A COMPREHENSIVE GUIDE TO A – LEVEL
CHEMISTRY PRACTICALS

STUDENT'S NAME: …………………………………………………………...

COMBINATION: ……………………………………………………………

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MAGALA RASHID
CHEMISTRY DEPARTMENT, KIBULI SS
BSc. ED (Hons) (MUK), MSc. PGP (MUK)
0702400181 / 0774400181
rashidmagalasnr@gmail.com

PREFACE

The purpose of this book is to provide a complete course of advanced level practical chemistry P525/3. This book is a hot cake; it has tremendously contributed to academic excellence in a number of schools. In schools where this book is used students score over 80% distinction in P525/3. These include Kibuli SS (88%), Ubuntu Hill School(86%), among others.

This book contains a simplified outline of the information required by a candidate in order to excel in practicals. This will enable students do a lot of self reading.

All experiments in this book are designed to require minimum amounts of reagents possible without compromising the experiments' objectives.

Students are advised to carry out experiments with hands on, rather than waiting to record results obtained by their colleagues.

The secret of excelling in chemistry hides in practicals and students must realise that acquisition of practical skills is a step by step process which requires one to have done a number of experiments with hands on using an accurately prepared practical manual.

ADVICE TO STUDENTS

Passing chemistry practicals requires;

- Being committed
- Regular attendance for practical lessons and doing experiments with hands on
- A lot of practice on calculations in volumetric analysis
- Ensuring that every practical you have done is assessed
- Cooperating with your teachers and laboratory technicians
- Consulting your teachers
  Discipline

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Part I: Physical Chemistry

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PART I: PHYSICAL CHEMISTRY

CHAPTER 1: INTRODUCTION

VOLUMETRIC ANALYSIS

Volumetric analysis involves measuring the volume of a solution of a substance that is required to react exactly with a measured volume of a solution of another substance.

Apparatus used in volumetric analysis

Pipette: This is used to measure a fixed volume of solution to be used in titration.
The volume of solution in a pipette is called an aliquot.
The volume of pipette used is normally recorded to one decimal place e.g. 10.0cm^3 or 20.0cm^3 or 25.0cm^3.

How to use a pipette

- Suck up the solution until it is a little above the mark of the stem.
- To avoid sucking up a mouthful, keep the jet well below the level of the solution.
- Close the pipette with the first fore finger or the thumb.
- Gently relax the pressure of the finger until the solution level has fallen to the mark. When doing this, hold the pipette at eye level. When the bottom of the meniscus is on the mark, increase the finger pressure to prevent further loss of liquid.
- Transfer the pipetted solution into the conical flask.
- After emptying, allow the pipette to drain against the side of the vessel for about 10 seconds.

N.B: Do not blow out the last drop of the solution into the pipette.

Burette: This is graduated from the top down wards in cubic centimeters and tenths of cubic centimeters. The level of the liquid is read at the bottom of the meniscus. Care must be taken when reading the burette to have the eye level with the bottom of the meniscus.

How to use a burette

- The burette is clamped in a vertical position in a burette holder.
- The burette is filled using a funnel.
- The tap of the burette is quickly opened and closed so as to fill its jet with the solution.
- The initial level of the solution is then read and recorded.
- The solution is run out of the burette slowly using the tap, into the solution in the conical flask until the end-point. The final level of the burette is read and recorded.
- The volume of the solution used is obtained by subtracting the initial reading from the final reading.
- The readings on the burette are recorded to two decimal places e.g. a burette reading of 16cm^3 is written as 16.00cm^3, 16.5cm^3 as 16.50cm^3 etc.

Conical flask: This is where the pipetted aliquot is transferred. During titration, the conical flask is placed below the burette jet on a white back ground. While titrating, the candidate's eyes must be directed towards the conical flask. A permanent colour change of the indicator during titration indicates the end-point of the titration.

Volumetric flask: This is normally used during preparation of a solutions of a solid in a specific volume of water. They are made in various volumes but the most common is one which measures 250cm^3.

Precautions on a volumetric flask include:

- Never pipette a solution directly from a volumetric flask.
- Never hold a volumetric flask by its neck.

Terms used in volumetric analysis

Titration: This is the process of adding a solution from the burette into a conical flask containing another solution.

Equivalence point: This is the point of a titration where the two substances have been mixed in exactly their stoichiometric proportions.

End-point: This is the point during a titration at which the substance added from the burette is just enough to completely react with all the substance in the conical flask and a slight excess has been added, causing a change in colour of the indicator used.

PRESENTATION AND TREATMENT OF RESULTS

Sample table of results

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

Experiment number	1 (rough)	2	3
Final burette reading (cm^3)	**22.20**	**19.50**	**23.90**
Initial burette reading (cm^3)	**3.00**	**1.50**	**6.00**
Volume used (cm^3)	**19.20**	**18.00**	**17.90**

Values used to calculate average titre **18.00cm^3 and 17.90cm^3**

$$\text{Average titre value} = \frac{18.00 + 17.90}{2}$$

$$= 17.95\text{cm}^3$$

N.B:

- The first titration may be treated as rough and it should only give a clue about the end point so that more accurate results can be determined in the subsequent titrations.
- The titration flask must be rinsed thoroughly with clean water before conducting the next titration.
- The titration must be repeated until two or more readings agree within ± 0.1cm^3.

Why sodium hydroxide, potassium manganate (VII) and concentrated sulphuric acid are not regarded as primary standards

Sodium hydroxide is deliquescent; once exposed to air, it quickly absorbs water from air and combines with it to form a solution, and hence it ceases to be pure.

In addition, when exposed to air, sodium hydroxide solution absorbs carbon dioxide from the air to form sodium carbonate and water.

$2NaOH(aq) + CO_2(g) \longrightarrow Na_2CO_3(aq) + H_2O(l)$

The sodium carbonate and water formed react with more carbon dioxide in the air to form sodium hydrogen carbonate, a white precipitate.

$Na_2CO_3(aq) + H_2O(l) + CO_2(g) \longrightarrow 2NaHCO_3(s)$

Potassium manganate (VII) cannot be obtained in a very pure form. Its aqueous solution is always contaminated with manganese (IV) oxide. This is because the manganate (VII) ion is a strong oxidizing agent and therefore unstable as it oxidizes water to oxygen and hydroxyl ions and itself reduced to manganese (IV) oxide.

$4MnO_4^-(aq) + 2H_2O(l) \longrightarrow 4MnO_2(s) + 3O_2(g) + 4OH^-(aq)$

Concentrated sulphuric acid is hygroscopic; once exposed to air, it quickly absorbs water from the air but does not form a solution, and hence it ceases to be pure.

STANDARD SOLUTIONS

A standard solution is one whose concentration is accurately known.

The concentration of a standard solution can be expressed both in terms of moles per litre (molar concentration / molarity) and in terms of grams per litre (mass concentration).

PREPARATION OF STANDARD SOLUTIONS

A standard solution is prepared by dissolving a known amount (mass or volume) of a primary standard in water to make a known volume of solution.

PREPARATION OF A STANDARD SOLUTION FROM A SOLID PRIMARY STANDARD

A calculated mass of the solid primary standard is dissolved in water to make a known volume of solution.

Example

Describe how you would prepare 2 litres of 0.1M sodium carbonate solution using anhydrous sodium carbonate. (Na = 23, C = 12, O = 16)

Solution

1 litre of 0.1M Na_2CO_3 solution contains 0.1 moles of Na_2CO_3

2 litres of 0.1M Na_2CO_3 solution contain (2 × 0.1) moles of Na_2CO_3

= 0.2 moles of Na_2CO_3

RFM of $Na_2CO_3 = 2\times23 + 12 + 16\times3 = 106$

1 mole of Na_2CO_3 weighs 106g

0.2 moles of Na_2CO_3 weigh $\frac{106\times0.2}{1}$ g

∴ mass required = 21.2g

What to do

Weigh 21.2g of anhydrous Na_2CO_3 into a beaker, add a small volume of water (e.g. approximately 100 cm^3) and shake well to dissolve. Transfer the mixture into a 2 litre (2000 cm^3) measuring cylinder, rinse the beaker with more small volumes of water and top up the measuring cylinder to the 2000 cm^3 mark with this water.

Note: The same method is used to prepare solutions of approximately known concentrations of non-primary standards such as sodium hydroxide, potassium manganate (VII), etc.

Exercise

1. Calculate the mass of borax (sodium tetraborate, $Na_2B_4O_7 \cdot 10H_2O$) required to make 250cm^3 of 0.05M borax solution and describe how the solution can be prepared. (Na = 23, B = 11, O = 16, H = 1)
2. Calculate the mass of sodium oxalate, $Na_2C_2O_4$ required to make 800cm^3 of 0.04M sodium oxalate solution and describe how the solution can be prepared. (Na = 23, C = 12, O = 16)
3. Describe how you would prepare 1500 cm^3 of 0.02M potassium manganate (VII) solution. (K = 39, Mn = 55, O =16)

PREPARATION OF A STANDARD SOLUTION FROM A 'LIQUID' PRIMARY STANDARD

The 'liquid' primary standard is actually a solution of the primary standard in water with a very high concentration. It is commonly called a **stock solution.** The standard solution is prepared by diluting a calculated volume of the stock solution with water to make a known volume of solution.

The same method is used to prepare solutions of approximately known concentration of non-primary standards such as sulphuric acid, ammonia solution, etc.

Below are the steps taken:

Step 1. Calculating the molarity of the stock solution

The required information for this calculation is usually found on the bottle containing the stock solution. It is as follows:

- Minimum assay (percentage by mass) w/w of the stock solution
- Specific gravity (density) of the stock solution
- Molecular weight (RMM) of the primary standard

Examples

a. Calculate the molarity of a stock solution of concentrated sulphuric acid whose specifications are:

Minimum assay = 98% (w/w)
Specific gravity = 1.84gcm^{-3}
Molecular weight = 98

Solution

$$\text{Mass of } H_2SO_4 \text{ alone in 1 } cm^3 \text{ of stock solution } = \frac{98}{100} x \; 1.84 \; g$$

$$\text{Mass of } H_2SO_4 \text{ alone in } 1000cm^3 \text{ of stock solution } = \frac{98}{100} \; x \; 1.84 \; x \; 1000 \; g$$

Molecular weight = 63

(b) Calculate the volume of the stock solution required to prepare 800 cm^3 of 0.2M nitric acid.

2. (a) Calculate the molarity of a stock solution of concentrated ammonia solution whose specifications are:
Minimum assay = 30% (w/w)
Specific gravity = $0.89gcm^{-3}$
Molecular weight = 17
(b) Calculate the volume of the stock solution required to prepare 1100 cm^3 of 2M ammonia solution.

STANDARDISATION OF SOLUTIONS

Once a standard solution is prepared from a primary standard, it can be used to standardize other solutions. Standardization refers to the process of determining the concentration of a solution by titrating it with another solution whose concentration is known.

Once the concentration of a solution is determined, the following can be determined:

(i) The relative formula mass of a compound.
(ii) The relative atomic mass of an element in a compound.
(iii) The number of moles of water of crystallization per mole of a hydrated compound.
(iv) The percentage of water of crystallization in a hydrated compound.
(v) The formula of a compound.
(vi) The valence of an element in a compound.
(vii) The stoichiometry (mole ratio) of a reaction.
(viii) The basicity of an acid.
(ix) The percentage purity or percentage impurity of an impure substance.
(x) The percentage composition of an element in a compound.
(xi) The solubility and solubility product of a sparingly soluble ionic compound.
(xii) The partition coefficient of a solute between two immiscible liquids.

CHAPTER 2: ACID - BASE TITRATIONS

Acid-base titrations are titrations involving reactions between acids and bases. One solution in a burette is slowly added to the other solution in a conical flask until a point is reached when the substance added from the burette is just enough to completely react with all the substance in the conical flask without any excess. This point is referred to as the equivalence point of the titration.

Equivalence point of an acid-base titration is the point in an acid-base titration where the acid and base have been mixed in exactly their stoichiometric proportions. An equivalence point cannot be obtained practically; it is only theoretical.

End-point is the point at which a slight excess of the reagent from the burette has been added to the reagent in the conical flask during a titration. At the end point, the equivalence point has been only slightly exceeded. The end point is indicated by a colour change of the indicator used. It is used as an approximation for the equivalence point.

Acid-base indicators (also less commonly known as a pH indicators) are weak acids or weak bases which change colour with change in pH.

Working range of an acid-base indicator is the range of pH values over which the colour change of the indicator takes place during an acid-base titration in which the indicator has been applied.

Qualities of a good indicator

It should have an easily detectable colour change at end point.
It should have a sharp colour change.
It should be non-toxic.

The table below summarises the colours of some common indicators in various media.

	COLOUR IN			
INDICATOR	***Neutral medium***	***Acidic medium***	***Alkaline medium***	***Working range***
Litmus	Blue/Red	Red	Blue	6.0 – 8.0
Methyl orange	Orange	Red	Yellow	3.0 – 4.4
Phenolphthalein	Colourless	Colourless	Purple	8.2 - 10.0

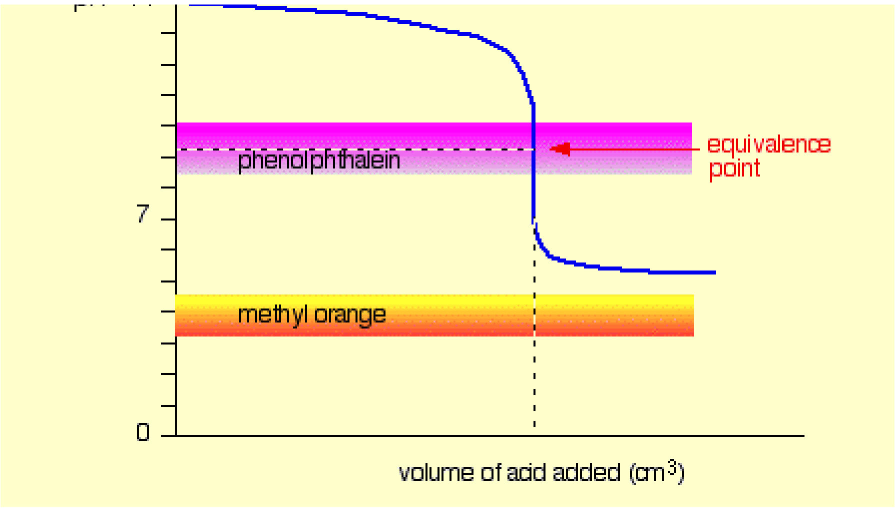
Choice of an indicator

In a titration, we need to choose an indicator which changes colour as close as possible to the equivalence point of the titration, that is, an indicator whose working range coincides with the equivalence point of the titration. The equivalence point varies from titration to titration.

The most commonly used acid-base indicators are phenolphthalein indicator and methyl orange indicator.

3. **Weak acid versus strong base**

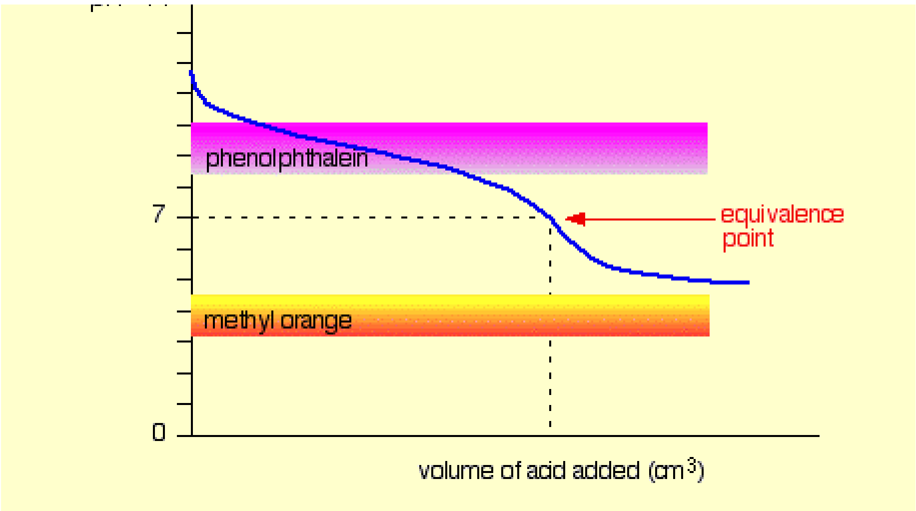
pH



The methyl orange is hopeless! However, the phenolphthalein changes colour exactly where you want it to.

4. **Weak acid versus weak base:** The curve is for a case where the acid and base are both equally weak.

pH



Neither indicator is of any use. Phenolphthalein will have finished changing well before the equivalence point, and methyl orange falls off the graph altogether.

NB: We would never titrate a weak acid and a weak base in the presence of an indicator.

Conclusion:

Phenolphthalein is colourless below a pH of 8.2 and purple above a pH of 10.0. It is therefore suitable for use in the following titrations:

1. Strong acid versus strong base
2. Weak acid versus strong base

EXPERIMENT 2.1

Standardization of hydrochloric acid using sodium carbonate

You are provided with the following:

FA1 which is a solution of hydrochloric acid of unknown concentration.

Solid X which is anhydrous sodium carbonate.

Methyl orange indicator.

You are required to prepare a standard solution of sodium carbonate and use it to standardize the acid.

Procedure

Weigh accurately 1.33g of solid X and pour it into beaker. Add about 100cm^3 of distilled water and shake the mixture well to dissolve. Transfer this solution into a 250cm^3 volumetric flask. Rinse the beaker several times with small portions of distilled water and use this water to make the solution in the volumetric flask up to the 250cm^3 mark. Pour this solution in a clean beaker and label it FA2. [use a similar procedure whenever dissolving a substance in water to make a specific volume of solution]

Pipette 20 or 25cm^3 of FA2 into a conical flask. Add 2-3 drops of methyl orange indicator and titrate it with FA1 from the burette until the yellow solution just turns red. Repeat the titration to obtain consistent results and record your results in the table below.

Results

Mass of weighing bottle + solid X = ……………………………………

Mass of weighing bottle alone = ………………………………………

Mass of solid X = ………………………………………………………...

Volume of pipette used = ……..……………………………………… cm^3

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

Titre values used to calculate the average volume of FA1 used

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Average volume of FA1 used ……………………………………………………………….

(a) Calculate the:

(i) molarity of FA2

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EXPERIMENT 2.2

Standardization of hydrochloric acid using borax

You are provided with the following:

Solid R which is sodium tetraborate (borax), $Na_2B_4O_7 \cdot 10H_2O$

FA1 which is hydrochloric acid of unknown concentration.

Methyl orange indicator.

You are required to prepare a standard solution of sodium tetraborate and use it to standardize the acid.

Theory

Borax reacts with hydrochloric acid according to the equation

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

Ionic equation:

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

Procedure

Weigh accurately about 4.77g of R and dissolve in about 100cm^3 of distilled water in a beaker. Transfer into 250cm^3 volumetric flask and make it up to the mark with more distilled water. Label the solution FA2.

Pipette 20 or 25cm^3 of FA2 into a conical flask and titrate with FA1 from the burette using methyl orange indicator.

Repeat the titration until you obtain consistent results and record the results in the table below.

Results

Mass of weighing bottle + R = ……………………………………………….

Mass of weighing bottle alone = ………………………………………………...

Mass of R = …………………………………………………………………

Volume of pipette used = ……………………………………………….. cm^3

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

Values of acid used to calculate average volume of FA1

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Average volume of FA1 used

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EXPERIMENT 2.3

To determine the number of moles of water of crystallization

You are provided with the following:

Solid T which is ethanedioic acid (oxalic acid), $H_2C_2O_4 \cdot nH_2O$ crystals

FA2 which is 0.1M sodium hydroxide solution.

Phenolphthalein indicator.

You are required to determine the number of moles of water of crystallization, n per mole of ethanedioic acid $H_2C_2O_4 \cdot nH_2O$

Procedure

Weigh 1.5g of T and dissolve it in distilled water to make 250cm^3 of solution using a volumetric flask. Label this solution FA1. Pipette 20cm^3 or 25cm^3 of FA1 and titrate it with FA2 from the burette using phenolphthalein indicator.

Repeat the titration to obtain consistent results and record the results in the table below.

Results

Mass of weighing bottle + T = …………………………………………

Mass of weighing bottle alone = ………………………………..

Mass of T = ………………………………………………..

Volume of pipette used = ……………………………...……………………….cm^3.

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

Titre values used to calculate the average volume of FA2 used.

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Average volume of FA2 used

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(a) Calculate the molarity of FA1

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

Titre values of FA1 used to calculate average volume.

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Average volume of FA1 used.

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(a) Calculate the;

(i) number of moles of sodium hydroxide in FA2 that reacted

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(ii) concentration of FA1 in moles of $MHSO_4$ per litre of solution.

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(b) Determine the relative atomic mass of M in $MHSO_4$

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EXPERIMENT 2.5

Determination of the value of y in $H(CH_2)_yCOOH$

You are provided with the following:

FA1 which is a solution of a monobasic organic acid $H(CH_2)_yCOOH$ of concentration 6.0g/dm^3.

FA2 is 1.5M sodium hydroxide solution

Methyl orange indicator.

You are required to determine the value of y in $H(CH_2)_yCOOH$

Procedure

Measure 15cm^3 of FA2 into a 250cm^3 volumetric flask and dilute with distilled water to the mark and label the resultant solution FA3. Pipette 20 or 25cm^3 of FA3 and titrate with FA1 using methyl orange indicator. Repeat the titration to obtain consistent results and record your results in the table below.

Volume of pipette ………………………………………… cm^3

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

Titre values of FA1 used to calculate average volume

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Average volume of FA1 used

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(a) Calculate the:-

(i) Number of moles of sodium hydroxide in FA3 that reacted

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(ii) Concentration of FA1 in moles per litre of solution

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Questions:
Calculate:

(i) the molarity of FA11

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(ii) the molarity of FA10

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CHAPTER 3: BACK TITRATION

The back titration technique is normally used to analyse substances that cannot be analysed by direct titration methods.

Some of the reasons why certain substances cannot be analysed by direct titration methods are:

1. When the substance to be analysed does not (readily) dissolve in water but can dissolve in another substance through reacting with it. For example,
   (a) The following substances do not dissolve readily in water but can readily dissolve in most dilute acids;
      (i) Moderately reactive metals such as Ca, Mg, Fe, Zn, Al, Pb, Sn, etc.
      (ii) Insoluble metal oxides such as CaO, MgO, FeO, Fe_2O_3, Al_2O_3, ZnO, PbO, SnO, CuO, etc.
      (iii) Insoluble metal carbonates such as $CaCO_3$, $MgCO_3$, $ZnCO_3$, etc.
      (iv) Insoluble metal hydroxides such as $Ca(OH)_2$, $Mg(OH)_2$, $Al(OH)_3$, etc.
   (b) Manganese (IV) oxide is insoluble in water but readily dissolves in a solution containing oxalate ions
2. When the substance to be analysed can dissolve in water but there is no substance it can be titrated directly with. For example,
   (a) Ammonium salts such as NH_4Cl, $(NH_4)_2SO_4$, NH_4NO_3, etc.
   (b) Sodium sulphite

Note: It is also possible to carry out back titration analysis even when the substance to be analysed does not fall in any of the two categories above. (see example 4 below)

General Experimental Procedure

- A known mass (or volume) of the substance to be analysed (say X) is reacted with an excess known volume, V of a standard solution of another reagent (say Y) such that some Y reacts with X, some Y remains un reacted, while all the X reacts and gets finished.
- The unreacted Y may be diluted by adding water to it (if its concentration is too high) or may not be diluted (if its concentration is fairly low).
- The unreacted Y (usually a portion of it) is titrated with a standard solution of another reagent (say Z) and the volumes of Y and Z that reacted are noted.
- By calculating backwards, the amount of X that reacted with Y can be determined.

General Calculation Procedure

[Hint: It is important that you first put the experimental procedure into a drawn-out sketch (as side work) to ease your calculations]

- Calculate the number of moles of Z in the reacted volume (this volume can either be the pipetted volume or the average titre value, depending on the experimental procedure used).
- Write equation for the reaction between Y and Z, obtain its mole ratio and use it to determine the number of moles of Y that reacted with Z during the titration.
- Determine the total number of moles of the unreacted Y.

(e) Calculate the original number of moles of sodium hydroxide in the 150 cm^3 of 1M sodium hydroxide. (0.15 moles)

(f) Calculate the number of moles of sodium hydroxide that reacted with ammonium chloride. (0.1022 moles)

(g) Write equation for the reaction between sodium hydroxide and ammonium chloride.

(h) Calculate the number of moles of ammonium chloride that reacted with sodium hydroxide. (0.1022 moles)

(i) Calculate the actual mass of ammonium chloride in the 6.12g of the impure sample. (5.4677g)

(j) Calculate the percentage purity of ammonium chloride in the impure sample. (89.34%)

3. 0.4g of MO (an oxide of a divalent metal) was dissolved in 100cm^3 of 0.5M nitric acid. The resultant solution was transferred into a 250cm^3 volumetric flask and made to the mark with distilled water. 16.75cm^3 of this solution was required to completely neutralize 20.0cm^3 of 0.1M sodium hydroxide solution. Determine the atomic mass of metal M. (24)

4. 60cm^3 of 0.25M sulphuric acid were added to 20cm^3 of yM potassium hydroxide solution and the resultant solution required 33.30cm^3 of 0.3M potassium hydroxide solution for complete neutralization. Determine the value of y. (1)

5. 1.8 g of the organic acid $C_nH_{n-1}COOH$, was reacted with 80cm^3 of 0.4M potassium hydroxide solution and then diluted to 200cm^3. 25cm^3 of the this solution containing unreacted potassium hydroxide was titrated with 0.1M sulphuric acid and 10.8 cm^3 of the acid was required to reach the end point. Calculate the relative molecular mass of the organic acid and hence determine its formula.

6. 0.3g of solid Z containing impure magnesium metal was dissolved in 50cm^3 of IM nitric acid. The resultant solution was diluted to 250cm^3 and labelled FA1. 25 cm^3 of 0.1M sodium hydroxide solution required 21.95 cm^3 of FA1 complete neutralization. Determine the percentage purity of magnesium in Z.

7. 6.58g of a fertilizer containing impure ammonium sulphate was boiled with excess potassium hydroxide solution and all the ammonia gas expelled was absorbed in 100cm^3 of 0.25M sulphuric acid. The resultant solution required 19.95cm^3 of 0.1M sodium hydroxide for complete neutralization.

(a) Calculate the number of moles of excess acid HCl (in the average volume of FA3 used.)

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(b) Determine the:-

(i) Original number of moles of HCl (in 40 cm^3 of FA1.)

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(ii) number of moles of HCl that reacted with Q
(number of moles of acid HCl that reacted with the 1.0g of the metal carbonate)

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(iii) number of moles of Q that reacted with HCl.

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(c) Calculate the value of M in MCO_3

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EXPERIMENT 3.2

Determination of the percentage purity of an organic acid

You are provided with the following:

FA1 is 0.5M sodium hydroxide solution.

FA2 is 0.1 M nitric acid.

Solid Y which is a dibasic organic acid

Procedure

Weigh accurately 1.0g of Y and transfer it to conical flask containing 100 cm^3 of distilled water. Add 40 cm^3 of FA1 and boil the mixture gently for about 15 minutes.

Cool and transfer the mixture into a 250cm^3 volumetric flask. Dilute with distilled water to the mark. Label the solution FA3.

Pipette 25 cm^3 of FA3 into a conical flask add 2-3 drops of phenolphthalein indicator and titrate it with FA2 from the burette. Repeat the titration to obtain consistent results and record your results in the table below.

Mass of weighing bottle + Y = ……………………………………………

Mass of weighing bottle = ……………………………………………

Mass of FA3 = ……………………………………………..

Volume of pipette……………………cm^3

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

Titre values of FA2 used to calculate the average volume

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Average volume of FA2 used

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(a) Calculate

(i) the number of moles of excess sodium hydroxide that did not react with the acid in Y

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EXPERIMENT 3.3

Determination of the percentage purity of an ammonium salt

You are provided with the following:

FA1 which is 1M sodium hydroxide solution.

FA2 which is 0.1M sulphuric acid.

Solid V which is impure ammonium chloride.

You are required to determine the percentage purity of V

Procedure

Weigh accurately 5.9 g of V and transfer it to a conical flask containing 150cm^3 of FA1. Boil the mixture gently until no more ammonia is expelled (test using moist red litmus paper). Cool and transfer the mixture into a 250 cm^3 volumetric flask. Make the solution up to the mark with distilled water and label it FA3.

Pipette 25 cm^3 (or 20 cm^3) of FA3 and titrate with FA2 using methyl orange indicator.

Repeat the titration to obtain consistent results.

Record the results in the table below.

Results

Mass of weighing bottle + V = ……………………………………

Mass of weighing bottle = ……………………………………

Mass of compound V = ……………………………………

Volume of pipette = …………………… cm^3

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

Titre values of FA2 used to calculate the average volume.

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Average volume of FA2 used

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Calculate the

(a)(i) number of moles excess sodium hydroxide that reacted with FA2

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(ii) number of moles of sodium hydroxide that reacted with ammonium chloride in V.

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(c) Determine:

(i) the mass of ammonium chloride in V that reacted with sodium hydroxide.

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(ii) the percentage purity of ammonium chloride in V.

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EXPERIMENT 3.4

You are provided with the following:

FA1, which is approximately a 1.0M sodium hydroxide solution.

FA2, which is a 1.0M hydrochloric acid.

FA3, which is 0.02M hydrochloric acid.

Q which is an acidic solid.

You are required to determine the percentage of the acid in **Q**.

Procedure A:

Pipette 25.0 (or 20.0) cm^3 of **FA1** into a conical flask and titrate it with **FA2** using phenolphthalein indicator. Repeat the titration until you get consistent results.

Record your results in Table A below.

Results:

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

Table A

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

Average volume of **FA2** used ………………………………………… cm^3.

Question:

Calculate the molar concentration of sodium hydroxide in **FA1**.

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Procedure B

Weigh accurately 1.5g of **Q** and transfer it to a conical flask containing about 25cm^3 of distilled water. Add 25cm^3 of **FA1** and boil the mixture gently for about 15 minutes (Add more water during the boiling to maintain the volume nearly constant if necessary).

Cool and transfer the mixture into a 250cm^3 volumetric flask. Dilute with distilled water to the mark. Label the solution **FA4.**

Pipette 25.0(or 20.0) cm^3 of **FA4** into a conical flask and titrate with **FA3**, using phenolphthalein indicator.

Repeat the titration until you obtain consistent results. Record your results in table **B** below.

Results:

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

Table B:

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

Volume of **FA3** used for calculation of the average titre values

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Average volume of **FA3** used

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Questions:

(a) Calculate:

(i) the number of moles of excess sodium hydroxide that did not react with the acid in **Q**.

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(ii) the number of moles of sodium hydroxide that reacted with the acid in **Q**.

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EXPERIMENT 3.5

You are provided with the following:

FA1, which is a 0.1M hydrochloric acid.

FA2, which is approximately a 0.1M sodium hydroxide solution.

FA3, which is a 2M hydrochloric acid.

Solid **W**, which is an impure metal carbonate.

You are required to standardize **FA2** and use it to determine the percentage of the impurity in **W**.

Procedure:

(a) Pipette 20 (or 25cm^3) of **FA2** into a conical flask.
Titrate with **FA1** using phenolphthalein as the indicator.
Repeat the titration until you obtain consistent results. Record your results in table 1 below.

Table 1

Volume of pipette used...cm^3.

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

Titrate values used for calculating the average volume of **FA1** used.

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Average volume of **FA1** used...cm^3.

Question:

(a) Calculate the concentration in moles per litre of solution **FA2**.

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(b) Weigh 1.5g of W and dissolve it in 20.0cm^3 of FA3 in a beaker. Add 80cm^3 of distilled water and label this solution FA4. Pipette 20 (or 25cm^3) of FA2 into a conical flask and titrate it with FA4 using phenolphthalein as the indicator. Record your results in Table 2 below.

Results:

Mass of container + **W**=………………………………………………….

Mass of empty container =………………………………………………..

Mass of **W** used = ……………………………………………………………

Volume of pipette used…………………………………………….cm³.

Table 2

Final reading (cm³)			
Initial reading (cm³)			
Volume of **FA4** used (cm³)			

Titre values used for calculating the average volume of **FA4** used.

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Average volume of **FA4** used……………………………………………….cm³.

Questions:

Calculate the

(a) (i) number of moles of hydrochloric acid that was in 100cm³ of **FA4**.

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(ii) number of moles of the metal carbonate that reacted. (the metal carbonate reacts with hydrochloric acid in the ratio: **1:2**)

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(b) Determine the percentage of the impurity in W.

(Relative formula mass of the metal carbonate = 100)

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CHAPTER 4: DOUBLE INDICATOR TITRATION

This is the type of titration used to determine the amount of each compound in a mixture using two different indicators in acid base titrations. Examples of such mixtures include: -

- Sodium hydroxide and sodium carbonate.
- Sodium hydroxide and sodium hydrogen carbonate
- Sodium hydrogen carbonate and sodium carbonate

The indicators commonly used are methyl orange and phenolphthalein.

Double indicator titration can be done in two different methods namely.

- **Continuous titration** – involves use of both indicators one at a time in the same pipetted volume of the solution mixture.
- **Titrate and pour** – involves pipetting two different aliquots of the mixture and each is titrated separately with phenolphthalein and methyl orange.

1. Mixture of sodium hydroxide and sodium carbonate

(a) Method 1 (Continuous titration)

Theory

A fixed volume of solution mixture is pipetted and 2-3 drops of phenolphthalein indicator is added. This is then titrated with the acid until the first end point is attained (purple to colourless).

The volume V_p of acid used is noted.

To the same solution, 2 to 3 drops of methyl orange indicator are added and the titration continued until the second point, end point is attained (yellow to red) The volume V_m of acid is again noted.

Treatment of results

During the titration with phenolphthalein as indicator, all the Sodium hydroxide is neutralized to sodium chloride while the sodium carbonate is half neutralized to sodium hydrogen carbonate.

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

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

The amount of acid used in the above two reactions is V_p.

During further titration with methyl orange, the sodium hydrogen carbonate formed is now neutralized to sodium chloride and carbon dioxide.

$NaHCO_{3(aq)} + HCl_{(aq)} \longrightarrow NaCl_{(aq)} + CO_{2(g)} + H_2O_{(l)}$

The volume of acid used is V_m

Since sodium carbonate is half neutralized in the titration using phenolphthalein indicator and the other half is neutralized in the titration using methyl orange indicator, the volume of acid needed for complete neutralization of sodium carbonate is twice V_m i.e volume of acid to neutralise $Na_2CO_3 = 2V_m$ cm^3.

Since all the sodium hydroxide in the first titration with phenolphthalein is neutralized, the volume of acid used is the difference between V_p and V_m i.e volume of acid to neutralise NaOH = $(V_p - V_m)$ cm^3.

Example

25cm^3 of a mixture of sodium hydroxide and sodium carbonate was pipetted into a conical flask, 3 drops of phenolphthalein indicator were added and the resultant mixture was titrated with 0.1M hydrochloric acid of which 22.50cm^3 were required to reach the end point. To the same mixure, 3 drops of methyl orange indicator were added and the titration continued using the same acid. 8.5cm^3 of the acid were required to reach the end point. Calculate the concentration in grams per litre of mixture in terms of:

(i) sodium hydroxide

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(ii) sodium carbonate

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EXPERIMENT 4.1

Determination of the amount sodium hydroxide and sodium carbonate in a mixture using standard hydrochloric

You are provided with;

FA1 which is a solution mixture containing sodium hydroxide and sodium carbonate.

FA2 is a standard solution of 0.1M hydrochloric acid.

Procedure

Pipette 20 or 25cm^3 of FA1 into a conical flask and titrate with FA2 using 2-3 drops of phenolphthalein indicator until the end point is attained.

Record the results in table I

Without pouring away, the resultant solution in the conical flask, add 2-3 drops of methyl orange indicator and continue to titrate with FA2 until the second end point is attained.

Record the results in table II.

Repeat the procedure 2 more times to obtain consistent results.

Volume of pipette = …………………………………………………………….cm^3.

Table I: (Phenolphthalein indicator)

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

Titrate values of FA2 used to calculate average volume

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Average volume of using phenolphthalein

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Table II (methyl orange indicator)

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

Titre values of FA2 used to calculate average volume

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Average volume of FA2 using methyl orange

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(a) Determine the volume of FA2 used to titrate

$NaOH_{(aq)} + HCl_{(aq)} \longrightarrow NaCl(aq) + H_2O_{(l)}$

$Na_2CO_{3(aq)} + HCl_{(aq)} \longrightarrow NaCl_{(aq)} + NaHCO_{3(aq)}$

V_p is the volume of acid used in the above two reactions.

During the titration with methyl orange all the sodium hydroxide and all the sodium carbonate are completely neutralized.

$NaOH_{(aq)} + HCl_{(aq)} \longrightarrow NaCl(aq) + H_2O_{(l)}$

$Na_2CO_{3(aq)} + 2HCl_{(aq)} \longrightarrow 2NaCl_{(aq)} + CO_{2(g)} + H_2O_{(l)}$

The volume V_m is the volume of acid used in the above two reactions.

Since sodium hydroxide is fully neutralized with both indicators and only half of sodium carbonate is neutralized to sodium hydrogen carbonate with phenolphthalein indicator, the volume of acid for half neutralization of sodium carbonate is the difference between V_m and V_p i.e

Volume of acid for half neutralization of Na_2CO_3 = $(V_m-V_p)cm^3$

Therefore for full neutralization of Na_2CO_3, volume of acid = $2(V_m-V_p)cm^3$. Since all the NaOH and Na_2CO_3, are neutralized with methyl orange indicator, the volume of acid used to neutralize NaOH only = $V_m-2(V_m-V_p)cm^3$.

$= (2V_p - V_m)\ cm^3$

Example

When $25cm^3$ of a mixture of sodium hydroxide and sodium carbonate was titrated with 0.1M sulphuric acid using phenolphthalein indicator, $29.90cm^3$ of the acid was required to reach the end point. Another $25cm^3$ of the mixture required $42.10cm^3$ of the same acid on titration using methyl orange indicator. Calculate the concentration in g/l of sodium hydroxide and sodium carbonate in the mixture.

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(ii) sodium carbonate in FA3

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2. Mixture of sodium carbonate and sodium hydrogen carbonate

(a) Method 1 (Continuous titration)

Theory

A fixed volume of the mixture is pipetted and titrated with a standard solution of an acid using phenolphthalein indicator and the volume V_p of acid required to reach the first end point is noted.

Methyl orange is then added to the resultant mixture and the titration is continued using the same acid solution until the second end point is attained. The volume V_m of acid required to reach the second end point is noted.

Treatment of results

During the titration using phenolphthalein indicator, sodium carbonate is only half neutralized while sodium hydrogen carbonate does not react at all.

$$Na_2CO_{3(aq)} + HCl_{(aq)} \longrightarrow NaCl_{(aq)} + NaHCO_{3(aq)}$$

Volume V_p is the volume of acid used in this reaction.

During the titration using methyl orange, the original sodium hydrogen carbonate in the mixture and the sodium hydrogen carbonate formed from the phenolphthalein titration are neutralized as in equations below.

$$\underset{\text{Original}}{NaHCO_{3(aq)}} + HCl_{(aq)} \longrightarrow NaCl_{(aq)} + CO_{2(g)} + H_2O_{(l)}$$

$$\underset{\text{Formed}}{NaHCO_{3(aq)}} + HCl_{(aq)} \longrightarrow NaCl_{(aq)} + CO_{2(g)} + H_2O_{(l)}$$

V_m is the volume of acid used in the above two reactions.

Since sodium carbonate is half neutralized in the phenolphthalein titration, the volume of acid required for complete neutralization of sodium carbonate is twice V_p i.e. volume of acid to neutralize $Na_2CO_3 = (2V_p)cm^3$.

EXPERIMENT 4.3

To determine the concentration (in g/dm^3) of sodium carbonate and sodium hydrogen carbonate in a mixture.

You are provided with;

FA1 which is a solution of a mixture of sodium carbonate and sodium hydrogen carbonate

FA2 is 0.1M hydrochloric acid.

Procedure

Pipette 20 or 25cm^3 of FA1 and titrate with FA2 using 2-3 drops of phenolphthalein indicator until the end point is attained.

Record the volume of FA2 used in table I.

Without pouring away the resultant solution in the conical flask, add 2-3 drops of methyl orange indicator and continue to titrate with FA2 until the second end point is attained.

Record the results in the table II.

Repeat the titration to obtain consistent results.

Volume of pipette used = ……………………………………………………cm^3.

Table I (phenolphthalein indicator)

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

Titre values of FA2 used to calculate average volume

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Average volume of FA2 used

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Table II (methyl orange indicator)

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

Titre values of FA2 used to calculate average volume

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Average volume of FA2 used

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Calculate the concentration in grams per dm^3 of:

Example

When 25cm^3 of a mixture of sodium carbonate and sodium hydrogen carbonate was titrated with 0.2M hydrochloric acid using phenolphthalein indicator, 15.00cm^3 of the acid was required to reach the end point. Another 25cm^3 of the same mixture required 40cm^3 of the same acid to reach the end point when using methyl orange as indicator. Calculate the concentration in g/l of sodium carbonate and sodium hydrogen carbonate in the mixture.

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EXPERIMENT 4.4

To determine the concentration (in g/dm^3) of sodium carbonate and sodium hydrogen carbonate in a mixture.

You are provided with;

FA3 which is a solution mixture containing sodium carbonate and sodium hydrogen carbonate.

FA4 is a solution of 0.1M hydrochloric acid.

Procedure

Pipette 20 or 25cm^3 of FA3 titrate with FA4 using 2-3 drops of phenolphthalein indicator until the end point is attained. Record the volume of FA4 used in table I

Pipette a fresh sample of FA3 and gain titrate with FA4 using 2-3 drops of methyl orange indicator until the end point is attained.

Record the volume of FA4 used in table II

Repeat the procedure to obtain consistent values.

Volume of pipette used = ……………………………………………………….cm^3.

Table I (using phenolphthalein indicator)

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

Titre values of FA4 used to calculate average volume

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Average volume of FA4 used

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Table II (Using methyl orange indicator)

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

Titre values of FA4 used to calculate average volume

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Average volume of FA4 used

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Calculate the concentration in grams per dm^3 of:

(i) sodium carbonate

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(ii) sodium hydrogen carbonate

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3. Mixture of sodium hydroxide and sodium hydrogen carbonate

(a) Method I (Continuous titration)

Theory

A fixed volume of solution mixture is pipetted and 2-3 drops of phenolphthalein indicator is added. This is then titrated with the acid until the first end point is attained (purple to colourless).

The volume V_p of acid used is noted.

To the same solution, without pouring it away, 2 to 3 drops of methyl orange indicator are added and the titration continued until the second point is attained (yellow to red) The volume V_m of acid is again noted.

Treatment of results

During the titration with phenolphthalein as indicator, all the Sodium hydroxide is neutralized to sodium chloride while the sodium hydrogen carbonate does not react with the acid.

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

The amount of acid used in the above reaction is V_p.

EXPERIMENT 4.5

Analysis of a mixture of sodium hydroxide and sodium hydrogen carbonate

You are required with the following:

FA1 which is a solution containing a mixture of sodium hydroxide and sodium hydrogen carbonate.

FA2 is 0.1M hydrochloric acid.

Phenolphthalein indicator

Methyl orange indicator

Procedure

Pipette 25cm^3 of FA1 and pour it into a 250cm^3 volumetric flask. Make the solution to the mark with distilled water and label the solution FA3. Pipette 20cm^3 or 25cm^3 of FA3 into a conical flask, add 2-3 drops of phenolphthalein indicator and titrate with FA2 from the burette until end point. Record your results in table I. Add 2-3 drops of methyl orange indicator to the resultant mixture and continue the titration until the second end point is reached. Record your results in table II. Repeat the procedure to obtain consistent results.

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

Burette readings

	TABLE I			TABLE II		
	Using phenolphthalein indicator			Using methyl orange indicator		
Final reading (cm^3)						
Initial reading (cm^3)						
Volume of FA2 used (cm^3)						

Average volume of FA2 used in table I: ……………………………………………….

Average volume of FA2 used in table II: ……………………………………………….

Calculate the:

(i) Volume of FA2 that reacted with NaOH in FA3

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(ii) Volume of FA2 that reacted with $NaHCO_3$ in FA3

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(iii) Concentration of NaOH in FA1 in g/l

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Calculate the concentration in grams per dm^3 of

(i) sodium hydroxide in the mixture

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(ii) sodium hydrogen carbonate in the mixture

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EXPERIMENT 4.7

You are provided with the following solutions:

FA1 which is hydrochloric acid of unknown concentration.

FA2 is a solution containing a mixture of sodium carbonate, Na_2CO_3 and sodium hydrogen carbonate, $NaHCO_3$.

FA3 is a solution containing 19.10g of borax, $Na_2B_4O_7 \cdot 10H_2O$ in 1.0dm^3 of solution. You are required to standardize the acid in **FA1** using **FA3**. Then, use the standardized **FA1** to determine the mass composition of the mixture **FA2**.

(Molecular mass of $Na_2B_4O_7 \cdot 10H_2O$= 381)

Borax reacts with hydrochloric acid according to the equation.

$$Na_2B_4O_7.\ 10H_2O + 2HCl \longrightarrow 2NaCl + 4H_3BO_3 + 5H_2O$$

Procedure of standardization of FA1

Pipette 25cm^3 (or 20cm^3) of FA_3 into a conical flask, add 3 drops of methyl orange indicator and titrate with FA1 from the burette. Repeat the titration until you obtain consistent results. Record your results in table 1.

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

Table 1

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

Average volume of **FA1** used ……………………………………………cm^3

(a) Determine the

(i) molarity of **FA3**

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(ii) molarity of **FA1**

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Procedure for the determination of the mass composition of FA2

Pipette 25cm^3 (or 20cm^3) of **FA2** into a conical flask, add 3 drops of phenolphthalein indicator and titrate with **FA1** from the burette. Stop the titration when you reach the end point and record the burette reading in the appropriate section of table 2, (using phenolphthalein). Add 3 drops of methyl orange indicator to the mixture in the flask and continue the titration. When you reach the end point, stop the titration and record the burette reading in the section (using methyl orange), in Table 2. Repeat the titration until you obtain consistent results.

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

Table 2

	Using Phenolphthalein			**Using Methyl Orange**		
Final reading (cm^3)						
Initial reading (cm^3)						
Volume of **FA1** used (cm^3)						

Average volume used with phenolphthalein

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Average volume used with methyl orange

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(b) Determine the:

(i) volume of **FA1** that reacted with Na_2CO_3.

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(ii) volume of **FA1** that reacted with $NaHCO_3$.

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(c) Calculate the mass of:

(i) Na_2CO_3 in 1 dm^3 of FA2.

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(ii) $NaHCO_3$ in 1 dm^3 of FA2.

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CHAPTER 5: REDOX TITRATIONS

Redox titrations are titrations involving redox reactions. A redox reaction is a reaction in which both oxidation and reduction take place simultaneously;

Oxidation

This can be defined as;

The addition of oxygen to a substance or the removal of hydrogen from a substance or **the removal of electrons from a substance**.

Examples of oxidation processes;

$$Fe^{2+}(aq) \longrightarrow Fe^{3+}(aq) + e$$

$$Zn(s) \longrightarrow Zn^{2+}(aq) + 2e$$

Note that electrons are on the right-hand side of the equation.

Oxidizing agent: This is a substance which removes electron(s) from another substance. It is also referred to an electron acceptor.

Examples of oxidizing agents:

Highly reactive non-metals such as Halogens like fluorine, chlorine.

Oxo-anions of transition elements

e.g $M_nO_4^-$, $Cr_2O_7^{2-}$ etc.

Oxo-anions of halogens.

e.g ClO_3^-, ClO^-, IO_3^- etc.

Reduction

This can be defined as;

The addition of hydrogen to a substance or the removal of oxygen from a substance or **the addition of electrons to a substance.**

Examples of reduction procecces;

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

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

Note that electrons are on the left-hand side.

Reducing agent: This is a substance which adds electron(s) to another substance. It can also be referred to as an electron donor.

Examples of reducing agents:

Highly reactive and moderately reactive metals e.g. Potassium, Sodium, Calcium, Zinc etc.

6. The overall charge on a compound or ion is equal to the sum of oxidation states of all elements in the compound/ion.

Examples

Determine the oxidation state of;

a) F in F_2 ; ans : 0
b) Cl in Cl^- ; ans : -1.
c) Zn in Zn ; ans : 0
d) Mn in MnO_4^-
Let w = oxidation state of Mn
w + (-2 x 4) = -1
w – 8 = -1
w = -1 + 8
w = +7
e) Mn in $KMnO_4$
Let y = oxidation state of Mn
1 + y + (4 x 2)=0
y + 1- 8 = 0
y – 7 = 0
y = +7
f) Mn in K_2MnO_4 (Potassium manganate (VI))
Let m be = oxidation state of Mn
(1x 2) + m + (4 x-2) = 0
2 + m - 8 = 0
m = +6
g) V in VO_3^+
Let z = oxidation state of V
z+ 3x -2 = +1
z – 6 = 1
z = +7

WRITING REDOX EQUATIONS.

This involves two steps:

1. Writing the half equations
2. Combining the half equations to obtain the overall redox equation

HOW TO WRITE HALF EQUATIONS

There are two types of half equation;

1. **Oxidation half equation**

This shows how the reducing agent loses the electron(s).

e.g.

$$Fe^{2+}(aq) \longrightarrow Fe^{3+}(aq) + e$$

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

Oxidizing agent	**Reduction product**
Manganate (VII) ion, MnO_4^-	Manganese (II) ion, Mn^{2+}
Dichromate ion, $Cr_2O_7^{2-}$	Chromium (III) ion, Cr^{3+}
Chlorate(V) ion ClO_3^-	Choride ion, Cl^-
Chlorate(I) ion ClO^-	Choride ion, Cl^-
Manganese (IV) oxide, M_nO_2	Manganese (II) ion, Mn^{2+}
Chorine, Cl_2	Chloride ion, Cl^-
Iodine, I_2	Iodide ion, I^-
H2O2	H_2O
Iodate(V) ion, IO_3^-	Iodine, I_2
Peroxodisulphate or persulphate ion, $S_2O_8^{2-}$	Sulphate ion, SO_4^{2-}

(b) Reducing agents

Hydrogen peroxide, H_2O_2	Oxygen, O_2

Illustration for step 1:

$Cr_2O_7^{2-} \longrightarrow Cr^{3+}$

Step 2:

Balance the number of any other atoms on either side of the equation before balancing oxygen or hydrogen.

Illustration for step 2: The Cr^{3+} is multiplied by 2 in order to balance Cr.

$Cr_2O_7^{2-} \longrightarrow 2Cr^{3+}$

Step 3:

Balance each oxygen atom by adding a water molecule for each oxygen atom required.

Illustration for step 3: 7 water molecules are added on the R.H.S in order to balance O.

$Cr_2O_7^{2-} \longrightarrow 2Cr^{3+} + 7H_2O$

Step 4:

Balance each excess hydrogen atom by adding a hydrogen ion for each hydrogen atom required.

Illustration for step 4: 14 H^+ are added on the L.H.S in order to balance H.

$Cr_2O_7^{2-} + 14H^+ \longrightarrow 2Cr^{3+} + 7H_2O$

Step 5:

Balance the charges by adding the appropriate number of electrons to the side which has an excess of positive charge.

Illustration for step 5: 6e are on the L.H.S in order to balance the charges.

$Cr_2O_7^{2-} + 14H^+ + 6e \longrightarrow 2Cr^{3+} + 7H2O$

Examples

1. Write the reduction half equation for the manganate (VII) ion

   Solution

   Step 1: $MnO_4^- \longrightarrow Mn^{2+}$

   Step 2 is irrelevant here

   Step 3: $MnO_4^- \longrightarrow Mn^{2+} + 4H_2O$

   Step 4: $MnO_4^- + 8H^+ \longrightarrow Mn^{2+} + 4H2O$

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

2. Write the oxidation half equation for the nitrite ion.

   Solution

   Step 1: $NO_2^- \longrightarrow NO_3^-$

   Step 2 is irrelevant here

   Step 3: $NO_2^- + H_2O \longrightarrow NO_3^-$

   Step 4: $NO_2^-(aq) + H_2O(l) \longrightarrow NO_3^-(aq) + 2H^+(aq)$

   Step 5: $NO_2^-(aq) + H_2O(l) \longrightarrow NO_3^-(aq) + 2H^+(aq) + 2e$

Step 2:

$(MnO_4^- + 8H^+ + 5e \longrightarrow Mn^{2+}{}_{(aq)} + 4H_2O_{(l)}) \times 2$

$(C_2O_4^{2-} \longrightarrow 2CO_2 + 2e) \times 5$

Step 3:

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

Step 4:

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

CATEGORIES OF REDOX TITRATIONS

1. POTASSIUM PERMANGANATE TITRATIONS
2. POTASSIUM DICHROMATE TITRATIONS
3. IODIMETRY AND IODOMETRY

1. POTASSIUM PERMANGANATE TITRATIONS

Potassium permanganate or Potassium manganate(VII) contains the manganate(VII) ion, $MnO4^-$ which is a powerful oxidizing agent. It is therefore very useful in volumetric analysis, particularly since it requires no indicator.

In acidic medium, the manganate(VII) ion (purple solution) is reduced by many reducing agents to manganese(II) ions (colourless or faint pink solution)

Reactions between manganate(VII) ions and oxalate ions are carried out at about 60°C since they tend to be slow at room temperature. The slowness is caused by the repulsion between the anions (manganate(VII) ions and oxalate ions), which reduces their frequency of collision at room temperature.

Potassium manganate(VII) is only stable in acidic medium and only sulphuric acid can be used to acidify potassium permanganate but not hydrochloric acid or nitric acid.

- With hydrochloric acid, Potassium permanganate being a strong oxidizing agent, it oxidises chloride ions in hydrochloric acid to chlorine.
  $2MnO_4^-(aq) + 16H^+ (aq) + 10Cl^-(aq) \longrightarrow 2Mn^{2+} (aq) + 8H_2O (l) + 10Cl_2(g)$
- This means that the oxidizing agent would be consumed before performing its intended oxidation role.
- With nitric acid, nitric acid itself being a powerful oxidizing agent, it would compete with the potassium permanganate for the reducing agent.

Potassium permanganate is not a primary standard. This is because it is always with impurities such as manganese (IV) oxide. Therefore if it is to be used in volumetric analysis, it's good to standardize it first. Potassium permanganate is normally standardized using the following primary standards:

(i) Sodium ethanedioate (sodium oxalate)
(ii) Ethanedioic acid (oxalic acid)

Calculate the concentration of:

(a) iron(II) ions in the mixture in mol dm^{-3} (Ans: 0.08 moldm^{-3})
(b) iron(III) ions in the mixture in mol dm^{-3} (Ans: 0.016 moldm^{-3})

5. GA1 is a solution containing 9.40g of a mixture of anhydrous oxalic acid (ethanedioic acid) and sodium oxalate (sodium ethanedioate) in a litre of solution.
   GA2 is 0.1M sodium hydroxide.
   GA3 is 0.02M potassium manganate(VII).
   On titration, 25 cm^3 of warm (60˚C) acidified GA1 required 35.60 cm^3 of GA3 and, 25 cm^3 of GA1 required 24.0 cm^3 of GA2 for complete reaction.
   Determine the:
   (a) Molar concentration of ethanedioic acid (Ans: 0.048 moldm^{-3})
   (b) Percentage of sodium ethanedioate in GA1.(Ans: 34%)
6. In a redox reaction involving manganate (VII) ions, 25.0 cm^3 of 30.60g/l of a contaminated iron(II) sulphate solution was pipetted then acidified. The resultant solution required 25.00 cm^3 of a solution containing 2.38g/l of manganate(VII) ions for complete reaction.
   Calculate the:
   (a) Molar concentration of iron(II) ions (Ans: 0.1 moldm^{-3})
   (b) Concentration of iron(II) sulphate in g/l (Ans: 27.8 g/l)
   (c) Percentage purity of iron(II) sulphate in the sample (Ans: 90.8%)
7. 3.69g of a metal peroxodisulphate (persulphate), $M_2S_2O_8$ was dissolved in water to make 250 cm^3 of solution. To 20 cm^3 of this solution was added 3.14g of ammonium ferrous sulphate, $(NH_4)_2Fe(SO_4)_2 \bullet 6H_2O$ and the solution made up to 100 cm^3 with distilled water. The mixture was shaken well and labeled FA1. 25 cm^3 of FA1 required 14.4 cm^3 of 0.02M acidified potassium permanganate solution to react with the unreacted iron(II) ions in the mixture. Determine the relative atomic mass of M.
   (Ans: 39)
8. 25 cm^3 of a solution prepared by dissolving 1.6g of $K_aH_b(C_2O_4)_c \bullet nH_2O$ in water to make 200 cm^3 of solution required 24.00 cm^3 of 0.1M sodium hydroxide solution for complete reaction. Another 25 cm^3 of the same solution 31.8 cm^3 of 0.02M potassium permanganate for complete reaction. Determine the values of a, b, c and n.
   (Ans: a=1, b=3, c=1, n=2)
9. 25 cm^3 of a solution prepared by dissolving 1.6g of iron(II) oxalate, $FeC_2O_4 \bullet nH_2O$ in water to make 250 cm^3 of solution required 27.00 cm^3 of potassium manganate(VII) to reach the end point. Determine the value of n in $FeC_2O_4 \bullet nH_2O$. (Ans: n=2)

(c) Calculate the:

(i) Concentration of sodium oxalate in FA2 in moldm^{-3}
(Na = 23, O = 16, C = 12)

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(ii) Concentration of potassium permanganate in moles per dm^3 and in g/l of FA1 (K = 39, Mn = 55)

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EXPERIMENT 5.2

Standardisation of potassium permanganate using ammonium iron (II) sulphate

You are provided with the following:

FA1 which is potassium permanganate solution

FA2 which is 2M sulphuric acid

Solid G which is ammonium iron (II) sulphate -6- water, $(NH_4)_2Fe(SO_4)_2•6H_2O$

You are required to determine the concentration of potassium permanganate in FA1.

Procedure

Weigh accurately 9.8g of G and dissolve it in 100cm^3 of water in a beaker.

Transfer the solution quantitatively into a 250cm^3 volumetric flask and add distilled water up to the mark. Label the solution FA3.

Pipette 20 or 25cm^3 of FA3 into a conical flask, add an equal volume of FA2 measured using a measuring cylinder and titrate with FA1 from the burette. Repeat the titration until you obtain consistent results and record your results in the table below.

Results

Mass of weighing bottle + G = ……………………………………

Mass of weighing bottle alone = ……………………………………

Mass of G = ……………………………………

Volume of pipette used = …………………………… cm^3.

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

Titre values of FA1 used to calculate average volume.

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Average volume of FA1 used.

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(a) Write the overall equation for the reaction which occurs.

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(b) Calculate:-

(i) the molar concentration of ammonium iron(II) sulphate in FA3

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EXPERIMENT 5.3

Determination of the percentage purity of pyrolusite through a redox back titration

You are provided with the following:

FA1 which is oxalic acid solution.

FA2 which is 0.02M potassium Manganate (VII) solution.

Solid **R** which is pyrolusite (impure manganese (IV) oxide)

FA5 which is 1M sulphuric acid.

You are required to determine the;

(i) Molar concentration of FA1.

(ii) Percentage purity of manganese(IV) oxide in R.

Theory:

Pyrolusite is an impure form of Manganese (IV) oxide. In acidic medium, manganese (IV) oxide reacts with oxalate ions as follows:

$$MnO_2(s) + C_2O_4^{2-}(aq) + 4H^+(aq) \longrightarrow Mn^{2+}(aq) + 2CO_2(aq) + 2H_2O(l)$$

Oxalate ions react with acidified potassium permanganate as follows:

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

Procedure A

Using a measuring cylinder, measure 100cm^3 of **FA1** and transfer it into a 250cm^3 volumetric flask. Add distilled water and make up to the mark. Label resultant solution **FA3**.

Pipette 25cm^3 of **FA3** into a clean conical flask followed by an equal volume of FA5 and heat the mixture to about 60^{o}C. Titrate the hot solution immediately with **FA2** from the burette. Repeat the procedure to obtain consistent readings. Record your results in the table below.

Results

Volume of pipette used ……………………………………………… (cm^3)

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

Volume of **FA2** used to calculate average………………………………………..(cm^3)

Average volume of **FA2**…………………………………………………………..(cm^3)

Questions:

(a) Calculate the;

(i) number of moles of MnO_4^- in **FA2** that reacted.

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(ii) number of moles of $C_2O_4^{2-}$ in **FA3** that reacted.
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(iii) Molar concentration of $C_2O_4^{2-}$ in **FA1**
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Procedure B:

Weigh accurately 1.2g of R and transfer it into a conical flask. Add to it about 100cm^3 of **FA1** followed by about 20cm^3 of FA5. Boil the mixture gently for about 4-5 minutes (with a glass funnel in the neck of the flask) until the remaining solid particles become white or brown. Cool the mixture and transfer it into a 250cm^3 volumetric flask and then make up to the mark with distilled water.

Label the resultant solution **FA4**.

Pipette 25cm^3 of **FA4** into a clean conical flask, then add 20cm^3 of FA5 and heat the mixture to about 60^{o}C. Titrate the hot solution immediately with FA2 from the burette. Repeat the procedure to obtain consistent readings. Record your results in the table below.

(iv) MnO_2 that reacted with $C_2O_4^{2-}$ ions.

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(b) Determine the percentage of MnO_2 in R.

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EXPERIMENT 5.4

Determination of the number of molecules of water of crystallization in a hydrated salt

You are provided with the following:

FA1 which is approximately 0.02M potassium manganate(VII) solution

FA2 which is a solution containing 6.6 g of sodium ethanedioate, $Na_2C_2O_4$ per litre

FA3 which is a 2M sulphuric acid

Solid T which is hydrated iron(II) sulphate.

You are required to determine the:

(i) concentration of potassium manganate(VII) in FA1

(ii) number of moles of water of crystallization in T

Procedure:

(a) Pipette 25cm^3 of FA2 into a conical flask and add an equal volume of FA3.

Heat the mixture to about 60^{o}C and titrate the hot solution with FA1 from the burette until end point (when a permanent faint purple colour is obtained).

Repeat the titration to 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 FA1 used (cm^3)			

Titre values used to calculate average volume of FA1.

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Average volume of FA1 used.

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Questions:

(a) (i) Write equation for the reaction between potassium manganate(VII) and sodium ethanedioate.

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(ii)Calculate the molar concentration of potassium managanate(VII)

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(b) Weigh accurately about 8.8g of T and dissolve it in about 50cm^3 of FA3. Transfer the resultant solution quantitatively into a 250 cm^3 volumetric flask and make it up to the mark with distilled water. Label the solution FA4.

Pipette 25 (or 20cm^3) of FA4 into a conical flask. Add the same volume of FA3 and titrate the mixture with FA1.

Repeat the titration to obtain consistent results.

Results:

Mass of bottle + T = ……………………………………………………
Mass of bottle = ……………………………………………………………
Mass of T = …………………………………………………………..

Volume of pipette used = ………………………………………………...….cm^3.

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

Titre values used to calculate the average volume FA1.

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Average volume of FA1 used.

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(a) Calculate the molar concentration of iron(II) sulphate in FA4.

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(b) Determine the number of molecules of water of crystallization in T.

EXPERIMENT 5.5

Determination of the percentage of sodium oxalate in a mixture of oxalic acid and sodium oxalate

You are provided with the following solutions:
FA1 which contains a mixture of oxalic acid and sodium oxalate
FA2 which is a solution containing 4g/dm^3 of sodium hydroxide.
FA3 which is a 0.02M Potassium manganate (VII) solution
2M sulphuric acid
You are required to find the percentage of sodium oxalate in the mixture.
(Na = 23; O = 16; C = 12; H = 1)

Procedure

Pipette 25 cm^3 of FA1 and titrate it with FA2 using phenolphthalein indicator until the end point is attained. Repeat the titration to 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 FA2 used (cm^3)			

Average volume FA2 used

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Pipette 10 cm^3 of FA1 into a conical flask. Add 10 cm^3 of 2M sulphuric acid using a measuring cylinder. Warm the mixture to 60°C and titrate it with FA3 from the burette. Repeat the titration to 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 FA3 used (cm^3)			

Average volume of FA3 used

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EXPERIMENT 5.6

You are provided with the following:

FA1, which is a solution prepared by dissolving 31.4 g of ammonium ferrous sulphate $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$ in water to make 1 litre of solution

FA2, which is potassium manganate(VII) solution

FA3, which is IM sulphuric acid

Solid R, which is hydrated iron(II) oxalate, $FeC_2O_4 \cdot nH_2O$

You are required to standardize FA2 using FA1 and use the standardized FA2 to determine the value of n in $FeC_2O_4 \cdot nH_2O$

Procedure I

Pipette 25cm^3 or 20cm^3 of FA1 into a clean conical flask, add an equal volume of FA3 using a measuring cylinder and titrate with FA2 from the burette. Repeat the titration until you obtain consistent results and record your results in table 1 below.

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

Table 1

Final burette reading / cm^3			
Initial burette reading /cm^3			
Volume of FA2 used /cm^3			

Titre values used for calculating average volume of FA2

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Average volume of FA2 used

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QUESTIONS:

a. Calculate the molar concentration of FA2

(Fe = 56, S = 32, O=16, N=14, H= 1)

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(i) number of moles of manganate(VII) ions in FA2 that reacted

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(ii) relative formula mass of R

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(iii) value of n in $FeC_2O_4 \bullet nH_2O$

(Fe = 56, C = 12, O=16, H= 1)

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ANALYSIS OF HYDROGEN PEROXIDE

Facts about hydrogen peroxide

- Hydrogen peroxide behaves both as an oxidizing agent and as a reducing agent. When reacting with an oxidizing agent such as MnO_4^- it will behave as a reducing agent. In this case it will be oxidized to oxygen as shown in the oxidation half equation below:

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

  Activity:

Write a redox equation for the reaction between H_2O_2 and MnO_4^-

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When reacting with a reducing agent such as I^- it will behave as an oxidising agent. In this case it will be reduced to water as shown in the reduction half equation below:

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

Activity:

Write a redox equation for the reaction between H_2O_2 and I^-

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- Hydrogen peroxide spontaneously decomposes with or without a catalyst to produce water and oxygen

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

Volume strength of hydrogen peroxide solution

The concentration of a solution of hydrogen peroxide is usually expressed in terms **volume strength.**

Volume strength of a solution of hydrogen peroxide is the volume of oxygen gas measured at s.t.p produced by one volume of the solution of hydrogen peroxide upon complete decomposition.

For example:

Volume strength of a solution of hydrogen peroxide is the number of dm^3 of oxygen gas measured at s.t.p produced by one dm^3 of the solution of hydrogen peroxide upon complete decomposition.

Or

Volume strength of a solution of hydrogen peroxide is the number of cm^3 of oxygen gas measured at s.t.p produced by one cm^3 of the solution of hydrogen peroxide upon complete decomposition.

Volume strength can be calculated once the molar concentration of the hydrogen peroxide solution is known.

The unit of volume strength is: 'volume'.

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(b) the volume strength of hydrogen peroxide.

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EXPERIMENT 5.7

Determination of the volume strength of hydrogen peroxide solution

You are provided with the following:

FA1 which is a solution made by dissolving 39.2 g of ammonium iron(II) sulphate srystals, $(NH_4)_2Fe(SO_4)_2 \bullet 6H_2O$ in water to make one litre of solution.

FA2 which is a solution of potassium permanganate of unknown concentration.

FA4 which is 1M Sulphuric acid.

Liquid L which is a solution of hydrogen peroxide.

You are required to standardize FA2 and use it to determine the volume strength of hydrogen peroxide in liquid L.

Procedure 1

(a) Pipette 20cm^3 or 25cm^3 of FA1 into a conical flask. Add an equal volume of FA4 using a measuring cylinder and titrate with FA2 from the burette. Repeat the titration until you get consistent results. Record you results in the table I below.

RESULTS:

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

Table I

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

Average volume of FA2 used ………………………… cm^3

Questions:

(i) Write equation for the reaction between FA1 and FA2.

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(ii) Calculate the molarity of FA2.

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(iv) Determine the volume strength of hydrogen peroxide in liquid L
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3. POTASSIUM DICHROMATE TITRATIONS

Potassium dichromate is an oxidizing agent because it contains the dichromate ion, $Cr_2O_7^{2-}$. In aqueous acidic medium, the dichromate ion (orange solution) is readily reduced by a number of reducing agents to chromium (III) (green solution) ions as shown in the equation below:

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

Potassium dichromate is a primary standard because it has all the qualities of a primary standard.

$Cr_2O_7^{2-}$ is a weaker oxidizing agent than MnO_4^-, it cannot oxidize Cl^- in hydrochloric acid to Cl_2. Therefore, potassium dichromate can also be acidified using hydrochloric acid in addition to using sulphuric acid.

The most important application of dichromate is in its reaction with iron(II) in which it is often preferred to permanganate.

Unlike permanganate, dichromate titrations require an indicator. This is because it is not possible to see a sharp colour change from orange to green. There are three indicators that may be used for the titration of Fe^{2+} with $K_2Cr_2O_7$. These are diphenylamine, diphenylbenzidine and diphenylamine sulfonate. The colour change for all three indicators is green to violet, and this occurs when a slight excess of potassium dichromate has been added. Phosphoric(V) acid must be present to form a complex with the Fe^{3+} ions formed during the oxidation reaction; otherwise Fe^{3+} ions affect the colour change of the indicator.

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(b) Calculate the molarity of FA4

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(c) Determine the value of n in $(NH_4)_2SO_4 \bullet FeSO_4 \bullet nH_2O$
(N = 14, H = 1, S = 32, O = 16, Fe = 56)

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IODIMETRY AND IODOMETRY
(IODINE-THIOSULPHATE TITRATIONS)

IODIMETRY (THE DIRECT METHOD)

This is a titration technique in which a solution of iodine is prepared and directly titrated with another substance, usually sodium thiosulphate solution.

Iodine is an oxidizing agent, though a relatively weak one. It can oxidize easily oxidizeable species such as:

- Thiosulphate ions ($S_2O_3^{2-}$) to tetrathionate ions ($S_2O_6^{2-}$),

  $$2S_2O_3^{2-}\,(aq) \rightarrow S_2O_6^{2-}\,(aq) + 2e$$

- Sulphite ions (SO_3^{2-}) to sulphate ions (SO_4^{2-})

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

- Tin(II) ions (Sn^{2+}) to tin (IV) ions (Sn^{4+})

  $$Sn^{2+}\,(aq) \rightarrow Sn^{4+}\,(aq) + 2e$$

The iodine itself is reduced to iodide ions:

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

Preparation of iodine solution

Iodine is sparingly soluble in water and therefore an aqueous solution of iodine is not simply prepared by dissolving iodine crystals in water. A solution of iodine (iodine solution) is prepared by dissolving iodine crystals in potassium iodide solution. This is because iodine reacts with iodide ions to form a soluble complex of tri-iodide ions:

$$I_2\,(aq) + I^-(aq) \rightleftharpoons I_3^-(aq)$$

An equilibrium is set up between iodine and tri-iodide ions, and if iodine molecules are removed from solution by a reaction, tri-iodide ions dissociate to form more iodine molecules:

$$I_3^-(aq) \rightleftharpoons I_2\,(aq) + I^-(aq)$$

Thus, a solution of iodine in potassium iodide can be titrated as though it were a solution of iodine in water.

The iodine-thiosulphate titration

A solution of iodine is best titrated with sodium thiosulphate solution. The sodium thiosulphate solution is placed in the burette and, as it is added to iodine solution in the conical flask, it reacts with the iodine according to the equation:

$$I_2\,(aq) + 2S_2O_3^{2-}\,(aq) \rightarrow 2I^-\,(aq) + S_4O_6^{2-}\,(aq)$$

During titration, the brown colour of the iodine solution fades gradually, and when it reaches a pale yellow colour, a few drops of a freshly prepared starch solution are added as the indicator for the titration. The solution becomes blue-black, and the titration is continued until it goes colourless. This marks the end-point.

sodium hydrogen carbonate and 25 cm^3 0.1M iodine solution. The mixture was stirred and labelled FA2. 25 cm^3 of FA2 required 14.35 cm^3 of 0.1M sodium thiosulphate solution to reach the point using starch indicator. Determine the value of n in $Na_2SO_3 \bullet nH_2O$.

EXPERIMENT 5.9

You are provided with the following solutions:
DA1: is a solution containing 13 g of an impure iodine in 1 dm^3 of aqueous solution
DA2: is a solution containing 0.025 moles of $Na_2S_2O_3 \bullet 5H_2O$ in 250 cm^3 of aqueous solution.
Starch Indicator.

Procedure:
(a) Pipette 25 cm^3 (or 20cm^3) of DA1 into a conical flask. Titrate DA1 with DA2 from the burette until the solution is pale yellow. Add starch indicator and continue to titrate until the blue-black colour is discharged. Repeat procedures to obtain consistent readings. Record your results in the table as shown below.

Results:
Volume of pipette used = …………………………… cm^3

Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of DA2 used (cm^3)			

Average volume of DA2 used ………………………………………

Questions:
(a) Calculate the concentration of DA2 in moles per dm^3.

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(b) Calculate:
(i) The molarity of iodine in DA1

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EXPERIMENT 5.10

To determine the percentage of water of crystallization in $Na_2SO_3 \cdot nH_2O$ through a back titration

You are provided with the following:

FA1 which is 0.05M iodine solution

FA2 which is sodium thiosulphate solution

FA3 which is sodium sulphite solution made by dissolving 12.8g of sodium sulphite crystals ($Na_2SO_3 \cdot nH_2O$) in water to make one litre of solution.

Solid G which is sodium hydrogen carbonate.

Starch indicator.

You are required to standardize FA1 and use it to determine the percentage of water of crystallization in the salt $Na_2SO_3 \cdot nH_2O$.

Theory:

Sodium sulphite is added to excess iodine solution, sulphite ions are oxidized to sulphate ions by iodine according to the equation:

$$SO_3^{2-} (aq) + I_2 (aq) + H_2O (l) \rightarrow SO_4^{2-} (aq) + 2I^- (aq) + 2H^+ (aq)$$

The reaction produces H^+ which must be removed immediately. Thus sodium hydrogen carbonate must be added to remove the H^+ .

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

The combined equation is:

$$SO_3^{2-} (aq) + I_2 (aq) + 2HCO_3^- (aq) \rightarrow SO_4^{2-} + 2I^- (aq) + CO_2 (g) + H_2O (l)$$

The unreacted iodine is titrated with a standard solution of sodium thiosulphate.

$$I_2 (aq) + 2S_2O_3^{2-} (aq) \rightarrow 2I^- (aq) + S_4O_6^{2-} (aq)$$

Procedure I

Pipette 10 cm^3 of FA1 into a clean conical flask. Titrate this solution with FA2 using starch indicator. Repeat the titration until you obtain consistent results. Record your results in table I below:

Results:

Volume of pipette used = …………………………… cm^3

Table I

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

Average volume of FA2 used ……………………………………… cm^3

(a) Calculate the molarity of FA2

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(ii) number of moles of excess iodine in 70 cm^3 of FA4

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(c) Determine the number of moles of sodium sulphite that reacted with the 50 cm^3 of FA1.

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(d) Determine the percentage of water of crystallization in $Na_2SO_3 \bullet nH_2O$

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IODOMETRY (THE INDIRECT METHOD)
This indirect technique involves titration of iodine liberated in a chemical reaction. In this method an oxidizing agent such as MnO_4^-, $Cr_2O_7^{2-}$, ClO_3^-, ClO^-, IO_3^-, $S_2O_8^{2-}$, Cl_2, Br_2, H_2O_2 etc. is treated with excess I^-(usually from KI) in acidic medium. The oxidizing agent oxidizes iodide ions to iodine, hence liberating iodine from iodide ions. The half equation is:
$2I^-(aq) \rightarrow I_2(aq) + 2e$

The amount of iodine liberated is stoichiometrically related to the amount of oxidizing agent used. Either the amount of the liberated is inused to standardize a solution of sodium thiosulphate or the amount of the liberated iodine is determined by titration with a standard solution of sodium thiosulphate using starch indicator.

NB: ***The amount of iodide ions and hydrogen ions is not required or used anywhere in these calculations. Thus they should not be involved in any calculations. Why? And what is their role?***

Standardisation of thiosulphate using potassium iodate (V)
Potassium iodate(V) is a primary standard. It reacts with iodide ions in the presence of acid to form iodine:

$IO_3^-(aq) + 5I^-(aq) + 6H^+(aq) \rightarrow 3I_2(aq) + 3H_2O(l)$

A standard solution of iodine can be prepared by weighing out the necessary quantity of potassium iodate(V) and making up a known volume of solution. When a portion of this solution is added to an excess of potassium iodide in acid solution, a calculated amount of iodine is liberated. Other primary standards that can be used to standardize thiosulphate include: Potassium dichromate

Questions:

1. 1.015 g of potassium iodate(V) were dissolved and made up to 250 cm^3. To a 25.0 cm^3 portion of this solution were added an excess of potassium iodide solution and dilute sulphuric acid. The solution was titrated with a solution of sodium thiosulphate, starch solution being added near the end point. 29.8 cm^3 thiosulphate were required. Calculate the molar concentration of the thiosulphate solution.
   (Ans: 0.095M)

2. A standard solution was made by dissolving 1.015 g of potassium dichromate and making up to 250 cm^3. A 25.0 cm^3 portion was added to an excess of potassium iodide and dilute sulphuric, and the iodine liberated was titrated with sodium thiosulphate solution. 19.20 cm^3 of this solution were needed. Find the molar concentration of thiosulphate solution.
   (Ans: 0.108M)

Estimation of chlorine
Chlorine displaces iodine from iodides. The iodine formed can be determined by titration with a standard thiosulphate solution. Chlorate(I) solutions are often used as a source of chlorine as they liberate chlorine readily on reaction with acid:

$ClO^-(aq) + 2H^+(aq) + Cl^-(aq) \rightarrow Cl_2(aq) + H_2O(l)$
The amount of chlorine available in a domestic bleach which contains sodium chlorate(I) can be found by allowing the bleach to react with an iodide solution to form iodine, and then titrating with thiosulphate solution:

EXPERIMENT 5.11

You are provided with the following:

FA1, which is a 0.012 M potassium iodate solution.

Solid W, which is sodium thiosulphate, $Na_2S_2O_3 \bullet 5H_2O$.

Liquid Z, which is a liquid bleaching agent containing chlorate(I).

You are required to prepare and standardize a solution of sodium thiosulphate and use it to determine the mass of chlorine required to produce 1 dm³ of the bleaching agent.

Potassium iodate reacts with potassium iodide according to the following equation;

$IO_3^-(aq) + 5I^-(aq) + 6H^+(aq) \rightarrow 3I_2(aq) + 3H_2O(l)$

Iodine reacts with sodium thiosulphate according to the equation

$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow S_4O_6^{2-}(aq) + 2I^-(aq).$

On combining the two equations above we get;

$IO_3^-(aq) + 6S_2O_3^{2-}(aq) + 6H^+(aq) \rightarrow I^-(aq) + 3S_4O_6^{2-}(aq) + 3H_2O(l).$

Chlorate(I) reacts with potassium iodide to form iodine according to the following equation;

$ClO^-(aq) + 2I^-(aq) + 2H^+(aq) \rightarrow I_2(aq) + Cl^-(aq) + H_2O(l).$

Procedure:

(a) Weigh accurately 6.0 g of **W** and dissolve it in about 100 cm³ of distilled water. Transfer the solution to a 250 cm³ volumetric flask and make it up to the mark with distilled water. Label the solution **FA2.**

Pipette 25.0 (or 20.0) cm³ of **FA1** into a conical flask. Add 20 cm³ of 1 M sulphuric acid, followed by 20 cm³ of 0.5 M potassium iodide solution. Titrate the mixture with **FA2** until the brown colour changes to yellow. Add 1 cm³ of starch indicator and continue the titration until the solution turns colourless.

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

Results:

Mass of **W** and weighing bottle …………………. g

Mass of weighing bottle alone …………………… g

Mass of **W** alone ……………….………………… g

Volume of pipette used …………………………….. cm³ .

Table B

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

Volumes of **FA2** used in calculating average volume

………………………………………………………………………………… cm^3

Average volume of **FA2** used ……………………………………………… cm^3

Questions:

(c) Calculate:

(i) the number of moles of iodine liberated.

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(ii) the number of moles of chloride ions that would be liberated from 10 cm^3 of **Z.**

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(iii) the number of moles of chlorine required to produce 1 dm^3 of **Z.**

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(iv) the mass of chlorine required to produce dm^3 of **Z.** $[Cl = 35.5]$

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EXPERIMENT 5.12

Determination of the percentage of copper

You are provided with the following:

FA1 which is sodium thiosulphate solution of unknown concentration.

FA2 which is a solution containing 0.9g of potassium iodate in water to make 250 cm^3 of solution.

Solid J which is a copper(II) salt

FA4 which is 10% potassium iodide solution

FA5 which is 1M hydrochloric acid.

Sodium carbonate solution.

Ethanoic acid solution.

You are required to standardize FA1 and use it to determine the percentage of copper in J.

Procedure I

Pipette 25cm^3 or 20cm^3 of FA2 into a conical flask, add an equal volume of FA5 followed by 10cm^3 of FA4.

Titrate the mixture with FA1 from the burette until the solution in the flask turns pale yellow. Add 3-4 drops of the starch indicator and continue the titration until the blue-black colouration is discharged to form a colourless solution. Repeat the procedure until you obtain consistent results and record your results in table I below:

Volume of pipette used = ………………………………………. cm^3.

Table I

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

Average volume of **FA1 used** ………………………………………… cm^3.

Questions:

(a) Calculate the molarity **FA1** in terms of thiosulphate ions.

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EXPERIMENT 5.13

You are provided with the following:

FA1, which is approximately a 0.1M sodium thiosulphate-5-water solution.

FA2, which is potassium manganate (VII) solution.

Solid Y, which is potassium dichromate (VI)

You are required to standardize FA1 and then use it to determine the molarity of FA2. Under acidic condition, iodide ions react with manganate (VII) ions and dichromate (VI) ions according to the following equations:

$Cr2O7^{2-}(aq) + 14H^+ (aq) + 6I^- (aq) \rightarrow 2Cr^{3+}(aq) + 3I_2(aq) + 7H_2O(l)$

$2MnO_4^-(aq) + 16H^+(aq) + 10I^- (aq) \rightarrow 2Mn^{2+} (aq) + 5I_2(aq) + 8H_2O(l)$

Thiosulphate (VI) ions reacts with iodine according to the equation:

$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow 2I^- (aq) + S_4O_6^{2-}(aq)$

Procedure:

(a) Weigh out accurately 1.0g of **Y** and dissolve it in about 50cm^3 of 2M sulphuric acid in a beaker. Transfer the solution into a 250cm^3 volumetric flask and make it up to the mark with distilled water. Label this solution **FA3**.

Results:

Mass of weighing container + **Y** = -------------------------------------- g

Mass of weighing container = -- g

Mass of **Y** = -- g

(b) Pipette 25.0 (or 20.0) cm^3 of **FA3** into a conical flask. Add 10cm^3 of potassium iodide solution followed by 10cm^3 of 2M sulphuric acid. Titrate the mixture with **FA1** until the solution is pale yellow. Add 5 drops of starch indicator and continue the titration until the blue colour turns green.

Repeat the titration to obtain consistent titre values. Record your results in Table I below.

Results:

Volume of pipette used ……………………… cm^3

Table I

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

Average volume of **FA1** used …………………………………….

EXPERIMENT 5.14

You are provided with the following:

GA1, which is a thiosulphate with the formula; $\mathbf{X_2S_2O_3.nH_2O}$, made by dissolving 12.4 g of the thiosulphate to make 250 cm^3 of solution.

GA2, which contains 3.5 g of a mixture of potassium chloride and potassium chlorate(V) in 1 litre of solution.

GA3, which is 1.0 M hydrochloric acid.

GA4, which is 10% potassium iodide solution.

Solid **Q**, which is potassium dichromate(VI)

You are required to determine the number of moles of water of crystallization **n** and to determine the percentage of potassium chloride in **GA2**.

In acid medium, chlorate(V) ions oxidize iodide ions to iodine. The chlorate(V) is reduced to chloride.

Dichromate(VI) ions in acid medium oxidize iodide ions to iodine. The dichromate(VI) are reduced to chromium(III) ions and water.

Procedure A:

Weigh accurately 2.0 g of **Q** and dissolve in about 100 cm^3 of distilled water. Transfer the resultant solution into a 250 cm^3 volumetric flask and make to mark with more distilled water. Label this solution **GA5**.

Results:

Mass of weighing container = ... g

Mass of weighing container = ... g

Mass of **Q** used = ... g

Procedure B:

Pipette 20.0 cm^3 (or 25.0 cm^3) of **GA5** into a conical flask, add an equal volume of **GA3** followed by 10 cm^3 of **GA4**. Titrate the resultant solution with **GA1** from a burette until the solution is pale yellow. Add 2 cm^3 of starch and continue the titration until the solution changes from deep blue to pale blue. Record the volume of **GA1** used. Repeat the titration until you get consistent results.

Record your results in table **I**.

Results:

Volume of pipette used = ... cm^3

Table I

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

(b) Determine the;

(i) Number of moles of dichromate(VI) ions that reacted with thiosulphate ions.

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(ii) Molar concentration of thiosulphate ions in **GA1**.

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(iii) the value of n.

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(c) Calculate the:

(i) the concentration of chlorate(V) in **GA2** in moles per dm^3

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(ii) mass of potassium chlorate(V) in **GA2**

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(iii) percentage of potassium chloride in **GA2**

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CHAPTER 6: COLLIGATIVE PROPERTIES

EXPERIMENT 6.1

You are provided with solids **F** and **G**.
You are required to determine the:

(i) Depression in the freezing point of **F**.
(ii) freezing point depression constant, k_f per kg of **F**.

Procedure:

(a) Pour about 200cm^3 of water into a 250cm^3 beaker.
Heat the water to boiling on a tripod stand.
In the meantime, weigh separately and accurately about 5.0g of F and 1.0g of **G**. Record the results of your measurements in the space provided below.

Results:

Mass of container + **F** = ……………………………………………..
Mass of empty container = …………………………………………..
Mass of **F** = ……………………………………………………………….
Mass of container + **G** = ……………………………………………..
Mass of empty container = ………………………………………….
Mass of **G** = ……………………………………………………………...

(b) Transfer the whole of **F** into a clean dry boiling tube. Immerse the boiling tube containing **F** into the beaker of hot water and continue heating the water until the whole of **F** melts.

Insert a thermometer in the liquid formed and heat to about 87^{o}C. Remove the boiling tube from the hot water and start the stop clock when its temperature drops to 85^{o}C.

Allow the liquid to cool while stirring with the thermometer and record its temperature after every half a minute for three minutes. Enter your results in the table below.

(c) Transfer the whole of **G** into the boiling tube containing **F**.
Immerse the boiling tube into the beaker of hot water and continue heating the water until the mixture melts. Continue heating until the temperature of the molten mixture is about 87°C. Remove the boiling tube from the hot water and start the stop clock when the temperature of mixture drops to 85^{o}C.

While stirring gently with the thermometer, allow the mixture to cool and record its temperature after every half a minute for three minutes.

Enter your results in the table below.

(c) From your graphs, read off the temperature after 2.5 minutes of

(i) pure**F**…………………………………………………………………...

(ii) mixture of **F** and **G**…………………………………………………

(d) Use the temperatures you have obtained in (c) above to determine the depression in the freezing point of **F**.

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(e) Calculate the freezing point depression constant, k_f per kg of **F**.

(Relative molecular mass of solid ***G*** *is 152).*

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CHAPTER 7: PARTITION EQUILIBRIA

EXPERIMENT 7.1

You are provided with the following:
FA1, which is aqueous ammonia.
FA2, which is trichloromethane.
FA3, which is 0.5M hydrochloric acid.
FA4, which is 0.05M hydrochloric acid.
You are required to determine the distribution (partition) coefficient of ammonia between water and trichloromethane.

Procedure

Add 10cm^3 of FA1 to 50cm^3 of FA2 in a conical flask. To this mixture, add 50cm^3 of distilled water. Shake the mixture vigorously for about 5 minutes and then allow to stand for about 10 minutes.

Carefully decant the upper (aqueous) layer into a boiling tube (or small conical flask/ beaker).

Transfer the lower (organic) layer into another boiling tube (or small flask/beaker) and cover.

Read and record the temperature in the results.

(i) Pipette 10cm^3 of the aqueous layer into a clean conical flask. Add 2 to 3 drops of phenolphthalein indicator and titrate with FA3.

Repeat the titration until you obtain consistent results and record your results in table 1.

Results:

Room temperature ……………………………………………………………….. ˚C.

Table 1

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

Average volume of FA3 used ………………………………………… cm^3

(ii) Pipette 10cm^3 of the organic layer into a clean conical flask. Add 2 to 3 drops of phenolphthalein indicator and titrate with FA4. Repeat the titration until you obtain consistent results. Record your results in table 2. xxxxxxxxxxxxxxxxx

(b) Determine the value of the distribution coefficient of ammonia between water and trichloromethane at the temperature you have recorded.

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EXPERIMENT 7.2

You are provided with the following;
YA1, which is a solution containing 10g of butanoic acid per litre.
YA2, which is a solution made by dissolving 3.2g of sodium hydroxide in water to make 1 litre of solution.
Liquid R, which is ether (ethoxyethane).
You are required to determine the distribution (partition) coefficient of butanoic acid between water and ether.
Caution!! Make sure there is no flame in the room

Procedure
Using a measuring, transfer 50cm^3 of YA1 and 50cm^3 of W into a conical flask.
Stopper the flask and shake vigorously for 5 minutes. Repeat the procedure using two other conical flasks with volumes:-

(i) 40cm^3 of YA1 and 60cm^3 of R.
(ii) 60cm^3 of YA1 and 40cm^3 of R

Leave the three flasks to stand after shaking to allow the layers to separate out. Note the temperature in each case.
Pipette 10cm^3 of the aqueous (lower) layer and titrate with YA2 using phenolphthalein indicator.

Record your results in the table.
Pipette 10cm^3 of the organic (upper) layer and titrate with YA2 using phenolphthalein indicator. Again, record your results in the table.
Repeat the procedure with the remaining two flasks as summarized in the table and record your results.

Mixture	Temp (˚C)	Layer	Final burette Reading (cm^3)	Initial burette reading (cm^3)	Volume of YA2 used (cm^3)
50cm^3 of YA1 and 50cm^3 of R		Aqueous			
50cm^3 of YA1 and 50cm^3 of R		Organic			
40cm^3 of YA1 and 60cm^3 of R		Aqueous			
40cm^3 of YA1 and 60cm^3 of R		Organic			
60cm^3 of YA1 and 40cm^3 of R		Aqueous			

EXPERIMENT 8.1

You are provided with the following:
FA1 is 0.02M iodine solution.
FA2 is 1.0M propanone.
FA3 is 0.01M sodium thiosulphate solution
1M Sulphuric acid
FA4 0.5M sodium hydrogen carbonate solution.

You are required to determine of the order of reaction with respect to iodine in the reaction between propanone and iodine.

Theory

In acidic medium, iodine reacts with propanone according to the equation

$$CH_3\,COCH_3(l) + I_{2(aq)} \rightarrow \quad CH_3COCH_2I_{(aq)} + \mathrm{HI}_{(aq)}$$

The reaction is acid catalyzed, and can be quenched (i.e. stopped) by removing the acid catalyst e.g. by addition of sodium hydrogen carbonate.

The amount of iodine left (unreacted) after a certain time (t) is determined by titration with standard sodium thiosulphate solution using starch indicator.

$$I_{2(aq)} + 2S_2O_{3\,(aq)}^{2-} \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$$

The amount of sodium thiosulphate required to reach endpoint is directly proportional to the amount of iodine remaining.

i.e. volume of $Na_2S_2O_3$ is directly proportional to the amount of iodine remaining.

Procedure

(i) Transfer 25cm^3 of FA2 and 25cm^3 of 2M Sulphuric acid into a conical flask.

(ii) Quickly add 50cm^3 of FA1 and at the same time start the stop clock. Shake the mixture well.

(iii) Using a clean measuring cylinder, transfer 10 cm^3 of FA4 into each of the conical flasks labeled 1, 2, 3, 4 and 5.

(iv) Pipette 10cm^3 of the acidified mixture of FA1 and FA2 obtained in step (ii) after exactly 5minutes and immediately pour it into the conical flask labeled 1. Shake well until effervescence stops. Titrate the residual iodine with FA3 using starch indicator until the end point is attained.

(v) Repeat the procedure in (iv) at 5 minutes interval using conical flasks labeled 2, 3, 4 and 5 respectively. Record the results in the table below.

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EXPERIMENT 8.2

You are provided with the following:
FA1 is 0.02M hydrogen peroxide
FA2 is potassium permanganate solution
FA3 which is 1M suphuric acid
1M iron (III) chloride solution.
You are required to determine the order of reaction with respect to hydrogen peroxide in the decomposition of hydrogen peroxide.

Theory

Hydrogen peroxide decomposes to form water and oxygen according to the equation below:

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

The reaction occurs in at a moderately high rate in an alkaline medium and in the presence of iron(III) chloride catalyst. It can therefore be quenched (i.e. stopped) by removing the alkali e.g. by addition of sulphuric acid.

The amount of H_2O_2 left un decomposed is determined by titration with standard acidified $KMnO_4$ solution. Manganate (VII) ions react with hydrogen peroxide according to the equation below:

$$MnO_4^-\,(aq) + 5H_2O_{2\,(aq)} + 16H^+_{(aq)} \rightarrow 2Mn^{2+}(aq) + 5O_2(g) + 8H_2O(l)$$

The volume of standard $KMnO_4$ solution required to reach end point is directly proportional to the amount of H_2O_2 remaining after a certain time.

Procedure

(i) Pipette 10.0cm^3 of FA1 into a clean conical flask, add 1cm^3 of sodium hydroxide solution followed by 2.0cm^3 of iron (III) chloride solution and start the stop clock immediately. Shake the mixture and allow it to stand for 3 minutes.

(ii) At the end of 3 minutes add 20cm^3 of FA3 and titrate the mixture with FA2 until the end point is attained.

(iii) Repeat the experiment but now add sulphuric acid after the time is 6, 9 and 12 minutes.

(iv) To determine the original concentration of hydrogen peroxide at t = 0 titrate 10.0cm^3 of the peroxide with FA2 directly without adding iron (III) chloride, record the results in the table below.

(c) Determine the rate constant K

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EXPERIMENT 8.3

You are provided with the following:

FA1 which is a **1M** solution of potassium iodide

FA2 which is a **0.05M** solution of sodium thiosulphate

FA3 which is a **0.1M** solution of hydrogen peroxide

FA4 which is **2M** sulphuric acid.

Starch solution.

You are required to investigate the effect of concentration of hydrogen peroxide on the reaction between potassium iodide and hydrogen peroxide.

Theory:
In acidic medium, hydrogen peroxide reacts with potassium iodide to liberate iodine as shown below.

$$H_2O_2(aq) + 2H^+(aq) + 2I^-(aq) \longrightarrow I_2(aq) + 2H_2O(l)$$

The reaction occurs slowly and the iodine liberated can be absorbed by sodium thiosulphate according to the equation.

$$I_2(aq) + 2S_2O_3^{2-}(aq) \longrightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$$

After some time t, the sodium thiosulphate gets used up and thus the iodine can no longer be absorbed. The excess iodine forms a dark blue colour with starch. The time t taken for the dark blue colour to be observed is noted.

NB: Potassium iodide is normally used in excess and sodium thiosulphate in a limited amount.

Procedure:

(a) Run 25.0cm^3 of **FA1** from the burette into a conical flask.

(b) Add 10cm^3 of **FA2** followed by 5cm^3 of **FA4** and 2cm^3 of starch.

(c) Transfer at once using a measuring cylinder 5cm^3 of **FA3** and immediately start the stop clock.

(d) Shake the mixture and record the time, **t** at which the blue colouration just appears.

(e) Repeat procedures (a) to (d) with the volumes of **FA3** shown in the table below.

CHAPTER 9: THERMOCHEMISTRY (CHEMICAL ENERGETICS)

EXPERIMENT 9.1

You are provided with the following.

FA1 which is 2M sodium hydroxide.

FA2 which is 2M hydrochloric acid

You are required to determine the enthalpy of neutralization of hydrochloric acid by sodium hydroxide solution.

Procedure:

(a) Using a measuring cylinder, measure out 50cm^3 of FA1 and place it into a plastic cup.

(b) Record the temperature t_1 (˚C) of this solution.

(c) Rinse the thermometer in distilled water and dry it.

(d) Rinse the measuring cylinder with distilled water and then measure out 50cm^3 of FA2.

(e) Measure and then record the temperature t_2 (˚C) of FA2.

Now add the 50cm^3 of FA2 to the 50cm^3 of FA1 in the plastic cup. Stir the mixture carefully with the thermometer and record the highest temperature t_3 attained.

Results

Volume of FA1 used = ……………………………………………………….

Volume of FA2 used = ……………………………………………………….

Total volume of mixture = ………………………………………………….

Initial temperature of FA1 (t_1) = ………………………………………

Initial temperature of FA2 (t_2) = ……………………………………...

Average initial temperature (t) = ………………………………………….

Highest temperature attained (t_3) = ……………………………………….

Temperature change (t_3 – t) = …………………………………………...….

Questions:

(a) Write an equation for reaction between FA1 and FA2.

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(b) Calculate the enthalpy of neutralization of hydrochloric acid

(Specific heat capacity of solution = 4.2J/g/˚C, Density of solution = 1g/cm^3)

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(ii)Calculate the number of moles of sulphuric acid in the volume you have obtained in (b) (i) above.

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(c) Calculate the enthalpy of neutralization of sodium hydroxide by sulphuric acid. (Specific heat capacity of solution = 4.2J/g/^{o}C density of solution = 1g/cm^3.)

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EXPERIMENT 9.3

You are provided with the following:

FA1 which is 2M copper (II) sulphate solution

Solid Z which is zinc powder

A stop clock.

You are required to determine the enthalpy of displacement of copper by zinc.

Procedure

(a) Measure 2g of Z and keep it.

(b) Using a measuring cylinder, transfer 50cm^3 of FA1 into a plastic cup and start the stop clock.

(c) Using a thermometer, measure and record the temperature of the solution every after half a minute for 2 minutes.

(d) After 2 minutes, add the Z you measured in procedure (a) above and stir well but carefully with the thermometer. Note and record the temperature after of the mixture every after half minute for five more minutes. Record your results in the table below.

Results

Time (min)	0	0.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0	4.5	5.0	5.5	6.0	6.5	7.0
Temp (°C)															

Questions

(b) Plot a graph of temperature against time.

(c) From the graph determine the;

(i) temperature change in the reaction

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(ii) heat change in the reaction

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(d) Calculate the enthalpy of displacement of copper by zinc

(Specific heat capacity of solution is 4.2J/g/^{o}C, density of solution = 1g/cm^3)

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CHAPTER 10: SOLUBILITY EQUILIBRIA

EXPERIMENT 10.1

You are provided with the following:

FA1 which is 0.1M sodium thiosulphate solution.

FA2 which is 10% potassium iodide solution.

FA3 which is 2M sulphuric acid

Solid R which is calcium iodate.

You are required to determine the solubility product of calcium iodate.

Procedure

Weigh accurately 1.5g of R into a 250cm ^{3} beaker. Add 100cm^3 of distilled water, stir with a stirring rod and shake vigorously. Transfer the mixture into a 250cm^3 volumetric flask and make the solution to the mark with more distilled water. Filter the solution into a beaker and label the filtrate FA4.

Pipette 10cm ^{3} of FA4 into a clean conical flask, add 10cm^3 of FA2 followed by 10cm^3 of FA3. Shake to mix and titrate with FA1 from the burette using starch indicator. Repeat the titration to obtain consistent results and record your results in the table below;

Mass of weighing container + R = ……………………… g

Mass of weighing container alone = ………………….. g

Mass of R = ……….……………………………………… g

Volume of pipette used ……………………………..cm ^{3}

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

Questions

(a) Calculate the number of moles of thiosulphate ions in FA1 used.

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(b) Determine the molar concentration of;

(i) iodate ions in FA4

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EXPERIMENT 10.2

You are provided with the following:
FA1 which is 0.05M hydrochloric acid.
Solid Q which is calcium hydroxide.
You are required to determine the solubility product of calcium hydroxide.

Procedure

Weigh accurately 3.0g of Q into a clean beaker. Add about 100cm^3 of distilled water and stir with a glass rod. Transfer the solution into a 250cm^3 volumetric flask. Rinse the beaker with more 50cm^3 distilled water and pour into the volumetric flask. Cork the flask and shake it and its contents vigorously. Make the solution to the mark with more distilled water. Filter the mixture into a clean beaker and label the filtrate FA2.

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

Mass of weighing bottle + Q = ……………………………….. g
Mass of weighing bottle = …………..………………………… g
Mass of Q alone = …………………………………..……….. g

Volume of pipette used = ………………………..………… cm^3

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

Average volume of FA1 used ………………………………………… cm^3

(a) Calculate the

(i) number of moles of HCl in FA1 that reacted

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(ii) molar concentration of hydroxide ions in FA2

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(iii) molar concentration of calcium ions in FA2.

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(c) Calculate the solubility product of calcium hydroxide

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PART II: INORGANIC CHEMISTRY

CHAPTER 12: INORGANIC QUALITATIVE ANALYSIS

Involves carrying out tests on a solid sample given to determine the cations and anions in it.

1. CATIONS

These are grouped in two categories:

(a) Non-coloured cations

These are:- Mg^{2+}, Ba^{2+}, Ca^{2+}, Pb^{2+}, Al^{3+}, Zn^{2+}, Sn^{2+}, Sn^{4+} and NH_4^+

(b) Coloured cations

These are:- Cu^{2+}, Fe^{2+}, Cr^{3+}, Ni^{2+}, Fe^{3+}, Mn^{2+} and Co^{2+}

2. ANIONS

These are:- SO_4^{2-}, SO_3^{2-}, CO_3^{2-}, NO_3^-, CI^-, Br^-, I^-, CH_3COO^- and $C_2O_4^{2-}$

Other anions are:- CrO_4^{2-}, $Cr_2O_7^{2-}$, HCO_3^-, NO_2^-, PO_4^{3-} and $S_2O_3^{2-}$

A. PRELIMINARY TESTS

These are the tests which the candidate carries out in order to get a clue about the nature of the substance being analysed.

Preliminary tests involve observing the:

1. Appearance of the substance
2. Action of heat on the substance

1. APPEARANCE

The appearance of substances can be analysed in terms of:

(a) texture

(b) smell

(c) colour

(a) **Texture:** This serves as a guideline as to what **anion(s)** may be present in the given solid sample. Here we are to note whether the solid given is in **crystalline** form or **powder** form.

OBSERVATION	**DEDUCTION**
Crystalline solid	SO_4^{2-}, SO_3^{2-}, NO_3^-, NO_2^-, CI^-, Br^-, I^-, CH_3COO^-, $C_2O_4^{2-}$present
Powdery solid	CO_3^{2-} , O^{2-} present

(b) **Smell:** Some compounds especially ammonium compounds have a noticeable smell.

OBSERVATION	**DEDUCTION**
Smell of ammonia	NH_4^+ present

Note: The observations and deductions for texture and smell may not be recorded on paper. They only guide the candidate's mind.

(c) Colour of solid / solution:

A solid can be the fresh solid sample given or the residue obtained from filtration. A solution can be obtained by dissolving the given solid sample in water or in a dilute acid or as a filtrate from filtration.

The colour of a solid or solution serves as a good guideline as to what **cation(s)** may be present in the given solid or solution.

OBSERVATION	**DEDUCTION**
Green solid / solution	Ni^{2+}, Cr^{3+}, Fe^{2+}, Cu^{2+} probably present
Blue solid / solution	Cu^{2+} probably present
Brown solid / Brown (or yellow) solution	Fe^{3+} probably present
White solid / colourless solution	Mg^{2+}, Ba^{2+}, Ca^{2+}, Pb^{2+}, Al^{3+}, Zn^{2+}, Sn^{2+}, Sn^{4+} and NH_4^+
Pink solid / pink solution	Co^{2+}, Mn^{2+}

2. ACTION OF HEAT

A little of the solid substance under test is heated in a clean **dry** hard glass boiling tube or test tube, gently first and then strongly until no further change occurs. Heating a compound can make it sublime (for those compounds which sublime when heated) or lose its water of crystallization (if it is hydrated) or produce a sound (e.g. a cracking sound by lead (II) nitrate) or decompose to give off a gas and leave behind a solid residue.

Note: Before heating, ensure that your test tube is clean and dry but not hot.

When heating, keen observation must be made on the following:

- Sublimate formed
- Condensate formed
- Sound produced
- Gas(es) given off
- Residue left behind.

(a) Sublimate: This is a solid deposited on the cooler inner parts of the test tube after heating a solid substance in a test tube. It is normally formed when compounds which sublime on heating are heated. Such compounds include ammonium salts, aluminium salts and iron (III) salts.

Reddish brown fumes with an irritating smell, turn moist blue litmus paper red and form a brown ring with freshly prepared $FeSO_4$ solution and conc. H_2SO_4	NO_2 given off ∴ NO_3^- present
Colourless gas with a pungent smell, turns moist blue litmus paper red then bleaches it, turns (acidified potassium dichromate solution from orange to green) or (acidified $KMnO_4$ solution from purple to colourless).	SO_2 given off ∴ SO_4^{2-}present
White fumes of a gas with an irritating smell, turn moist blue litmus paper red and forms dense white fumes with ammonia.	HCl given off ∴ Cl^- present
White fumes with a sweet smell, form an orange precipitate with 2,4dinitrophenyl hydrazine	CH_3COCH_3 vapour given off, :. CH_3COO^- present
White fumes of a gas with an irritating smell, turns moist blue litmus paper red and forms a white precipitate with acidified barium nitrate solution	SO_3 given off :. SO_4^{2-}present

NB: A candidate should have the ability to identify SO_2, NH_3, CH_3COCH_3, and CH_3COOH by their smell.

(e) Residue: This is the solid left in the test tube after heating the given solid sample in a test tube until no further change occurs. The colour of the residue gives some important information about the nature of the original sample.

OBSERVATIONS	DEDUCTIONS
A yellow solid residue when hot and turns white on cooling	ZnO formed :. Zn^{2+} present.
A brown solid residue when hot and turns yellow on cooling	PbO formed :. Pb^{2+} present.
A black solid residue	CuO or NiO or FeOformed :. Cu^{2+} or Ni^{2+} or Fe^{2+} present.
A brown solid residue both when hot and when cold	Fe_2O_3 formed :. Fe^{3+} present or Fe^{2+} oxidized to Fe^{3+}

B. TESTING FOR CATIONS AND ANIONS IN AQUEOUS SOLUTION

TESTS FOR CATIONS IN AQUEOUS SOLUTION

Cation	**With dil. NaOH**	**With dil. NH_4OH**	**With dil. H_2SO_4**	**Confirmatory test**	
Ba^{2+}	white precipitate insoluble in excess	white precipitate insoluble in excess	White precipitate	**Test**	**Observation**
				Add $(NH_4)_2C_2O_4$ (aq) followed by CH_3COOH or dil.HNO_3	White precipitate soluble in acid.
				Add K_2CrO_4 (aq) followed by CH_3COOH or dil. HNO_3	Yellow precipitate insoluble in acid.
				Add K_2CrO_4 followed by NaOH(aq)	Yellow precipitate insoluble in acid.
Mg^{2+}	White precipitate insoluble in excess	White precipitate insoluble in excess	No observable change	Add solid NH_4Cl followed by Na_2HPO_4(aq) and then NH_3(aq) dropwise till in excess	White precipitate insoluble in ammonia solution.
				Add $NaHCO_3$(aq) and heat	White precipitate
Zn^{2+}	White precipitate soluble in excess to form a colourless solution	White precipitate soluble in excess to form a colourless solution	No observable change	Add solid NH_4Cl followed by Na_2HPO_4(aq) and then NH_3(aq) dropwise till in excess.	White precipitate soluble in ammonia solution

NH_4^+	No observable. Change in the cold but on warming bubbles of a colourless gas with a choking smell, turns moist red litmus paper blue and forms dense white fumes with conc. HCl.	No observable change	No observable change	Add NaOH(aq) and warm	Bubbles of a colourless with a choking smell, turns moist red litmus paper blue and forms dense white fumes with conc. HCl

TESTS FOR ANIONS IN AQUEOUS SOLUTION

Anion	With $Pb(NO_3)_2$ (aq)	With $Ba(NO_3)_2$ (aq) or $BaCl_2$(aq)	With $AgNO_3$(aq)	With acidified $KMnO_4$	With dil. HNO_3 followed by bleaching powder and then CCl_4
SO_4^{2-}	White precipitate does not dissolve on heating and in acid	White precipitate does not dissolve in acid			
Cl^-	White precipitate does not dissolve in acid but dissolves on heating and re-appears on cooling	No observable change	White precipitate which does not dissolve in acid but dissolves in NH_3(aq) to form a colourless solution	Bubbles of a greenish yellow gas, purple solution turns colourless	Colourless solution in the organic layer

	acid with no effervescence				

TESTS FOR THE NITRATE ION AND THE ETHANOATE ION

The NO_3^- ion

TEST	OBSERVATION
To the solid, add conc. H_2SO_4 and heat	Reddish brown fumes that turn moist blue litmus paper red and form a brown ring with freshly prepared $FeSO_4$ solution and conc. H_2SO_4
To the solution, add copper turnings followed by conc. H_2SO_4 and heat.	Reddish brown fumes that turn moist blue litmus paper red and form a brown ring with freshly prepared $FeSO_4$ solution and conc. H_2SO_4
To the solution, add zinc powder (or aluminium powder or Devarda's alloy) followed by NaOH solution and warm.	A colourless with a choking smell, turns moist red litmas paper blue and forms dense white fumes with conc. HCl
Add freshly prepared $FeSO_4$ solution followed by conc. H_2SO_4 down the sides of a slanting test tube.	A brown ring

The CH_3COO^- ion

TEST	OBSERVATION
To the solid, add conc. H_2SO_4 and heat	White fumes with a sharp vinegar smell

To the solution, add conc. H_2SO_4 followed by an alcohol and warm. Pour the resultant mixture in a beaker containing cold water.	A sweet fruity smell.
To the solution add neutral iron (III) chloride solution	A brown precipitate

SEPARATION OF MIXTURES IN QUALITATIVE ANALYSIS

Whenever a candidate is to do a practical exercise on inorganic qualitative analysis, they are always given a filter paper. Why? The answer is that, he/she is to use it to separate a mixture by filtration. **Why do we separate mixtures?** The answer is that, the substance to be analyzed is usually a mixture of cations and anions, and most cations and anions can only be analyzed fully when not mixed with others. For example, you cannot carry out a confirmatory test on calcium ions when mixed with barium ions in the same solution. This is the same case even for almost all other cations. A cation must therefore be isolated first from other 'disturbing' before specific confirmatory tests are carried out on it.

How do we separate these ions? There are three substances used when separating ions. These are: Water, Sodium hydroxide solution and ammonia solution.

1. **Separation using water**
   Water is used when the mixture to be separated consists of an insoluble (in water) compound and a soluble compound. E.g. $CaCO_3$ and $Ba(NO_3)_2$, CuO and $ZnSO_4$, $NiCO_3$ and $MnSO_4$, etc.
   Separation procedure for the $CaCO_3$ / $Ba(NO_3)_2$ mixture

TEST	OBSERVATION	DEDUCTION
To 2 spatula endfuls of the sample (mixture), add some water (e.g. 5cm^3) and shake.	Partially dissolves forming a white suspension	Mg^{2+}, Ba^{2+}, Ca^{2+}, Pb^{2+}, Al^{3+}, Zn^{2+}, Sn^{2+}, Sn^{4+} present
Filter and keep both the filtrate and residue.	A colourless filtrate	Mg^{2+}, **Ba^{2+}**, Ca^{2+}, Pb^{2+}, Al^{3+}, Zn^{2+}, Sn^{2+}, Sn^{4+}, NH_4^+ present
	A white residue	Mg^{2+}, Ba^{2+}, **Ca^{2+}**, Pb^{2+}, Al^{3+}, Zn^{2+}, Sn^{2+}, Sn^{4+} present

The two compounds have now been separated. The filtrate consists of $Ba(NO_3)_2$ solution, while the residue consists of $CaCO_3$. Individual tests for the cations and anions can now be carried out without interference. Before carrying out tests on the Ca^{2+} ions, the solid $CaCO_3$ must be washed and then dissolved in a suitable acid first.

TEST	OBSERVATION	DEDUCTION
Wash the residue and dissolve it in a minimum amount of dilute nitric acid.	Bubbles of a colourless gas that turns moist blue litmus paper red and lime water milky	CO_2 gas given off :. CO_3^{2-} confirmed present
	The white solid dissolves to form a colourless solution	Mg^{2+}, Ba^{2+}, Ca^{2+}, Pb^{2+}, Al^{3+}, Zn^{2+}, Sn^{2+}, Sn^{4+} NH_4^+ present

Note:

- Washing the residue helps to remove some remains of the filtrate from the residue.
- Unless otherwise stated, the residue is usually washed using water.
  Question: Why do we confirm the CO_3^{2-} here?

2. Separation using sodium hydroxide solution

This one is specifically for separating cations and not anions. Sodium hydroxide solution is used when the mixture to be separated is an aqueous solution containing two cations; whereby, on treating the mixture with sodium hydroxide solution dropwise until in excess, one cation will form an amphoteric hydroxide (i.e. a hydroxide which is soluble in excess sodium hydroxide solution) while the other will form a basic hydroxide (i.e. a hydroxide which is insoluble in excess sodium hydroxide solution).

Cations which form amphoteric hydroxides are: Pb^{2+}, Al^{3+}, Zn^{2+}, Sn^{2+}, Sn^{4+}, and Cr^{3+}.

Cations which form basic hydroxides are: Mg^{2+}, Ba^{2+}, Ca^{2+}, Cu^{2+}, Fe^{2+}, Ni^{2+}, Fe^{3+}, Mn^{2+} and Co^{2+}.

Consider a solution containing calcium ions and lead (II) ions.

TEST	OBSERVATION	DEDUCTION
To about 1 cm^3 of solution in a test tube, add sodium hydroxide solution dropwise until in excess.	A white precipitate insoluble in excess	Mg^{2+}, **Ca^{2+}** present
Filter and keep both the filtrate and residue	A colourless filtrate	**Pb^{2+}**, Zn^{2+}, Al^{3+} present in filtrate
	A white residue	Mg^{2+}, **Ca^{2+}** present in residue

Note:

(i) Ca^{2+} forms a white precipitate insoluble in excess alkali,

$Ca^{2+}(aq) + 2OH^-(aq) \longrightarrow Ca(OH)_2(s)$

while Pb^{2+} forms a white precipitate soluble in excess alkali,

$Pb^{2+}(aq) + 2OH^-(aq) \longrightarrow Pb(OH)_2(s)$

$Pb(OH)_2(s) + 2OH^-(aq) \longrightarrow [Pb(OH)_4]^{2-}(aq)$

The overall observation is 'white precipitate insoluble in excess'

(ii) Filtration **separates** the soluble complex solution $[Pb(OH)_4]^{2-}$ (as filtrate) from the precipitate $Ca(OH)_2$ (as residue).

(iii) To obtain $Ca^{2+}(aq)$ from the residue, the residue is dissolved in a suitable dilute acid

$Ca(OH)_2(s) + 2H^+(aq) \longrightarrow Ca^{2+}(aq) + 2H_2O(l)$

form a hydroxide which is soluble in excess ammonia solution while the other will form a hydroxide which is insoluble in excess ammonia solution.

Cations which form hydroxides that are soluble in excess ammonia solution are: Zn^{2+}, Cu^{2+} and Ni^{2+}

Cations which form hydroxides that are insoluble in excess ammonia solution are: Mg^{2+}, Ba^{2+}, Ca^{2+}, Pb^{2+}, Al^{3+}, Sn^{2+}, Sn^{4+}, Cr^{3+}, Fe^{2+}, Fe^{3+}, Mn^{2+} and Co^{2+}.

Consider a solution containing zinc ions and iron(III) ions.

TEST	**OBSERVATION**	**DEDUCTION**
To about 1 cm^3 of solution in a test tube, add ammonia solution dropwise until in excess.	A brown precipitate insoluble in excess	Fe^{2+} present
Filter and keep both the filtrate and residue	A colourless filtrate A brown residue	Zn^{2+}present in filtrate Fe^{3+} present in residue

Note:

(i) Fe^{3+} forms a brown precipitate insoluble in excess alkali,

$$Fe^{3+}(aq) + 3OH^-(aq) \longrightarrow Fe(OH)_3(s)$$

while Zn^{2+} forms a white precipitate soluble in excess alkali,

$$Zn^{2+}(aq) + 2OH^-(aq) \longrightarrow Zn(OH)_2(s)$$
$$Zn(OH)_2(s) + 4NH_3(aq) \longrightarrow [Zn(NH_3)_4]^{2+}(aq) + 2OH^-(aq)$$

The overall observation is 'brown precipitate insoluble in excess'

(ii) Filtration **separates** the soluble complex solution $[Zn(NH_3)_4]^{2+}$ (as filtrate) from the precipitate $Fe(OH)_3$ (as residue).

(iii) To obtain $Fe^{3+}(aq)$ from the residue, the residue is washed and then dissolved in a suitable dilute acid

$$Fe(OH)_3(s) + 3H^+(aq) \longrightarrow Fe^{3+}(aq) + 3H_2O(l)$$

TEST	OBSERVATION	DEDUCTION
Wash the residue and dissolve it in a minimum amount of dilute nitric acid	The brown solid dissolves to form a brown solution	Fe^{3+} present.

(iv) The Zn in the filtrate above is in the form of $[Zn(NH_3)_4]^{2+}$ and therefore cannot undergo the usual reactions of Zn^{2+}. To obtain $Zn^{2+}(aq)$ from the filtrate, it must be treated with a suitable acid until the solution becomes **just acidic** as shown in the table below.

(b) Place another sample of Y in a test tube. Add dilute nitric acid drop wise until there is no further change. Divide the solution into **four** portions.		
(i) To the first portion add sodium hydroxide solution drop wise until in excess. Warm the mixture.		
(ii) To the second portion add 4 drops of dilute sulphuric acid.		
(iii) To the third portion add ammonium hydroxide solution drop wise until in excess.		
(iv) To the fourth portion add lead nitrate solution. Heat the solution and allow it to cool.		
(v) To the fifth portion carry out another test to confirm the anion in Y. TEST; …………………………………… …………………………………… …………………………………… …………………………………… …………………………………… ……………………………………		

Cations in Y are ……………………… and ……………………………

Anions in Y are ………………………… and ……………………………

PRACTICAL 2

You are provided with substance Z which contains **two** cations and **two** anions. You are to identify the cations and anions in Z. Carry out the tests below on Z and record your observations and deductions in the table. Identify any gas (es) evolved.

TESTS	OBSERVATIONS	DEDUCTIONS
(a) Heat one spatula end- ful of Z strongly in a dry test tube until there is no further change.		
(b) To a spatula end-ful of Z add 2-3 drops of concentrated sulphuric acid and warm.		
(c) To two spatula end-ful of Z add dilute nitric acid until there is no further change. Add sodium hydroxide solution drop-wise until in excess. Filter and keep the filtrate. Wash the residue.		
(d) To the filtrate add dilute nitric acid until the solution is just acidic. Divide the resultant solution into **four** parts.		
(i) To the first part of the acidic solution add sodium hydroxide solution drop-wise until in excess.		
(ii) To the second part of the acidic solution add aqueous ammonia drop wise until in excess.		

(iii) To the third part of the acidic solution add dilute sulphuric acid.		
(iv) Use the fourth part of the acidic solution to carry out a test of your choice to confirm one of the cations in Z. **Test** …………………………………… …………………………………… …………………………………… …………………………………… …………………………………… …………………………………...		
(e) To two spatula end-fuls of Z add about 5.0 cm^3 of water, shake and filter. Divide the filtrate into five parts. (i) To the **first** part of the filtrate add sodium hydroxide solution drop- wise until in excess.		
(ii) To the second part of the filtrate add aqueous ammonia drop-wise until in excess.		
(iii) Use the third part of the filtrate to carry out a test of your own choice to confirm one of the cations in Z. **TEST;** …………………………………… …………………………………… …………………………………… …………………………………… ……………………………………		
(iii) To the fourth part of the filtrate add 2-3 drops of lead (II) nitrate solution and heat.		

Divide the filtrate into three portions. (i) To the first portion add 3-4 drops of hydrogen peroxide.		
(ii) To the second portion add silver nitrate solution.		
(iii) To the third portion add lead nitrate solution.		
(c) Place the residue in a boiling tube. Add dilute nitric acid drop-wise until there is no further change. Add sodium hydroxide solution drop-wise until in excess. Filter the mixture and keep both the residue and filtrate.		
(i) To the first portion of the filtrate add sodium hydroxide solution drop-wise until in excess.		
(ii) To the second portion of the filtrate add ammonium hydroxide solution drop-wise until in excess.		
(iii) To the third portion of the filtrate add 3-4 drops of dilute sulphuric acid.		
(iv) To the fourth portion of the filtrate add 3-4 drops of potassium chromate solution followed by sodium hydroxide solution drop-wise until in excess.		
(d) Transfer the residue into a test tube. Add dilute nitric acid drop-wise until there is no further change. Divide the solution into three portions.		
(i) To the first portion add sodium hydroxide solution drop-wise until in excess.		

(ii)To the second portion add potassium hexacyanoferrate (II) solution.		
(iii) To the third portion add potassium iodide solution		

Cations in Q ………………………………………………….

Anions in Q ………………………………………………….

PRACTICAL 5

You are provided with substance Y which contains **one** cation and **one** anion.You are to identify the cation and anion in Y .Carry out the following tests below on Y and record your observations and deductions in the table.

TESTS	OBSERVATIONS	DEDUCTIONS
(a)Place two spatula end-ful of Y in a test tube. Add 5.0 cm^3 of water and shake the mixture.		
(i) To a little amount of the solution add lead nitrate solution followed by dilute nitric acid.		
(ii) To a little amount of the solution carry a test to confirm the anion in Y. TEST;		
(i)To a little amount of the solution add ammonium hydroxide solution drop-wise until in excess.		
(ii) To the remaining amount of the solution add sodium hydroxide solution drop-wise until in excess. Add 5-6 drops of hydrogen peroxide solution. Warm the mixture and allow the solution to cool. Divide the solution into six portions		
(i) To the first portion add 3-4 drops of lead(II) nitrate solution.		

PRACTICAL 6

You are provided with substance R which contains two cations and two anions. You are to identify the cations and anions in R .Carry out the following tests below on R and record your observations and deductions in the table. Identify any gas(es) evolved.

TESTS	OBSERVATIONS	DEDUCTIONS
(a) (i) Place one spatula end-ful of R in a test tube. Add 3-4 drops of concentrated sulphuric acid and warm the mixture.		
(b) (i) Place two spatula end-full of substance R in a boiling tube. Add 5.0 cm^3 of water. Shake the mixture.		
(ii) To a little amount of the solution add iron (III) chloride solution and warm the mixture.		
(c) To the remaining amount of the solution in (b) i add sodium hydroxide solution drop-wise until in excess. Filter the mixture. Keep both the filtrate and residue.		
(c) To the filtrate add dilute nitric acid drop-wise until when the solution is **just** acidic.Divide the resultant solution into six portions.		
(i) To the first portion add sodium hydroxide solution drop-wise until in excess.		

(iii) To the third part carry out a test to confirm one of the cation in substance R. TEST;		

Cations in R………………………………………………………………
Anions in R …………………………………………………………..

PRACTICAL 7

You are provided with substance T which contains two cations and two anions. You are to identify the cations and anions in T. Carry out the following tests below on T and record your observations and deductions in the table. Identify any gas(es) evolved

TESTS	OBSERVATIONS	DEDUCTIONS
(a) Place a half spatula endful of substance T in a test tube and heat.		
(b) Place a half spatula endful of substance T in a test tube. Add 3-4 drops of concentrated sulphuric acid. Warm the mixture.		
(c) Place two spatula endfuls of substance T in a test tube. Add 5.0cm^3 of water and shake the mixture. Filter the mixture and keep both the filtrate and residue. Wash the residue.		
Divide the filtrate into three portions. (i) To the first portion add 3-4 drops of lead (II) nitrate solution followed by 2-3 drops of dilute nitric acid.		
(ii) To the second portion add 3-4 drops of chlorine water (or half spatula endful of bleaching powder) followed about 1.0cm^3 of carbon tetrachloride.		
(iii) To the third portion add silver nitrate solution		

(f) Transfer the residue into a test tube. Add dilute nitric acid drop-wise until there is no further change. Divide the solution into three portions.		
(i) To the first portion add sodium hydroxide solution drop-wise until in excess.		
(ii) To the second portion add ammonium hydroxide solution drop-wise until in excess.		
(iii) To the third portion add potassium chromate solution.		
() To the fourth portion carry out a test to confirm the cation in substance T. TEST;		

Cations in T ……………………………………
Anions in T………………………………..

PRACTICAL 8

You are provided with substance F which contains two cations and two anions. You are to identify the cations and anions in F. Carry out the following tests below on F and record your observations and deductions in the table. Identify any gas(es) evolved.

TESTS	OBSERVATIONS	DEDUCTIONS
() Place a half spatula end-ful of substance F in a test tube. Heat gently and then strongly.		
() Place two spatula end-ful of substance F into a test tube. Add 5.0 cm^3 of water and shake the mixture. Divide the solution into six portions.		
() To the first portion add sodium hydroxide solution drop-wise until in excess. Warm the mixture.		
() To the second portion add ammonium hydroxide solution drop-wise until in excess.		
() To the third portion add potassium hexacyanoferrate (II) solution		

solution dropwise until in excess.		
(vii) To the seventh part of the filtrate add 2-3 drops of lead (II) nitrate solution.		
(d) Wash the residue with water and then dissolve it in dilute hydrochloric acid. Divide the acidic solution into three parts.		
(i) To the first part of the acidic solution add dilute sodium hydroxide solution dropwise until in excess.		
(ii) To the second part of the acidic solution add dilute ammonia solution dropwise until in excess.		
(iii) To the third part of the acidic solution add a small amount of solid ammonium chloride. Shake and add 2-3 drops of disodium hydrogen phosphate solution followed by dilute ammonia solution dropwise until in excess.		

(e) Cations in K are ………………………………………………

and the anions in K are …………………………………...

(b) To two spatula ends-full of W, add about 10 cm^3 of water. Shake well and filter. Keep both the filtrate and residue. Divide the filtrate into four portions.		
(i) To the first portion add dilute sodium hydroxide dropwise until in excess.		
(ii) To the second portion add a half a spatula endful of solid ammonium chloride and shake to dissolve. Then add 2-3 drops of aqueous disodium hydrogen phosphate, followed by aqueous ammonia dropwise until in excess.		
(iii) To the third portion add dilute nitric acid followed by lead (II) nitrate solution and heat.		
(iv) Use the fourth portion to carry out a test of your choice to confirm one of the anions in W. ……………………… ……………………… ………………………. ………………………. ………………………. ……………………….		
(c) Dissolve the residue in about 5 cm^3 of dilute hydrochloric acid. Divide the solution into two parts.		
(i) To the first part add dilute sodium hydroxide solution dropwise until in excess.		

(ii) To the second part add aqueous ammonia dropwise until in excess. Add 2-3 drops of dimethyl glyoxime to the mixture.		

Identify:

the cations in W ……………………………………………..

the anions in W ……………………………………………...

PRACTICAL 11

You are provided with substance Q which contains two cations and two anions. Carry out the following tests on Q and identify the cations and anions in it. Identify any gases evolved. Record your observations and deductions in the table below.

TEST	OBSERVATION	DEDUCTION
(a) Heat a spatula endful of Q strongly in a dry test tube.		
(b) Shake two spatula endfuls of Q with 10 cm^3 of water and filter (keep both the filtrate and residue).		
(c) Divide the filtrate into five parts. (i) To the first part add dilute sodium hydroxide solution dropwise until in excess.		
(ii) To the second part add aqueous ammonia dropwise until in excess.		

(iii) To the third part add concentrated potassium thiocyanate.		
(iv) To the fourth part add 2-3 drops of lead(II) nitrate solution.		
(v) Use the fifth part to carry out a test of your own to confirm the anion in Q. ……………………… …………………….. ……………………… ……………………… ………………………		
(d) Wash the residue and transfer it to a test tube, add dilute hydrochloric acid dropwise until there is no further change and divide the product into 4 parts. (i) To the first part add dilute sodium hydroxide solution dropwise until in excess.		
(ii) To the second part add aqueous ammonia solution dropwise until in excess.		
(iii) To the third part add a few drops of potassium iodide solution.		
(iv) To the fourth part add a few drops of potassium thiocyanate solution.		

(a) The cations in Q are …………………………………………………..

(b) The anions in Q are …………………………………………………..

PRACTICAL 12

You are provided with substance, W, which contains two cations and two anions. Carry out the following tests on W and identify the cations and anions in it. Identify any gases evolved. Record your observations and deductions in the table below.

TEST	OBSERVATION	DEDUCTION
(a) Heat two spatula endfuls of W strongly in dry test tube.		
(b) To two spatula endfuls of W in a dry test tube, add 5 drops of concentrated sulphuric acid and warm.		
(c) Dissolve three spatula endfuls of W in about 5 cm^3 of water to make a solution. (i) Use 1 cm^3 of the solution of W to carry out a test of your own choice to confirm one of the anions in W. ………………………. ……………………….. ………………………. ………………………. ………………………		
(ii) To the remaining solution of W add dilute sodium hydroxide solution dropwise until there is no further change. Filter and keep both the filtrate and the residue.		

(d) Add dilute hydrochloric acid dropwise to the filtrate until the solution is just acidic. Divide the solution into four portions. (i) To the first portion of the acidified filtrate, add dilute sodium hydroxide solution dropwise until in excess.		
(ii) To the second portion of the acidified filtrate, add potassium iodide solution.		
(iii) To the third portion of the acidified filtrate, add 5 drops of litmus solution followed by dilute ammonia solution dropwise until in excess.		
(iv) To the fourth portion of the acidified filtrate, add 5 drops of barium nitrate solution.		
(e) Wash the residue with water and dissolve in dilute hydrochloric acid. Divide the acidic solution into three portions. (i) To the first portion of the acidic solution, add sodium hydroxide solution dropwise until in excess.		
(ii) To the second portion of the acidic solution, add dilute ammonia solution dropwise until in excess.		

(iii) Use the third portion of the acidic solution to carry out a test of your own to confirm one of the cations in W. ……………………… ……………………….. ………………………. ………………………. ………………………		

(f) The cations in W are ……………………………………………………………

(g) The anions in W are …………………………………………………………….

PRACTICAL 13

You are provided with substance, P, which contains two cations and two anions. Carry out the following tests on P and identify the cations and anions in it. Identify any gases evolved. Record your observations and deductions in the table below.

TESTS	OBSERVATIONS	DEDUCTIONS
(a) Heat one spatula endful of P in a dry test tube.		
(b) To two spatula endfuls of P, add about 5 cm^3 of water. Shake vigorously and filter. Divide the filtrate into 5 parts. Keep the residue.		
(i) To the first part of the filtrate, add dilute sodium hydroxide solution dropwise until in excess.		

(ii) To the second part of the filtrate, add dilute ammonia solution dropwise until in excess.		
(iii)To the third part of the filtrate, add 3-4 drops of concentrated nitric acid followed by 2-3 drops potassium thiocyanate.		
(iv) To the fourth part of the filtrate, add 2-3 drops of lead(II) nitrate solution. Heat and allow to cool.		
(v) Use the fifth part of the filtrate to carry out a test of your own choice to confirm one of the anions in P. ………………………. ……………………….. ………………………. ………………………. ………………………		
(c) Wash the residue with water and dissolve it in dilute hydrochloric acid. Divide the resultant solution into three portions.		
(i) To the first portion of the solution, add dilute sodium hydroxide solution dropwise until in excess.		
(ii)To the second portion of the solution, add dilute ammonia solution dropwise until in excess.		
Use the third portion of the solution to carry out a test of your own choice to confirm one of the cations in P. ………………………. ……………………….. ………………………. ………………………. ……………………….		

(d) Identify the:

(i) cations in P ……………………………………………………

(ii) anions in P ……………………………………………………

PRACTICAL 14

You are provided with substance, T, which contains two cations and two anions. Carry out the following tests on T and identify the cations and anions in it. Identify any gases evolved. Record your observations and deductions in the table below.

TESTS	OBSERVATIONS	DEDUCTIONS
(a) Heat one spatula endful of T in a dry test tube.		
(b) To two spatula endfuls of T add about 5 cm^3 of water and shake vigorously. Add ammonia solution dropwise until in excess. Filter and keep both the filtrate and residue.		
(c) To the filtrate, add dilute nitric acid until the solution is just acidic. Divide the acidic solution into seven portions.		
(i)To the first portion of the acidified filtrate, add sodium hydroxide solution dropwise until in excess.		

(ii)To the second portion, add ammonia solution dropwise until in excess.		
(iii) Use the third portion to carry out a test of your own choice to confirm the cation in the filtrate. ……………………… ……………………….. ……………………… ……………………… ………………………		
(iv)To the fourth portion, Add 2-3 drops of lead(II) nitrate solution.		
(v)To the fifth portion, add 3-4 drops of barium nitrate solution.		
(vi)To the sixth portion, add 3-4 drops of silver nitrate solution followed by excess ammonia solution.		
(vii)To the seventh portion, add one spatula endful of bleaching powder followed by about 1 cm^3 of carbon tetrachloride. Shake the mixture and allow to stand.		
(d) Wash the residue and dissolve it in dilute hydrochloric acid. Divide the acidic solution into four portions. (i)To the first portion, add sodium hydroxide solution dropwise until in excess.		

(ii)To the second portion, add ammonia solution dropwise until in excess.		
(iii)To the third portion, add potassium iodide solution.		
(iv)Use the fourth portion to carry out a test of your own choice to confirm the cation in the residue. ………………………. ……………………….. ………………………. ………………………. ……………………….		

PART III: ORGANIC CHEMISTRY

CHAPTER 13: ORGANIC QUALITATIVE ANALYSIS

The aim of organic qualitative analysis is to investigate an organic compound and then comment on its nature.

Below are the five parameters considered when commenting on the nature of an organic compound.

- Aromatic or aliphatic
- Saturated or unsaturated
- Functional group / homologous series e.g. alcohol, aldehyde, ketone, carboxylic acid, alkene, alkyne, amine etc.
- Class in a particular homologous series e.g. primary, secondary or tertiary for alcohols, alkyl halides and amines
- Other group(s) e.g. methyl group, chloro group, bromo group, iodo group, etc.

Below are the 20 common reagents / tests, the possible observations from them and the corresponding deductions for each observation.

Test	Possible observations	Corresponding deductions
1. Burn a small amount of the substance on a spatula end or crucible lid.	A blue non-sooty flame	Aliphatic compound with a **low carbon content or low molecular mass or low C:H ratio**.
	A yellow non-sooty flame	Aliphatic compound with a **low molecular mass** (or **low C: H ratio**).
	A yellow sooty flame	Aromatic compound
2. To a small amount of the substance add a little water and shake	Miscible (if liquid) / soluble (if solid)	Polar organic compound with a low molecular mass, e.g. alcohol, carboxylic acid, carbonyl compound, etc.
	Immiscible (if liquid)/ insoluble (if solid)	Non- polar organic compound.
	Partially miscible (if liquid) / partially soluble (if solid) [but dissolves on heating]	Polar organic compound with a high molecular mass, e.g. aromatic carboxylic acid (where applicable)
3. Test the solution with litmus paper/ solution	Solution turns blue litmus red	**Acidic** compound e.g. phenol or carboxylic acid

	No observable change	Hydroxyl group absent.
20. Add soda lime and heat	A colourless vapour which burns with a non- sooty flame.	Alkane evolved ∴ Aliphatic carboxylic acid present.
	A colourless vapour which burns with a yellow sooty flame-	Aromatic hydrocarbon produced. ∴ Aromatic carboxylic acid present.
	No observable change	Carboxylic acid absent.

PRACTICAL 1

You are provided with an organic substance D. You are required to determine the nature of D. Carry out the following tests on D and record your observations and deductions in the table below.

TEST	OBSERVATION	DEDUCTION
(a) Burn little amount of D on a spatula end.		
(b) To 3-4 drops of D add little amount of water. Shake the mixture and test the solution with litmus paper.		
(c) To 3-4 drops of D add 3 drops of acidified potassium dichromate solution. Heat the mixture.		
(d) To 2-3 drops of D add 3 drops of bradys reagent.		
(e) To 3-4 drops of D add an equal volume of ethanoic acid then followed by 2 drops of concentrated sulphuric acid. Heat the mixture and pour the contents in a beaker of cold water.		
(f) To 3-4 drops of D add 5 drops of concentrated sulphuric acid. Heat the mixture and pass the vapour formed through acidified potassium permanganate solution.		
(g) To 3-4 drops of D add 4 drops of Lucas reagent. Leave the solution to stand.		
(h) To 3-4 drops of D add 5-6 drops of iodine solution followed by drop wise addition of sodium hydroxide solution until when the brown colour of the solution is just discharged.		

Name the functional group in D ……………………………………..

Comment nature of D……………………………………………………………

PRACTICAL 2

You are provided with an organic substance E. You are required to determine the nature of E. Carry out the following tests on E and record your observations and deductions in the table below.

TEST	OBSERVATION	DEDUCTION
(a) Burn little amount of E on spatula end.		
(b) To 3-4 drops of E add little amount of water. Shake the mixture and test the solution with litmus paper.		
(c) To 3-4 drops of E add 4-5 drops of concentrated sulphuric acid. Heat the mixture and pass the vapour through acidified potassium permanganate solution.		
(d) To 3-4 drops of E add an equal volume of acidified potassium dichromate. Heat the mixture and pass the vapour through Brady's reagent.		
(e) To 3-4 drops of E add an equal volume of acidified potassium dichromate solution. Heat the mixture and allow the solution to cool. Divide the solution into **two** portions.		
(i) To the first portion add 3 drops of bradys reagent.		

(ii) To the second portion add 3-4 drops of silver nitrate solution followed by drop wise addition of ammonium hydroxide solution until when the precipitate just dissolves. Warm the mixture in a water bath.		
(f) To 3-4 drops of E add 5-6 drops of iodine solution followed by drop wise addition of sodium hydroxide solution until when the brown colour of the solution is just discharged.		

Name the functional group in E …………………………………………..

Comment on nature of substance E………………………………………….

PRACTICAL 3

You are provided with an organic substance G. You are required to determine the nature of G. Carry out the following tests on G and record your observations and deductions in the table below

TEST	OBSERVATION	DEDUCTION
(a) Burn little amount of G on spatula end.		
(b) To about 1.0 cm^3 of G add 3.0 cm^3 of water. Shake the mixture and test the solution with litmus paper. Divide the solution into **four** portions.		
(i) To the first portion add 2-3 drops of neutral iron (III) chloride solution.		
(ii) To the **second** portion add 2-3 drops of sodium carbonate solution.		
(iii) To the **third** portion add Brady's reagent.		
(iv) To the **fourth** portion of the mixture add 2-3 drops of acidified potassium dichromate solution and heat the mixture.		
(c) To about 1 cm^3 of G add 4-5 drops of Lucas reagent.		

(d) To about 1.0 cm^3 of G add an equal volume of ethanoic acid followed by 4-5 drops of concentrated sulphuric acid. Heat the mixture and pour the mixture in a beaker of water.		
(e) To about 1.0 cm^3 of G add about an equal volume of concentrated sulphuric acid. Heat the mixture and pass the vapour formed through acidified potassium permanganate solution.		

Comment on nature of G

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PRACTICAL 4

You are provided with an organic substance J. You are required to determine the nature of J. Carry out the following tests on J and record your observations and deductions in the table below.

TEST	OBSERVATION	DEDUCTION
(a) Burn small amount of J on a spatula end.		
(b) To 2-3 drops of J add little amount of water. Shake the mixture. Add 3 drops of litmus solution.		
(c) To 2-3 drops of J add 3 drops of sodium carbonate solution.		
(d) To 2-3 drops of Y add 3 drops of iron (III) chloride solution.		
(e) To 3-4 drops of J add 3drops of acidified potassium dichromate solution. Heat the mixture.		
(f) To 2-3 drops of J add 3 drops of Brady's reagent.		

(g) To 3-4 drops of J add an equal volume of ethanoic acid followed by three drops of concentrated sulphuric acid. Heat the mixture and pour the contents in a beaker of cold water.		
(h) To 3-4 drops of J add 5-6 drops of iodine solution followed by drop wise addition of sodium hydroxide solution until when the brown colour of the solution is just discharged.		

Name the functional in J …………………………………………………………………………..

Comment on nature of J …………………………………………………………………………

PRACTICAL 5

You are provided with an organic substance M. You are required to determine the nature of M. Carry out the following tests on M and record your observations and deductions in the table below.

TEST	OBSERVATION	DEDUCTION
(a) Burn small amount of M on spatula end.		
(b) To 2-3 drops of M add little amount of water. Add 2 drops of litmus solution.		
(c) To 2 drops of M add 3 drops of sodium carbonate solution.		
(d) To 2 drops of M add 3 drops of iron(III) chloride solution.		
(e) To 2-3 drops of M add 3-4 drops of acidified potassium dichromate solution. Heat the mixture.		
(f) To 3-4 drops of M add 4-5 drops of Brady's reagent.		

(g) To 4-5 drops of M add 5-6 drops of iodine solution followed by drop wise addition of sodium hydroxide solution until when the brown colour of the solution is just discharged.		

Name the functional group in M …………………………………………………………

Comment on nature of M ………………………………………………………………

PRACTICAL 6

You are provided with an organic substance Q. You are required to determine the nature of Q . Carry out the following tests on Q and record your observations and deductions in the table below.

(a) Burn little amount of Q on a spatula end.		
(b) Place about three spatula end-ful of Q in a test tube and add about 5 cm^3 of water. Shake the mixture and test the solution with litmus. Divide the solution into **seven** portions.		
(c) To the first portion add sodium carbonate solution.		
(d) To the second portion add iron (III) chloride solution.		
(e) To the third portion add 3-4 drops of acidified potassium dichromate solution. Heat the mixture.		
(f) To the fourth portion add Brady's reagent.		
(g) To the fifth portion add 3-4 drops of Fehling's solution. Warm the mixture.		
(h) To the sixth portion add 3 drops of sodium hydroxide solution followed 5-6 drops		

of silver nitrate solution then add ammonium hydroxide solution drop wise until when the precipitate just dissolves. Warm the mixture in water bath for about five minutes.		
(i) To the seventh portion add 5-6 drops of iodine solution followed by sodium hydroxide solution drop wise until when the brown colour of the solution is just discharged.		

Name the functional group in Q …………………………………………………………………….
Comment on nature of Q
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PRACTICAL 7

You are provided with an organic substance R. You are required to determine the nature of R. Carry out the following tests on R and record your observations and deductions in the table below.

TEST	OBSERVATION	DEDUCTION
(a) Burn small amount of R on a spatula end.		
(b) To 2-3 drops of R add little amount of water. Shake the mixture and add 3 drops of universal indicator.		
(c) To 2-3 drops of R add 3-4 drops of iron (III) chloride solution.		
(d) To 2-3 drops of R add a half spatula end ful of sodium hydrogen carbonate.		
(e) To 3-4 drops of R add an equal volume of butan-1-ol followed by 3 drops of concentrated sulphuric acid. Heat the mixture and pour the contents in a beaker of cold water.		
(f) To 3-4 drops of R add 3 drops of acidified potassium permanganate solution. Warm the mixture.		
(g) To 3-4 drops of R add silver nitrate solution followed by drop wise addition of ammonium hydroxide solution until when the precipitate just dissolves. Warm the mixture in hot water.		
(g) To 3-4 drops of R add Fehling's solution and warm the mixture.		

Name the functional group in R…………………………………………………………………….

Comment on nature of R

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PRACTICAL 8

You are provided with an organic substance S.You are required to determine the nature of S. Carry out the following tests on S and record your observations and deductions in the table below.

TEST	OBSERVATION	DEDUCTION
(a) Burn small amount of S on a spatula end.		
(b) To 2-3 drops of S add little amount of water. Shake the mixture. Add 3 drops of universal indicator.		
(c) To 2-3 drops of S add 3-4 drops of iron (III) chloride solution.		
(d) To 2-3 drops of S add half spatula end-ful of sodium hydrogen carbonate.		
(e) To 3-4 drops of S add an equal volume of Butan-1 -ol followed by 3 drops of concentrated sulphuric acid. Heat the mixture and pour the contents in a beaker of cold water.		
(f) To 3-4 drops of S add 3 drops of acidified potassium permanganate solution. Heat the mixture.		
(g) To about 5 drops of silver nitrate solution add 3 drops of sodium hydroxide solution followed by ammonia solution drop wise until the precipitate just dissolves. Add 4 drops of S , shake and warm the mixture in a water bath for about five minutes.		

(h) To 3-4 drops of S add 2-3 drops of Fehling’s solution. Warm the mixture.		

Name the functional group in S ………………………………………………

Comment on the nature of S ………………………………………..........

PRACTICAL 9

You are provided with an organic substance T. You are required to determine the nature of T. Carry out the following tests on T and record your observations and deductions in the table below.

TEST	OBSERVATION	DEDUCTION
(a) Burn little amount of T on spatula end.		
(b) To a little amount of T add sodium hydroxide solution.		
(c) To 2 spatula endful of T add about 5cm^3 of water. Warm the mixture. Divide the solution into **five** portions. (In each case if the mixture solidifies **warm** with hot water before carrying out the test)		
(i) To the first portion add 3 drops of iron (III) chloride solution.		
(ii) To the second portion add a half spatula endful of sodium hydrogen carbonate.		
(iii) To the third portion add 3 drops of acidified potassium permanganate solution. Warm the mixture.		
(iv) To the third portion add an equal volume of ethanol followed by 3 drops of concentrated sulphuric acid. Heat the mixture and pour the contents in a beaker of cold water.		

Name the functional group in T…………………………………………………..

Comment on nature of T…………………………………………………………..

Practical 10

You are provided with an organic substance M. You are required to determine the nature of M. Carry out the following tests on M and record your observations and deductions in the table below.

TESTS	OBSERVATIONS	DEDUCTIONS
(a) Burn a small amount of M on the tip of a dry spatula or porcelain.		
(b) (i) Shake a half spatula endful of M with about 2 cm^3 of dilute sodium hydroxide solution.		
(ii) Shake a half spatula endful of M with about 2 cm^3 of water and add 2-3 drops of litmus solution.		
(c) Shake a spatula endful of M with about 5 cm^3 of water and divide the solution into three parts.		
(i) To the first part of the solution add 2-3 drops of sodium hydrogen carbonate solution.		
(ii) To the second part of the solution add 2-3 drops of 2,4-dinitrophenylhydrazine (Brady's) solution.		
(iii) To the third part of the solution add 2-3 drops of iron(III) chloride solution and warm.		
(d) Dissolve a spatula endful of M, in about 5 cm^3 of water. To the solution add about 1-2 cm^3 of dilute sodium hydroxide solution. Heat the mixture, cool, add 2-3 drops of silver nitrate solution and		

filter. Keep both the filtrate and residue.		
(e) To the residue add dilute ammonia solution dropwise until in excess.		
(f) To the filtrate add about equal volume of ethanol followed by 3-4 drops of concentrated sulphuric acid. Heat the mixture and cool.		

(g) State the nature of M.

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Practical 11

You are provided with an organic substance B .You are required to determine the nature of B. Carry out the following tests on B and record your observations and deductions in the table below.

TESTS	OBSERVATIONS	DEDUCTIONS
(a) Burn a small amount of B on a spatula end or porcelain dish.		
(b) Shake 2-3 drops of B with about 2 cm^3 of water and allow to stand.		
(c) Add 2-3 drops of potassium dichromate to a few drops of B and warm.		
(d) Add 2-3 drops of 2,4-dinitrophenylhydrazine solution (Brady's reagent) to about 0.5 cm^3 of B.		

(e) Dissolve 4-5 drops of B in about 1 cm^3 of methanol then add about 1 cm^3 of dilute sodium hydroxide solution followed by iodine solution until the colour of iodine persists then warm and allow to stand.		

(f) Comment on the nature of B
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Practical 12

You are provided with an organic substance T .You are required to determine the nature of T. Carry out the following tests on T and record your observations and deductions in the table below.

TESTS	OBSERVATIONS	DEDUCTIONS
(a) Burn a small amount of T on a spatula end or on a crucible lid.		
(b) To a spatula endful of T in a test tube, add 3 cm^3 of sodium hydroxide solution followed by dilute sulphuric acid.		
(c) To a spatula endful of T in a test tube, add about 4 cm^3 of water, warm the mixture and test the solution with litmus. Divide the solution into five portions.		
(i)To the first portion, add a half spatula endful of sodium carbonate.		
(ii) To the second portion, add neutral iron(III) chloride solution		

(iii) To the third portion, add 3-4 drops of acidified potassium manganate(VII) solution and warm.		
(iv) To the fourth portion, add 2-3 drops of Brady's reagent.		
(v) To the fifth portion, add Fehling's solution and heat.		
(d) To about 3 cm^3 of silver nitrate solution, add 2 drops of sodium hydroxide solution, followed by ammonia solution until the precipitate just dissolves. Add a spatula endful of T, shake and heat in a water bath for about five minutes.		

(e) Comment on the nature of T.

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Practical 13

You are provided with an organic substance T .You are required to determine the nature of T. Carry out the following tests on T and record your observations and deductions in the table below.

TESTS	OBSERVATIONS	DEDUCTIONS
(a) Burn a spatula endful of T in a porcelain dish or at the end of a spatula.		
(b) Shake two spatula endfuls of T with about 5 cm^3 of water. Warm and test with litmus.		

(c) Dissolve two spatula endfuls of T in about 5 cm^3 of methanol and divide the solution into four parts. (i) To the first part of the solution, add 2-3 drops of iron(III) chloride solution.		
(ii) To the second part of the solution add 2-3 drops of Brady's reagent.		
(iii) To the third part of the solution, add about 1 cm^3 of Fehling's solution and boil.		
(iv) To the fourth part of the solution, add about 1 cm^3 of iodine solution, followed by dropwise addition sodium hydroxide solution until the solution is pale yellow and warm.		

(d) Comment on the nature of T.

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Practical 14

You are provided with an organic substance W. You are required to determine the nature of W. Carry out the following tests on W and record your observations and deductions in the table below.

TESTS	OBSERVATIONS	DEDUCTIONS
(a) Burn a small amount of W on a spatula end or crucible lid.		

(b) Add a spatula endful of W to about 5 cm^3 of dilute sodium hydroxide solution and shake.		
(c) To a spatula endful of W in a test tube add about 5 cm^3 of water. Shake vigorously and warm. Test the solution with litmus paper and divide into three equal portions.		
(i) To the first portion of the solution add 2-3 drops of 2,4-dinitrophenyl hydrazine.		
(ii) To the second portion of the solution, add 2-3 drops of iron(III) chloride solution.		
(iii) To the third portion of the solution, add a spatula endful of sodium hydrogen carbonate.		
(d) To 2 cm^3 of ethanol, add a spatula endful of W and shake. Add 3-4 drops of concentrated sulphuric acid and warm the mixture.		

(e) Comment on the nature of W.

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