PRECISE ADVANCED LEVEL CHEMISTRY PRACTICAL GUIDE

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DECLARATION

Appreciation goes Mr. Nagaya Henry, Mr. muhama Geofrey, Ms Naume Mr. Buyi Dick, Ms Akello, Mr. mukatabala alloycius, Mr. issat Ignatius, Mr. okitoi, Ms Abinyo Racheal, Mr Napokooli Isaac, Mr. mulegi john, Ms amole, Mr. wozemba, for the tremendous, endless effort in shaping and guiding me academic pursuit

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Contents

1.0 VOLUMETRIC ANALYSIS

The term volumetric analysis refers to quantitative chemical analysis carried out by determining the volume of a solution of accurately known concentration (standard solution) which is required to react quantitatively with a measured volume of the solution of a substance to be determined. With this information, various calculations can be made

In volumetric analysis, the reagent of known concentration is called titrant and the substance being titrated is the titrand. In this analysis, a series of titrations are carried out, where by the standard solution is usually added from the burette in small measured quantities to a fixed volume of a solution measured with a pipette. The process of adding a standard solution until the reaction is just complete is termed as titration. The point at which the reaction is just complete is referred to as equivalence point or theoretical end-point

The completion of a titration is detected by some physical change produced by the standard solution itself (e.g faint pink colour formed by potassium permanganate) or more usually by addition of an auxiliary reagent known as indicator which should give a clear visual change (either of colour change or formation of a turbidity) in the liquid titrated. The point at which this occurs is called the end-point. In ideal titration, the visible end-point will coincide with the stoichiometric end-point or theoretical end-point. In practise however, a very small difference usually occurs, this represents a titration error. The indicator and the experimental condition should be selected such that the difference between the visible end-point and the equivalence point is as small as possible.

TITRIMETRIC MTHODS

Titrimetric methods are widely used in chemistry to determine oxidants, reductants, acids, bases, metal ions, etc. Titration is based on a reaction between the analyte (unknown sample) and the regent of known concentration and reaction stoichiometry. The volume or mass of the reagent needed to react completely with a fixed quantity of the analyte is obtained from which the amount of analyte is determined. Titrimetric methods include powerful group of quantitative procedures that are based on measuring the amount of reagent consumed by the analyte. These methods include,

i. **Volumetric titrimetry**. This involves measuring volume of solution of known concentration that is needed to react completely with the sample.
   The reagent of exactly known concentration is refered to as standard reagent**. Titration** is the process in which the standard reagent is added to a solution of the sample until the reaction is judged to be complete**. Back titration** is the process by which the excess of the standard solution used to consume the sample is determined by titration with a second standard solution. Equivalence point and end-point are confused to mean the same but they are totally different**. Equivalence point** is a point in titration when the amount of standard solution added is exactly equal to the amount of the sample whereas **end-poin**t is the point in titration when a physical change occurs that is associated with a condition of chemical equivalence. The two values are usually different and the difference gives the

titration error. Indicators are added to the solution mixtures to produce an observable physical change at the end-point or near equivalence point

ii. **Gravimetric titrimetry**. This involves measuring mass of reagent that reacts completely with the sample.

iii. **Coulometric titrimetry**. Here the reagent is a constant direct electrical current of known magnitude that consumes the sample. Here the time is required, and the total charge to complete the electrochemical reaction

1.1 CLASSIFICATION OF REACTIONS IN VOLUMETRIC ANALYSIS

a) Neutralisation reactions or acidemetry and alkalimetry.

These includes neutralisation of free base or those formed from the salts of weak acids by hydrolysis with standard acid (acidimetry) and titration of free acids or those formed by hydrolysis of salts of weak bases with standard base (alkalimetry). The reaction involves the combination of hydrogen and hydroxide ions to form water

b) Oxidation-reduction reactions

These include all reactions involving changes of oxidation numbers or transfer of electrons among the reacting substances. The standard substances are either reducing or oxidising reagents

c) Precipitation reactions

These depend upon the combination of ions to form a simple precipitate as in the titration of silver ion with a chloride. There is no change in the oxidation state

$$Ag^+_{(aq)} + Cl^-_{(aq)} \longrightarrow AgCl_{(s)}$$

d) Complex formation reactions

These depend upon the combination of ions, other than hydrogen or hydroxyl ions to form a soluble, slightly dissociated ion or compound as in titration of a solution of cyanide with silver nitrate

$$2CN^-_{(aq)} + Ag^+_{(aq)} \longrightarrow AgCN_{(s)}$$

1.2 STANDARD SOLUTIONS

A standard solution is one whose concentration is accurately known. Standard solutions are expressed in terms of molar concentration or molarity; such standard solutions are specified in terms of moles of solute dissolve in 1 litre of solution.

$$\text{Molarity} = \frac{\text{moles of solute}}{\text{volume of solution in litres}}$$

The relative molecular mass for sulphuric acid, H_2SO_4 is calculated from the relative atomic masses as follows

$$RMM = (2 X 1) + (32 X 1) + (16 X 4) = 98$$

It follows that a molar solution of sulphuric acid will contain 98g of H_2SO_4 in 1 litre of solution. So the concentration of any solution can be expressed in terms of molar concentration so long as the mass of a substance in a specified volume is known.

1.2.1 PRIMARY STANDARD

A primary standard is a compound of sufficient purity from which standard solution can be prepared by direct weighing a quantity of it, followed by dilution to give a defined volume of solution. A primary standard should satisfy the following requirements

a) It must have high degree of analytical purity, easy to obtain, easy to dry, and preserve in a pure state.
b) The substance should be unaltered in air during weighing. It should not be hygroscopic, oxidised by air, ore affected by carbon dioxide
c) It should have high relative molecular mass to minimise gravimetric errors (errors due to weighing)
d) The substance should be readily soluble under the condition in which it is employed
e) It should have no interfering products during titration

The reaction with the standard solution should be stoichiometric and practically instantaneous. In practise an ideal standard is difficult to obtain. The substances commonly employed as primary standards include,

i) Acid-base reactions. Sodium carbonate (Na_2CO_3), sodium tetra borate ($Na_2B_4O_7$) and constant point boiling hydrochloric acid.
ii) Precipitation reactions. Silver nitrate, sodium chloride, potassium chloride, and potassium bromide.
iii) Oxidation-reduction reaction. Potassium dichromate ($K_2Cr_2O_7$), potassium iodate (KIO_3), sodium ethanediote ($Na_2C_2O_4$) and arsenic (II) oxide (A_2O_3)

PREPARATION OF A STANDARD SOLUTION OF SODIUM CARBONATE

Apparatus

- Weighing bottle , 250ml beaker, Spatula, Stirring rod, Filter funnel, Balance sensitive to 0.01g, Distilled water

The anhydrous sodium carbonate is best made from sodium hydrogen carbonate

$$NaHCO_{3(s)} \longrightarrow Na_2CO_{3(s)} + H_2O_{(l)} + CO_{2(g)}$$

The anhydrous sodium carbonate formed is very pure and can be used in the ordinary weighing with no appreciable change in composition

Procedure

i) Work out the mass of sodium carbonate needed to make up the solution e.g the mass of sodium carbonate dissolved in 250m to make 0.1M solution is calculated as
1M of sodium carbonate weighs 106g

0.1M of sodium carbonate will weigh 0.1 X 106 = 10.6g

This implies that 1000cm^3 of solution dissolves 10.6g

Therefore 250cm^3 of solution will dissolve $\frac{10.6 \text{ X } 250}{1000}$ = 2.65g

ii) Note the mass of the empty weighing bottle, M_1. Weigh accurately, an approximate mass of sodium carbonate as calculated, M_2 into the weighing bottle. Tap off the sodium carbonate into a 400ml capacity beaker and then weigh the bottle again, M_3. The actual mass is obtained by difference to minimise gravimetric errors, $M_2 – M_3$

iii) Dissolve the sample carefully in about 125ml of distilled water swirl for homogeneity and transfer using a funnel to a clean volumetric flask.

iv) Remove the funnel and carefully add distilled water to the mark. All the solution to mix well by shaking the flask. The sample has been dissolved in 250ml of the solution, the actual concentration of the solution is $\frac{(M2-M3) \text{ X } 1000}{106 \text{ X } 250}$ M

PREPARATION OF APPROXIMATELY 0.1M SULPHURIC ACID AND STANDARDISE IT AGAINST STANDARD SODIUM CARBONATE SOLUTION

a) Diluting the acid. Pure concentrated sulphuric acid contains 98% by mass of sulphuric acid.

i) Take 5.5 – 6.0 cm^3 (care) of concentrated sulphuric acid in a small measuring cylinder, pour it, with stirring into about 100cm^3 of cold distilled water in a beaker

ii) Pour this solution into 700ml 0f of cold distilled water in a measuring cylinder of capacity 1000ml

iii) Wash out the beaker with cold distilled water twice and add to washing to the measuring cylinder, then add distilled water approximately to the mark, stir well. This should give you sulphuric acid of concentration a little more than 0.1M

b) Filling the burette with the diluted acid.

i) Wash the burette with water, test the burette of the glass tap to check whether it allows water to run easily

ii) Wash out the burette twice with few ml of approximately 0.1M sulphuric acid. Allow the acid to run through the tap and pour out the rest of the liquid from the top of burette. This leaves the burette wet with the acid and nothing is left to contaminate it.

iii) Fill the burette with the acid to a level above the zero mark. Open the tap to ensure that the jet is filled with acid and not a bubble. Level of the acid need not to be zero, but must be above it.

iv) Place your eye level with bottom of the meniscus and note the reading of the burette to 0.05cm^3 at least

c) Filling the 23cm^3 pipette with the 0.1M sodium carbonate solution

i) Wash out the pipette (25cm^3) with a little of exactly 0.1M sodium carbonate solution

ii) Draw this solution into the pipette above the mark, and with the mark at the eye level, allow the solution to run out very easily till the lowest level of the meniscus is on the mark.

iii) Allow the liquid to run from the pipette into the conical flask which has been washed out with cold distilled water only

d) Titration of sodium carbonate with the acid

i) Add 2-3 drops of methyl orange indicator in the conical flask. The solution turns yellow

ii) Run the acid into the conical flask 2-3cm^3 at a time with shaking until the liquid in the flask turns pink, and then add drop by drop until the colour of the liquid is orange. This is the end-point of the titration

iii) Repeat the titration to obtain consistent results with minimal errors

Sample reading

Titration number	**1**	**2**	**3**
Final burette reading (cm^3)	24.10	36.10	33.80
Initial burette reading (cm^3)	0.00	12.20	10.00
Volume of acid used (cm^3)	24.10	23.90	23.80

$$\text{Average volume } \frac{23.90+23.80}{2} = 23.85\text{cm}^3$$

Since the first titration may be regarded as trial run, it is usual in such experiments to neglect this reading and the average of the subsequent closest results is taken

Calculations

Sodium carbonate reacts with sulphuric acid according the following equation

$$Na_2CO_{3(aq)} + H_2SO_{4(aq)} \longrightarrow Na_2SO_{4(aq)} + CO_{2(g)} + H_2O_{(l)}$$

1000cm^3 of solution contains 0.1 moles of sodium carbonate

25cm^3 of solution will contain $\frac{25 \text{ X } 0.1}{1000} = 2.5 \text{ X } 10^{-3}$ moles

Since the mole ratio of $Na_2CO_3 : H_2SO_4 = 1:1$

Therefore 23.85cm^3 of solution contains 2.5 X 10^{-3} moles of sulphuric acid

1000cm^3 of solution will contain $\frac{2.5 \text{ X } 10-3 \text{ X } 1000}{23.85} = 1.05\text{M}$

The molarity of sulphuric acid = 1.05M

To make the acid exactly 1M, the volume of 1.05M sulphuric acid required to make 1 litre of 0.1 M H_2SO_4 is given by $V_1 = \frac{0.1 \text{ X } 1000}{0.1058} = 945\text{cm}^3$

Therefore 55cm^3 of distilled water should be added to 945cm^3 of 1.058M H_2SO_4 to make 0.1M

2.0 PH AND CHOICE FOR INDICATOR

PH scale is used for the purpose of comparison of acidity of solutions. PH stands for the power of hydrogen ions. PH is defined as the negative logarithm to the base ten of the molar concentration of hydrogen ions.H_3O^+

$$PH = -\log [H_3O^+_{aq}] \text{ or } \log[\frac{1}{H3O+}]$$

Therefore, acidic solutions will have PH values, which are low. A 0.001M solution of hydrochloric acid has hydrogen ion concentration of 10^{-3}

$$\text{Hence } PH = \log[\frac{1}{H3O+}] = \log[\frac{1}{10-3}] = 3$$

Pure water is neutral and has a PH value of 7

The self-ionisation of water can be written as an acid base equilibrium

$$H_2O_{(l)} + H_2O_{(l)} \longrightarrow H_3O^+_{(aq)} + OH^-_{(aq)}$$

$$\text{For this equilibrium } \frac{[H3O+aq]\ [OH(aq]}{[H2O(l)]}$$

The water concentration is so high compared to those of H_3O^+ and OH^- that can be regarded as constant and the dissociation constant for water is defined as $K_w = [H_3O^+_{aq}]\ [OH_{(aq}]$, the value of K_w at 25^oC is 10^{-14}

$$K_w = [H_3O^+_{aq}]\ [OH_{(aq}] = 10^{-14}$$

$$\text{Hence } [H_3O^+_{aq}] = 10^{-7}$$

And the PH of water is 7. The PH of 7 is defined as neutrality. K_w enables the relationship between $[H_3O^+_{aq}]$ and $[OH_{(aq}]$ to be worked out simply for any aqueous equilibrium. For instance a 0.01M solution of sodium hydroxide has hydroxyl ion concentration of 10^{-2} moles per litre.

$$\text{Concentration of } OH^- \text{ ions } = 0.01 mol\ l^{-1}$$

$$\text{Since } [OH_{(aq}] = [H_3O^+_{aq}] = 10^{-7} mol\ l^{-1}$$

$$[OH_{(aq}]\ [H_3O^+_{aq}] = 10^{-14} mol^2\ l^{-2}$$

$$[H_3O^+_{aq}][10^{-2}] = 10^{-14}$$

$$[H_3O^+_{aq}] = 10^{-12}$$

$$\text{Hence } PH = 12$$

2.1 CHOICE OF INDICATORS

In titration, indicators are chemical substances which functions to show visibly the equivalence point of a reaction has been reached. Usually the colour of the indicator changes sharply with the reagent employed in titration so that the presence of an excess of that reagent may be detected, the stage in titration when the colour change occurs is called the end-point of the titration.

2.2 ACID-BASE INDICATORS

An acid base indicator is a substance that changes colour according to the hydrogen ion concentration of a liquid in which they are placed. They are either weak bases or weak acids and are therefore slightly dissociated when dissolved in water. The colour of the indicator depends on the colour of the un dissociated molecules and the colour of the ions produced. The process is best described in terms of a particular indicator. Phenolphthalein is an indicator which behaves as a weak acid. The formula of phenolphthalein and the products is into which it partially dissociated in water.

$$HIn_{(aq)} + H_2O_{(l)} \longrightarrow H_3O^+_{(aq)} + In^-_{(aq)}$$

In this case, the un dissociated (HIn) are colourless and the anion In^- is pink. The dissociation represents reaction in equilibrium and we can therefore say, that the equilibrium constant for a reaction is given by

$$K_a = \frac{[H3O+(aq)][In-(aq)]}{HIn(aq)}$$

$$\text{Log } K_a = \log \frac{[H3O+(aq)][In-(aq)]}{[HIn(aq)]}$$

$$\text{Log } K_a = \log H3O+] + \log [In-] - \log[HIn]$$

We notice that -log [H3O+] is a definition of PH

$$\text{Log } K_a = -PH + \log [In-] - \log[HIn]$$

$$PH = -\text{Log } K_a + \log \frac{[In-(aq)]}{[HIn(aq)]}$$

Thus can be seen that the hydrogen in concentration will affect the equilibrium position of the indicator, if the hydrogen concentration is high, the equilibrium position will be shifted to the right and the solution becomes pink. The equilibrium will be in its neutral position when the HIn concentration is equal to In^- concentration. At this point, the colour will be mixture of colourless and pink, it appears as pink.

$$\text{If } [In^-] = [HIn]$$

$$\text{Then } \frac{[In-]}{[HIn]} = 1$$

$$\text{Log } \frac{[In-]}{[HIn]} = 0$$

Thus at the balance point, PH = -log k_a or P^{ka}

Below is a list of some of the common indicators in use, with the range of the values over which the colour range takes place.

Indicator	Pk$_a$	Transition interval	Colour in acid	Colour in base
Methyl orange	3.4	3.2-4.4	Red	Yellow
Methyl red	5.0	4.2-6.2	Red	Yellow
Bromothymol blue	7.3	6.0-7.6	Yellow	Blue
Phenolphthalein	9.7	8.2-10.0	colourless	Pink

2.3 TITRATION CURVES

One of the remarkable things about the acid-base titration performed with indicators is the fact that the colour of the indicator changes sharply where only one drop of the acid or base is added at the equivalence point. This is particularly marked in the case of the case of the strong acid and a strong base. Titration curves illustrate this phenomenon. It shows variation of PH with of the mixtures during the course of titration. A typical result for a strong acid and strong base is given by the titration curve for 0.1M by hydrochloric acid titrated with 0.1M sodium hydroxide. A very large change in PH takes place for a small addition of sodium hydroxide near the equivalence point. This accounts for a sharp change in colour of an indicator.

Diagram

Fig (2.1b) shows the results of the similar experiment performed with hydrochloric acid and ammonia solution. The result is typical for a strong acid and weak base. In this case, the first part of the curve is shorter and the equivalence point is at about the PH of 5. Therefore the suitable indicator is methyl orange or methyl red.

Diagram

Fig (2.1c) shows the change in PH when a strong base is titrated against a weak acid. The base is sodium hydroxide and the acid is ethanoic acid. The flat point of the curve is shorter than in the fig (2.1a). But in this case, the equivalence point is on the alkali acid at about PH 9

f ig (2.1d) shows results of titration of a weak acid against a weak base, the acid is ethanoic acid and the base is ammonia solution. It is noticed in this case that there is no straight part of the curve, only a point of inflection. This implies that there is no sudden change in the PH when a small amount of the acid or base is added near the equivalence point. Therefore there is no satisfactory indicator used for this titration.

2.4 REDOX INDICATORS

Redox indicator should mark the sudden change in the oxidation potential in the equivalence point in a redox titration. The ideal redox indicator will be one with an oxidation potential immediate between that of the solution titrated and that of the titrant and should exhibit sharp readily detectable colour change. A redox indicator is a compound which exhibits different colours in the oxidised and reduced forms

Some of the oxidation-reduction indicators include

Indicator	**Colour change**	
	Oxidised form	**Reduced form**
Diphenyl benzidine	Violet	Colourless
Diphenylamine	Violet	Colourless
Starch, I_3^-, KI	Blue	Colourless
Diphenylamminosulphonic acid	Red-violet	Colourless
Methylene blue	Blue	Colourless

Note. Potassium permanganate acts as its own indicator, end-point being the first permanganate pink colour. During titration, the smallest possible quantity of the suitable indicator possible should be used as some are acids and base themselves

3.0 ACID-BASE TITRATION

An acid is compound with a tendency to donate a proton to water molecule.

$$HX_{(aq)} + H_2O_{(l)} \longrightarrow X^-_{(aq)} + H_3O^+_{(aq)}$$

Whereas the base has a tendency to gain a proton from a water molecule

$$B_{(aq)} + H_2O_{(l)} \longrightarrow BH^+_{(aq)} + OH^-_{(aq)}$$

In aqueous alkali, the H_3O^+ ions in the acid combine with the OH^- ions in the alkali to form water

$$H_3O^+_{(aq)} + OH^-_{(aq)} \longrightarrow H_2O_{(l)}$$

In some acid-base reactions, the end-point shown by one indicator denotes different reactions from that shown another. In the case of sodium carbonate titration, phenolphthalein turns colourless on addition of an acid when all the sodium carbonate is converted to sodium hydrogen carbonate.

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

But the carbonate ion has to be converted completely to carbondioxide and water before mthyl orange changes from yellow to red

$$CO_3^{2-}{}_{(aq)} + 2H^+{}_{(aq)} \longrightarrow CO_{2(aq)} + H_2O_{(l)}$$

3.1 STANDARDISATION OF SODIUM HYDROXIDE WITH 0.1M HYDROCHLORIC ACID

Requirements

- 1 Burette (50cm^3), 2 Conical flask (250cm^3), 1 Pipette (25cm^3), 1 Stand, 1 White tile, Phenolphthalein indicator, 100cm^3 of 0.1M hydrochloric acid, Weighing scale sensitive to 0.1g, Weighing bottle, 1.0g sodium hydroxide

Procedure,

a) Weighing accurately 1.0g of pure sodium hydroxide in a weighing bottle or beaker

b) Transfer the content into volumetric flask, add about 100cm^3 of distilled water, shake well to dissolve and then make it to the mark with more distilled water.

c) Shake the flask well, then pipette 25cm^3 of the solution into a conical flask, add 3 drops of phenolphthalein indicator

d) Place the standard hydrochloric acid in the burette, titrate a portion of alkali solution with acid from the burette until the indicator change colour. Repeat the titration until you get a consistent titre. Individual titration should not differ by more than 0.1cm^3. Record your results in the table below.

Results

Mass of weighing bottle + sodium hydroxide...

Mass of the weighing bottle...

Mass of sodium hydroxide...

Volume of the pipette used...

Titration number	1	2	3
Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of hydrochloric acid used (cm^3)			

Titre values used to calculate average volume of acid..

Average volume of a acid..
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a) Write the equation for the reaction
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b) Calculate the number of moles of acid used
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c) Calculate the number of moles of sodium hydroxide that reacted
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d) Calculate the concentration of sodium hydroxide solution in mol dm^{-3}
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e) Calculate the mass of sodium hydroxide dissolved in 1 litre of solution
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3.2 TITRATION OF STANDARD ANHYDROUS SODIUM CARBONATE SOLUTION WITH HYDROCHLORIC ACID

Requirements

- 1 Burette (50cm^3), 2 conical flasks (250 cm^3), 1 volumetric flask(250 cm^3), pipette (25 0r 25 cm^3), white tile, 100 cm^3 of hydrochloric acid solution, 2.0g of anhydrous sodium carbonate, methyl orange indicator,1 weighing bottle, 1 weighing balance sensitive to 0.1g

Procedure

a) Weigh out accurately about 1.5g of pure sodium carbonate in a weighing bottle

b) Transfer it into 250 cm^3 volumetric flask, add about 150 cm^3 of distilled water and shake well to dissolve, and then make to the mark with more water.

c) Shake the flask well, then pipette 25 cm^3 of the solution into a conical flask, then add 3 drops of methyl orange indicator

d) Titrate the portion with the acid from the burette until the indicator changes colour. Repeat the titration to obtain consistent results and tabulate your results,

Results

Mass of the weighing bottle + sodium carbonate...
Mass of the weighing bottle..
Mass of sodium carbonate...
Capacity of the pipette used..

Titration number	**1**	**2**	**3**	**4**
Final burette reading (cm^3)				
Initial burette reading (cm^3)				
Volume of hydrochloric acid used (cm^3)				

Titre values used to calculate average volume of acid...

Average volume of a acid...
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i) Write the equation for the reaction

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ii) Calculate the number of moles of sodium carbonate used

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iii) Calculate the number of moles of acid used

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iv) Calculate the concentration acid solution in mol dm^{-3}

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v) Calculate the mass of hydrochloric acid dissolved in 1 litre of solution

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3.3 TITRATION OF STANDARD SODIUM TETRABORATE WITH HYDROCHLORIC ACID

Equation of reaction,

$$B_4O_7^{2-}{}_{(aq)} + 2H^{+}{}_{(aq)} + 5H_2O_{(l)} \longrightarrow 4H_3BO_3^{-}{}_{(aq)}$$

Requirements

- 1 Burette (50cm^3), 2 conical flasks (250 cm^3), 1 volumetric flask(250 cm^3), pipette (25 0r 25 cm^3), white tile, 100 cm^3 of hydrochloric acid solution, 5.0g of borax (analyticalgrade), methyl orange indicator,1 weighing bottle, 1 weighing balance sensitive to 0.1g

Procedure

a) Weigh out accurately about 4.8g of borax (analytical grade) in a weighing bottle

b) Transfer it into 250 cm^3 volumetric flask, add about 150 cm^3 of distilled water and shake well to dissolve, and then make to the mark with more water.

c) Shake the flask well, then pipette 25 cm^3 of the solution into a conical flask, then add 3 drops of methyl orange indicator

d) Titrate the portion with the acid from the burette until the indicator changes colour. Repeat the titration to obtain consistent results and tabulate your results,

Results

Mass of the weighing bottle + borax...
Mass of the weighing bottle...
Mass of sodium carbonate...
Capacity of the pipette used..

Titration number	**1**	**2**	**3**	**4**
Final burette reading (cm^3)				
Initial burette reading (cm^3)				
Volume of hydrochloric acid used (cm^3)				

Titre values used to calculate average volume of acid..

Average volume of a acid...
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a) Write the equation for the reaction

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Calculate the concentration of borax in mol dm^{-3}

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b) Calculate the number of moles of hydrochloric acid that reacted with borax

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c) Calculate the concentration acid solution in mol dm^{-3}

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d) Calculate the concentration of hydrochloric acid in grams per litre

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3.4 ANALYSIS OF A MIXTURE OF SODIUM HYDROXIDE AND SODIUM CARBONATE

Method 1 (continuous neutralisation)

Requirements

- 1 Burette ($50cm^3$), 2 conical flasks ($250\ cm^3$), pipette (25 0r $25\ cm^3$), white tile, BA1 which is a mixture of sodium carbonate and sodium hydroxide solutions, BA2 which is 0.1M hydrochloric acid, methyl orange indicator, phenolphthalein indicator

Procedure

a) Pipette $25cm^3$ of BA1 into a conical flask, add 2 drops of phenolphthalein indicator.

b) Titrate BA1 with BA2 until the indicator just changes colour. Record your results in the table A

c) Without pouring away the solution in the flask, add 2 drops of methyl orange indicator and continue with the titration with BA2 until the colour of methyl orange indicator just changes. Record your results in table B

Results

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

Table A

Titration number	**1**	**2**	**3**	**4**
Final burette reading (cm^3)				
Initial burette reading (cm^3)				
Volume of BA2 used (cm^3)				

Titre values used to calculate average volume of BA2………………………………………………

Average volume of a BA2 (V_1)………
……
……
…………………………………………………………………………………………………

Table B

Titration number	**1**	**2**	**3**	**4**
Final burette reading (cm^3)				
Initial burette reading (cm^3)				
Volume of hydrochloric acid used (cm^3)				

Titre values used to calculate average volume of BA2...

Additional average volume of BA2(V_2)
....
....
....
....

a) Write the equations of reactions corresponding to the indicators used
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b) Calculate the volume of BA2 required to react with sodium carbonate in BA1
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c) Calculate the concentration of sodium carbonate in gl^{-1} contained in BA1
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d) Calculate the volume of acid that reacted with sodium hydroxide in BA1
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e) Calculate the mass of sodium hydroxide dissolved in $1000cm^3$ of solution
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3.5 ANALYSIS OF A MIXTURE OF SODIUM CARBONATE AND SODIUM HYDROXIDE

Method 2 (step wise neutralisation)

Requirements

- 1 Burette (50cm^3), 2 conical flasks (250 cm^3), pipette (25 0r 25 cm^3), white tile, BA1 which is a mixture of sodium carbonate and sodium hydroxide solutions, BA2 which is 0.1M hydrochloric acid, methyl orange indicator, phenolphthalein indicator

Procedure

a) Pipette 25cm^3 of BA1 into a conical flask, add 2 drops of phenolphthalein indicator. Titrate BA1 with BA2 until the indicator just changes colour. Repeat titration to obtain consistent results, Record your results in the table A

Results

Volume of pipette used..cm^3

Table A

Titration number	1	2	3	4
Final burette reading (cm^3)				
Initial burette reading (cm^3)				
Volume of BA2 used (cm^3)				

Titre values used to calculate average volume of BA2..

Average volume of a BA2 (V_1)...

....

....

....

d) Pipette another fresh 25 or 20cm^3 of BA1 into a clean conical flask and titrate with BA2 using methyl orange indicator. Repeat the titration to obtain consistent titres and record in table B

Table B

Titration number	**1**	**2**	**3**	**4**
Final burette reading (cm^3)				
Initial burette reading (cm^3)				
Volume of hydrochloric acid used (cm^3)				

Titre values used to calculate average volume of BA2...

average volume of BA2(V$_2$)
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....
....
....

a) Calculate the volume of BA2 required for complete neutralisation of sodium carbonate
....
....
....

b) Calculate the concentration of sodium carbonate in gl^{-1}
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c) Calculate the volume of BA2 required to neutralise sodium hydroxide completely
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d) Calculate the mass of sodium hydroxide dissolved in 1dm^3 of solution
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3.6 DETERMINATION OF A MIXTURE OF SODIUM CARBONATE AND SODIUM HYDROGEN CARBONATE

Requirements

- 1 Burette (50cm^3), 2 conical flasks (250 cm^3), pipette (25 0r 25 cm^3), white tile, FA1 which is a mixture of sodium carbonate and sodium hydrogen carbonate solutions, FA2 which is 0.1M hydrochloric acid, phenolphthalein indicator, methyl orange indicator.

Procedure

a) Pipette 25cm^3 of FA1 into a conical flask, add 3 drops of phenolphthalein indicator.
b) Titrate FA1 with BA2 until the indicator just changes colour. Repeat titration to obtain consistent results, Record your results in the table A

Results

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

Table A

Titration number	**1**	**2**	**3**	**4**
Final burette reading (cm^3)				
Initial burette reading (cm^3)				
Volume of FA2 used (cm^3)				

Titre values used to calculate average volume of FA2…………………………………………………

Average volume of a FA2 (V_1)……………………………………………………………………………………

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…………………………………………………………………………………………………

c) Pipette another fresh 25 or 20cm^3 of FA1 into a clean conical flask and titrate with FA2 using methyl orange indicator. Repeat the titration to obtain consistent titres and record in table B

Table B

Titration number	**1**	**2**	**3**	**4**

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

Titre values used to calculate average volume of FA2..

average volume of FA2(V_2)

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....

a) Calculate the volume of FA2 required for complete neutralisation of sodium carbonate

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b) Calculate the concentration of sodium carbonate in gl^{-1}

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c) Calculate the volume of FA2 required to neutralise sodium hydrogen carbonate completely

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d) Calculate the mass of sodium hydrogen carbonate dissolved in $1dm^3$ of solution

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3.7 DETERMINATION OF A MIXTURE OF SODIUM HYDROXIDE AND SODIUM HYDROGEN CARBONATE

Requirements

- 1 Burette (50cm^3), 2 conical flasks (250 cm^3), pipette (25 0r 25 cm^3), white tile, A1 which is a mixture of sodium hydrogen carbonate and sodium hydroxide solutions, A2 which is 0.05M hydrochloric acid, methyl orange indicator, phenolphthalein indicator

Procedure

e) Pipette 25cm^3 of A1 into a conical flask, add 3 drops of phenolphthalein indicator.

f) Titrate A1 with A2 until the indicator just changes colour. Record your results in the table A

g) Without pouring away the solution in the flask, add 3 drops of methyl orange indicator and continue with the titration with A2 until the colour of methyl orange indicator just changes. Record your results in table B

Results

Volume of the pipette used...cm^3

Table A

Titration number	**1**	**2**	**3**	**4**
Final burette reading (cm^3)				
Initial burette reading (cm^3)				
Volume of A2 used (cm^3)				

Titre values used to calculate average volume of A2..

Average volume of a A2 (V$_1$)..
....
....
....

Table B

Titration number	**1**	**2**	**3**	**4**
Final burette reading (cm^3)				
Initial burette reading (cm^3)				
Volume of hydrochloric acid used (cm^3)				

Titre values used to calculate average volume of A2..

Additional average volume of A2(V_2)

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a) Write the equations of reactions corresponding to the indicators used

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b) Calculate the volume of A2 required to react with sodium hydroxide in A1

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c) Calculate the concentration of sodium hydroxide in gl^{-1} contained in A1

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d) Calculate the volume of acid that reacted with sodium hydrogen carbonate in A1

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e) Calculate the mass of sodium hydrogen carbonate dissolved in $1000cm^3$ of solution

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4.0 DETERMINATION OF RELATIVE MOLECULAR MASS OF AN ORGANIC ACID

Requirements

- 1 burette ($50cm^3$), conical flask ($250cm^3$), pipette (25 0r $20cm^3$), pure organic acid of molecular formula RCOOH, $50cm^3$ of 0.2M sodium hydroxide solution, balance sensitive to 0.1g, volumetric flask ($250cm^3$), phenolphthalein indicator.

Procedure

a) Weigh accurately 5.0g of pure organic acid RCOOH and dissolve in 1.1 (V/V) ethanol/water mixtures.
b) Transfer the solution to $250cm^3$ volumetric flask, shake well to ensure that the solution is homogenous and make up to the mark.
c) Pipette $25cm^3$ aliquot into the conical flask, add 3 drops of phenolphthalein indicator
d) Titrate with standard 0.2M sodium hydroxide until the colour of the solution becomes faint pink, repeat with further $25cm^3$ volume of the acid solution for consistency

Results

Mass of weighing bottle + acid RCOOH..

Mass of weighing bottle...

Mass of acid RCOOH...

Volume of pipette used..

Titration number	**1**	**2**	**3**	**4**
Final burette reading (cm^3)				
Initial burette reading (cm^3)				
Volume of sodium hydroxide used (cm^3)				

Titre values used to calculate average volume..

Average volume of sodium hydroxide
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a) Write the equation for the reaction

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b) Calculate the relative molecular mass of the acid, RCOOH

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c) Calculate the value of R in RCOOH

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5.1 DETERMINATION OF WATER OF CRYSTALLISATION IN A HYDRATED COMPOUND

Requirements

1 burette (50cm^3), 2 conical flasks (250cm^3), pipette (25 0r 20cm^3), volumetric flask (250cm^3), phenolphthalein indicator, 2.0g of R which is an acid of formula $H_2A.nH_2O$ where n is the number of molecules of water of crystallisation, 100cm^3 of BA1 which is 0.1M sodium hydroxide solution, weighing balance sensitive to 0.1g

Procedure

a) Weigh accurately 1.6g of X and transfer the solution to 250cm^3 volumetric flask, dissolve in water shake well to ensure that the solution is homogenous and make up to the mark, label the solution BA2.

b) Pipette 25cm^3 BA1aliquot into the conical flask, add 3 drops of phenolphthalein indicator

c) Titrate with BA2 until the end-point. Repeat the titration for consistency

Results

Mass of weighing bottle + X..

Mass of weighing bottle..

Mass of acid X...

Volume of pipette used..

Titration number	**1**	**2**	**3**	**4**
Final burette reading (cm^3)				
Initial burette reading (cm^3)				
Volume of BA2 (cm^3)				

Titre values used to calculate average volume...

Average volume of BA2
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a) Write the equation for the reaction
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b) Calculate the molarity of BA2
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c) Determine the value of n (formula mass of A = 48)
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5.2 DETERMINATION OF THE PERCENTAGE OF AMMONIUM CHLORIDE IN THE IMPURE SAMPLE

Requirements

- 1 Burette (50cm^3), 2 conical flasks (250 cm^3), 1 volumetric flask(250 cm^3), pipette (25 0r 25 cm^3), 100 cm^3 of 2M sodium hydroxide solution, 100cm^3 of 0.5M hydrochloric acid, Bunsen burner, 1 weighing bottle, 1 weighing balance sensitive to 0.1g

Procedure

a) Weigh accurately 5.0g 0f a sample of ammonium chloride into a weighing bottle
b) Transfer it into 250cm^3 conical flask. Add 100cm^3 of 2M sodium hydroxide and boil the mixture until ammonia has ceased to evolve (test with litmus paper)
c) Cool and transfer into 250cm^3 volumetric flask, make the solution to the mark with water and label it BA2
d) Pipette 25 or 20cm^3 of this solution into a conical flask and titrate it with 0.5M hydrochloric acid using phenolphthalein indicator. Repeat the titration to obtain consistent results and record your values in the table below
e)

Results
Mass of weighing bottle + sample..
Mass of weighing bottle...
Mass of acid sample..
Capacity of pipette used...

Titration number	**1**	**2**	**3**
Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of hydrochloric acid used (cm^3)			

Titre values used to calculate average volume...
Average volume of hydrochloric acid
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a) Calculate the number of moles of excess sodium hydroxide solution

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b) Calculate the number of moles of sodium hydroxide that reacted with ammonium chloride solution.

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c) Calculate the percentage of ammonia in ammonium chloride salt

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5.3 DETERMINATION OF PERCENTAGE PURITY

Requirements

- 1 Burette (50cm^3), 2 conical flasks (250 cm^3), 1 volumetric flask (250 cm^3), pipette (25 0r 25 cm^3), 250 cm^3 of BA2 which is 0.1M sodium hydroxide solution, 100cm^3 of BA3 which is 0.05M sulphuric acid, 1 weighing bottle, 1 weighing balance sensitive to 0.1g, phenolphthalein indicator, 2.0g of solid BA1 which is an impure sample containing an organic compound R, heat source.

Procedure

a) Weigh accurately 1.0g of BA1 in a weighing bottle, transfer it into a conical flask. Add 200cm^3 of BA2 and boil the mixture for 15 minutes

b) Cool and transfer it into 250cm^3 volumetric flask and make the solution to the mark with BA2

c) Pipette 50cm^3 of the resultant solution into the conical flask. Titrate with BA3 using phenolphthalein indicator. Repeat the titration to obtain consistent titres and record in table below.

Results

Mass of weighing bottle + BA1...

Mass of weighing bottle..

Mass of acid BA1..

Volume of pipette used...

Titration number	**1**	**2**	**3**
Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of BA3 used (cm^3)			

Titre values used to calculate average volume..

Average volume of BA3
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....
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a) Calculate;

I) The number of moles of excess sodium hydroxide that reacted with sulphuric acid
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II) The number of moles of sodium hydroxide that reacted with R
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III) The mass of R that reacted with sodium hydroxide (mole ratio of R : NaOH = 1 : 2 and RFM of R = 180)

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IV) The percentage of R in BA1

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6.0 PHYSICAL CHEMISTRY EXPERIMENTS

6.1 DETERMINATION OF MOLECULAR MASS BY FREEZING POINT DEPRESSION

Requirements

- 10.0g of naphthalene, 1.0g of camphor, thermometer sensitive to 0.1°C, weighing scale sensitive to 0.1g

Procedure

a) Weigh accurately 9.0g of naphthalene into a boiling tube

b) Insert the thermometer in the tube in the beaker of boiling water; stir the naphthalene to ensure complete melting.

c) Put off the flame; allow the boiling tube to cooling the beaker of water. When crystals start to appear, note the steady temperature, T_1

d) Now weigh accurately 0.8g of camphor and put it into the boiling tube containing naphthalene and note the steady temperature, T_2 when crystals start to form

e) Record your results in the below

Results

Mass of naphthalene...

Mass of camphor...

Temperature, T_1...

Temperature, T_2..

$\Delta T = T_1 - T_2$...

a) Calculate the molecular mass of camphor from first principles (the cryoscopic constant, K_f for 1g of naphthalene is 6.9°C per mole of the solute)

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6.2 DETERMINATION OF SOLUBILITY PRODUCT CONSTANT

Requirements

- 1 burette ($50cm^3$), 2 conical flasks ($250cm^3$), 1pipette (25 or $20cm^3$), 150 0f BA1 which is 0.05M hydrochloric acid, 2g of sparingly soluble hydroxide, $M(OH)_2$ of molecular mass 74, phenolphthalein indicator.

Procedure

a) Put into the beaker or flask all the 2g of $M(OH)_2$ supplied and add 150cm^3 of distilled water

b) Stopper the flask and shake the mixture vigorously for 15 minutes and then filter using a dry filter paper.

c) Pipette 25 or 20cm^3 of the filtrate into the conical flask

d) Titrate the solution with BA1 using phenolphthalein indicator. Repeat titration to ensure consistency. Report the results in the table below.

Volume of pipette used..

Titration number	**1**	**2**	**3**
Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of BA1 used (cm^3)			

Titre values used to calculate average volume...

Average volume of BA1

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a) Calculate the concentration of $M(OH)_2$ in gl^{-1}

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b) Calculate the solubility product of $M(OH)_2$

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6.3 DETERMINATION OF SOLUBULITY PRODUCT CONSTANT

Requirements

- 1 burette (50cm^3), 2 conical flasks (250cm^3), 1pipette (25 or 20cm^3), 150 0f BA1 which is 0.1M sodium hydroxide solution, BA2 which is 0.1M hydrochloric acid, 2g of calcium hydroxide, phenolphthalein indicator.

Procedure

a) Put into a 250cm^3 conical flask 100cm^3 of BA1 solution.

b) Add to it the whole mass of calcium hydroxide, stopper the flask, and shake for 15 minutes and then filter using a dry filter paper

c) Pipette 25 or 20cm^3 of the filtrate into the conical flask

d) Titrate the solution with BA2 using phenolphthalein indicator. Repeat the titration for consistency and tabulate your results.

Results

Volume of pipette used..

Titration number	**1**	**2**	**3**
Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of BA2 used (cm^3)			

Titre values used to calculate average volume..

Average volume of BA2
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a) Calculate the number of moles of hydrochloric acid required for reaction with the total OH^- in saturated solution

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c) Calculate the concentration of $Ca(OH)_2$ in gl^{-1}

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d) Calculate the solubility product of $Ca(OH)_2$

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6.4 DETERMINATION OF THE DISTRIBUTION CONSTANT K_D OF BUTANEDIOC ACID BETWEEN WATER AND ETHER

Requirements

Titration apparatus, FA1 which is a solution containing 10g of butanedioc acid per litre, FA2 which is a solution containing 1.3g of hydroxide ions per litre, W which is ether immiscible with water.

Procedure

a) Using separate cylinders transfer 40cm^3 of FA1 and 40cm^3 of W into a clean conical flask. Stopper the flask and shake vigorously for 5 minutes
b) Repeat the procedure using other flasks with the volumes of 30cm^3 of FA1 and 50cm^3 of W, 50cm^3 of FA1 and 30cm^3 of W
c) Leave the flasks to stand for after shaking for some time to allow the layers to separate out and note the temperature in each case
d) Pipette 10cm^3 of the lower aqueous layer and titrate it with FA2 using phenolphthalein indicator
e) Pipette another 10cm^3 of the upper layer (organic layer) and titrate it with FA2 using phenolphthalein indicator
f) Repeat the procedure with the other remaining two flasks and tabulate your results

Results

Mixture	Temperature	Layer	Final reading	Initial reading	Volume of FA2 used
40cm^3 of FA1 and 40cm^3 of W		lower upper			
30cm^3 of FA1 and 50cm^3 of W		lower upper			
50cm^3 of FA1 and 30cm^3 of W		lower upper			

a) Calculate the concentration of FA2 in mol dm^{-3}

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b) Calculate the number of moles of butanedioc acid in the aqueous layer in mol dm^{-3}

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c) The ratio, $\frac{\text{concentration of butanedioc acid in aqeueos layer}}{\text{concentration of butanedioc acid in the solvent layer}}$

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d) Get the ratio for second and third flasks

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e) Comment on your results

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7.0 REDOX TITRATIONS

The formal concept of oxidation number is useful in the balancing of the redox equations especially those containing oxo-anions

1.1. Rules for finding the oxidation number

I) Atoms of elements are given oxidation number zero. Oxygen has oxidation number of -2 except in peroxides where it is -1

II) F, Cl, Br, I when present in halides have an oxidation number of -1

III) Hydrogen always has the oxidation number of +1 except in ionic hydrides

IV) Sodium and potassium have +1, Mg and Ca +2, Al +3

V) In any compound, the sum of the oxidation number is zero

VI) In a compound of metal and non-metal, the metal is given a positive oxidation number and non-metal given a negative oxidation number. In other cases, (I-IV) must be applied eg in SO_2, oxygen always has O.N of -2, therefore the total for oxygen is -4, sulphur is X

$$X - 4 = 0$$
$$X = 4$$

Therefore sulphur has +4

For MnO_4^-, O.N for manganese can be calculated as below

$$X - 8 = -1$$
$$X = +7$$

Therefore O.N for Mn = +7

Oxidation number is used in balancing of redox equations. This can be illustrated by the reduction of chlorate to chloride ion. In the ClO_3^-, chlorine has oxidation number of +5 whereas Cl^- has -1 thus electrons are required for the reduction of ClO_3^- to Cl^-

In acid solution, the half equation is

$$ClO_3^-{}_{(aq)} + 6e + 6H^+{}_{(aq)} \longrightarrow Cl^-{}_{(aq)} + 3H_2O_{(l)}$$

In half equation, for the reduction of the oxo-anion in the acid solution, it is usually necessary to balance the charge on the two sides by adding H^+ ion at one side and water molecule on the other side. In this case, $6H^+$ ions on the left and $3H_2O$ molecules on the right are necessary to ensure that the total charge and stoichiometry are both balanced.

As the equation of Fe^{2+} to Fe^{3+} releases only one electron

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

6 Fe^{2+} ions are necessary to reduce one ClO_3^- to Cl^-. The complete redox equations obtained by adding six times the second equation and add to the first one.

$$ClO_3^-{}_{(aq)} + 6e + 6H^+{}_{(aq)} \longrightarrow Cl^-{}_{(aq)} + 3H_2O_{(l)}$$
$$6Fe^{2+}{}_{(aq)} \longrightarrow 6Fe^{3+} + 6e$$

$$ClO_3^-{}_{(aq)} + 6Fe^{2+}{}_{(aq)} + 6H^+{}_{(aq)} \longrightarrow Cl^-{}_{(aq)} + 3H_2O_{(l)} + 6Fe^{3+}{}_{(aq)}$$

Note. The electrons cancel out

Oxidation-reduction reaction includes reactions involving change in oxidation number or transfer of electrons among the reacting substances. The standard solutions are either reducing or oxidising agents.

7.1 Principle oxidising agents include

a) Potassium manganate (VII) titration

Potassium permanganate (VII) is a useful oxidising agent in acidic medium. On reduction, the purple colour of MnO_4^- in turned to colourless Mn^{2+} ions; as a result no indicator is needed.

Manganate (VII) is a strong oxidising agent, it is usually acidified with sulphuric acid

$$2MnO_4^-{}_{(aq)} + 10Cl^-{}_{(aq)} + 16Cl^-{}_{(aq)} \longrightarrow 2Mn^{2+}{}_{(aq)} + 5Cl_{2(g)} + 8H_2O_{(l)}$$

The manganate (VII) solution in acidic medium is used mainly in redox titration with

i. Iron (II) salts

$$MnO_4^-{}_{(aq)} + 5Fe^{2+}{}_{(aq)} + 8H^+{}_{(aq)} \longrightarrow 5Fe^{3+}{}_{(aq)} + Mn^{2+}{}_{(aq)} + 4H_2O_{(l)}$$

ii. Ethanedioate

$$5C_2O_4^{2-}{}_{(aq)} + 2MnO_4^-{}_{(aq)}\ 16H^+{}_{(aq)} \longrightarrow 10CO_{2(g)} + 2Mn^{2+}{}_{(aq)} + 4H_2O_{(l)}$$

iii. Hydrogen peroxide

$$2\ MnO_4^-{}_{(aq)} + 16H^+{}_{(aq)}\ 5H_2O_{2(aq)} \longrightarrow 10H^+{}_{(aq)} + 5O_{2(g)} + 2Mn^{2+}{}_{(aq)} + 8H_2O_{(l)}$$

or

$$2\ MnO_4^-{}_{(aq)} + 6H^+{}_{(aq)}\ 5H_2O_{2(aq)} \longrightarrow 5O_{2(g)} + 2Mn^{2+}{}_{(aq)} + 8H_2O_{(l)}$$

b) Dichromate (VI) titration

Dichromate is majorly used in the oxidation of iron (II) salts

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

In acidic medium, potassium dichromate (VI) acts a powerful oxidising agent and it is normally acidified by hydrochloric acid. The half equation for oxidation is,

$$Cr_2O_7^{2-}{}_{(aq)} + 14H^+{}_{(aq)}\ 6e \longrightarrow 2Cr^{3+}{}_{(aq)} + 7H_2O_{(l)}$$

$Cr_2O_7^{2-}{}_{(aq)}$ is orange, $Cr^{3+}{}_{(aq)}$ is green, $Cr_2O_4^{2-}{}_{(aq)}$ is yellow

The chromate (VI) ion in the presence of acid is converted to dichromate (VI) ions

$$2Cr_2O_4^{2-}{}_{(aq)} + 2H^+{}_{(aq)} \longrightarrow Cr_2O_7^{2-}{}_{(aq)} + H_2O_{(aq)}$$

c) Iodine titration

The direct iodimetric titration, iodimetry refers to titration with standard solution of iodine. Iodine dissolves in potassium iodide solution to give intense yellow brown colour containing the I_3^- ion.

$$I_{2(aq)} + I^-{}_{(aq)} \longrightarrow I_3^-{}_{(aq)}$$

The strength of the solution can be determined with standard solution of sodium thiosulphate penta hydrate, $Na_2S_2O_3.5H_2O$, the reaction is represented by the following equation

$$I_{2(aq)} + 2S_2O_3^{2-}{}_{(aq)} \longrightarrow 2I^-{}_{(aq)} + S_4O^{2-}{}_{6(aq)}$$

The colour of the iodine is weakened as standard thiosulphate is added to it. Near the end-point when the solution has a pale straw colour, a little starch is added. A deep blue colour appears which is discharged at the end-point. When the standard thiosulphate ions are added to convert the last of iodine to iodide

The indirect iodometric titration, iodometry deals with the titration of iodine liberated in a chemical reaction. If a strong oxidising agent is treated with a large excess of iodide ions in a neutral or more usually acid solution, the iodide ion reacts with the reducing agent and the oxidant will quantitatively be reduced. In such cases, an equivalent amount of iodine liberated and is then titrated with a standard solution of a reducing agent which is usually sodium thiosulphate e.g

$$Cu^{2+}_{(aq)} + 4I^{-}_{(aq)} \longrightarrow CuI_{(aq)} + I_{2(aq)}$$

$$Cl_{2(g)} + 2I^{-}_{(aq)} \longrightarrow 2Cl^{-}_{(aq)} + I_{2(aq)}$$

$$6H^{+}_{(aq)} + IO_{3}^{-}{}_{(aq)} + 5I^{-}_{(aq)} \longrightarrow 3H_2O_{(l)} + 3I_{2(aq)}$$

Weakly acidic conditions are necessary in all these reactions, but too low PH causes the sodium thiosulphate, $Na_2S_2O_3$ to decompose.

7.2 STANDARDISATION OF MANGANATE (VII) SOLUTIONS USING AQEUEOUS ETHANEDIOATE

Requirements

Burette (50ml), pipette (10, 20, 25ml), 2 conical flasks (250ml), weighing scale sensitive to 0.1g, 100ml of FA1 which is a solution of potassium permanganate (VII), 1.7g of P which is sodium oxalate, weighing bottle, 1M sulphuric acid solution, 1 volumetric flask (250ml)

Procedure

a) Weigh accurately 1.7g of sodium oxalate in a weighing bottle and dissolve in 100ml of distilled water

b) Transfer it quantitatively into 250ml volumetric flask and make up to the mark with distilled water, shake well.

c) Pipette 25ml of this solution into the clean conical flask and add equal volume of 1M sulphuric acid. Heat up to about 65°C as rapidly as possible. Titrate the hot mixture with manganate (VII) solution until the permanent faint pink colour is obtained

d) Repeat the titration for consistency and tabulate your results.

Results

Mass of the weighing bottle + P...
Mass of the weighing bottle...
Mass of P...
Volume of the pipette used...

Titration number	**1**	**2**	**3**
Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA1 used (cm^3)			

Titre values used to calculate average volume..

Average volume of BA1

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a) Write the overall equation for the reaction

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b) Calculate the number of moles of sodium oxalate used

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c) Calculate the number of moles of potassium permanganate (VII)

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d) Calculate Molarity of potassium permanganate (VII)

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e) Calculate the mass of the permanganate dissolved in 1 litre of solution

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7.3 DETERMINATION OF THE PROPORTION OF Fe^{2+} IN THE SALT

Requirements

Burette (50ml), pipette (10, 20, 25ml), 2 conical flasks (250ml), weighing scale sensitive to 0.1g, 150ml of 0.02M solution of potassium permanganate (VII), 12g of X which is ammonium iron (II) sulphate 12-water $[NH_4Fe(SO_4)_2.12H_2O]$, zinc powder/granules, weighing bottle, 1M copper (II) sulphate solution, 1 volumetric flask (250ml),1M sulphuric acid

Procedure

a) Weigh accurately 12g of ammonium iron (II) sulphate 12-water $[NH_4Fe(SO_4)_2.12H_2O$ in a weighing bottle and dissolve in the 75ml of 1M sulphuric acid and transfer it quantitatively into 250ml volumetric flask and make to the mark with distilled water

b) Pipette 25ml portions of the solution into three conical flasks, add equal volume of 1Msulphuric acid and 1ml of 1M copper (II) sulphate and then about 2g of zinc dust/granules. Wait for the solution to turn colourless or a very pale green

c) Filter each solution of iron solution and rinse the remaining zinc adding the rinsing to the filtrate. Titrate each batch with standard permanganate solution until the permanent pale pink tinge is seen.

Results

Mass of the weighing bottle + X…………………………………………………
Mass of the weighing bottle………………………………………………………
Mass of X…………………………………………………………………………………
Volume of the pipette used…………………………………………………………

Titration number	**1**	**2**	**3**
Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of manganate (VII) used (cm^3)			

Titre values used to calculate average volume…………………………………………………

Average volume of permanganate (VII)
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a) Write the overall equation for the reaction
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b) Calculate the number of moles of potassium permanganate (VII) used
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c) Calculate the concentration of Fe^{2+} in moles per litre in X

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7.4 DETERMINATION OF WATER OF CRYSTALLISATION IN A HYDRATED SALT

Requirements

Burette (50ml), pipette (10 or 20 or 25ml), 2 conical flasks (250ml), weighing scale sensitive to 0.1g, BA1 which is a solution containing 3.2g of potassium permanganate (VII) per litre, weighing bottle, 1 volumetric flask (250ml), 50ml of 1M sulphuric acid, 7g of the hydrated salt[$FeSO_4.nH_2O$], X

Procedure

a) Weigh accurately about 6.4g of the hydrated salt and transfer it into a 250ml volumetric flask, dissolve in 50ml of 1M sulphuric acid and make it to the mark with distilled water, label it BA2
b) Pipette 25ml or 20ml of BA2 into the clean conical flask and titrate it with BA1 from the burette. Repeat the titration for consistency and tabulate your results

Results

Mass of the weighing bottle + X..
Mass of the weighing bottle..
Mass of X..
Volume of the pipette used..

Titration number	**1**	**2**	**3**
Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of BA1 used (cm^3)			

Titre values used to calculate average volume..

Average volume of BA1
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a) Write the overall equation for the reaction
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b) Calculate the molarity of BA1 used
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c) Calculate the concentration of BA2 used in moles per litre
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d) Calculate the relative formula mass of the hydrated salt (Fe = 56, O = 16, H = 1)
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e) Determine the value of n in the salt, $FeSO_4.nH_2O$

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7.5 DETERMINATION OF THE STOICHIOMETRY OF THE REACTIONS

Requirements

Burette (50ml), pipette (10 or 20 or 25ml), 2 conical flasks (250ml), weighing scale sensitive to 0.1g, FA1 which is a solution containing 2.41g of potassium permanganate (VII) per litre, FA3 which is a solution containing 2.5g of Fe^{2+} in 250ml ,M which is a metal, weighing bottle, 1 volumetric flask (250ml)

You are required to determine the stiochiometry of the reaction between Fe^{2+} and metal M

Procedure

a) Weigh accurately about 0.2g of M and transfer the whole mass into a conical flask, add 100ml of FA3 and boil gently until the whole of M is dissolved, cool and label FA4

b) Pipette 25ml of FA4 into a conical flask and add 25ml of dilute sulphuric acid. Titrate the resulting solution with FA1 and tabulate results

Results

Mass of the weighing bottle + M..
Mass of the weighing bottle...
Mass of M..
Volume of the pipette used..

Titration number	**1**	**2**	**3**
Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of FA1 used (cm^3)			

Titre values used to calculate average volume...

Average volume of FA1

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a) Write the overall equation for the reaction

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b) Calculate the molarity of FA1 used

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c) Calculate the number of moles of Fe^{2+} ions that was produced when M reacts with FA3concentration of BA2 used in moles per litre

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d) Calculate the number of moles of M used (M=128)..
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e) Determine the stoichiometry of the reaction between M and Fe^{2+} ions
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7.6 STANDARDISATION OF HYDROGEN PEROXIDE BY POTASSIUM PERMANGANATE (VII)

Requirements

Burette (50ml), pipette (10 or 20 or 25ml), 2 conical flasks (250ml), BA1 which is a solution containing 2.41g of MnO_4^- per litre, BA2 which is a solution of hydrogen peroxide, 50ml of 2M sulphuric acid

Procedure

a) Pipette 10ml of BA2 into a clean conical flask, add 10ml of 2M sulphuric acid and titrate the mixture with BA1. Repeat the titration for consistency and tabulate the results

Results

Volume of the pipette used..

Titration number	**1**	**2**	**3**
Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of BA1 used (cm^3)			

Titre values used to calculate average volume..

Average volume of BA1..
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a) Write the all equations for the reactions and overall equation
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b) Calculate the concentration of BA2 in mol dm^{-3}
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c) Calculate the concentration of BA2 in grams per litre

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7.7 ANALYSIS OF HYDROGEN PEROXIDE

Hydrogen peroxide is usually encountered in aqueous solution and frequently referred to as 20-volume, 40-volume and 100-volume hydrogen peroxide. These volumes contain 6%, 12%, 30% of hydrogen peroxide respectively. The term volume is based up on the volume of oxygen liberated on decomposition of the peroxide. Thus 1cm^3 of 100-volume hydrogen peroxide will yield 100cm^3 of oxygen measures at s.t.p.

The following reaction occurs when potassium permanganate (VII) is added to hydrogen peroxide solution acidified with dilute sulphuric acid

$$2MnO_4^-{}_{(aq)} + 5H_2O_{(aq)} + 6H^+{}_{(aq)} \longrightarrow 2Mn^{2+}{}_{(aq)} + 5O_{2(g)} + 8H_2O_{(l)}$$

It is reasonably good to fairly use high concentration of acid and low rate of addition in order to reduce the danger of forming manganese dioxide which is an active catalyst for decomposition of hydrogen peroxide

Requirements

Burette (50ml), pipette (10 or 20 or 25ml), 2 conical flasks (250ml), solution of hydrogen peroxide, BA1 which is 0.02M potassium permanganate (VII) solution, 150ml of 1M sulphuric acid

Procedure

Results

a) Transfer carefully 25ml of hydrogen peroxide solution into 250ml volumetric flask, dilute to the mark with water, shake thoroughly
b) Pipette 25ml of this solution into the clean conical flask, dilute with 10ml of distilled water, and then add 10ml of 1M sulphuric acid. Titrate the resultant mixture with

standard 0.02M potassium permanganate (VII) to the first permanent faint pink colour. Repeat the titration for consistency and tabulate the titres

Volume of the pipette used..

Titration number	**1**	**2**	**3**
Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of BA1 used (cm^3)			

Titre values used to calculate average volume..

Average volume of BA1...
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a) Write the all equations for the reactions and overall equation
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b) Calculate the weight of hydrogen peroxide per litre of the original solution
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c) Calculate the volume strength ie the number of millimetres of oxygen at s.t.p that can be obtained from 1cm^3 of the original solution

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7.8 DETERMINATION OF THE COMPOSITION OF A MIXTURE OF ETHANEDIOIC ACID AND SODIUM ETHANEDIOATE

Requirements

Burette (50ml), pipette (10 or 20 or 25ml), 2 conical flasks (250ml), 200ml of solution of mixture,150ml of 0.02M potassium permanganate (VII) solution, 150ml of 0.1M sodium hydroxide (BA1), weighing balance sensitive to 0.1g

Procedure

a) Pipette 25ml of the solution mixture into a clean conical flask; add 3 drops of the phenolphthalein indicator. Titrate with 0.1M sodium hydroxide solution until the solution just turns pink
b) Repeat the titration to get consistent values, tabulate the results.

Results

Volume of the pipette used..

Titration number	**1**	**2**	**3**
Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of BA1 used (cm^3)			

Titre values used to calculate average volume..

Average volume of BA1...
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c) Pipette another 25ml portion of the solution into a conical flask, add an equal volume of 2M sulphuric acid, heat the solution to about 57°C and immediately titrate with 0.02M potassium permanganate (VII), the end-point is reached when the pink coloration that persist is obtained. Repeat the titration for consistency and tabulate the results

Results

Volume of the pipette used..

Titration number	**1**	**2**	**3**
Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of MnO_4^- used (cm^3)			

Titre values used to calculate average volume..

Average volume of MnO_4^-

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a) Calculate the molarity of ethanedioic acid in the mixture

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b) Calculate the molarity of sodium ethanedioate

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c) Calculate the concentration of ethanedioic acid in grams per litre in the original mixture
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d) Calculate the concentration of sodium ethanedioate in grams per litre in the original mixture
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e) Calculate the percentage of ethanedioic acid in the original mixture
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f) Calculate the percentage of sodium ethanedioate in the original mixture

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7.9 STANDARDISATION OF SODIUM THIOSULPHATE

Requirements

- Burette (50ml), pipette (10 or 20 or 25ml), 2 conical flasks (250ml), M which is 1.5g of potassium dichromate (VI), sodium thiosulphate ($Na_2S_2O_3.5H_2O$), weighing balance sensitive to 0.1g, starch indicator, 1M HCl

Procedure

a) Weigh out accurately 1.2g of potassium dichromate (VI) salt and dissolve it in the minimum quantity of distilled water in volumetric flask (250ml), make to the mark.

b) Pipette 25ml of this solution and add 25ml 1M HCl then 10ml of 10% solution of KI. Titrate the liberated I_2 with sodium thiosulphate solution with constant shaking, when the colour of the liquid becomes pale yellow, add 2ml of starch solution and continue the titration until the colour of the solution changes from blue to green. Repeat the titration to obtain consistent results and tabulate results.

Results

Mass of the weighing bottle + M...
Mass of the weighing bottle...
Mass of M...
Volume of the pipette used...

Titration number	**1**	**2**	**3**
Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of thiosulphate used (cm^3)			

Titre values used to calculate average volume..

Average volume of thiosulphate
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a) Write the equation for the reaction that leads to liberation of iodine
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b) Calculate the molarity of potassium dichromate (VI)
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c) Calculate the number of moles of iodine liberated by potassium dichromate (VI)
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d) Calculate the concentration of sodium thiosulphate in mol dm^{-3}
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7.10 STANDARDISATION OF SODIUM THIOSULPHATE SOLUTION

Requirements

- Burette (50ml), pipette (10 or 20 or 25ml), 2 conical flasks (250ml), Z which is 1.0g of potassium iodate, BA1 which is 0.1M sodium thiosulphate, weighing balance sensitive to 0.1g, starch indicator, 1M sulphuric acid

Procedure

c) Weigh out accurately 1.0g of potassium iodate and dissolve it in distilled water in volumetric flask (250ml), make to the mark.

d) Pipette 25ml of this solution and add 10ml of 10% solution of KI followed by 20ml of 1M sulphuric acid. Titrate the liberated I_2 with sodium thiosulphate solution with constant shaking, when the colour of the liquid becomes pale yellow, add 2ml of starch solution and continue the titration until the colour of the solution changes from blue to colourless. Repeat the titration to obtain consistent results and tabulate results.

Results

Mass of the weighing bottle + Z..
Mass of the weighing bottle..
Mass of Z...
Volume of the pipette used...

Titration number	**1**	**2**	**3**
Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of thiosulphate used (cm^3)			

Titre values used to calculate average volume..

Average volume of thiosulphate

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a) Write the equation for the reaction that leads to liberation of iodine

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b) Calculate the molarity of potassium iodate (I = 127, K = 39, O = 16)

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c) Calculate the number of moles of iodine

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d) Calculate the concentration of sodium thiosulphate in mol dm^{-3}

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7.11 DETERMINATION OF COPPER IN CRYSTALLISED COPPER

Requirements

- Burette (50ml), pipette (10 or 20 or 25ml), 2 conical flasks (250ml), Y which copper (II) sulphate crystals, BA1 which is 0.1M sodium thiosulphate , weighing balance sensitive to 0.1g, starch indicator, ethanoic acid solution, sodium carbonate solution

Procedure

a) Weigh out accurately 6.0g of copper (II) sulphate and place in volumetric flask (250ml), add 100ml of distilled water to dissolve followed by sodium carbonate solution until the slight ppt forms and dilute ethanoic acid drop wise until the ppt just dissolves. Top up the copper (II) sulphate solution to the mark with distilled water, shake the mixture

b) Pipette 25ml of this solution and add 10ml of 10% solution of KI. Titrate the liberated I_2 with standard 0.1M sodium thiosulphate solution with constant shaking until the brown colour of iodine fades, then add 2ml of starch solution and continue the titration until the colour of the solution changes from blue to colourless. Repeat the titration to obtain consistent results and tabulate results.

Results

Mass of the weighing bottle + Y..

Mass of the weighing bottle..

Mass of Y...

Volume of the pipette used..

Titration number	**1**	**2**	**3**
Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of thiosulphate used (cm^3)			

Titre values used to calculate average volume..

Average volume of thiosulphate

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Copper (II) sulphate reacts with potassium iodide according to the following equation

$$CuSO_{4(aq)} + 4KI_{(aq)} \longrightarrow 2CuI_{(s)} + I_{2(aq)} + 2K_2SO_{4(aq)}$$

a) Calculate the amount of copper in the crystallised copper sulphate

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b) Calculate the percentage of copper in the crystallised copper (II) sulphate

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7.12 DETERMINATION OF AVAILABLE CHLORINE IN HYPOCHLORITES (COMMERCIAL BLEACHING POWDER)

Commercial bleaching powder essentially contains a mixture of calcium hypochlorite, $Ca(OCl)_2$ and basic chloride, $CaCl_2.Ca(OH)_2.N_2O$ and some free slaked lime is usually present. The active constituent is the hypochlorite which is responsible for the bleaching action. The available chlorine refers to the chlorine liberated by action of dilute acids on hypochlorite and is expressed as a percentage in the case of bleaching powder.

$$OCl^-_{(aq)} + Cl^-_{(aq)} + 2H^+_{(aq)} \longrightarrow Cl_{2(g)} + H_2O_{(l)}$$

Requirements

- Burette (50ml), pipette (10 or 20 or 25ml), 2 conical flasks (250ml), 10% KI, BA1 which is 0.1M sodium thiosulphate, weighing balance sensitive to 0.1g, starch indicator, Glacial ethanoic acid solution, bleaching powder(M)

Procedure

a) Weigh accurately 2.5g of bleaching powder into a clean mortar, add a little of water and grind the mixture to a smooth paste, add a little of more water and pour the milky liquid into a 250ml volumetric flask. Grind the residue with little more water and repeat the process until the whole sample has been transferred to the flask, either in form of solution or state of very fine suspension and the mortar washed quite clean. The flask is then filled to the mark with distilled water and properly shaken

b) Pipette 25ml of the liquid in the conical flask and immediately add 20ml of 10% KI solution followed by 10ml of glacial ethanoic acid. Titrate the liberated iodine with standard solution of thiosulphate solution with constant shaking .when the colour of the liquid becomes pale yellow, add 2ml of starch solution, and continue titration until the colour of solution turns from blue to colourless. Repeat the titration to obtain consistent results and tabulate them.

Equations of reaction are,

$$OCl^-_{(aq)} + 2I^-_{(aq)} \longrightarrow I_{2(aq)} + Cl^-_{(aq)} + H_2O_{(l)}$$
$$I_{2(aq)} + 2S_2O_{3(aq)} \longrightarrow 2I^-_{(aq)} + S_4O_{6(aq)}$$

Results

Mass of the weighing bottle + M...

Mass of the weighing bottle..

Mass of M...

Volume of the pipette used..

Titration number	**1**	**2**	**3**
Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of thiosulphate used (cm^3)			

Titre values used to calculate average volume...

Average volume of thiosulphate

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a) Determine the number of moles of iodine liberated

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b) Calculate the number of moles of OCl^- dissolved in 1 litre in bleaching powder

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c) The mass of $Ca(OH)_2$ in the sample of bleaching powder

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7.13 DETERMINATION OF SODIUM CHLORATE (I) CONTENT IN JIK

Requirements

- Burette (50ml), pipette (10 or 20 or 25ml), 2 conical flasks (250ml), BA2 which is 10% KI solution, BA3 which is 0.1M sodium thiosulphate, BA1 which is JIK, BA4 which is 2M sulphuric acid, starch indicator.

Procedure

a) Transfer 15ml of BA1 into 250ml volumetric flask, add distilled water to the mark. Transfer 25ml of this solution into a clean conical flask, add 10ml of BA4 solution. Titrate the liberated iodine with BA3 using starch indicator up to near end-point. Repeat the titration for consistency and tabulate your results

Results

Volume of the pipette used..

Titration number	**1**	**2**	**3**
Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of thiosulphate used (cm^3)			

Titre values used to calculate average volume..

Average volume of thiosulphate

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a) Determine the number of moles of iodine liberated

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b) Calculate the number of moles of OCl^- dissolved in 1 litre in the original BA1 solution

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c) The concentration of NaOCl in the original BA1 in gl^{-1}

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7.14 DETERMINATION OF RELATIVE ATOMIC MASS OF X IN THE XO^- ANION

Requirements

- Burette (50ml), pipette (10 or 20 or 25ml), 2 conical flasks (250ml), 10% KI solution, 0.1M sodium thiosulphate, 1M HCl, starch indicator, solution os potassium iodate made by dissolving 2.69g in 1 litre

  Equation of reaction is,

$$XO^-_{(aq)} + 6I^-_{(aq)} + 6H^+_{(aq)} \longrightarrow X^-_{(aq)} + 3I_{2(aq)} + 3H_2O_{(l)}$$

Procedure

a) Pipette 25ml of the iodate solution into a clean conical flask, add equal volume of dilute HCl acid then add 10% KI solution.

b) Titrate the iodine liberated with sodium thiosulphate solution from the burette with constant shaking, when the colour of the mixture becomes pale yellow, add 2ml of starch solution and continue titration until the colour turns from blue to colourless. Repeat the titration for consistency and record the titres in the table below.

Results

Volume of the pipette used...

Titration number	**1**	**2**	**3**
Final burette reading (cm^3)			
Initial burette reading (cm^3)			
Volume of thiosulphate used (cm^3)			

Titre values used to calculate average volume..

Average volume of thiosulphate

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a) Calculate the concentration of the iodate, XO_3^- in moles per litre

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b) Calculate the molar mass of iodate, XO_3^-

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Determine the relative atomic mass of X

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8.0 QUALITATIVE ANALYSIS PRACTICALS (INORGANIC PRACTICALS)

Safety is the most important aspect for students of chemistry in the laboratory; therefore students should take caution and responsibility. Students should revise all rules and regulations of the laboratory (O level chemistry) before performing practical.

Qualitative analysis requires a student to carry out a number of sample tests on one or more substances mixed the observation a recorded from which deduction about the substance is made. The common apparatus used in this case include,

- Test tube rack, test tubes, boiling tubes, spatula, Bunsen burner, test tube holder, glass rod, filter paper, filter funnel, wash bottle, a set of reagent bottles

Students at this level are required to make use of preliminary test and few specific tests. They are expected to know the reactions of the following ions and therefore are able to identify them

- **ANIONS**

Carbonate (CO_3^{2-}), chloride (Cl^-), bromide (Br^-), chlorite (ClO^-), thiosulphate ($S_2O_3^{2-}$), iodide (I^-), nitrate (NO_3^-), sulphate (SO_4^{2-}), sulphite (SO_3^{2-}), oxalate ($C_2O_4^{2-}$), chromate (CrO_4^{2-}), dichromate ($Cr_2O_7^{2-}$), hydrogen carbonate (HCO_3^-), manganate (MnO_4^-), ethanoate (CH_3COO^-), phosphate (PO_4^{3-})

- **CATIONS**

Aluminium (Al^{3+}), ammonium (NH_4^+), calcium (Ca^{2+}), iron (II) (Fe^{2+}), iron (III) (Fe^{3+}), lead (Pb^{2+}), zinc (Zn^{2+}), Copper (Cu^{2+}), Nickel (Ni^{2+}), Magnessium (Mg^{2+}), chromium (Cr^{3+}), vanadium (V^{5+}), cobalt (Co^{2+}), manganese (Mn^{2+}), barium (Ba^{2+})

8.1 QUANTITIES OF SUBSTANCES FOE TESTS

a) Volumes of liquids

Volumes of liquids may be measured accurately using the following apparatus

i. Measuring cylinders (100, 50,25, 10, 5cm^3)
ii. Beakers (500, 250, 150, 100cm^3)
iii. Teat pipettes etc

Accurate volumes are rare in qualitative analysis but if required a measuring cylinder is used. Place the cylinder on the flat bench and pour the liquid. Care should be taken when approaching the specified quantity, put your eyes on the same level with the meniscus of the liquid. Take the reading from the bottom of the meniscus except for mercury. Volume of liquids can also be estimated if it is small less 10ml, using a test tube of 1cm diameter. The volume of liquid can be done drop wise, a teat pipette works for this case, though reagent bottles can also be used. Hold the test tube containing the test substance in left hand, using your right hand, get the reagent bottle and tip off the liquid from the reagent bottle in small bits. Make sure the level on the bottle is against your palm. This is to avoid damage to the label by some run-off from the bottle. Shake the test tube; take observations as the number of drops increase. Add more of the liquid until it is five times as the original volume (then you have added in excess)

b) **Making solutions**

i. Dissolving in water.

At this level, water and dilute acids will be used as solvents, normally you are provided with unknown substances in a solid form and you are required to make an aqueous solution of it before you carry out any tests. Put a spatula end-full of the solid into a test tube and about 5ml of water and shake vigorously, if it does not dissolve easily warm gently. Note whether it dissolves completely or partially, if it dissolves completely note the colour of the solution. If it is partially soluble, filter and note the colour of both filtrate and residue, Heat change is sometimes observed and recorded. Usually, dissolving of solids in water does not evolve gases.

ii. Dissolving in acids

Put a spatula end-full of the solid in the test tube. Add cold dilute acid (0.5ml) to start with, the acid is usually dilute nitric acid, however sometimes dilute or concentrated hydrochloric acid is used instead. If there is effervescence, try to smell the gas if any, use litmus paper and other tests like glowing splint and lime water. While you are getting ready, put your thumb across the mouth of the test tube. Add more and slowly in as the instruction require, note the colours, gases, heat changes and record them. You should be able to make up this solution correctly as required for it is the stock you will use for experiments.

iii. To acidify

This is to add a little of the acid to the liquid, there is visible reaction normally with concentrated acids, be careful and use a dropper always if possible. If asked to divide the solution into parts or several portions. The portions should be 1-2cm^3 and leave some of the solution for emergency.

c) **weighing**

Accurate weighing is not quite necessary; the mass of the solid can be estimated using a spatula. For example small tip of the spatula (0.1g), spatula end full (0.5g), a heaped spatula head (1.0g)

8.2 PRELIMINARY TESTS

a) Appearance of substances

Note the colour of the solid and its aqueous solution whether asked for or not. Colour can be a good guide to what metallic ions may be present because most compounds of the particular metallic ion have the same general colour in solution. But never identify the compound on the basis of colour alone. The colour of the solids can be very deceptive at times e.g anhydrous copper (II) oxalate is a yellow powder.

Note

i. If the compound is white and its solution in water is colourless, this indicates that the transition metal ions are probably absent. The most likely metallic ions are present are Ca^{2+}, Mg^{2+}, Ba^{2+}, Al^{3+}, Pb^{2+}, Zn^{2+}, NH_4^+, Sn^{2+}.NH_4^+ has a smell of ammonia.

ii. If the compound is black solid or powder, it is probably oxide or sulphide of carbon

iii. If the solid compound and its solution is coloured, this indicates that the transition metal ion is probably present. These include
Cu^{2+} (blue), Fe^{2+} (green), F^{3+} (brown), Ni^{2+} (green), Co^{2+} (pink), Cr^{3+} (green), Cr^{6+} (yellow/orange), Mn^{2+}(very pale pink, not visible in solution), Mn^{7+} (very dark purple, pink in dilute acid)

b) **Flame test.**

These tests are rarely asked for your examination, the flame test rod must be thoroughly cleaned with concentrated hydrochloric acid .chlorides are preferred because they are volatile than other salts, drops of the solution are heated in the flame and then colour of the flame observed. Flame tests are common in organic chemistry practical.

c) **Action of heat on solids**

Heating a solid may result into decomposition of the compound. This may result into formation of the sublimate and evolution of gases or formation of the residue which is left behind. If the solid sublimes then it is possibly covalent compound like NH_4^+

i.Residue

The residue is usually the oxide of the metal, the colour of the oxide gives a clue about the metal as below

Observations	**Deductions**
Yellow when hot and white when cold	Zinc oxide, ZnO
Brown when hot and yellow when cold	Lead oxide, PbO
Black when hot and brown when cold	Iron (III) oxide, F_2O_3
Green	Chromium (III) oxide, Cr_2O_3
Black	MnO, CuO, FeO, NiO
White	MgO, Al_2O_3

ii. Gases

Observations	**Deductions**
Water vapour	Hydrated salt present
CO_2	CO_3^{2-}, $C_2O_4^{2-}$, HCO_3^- probably present
NO_2 and O_2	NO_3^- other than those of Na^+, K^+, NH_4^+
SO_2	SO_4^{2-}, $S_2O_3^{2-}$
NH_3/sublimate	NH_4^+
HCl gas	Certain hydrated chlorides except those of group 1
Cl_2 gas	Unstable chlorides eg $CoCl_2$, $CuCl_2$ present
SO_3	Sulphite apart from those of gp 1,Ca^{2+}, Ba^{2+}

d) Reactions of cations

These cations are categorised into coloured and non-coloured. They are identified by use of sodium hydroxide and ammonium solutions. The reactions involve precipitation of the insoluble metal hydroxides, certain hydroxides dissolve in excess alkali due to formation of soluble complexes.

Reactions of cations with sodium hydroxide solution

Reagent	**Observation**	**Deduction**
Addition of 2M NaOH solution drop wise until excess	White ppt insoluble in excess	Mg^{2+}, $Ca^{2+,}$ Ba^{2+} probably present
	White ppt soluble in excess	Al^{3+}, Zn^{2+}, Pb^{2+} probably present
	Solution remains colourless with evolution of a colourless chocking smell gas, gas turns moist red litmus paper blue and forms dense white fumes with conc. HCl	Gas is NH_3 White dense fumes is NH_4Cl Therefore NH_4^+ probably present
	White ppt which rapidly turns brown	Mn^{2+} probably present
	Blue ppt insoluble in excess	Cu^{2+} probably present
	Blue ppt insoluble in excess, turns pink and brown on standing	Co^{2+} probably present
	Green ppt insoluble in excess	Ni^{2+}. Cu^{+} probably present
	Green ppt insoluble in excess, turns brown on standing	Fe^{3+} probably present
	Green ppt soluble in excess	Cr^{3+} present
	Brown ppt insoluble in excess	Fe^{3+} probably present

Reactions of cations with ammonia solution

Reagent	**Observations**	**Deductions**
add 2m ammonia solution drop wise unil excess	White ppt insoluble in excess	Al^{3+}, Pb^{2+} probably present
	White ppt soluble in excess	Zn^{2+} present,[$Zn(OH)_4]^{2-}$ formed
	White ppt insoluble in excess, though because of the weak basic character of ammonia, the ppt may not show	Mg^{2+} probably present
	Blue ppt soluble in excess forming a deep blue solution	Cu^{2+}, Co^{2+} probably present
	White ppt which rapidly turns brown	Mn^{2+} probably present
	Green ppt soluble in excess Note. Light blue solution is formed for Ni^{2+}	Cr^{3+}, Ni^{2+} probably present
	Green ppt insoluble in excess	Fe^{2+} probably present
	Brown ppt insoluble in excess	Fe^{3+} probably present

8.3 CONFIRMATORY TESTS FOR CATIONS

TEST	OBSERVATION	DEDUCTION
1. To the solution, add aqueous ammonia drop wise until the solution is alkaline, then 2 drops of alizarin solution	Pink colouration formed	Al^{3+} confirmed present
2. To the aqueous solution of the unknown, add few drops of mercury (II) chloride solution followed by potassium iodide solution until the red ppt of mercury (II) iodide just dissolves due to formation of the complex, now add NaOH until excess	Yellow or brown ppt or solution is formed	NH_4^+ confirmed present
3. To the solution of the unknown, add dilute NaOH until the solution is alkaline and then H_2O_2 solution to the mixture followed by lead (II) ethanoate	Yellow solution which turns into a yellow ppt on addition of lead (II) ethanoate	Cr^{3+} confimed present
4. To the unknown solution, add 3 drops of fairly conc. Potassium or ammonium thiocynate solution **OR.** Add ethanoic acid to the unknown until the solution is acidic, then add 3 drops of KNO_3 solution and shake	Blue solution formed Yellow crystalline ppt formed	Co^{2+} confirmed present
5. Add 3 drops of aqueous potassium hexacynaferare (II) to the solution of un known **OR.** To the solution of the un known add few drops of potassium iodide	Brown ppt is formed Brown colour and an off white ppt	Cu^{2+} confirmed present
6. To the solution of the un known, add drops of conc. Nitric acid followed by a small quantity of solid sodium bismuthate and boil	A violet (purple) colouration formed	Mn^{2+} confirmed present
7. To the solution of the un known, add solid NH_4Cl followed by 3-4 drops of di-sodium monohydrogen phosphate, then add aqueous ammonia until excess	White crystalline ppt insoluble in excess ammonia	Mg^{2+} confirmed present
8. To the solution of the un known, add solid NH_4Cl followed by 3-4 drops of di-sodium monohydrogen monophosphate, then aqueous ammonia until excess	White ppt soluble in excess ammonia	Zn^{2+} confirmed present
9. Add aqueous ammonia to the solution of the un known until it	A red ppt is formed	Ni^{2+} confirmed present

is alkaline, add 3 drops of dimethylglycoxime		
10. Add potassium iodide solution drop wise to the u known **OR.** add 2-3drops of aqueous potassium chromate to the un known **OR.** add 3-4 drops of dilute HCl or aqueous NaCl to the un known and heat	Yellow ppt is formed Yellow ppt is formed White ppt soluble on warming and reappears on cooling	PbI_2 formed Pb^{2+} confirmed present $PbCrO_4$ formed Pb^{2+} confirmed present $PbCl_2$ formed Pb^{2+} confirmed present
11. Add potassium hexacynoferate (III) solution to the un known **OR.** add conc. Nitric acid to the solution of un known and boil the mixture	Dark blue ppt is formed Yellow or brown solution with evolution of a brown gas (NO_2)	Fe^{2+} confirmed present
12. Add potassium hexacynoferate (II) solution to the un known **OR**. add 2-3 drops of potassium thiocynate solution to the solution of the un known	Dark blue ppt formed Red colour observed	Fe^{3+} confirmed present
13. Add 3-4 drops of sulphuric acid or sodium sulphate solution to the un known **OR.** add 2-3 drops of aqueous potassium chromate followed by ethanoic acid	White ppt formed Yellow ppt insoluble in ethanoic acid	$BaSO_4$ formed Ba^{2+} confirmed present $BaCrO_4$ formed Ba^{2+} confirmed present
14. To the solution of the un known, add aqueous ammonia until the solution is alkaline, then add a few drops of ammonia ethanedioate to the the mixture	White ppt formed	CaC_2O_4 formed Ca^{2+} confirmed present

8.4 CONFIRMATORY TESTS FOR ANIONS

TEST	OBSERVATION	DEDUCTION
1. (a) To the solution of the un known, add 2-3 drops of aqueous silver nitrate followed by excess nitric acid	i) White ppt formed ii) Pale yellow ppt formed iii)Yellow ppt formed N.B. AgCl and AgBr dissolve in excess ammonia	i) Cl^- confirmed present ii) Br^- confirmed present iii) I^- confirmed present

(b)To the solution of the un known add aqueous solution of lead (II) ions	i) yellow ppt formed ii) white ppt soluble on warming and reappears on cooling	i. I^- confirmed present . ii. Cl^- confirmed present
(c) To solution of the un known, add aqueous solution of copper (II) sulphate	Brown ppt is formed	CuI formed I- confirmed present
2. (a) To the un known solution, add aqueous barium nitrate followed by excess dilute nitric acid **OR.** To the un known solution, add aqueous barium chloride followed by dilute HCl acid	White ppt insoluble in acid	$BaSO_4$ formed SO_4^{2-} confirmed present
(b) To the un known solution, add lead (II) ethanoate solution **OR.** lead (II) nitrate followed by dilute nitric acid	White ppt insoluble in the acid	$PbSO_4$ formed SO_4^{2-} confirmed present
3. (a) to the un known solid, add conc. H_2SO_4 and warm if necessary	Brown fumes evolved which condense into an oily liquid at the sides of test tube	NO_3^- confirmed present
(b) to the solution of un known, add aqueous solution of freshly prepared iron (II) sulphate followed by few drops of conc. H_2SO_4 acid	Brown ring formed	NO_3^- confirmed present
(c) To the un known solution, add few pieces of copper turnings followed by about 5ml of conc. H_2SO_4 and heat	Brown fumes evolved	NO_3^- confirmed present
(d) To the solution of un known or solid and add NaOH solution followed by Zinc or Aluminium powder and heat	colourless gas with chocking smell evolved, gas turns moist red litmus paper blue and forms dense white fumes with Conc. HCl acid	Gas is NH_3 NO_3^- confirmed present
4. (a) To the unknown solution, add dilute HCl acid or nitric acid	Colourless gas evolved, gas turns moist blue litmus paper red, and turns lime water milky	CO_2 gas evolved CO_3^{2-} confirmed present
(b) Add a few drops of $MgSO_4$ solution to the un known	White ppt formed	$MgCO_3$ formed CO_3^{2-} confirmed present

5. (a) To the solution of the un known, add freshly prepared aqueous iron (II) sulphate followed by dilute ethanoic acid	Brown solution formed	$Fe(NO)^-.5H_2O$ formed NO_2^- confirmed present
6. (a) add oxidising agent like H_2O_2 to un known solution. Test for a sulphate in the resultant mixture	Positive result for SO_4^{2-} observed	SO_3^{2-} confirmed present
(b) To the un known solution. Add aqueous silver nitrate drop wise until excess	White ppt soluble in excess $AgNO_3$	SO_3^{2-} confirmed present
7. (a) To the solution of the un known, add 2-3 drops of silver nitrate solution followed by excess aqueous ammonia	White ppt soluble in excess ammonia	C_2O_4 confirmed present
(b) To un known solution, add dilute H_2SO_4 drop wise until the solution is acidic, heat till uncomfortable to touch, then add drops of $KMnO_4$ solution to the hot solution	Colour of permanganate turns from purple to colourless. Colourless gas evolved, gas turns damp blue litmus paper red and lime water milky	MnO_4^- reduced to Mn^{2+} Gas is CO_2 $C_2O_4^{2-}$ confirmed present

8.5 SUMMARY OF TESTS FOR CATIONS

CATION	TEST	OBSERVATION
Ba^{2+}	i. Heat a little of the solid on a spatula	Green flame
	ii. Addition of NH_3 and NaOH solutions	white ppt insoluble in excess
	iii. Addition of CO_3^{2-}	White ppt soluble in the acid
	iv. Addition of $C_2O_4^{2-}$	White ppt, insoluble in H_2O but soluble in hot ethanoic acid, acetic acid, mineral acids
	v. Addition of CrO_4^{2-}	Yellow ppt insoluble in ethanoic acid but soluble in mineral acids to form red solution
	vi. Addition of SO_4^{2-}	White ppt formed
Al^{2+}	i. Addition of NaOH solution	White ppt soluble in excess
	ii. Addition of NH_3 solution	White ppt insoluble in excess
	iii. Addition of $(NH4)_2S$	White ppt, evolution of H_2S due to hydrolysis
	iv. Addition of OH^-, separate the ppt $Al(OH)_3$ from the liquid. Dissolve	A red ppt formed/pink colouration

	the ppt in dilute HCl, add little ammonium ethanoate solution on aluminon reagent and excess$(NH_4)_2CO_3$ solid	
	v. Addition of CO_3^{2-}	White ppt, Colourless gas evolved, gas turns moist blue litmus paper red, and turns lime water milky (CO_2) [due to hydrolysis of $Al_2(CO_3)_3$
Zn^{2+}	i. Addition of NaOH solution	White ppt soluble in excess
	ii. Addition of NH_3 solution	White ppt soluble in excess
	iii. Addition of CO_3^{2-}	White ppt
	iv. Filter off its ppt, heat strongly until no further change, allow to cool	Residue is yellow hot, white cold.
	v. Addition of solid NH_4Cl followed by di-sodium monohydrogenphospate solution and then ammonia solution	White ppt, soluble in ammonia solution
	vi. Addition of H_2S in neutral solution	Yellowish partial ppt (ZnS)
	vii.Addition of H_2S in acidic solution	No ppt
	viii. Addition of H_2S in alkaline solution	Complete yellowish ppt of ZnS
	ix. Addition of $(NH_4)_2S$	Yellowish ppt of ZnS soluble in acid
	x. Addition of acid (acidify) + 0.5ml of very dilute $CuSO_4$ + 2ml of ammonium mercuric thiocynate	Violet colouration
	xi. Repeat the above test with dilute $CuSO_4$ or any cobalt salt	Blue ppt is formed
Pb^{2+}	i. Addition of NaOH solution	White ppt soluble in excess
	ii. Addition of aqueous NH_3	White ppt insoluble in excess
	iii. Addition of dilute HCl or NaCl solution	White ppt soluble on warming and reappears on cooling
	iv. Decant as much as of the supernatant liquid as possible from the above ppt, add conc.HCl	Ppt dissolves due to formation of a complex $PbCl_{2(s)} + 2HCl_{(aq)} \longrightarrow H_2PbCl_{4(aq)}$
	v. Addition of CrO_4^{2-}	Yellow ppt formed, insoluble in ethanoic acid and NH_3 solution

	vi. Addition of $(NH_4)_2S$ or H_2S	Black ppt of PbS
	vii.Addition of I^-	Yellow ppt, dissolves in excess
	viii. Addition of SO_4^{2-}	White ppt
	ix. To the ppt above, add conc. Ammonium acetate solution	Ppt dissolves
Cr^{3+}	i. Addition of NaOH solution	Green ppt soluble in excess forming pink/violet solution
	ii. Addition of aq NH_3	Green ppt soluble in excess forming green solution
	iii. To solution or ppt above (ii), add H_2O_2	Yellow solution of chromate formed
	iv. To the solution in (iii) add $AgNO_3$	Red ppt of $Ag_2Cr_2O_4$
	v. Addition of CO_3^{2-}	Green ppt of $Cr_2(CO_3)_3$ with evolution Colourless gas, gas turns moist blue litmus paper red, and turns lime water milky (CO_2) due to hydrolysis of $Cr_2(CO_3)_3$
	vi. Addition of 1ml of butan-1-ol + dilute H_2SO_4 + 0.5ml of H_2O_2 (heating may be necessary)	Organic layer is coloured blue due to formation of chromium peroxide
	iii. vAddition of $(NH_4)_2S$	Green ppt withevolution of H_2S gas
Mn^{2+}	i. Addition of $NaOH/NH_3$ solution	White ppt which rapidly turns brown
	ii. Addition of $(NH_4)_2S$	Pink ppt, darkens on standing dissolves in acid or alkali to form faint yellowish solution
	iii. Addition of cold solution + HNO_3 + solid sodium bismuthate and shake	Violet solution due to formation of permanganic acid, $HMnO_4$
	iv. Addition of conc. HNO_3 + PbO or Pb_3O_4 boil the mixture and allow to cool	Violet solution results
Fe^{2+}	i. Addition of $NaOH/NH_3$ solution	Green ppt insoluble in excess, turns brown on standing
	ii. Addition of CO_3^{2-}	Green ppt, with evolution Colourless gas, gas turns moist blue litmus paper red, and turns lime water milky (CO_2) due to hydrolysis of $Fe(CO_3)$
	iii. Addition of potassium hexacynoferate (III) or potassium ferricynide	Dark blue ppt
	iv. Addition of NH_3 solution followed by dimethylglycoxime	Ppt formed (not formed by Fe^{3+} but Ni^{2+}, Co^{2+}, Cu^{2+} may interfere

	v. Addition of H_2S a) Acid b) Alkali c) In acid in which [H^+] has been reduced by adding sodium ethanoate	i. No observable change ii. Black ppt of FeS iii. Black ppt of FeS
	d) Acidify then add $KMnO_4$ solution	$KMnO_4$ turns from purple to colourless
	e) Addition of $(NH_4)_2S$	Black ppt/ pale green ppt
Fe^{3+}	a) Addition of $NaOH/NH_3$ solutions	Brown ppt insoluble in excess
	b) Addition of $(NH_4)_2S$	Black mixture of Fe_2S_3 and S dissolves in acid leaving yellow sulphur
	c) Addition of potassium hexacynoferate (II)/ferrocyanide	Dark/intense blue ppt
	d) Addition of sodium ethanoate	Red/brown ppt of iron (III) ethanoate
	e) Addition of thiocyanate/potassium thiocyanate	Deep red solution $Fe^{3+}_{(aq)} + 3SCN^{-(aq)} \longrightarrow Fe(SCN)_{3(aq)}$
Ni^{2+}	a) Addition of NaOH solution	Green ppt insoluble in excess
	b) Addition of NH_3 solution	Green ppt soluble in excess forming light blue solution
	c) Addition of H_2S in i. In neutral media ii. In acid iii. In alkali	Light ppt (partial precipitation of black NiS Dense ppt No ppt formed
	d) Addition of $(NH_4)_2S$	Black ppt soluble in acid
	e) Addition of CO_3^{2-}	Green ppt
	f) Addition of NH_3 solution followed by dimethylglycoxime	Red ppt is formed
	g) Addition of dilute H_2SO_4 and then $KMnO_4$ solution	No observable change unlike Fe^{2+}
Cu^{2+}	i. Flame test	Green flame
	ii. Addition of NaOH solution	Blue ppt insoluble in excess
	iii. Boil the above ppt	Black ppt results
	iv. Addition of NH_3 solution	Blue ppt soluble in excess forming deep blue solution
	v. Addition of CO_3^{2-}	Blue/green ppt of $CUCO_3$
	vi. Addition of H_2S in acid/neutral solution or $(NH_4)_2S$	Black ppt of CuS soluble in hot dilute HNO_3 acid
	vii. Addition of KI solution	CuI_2 formed which decomposes to CuI and free I_2 that dissolves in KI and turns solution brown Addition of $Na_2S_2O_3$ removes the I_2

	iii. Addition of potassium hexacynoferate (II) solution	Brown ppt insoluble in excess
Ag^+	i. Addition of NaOH solution	Brown ppt of AgO insoluble in excess
	ii. Addition of NH_3 solution	Brown ppt soluble in excess due formation of $Ag(NH3)_2^+$ complex
	iii. Addition of Cl^-	White ppt, darkens on standing due to decomposition into Ag and Cl^-
	iv. To ppt in (iii) above add NH_3 solution	Ppt dissolves
	v. To solution in (iv), add dilute HNO_3 acid	White ppt reappears
	vi. Addition of K_2CrO_4 in neutral solution	Red ppt of Ag_2CrO_4 soluble in dilute nitric acid/NH_3 solution
	vii. Addition of KI solution	Yellow ppt insoluble in conc. NH_3 solution.
Co^{2+}	i. Addition of NaOH solution	Blue ppt insoluble in excess, turns pink and brown on standing
	ii. Addition of NH_3 solution	Blue ppt turns pink and brown
	iii. $Mt_{3(aq)}$	Blue ppt darkens in conc. NH_3 to form yellow brown solution
	iv. Addition of Conc. HCl	Pink solution turns deep blue
	v. Addition of conc. HCl + Co^{2+} + pentanol + solid NH_4SCN and shake	Blue colour appears in pentanol layer. This confirms Co^{2+}

1.2. SUMMARY OF TESTS FOR ANIONS

ANION	TEST	OBSERVATION
CO_3^{2-}	solubility	Insoluble except carbonates of K^+, Na^+, NH_4^+
	If soluble add i. Phenolphthalein indicator ii. Add Ca^{2+}, Mg^{2+}	Pink colouration White ppt formed
	Add dilute acid and test gas with lime water	Dissolves with effervescence and Colourless gas evolved, gas turns damp blue litmus paper red and lime water milky
	Heat the solid strongly and test gas with lime water	Colourless gas evolved, gas turns damp blue litmus paper red and lime water milky (most carbonates decompose on heating to give the oxide and CO_2 except those of K^+, Na^+. $(NH_4)_2CO_3$ decomposes to H_2O, NH_3, CO_2. $HgCO_3$ and Ag_2CO_3 decomposes to CO_2 and O_2

HCO_3^-	solubility	Solid dissolves
	Heat (observe the sides of boiling tube, test gas with lime water	Colourless gas evolved, gas turns damp blue litmus paper red and lime water milky. Colourless neutral liquid condenses on sides of boiling tube, liquid turns white anhydrous copper (II) sulphate blue
	Addition of acid, test gas with lime water	Dissolves with effervescence and Colourless gas evolved, gas turns damp blue litmus paper red and lime water milky
	Add Ca^{2+}/Mg^{2+} and boil	No ppt forms in the cold, ppt formed on boiling
$C_2O_4^{2-}$	Addition of dilute H_2SO_4 + $KMnO_4$ and warm	Purple colour turns colourless unlike CO_3^{2-} $2MnO_4^-{}_{(aq)} + 16H^+{}_{(aq)} + 5H_2O_{2(aq)} \rightarrow 10H^+{}_{(aq)} + 5O_{2(g)} + 2Mn^{2+} + 8H_2O_{(l)}$
	Addition of Ca^{2+} or Ba^{2+} or Pb^{2+} in the presence of NH_3 solution	White ppt formed
	Addition of conc. H_2SO_4 acid, test gas with lime water	Colourless gas evolved, gas turns damp blue litmus paper red and lime water milky.
	Add Ag^+ then NH_3 or HNO_3 solution	White ppt dissolves in NH_3 or HNO_3 solution. some oxalates decompose in presence of acid and CO_2 is given off
CH_3COO^-	Add Na_2CO_3 solution boil and filter, neutralise with HNO_3 and NH_3 solution then add $FeCl_3$ solution	Red ppt formed dissolves in dilute HCl acid
	Dissolve in little ethanol, warm then pour the mixture in 10ml of H_2O. Detect the smell	Sweet /fruity smell of ethyl ethanoate detected
NO_3^-	Add freshly prepared $FeSO_4$ then a little dilute H_2SO_4 then carefully conc. H_2SO_4 down the sides of the tube so that there are two separate layers	Brown ring formed Conc. H_2SO_4 reacts with nitrate to form HNO_3 $NO_3^-{}_{(aq)} + H_2SO_{4(aq)} \rightarrow HSO_4^-{}_{(aq)} + HNO_{3(aq)}$ HNO_3 is reduced by $FeSO_4$ in presence of H_2SO_4 to NO $6FeSO_{4(aq)} + 2HNO_{3(aq)} + 3H_2SO_{4(aq)} \rightarrow 3Fe(SO_4)_{3(aq)} + 4H_2O_{(l)} + 2NO$ The NO reacts with the remaining $FeSO_4$ brown complex which appears as brown ring. $FeSO_{4(aq)} + NO \rightarrow FeSO_4.NO$
	Add NaOH + devada's alloy and warm (test gas with moist red litmus paper or open bottle of conc. HCl acid	Moist red litmus paper turns blue, white fumes are formed with conc.HCl (NH_3 given off) devada's alloy is composed of 50% Cu, 45% Al, 5% Zn
	Heat strongly	Brown fumes which turns moist blue litmus paper red and darkens $FeSO_4$ solution (NO_2 gas)

		Most NO_3^- decompose to NO_2, metal oxide and O_2 except those of K^+, Na^+, which decompose to metal nitrite and O_2. But those of Hg and Ag give metal, NO_2 and O_2
NO_2^-	Addition of dilute acid	Brown fumes which turns moist blue litmus paper red and darkens $FeSO_4$ solution (NO_2)
	Addition of $FeSO_4$ and then dilute H_2SO_4 acid	Brown coloration, due to formation of brown complex $[Fe(NO)]^{2+}$
	Addition of dilute H_2SO_4 + $KMnO_4$	Purple colour turns colourless, NO_2^- is a reducing agent unlike NO_3^- $2MnO_4^-{}_{(aq)} + 5NO_2^-{}_{(aq)} + 6H^+_{(aq)} \rightarrow 2Mn^{2+}_{(aq)} + 5HNO_{3(aq)} + 3H_2O_{(aq)}$
	Add NaOH + Zn (test gas with conc. HCl	Colourless chocking gas evolved, gas turns moist red litmus paper blue, forms dense white fumes with conc.HCl $NO_2^-{}_{(aq)} + 3Zn_{(s)} + 5NaOH_{(aq)} \rightarrow NH_{3(g)} + 2Na(ZnO_2) + H_2O_{(l)}$
	Addition of KI + dilute HCl then ethanoic acid	Deep red colouration $HCl_{(aq)} + NO_2^-{}_{(aq)} \rightarrow HNO_{2(aq)} + Cl^-_{(aq)}$ $2KI_{(aq)} + 2NO_2^-{}_{(aq)} + CH_3COOH_{(aq)} \rightarrow 2CH_3COOK_{(aq)} + I_{2(aq)}$ $2NO_{(g)} + 2H_2O_{(l)}$
SO_4^{2-}	Addition of Ba^{2+}	White ppt formed
	Heat strongly	Evolution of acidic gases which turns purple MnO_4^- colourless/ turns orange $Cr_2O_7^{2-}$ green and darkens Ba^{2+} ions ie SO_2 and SO_3
SO_3^{2-}	Add dilute acid (test gas by passing it through MnO_4^- or $Cr_2O_7^{2-}$)	Evolution of acidic gas which turns purple MnO_4^- colourless/ turns orange $Cr_2O_7^{2-}$ green $5SO_{2(g)} + 2MnO_4^-{}_{(aq)} + 4H^+_{(aq)} \rightarrow 5SO_4^{2-}{}_{(aq)} + 2Mn^{2+}_{(aq)} + 2H_2O_{(l)}$ $3SO_{2(g)} + Cr_2O_7^{2-}{}_{(aq)} + 2H^+_{(aq)} \rightarrow 2Cr^{3+}_{(aq)} + SO_4^{2-}{}_{(aq)} + H_2O_{(l)}$
	Add Ba^{2+} then dilute acid	White ppt soluble in acid with Evolution of acidic gas which turns purple MnO_4^- colourless/ turns orange $Cr_2O_7^{2-}$ green (SO_2 gas)
	Heat in a test tube	Evolution of acidic gas which turns purple MnO_4^- colourless/ turns orange $Cr_2O_7^{2-}$ green (SO_2 gas), colour of the oxide when hot and cold required

$S_2O_3^{2-}$	Add dilute acid (test gas by passing it through MnO_4^- or $Cr_2O_7^{2-}$)	Yellow ppt of Sulphur formed with Evolution of acidic gas which turns purple MnO_4^- colourless/ turns orange $Cr_2O_7^{2-}$ green (SO_2 gas)
	Heat the solid	Evolution of acidic gas which turns purple MnO_4^- colourless/ turns orange $Cr_2O_7^{2-}$ green (SO_2 gas), there may be some traces of H_2S
	Addition of I_2 in KI	Solution turns from brown to colourless
	Addition of Ba^{2+} solution	No observable change, solution remains colourless **unlike SO_4^{2-} and SO_3^{2-} which form white ppt**
	Addition of $AgNO_3$ solution slowly	White ppt which rapidly turns brown and dissolves in excess reagent $S_2O_3^{2-}{}_{(aq)} + Ag^+{}_{(aq)} \longrightarrow Ag_2S_2O_{3(s)}$ dissolves in excess to form $Ag^+[\ Ag_2S_2O_3]^-{}_{(aq)}$
PO_4^{3-}	Addition of Ag	Yellow ppt of Ag_3PO_4 soluble in dilute HNO_3/NH_3 solution
	If insoluble solid, add conc.HNO_3 + excess ammonium molybdate and warm	Yellow ppt soluble in hot or alkaline solution
	To solution + solution containing $MgCl_2$, NH_4Cl and ammonia	White ppt formed (H_4NMgPO_4)
CrO_4^{2-}	Addition of Ag^+	Red ppt soluble in dilute HCl acid/NH_3 solution
	Addition of an acid	Solution turns from yellow to orange due to formation of the dichromate $2CrO_4^{2-}{}_{(aq)} + 2H^+{}_{(aq)} \longrightarrow 2Cr_2O_7^{2-}{}_{(aq)} + H_2O_{(l)}$
	Addition of a reducing agent like SO_2 or H_2S	Solution turns from yellow to green due to formation of Cr^{3+}
	Addition of acid + ether + H_2O_2 **(shake but not near a flame)**	Blue colour appears in the ether layer
	Solid + ethanol + dilute H_2SO_4 warm and detect the smell	Mixture turns green (formation of Cr^{3+}), fruity smell detected
	Add oxidising agent like H_2O_2, MnO_4^-, PbO_2, MnO_2, Cl_2, $Cr_2O_7^{2-}$, HNO_3, NO_3^-, Br_2, Fe^{2+} then dilute H_2SO_4 + KI and starch	Brown colouration which turns deep blue on addition of starch
	Add reducing agents like Cr^{2+}, Sn^{2+}, $HCOO^-$, $C_2O_2^{2-}$, $S_2O_3^{2-}$, S^{2-}, SO_3^{2-}, NO_2^- then dilute H_2SO_4 + $KMnO_4$ solution drop wise	Purple colour of solution turns colourless
Cl^-	Addition of $AgNO_3$ in dilute HNO_3 acid/NH_3 solution	White ppt soluble in excess NH_3/HNO_3 solutions

	Solid + conc. H_2SO_4 acid (test the gas with conc. NH_3)	Colourless gas with chocking smell, gas turns moist red litmus paper blue and forms dense white fumes with NH_3
	Addition of $K_2Cr_2O_7$ followed by conc. H_2SO_4 and warm	Red brown gas evolved, soluble in H_2O forming yellow solution
	Solid +MnO_2 +conc. H_2SO_4 (test gas with KI or KBr solutions)	Yellowish-green gas liberated, gas gives deep brown colouration with KI or deep red colouration with KBr (MnO_4^- may be an oxidising agent)
ClO^-	Add acid and detect smell	Smell of chlorine
	Add Co^{2+} solution	Black ppt of Co_3O_4 formed with evolution of a colourless neutral gas that relights a glowing split. evolution of O_2 becomes rapid due catalytic action of Co^{2+} and Co^{3+} which are (contained in Co_3O_4) on decomposition
	Addition of Pb^{2+} and boil	Ppt of PbO_2 formed gradually turns brown $ClO^-_{(aq)} + Pb^{2+}_{(aq)} + 2OH^-_{(aq)} \xrightarrow{} PbO_{2(s)} + Cl^-_{(aq)} + H_2O_{(l)}$
Br^-	Addition of $AgNO_3$ in dilute HNO_3	Pale yellow ppt formed insoluble in acid but dissolves in NH_3 solution
	Addition of chlorine water or bleaching powder + BrO_3	The solution slowly becomes dark brown due to displaced Br_2
	To solid add conc. H_2SO_4 (pass the gas through KI)	Brown chocking fumes of gas evolved which turns KI solution deep red.
I^-	Addition of $AgNO_3$ in dilute HNO_3	Yellow ppt insoluble in the acid
	Solid + MnO_2 + conc. H_2SO_4(warm)	Violet fumes of I_2 soluble in sodium thiosulphate
	Solid + bleaching powder + dilute HNO_3 + CCl_4	Purple colour in CCl_4 layer
Cl^-, Br^-, I^-	To distinguish between Cl^-, Br^-, I^-. Carry out the following tests Solid + CCl_4 + MnO_2 + conc. H_2SO_4 and shake	i. CCl_4- layer is colourless, Cl^- ii. CCl_4-layer is red brown, Br^- iii. CCl_4-layer is violet, I^-
S^{2-}	Add excess MnO_2 and heat (test gas by passing it through acidified $KMnO_4$ or $K_2Cr_2O_7$ solution	Gas evolved that turns purple MnO_4^- colourless/turns orange $Cr_2O_7^{2-}$ green (SO_2)
	If soluble, add PbO_2	Black ppt of PbS
	To solution, add dervada's alloy + dilute HCl and warm (test gas	Gas with smell of rotten eggs evolved, turns lead acetate paper black, turns purple MnO_4^- colourless/turns orange $Cr_2O_7^{2-}$ green leaving yellow deposit of sulphur

	with acidified $KMnO_4$ or $K_2Cr_2O_7$ solution)	$Cr_2O_7^{2-}{}_{(aq)}+8H^+{}_{(aq)} \rightarrow 3H_2S_{(g)}+Cr^{3+}{}_{(aq)}+3SO_4^{2-}{}_{(aq)}+7H_2O_{(l)} + S$
	Add $Na_2Fe(CN)_2NO$ or sodium nitroprusside)	Purple colouration appears
	Addition of $AgNO_3$ solution	Ppt of Ag_2S insoluble in cold dilute HNO_3 acid

8.6TESTING FOR GASES

First observe the gas then smell it (do not put your nose on the mouth of the test tube where the gas is coming from), put the test tube at a distance from your nose then turn the gas towards your nose with your hand. Then test the gas chemically. Tests which involve change in colour are best done using a strip of litmus paper dipped in testing reagent. If you suspect the gas to be poisonous, carry out the test in the fume cupboard. Never allow poisonous gases to spread in the lab and do not smell a gas you suspect to be poisonous. Some gases irritate the reparatory system although they are not poisonous, therefore do not allow evolution of a gas to continue after you have identified it, stop the reaction by pouring away the reagents and washing with plenty of water.

Gas	**Appearance**	**smell**	**action on moist litmus paper**	**confirmatory test**
Br_2	Deep brown fumes, turns to liquid	Pungent (poisonous)	Bleaches	i. Forms deep red/brown solution with CCl_4 ii. Turns fluorescent paper pink
CH_4				Burns with a bluish flame
C_2H_4		sweet		i. Burns with a yellow flame ii. Turns brown Br_2 water colourless
C_2H_2		None when pure		i. Burns with a yellow sooty flame ii. Gives dirty white ppt with ammoniac
CO		poisonous		Burns with a blue flame to form CO_2
CO_2			Turns moist blue litmus paper red (weakly acidic)	Turns lime water milky
Cl_2	Pale green/yellow green	Pungent (poisonous)	Turns moist blue litmus paper red and bleaches (acidic)	
H_2				Burns with soft pop sound
HCl	Steamy fumes	Pungent (poisonous)	Turns moist blue litmus paper red (strongly acidic)	i. Turns $AgNO_3$ solution milky like HBr, HI ii. Forms dense white fumes with NH_3 iii. Forms white ppt with $Pb(NO_3)_2$ insoluble in hot water
H_2S		Rotten eggs	Turns moist blue litmus paper red (weakly acidic)	i. Gives black ppt with $Pb(NO_3)_2$/lead acetate

				ii. Generally forms black insoluble sulphides with many metals
N_2				Very un reactive, form Mg_3N_2 with burning Mg which dissolves in water to form NH_3 (use tests for NH_3)
I_2	Purple (black solid)	Pungent (poisonous)	Slowly bleaches	i. Turns blue with starch ii. Forms purple colour in CC_4
NH_3		Pungent	Turns moist red litmus paper blue	I. Forms dense white fumes with conc. HCl II. Turns blue $CuSO_4$ solution deep blue (refer to tests for Cu^{2+})
NO_2	Brown	Strong and un pleasant (poisonous)	Turns moist blue litmus paper red	Darkens $FeSO_4$ solution
N_2O		Sweetish (sticky smell)		Rekindles a glowing splint and soluble in water unlike O_2 which is practically insoluble in water
NO	Colourless but readily turns brown in air	No smell but that of NO_2 usually detected	Neutral though NO_2 is detected	Darkens $FeSO_4$ solution
PH_3		Rotten fish		Very inflammable, sometimes without ignition forms white fumes
O_2				Rekindles a glowing splint (practically insoluble in water)
SO_2		Sharp chocking (poisonous)	Turns moist blue litmus paper red	i. Turns purple acidified MnO_4^- colourless ii. Turns orange acidified $Cr_2O_7^{2-}$ green
SO_3	Smoky white fumes	Chocking (poisonous)	Turns moist blue litmus paper red	Forms white ppt with Ba^{2+} soluble in the acid

9.0 QUALITITATIVE ANALYSIS OF ORGANIC COMPOUNDS (ORGANIC PRACTICALS)

1.3. Flame test

Aliphatic compounds burn with non-sooty flame

Aromatic compounds burn with sooty flame

1.4. Solubility tests

Solvent	**Observation**	**Deduction**
Distilled water	insoluble	Higher carboxylic acid and amine probably present
	Soluble giving a solution that turns blue litmus paper red (acidic)	Simple carboxylic acid, salt of amine acylhalide, acid anhydride probably present

	Soluble giving a solution that turns red litmus paper blue	Lower amine, salt of carboxylic acid with strong bases
	Soluble giving a solution neutral to litmus paper	Alcohol, alkanal, alkanone probably present
Dilute sodium hydroxide solution	soluble	Carboxylic acid, phenol probably present
Dilute HCl acid	soluble	Amine probably present
Sodium hydrogen carbonate	soluble	Phenol, carboxylic acid probably present

1.5. Functional groups

Functional group	reagent	Observation	Deduction
Alcohols (R-OH) and phenols (Ar-OH)	Sodium metal	Vigorous effervescence with evolution of colourless neutral gas which burns with soft pop sound	Gas is H_2 Alcohol present (R-OH)
	Acidified $K_2Cr_2O_7$ solution and boil gently	i. Rapid colour change from orange to green ii. slow colour change from orange to green iii. No apparent colour change, solution remains orange	i. Primary alcohol present ii. Secondary alcohol present iii. Tertiary alcohol present
	Luca's regent	i. Clear solution, though some darkening may occur ii. Clear solution turns cloudy after 5mins iii. Clear solution turns cloudy immediately	i. Primary alcohol ii. Secondary alcohol iii. Tertiary alcohol
	Carboxylic acid/acid chloride in presence of conc. H_2SO_4	Sweet smell produced	Alcohol present
	Iodine solution with dilute NaOH	Yellow ppt formed	CH_3CH_2OH and $CH_3CHOHCH_3$ present
	Conc. H_2SO_4	Evolution of acidic chocking gas that decolourises bromine water/$KMnO_4$ solution (SO_2)	Alcohol present
	Iron (III) chloride	Violet colour formed	Phenol confirmed present
Carbonyl compound RC=O	KI and NaOH (iodoform)	Yellow ppt formed	Methyl carbonyl compound ($RCOCH_3$) present

	Brady's reagent (2,4 dinitrophenylhydrazine)	Yellow/orange ppt formed	Carbonyl compound present
	Fehling's solution	Yellow/red/brown ppt	Aldehyde confirmed present
	Tollen's reagent (ammoniacal silver nitrate)	Silver mirror formed on sides of test tube	Aldehyde confirmed present
Carboxylic acids	Soda lime (NaOH)	A gas that burns with yellow flame evolved	Carboxylic acid confirmed present
	Ethanol/conc. H_2SO_4	Sweet smell produced	Carboxylic acid or acid dichloride present
	Fehling's solution	Yellow ppt formed	Methanoic acid (HCOOH) present
	Tollen's reagent	Silver mirror	HCOOH present
amines		i. Clear solution with evolution of acidic brown fumes of gas that darkens $FeSO_4$ solution (NO_2) ii. Clear solution with no evolution of gas iii. Yellow oil	i. Primary aliphatic amine ii. Primary aromatic, tertiary amine iii. Secondary amine present
	Conc. HCl /$NaNO_2$ /2-naphthol in NaOH solution	Formation of bright red dye	Primary aromatic amine confirmed present

1.6. GENERAL REACTIONS AND PROPERTIES OF ORGANIC COMPOUNDS

	Test	Possibilities
Physical state	Solid	Salts, aromatic carboxylic acid, phenols, alkanedioic acids
	Liquid	Alkanols, alkanals, alkanoic acid, ethers and aromatic hydrocarbon
	Smell	Most organic compounds have characteristic smell, however smell should not be relied on in identifying compounds because most of them have similar smell
Solubility (use small amounts)	Soluble in water (test resultant solution with litmus paper)	• C_1-C_3 compounds are soluble • Alkanols, alkanals, alkanones, amide(urea) form neutral solutions • Carboxylic acids, salts of amines or acid chlorides, phenols form acidic solutions • Amines or salt of carboxylic acid and strong base form alkaline solutions
	Soluble in dilute HCl	Basic substances, most amines
	Solule in dilute NaOH	Acidic, most alkanoic acids and phenols

	Soluble in dilute $NaHCO_3$ with effervescence	Strongly acidic, most alkanoic acids
	Insoluble in acid and alkali, neutral to litmus paper	Hydrocarbon, nitrohydrocarbon, high RFM alkanols, alkanals, alkanone, ether, ester
	Ignite on a spatula end	a) Luminous sooty flame for aromatic, unsaturated aliphatic compounds, high molecular weight aliphatic compounds b) Clean non-luminous flame for aliphatic compound of low % Carbon c) Doesnot burn- certain high proportion of non-combustible material like halogens, nitrogen, metal d) Charr rapidly – carbohydrates, hydroxacid e) Residue ash (not carbon)- metal present f) Violet vapour – contain high % of iodine

9.1 TESTING FOR A CERTAIN CLASS OF ORGANIC COMPOUNDS

Compound	Class	Test	Observation
Alcohols	Primary alcohol (RCH_2OH)	Add glacial ethanoic acid followed by conc.H_2SO_4 and warm (detect the smell)	A pleasant (sweet) fruity smell of an ester detected
	Secondary alcohol (R_2CHOH)	Add cariammonium nitrite reagent	Intense colouration/ppt formed. Phenols also give same observation, so carry out the test below
	Tertiary alcohol ($R_1R_2COHR_3$)	Add $FeCl_3$ solution	No observable change. Solution develops brown colouration due to Fe^{3+}. Violet colouration forms with phenol
	Where R_1, R_2, R_3 are alkyl groups not H	Add $K_2Cr_2O_7$ + dilute H_2SO_4 boil gently	Solution turns from orange to green
		Add Na metal (care)	Colourless neutral gas which burns with soft pop sound liberated. Gas is H_2
		Add CrO_3 in dilute H_2SO_4	Solution turns from blue to green, given by primary alcohols only
		Add conc.HCl + anhydrous $ZnCl_2$ shake vigorously and allow to stand(luca's test)	i. Clear solution (although a little darkening may occur) – primary alcohol ii. Clear solution turns cloudy – secondary alcohol iii. Clear solution turns cloudy immediately – tertiary alcohol

		Add I_2 in KI + NaOH until brown colour discharged, boil for 1min (iodoform test)	Yellow ppt formed. This tests alcohols of the structure CH_3CHOR
Aldehydes (RCHO)		Add brady's reagent	Yellow ppt
		Add schif's reagent	Red colour of the dye restored immediately
		Add NaOH and warm on water bath	A brownish oily strong smelling resin formed. Given by only lower aliphatic aldehydes)
		Add cold dilute $KMnO_4$	Brown ppt of MnO_2 formed
		Add CrO_3 in dilute H_2SO_4 (see ketones)	Solution turns blue-green and becomes opaque
Aromatic aldehydes (ArCHO)		In addition to the general tests for aldehydes above, Add NaOH warm while shaking, allow the mixture to settle and pour off some of it in the test tube, add conc.HCl and allow to cool	Small white crystals as the mixture cools
Ketones (R_1COR_2)		Carry out brady's reagent test, iodoform test	As in aldehydes
		Add Br_2 in CCl_4	Reddish brown solution turns to colourless with evolution of HBr (only given by carbonyl compounds with alpha hydrogen)
		Add saturatedNaHSO$_3$	White ppt formed
		Add tollen's reagent	No observable change for ketones Black colouration occurs and silver mirror for aldehydes
		Add fehling's solution	No observable change for ketone Reddish brown ppt formed for aldehydes
Carboxylic acids (RCOOH)		Add ethanol + conc.H_2SO_4 and warm (detect the smell)	Pleasant/sweet/fruity smell of an ester (benzoic acid gives a negative result)
		Add solid NaOH (soda lime) and heat, test the gas by igniting it	Compound dissolves and, a) Burns with a clean non-luminous flame- aliphatic carboxylic acid b) Burns with luminous sooty flame- aromatic carboxylic acid

		Add Na_2CO_3 or $NaHCO_3$ and test gas with lime water	Colourless acidic gas which turns lime water milky evolved (not given by phenols)
		Add acidified MnO_4^- and warm	Turns purple MnO_4^- colourless If Oxalic acid CO_2 is evolved unlike other carboxylic acids
		Detect smell (take care)	Characteristic carbolic smell and very corrosive to skin and textiles
		Add ceriammonium nitrite	Intense coloration/ppt formed
		Add $FeCl_3$ solution	Intense violet/purple colouration
		Add NaOH solution	Compound dissolves
		Bromine water	White ppt formed
Esters (R_1COOR_2)		i. Detect smell ii. Add equal volume of water + a drop of NaOH + 2 drops of phenolphthalein shake vigorously and allow to stand	i. Generally have strong pleasant fruity smell ii. Pink colour of phenolphthalein disappears due to formation of the parent acid and alcohol
		Add conc. H_2SO_4	Solid dissolves unlike hydrocarbons
Ammines (R_1NHR_2)		Detect smell (care)	Ammonia like fishy smell for aliphatic ammines. Primary amines smell more like ammonia
	Add dilute HCl		
Amides			
Urea (H_2NCONH_2)		Heat part of the solid greatly (test gas with moist litmus paper or conc. HCl), continue heating, cool to solidified product then add NaOH and a drop of $CuSO_4$(biurette test)	Solid melts with evolution of colourless alkaline gas with chocking smell that forms dense white fumes with conc.HCl