarrow Pb(OH)_4^{2-}(aq) + CrO_4^{2-}(aq)$

Colourless solution *Yellow solution*

3) Potassium chromate solution, however, may also be used in conjunction with dilute hydrochloric acid to confirm the presence of Pb^{2+}, and also in conjunction with both dilute hydrochloric acid and dilute sulphuric acid to confirm the presence of Ba^{2+}.

Test	**Observation**	**Deduction**
To the test solution, add 2-3 drops of potassium chromate solution followed by dilute hydrochloric acid.	A yellow precipitate insoluble in dilute hydrochloric acid.	Pb^{2+} confirmed present
To the test solution, add 2-3 drops of potassium chromate solution followed by dilute hydrochloric acid and then dilute sulphuric acid.	A yellow precipitate soluble in dilute hydrochloric acid, precipitate reappears on addition of dilute sulphuric acid.	Ba^{2+} confirmed present

j) Action of ammonium oxalate solution

Ammonium oxalate solution is used in conjunction with dilute hydrochloric acid/dilute nitric acid or ethanoic acid. It is used to test for barium ions,$Ba^{2+}$and calcium ions,Ca^{2+}.

Test	Observation	Deduction

To the test solution, add ammonium oxalate solution followed by dilute hydrochloric acid/nitric acid.	A white precipitate soluble in the acid.	Ba^{2+}, Ca^{2+}
To the test solution, add ammonium oxalate solution followed by ethanoic acid.	A white precipitate soluble in ethanoic acid.	Ba^{2+} confirmed present.
	A white precipitate insoluble in ethanoic acid.	Ca^{2+}confirmed present.

k) Action of potassium hexacyanoferrate(III) solution
(potassium ferricyanide solution)

The presence of $Fe^{2+}$is confirmed by addition of 2-3 drops of potassium hexacyanoferrate(III) solution to the test solution. Formation of a **dark blue precipitate** confirms the presence of Fe^{2+}.

$$Fe^{2+}(aq) + K^{+}(aq) + Fe(CN)_6^{3-}(aq) \rightarrow KFe[Fe(CN)_6](s)$$

Dark blue precipitate

Note:

1) Addition of potassium hexacyanoferrate(III) solution to a solution containing Fe^{3+} results in formation of a brown solution.

2) Addition of potassium hexacyanoferrate(III) solution can also be used to confirm the presence of Ni^{2+} ions. Formation of a brown precipitate confirms the presence of Ni^{2+} ions.

l) Action of potassium hexacyanoferrate(II) solution
(potassium ferrocyanide solution)

The presence of Fe^{3+} is confirmed by use of potassium hexacyanoferrate(II)solution. Formation of a **dark blue precipitate** confirms the presence of Fe^{3+}.

$$Fe^{3+}(aq) + K^{+}(aq) + Fe(CN)_6^{4-}(aq) \rightarrow KFe[Fe(CN)_6](s)$$

Dark blue precipitate

Note:

1) Addition of potassium hexacyanoferrate(III) solution to a solution containing Fe^{2+} results in formation of a pale blue precipitate (light blue precipitate).

2) Addition of potassium hexacyanoferrate(II) solution can also be used to confirm the presence of Cu^{2+} ions. Formation of a brown precipitate confirms the presence of Cu^{2+} ions.

m) Action of ammonium thiocyanate solution or potassium thiocyanate solution

Formation of a deep red solution(formation of a red solution) confirms the presence of Fe^{3+}.

$$Fe(H_2O)_6^{3+}(aq) + SCN^{-}(aq) \rightarrow [Fe(H_2O)_5(SCN)]^{2+}(aq) + H_2O(l)$$

Deep red solution

n) Action of dimethyl glyoxime solution

Dimethyl glyoxime solution is used in confirming the presence of Ni^{2+}. To the test solution, a few drops of dilute ammonia solution is added followed by dimethyl glyoxime solution. Formation of a bright red precipitate/red precipitate/pink precipitate confirms the presence of Ni^{2+}.

Note: The presence of Ni^{2+} ions can also be confirmed by addition of potassium hexacyanoferrate(III) solution. Formation of a brown precipitate confirms the presence of Ni^{2+}.

o) Action of concentrated nitric acid

When concentrated nitric acid added to a solution containing Fe^{2+}, and the resultant solution warmed, the pale green solution turns brown on warming. This is because concentrated nitric acid is an oxidizing agent and therefore oxidizes Fe^{2+} which appear pale green in solution to Fe^{3+}which appear brown in solution.

$$Fe^{2+}(aq) \rightarrow Fe^{3+}(aq) + e^-$$

Note: Concentrated nitric acid can also be used in conjunction with solid sodium bismuthate or lead(IV) oxide followed by warming/heating to confirm the presence of Mn^{2+} whereby a purple solution is formed. This is because Mn^{2+} which are very pale pink (basically appear colourless in solution) are oxidized to MnO_4^- which appear purple in solution.

$$2Mn^{2+}(aq) + 5BiO_3^-(s) + 14H^+(aq) \rightarrow 2MnO_4^-(aq) + 5Bi^{3+}(aq) + 7H_2O(l)$$
$$2Mn^{2+}(aq) + 5PbO_2(s) + 4H^+(aq) \rightarrow 2MnO_4^-(aq) + 5Pb^{2+}(aq) + 2H_2O(l)$$

p) Action of concentrated hydrochloric acid.

Concentrated hydrochloric acid can be used to confirm the presence of Cu^{2+}. Addition of excess concentrated hydrochloric acid to a solution containing Cu^{2+}, results in formation of a yellow solution. This is due to formation of a complex ion called the tetrachlorocuprate(II) ion, $CuCl_4^{2-}$.

$$Cu^{2+}(aq) + 4Cl^-(aq) \rightarrow \underset{\textit{Yellow solution}}{CuCl_4^{2-}(aq)}$$

OR $$Cu(H_2O)_6^{2+}(aq) + 4Cl^-(aq) \rightarrow \underset{\textit{Yellow solution}}{CuCl_4^{2-}(aq)} + 6H_2O(l)$$

The presence of cobalt(II) ions, Co^{2+} can also be confirmed using concentrated hydrochloric acid.Addition of excess concentrated hydrochloric acid to a solution containing Co^{2+}, results in formation of a blue solution. This is due to formation of a soluble complex called the tetrachlorocobaltate(II) ion, $CoCl_4^{2-}$.

$$Co(H_2O)_6^{2+}(aq) + 4Cl^-(aq) \rightarrow \underset{\textit{Blue solution}}{CoCl_4^{2-}(aq)} + 6H_2O(l)$$

Similarly, when concentrated hydrochloric acid is added to a solution containing Co^{2+}, followed by solid ammonium thiocyanate and then pentanol(amylalcohol) and gentle shaking done, a blue solution in the organic layer (blue solution in upper layer) and a purple solution in the inorganic layer(purple solution in the lower layer) is observed. This also confirms the presence of Co^{2+}.

Concentrated hydrochloric acid can also be used to confirm the presence of Pb^{2+}.

When concentrated hydrochloric acid is added drop wise until in excess, to a solution containing lead(II) ions, a white precipitate is formed which dissolves in excess to form a pale yellow solution. The white precipitate is due to formation of insoluble lead(II) chloride while the pale yellow solution is due to formation of a soluble complex called the tetrachloroplumbate(II) ion, $PbCl_4^{2-}$.

$$Pb^{2+}(aq) + 2Cl^-(aq) \rightarrow \underset{\textit{White precipitate}}{PbCl_2(s)}$$
$$PbCl_2(s) + 2Cl^-(aq) \rightarrow \underset{\textit{Pale yellow soluiion}}{PbCl_4^{2-}(aq)}$$

q) Action of concentrated sulphuric acid

A few drops(about 3 to 5 drops) of concentrated sulphuric acid are added to a solid followed by gentle warming. This is usually used to test for cations which have a different colour while in anhydrous form (in form of an anhydrous salt) and while in a hydrated form (in form of a hydrated salt). Such cations include $Cu^{2+}$and Co^{2+}.

Test	Observation	Deduction
To the unknown solid, add 2 to 3 drops of concentrated sulphuric acid and warm gently.	The blue solid turns white	Hydrated Cu^{2+}salt turns to anhydrous Cu^{2+}salt.
	The pink solid turns blue	Hydrated Co^{2+}salt turns to anhydrous Co^{2+}salt.

r)Action of hydrogen peroxide solution

Hydrogen peroxide solution, in the presence of excess alkali(e.g. excess sodium hydroxide solution), and then warming, oxidizes Cr^{3+} which appear green in solution to chromate(VI) ions, CrO_4^{2-} which appear yellow in solution.

$$2Cr^{3+}(aq) + 10OH^-(aq) + 3H_2O_2(aq) \rightarrow 2CrO_4^{2-}(aq) + 8H_2O(l)$$

When amylalcohol is added to the resultant solution, followed by dilute sulphuric acid, a blue solution in the organiclayer is formed.

s) Action of acidified potassium manganate(VII) solution

Tin(II) ions, Sn^{2+}, turn acidified potassium manganate(VII) solution from purple to colourless. $Sn^{2+}$are strong reducing agents and therefore reduce manganate(VII) ions, MnO_4^- which appear purple in solution, to Mn^{2+} which are very pale pink and hence basically appear colourless in solution. Meanwhile the Sn^{2+} are oxidized to Sn^{4+}.

$$5Sn^{2+}(aq) + 2MnO_4^-(aq) + 16H^+(aq) \rightarrow 2Mn^{2+}(aq) + 5Sn^{4+}(aq) + 8H_2O(l)$$

t) Action of silver nitrate solution

Silver nitrate solution, when added to a solution of Sn^{2+} in the presence of excess dilute sodium hydroxide solution, a grey precipitate is formed. This is because in excess dilute sodium hydroxide solution, Sn^{2+} exists as the tetrahydroxostannate(II) ion which reduces the Ag^+ from silver nitrate to silver metal which appears as a grey precipitate. Meanwhile the Sn^{2+} are oxidized to Sn^{4+}.

$$Sn(OH)_4^{2-}(aq) + 2Ag^+(aq) \rightarrow Sn^{4+}(aq) + 2Ag(s) + 4OH^-(aq)$$

8.3.1.1 The "Just Acidic" Concept

This concept is used to test for cations using either addition of dilute sodium hydroxide or dilute ammonia solution drop wise until in excess and carrying out filtration, followed by drop wise addition of a dilute mineral acid to the resultant filtrate until there is no further change.

1) With dilute sodium hydroxide solution

Dilute sodium hydroxide solution is added drop wise until in excess to a test solution containing two cations, one of which forms a precipitate soluble in excess while the other cation forms a precipitate insoluble in excess dilute sodium hydroxide solution. The addition of excess dilute sodium hydroxide solution is followed by filtration and to the filtrate, a dilute mineral acid such as dilute nitric acid, dilute hydrochloric acid or dilute sulphuric acid is added drop by drop while shaking the test tube with its contents until the solution is **"just acidic".**

How does one tell that the filtrate has become just acidic?

When the dilute mineral acid is added to the filtrate, at first, a white precipitate is formed but with continued addition of the acid drop by drop, while shaking the test tube with its contents, the white precipitate dissolves in the acid to form a colourless solution and at this point when the precipitate has just dissolved in the acid to form a colourless solution, the solution is now **"just acidic".**

Explanation

The test solution contains one cation from this category of cations: Zn^{2+}, Pb^{2+}, Al^{3+}, Sn^{2+} or Cr^{3+} and another from this category of cations: Ca^{2+}, Mg^{2+}, Ba^{2+}, Mn^{2+}, Cu^{2+}, Fe^{2+}, Ni^{2+}, $Fe^{3+}$and Co^{2+}.

a) The cation from the first category forms **a precipitate with dilute sodium hydroxide solution, which dissolves in excess to form a solution** and therefore, on filtration, this cation will be present in the filtrate in form of a soluble complex ion (in form of the tetrahydroxozincate(II) ion, tetrahydroxoplumbate(II) ion, tetrahydroxoaluminate(III) ionor tetrahydroxostannate(II) ion).
On addition of the dilute mineral acid to the filtrate**, a white precipitate is formed which dissolves in the acid to form a colourless solution if Zn^{2+}, Pb^{2+}, Al^{3+}or Sn^{2+} are present.** This is due to the following reactions.

i) For Zn^{2+},

$$\underset{\text{Tetrahydroxozincate(II)ion}}{[Zn(OH)_4]^{2-}(aq)} + 2H^+(aq) \rightarrow \underset{\text{White ppt of zinc hydroxide}}{Zn(OH)_2(s)} + 2H_2O(l)$$

$$\underset{\text{Colourless solution of zinc ions}}{Zn(OH)_2(s)} + 2H^+(aq) \rightarrow \underset{\text{Colourless solution of zinc ions}}{Zn^{2+}(aq)} + 2H_2O(l)$$

ii) For Pb^{2+},

$$\underset{\text{Tetrahydroxoplumbate(II)ion}}{[Pb(OH)_4]^{2-}(aq)} + 2H^+(aq) \rightarrow \underset{\text{White ppt of lead(II) hydroxide}}{Pb(OH)_2(s)} + 2H_2O(l)$$

$$Pb(OH)_2(s) + 2H^+(aq) \rightarrow \underset{\text{Colourless solution of lead(II) ions}}{Pb^{2+}(aq)} + 2H_2O(l)$$

iii) For Al^{3+},

$$\underset{\text{Tetrahydroxoaluminate(III) ion}}{[Al(OH)_4]^-(aq)} + H^+(aq) \rightarrow \underset{\text{White ppt of aluminium hydroxide}}{Al(OH)_3(s)} + H_2O(l)$$

$$Al(OH)_3(s) + 3H^+(aq) \rightarrow \underset{\text{Colourless solution of aluminium ions}}{Al^{3+}(aq)} + 3H_2O(l)$$

iv) For Sn^{2+},

$$\underset{\text{Tetrahydroxostannate(II) ion}}{[Sn(OH)_4]^{2-}(aq)} + 2H^+(aq) \rightarrow \underset{\text{White ppt of tin(II) hydroxide}}{Sn(OH)_2(s)} + 2H_2O(l)$$

$$Sn(OH)_2(s) + 2H^+(aq) \rightarrow \underset{\text{Colourless solution of tin(II) ions}}{Sn^{2+}(aq)} + 2H_2O(l)$$

Note: The above are the only key reactions that occur in case the mineral acid used is dilute nitric acid since the metal nitrates that can be formed by the free metal ions (Zn^{2+}, Pb^{2+}, Al^{3+} or Sn^{2+}) are soluble and in such a case, formation of **a white precipitate which dissolves in the acid to form a colourless solution**indicates presence of Zn^{2+}, Pb^{2+}, Al^{3+} or Sn^{2+}. However, if the mineral acid used is dilute hydrochloric acid or dilute sulphuric acid, formation of **a white precipitate which dissolves in the acid to form a colourless solution**indicates presence of Zn^{2+}, Al^{3+} orSn^{2+}. In this case, Pb^{2+} are excluded because apart from the reactions above, the free lead(II) ions formed when the solution turns just acidic, also undergo other important reactions where they combine with chloride ions from dilute hydrochloric

acid or sulphate ions from dilute sulphuric acid to form insoluble lead(II) chloride or lead(II) sulphate respectively, both of which appear as white precipitates.

$$Pb^{2+}(aq) + 2Cl^{-}(aq) \rightarrow \underset{\textit{white precipitate}}{PbCl_2(s)}$$

$$Pb^{2+}(aq) + SO_4^{2-}(aq) \rightarrow \underset{\textit{white precipitate}}{PbSO_4(s)}$$

This implies that iflead(II) ions were present in the filtrate, the white precipitate formed on addition of dilute hydrochloric acid or dilute sulphuric acid would never dissolve in the acid to form a colourless solution since lead(II) chloride and lead(II) sulphate do not react with acids.

Alternatively, on addition of the dilute mineral acid to the filtrate, **a green precipitate (grey-green precipitate) is formed which dissolves in the acidto form a green solution. This occurs if Cr^{3+} are present.** This is due to the following series of reactions:

$$\underset{\textit{Tetrahydroxochromate(III) ion}}{[Cr(OH)_4]^{-}(aq)} + H^{+}(aq) \rightarrow \underset{\textit{Green ppt of chromium(III) hydroxide}}{Cr(OH)_3(s)} + H_2O(l)$$

$$Cr(OH)_3(s) + 3H^{+}(aq) \rightarrow \underset{\textit{Green solution of chromium(III) ions}}{Cr^{3+}(aq)} + 3H_2O(l)$$

b) The cation from the second category forms a precipitate of the metal hydroxide that is insoluble in excess and will be present in the residue after filtration. The insoluble precipitate may be white (ifCa^{2+}, Mg^{2+}or $Ba^{2+}$are present), dirty white if Mn^{2+} are present, blue (ifCu^{2+}or Co^{2+} are present), pale green if Ni^{2+} are present, dirty green (if Fe^{2+} are present) or brown/reddish-brown (if Fe^{3+} are present).

$Ca^{2+}(aq) + 2OH^{-}(aq) \rightarrow Ca(OH)_2(s)$
$Mg^{2+}(aq) + 2OH^{-}(aq) \rightarrow Mg(OH)_2(s)$
$Ba^{2+}(aq) + 2OH^{-}(aq) \rightarrow Ba(OH)_2(s)$
$Mn^{2+}(aq) + 2OH^{-}(aq) \rightarrow Mn(OH)_2(s)$
$Cu^{2+}(aq) + 2OH^{-}(aq) \rightarrow Cu(OH)_2(s)$
$Ni^{2+}(aq) + 2OH^{-}(aq) \rightarrow Ni(OH)_2(s)$
$Fe^{2+}(aq) + 2OH^{-}(aq) \rightarrow Fe(OH)_2(s)$
$Fe^{3+}(aq) + 3OH^{-}(aq) \rightarrow Fe(OH)_3(s)$
$Co^{2+}(aq) + 2OH^{-}(aq) \rightarrow Co(OH)_2(s)$

2) With dilute ammonia solution

Dilute ammonia solution is added drop wise until in excess to a test solution containing two cations, one of which forms a white precipitate soluble in excess to form a colourless solution (if $Zn^{2+}$are present) or a pale blue precipitate soluble in excess to form a deep blue solution (if $Cu^{2+}$are present) while the second cation forms a precipitate insoluble in excess dilute ammonia solution (e.g Pb^{2+}, Al^{3+}, Mg^{2+},Mn^{2+}, Cr^{3+}, Fe^{2+}or Fe^{3+}). The addition of excess dilute ammonia solution is followed by filtration and to the filtrate, a dilute mineral acid such as dilute nitric acid, dilute hydrochloric acid or dilute sulphuric acid is added drop by drop while shaking the test tube with its contents until the solution is **"just acidic".**Only three cations (Zn^{2+}, Cu^{2+} and Ni^{2+}) form precipitates that dissolve in excess dilute ammonia solution whereby, on filtration, the filtrate can either be a colourless solution due to the tetraamminezinc(II) ion, a deep blue solution due to the tetraamminecopper(II) ion or a blue solution due to the hexaamminenickel(II) ion.

How does one tell that the filtrate has become just acidic?

When the dilute mineral acid is added to the filtrate, at first, a white precipitateis formed (if $Zn^{2+}$are present) or pale blue precipitate is formed (if $Cu^{2+}$are present) or a pale green precipitate is formed (if Ni^{2+} are present), but with continued addition of the acid drop by drop, while shaking the test tube with its contents, the white precipitate (if $Zn^{2+}$are present) dissolves in the acid to form a colourless solution,the pale blue precipitate (if $Cu^{2+}$are present) dissolves in the acid to form a blue solution or the

pale green precipitate dissolves in the acid to form a green solution (if Ni^{2+} are present) and at this point when the precipitate has just dissolved in the acid to form a solution, the solution is now **"just acidic".**

Explanation

The test solution contains one cation from this category of cations: Zn^{2+}, Cu^{2+}or $Ni^{2+}$and another from this category of cations: Pb^{2+}, Al^{3+}, Mg^{2+}, Ba^{2+}, Mn^{2+}, Co^{2+}, Cr^{3+}, Fe^{2+}or Fe^{3+}.

a) The cation from the first category forms **a precipitate with dilute ammonia solution, which dissolves in excess to form a solution** and therefore on filtration, this cation will be present in the filtrate in form of a soluble complex ion (in form of the tetraamminezinc(II) ion, tetraamminecopper(II) ion or hexaamminenickel(II) ion).

On addition of the dilute mineral acid to the filtrate**, a white precipitate is formed which dissolves in the acid to form a colourless solution (**if $Zn^{2+}$are present),**a pale blue precipitate is formed which dissolves in the acid to form a blue solution** (if $Cu^{2+}$are present)or a pale green precipitate soluble in the acid to form green solution (if $Ni^{2+}$are present). This is due to the following series of reactions.

i) For Zn^{2+},

$$\underset{\text{Tetraamminezinc(II) ion}}{[Zn(NH_3)_4]^{2+}(aq)} + 2H^+(aq) + 2H_2O(l) \rightarrow \underset{\text{White ppt of zinc hydroxide}}{Zn(OH)_2(s)} + 4NH_4^+(aq)$$

$$Zn(OH)_2(s) + 2H^+(aq) \rightarrow \underset{\text{Colourless solution of zinc ions}}{Zn^{2+}(aq)} + 2H_2O(l)$$

ii) For Cu^{2+},

$$\underset{\text{Tetraamminecopper(II) ion}}{[Cu(NH_3)_4]^{2+}(aq)} + 2H^+(aq) + 2H_2O(l) \rightarrow \underset{\text{Pale blue precipitate of copper(II) hydroxide}}{Cu(OH)_2(s)} + 4NH_4^+(aq)$$

$$Cu(OH)_2(s) + 2H^+(aq) \rightarrow \underset{\text{Blue solution of copper(II) ions}}{Cu^{2+}(aq)} + 2H_2O(l)$$

White precipitate of barium hydroxide

iii) For Ni^{2+},

$$\underset{\text{Hexaamminenickel(II) ion}}{[Ni(NH_3)_6]^{2+}(aq)} + 4H^+(aq) + 2H_2O(l) \rightarrow \underset{\text{Pale green precipitate of nickel(II) hydroxide}}{Ni(OH)_2(s)} + 6NH_4^+(aq)$$

$$Ni(OH)_2(s) + 2H^+(aq) \rightarrow \underset{\text{Green solution of nickel(II) ions}}{Ni^{2+}(aq)} + 2H_2O(l)$$

b) The cation from the second category forms a precipitate of the metal hydroxide that is insoluble in excess dilute ammonia solution and will be present in the residue after filtration. The insoluble precipitate may be white (ifPb^{2+}, Al^{3+},Sn^{2+}, Mg^{2+},$Ba^{2+}$are present), dirty white (if Mn^{2+} are present), blue but turns pink on standing (if Co^{2+} are present),green/grey-green (if Cr^{3+} are present), dirty green (if Fe^{2+} are present) or brown/reddish-brown (if Fe^{3+} are present).

$Pb^{2+}(aq) + 2OH^-(aq) \rightarrow Pb(OH)_2(s)$
$Al^{3+}(aq) + 3OH^-(aq) \rightarrow Al(OH)_3(s)$
$Mg^{2+}(aq) + 2OH^-(aq) \rightarrow Mg(OH)_2(s)$
$Ba^{2+}(aq) + 2OH^-(aq) \rightarrow Ba(OH)_2(s)$
$Sn^{2+}(aq) + 2OH^-(aq) \rightarrow Sn(OH)_2(s)$
$Mn^{2+}(aq) + 2OH^-(aq) \rightarrow Mn(OH)_2(s)$

$Co^{2+}(aq) + 2OH^-(aq) \rightarrow Co(OH)_2(s)$
$Cr^{3+}(aq) + 3OH^-(aq) \rightarrow Cr(OH)_3(s)$
$Fe^{2+}(aq) + 2OH^-(aq) \rightarrow Fe(OH)_2(s)$
$Fe^{3+}(aq) + 3OH^-(aq) \rightarrow Fe(OH)_3(s)$

Table showing a summary of preliminary and confirmatory tests forcommon cations

Cation	Preliminary/ Confirmatory tests	Test procedure	Observation
Zn^{2+}	**Preliminary tests**	Heat two spatula endfuls of the solid in a dry test tube strongly until there is no further change.	The solid turns into a yellow residue when hot, turns white on cooling.
		To the test solution, add dilute sodium hydroxide solution drop wise until in excess.	A white precipitate soluble in excess to form a colourless solution.
		To the test solution, add dilute ammonium hydroxide solution drop wise until in excess.	A white precipitate soluble in excess to form a colourless solution.
		To the test solution, add sodium carbonate solution drop wise until in excess.	A white precipitate insoluble in excess.
	Confirmatory test	To the test solution, add solid ammonium chloride followed by 3-4 drops of disodium hydrogenphosphate solution and then dilute ammonia solution drop wiseuntil in excess.	A white precipitate soluble in excess ammonia solution to form a colourless solution.
		To the test solution, add potassium hexacyanoferrate(II) solution.	A white precipitate is formed.
Pb^{2+}	**Preliminary tests**	Heat two spatula endfuls of the solid in a dry test tube strongly until there is no further change.	The solid turns into a reddish-brown residue when hot, turns yellow on cooling.
		To the test solution, add dilute sodium hydroxide solution drop wise until in excess.	A white precipitate soluble in excess to form a colourless solution.
		To the test solution, add dilute ammonium hydroxide solution drop wise until in excess.	A white precipitate insoluble in excess
		To the test solution, add sodium carbonate solution drop wise until in excess.	A white precipitate insoluble in excess.
		To the test solution, add 1-2 drops of dilute hydrochloric acid solution and warm; then cool under running tap water. **Note:** Sodium chloride solution can also be used in place of dilute hydrochloric acid in this test.	A white precipitate which dissolves on warming, precipitate reappears on cooling.
		To the test solution, add 1-3 drops of dilute sulphuric acid solution. **Note:**Sodium sulphate can also be used in place of dilute sulphuric acid for the same purpose in this test.	A white precipitate is formed.
	Confirmatory tests	To the test solution, add 2–3 drops of potassium iodide solution.	A yellow precipitate is formed.

		To the test solution, add potassium iodide solution drop wise until in excess.	A yellow precipitate soluble in excess to form a colourless solution.
		To the test solution, add potassium chromate solution followed by dilute sodium hydroxide solution until in excess.	A yellow precipitate soluble in excess sodium hydroxide solution.
		To the test solution, add potassium chromate solution followed by dilute hydrochloric acid.	A yellow precipitate insoluble in the acid.
		To the test solution, add concentrated hydrochloric acid drop wise until in excess.	A white precipitate soluble in excess to form a pale yellow solution.
Al^{3+}	**Preliminary tests**	Heat two spatula endfuls of the solid in a dry test tube strongly until there is no further change.	The solid forms a white residue.
		To the test solution, add dilute sodium hydroxide solution drop wise until in excess.	A white precipitate soluble in excess to form a colourless solution.
		To the test solution, add dilute ammonium hydroxide solution drop wise until in excess.	A white precipitate insoluble in excess.
		To the test solution, add sodium carbonate solution drop wise until in excess.	A white precipitate insoluble in excess with effervescence of a colourless gas which turns moist blue litmus paper red and limewater milky.
		To the test solution, add 1–2 drops of Potassium iodide solution.	No observable change.
	Confirmatory test	To the test solution, add 2-3drops of dilute nitric acid are added, followed by 3-4 drops of litmus solution and then dilute ammonia solution drop wise until in excess.	A blue lake solution is formed.
		To the test solution, add 2drops of litmus solution, followed by about 1cm^3 of dilute hydrochloric acid and then dilute ammonia solution until the solution is just alkaline, then add Alizarin Reagent.	A pink colouration is formed.
Sn^{2+}	**Preliminary tests**	Heat two spatula endfuls of the solid in a dry test tube strongly until there is no further change.	The solid forms a white residue.
		To the test solution, add dilute sodium hydroxide solution drop wise until in excess.	A white precipitate soluble in excess to form a colourless solution.
		To the test solution, add dilute ammonium hydroxide solution drop wise until in excess.	A white precipitate insoluble in excess.
	Confirmatory tests	To the test solution, add acidified potassium manganate(VII) solution.	The purple solution turns colourless.

		To the test solution, add iron(III) chloride solution.	The brown/yellow solution turns green.
NH_4^+	**Preliminary test**	Heat two spatula endfuls of the solid in a dry test tube strongly until there is no further change.	A colourless, pungent, chocking gas, which turns moist red litmus paper blue and forms dense white fumes with concentrated hydrochloric acid, is evolved. A white sublimate is formed.
	Confirmatory tests	To the test solution, add dilute sodium hydroxide solution drop wise until in excess and then warm.	No observable change, but on warming, a colourless pungent, chocking gas, which turns moist red litmus paper blue and forms dense white fumes with concentrated hydrochloric acid is evolved.
		Grind half a spatula endful of the solid with soda lime using the bottom of a test tube.	A colourless, pungent, chocking gas, which turns moist red litmus paper blue and forms dense white fumes with concentrated hydrochloric acid, is evolved.
Ca^{2+}	**Preliminary tests**	Heat two spatula endfuls of the solid in a dry test tube strongly until there is no further change.	The solid forms a white residue.
		To the test solution, add sodium sulphate solution (or dilute sulphuric acid).	A white precipitate is formed.
		To the test solution, add sodium hydroxide solution drop wise until in excess.	A white precipitate insoluble in excess.
		To the test solution, add dilute ammonium hydroxide solution drop wise until in excess.	No observable change
		To the test solution, add sodium carbonate solution drop wise until in excess.	A white precipitate insoluble in excess.
	Confirmatory tests	To the test solution, add ammonium oxalate solution followed by dilute hydrochloric acid/nitric acid.	A white precipitate soluble in the acid.
		To the test solution, add ammonium oxalate solution followed by ethanoic acid.	A white precipitate insoluble in ethanoic acid.
		To the test solution, add potassium chromate solution followed by excess ethanoic acid.	A yellow precipitate soluble in excessethanoic acid.
		To the test solution, add potassium chromate solution followed by dilute sodium hydroxide solution drop wise until in excess.	A yellow precipitate soluble in excess dilute sodium hydroxide solution.
Mg^{2+}	**Preliminary tests**	Heat two spatula endfuls of the solid in a dry test tube strongly until there is no further change.	The solid forms a white residue.

		To the test solution, add dilute sodium hydroxide solution drop wise until in excess.	A white precipitate insoluble in excess.
		To the test solution, add dilute ammonium hydroxide solution drop wise until in excess.	A white precipitate insoluble in excess.
		To the test solution, add Sodium carbonate solution drop wise until in excess.	A white precipitate insoluble in excess.
		To the test solution, add sodium sulphate solution (or dilute sulphuric acid).	No observable change.
	Confirmatory test	To the test solution, add solid ammonium chloride followed by 3-4 drops of disodium hydrogenphosphate solution and then dilute ammonia solution drop wise until in excess.	A white precipitate insoluble in excess ammonia solution.
Ba^{2+}	**Preliminary tests**	Heat two spatula endfuls of the solid in a dry test tube strongly until there is no further change.	The solid forms a white residue.
		To the test solution, add dilute sodium hydroxide solution drop wise until in excess.	A white precipitate insoluble in excess.
		To the test solution, add dilute ammonium hydroxide solution drop wise until in excess.	A white precipitate insoluble in excess.
		To the test solution, add sodium carbonate solution drop wise until in excess.	A white precipitate is insoluble in excess.
		To the test solution, add sodium sulphate solution (or dilute sulphuric acid).	A white precipitate is formed.
	Confirmatory tests	To the test solution, add ammonium oxalate solution followed by dilute hydrochloric acid/nitric acid.	A white precipitate soluble in the acid.
		To the test solution, add potassium chromate solution, followed by dilute sodium hydroxide solution drop wise until in excess.	A yellow precipitate insoluble in excess Sodium hydroxide solution.
		To the test solution, add potassium chromate solution, followed by dilute hydrochloric acid and then dilute sulphuric acid.	A yellow precipitate soluble in dilute hydrochloric acid, precipitate reappears on addition of dilute sulphuric acid.
		To the test solution, add ammonium oxalate solution followed by ethanoic acid.	A white precipitate soluble in ethanoic acid.
Cu^{2+}	**Preliminary tests**	Heat two spatula endfuls of the solid in a dry test tube strongly until there is no further change.	The blue/green solid turns into a black residue.
		To the test solution, add sodium carbonate solution drop wise until in excess.	A green precipitate insoluble in excess **or** blue precipitate insoluble in excess.

		To the test solution, add dilute sodium hydroxide solution drop wise until in excess (and heat).	A pale blue precipitate insoluble in excess (turns black on heating). **Note:** The black solid is copper(II) oxide.
		To the test solution, add a little zinc powder and leave to stand. **Note:** Magnesium powder can also be used in place of zinc powder.	A brown solid is formed. Blue solution turns colourless. **Note:** The brown solid (reddish-brown solid) is copper metal, formed by the displacement reaction (also redox reaction) below. $Cu^{2+}(aq)+Zn(s) \rightarrow Cu(s)+Zn^{2+}(aq)$ The colourless solution is due to $Zn^{2+}(aq)$.
	Confirmatory tests	To the test solution, add dilute ammonium hydroxide solution drop wise until in excess.	A pale blue precipitate soluble in excess to form a deep blue solution.
		To the test solution, add 3–4 drops of potassium iodide solution.	A white precipitate in a brown solution (**OR** a white precipitate stained brown).
		To the test solution, add 3–4 drops of potassium iodide solution followed by sodium thiosulphate solution.	A white precipitate in a brown solution, brown solution turns colourless but white precipitate remains on addition of sodium thiosulphate solution.
		To the test solution, add 2–3drops of potassium hexacyanoferrate(II) solution.	A brown precipitate is formed.
		To the test solution, add excess concentrated hydrochloric acid.	A yellow solution is formed.
Fe^{2+}	**Preliminary tests**	Heat two spatula endfuls of the solid in a dry test tube strongly until there is no further change.	The pale green solid turns into a brown residue.
		To the test solution, add dilute sodium hydroxide solution drop wise until in excess.	A dirty green precipitate insoluble in excess, turns brown on standing in air.
		To the test solution, add dilute ammonium hydroxide solution drop wise until in excess.	A dirty green precipitate insoluble in excess, turns brown on standing in air.
		To the test solution, add sodium carbonate solution drop wise until in excess.	A dirty green precipitate insoluble in excess, turns brown on standing in air.
	Confirmatory tests	To the test solution, add 3-4 drops of potassium hexacyanoferrate(III) solution.	A dark blue precipitate is formed.
		To the test solution, add concentrated nitric acid and warm.	Pale green solution turns brown.

		To the test solution, add hydrogen peroxide solution and warm.	Pale green solution turns brown.
Fe^{3+}	**Preliminary tests**	Heat two spatula endfuls of the solid in a dry test tube strongly until there is no further change.	The brown/yellow solid forms a brown residue.
		To the test solution, add dilute sodium hydroxide solution drop wise until in excess.	A brown/reddish-brown/rusty brown precipitate insoluble in excess.
		To the test solution, add dilute ammonium hydroxide solution drop wise until in excess.	A brown/reddish-brown/rusty brown precipitate insoluble in excess
		To the test solution, add sodium carbonate solution drop wise until in excess.	A brown/reddish-brown/rusty brown precipitate insoluble in excess with effervescence of a colourless gas which turns moist blue litmus paper red and limewater milky.
	Confirmatory tests	To the test solution, add 3-4 drops of Potassium hexacyanoferrate(II) solution.	A dark blue precipitate is formed.
		To the test solution, add ammonium thiocyanate solution **or** potassium thiocyanate solution.	A deep red solution is formed.
Ni^{2+}	**Preliminary tests**	Heat two spatula endfuls of the solid in a dry test tube strongly until there is no further change.	The pale green solid forms a black residue.
		To the test solution, add dilute sodium hydroxide solution drop wise until in excess.	A pale green precipitate insoluble in excess.
		To the test solution, add dilute ammonium hydroxide solution drop wise until in excess.	A pale green precipitate soluble in excess to form a blue solution.
		To the test solution, add sodium carbonate solution drop wise until in excess.	A pale green precipitate insoluble in excess.
	Confirmatory tests	To the test solution, add a little dilute ammonia solution followed by dimethyl glyoxime solution.	A bright red precipitate/red precipitate/pink precipitate is formed.
		To the test solution, add a few drops of potassium hexacyanoferrate(III) solution.	A brown precipitate is formed.
Cr^{3+}	**Preliminary tests**	To the test solution, add dilute Sodium hydroxide solution drop wise until in excess.	A green precipitate (grey-green precipitate) soluble in excess to form a green solution.
		To the test solution, add dilute ammonium hydroxide solution drop wise until in excess.	A green precipitate(grey-green precipitate) insoluble in excess.
		To the test solution, add sodium carbonate solution drop wise until in excess.	A green precipitate (blue-green precipitate)insoluble in excess with effervescence of a colourless gas which turns moist blue litmus paper red and limewater milky.

	Confirmatory tests	To the test solution, add dilute sodium hydroxide solution drop wise until in excess, followed by hydrogen peroxide solution and warm.	A green precipitate soluble in excess sodium hydroxide solution to form a green solution, turns yellow on addition of hydrogen peroxide solution.
		To the test solution, add excess sodium hydroxide solution, followed by hydrogen peroxide solution and warm, then add amylalcohol followed by dilute sulphuric acid.	A green solution which turns yellow on warming, and then a blue solution in the organic layer.
		To the test solution, add lead(II) nitrate or lead(II) ethanoate solution followed by dilute sodium hydroxide solution drop wise until in excess.	A yellow precipitate soluble in excess sodium hydroxide solution.
Mn^{2+}	**Preliminary tests**	To the test solution, add dilute sodium hydroxide solution drop wise until in excess.	A dirty white precipitate insoluble in excess, turns brown on standing.
		To the test solution, add dilute ammonium hydroxide solution drop wise until in excess.	A dirty white precipitate insoluble in excess, turns brown on standing.
		To the test solution, add sodium carbonate solution drop wise until in excess.	A dirty white precipitate insoluble in excess, turns brown on standing.
	Confirmatory tests	To the test solution, add a few drops of concentrated nitric acid followed bya little solid sodium bismuthate and warm gently.	A purple solution is formed.
		To the test solution, add a few drops of concentrated nitric acid followed by lead(IV) oxide and warm gently.	A purple solution is formed.
		To the test solution, add a few drops of hydrogen peroxide solution followed by dilute sodium hydroxide solution drop wise until in excess.	A dark brown precipitate with rapid effervescence of a colourless gas that relights a glowing splint.
		Fuse/meltthe unknown solid with a mixture of solid sodium carbonate and potassium nitrate.	A green mass is formed.
Co^{2+}	**Preliminary tests**	Heat two spatula endfuls of the solid in a dry test tube strongly until there is no further change.	The solid forms a black residue.
		To the test solution, add dilute sodium hydroxide solution drop wise until in excess.	A blue precipitate insoluble in excess, turns pink on standing in air, turns brown on further standing in air.
		To the test solution, add dilute ammonium hydroxide solution drop wise until in excess.	A blue precipitate insoluble in excess (it may turn pink on standing and may turn brown on further standing in air).
		To the test solution, add sodium carbonate solution drop wise until in excess.	Pink-violet precipitate insoluble in excess.

	Confirmatory tests	To the test solution, add solid ammonium thiocyanate followed by amyl alcohol.	A blue solution in the organic layer.
		To the test solution, add concentrated ammonia solution drop wise until in excess followed by a few drops of hydrogen peroxide solution.	A green precipitate soluble in excess concentrated ammonia solution to form a brown solution and rapid effervescence of a colourless gas on addition of hydrogen peroxide solution.
		To the test solution, add concentrated hydrochloric acid, followed by solid ammonium thiocyanate and then pentanol (amylalcohol) and shake gently.	A blue solution in the organic layer(blue solution in upper layer) and a purple solution in the inorganic layer(purple solution in the lower layer).
		To the test solution, add excess concentrated hydrochloric acid.	A blue solution is formed.

8.3.2Reagents used in the identification of Anions in Solids and Solutions

The major reagents used in detection of anions in solution include: dilute hydrochloric acid, dilute nitric acid, dilute sulphuric acid, concentrated nitric acid, concentrated sulphuric acid, freshly prepared iron(II) sulphate solution, lead(II) nitrate solution/lead(II) acetate solution, barium nitrate solution/barium chloride solution, acidified silver nitrate solution, silver nitrate solution with excess ammonia solution, aluminium metal, zinc metal, acidified potassium manganate(VII) solution, ethanol, chloroform, iodine solution,magnesium sulphate solution/magnesium nitrate solution/magnesium chloride solution and many other reagents.

a) Action of dilute hydrochloric acid

The dilute hydrochloric acid may be either added to a solid or to a test solution. In case there is no reaction at room temperature, some gentle warming may be required.

Test	Observation	Deduction
i) To the unknown solid, add $2cm^3$ of dilute hydrochloric acid.	Effervescence of a colourless gas which turns moist blue litmus paper pink/red and limewater milky(forms a white precipitate with calcium hydroxide solution).	$CO_2(g)$ evolved CO_3^{2-} (or HCO_3^-) present.
ii)To the unknown solid, add $2cm^3$ of dilute hydrochloric acid and warm.	No observable change at room temperature, but on warming, there is evolution of a colourless, pungent gas which turns moist blue litmus paper red and bleaches it, and turns acidified potassium dichromate(VI) solution from orange to green/turns acidified potassium manganate(VII) solution from purple to colourless.	$SO_2(g)$ evolved. SO_3^{2-} present.

iii) To the test solution, add $2cm^3$ of dilute hydrochloric acid and warm.	Cream precipitate/yellow precipitate; On warming, there is evolution of a colourless, pungent gas which turns moist blue litmus paper red and bleaches it, and turns acidified potassium dichromate(VI) solution from orange to green/turns acidified potassium manganate(VII) solution from purple to colourless.	S(s) precipitated. $SO_2(g)$ evolved. $S_2O_3^{2-}$ confirmed.present
iv)To the unknown solid, add $2cm^3$ of dilute hydrochloric acid and warm.	No observable change even on warming	SO_4^{2-} probably present

b) Action of dilute nitric acid

Just like dilute hydrochloric acid, dilute nitric acid can be effectively used to test for the presence of carbonate ions, CO_3^{2-} as in test (a)(i) above.

Note: Dilute nitric acid can also be used to confirm the presence of the chloric(I) ion/hypochlorous ion, ClO^-.Addition of dilute nitric acid to a test solution containing ClO^-, results in evolution of chlorine gas.

Test	Observation	Deduction
To the test solution, add dilutenitric acid.	A greenish-yellow gas which turns moist blue litmus paper red and bleaches it.	ClO^- confirmed present

c) Action of dilute sulphuric acid

Similarly, dilute sulphuric acid can also be used effectively to test for the presence of carbonate ions, CO_3^{2-} as in test (a)(i) above.However, in case dilute sulphuric acid is added to a test solution and a white precipitate is formed, then Pb^{2+}, Ca^{2+} or Ba^{2+}ions are likely to be present in the compound under analysis. Therefore, dilute sulphuric acid is used to test for carbonates usually when the cations present in the sample under analysis are neither Pb^{2+}, Ca^{2+}nor Ba^{2+}.

Apart from testing for carbonate ions, dilute sulphuric acid can also be used to test for the presence of nitrite ions, NO_2^-. To a cold solution suspected to contain nitrite ions, iron(II) sulphate solution is added followed by dilutesulphuric acid. Formation of a dark brown complex confirms thepresence of the NO_2.

Test	Observation	Deduction
To the cold test solution, add iron(II) sulphate solution followed by dilute sulphuric acid.	A dark brown complex is formed.	NO_2^- confirmed present

Dilute sulphuric acid, when used together with hydrogen peroxide solution, can be used to confirm the presence of the chromate(VI) ion, CrO_4^{2-}.

Test	Observation	Deduction
To the test solution, add dilute sulphuric acid followed by hydrogen peroxide solution.	Yellow solution turns orange and then to an intense blue solution which quickly fades, leaving behind a green solution.	CrO_4^{2-}confirmed present

d) Action of concentrated sulphuric acid

Concentrated sulphuric acid is mainly added to unknown solids rather than test solutions. In this way, a few drops (2 to 3 drops) of the concentrated sulphuric acid are added to the solid in a test tube. Concentrated sulphuric acid is used to test for anions such as CO_3^{2-}, Cl^-, Br^-, I^-,$CH_3COO^-$and SO_3^{2-}. Unlike the CO_3^{2-} which reacts with the concentrated sulphuric acid at room temperature, the rest only react when addition of the concentrated sulphuric acid is followed by gentle warming. In each case, a gas or a mixture of gases is given off/evolved.The gas or mixture of gases evolved depends on the type of anion present in the solid.

For a CO_3^{2-}, carbon dioxide gas is given off, for the Cl^-, hydrogen chloride gas is evolved, for the Br^-, bromine gas and hydrogen bromide gas are evolved, for the I^-, Iodine vapour and hydrogen iodide gas are given off, for CH_3COO^-, acetic acid fumes are given off while for SO_3^{2-}, sulphur dioxide gas is given off.

Concentrated sulphuric acid can be added to a test solution, particularly when used together with a freshly prepared solution of iron(II) sulphate while testing for the nitrate ions**(The brown ring test).**To the test solution in a test tube, add a freshly prepared solution of iron(II) sulphate. In the brown ring test, the test tube containing the solution suspected to contain a nitrate is held in a slanting position and then concentrated sulphuric acid added drop by drop down the walls of the test tube. Formation of a brown ring at the interface of the acid and aqueous layer confirms the presence of a nitrate. Avoid shaking. In the brown ring test, it is important to avoid boiling because this might turn out to be explosive hence causing accidents. The brown ring formed is of a complex ion formed as shown below.

$$3Fe^{2+}(aq) + NO_3^-(aq) + 4H^+(aq) \rightarrow \underset{\{Brown\ ring\}}{3Fe^{2+}(aq)+NO(g)+2H_2O(l)}$$

Note:

1)In a similar way, addition of a few drops of concentrated sulphuric acid to a solid containing a nitrate, NO_3^-, followed by gentle warming, results in evolution of brown fumes of nitrogen dioxide gas.

2) Also, addition of a few copper turnings to a solution containing a nitrate, NO_3^-, followed by a few drops of concentrated sulphuric acid and gentle warming, results in evolution of brown fumes of nitrogen dioxide gas.

3)When to a solid containing a chloride, manganese(IV) oxide is added followed by a few drops (2-3 drops) of concentrated sulphuric acid, chlorine gas is evolved (chlorine gas is a greenish-yellow gas).

Test	Observation	Deduction
To the unknown solid, add 3 to5 drops of concentrated sulphuric and warm gently.	Rapid effervescence of a colourless gas which turns moist blue litmus paper red/pink and turns limewater milky(forms a white precipitate with calcium hydroxide solution).	$CO_2(g)$ evolved CO_3^{2-} confirmed present.
	Effervescence of misty fumes with a chocking smell, which turn moist blue litmus paper red and form dense white fumes with concentrated ammonia.	HCl(g) evolved Cl^- suspected.

	Effervescence of a brown vapour which turns moist blue litmus paper red and bleaches it. White fumes which turn moist blue litmus paper red.	$Br_2(g)$ evolved. HBr(g) evolved. Br^- suspected.
	Effervescence of a purple vapour which turns moist blue litmus paper red and sublimes to form a black/purple/purplish-black solid. White fumes which turn moist blue litmus paper red.	$I_2(g)$ HI(g) I^- suspected.
	Effervescence of white fumes with a sharp vinegar smell and turn moist blue litmus paper red.	Acetic acid fumes CH_3COO^- suspected.
	Effervescence of a colourless gas with a pungent smell, which turns moist blue litmus paper red and turns acidified potassium dichromate(VI) solution from orange to green/turns acidified potassium manganate(VII) solution from purple to colourless.	SO_2 (g) evolved. SO_3^{2-} suspected.
	Brown fumes with a pungent smell, which turn moist blue litmus paper red.	$NO_2(g)$ evolved. NO_3^- suspected.
To the unknown solid, add half a spatula endful of manganese(IV) oxide followed by 2-3 drops of concentrated sulphuric acid and warm gently.	Effervescence of a greenish-yellow gas which turns moist blue litmus paper red and bleaches it.	$Cl_2(g)$ evolved. Cl^- suspected.
	Effervescence of a brown vapour which turns moist blue litmus paper red and bleaches it.	$Br_2(g)$ evolved. Br^- suspected.
	Effervescence of a purple vapour which turns moist blue litmus paper red and subimes to form a black/purple/purplish-black solid.	$I_2(g)$ evolved. I^- suspected.
To the test solution, add a few pieces of copper turnings followed by 3-4 drops of concentrated sulphuric acid and warm gently.	Brown fumes with a pungent smell, which turn moist blue litmus paper red.	$NO_2(g)$ evolved NO_3^- suspected
To the test solution in a test tube, add a freshly prepared solution of iron(II) sulphate. Hold the test tube in a slanting position and then add concentrated sulphuric acid drop wise down the walls of the test tube.(Avoid shaking).	A brown ring is formed at the interface of the acid and aqueous layer.	NO_3^- confirmed present

e) Action of Devarda's Alloy

Devarda's Alloy is a uniform mixture of zinc, aluminium and copper.

To the test solution suspected to contain a nitrate, Devarda's Alloy is added followed by excess dilute sodium hydroxide solution and boil the mixture. Evolution of ammonia gas confirms the presence of a nitrate ion, NO_3^-.

The zinc metal in Devarda's alloy reacts according to the equation below.

$4Zn(s) + NO_3^-(aq) + 7OH^-(aq) + 6H_2O(l) \rightarrow 4Zn(OH)_4^{2-}(aq) + NH_3(g)$

Test	Observation	Deduction
To the test solution, add Devarda's Alloy followed by excess dilute sodium hydroxide solution and boil.	A colourless, pungent, chocking gas which turns moist red litmus paper blue and forms dense white fumes with concentrated hydrochloric acid.	$NH_3(g)$ evolved NO_3^- confirmed present

Note: In case Devarda's Alloy is not available, either zinc metal powder or aluminium metal powder is added to the test solution followed by excess dilute sodium hydroxide solution and the mixture warmed. Evolution of ammonia gas confirms the presence of a nitrate.

Test	Observation	Deduction
To the test solution, add zinc metal powder **or** aluminium metal powder followed by excess dilute sodium hydroxide solution and warm.	A colourless, pungent, chocking gas which turns moist red litmus paper blue and forms dense white fumes with concentrated hydrochloric acid.	$NH_3(g)$ evolved NO_3^- confirmed present

Note: Thenitrite ion, NO_2^- can also be tested for in a similar way using Devarda's Alloy or alternatively, zinc metal powder or aluminium metal powder whereby the observation is the same (i.e. ammonia gas is given off) just like it is in the test for a nitrate, NO_3^-.

f) Action of lead(II) nitrate solution (or lead(II) acetate solution)

Lead(II) nitrate solution **(or** lead(II) acetate solution) is used to test for sulphates, sulphites, oxalates, chlorides, bromides, iodides,carbonates and hypochlorites. The solution may be used in isolation or in combination with dilute nitric acid.

Test	Observation	Deduction
To the test solution, add lead(II) nitrate solution.	A white precipitate.	SO_4^{2-}, SO_3^{2-}, Cl^-, Br^-, $C_2O_4^{2-}$, CO_3^{2-}, PO_4^{3-}, ClO^-.
To the test solution, add lead(II) nitrate solution	A yellow precipitate.	I^-, CrO_4^{2-}
To the test solution, add lead(II) nitrate solution and warm (then cool). **Note:** Cooling is optional.	A white precipitate soluble on warming to form a colourless solution (precipitate reappears on cooling).	Cl^-
To the test solution, add lead(II) nitrate solution and warm.	A white precipitate insoluble on warming.	SO_4^{2-}, SO_3^{2-}

To the test solution, add lead(II) nitrate solution, followed by dilute nitric acid.	A white precipitate insoluble in the acid.	SO_4^{2-}, Cl^-, Br^-
To the test solution, add lead(II) nitrate solution, followed by dilute nitric acid.	A white precipitate soluble in the acid with effervescence of a colourless gas which turns moist blue litmus paper pink/red and limewater milky (forms a white precipitate with calcium hydroxide solution).	CO_2 (g) evolved. CO_3^{2-} present.
To the test solution, add lead(II) nitrate solution, followed by dilute nitric acid.	A white precipitate soluble in the acid without effervescence.	SO_3^{2-}, $C_2O_4^{2-}$, PO_4^{3-}

g) Action of Barium nitrate solution

Barium nitrate solution may be used in isolation e.g. in testing for CrO_4^{2-}. In most cases, however, Barium nitrate solution isused together with dilute nitric acid. The test procedure is such that 2 to 3drops of Barium nitrate solution are added to the test solution of the unknownfollowed by a few drops of dilute nitric acid. When combined with dilute nitric acid, Barium nitrate solution is used to test for SO_4^{2-}, SO_3^{2-}, $C_2O_4^{2-}$, CO_3^{2-}, PO_4^{3-}.

Note: The above combination, however, is mainly used to confirm the presence of a SO_4^{2-}.

Test	Observation	Deduction
To the test solution, add Barium nitrate solution.	A yellow precipitate is formed.	CrO_4^{2-} confirmed present.
To the test solution, add Barium nitrate solution followed by dilute nitric acid.	A white precipitate, insoluble in the acid.	SO_4^{2-} confirmed present.
To the test solution, add dilute nitric acid followed by Barium nitrate solution.	A white precipitate is formed.	SO_4^{2-} confirmed present
To the test solution, add Barium nitrate solution followed by dilute nitric acid.	A white precipitate soluble in dilute nitric acidwithout effervescence.	SO_3^{2-}, $C_2O_4^{2-}$, PO_4^{3-}
To the test solution, add Barium nitrate solution followed by dilute nitric acid.	A white precipitate soluble in the acid with effervescence of a colourless gas which turns moist blue litmus paper red/pink and limewater milky(forms a white precipitate with calcium hydroxide solution).	CO_2 (g) given off CO_3^{2-} present
To the test solution, add Barium nitrate solution followed by dilute nitric acid.	A yellow precipitate which dissolves in the acid to form an orange solution.	CrO_4^{2-}

To the test solution, add Barium nitrate solution followed by dilute nitric acid.	No observable change.	Cl^-, $S_2O_3^{2-}$

h) Action of Barium chloride solution

Barium chloride solution is usually used together with dilute hydrochloric acid. This combination is basically used to test for the same anions as in part (f) above except that it can not be used to test for the chloride since the dilute hydrochloric acid itself contains a chloride. Otherwise, the observations and deductions are the same as in (f) above only that in case a SO_3^{2-} is present, addition of Barium chloride solution followed by dilute hydrochloric acid gives a white precipitate soluble in the acid with evolution of a colourless, pungent gas which turns moist blue litmus paper red and turns acidified potassium dichromate solution from orange to green. (The gas evolved is Sulphur dioxide).

i)Action ofsilver nitrate solution with dilute nitric acid

Silver nitrate solution can be used in isolation (alone) but in most cases, it is used when acidified. Silver nitrate is usually acidified using dilute nitric acid. This combination of reagents is used to test for the presence of Cl^-, Br^-, I^-, SO_3^{2-}, $C_2O_4^{2-}$, CO_3^{2-}, CrO_4^{2-}, PO_4^{3-}, e.t.c. The silver nitrate solution may be added first followed by the acid or the acid may be added first followed by silver nitrate solution.

Test	Observation	Deduction
To the test solution, add a few drops of silver nitrate solution followed by dilute nitric acid.	A white precipitate insoluble in the acid.	Cl^- confirmed present
	A cream precipitate insoluble in the acid.	Br^-
	A pale yellow precipitate insoluble in the acid.	I^-
	A pale yellow precipitate soluble in the acid without effervescence.	PO_4^{3-}
	A white precipitate dissolves in the acid with effervescence of a colourless gas which turns moist blue litmus red and limewater milky(forms a white precipitate with calcium hydroxide solution).	CO_2(g) evolved CO_3^{2-} present
	A white precipitate soluble in the acid without effervescence.	$C_2O_4^{2-}$
	A red precipitate soluble in the acid with no effervescence to form an orange solution.	CrO_4^{2-}
	No observable change.	SO_4^{2-}
To the test solution, add dilute nitric acid followed by a few drops of silver nitrate solution.	A white precipitate is formed.	Cl^- confirmed present
	A pale yellow precipitate is formed.	I^-
	A cream precipitate (very pale yellow precipitate).	Br^-
	No observable change.	SO_4^{2-}

j) Action of silver nitrate solution with excess ammonia solution

Test	Observation	Deduction
To the test solution, add a few drops of silver nitrate solution followed by ammonia solution drop wise until in excess.	A white precipitate soluble in excess ammonia solution, forming a colourless solution.	Cl^- confirmed present
	A pale yellow precipitate insoluble in excess ammonia solution.	I^-
	A cream precipitate dissolves with difficulty in excess ammonia solution.	Br^-
	A white precipitate solublein excess ammonia solution.	$C_2O_4^{2-}$, CO_3^{2-}
	A white precipitate insoluble in excess ammonia solution.	SO_3^{2-}
	A red precipitate soluble in excess ammonia solution to form a yellow solution.	CrO_4^{2-}
	No observable change.	SO_4^{2-}

Note: When to a solution containing Cl^- ions, silver nitrate solution is added, followed by excess ammonia solution, the sequence of chemical reactionsthat occur is as shown below:

$$Ag^+(aq) + Cl^-(aq) \rightarrow AgCl(s)$$

White precipitate of silver chloride

$$AgCl(s) + 2NH_3(aq) \rightarrow [Ag(NH_3)_2]^+(aq) + Cl^-(aq)$$

Colourless solution of the diamminesilver(I) ion

k) Action of magnesium sulphate/magnesium nitrate/magnesium chloride solution

All the three magnesium salts indicated above are soluble salts. Any of them can be used to differentiate between a soluble carbonate from a soluble hydrogencarbonate.

Test	Observation	Deduction
To the test solution, add magnesium sulphate solution.	A white precipitate is formed.	CO_3^{2-} present
	No observable change.	HCO_3^- present

The observations above are based on the fact that in case the carbonate ion is present in the test solution, the magnesium ions react with the carbonate to form the insoluble magnesium carbonate which appears as a white precipitate.

$$Mg^{2+}(aq) + CO_3^{2-}(aq) \rightarrow MgCO_3(s)$$

White precipitate

In case the Hydrogencarbonate ion is present in the test solution, the Magnesium ions react with the hydrogencarbonate ions to form soluble Magnesium hydrogencarbonate which is appears as a colourless solution.

$$Mg^{2+}(aq) + 2HCO_3^-(aq) \rightarrow Mg(HCO_3)_2(aq)$$

Colourless solution

l)Action of acidified potassium manganate(VII) solution

Acidifiedpotassium manganate(VII) solution/Acidified potassium permanaganate solution is a powerful oxidizing agent. A series of reducing agents can therefore reduce the acidified potassium manganate(VII) solution, turning it from a purple solution to a colourless solution. The purple colour is due to the manganate(VII) ions while the seemingly colourless solution is due to the manganese(II) ions which are very pale pink in solution, hence appearing colourless. Therefore,acidified potassium manganate(VII) solution is used to test for anions that are reducing agents such as $C_2O_4^{2-}$, SO_3^{2-}, $S_2O_3^{2-}$, NO_2^-, Cl^-, $Br^-$and I^-. All these anions therefore reduce MnO_4^- to Mn^{2+}.

Test	Observation	Deduction
To the test solution, add acidified potassium manganate(VII) solution.	Purple solution turns colourless.	SO_3^{2-}, $S_2O_3^{2-}$, NO_2^-, Cl^-, Br^-, I^-
To the test solution, add acidified potassium manganate(VII) solution and heat.	Purple solution turns colourless on heating.	$C_2O_4^{2-}$confirmed present.

Note: Oxalate ions, $C_2O_4^{2-}$ only react with acidified potassium manganate(VII) solution when heated.

m) Action of neutral iron(III) chloride solution

Neutral iron(III) chloride solution is used to confirm the presence of ethanoate ions, CH_3COO^-.
Formation of a red colouration confirms the presence of CH_3COO^-.
However, when addition of neutral iron(III) chloride solution to a solution containing CH_3COO^-, is followed by boiling, a brown precipitate is formed on boiling.

$$CH_3COO^-(aq) + H_2O(l) \rightleftharpoons CH_3COOH(aq) + OH^-(aq)$$

$$Fe^{3+}(aq) + 3OH^-(aq) \rightarrow Fe(OH)_3(s)$$

Brown precipitate

OR: $$Fe^{3+}(aq) + 3CH_3COO^-(aq) \rightarrow (CH_3COO)_3Fe(s)$$

Brown precipitate

Test	Observation	Deduction
To the test solution, add neutral iron(III) chloride solution.	A red colouration is formed.	CH_3COO^- confirmed present.
To the test solution, add neutral iron(III) chloride solution and boil/heat.	Abrown precipitate (or reddish-brown precipitate) is formed.	CH_3COO^- confirmed present.

n) Action of Chloroform

Chloroform is used in confirming the presence of both the Br^- and I^-. The chloroform is used in combination with bleaching powder (or solution of a bleaching agent) together with dilute nitric acid.

Test	Observation	Deduction
To the test solution, add a little bleaching powder (**or** add $1cm^3$ of a solution of a bleaching agent), followed by $1cm^3$ of dilute nitric acid and then $1cm^3$ of chloroform and shake gently.	An orange solution in the organic layer/lower layer. (orange lower layer).	Br^- confirmed present.
	Purple solution in the organic layer/lower layer. (purple lower layer).	I^- confirmed present.

Note: A solution of a bleaching agent in the form of sodium hypochlorite e.g. JIK can work effectively for the above stated chemical test.

o) Action of ethanol

Ethanol is used in combination with concentrated sulphuric acid to test for ethanoate ions. Ethanol and a few drops of concentrated sulphuric acid are added to a test solution containing the ethanoate ion, CH_3COO^-. A sweet, fruity smell confirms the presence of CH_3COO^-. This chemical test is based on the **esterification reaction** (Formation of an ester from an alcohol, a carboxylic acid and a concentrated mineral acid, which is usually concentrated sulphuric acid).

Test	Observation	Deduction
To the test solution, add $1cm^3$ of Ethanol followed by 3 to 5 drops of concentrated sulphuric acid and warm. Pour the product in a test tube containing water.	A sweet, fruity smell is detected.	Esterification reaction. CH_3COO^- confirmed present.

p) Action of iodine solution

Iodine solution is commonly used to test for anions such as SO_3^{2-}, $S_2O_3^{2-}$, $NO_2^-$and $C_2O_4^{2-}$. These anions are reducing agents which reduce aqueous iodine to iodide ions. Hence addition of iodine solution to a test solution containing SO_3^{2-}, $S_2O_3^{2-}$and NO_2^- turns the brown iodine solution colourless. However, with the$C_2O_4^{2-}$, the brown solution turns colourless only after the mixture has been heated.

Test	Observation	Deduction
To the test solution, add iodine solution.	Brown solution turns colourless.	SO_3^{2-}, $S_2O_3^{2-}$, NO_2^-
To the test solution, add iodine solution and heat.	Brown solution turns colourless.	$C_2O_4^{2-}$

q) Action of ammonium molybdate solution

Ammonium molydate solution is used in conjunction with concentrated nitric acid to test for phosphate ions, PO_4^{3-}. When to a solution containing the PO_4^{3-}, ammonium molybdate solution is added followed by a few drops of concencentrated nitric acid and the mixture warmed, a yellow precipitate is formed.

Table showing a summary of Preliminary and Confirmatory tests for Common Anions

Anions	Preliminary/ Confirmatory tests	Test procedure	Observation
CO_3^{2-}	**Preliminary test**	Heat 2 spatula endfuls of the solid strongly in a dry test tube until there is no further change.	A colourless gas that turns moist blue litmus paper red and limewater milky (forms a white precipitate with calcium hydroxide solution).
	Confirmatory tests	i) To the unknown solid add $2cm^3$ of dilute hydrochloric acid or dilute nitric acid.	Effervescence of a colourless gas that turns moist blue litmus paper red and limewater milky.

		ii) To the unknown solid, add 2 to 3 drops of concentrated sulphuric acid.	Rapid effervescence of a colourless gas that turns moist blue litmus paper red and limewater milky.
		iii) To the test solution, add lead(II) nitrate solution followed by dilute nitric acid.	A white precipitate soluble in the acid with effervescence of a colourless gas that turns moist blue litmus paper red and limewatermilky.
		iv) To the test solution, add Barium nitrate solution followed by dilute nitric acid.	A white precipitate soluble in the acid with effervescence of a colourless gas which turns moist blue litmus paper red and limewater milky.
		v) To the test solution, add Barium chloride solution followed by dilute hydrochloric acid.	A white precipitate soluble in the acid with effervescence of a colourless gas which turns moist blue litmus paper red and limewater milky.
		vi) To the test solution, add silver nitrate solution followed by dilute nitric acid.	A white precipitate soluble in the acid with effervescence of a colourless gas which turns moist blue litmus paper red and limewater milky.
		vii) To the test solution, add silver nitrate solution followed by excess dilute ammonia solution.	A white precipitate in excess ammonia solution.
SO_4^{2-}	**Preliminary tests**	i) Heat 2 spatula endfuls of the solid strongly in a dry test tube until there is no further change.	A colourless, pungent gas, which turns moist blue litmus paper red and bleaches it, and turns acidified potassium dichromate(VI) solution from orange to green, is evolved. Also white fumes which turn moist blue litmus paper red.
		ii) To the test solution, add lead(II) nitrate solution.	A white precipitate is formed.
		iii) To the test solution, add lead(II) nitrate solution and warm.	A white precipitate insoluble on warming.
		iv) To the test solution, add lead(II) nitrate solution, followed by dilute nitric acid.	A white precipitate insoluble in the acid.
		v) To the test solution, add silver nitrate solution.	No observable change.
	Confirmatory tests	i) To the test solution, add Barium nitrate solution, followed by dilute nitric acid.	A white precipitate insoluble in the acid.
		ii) To the test solution, add Barium chloride solution followed by dilute hydrochloric acid.	A white precipitate insoluble in the acid.

SO_3^{2-}	**Preliminary tests**	i)To the unknown solid, add 2cm^3 of dilute hydrochloric acid and warm.	No observable change at room temperature, but on warming, there is effervescence of a colourless, pungent gas, turns moist blue litmus paper red and bleaches it, and turns acidified potassium dichromate(VI) solution from orange to green/turns acidified potassium manganate(VII) solution from purple to colourless.
		ii) To the unknown solid, add 2-3 drops of concentrated sulphuric acid and warm gently.	Effervescence of a colourless, pungent gas, turns moist blue litmus paper red and bleaches it, and turns acidified potassium dichromate(VI) solution from orange to green/turns acidified potassium manganate(VII) solution from purple to colourless.
		iii) To the test solution, add 2cm^3 of dilute hydrochloric acid and warm.	Effervescence of a colourless, pungent gas, turns moist blue litmus paper red and bleaches it, and turns acidified potassium dichromate(VI) solution from orange to green/turns acidified potassium manganate(VII) solution from purple to colourless.
		iv) To the test solution, add lead(II) nitrate solution.	A white precipitate.
		v) To the test solution, add lead(II) nitrate solution followed by dilute nitric acid.	A white precipitate soluble in the acid without effervescence.
		vi) To the test solution, add Barium nitrate solution followed by dilute nitric acid.	A white precipitate soluble in the acid without effervescence.
		vii) To the test solution, add Barium chloride solution followed by dilute hydrochloric acid.	A white precipitate soluble in the acid with evolution of a colourless, pungent gas which turns moist blue litmus paper red and bleaches it, and turns acidified potassium dichromate(VI) solution from orange to green.
		viii) To the test solution, add silver nitrate solution followed by dilute nitric acid.	A white precipitate soluble in the acid without effervescence.
	Confirmatory tests	i) To the test solution, add acidified potassium manganate(VII) solution.	Purple solution turns colourless.
		ii) To the test solution, add a few drops of iodine solution.	Brown solution turns colourless.
$C_2O_4^{2-}$	**Preliminary tests**	i) Heat 2 spatula endfuls of the solid strongly in a dry test tube until there is no further change.	A colourless gas that turns moist blue litmus paper red and limewater milky is evolved.

		ii) To the unknown solid, add 2 to 3 drops of concentrated sulphuric acid and warm.	Effervescence of a colourless gas that turns moist blue litmus paper red and limewater milky is evolved. Effervescence of another colourless gas neutral to litmus paper and burns with a blue flame.
		iii) To the test solution, add lead(II) nitrate solution.	A white precipitate.
		iv) To the test solution, add lead(II) nitrate solution followed by dilute nitric acid.	A white precipitate soluble in the acid without effervescence.
		v) To the test solution, add Barium nitrate solution followed by dilute nitric acid.	A white precipitate soluble in the acid without effervescence.
		vi) To the test solution, add silver nitrate solution followed by dilute nitric acid.	A white precipitate soluble in the acid without effervescence.
		vii) To the test solution, add silver nitrate solution followed by excess dilute ammonia solution.	A white precipitate soluble in excess ammonia solution.
	Confirmatory tests	i) To the test solution, add acidified potassium manganate(VII) solution and heat.	Purple solution turns colourless.
		ii) To the test solution, add iodine solution and heat.	Brown solution turns colourless.
$S_2O_3^{2-}$	**Preliminary tests** ***Note: These two preliminary testscan also act as confirmatory tests in case none of the test procedures for the confirmatory tests below has been provided.***	i) To the unknown solid, add 2-3 drops of concentrated sulphuric acid and warm gently.	Effervescence of a colourless, pungent gas, turns moist blue litmus paper red and bleaches it, and turns acidified potassium dichromate(VI) solution from orange to green/turns acidified potassium manganate(VII) solution from purple to colourless and **a cream precipitate(pale yellow precipitate) is formed.**
		ii) To the test solution, add $2cm^3$ of dilute hydrochloric acid and warm.	Effervescence of a colourless, pungent gas, turns moist blue litmus paper red and bleaches it, and turns acidified potassium dichromate(VI) solution from orange to green/turns acidified potassium manganate(VII) solution from purple to colourless and **a cream precipitate (pale yellow precipitate) is formed.**
	Confirmatory tests	i) To the test solution, addiodine solution.	Brown solution turns colourless.
		ii) To the test solution, add acidified potassium manganate(VII) solution.	Purple solution turns colourless.

Cl^-	**Preliminary tests**	i) Heat 2 spatula endfuls of the solid strongly in a dry test tube until there is no further change.	Misty, chocking fumes, which turn moist blue litmus paper red and form dense white fumes with concentrated ammonia solution. Also a greenish-yellow gas, turns mosit blue litmus paper red and bleaches it.
		ii) To the unknown solid, add a few drops of concentrated sulphuric acid and warm.	Effervescence of misty, chocking fumes, which turn moist blue litmus paper red and form dense white fumes with concentrated ammonia solution.
		iii) To the unknown solid, add a little manganese(IV) oxide followed by a few drops of concentrated sulphuric acid and warm.	Effervescence of a greenish–yellow gas with an irritating smell, which turns moist blue litmus paper red and bleaches it.
		iv) To the test solution, add lead(II) nitrate solution.	A white precipitate is formed.
		vi) To the test solution, add lead(II) nitrate solution followed by dilute nitric acid.	A white precipitate insoluble in the acid.
		v) To the test solution, add Barium nitrate solution, followed by dilute nitric acid.	No observable change.
	Confirmatory tests	i) To the test solution, add 2 drops of lead(II) nitrate solution and warm, then cool under running tap water.	A white precipitate soluble on warming, precipitate reappears on cooling.
		ii) To the test solution, add silver nitrate solution followed by dilute nitric acid.	A white precipitate insoluble in the acid.
		iii) To the test solution, add silver nitrate solution followed by excess ammonia solution.	A white precipitate soluble in excess ammonia.
I^-	**Preliminary tests**	i) Heat 2 spatula endfuls of the solid strongly in a dry test tube until there is no further change.	A purple vapour which turns moist blue litmus paper red and sublimes to form a black/purple/purplish-black solid.
		ii) To the unknown solid, add 2-3 drops of concentrated sulphuric acid and warm.	Apurple vapour which turns moist blue litmus paper red and sublimes to form a black/purple/purplish-black solid. White fumes which turn moist blue litmus paper red.
		iii) To the unknown solid, add half a spatula endful of manganese(IV) oxide followed by 2-3 drops of concentrated sulphuric acid and warm.	A purple vapour which turns moist blue litmus paper red and sublimes to form a black/purple/purplish-black solid.
		iv) To the test solution, add lead(II) nitrate solution.	A yellow precipitate.

		v) To the test solution, add dilute nitric acid followed by lead(II) nitrate solution.	A yellow precipitate.
		vi) To the test solution, add Barium nitrate solution.	No observable change.
		vii) To the test solution, add silver nitrate solution.	A pale yellow precipitate.
		viii) To the test solution, add dilute nitric acid followed by silver nitrate solution.	A pale yellow precipitate.
		ix) To the test solution, add silver nitrate solution followed by dilute nitric acid.	A pale yellow precipitate insoluble in the acid.
		x) To the test solution, add silver nitrate solution followed by dilute ammonia solution until in excess.	A pale yellow precipitate insoluble excess ammonia solution.
	Confirmatory tests	i) To the test solution, add 3-5 drops of concentrated nitric acid and warm. Cool and then add starch solution.	Colourless solution turns brown and then to a blue-black solution
		ii) To the test solution, add 5-6 drops of concentrated sulphuric acid and warm. To the mixture, add sodium thiosulphate solution.	Colourless solution turns brown solution and then colourless on addition of sodium thiosulphate solution.
		iii) To the test solution, add a little bleaching powder (**or** add $1cm^3$ of sodium hypochlorite solution), followed by $1cm^3$ of dilute nitric acid and then $1cm^3$ of chloroform (**or** $1cm^3$ of tetrachloromethane) and shake gently.	A purple solution in the organic layer/lower layer. (Purple lower layer)
Br-	**Preliminary tests**	i) Heat 2 spatula endfuls of the solid strongly in a dry test tube until there is no further change.	A brown vapour which turns moist blue litmus paper red and bleaches it.
		ii) To the unknown solid, add a few drops of concentrated sulphuric acid and warm.	A brown vapour which turns moist blue litmus paper red and bleaches it. White fumes which turn moist blue litmus paper red.
		iii) To the unknown solid, add half a spatula endful of manganese(IV) oxide followed by 2-3 drops of concentrated sulphuric acid and warm.	A brown vapour which turns moist blue litmus paper red and bleaches it.
		iv) To the test solution, add lead(II) nitrate solution	A white precipitate.
		v) To the test solution, add dilute nitric acid followed by lead(II) nitrate solution.	A white precipitate.

		vi) To the test solution, add Barium nitrate solution.	No observable change.
		vii) To the test solution, add silver nitrate solution.	A cream precipitate.
		viii) To the test solution, add silver nitrate solution followed by dilute nitric acid.	A cream precipitate insoluble in the acid.
		ix) To the test solution, add dilute nitric acid followed by silver nitrate solution.	A cream precipitate.
		x) To the test solution, add silver nitrate solution followed by dilute ammonia solution until in excess.	A cream precipitate dissolves with difficulty in excess ammonia solution.
	Confirmatory test	To the test solution, add a little bleaching powder (**or** add 1cm^3 of sodium hypochlorite solution), followed by 1cm^3 of dilute nitric acid and then 1cm^3 of chloroform (**or** 1cm^3 of tetrachloromethane) and shake gently.	An orange solution in the organic layer/lower layer. (orange lower layer).
NO_3^-	**Preliminary tests**	i) Heat 2 spatula endfuls of the solid strongly in a test tube until there is no further change.	Brown, pungent fumes, which turn moist blue litmus paper red.
		i) To the unknown solid, add 2 to 3 drops of concentrated sulphuric to the unknown solid and warm gently.	Brown, pungent fumes, which turn moist blue litmus paper red.
		ii) To the test solution, add a few pieces of copper turnings followed by 3 to 4 drops of concentrated sulphuric acid and warm gently.	Brown, pungent fumes, which turn moist blue litmus paper red.
	Confirmatory test	i) To the test solution in a test tube, add a freshly prepared solution of iron(II) sulphate. Hold the test tube in a slanting position and then add concentrated sulphuric acid drop by drop down the wall of the test tube.	A brown ring is formed at the interface of the acid and aqueous layer.
		ii) To the test solution, add Devarda's alloy followed by excess dilute sodium hydroxide solution and warm gently.	A colourless, pungent, chocking gas which turns moist red litmus paper blue and forms dense white fumes with concentrated hydrochloric acid.
		iii) To the test solution, add aluminium metal powder followed by excess dilute sodium hydroxide solution and warm gently. ***Note: Zinc metal powder can also be used in place of aluminium metal powder for the same purpose in this test.***	A colourless, pungent, chocking gas which turns moist red litmus paper blue and forms dense white fumes with concentrated hydrochloric acid.

NO_2^-	**Preliminary test**	i)To the unknown solid, add 1cm^3 of dilute hydrochloric acid and warm gently	Effervescence of brown, pungent fumes which turn moist blue litmus paper red. Brown, pungent solution turns pale blue.
	Confirmatory tests	i) To test solution, add a freshly prepared solution of iron(II) sulphate followed by dilute sulphate solution.	A dark brown complex is formed.
		ii) To the test solution, add acidified potassium manganate(VII) solution	Purple solution turns colourless.
		iii) To the test solution, add iodine solution.	Brown solution turns colourless.
CH_3COO^-	**Preliminary tests**	i) Heat 2 spatula endfuls of the solid strongly in a dry test tube until there is no further change.	White fumes with a sweet odour which form a yellow precipitate with 2,4-dinitrophenylhydrazine solution (Brady's reagent). Also a colourless gas that turns moist blue litmus paper red and limewater milkyis evolved.
		ii) To the test solution, add 2 to 3 drops of concentrated sulphuric acid and warm gently.	White fumes with a vinegar smell, turn moist blue litmus paper red.
	Confirmatory tests	i) To the test solution, add neutral iron(III) chloride solution.	A red colouration is formed. (A red solution is formed.)
		ii) To the test solution, add Neutral iron(III) chloride solution and boil.	A brown precipitate is formed.
		iii) To the test solution, add ethanol followed by 3 to 5 drops of concentrated sulphuric acid and warm. Pour the product into a beaker of cold water.	A sweet, fruity smell.
PO_4^{3-}	**Preliminary tests**	i) To the test solution, add lead(II) nitrate solution	A white precipitate is formed.
		ii) To the test solution, add lead(II) nitrate solution followed by dilute nitric acid.	A white precipitate soluble in the acid without effervescence.
		iii) To the test solution, add Barium nitrate solution followed by dilute nitric acid.	A white precipitate soluble in the acid without effervescence.
		iv) To the test solution, add silver nitrate solution followed by dilute nitric acid.	A pale yellow precipitate soluble in the acid without effervescence.
		v) To the test solution, add silver nitrate solution followed by excess dilute ammonia solution.	A pale yellow precipitate soluble in excess ammonia solution.
	Confirmatory test	To the test solution, add ammonium molybdate solution followed by concentrated nitric acid and warm.	A yellow precipitate is formed.

CrO_4^{2-}	**Preliminary tests**	i) To the test solution, add lead(II) nitrate solution.	A yellow precipitate is formed.
		ii) To the test solution, add Barium nitrate solution.	A yellow precipitate is formed.
	Confirmatory tests	i) To the test solution, add dilute hydrochloric acid.	Yellow solution turns into orange solution.
		ii) To the test solution, add dilute Sulphuric acid followed by hydrogen peroxide solution.	Yellow solution turns orange and then to an intense blue solution which quickly fades, leaving behind a green solution.
		iii) To the test solution, add 2-3 drops of silver nitrate solution followed by dilute nitric acid.	A red precipitate soluble in the acid without effervescence to form an orange solution.
		iv) To the test solution, add 2-3 drops of silver nitrate solution followed by excess dilute ammonia solution.	A red precipitate soluble in excess ammonia solution to form a yellow solution.
ClO^-	**Preliminary tests**	i) To the test solution, add lead(II) nitrate solution.	A white precipitate.
		ii) To the test solution, add silver nitrate solution.	A white precipitate.
	Confirmatory test	To the test solution, add dilute nitric acid.	A greenish-yellow gas; turns moist blue litmus paper red and bleaches it.

8.3.3 Rules followed while answeringInorganic Qualitative Analysis Questions (The Dos and Don'ts of Inorganic Qualitative Analysis)

- Observations should be writtenprecisely and completely.
- Deductions should relate to observations.
- In the deduction column, a wrong answer written counsels out with a correct answer.
- The use of words inappropriately will lead to loss of marks and should therefore be avoided.
- Words such as "suspected", "probably present" or "present" carry no extra marks. However, in case of a confirmatory test for any ion, the statement **"confirmed present"** must accompany the cation or anion referred to while making the deduction.
- Giving the identity of a cation or anion at the end of the experiment can only earn the candidate a mark if the ionwas properly identified at the confirmatory test during the course of the experiment. For example, the test procedure (where applicable), observation and deduction for confirming the cation or anion should be correct and complete.
- Any chemical formula or symbol or charge on any ion should be correctly written. For example, the charge on the ion should be as close as possible to the constituent letters of the symbol and for a symbol with more than one letter in it, the the letters that constitute the symbol should be written as close as possible but should remain separate. ***(Refer to the Table below for details)***

Ion or compound	Correct presentation of symbol or formula	Wrong presentation of symbol or formula
Zinc ion	Zn^{2+}	$2n^{2+}$, ZN^{2+}, Zn^{2+}, Zn^{2+}, Zn^{+2}
Lead(II) ion	Pb^{2+}	P_b^{2+}, Pb^{2+}, Pb^{2+}, Pb^{2+}, Pb^{+2}
Aluminium ion	Al^{3+}	Al^{3+}, Al^{3+}, Al^{3+}, Al^{3+}, Al^{+3}
Calcium ion	Ca^{2+}	Ca^{2+}, Ca^{2+}, Ca^{2+}, Ca^{+2}
Magnesium ion	Mg^{2+}	Mg^{2+}, mg^{2+}, Mg^{2+}, Mg^{2+}, Mg^{+2}
Barium ion	Ba^{2+}	Ba^{2+}, Ba^{2+}, Ba^{2+}, Ba^{2+}, Ba^{+2}
Copper(II) ion	Cu^{2+}	Cu^{2+}, CU^{2+}, Cu^{2+}, Cu^{+2}
Iron(II) ion	Fe^{2+}	Fe^{2+}, Fe^{2+}, Fe^{2+}, Fe^{2+}, fe^{2+}, fe^{2+}
Nickel(II) ion	Ni^{2+}	Ni^{2+}, Ni^{2+}, Ni^{2+}, Ni^{2+}, Ni^{+2}
Ammonium ion	NH_4^+	NH_4^+, NH_4^+, NH_3^+
Carbonate ion	CO_3^{2-}	Co_3^{2-}, CO_3^{2-}, CO_3^{2-}, CO_3^{-2}
Sulphate ion	SO_4^{2-}	So_4^{2-}, SO_4^{2-}, SO_4^{2-}, SO_4^{-2}
Chloride ion	Cl^-	Ci^-, Cl^-, CL^-, Cl^-, cl^-
Iodide ion	I^-	I^-, l^-, i^-
Carbon dioxide	CO_2	Co_2, CO_2, CO_2, cO_2
Zinc oxide	ZnO	Zno, ZnO, $2nO$, ZnO
Magnesium oxide	MgO	Mgo, MgO, MgO, MgO
Lead(II) oxide	PbO	Pbo, PbO, PbO, Pb^{2+}

8.4 Worked out examples on Inorganic Qualitative Analysis

Worked out example 8.4.1

You are provided with substance A, which contains two cations and two anions. Carry out the following tests and identify them. Identify any gases which may be evolved. Write your observations and deductions in the table below.

Test	Observation	Deduction
a) Heat a spatula endful of A in a dry test tube strongly until there is no further change.	*Pale green crystalline solid.* *A colourless condensate; which turns anhydrous copper(II) sulphate from white to blue.* *A colourless gas which turns moist blue litmus paper red; and limewater milky.* *Acolourless, pungent gas; turns moist blue litmus paper red; and turns acidified potassium dichromate solution from orange to green.* *A white residue.* *A black residue.*	*Ni^{2+}, Fe^{2+}, Cr^{3+},Cu^{2+}* *Water of crystallization* *(**or** H_2O given off from a hydrated salt)* *CO_2 (g) evolved.* *CO_3^{2-} or $C_2O_4^{2-}$suspected present.* *SO_2(g) evolved* *SO_4^{2-} or SO_3^{2-}suspected present.* *Al_2O_3(s),MgO(s), CaO(s) **or** BaO(s) formed hence Al^{3+},Mg^{2+}, Ca^{2+}**or**Ba^{2+}* *CuO(s)formed hence Cu^{2+}**or** NiO(s) formed henceNi^{2+}.*
b) Dissolve two spatula endfuls of A in dilute nitric acid. To 3cm^3 of the resultant solution, add 7cm^3 of dilute sodium hydroxide solution drop wise and then filter. Keep both the residue and filtrate.	*Pale green crystalline solid dissolves in the acid with effervescence of a colourless gas which turns moist blue litmus paper red; and limewater milky; to form a pale green solution.* *A pale green precipitate insoluble in excess.* *A colourless filtrate.* *A pale green residue.*	*CO_2(g) evolved.* *CO_3^{2-} confirmed present.* *Ni^{2+} orCr^{3+}suspected present.* *Ni^{2+}suspected present.* *Zn^{2+}, Pb^{2+}, Al^{3+}or Sn^{2+}suspected present in filtrate* *Ni^{2+}suspected in residue.*
c) To 4cm^3 of the filtrate obtained in (b) above, add dilute nitric acid drop by drop until the solution is just acidic. Divide the acidic solution into **six** parts.	*A white precipitate soluble in the acid to form a colourless solution*	*Zn^{2+},Pb^{2+},Al^{3+} orSn^{2+}*

i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.	*A white precipitate soluble in excess to form a colourless solution.*	*Zn^{2+}, Pb^{2+}, Al^{3+} or Sn^{2+}*
ii) To the second part, add dilute ammonium hydroxide solution drop wise until in excess.	*A white precipitate insoluble in excess.*	*Pb^{2+} or Al^{3+}*
iii) To the third part, add a few drops of potassium iodide solution	*No observable change.*	*Pb^{2+} <u>absent</u>* *Al^{3+} suspected present*
iv) To the fourth part, add 2-3 drops of dilute nitric acid followed by 3-4 drops of litmus solution, followed by dilute dilute ammonia solution drop wise until in excess.	*A blue lake solution.*	*Al^{3+} confirmed present.*
v) To the first part, add dilute nitric acid followed by lead(II) nitrate solution.	*A white precipitate is formed.*	*SO_4^{2-}, Cl^- or Br^-*
vi) Carry out a test of your own choice to the sixth part, to confirm the anion present in A. *<u>Add Barium nitrate solution followed by dilute nitric acid.</u>*	*A white precipitate insoluble in the acid.*	*SO_4^{2-} confirmed present.*
d) Wash the residue obtained in (b) above with water and to the washed residue, add dilute nitric acid until it just dissolves. Divide the resultant solution into **three** parts.	*Pale green residue dissolves in the acid to form a pale green solution.*	*Ni^{2+}*
i) To the first part, added dilute sodium hydroxide solution, drop wise until in excess.	*A pale green precipitate insoluble in excess.*	*Ni^{2+}*
ii) To the second part, add dilute ammonia solution drop wise until in excess.	*A pale green precipitate soluble in excess to form a blue solution.*	*Ni^{2+}*
iii) Carry out a test of your own choice to the third part to confirm one of the cations present in A. *<u>Add a little dilute ammonia solution followed by dimethyl glyoxime solution.</u>*	*A bright red precipitate (red / pink precipitate)*	*Ni^{2+} confirmed present.*

Cations in A……..….Al^{3+}…… and ..……Ni^{2+}……..
Anions in A…….CO_3^{2-}…….. and ……..SO_4^{2-}……

Worked out example 8.4.2

You are provided with substance B_1, which contains two cations and two anions. Carry out the following tests and identify them. Identify any gases which may be evolved. Write your observations and deductions in the table below.

Test	Observation	Deduction
a) Heat a spatula endful of B_1 in a dry test tube strongly until there is no further change.	*Yellowpowdered solid.* *A colourless condensate which turns anhydrous copper(II) sulphate from white to blue.* *White fumes with a sweet odourand form a yellow precipitate with 2,4-dinitrophenylhydrazine solution (Brady's Reagent).* *A colourless gas which turns moist blue litmus paper red; and limewater milky.* *Brown; pungent fumes, which turn moist blue litmus paper red;* *A reddish-brownresidue when hot; turns yellowon cooling.*	*PbO(s) hence Pb^{2+} **or** CrO_4^{2-}.* *Water of crystallization (**or** H_2O given off from a hydrated salt.)* *$CH_3COCH_3(g)$ evolved.* *CH_3COO^- suspected present.* *$CO_2(g)$ evolved.* *CO_3^{2-} or $C_2O_4^{2-}$ suspected.* *$NO_2(g)$ evolved.* *NO_3^- suspected present.* *PbO(s) formed; hence Pb^{2+}*
b) To a spatula endful of B_1, add 3 to 5 drops of concentrated sulphuric acid and warm gently.	*White fumes with a vinegar smell; turn moist blue litmus paper red.* *Brown; pungent fumes, which turn moist blue litmus paper red.*	*Acetic acid fumes;* *CH_3COO^- suspected present.* *$NO_2(g)$ evolved.* *NO_3^- suspected present.*
c) To two spatula endfuls of B_1, add dilute nitric acid and warm gently to dissolve. To 4cm^3 of the resultant solution, add 7cm^3 of dilute ammonia solution drop wise and then filter.Keep both the residue and filtrate.	*Yellow crystalline solid dissolves in the acid to form a colourless solution.* *A whiteprecipitate insoluble in excess.* *A colourless filtrate.* *A white residue.*	*Zn^{2+},Pb^{2+},Al^{3+}, Ba^{2+},Mg^{2+}or Ca^{2+}suspected present.* *Pb^{2+},Al^{3+},Sn^{2+}, Ba^{2+}orMg^{2+}suspected present.* *Zn^{2+} suspected present in filtrate* *Pb^{2+},Al^{3+}, Sn^{2+}, Ba^{2+}or Mg^{2+}suspected present in residue.*

d) To 4cm^3 of the filtrate obtained in (b) above, add dilute nitric acid drop by drop to acidify. Divide the acidic solution into **five** portions. i) To the first portion, add dilute sodium hydroxide solution drop wise until in excess.	*A white precipitate which dissolves in the acid to form a colourless solution.* *A white precipitate soluble in excess to form a colourless solution.*	Zn^{2+} Zn^{2+}
ii) To the portion, add dilute ammonium hydroxide solution drop wise until in excess.	*A white precipitate soluble in excess to form a colourless solution.*	Zn^{2+}
iii) Carry out a test of your own choice to the third portion to confirm one of the cations present in B_1. *Add solid ammonium chloride, followed by 3-4 drops of disodium hydrogenphosphate solution and then dilute ammonia solution drop wise until in excess.*	*A white precipitate soluble in excess ammonia solution to form a colourless solution.*	Zn^{2+} *confirmed present.*
iv) To the fourth portion, add neutral iron(III) chloride solution and boil.	*A brown precipitate is formed.*	CH_3COO^- *confirmed present.*
v) To the fifth portion, add zinc metal powder followed by excess sodium hydroxide solution and warm.	*A colourless, pungent, chocking gas which turns moist red litmus paper blue; and forms dense white fumes with concentrated hydrochloric acid.*	$NH_3(g)$ *evolved.* NO_3^- *confirmed present.*
e) Wash the residue obtained in (c) above with dilute ammonia solution and dissolve the washed residue in dilute nitric acid. Divide the resultant solution into**four**portions. i) To the first portion, add dilute sodium hydroxide solution drop wise until in excess.	*White residue dissolves in the acid to form a colourless solution.* *A white precipitate soluble in excess to form a colourless solution.*	Pb^{2+}, Al^{3+}, Sn^{2+}, Ba^{2+} or Mg^{2+} Pb^{2+}, Al^{3+} or Sn^{2+}
ii) To the second portion, add dilute ammonia solution drop wise until in excess.	*A white precipitate insoluble in excess.*	Pb^{2+}, Al^{3+} or Sn^{2+}
iii) To the third portion, add dilute sulphuric acid.	*A white precipitate.*	Pb^{2+}

iv) To the fourth portion, add potassium chromate solution followed by sodium hydroxide solution drop wise until in excess.	*A yellow precipitate soluble in excess sodium hydroxide solution to form a yellow solution.*	*Pb^{2+} confirmed present.*

Cations in B_1……..…*Zn^{2+}*…….. and..……*Pb^{2+}*……..
Anions in B_1……. *CH_3COO^-*…….. and ……..*NO_3^-*……...

Worked out example 8.4.3

You are provided with substance B_2, which contains two cations and two anions. Carry out the following tests and identify them. Identify any gases which may be evolved. Write your observations and deductions in the table below.

Test	Observation	Deduction
a) Heat a spatula endful of B_2 in a dry test tube strongly until there is no further change.	*White crystalline solid.* *A colourless condensate which turns anhydrous copper(II) sulphate from white to blue.* *Colourless gas with a pungent smell; turns moist blue litmus paper red; and acidified potassium dichromate(VI) solution from orange to green.* *White fumes turn moist blue litmus paper red.* *Whiteresidue.*	*Zn^{2+},Pb^{2+},Al^{3+}, Ba^{2+},Mg^{2+}, Ca^{2}orSn^{2+} suspected present.* *Water of crystallization (**or** H_2O given off from a hydrated salt.)* *SO_2 (g) evolved.* *SO_4^{2} or SO_3^{2-} suspected present.* *SO_3 (g) evolved.* *SO_4^{2-} suspected present.* *CaO(s), MgO(s), Al_2O_3(s), BaO(s) formed;hence Ca^{2+}, Mg^{2+}, Al^{3+} or Ba^{2+}.*
b) To a spatula endful of B_2, add 3 to 5 drops of concentrated sulphuric acid and warm gently.	*Effervescence of a brown vapour; turns moist blue litmus paper red; and bleaches it.*	*Br_2(g) evolved;* *Br^- suspected present.*
c) To two spatula endfuls of B_2, in a boiling tube, add 5cm^3 of water and shake well to dissolve. To 4cm^3 of the resultant solution, add dilute ammonia solution drop wise until in excess and then filter.Keep both the filtrate and residue.	*White crystalline solid dissolves in water to form a colourless solution.* *Whiteprecipitate insoluble in excess.* *Colourless filtrate.* *White residue.*	*Zn^{2+},Pb^{2+},Al^{3+}, Sn^{2+}, Ba^{2+},Mg^{2+}orCa^{2+}suspected present.* *Pb^{2+},Al^{3+}, Sn^{2+}, Ba^{2+}or Mg^{2+}suspected.* *Zn^{2+} suspected present in filtrate* *Pb^{2+},Al^{3+}, Sn^{2+}, Ba^{2+}or Mg^{2+}suspected present in residue.*

d) To 5cm^3 of the filtrate obtained in (c) above, add dilute nitric acid drop wise until the solution is just acidic. Divide the acidic solution into **seven** portions. i) To the first portion, add dilute sodium hydroxide solution drop wise until in excess.	*A white precipitate which dissolves in the acid to form a colourless solution.* *A white precipitate soluble in excess to form a colourless solution.*	Zn^{2+} Zn^{2+}
ii) To the second portion, add add dilute ammonia solution drop wise until in excess.	*A white precipitate soluble in excess.*	Zn^{2+}
iii) Carry out a test of your own choice to the thirdportion to confirm one of the cations present in B_2. *Add solid ammonium chloride, followed by 3-4 drops of disodium hydrogenphosphate solution and then dilute ammonia solution drop wise until in excess.*	*A white precipitate soluble in excess ammonia solution forming a colourless solution.*	Zn^{2+} *confirmed present.*
iv) To the fourth portion, add 2-3 drops of lead(II) nitrate solution and warm.	*A white precipitate insoluble on warming.*	SO_4^{2-}
v) To the fifthportion, add barium nitrate solution.	*A white precipitate is formed.*	SO_4^{2-} *confirmed present.*
vi) To the sixth portion, add 2-3 drops of siver nitrate solution followed by dilute ammonia solution dropwise until in excess.	*A cream precipitate; dissolves with difficultyin excess ammonia solution.*	Br^-
(vii) To the seventh portion, add1 drops of dilute nitric acid followed by 1 drops of a solution of a bleaching agent, and then 2-3dropsof chloroform and shake gently.	*An orange solution in the organic layer/lower layer.*	Br^- *confirmed present*
e) Wash the residue obtained in (c) above with dilute ammonia solution and dissolve the washed residue in dilute sulphuric acid. Divide the resultant solution into **three** portions.	*White residue dissolves in the acid to form a colourless solution.*	Al^{3+}, Sn^{2+} *or* Mg^{2+}

i) To the first portion, add dilute sodium hydroxide solution drop wise until in excess.	*A white precipitate soluble in excess to form a colourless solution.*	Al^{3+} *or* Sn^{2+}
ii) To the second portion, add dilute ammonia solution drop wise until in excess.	*A white precipitate insoluble in excess.*	Al^{3+} *or* Sn^{2+}
iii) To the test solution, add 2-3 drops of dilute nitric acid are added, followed by 3-4 drops of litmus solution and then dilute ammonia solution drop wise until in excess.	*A blue lake solution.*	Al^{3+} *confirmed present.*

Cations in B_2……..…Zn^{2+}…… and..……Al^{3+}……..
Anions in B_2……. SO_4^{2-}……and ……Br^-……..

8.5 Practical Exercises on Inorganic Qualitative Analysis

Experiment 8.5.1

You are provided with substance C, which contains two cations and two anions. Carry out the following tests to identify them. Identify any gases which may be evolved. Write your observations and deductions in the table below.

Test	Observation	Deduction
a) Heat two spatula endfuls of C in a dry test tube strongly until there is no further change.		
b) To two spatula endfuls of C, add 6cm^3 of water. Shake well and then filter. Keep both the filtrate and residue. Divide the filtrate into **three** portions. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess and then warm.		

ii) To the second part, add 2 drops of lead(II) nitrate solution and warm.		
iii) Carry out a test of your own choice to the third part to confirm one of the anions present in C. …… …… …… …… ……		
c) Dissolve the residue obtained in part (b) above in $5cm^3$ of dilute nitric acid. Divide the resultant solution into **four** portions. i) To the first portion, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the third portion, add 2 to 3 drops of potassium iodide solution.		
iii) To the third portion, add dilute ammonia solution drop wise until in excess.		
iv) To the fourth portion, add solid ammonium chloride followed by 3-4drops of disodium hydrogen phosphate solution and then dilute ammonia solution drop wise until in excess.		

d)(i) Cations in C……..……..and…….…….

(ii) Anions in C……..……..and……..

Experiment 8.5.2

You are provided with substance D, which contains two cations and two anions. Carry out the following tests to identify them. Identify any gases which may be evolved. Write your observations and deductions in the table below.

Test	Observation	Deduction
a) Heat two spatula endfuls of D in a dry test tube strongly until there is no further change.		
b) To two spatula endfuls of D, add $5cm^3$ of dilute nitric acid and warm gently to dissolve. Cool the resultant solution. To $4cm^3$ of the resultant solution, add $7cm^3$ of dilute sodium hydroxide solution drop wise and then filter. Keep both the filtrate and residue.		
c) To $4cm^3$ of the filtrate obtained in (b) above, add dilute nitric acid drop by drop until the solution is just acidic. Divide the resultant solution into **six** parts. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second part, add dilute ammonia solution drop wise until in excess.		
iii) To the third part, add dilute sulphuric acid.		
iv) Carry out a test of your own choice to the third part to confirm one of the cations present in D. ……..... ……..... ……..……...…… …….		

v) To the fifth part, add a few pieces of Copper turnings followed by 3 to 4 drops of concentrated sulphuric acid and warmgently.		
vi) To the sixth part, add freshly prepared iron(II) sulphate solution; then hold the test tube in a slanting position and then add concentrated sulphuric acid drop by drop along the wall of the test tube.(Avoid shaking).		
d) Dissolve the residue you have obtained in (b) above in dilute nitric acid. Divide the resultant solution into **three** parts. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second part, add dilute ammonia solution drop wise until in excess.		
iii) To the third part, add solid ammonium chloride followed by 3-4 drops of disodium hydrogen phosphate solution and then dilute ammonia solutiondrop wise until in excess.		

e) (i) Cations in D…….....and …….……..……

(ii) Anions in D……..……..and……

Experiment 8.5.3

You are provided with substance E, which contains two cations and two anions. Carry out the following tests to identify them. Identify any gases which may be evolved. Write your observations and deductions in the table below.

Test	Observation	Deduction
a) To two spatula endfuls of E, add 6 drops of concentrated sulphuric acid and warm.		

b) Totwo spatula endful of E, add $8cm^3$ of water. Shake well and filter. Keep both the filtrate and residue.		
c) Divide the filtrate into **seven** parts. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second part, add dilute ammonia solution drop wise until in excess.		
iii) To the third part, add sodium sulphate solution.		
iv) To fourth part,add potassium chromate(VI) solution followed by dilute hydrochloric acid and then dilute sulphuric acid.		
v) To the fifth part, add 1 to 2 drops of Lead(II) nitrate solution and warm.		
vi) To the sixth part, add silver nitrate solution followed by dilute nitric acid		
vii) To the seventh part, add zinc metal powder followed by excess sodium hydroxide solution and then warm gently.		
d) Wash the residue obtained in (b) above with water and dissolve the washed residue in $5cm^3$ of dilute nitric acid and warm to dissolve. Cool the resultant solution and divide it into **four** parts. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second part, add dilute ammonia solution drop wise until in excess.		
iii) To the third part, add sodium sulphate solution.		

iv) Carry out a test of your own choice to the fourth part to confirm one of the cations present in E. ……... ……... ……... ……...		

e) (i)Cations in E…….....and ……..……

(ii) Anions in E…….and……..

Experiment 8.5.4

You are provided with substance F, which contains two cations and two anions. Carry out the following tests to identify them. Identify any gases which may be evolved. Write your observations and deductions in the table below.

Test	Observation	Deduction
a) Heat two spatula endfuls of F in a dry test tube strongly until there is no further change.		
b) To a spatula endful of F in a test tube, add 4cm^3 of dilute nitric acid.Divide the resultant solution into **two** parts. i) To the first part, add2 to 3 drops oflead(II) nitrate solution and warm, then cool under running tap water.		
ii) To the second part, add 2 to 3drops of silver nitrate solution followed by excess dilute ammonia solution.		
c) To two spatula endfuls of F, add 6cm^3 of dilute nitric acid, little at a time. To 4cm^3 of the resultant solution, add 7cm^3 of dilute sodium hydroxide solution drop wise and then filter. Keep both the filtrate and residue.		

d) To 4cm^3 of the filtrate obtained in (c) above, add dilute nitric acid drop by drop until there is no further change. Divide the resultant solution into **three** parts. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second part, add 2 to 3 drops of potassium iodide solution.		
iii) To the second part, add dilute ammonia solution drop wise until in excess.		
iv) Carry out a test of your own choice to the third part to confirm the cation present in the filtrate. ……..…... ……...…… …….. ……..		
e) Wash the residue obtained in (c) above with dilute sodium hydroxide solution and dissolve the washed residue in 5cm^3 of dilute nitric acid. Divide the resultant solution into **four** parts. i) To the first part, add dilute Sodium hydroxide solution drop wise until in excess.		
ii) To the second part, add dilute ammonia solution drop wise until in excess.		
iii) To the third part, add sodium sulphate solution.		
iv) To the fourth part, add ammonium oxalate solution followed by dilute hydrochloric acid.		

f) (i) Cations in F…….....and ……..…….

(ii) Anions in F……and……

Experiment 8.5.5

You are provided with substance G, which contains two cations and two anions. Carry out the following tests to identify them. Identify any gases which may be evolved. Write your observations and deductions in the table below.

Test	Observation	Deduction
a) Heat two spatula endfuls of G in a dry test tube strongly until there is no further change.		
b) To a spatula endful of G, add 5 drops of concentrated sulphuric acid and warm.		
c) To 3 spatula endful of G, add 8cm^3 of water. Shake well and then filter. Keep both the residue and filtrate. Divide the filtrate into **seven** portions. i) To the first portion, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second portion, add a few drops of potassium iodide solution.		
iii) To the third portion, add dilute ammonia solution drop wise until in excess.		
iv) Carry out a test of your own choice to the third portion to confirm one of the cations present in G. ……..... ……..... ……... ……..…….		

v) To the fifth portion, add neutral iron(III) chloride solution and boil.		
vi) To the sixth portion, add 2 drops of lead(II) nitrate solution followed by dilute nitric acid.		
vii) To the seventh portion, add 1cm^3 of dilute hydrochloric acid and warm.		
viii) To the eighth portion, add acidified potassium manganate(VII) solution.		
d) Wash the residue obtained in (c) above with water and to the washed residue, add5cm^3 of dilute nitric acid and warm gently to dissolve. Cool the resultant solution. Decant off the clear upper solution and divide it into **three** parts. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second part, add a little zinc powder and warm gently.		
iii) To the third part, add potassium iodide solution.		

e) (i) Cations in G…….....and ……...…….

(ii) Anions in G……and…….

Experiment 8.5.6

You are provided with substance H, which contains two cations and two anions. Carry out the following tests to identify them. Identify any gases which may be evolved. Write your observations and deductions in the table below.

Test	Observation	Deduction
a) To two spatula endfuls of H, add 5 drops of concentrated sulphuric acid and warm.		

b) To 3 spatula endfuls of H, add $9cm^3$ of water. Shake well and then filter. Keep both the residue and filtrate. Divide the filtrate into **eight** portions. i) To the first portion, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second part, add dilute ammonia solution drop wise until in excess.		
iii) To the third part, add dilute sulphuric acid.		
iv) Carry out a test of your own choice to the fourth part to confirm one of the cations present in G. ……..... ……..... ……..... ……....		
v) To the fifth part, add aluminium metal powder followed by excess dilute sodium hydroxide solution and warm.		
vi) To the sixth part, add 2 drops of lead(II) nitrate solution.		
vii) To the seventh part, add silver nitrate solution.		
viii) To the eighth part, add a spatula endful of bleaching powder (or $1cm^3$ of the bleaching agent solution) followed by $1cm^3$ of dilute nitric acid and then $1cm^3$of chloroform and shake gently.		
d) Wash the residue obtained in (c) above with water and to the washed residue, add $6cm^3$ of dilute nitric acid and warm gently to dissolve. Cool the resultant solution and Divide the cold resultant solution into **three** parts.		

i) To the first part, add dilute sodium hydroxide solution drop wise until in excess and then heat.		
ii) To the second part, add a little magnesium metal powder and leave to stand.		
iii) To the third part, add 2 to 3 drops of potassium iodide solution followed by sodium thiosulphate solution.		

e) (i) Cations in H..…….....and ……...…….

(ii) Anions in H……and……

Experiment 8.5.7

You are provided with substance I, which contains three cations and a single anion. Carry out the following tests to identify them. Identify any gases which may be evolved. Write your observations and deductions in the table below.

Test	Observation	Deduction
a) Dissolve two spatula endfuls of R in about $6cm^3$ of water. Keep the resultant solution for part (c).		
b) To $4cm^3$ of the solution obtained in (b) above, add dilute sodium hydroxide solution drop wise as you shake until in excess and then filter. Keep both the residue and filtrate.		
c) To $3cm^3$ of the filtrate obtained in (b) above, add dilute hydrochloric acid drop by drop until there is no further change. Divide the resultant solution into **six** parts. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		

ii) To the second part, add dilute ammonia solution drop wise until in excess.		
iii) To the third part, add sodium carbonate drop wise until in excess.		
iv) To the fourth part, add 2-3 of dilute nitric acid followed by 3-4 drops of litmus solution followed by dilute ammonia solution drop wise until in excess.		
v) To the fifth part, add lead(II) nitrate solution followed by dilute nitric acid.		
vi) Carry out a test of your own choice to confirm the anion present in I. ……..…….. ……..…….. ……... ……...		
d) Wash the residue obtained in (b) above with water and dissolve the washed residue in $4cm^3$ of dilute nitric acid. Divide the resultant solution into **two** parts.		
i) To the first part, add dilute sodium hydroxide solution drop wise until in excess and heat.		
ii) To the second part, add dilute ammonia solution drop wise until in excess.		
iii) To the second part, add potassium hexacyanoferrate(II) solution.		
d) Grind half a spatula end full of I with soda lime using the bottom of a test tube.		

e) (i)Cations in I…….....**,**…….. and……..……

(ii)Anion in I..……..

Experiment 8.5.8

You are provided with substance J, which contains two cations and two anions. Carry out the following tests to identify them. Identify any gases which may be evolved. Write your observations and deductions in the table below.

Test	Observation	Deduction
a) Heat two spatula endfuls of Jstrongly in a dry test tube until there is no further change.		
b) To a spatula endful of J, add 6cm^3 of dilute nitric acid and warm gently to dissolve. Cool the resultant solution. To 5cm^3 of the cold resultant solution, add dilute sodium hydroxide solution drop wise until in excess and then filter. Keep both the filtrate and residue.		
c) To the filtrate obtained in (c) above, add dilute nitric acid drop by drop to acidify. Divide the resultant solution into **seven** parts. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second part, dilute ammonia solution drop wise until in excess.		
iii) To the third part, add solid ammonium chloride followed by 3-4 drops of disodium hydrogenphosphate solution and then dilute ammonia solution drop wise until in excess.		
iv) To the fourth part, add add 1 to 2 drops of lead(II) nitrate solution followed by dilute nitric acid.		
v) To the fifth part, add Barium nitrate solution followed by dilute nitric acid.		

vi) To the sixth part, add $2cm^3$ of dilute hydrochloric acid and warm gently.		
vii) Carry out a test of your own choice to the seventh part to confirm one of the anions present in J. …… …… …… ……		
d) Wash the residue obtained in (b) above with dilute sodium hydroxide solution and dissolve the washed residue in $4cm^3$ of dilute hydrochloric acid.Divide the resultant solution into **four**parts. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second part, add dilute ammonia solution drop wise until in excess.		
iii) Carry out a test of your own choice to confirm the other cation present in J. ……. ……. ……..... ……..		

e) (i) Cations in J..…….....and ……...…….

(ii) Anions in J……and……

Experiment 8.5.9

You are provided with substance K, which contains two cations and two anions. Carry out the following tests to identify them. Identify any gases which may be evolved. Write your observations and deductions in the table below.

Test	Observation	Deduction
a) Heat two spatula endfuls of L in a dry test tube strongly until there is no further change.		
b) To 2 spatula endfuls of K, add 8cm^3 of water. Shake well and then filter. Keep both the residue and filtrate. Divide the filtrate into **four** parts. i) To the first part, add 2 to 3 drops of lead(II) nitrate solution followed by dilute nitric acid.		
ii) To the second part, add 2 to 3 drops of Barium nitrate solution followed by dilute nitric acid.		
iii) To the fourth part, add acidified potassium manganate(VII) solution and heat.		
c) Wash the residue obtained in (b) above with water and to the washed residue, add 5cm^{3}ofdilute nitric acid and warm gently to dissolve. Cool the resultant solution. To 4cm^3 of the cold resultant solution, add 7cm^3 of dilute ammonia solution drop wise and then filter. Keep both the residue and filtrate.		
d) Dissolve the residue obtained in part (c) above in about 4cm^3 of dilute hydrochloric acid. Divide the resultant solution into**three** parts. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		

ii) To the second part, add dilute ammonium hydroxide solution drop wise until in excess.		
iii) To the third part, add concentrated nitric acid followed by solid sodium bismuthate and warm.		
d) To the filtrate obtained in (c) above, add dilute nitric acid drop by drop to acidify. Divide the acidic solution into **four**portions. i) To the first portion, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second portion, add sodium carbonate solution drop wise until in excess.		
iii) To the third portion, add dilute ammonium hydroxide solution drop wise until in excess.		
iv) Carry out a test of your own choice to the fourth portion to confirm one of the cations present in K. ……. …… ……. ……		

e) (i) Cations in K…….....and ……...…….

(ii) Anions in K…….and…….

Experiment 8.5.10

You are provided with substance L, which contains three cations and a single anion. Carry out the following tests to identify them. Identify any gases which may be evolved. Write your observations and deductions in the table below.

Test	Observation	Deduction
a) Heat two spatula endfuls of L in a dry test tube strongly until there is no further change.		
b) Grind half a spatula endful of L with soda lime using the bottom of a test tube.		
c) To two spatula endful of L, add $7cm^3$ of water. Shake well and then filter. Keep both the residue and filtrate.		
d) Divide the filtrate into **two parts, each of $1cm^3$.** i) To the first part, add 1-2 drops of lead(II) nitrate solution and warm, then cool under running tap water.		
ii) To the second part, add dilute nitric acid followed by silver nitrate solution.		
e) Wash the residue obtained in (c) above with water and to the washed residue, add $6cm^3$ of dilute nitric acid and heat for two minutes to disolve. Cool the resultant solution. To $3cm^3$ of the cold resultant solution, add $7cm^3$ of dilute ammonia solution drop wise and then filter. Keep both the residue and filtrate.		
f) To the filtrate obtained in (e) above, add dilute nitric acid drop by drop until the solution is just acidic. Divide the acidic solution into **four** parts.		

i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second part, add dilute ammonia solution drop wise until in excess.		
iii) To the third part, add sodium sulphate solution.		
iv) To the fourth part, add potassium iodide solution drop wise until in excess.		
g) Dissolve the residue obtained in part (e) above in about $4cm^3$ of dilute hydrochloric acid. Divide the resultant solution into **three** parts. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess and heat.		
ii) To the second part, add dilute ammonium hydroxide solution drop wise until in excess.		
iii) To the third part, add excess concentrated hydrochloric acid.		

h) (i) Cations in L……....**,** .....and ……..……..

(ii) Anions in L……

Experiment 8.5.11

You are provided with substance M, which contains two cations and two anions. Carry out the following tests to identify them. Identify any gases which may be evolved. Write your observations and deductions in the table below.

Test	Observation	Deduction
a) Heat two spatula endfuls of M in a dry test tube strongly until there is no further change.		

b) To a spatula endful of M, add 3 to 5 drops of concentrated sulphuric acid and warm.		
c) Totwo spatula endfuls of M, add 10cm^3 of water. Shake well and filter. Keep both the filtrate and residue. Divide the filtrate into **seven** portions. i) To the first portion, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second portion, add sodium carbonate solution drop wise until in excess.		
iii) To the third portion, add solid ammonium chloride followed by 3-4 drops of disodium hydrogen phosphate solution and then dilute ammonia solution drop wise until in excess.		
iv) To the fourth portion, add neutral iron(III) chloride solution.		
v) To the fifth portion, add ethanol followed by 5 drops of concentrated sulphuric acid and heat. Pour the product in a beaker of cold water.		
vi) To the sixth portion, add 2 drops of lead(II) nitrate solution and warm.		
vii) Carry out a test of your own choice to the seventh portion to confirm one of the anions present in M. ……..... ……..... ……..... ……..		
d) Wash the residue obtained in (c) above and to the washed residue, add 3cm^3 of dilute nitric acid and warm to dissolve, then cool the resultant solution. Divide the cold resultant solution into **three** portions. i) To the first portion, add sodium hydroxide solution drop wise until in excess.		

ii) To the second portion, add dilute ammonia solution drop wise until in excess.		
iii) To the third portion, add excess concentrated hydrochloric acid.		

e) (i) Cations in M…….....and ……..…….

(ii) Anions in M…….and……

Experiment 8.5.12

You are provided with substance N, which contains two cations and two anions. Carry out the following tests to identify them. Identify any gases which may be evolved. Write your observations and deductions in the table below.

Test	Observation	Deduction
a) Heat two spatula endfuls of M in a dry test tube strongly until there is no further change.		
b) To a spatula endful of M, add 5cm^3 of water and shake well to mix and then filter. Keep both the filtrate and residue.		
c) Divide the filtrate obtained in (b) above into **three** portions. i) To the first portion, add lead(II) nitrate solution.		
ii) To the second portion, add Silver nitrate solution.		
iii) To the third portion, add a little bleaching powder (or 1cm^3 of a solution of a bleaching agent), followed by 1cm^3 of dilute nitric acid and then 1cm^3 of chloroform and shake gently.		

d) Wash the residue obtained in (b) above with water, and to the washed residue, add 5cm^3 of dilute nitric acid. Shake gently for one minute. Warm patientlyto dissolve and then cool the resultant solution.		
e) To 3cm^3 of the cool resultant solution obtained in (d) above, add 7cm^3 of dilute sodium hydroxide solution drop wise as you shake and then filter. Keep both the residue and filtrate.		
(f) Dissolve the residue obtained in (e) above in about 3cm^{3}of dilute nitric acid. Divide the resultant solution into **three** parts. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second part, add dilute ammonia solution drop wise until in excess.		
iii) Carry out a test of your own choice to the third part to confirm one of the cations present in N. …….. …….. …….. ……..		
g) To 4cm^3 of the filtrate obtained in (e) above, add dilute nitric acid drop by drop to acidify. Divide the acidic solution into **three** parts. i) To the first part, add dilute sodum hydroxide solution drop wise until in excess		
ii) To the second part, add dilute ammonia solution drop wise until in excess.		

iii) Carry out a test of your own choice to the fourth part to confirm the other cation present in N. …… …… …… ……		

h) (i) Cations in N…….....and ……...…….

(ii) Anions in N.……and……

Experiment 8.5.13

You are provided with substance P, which contains two cations and two anions. Carry out the following tests to identify them. Identify any gases which may be evolved. Write your observations and deductions in the table below.

Test	Observation	Deduction
a) To two spatula endfuls of P, add 7cm^3 of water and shake well; then filter. Keep both the filtrate and residue.		
b) Divide the filtrate obtained in (a) above into **four** parts. i) To the first part, add lead(II) nitrate solution followed by dilute nitric acid.		
ii) To the second part, add Barium nitrate solution followed by dilute nitric acid.		
iii) To the third part, add 2 drops of silver nitrate solution followed by 5 drops of dilute nitric acid.		
iv) To the fourth part, add ammonium molybdate solution followed by concentrated nitric acid and warm.		

c) Wash the residue obtained in (a) above with water and to the washed residue, add 6cm^3 of dilute nitric acid, then warm to dissolve better. Cool the resultant solution. To 4cm^3 of the cold resultant solution, add dilute ammonia solution drop wise until in excess and then filter. Keep both the filtrate and residue.		
d) To 4cm^3 of the filtrate, add dilute nitric acid dop by drop to acidify. Divide the acidic solution into **four** parts. i) To the first part, add dilute sodium hydroxide solution dropwise until in excess.		
ii) To the second part, add potassium iodide solution.		
iii) To the third part, add dilute ammonia solution drop wise untilin excess.		
iv) Carry out a test of your own choice to the fourth part to confirm one of the cations present in P. ……..... ……..... ……..... …….....		
d) Wash the residue obtained in (c) above with dilute ammonia solution, and to the washed residue, add 5cm^3 of dilute nitric acid. Divide the resultant solution into **four** parts. i) To the first portion, add dilute sodium hydroxide solution drop wise until in excess.		

ii) To the second part, add dilute ammonia solution drop wise until in excess.		
iii) To the third part, add sodium sulphate solution.		
iv) Carry out a test of your own choice to the fourth part to confirm one of the cations present in P. ……..... ……..... ……..... ……		

e) (i) Cations in P…….....and ……...…….

(ii) Anions in P……and……

Experiment 8.5.14

You are provided with substance Q, which contains two cations and two anions. Carry out the following tests to identify them. Identify any gases which may be evolved.

Test	Observation	Deduction
a) Totwo spatula endfuls of Q, add 7cm^{3}of water. Shake well and then filter. Keep both the filtrate and residue.		
b) Divide the filtrate obtained in (b) above into **six** parts. i) To the first part, add sodium hydroxide drop wise until in excess.		
ii) To the second part, add dilute ammonia solution drop wise until in excess.		

iii) To the third part, add sodium carbonate solution drop wise until in excess.		
iv) To the fourth part, add excess sodium hydroxide solution, followed by hydrogen peroxide solution and warm, then add amylalcohol followed by dilute sulphuric acid.		
v) To the fifth part, add lead(II) nitrate solution followed by dilute nitric acid.		
vi) Carry out a test of your own choice to the sixth part, to confirm one of the anions present in Q. …… …… …… ……		
c) Wash the residue obtained in (a) above with water and dissolve the washed residue in $4cm^3$ of dilute nitric acid. Divide the resultant solution into **four** parts. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second part, add dilute ammonia solution drop wise until in excess.		
iii) To the third part, add sodium sulphate solution.		
iv) Carry out a test of your own choice to the fourth part to confirm the cation present in the residue. …… …… ……		

d) (i) Cations in Q…….....and ……...……....

(ii) Anions in Q.……and……

Experiment 8.5.15

You are provided with substance R, which contains two cations and two anions. Carry out the following tests to identify them. Identify any gases which may be evolved.

Test	Observation	Deduction
a) To a spatula endfuls of R, add 7cm^3 of water. Shake well and then filter. Keep both the filtrate and residue.		
b) Divide the filtrate obtained in (a) above into **three**parts. i) To the first part, add lead(II) nitrate solution followed by dilute nitric acid.		
ii) To the second part, add Barium nitrate solution followed by dilute nitric acid.		
iii) Carry out a test of your own choice to the third part to confirm one of the anions present in R. ……..... ……..... ……..... …….....		
c) Wash the residue obtained in (a) above with water and to the washed residue, add 6cm^3 of dilute nitric acid and warm to dissolve; then cool the resultant solution. To 4cm^3 of the cold resultant solution, add 7cm^3 of dilute sodium hydroxide solution drop wise and then filter. Keep both the filtrate and residue.		
d) To 4cm^3 of the filtrate obtained, add dilute sulphuric acid drop by drop until the solution is just acidic. Divide the acidic solution into **four**parts.		

i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second part, add sodium carbonate solution drop wise until in excess.		
iii) To the third part, add dilute ammonia solution drop wise until in excess.		
iv) Carry out a test of your own choice to the fourth part to confirm one of the cations present in R. …… …… ……..... ……		
e) Wash the residue obtained in (c) above with dilute sodium hydroxide solution and to the washed residue, add 4cm^3 of dilute nitric acid and shake well to dissolve. Divide the resultant solution into **four** parts. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second part, add dilute ammonia solution drop wise until in excess.		
iii) To the third part, add sodium sulphate solution.		
iv) To the fourth part, add solidammonium chloride followed by 3-4 drops of disodium hydrogenphosphate solution and then dilute ammonia solution drop wise until in excess.		

f) (i) Cations in R…….....and ……...…….

(ii) Anions in R.……and……

Experiment 8.5.16

You are provided with substance S, which contains two cations and two anions. Carry out the following tests to identify them. Identify any gases which may be evolved.

Test	Observation	Deduction
a) To a spatula endful of **S**, add 5 drops of concentrated sulphuric acid and warm.		
b) To two spatula endfuls of S, add $6cm^3$ of water and shake well to dissolve. To $5cm^3$ of the resultant solution, add $8cm^3$ of dilute sodium hydroxide solution drop wise and then filter. Keep both the residue and filtrate.		
c) Dissolve the residue obtained in (b) above in $4cm^3$ of dilute sulphuric acid. Divide the resultant solution into **three** parts. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second part, add dilute ammonium hydroxide solution drop wise until in excess.		
iii) To the third part, add a few drops of Potassium hexacyanoferrate(III) solution.		
d) To the filtrate obtained in (b) above, add dilute nitric acid drop by drop until there is no further change. Divide the resultant solution into **seven** parts. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second part, add dilute ammonium hydroxide solution drop wise until in excess.		

iii) To the third part, add dilute sulphuric acid.		
iv) To the fouth part, add Devarda's alloy followed by excess sodium hydroxide solution and boil.		
v) To the fifth part, add lead(II) nitrate solution followed by dilute nitric acid.		
vi) Carry out a test of your own choice to the sixth part, to confirm one of the anions present in S. …… …… …… ……		
vii) To the seventh part, silver nitrate solution followed by excess ammonia solution.		

e) (i) Cations in S…….....and ……...…….

(ii) Anions in S.……and……

Experiment 8.5.17

You are provided with substance T, which contains three cations and two anions. Carry out the following tests to identify them. Identify any gases which may be evolved.

Test	Observation	Deduction
a) Heat two spatula endfuls of T strongly in a dry test tube until there is no further change.		
b) To a spatula endful of T, add 5 drops of concentrated sulphuric acid and warm.		

c) To three spatula endfuls of T in a boiling tube, add 12cm^3 of water and shake well; then filter. Keep both the residue and filtrate. Divide the filtrate obtained above into **nine** parts. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To second part, add sodium carbonate solution drop wise until in excess.		
iii) To the third part, add dilute ammonium hydroxide solution drop wise until in excess.		
iv) Carry out a test of your own choice to thefourth part to confirm one of the cations present in T. …………………………………….......... …………………………………….......... ……………………………………........ ……………………………………........		
v) To the fifh part, add ethanol followed by 5 drops of concentrated sulphuric acid and heat. Pour the product in a beaker containing cold water.		
vi) Carry out a test of your own choice to the sixth part to confirm one of the anions present in T. …………………………………….......... …………………………………….......... ……………………………………..........		
vii) To theseventh part, add 2 to 3 drops of dilute nitric acid followed by lead(II) nitrate solution.		
viii) To the eighth part, add 2 to 3 drops of dilute nitric acid followed by silver nitrate solution.		

ix) To the ninth part, add a little bleaching powder (or 1cm^3 of the bleaching agent solution) followed by 1cm^3 of dilute nitric acid and then 1cm^3 of chloroform. Shake gently.		
d) Wash the residue obtained in (c) above with water and dissolve the washed residue in 3cm^3 of dilute nitric acid. Divide the resultant solution into **four** parts. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second part, add zinc metal powder and warm gently.		
iii) To the third part, add dilute ammonia solution drop wise until in excess.		
iv) To the fourth part, add potassium hexacyanoferrate(II) solution.		

e) (i) Cations in T…….....and ……...…….

(ii) Anions in T…….and…….

Experiment 8.5.18

You are provided with substance U, which contains two cations and two anions. Carry out the following tests to identify them. Identify any gases which may be evolved.

Test	Observation	Deduction
a) To a spatula endful of U, add 7cm^3 of water. Shake well and then filter. Keep both the filtrate and residue.		
b) To the filtrate obtained in (a) above, add dilute sodium hydroxide solution drop wise until in excess and then filter. Keep both the filtrate and residue.		

c) To the filtrate obtained in (b) above, add dilute nitric acid drop by drop until the solution is just acidic. Divide the acidic solution into **six** parts. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second part, add potassium iodide solution.		
iii) To the third part, add dilute ammonia solution drop wise until in excess.		
iv) To the fourth part, add solid ammonium chloride followed by 3-4 drops of disodium hydrogen phosphate solution and then dilute ammonia solution drop wise until in excess.		
ii) To the fifth part, add 2 to 3 drops of lead(II) nitrate solution and warm.		
iii) Carry out a test of your own choice to the sixth part to confirm one of the anions present in U. ……..... ……..... ……..... ……..... 		
d) Wash the residue obtained in (b) above with d andilute sodium hydroxide solution andto the washed residue, add $5cm^3$ of dilute nitric acidand divide the resultant solution into **four** parts. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		

ii) To the second part, add sodium carbonate solution drop wise until in excess.		
iii) To the third part, add dilute ammonia solution drop wise until in excess.		
iv) Carry out a test of your own choice to the fourth part to confirm one of the cations present in U. …… …… …… ……		
e) Wash the residue obtained in (a) above with waterand to the washed residue, add 4cm^3 of dilute nitric acid and shake well to dissolve. Divide the resultant solution into **four** parts. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second part, add sodium carbonate solution drop wise until in excess.		
iii) To the third part, add dilute ammonia solution drop wise until in excess.		
iv) To the fourth part, add potassium iodide solution followed by sodium thiosulphate solution.		

f) (i) Cations in U…….....and ……...…….

(ii) Anions in U.……and……

Experiment 8.5.19

You are provided with substance V, which contains two cations and two anions. Carry out the following tests to identify them. Identify any gases which may be evolved.

Test	Observation	Deduction
a) To a spatula enful of S, add 5 drops of concentrated sulphuric acid and warm.		
b) To two spatula endfuls of S, add 7cm^3 of dilute nitric acid and warm to dissolve. To 5cm^{3}of the resultant solution, add dilute ammonia solution drop wise until in excess and then filter. Keep both the residue and filtrate.		
c) To 4cm^3 of the filtrate obtained in (b) above, add dilute nitric acid drop by drop until the solution is just acidic. Divide the resultant solution into **five**parts. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second part, add dilute ammonia solution drop wise until in excess.		
iii) To the third part, add excess concentrated hydrochloric acid.		
iv) To the fourth part, add 2 to 3 drops of lead(II) nitrate solution.		
v) To the fifth part, add silver nitrate solution followed by dilute nitric acid.		
d) Wash the residue obtained in (b) above twice with water and dissolve the washed residue in dilutenitric acid. Divide the resultant solution into **four** parts. i) To the first part, add dilute sodium hydroxide solution drop wise until in excess.		

ii) To the second part, add dilute ammonium hydroxide solution drop wise until in excess.		
iii) To the third part, add sodium sulphate solution.		
iv) Carry out a test of your own choice to the fourth part to confirm one of the cations present in V. ……..... ……..... ……		
e) To a spatula endful of V, add $5cm^3$ of water. Shake well and filter. Keep the filtrate, but discard the residue. Divide the filtrate into **two** parts. i) To the first part, add a few copper turnings followed by concentrated sulphuric acid and then warm.		
ii) Carry out a test of your own choice to the second part to confirm one of the anions present in V. …… …… …… ……		

e) (i) Cations in V…….....and ……...…….

(ii) Anions in V…….and……

Experiment 8.5.20

You are provided with substance W, which contains two cations and two anions. Carry out the following tests and identify them. Identify any gases which may be evolved. Write your observations and deductions in the table below.

Test	Observation	Deduction
a) Heat a spatula endful of W in a dry test tube strongly until there is no further change.		
b) To a spatula endful of W, add 3 to 5 drops of concentrated sulphuric acid and warm gently.		
c) To two spatula endfuls of B_2, in a boiling tube, add $5cm^3$ of water and shake well to dissolve. To $4cm^3$ of the resultant solution, add dilute ammonia solution drop wise until in excess and then filter.Keep both the filtrate and residue.		
d) To $5cm^3$ of the filtrate obtained in (c) above, add dilute nitric acid drop wise until the solution is just acidic. Divide the acidic solution into **seven** portions. i) To the first portion, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second portion, add add dilute ammonia solution drop wise until in excess.	.	
iii) Carry out a test of your own choice to the thirdportion to confirm one of the cations present in B_2. ……... ……..... ……... ……...		

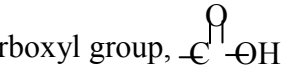
iv) To the fourth portion, add 3-4 drops of lead(II) nitrate solution and warm.		
v) To the fifthportion, add barium nitrate solution followed by diute nitric acid.		
vi) To the sixth portion, add 2-3 drops of siver nitrate solution followed by dilute ammonia solution dropwise until in excess.		
(vii) To the seventh portion, add 2 drops of a solution of a bleaching agent, followed by 2 drops of dilute nitric acid and then 3-4 drops of chloroform and shake gently.		
e) Wash the residue obtained in (c) above with dilute ammonia solution and dissolve the washed residue in dilute sulphuric acid. Divide the resultant solution into **three** portions. i) To the first portion, add dilute sodium hydroxide solution drop wise until in excess.		
ii) To the second portion, add dilute ammonia solution drop wise until in excess.		
iii) To the third portion, add 2-3 drops of litmus solution followed by dilute ammonia solution drop wise until in excess.		

Cations in W……..…….and..……

Anions in W…….……...and ……. ……..

CHAPTER NINE
9.0 ORGANIC QUALITATIVE ANALYSIS

9.1 INTRODUCTION
The categories of organic compounds dealt with at this level include: alcohols, carbonyls (aldehydes and ketones), carboxylic acids and salts of carboxylic acids,halocarboxylic acids (e.g chloroethanoic acid),phenols/phenolic compounds, amines, e.t.c.

9.1.1 Key aims of analyzing Organic Compounds
Analysis of organic compounds aims at the following:
1) Categorising an organic compound as either aliphatic or aromatic.
2) Determination of the carbon to hydrogen ratio in an organic compound.
3) Categorising an organic compound as either saturated or unsaturated.
4) Identification of the functional group of an organic compound. The functional groups include the following:
    - Hydroxyl group, -OH
    - Carbonyl group, 
    - Carboxyl group, 
    - Amino group,-NH_2
5) Distinguishing between different classes of organic compounds, for instance primary, secondary or tertiary alcohols.

9.2 PRELIMINARY TESTS

9.2.1 Physical appearance of the compound
The physical appearance of an organic compound at room temperature may give us an overview of the nature of organic compound.

Observation	Deduction
A colourless liquid.	Lower aliphatic compound (Aliphatic compound with 6 carbon atoms or less)
Solid compound.	Aromatic compound **or**higher aliphatic compound (Aliphatic compound with more than 6 carbon atoms)**or**organic salt.
Pink crystals which dissolve to form a pink solution on exposure to the atmosphere.	Phenol probably present.

9.2.2 The Odour /Smell of an organic compound

There is a great variety of odours of organic compounds in spite of the fact that not all of them can be easily described. Below are some of the common odours and the corresponding organic compounds that they point to.

Observation	Deduction
Carbolic smell	Phenol
Antiseptic smell	Triiodomethane
Sweet, fruity smell	Ester
Sweet smell (but not fruity)	Ketones, aromatic aldehydes **or**lower alkylhalides
Pungent	Lower carboxylic acids and acid chlorides
Smell of petrol	Liquid alkanes
Fishy smell	Amines
Odourless	Ionic organic compound

9.2.3 The Flame Test

The flame Test is used for:

- Categorising an organic compound as either aliphatic or aromatic.
- Determination of the carbon to hydrogen ratio (carbon content) in an organic compound.
- Categorising an organic compound as either saturated or unsaturated.

In the flame test, a small amount of an organic compound is burnt on a spatula end or in a crucible. The observation made should focus on the colour of the flame, but most importantly whether the flame is sooty or non-sooty.

Test	Observation	Deduction
Burn a small amount of the organic compound on a spatula end or crucible	Burns readily with a blue non-sooty flame.	Aliphatic, saturated compound with a low carbon to hydrogen ratio(with a low carbon content).
	Burns readily with a yellow sooty flame.	Aromatic, unsaturated compound with a high C:H ratio (with a high carbon content)**or**long chain aliphatic, unsaturated compound with a high C:H ratio (with a high carbon content).
	Burns with great difficulty with a smell of burnt sugar.	Carbohydrate in form of a sugar present e.g. Glucose or Sucrose.
	Does not burn.	Alkyl halide, nitrogen containing compound **or** salt of carboxylic acid.

9.2.4 Solubility in water

The solubility of an organic compound in water can help us to analyse an organic compound in the following aspects:

- To determine whether the compound is polar or non-polar.
- If the compound is polar, we can determine whether it is a polar aliphatic or a polar aromatic compound.
- If the compound is polar and aliphatic, we can determine whether it is of a low molecular weight/low molecular mass or a high molecular weight/high molecular mass.

In principle, an organic compound that is soluble in water or miscible with water should be polar since water is a polar solvent.

Therefore, if a compound is soluble in water or miscible with water, then it is a polar aliphatic compound with a low molecular weight (low molecular mass).

If the compound is partially soluble in water or partially miscible with water, then it is a polar aromatic compound or a polar aliphatic compound with a high molecular weight (high molecular mass)

Note: The lower the molecular weight (molecular mass), the shorter the hydrophobic hydrocarbon portion of the molecule and hence the greater the solubility in water (the greater the miscibility with water) and the higher the molecular weight (molecular mass), the longer the hydrophobic hydrocarbon portion of the molecule and hence the lower the solubility in water (the lower the miscibility with water).

If the compound is insoluble in water or immiscible with water, then the compound is on-polar.

Note:1) The word soluble, partially solubleand insoluble in water are used when dealing with a solid organic compound while the words miscible, partially miscible or immiscible with water are used when dealing with a liquid organic compound.

2) The hydroxyl group, carbonyl group, carboxyl group and amino group are always associated with polarity whenever they are present in a molecule. In general, therefore, functional groups that contain oxygen atoms and nitrogen atoms are significantly polar and hence molecules that possess such functional groups tend to dissolve readily in wat

Test	Observation	Deduction
To 1cm^3 of the organic compound, add 2cm^3 of water.	Soluble in water/miscible with water to form a colourless solution	Polar aliphatic compound with a low molecular mass e.g.alcohol, carbonyl, carboxylic acid **or**ester.
To 1cm^3 of the organic compound, add 2cm^3 of water.	Partially miscible with water to form a colourless solution	Polar aliphatic compound with a high molecular mass e.g. alcohol, carbonyl, carboxylic acid**or**ester.
Shake a spatula endful of the organic compound with 4cm^3 of water.	Sparingly soluble in water to form a colourless solution	Polar aromatic compound e.g. aromatic carbonyl compound, carboxylic acid, ester**or**phenol.
To 1cm^3 of the organic compound, add 2cm^3 of water.	Immiscible with water	Non-polar aliphatic compound.

Shake a spatula endful of the organic compound with $4cm^3$ of water.	Insoluble in water	Non-polar aromatic compound

9.2.5Use of Indicators

The most commonly used indicators include litmus paper and Universal indicator.

Indicator Used			Deduction
Litmus paper	**Litmus solution**	**Universal Indicator Solution**	
No effect on both blue and red litmus paper.	No effect on both blue and red litmus solution.	Solution remains green.	Alcohols, carbonyl **or** Ester.
Blue litmus paper turns red/pink.	Blue litmus solution turns red.	Solution turns red or pink or orange.	Carboxylic acid **or**phenol.
Red litmus paper turns blue.	Red litmus solution turns blue.	Solution turns blue, purple or violet.	Amine **or**salt of carboxylic acid.

9.3 REAGENTS USED TO TEST FOR ORGANIC COMPOUNDS

a) Action of sodium hydroxide solution

Sodium hydroxide solution is used to test for the presence of carboxylic acids and phenol in a neutralisation reaction.

If the compound is a lower aliphatic carboxylic acid, then the colourless solution of the carboxylic acid is miscible with sodium hydroxide solution to form a colourless solution with no gas or vapour evolved.

If the compound is an aromatic carboxylic acid or phenol, the solid dissolvesin the sodium hydroxide solution on warming to form a colourless solution with no gas or vapour evolved.

If the compound is an ester, the compound loses its sweet, fruity smell on boiling. This is due to an ester hydrolysis reaction which results in formation of an alcohol and a salt of a carboxylic acid.

If the compound is an aliphatic amine, the compound is miscible with sodium hydroxide solution on warming with evolution of a colourless gas that turns moist red litmus paper blue (The gas evolved is an ammonium salt which is alkaline).

If the compound is an amide, the compound is miscible with sodium hydroxide solution and on warming, a colourless gas which turns moist red litmus paper blue.

Test	Observation	Deduction
To 1cm^3 of the organic compound, add 4cm^3 of sodium hydroxide solution.	Miscible to form a colourless solution with no evolution of a gas.	Neutralisation reaction. aliphatic carboxylic acid**or**phenol present.
To a spatula endful of the solid, add 4cm^3 of sodium hydroxide solution and warm.	Dissolves to form a colourless solution without evolution of a gas.	Neutralisation reaction aromatic carboxylic acid **or** phenol present.
To 1cm^3 of the organic compound, add 4cm^3 of sodium hydroxide solution and boil/heat.	Miscible to form a colourless solution without evolution of a gas and on boiling/heating, the sweet fruity smell is lost.	Ester hydrolysis to form alcohol and sodium salt of a carboxylic acid. Ester present.
To 1cm^3 of the organic compound, add 4cm^3 of sodium hydroxide solution.	Miscible with evolution of a colourless gas that turns moist red litmus paper blue.	Aliphatic amine present.
To 1cm^3 of the organic compound, add 4cm^3 of sodium hydroxide solution and warm.	Miscible on warming with evolution of a colourless gas that turns moist red litmus paper blue.	Aliphatic amide present.

b) Action of sodium carbonate or sodium hydrogencarbonate

The sodium carbonate or sodium hydrogencarbonate may either be added in solid or solution form. Carboxylic acids ionize partially to form hydrogen ions which react with sodium carbonate or sodium hydrogencarbonate, evolving carbon dioxide gas (a colourless gas which turns moist blue litmus paper red and limewater milky).

$RCOOH(aq) \rightleftharpoons RCOO^-(aq) + H^+(aq)$
$2H^+(aq) + CO_3^{2-}(aq) \rightarrow H_2O(l) + CO_2(g)$
$H^+(aq) + HCO_3^-(aq) \rightarrow H_2O(l) + CO_2(g)$

Test	Observation	Deduction
To 1cm^3 of the solution, add a little solid sodium carbonate/sodium hydrogencarbonate.	Effervescence of a colourless gas which turns moist blue litmus paper red and limewater milky.	$CO_2(g)$ evolved. Carboxylic acid present.
ORTo 1cm^3 of the solution, add sodium carbonate/sodium hydrogencarbonate solution.		
To 1cm^3 of the solution, add a little solid sodium carbonate/sodium hydrogencarbonate.	No observable change.	Carboxylic acid absent.
OR To 1cm^3 of the solution, add sodium carbonate/sodium hydrogencarbonate solution.		

Note: In spite of being acidic, phenol is a weak acid which cannot liberate carbon dioxide gas from sodium carbonate or sodium hydrogencarbonate.

c) Action of iron(III) chloride solution

Iron(III) chloride solution is used to test fora phenol (or phenolic compound). Formation of a violet colouration (purple colouration) indicates presence of a phenol/phenolic compound. While testing for phenol (or phenolic compounds), no heating is required.

Test	Observation	Deduction
To 1cm^3 of the solution, add iron(III) chloride solution.	A violet colouration (purple colouration) is formed.	Phenol/phenolic compound present.
	No observable change.	Phenol/phenolic compoundabsent.

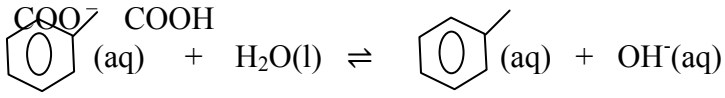
d)Action of neutral iron(III) chloride solution

Neutral iron(III) chloride solution is used to test for a phenol (or phenolic compound), salts of carboxylic acids (for instance the **benzoate ion and alkanoates** such as **ethanoate and methanoate)**or aliphatic carboxylic acids.

If addition of neutral iron(III) chloride solution is not followed by heating, then the test is for a phenol (or phenolic compound) whereby formation of a violet colouration (purple colouration) indicates presence ofa phenol (or phenolic compound) yet if there is no observable change (if no violet/purple colouration is formed), this indicates absence of aphenol (or phenolic compound).

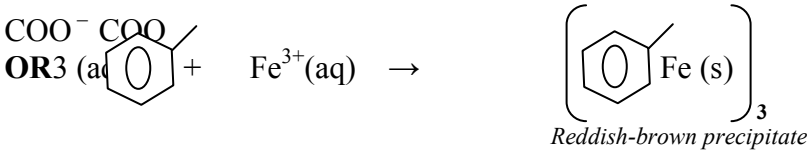
On the other hand, if addition of neutral iron(III) chloride solution is followed by heating, then the test is for either a salt of a carboxylic acid (e.g. benzoate, ethanoate or methanoate) or an aliphaticcarboxylic acidwhereby formation of a brown precipitate (reddish-brown precipitate) indicates the presence of a salt of a carboxylic acid (e.g. benzoate, ethanoate or methanoate) while if red colouration is formed which turns to a reddish-brown precipitate on heating, then an aliphatic carboxylic acid is present.

(a) The reddish-brown precipitate (brown precipitate) is due to the following sequence of reactions:



$$Fe^{3+}(aq) + 3OH^-(aq) \rightarrow Fe(OH)_3(s)$$

Reddish-brown precipitate



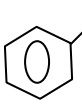
Reddish-brown precipitate

(b) The red colourationwhich turns to a reddish-brown precipitate on heating is due to the following reaction.

$$3RCOOH(aq) + Fe^{3+}(aq) \rightarrow (RCOO)_3Fe(s) + 3H^+(aq)$$

Brown precipitate

Test	Observation	Deduction

To 1cm^3 of the solution, add neutral iron(III) chloride solution and heat.	A brown precipitate is formed on heating.	 COO^- present
	No observable change evwn on heating.	 COO^- **Or** salt of aliphatic carboxylic acid absent.
	A red colouration which turns to a reddish-brown precipitate on heating	Aliphatic carboxylic acid present

e) Action of 2,4-dinitrophenylhydrazine solution (Brady's Reagent)

2,4-dinitrophenylhydrazine solution is used to test for the presence of carbonyl compounds.

When 2, 4-dinitrophenylhydrazine solution is added to a solution of a carbonyl compound, whether it is a ketone or aldehyde, a yellow precipitate or orange precipitate is formed. A yellow precipitate is usually formed from an aliphatic carbonyl compound while an orange precipitate is usually formed from an aromatic carbonyl compound.

Test	Observation	Deduction
To 1cm^3 of the solution, add 2-3 drops of Brady's Reagent.	A yellow precipitate (or an orange precipitate) is formed.	Carbonyl compound present.
	No observable change.	Carbonyl compound absent.

f) Action of saturated sodium hydrogensulphite solution

Saturated sodium hydrogensulphite solution is another reagent that can be used to confirm the presence of carbonyl compounds. All carbonyl compounds, whether **ketones or aldehydes,** form a **white precipitate** when saturated sodium hydrogensulphite solution is added to them.

Test	Observation	Deduction
To 1cm^3 of the solution, addsaturated sodium hydrogensulphite solution.	A white precipitate is formed.	Carbonyl compound present.
	No observable change.	Carbonyl compound absent.

Note: Acidified sodium sulphite solution can also be used for the same purpose in case saturated sodium hydrogensulphite solution is not available.

g) Action of ammoniacal silver nitrate solution (Tollen's Reagent)

Since aldehydes and ketones behave similarly in several aspects, there is need for a mechanism of differentiating between them. Tollen's Reagent is one of those reagents that can be used to differentiate between the two. When Tollen's Reagent is added to a solution containing an aldehyde and the mixture warmed and the mixture allowed tostand, a **silver mirror** is formed at the walls of the test tube. Sometimes instead of a silver mirror, a grey precipitate is observed. Methanoic acid and aldose sugars such as Glucose also give a positive test with Tollen's Reagent since these compounds also possess aldehyde groups.

Note: 1) In some situations, the student may be indirectly required to prepare his/her own Tollen's reagent.; for instance, the procedure may read: *To 1cm^3 of silver nitrate solution, add 1cm^3 of sodium hydroxide solution followed by diluteammonia solution drop wise until the precipitate just dissolves, then add 2cm^3 of the test solution and warm, then allow to stand.*

2) Aldose sugars, due to possession of an aldehyde group which has reducing properties, are also called Reducing sugars.

For Methanoic acid,

$$HCOOH(aq) + 2Ag^+(aq) + 2OH^-(aq) \rightarrow 2Ag(s) + 2H_2O(l) + CO_2(g)$$

Test	Observation	Deduction
To 1cm^3 of the solution, add 2cm^3 of Tollen's Reagent and warm; then allow to stand.	A silver mirror is formed on the walls of the test tube	Reducing agent present such as aldehyde, methanoic acid **or** Reducing sugar (such as Glucose).

h) Fehling's solution

Fehling's solution can also be used effectively in differentiating between aldehydes and ketones. When Fehling's solution is added to an aldehyde and the mixture boiled, a reddish-brown precipitate (red precipitate) is formed. This observation, however, only applies to aliphatic aldehydes and aldose sugars (reducing sugars) such as glucose, but does not apply to aromatic aldehydes in which the aldehyde group is directly attached to the Benzene ring (Benzaldehyde and its derivatives).

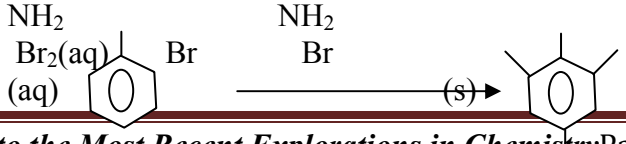
Test	Observation	Deduction
To 1cm^3 of the solution , add 2cm^3 of Fehling's solution and boil	A reddish-brown precipitate is formed.	Reducing agent present such asaliphatic aldehyde, **or** reducing sugar (such as Glucose)

i)Action of bromine water

Bromine water is used to test for unsaturation whereby when bromine water is reacted with any compound with multiple carbon-carbon bonds (double or triple carbon-carbon bonds), the reddish-brown solution of bromine water turns colourless.

$$RCH{=}CHR' \xrightarrow{Br_2/H_2O} \underset{OH\ Br}{RCH\text{-}CHR'}$$

However, in the practical paper, it is also common to use bromine water to test for aliphatic amines, aromatic amines and phenols in which case, apart from the Bromine water turning from brown to colourless, there are other observations made, for instance formation of white fumes or formation of a white precipitate, depending on the type of organic compound present. For aryl amines (aromatic amines) and Phenol, a white precipitate is formed. This is due to formation of a 2,4,6-tribromoproduct on addition of excess Bromine water.



Br

White precipitate of 2,4,6-tribromoaminobenzene

OHOH

Test	Observation	Deduction
To 1cm^3 of the solution **or** to half a spatula endful of the solid, add Bromine water, little at a time, shaking after each addition (until the Bromine water has turned from brown to colourless).	Brown solution turns colourless; with no white fumes; and formation of a second liquid layer.	Unsaturated compound with multiple carbon-carbon bonds e.g. alkene **or**alkyne.
	Brown solution turns colourless; with formation of white fumes; and a product that is completely miscible with water.	Aliphatic amine **or** aromatic amine whose amino group is not directly attached to the Benzene ring.
	Brown solution turns colourless; with formation of white fumes; and a product that is immiscible with water; white precipitate formed on addition of excess Bromine water.	Aryl amine (aromatic amine with amino group directly attached to the Benzene ring) present.
	Brown solution turns colourless; with no white fumes; and a product that is immiscible with water; white precipitate formed on addition of excess Bromine water.	Phenol present.

Note: The reagent is used as either a saturated solution of Bromine or as a solution of 5%Bromine in carbon tetrachloride.

j) Action of acidified potassium permanganate solution [acidified potassium manganate(VII) solution]

Acidified potassium permanganate solution is a very effective oxidizing agent which oxidizes various classes of organic compounds which have reducing properties in their functional groups.

Therefore,acidified potassium permanganate solution, just like bromine water, is used to test for unsaturation whereby when the reagent is reacted with any unsaturated organic compoundwith multiple carbon-carbon bonds ***(e.g. alkenes and alkynes),***the purple solution turns colourless.

Acidified Potassium permanganate solution is also used to test for other reducing agents such as:

- Primary alcohols
- Secondary alcohols
- Aldehydes
- Methanoic acid.
- Aldose sugar (Reducing sugar e.g. Glucose).
- Oxalic acid and salts of oxalic acid.

In the process, primary alcohols are oxidized to aldehydes.

$RCH_2OH \rightarrow RCHO$

Secondary alcohols are oxidized to ketones.

$RCHOHR' \rightarrow RCOR'$

Aldehydes are oxidized to carboxylic acids.

$RCHO \rightarrow RCOOH$

Oxalate ions from oxalic acid and a salt of oxalic acid are oxidized to carbon dioxide gas.

$C_2O_4^{2-}(aq) \rightarrow 2CO_2(g) + 2e^-$

Methanoic acid is oxidized to Carbon dioxide.

$HCOOH(aq) \rightarrow CO_2(g) + 2H^+(aq) + 2e^-$

Test	Observation	Deduction
To $1cm^3$ of the solution, add 1- 2 drops of acidified potassium permanganate solution	Purple solution turns colourless in the cold and the product is immiscible with water.	Unsaturated compound with multiple carbon-carbon bond(s) e.g. alkene **or** alkyne present.
To $1cm^3$ of the solution, add 1- 2 drops of acidified potassium permanganate solution and **warm/heat.**	Purple solution turns colourless on warming and the product is miscible with water.	Primary alcohol, secondary alcohol, aldehyde, methanoic acid, aldose sugar (e.g. Glucose), oxalic acid **or** salt of oxalic acid present.

Note:

1)Unsaturated compoundswith multiple carbon-carbon bonds such as alkenesandalkynes can reduce acidified potassium permanganate solution in the cold. No warming/heating is required at all.

2)Apart from oxalic acid and salts of oxalic acid which can only reduce acidified potassium permanganate after warming or heating, the other reducing agents in the category of primary or secondary alcohols, aldehydes, methanoic acid and aldose sugars can reduce acidified potassium permanganate solution whether in the cold or on warming/heating.

k) Action of acidified potassium dichromate solution [acidified potassium dichromate (VI) solution]
Acidified potassium dichromate solution is also an oxidizing agent and is used to test for reducing agents such as primary alcohols, secondary alcohols, aldehydes, methanoic acid and aldose sugars exactly in the same way as acidified potassium permanganate solution except that it cannot be used to test for unsaturation (the presence of multiple carbon-carbon bonds) in an organic compound. The positive test is when the orange solution turns green while the negative test is when there is no observable change.

l)Action of ethanoic acid and concentrated sulphuric acid
This combination of reagents is used to confirm the presence of the –OH functional group in primary alcohols. It is based on the fact that when to a**primaryalcohol (primary akanol)**, a carboxylic acid (such as ethanoic acid) is added followed by concentrated sulphuric acid followed by warming/heating, an ester is formed. This is known as *esterification reaction* and the formation of an ester is detected by a sweet, fruity smell.

Test	Observation	Deduction
To $1cm^3$ of the solution of the organic compound, add $1cm^3$ of Ethanoic acid followed by about 5 drops of concentrated sulphuric acid and warm/heat. Pour the product in a beaker containing cold water.	A sweet, fruity smell.	Esterification reaction. (Ester formed) Primary alcohol confirmed present.

m)Action of ethanol or methanol and concentrated sulphuric acid
This combination of reagents is used to confirm the presence of the –COOH functional group in carboxylic acids. It is based on the fact that when to a **carboxylic acid**, a primaryalcohol (primary alkanol) is added followed by concentrated sulphuric acid and then followed by warming/heating, an **ester** is formed. This is known as *esterification reaction* and formation of an ester is detected by a sweet, fruity smell.

Test	Observation	Deduction
To $1cm^3$ of the solution of the organic compound, add $1cm^3$ of ethanol/methanol followed by about 5 drops of concentrated sulphuric acid and warm/heat. Pour the product in a beaker containing cold water.	A sweet, fruity smell.	Esterification reaction (ester formed). Carboxylic acid confirmed present.

n) Action of Sodium metal
Sodium is a highly electropositive metal which reacts with all hydroxy compounds(all compounds that have the –OH functional group), with effervescence of a colourless gas that burns with a pop sound. The gas evolved is hydrogen gas.

$ROH(aq) + Na(s) \rightarrow RO^-Na^+(aq) + \frac{1}{2} H_2(g)$

Note: Carboxylic acids also liberate hydrogen gas when reacted with sodium metal.

$RCOOH(aq) + Na(s) \rightarrow RCOO^-Na^+(aq) + \frac{1}{2} H_2(g)$

o) Action of phosphorus pentachloride

To about 0.25g of the dry organic compound, in a dry test tube, approximately 0.1g of phosphorous pentachloride is added. This is followed by observing whether misty fumes are given off, which form dense white fumes with concentrated ammonia solution. *(The misty fumes are of hydrogen chloride gas).*
phosphorus pentachloride reacts with organic compounds that contain the –OH group, for instance alcohols, phenols and carboxylic acids.

Phosphorus pentachloride, also, when reacted with amines, dense white fumes of ammonium chloride are evolved.
Note: Phosphorus pentachloride solution can also be used for the same purpose.

Observation	Deduction
Misty fumes which form dense white fumes with concentrated ammonia solution.	Compound withan –OH group such as**alcohol, phenol** or **carboxylic acid.**
Dense white fumes.	Basic compound present such as **amine**.

p) Action of anhydrous zinc chloride and concentrated hydrochloric acid (Lucas' Reagent)

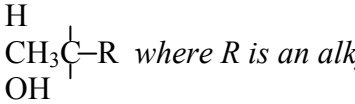
Lucas' reagent is used to differentiate between different classes of alcohols.
The reagent is used at room temperature. When Lucas' Reagent is reacted with a primary alcohol, there is no observable change at room temperature; with a secondary alcohol, a cloudy solution is formed within 5 to 10 minutes; while with a tertiary alcohol, a cloudy solution is formed immediately.

Observation	Deduction
No observable change at room temperature.	Primary alcohol present.
A cloudy solution is formed within 5 to 10 minutes.	Secondary alcohol present.
A cloudy solution is formed immediately.	Tertiary alcohol present.

Note: Lucas' reagent is prepared by dissolving 2.5g of anhydrous zinc chloride in $100cm^3$ of concentrated hydrochloric acid. (The reagent is used when freshly prepared).

q) Action of iodine solution and sodium hydroxide solution (Iodoform Test)

This combination of reagents is used to test for alcohols with the structure**:**



where R is an alkyl group or hydrogen

Note: All secondary alcohols that conform to the above structure give a positive iodoform test while ethanol is the only primary alcohol that gives a positive iodoform test. None of the tertiary alcohols gives a positive iodoform test.

The combination of reagents is also used to test for the presence of carbonyl compounds with the structure:

$CH_3\overset{\overset{O}{\|}}{C}-R$ *where R is an alkyl group or hydrogen*

$$CH_3\overset{\overset{O}{\|}}{C}-R(aq) + 3I_2(aq) + 4NaOH(aq) \rightarrow RCOONa(aq) + CHI_3(s) + 3NaI(aq) + 3H_2O(l)$$

When iodine solution is added to the test solution followed by dilute sodium hydroxide solution drop wise, followed by gentle warming and then cooling, to the primary and secondary alcohols, together with carbonyls of the specified structures above, a yellow precipitate with an antiseptic smell is formed.The yellow precipitate is for triiodomethane, CHI_3.

Test	Observation	Deduction
To 1cm^3 of the solution, add 1 cm^3 of Iodine solution followed by sodium hydroxide solution drop wise until the solution turns pale yellow. Warm gently and then cool under running tap water.	A yellow precipitate with an antiseptic smell.	$CHI_3(s)$ formed Alcohol with the structure: $CH_3\overset{\overset{H}{|}}{C}-\overset{|}{OH}$ **OR**Carbonyl with the structure: $CH_3\overset{\overset{O}{\|}}{C}-$

r)Action of soda lime

Soda lime is a solid mixture of sodium hydroxide and calcium oxide. It reacts with aliphatic carboxylic acids to liberate alkane vapours.

$$RCOOH(aq) + 2NaOH(s) \xrightarrow[heat]{CaO} \underset{\textit{Alkane vapour}}{RH(g)} + Na_2CO_3(s)$$

It reacts with aromatic carboxylic acids (specifically benzoic acid) to evolve benzene vapour.
It reacts with hydroxyl aromatic carboxylic acids to evolve phenol vapour.
Soda lime also, when reacted with simple aliphatic amides and substituted amides evolve vapours of amines.

It further reacts with ammonium salts to evolve ammonia gas.

When using soda lime, to about1cm^3 of the organic compound or to half a spatula endful of the organic compound, about 2 spatula end fulls of soda lime are added and the mixture warmed first gently and then warmed more strongly and any gases/vapours evolved are noted, taking keen interest in their odours, effect on moist litmus papers and in some situations, the vapours evolved are allowed to burn especially when an aliphatic carboxylic acid or Benzoic acid is being suspected.

Test	Observation	Deduction
To 1cm^3 of the organic compound or to half a spatula endful of the organic compound, add 2 spatula endfuls of soda lime and warm the mixture, first gently and then more strongly.	A colourless vapour with a smell of petrol or paraffin which burns with a non-sooty flame.	Alkane evolved. Aliphatic carboxylic acid present.
	A colourless vapour which burns with a sooty flame.	Benzene vapour evolved Aromatic carboxylic acid (e.g Benzoic acid) present.
	A colourless vapour with a carbolic smell which burns with a sooty flame.	Phenol evolved. Hydroxyl aromatic carboxylic acid present.
	A colourless vapour with a characteristic fishy smell which turns moist red litmus paper blue and burns with a non-sooty flame.	Lower aliphatic amine evolved. Lower aliphatic amide **or**substituted amide present.
	A colourless, pungent, chocking gas, turns moist red litmus paper blue and forms dense white fumes with concentrated hydrochloric acid.	$NH_3(g)$ evolved. Ammonium salt **or** Simplest aliphatic amide present.

s) Action of concentrated hydrochloric acid and sodium nitrite solution(at 0^0C)

This combination is used to test for classes of amines. The reaction is carried out at 0^0C (<5^0C). The combination forms nitrous acid, HNO_2.

With aliphatic primary amines, a colourless solution of an alcohol is observed with effervescence of a colourless gas neutral to litmus paper (the gas evolved is nitrogen gas).

$$RCH_2NH_2(s) \xrightarrow[0^0C]{NaNO_2/\ Conc.\ HCl} CH_2OH(aq) + \tfrac{1}{2}N_2(g)$$

Note:

1)A primary aliphatic amide may also give the same observation as aprimary aliphaticamine.

2) Withsecondary aliphatic amines, a yellow oily liquid is observed with no effervescence ofa colourless gas (the yellow oily liquid is a nitrosoamine).

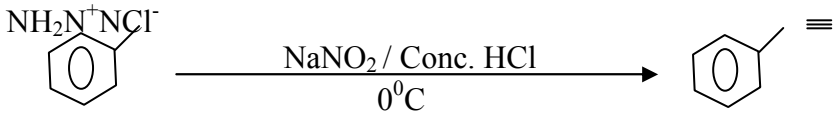
$$\begin{matrix} R' \\ RNHR' \\ R \end{matrix} \xrightarrow[0^0C]{NaNO_2\ /\ Conc.\ HCl} N\text{-}N{=}O + H_2O$$

Yellow oily liquid
(Nitrosoamine)

3)With a Tertiary aliphatic amine, a colourless solution is observed with no effervescence of a colourless gas.

$$RNR'(R'') \xrightarrow[0^0C]{NaNO_2\ /Conc.\ HCl} RN^+ R'(R'')(H)$$

4) Similarly, with a primary aromatic amine ($C_6H_5NH_2$), a colourless solution is observed with no effervescence of a colourless gas (the colourless solution is due to formation of a **diazonium salt**).



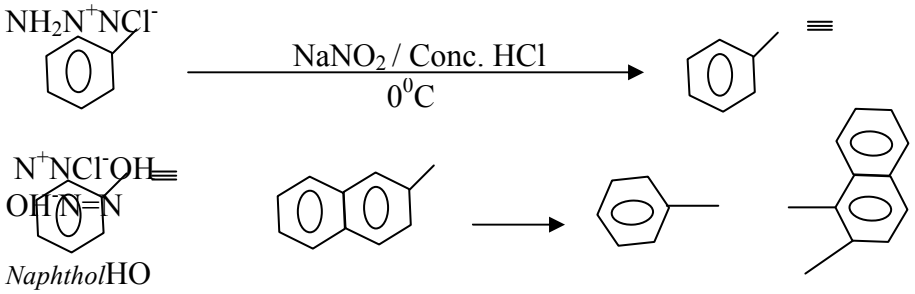
Colourless solution of a daizonium salt (Benzene diazonium chloride)

However, on warming, effervescence of a colourless gas neutral to litmus paper occurs. The colourless gas is nitrogen.

Test	Observation	Deduction
To 1cm^3 of the organic compound or to half a spatula end full of the organic compound, add 5 drops of concentrated hydrochloric acid followed by 1cm^3 of sodium nitrite solution (care is taken that the reaction mixture is maintained at 0^0C i.e. at a temperature less than 5^0C).	Effervescence of a colourless gas neutral to litmus paper.	N_2(g) evolved. Primary aliphatic amine present.
	A yellow oily liquid is formed with no effervescence.	Nitrosoamine formed. Secondary aliphatic amine present.
	No observable change even on warming.	Tertiary aliphatic amine present.
	No observable change in the cold, but on warming, effervescence occurs, of a colourless gas neutral to litmus paper.	Primary aromatic amine present.

t)Action of concentrated hydrochloric acid, followed by sodium nitrite solution, sodium hydroxide solution and then 2-naphthol

When concentrated hydrochloric acid is added to an organic compound followed by sodium nitrite solution, sodium hydroxide solution and then 2-naphthol, a bright yellow precipitate (or an orange precipitate) is formed. The precipitate formed is of an azo dye.



*Naphthol*HO

An Azo Dye (Bright yellow/orange precipitate)

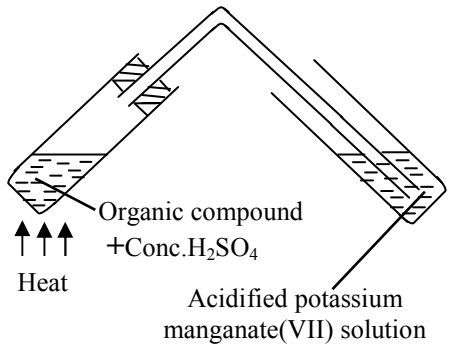
u)Action of copper(II) sulphate solution

When copper(II) sulphate solution is added to a solution containing an aliphatic amine, a deep blue solution is formed. This is due to formation of a complex ion, i.e. the tetraammine copper(II) ion which is deep blue.

Test	Observation	Deduction
To $3cm^3$ of the organic compound, add $1cm^3$ of copper(II) sulphate solution.	A deep blue solution is formed.	Aliphatic amine present.

v) Action of hot concentrated sulphuric acid

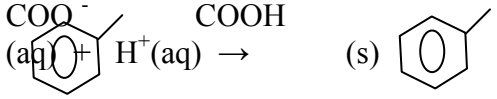
Hot concentrated sulphuric acid is used to dehydrate alcohols/alkanols which results in formation of alkenes. The alkene formed can thus turn acidified potassium manganate(VII) solution from purple to colourless.

Test	Observation	Deduction
To $1cm^3$ of the organic compound, add 5 drops of concentrated sulphuric acid and heat. Pass the vapour formed through acidified potassium manganate(VII) solution as shown below. 	White fumes which turn acidified potassium manganate(VII) solution from purple to colourless	Alcohol dehydrated to form alkene.

w) Action of dilute sulphuric acid

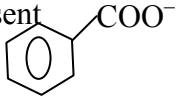
Dilute sulphuric acid is used normally to precipitate out an aromatic carboxylic acid from a solution of its salt. Usually an aromatic carboxylic acid is dissolved in sodium hydroxide solution and to a portion of the resultant solution of the sodium salt of the aromatic carboxylic acid, dilute sulphuric acid is added.

A white precipitate is formed.Therefore, dilute sulphuric acid can be used to test for the presence of the Benzoate ion.



white precipitate

Addition of dilute sulphuric acid can also be used to test for the presence of a basic substance such as an amine. Such a basic substance will dissolve readily in dilute sulphuric acid with no effervescence of a gas. Similarly, dilute sulphuric acid readily dissolves any salt of alower organic acid e.g methanoates, ethanoates and propanoates.

Test	Observation	Deduction
To 1cm^3 of the solution, add A few drops of dilute sulphuric acid.	A white precipitate is formed	present 
To a spatula endful of the solid, add 4cm^3 of dilute sulphuric acid and shake well to dissolve.	The solid dissolves in the acid in the cold with no effervescence.	Basic compound present e.g. amine
To a spatula endful of the solid, add 4cm^3 of dilute sulphuric acid and warm gently to dissolve.	The solid dissolves in the acid on warming with no effervescence.	Salt of a lower organic acid e.g methanoate, ethanoate or propanoate.

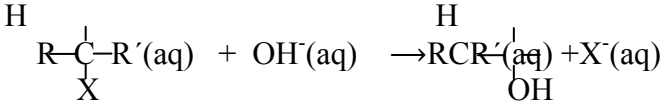
x) Action of dilute hydrochloric acid

Dilute hydrochloric acid can be used in a similar way like dilute sulphuric acid to test for the functional groups stated above.

y) Action of hot dilute sodium hydroxide solution and silver nitrate solution.

This combination of reagents is used in testing for alkylhalides or any other organic compounds that have got halogen atoms as one of their functional groups.

To 5cm^3 of the organic compound, 1-2cm^3 of dilute sodium hydroxide solution is added. If the compound under analysis is a solid, a spatula endful of the sample is first dissolved in about 5cm^3 of water before the dilute sodium hydroxide solution is added and the mixture heated. The -X group is substituted by the –OH group.



The mixture is thencooled and 2-3 drops of silver nitrate solution is added followed by filtration.

$$Ag^+(aq) + X^-(aq) \rightarrow AgX(s)$$

Note: *The colour of the precipitate* ***AgX(s)*** *depends on which halide* ***X$^-$*** *is. In case it is a* ***Cl$^-$,*** *a white precipitate is formed; if it is a* ***Br$^-$,*** *a cream precipitateis formed and if it is an* ***I$^-$,*** *a pale yellow precipitate is formed.*

To the residue, dilute ammonia solution is added drop wise until in excess.

$$AgX(s) + 2NH_3(aq) \rightarrow [Ag(NH_3)_2]^+(aq) + X^-(aq)$$

To the filtrate, an equal volume of ethanoic acid is added followed by 4-5drops of concentrated sulphuric acid and the mixture heated and cooled. The product is then poured into a beaker containing cold water. A sweet, fruity smell is detected due to formation of an ester (Esterification reaction).

Note: *In case the organic compound under analysis has a carboxyl group, instead of addition of Ethanoic acid, Ethanol may be added together with concentrated sulphuric acid in which case esterification also occurs.*

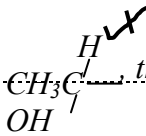
Test	Observation	Deduction
a) To a spatula endful of Q, add $4cm^3$ of water. To the resultant solution, add $2cm^3$ of dilute sodium hydroxide solution. Shake well and heat the mixture, then cool and add 2-3 drops of silver nitrate solution and filter. Keep both the filtrate and residue.	*White crystalline solid dissolves in water to form a colourless solution.* *Colourless solution miscible with dilute sodium hydroxide solution.* *A white precipitate is formed on addition of silver nitrate solution.* *A colourless filtrate is formed.* *A white residue is formed.*	*Polaraliphatic compound with a low molecular mass e.g. alcohol, carbonyl, carboxylic acid, ester, alkylhalide.* *Halidegroup substituted by -OH group.* *Cl^- released; from a Chlorocompound.* *Hydroxycompound formed.* *AgCl(s) formed.*
b) To the filtrate obtained in (a) above, add an equal volume of ethanol followed by 4-5 drops of concentrated sulphuric acid. Heat the mixture, cool, then pour the product in a beaker containing cold water.	*A sweet, fruity smell.*	*Ester formed;* *Carboxyl group(-COOH group) present.*
c) To the residue obtained in (a) above, add dilute ammonia solution drop wise until in excess.	*White precipitate dissolves in excess dilute ammonia solution.*	*AgCl(s) dissolves in excess dilute ammonia solution to form $[Ag(NH_3)_2]^+(aq)$.* *Cl^- confirmed to have been released; from a Chlorocompound*

9.4 Worked out Examples on Organic Qualitative Analysis

Worked out Example 9.4.1
You are provided with substance A_1 which is an organic compound. Carry out the following tests to identify its nature.

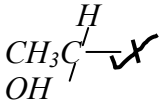
Test	Observation	Deduction
a) Burn a small amount of A_1 on a spatula end or crucible lid.	*Colourless liquid burns with a yellow <u>non-sooty</u> flame.*	*<u>Aliphatic</u>; <u>saturated</u> compound with a low carbon to hydrogen ratio (with a low carbon content).*
b) Shake $2cm^3$ of A_1 with $1cm^3$ of water. Test the resultant with litmus paper. Divide the resultant solution into **2** parts.	*<u>Miscible</u> with water to form a colourless solution.* *Colourless solution has <u>no effect on both blue and red litmus paper</u>.*	*<u>Polar</u>, aliphatic compound with a <u>low molecular mass</u> e.g. Alcohol, Carbonyl compound, Carboxylic acid or Ester.* *<u>Neutral compound</u> present e.g. alcohol, carbonyl or ester.*
i) To the first part, add Brady's Reagent	*No observable change.*	*<u>Carbonyl compound absent</u>.*
ii) To the second part, add $2cm^3$ of iodine solution followed by sodium hydroxide solution drop by drop until the solution turns pale yellow. Warm gently and then cool under running tap water.	*A <u>yellow precipitate</u> with an antiseptic smell.*	*$CHI_3(s)$ formed* *<u>Alcohol</u>; with the structure:* $CH_3C(H)(OH)-$
c) To $2cm^3$ of A_1 in a boiling tube, add $4cm^3$ of acidified potassium permanganate solution and heat. Divide the resultant solution into 2 parts.	*Purple solution turns colourless.*	*<u>Primary</u> or <u>secondary alcohol</u> present.*
d) To $1cm^3$ of A_1, add $1cm^3$ of ethanoic acid followed by 5 drops of concentrated sulphuric acid and then warm. Pour the resultant solution into a beaker of cold water.	*A sweet, fruity smell.*	*Esterification reaction. (Ester formed).* *<u>Primary alcohol</u> present.*
(e) To $1cm^3$ of B_3, add anhydrous zinc chloride in concentrated hydrochloric (Lucas' reagent). Shake and allow to stand.	*No observable change.*	*<u>Primary alcohol</u> confirmed present.*

f) Comment on the nature of A_1.

A_1 is an aliphatic, saturated,primary alcohol with the structure:  *, that is, ethanol.*

Worked out Example 9.4.2

You are provided with substance A_2 which is an organic compound. Carry out the following tests to identify its nature.

Test	Observation	Deduction
a) Burn a small amount of A_2 on a spatula end or crucible lid.	*Colourless liquid burns with a yellow non-sooty flame.*	*Aliphatic, saturated compound with a low carbon to hydrogen ratio(with a low carbon content).*
b) Shake $4cm^3$ of A_2 with $3cm^3$ of water and divide the resultant solution into 3 parts.	*Miscible with water to form a colourless solution.*	*Polar aliphatic compound with a low molecular mass e.g. Alcohol, Carbonyl compound, Carboxylic acid or Ester.*
i) Test the first part with litmus paper.	*Colourless solution has no effect on both blue and red litmus paper.*	*Neutral compoundpresent e.g. alcohol or carbonyl.*
ii) To the second part, add Brady's Reagent	*No observable change.*	*Carbonyl compound absent.*
iii) To the third part, add $2cm^3$ of iodine solution followed by sodium hydroxide solution drop by drop until the solution turns pale yellow. Warm gently and then cool under running tap water.	*A yellow precipitate with an antiseptic smell.*	*$CHI_3(s)$ formed* *Alcohol with the structure:* 
c) To $2cm^3$ of A_2 in a boiling tube, add $4cm^3$ of acidified potassium permanganate solution and heat. Divide the resultant solution into 3 parts.	*Purple solution turns colourless.*	*Reducing agent present e.g. primary or secondary alcohol.*
i) To the first part, add Brady's reagent.	*A yellow precipitate is formed.*	*Primary or secondary alcoholoxidized to a Carbonyl.*
ii) To the second part, add ammoniacal silver nitrate solution.	*No observable change.*	*Secondary alcoholoxidized to Ketone.*

d) Comment on the nature of A_2.

A_2 is an aliphatic,saturated,secondary alcohol with the structure: $CH_3C(H)(OH)CH_3$

Worked out Example 9.4.3

You are provided with substance B_1 which is an organic compound. Carry out the following tests to identify its nature.

Test	Observation	Deduction
a) Burn a small amount of B_1 on a spatula end or crucible lid.	*White crystalline solid melts to form a colourless liquid which burns with a yellow sooty flame.*	*Aromatic compound with a high carbon to hydrogen ratio(with a high carbon content)* ***OR*** *Long chain aliphatic, unsaturated compound with a high carbon to hydrogen ratio (with a high carbon content).*
b) Shake half a spatula endful of B_1 with about $4cm^3$ of water. Test the resultant solution with litmus paper.	*White crystalline solid partially dissolves in water to form a colourless solution.*	*Polar aromatic compound* ***OR*** *Polar aliphatic compound with a high molecular mass e.g. Alcohol, Carbonyl compound, Carboxylic acid or Ester.*
	Colourless solution turns blue litmus paper red.	*Acidic compound present e.g. Carboxylic acid* ***or*** *Phenol.*
(c) Divide the solution obtained in (b) above into two parts i) To the first part, add iron(III) chloride solution.	*No observable change.*	*Phenol absent.*
ii) To the second part, add a little solid sodium hydrogencarbonate	*Effervescence of a colourless gas.*	*Carboxylic acid present.*
d) To a spatula endful of B_1, add $2cm^3$ of sodium hydroxide solution and warm to dissolve. Cool the resultant solution and divide the resultant solution into two parts. i) To the first part, add neutral iron (III) chloride solution and heat.	*White crystalline solid dissolves in sodium hydroxide solution to form a colourless solution.* *A reddish-brown precipitate is formed.*	*Neutralisation reaction.* *Acidic compound present e.g. Carboxylic acid.* C_6H_5COO

ii) To the second part, add dilute sulphuric acid.	*A white precipitate is formed.*	*$C_6H_5COOH(s)$ formed from $C_6H_5COO^-(aq)$.*
e) Carry out a test of your own choice to determine the functional group of organic substance B_1 *To a spatula endful of B_1, add about $2cm^3$ of ethanol, shake well to dissolve the solid, then add 5 drops of concentrated sulphuric acid and heat the mixture. Pour the product in a beaker of cold water.*	*A sweet, fruity smell.*	*Esterification reaction (Ester formed)* *Carboxylic acid confirmed present e.g. Benzoic acid.*

d) Comment on the nature of B_1.

*B is an aromatic, unsaturatedcarboxylic acid; that is Benzoic acid(**OR**Aromaticunsaturatedcarboxylic acidwith the carboxyl group directly attached to the Benzene ring).*

Worked out Example 9.4.4

You are provided with substance B_2 which is an organic compound. Carry out the following tests to determine its nature.

Test	Observation	Deduction
a) Burn a small amount of B_2 on a spatula end or porcelain dish.	*Colourless liquid burns with a yellow non-sooty flame.*	*Aliphatic, saturated compound with a low carbon to hydrogen ratio (with a low carbon content).*
b) Shake $1cm^3$ of B_2 with $3cm^3$ of water. Test the resultant solution with litmus paper. Divide the resultant solution into 2parts.	*Miscible with water to form a colourless solution.* *Colourless solution has no effect on both blue and red litmus paper.*	*Polar aliphatic compound with a low molecular mass e.g. Alcohol, carbonyl compound, carboxylic acid or ester.* *Neutral compound present e.g. alcohol or carbonyl.*
i) Test the **first**part, add 2-3 drops of iron(III) chloride solution.	*No observable change.*	*Phenol absent.*
ii) To the **second** part, add 2-3 drops of acidified potassium dichromate(VI) solution and warm.	*No observable change.*	*Reducing agentabsent.* *Tertiary alcohol;or ketone probably present.*

(c) To 2-3 drops of B_2, add 4-5 drops of 2,4-dinitrophenylhydrazine solution.	*Yellow precipitate.*	*Ketone present.*
d) To 0.5cm^3 of B_2, add 1cm^3 of methanol and shake well to dissolve.To the resultant solution, add about 4cm^3 of iodine solution followed by sodium hydroxide solution drop wise until the solution turns pale yellow. Warm the mixture and then allow to stand.	*A yellow precipitate with an antiseptic smell.*	*CHI_3(s) formed.* *Ketone with the structure:* $CH_3C(=O)-$
e) To 1cm^3 of B_2, add an equal volume of Fehling's solution and heat the mixture.	*No observable change.*	*Aldehyde absent.* *Ketone confirmed present.*

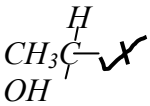
f) Comment on the nature of B_2.

A is an aliphatic, saturated,ketone with the structure: $CH_3C(=O)-$

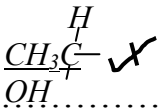
Worked out Example 9.4.5

You are provided with substance B_3 which is an organic compound. Carry out the following tests to determine its nature.

Test	Observation	Deduction
(a) Burn a small amount of B_3 on a spatula end or porcelain dish.	*Colourless liquid burns with a yellow sooty flame.*	*Aromatic compound with a high carbon to hydrogen ratio.* ***OR** Long chainaliphatic, unsaturated compound with a high carbon to hydrogen ratio (with a high carbon content).*
(b) Shake 1cm^3 of B_3 with 3cm^3 of water. Test the resultant solution with litmus paper.	*Partially miscible with water to form a colourless solution.* *Colourless solution has no effect on both blue and red litmus paper.*	*Polar aromatic compound* ***OR**Polaraliphatic compound with a high molecular mass e.g. Alcohol, carbonyl compound, carboxylic acid or ester.* *Neutral compound present e.g. alcohol,carbonyl or ester.*
(c) To 3-4 drops of B_3, add a few drops of 2,4-dinitrophenylhydrazine solution.	*No observable change.*	*Carbonyl compoundabsent*

(d) To 2 drops of B_3, add 2 drops of bromine water.	*No observable change.*	*Unsaturated compound absent (**or** saturated compound present)*
(e) To 1cm^3 of B_3, add 2cm^3 of acidified potassium dichromate(VI) solution. To the resultant product, add 3-4 drops of 2,4-dinitrophenyl-hydrazine solution.	*Orange solution turns green. Then a yellow precipitate is formed.*	*Primary or secondary alcohol oxidized to a carbonyl.*
(f) To 1cm^3 of B_3, add 1cm^3 of methanoic acid followed by 3-4 drops of concentrated sulphuric acid and heat the mixture. Pour the product in a beaker containing cold water.	*No observable change.*	*Primary alcohol absent.*
(g) To 1cm^3 of B_3, add anhydrous zinc chloride in concentrated hydrochloric (Lucas' reagent). Shake and allow to stand.	*Cloudy solution formed after 7 minutes.*	*Secondary alcohol present.*
(h) To the third part, add 2cm^3 of iodine solution followed by sodium hydroxide solution drop by drop until the solution turns pale yellow. Warm gently and then allow to cool.	*No observable change.*	*Alcohol without the structure:* 

(i) Comment on the nature of compound B_3.

Compound B_3 is an aromatic secondary alcohol without the structure . 

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9.5Practical Exercises onOrganic Qualitative Analysis

Experiment 9.5.1

You are provided with substance C which is an organic compound. Carry out the following tests to identify its nature.

Test	Observation	Deduction
a) Burn a small amount of C on a dry spatula end or dry crucible lid.		
b) Shake 4cm^3 of C with 3cm^3 of water and test the resultant solution with litmus paper. Divide the solution into **four** parts.		

i) To the first part, add solid sodium carbonate.		
ii) To the second part, add 1cm^3 of iodine solution followed by sodium hydroxide solution drop by drop until the solution turns pale yellow. Warm gently and then cool under running tap water.		
iii) To the third part, add 2 to 3 drops of 2,4-dinitrophenylhydrazine solution (Brady's reagent).		
iv) To the fourth part, add 1cm^3 of ethanoic acid followed by 5 drops of concentrated sulphuric acid and then warm. Pour the product in a beaker containing cold water.		
c) To 2cm^3 of C, add 4cm^3 of acidified potassium manganate(VII) solution and heat. Cool the resultant solution and divide it into **two**parts.		
i) To the first part,2 to 3 drops of2,4-dinitrophenylhydrazine solution (Brady's reagent).		
ii) To the second part, add ammoniacal silver nitrate solution and warm.		
d) To 1cm^3 of C, add 4 drops of Lucas' reagent and leave to stand.		

d) Comment on the nature of C.

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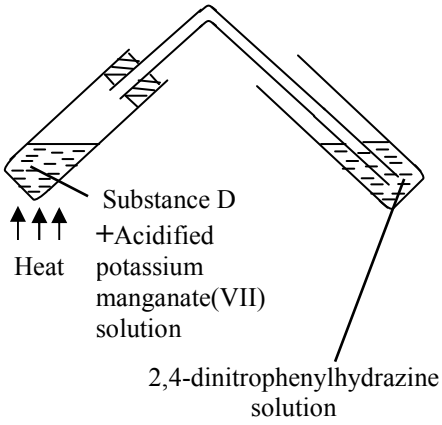
Experiment 9.5.2

You are provided with substance D which is an organic compound. Carry out the following tests to identify its nature.

Test	Observation	Deduction
a) Burn a small amount of D on a dry spatula end or dry crucible lid.		
b) Shake $3cm^3$ of C with $3cm^3$ of water and test the resultant solution with litmus paper. Divide the resultant solution into**three** parts.		
i) To the first part, add 2-3 drops of 2,4-dinitrophenylhydrazine solution (Brady's reagent).		
ii) To the second part, add $2cm^3$ of Iodine solution followed by sodium hydroxide solution drop by drop until the solution turns pale yellow. Warm gently and then cool under running tap water.		
iii) To the third part, add $1cm^3$ of ethanoic acid followed by 5 drops of concentrated sulphuric acid and then warm. Pour the product in a beaker containing cold water.		
c) To $2cm^3$ of D, add $1cm^3$ of acidified potassium manganate(VII) solution and heat in a boiling tube which is corked and pass the vapour produced through 2,4-dinitro-phenylhydrazine solution (Brady's reagent) which is in a test tube by use of a delivery tube as shown below.  Cool the resultant solution in the boiling tube and keep it for use in part (d) below.		

d) To cool resultant solution from part (c) above, add ammoniacal silver nitrate solution.Warm and allow to stand.		
e) To 1cm^3 of D, add 4 drops of Lucas' reagentand allow tostand.		

f) Comment on the nature of D.

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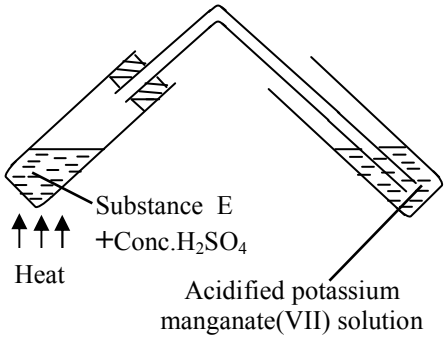
Experiment 9.5.3
You are provided with substance E which is an organic compound. Carry out the following tests to identify its nature.

Test	Observation	Deduction
a) Burn a small amount of E on a dry spatula end or dry crucible lid.		
b) To 1 cm^3 of E, add 1cm^3 of water and shake. Test the resultant solution with Litmus paper. Divide the resultant solution into **three** parts.		
i) To the first part, add 2-3 drops of sodium carbonate solution.		
ii) To the second part, add 2-3 drops of 2,4-dinitrophenylhydra-zine solution (Brady'sreagent).		
iii)To the third part, add 2-3 drops of acidified potassium dichromate(VI) solution and heat the mixture.		

c) To 1cm^3 of E, add an equal volume of ethanoic acid followed by 5 drops of concentrated sulphuric acid. Heat the mixture and pour the product in a beaker of cold water.		
d) To 1cm^3 of E, add 1cm^3 of concentrated sulphuric acid. Heat the mixture and pass the gas produced through acidified potassium manganate(VII) solution by use of a delivery tube as shown below.  		
e) To 1cm^3 of E, add 4 drops of Lucas' reagent.		

f) Comment on the nature of E.

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Experiment 9.5.4

You are provided with substance F which is an organic compound. Carry out the following tests to identify its nature.

Test	Observation	Deduction
a) Burn a small amount of F on a spatula end or crucible lid.		

b) To 0.5 cm^3 of F, add 1cm^3 of water and shake. Test the resultant solution with litmus paper.		
c) To 0.5 cm^3 of F, add 2-3 drops of sodium carbonate solution.		
d) To 0.5cm^3 of F, add 2cm^3 of Iodine solution followed by sodium hydroxide solution drop by drop until the solution turns pale yellow. Warm gently and then cool under running tap water.		
e)To 0.5cm^3 of F, add 2-3 drops of 2,4-dinitrophenylhydrazine solution (Brady's reagent).		
f) To 1cm^3 of F, add 2cm^3 of acidified potassium dichromate(VI) solution and heat.		
g) To 0.5cm^3 of F,add an equal volume of ammoniacal silver nitrate solution and warm; then allow to stand.		
h) To 1cm^3 of F, add 2cm^3 of Fehling's solution and boil.		

d) Comment on the nature of F.

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Experiment 9.5.5

You are provided with substance G which is an organic compound. Carry out the following tests to identify its nature.

Test	Observation	Deduction
a) Burn a small amount of G on a dry spatula end or dry crucible lid.		

b) Shake $4cm^3$ of G with $2cm^3$ of water. Test the solution with litmus paper. Divide the resultant solution into **four** parts.		
i) To the first part, add saturated sodium hydrogensulphite solution and shake strongly.		
ii) To the second part, add $2cm^3$ of acidified potassium dichromate solution and then heat.		
iii) To the third part, add $2cm^3$ of iodine solution followed by sodium hydroxide solution drop by drop until the brown solution is discharged. Warm gently and then cool under running tap water.		
iv) To $1cm^3$ of silver nitrate solution, add $1cm^3$ of sodium hydroxide solution followed by ammonia solution drop wise until the precipitate just dissolves, then add $2cm^3$ of the fourth part and warm, then allow to stand.		

d) Comment on the nature of G.

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Experiment 9.5.6

You are provided with substance H which is an organic compound. Carry out the following tests to identify the functional group of **H**.

Test	Observation	Deduction
a) Burn a small amount of H on a spatula end or crucible lid.		

b) Shake half a spatula endful of H with about 6cm^3 of water. Test the resultant solution with litmus paper.		
c) Divide the solution obtained in (b) above into **five**parts. i) To the first part, add iron(III) chloride solution.		
ii) To the second part, add 2-3 drops ofsodium hydrogencarbonate solution.		
iii) To the third part, add 2,4-dinitrophenylhydrazine solution.		
iv) To the fourth part, add 2cm^{3}of acidified potassium manganate(VII) solution and heat.		
v) To the fifth part, add 2cm^3 of Tollen's reagent and warm; then allow the mixture to stand.		

d) Identify the functional group of H.

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Experiment 9.5.7

You are provided with substance **I** which is an organic compound. Carry out the following tests to identify its functional group.

Test	Observation	Deduction
a) Burn a small amount of **I**on a spatula end or crucible lid.		
b) Shake 2cm^3 of **I** with about 2cm^3 of water. Test the resultant solution with litmus paper. Divide the resultant solution into **four** parts.		

i) To the first part, add 2-3 drops of Universal Indicator.		
ii) To the second part, add 2,4-dinitrophenylhydrazine solution.		
iii) To the third part, add neutral iron(III) chloride solution and heat.		
iv) To the fourth part, add a littlesodium carbonate powder.		
c) To 1cm^3 of **I,** add soda lime and heat.		
d) To 1cm^3 of **I,** add 1cm^3 of ethanol followed by 5 drops of concentrated sulphuric acid and heat. Pour the product in a beaker of cold water.		

e) Basing on the result you have obtained in (d) above, identify the functional group of **I.**

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Experiment 9.5.8

You are provided with substance J which is an organic compound. Carry out the following tests to identify substance J.

Test	Observation	Deduction
a) Burn a small amount of J on a dry spatula end or dry porcelain.		
b) To 1cm^3 of J, add an equal volume of sodium hydroxide solution.		
c) Shake 2cm^3 of J with about 2cm^3 of water. Test the resultant solution with litmus paper. Divide the resultant solution into **three** parts.		

i) To the first part, add 2-3 drops of neutral iron(III) chloride solution and heat.		
ii) To the second part, add a little sodium carbonate powder.		
iii) To the third part, add 1cm^3 of acidified potassium dichromate solution and heat.		
d) To 1cm^3 of J, add ammoniacal silver nitrate solution and warm; then allow the mixture to stand.		

e) State the identity of J.

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Experiment 9.5.9
You are provided with substance K which is an organic compound. Carry out the following tests to identify its functional group.

Test	Observation	Deduction
a) Burn a small amount of K on a dry spatula end or dry crucible lid.		
b) Shake half a spatula endful of G with about 5cm^3 of water. Test the resultant solution with litmus paper.		
c) Divide the solution obtained in (b) above into**two** parts. i) To the first part, add 2-3 drops ofiron(III) chloride solution.		

ii) To the second part, add a little solidsodium hydrogencarbonate.		
d) To a spatula endful of K, add about $2cm^3$ of dilute sodium hydroxide solution and warm to dissolve. Cool and divide the resultant solution into **two** parts.		
i) To the first part, add 2-3 drops of neutral iron(III) chloride solution and heat.		
ii) To the second part, add dilute sulphuric acid.		
e) To half a spatula endful of K, add 2 spatula endfuls of soda lime and warm the mixture, first gently and then more strongly.		
f) Usinga spatula endfuls of K, carry out a test of your own choice to identify the functional group of K. ……. ……. ……. ……. ……. ……. ……..		

d) Basing on the observations made in the table above, deduce thefunctional group of K.

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Experiment 9.5.10
You are provided with substance L which is an organic compound. Carry out the following tests to identify its nature.

Test	Observation	Deduction
a) Burn a small amount of L on a dry spatula end or dry crucible lid.		
b) Shake two spatula endfuls of L with about $6cm^3$ of water. Test the resultant solution with litmus paper. Divide the resultant solution into **four**portions.		
i) To the first portion, add sodium hydroxide solution.		
ii) To the second portion, add a little solid sodium hydrogencarbonate.		
iii) To the third portion, add $1cm^3$ of bromine water.		
iv) To the fourth part, add neutral iron(III) chloride solution drop wise as you shake.		

d) Comment on the nature of L.

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Experiment 9.5.11

You are provided with substance M which is an organic compound. Carry out the following tests to identify its nature.

Test	Observation	Deduction
a) Burn a small amount of M on a dry spatula end or dry porcelain.		
b) Shake half a spatula endful of L with about $7cm^3$ of water and warm to dissolve. Test the resultant solution with litmus paper. Divide the solution obtained in (b) above into **three** parts.		
i) To the first part, add iron(III) chloride solution.		
ii) To the second part, add a little solid sodium hydrogencarbonate.		
iii) To the third part, add dilute sulphuric acid.		
c) To a spatula endful of M, add about $2cm^3$ of dilute sodium hydroxide solution and warm. To the resultant solution, add 2-3 drops of neutral iron(III) chloride solution and heat.		
d) To a spatula endful of M, add $2cm^3$ of methanol, shake well to dissolve the solid and then add 5 drops of concentrated sulphuric acid and heat the mixture. Pour the product in a beaker containing cold water.		

d) Comment on the nature of M.

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Experiment 9.5.12
You are provided with substance N which is an organic compound. Carry out the following tests to identify its nature.

Test	Observation	Deduction
a) Burn a small amount of N on a dry spatula end or dry porcelain.		
b) Shake a spatula endful of N with about $2cm^3$ of methanol. Add $1cm^3$ of water and test the resultant solution with litmus paper. Divide the resultant solution into**two** portions.		
i) To the first portion, add iron(III) chloride solution.		
ii) To the second portion, add an equal volume ofsodium hydrogencarbonate solution.		
c) To a spatula endful of N in $3cm^3$ of sodium hydroxide solution and warm to dissolve. Cool the resultant solution and divide it into **two**portions.		
i) To the first portion, add neutral iron(III) chloride solution and heat.		
ii) To the second part, add dilute hydrochloric acid.		

d) To half a spatula endful of N, add 2 spatula endfuls of soda lime and warm the mixture, first gently and then more strongly.		

d) Comment on the nature of N.

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Experiment 9.5.13
You are provided with substance P which is an organic compound. Carry out the following tests to identify its nature.

Test	Observation	Deduction
a) Burn a small amount of P on a dry spatula end or crucible lid.		
b) Shake two spatula endfuls of P with about $4cm^3$ of methanol. Add $2cm^3$ of water and test the resultant solution with litmus paper. Divide the resultant solution into **five**portions.		
i) To the first portion, addiron(III) chloride solution.		
ii) To the second portion, add an equal volume of sodium carbonatesolution.		
iii) To the third part, add 2-3 drops of Brady's reagent.		
iv) To the fourth portion, add $2cm^3$ of acidified potassium dichromate(VI) solution and heat.		
v) To the fifth portion, add ammoniacal silver nitrate solution and warm; then allow to stand.		

c) To a spatula endful of P in $3cm^3$ of sodium hydroxide solution and warm to dissolve. Cool the resultant solution and keep the cold resultant solution for part (d) below.		
d) To $1cm^3$ of the cold resultant solution obtained in (c) above, adddilute sulphuric acid.		

e) Comment on the nature of P.

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Experiment 9.5.14

You are provided with substance Q which is an organic compound. Carry out the following tests to determine the nature of substance Q. Record your observations and deductions in the table below.

Test	Observation	Deduction
a) Burn a small amount of Q on a dry spatula end or dry porcelain.		
b) Shake half a spatula endful of Q with about $2cm^3$ of sodium hydroxide solution.		
c) Shake a spatula endful of Q with about $5cm^3$ of water.Divide the resultant solution into **four** portions. i) To the first portion, add 2-3 drops of litmus solution.		
ii) To the second portion, add an equal volume of sodium carbonatesolution.		
iii) To the third portion, add 2-3 drops of 2,4-dinitrophenylhydrazine solution (Brady's reagent).		

iv) To the fourth portion, add 2-3 drops of iron(III) chloride solution and warm.		
d) To a spatula endful of Q, add $4cm^3$ of water. To the resultant solution, add $2cm^3$ of dilute sodium hydroxide solution. Shake well and heat the mixture, then cool and add 2-3 drops of silver nitrate solution and filter. Keep both the filtrate and residue.		
e) To the filtrate obtained in (d) above, add an equal volume of ethanol followed by 4-5 drops of concentrated sulphuric acid. Heat the mixture, cool then pour the product in a beaker containing cold water.		
f) To the residue obtained in (d) above, add dilute ammonia solution drop wise until in excess.		

g) Comment on the nature of Q.

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CHAPTER TEN
10.0 CHEMICAL ENERGETICS

10.1 Introduction
Chemical Energetics is a branch of chemistry that deals with heat changes that take place in a variety of chemical reactions.

The total energy content of a substance stored in its bonds is known as the enthalpy, H. Therefore, when there is a change in the total energy content of the reacting system at the end of a chemical reaction, then we have enthalpy change, ΔH.

The practical branch of Chemical Energetics is utilized in the determination of:
- Concentrations of solutions (standardization of solutions).
- Enthalpy of neutralization of a base by an acid/an acid by a base.
- Enthalpy of displacement of copper(II) ions by magnesium, zinc,iron, e.t.c.
- Enthalpy of dissociation of weak acids (organic acids).
- Basicity of an acid.

10.2 Worked out Examples on Chemical Energetics

Worked out example 10.2.1
You are provided with the following:
FA1 which isnitric acid
FA2 which is sodium hydroxide solution
Solid T which is anhydrous sodium carbonate
You are required to determine the concentration of sodium hydroxide in grams per litre by thermometric titration with nitric acid

Procedure I
Weigh accurately, 15.4g of T into a clean beaker. Add 100cm^3 of water and stir well to dissolve. Transfer the solution into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label the solution FA3.

Pipette 20 or 25cm^3 of FA3 into a clean conical flask. Add 2-3 drops of methyl orange indicator and titrate with FA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below.

Mass of container + T……..……..*54.0*……..g
Mass of container alone……*38.6*……..g
Mass of solid T…….……*15.4*……...g
Volume of pipette used……..*25*……cm^3

Final burette reading (cm^3)	*19.50*	*19.30*	*29.30*
Initial burette reading (cm^3)	*0.00*	*0.00*	*10.00*
Volume of FA1 used (cm^3)	*19.50*	*19.30*	*19.30*

Values used to calculate average……*19.30, 19.30*……cm^3

Average volume of FA1 used……$\left(\frac{19.30 + 19.30}{2}\right)$ = ***19.30***……cm^3

Questions

a) Calculate the molar concentration of:

i)sodium carbonate in FA3.(Na=23, C=12, O=16)

Molar Mass of Na_2CO_3 = [(23x2)+(12x1)+(16x3)]g

= (46+12+48)g

= 106*g*

106g of sodium carbonate contain 1 mole

15.4g of sodium carbonate contain $\left(\frac{1}{106}\right)$*moles*

= 0.145 moles

250cm^3 of FA3 contain 0.145 moles of sodium carbonate

1000cm^3 of FA3 contain $\left(\frac{0.145}{250}\text{x}1000\right)$ *moles of sodium carbonate*

= ***0.58 M***

ii) nitric acid in FA1.

$Na_2CO_3(aq) + 2HNO_3(aq) \rightarrow 2NaNO_3(aq) + H_2O(l) + CO_2(g)$

1000cm^3 of FA3 contain 0.58 moles of sodium carbonate

25cm^3 of FA3 contain $\left(\frac{0.58}{1000}\ x25\right)$*moles of sodium carbonate*

= ***0.0145*** *moles of sodium carbonate*

Moles of nitric acid = (2xmoles of sodium carbonate)

= (2x0.0145)

= ***0.029*** *moles of nitric acid*

19.30cm^3 of FA1 contain 0.029 moles of nitric acid

1000cm^3 of FA1 contain $\left(\frac{0.029}{19.30}x\ 1000\right)$*moles of nitric acid*

= 1.503 M

≃***1.5 M***

Procedure II

i) Using a measuring cylinder, measure 30cm^3 of FA1 into a clean plastic beaker.

ii) Using a thermometer, note and record the initial temperature, T_0 of solution FA1.

iii) Using a burette, run 10cm^3 of FA2 into the FA1 solution in the plastic beaker. Stir the mixture with the thermometer and note the maximum temperature, T reached by the mixture.

iv) Repeat procedures (i) to (iii) for volumes of FA2 added equal to 15, 20, 25, 30 and 35cm^3, and record your results in the table below.

Volume of FA2 added (cm^3)	*0*	*10*	*15*	*20*	*25*	*30*	*35*
Maximum temperature of mixture, T (^0C)	*25.0*	*29.0*	*31.0*	*32.0*	*32.0*	*31.0*	*30.0*
Temperature rise (T- T_0) (^0C)	*0.0*	*4.0*	*6.0*	*7.0*	*7.0*	*6.0*	*5.0*

Questions

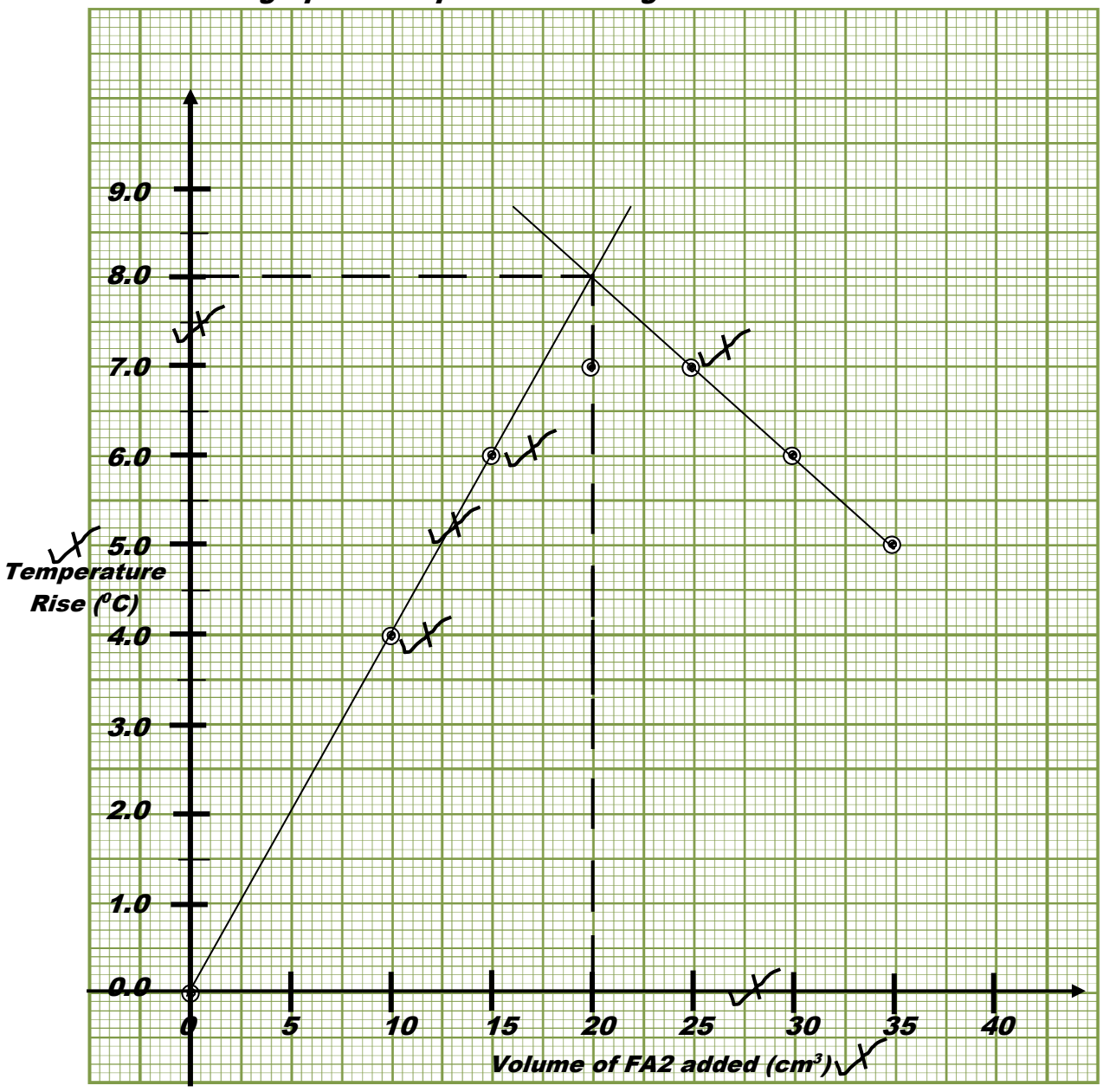
a) Plot a graph of temperature rise against volume of FA2 added.

b) By use of the graph, determine the:

i) maximum temperature rise, ΔT:……...…….*ΔT = 8.0*……..^0C

ii) volume of FA2 that completely neutralized the 30cm^3 of FA1: …….= *20*……..…..cm^3

A graph of temperature rise against volume of FA2



c) Calculate the:

i)number of moles of nitric acid which reacted.

1000cm^3 of FA1 contain 1.5 moles of nitric acid

30cm^3 of BA1 contain $\left(\frac{1.5 \times 30}{1000} x30\right)$ *moles of nitric acid*

***=0.045** moles of nitric acid*

ii) molar enthalpy of neutralization of nitric acid.(Density of mixture = 1g cm^{-3}; specific heat capacity of mixture= 4.2 Jg^{-1} ^{0}C^{-1}).

Total volume of mixture at neutralization point = (30+20)cm^3 ***= 50cm^3***

Total mass of mixture at neutralization point = (Density x volume)

= (1 x 50)g ***= 50 g***

ΔH = (Mass of mixture)x(Specific heat capacity of mixture)x(Max. temp. rise)

= (50 x 4.2 x 8.0) J ***= 1680 J***

$NaOH(aq) + HNO_3(aq) \rightarrow NaNO_3(aq) + H_2O(l)$

Moles of water = (1xmoles of nitric acid)

= (1 x 0.045)

= 0.045 moles of water

0.045 moles of water are formed with evolution of 1680 J

1 mole of water is formed with evolution of: $\left(\frac{1680}{0.045}\right)$ *Jmol^{-1}*

= 37333.33 Jmol^{-1}

$=\left(\frac{37333.33}{1000}\right)$ *kJmol^{-1}*

= 37.33kJmol^{-1}

iii) concentration of sodium hydroxide in grams per litre.(Na=23, O=16, H=1)

Moles of sodium hydroxide = (1xmoles of nitric acid)

Moles of sodium hydroxide = (1 x 0.045)

=0.045 moles of sodium hydroxide

20cm^3 of FA2 contain 0.045 moles of sodium hydroxide

1000cm^3 of FA2 contain $\left(\frac{0.045}{20} x1000\right)$ *moles of sodium hydroxide*

***= 2.25** moles of sodium hydroxide per litre*

Molar mass of NaOH = [(23x1) + (16x1) + (1x1)] ***= 40g***

1 mole of sodium hydroxide weighs 40g

2.25 moles of sodium hydroxide weigh: (40 x 2.25) g

=90 grams per litre

Worked out example 10.2.2

You are provided with the following:
HA1 which is 2.0M sulphuric acid
HA2 which is sodium hydroxide solution
HA3 which is 2.0M ethanoic acid
You are required to determine the:
i) concentration of sodium hydroxide in HA2 in moldm^{-3}.
ii) molar enthalpy of neutralisation between sulphuric acid andsodium hydroxide, and that between ethanoic acid and sodium hydroxide.
iii) enthalpy of dissociation of ethanoic acid.

Theory

Sodium hydroxide reacts with sulphuric acid and ethanoic acid according to the equations below.

$2NaOH(aq) + H_2SO_4(aq) \rightarrow Na_2SO_4(aq) + 2H_2O(l)$

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

Ethanoic acid dissociates according to the following equation:

$CH_3COOH(aq) \rightleftharpoons CH_3COO^-(aq) + H^+(aq)$

Produre I

i) Place HA1 into the burette.
ii) Using a measuring cylinder, measure and transfer 40cm^3 of HA2 into a plastic beaker. Measure and record its temperature into Table I below.
iii) Add 5cm^3 of HA1 from the burette. Stir the mixture carefully with a thermometer and record the highest temperature reached by the mixture in Table I below.
iv) Repeat procedure (iii) at intervals as shown in the table below.

Table I

Volume of HA1 (cm^3)	0	5	10	15	20	25	30
Highest temperature reached (^{0}C)	*22.0* ✓	*28.0* ✓	*32.5* ✓	*36.0* ✓	*39.0* ✓	*38.0* ✓	*36.0* ✓

Procedure II

i) Place HA3 into the burette.
ii) Using a measuring cylinder, measure and transfer 40cm^3 of HA2 into a plastic beaker. Measure and record its temperature into Table II below.
iii) Add 5cm^3 of HA3 from the burette. Stir the mixture carefully with a thermometer and record the highest temperature reached by the mixture in Table II below.
v) Repeat procedure (iii) at intervals as shown in Table II below.

Table II

Volume of HA1 (cm^3)	0	5	10	15	20	25	30
Highest temperature reached (^{0}C)	*22.0* ✓	*26.0* ✓	*29.0* ✓	*31.0* ✓	*32.0* ✓	*31.0* ✓	*30.0* ✓

Questions

a) Plot a graph of highest temperature against volume of sulphuric acid added.
b) Using the graph plotted in (a) above, determine the:

i) volume of sulphuric acid required for the neutralisation of sodium hydroxide.

……..…*20cm*^{3} ……

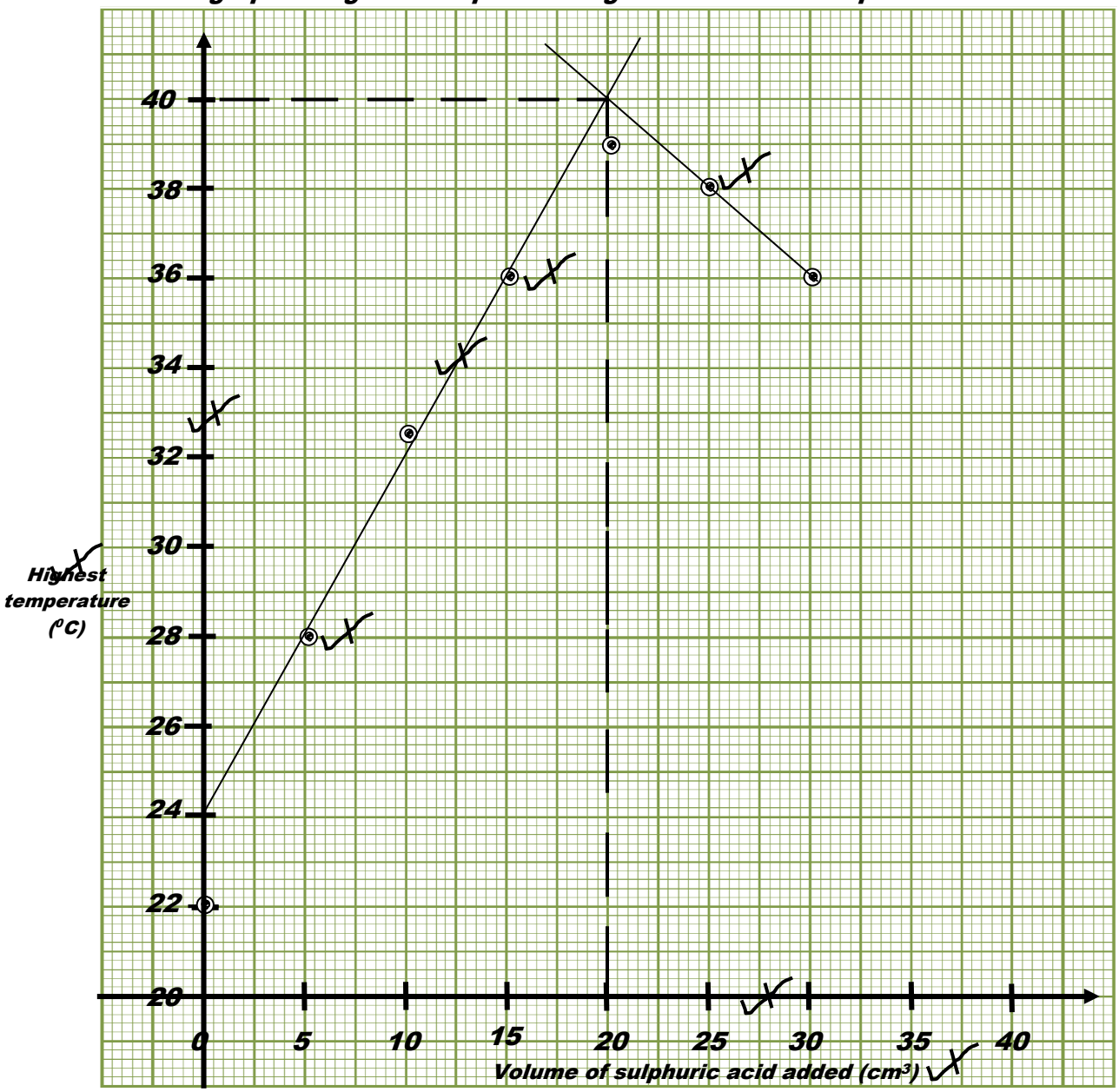
ii) maximum temperature reached at the neutralisation point.

……*40.0*^{0}*C* ……

iii) temperature change, ΔT at the neutralisation point.

……$\Delta T = (40.0-24.2)$ …= *15.8*^{0}*C* ……..

A graph of highest temperature against volume of sulphuric acid added



c) Calculate the concentration of sodium hydroxide in HA2 in moldm^{-3}.

1000cm^3 of HA1 contain 2.0 moles of sulphuric acid

20cm^3 of HA1 contain $\left(\frac{2.0}{1000} x20\right)$ *moles of sulphuric acid*

=0.04 moles of sulphuric acid

Moles of NaOH = (2xmoles of sulphuric acid)

= (2x0.04)= 0.08moles of sodium hydroxide

40cm^3 of HA2 contain 0.08 moles of sodium hydroxide

1000cm^3 of HA2 contain $\left(\frac{0.08}{40} x\ 1000\right)$ *moles of sodium hydroxide*

Concentration of sodium hydroxide in HA2= 2 moldm^{-3}

d) Calculate the molar enthalpy of neutralisation between sulphuric acid and sodium hydroxide in kJmol^{-1}.(Density of solution=1gcm^{-3}, Specific heat capacity of solution = 4.2 Jg^{-1} ^{0}C^{-1}).

Total volume of mixture at neutralisation point = (40+20) = 60cm^3

Mass of mixture = (density x volume)

*= (1x60)g = **60g***

ΔH *= mass x specific heat capacity x temperature change*

= (60x4.2x15.8)J

= - 3981.6 J

Moles of water = (2xmoles of sulphuric acid)

= (2x0.04)

= 0.08 moles of water

0.08 moles of water are formed with evolution of 3981.6J

1 mole of water is formed with evolution of $\left(\frac{3981.6}{0.08}\right)J$

$= -49770\ Jmol^{-1}$

$= \left(\frac{-49770}{1000}\right) kJmol^{-1} = -49.77 kJmol^{-1}$

e) Plot a graph of highest temperature againstvolume of ethanoic acid added.

f) Using the graph plotted in (e) above, determine the:

i) volume of ethanoic acid required for the neutralisation of sodium hydroxide

……***18cm^3***……

ii) maximum temperature reached at the neutralisation point.

……***32.4^{0}C***……..

iii) temperature change, ΔT at the neutralisation point.

……***(32.4 -23.6)= 8.8^{0}C*** ……..

g) Calculate the enthalpy of neutralisation between ethanoic acid and sodium hydroxide in kJmol^{-1}.

Total volume of mixture at neutralisation point = (40 + 18) = 58cm^3

Mass of mixture = (density x volume)

= (1x58)g= ***58g***

ΔH = mass x specific heat capacity x temperature change

= (58x4.2x8.8)J

= - 2143.68 J

1000cm^3 of FA3 contain 2.0 moles of ethanoic acid

18cm^3 of FA3 contain $\left(\frac{2.0}{1000} \times 18\right)$ *moles of ethanoic acid*

= 0.036 *moles of ethanoic acid*

Moles of water = (1xmoles of ethanoic acid)

= (1x0.036)= ***0.036*** *moles of water*

0.036 moles of water are formed with evolution of 2143.68J

1 mole of water is formed with evolution of $\left(\frac{2143.68}{0.036}\right)J$

= -59546.67Jmol^{-1}

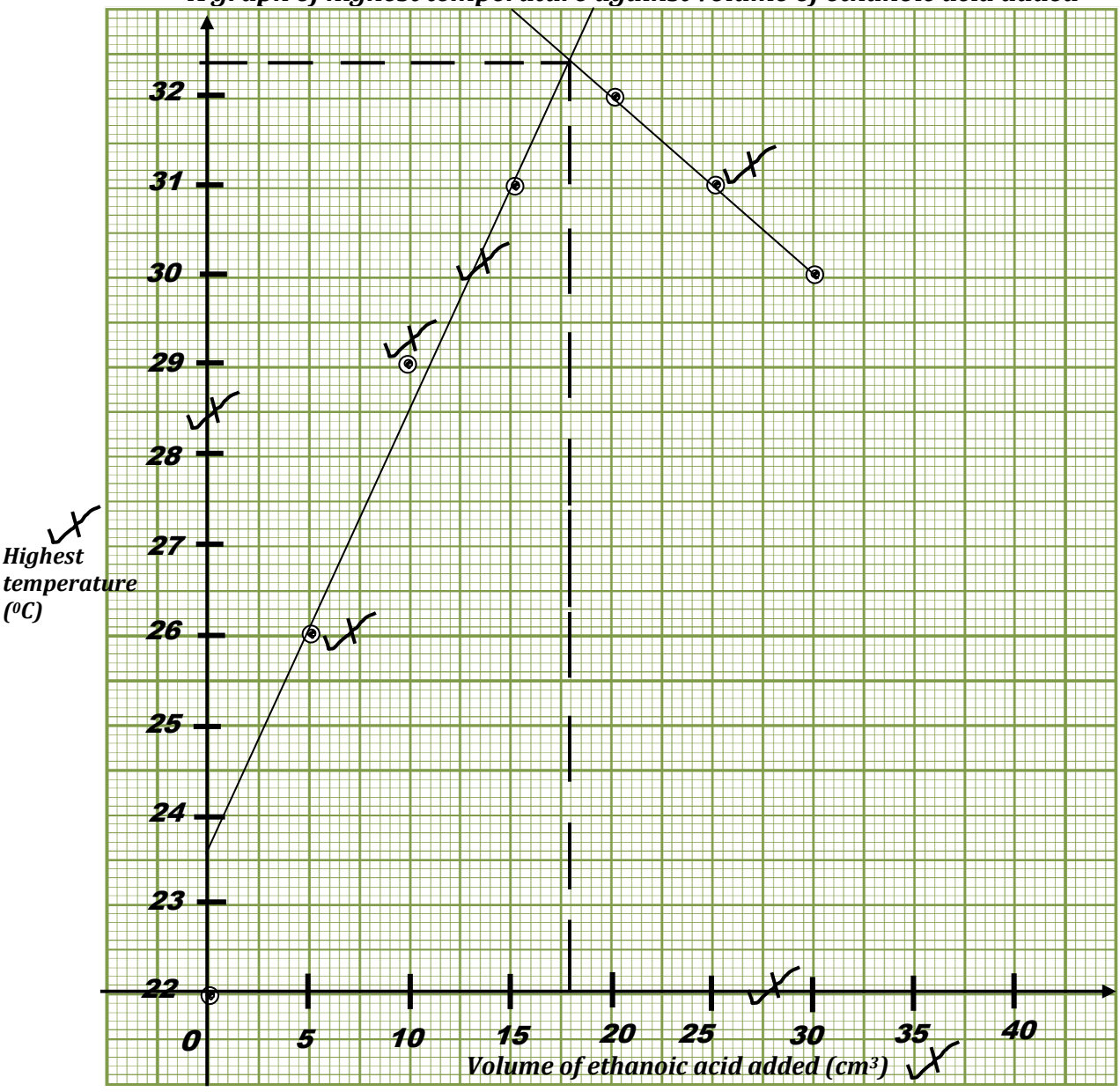
$=\left(\frac{-59546.67}{1000}\right)=$ ***-59.55kJmol^{-1}***

h) Determine the enthalpy of dissociation of ethanoic acid in kJmol^{-1}.

Enthalpy of dissociation of ethanoic acid = (-49.77-$^-$59.55) kJmol^{-1}

= (-49.77+59.55) kJmol^{-1}= ***+9.78 kJmol^{-1}***

A graph of highest temperature against volume of ethanoic acid added



Worked out example 10.2.3

You are provided with the following:

FA1 which is a solution of 1.0M copper(II) sulphate pentahydrate, $CuSO_4.5H_2O$

Solid **M** which is zinc metal powder.

You are required to determine the molar enthalpy of displacement of copper(II) ions by zinc metal.

Procedure

i) Using a measuring cylinder, measure and transfer $30cm^3$ of FA1 into a clean plastic beaker. Measure and record the initial temperature of FA1.

ii) Weigh accurately, 2.5g of zinc metal powder and add it to the FA1 solution in the plastic beaker. Stir well with a thermometer for about 20 seconds. Record the temperature reading of the mixture at the end of every 1 minute as shown in the table below.

Time (minutes)	0.0	1.0	2.0	3.0	4.0	5.0	6.0	7.0
Temperature (^{0}C)	*27.0*	*46.0*	*62.5*	*71.5*	*70.0*	*67.5*	*65.0*	*62.5*

Questions

(a) Plot a graph of temperature change against time.

(b) Using the graph you have plotted, determine the maximum temperature change, ΔT_{max} for the reaction……*= (77.5 – 27.0) ^{0}C* ***= 50.5*** ……^{0}C

(c) Determine the heat evolved in kJ mol^{-1} in the process of the displacement reactionin kJ mol^{-1}.

(Density of water =1gcm^{-3}, Specific heat capacity of water = 4.2$Jg^{-1}\,{}^0C^{-1}$).

Volume of solution = 30 cm^3

Mass of solution = (volume x density) = (30 x 1) = 30g

ΔH = (mass x specific heat capacity x temperature change)= (30 x 4.2 x 50.5) ***J*** *= 6363 J*

$= \left(\frac{6363}{1000}\right) kJ$

= 6.363kJ

d) Calculate the:

i) number of moles of copper(II) ions present in the volume of FA1 used in the experiment.

1000cm^3 of FA1 contan 1.0 moles of copper(II) ions.

30cm^3 of FA1 contain $\left(\frac{1.0}{1000} \text{ x } 30\right)$ *moles*

= 0.03moles

ii) mass of zinc metal used in the displacement reaction. (Zn = 65)

$Cu^{2+}(aq) + Zn(s) \rightarrow Cu(s) + Zn^{2+}(s)$

Moles of Zinc metal =(1xmoles of copper(II) ions)

= (1x0.03) ***= 0.03 moles***

1 mole of Zinc metal weighs 65g

0.03 moles of Zinc metal weigh (65x0.03)g

=1.95g

(iii) molar enthalpy of displacement of copper(II) ions by zinc metal.

0.03 moles of copper(II) ions evolve 6.363kJ

1 mole of copper(II) ions evolve $\left(\frac{6.363}{0.03}\right)$*kJ mol*$^{-1}$

$= -212.1$ *kJ mol*$^{-1}$

e) Outline atleast**four (4)** assumptions made in this experiment.

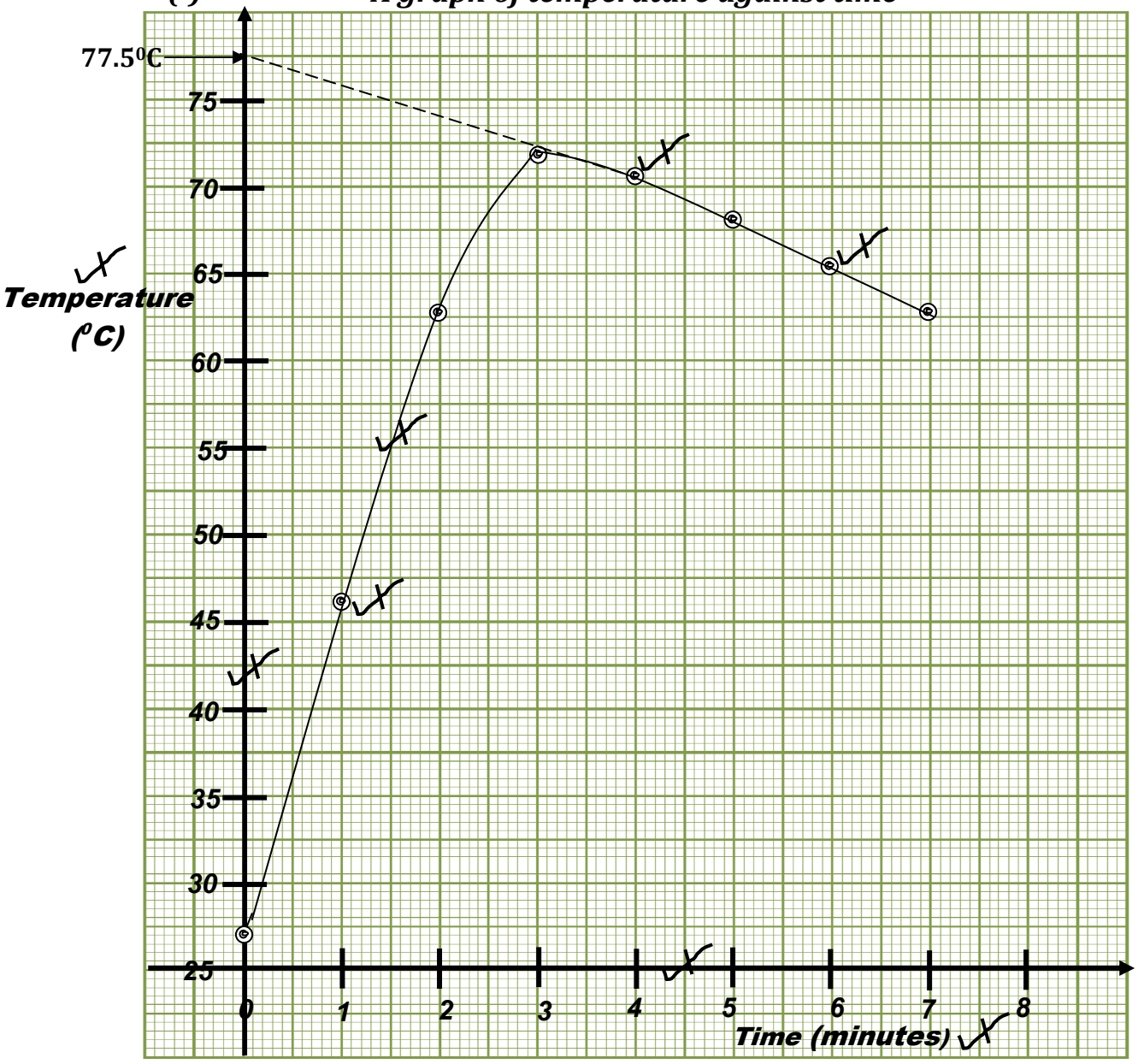
i) No heat is lost to the sorroundings.

ii) The heat capacity of the beaker is negligible.

iii) The specific heat capacity of the solution is equal to that of water.

iv) The density of the solution is equal to that of water.

(i) A graph of temperature against time



10.3 Practical Exercises on Chemical Energetics

Experiment 10.3.1

You are provided with the following:

FA1 which is a 0.5M solution of the acid, H_xP.

FA2 which is 1M potassium hydroxide solution.

You are required to determine the basicity of the acid, H_xP and the molar enthalpy of neutralisation of the acid by potassium hydroxide.

Procedure

i) Measure and transfer $40cm^3$ of FA1 into a clean plastic beaker.

ii) By use of a thermometer, determine and record the initial temperature of FA1, t_1 and the initial temperature of FA2, t_2.

iii) By use of another measuring cylinder, measure and add $10cm^3$ of FA2 to the $40cm^3$ of FA1 in the plastic beaker. Stir the mixture gently with the thermometer and record the maximum temperature reached by the mixture, t_4.

iv) Pour away the mixture and rinse the plastic beaker thoroughly with water.

v) Repeat procedure (i) to (iv) using the volumes of FA1 and FA2 indicated below. In a similar way, also indicate the values of t_1, t_2, t_3, plus the temperature change, $\Delta t = (t_4 - t_3)$ for each of the experiments carried out.

Experiment number	1	2	3	4	5	6	7
Volume of FA1 (cm^3)	40	35	30	25	20	15	10
Volume of FA2 (cm^3)	10	15	20	25	30	35	40
Initial temperature of FA1, t_1 (^0C)							
Initial temperature of FA2, t_2 (^0C)							
Average temperature, $t_3 = \left(\frac{t_1 + t_2}{2}\right)$ (^0C)							
Highest temperature reached, t_4 (^0C)							
Temperature change, $\Delta t = (t_4 - t_3)$ (^0C)							

Questions

a) Plot a graph of Δt against volume of FA1.

b) From the graph plotted, determine the following:

i) Maximum value of Δt …….....^0C

ii) Volume of the the acid, H_xT in FA1 that is required for neutralisation of the potassium hydroxide inFA2.

……...cm^3

iii) Volume of potassium hydroxide in FA2 that reacted with the acid, H_xP in FA1 to give the maximum value of Δt……..cm^3

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c) Determine the:

i) stoichiometry between the acid H_xP and the potassium hydroxide.

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ii) basicity of the acid, H_xP.

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d) Calculate the molar heat of neutralisation of the acid, H_xP. (Density of solution=1gcm^{-3}, Specific heat capacity of solution = 4.2 Jg^{-1} ^{0}C^{-1})

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Experiment 10.3.2
You are provided with the following:
FA1 which is sodium hydroxide solution
FA2 which is nitric acid
Solid F which is anhydrous sodium carbonate
You are required to determine the concentration of sodium hydroxide by thermometric titration with hydrochloric acid.

Procedure I
Weigh accurately, 12.3g of F into a clean beaker. Add 100cm^3 of water and stir well to dissolve. Transfer the solution into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label the solution FA3.

Pipette 20 or 25cm^3 of FA3 into a clean conical flask. Add 2-3 drops of methyl orange indicator and titrate with FA2 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below.

Mass of container + F……..……..…….……..g

Mass of container …….....g

Mass of solid F...…….……g

Volume of pipette used……..……cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA2 used (cm^3)			

Values used to calculate average……..……cm^3

Average volume of FA2 used……..……cm^3

Questions

a) Determine the concentration of:

i) sodium carbonate in FA3 in mol dm^{-3}.(Na=23, C=12, O=16)

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ii) hydrochloric acid in FA2 in mol dm^{-3}.

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Procedure II

i) Using a measuring cylinder, measure $15cm^3$ of FA2 into a clean plastic beaker.

ii) Using a thermometer, note and record the initial temperature, T_0 of solution FA2.

iii) Using a burette, run $5cm^3$ of FA1 into the FA2 solution in the plastic beaker. Stir the mixture with the thermometer and note the maximum temperature, T reached by the mixture.

iv) Repeat procedures (iii) for volumes of FA1 added equal to 10,15, 20, 25, 30 and$35cm^3$, each time adding an extra $5cm^3$ to the solution in the plastic beakerand record your results in the table below.

Volume of FA1 added (cm^3)	*0*	*5*	*10*	*15*	*20*	*25*	*30*	*35*
Maximum temperature of mixture, T (^0C)								
Temperature rise (T- T_0) (^0C)								

Questions

a) Plot a graph of temperature rise against volume of FA2 added.

b) By use of the graph you have plotted, determine the:

i) maximum temperature rise, ΔT.

……

ii) volume of FA2 that completely neutralized the $40cm^3$ of FA1

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c) Calculate the:

i)number of moles of hydrochloric acid which reacted.

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ii) molar enthalpy of neutralization of nitric acid. (Density of mixture = 1g cm^{-3}; specific heat capacity of mixture= 4.2 $Jg^{-1}\,{}^{0}C^{-1}$).

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iii) concentration of sodium hydroxide in grams per litre. (Na=23, O=16, H=1)

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Experiment 10.3.3

You are provided with the following:
FA1 which is 2.5M sulphuric acid
FA2 which is sodium hydroxide solution
FA3 which is 2.5M methanoic acid
You are required to determine the:
i) concentration of sodium hydroxide in FA2 in moles per litre.
ii) molar enthalpy of neutralisation between sulphuric acid and sodium hydroxide,and that betweenmethanoic acid and sodium hydroxide.
iii) enthalpy of dissociation of methanoic acid.

Theory

Sodium hydroxide reacts with sulphuric acid and Methanoic acid according to the equations below.

$2NaOH(aq) + H_2SO_4(aq) \rightarrow Na_2SO_4(aq) + 2H_2O(l)$

$NaOH(aq) + HCOOH(aq) \rightarrow HCOONa(aq) + H_2O(l)$

Methanoic acid dissociates according to the following equation:

$HCOOH(aq) \rightleftharpoons HCOO^-(aq) + H^+(aq)$

Produre I

i) Place FA1 into the burette.

ii) Using a measuring cylinder, measure and transfer 40cm^3 of FA2 into a plastic beaker. Measure and record its temperature into Table I below.

iii)Add 5cm^3 of FA1 from the burette. Stir the mixture carefully with a thermometer and record the highest temperature reached by the mixture in Table I below.

iv) Repeat procedure (iii) at intervals, each time adding an extra 5cm^3 of FA1 as shown in table Ibelow.

Table I

Volume of FA1 (cm^3)	0	5	10	15	20	25	30	35	40
Highest temperature reached (^{0}C)									

Procedure II

i) Place FA3 into the burette.

ii) Using a measuring cylinder, measure and transfer 40cm^3 of FA2 into a plastic beaker. Measure and record its temperature into Table II below.

iii) Add 5cm^3 of FA3 from the burette. Stir the mixture carefully with a thermometer and record the highest temperature reached by the mixture in Table II below.

iv) Repeat procedure (iii) at intervals, each time adding an extra 5cm^3 as shown in Table IIbelow.

Table II

Volume of FA3 (cm^3)	0	5	10	15	20	25	30
Highest temperature reached (^{0}C)							

Questions

a) Plot a graph of highest temperature against volume of sulphuric acid added.

b) Using the graph plotted in (a) above, determine the:

i) volume of sulphuric acid required for the neutralisation of sodium hydroxide

……..……

ii) maximum temperature reached at the neutralisation point.

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iii) temperature change, ΔT at the neutralisation point.

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c) Calculate the concentration of sodium hydroxide in HA2 in moldm^{-3}.

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d) Calculate the molar enthalpy of neutralisation between sulphuric acid and sodium hydroxide in kJmol^{-1}.(Density of solution=1gcm^{-3}, Specific heat capacity of solution = 4.2 Jg^{-1} ^{0}C^{-1})

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e) Plot a graph of highest temperature against volume of methanoic acid added.

f) Using the graph plotted in (e) above, determine the:

i) volume of methanoic acid required for the neutralisation of sodium hydroxide

……..……..

ii) maximum temperature reached at the neutralisation point.

……

iii) temperature change, ΔT at the neutralisation point.

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g) Calculate the enthalpy of neutralisation between methanoic acid and sodium hydroxide in kJmol^{-1}.

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h) Determine the enthalpy of dissociation of methanoic acid in $kJmol^{-1}$.

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Experiment 10.3.4

You are provided with the following:

GA1 which is a solution of 0.25M copper(II) chloride dihydrate, $CuCl_2.2H_2O$

Solid **Y** which is magnesium metal powder.

You are required to determine the molar enthalpy of displacement of copper(II) ions by magnesium metal.

Procedure

i) Using a measuring cylinder, measure and transfer $60cm^3$ of GA1 into a clean plastic beaker. Measure and record the initial temperature of GA1.

ii) Weigh accurately, 1.5g of Magnesium metal powder and add it to the GA1 solution in the plastic beaker. Stir well with a thermometer for about 20 seconds. Record the temperature reading of the mixture at the end of every 30 seconds as shown in the table below.

Time (s)	0	30	60	90	120	150	180	210	240	270	300
Temperature (^0C)											

Questions

a) Plot a graph of temperature change against time.

b) Using the graph you have plotted, determine the maximum temperature change, ΔT_{max} for the reaction……...……^0C

c) Determine the heat evolved in the process of the displacement reaction.

(Density of water =$1gcm^{-3}$, Specific heat capacity of water = $4.2Jg^{-1}\,{}^0C^{-1}$).

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d) Calculate the:

i) number of moles of copper(II) ions present in the volume of GA1 used in the experiment.

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ii)mass of magnesium metalused in the displacement reaction. (Mg=24).

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iii) molar enthalpy of displacement of copper(II) ions by magnesium metal.

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d) Outline atleast**three (3)** assumptions made in this experiment.

i)……

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ii)……

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iii)……

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CHAPTER ELEVEN
11.0 CHEMICAL KINETICS

11.1 Introduction

A wide range of chemical reactions occur at differing rates. In this chapter, however, for experimental purposes, we shall deal with reactions that go to completion within seconds or a few minutes' time.

The rate of a chemical reaction is the change in concentration of (a) reactant(s) or product(s) per unit time. In these experiments, we shall focus on determination of orders of reactions as well as activation energy, Ea of specific reactions. The reciprocal of time, $^{1}/t$ gives an indication of the rate of reaction.

Order of a reaction is the sum of the powers to which the concentrations of reactants are raised in an experimental rate equation.
Consider the reaction below with reactants A, B and C reacting to form theproducts.

$wA + xB + yC \longrightarrow Products$

$Rate\ of\ reaction = k[A]^{w}[B]^{x}[C]^{y}$

Where [A],[B] and[C]are the molar concentrations of reactants A, B and C;
w, x and y are the orders of reaction with respect to A, B and C respectively;
kis the rate constant.

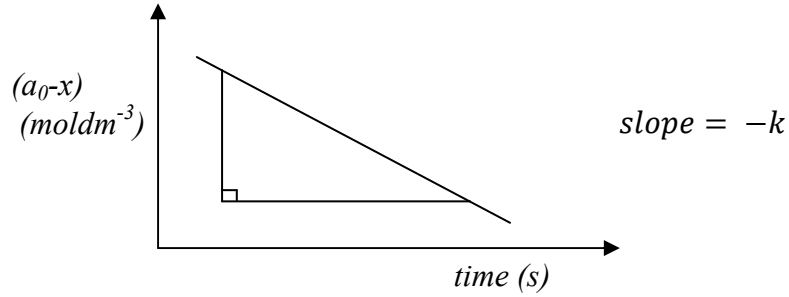
Note:*Overall order of reaction = (w+x+y)*

Graphical determination of the Orders of Reactions

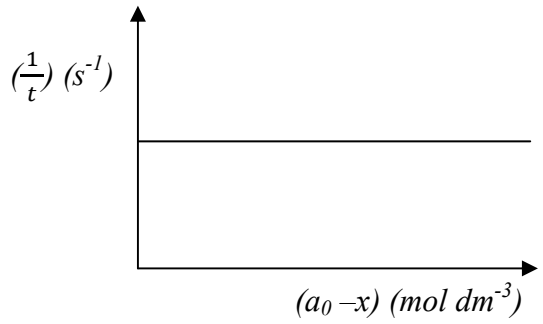
If reactants B and C are used in a large excess, then the rate of reaction will depend on the concentration of A and hence the value of **w** which is the order of reaction with respect to reactant A can be obtained experimentally.

a) Zero Order Reaction

A reaction will be proved to be **zero order** with respect to reactant A (the value of w will be zero) if:
i)a plot of(a_0-x) against time, t gives a straight line with a negative gradient whose slope is **-k**.
Where: a_0 = initial concentration of reactant A at t=0
x = amount of reactant A that has disintegrated/reacted.
(a_0-x) = concentration of reactant A at time t.
t = time taken.



ii) a plot of the rate of reaction, $^{1}/t$ against (a_0-x) gives a straight line parallelto the horizontal axis.



b) First Order Reaction

A reaction will be proved to be **first order** with respect to reactant A (the value of w will be 1) if:

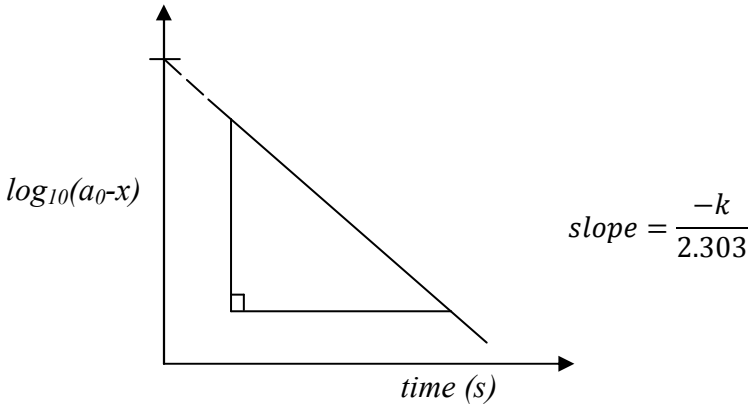
i) a plot of $\log_{10}(a_0\text{-}x)$ against time, t gives a straight line with a negative gradient. The slope of the graph is $\frac{-k}{2.303}$ andthe intercept on the vertical axisis equal to **$\log_{10} a_0$.**

Where: a_0 = initial concentration of reactant Aat t=0.

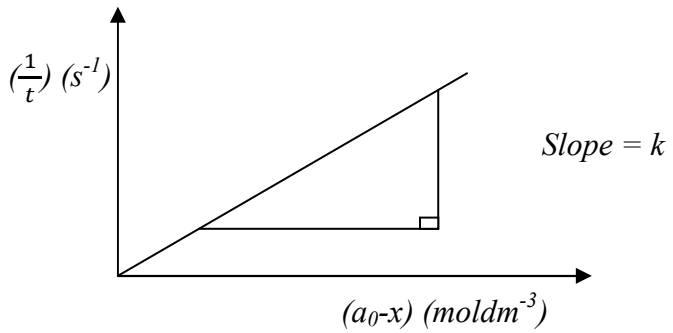
x = amount of reactant A that has disintegrated

(a_0-x) = concentration of reactant A at time t.

t = time taken.

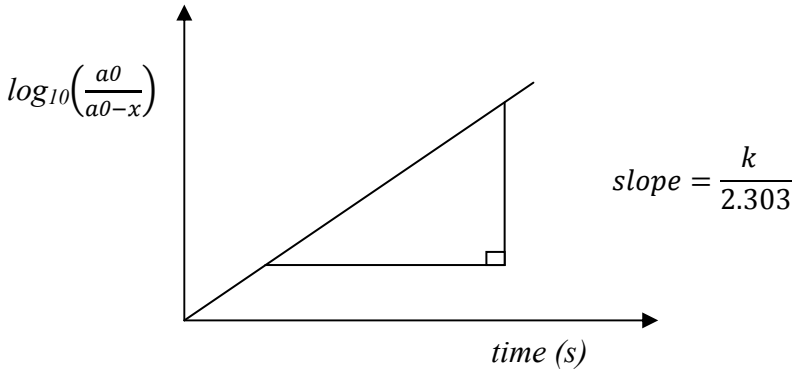


ii) a plot of $^{1}/t$ against (a_0-x) gives a straight line through the origin (with apositive gradient). Its slope isequal to the rate constant, **k**.

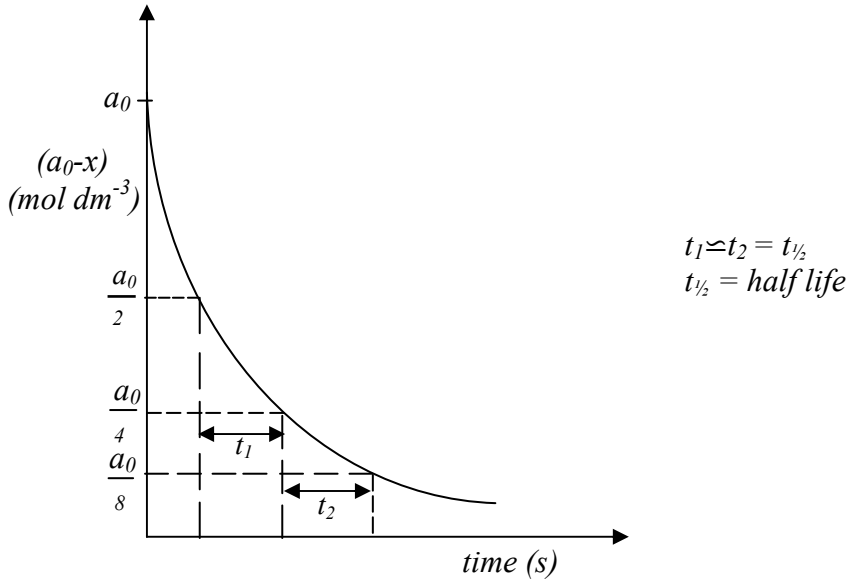


Note: *A plot of (a_0-x) against $^{1}/t$ gives a graph of a similar shapewhose slope is* ***$^{1}/k$.***

iii) a plot of $log_{10}\left(\frac{a0}{a0-x}\right)$ against time, t gives a straight line through the origin with a positive gradient whose slope is $\frac{k}{2.303}$.



iv) a plot of (a_0-x) against time gives a smooth curve with a negative gradient with equal half life.

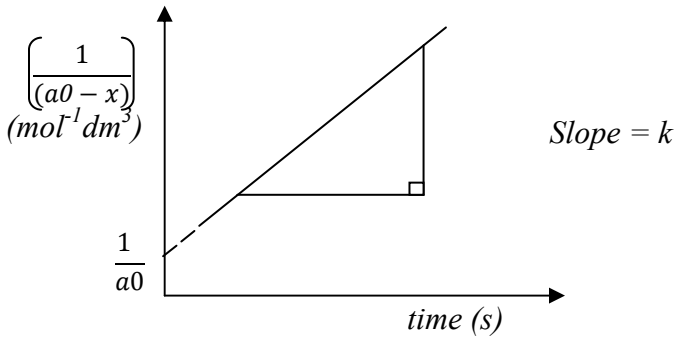


c) Second Order Reaction

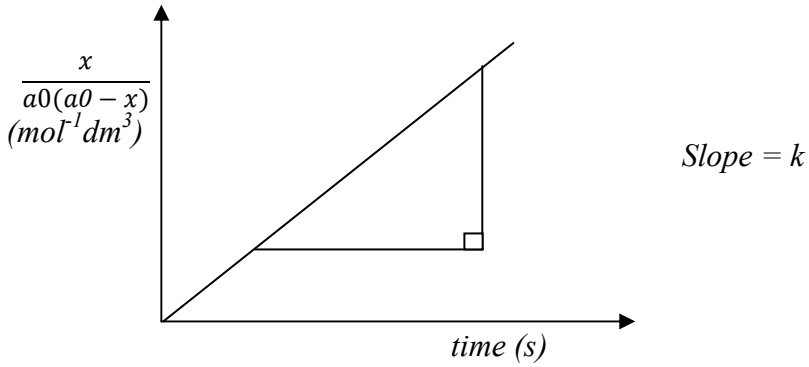
A reaction will be proved to be **second order** with respect to reactant A (the value of w will be 2) if:

i) a plot of $\frac{1}{a0-x}$ against time, t gives a straight line with a positive gradient. The slope of the graph is ***k*** and the intercept on the vertical axis is equal to $\frac{1}{a0}$.

Where: a_0 = initial concentration of reactant A at t=0
x = amount of reactant A that has disintegrated
(a_0-x) = concentration of reactant A at time t.
t = time taken.



ii)a plot of $\frac{x}{a0(a0-x)}$ against time gives a straight line through the origin, with a positive gradient whose slope is *k.*



Note:
1) Any other variable that is directly proportional to the one indicated in the graphs above can also be used in plotting. For instance volume of reactant A can be used instead of (a_0-x) while ^{1}/volume of reactant A can be usedinstead of $\frac{1}{a0-x}$ and curves of the same shape are obtained as above.
2) Similarly, in case the variable on the vertical axis is interchanged with that on the horizontal axis, curves of similar shapes are obtained as above.

11.2 Worked out Examples on Chemical Kinetics

Worked out Example 11.2.1
You are provided with the following:
FA1 which is iodine solution.
FA2 which is 0.5M propanone solution.
FA3 which is 0.01M sodium thiosulphate solution.
FA4 which is 0.5M sodium hydrogencarbonate solution.
1M sulphuric acid.
Starch solution
You are required to determine the order of reaction with respect to iodine in its reaction with propanone in the presence of an acid catalyst.

Theory

Aqueous iodine reacts with propanone in the presence of an acid catalyst to form 1-iodopropanone. During the progress of the reaction, when sodium hydrogencarbonate is added to the reaction mixture, the acid catalyst is neutralized and the reaction stops immediately so that some iodine remains unreacted. A portion of the reaction mixture containing the unreacted iodine is pipetted and titrated against standard sodium thiosulphate solution. The volume of sodium thiosulphate used is directly proportional to the concentration of iodine in the pipetted volume of the reaction mixture at any given time. Hence instead of plotting concentration of iodine, volume of sodium thiosulphate can be plotted against time.

Assumption: *The propanone and the acid are in large excess such that their concentration will be assumed to have no effect on the rate of reaction.*

Procedure

i) Pipette 25.0cm^3 of **FA2** into a conical flask.Using a measuring cylinder, add 25.0cm^3 of 1M sulphuric acid followed by 50cm^3 of **FA1**, and at the same time, start the stop clock. Shake the mixture gently and label it **FA5**.

ii) Using another measuring cylinder, transfer 10cm^3 of **FA4** into another conical flask. At the end of 2 minutes, pipette 10cm^3 of **FA5** into the conical flask containing the 10cm^3 of **FA4 (MAKE SURE YOU DO NOT STOP THE STOP CLOCK).** Shake the mixture until no more effervescence occurs. Add 1cm^3 of starch solution and titrate the mixture with **FA3** from the burette.

iii) Record your results in the table below.

iv) Repeatprocedure (ii) above after every **6 minutes** for the period of time indicated in the table below.

Time from the start of the reaction (min)	2	8	14	20	26	32	38
Final burette reading (cm^3)	*8.50* ✓	*16.60* ✓	*22.50* ✓	*27.60* ✓	*30.80* ✓	*32.80* ✓	*33.70* ✓
Initial burette reading (cm^3)	*0.10* ✓	*8.50* ✓	*16.60* ✓	*22.50* ✓	*27.60* ✓	*30.80* ✓	*32.80* ✓
Volume of **FA3**used (cm^3)	*8.40* ✓	*8.10* ✓	*5.90* ✓	*5.10* ✓	*3.20* ✓	*2.00* ✓	*0.90* ✓

(10 ½ marks)

Questions

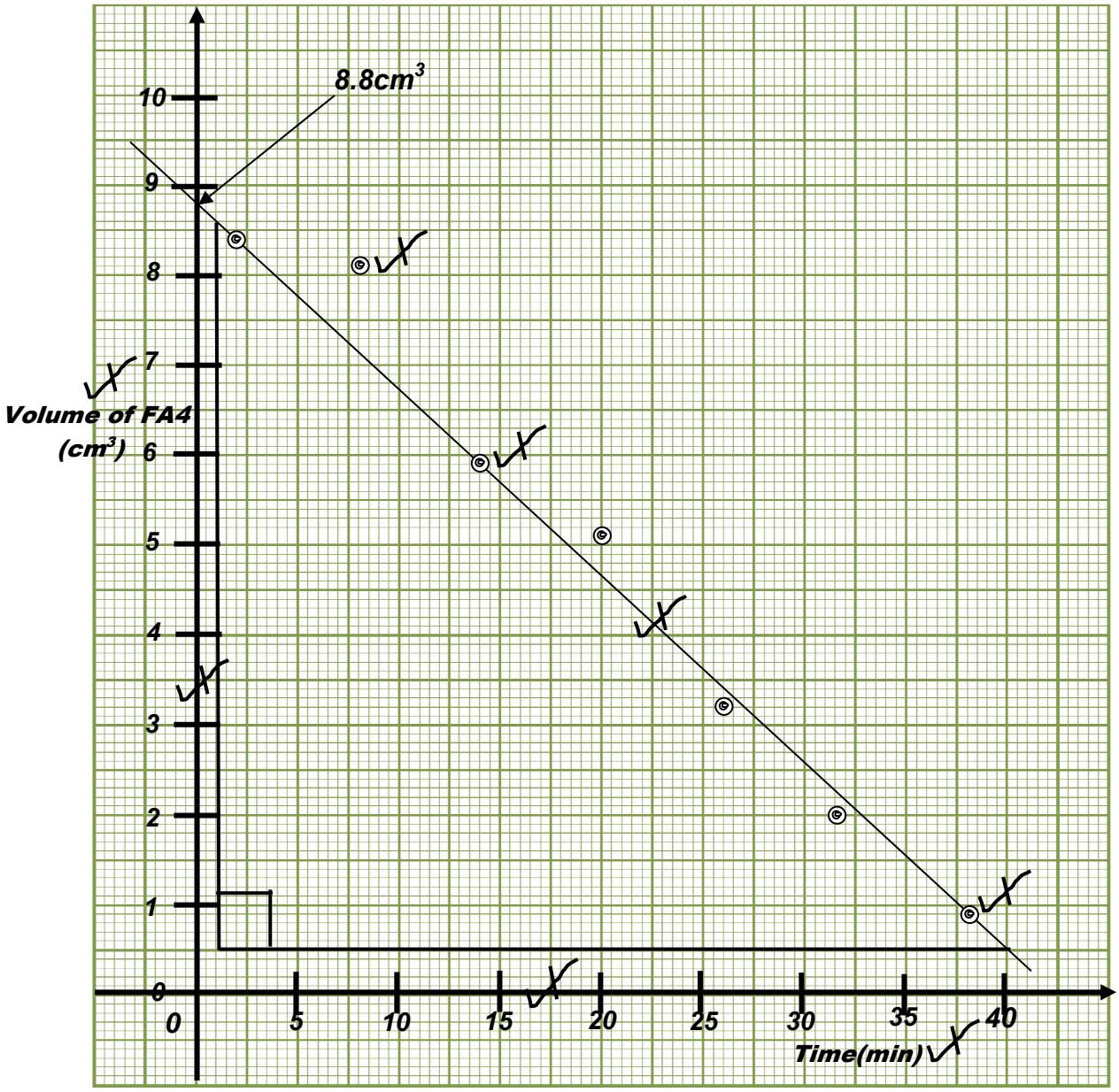
a) Plot a graph of volume of FA4 against time.

b) Using the graph you have plotted in (a) above:

(i) deduce the order of reaction with respect to iodine. (include a reason for the order of reaction you have stated) (10 ½ marks)

The reaction is zero order with respect to iodine.✓This is because a graph of volume of FA4 (sodium thiosulphate) against time gives a straight line with a negative gradient.✓This implies that the rate of reaction is independent of the concentration of iodine.✓OR This implies that the rate of reaction is constant (slope of the graph is constant) irrespective of the concentration of iodine].

(a)(iii) **A graph of volume of FA4 against time**



(ii) determine the volume of FA4 at t = 0 minutes.

8.8cm^3

……

(iii) determine the value of the rate constant, *k* and **include its units** given that the slope for the graph you have plotted in (a) above is given by: ***slope = -k.***

Slope $= \frac{\text{Change in volume of FA4 values}}{\text{Change in time}} = \frac{(0.5 - 8.6)\,cm^3}{(40 - 1)\,min} = \frac{-8.1\,cm^3}{39\,min} = -0.208\ cm^3\ min^{-1}$

$Slope = -k; \quad -0.208\ cm^3\ min^{-1} = -k; \quad k = 0.208\ cm^3\ min^{-1}$

c) Using the answer you have obtained in (b)(ii) above, determine the molar concentration of the iodine solution in FA1.

1000cm^3 of FA4 contain 0.01 moles of $S_2O_3^{2-}$

8.8cm^3 of FA4 contain $\left(\frac{0.01}{1000} \text{ X } 8.8\right)$ *moles of $S_2O_3^{2-}$*

= 8.8x10^{-5} moles of $S_2O_3^{2-}$

$I_2(aq) + 2S_2O_3^{2-}(aq) \longrightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$

Moles of I_2 = (½ x moles of $S_2O_3^{2-}$) = (½ x 8.8x10^{-5}) = 4.4 x10^{-5}

50cm^3 of FA1 contain 4.4 x10^{-5} moles of I_2

1000cm^3 of FA1 contain $\left(\frac{4.4 \text{ x } 10-5}{50} \text{ X } 1000\right)$ *moles of I_2*

= 8.8x10^{-4} moles of I_2 per dm^3

Worked out Example 11.2.2

You are provided with the following:

FA1 which is 0.1M hydrogen peroxide solution

FA2 which is 0.06M sodium thiosulphate

FA3 which 0.1M potassium iodide solution acidified with sulphuric acid

Starch solution

You are required to investigatethe rate of reaction between hydrogen peroxide and potassium iodide and to determine the order of reaction with respect to hydrogren peroxide.

Theory

Hydrogen peroxide reacts with acidified potassium iodide solution to liberate aqueous iodine.

The liberated iodine then reacts with thiosulphate ions according to the equation below.

$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$

Since the iodine is liberated in excess, once all the thiosulphate ions available have reacted with iodine, the unreacted iodine then combines with the starch solution in the reaction mixture to form the blue-black starch-iodine complex. Hence appearance of the blue colour in the reaction mixture is an indication that all the sodium thiosulphate has reacted with iodine (the reaction between the two is complete).

Procedure

i) Using ameasuring cylinder, transfer 25cm^3 of FA3 into a conical flask.

ii) Add 15cm^3 of FA2 from the burette followed by addition of 25cm^3 of distilled water and then 2cm^3 of starch indicator solution using suitable measuring cylinders. Shake gently to mix.

iii) To the mixture in part (ii) above, add at once 10cm^3 of FA1 and simultaneously start the stop clock. Shake the mixture strongly and continuosly until the blue colour appears in the solution. Note and record the time taken for the blue colour to appear in the solution. Record your results in the table below.

iv)Repeat procedures (i) to (iii), but this time using 15, 20, 25 and 30cm^3 of FA1 for experiment 2, 3, 4 and 5respectively as indicated in the table below.

Experiment number	1	2	3	4	5

Volume of FA3 (cm^3)	25	25	25	25	25
Volume of FA2 (cm^3)	15	15	15	15	15
Volume of distilled water (cm^3)	25	20	15	10	5
Volume of starch solution (cm^3)	2	2	2	2	2
Volume of FA1 (cm^3)	10	15	20	25	30
log_{10} (volume of FA1)	*1.000* ✓	*1.176* ✓	*1.301* ✓	*1.398* ✓	*1.477* ✓
Time(s)	*80.0* ✓	*60.0* ✓	*45.0* ✓	*37.0* ✓	*29.0* ✓
Reciprocal of time, $^1/t$ (s^{-1})	*$1.25x10^{-2}$* ✓	*$1.67x10^{-2}$* ✓	*$2.22x10^{-2}$* ✓	*$2.70x10^{-2}$* ✓	*$3.45x10^{-2}$* ✓

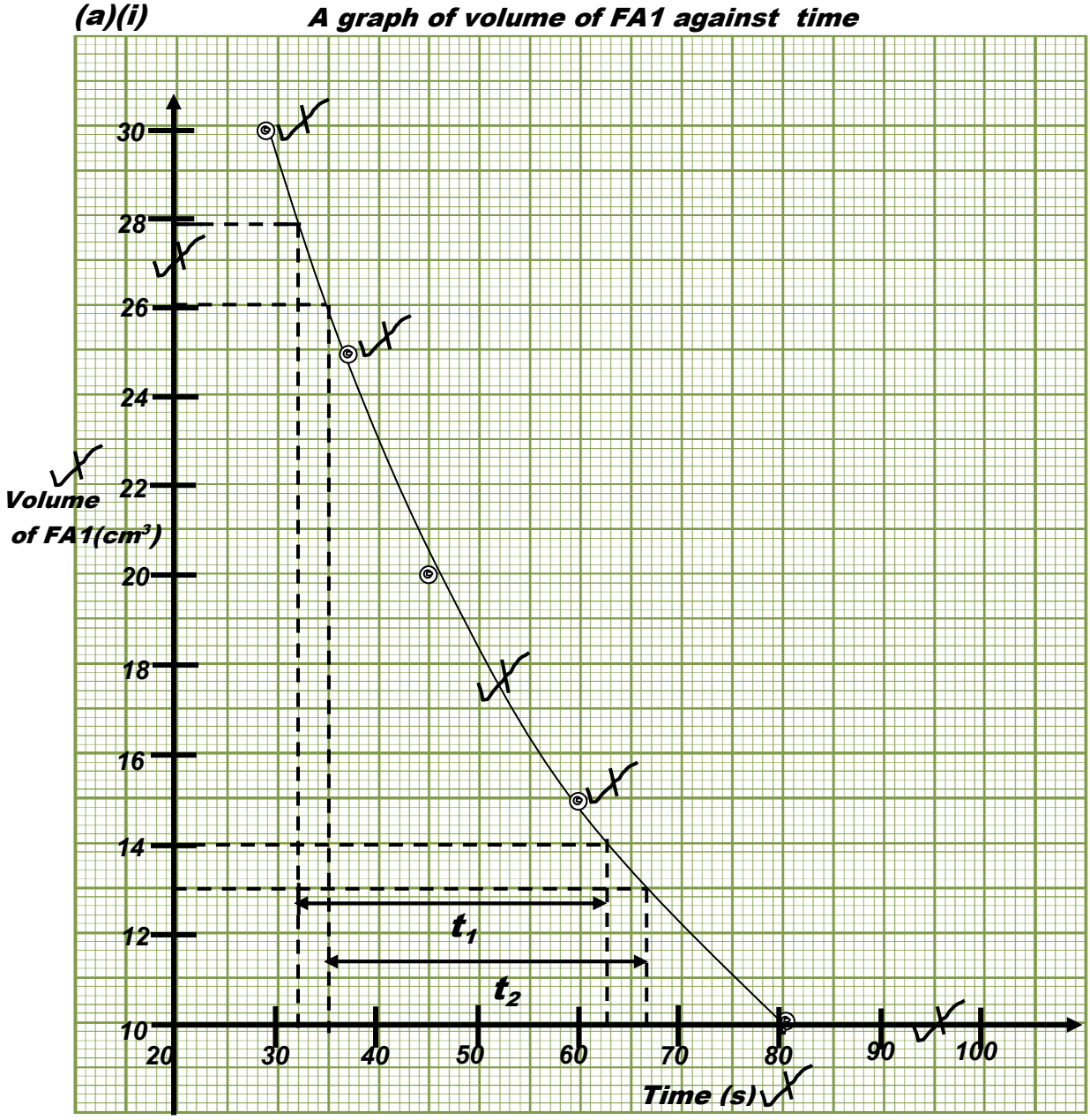
Questions

a) Plot a graph of:

i) volume of FA1 against time.

ii) volume of FA1 against $^1/t$.

iii) log_{10}(volume of FA1) against time.



b) Basing on the graph plotted for (a)(i) above:
(i)Deduce the order of reaction with respect to hydrogen peroxide.

Volume of FA1 (cm^3)	*Time taken (s)*	*Change in time (s)*
From 28	*32*	t_1 = (63-32)
to 14	*63*	= 31
From 26	*35*	t_2 = (67-35)
to 13	*67*	= 32

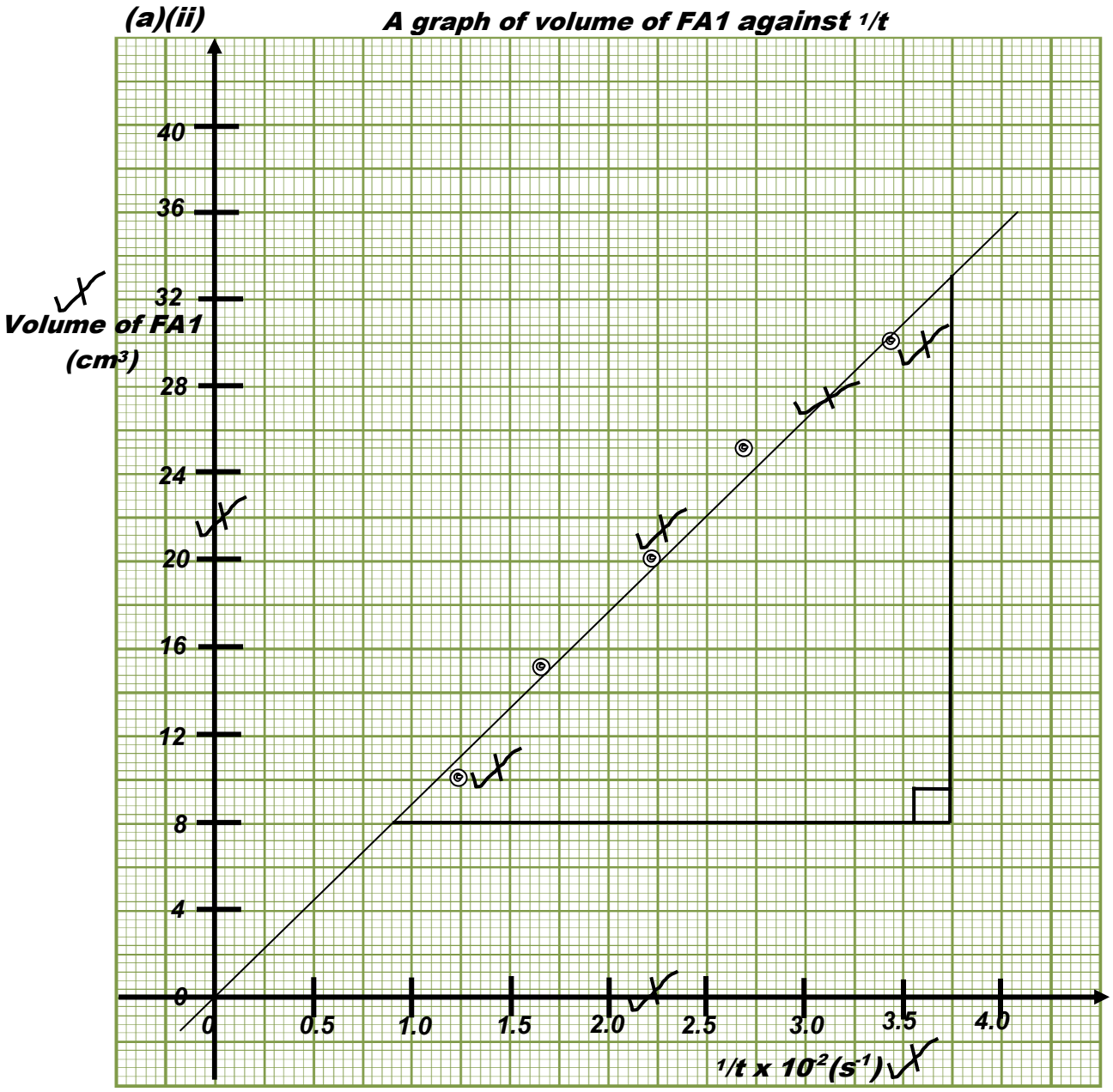
$t_1 \backsimeq t_2 = t_{½}$ (where $t_{½}$ = half life)

The reaction is first order with respect to hydrogen peroxide. This is because half life is independent of the initial volume (initial concentration) of hydrogen peroxide.

(ii)Determine the rate constant, *k*.

$t_{½} = \frac{t1 + t2}{2} = \frac{31 + 32}{2} = 31.5s$

$k = \frac{In\ 2}{t½} = \frac{0.693}{31.5\ s} = 0.022s^{-1}$



c) Determine the slope of the graph plotted for (a)(ii) above (include its units).

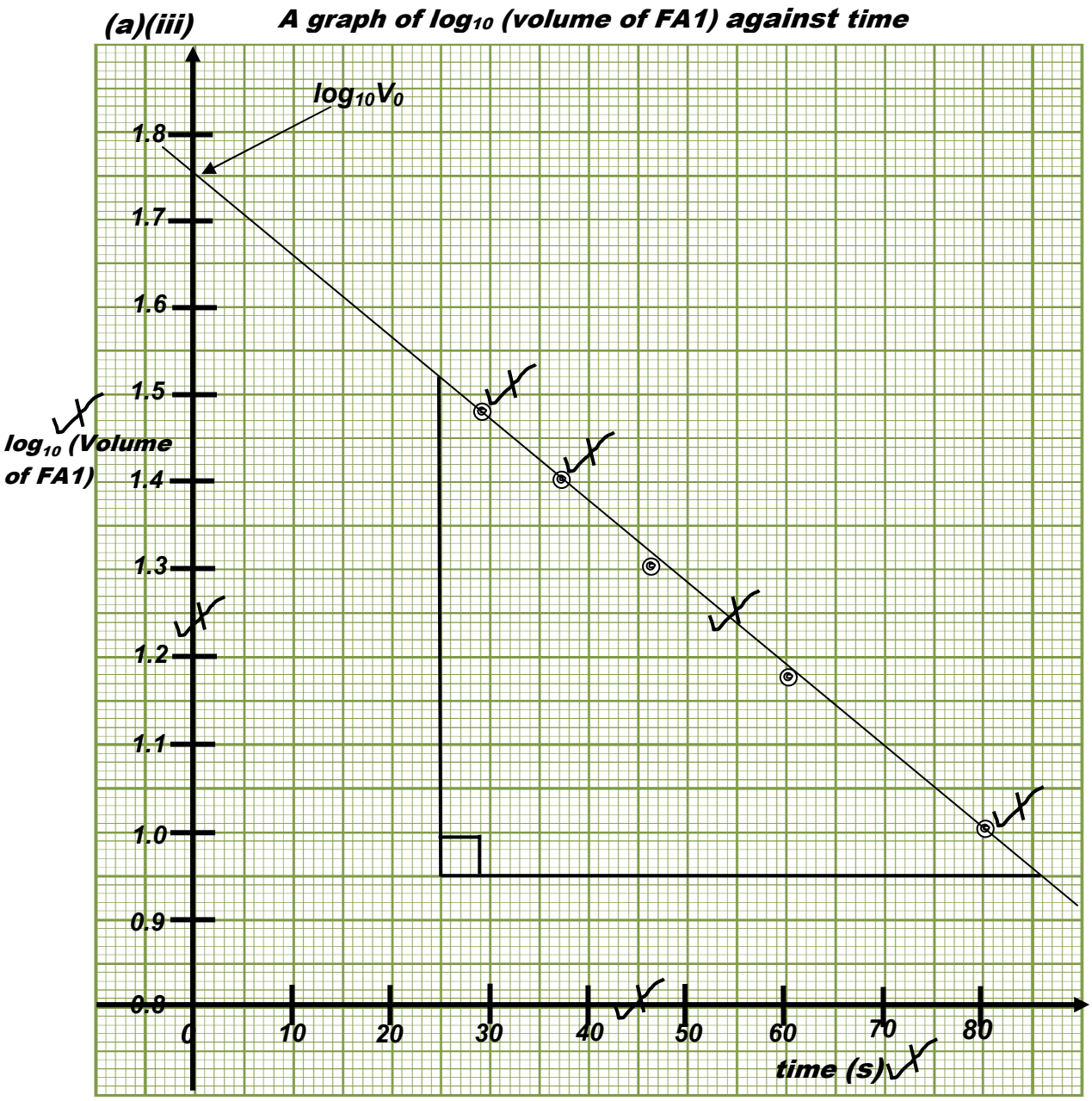
$$Slope = \frac{Change\ in\ volume\ of\ FA1\ values}{change\ in \frac{1}{t}\ values}$$

$$= \frac{(33.2-8.0)cm3}{(3.75-0.9)x10-2\ s-1} = \frac{(25.2)cm3}{2.85x10-2\ s-1} = \textbf{884.21 cm}^3\textbf{s}$$

d) Basing on the graph you have plotted for (a)(ii) above, deduce the order of reaction with respect to hydrogen peroxideperoxide.

The reaction is first order with respect to hydrogen peroxide. This is because a plot ofvolume of hydrogen peroxide (concentration of hydrogen peroxide) against $^1/t$ gives a straight line through the origin; with a positive gradient.

Note:*Since this is a first order reaction, if a graph of $^1/t$ against volume/concentration of hydrogen peroxide was plotted, a graph of a similar shape would be obtained, only that its slope would be equivalent to the value of the rate constant,* ***k****instead of* $^1/\mathbf{k}$.



e) (i) Calculate the slope for the graph plotted in **(a)(iii)** above and use it to determine the value of the rate constant, ***k*** using the expression below:

$(\frac{-k}{2.303} = slope)$ (*where **k**=Rate constant)* (include the units for ***k***).

$$Slope = \frac{Change\ in\ log10(volume\ of\ FA1)\ values}{change\ in\ time}$$

$$= \frac{(1.52-0.95)}{(25-86)s} = \frac{0.57}{-61s} = \textbf{–9.344x10}^{-3}\textbf{s}^{-1}$$

$But \frac{-k}{2.303} = slope$
$\therefore \frac{-k}{2.303} = -9.344x10^{-3}\ s^{-1}$ ✓
$k = 2.303 \times 9.344x10^{-3}\ s^{-1}$
$\boldsymbol{k = 2.15x10^{-2}s^{-1}}$ ✓

(ii) Using the graph plotted in **(a)(iii)** above, determine the volume of hydrogen peroxide, V_0, *at t=0. (Intercept on the vertical axis=*$log_{10} V_0$*)*

$log_{10} V_0 = 1.75$
$V_0 = 10^{1.75}$ ✓
$\boldsymbol{V_0 = 56.23cm^3}$ ✓

f) Basing on the graph you have plotted for **(a)(iii)** above, deduce the order of reaction with respect to hydrogen peroxideperoxide.

The reaction is first order with respect to hydrogen peroxide. ✓ *This is because a plot of* log_{10}*(volume of hydrogen peroxide) against time gives a straight line with a negative gradient.* ✓

Worked out Example 11.2.3
You are provided with the following:
HA1 which is sodium thiosulphate solution
HA2 which is hydrochloric acid
White piece of paper

You are required to determine theorder of reaction with respect to hydrochloric acidin its reaction with sodium thiosulphate.

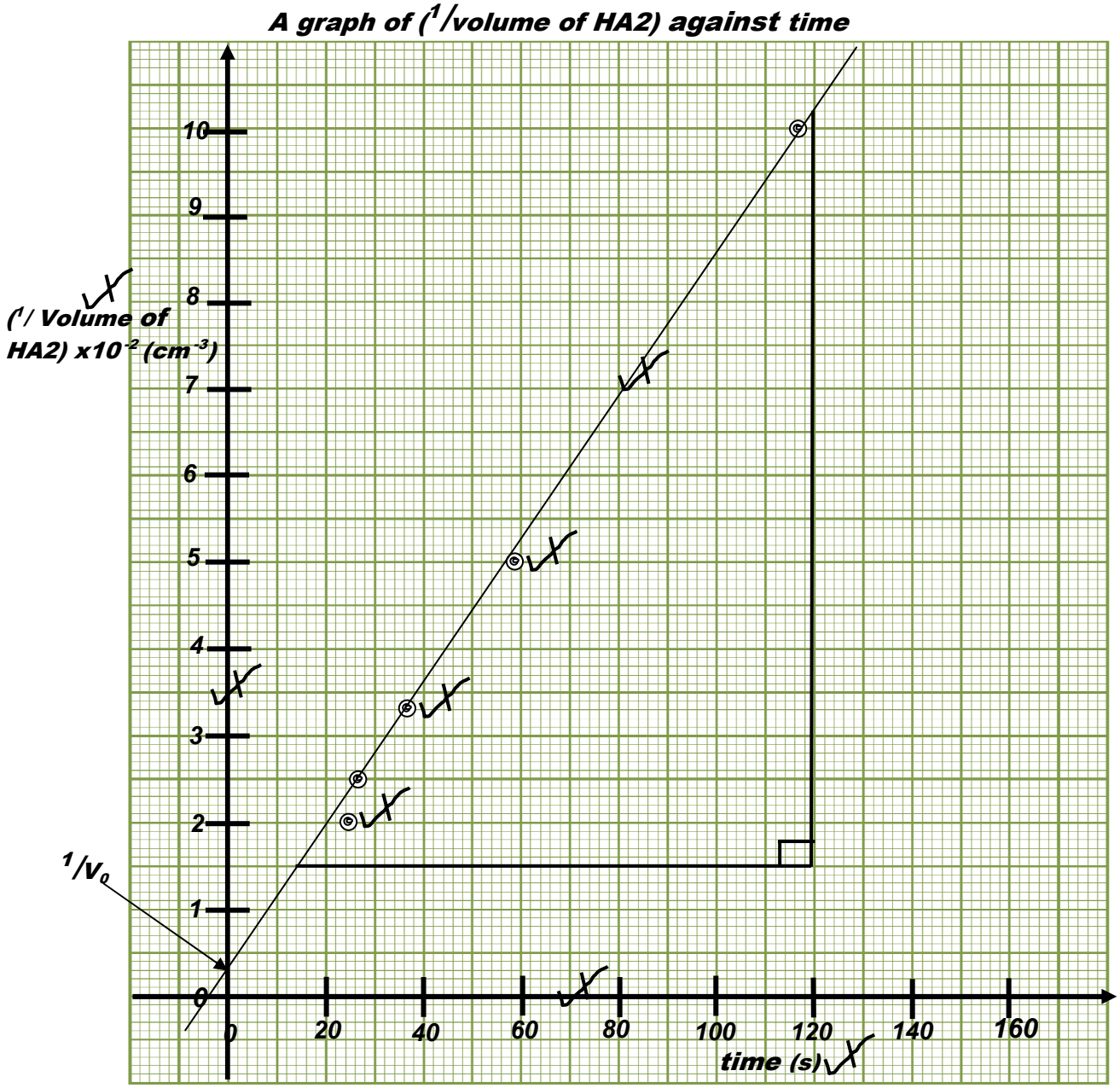
Theory
Hydrochloric acid reacts with sodium thiosulphate resulting in precipitation of sulphur. The precipitation of sulphur prevents the viewer from observing a cross or dot on the white sheet of paper.

Procedure
i) Mark a cross or dot on the white sheet of paper provided with blue or black ink.
ii) By use of a measuring cylinder, measure and transfer 50cm^3 of HA2 into a clean glass beaker followed by 10cm^3 of distilled water.
iii) Add 10cm^3 of HA1 and simultaneously start the stop clock.
iv) Gently shake the beaker for the solution to mix well and place the beaker on top of the white sheet of paper over the cross or dot.
v) Watch the cross through the solution carefully from above the beaker. Immediately stop the stop clock when the cross or dot disappears and note the time taken for the cross or dot to disappear.
vi) Pour away the reaction mixture and rinse the beaker thoroughly.
vii) Repeat procedures (i) to (vi) but this time adding 40, 30, 20 and 10cm^3 of HA2 to the beaker for the 2nd, 3rd, 4th and 5th experiments respectively.

Experiment number	1	2	3	4	5
Volume of HA2 (cm^3)	50	40	30	20	10
Volume of distilled water (cm^3)	10	20	30	40	50
Volume of HA1 (cm^3)	10	10	10	10	10
Time (s)	*24.0* ✓	*26.0* ✓	*36.0* ✓	*58.0* ✓	*116.0* ✓
^{1}/ volume of HA2 (cm^{-3})	*$2.00x10^{-2}$* ✓	*$2.50x10^{-2}$* ✓	*$3.33x10^{-2}$* ✓	*$5.00x10^{-2}$* ✓	*$10.0x10^{-2}$* ✓

Questions

a) Plot a graph of (^{1}/ volume of HA2) against time.



b) Basing on the graph plotted for (a) above, deduce the order of reaction with respect to hydrochloric acid.

The reaction is second order with respect to hydrochloric acid. This is because a plot of $^{1}/$ volume of HA2 ($^{1}/$ concentration of hydrochloric acid) against time gives a straight line with a positive gradient.

c) Determine the slope of the graph plotted for (a) above (indicate its units).

$$Slope = \frac{Change\ in\ (1/volume\ of\ HA2)\ values}{Change\ in\ time}$$

$$= \frac{(10.2-1.5)\ x\ 10-2\ cm3}{(120-14)s} = \frac{8.7x10-2}{106s} = \mathbf{8.208x10^{-4}\ cm^{-3}s^{-1}}$$

d) (i) Determine the value of the intercept on the vertical axis, $^{1}/V_0$(include its units).

$$\frac{1}{V0} = \mathbf{0.3x10^{-2}cm^{-3}} = \mathbf{3.0x10^{-3}cm^{-3}}$$

Note:

1)*Whenever you are required to determine the value of* ***anintercept on the vertical axis,*** *asyou plot the graph, the readings on the horizontal axis* ***must*** *start from* ***zero.***

2) *Incase* ***an intercept on the horizontal axis*** *is required, as you plot the graph, the readings on the vertical axis* ***must*** *start from* ***zero.***

3) *In case* ***no intercept is required,*** *then as you plot the graph, the readings on both the horizontal andvertical axis* ***do not necessarily*** *have to start from* ***zero.***

(ii) Usethe value of $^{1}/V_0$ obtained in (d) (i) above to determine the value of V_0.

(where V_0 = volume of hydrochloric acid at ***t=0****)*

$$\frac{1}{V0} = 3.0x10^{-3}cm^{-3}; V_0 = \frac{1}{0.003} V_0 = 333.33\ cm^3$$

Worked out Example 11.2.4

You are provided with the following:

FA1 which is 0.018M potassium manganate(VII) solution prepared using 2M sulphuric acid.

FA2 which is 0.045M oxalic acid

You are required to determine the activation energy of the reaction between potassium manganate(VII) and oxalic acid.

Theory

Oxalate ions from oxalic acid in FA2 react with manganate(VII) ions from acidified potassium manganate(VII) in FA1 according to the following equation.

$$2MnO_4^-(aq) + 16H^+(aq) + 5C_2O_4^{2-}(aq) \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) + 10CO_2(g)$$

When the reaction is complete, the purple manganate(VII) ions are reduced to very pale pink managanese(II) ions which appear colourless in dilute solution hence the purple solution turns colourless.

The activation energy, Ea of the reaction can be determined basing on the equation below:

$k = Ae^{-Ea/RT}$

Taking the natural logarithms,

$\ln k = \ln (Ae^{-Ea/RT})$ *{But In xy= (In x + In y)}*

$\ln k = \ln A + \ln e^{-Ea/RT}$ *{But In x^y = yIn x}*

$\ln k = \ln A + (-\frac{Ea}{RT})\ln e$ *{But Ine = 1}*

$\ln k = \ln A - \frac{Ea}{RT}$ *{But Inx = 2.303log$_{10}$x}*

$2.303\log_{10} k = 2.303\log_{10} A - \frac{Ea}{RT}$

Dividing through by 2.303, we have:

$\log_{10} k = \log_{10} A - \frac{Ea}{2.303RT}$

On rearranging the equation, we have:

$\log_{10} k = \log_{10} A - (\frac{Ea}{2.303R})\frac{1}{T}$

Where k is the Rate constant, A is the exponential constant, R is the molar gas constant ($R=8.314\ Jmol^{-1}K^{-1}$) while T is temperature in Kelvin.

Since k is the rate constant, it is directly proportional to the rate of reaction, which isalso directly proportional to$\frac{1}{t}$, hence instead of plotting $\log_{10}k$ against$\frac{1}{T}$, a graph of$\log_{10}(\frac{1}{t})$ can be plotted against$\frac{1}{T}$, whose slope is equal to$\frac{-Ea}{2.303R}$.

$\log_{10}(\frac{1}{t}) = \log_{10}A - (\frac{Ea}{2.303R})\frac{1}{T}$

Dividing through by -1 gives:

$-\log_{10}(\frac{1}{t}) = -\log_{10} A + (\frac{Ea}{2.303R})\frac{1}{T}$

{But-log$_{10}$ (^{1}/t)= -log$_{10}$ (t^{-1})=log$_{10}$ (t^{-1})$^{-1}$=log$_{10}$ t}

Therefore,

$\mathbf{\log_{10} t = -\log_{10} A + (\frac{Ea}{2.303R})\frac{1}{T}}$

Procedure

i) Use the thermometer provided to read and record the room temperature, $\mathbf{T_0}$ in the space provided in the table below.

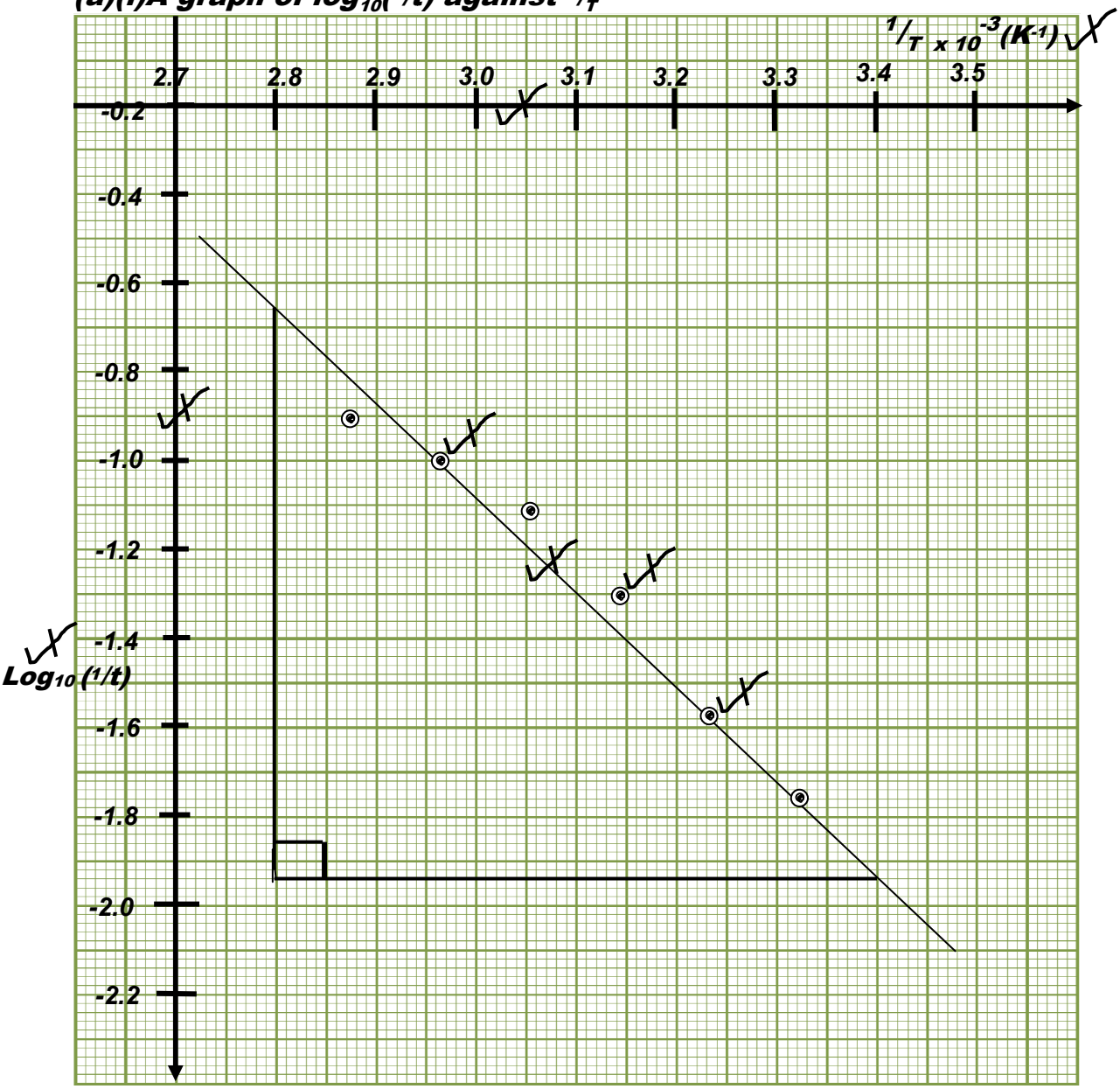
ii) Using a measuring cylinder, measure and transfer 30cm^3 of FA2 into a clean conical flask.

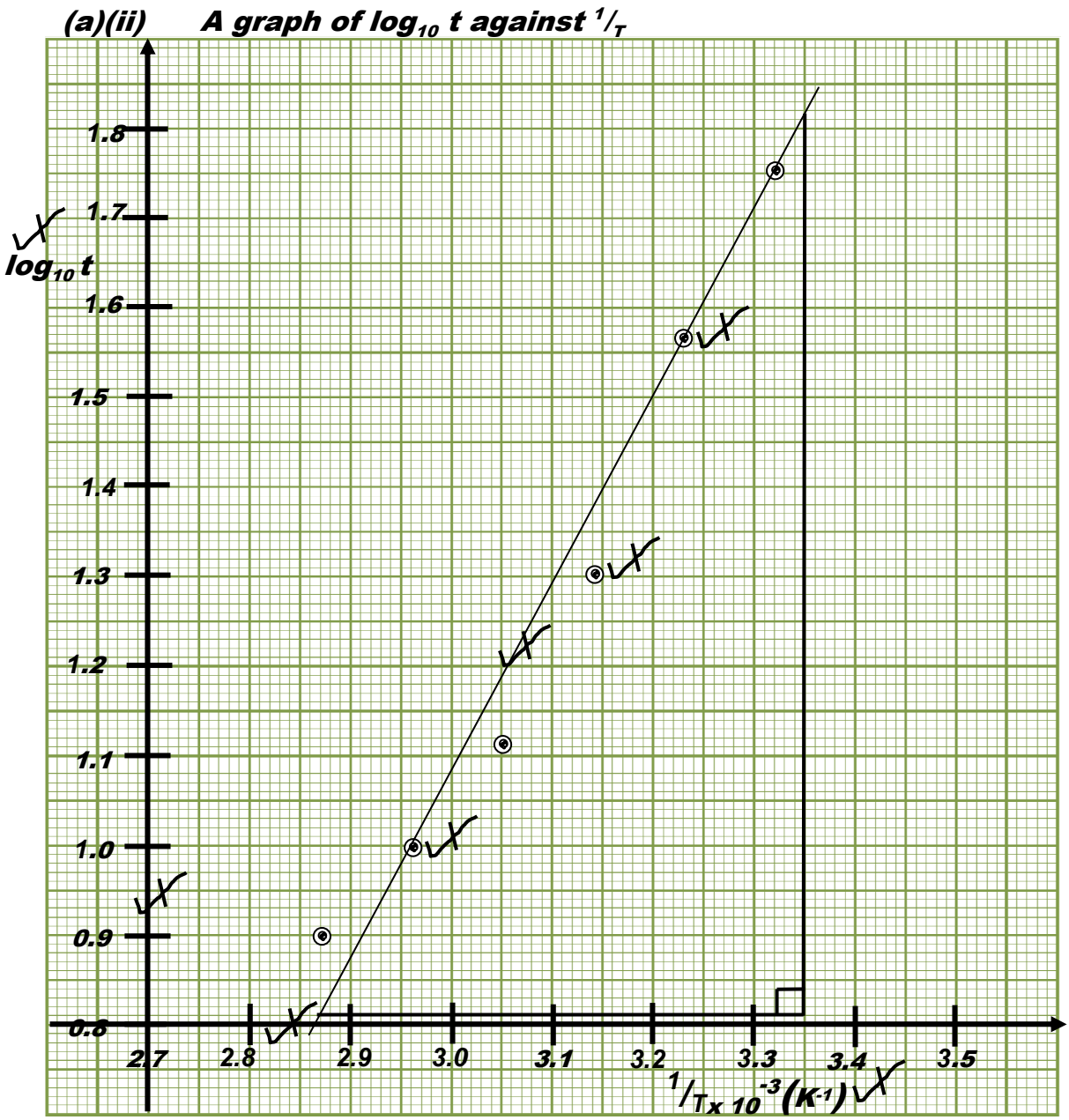
iii) By use of another measuring cylinder, measure $30cm^3$ of FA1 and keep the solution in the measuring cylinder.
iv) Heat solution FA2 in the glass beaker to a temperature of 75^0C.
v) Remove the hot FA2 solution in the glass beaker from the heat source and immediately add the $30cm^3$ of FA1 to the hot FA2 solution in the glass beaker and simultaneously start the stop clock.
vi) Keep shaking the solution gently once in a while and record the time taken for the the solution to turn from purple to faint pink.
vii) Repeat procedures (ii) to (vi) for temperatures of the FA2 solution equal to 65^0C, 55^0C, 45^0C, 35^0C and finally room temperature.
viii) Enter all your results in the table below.

Temperature (^0C)	75	65	55	45	35	$\mathbf{T_0 = 28}$
Time, **t** (s)	*8.0*	*10.0*	*13.0*	*20.0*	*37.0*	*57.0*
Rate, $(\frac{1}{t})(s^{-1})$	$12.5x10^{-2}$	$10.0x10^{-2}$	$7.69x10^{-2}$	$5.00x10^{-2}$	$2.70x10^{-2}$	$1.75x10^{-2}$
Log $_{10}(\frac{1}{t})$	*-0.903*	*-1.000*	*-1.114*	*-1.301*	*-1.569*	*-1.757*
Log $_{10}$t	*0.903*	*1.000*	*1.114*	*1.301*	*1.568*	*1.757*
Absolute temperature, **T** (K)	*348*	*338*	*328*	*318*	*310*	*301*
$(\frac{1}{T})$***x10^{-3}*** (K^{-1})	*2.87*	*2.96*	*3.05*	*3.14*	*3.23*	*3.32*

a) Plot a graph of:
(i) log $_{10}(\frac{1}{t})$ against $(\frac{1}{T})$.
(ii) $\log_{10}$ t against $(\frac{1}{T})$.

(a)(i)A graph of $\log_{10}(1/t)$ against $1/T$





b) Using the graph plotted in (a)(i) above, determine the activation energy, E_a of the reaction in **Jmol^{-1}** from the expression below:

Slope $= \frac{-Ea}{2.303R}$

$$Slope = \frac{Change\ in\ log10\left(\frac{1}{t}\right)values}{Change\ in\left(\frac{1}{T}\right)values}$$

$$= \frac{(-1.94- -0.66)}{(3.4-2.8)x10-3\ K-1} = \frac{(-1.94+0.66)}{(3.4-2.8)x10-3\ K-1} = \frac{-1.28}{0.6\ x\ 10-3\ K-1} = \textbf{-2133.33K}$$

$$Slope = \frac{-Ea}{2.303R}; -2133.33K = \frac{-Ea}{2.303\ x\ 8.314\ J\ mol-1\ K-1}$$

$-E_a$ = *-2133.33K x2.303x8.314Jmol^{-1}K^{-1}*

$$\boldsymbol{E_a = 40847.17Jmol^{-1}}$$

(c) Using the graph plotted in (a)(ii) above, determine the activation energy, E_a of the reaction inJmol^{-1} from the expression below:

Slope $= \frac{Ea}{2.303R}$

$$Slope = \frac{Change\ in\ log10\ t\ values}{Change\ in\left(\frac{1}{T}\right)values}$$

$$= \frac{(1.82- 0.81)}{(3.35-2.87)x10-3\ K-1} = \frac{1.01}{0.48x10-3\ K-1} = \textbf{2104.17K}$$

$$Slope = \frac{Ea}{2.303R}; 2104.17K = \frac{Ea}{2.303\ x\ 8.314\ J\ mol-1\ K-1}$$

E_a = *2104.17K x2.303x8.314Jmol^{-1}K^{-1}*

$$\boldsymbol{E_a = 40288.77\ Jmol^{-1}}$$

Worked out Example 11.2.5

You are provided with the following:

FA1 which is potassium manganate(VII) solution.

FA2 which is a solution of sodium oxalate prepared using dilute sulphuric acid.

You are required to determine the activation energy, E_a and the exponential constant, A for the reaction between potassium manganate(VII) and sodium oxalate.

Theory

Oxalate ions from the acidified sodium oxalate in FA2 react with manganate(VII) ions from potassium manganate(VII) in FA1 according to the following equation.

$2MnO_4^-(aq) + 16H^+(aq) + 5C_2O_4^{2-}(aq) \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) + 10CO_2(g)$

When the reaction is complete, the purple manganate(VII) ions are reduced to very pale pink managanese(II) ions which appear colourless in dilute solution hence the purple solution turns colourless.

The activation energy, E_a of the reaction is related to the rate constant, k, absolute temperature, T, exponential constant, A, and the molar gas constant, R as shown by the equation below:

$$\log_{10}k = \log_{10}A - \left(\frac{Ea}{2.303R}\right)\frac{1}{T}$$

Dividing through the equation above by -1 gives:

$$-\log_{10}k = -\log_{10}A + \left(\frac{Ea}{2.303R}\right)\frac{1}{T}$$

(Note:R=8.314 $Jmol^{-1}K^{-1}$**)**

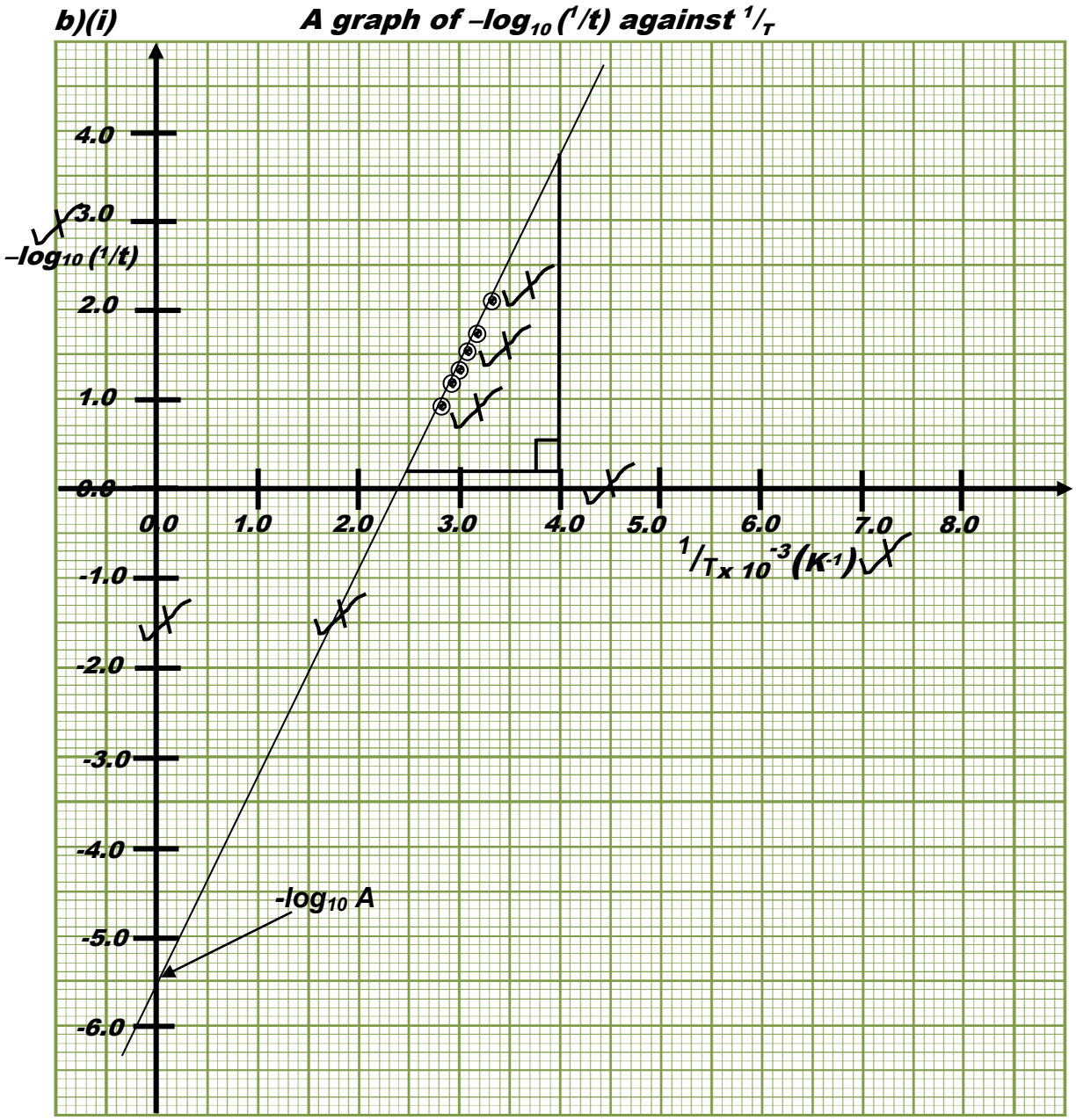
Since k is the rate constant, it is directly proportional to the rate of reaction, which is also directly proportional to $^1/t$, thus instead of plotting$\log_{10}k$ against$\frac{1}{T}$, a graph of $\log_{10}(\frac{1}{t})$ can be plotted against$\frac{1}{T}$, whose slope is equal to$\frac{-Ea}{2.303R}$and an intercept on the vertical axis equal to$\log_{10}A$.
Alternatively, a graph of $-\log_{10}(\frac{1}{t})$ can be plotted against$\frac{1}{T}$, whose slope is equal to $\frac{Ea}{2.303R}$and an intercept on the vertical axis equal to$-\log_{10}A$.

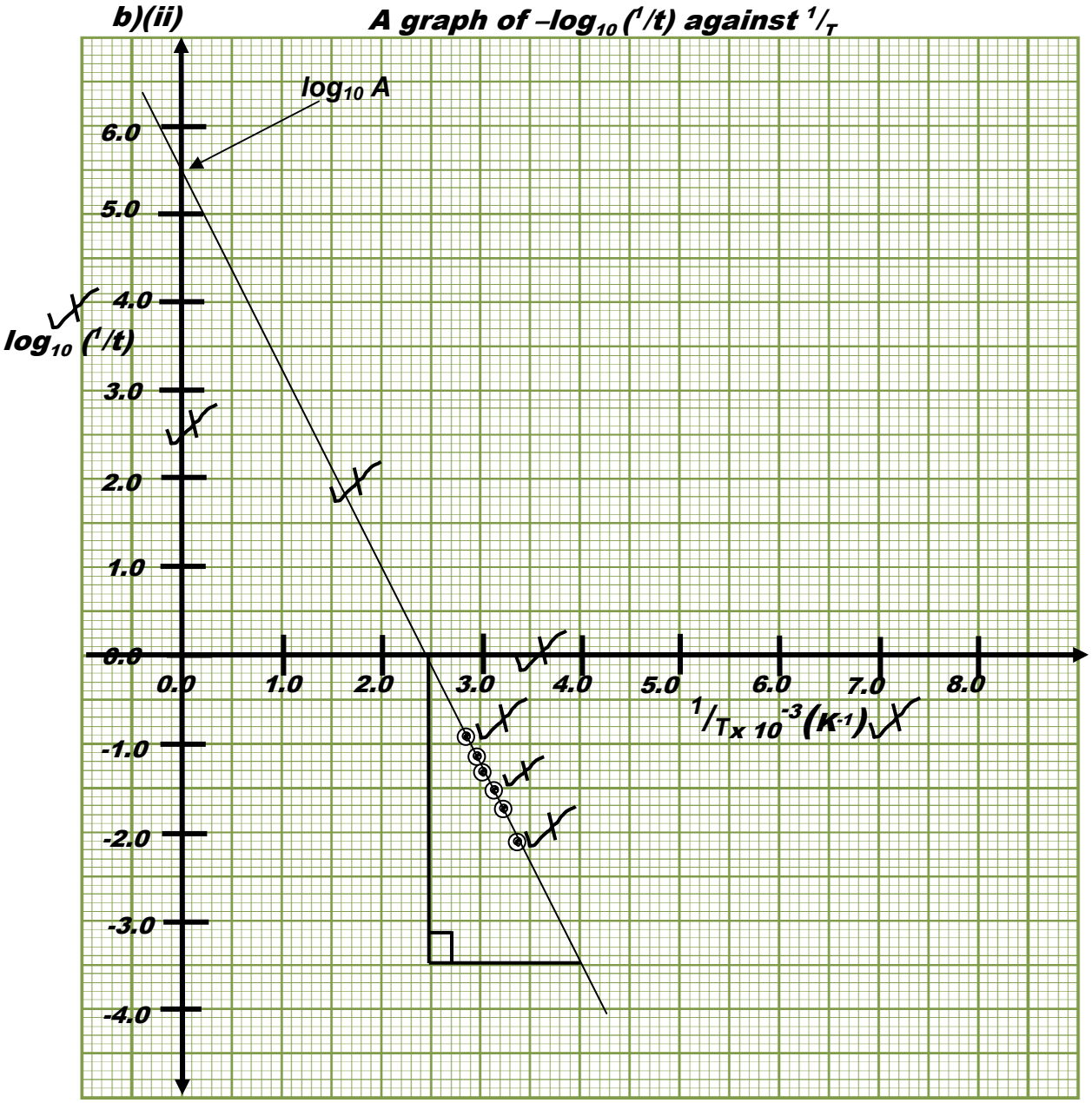
Procedure
(i) Using the thermometer provided, read and record the room temperature in the space in the table.
(ii) By use of a measuring cylinder, measure $20cm^3$ of FA2 into a clean conical flask.
(iii) To the solution in the conical flask, add $20cm^3$ of FA1 using another measuring cylinder and simultaneously start the stop clock.
(iv) Shake the contents of the conical flask and leave to stand.
(v) Note and record the time, t, taken for the solution to turn from purple to colourless.
(vi) Repeat procedures (ii) to (v), but this time, before FA1 is added, the FA2 solution is first heated to 40, 50, 60, 70 and 80^0C.
(vi) Record your results in the table below.

Temperature (^0C)	Room Temperature=***26***	40	50	60	70	80
Absolute temperature, **T** (K)	*299* ✓	*313* ✓	*323* ✓	*333* ✓	*343* ✓	*353* ✓
$(\frac{1}{T})$ (K^{-1})	3.34×10^{-3} ✓	3.19×10^{-3} ✓	3.09×10^{-3} ✓	3.00×10^{-3} ✓	2.92×10^{-3} ✓	2.83×10^{-3} ✓
Time, **t** (s)	*136.0* ✓	*56.0* ✓	*36.0* ✓	*22.0* ✓	*15.0* ✓	*9.0* ✓
Reciprocal of time, $(\frac{1}{t})$ (s^{-1})	7.35×10^{-3} ✓	17.8×10^{-3} ✓	27.7×10^{-3} ✓	45.4×10^{-3} ✓	66.6×10^{-3} ✓	111×10^{-3} ✓
$-\log_{10}(\frac{1}{t})$	*2.134* ✓	*1.748* ✓	*1.556* ✓	*1.342* ✓	*1.176* ✓	*0.954* ✓
$\log_{10}(\frac{1}{t})$	*-2.134* ✓	*-1.748* ✓	*-1.556* ✓	*-1.342* ✓	*-1.176* ✓	*-0.954* ✓

Questions
(a) Determine the absolute temperature, T from the expression:
$T = (^0C+273)$

(b)Plot graphs of:
(i)$-\log_{10}(\frac{1}{t})$ against$\frac{1}{T}$.
(ii) $\log_{10}(\frac{1}{t})$ against$\frac{1}{T}$


b)(i)
A graph of –log10 (1/t) against 1/T
4.0
3.0
–log10 (1/t)
2.0
1.0
0.0
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
1/T x 10-3 (K-1)
-1.0
-2.0
-3.0
-4.0
-log10 A
-5.0
-6.0



(c) Using the graph you have plotted in (b)(i), determine the:

(i) activation energy, E_a, of the reaction in **kJmol^{-1}** from the expression below:

$$\text{Slope} = \frac{Ea}{2.303R}$$

$$Slope = \frac{Change\ in - log10\ (\frac{1}{t})\ values}{Change\ in \left(\frac{1}{T}\right) values}$$

$$= \frac{(0.2 - 3.7)}{(2.5-4.0)x10-3\ K-1} = \frac{-3.5}{-1.5x10-3\ K-1} = \mathbf{2333.33K}$$

$$Slope = \frac{Ea}{2.303R}; 2333.33K = \frac{Ea}{2.303\ x\ 8.314\ J\ mol-1\ K-1}$$

E_a = 2333.33K x2.303x8.314Jmol^{-1}K^{-1}

= 44676.601 J mol^{-1}

$$= \frac{44676.601\ J\ mol-1}{1000} = \mathbf{44.677\ kJ\ mol^{-1}}$$

(ii) value of theexponential constant**,** **A**. *(Intercept on the vertical axis = -log_{10} A).*

-log_{10} A=-5.5

$log_{10}(^1/A)$ = -5.5

$10^{-5.5} = \frac{1}{A}$; $10^{-5.5}A = 1$; $A = (10^{-5.5})^{-1}$; $A = 10^{5.5}$; **$A = 3.16x10^5$**

d) Using the graph you have plotted in (b)(ii), determine the:

(i) activation energy, E_a, of the reaction in **kJmol^{-1}**from the expression below:

$$\text{Slope} = \frac{Ea}{2.303R}$$

$$Slope = \frac{Change\ in\ log10\ (\frac{1}{t})\ values}{Change\ in (\frac{1}{T}) values}$$

$$= \frac{(0.0 - 3.5)}{(2.5-4.0)x10-3\ K-1} = \frac{-3.5}{-1.5x10-3\ K-1} = \mathbf{2333.33K}$$

$$Slope = \frac{Ea}{2.303R}; 2333.33K = \frac{Ea}{2.303\ x\ 8.314\ J\ mol-1\ K-1}$$

E_a = 2333.33K x2.303 x 8.314 Jmol^{-1}K^{-1}

= 44676.601 J mol^{-1}

$$= \frac{44676.601\ J\ mol-1}{1000} = \mathbf{44.677\ kJ\ mol^{-1}}$$

(ii) value of theexponential constant**,** **A**. *(Intercept on the vertical axis =log_{10} A)*

log_{10} A=5.5

$A = 10^{5.5}$; **$A = 3.16x10^5$**

Reminder:

1) *Whenever you are required to determine the value of* ***anintercept on the vertical axis,*** *as you plot the graph, the readings on the horizontal axis* ***must*** *start from* ***zero.***

2) *Incase* ***an intercept on the horizontal axis*** *is required, as you plot the graph, the readings on the vertical axis* ***must*** *start from* ***zero.***

3) *In case* ***no intercept is required,*** *then as you plot the graph, the readings on both the horizontal and vertical axis* ***do not necessarily*** *have to start from* ***zero.***

11.3 Practical Exercises on Chemical Kinetics

Experiment 11.3.1
You are provided with the following:
FA1 which isiodine solution.
FA2 which is 0.4M propanone solution.
FA3 which is 0.01M sodium thiosulphate solution.
FA4 which is 0.8M sodium hydrogencarbonate solution.
1M sulphuric acid.
Starch solution

You are required to determine the:
i) order of reaction with respect to iodine in its reaction with propanone in the presence of an acid catalyst.
ii) rate constant for the reaction.

Theory
Aqueous Iodine reacts with propanone in the presence of an acid catalyst to form a compound known as1-iodopropanone.
During the progress of the reaction, when sodium hydrogencarbonate is added to the reaction mixture, the acid catalyst is neutralized and the reaction stops immediately so that some iodine remains unreacted. A portion of the reaction mixture containing the unreacted iodine is pipetted and titrated against standard sodium thiosulphate solution. The volume of sodium thiosulphate used is directly proportional to the concentration of iodine in the pipetted volume of the reaction mixture at any given time. Hence instead of plotting concentration of iodine, volume of sodium thiosulphate can be plotted against time (alternatively, the reciprocal of time can be plotted against volume of sodium thiosulphate).
Note: *In order to focus on iodine as the reactant of interest, we shall assume that the propanone and the acid are in large excess such that their concentration will be assumed to have no effect on the rate of reaction.*

Procedure
i) Using a measuring cylinder, transfer 20cm^3 of **FA1** into a conical flask. Add 20 cm^3 of 1M sulphuric acid followed by 40cm^3 of **FA2**, and at the same time, start the stop clock. Shake the mixture gently and label it **FA5**.
ii) Using another measuring cylinder, transfer 10cm^3 of **FA4** into another conical flask. At the end of **5 minutes,** pipette 10cm^3 of **FA5** into the conical flask containing the 10cm^3 of **FA4 (MAKE SURE YOU DO NOT STOP THE STOP CLOCK).** Shake the mixture until no more effervescence occurs. Add 1cm^3 of starch solution and titrate the mixture with **FA3** from the burette.
iii) Record your results in the table below.
iv) Repeat procedure (ii) above after every **5 minutes**(i.e. at the end of 10, 15, 20, 25, 30 and 35minutes) as shown in the table below.

Volume of pipette used……..cm^3

Time from the start of the reaction (min)	5	10	15	20	25	30	35
Final burette reading (cm^3)							
Initial burette reading (cm^3)							
Volume of FA3 used (cm^3)							

Questions

a)Plot a graph of volume of FA3 against time.

b) Using the graph you have plotted in (a) above:

(i) deduce the order of reaction with respect to iodine.

(include a reason for the order of reaction you have stated)

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(ii) determine the volume of FA4 at t = 0 minutes.

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(iii) determine the value of the rate constant, *k* and **include its units**(the slope for the graph you have plotted in (a) above is given by: ***slope =— k***).

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c) Using the answer you have obtained in (b)(ii) above, determine the molar concentration of the iodine solution in FA1.

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Experiment 11.3.2

You are provided with the following:
GA1 which is 0.1M hydrogen peroxide solution
GA2 which is 0.06M sodium thiosulphate
GA3 which 0.1M potassium iodide solution acidified with sulphuric acid
Starch solution
You are required to determine thevalue of the rate constant and the order of reaction with respect to hydrogren peroxide.

Theory

Hydrogen peroxide reacts with acidified potassium iodide solution to liberate aqueous iodine.
The liberated iodine then reacts with thiosulphate ions according to the equation below.
$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$
Since the iodine is liberated in excess, once all the thiosulphate ions available have reacted with iodine, the unreacted iodine then combines with the starch solution in the reaction mixture to form the blue-black starch-iodine complex. Hence appearance of the blue colour in the reaction mixture is an indicates that all the sodium thiosulphate has reacted with iodine (the reaction between the two is complete).

Procedure

(i) Using ameasuring cylinder, transfer 25cm^3 of GA3 into a conical flask.
(ii) Add 15cm^3 of GA2 from the burette followed by addition of 25cm^3 of distilled water and then 2cm^3 of starch indicator solution using suitable measuring cylinders. Shake gently to mix.
(iii) To the mixture in part (ii) above, add at once 10cm^3 of GA1 and simultaneously start the stop clock. Shake the mixture strongly and continuosly until the blue colour appears in the solution. Note and record the time taken for the blue colour to appear in the solution. Record your results in the table below.
(iv) Repeat procedures (i) to (iii), while adding the volumes of GA1 shown in the table below.

Experiment number	1	2	3	4	5
Volume of GA3 (cm^3)	25	25	25	25	25
Volume of GA2 (cm^3)	15	15	15	15	15
Volume of distilled water (cm^3)	25	20	15	10	5
Volume of starch solution (cm^3)	2	2	2	2	2
Volume of GA1 (cm^3)	10	15	20	25	30
$\log_{10}$ (volume of GA1)					
Time(s)					
Reciprocal of time, $\frac{1}{t}$(s^{-1})					

Questions

a) Plot a graph of:

i) volume of GA1 against time.

ii) $\frac{1}{t}$against volume of GA1.

iii) $\log_{10}$ (volume of GA1) against time.

b) Basing on the graph plotted for (a)(i) below:

(i) deduce the order of reaction with respect to hydrogen peroxide.

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(ii) determine the rate constant, *k* by using the half life of the decomposition of hydrogen peroxide.

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b)(i) Determine the slope of the graph plotted for part (a)(ii) above (include its units).

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(ii) Basing on the graph you have plotted for part (a)(ii) above, deduce the order of reaction with respect to hydrogen peroxideperoxide.

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d) (i) Calculate the slope for the graph plotted for part**(a)(iii)** above and use it to determine the value of the rate constant, ***k*** using the expression below:
($^{-k}/_{2.303}$= slope) (*where **k**=Rate constant)* (include the units for ***k***).

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(ii) Using the graph plotted for part**(a)(iii)** above, determine the volume of hydrogen peroxide, V_0, *at t=0. (Intercept on the vertical axis =*$log_{10} V_0$*)*

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e) Basing on the graph you have plotted for **(a)(iii)** above, deduce the order of reaction with respect to hydrogen peroxide.

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Experiment 11.3.3
You are provided with the following:
HA1 which ishydrogen peroxide solution
HA2 which is 0.02M potasssium manganate(VII) solution
HA3 which is1M iron(III) chloride solution.
2M sodium hydroxide solution
1M sulphuric acid

You are required to determine the:
(i) molar concentration of the hydrogen peroxide solution in HA1.
(ii) half life and rate constant for the decomposition of hydrogen peroxide.
(iii) order of reaction with respect to hydrogen peroxide in its catalytic decomposition reaction.

Theory

The decomposition of hydrogen peroxide is catalysed by iron(III) ions provided byiron(III) chloridesolution.

$2H_2O_2(aq) \quad FeCl_3 \quad 2H_2O(l) + O_2(g)$

Sodium hydroxide solution is added to neutralise the acidity of iron(III) chloride while the reaction is quenched/stopped by addition of the acid (sulphuric acid).

After a given period of time, t, the hydrogen peroxide that remains undecomposed, can be titrated against acidified potassium manganate(VII) solution as shown by the equation below:

$2MnO_4^-(aq) + 6H^+(aq) + 5H_2O_2(aq) \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) + 5O_2(g)$

The volume of potassium manganate(VII) solution required to reach the end point is directly proportional to the amount of undecomposed hydrogen peroxide left after any given period of time, t.

Procedure I

(i) By use of a measuring cylinder, transfer 25cm^3 of HA1 into a 250cm^3 volumetric flask and make up to the mark with distilled water. Label this solution HA4.

(ii) Pipette 10.0cm^3 of HA4 into a conical flask. Add 25cm^3 of 1M sulphuric acid and titrate with HA2 from the burette until the solution in the conical flask just faint turns pink. Repeat the titration until you obtain consistent titre values. Record your results in the table below.

Volume of pipette used……..cm^3

Table I

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of HA2 used (cm^3)			

Values used to calculate average……..……..……cm^3

Average volume of HA2 used =…….……cm^3

(a) Determine the molar concentration of hydrogen peroxide in HA1.

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Procedure II

(i) Pipette 10.0cm^3 of HA4 into a conical flask. Add **4 drops of 2M sodium hydroxide solution** followed by **4 drops of HA3** and immediately start the stop clock.Occasionally, shake the mixture gently.
(ii) At the end of **4 minutes,** add 25cm^3 of **1M sulphuric acid**(the acid stops further decomposition of hydrogen peroxide) and titrate the mixture with **HA2** from the burette until the solution just turns faint pink.
iii) Record your results in the table below.
iv) Repeat procedures (i) to (iii) above after every **4 minutes**(i.e. at the end of 8, 12, 16 and 20minutes) as shown in the table below.

Time taken from addition of HA3 and addition of 1M sulphuric acid (minutes)	0	4	8	12	16	20
Final burette reading (cm^3)						
Initial burette reading (cm^3)						
Volume of **HA2** used (cm^3)						
Log_{10} (Volume of HA2 used)						

Questions
b)Plot a graph of log_{10} (Volume of HA2 used) against time.
c) Using the graph you have plotted in (b) above:
(i) determine the half lifeforthe decomposition of hydrogen peroxide.

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(ii) rate constant for the decomposition of hydrogen peroxide.

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(ii) deduce the order of reaction with respect to hydrogen peroxide in its catalytic decomposition reaction. (Include a reason for the order of reaction you have stated).

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Experiment 11.3.4

You are provided with the following:
FA1 which is hydrochloric acid
FA2 which is sodium thiosulphate solution
White piece of paper
You are required to determine theorder of reaction with respect to hydrochloric acidin its reaction with sodium thiosulphate.

Theory

The reaction between hydrochloric acid and sodium thiosulphate results in precipitation of sulphur. The precipitation of sulphur prevents the viewer from observing a cross or dot on the white sheet of paper provided.

Procedure

i) Mark a cross or dot on the white sheet of paper provided with blue or black ink.By use of a measuring cylinder, measure and transfer $50cm^3$ of FA1 into a clean glass beaker.Add $10cm^3$ of FA2 and simultaneously start the stop clock. Gently shake the beaker for the solution to mix well and place the beaker on top of the white sheet of paper over the cross or dot.
ii) Watch the cross through the solution carefully from above the beaker. Immediately stop the stop clock when the cross or dot disappears and note the time taken for the cross or dot to disappear.
iii) Record your results in the table below.
iv) Pour away the reaction mixture and rinse the beaker thoroughly.
v) Repeat procedures (i) to (iv) but this time adding 40, 30, 20 and $10cm^3$ of FA1 to the beaker for the 2^{nd}, 3^{rd}, 4^{th} and 5^{th} experiments respectively.

Experiment number	1	2	3	4	5
Volume of FA1 (cm^3)	50	40	30	20	10
Volume of distilled water (cm^3)	10	20	30	40	50
Volume of FA2 (cm^3)	10	10	10	10	10
Time (s)					
^{1}/ volume of FA1 (cm^{-3})					

Questions

a) Plot a graph of (^{1}/ volume of FA1) against time.
b) Basing on the graph plotted for (a), deduce the order of reaction with respect to hydrochloric acid.

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c) Determine the slope of the graph plotted in (a) above (indicate its units).

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d) Determine the value of the intercept on the vertical axis, $^{1}/_{V_0}$ and use it to determine the value of V_0 *(where* V_0 *= volume of hydrochloric acid at t=0)*

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Experiment 11.3.5

You are provided with the following:

FA1 which is 0.02M potassium manganate(VII) solution prepared using 2M sulphuric acid

FA2 which is 0.05M oxalic acid

You are required to determine the activation energy, E_a for the reaction between oxalic acid and potassium manganate(VII).

Theory

Oxalate ions from oxalic acid in FA2 react with manganate(VII) ions from acidified potassium manganate(VII) in FA1 according to the following equation.

$2MnO_4^-(aq) + 16H^+(aq) + 5C_2O_4^{2-}(aq) \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) + 10CO_2(g)$

When the reaction is complete, the purple manganate(VII) ions are reduced to very pale pink managanese(II) ions which appear colourless in dilute solution hence the purple solution turns colourless.

The activation energy, Ea can be determined basing on the equation below:

$k = Ae^{-Ea/RT}$

Taking the natural logarithms,

$\ln k = \ln (Ae^{-Ea/RT})$ *{But In xy= (In x + In y)}*

$\ln k = \ln A + \ln e^{-Ea/RT}$ *{But In* x^y *= yIn x}*

$\ln k = \ln A + (-\frac{Ea}{RT}) \ln e$ *{But Ine = 1}*

$\ln k = \ln A - \frac{Ea}{RT}$ *{But* $\ln x = 2.303\log_{10} x$*}*

$2.303\log_{10} k = 2.303\log_{10} A - \frac{Ea}{RT}$

Dividing through by 2.303, we have:

$\log_{10} k = \log_{10} A - \frac{Ea}{2.303RT}$

On rearranging the equation, we have:

$\log_{10} k = \log_{10} A - (\frac{Ea}{2.303R})\frac{1}{T}$

Dividing through by -1 gives:

$-\log_{10} k = -\log_{10} A + (\frac{Ea}{2.303R})\frac{1}{T}$

Where k is the Rate constant, A is the exponential constant, R is the molar gas constant ($R=8.314\ Jmol^{-1}K^{-1}$) while T is temperature in Kelvin.

Since k is the rate constant, it is directly proportional to the rate of reaction, which is also directly proportional to $(\frac{1}{t})$, hence instead of plotting $-\log_{10}k$ against $\frac{1}{T}$, a graph of $-\log_{10}(\frac{1}{t})$ can be plotted against $\frac{1}{T}$, whose slope is equal to $\frac{Ea}{2.303R}$.

$-\log_{10}(\frac{1}{t}) = -\log_{10} A + (\frac{Ea}{2.303R})\frac{1}{T}$

Dividing through by -1 gives:

$\log_{10}(\frac{1}{t}) = \log_{10} A - (\frac{Ea}{2.303R})\frac{1}{T}$

Procedure

i) Use the thermometer provided to read and record the room temperature, $\mathbf{T_0}$ in the space provided in the table below.
ii) Using a measuring cylinder, measure and transfer $20cm^3$ of FA2 into a clean glass beaker.
iii) By use of another measuring cylinder, measure $20cm^3$ of FA1 and keep the solution in that measuring cylinder.
iv) Heat solution FA2 in the glass beaker to a temperature of 65^0C.
v) Remove the hot FA2 solution in the glass beaker from the heat source and immediately add the $20cm^3$ of FA1 to the hot FA2 solution in the glass beaker and simultaneously start the stop clock.
vi) Keep shaking the solution gently once in a while and record the time taken for the the solution to turn from purple to colourless.
vii) Repeat procedures (ii) to (vi) for temperatures of the FA2 solution equal 55^0C, 45^0C, 35^0C and finally room temperature, T_0.
viii) Enter all your results in the table below.

Temperature (^{0}C)	65	55	45	35	T_0=
Time, **t** (s)					
Rate, $\frac{1}{t}$(s^{-1})					
Log ($\frac{1}{t}$)					
-Log ($\frac{1}{t}$)					
Absolute temperature, T (K)					
($\frac{1}{T}$)(K^{-1})					

a) Plot a graph of -log ($\frac{1}{t}$)against $\frac{1}{T}$.

b) By use of the graph plotted in (a) above, determine the:

i) slope of the graph (indicate its units).

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ii) activation energy, E_a of the reaction from the expression below.

$Slope = \frac{Ea}{2.303R}$

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c) Plot a graph of log $(\frac{1}{t})$ against $\frac{1}{T}$.

d) By use of the graph plotted forpart (c), determine the:

i) slope of the graph (indicate its units).

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ii) activation energy, E_a of the reaction from the expression below.

$Slope = \frac{-Ea}{2.303R}$

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Experiment 11.3.6
You are provided with the following:
FA1 which ispotassium manganate(VII) solution
FA2 which is a solution of sodium oxalate acidified using dilute sulphuric acid.
You are required to determine the:
(i) volume, V of FA1 which reacts completely with 25cm^3 of FA2.
(ii) activation energy, Ea for the reaction between sodium oxalate and potassium manganate(VII).
(iii) exponential constant, A for the reaction.

Theory
Oxalate ions from the acidified sodium oxalate in FA2 react with manganate(VII) ions from potassium manganate(VII) in FA1 according to the following equation.

$$2MnO_4^-(aq) + 16H^+(aq) + 5C_2O_4^{2-}(aq) \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) + 10CO_2(g)$$

When the reaction is complete, the purplesolution turns colourless.
The activation energy, Ea can be determined basing on the equation below:

$$log_{10}k = log_{10}A - \frac{Ea}{2.303RT}$$

Where **k** is the Rate constant, A is the exponential constant, R is the molar gas constant (R=8.314 Jmol^{-1}K^{-1}), T is temperature in Kelvin.

On rearranging the equation above, we have:

$$log_{10}k = log_{10}A - (\frac{Ea}{2.303R})\frac{1}{T}$$

Dividing through by -1 gives:

$$-log_{10}k = -log_{10}A + (\frac{Ea}{2.303R})\frac{1}{T}$$

Since k is the rate constant, it is directly proportional to the rate of reaction, which is also directly proportional to $\frac{1}{t}$, hence instead of plotting $-\log_{10}k$ against$\frac{1}{T}$, a graph of$-\log_{10}(\frac{1}{t})$ can be plotted against$\frac{1}{T}$, whose slope is equal to $\frac{Ea}{2.303R}$ and intercept on the vertical axis of $-\log_{10}A$ as shown by the equation below.

$$-log_{10}(\frac{1}{t}) = -log_{10}A + (\frac{Ea}{2.303R})\frac{1}{T}$$

{Note:-**$log_{10}(1/t) = log_{10}t$**}

Therefore,

$$\boldsymbol{log_{10}t = -log_{10}A\left(\frac{Ea}{2.303R}\right)\frac{1}{T}}$$

Procedure I
(i) Using a measuring cylinder, transfer 25cm^3 of FA2 into a clean conical flask.
(ii) Heat the FA2 solution in the conical flask to a temperature of 80^{0}C and titrate the hot solution immediately with FA1 from the burette until the end point is reached.
(iii) Repeat procedures (i) to (ii) until you obtain consistent results.Record your results in table 1 below.

Volume of FA2 used……..……..cm^3

Table I

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA1 used (cm^3)			

Values used to calculate average……..……cm^3

Average volume of FA1 used, V=……cm^3

Procedure II

(i) Use the thermometer provided to read and record the room temperature, $\mathbf{T_R}$ in the space provided in the table below.
(ii) Run 25cm^3 of FA2 from the burette into a clean conical flask and heat the solution to a temperature, p=80^{0}C.
(iii)By use of a measuring cylinder, add Vcm^3 of FA1 to the hot solution in the conical flask and simultaneously start the stopclock (or stop watch).
(iv) Shake the contents of the conical flask and once to mix and leave to stand.
(v) Note and record the time, t, taken for the solution to turn colourless.
(vi) Repeat procedures (ii) to (v) for the values of temperature, p=70, 60, 50, 40^{0}C and room temperature, T_R.
(vii) Record your results in table II below.

Table II

Temperature, P (^{0}C)	80	70	60	50	40	Room temperature, T_R =……
Absolute temperature, T(K)						
$\left(\frac{1}{T}\right)$ (K^{-1})						
Time, **t** (s)						
Log_{10} t						

a) Complete the table above table by filling in the following:
   (i) Absolute temperature, T using the formula: T=(p+273).
   (ii) Reciprocal of T.
   (iii) Log_{10} t.

b) Plot a graph of log_{10} t against $\left(\frac{1}{T}\right)$.

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c) By use of the graph plotted for part (b) above, determine the:
i) slope of the graph (indicate its units).

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ii) activation energy, E_a of the reaction in **kJmol^{-1}** from the expression below.

$Slope = \frac{Ea}{2.303R}$

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CHAPTER TWELVE
12.0 COLLIGATIVE PROPERTIES

12.1 Introduction
A Colligative property is a physical property of a dilute solution which depends on the number of moles of a non-volatile solute dissolved in a fixed mass of a solvent but is independent of its chemical nature.

Examples of colligative properties include:
- Relative lowering of vapour pressure.
- Elevation in boiling point.
- Depression in freezing point.
- Osmotic pressure.

Colligative properties are used in determination of relative formula masses of non-volatile solutes. Nevertheless,using colligative properties to determine relative formula masses of solutes has the following limitations:

i) The solution should be dilute.
ii) The solute particles should not dissociate within the solvent.
iii) The solute particles should not associate within the solvent.
iv) The solute particles should not react with the solvent particles.
v) The solute should be non-volatile.

12.2 Worked Out Examples on Colligative Properties

Worked Out Example 12.2.1
You are provided with the following:
Compound**X**
Compound**Y**
You are required to determine the relative formula mass of Compound Y using the depression in freezing point method.

Procedure
i) Weigh accurately 6.2g of substance X into a clean boiling tube.
ii) Immerse the boiling tube containing substance X into a beaker of boiling water and heat until substance X melts.
iii) Insert the thermometer into liquid X and continue heating to 95^0C.
iv) Remove the boiling tube from the boiling water in order to allow it to cool in air and immediately start the stop clock (or stop watch).
v) Read and record the temperature of the liquid every after one minute and record the results in the table below.
vi) Weigh accurately 1.0g of substance Y and add it to the boiling tube containing substance A.
vii) Immerse the mixture into the beaker containing boiling water until the mixture melts. Continue heating until the molten mixture is at 95^0C.

viii) Remove the boiling tube from the boiling water to allow the mixture to cool in air and immediately start the stop clock(or stop watch).
ix) Read and record the temperature of the mixture every after one minute and record the results in the table below.

RESULTS

Mass of beaker + substance X……...*46.2*……....g

Mass of empty beaker……*39.8*……g

Mass of substance X……*6.4*……g

Mass of beaker + substance Y……*40.8*……...g

Mass of empty beaker……*39.8*…….....g

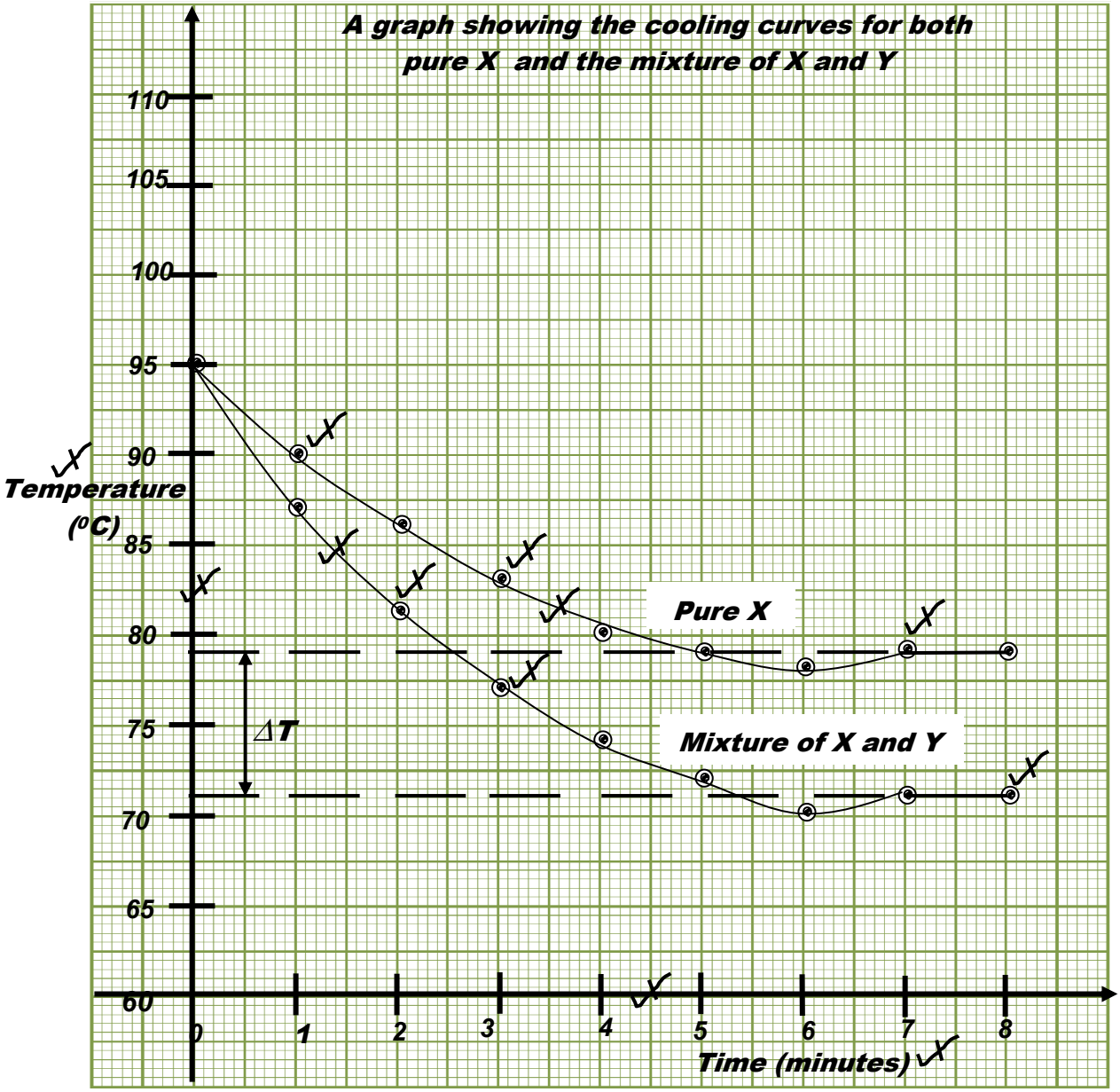
Mass of substance Y……*1.0*……....g

TABLE OF RESULTS

Time (minutes)	0	1	2	3	4	5	6	7	8
Temperature of pure X (^{0}C)	*95.0*	*90.0*	*86.0*	*83.0*	*80.0*	*79.0*	*78.0*	*79.0*	*79.0*
Temperature of mixture of X and Y(^{0}C)	*95.0*	*87.0*	*81.0*	*77.0*	*74.0*	*72.0*	*70.0*	*71.0*	*71.0*

Question

a) Plot on the same axes, the cooling curves of both pure X and the mixture of X and Y.



a) By use of the graph you have plotted in (a) above, determine the:

i) freezing point of pure substance X.

……*79.0^{0}C*……

ii) freezing point of the mixture of substances X and Y.

……*71.0^{0}C*……

iii) depression in freezing point of substance X caused by 1.0g of substance Y.

……$\Delta T=(79.0-71.0)^0C$……..

……*= 8.0^{0}C*……....

b) Relative formula mass of substance Y.
(freezing point constant of substance X is $6.94^0Cmol^{-1}kg^{-1}$)

6.2g of substance X dissolve 1.0g of substance Y
1000gof substance X dissolve $\left(\frac{1.0}{6.2} x\ 1000\right)g$ *of substance Y.*
=161.29g of substance Y

8^0C is the depression in freezing point caused by 161.29g of Y.
6.94^0C is the depression in freezing point caused by: $\left(\frac{161.29}{8} x\ 6.94\right)g$ *of substance Y.*
=139.92 g of substance Y
*Relative formula mass of substance Y=**139.92***

12.3 Practical Exercises on Colligative Properties

Experiment 12.3.1
You are provided with the following:
Substance **A**
Substance **B**
You are required to determine the relative formula mass of substance B using the depression in freezing point method.

Procedure
i) Weigh accurately 6.6g of substance A into a clean boiling tube.
ii) Immerse the boiling tube containing substance A into a beaker of boiling water and heat until substance A melts.
iii) Insert the thermometer into liquid A and continue heating to 95^0C.
iv) Remove the boiling tube from the boiling water in order to allow it to cool in air and immediately start the stop clock (or stop watch).
v) Read and record the temperature of the liquid every after one minute and record the results in the table below.
vi) Weigh accurately 1.1g of substance B and add it to the boiling tube containing substance A.
vii) Immerse the mixture into the beaker containing boiling water until the mixture melts. Continue heating until the molten mixture is at 95^0C.
viii) Remove the boiling tube from the boiling water to allow the mixture to cool in air and immediately start the stop clock(or stop watch).
ix) Read and record the temperature of the mixture every after one minute and record the results in the table below.

RESULTS

Mass of beaker + substance A……..g

Mass of empty beaker……...g

Mass of substance A…….g

Mass of beaker + substance B……..g

Mass of empty beaker……...g

Mass of substance B…….g

TABLE OF RESULTS

Time (minutes)	0	1	2	3	4	5	6	7	8
Temperature of pure A (^{0}C)									
Temperature of mixture of A and B(^{0}C)									

Question

a) Plot on the same axes, the cooling curves of both pure A and the mixture of A and B.

b) By use of the graph you have plotted in (a) above, determine the:

i) freezing point of pure substance A.

…….

ii) freezing point of the mixture of substances A and B.

…….

iii) depression in freezing point of substance A caused by 1.1g of substance B.

…….

…….

c) Relative formula mass of substance B.

(freezing point constant of substance A is $6.9^0Cmol^{-1}kg^{-1}$)

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GRAPH PAGE

**********THE END**********