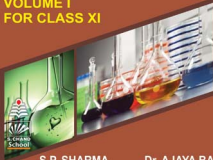


ISC PRACTICAL CHEMISTRY

VOLUME I
FOR CLASS XI



S.P. SHARMA

Dr. AJAYA BABOO

*Strictly according to the latest Syllabus of ISC (Class XI) prescribed by the
Council for the Indian School Certificate Examinations, New Delhi*

ISC

PRACTICAL CHEMISTRY

VOLUME – I

(FOR CLASS XI)

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PREFACE TO THE REVISED EDITION

In order to respond to the long pressing demand and constructive criticism of the experimentors of class XI studying in accordance with the latest syllabus prescribed by Council for the Indian School Certificate Examinations, New Delhi, the book has been thoroughly revised and updated.

The chapters have been arranged sequentially. Along with this, new additions on 'Preparation of Inorganic Compounds' and 'Paper Chromatography' have been incorporated as per the demands of new syllabus. Thus students shall find this edition completely in sync with the latest syllabus. New Experiments on Practical Chemistry have been incorporated.

We wish to extend our special thanks, in anticipation, to the students, our follow teachers and management of S. Chand & Company Pvt. Ltd. for their valuable suggestions to improve the book.

Hope the new edition will provide necessary guidance to carrying out the experiments.

S.P. SHARMA
Dr. AJAYA BABOO

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PREFACE TO THE FIRST EDITION

“Those sciences are vain and full of errors which are not born from experimentation, the mother of all certainties”. A systematic ordering and reordering of knowledge is an essential part of teaching. Discussions, refine this expression of knowledge. However, to find out how the material world behaves, merely thinking about it will not serve our purpose. We must observe and therefore the need for experimentation.

In Chemistry it is only through experimentation, through the laboratory process, that the unifying principle can be developed and understood. Once we become familiar with these widely applicable and recognized principles, there remains no longer any need for endless memorization of innumerable chemical facts. The men who established the first laboratories way back in the Middle Ages, were known as ‘alchemists’. Today we laugh at them but their researches became the basis of a host of great inventions and discoveries. Who knows what wonderful new materials, our young experimenters may give to us, for future use.

We are sure that the students will find this book a veritable storehouse of knowledge about practical chemistry. Their genius, unmanifested though rapidly developing, will enable them to enjoy and understand these pages. We hope for no greater reward than that young readers, with their lives before them, may find in this volume something to inspire them with an earnest and unflagging zeal for finding something new in the realms of chemistry. The story of science is like the story of life—ever moving and never ending. Each new generation has added new chapters to the book of science. And the process must continue.

We extend our sincere thanks and gratitude to our Principal Rev. Father George Paul who has been a source of constant encouragement to us. Also, we express our heart - felt thanks to the brilliant students of our esteemed institution, St. Peter’s College, Agra. Our association with each one of them has been enriching for us, without which our experience in the field of Practical Chemistry would have remained insufficient.

S.P. SHARMA
Dr. AJAYA BABOO

PRACTICAL CHEMISTRY

(Syllabus ISC Class XI)

PRACTICAL WORK

20 Marks

Candidates are required to complete the following experiments:

1. Basic laboratory techniques:

- Cutting a glass tube.
- Bending a glass tube.
- Drawing out a glass jet.
- Boring a cork.

2. Titration: Acid-base titration involving molarity.

Titration involving:

- Sodium carbonate solution/ dil H_2SO_4 or dil HCl using methyl orange indicator.
- $NaOH$ or KOH solution/ dil H_2SO_4 or dil. HCl using methyl orange indicator.
- Calculations involving molarity, concentration in grams l^{-1} / number of ions, water of crystallisation and percentage purity.

NOTE: Calculation of molarity must be upto 4 decimal places at least, in order to avoid error.

3. Qualitative analysis: identification of the following in a given mixture (containing two anions and two cations):

Anions: CO_3^{2-} , NO_2^- , S^{2-} , SO_3^{2-} , SO_4^{2-} , NO_3^- , CH_3COO^- , Cl^- , Br^- , I^- , $C_2O_4^{2-}$.

Cations: NH_4^+ , Pb^{2+} , Cu^{2+} , Al^{3+} , Fe^{3+} , Zn^{2+} , Mn^{2+} , Ni^{2+} , Ba^{2+} , Sr^{2+} , Ca^{2+} , Mg^{2+} .

Formal analytical procedure required.

Anions: Dilute acid group: CO_3^{2-} , NO_2^- , S^{2-} , SO_3^{2-}

Concentrated acid group: NO_3^- , Cl^- , Br^- , I^-

Special group: SO_4^{2-} , CH_3COO^- , $C_2O_4^{2-}$

Cations: Group zero: NH_4^+

Group I: Pb^{2+}

Group II: Cu^{2+} , Pb^{2+}

Group III: Al^{3+} , Fe^{3+}

Group IV: Zn^{2+} , Mn^{2+} , Ni^{2+}

Group V: Ba^{2+} , Sr^{2+} , Ca^{2+}

Group VI: Mg^{2+}

NOTE:

- More than one radical will not be given from the same group of anions and cations.
- For wet test of anions, sodium carbonate extract must be used (except for carbonate). (Insoluble salts excluded)

4. Preparation of inorganic compounds

(i) *Preparation of potash alum/Mohr's salt.*

(ii) *Preparation of crystalline FeSO_4 / CuSO_4 .*

5. Paper Chromatography

Preparation of chromatogram, separation of pigments from extracts of leaves and flowers/ink mixtures; determination of R_f value.

PROJECT WORK AND PRACTICAL FILE

10 Marks

Project Work - 7 Marks

The candidate is to creatively execute one project/assignment on a selected topic of Chemistry. Teachers may assign or students may choose **any one** project of their choice.

Suggested Evaluation criteria for Project Work:

- Introduction/purpose
- Contents
- Analysis/material aid (graph, data, structure, pie charts, histograms, diagrams, etc)
- Presentation
- Bibliography

Practical File - 3 Marks

Teachers are required to assess students on the basis of the Chemistry Practical File maintained by them during the academic year.

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SAFETY RULES FOR CHEMICAL LABORATORY

1. Never work alone in the laboratory.
2. Never attempt unauthorised experiment.
3. Never taste any chemical. Should a chemical enter the mouth, rinse out with water at once and report the matter to the teacher.
4. Never inhale poisonous gases or volatile liquids such as chloroform, carbon tetrachloride etc. Smell them carefully.
5. When heating any substance in a test tube, be careful to point the mouth of the test tube away from yourself and your neighbours.
6. Never add more than the quantity of the reagent you have been told to add.
7. When mixing acid with water, add acid slowly to water and not water to acid.
8. Do not pour hot conc. acid directly into the sink. Allow it to cool and then pour carefully while the water tap is kept open.
9. Always allow a hot test tube to cool before replacing it in the rack, if the rack is wet.
10. Be cautious with heated glass.
11. Never heat a funnel or a watch glass over a flame.
12. Never use cracked or broken glass ware.
13. Reagents once taken out of the bottle should not be poured back.
14. Replace the stoppers of the reagent bottles immediately after use. Do not interchange them.
15. The test tube must be kept at eye-level and away from you when making an observation.
16. Open the water taps gently and close when not in use. Close the gas taps immediately after use.
17. At the end of the experiment wash the used glass ware and arrange them in order.
18. Wash your hands before leaving the lab.

ISC
PRACTICAL CHEMISTRY
VOLUME – I

Basic Laboratory Techniques

Regarding a chemical laboratory Michael Faraday's words come true

“We come here to be philosophers, and I hope you will always remember that whenever a result happens, specially if it be new, you would say, what is the cause ? Why does it occur ? And you will in course of time find out the reason.”

Chemistry laboratory is a centre of learning where the students develop their power of observation by performing the experiments of various types using the apparatus of different kinds. Heating device in the chemistry laboratory is the Bunsen burner. It has a simple construction with following parts.

- | | | |
|---------------------------------|-------------------|----------------|
| (a) The base | (b) The gas tube | (c) The nipple |
| (d) The metallic tube or barrel | (e) Air regulator | |

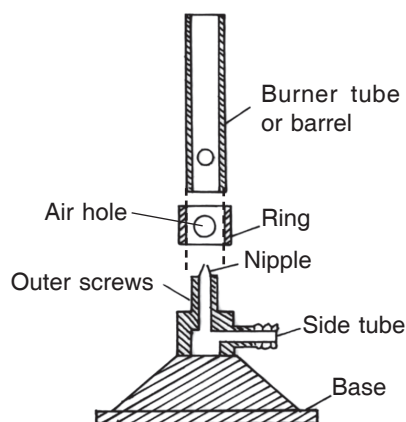


Figure – 1. Bunsen burner

Points to remember for the Bunsen burner

1. Before lighting the burner always make sure that the air regulator works freely and all connections are air tight.
2. Never blow to put out the flame of the burner. Always turn it off from the gas tap.
3. Never throw solid material into the flame. This will spoil the working of the burner.
4. For heating purpose always use the tip of the burner flame and not the middle portion.

Some time the flame of a burner is found to strike back inside the barrel, it indicates that the air hole is too big to give a non-smoky flame or the pressure of the gas coming in the burner is low. The flame strikes back in order to get more fuel. In this case the air hole is adjusted properly to get a non-smoky flame. Since the barrel has become hot, it is not advisable to touch it.

Handling and manipulation of glass tubing

Cutting a glass tube or rod.

Place the glass tube or rod flat on the bench top. Hold it firmly with the help of your left hand and make one sharp scratch with the help of a sharp triangular file at the point where the tube to be cut as desired. Push the file away from you to make this scratch. It should not be moved back and forth, only one firm stroke is required for the purpose.

Now hold the rod or tube firmly placing one hand on each side of the scratch mark with the two thumbs, touching each other as shown in figure – 2.

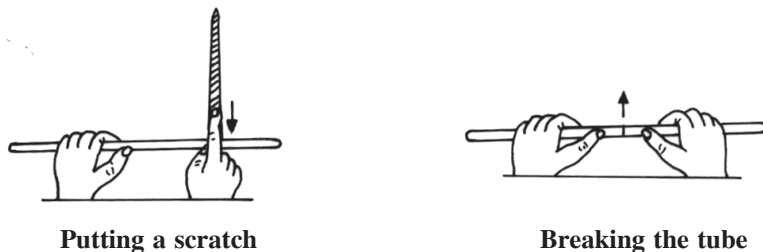


Figure – 2

Your thumb should be on opposite side of scratch. Now pull the tube or rod back gently by pressing thumbs forward (against the glass). It will give a clear and sharp break. In case if the break is not clear, keep the sharp freshly cut edge rotating in the flame for a while so that it is rounded off as shown in the figure – 3.

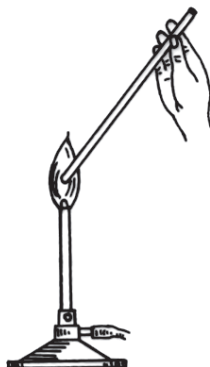


Figure – 3 (Rounding off the edge)

Bending a glass tube

It needs a horizontal flame. Use a flame spreader to obtain a horizontal flame from Bunsen burner. Hold the glass tube to be bent with the help of both the hands, one on each end as shown in figure – 4. (Be sure that the ends are fire polished and cool).

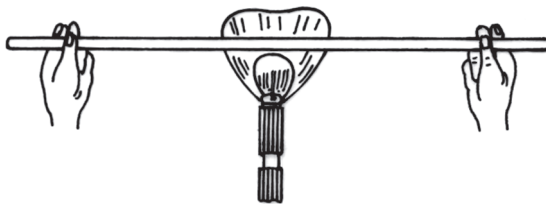


Figure – 4
(delivery tube in flame)

Hold the tube lengthwise in the horizontal flame at the point where bend is required. Now rotate the tube evenly and carefully to make the glass soft and the heated portion begins to sag.

Bring away the tube from the flame and bend it gently to the required shape holding it in this position until the glass becomes hard. The bend should be in one plane. Good and poor bends are shown in the figure – 5.

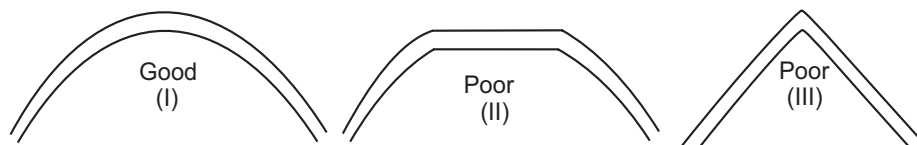


Figure – 5

Note : It should be noted that the bend should never be at 90° as shown in figure 5 (III). In this case the inner ends of the arms are nearly blocked and will not allow the fluid to pass. Figure – 5 (I) shows a proper bend.

Drawing out a glass Jet

Light a Bunsen burner. Hold the required glass tube, with both the hands, one on each end. Hold the tube lengthwise and rotate it gently and evenly until the glass becomes soft and heated portion begins to sag. Now remove the tube away from the flame and allow it to cool slightly. Now rotate it while pulling the ends apart till the capillary of required size is obtained as shown in figure – 6.

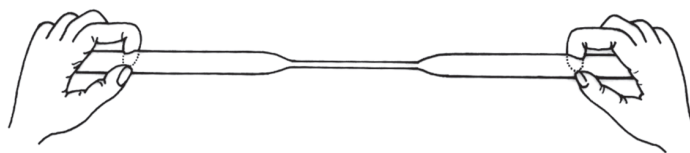


Figure – 6
(Drawing jet outside the flame)

The middle portion can be cut and removed using a sharp triangular file as shown in figure – 7 and you get two droppers in hand.

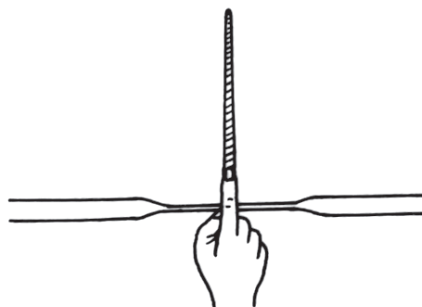


Figure – 7
(Cutting the drawn jet)

The jets so obtained are used for different purposes such as making wash bottles, medicine droppers, melting point tubes, for washing precipitates etc.

Boring a cork

Many times in laboratory we need to pass a glass tube through a cork stopper such as preparation of gases, making wash bottles for different purposes.

For this it is required to bore hole, one, two or more through cork stoppers.

(i) Selection of cork of the correct size

The cork should be of the size which fits well in the neck of the vessel in use and should be strong enough to bear the pressure when hole of proper size is bored in it.

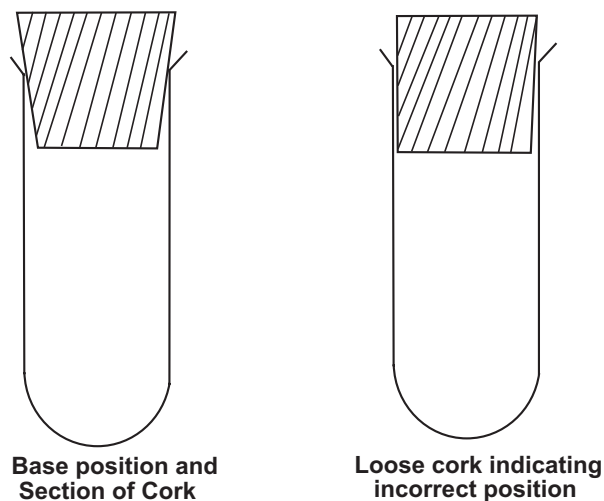


Figure – 8

(ii) Pressing of Cork

Press the cork with the help of a cork presser as shown in figure – 9. Press the cork between the wheel of the presser and the grooved plate. Now press the handle lever at times.

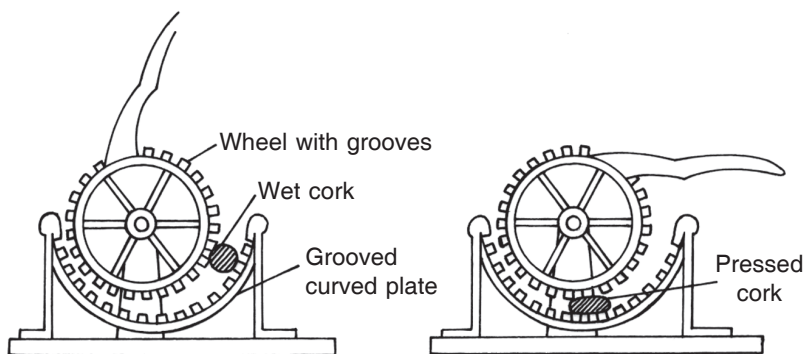


Figure – 9
Pressing cork with a wheel type cork presser

(iii) Selection of Cork Borer

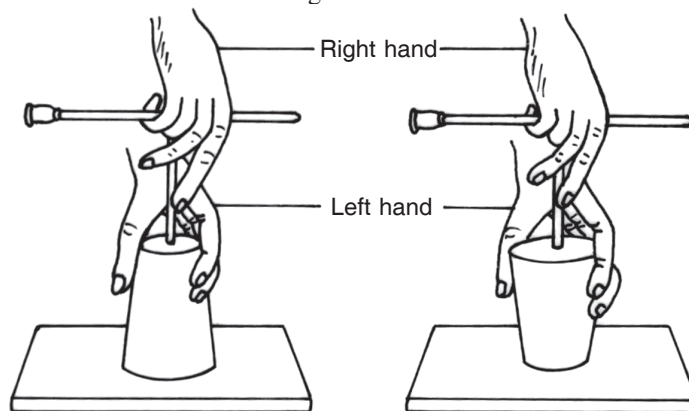
Cork borers are available in various sizes. Since the glass tube is to be fixed tightly in the cork a borer is selected which is slightly smaller in diameter than the glass tube.

What are cork borers ?

They are metallic tubes having a sharp edge at one end and handle at the other. When a borer is used for long time it loses in sharpness and becomes dull. A special sharpener is used to sharpen it to make it useful.

(iv) Actual boring

Wet the cork borer with little glycerol in order to give it smoothness. Now hold the borer in right hand and the cork in the left pointing the narrow end upwards. Place the sharp end of the borer at the centre of the cork as shown in figure – 10.

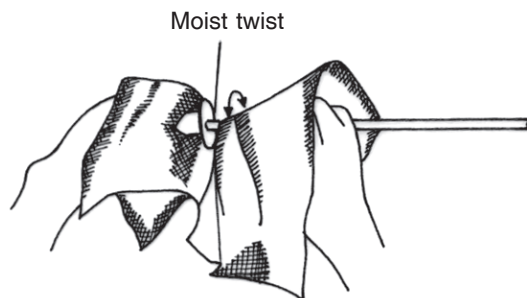
**Figure – 10****(Borer driven half way
from narrow end)****(Boring from broad end)**

Align the borer parallel to the axis of the cork. Twist and press till the borer is half way through the cork. Check the alignment after each twist. Now place the cork on a wooden borer and twist the borer vigorously until it cuts through the cork. Remove the stem of the cork from the borer with the help of a cleaning rod.

(v) How to insert glass tubing in stoppers ?

Make the tube and stopper wet with soap solution or glycerol or simply with water. Before using the tube be sure that the ends of the tube are fire polished. Now grasp the tube near the point of insertion and rotate the tube back and forth while pushing it gently into the stopper.

- Note.** (i) Do not apply too much force to push the tubing through the stopper.
(ii) Wrap your hands in a towel for protection.

**Figure – 11****(Fitting a delivery tube in the cork)****(vi) How to remove glass tubing from stopper ?**

Apply soap solution or glycerol to lubricate the tubing which is to be pulled through the stopper. Wrap the tubing with a towel. Pull the tubing from the stopper with a gentle twisting motion. If the tube gets stuck to the stopper, insert the end of the stout wire between the tubing and the stopper and rotate it gently.

If this procedure fails, take the smallest size cork borer available which fits over the tubing through the stopper and work it out.

Chapter 2 Titrations

Acid base titrations involving molarity

Out of the methods of chemical analysis, the qualitative one has already been discussed in Chapter II. The other method *i.e.* quantitative analysis is divided into volumetric and gravimetric analysis. This chapter includes the details of the former.

The principle of volumetric analysis is to take a measured volume of a solution of unknown concentration and to this solution an equivalent amount of a solution of suitable reagent of known concentration is added. The process of adding the solution till the reaction is complete" is known as *titration*. The sharp change in coloration or the coloured precipitate formation indicates the completion of the *titration*. The change in coloration can be detected by the addition of a chemical known as *indicator*. In a titration like KMnO_4 solution against oxalic acid solution, no indicator is required. (self indicator)

The terms used in this method of investigation are the following :

Titration

It is a process of adding one solution through burette to the known volume of the other solution till the reaction is complete.

The two solutions are such that the strength of one is known to us and is known as *titrant* or *standard solution*. The other solution whose concentration is to be determined - by this process is known as *titrate*.

Indicator

A chemical substance which helps in visual detection of completion of titration is called indicator.

The completion of reaction is indicated by producing turbidity or a visible change in colour.

In a redox titration, such as oxalic acid and potassium permanganate solution, no indicator is required and the completion of reaction is indicated by the excess drop of potassium permanganate solution. Such a titration is known as self-indicator titration.

End Point

A sharp point at which completion of reaction takes place is known as *end point* of the titration. It is also known as titre value.

Concentration of the solution

It is the number of moles, equivalents or grams present per litre of the solution.

Normality

It is the number of gram equivalent weight of solute dissolved in one litre of the solution. Normality is denoted by 'N'.

Gram Equivalent Weight

The equivalent weight of a substance expressed in grams is known as its gram equivalent weight.

The strength of a solution, normality and gram equivalent weight can be related as -

$$\text{Strength} = \text{Normality} \times \text{Gram Equivalent Weight}$$

Normal Solution

It is a solution which contains gram equivalent weight of solute in a litre of solution.

It can be expressed as 1N solution. When 1/2 gram equivalent of solute is dissolved in 1 litre of solution, it is called *seminormal* solution and is denoted as N/2.

Similarly *deci normal* and *centi normal* solutions are denoted as N/10 and N/100 respectively.

Gram Molecular Weight

Molecular weight expressed in grams is known as gram molecular weight. It is also known as one mole.

Molarity

It is the number of gram molecular weight of solute dissolved in one litre of solution. The molarity is denoted by "M".

Molar Solution

It is a solution which contains gram molecular weight of solute dissolved in a litre of solution.

When 1/2 gram molecular weight of solute is dissolved in one litre of solution, it is called semi molar solution and is denoted as M/2.

Similarly, deci and centi molar solutions can be denoted as M/10 and M/100 respectively.

Molal Solution

It is a solution, which contains a gram molecular weight of solute in one thousand grams of solvent.

The molarity, gram molecular weight and strength are related as.

$$\text{Strength} = \text{Molarity} \times \text{Gram Molecular Weight}$$

Normality and Molarity can be related as $N = nM$.

Equipments used in Volumetric Analysis**(1) Burette**

It is a long cylindrical glass tube of uniform bore. It is graduated along most of its length from zero to fifty in milli - litres such that one small division reads one tenth of a ml *i.e.* 0.1 ml. The lower end is provided with a glass stopper (or a pinch cock) for controlling the delivery of variable volumes of solutions.

The surface of a solution touching the inner wall of the burette is often curved and is known as meniscus. While reading a burette the bottom of the meniscus opposite the graduation is considered.

Use of Burette

Clean the burette thoroughly with chromic acid. (To prepare chromic acid, a mixture of solid potassium di-chromate ($K_2Cr_2O_7$) and conc. sulphuric acid can be boiled in a beaker), and then with tap water. Make sure that it does not leak. If it does, apply a little grease or vaseline to the stopper. Insert it in its position and incite it to and fro to check its free movement.

Now, wash the burette with distilled water and then rinse (wash with the solution to be filled) and allow a part of the solution to run down through the jet.

Fix the burette vertically on a burette stand. Fill the solution in it. Open the tap to remove air bubble, if it persists.

Adjust the concavity of meniscus to the zero mark or to a point convenient according to the level of your eye.

A piece of white paper can be fixed behind the level of the liquid to observe the reading accurately.

Precautions

1. Make it a point to wash the burette with distilled water after use.
2. Allow some time for the sticking liquid to come down to the desired mark before adjusting the concavity of meniscus.
3. The oozed out drop sticking with the jet of burette should be wiped off with a filter paper.
4. Filling of hot liquid should be avoided, as it is graduated at a much lower temperature.
5. Avoid the use of rubber tubing burette when solutions like that of KMnO_4 or $\text{K}_2\text{Cr}_2\text{O}_7$ are to be filled. In this case read top level of the meniscus.

(2) Pipette

Pipette is an apparatus designed to deliver a fixed volume of liquid. It is a specially designed glass tube with a bulb in the middle, a jet at the bottom and an etched circular mark at an appropriate height above the bulb.

Pipette is filled upto this circular mark, measuring a definite volume. The capacity of the pipette is stated on its bulb in ml at stated temperature. The pipettes in common use are of 25 ml, 20 ml and 10 ml.

Use of pipette

Wash the pipette in the same way as described for burette. Rinse the pipette with the solution to be measured.

Now dip the jet of the pipette in the solution and suck it from the other end gently, such that the liquid rises a little above the etched mark. Stop sucking and quickly close the upper end with the fore finger. Rotate the pipette slowly to allow the liquid to triple down upto the etched mark.

The measured liquid is transferred in a clean titration flask by releasing the pressure of the forefinger. When the solution has run out from the pipette, allow its tip to touch the flask held in a slanting position.

Precautions

1. It should not be used for measuring poisonous liquids.
2. The solution retained in the tip should not be blown out.
3. Liquid should be sucked very carefully. During sucking, the lower end of the pipette should be well dipped in the solution, otherwise the sucked solution may go into the mouth.

(3) Measuring Flask

It is a flat bottomed glass flask with a long narrow neck bearing a ground glass stopper. The neck has a circular line etched around it. The capacity upto the circular mark at the stated temperature is etched on each flask. It is also called a volumetric flask or graduated flask or standard flask. The measuring flask in common use are of 1000 ml, 500 ml, 250 ml and 100 ml.

Use of Measuring flask

Clean the flask with chromic acid followed by tap water and finally with distilled water. Now the flask is ready to prepare the solution.

Preparation of standard solution

Collect the clean funnel and insert its stem in the washed measuring flask. Transfer the accurately weighed substance on to the funnel. Pour a little distilled water on it to allow the substance to run down into the flask. Place the stopper properly and shake the contents to dissolve the substance. Dilute it with distilled water upto the mark. Carefully invert it 2-3 times and allow it to stand for some time. The so - prepared solution is known as *standard solution*.

(4) Titration Flask

It is a conical flask made of glass. The titration flasks in common use are of 250 ml and 150 ml.

Use of a titration flask

Wash the flask two to three times with tap water followed by distilled water. Now it is ready for use.

(5) Chemical Balance

It is a delicate instrument which is used to weigh small amounts of substance accurately upto the fourth place of decimal. It is an absolute necessity in volumetric analysis, as the substances used to prepare standard solutions are weighed, using this.

The essential parts of an analytical balance are described as under.

1. *Base* - It is rectangular piece of wood or marble which rests on three Levelling screws. Two of these are in front and are used to maintain the uniform level of the base. The screws are adjusted such that the plumb line indicates the correct position. The third screw is like a nail which is behind the front screws (bearing central back position).
2. *Pillar* - It is a hollow metallic structure with a broad circular base fixed on the centre of the rectangular piece *i. e.* the base. The beam rests on it. A little above the base there is a scale fixed on the foot of a pillar.
3. *Beam* - It is a long, rigid, specially designed metallic rod bearing divisions on it. It is supported on three agate knife, edges, two on the sides and one in the centre. The side agate pieces have the edges pointing upwards, whereas the middle one points inwards. The beam rests on the bifurcated shaft by means of the middle agate piece. There are 50 or 100 equal divisions on either side of the beam.
4. *Bearing* - On each sharp agate piece, at the end of the beam, rests a bearing which has two parts. The lower part of it consists of two hooks one above the other.
5. *Adjusting screws* - There are two adjusting screws, one at each end of the beam. These are adjusted to keep the position of the pointer in equilibrium.
6. *Pans* - There are two metallic pans of equal weight hanging on the hooks.
7. *Plumb-line* - There is a pointed cone tied to a thread which hangs vertically behind the pillar towards the right of the scale. This is known as plumb line. This is used to find out the correct levelling of the balance. There is a metallic piece attached to the bottom of the pillar pointed upwards. The plumb line remains vertically above the metallic piece in the position of correct levelling.
8. *Handle* - Handle is a metallic lever attached to the base of the balance which, when turned towards right, raises the beam to make the pointer swing.

9. *Scale* - It is a white rectangular porcelain piece fixed on the base. It has a zero in the middle and equal number of divisions on the two sides of it. The pointer moves to and fro over it when the beam oscillates.
10. *Beam arrests and pan - arrests* - The balance has a beam arrest. The beam rests on it when not in use.

There are two pan - arrests and are used to arrest the pans when the balance is not in use.

11. *Rider* - A rider is a thin wire of aluminium or platinum usually in U-Shape weighing 10 mg. It can weigh accurately upto 0.001 gm. It can be placed any where on the graduated beam with the help of rider hook.
12. *Rider Hook* - This is a hook on which the rider is suspended. This can be made to move over the graduated beam with the help of a metallic rod known as rider carrier and is used to place the rider at any division on the beam.

Glass case

The balance is enclosed in a glass case with a front door and two side windows for keeping the weights and the substance to be weighed, on the pans. This protects the balance from air current, dust and moisture.

(6) Weight Box

This is a wooden box which contains the weights. The weights are of two types namely, the 'gram weights' and the milligram weights.

The gram weights are usually cylindrical in shape provided with a narrow stem. They are made of brass and coated with nickel or chromium.

The milligram weights or fractional weights are made of aluminium or stainless steel. They are leaf shaped with one corner turned up to facilitate handling. Their shapes differ in different boxes but most of them are rectangular.

The weights are arranged generally as –

1. *Grams* - 100, 50, 20, 20, 10, 5, 2, 2, 1.
2. *Milligram* - 500, 200, 200, 100, 50, 20, 20, 10.
3. *Rider*.

Some times the box contains weights smaller than 10 mg. These are not generally used but a rider is used instead.

The weights are placed in specific wooden partition. The fractional weights are covered by a glass slab.

(7) Forceps

It is a modified form of nickel plated brass tongs used to lift weights.

Use of a balance

The use of a balance can be divided under two headings.

1. Adjustment (setting) of balance.
2. Process of weighing.

1. Setting

Before the weighing is started, the experimenter must see whether the balance is properly set or not. The following steps must be taken in this concern

1. Clean the glass shutters gently with a cloth if dirty.

2. Open the shutters and clean the pans and the platform.
3. See the plumb line. It must be just above the cone. If not, turn the levelling screws to bring it in the correct position.
4. Turn the key towards the right to make the pans swing to and fro and the pointer over the scale covering equal number of divisions on the two sides of the zero, indicates the accuracy of the balance.

If the pointer covers more distance to one side and then the other side of this zero, turn the key to left to bring the beam and the pointer to rest. Move the adjusting screw of the heavier pan inward and the lighter pan outward and check again the accuracy of the balance by turning the key to right. Do it till the balance is set.

Process of Weighing

1. Open the left window and place the substance to be weighed on the left pan. Close the window.
2. Now open the right window, use the forceps to place a weight which you think is slightly heavier than the substance.
3. Arrest the pans, remove this weight and place a weight just lighter than this. Turn the key and check the lightness of the weight.
4. Now place the smaller gram weights and then fractional weights.
5. Try weights substantially till the oscillation of the pointer covers equal divisions on both sides of the zero mark of the scale.
6. Now close the side windows and try correct weighing.
7. Arrest the beam and note down the weights.
8. Replace the weights in their places in the weight box.

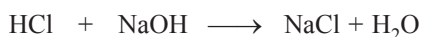
Precautions to be observed while weighing

1. Turn the key gently.
2. Transfer of any thing to and from the pan should be done when the beam is at rest.
3. Place the article to be weighed to the left pan and the weights to the right.
4. Never place a hot article in the pan.
5. Side windows must be closed before the final reading is taken as the pointer is disturbed by air movement.
6. Use forceps to lift weights.

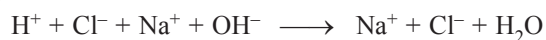
The principle of Volumetric analysis

In volumetric analysis, a standard solution of a reagent is put in the burette and is gradually added to a known volume of the other reagent until it is found that the amount of the reagent added is equivalent to the amount of substance being determined.

Let us consider a very common neutralization reaction of monobasic acid and monoacidic base say HCl and NaOH as the reagents.



Ionically,



The above neutralization reaction conveys the following information -

- (i) That 36.5 gm of hydrochloric acid exactly neutralises 40 gm of sodium hydroxide.

- (ii) That the Molar (or normal) solution of acid will exactly neutralise, the molar (or normal) solution of the alkali.
- (iii) That the known volume of any fraction of molar (or normal) solution of acid will exactly neutralise the equal volume of same fraction of the molar (or normal) solution of the alkali.

Acid-Alkali Titrations

(Neutralization reaction titrations)

To understand the neutralization reaction titrations one should be acquainted with the following two terms viz.

(i) Acidimetry

The titration of free bases with the standard acid is known as acidimetry.

(ii) Alkalimetry

The titration of free acids with the standard base is known as alkalimetry.

Neutralization

It is a process in which hydrogen ions (or hydronium ions H_3O^+ as per modern theory) combine with equal number of hydroxyl ions to form water.

The completion of the above process is indicated by some chemical substances known as indicators.

Indicators are the electrolytes which give colour change accordingly as they are introduced in acid or alkali solutions. The following combinations of acids and alkalies are in common use.

- (i) Strong acid * and strong base *
- (ii) Strong acid and weak base *

The indicators used in (i) case can be either methyl orange or phenolphthalein but in (ii) case methyl orange only can be used.

Note : Where there is choice of indicator, the phenolphthalein should be used as appearance of pink colour from colourless is sharp and can be distinguished easily.

Preparation of standard solutions using 250 ml measuring flask

Object

To prepare N/10 solution of caustic soda (NaOH).

Apparatus required

Volumetric flask (measuring flask), chemical balance, weight box, dropper, funnel, weighing bottle, 100 ml beaker and caustic soda (NaOH) pellets.

Procedure

- (1) Wash a 250 ml measuring flask with tap water followed by distilled water.
- (2) In a well cleaned dry weighing bottle weigh 1.0 gm caustic soda pellets and transfer it immediately ** to a 100 ml beaker. Add sufficient quantity of distilled water and stir the contents well to dissolve them.

* The strong acids in common use are dil. H_2SO_4 , HCl and HNO_3 . The strong bases in common use are NaOH and KOH . The weak bases in common use are NH_4OH and Na_2CO_3 solutions.

** Caustic soda is a highly deliquescent substance, therefore, the duration of its exposure to atmosphere should be minimised.

- (3) Very carefully transfer the solution to the measuring flask using a funnel.
- (4) Pour the distilled water on to the funnel in instalments to remove the adhering caustic soda solution. Remove the funnel. Fit the glass stopper of measuring flask well and allow it to stand for some time to make it homogeneous.
- (5) Shake the flask and add more water to reach its level little below the etched mark.
- (6) Remove the stopper and with the help of dropper add very carefully the distilled water to make it upto the etched mark. Finally fit the glass stopper.

Object

To prepare N/10 (deci normal) solution of hydrochloric acid using one litre measuring flask.

Apparatus required

Measuring flask, funnel, burette, beaker, conc. hydrochloric acid (density 1.16 g/cc).

Method

- (1) Wash a measuring flask with the help of clean distilled water and with the help of clean funnel pour approximately 500 ml of distilled water in it.
- (2) Measure the volume of hydrochloric acid as per following calculation. For N solution of HCl the amount of acid required = 36.5 gm /l, therefore for N/10 solution of HCl the amount of acid required = 3.65 gm /l.

$$\begin{aligned}\text{Volume} &= \frac{\text{mass}}{\text{density}} \\ &= \frac{3.65}{1.16} \\ &= 3.0 \text{ ml app.}\end{aligned}$$

Since it is inconvenient to weigh acids, the calculations for volume of it containing the above calculated amount are made.

3. From burette run down 3 ml of hydrochloric acid into the measuring flask (containing 500 ml of distilled water). Shake it thoroughly and add distilled water to make it upto the mark. Finally shake the flask and allow the acid solution to stand for some time to make it homogeneous.

Note : According to I. S. C. Chemistry Practical syllabus students are not supposed to prepare the volumetric solutions. The provided solutions of known concentration are treated as standard solutions.

Experiment No. 1**Object**

To determine the normality and concentration of a given hydrochloric acid solution by titrating it against a deci-normal solution of caustic soda using methyl orange as indicator.

Apparatus used

Burette, pipette, titration flask, burette stand, white glazed tile. Indicator - Methyl orange.

Procedure

1. Wash all the glass apparatus with tap water followed by distilled water.
2. Rinse and fill the burette with the given hydrochloric acid solution. Fix it vertically in

a burette stand keeping its tap on the right hand side. Adjust the level of acid * to zero mark and note it down as initial reading of burette.

3. Rinse the pipette with the alkali solution. Carefully suck up the solution into the pipette and adjust it upto etched circular mark.
4. Transfer this alkali solution into the titration flask. Add to it a few drops of the methyl orange indicator.
5. Place the titration flask on the glazed tile such that its mouth is just below the burette tip. (avoid inserting the tip into the neck of the flask).
6. Add the solution from burette in small instalments and go on shaking the flask by whirling motion. When the colour of the solution is about to change from light yellow to light pink add acid in drops. Stop additions from burette as soon as the colour of indicator is just changed. Wait for few seconds to ensure the stability of pink colour. The persistence ** of pink colour indicates the completion of neutralization reaction. Record the final reading of burette as the end point.

The difference in the initial and final readings of the burette represents the volume of the acid which reacts completely with the known volume of the alkali taken in the titration flask.

7. Note down the difference of the initial and final readings of the burette as end point.
8. Repeat the experiment till two concordant (same) readings are obtained.

Observations

Volume of alkali used (*i.e.* size of the pipette) = 25 ml.

S. No.	Burette Reading		Volume of acid solution used	End point or titre value
	Initial	Final		
1.	0.0 ml.	24.6 ml.	24.6 ml.	24.5 ml.
2.	0.0 ml.	24.5 ml.	24.5 ml.	
3.	0.0 ml.	24.5 ml.	24.5 ml.	

End point or titre value = 24.5 ml.

Calculations : (1) For Normality

$$N_1 V_1 = N_2 V_2$$

where N_1 and V_1 are the normality and volume of acid and N_2 and V_2 for alkali respectively.

$$N_1 \times 24.5 = \frac{1}{10} \times 25$$

$$N_1 \times \frac{25}{24.5 \times 10} = \frac{1}{9.8} = 0.10204$$

For Concentration

$$\text{Concentration} = \text{Normality} \times \text{gm eq. wt.}$$

$$= \frac{1}{9.8} \times 36.5 \text{ g/l OR } 0.10204 \times 36.5 \text{ g/l}$$

$$= 3.724 \text{ g/l.}$$

* If it is a rubber tubing burette, run out the solution rapidly through the tip to remove any air bubble entrapped in it.

** If pink colour fails to persist, add an acid drop by drop to reach the required stage.

Precautions

1. The solution retained in the tip of pipette should not be blown out.
2. Titration flask must be rinsed with distilled water only.
3. Do not allow any drop of acid solution to stick to the neck or the sides of titration flask.
4. The end point should be recorded at the appearance of the lightest possible pink colour.
5. The concordant readings and not the mean of the values should be used as end point in the calculations.

Experiment No. 2**Object**

You are provided with 3.5 gm /l of KOH solution. Determine the normality and concentration of sulphuric acid solution by titrating it against the given KOH solution using phenolphthalein as indicator.

Apparatus required

Burette, pipette, titration flask, burette stand, white glazed tile.

Procedure

1. Wash all the glass apparatus with distilled water.
2. Rinse and fill the burette with the given KOH solution. Fix it vertically in burette stand keeping its tap on right hand side. Adjust the level of the solution to zero mark and record it as initial reading of burette.
3. Rinse the pipette with given acid solution. Carefully suck--up the solution into the pipette and adjust it upto the etched circular mark. Transfer the measured solution to flask. Add to it 2-3 drops of phenolphthalein indicator.
4. Place the titration flask on a glazed tile such that the mouth is just below burette tip.
5. Add the alkali solution from burette in small instalments by whirling motion. When the lightest possible persistent pink colour appears. Stop adding the alkali solution and record the burette reading.
6. Note down the difference of initial and final readings of burette as end point.
7. Repeat the experiment to obtain the two concordant readings.

Observations

Volume of acid used *i.e.* the size of the pipette = 25.0 ml.

S. No.	Burette Reading		Volume of alkali solution used	Titre value
	Initial	Final		
1.	0.0 ml.	24.7 ml.	24.7 ml.	24.8 ml.
2.	0.0 ml.	24.8 ml.	24.8 ml.	
3.	0.0 ml.	24.8 ml.	24.8 ml.	

Calculations

For normality of KOH solution

$$\text{Normality} = \frac{\text{Concentration}}{\text{gm. eq. wt.}}$$

$$= \frac{3.5}{56} = \frac{1}{16}$$

For Normality of sulphuric acid solution

$$N_1 V_1 = N_2 V_2$$

where $N_1 V_1$ are the normality and volume for sulphuric acid solution, while $N_2 V_2$ are the normality and volume for KOH solution.

$$N_1 \times 25 = \frac{1}{16} \times 24.8$$

$$N_1 = \frac{24.8}{16 \times 25} = \frac{31}{500} = 0.062$$

For Concentration of sulphuric acid solution

$$\text{Concentration} = \text{Normality} \times \text{gm.eq. wt.}$$

$$= 0.062 \times 49$$

$$= 3.038 \text{ gm/l}$$

Note : Eq. wt. of sulphuric acid

$$= \frac{\text{molecular wt of H}_2\text{SO}_4}{\text{basicity of H}_2\text{SO}_4}$$

$$= \frac{98}{2} = 49$$

$$\text{Eq. wt. of KOH} = \frac{\text{Molecular wt. of KOH}}{\text{Acidity of KOH}}$$

$$= \frac{56}{1} = 56$$

Experiment No. 3

Object

Find out the normality and concentration of Na_2CO_3 solution by titrating against N/10 Nitric acid solution using methyl orange indicator.

Apparatus required

Burette, pipette, titration flask, Burette stand, white glazed tile.

Procedure

1. Rinse and fill the burette with nitric acid solution.
2. Pipette out 25 ml of sodium carbonate solution to the titration flask and add to it 3-4 drops of methyl orange indicator.
3. Run down the acid solution from the burette in small instalments with constant shaking the flask. Stop additions from burette as soon as the colour of indicator is just changed. Repeat the experiment till two concordant readings are obtained.

Observations

Size of pipette used = 25.0 ml.

S. No.	Burette Reading		Volume of acid solution used	Titre value
	Initial	Final		
1.	0.0 ml.	25.6 ml.	25.6 ml.	25.5 ml.
2.	10.0 ml.	35.5 ml.	25.5 ml.	
3.	10.0 ml.	35.5 ml.	25.5 ml.	

$$\begin{aligned}
 \text{Calculations : } \text{Eq.wt. of Na}_2\text{CO}_3 &= \frac{\text{Molecular wt. of Na}_2\text{CO}_3}{\text{No. of + vely charged ions of sodium}} \\
 &= \frac{106}{2} = 53 \\
 \text{Eq. wt. of Nitric acid} &= \frac{\text{Mol. wt. of Nitric acid}}{\text{Basicity of Nitric acid}} \\
 &= \frac{63}{1} = 63
 \end{aligned}$$

For Normality of Sodium Carbonate

$$\begin{aligned}
 N_1 V_1 &= N_2 V_2 \\
 (\text{for Na}_2\text{CO}_3) \quad (\text{For HNO}_3) \\
 N_1 \times 25 &= \frac{1}{10} \times 25.5 \\
 N_1 &= \frac{25.5}{10 \times 25} \\
 &= 0.102
 \end{aligned}$$

For Concentration of Na₂CO₃

$$\begin{aligned}
 \text{Concentration} &= \text{Normality} \times \text{gram eq.wt.} \\
 &= 0.102 \times 53 \\
 &= 5.406 \text{ gm/l}
 \end{aligned}$$

Experiment No. 4

To determine the molarity and concentration of a sulphuric acid soln. by titrating it against a caustic potash soln of concentration 1.5 gm/litre. Use methyl orange as indicator.

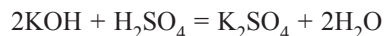
Apparatus required

Burette, Pipette, Titration flask, white glazed tile and a burette stand.

Procedure

1. Wash, rinse and fill the burette with the given sulphuric acid soln. check air bubble and bring the liquid level upto zero mark.
2. Wash, rinse and fill the pipette with caustic potash soln. and transfer it carefully to the titration flask.
3. Add 3-4 drops of methyl orange indicator to the above soln. Shake the contents of the flask and place it on the tile.
4. Run down the acid soln. from the burette in small instalments, shaking the flask constantly. Watch carefully, the colour of the solution in flask and stop addition from burette as soon as the colour of the solution changes from light yellow to light pink.

- Note down the final reading of the burette and subtract from it the initial reading. The difference is the end point or known as titre value.
- Repeat the experiment to obtain two concordant readings



Observations

Volume of KOH used *i.e.*, size of pipette = 25 ml

Burette Reading Volume of				
S. No.	Initial	Final	acid used	titre value
1.	0.0 ml	25.3 ml	25.3 ml	25.1 ml
2.	0.0 ml	25.1 ml	25.1 ml	
3.	0.0 ml	25.1 ml	25.1 ml	

Calculations

For molarity of KOH solu

$$\begin{aligned}\text{Molarity} &= \frac{\text{Concentration}}{\text{molecular weight}} \\ &= \frac{1.5}{56} \\ &= 0.026785714 \text{ M}\end{aligned}$$

For molarity of sulphuric acid.

$$\frac{M_1 V_1}{M_2 V_2} = \frac{n_1}{n_2}$$

Where M_1 and V_1 are molarity and volume for sulphuric acid solu and M_2 and V_2 are for caustic potsh solution; n_1 is the number of moles of H_2SO_4 white n_2 is the number of moles of KOH solution.

$$\begin{aligned}\frac{M_1 \times 25.1}{0.026785714 \times 25} &= \frac{1}{2} \\ M_1 &= \frac{0.026785714 \times 25}{2 \times 25.1} \\ &= 0.013339499 \text{ M.}\end{aligned}$$

For concentration of sulphuric acid

$$\begin{aligned}\text{Concentration (Strength)} &= \text{Molarity} \times \text{gram molecular weight} \\ &= 0.013339499 \times 98 \\ &= 1.307 \text{ g/litre}\end{aligned}$$

Experiment No. 5

You are provided with two solutions as follows

A-12 is a solution of hydrochloric acid

A-13 is a solution of potassium carbonate of concentration 3.45 gm/litre.

Fill the solution A-12 in the burette and titrate it against A-13 using methyl orange indicator.

Record observations

- State the titre value

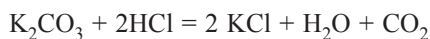
2. Calculate molarity of A-13
3. Calculate molarity of A-12 and its concentration in gm/litre.

Apparatus required

Burette pipette, Titration flask, white glazed tile and Burette stand.

Procedure

1. Wash, rinse and fill the burette with soln. A-12 ie HCl up to zero mark.
2. Wash and rinse the pipette; fill it upto mark with soln A-13 potassium carbonate soln. Transfer it carefully to the titration flask.
3. Add 3-4 drops of methyl orange indicator to the above solution and place the flask on a glazed tile.
4. Run down the acid solution from the burette in small instalment into the flask with constant shaking. Watch carefully the colour of the solution in the flask and stop additions from burette as soon as the colour of the indicator changes from light yellow to light pink
5. Note down the final reading of the burette and subtract from it the initial reading. The difference of these two is the titre value or the end point.
7. Repeat the experiment to obtain two concordant readings.

**Observations**

Volume of K_2CO_3 used i.e. size of the pipette = 25 ml

S.No.	Burette Intial	Reading Final	Vol. of acid Soln. used	End point
1.	0.0 ml	26.2 ml	26.2 ml	26.1 ml
2.	0.0 ml	26.1 ml	26.1 ml	
3.	0.0 ml	26.1 ml	26.1 ml	

Intended titre value = 26.1 ml.

Calculation

(i) Molarity of A-13 i.e. Potassium carbonate solution

$$= \frac{\text{Concentration}}{\text{Molecular Weight}}$$

$$= \frac{3.45}{138} = 0.025 \text{ M}$$

(ii) Molarity of solution A-12 i.e. dil hydrochloric acid

$$= \frac{M_1 V_1 (\text{acid})}{M_2 V_2 (\text{alkali})} = \frac{n_1}{n_2}$$

$$= \frac{M_1 \times 26.1}{0.025 \times 25} = \frac{2}{1}$$

$$M_1 = \frac{0.025 \times 25 \times 2}{26.1} = 0.04789272 \text{ M.}$$

$$\begin{aligned}
 \text{(iii) Concentration of solution A-12 i.e. dil hydrochloric acid} \\
 &= \text{Molarity} \times \text{Molecular weight} \\
 &= 0.04789272 \times 36.5 \\
 &= 1.7480 \text{ g/litre}
 \end{aligned}$$

Experiment No. 6

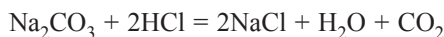
You are provided with a solution of an hydrous sodium carbonate (Na_2CO_3) labelled B-10 prepared by dissolving 10.25 gm of sodium carbonate per litre of the solution.

You are also provided with an other solution labelled B-11 of unknown concentration of dil HCl.

Perform titration between the two solution using methyl orange as indicator and calculate the following

1. Molarity of sodium carbonate solu B-10
2. Molarity of dil HCl solu B-11
3. Strength of dil HCl solu B-11 in gm/litre
4. Number of H^+ in B-11 in gm/litre.

The Chemical equation representing reaction between the two solutions is given as follows



Given: $\text{Na} = 23, \text{C} = 12, \text{O} = 16, \text{H} = 1, \text{Cl} = 35.5$

Apparatus required: Burette, Pipette, Titration flask, white glazed tile and Methyl orange soln.

Procedure 1. Wash, rinse and fill the burette with dil HCl.

2. Wash and rinse the pipette and transfer 25 ml of B-10 into a clean titration flask with its help.

3. Add to it two drops of methyl orange.

4. Run down the acid solution from the burette in small instalments, into the titration flask with constant shaking till the last drop gives permanent light pink colour.

5. Repeat the experiment to get two concordate readings

Observations – Size of pipette = 25.0 ml

S. No.	Burette Reading		Vol of acid used	End point or Titre value
	Initial	Final		
1.	0.0 ml	25.8 ml	25.8 ml	25.6 ml
2.	0.0 ml	25.7 ml	25.7 ml	
3.	0.0 ml	25.6 ml	25.6ml	
4.	0.0 ml	25.6 ml	25.6 ml	

Calculation

$$\text{Molecular weight of HCl} = 1 + 35.5 = 36.5$$

$$\begin{aligned}
 \text{Molecular weight of Na}_2\text{CO}_3 &= 2 \times 23 + 12 + 3 \times 16 \\
 &= 106
 \end{aligned}$$

$$\begin{aligned}
 \text{(i) Molarity of Na}_2\text{CO}_3 &= \frac{\text{Strength}}{\text{Molecular weight}} \\
 &= \frac{10.250}{106} \\
 &= 0.096698
 \end{aligned}$$

(ii) If M_1 and V_1 are molarity and volume of Na_2CO_3 and M_2 and V_2 are of HCl

$$\frac{M_1 V_1}{M_2 V_2} = \frac{n_1}{n_2} \quad n_1 \text{ and } n_2 \text{ are the number of moles reacting}$$

$$\frac{0.096698 \times 25}{M_2 \times 25.6} = \frac{1}{2}$$

So $M_2 = \text{molarity of HCl} = 0.18886328$

(iii) Strength of dil HCl = molarity \times molecular weight
 $= 0.18886328 \times 36.5$
 $= 6.8935$
 $= 6.894 \text{ gm/litre}$

(iv) 36.5 gm/litre of HCl contains $\text{H}^+ = 6.023 \times 10^{23}$

So 6.894 gm/litre of HCl contains $\text{H}^+ = \frac{6.023 \times 10^{23} \times 6.894}{36.5}$
 $= 11.376 \times 10^{22}$

Results

- (i) Molarity of $\text{Na}_2\text{CO}_3 = 0.096698 \text{ M}$
- (ii) Molarity of dil HCl = 0.18886328 M
- (iii) Strength of dil HCl = 6.894 gm/litre
- (iv) Number of H^+ in dil HCl = 11.376×10^{22}

Experiment No. 7

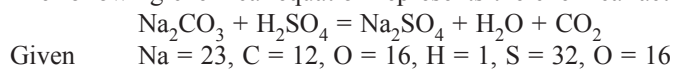
You are provided with

1. Soln. B-15 which is dil H_2SO_4 of concentration 9.450 gm/litre
2. Soln. B-16 which is of impure sodium carbonate (Na_2CO_3) of concentration 10.520 gm/litre

Perform titration taking B-15 in burette and B-16 in pipette using methyl orange as indicator and calculate the following.

1. Molarity of B-15 ie dil H_2SO_4 solu
2. Molarity of B-16 ie Impure Na_2CO_3 solu.
3. Strength of Na_2CO_3 solu.
4. Percentage purity of Na_2CO_3 in the sample used to make solution
5. Strength of CO_3^{2-} in the solution

The following chemical equation represents the chemical action between the solutions



Apparatus required

Burette, Pipette, Titration flask White glazed tile and methyl orange.

Procedure

1. Wash, rinse and fill the burette with B-15 up to mark.
2. Wash and rinse the pipette and use it to transfer 25 ml of B-16 in to well washed titration flask.
3. Add two drops of methyl orange to B-16 in titration flask.
4. Place the flask on white glazed tile and run down B-15 from the burette to add it to the contents of the flask in small instalments carefully shaking the flask and observing the colour of the solution till a permanent light pink colour is obtained.

5. Repeat the experiment till two concordant readings are obtained.

Observations – size of pipette = 25 ml.

S. No.	Burette Reading Initial	Final	volume of acid used	Titre value or End point
1.	0.0 ml	24.9 ml	24.9 ml	24.8 ml
2.	0.0 ml	24.8 ml	24.8 ml	
3.	0.0 ml	24.8 ml	24.8 ml	

Calculations

$$\begin{aligned}
 \text{(i) Molarity of H}_2\text{SO}_4 \text{ soln.} &= \frac{\text{Strength of H}_2\text{SO}_4 \text{ Solu.}}{\text{Molecular weight of H}_2\text{SO}_4} \\
 &= \frac{9.450}{98} = 0.09642857
 \end{aligned}$$

(ii) For molarity of Na_2CO_3 soln

$$\frac{M_1 V_1}{M_2 V_2} = \frac{n_1}{n_2}$$

Where M_1 V_1 are molarity and volume of H_2SO_4 and M_2 V_2 are for Na_2CO_3 Soln. n_1 , and n_2 are the number of moles reacting

$$\frac{0.09642857 \times 24.8}{M_2 \times 25} = \frac{1}{1}$$

$$M_2 = 0.095657142$$

$$\begin{aligned}
 \text{(iii) Strength of sodium carbonate soln.} &= \text{Molarity} \times \text{Molecular weight} \\
 &= 0.095657142 \times 106 \\
 &= 10.13965714 \\
 &= 10.140 \text{ gm/litre}
 \end{aligned}$$

(iv) For percentage purity of Na_2CO_3

10.520 gm of impure sample contains 10.140 gm of pure salt

$$\begin{aligned}
 \text{so 100 gm of impure sample contains} &= \frac{10.14 \times 100}{10.52} \\
 &= 96.3878\% \\
 &= 96.39\%
 \end{aligned}$$

(v) For concentration of CO_3^{2-}

$$\begin{aligned}
 \text{Molecular weight of Na}_2\text{CO}_3 &= 2 \times 23 + 12 + 3 \times 16 \\
 &= 46 + 12 + 48 = 106
 \end{aligned}$$

Since 106 gm of Na_2CO_3 has concentration of $\text{CO}_3^{2-} = 60$ gm/litre

$$\begin{aligned}
 \text{so 10.140 gm of Na}_2\text{CO}_3 \text{ has concentration of} &= \frac{60 \times 10.14}{106} \\
 &= 5.7396 \text{ gm/litre}
 \end{aligned}$$

Results–Molarity of $\text{H}_2\text{SO}_4 = 0.09642857$

(ii) Molarity of $\text{Na}_2\text{CO}_3 = 0.095657142$

(iii) Strength of Na_2CO_3 solu = 10.14 gm/litre

(iv) Percentage purity of $\text{Na}_2\text{CO}_3 = 96.39$

(v) Concentration of $\text{CO}_3^{2-} = 5.7396$ gm/litre.

EXPERIMENTS TO BE PERFORMED BY STUDENTS**Experiment No. 8.**

You are provided with two solutions as follows :

C_{10} is a solution of an acid H_xA , containing 0.90 mole per litre.

C_{11} is a solution of an alkali $M(OH)_2$ containing 1.085 mole per litre.

Fill the acid C_{10} in the burette and titrate 25 cm³ portions of C_{11} using methyl orange as indicator. Repeat the experiment to get the concordant readings.

Record the following :

(i) The titre value you intend to use in your calculations.

(ii) Molarity of C_{10}

(iii) Molarity of C_{11}

Calculate (i) the g - formula of the alkali which react with 1 g - formula of the acid.

(ii) The value of X

Write an ionic equation for the reaction between the alkali $M(OH)_2$ and the acid H_xA .

Experiment No. 9.

You are provided with two solutions as follows :

B_{10} is a solution of an alkali $X(OH)_n$ containing 0.120 g - formula per litre. B_{11} is a solution of hydrochloric acid containing 0.450 g - formula of it per litre.

Put the acid into the burette and titrate 25 cm³ portions of B_{10} using the indicator provided. Repeat the experiment to get concordant readings.

Calculate the following :

(i) Molarity of B_{10}

(ii) Molarity of B_{11}

(iii) Normality of B_{10}

(iv) Normality of B_{11}

(v) The value of n in $X(OH)_n$

(vi) The number of H^+ ions present per litre of the acid B_{11} .

Experiment No. 10.

You are provided with two solutions as follows :

C_{10} is a solution of certain alkali X_2CO_3 containing 5.6 g of it per litre.

C_{11} is a solution containing 5.0 g of sulphuric acid per litre.

Fill the sulphuric acid solution, C_{11} into the burette. Deliver 25 cm³ portions of the alkali solution C_{10} into a clean beaker.

Titrate this amount of the alkali solution C_{10} with sulphuric acid solution C_{11} using methyl-orange as indicator. Record the titre value you intend to use in your calculations.

Calculate the following :

(i) Molarity of sulphuric acid, C_{11}

(ii) Molarity of alkali solution, C_{10}

(iii) Normality of sulphuric acid, C_{11}

(iv) Normality of alkali solution, C_{10}

(v) The formula weight of the alkali X_2CO_3

- (vi) The atomic mass of the metal X present in the alkali, C_{10}
 (vii) The concentration of carbonate ion (CO_3^{2-}) in g/l present in the alkali C_{10}

Experiment No. 11

Object

You are provided with two solutions as follows :

- (a) Solution B-10 is a solution containing 9.850 g of sulphuric acid per litre.
 (b) Solution B-11 is a solution containing 28.650 g per litre of hydrated sodium carbonate ($\text{Na}_2\text{CO}_3 \cdot x\text{H}_2\text{O}$)

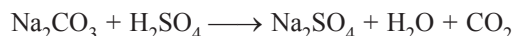
Procedure

Rinse and fill the burette with sulphuric acid solution B - 10, Ripette out 25 ml (or 20 ml) of solution B - 11 into a clean conical flask. To this add 2 drops of methyl orange indicator.

Rundown the solution of acid from burette dropwise with constant whorling the flask till the last drop of acid gives a permanent pale pink colour. This indicates the end point. Repeat the titration to get at least two concordant readings. Tabulate your readings.

State the size of pipette used in the titration and the titre value you wish to use in your calculations

The equation for the reaction may be written as below :



The relative atomic masses are:

$$\text{S} = 32, \text{Na} = 23, \text{C} = 12, \text{O} = 16, \text{H} = 1$$

Calculate the following:

- (a) The molarity of the solution of sulphuric acid B-10.
 (b) The molarity of the solution of hydrated sodium carbonate ($\text{Na}_2\text{CO}_3 \cdot x\text{H}_2\text{O}$) B-11.
 (c) The molecular mass of hydrated sodium carbonate.
 (d) The value of x .
 (e) The formula of the hydrated sodium carbonate.

Observation table

S.No.	Burette Reading		Valume of H_2SO_4 used	End Point
	Initial	Final		
1.	0.0 ml	25.0 ml	25.0 ml	24.9 ml
2.	0.0 ml	24.9 ml	24.9 ml	
3.	0.0 ml	24.9 ml	24.9 ml	

Capacity of pipette used = 25 ml

Titre value = 24.9 ml

Calculations

$$\begin{aligned} \text{(a) Molarity of } \text{H}_2\text{SO}_4 &= \frac{\text{Strength}}{\text{Molecular weight}} \\ &= \frac{9.850}{98} = 0.100510204 \text{ M} \end{aligned}$$

(b) Molarity of sodium carbonate solution:

$$\frac{M_1 V_1 (\text{acid})}{M_2 V_2 (\text{Sod. carbonate})} = \frac{n_1}{n_2}$$

$$\frac{0.100510204 \times 24.9}{M_2 \times 25} = \frac{1}{1}$$
$$M_2 = \frac{0.100510204 \times 24.9}{25} = 0.100108163 \text{ m}$$

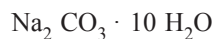
(c) Molecular mass of hydrated sodium carbonate

$$\text{Molecular mass} = \frac{\text{strength}}{\text{molarity}}$$
$$= \frac{28.650}{0.100108163} = 286.190 \text{ g}$$

(d) The value of x

$$\begin{aligned}\text{Na}_2\text{CO}_3 \cdot x \text{H}_2\text{O} &= 286.190 \\ 106 + x \text{H}_2\text{O} &= 286.190 \\ 18x &= 286.190 - 106 \\ 18x &= 180.190 \\ x &= \frac{180.190}{18} = 10.019 \\ x &= 10\end{aligned}$$

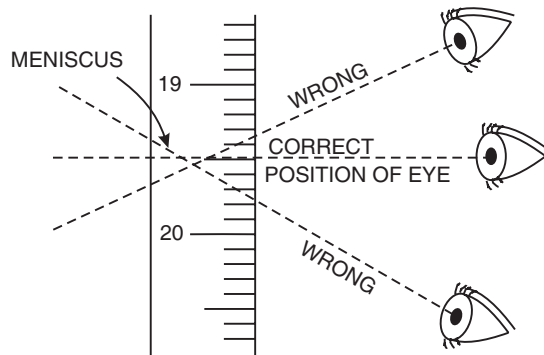
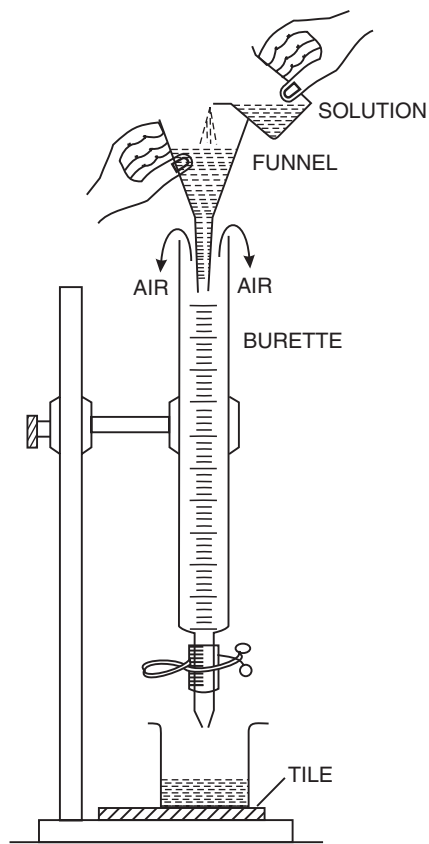
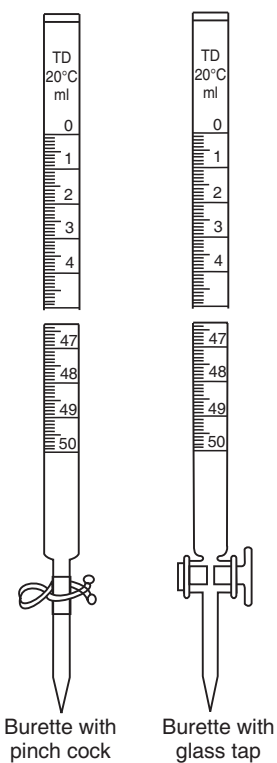
(e) The formula of hydrated sodium carbonate:



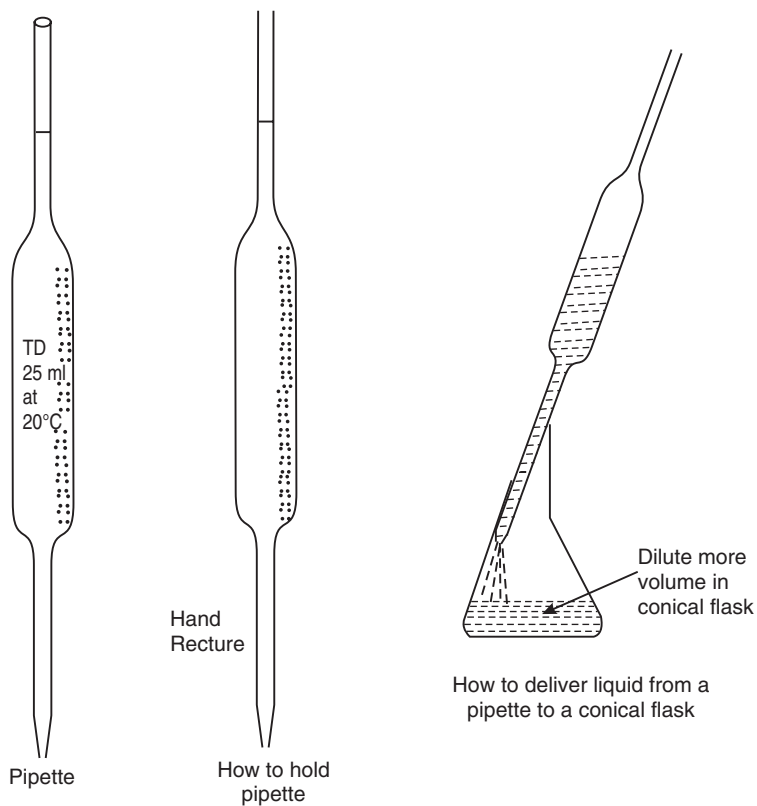
**DIAGRAMS OF THE APPARATUS USED IN VOLUMETRIC ANALYSIS
(TITRATION EXPERIMENTS)**

1. BURETTE

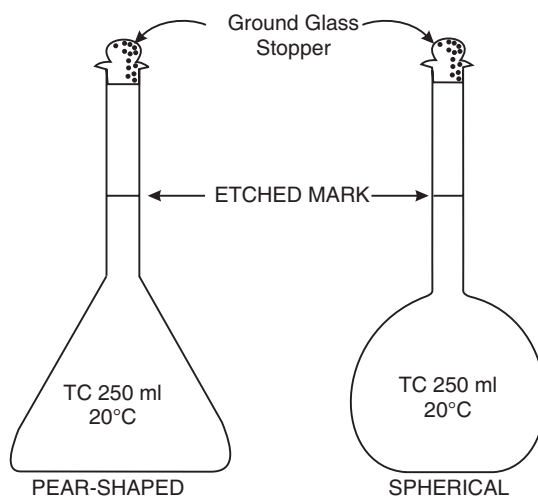
BURETTE



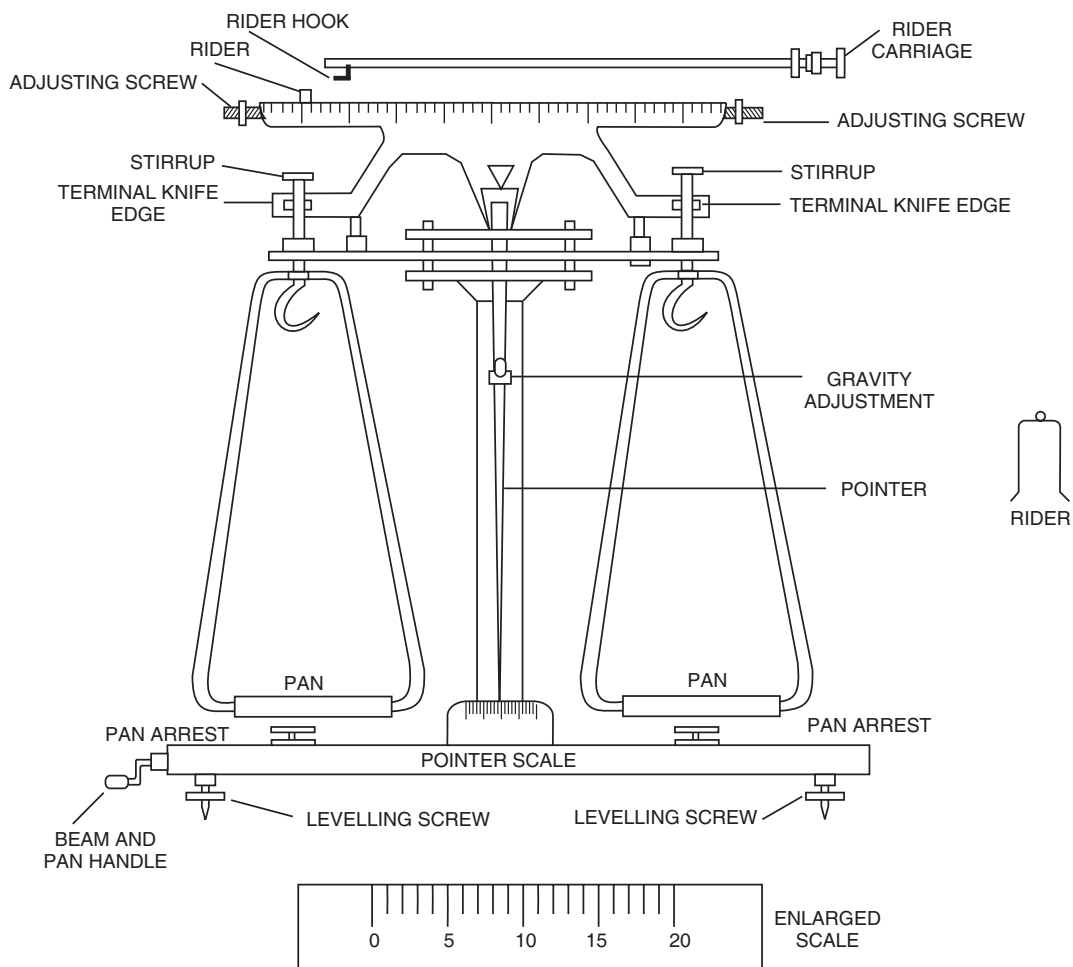
2. PIPETTE



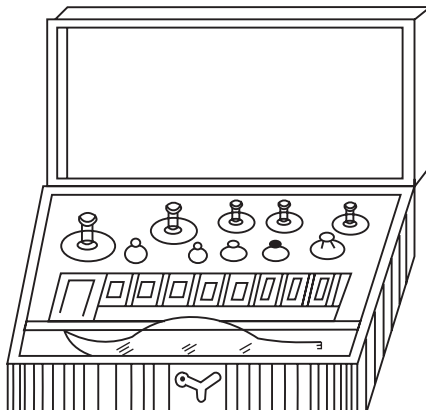
3. MEASURING FLASK

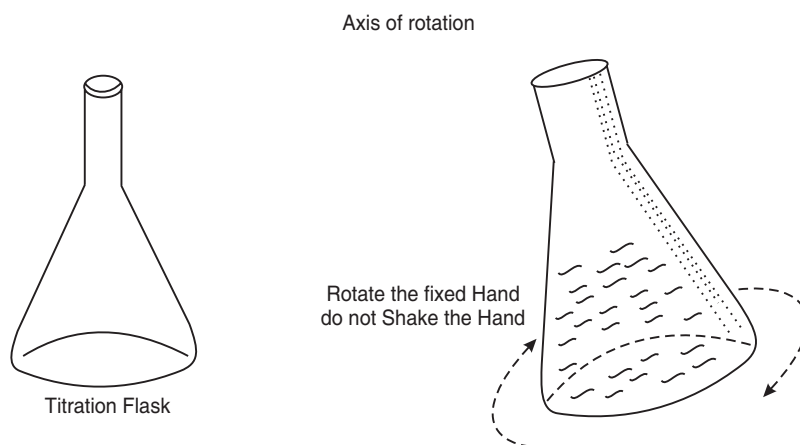


4. CHEMICAL BALANCE



5. WEIGHT BOX



7. TITRATION FLASK**6. FORCEPS**

Chapter 3 Qualitative Analysis

What is Analysis?

The word analysis means separating a substance into its elements or component parts. It stands for the determination of the quality and quantity of these constituents.

Analytical work is of utmost importance in the study of chemistry. The branch of chemistry in which all the operations carried out to determine the constituents of a chemical compound or a mixture of chemical compounds are studied, is known as Analytical Chemistry. It is sub-divided into two branches:—

1. Qualitative Analysis, and
2. Quantitative Analysis.

Qualitative Analysis. It deals with the identification of an unknown salt or a mixture of two or more salts.

Every salt consists of two parts or radicals (ions) known as basic radical and acid radical.

Basic Radical – It is the positive ion or the cation.

Acid Radical – It is the negative ion or the anion.

Every salt in its solution undergoes dissociation to produce cations and anions. The detection of cations and anions which a salt or a mixture of salts produce in solution is known as qualitative Analysis.

Quantitative Analysis– It includes those methods which are employed to know the exact quantities of the component present in any substance, which may be a pure chemical compound or a mixture containing several elements or compounds. This analysis is divided into the following two types:

- (i) *Volumetric Analysis* – In this analysis, the volume of a solution of known concentration which is required to react quantitatively with the known volume of the solution of the other substance, is determined.
- (ii) *Gravimetric Analysis* – In this analysis the quantitative estimation of a substance is performed by converting it into a pure, stable, soluble compound of known composition.

A systematic scheme is required to detect cations and anions in the inorganic quantitative analysis. The cations are divided into six groups, depending on their solubility and solubility products, in presence of some strong electrolyte having a common ion with those of the precipitating electrolyte. There is a separate group known as zero group, **which contains Ammonium ion (NH_4^+), with no group reagent and is identified by a separate test.** The group reagents for various groups, their cations and the respective colours of their precipitates are summarised below in a tabular form.

Group	Group Reagent	Cations	Formulae and colours of the precipitated salts
Zero	Caustic soda Solution	NH_4^+	No precipitate but a characteristic odour is felt when mixture is heated with Sodium Hydroxide solution.
Ist	dil. HCl	Pb^{2+}	PbCl_2 (White)
IIInd	H_2S in acidic medium	Pb^{2+} , Cu^{2+}	PbS (black), CuS (black)
IIIrd	NH_4OH in presence of NH_4Cl	Al^{3+} , Fe^{3+}	$\text{Al}(\text{OH})_3$ (White) $\text{Fe}(\text{OH})_3$, (reddish brown)
IVth	H_2S gas in presence of NH_4Cl and NH_4OH	Zn^{2+} , Mn^{2+} , Ni^{2+}	ZnS (White), MnS (Flesh coloured) NiS (Black)
Vth	$(\text{NH}_4)_2\text{CO}_3$ in presence of NH_4Cl and NH_4OH	Ba^{2+} , Sr^{2+} , Ca^{2+}	BaCO_3 (White) SrCO_3 (White) CaCO_3 (White)
VIth	Na_2HPO_4 in ammoniacal solution	Mg^{2+}	$\text{Mg}(\text{NH}_4)\text{PO}_4$ (White)

The detailed tests for various cations including the knowledge of reaction necessary for formal group analysis are described below.

Preparation of Original Solution (abbreviated as O.9)

It is necessary to bring the given mixture into solution to get it into ionic forms which bring about all inorganic chemical reactions. It is because inorganic reactions are ionic in nature and when the salts are dissolved in water or any other suitable solvent, they are ionised.

The selection for the suitable solvent for the given mixture must be made in the following order:

1. *Water* – Water is known as universal solvent and most of the salts dissolve in it. Therefore it should be tried as a solvent before selecting another. The solution so prepared is known as aqueous solution.

Place a small amount of the given substance in a clean, glass test tube and add sufficient quantity of water to it. Shake if required. If the substance does not dissolve, the contents are heated.

Note.

- All salts of sodium, potassium and ammonium are soluble in water.
 - All acetates, bicarbonates, halides (Chloride, Bromide, Iodides), Nitrites and Nitrates are soluble in water, except silver halides.
 - Most of the carbonates, sulphides except CaS , Sulphites, Sulphates and Phosphates are insoluble in water.
 - Exceptions-*lead chloride is soluble in hot water* and insoluble in cold water.
2. *Hydrochloric Acid*. If the substance does not dissolve in cold or hot water, it should be tried with a small amount of dil. HCl, first cold and then hot. In case the substance does not dissolve in dil. HCl, conc. HCl should be used with a fresh portion of the given substance, first at room temperature and then by heating. When O.S. is prepared in hydrochloric acid it indicates the absence of the cation of the first group (Pb^{2+}).

3. HNO_3 (Nitric Acid). If none of the above named solvents, works, dil. or conc. HNO_3 should be tried with a fresh portion of substance in a separate test tube to facilitate the dissolution of lead salts.

Note : Use of nitric acid should be avoided and if necessary, minimum quantity of acid should be used in order to prevent the oxidation of H_2S to colloidal Sulphur in 2nd group.

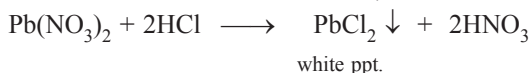
Group reagent is added to original solution one after another, till the precipitate in any group is obtained. The precipitate obtained indicates the presence of some radical in that group. Excess of group reagent should be added to ensure complete precipitation otherwise the unprecipitated ions in the solution will interfere in the identification of subsequent group radicals.

ANALYSIS OF GROUP – I

This group includes lead when dil. HCl is added to the cold solution of the given substance in water or nitric acid, a white precipitate of lead chloride is formed.

Reactions of Lead Ions

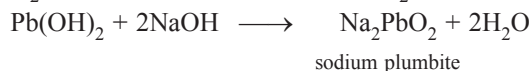
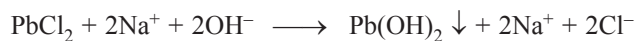
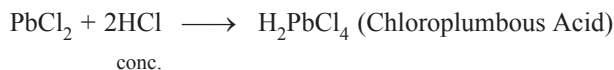
When dil. HCl is added to a lead nitrate solution, lead chloride is precipitated in cold.



Ionicly,



The lead chloride so precipitated is soluble in hot water. This property is the basis of the separation of lead from Silver Chloride and it gives a white ppt. again when the solution is cooled. It is also soluble in hot conc. HCl and strong alkalies forming complexes.

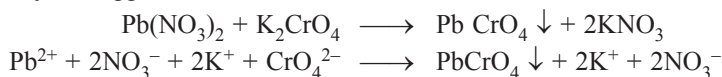


Confirmatory Tests for Lead

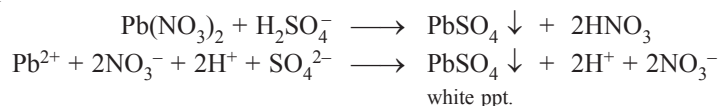
(a) *Potassium Iodide Test.* When KI solution is added to lead nitrate solution a canary yellow ppt. of lead iodide is formed.



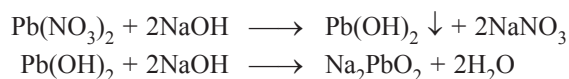
(b) *Potassium Chromate Solution.* When K_2CrO_4 solution is added to lead nitrate solution a mustard yellow ppt. of lead chromate is obtained.



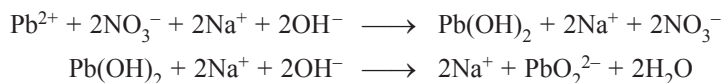
(c) *Dil. Sulphuric acid.* When dil. H_2SO_4 is added to lead nitrate solution, a white ppt. of lead sulphate is obtained.



(d) *Sodium Hydroxide Solution.* When NaOH solution is added to lead nitrate solution a white ppt. of lead hydroxide is obtained. The white precipitate is soluble in excess of NaOH solution as Sodium Plumbite is formed.



Ionically,



Detection and Separation of Lead Ion in First Group

To the original solution of the substance in a test tube add dil. HCl, if a white ppt. is produced, group I is present.

Filter the solution. Retain the filtrate for subsequent groups. Collect the ppt. and dissolve it in hot water. Dissolution of the ppt. indicates the presence of lead ion.

A tabular representation of Group I radicals

To 0.5 gm of the substance in test tube add dil. HCl. Appearance of white precipitate indicates the presence of Ist group. Filter the solution. Retain the filtrate for subsequent groups. Pour hot water on to the filter paper containing the white ppt.

Treatment of the solution

Divide this hot solution in four parts:

1. Cool the first part under tap water; white shining crystals observed.
2. Hot solution + $\text{K}_2\text{CrO}_4 \longrightarrow$ mustard
Yellow ppt. observed
3. Hot solution + KI \longrightarrow Canary
Yellow ppt. observed.
4. Hot solution + NaOH solution \longrightarrow
white ppt. observed.

Excess NaOH solution \longrightarrow white
ppt. dissolves. (Pb²⁺ confirmed)

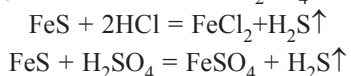
ANALYSIS OF GROUP II

This group includes lead and copper. To a small portion of filtrate of first group, pass H₂S gas. Appearance of black ppt. indicates the presence of 2nd group. (If no such change occurs, reject this solution and proceed to third and subsequent groups with remaining filtrate of Ist group.

When a black precipitate appears, pass H₂S gas, into remaining filtrate of Ist group. Filter the solution. Retain the filtrate for subsequent groups, while use the precipitate for IInd group.

Preparation of H₂S gas by kipp's apparatus :

Hydrogen sulphide gas, also known as sulphuretted hydrogen, is prepared in lab by treating ferrous sulphide pieces (not powder) with dil HCl or dil H₂SO₄ as shown below :



As the gas is required intermittently in the lab to precipitate the second and fourth group cations in to their sulphides, a woulfe's bottle can not be used for the purpose.

Our object is to use an apparatus which supplies us with the required amount of gas when needed and stop the reaction when not required. The purpose is best served using an apparatus known as Kipp's apparatus. It is known after the name of the scientist who designed it.

Construction—It consists of three glass bulbs(A), (B) & (C) as shown in the figure 1.

(A) and (B) are spherical and (C) is hemispherical. (A) is detachable.

Bulb A is opened on the top. Its bottom has a base stem, narrowing in diameter as it goes into the bulb (C) Bulb (B) has an outlet (E) which has a delivery tube fitted with a stop cock. The bulb (E) has an opening (F) fitted with a tight cork. The apparatus can be emptied through this.

Setting of apparatus—cork (E) is removed and pieces of ferrous sulphide are introduced gently through it. The pieces should be big enough to rest at the bottom of (B). If small in size, they will fall down into (C) and the purpose of using this especially designed apparatus will be defeated). Dil HCl or dil H_2SO_4 is filled through the upper bulb (A) which falls and fills (C).

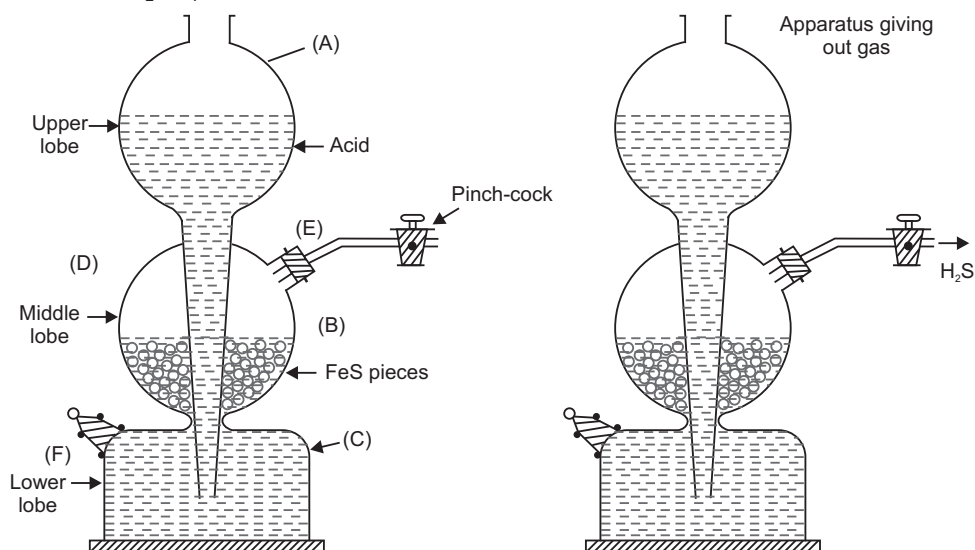


Fig. 1

Little more acid is filled to allow it to rise up to the bulb, (B) to touch the FeS pieces.

Function—The apparatus is ready for use. The reaction starts and the gas starts filling the lobe (B) which exerts a downward pressure on the acid, pushing it up to the sphere (A) through the stem (D). The acid and the FeS pieces in lobe (B) are out of touch and the reaction stops.

Gas, when required in lab is obtained on opening the stop cock. Escaping of gas from lobe (B) reduces pressure in it helping acid to fall down into (C) from (A) through (D) This keeps the reaction on till required.

The sample of the gas obtained by this method is quite impure particularly due to hydrogen gas. The commercial sample of ferrous sulphide used in the process is responsible for it.

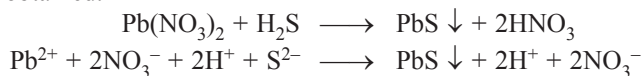
The gas can be purified by passing it through potassium permanganate solution but practically it is not done as the impure sample serves the purpose of Class XI syllabus.

Note-(I) While using the apparatus, we must be careful about keeping (E) and (F) tight. A little mishandling of the apparatus will disturb them and the apparatus will fail to work.

(II) Teachers are advised to prepare saturated solution of the gas in distilled water and store it in well stoppered bottle for students' use.

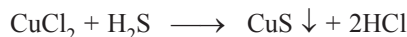
Reactions of Lead Ions

When H_2S gas is passed into a lead nitrate solution acidified with HCl, a black ppt. of lead sulphide is obtained.



Reactions of Copper Ions

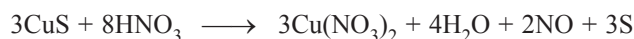
When H₂S gas is passed into the filtrate of Ist group, a black ppt. of Cupric Sulphide is formed.



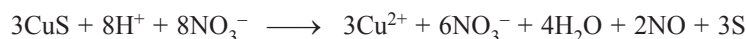
Ionically,



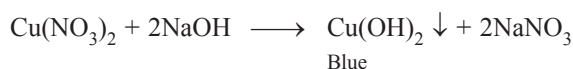
The black ppt. is dissolved in hot and dil. nitric acid.



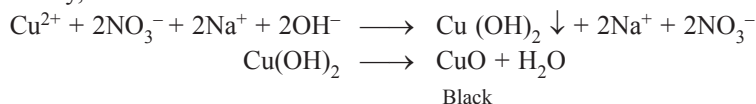
Ionically,

**Confirmatory Test for Copper Ions***(a) Sodium Hydroxide Solution*

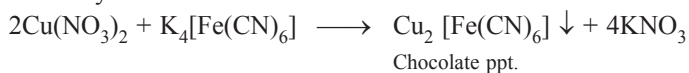
When a solution of sodium hydroxide is added to cupric nitrate solution a blue ppt. of cupric hydroxide is formed, which is insoluble in excess NaOH and turns black on heating, producing CuO.



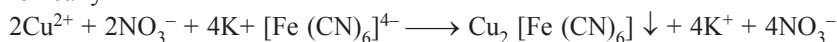
Ionically,

*(b) Potassium Ferro Cyanide Solution*

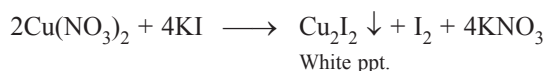
When potassium ferro cyanide solution is added to a solution of copper nitrate, a chocolate ppt. of cupric ferrocyanide is obtained.



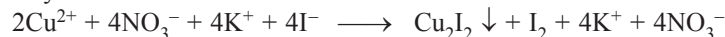
Ionically

*(c) Potassium Iodide Solution*

When KI solution is added to a cupric nitrate solution, cuprous iodide is formed and Iodine is set free



Ionically

**Detection and Separation of Lead and Copper**

When H₂S gas is passed in the filtrate of Ist group, a black ppt. indicates the presence of 2nd group. the solution is filtered. Filtrate is retained for 3rd and subsequent groups. Treat the black ppt. with dil. Sulphuric acid. Collect the filtrate to test copper (Cu²⁺) and the residue to test lead (Pb²⁺) ions.

A Tabular Representation of Group II Radicals

To the filtrate of 1st group pass H₂S gas. Appearance of black ppt. indicates the presence of IInd group.

Filter the solution. Retain the filtrate for subsequent groups. Pour dil. H_2SO_4 onto the filter paper containing black ppt. Add ethyl alcohol if available.

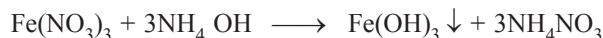
Treatment of Residue	Treatment of Filtrate
Dissolve the white ppt. in ammonium acetate. Add dil. acetic acid. Divide the solution in two parts 1. Add KI \longrightarrow canary Yellow ppt. 2. Add $\text{K}_2\text{CrO}_4 \longrightarrow$ mustard yellow ppt. (Pb^{2+} confirmed)	Divide the filtrate into two parts. 1. Add NaOH solution \longrightarrow Blue ppt. Heat \longrightarrow Black ppt. 2. Add NH_4OH solution dropwise and then in excess \longrightarrow bluish white ppt. formed, turns deep blue solution with excess ammonium hydroxide NH_4OH 3. Add $\text{K}_4[\text{Fe}(\text{CN})_6] \longrightarrow$ Chocolate coloured ppt. (Cu^{2+} confirmed)

ANALYSIS OF GROUP III

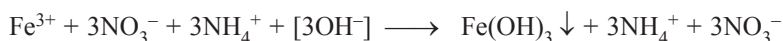
This group includes Fe^{+3} and Al^{+3} . Boil off H_2S Confirm by lead acetate paper which gives silver black ppt in presence of H_2S gas. from the filtrate of 2nd group and add 2-ml of conc. HNO_3 and boil to oxidise the ferrous ions to ferric ions if present in solution. Cool the solution. Add solid NH_4Cl and NH_4OH in excess (till the solution smells of ammonia). Appearance of Reddish Brown precipitate indicates the presence of ferric ions, while a white ppt. indicates the presence of Aluminium ion.

Reaction of Ferric Ions

When excess NH_4OH solution is added in presence of NH_4Cl in the cool filtrate of 2nd group after boiling off H_2S and due oxidation, a reddish brown ppt. of $\text{Fe}(\text{OH})_3$ is formed.

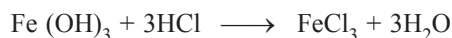


Ionicallly,



When the Reddish brown ppt. is dissolved in dil. HCl , ferric chloride and water are formed.

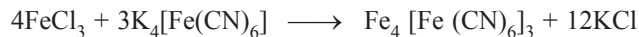
Ionicallly,



Confirmatory Test for Ferric Ion

Divide the solution in two parts

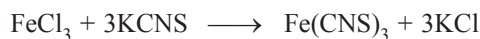
(a) Add Potassium Ferro Cyanide solution \longrightarrow deep blue ppt. is formed.



Deep Blue (Prussian blue)

(b) Add Potassium Thio Cyanate solution (KCNS)

A blood red coloration is obtained.

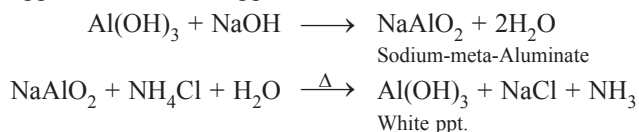


Blood Red

Confirmatory Test for Aluminium Ion

Dissolve white ppt in minimum amount of dil HCl. Now add caustic soda solution \longrightarrow white ppt soluble in excess caustic soda solution.

When white ppt. is dissolved in caustic soda solution and the soln. is treated with solid NH_4Cl and heated, reappearance of white ppt. confirms Al^{3+}

**Detection and Separation of Fe^{3+} and Al^{3+}**

Collect the filtrate of 2nd group. Boil off H_2S completely. Add NH_4Cl and NH_4OH in excess. Formation of reddish brown or white ppt. indicates the presence of 3rd group. Filter the solution. Retain the filtrate for 4th group and subsequent groups. Collect the filtrate for the identification of Fe^{3+} and Al^{3+} .

When white ppt. appears in 3rd group, it indicates the presence of Aluminium (Al^{3+}) only but the appearance of reddish brown ppt. indicates two possibilities.

- (i) Only Ferric ions are present.
- (ii) Both Ferric and Aluminium ions are present.

As the reddish brown coloured ppt. Fe(OH)_3 will mask the white coloured ppt. of Al(OH)_3 and the latter will not be visible.

Note: For ISC Chemistry practical exam. combination of two radicals of same group is avoided.

A Tabular Representation of IIIrd Group

Pour caustic soda solution to the filter paper containing the III group ppt. Collect the filtrate for Al^{3+} test and residue for Fe^{3+} .

Treatment of Residue	Treatment of Filtrate
1. Dissolve the residue in dil. HCl and divide the solution in two parts 1. Add $\text{K}_4[\text{Fe(CN)}_6]$ solution \longrightarrow Deep blue colour 2. Add KCNS solution \longrightarrow Blood Red Colour Fe^{3+} confirmed	1. Add solid NH_4Cl in a part of filtrate and heat to boil. Reappearance of white ppt. of Al(OH)_3 confirms the presence of Aluminium (Al^{3+}) ions. 2. To the second part add a few drops of blue litmus solution which turns red. Now add excess ammonium hydroxide. A blue floating precipitate in colourless solution is seen. Al^{3+} confirmed.

IDENTIFICATION OF GROUP IV RADICALS

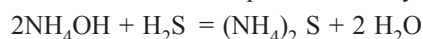
If first, second and third groups are absent, we shall begin with nearly 5 ml. of original solution, but if any of these groups is present, we shall begin with the filtrate of the corresponding group.

To a portion of the original solution add a pinch of ammonium chloride and excess ammonium hydroxide (the chemicals may not be added if already added for third group.) followed by hydrogen sulphide gas.

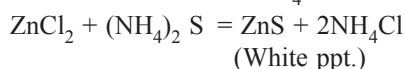
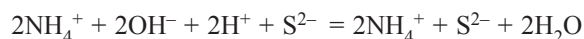
Details of the sulphide precipitates are given below.

<i>Colour of the Precipitate</i>	<i>Name of the Precipitate</i>	<i>Formulas of Sulphide</i>	<i>Cation Indicated</i>
White or grey	Zinc sulphide	ZnS	Zn ²⁺
Flesh coloured	Manganese sulphide	MnS	Mn ²⁺
Black	Nickel sulphide	NiS	Ni ²⁺

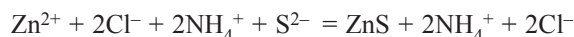
Filter the solution and retain the filtrate for the next group. Use residue for the fourth group. The following reactions show the formation of sulphides when hydrogen sulphide is passed.



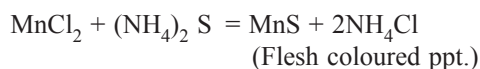
Ionicallly :



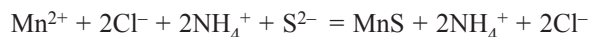
Ionicallly :



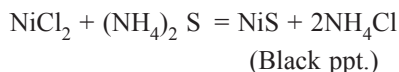
Similarly :



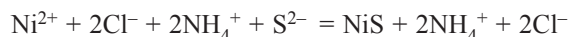
Ionicallly :



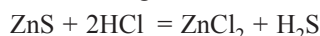
Similarly :



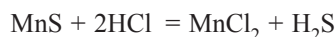
Ionicallly :



The precipitate obtained above is treated with minimum quantity of dil. hydrochloric acid. Solution is heated for a long time to boil off H₂S produced by the interaction of ZnS and dil hydrochloric acid. The sulphides of zinc and manganese dissolve but that of nickel does not.



Ionicallly :



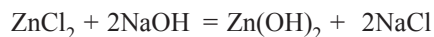
Ionicallly :



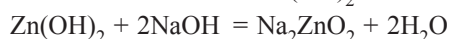
The chlorides of zinc and manganese are used to perform tests for them.

Test for Zinc Ions :

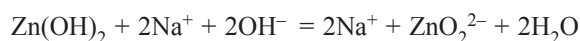
1. To the solution obtained above add caustic soda solution drop by drop. A white or greyish precipitate appears which dissolves in excess caustic soda solution due to its amphoteric nature.



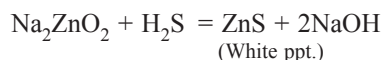
Ionicallly :



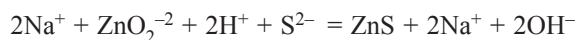
Ionicly :



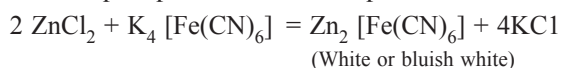
Pass hydrogen sulphide gas into it, a white precipitate of zinc sulphide confirms the presence of zinc ions in the solution.



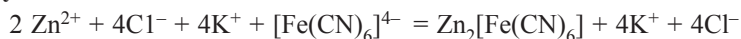
Ionicly :



2. Treat the second portion of the solution with a potassium ferrocyanide solution. Formation of a white or a bluish white precipitate confirms the presence of zinc ions.



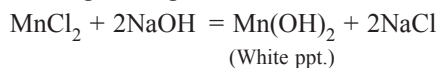
Ionicly :



Test for Manganese Ions :

1. Add caustic soda solution drop by drop till it is in excess. A white precipitate appears which remains insoluble in presence of excess caustic soda solution.

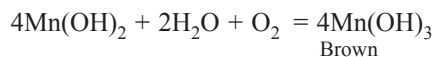
The reactions explaining the changes are given below :



Ionicly :

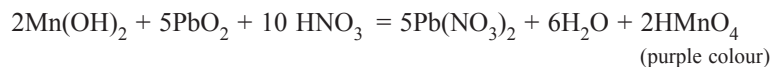


In contact with oxygen of air it turns brown. The following reaction explains the change.



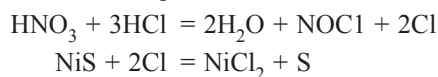
This confirms the presence of manganese ions.

2. To the solution of the ppt. (MnS) prepared in dil. hydrochloric acid add caustic soda soln. to convert it into its hydroxide. Filter the soln. and reject the filtrate. Dissolve the precipitate in conc. nitric acid in presence of lead dioxide. Boil it for some time and dilute it. A purple colour is observed due to the formation of permanganic acid. The reaction is as given below.



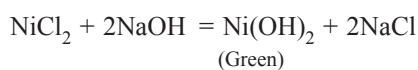
Test for Nickel Ions :

The black precipitate of nickel sulphide which is insoluble in dil. hydrochloric acid is treated with aqua regia to dissolve it. The reactions take place as shown below.



Boil it for some time to reduce its volume and divide the solution into two parts.

1. To one part add caustic soda solution; a green coloured precipitate confirms the presence of nickel ions.

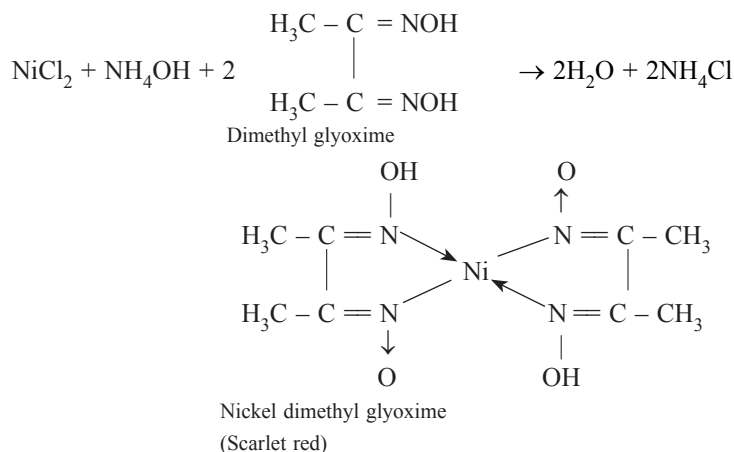


Ionicly :



2. To the second portion add excess ammonium hydroxide followed by some dimethyl glyoxime solution a scarlet red precipitate is observed due to the formation of nickel dimethyl glyoxime. This confirms the presence of nickel ions.

The appearance of scarlet red precipitate can be explained in the following manner:



The tests of members of 4th group can be summarised in the tabular form as given below.

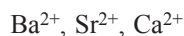
To the O.S. (if all three groups are absent) or to the filtrate of the previous group, add a pinch of NH_4Cl excess NH_4OH followed by H_2S gas passed into it.

Filter the solution. Retain the filtrate for the next group and use residue for the 4th group.

Treat the residue with dil. HCl .

<i>Precipitate</i> <i>Insoluble (Black colour) Ni^{2+}</i>	<i>Precipitate dissolves</i> <i>Zn^{2+}</i>	<i>Mn^{2+}</i>
Dissolve the ppt. in aqua regia and boil for long time.	1. Soln. + $\text{NaOH} \rightarrow$ dropwise till in excess \rightarrow white ppt. soluble in excess NaOH . Pass H_2S . A white ppt.	Soln. + $\text{NaOH} \rightarrow$ dropwise till in excess \rightarrow white ppt. insoluble in excess \rightarrow Turns brown after some time.
1. Soln. + $\text{NaOH} \rightarrow$ green ppt.		
2. Soln. + NH_4OH + Dimethyl glyoxime \rightarrow scarlet red ppt.	2. Soln. + $\text{K}_4[\text{Fe}(\text{CN})_6]$ Bluish white ppt.	

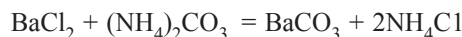
IDENTIFICATION OF GROUP V RADICALS



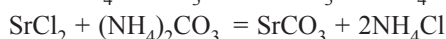
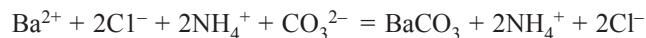
Begin with original soln. if all four groups are absent. If filtrate of fourth group is used boil it for some time to get rid of hydrogen sulphide as well as to concentrate the soln. to reduce it to one-third of original volume.

To this, add a pinch of ammonium chloride and excess ammonium hydroxide followed by ammonium carbonate soln. Formation of a white precipitate indicates the presence of fifth group.

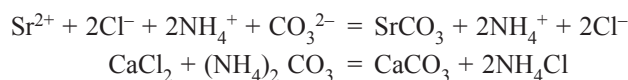
The following reactions explain the appearance of white precipitate due to the formation of the carbonates.



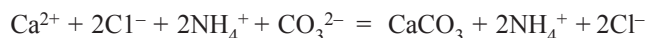
Ionicallly :



Ionicallly:



Ionicallly :

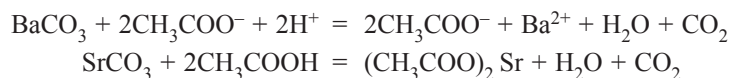


Filter this solution and retain the filtrate for the sixth group. Use residue for fifth group.

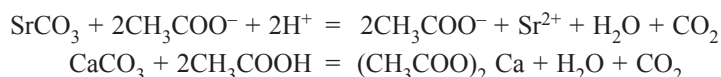
Dissolve the residue **in minimum quantity of hot acetic acid**. The dissolution of carbonate can be explained in the following way.



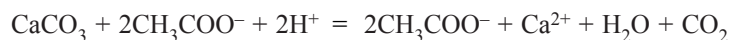
Ionicallly:



Ionicallly:

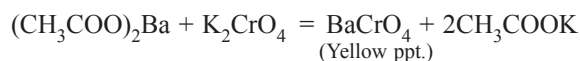


Ionicallly :

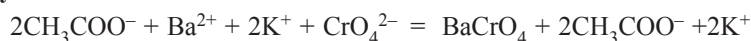


Use the solution to perform tests for barium, strontium and calcium in this very order.

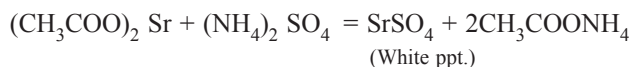
To the first portion add potassium chromate solution; a yellow precipitate confirms the presence of barium ions.



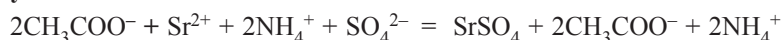
Ionicallly:



If barium ions are absent, add a concentrated solution of ammonium sulphate, boil it and allow to cool. A white ppt. confirms the presence of strontium ions.



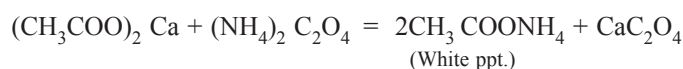
Ionicallly:



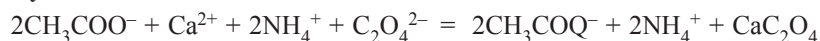
Note: Sometimes, the white precipitate appears only on scratching the sides of the test tube using a glass rod.

If barium and strontium ions are absent, use the third portion for calcium ions.

Add ammonium oxalate soln. to it and heat, a white precipitate confirms the presence of calcium ions. Calcium oxalate is formed in the reaction.



Ionicallly:



The tests for the members of fifth group can be summarised in the following table.

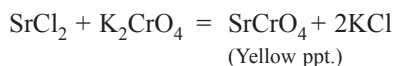
O.S. or filtrate of the previous group + NH_4Cl + NH_4OH (excess) + $(\text{NH}_4)_2\text{CO}_3 \rightarrow$ white ppt. Filter \rightarrow use filtrate for the next group and residue for the fifth group.

Residue is dissolved in minimum amount of hot and dilute acetic acid after the contents. Divide the solution into three parts

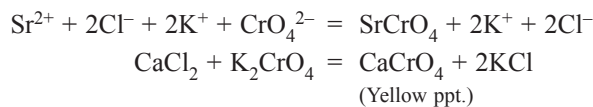
Ba^{2+}	Sr^{2+}	Ca^{2+}
Soln. + K_2CrO_4 Yellow ppt. Insoluble in acetic acid.	If Ba^{2+} is absent, soln. + $(NH_4)_2SO_4 \rightarrow$ white ppt. Insoluble in acetic acid	If both Ba^{2+} and Sr^{2+} are absent: soln. + $(NH_4)_2C_2O_4 \rightarrow$ white ppt. Insoluble in acetic acid.

It must be noted carefully that potassium chromate which is used to detect barium ions gives yellow precipitate with strontium and calcium salts too, as they also produce chromates. Same way ammonium oxalate solution gives a white precipitate not only with solution of calcium salts but also with barium and strontium due to the formation of their oxalates. However, we can distinguish them from each other on the basis of their solubility in acetic acid.

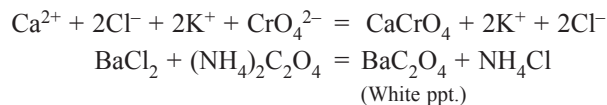
Formation of the chromates and oxalates can be explained with the help of following chemical equations :



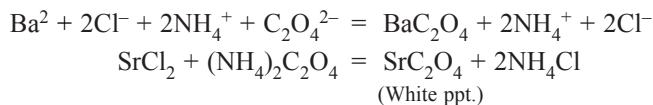
Ionicallly :



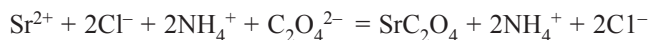
Ionicallly :



Ionicallly :



Ionicallly :



The following table states the effect of acetic acid on the named precipitates.

<i>Cation present in the salt</i>	<i>Yellow ppt. with K_2CrO_4 (Treated with CH_3COOH)</i>	<i>White ppt. with $(NH_4)_2C_2O_4$ (Treated with CH_3COOH)</i>
Ba^{2+}	Insoluble	Soluble in hot acid
Sr^{2+}	Soluble	Insoluble
Ca^{2+}	Soluble	Insoluble

Once an indication is made of the presence of fifth group by observing white ppt. on adding ammonium carbonate to the solution and the confirmatory test is performed for the particular member, flame test must be done to verify the result. The colours observed are given below.

Barium ions	Grassy green
Strontium ions	Crimson
Calcium ions	Brick red

Flame Test :

Flame test is one of the dry tests used to detect some selected cations which impart a particular colour to the oxidising flame of a Bunsen burner.

A clean platinum wire and conc. hydrochloric acid are used for the purpose.

Place about 8 ml of pure conc. hydrochloric acid in a watch glass and to it add a pinch of the given salt of mixture and prepare a thick paste of it using platinum wire to mix them so that some paste sticks to the loop of the platinum wire.

Now bring the loaded wire into the non-luminous flame of the Bunsen burner and observe the colour imparted to it by the paste. The colour of the flame changes when viewed through a cobalt glass.

The colours as seen with naked eye and through cobalt (blue) glass for different cations are given in the following table.

<i>S.No.</i>	<i>Colour Through Naked Eye</i>	<i>Colour Through Blue Glass</i>	<i>Cation Indicated</i>
1.	Bluish Green	—	Copper
2.	Brick Red	Light Green	Calcium
3.	Crimson Red	Crimson Red	Strontium
4.	Grassy Green	Bluish Green	Barium
5.	Golden Yellow	Invisible (Nil)	Sodium
6.	Lilac (Violet)	Pink or Purple	Potassium

Note : Sodium and potassium ions are not in course of I.S.C.

How to Clean a Platinum Wire ?

Bend a small loop at the end of the platinum wire and dip the looped end in concentrated hydrochloric acid taken in a test tube. Now bring it to the oxidising (non-luminous) flame of the burner. A change in colour of the flame indicates that the wire is not clean and needs the above operation. The operation should be repeated till the wire is clean. Platinum wire should always be cleaned after the flame test is performed.

Note : It should be noted that persistent golden yellow colour of sodium flame conceals all other flames, therefore, flame has to be observed carefully in presence of sodium.

Theory of Flame Test :

Ionisation of a salt can be achieved in two ways

1. *By dissolving it in water* – This is utilised in wet tests when original solution of the salt is used in analysis.
2. *By thermal ionisation* – This property is utilised in flame test.

All salts get ionised at some temperature or the other when heated. The halides of metals have lower thermal ionisation temperature whereas oxides have very high. Carbonates, nitrates, etc., decompose into their oxides and do not get vapourised when brought to the flame. This is why halides only are used. Also hydrochloric acid is cheap and easily available, therefore, all the salts are treated with hydrochloric acid to convert them into their chlorides which when brought to the flame ionise to set free the cation which imparts its characteristic colour to the flame.

Platinum wire only is used for the purpose simply because :

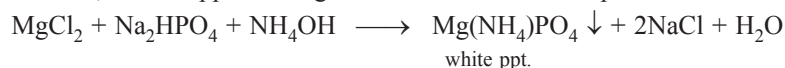
1. Platinum does not react with conc. hydrochloric acid nor with the chloride of the metal formed, even at the temperature of the flame.
2. Platinum does not impart any colour to the flame, therefore the characteristic colour of the cation present in the salt, remains interference-free.

ANALYSIS OF GROUP VI

Collect the filtrate of Vth group and reduce its volume. Add NH_4Cl and NH_4OH and disodium hydrogen phosphate (Na_2HPO_4) solution. A white precipitate of Magnesium Ammonium Phosphate indicates the presence of VIth gp and of Magnesium (Mg^{2+}) ions. Sometimes the ppt. appears only on boiling and the scratching the side of test tube with a glass rod.

Chemistry of Magnesium (Mg^{2+}) Ions

When Na_2HPO_4 is added to Magnesium Chloride solution containing sufficient amount of NH_4OH solution, a white ppt. of Magnesium Ammonium Phosphate is formed.



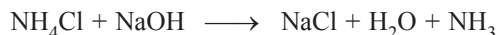
Detection of Magnesium (Mg^{2+}) Ions

To the filtrate of Vth group add NH_4Cl and NH_4OH solution followed by di-sodium hydrogen phosphate solution. Boil the solution and scratch the inner sides of test tube if required \longrightarrow A fine crystalline white ppt. of Magnesium Ammonium Phosphate confirms Magnesium (Mg^{2+}) ions and VI group.

ZERO GROUP

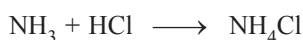
This group contains Ammonium (NH_4^+) ions which can be identified off the track of formal group analysis and therefore original solution for its identification is not required.

Place a small amount of given salt in a clean test tube and add to it NaOH solution. Heat the contents gently. Characteristic smell of Ammonia indicates the presence of zero group.



Identification of Ammonium (NH_4^+) Ions

1. Bring a moist Red Litmus strip in contact with the evolved gas \longrightarrow It turns blue.
2. Bring a glass rod moistened with conc. HCl in contact with the evolved gas \longrightarrow dense white fumes.



3. Pass the evolved gas in Nessler's Reagent \longrightarrow a brown ppt. is formed.



Nessler's Reagent



Iodine of Millon's base (brown)

ANALYSIS OF ACID RADICALS

It is not possible to divide the acid radicals or anions into systematic groups, as was done in the case of cations. However, for the sake of simplicity these may be divided into three groups.

1. **dilute acid group.** It includes carbonate (CO_3^{2-}), sulphite (SO_3^{2-}). Sulphide (S^{2-}), Nitrite (NO_2^-).
2. **Concentrated acid group.** It includes chloride (Cl^-), Bromide (Br^-), Iodide (I^-) nitrate (NO_3^-) and acetate (CH_3COO^-)
3. **Special group.** It contains sulphate (SO_4^{2-}).

Chemistry of Acid Radicals

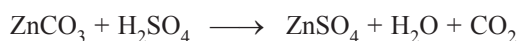
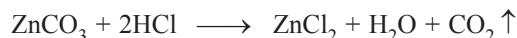
The confirmatory tests for anions (acid radicals) are done by treating the dry mixture with dil. H_2SO_4 or dil. HCl and then conc. H_2SO_4 . Moreover, the test of acid radical such as sulphate is done by a special method.

Chemistry of Dilute Acid Group

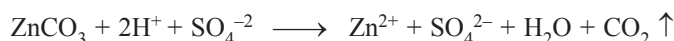
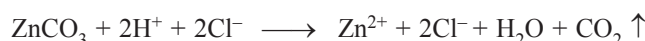
The theory of test for acid radicals is that when the given substance is treated with dil. H_2SO_4 or dil. HCl , it gives out a particular gas, for which a test can be performed to identify the corresponding anion.

Chemistry of Individual Radicals

(a) Carbonate (CO_3^{2-}). When dil. HCl or dil H_2SO_4 is added to a carbonate brisk effervescence takes place with the evolution of CO_2 which is odourless.

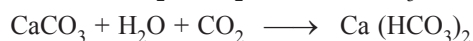
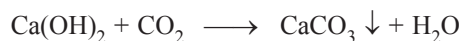


Ionically,

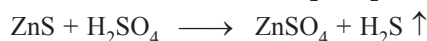
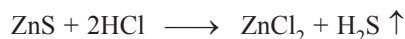


Confirmatory Test for Carbon-dioxide

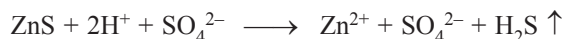
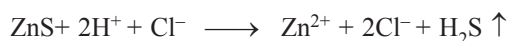
When CO_2 is passed through lime water, it turns turbid white due to the formation of carbonate. The white turbidity dissolves when excess CO_2 is passed through lime water, forming soluble calcium bi carbonate.



(b) Sulphide (S^{2-}). When dil. HCl or H_2SO_4 is added to a sulphide, a characteristic-rotten egg smelling gas, H_2S evolves.

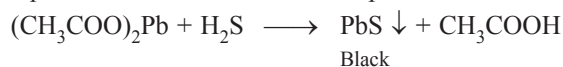


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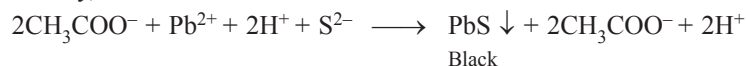


Confirmatory Test for H_2S gas

When H_2S gas reacts with a piece of filter paper moistened with lead acetate solution, a shining black precipitate is formed due to lead sulphide formation.

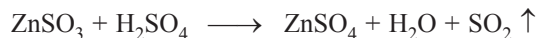
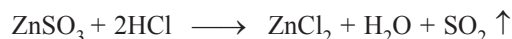


Ionically,

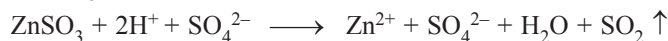
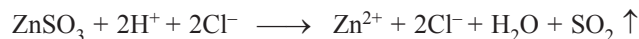


(c) Sulphite (SO_3^{2-}) :

When dil. HCl or H_2SO_4 is added to a sulphite a characteristic burning sulphur smelling sulphur dioxide gas evolves.

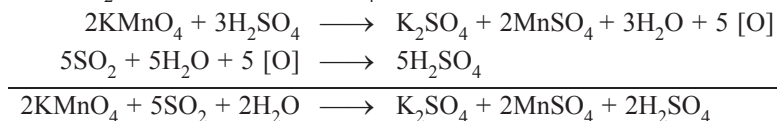


Ionically,



Confirmatory Test for Sulphur dioxide gas

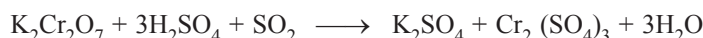
1. When SO_2 is passed in dil. KMnO_4 solution the pink colour of the latter is decolourised.



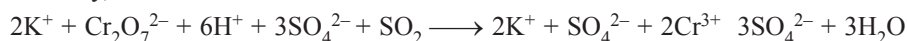
Ionically,



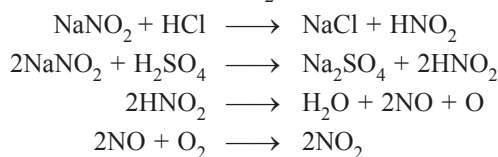
2. When a piece of filter paper moistened with $\text{K}_2\text{Cr}_2\text{O}_7$ solution is brought in contact with SO_2 gas, the orange colour of $\text{K}_2\text{Cr}_2\text{O}_7$ solution turns green, due to the formation of chromium (Cr^{3+}) ions.



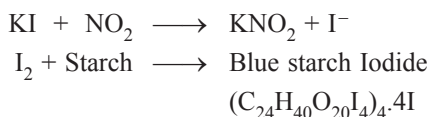
Ionically,



(d) Nitrite: When dil. HCl or H_2SO_4 is added in sodium nitrite, a colourless gas NO evolves at room temp. which readily oxidises to NO_2 and is observed as yellowish brown gas.

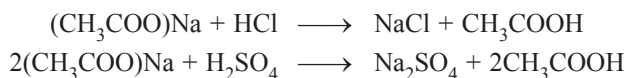


The evolved gas turns the paper dipped in starch iodide solution blue black.

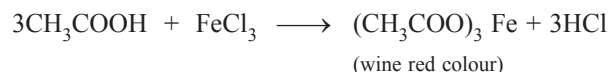


(e) Acetate (CH_3COO^-) :

When dil. HCl or H_2SO_4 is added in sodium acetate solution and the contents heated, acetic acid is formed, which can be detected by its characteristic vinegar odour.



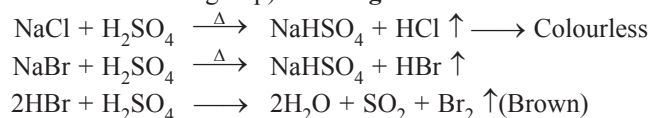
1. The presence of acetic acid can be confirmed by adding neutral ferric chloride solution into the above solution.

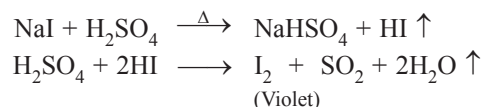


2. Mixture + conc. H_2SO_4 + Ethanol. Heat the contents and pour-out in a beaker containing water. Fruity odour confirms acetate ion.

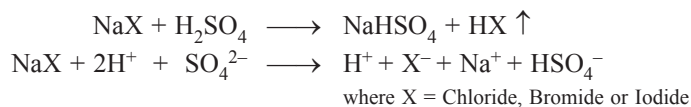
Concentrated Acid Group

Halides- They include chloride, bromide, iodide and nitrate. When conc. H_2SO_4 is added to the given substance containing halide ion, gases with characteristic colour and odour are evolved. (Conc. HCl is not used in the test of this group). **Heating is a must in this test.**





Ionically,

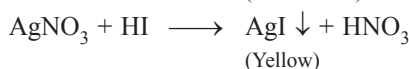
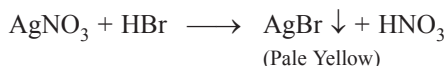
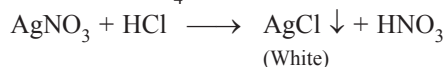


Preparation of Sodium Carbonate Extract :

Mix the given substance with solid sodium carbonate in the ratio 1 : 3 approximately and add about 15 cc of distilled water to it. Boil it for some time. Cool and filter. The filtrate is called sodium carbonate extract.

Neutralise soda extract with dil. HNO_3 . Add to it AgNO_3 solution and observe the colour of precipitate.

- (i) A white ppt. soluble in $\text{NH}_4\text{OH} \longrightarrow$ Chloride
- (ii) Pale yellow ppt. sparingly soluble in $\text{NH}_4\text{OH} \longrightarrow$ Bromide
- (iii) Yellow ppt. insoluble in $\text{NH}_4\text{OH} \longrightarrow$ Iodide



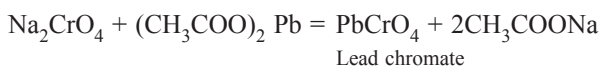
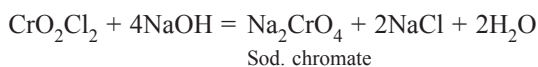
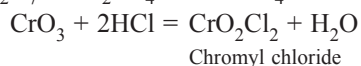
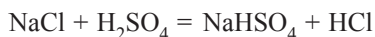
Chromyl Chloride Test for Chloride Ion

This is a very sensitive test for chloride ions.

The given chloride salt is heated with conc. sulphuric acid in presence of some crystals of potassium dichromate when deep red vapours of chromyl chloride are evolved. These vapours are passed through caustic soda soln. the solution turns yellow due to the formation of sodium chromate.

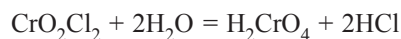
The yellow solution is neutralised with acetic acid and some lead acetate soln. is added, a yellow precipitate is observed due to the formation of lead chromate. This confirms the presence of chloride.

The reactions are given below.



Note (i) This test is not given by bromide and iodide ions.

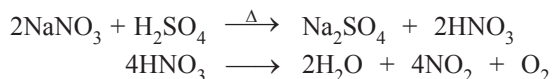
(ii) The heating of salt with $\text{K}_2\text{Cr}_2\text{O}_7$ and H_2SO_4 should be done in a dry test tube otherwise the chromyl chloride vapours will be hydrolysed in the test tube



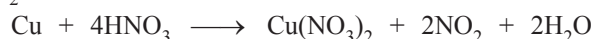
(iii) The test is said to be positive when all the three observation (red vapours, yellow soln and yellow ppt.) are correct.

Nitrate (NO_3^-)

When conc. H_2SO_4 is added to a nitrate, light brown fumes are evolved, which darken on heating.



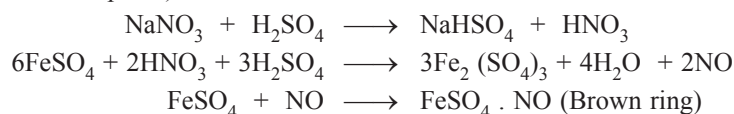
When copper turnings are added to the contents of test tube, dark brown fumes are evolved due to formation of NO_2 .



Note : Better results are obtained if paper pallet is used in place of Cu turnings.

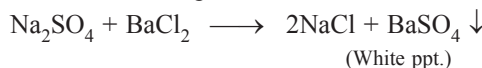
Ring Test

When to an aqueous solution of sodium nitrate, a freshly prepared saturated aqueous solution of ferrous sulphate is added and then conc. H_2SO_4 is poured by the side of the test tube, a brown ring is formed at the junction of the two solutions, due to the formation of $\text{FeSO}_4 \cdot \text{NO}$. (Nitrosoferrous sulphate)



Special Group Test

1. Sulphate (SO_4^{2-}). When to a solution of sodium sulphate a solution of barium chloride is added a white precipitate of barium sulphate is formed which is insoluble in mineral acids.



Ionically,



2. To the 2 ml of sodium carbonate extract neutralised with dil HCl, when barium chloride solution is added, a **white** ppt. is formed which is insoluble in mineral acids.

Identification of Oxalate Ion :

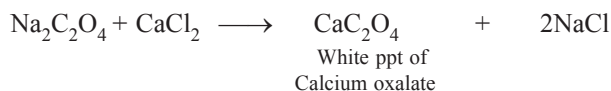
Oxalate ion when treated with conc. sulphuric acid and heated, carbon monoxide and carbon dioxide are formed. The carbon dioxide turns moist blue litmus strip red while carbon monoxide burns with a blue flame.

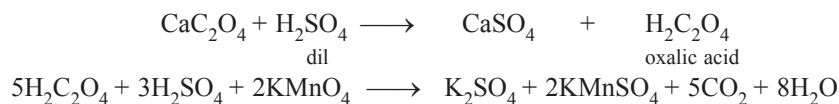
Tests for oxalate Ion ($\text{C}_2\text{O}_4^{2-}$)

1. Take five ml of sodium carbonate extract and neutralise it with dil. hydrochloric acid. Now add 2 ml of calcium chloride solution. A thick white precipitate confirms oxalate ion if this precipitate remains insoluble in acetic acid.

2. Take the white precipitate of this calcium oxalate obtained in the above test and add to it dil. H_2SO_4 . Now add a few drops of KMnO_4 and warm gently. The pink colour of KMnO_4 is discharged.

Chemical Reactions Involved in the Test of Oxalate Ion.





SOLVED EXERCISES BASED ON MIXTURE ANALYSIS

Experiment No. 1

Analyse qualitatively the given substance labelled A-12 for two cations and two anions.

Identify these ions. In testing for anions describe the colour, odour etc. of the gas/gases evolved and give confirmatory tests for them.

In testing for cations you must describe.

1. How the original solution for group analysis was formed.
2. The formal group analysis mentioning group reagent.
3. Give confirmatory test for each cations.

Identification of anions

S.No.	Experiment	Observations	Inference
1.	To a pinch of substance A-12 in a test tube add 2 ml of dil. HCl.	Brisk effervescence with evolution of colourless, odourless gas	CO_3^{2-} suspected
2.	Bring a moist blue litmus paper near the gas.	It turns red	
3.	Pass the evolved gas into lime water soln.	Milky turbidity	CO_3^{2-} confirmed
4.	Take another pinch of the given substance A-12 in a clean test tube and add 2 ml of conc. H_2SO_4 to it. Heat the test tube.	A colourless gas with pungent odour evolves	
5.	Bring a moist blue litmus paper near the gas.	It turns red.	Cl^- suspected
6.	Bring a glass rod moistened with NH_4OH near the gas.	Dense white fumes formed.	Cl^- suspected
7.	Pass the gas into silver Nitrate solution.	A curdy white ppt observed.	Cl^- confirmed
8.	Add NH_4OH soln to the above white ppt.	The ppt dissolves readily	

Identification of Cations

Preparation of original soln: The original solution is prepared in dil. HCl

Since the substance is soluble in dil HCl, it indicates that the first group is absent.

1.	Pass H_2S gas to a portion of the soln	No precipitate observed	II gp absent
2.	Reject the above solution and take an other portion of the original soln. Boil it with 1 ml of conc. HNO_3 and cool. To this add a small pinch of NH_4Cl followed by excess NH_4OH	No ppt. observed	III gp absent

3.	Pass H_2S gas to the above solution	No ppt. Observed	IV gp absent
4.	Reject the above solution and take an other portion of the original soln. To this add NH_4Cl followed by excess of NH_4OH and little of $(\text{NH}_4)_2\text{CO}_3$ soln.	White ppt observed	V gp present Ca^{2+} suspected
5.	Filter it, Retain the filtrate for the next group and use the precipitate for fifth group. Dissolve the precipitate in minimum quantity of hot dil. acetic acid and add ammonium oxalate soln	White ppt formed	Ca^{2+} confirmed
6.	Take the filtrate of the Vth gp. To this add little NH_4OH and di sodium hydrogen phosphate solution and scratch sides of the test tube with a glass rod	White ppt. formed	Mg^{2+} confirmed

Result – Substance A – 12 contains.

Cations – Calcium Ca^{2+}
 Magnesium Mg^{2+}
 Anions Carbonate CO_3^{2-}
 Chloride Cl^-

Experiment No. 2

Analyse qualitatively the given substance labelled A-14 for two cations and two anions.

Identify all the four ions

In testing for anions describe the colour and odour of the gas/gases evolved and any changes observed which lead to the name of the ions. Give confirmatory tests for the ions.

In testing for cations, describe

1. How the original soln for group analysis is formed.
2. The formal group analysis mentioning group reagent.
3. Give confirmatory tests for the cations.

Identification of Anions

S.No.	Experiment	Observations	Inference
1.	To a pinch of the substance A-14 add conc. sulphuric acid	Light brown coloured fumes with pungent odour	NO_3^- suspected NO_3^- confirmed
2.	Heat the test tube	The fumes darken	
3.	Add copper turnings to the contents of the test tube	brown fumes intensify	
4.	Ring test for NO_3^- confirmation : Prepare an aqueous soln of the substance. To this add equal volume of freshly prepared soln of ferrous sulphate. Now allow a few drops of conc. H_2SO_4 to flow along the sides of the test tube.	brown ring observed at the junction of two liquids.	

5.	Neutralise a portion of sodium carbonate extract with dil. HCl and add BaCl_2 solution.	White ppt.	SO_4^{2-} suspected
6.	To the above ppt. add little of dil HCl	The precipitate is insoluble	SO_4^{2-} confirmed

Identification of Cations

Preparation of O.S. \Rightarrow O.S. is prepared by dissolving the mixture in water.

1.	To a pinch of substance A-14 add 2 ml caustic soda solution and heat.	A colourless gas with pungent odour evolves	Zero group is suspected
2.	Bring a moist red litmus paper near the mouth of the test tube	It turns blue	Zero group is suspected
3.	Bring a glass rod moistened with conc. hydrochloric acid	Dense white fumes observed	NH_4^+ confirmed
4.	Pass the evolved gas in to Nessler's reagent	It turns brown	NH_4^+ further confirmed
5.	To a portion of the original soln add dil. HCl	No precipitate formed	Ist gp is absent
6.	Pass H_2S gas in to it	Black ppt. formed	II gp present
7.	Filter the contents. Dissolve the residue in minimum quantity of dil. HCl. Heat and divide the soln into two parts (i) To first part add excess NH_4OH (ii) To the second part add. Potassium Ferrocyanide solution.	Deep blue solution Chocolate brown ppt formed	Cu^{2+} confirmed Cu^{2+} confirmed

Result – The substance A-14 contains

Cations 1. Ammonium (NH_4^+)

2. Copper (Cu^{2+})

Anions 1. Nitrate (NO_3^-)

2. Sulphate (SO_4^{2-})

Experiment No. 3

Analyse qualitatively the substance A-16 for two cations and two anions. Identify all the four ions.

In testing for anions describe the colour and odour of the gas or gases evolved, state all the changes you observe. Give the confirmatory tests.

In testing for cations describe

(i) How the original solution is prepared ?

(ii) The formal group analysis with pertinent group reagents.

(iii) The confirmatory tests for cations.

Identification of Anions

S.No.	Experiment	Observation	Inference
1.	To a pinch of the substance A-16 add dil. HCl	A colourless characteristic rotten egg smelling gas evolved	S^{2-} suspected
2.	Bring a piece of filter paper moistened with lead acetate soln in contact with the evolved gas.	Shining black precipitate formed	S^{2-} confirmed
3.	To a pinch of the substance A-16 add conc. H_2SO_4 and heat.	Colourless gas with pungent odour evolves, the whole liquid turns brown.	Br^- suspected
4.	Pass the gas into silver nitrate soln	A curdy yellowish white ppt. formed	Br^- confirmed
5.	Add excess NH_4OH to the precipitate.	The precipitate dissolves with difficulty	
6.	Prepare sodium carbonate extract. Neutralise it with dil HNO_3 . Add silver nitrate to it.	Pale yellow precipitate formed	
7.	Add excess NH_4OH to it.	The precipitate dissolved	Br^- confirmed

Identification of Cations

Preparation of original soln: The solution is prepared in dil. HCl

Since original solution is prepared in dil. HCl, it indicates that the first group is absent: So we proceed further to the second group.

S.No.	Experiment	Observation	Inference
1.	To a portion of the soln pass H_2S .	No ppt formed	II gp absent
2.	Reject the above solution. Take fresh portion of the original soln. Add a pinch of NH_4Cl followed by excess NH_4OH .	Gelatinous white ppt.	III gp suspected
3.	Filter it. Retain filtrate for the next group. Use precipitate for this group. Dissolve the precipitate in dil HCl and add caustic soda soln. drop by drop.	A white precipitate appears which dissolves in excess caustic soda solution.	Al^{3+} suspected
4.	To the above solution add solid NH_4Cl and heat.	A white precipitate reappears	Al^{3+} confirmed
5.	To a portion of the filtrate retained previously, add little more of NH_4Cl (solid), excess NH_4OH and pass H_2S gas into it.	A white ppt appears	IV gp suspected
6.	Filter it and collect precipitate for this group. Dissolve the precipitate in dil. HCl, boil and cool add caustic soda soln drop by drop.	A white ppt. appears which dissolves in excess caustic soda soln	Zn^{2+} suspected

7.	To the other portion of solution add potassium ferrocyanide solution.	Bluish white ppt appears	Zn^{2+} confirmed
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Results—The given substance A-16 contains

Cations	Aluminium	(Al^{3+})
	Zinc	(Zn^{2+})
Anions	Sulphide	(S^{2-})
	Bromide	(Br^-)

Experiment No. 4

Identify two anions and two cations present in the given substance labelled A-18.

It is necessary to carry out formal group analysis and state how original soln. was prepared.

While reporting anions distinct colour and odour of evolved gases should be mentioned. Use of sodium carbonate extract shall be appreciated.

Identification of Anions

S.No.	Experiment	Observation	Inference
1.	Treat a pinch of the substance A-18 with conc. H_2SO_4 and warm	Colourless vapours with strong Vinegar odour evolved	CH_3COO^- confirmed
2.	Sodium carbonate extract neutralised with dil HCl is mixed with neutral ferric chloride soln	Wine red colour	
3.	The mixture is treated with conc. H_2SO_4 and Ethyl Alcohol and heated	Fruity odour is felt	CH_3COO^- confirmed
4.	To a pinch of the substance add conc. H_2SO_4 and warm	Light brown fumes with pungent odour	NO_3^- suspected
5.	Heat the contents with some copper turnings	The fumes become dark brown	
6.	To an aqueous soln of the substance, add freshly prepared aqueous soln of ferrous sulphate and then add a few drops of conc H_2SO_4 gently (By allowing them to flow along the sides of test tube.)	A brown ring observed at the junction of the two liquids	NO_3^- confirmed

Identification of Cations

The original solution is prepared in water and more readily in dil HNO_3 .

1.	To a portion of original soln add dil HCl	White ppt.	Ist gp present
2.	Filter the contents. Retain filtrate for next groups and residue for first group. Dissolve the residue in hot water and divide the solution into three parts		
3.	To the first part add potassium iodide soln	Canary yellow ppt	Pb^{2+} confirmed
4.	To the second portion add potassium chromate soln	Mustard yellow ppt	Pb^{2+} confirmed

5.	To the third part add dil H_2SO_4	White ppt	Pb^{2+} confirmed
6.	Use the filtrate retained previously. To a portion of it add dil HCl and pass H_2S gas.	No ppt	II gp absent
7.	Reject the above contents and take another portion of the filtrate. Add solid NH_4Cl and excess NH_4OH .	No change	III gp absent
8.	Pass H_2S into above contents	White ppt.	IV gp suspected
9.	Filter the contents and dissolve the ppt in dil HCl. Boil the contents Divide into two parts. To first part Add caustic soda soln drop by drop.	A white ppt appears which dissolves in excess Caustic soda soln.	Zn^{2+} suspected
10.	To the above pass H_2S gas	White ppt reappears	Zn^{2+} confirmed

Result : The given substance A – 18 contains

Cations	Lead	(Pb^{2+})
	Zinc	(Zn^{2+})
Anions	Acetate	(CH_3COO^-)
	Nitrate	(NO_3^-)

Experiment No. 5

Object : You are provided with a substance labelled A-20. Identify two anions and two cations present in it.

In testing for anions describe the colour and odour of the gas/gases evolved and other changes observed which help in the detection of ions. Also describe, how sodium carbonate extract was prepared. Give confirmatory tests for the ions.

In testing for cations, describe

1. the preparation of original soln. for group analysis.
2. the formal group analysis stating the group reagent.
3. give confirmatory tests for the ions identified.

Identification of Anions

Preparation of sodium carbonate extract : Mix a portion of the substance with solid sodium carbonate in the ratio 1 : 3 (approximately) and add about 15 ml distilled water in it. Boil for some time and filter. The filtrate is sodium carbonate extract. The residue is retained for original soln.

S.No.	Experiment	Observation	Inference
1.	To a pinch of substance A-20 in a clean test tube add 2 ml of dil. HCl	Colour less gas evolves which readily changes to yellowish brown colour	NO_2^- suspected
2.	Hold a starch iodide paper near the mouth of the test tube	It turns blue black	NO_2^- confirmed
3.	Take a pinch of A-20 in a clean test tube and add to it 2 ml of conc. H_2SO_4 . Heat the contents.	A colourless gas bearing a pungent odour evolves.	

4.	Bring a moist blue litmus paper near the gas	It turns red.	Cl^- suspected
5.	Bring a glass rod moistened with NH_4OH near the mouth of the test tube	Dense white fumes are formed	Cl^- suspected
6.	Take nearly 5 ml of Sodium carbonate extract. Neutralise it with dil HNO_3 and add 2 ml of AgNO_3 soln.	A curdy white precipitate is formed.	
7.	To the contents add ammonium hydroxide soln.	The curdy white ppt. readily dissolves	Cl^- confirmed

Preparation of Original Soln. The original soln. is prepared by dissolving the residue of sodium carbonate extract in dil. HCl .

Identification of Cations

S.No.	Experiment	Observation	Inference
1.	To a portion of O.S. add dil. HCl	No change	I group absent
2.	Pass H_2S gas to the above test-tube	No ppt.	II group absent
3.	Take a fresh portion of O.S. Boil it with 2 ml of conc. HNO_3 . Cool it add a pinch of NH_4Cl followed by excess NH_4OH .	Gelatinous white ppt. is formed.	III group suspected
4.	Filter the contents, retains the filtrate for the next group and precipitate for this group.		
5.	Dissolve the white ppt. in dil. HCl and add caustic soda soln. drop by drop.	The white ppt. dissolves in excess caustic soda soln.	
6.	To the above soln. add solid NH_4Cl and heat	A white ppt. reappears	Al^{3+} confirmed
7.	The filtrate retained for the next group above is taken for the next cation.		
	To a portion of it add a pinch of NH_4Cl and excess NH_4OH and pass H_2S gas	No change	IV gp. absent
8.	Reject the above soln. and take a portion of the above retained filtrate.		
	To this add NH_4Cl and excess NH_4OH . Now add a little of $(\text{NH}_4)_2\text{CO}_3$ soln.	White ppt. observed	V group present Ca^{2+} suspected
9.	Filter it. Reject the filtrate. Dissolve the ppt. in minimum quantity of hot dil. CH_3COOH and add amm. oxalate soln.	White ppt.	Ca^{2+} confirmed

Result : The given substance A-20 contains

Cations	Al^{3+}
	Ca^{2+}
Anions	NO_2^-
	Cl^-

Experiment No. 6

Identify two cations and two anions present in the given substance labelled A-22.

Carry out the formal group analysis to identify the ions. Give confirmatory tests for them. You must describe the preparation of sodium carbonate extract for anions and original soln. for cations. Dry tests shall not be accepted as confirmatory tests.

Identification of Anions

Preparation of Sodium Carbonate extract : To a pinch of substance A-22 in a test tube add sodium carbonate (solid) in the ratio 1 : 3 nearly. Add nearly 15 ml of distilled water. Boil, cool and filter the contents. The filtrate is the required extract. Residue is used for O.S. preparation.

S.No.	Experiment	Observation	Inference
1.	Take a pinch of A-22 in a clean test tube. Add to it 2 ml of dil. H_2SO_4 .	Brisk effervescence with evolution of colourless and odourless gas.	CO_3^{2-} suspected
2.	Pass the evolved gas into lime water	It turns milky.	CO_3^{2-} suspected
3.	Add little of conc. H_2SO_4 to a pinch of A-22 in a clean test tube and heat.	Vinegar odour is felt. It becomes stronger on heating.	CH_3COO^- suspected
4.	To a small portion of an aqueous soln. of A-22 add neutral ferric chloride soln.	Wine-red colour	CH_3COO^-
5.	To the mixture A-22 add ethyl alcohol followed by conc. H_2SO_4 . Heat and pour the contents in a beaker containing water	Fruity odour is felt	CH_3COO^- confirmed

Identification of Cations

Preparation of Original Soln. : Original soln. is prepared in dil. HCl by dissolving the residue of sodium carbonate extract.

S.No.	Experiment	Observation	Inference
1.	Add dil HCl to little of O.S.	No change	Gp I absent
2.	Pass H_2S gas into the above soln.	No ppt.	Gp II absent
3.	Boil off H_2S . Cool the solution. Or Take a fresh portion of O.S. – Add to it 1 ml of conc. HNO_3 Boil it for some time then cool it. To this add a pinch of solid NH_4Cl followed by excess NH_4OH	No change	Gp III absent
4.	Pass H_2S gas to the above soln.	No change	Gp IV absent

5.	Boil off H_2S . Cool and add little of solid NH_4Cl and excess NH_4OH . To this add excess $(\text{NH}_4)_2\text{CO}_3$ soln.	White ppt. observed	Gp V present
6.	Filter the contents of the test tube. Retain the filtrate for next group. Dissolve the residue in hot and dil CH_3COOH and add amm. oxalate soln.	White ppt.	Ca^{2+} confirmed
7.	Boil the filtrate of gp V to reduce its volume. Cool and add excess NH_4OH followed by little of di-sodium hydrogen phosphate soln. use a glass rod to scratch walls of the test tube.	fine crystalline white ppt.	gp VI present Mg^{2+} confirmed

Result : The given substance A-22 contains

Anions	CO_3^{2-}
	CH_3COO^-
Cations	Ca^{2+}
	Mg^{2+}

Experiment No. 7

You are provided with a substance labelled A-24. Analyse it for two cations and two anions.

In testing for anions describe the colour and odour of the gas/gases evolved. Describe the preparation of sodium carbonate extract required for the test of anions.

In case of cations describe the preparation of original soln. Describe the formal group analysis. Give confirmatory tests for all ions, you have identified.

Identificatin of Anions

Preparation of Sodium Carbonate Extract : To a pinch of A-24 add solid sodium carbonate in the ratio 1 : 3. Add nearly 15 ml of distilled water boil, cool and filter. The filtrate is the required extract. Dissolve the residue in dil HCl. This is the original soln. for cations.

S.No.	Experiment	Observation	Inference
1.	To a pinch of the substance A-24 in a clean test tube add little dil HCl	A colour less gas evolved which has the characteristic rotten eggs odour	S^{2-} suspected
2.	Dip a piece of filter paper in lead acetate soln and bring it in contact with the gas.	A shining black ppt collects on the piece of paper.	S^{2-} confirmed
3.	To a pinch of A-24 in a clean test tube add little conc. H_2SO_4 and heat.	A colourless gas evolves which has a pungent odour.	
4.	Bring a moist blue litmus paper near the gas	It turns red.	Cl^- suspected
5.	Bring a glass rod moistened with NH_4OH near the gas.	Dense white fumes formed.	Cl^- suspected

6.	Neutralise a portion of sodium carbonate extract with dil HNO_3 and add AgNO_3 soln.	A curdy white ppt. formed.	
7.	Add NH_4OH to the contents of the above test tube.	The white ppt. dissolves readily.	Cl^- confirmed

Identification of Cations

Since the original solution is formed in dil hydrochloric acid, I group is absent.

S.No.	Experiment	Observation	Inference
1.	To a portion of O.S. pass H_2S gas	No change	Gp II is absent
2.	Take a fresh portion of O.S. add to it a pinch of solid $\text{NH}_4\text{Cl} + \text{NH}_4\text{OH}$ excess	A dirty green ppt.	
3.	To an other portion of the above soln. add few drops of potassium ferricyanide soln.	A deep blue coloured soln.	Fe^{2+} confirmed
4.	Boil a portion of the O.S. with some conc. HNO_3 . Cool and add solid NH_4Cl followed by excess NH_4OH	Reddish brown ppt.	
5.	Filter it. Retain the filtrate for the next group. Dissolve the residue in dil. HCl .		
6.	To a portion of this soln. add potassium ferrocyanide.	Blue colour	Fe^{2+} is oxidised to Fe^{3+} and is confirmed
7.	To an other portion of the soln. add little potassium thiocyanate soln.	An intense blood red colour	
8.	Take a portion of the filtrate which was retained for the next group. To this add little of solid NH_4Cl , excess NH_4OH and pass H_2S gas	White ppt.	Gp IV is present
9.	Filter the contents of the test tube and dissolve the white ppt. in dil HCl . Boil, cool and divide it into three parts.	A flocculent gelatinous white ppt forms	
10.	To first part add NaOH soln. drop by drop and then in excess	A white ppt forms. The ppt. dissolves in excess NaOH	Zn^{2+} confirmed
11.	Add little $\text{K}_4[\text{Fe}(\text{CN})_6]$ to the second part.	A bluish white ppt.	
	Pass H_2S gas into the third part	White ppt.	Zn^{2+} confirmed

Result : The give substance A-24 contains

Anions	S^{2-}
	Cl^-
Cations	Fe^{3+}
	Zn^{2+}

Experiment No. 8

You are given a substance labelled A-26. Identify two cations and two anions in it. While testing for anions describe the colour and odour of the gas/gases evolved. In testing for cations you must describe how the original soln. was prepared.

Give formal group analysis with the group reagent.

Give confirmatory tests for the four ions you identify.

Identification of Anions

Preparation of Sodium Carbonate extract : Mix a pinch of the given substance with solid sodium carbonate in the ratio 1 : 3. To this add about 15 ml of distilled water. Boil, cool and filter. The filtrate is the required extract.

S.No.	Experiment	Observation	Inference
1.	To a pinch of the substance in a test tube add conc. H_2SO_4 and heat	A colourless gas with pungent odour evolves	Cl^- suspected
2.	Bring a moist blue litmus paper near the gas.	It turns red	
3.	Bring a glass rod moistened with NH_4OH near the gas.	Dense white fumes.	
4.	To a small portion of the extract in a test tube add dil HNO_3 to neutralise it and add AgNO_3 soln.	Curdy white ppt.	Cl^- confirmed
5.	Add NH_4OH to the above white ppt.	The ppt. readily dissolves.	
6.	To another portion of the extract in a test tube add dil HCl to neutralise it. Now add BaCl_2 soln.	A white ppt. obtained	
7.	Add dil HCl to the above white ppt.	It remains insoluble	SO_4^{2-} confirmed

Identification of Cations

Preparation of original soln.: The O.S. is prepared in distilled water.

S.No.	Experiment	Observation	Inference
1.	To a pinch of the substance in a clean test tube add caustic soda soln. and heat	A colourless gas with a strong pungent characteristic odour	NH_4^+ suspected
2.	Bring a moist rod litmus paper near the gas	It turns blue	
3.	A glass rod dipped in conc. HCl is brought near the gas	Dense white fumes observed	
4.	Pass the evolved gas into Nessler's reagent	It turns brown	NH_4^+ confirmed
5.	Add dil HCl to a portion of O.S.	No change	Gp I is absent
6.	Pass H_2S gas into it	No change	Gp II absent
7.	Reject the above soln. To a fresh portion of O.S. add conc. HNO_3 and boil. Cool it and add solid NH_4Cl followed by excess NH_4OH	Gelatinous white ppt.	Gp III suspected

8.	Filter it. Dissolve the ppt. in dil HCl and add NaOH soln. drop by drop	A white ppt. appears which dissolves in excess NaOH	Al^{3+} suspected
9.	To the above soln. add solid NH_4Cl and heat	The white ppt. reappears	Al^{3+} confirmed

Result : The given substance A-26 contains

Anions	Cl^- SO_4^{2-}
Cations	NH_4^+ Al^{3+}

Experiment No. 9

Analyse qualitatively the given substance A-28 for two cations and a common anion. Identify all the three ions.

In testing for anion, describe the colour and odour of the gas evolved. In testing for cations you must describe the preparation of the original solution and the formal group analysis with the pertinent group reagent. Give confirmatory tests for all the ions.

Identification of Anion

S.No.	Experiment	Observation	Inference
1.	To a pinch of the given substance in a clean test tube add dil HCl and heat	A colourless gas with strong odour of burning sulphur	SO_3^{2-} suspected SO_3^{2-} confirmed SO_3^{2-} confirmed
2.	Bring a moist blue litmus paper near the gas	The paper turns red and is finally bleached	
3.	Pass the gas into acidified potassium permanganate soln.	The pink colour is decolourised	
4.	Dip a piece of filter paper in acidified $\text{K}_2\text{Cr}_2\text{O}_7$ soln. and bring it in contact with the gas.	The orange colour of paper turns green	

Identification of Cations

Preparation of original solution : The original soln. is prepared in dil HCl. Since soln is prepared in dil HCl, gp I is absent.

S.No.	Experiment	Observation	Inference
1.	To a portion of O.S. pass H_2S gas	No change	Gp II is absent
2.	Boil off H_2S completely from the above soln.		
3.	Add 2 ml of conc. HNO_3 and boil. Cool it and add a little solid NH_4Cl followed by excess NH_4OH	No change	Gp III is absent
4.	To the above soln. pass H_2S Filter the above contents. Collect the ppt. for this group and retain filtrate for the next group.	White ppt.	Gp IV suspected
5.	Dissolve the ppt. in dil HCl. Boil and cool. To a part of this add caustic soda soln. drop by drop.	White ppt. appears which dissolves in excess NaOH soln.	Zn^{2+} confirmed

6.	To the other portion of the above soln. add potassium ferrocyanide soln.	Bluish white ppt. appears	Zn ²⁺ confirmed
7.	To the remaining portion of the soln. pass H ₂ S gas	White ppt	Zn ²⁺ confirmed
8.	Take the filtrate of gp IV retained above for the detection of next ion.		
9.	Heat it to expel H ₂ S gas as well as to reduce its volume.		
10.	Add solid NH ₄ Cl and excess NH ₄ OH to it followed by (NH ₄) ₂ CO ₃ soln.	White ppt	Gp V suspected
11.	Filter the contents. Dissolve the ppt in minimum quantity of hot dil. acetic acid and add Amm. Oxalate soln.	White ppt	Ca ²⁺ confirmed

Result : The given substance A-28 contains

Anion	SO ₃ ²⁻
Cations	Zn ²⁺
	Ca ²⁺

Experiment No. 10

Identify two cations and two anions present in the given substance labelled A-30.

While testing for anions, describe the preparation of sodium carbonate extract. Also mention the colour and odour of gas/gases evolved.

While testing for cations state how the original soln. was prepared and the formal group analysis with pertinent group reagent.

Give confirmatory tests for all ions.

Identification of Anions

Preparation of Sodium Carbonate extract : Mix a portion of the given substance with solid sodium carbonate in the ratio 1 : 3 nearly in a clean test tube. To this add nearly 15 ml of distilled water. Boil it for some time. Cool and filter the contents. The filtrate is sodium carbonate extract.

S.No.	Experiment	Observation	Inference
1.	To a pinch of substance A-30 add conc. H ₂ SO ₄ and heat	Colourless gas evolves with a pungent odour. The whole liquid turns brown.	Br ⁻ suspected
2.	Neutralise a portion of sodium carbonate extract with dil HNO ₃ . Add silver nitrate soln. to it.	Pale yellow precipitate	
3.	Add excess NH ₄ OH to the above ppt.	Sparingly soluble	Br ⁻ confirmed
4.	Neutralise the remaining portion of extract with dil HCl and add BaCl ₂ soln.	White ppt.	SO ₄ ²⁻ suspected
5.	To the above add dil HCl	The ppt. does not dissolve	SO ₄ ²⁻ confirmed

Identification of Cations

Preparation of original soln. : The solution is prepared in dil HCl. Also it shows that first group is absent, therefore the analysis will start from second group.

S.No.	Experiment	Observation	Inference
1.	To a portion of original soln mixed with dil HCl, pass H_2S gas	Black ppt.	Gp II suspected
2.	Filter the solution. Retain the filtrate for the next group. Take the ppt. Dissolve it in dil HNO_3 and divide in three parts		
3.	To the first part add NaOH soln. in excess	A pale blue precipitate	
4.	Heat the contents of the test tube	Black ppt.	Cu^{2+}
5.	To the second part of the soln. add NH_4OH drop by drop till in excess.	Bluish white ppt. which gives a deep blue coloured soln when in excess	Cu^{2+} confirmed
6.	To the third portion of the soln. add potassium ferrocyanide soln.	Chocolate brown precipitate forms.	Cu^{2+} confirmed
7.	Take the filtrate of gp II (which was retained for further analysis.) Boil it to expel all H_2S .		
8.	To a portion of it add 2 ml conc. HNO_3 and boil. Cool and add solid NH_4Cl followed by excess NH_4OH	No change	Gp III is absent
9.	To the above soln. pass H_2S gas	White ppt.	Gp IV suspected
10.	Filter the contents. Take the residue and dissolve it in dil HCl. Boil and cool.		
11.	To a portion of it add NaOH soln. drop by drop till in excess	A white ppt. which dissolves in excess NaOH	Zn^{2+} confirmed
	To the second portion of the soln. add potassium ferrocyanide soln.	Bluish white ppt.	Zn^{2+} confirmed
	To the remaining portion of soln. pass H_2S gas	White ppt. Reappears.	Zn^{2+} confirmed

Result : The given substance A-30 contains

Anions	Br^-
	SO_4^{2-}
Cations	Cu^{2+}
	Zn^{2+}

Experiment No. 11

You are provided with a mixture labelled as A-32. Apply formal group analysis to identify two anions and two cations present in it. Prepare sodium carbonate extract for anion identification while original solution for cation identification. Give one good confirmatory test for each ion you have identified. Report the result in tabular form.

Solve.**Identification of Anions**

Preparation of Sodium Carbonate extract : Treat a portion of given substance with sodium carbonate in 1 : 3 approximately in a test tube. Add to it 15 ml water and boil for sometime. Cool the contents and filter. The filtrate is sodium carbonate extract. Retain the residue for cation identification.

S.No.	Experiment	Observations	Inference
1.	Take a pinch of A-32 and add to it 2 ml dil hydrochloric acid.	Brisk effervescences are observed. A faint odour gas evolves.	CO_3^{2-} suspected
2.	Pass the evolved gas in lime water	Lime water turns milky	CO_3^{2-} confirmed
3.	To another part of A-32 add 2ml conc. H_2SO_4 and heat.	Evolution of colourless gas which burns with blue flame.	Oxalate ion is suspected
4.	5ml of sodium carbonate extract neutralised with dil. hydrochloric acid and treated with 2 ml of calcium chloride solution.	A white precipitate of calcium oxalate is formed which is insoluble in dil. H_2SO_4	Oxalate ($\text{C}_2\text{O}_4^{2-}$) confirmed

Preparation of original solution : The original solution is prepared in dil. hydrochloric acid.

S.No.	Experiment	Observation	Inference
1.	To a little portion of A-32 caustic soda solution is added and the contents are heated	A colourless characteristic pungent odour gas evolves out which turns moist red litmus strip blue.	The evolved gas is ammonia and zero group is present.
2.	Pass the evolved gas in Nessler's reagent.	The Nessler's reagent turns brown.	NH_4^+ ion confirmed
3.	Take 5 ml of original solution and add to it 1 ml dilute hydrochloric acid	Since O.S. is prepared in dil hydrochloric acid. There is no change with further addition of few ml of acid.	I gp absent
4.	Pass H_2S gas to the contents of this test tube.	No change is observed.	II gp absent
5.	Take fresh portion of original solution. Add to it few drops of conc. HNO_3 heat and cool. Now add little amount of ammonium chloride and excess ammonium hydroxide.	No change is observed	III gp absent
6.	Pass H_2S gas to the contents of this test tube	A white precipitate is observed.	IV gp present
7.	Filter the contents. Dissolve the residue in minimum amount of dilute hydrochloric acid and boil to remove H_2S gas formed due to interaction of the two reactants. To this solution add Potassium ferrocyanide solution.	A bluish white precipitate is formed.	Zn^{2+} is confirmed

Result : The give sample A-32 contains

Cations: NH_4^+ , and Zn^{2+}

Anions: CO_3^{2-} and $\text{C}_2\text{O}_4^{2-}$

Experiment No. 12

Analyse the given mixture A-19 for two cations and two anions and give a formal group analysis.

- (a) In testing for anions, you must describe how
- the soln/sodium carbonate extract was formed
 - how the gases were identified
 - confirmatory test for each anion
- (b) In testing for cations, you must describe
- how the original soln. was prepared
 - the confirmatory test for each cation

Dry tests are not accepted as confirmatory tests.

Preparation of Na_2CO_3 extract.

To a pinch of A-19 add Na_2CO_3 in the ratio 1:3. Add 10 ml water. Boil and cool. Filter the contents. The filtrate is sodium carbonate extract.

Identification of Anions

S.No.	Experiment	Observation	Inference
1.	To a pinch of A-19 add conc. H_2SO_4 . Heat. Add copper turnings.	Light brown fumes. The brown fumes become denser.	NO_3^- suspected
2.	To aq. soln. of A-19 add an equal volume of a saturated soln. of FeSO_4 crystals. Add conc. H_2SO_4 from the side of the test tube.	A brown ring is formed at the junction of two liquids.	NO_3^- confirmed
3.	Neutralise Na_2CO_3 extract with dil HNO_3 . Add BaCl_2 soln. Add sufficient dil HCl to it.	White ppt. formed. The ppt. does not dissolve	SO_4^{2-} confirmed

Preparation of O.S. The residue of Sodium Carbonate extract is dissolved in dil HCl . The soln. obtained is O.S. It will be used for cation identification.

Identification of Cations

S.No.	Experiment	Observation	Inference
1.	Add few drops of dil HCl to a part of O.S.	No change	Group I is absent.
2.	Pass H_2S gas into the above soln.	No change	Group II is absent.
3.	To a fresh portion of O.S. add 1 ml conc. HNO_3 . Boil and cool. Add a pinch of NH_4Cl + excess NH_4OH	No change	Group III is absent.
4.	Pass H_2S gas into the above soln. Filter. Dissolve the ppt in aqua regia and boil. Divide it in two parts.	Black ppt	Group IV is present.
	(a) In one part add NaOH soln.	Green ppt	Ni^{2+} confirmed

<p>(b) To the other part add NH_4OH and dimethyl glyoxime soln.</p> <p>To the filtrate add a pinch of NH_4Cl + excess NH_4OH + excess $(\text{NH}_4)_2\text{CO}_3$ soln.</p> <p>Filter the contents. Boil filtrate to reduce bulk.</p> <p>Now add excess NH_4OH and then di-sodium hydrogen phosphate soln. Scratch innerwalls of the test tube with a glass rod.</p>	<p>Scarlet red ppt</p> <p>No change</p> <p>A fine crystalline white ppt.</p>	<p>Ni^{2+} confirmed</p> <p>Group V is absent.</p> <p>Group VI is present.</p> <p>Mg^{2+} confirmed.</p>
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Result. The substance A-19 contains

Anions	Nitrate	NO_3^-
	Sulphate	SO_4^{2-}
Cations	Nickel	Ni^{2+}
	Magnesium	Mg^{2+}

Experiment No. 13

You are provided with a mixture A-20 containing two cations and two anions. Apply formal group analysis to identify these ions.

- (a) While testing for anions you must mention
- How the soln/Sodium carbonate extract was prepared.
 - How the gases were identified.
 - The confirmatory test for each anion.
- (b) While testing for cations you must mention
- How the original soln. for group analysis was formed.
 - The formal group analysis with pertinent group reagent.
 - The confirmatory test for each cation.

Preparation of Na_2CO_3 Extract. To a pinch of mixture A-20 add Na_2CO_3 in the ratio 1:3. Add 10 ml of distilled water. Boil for five minutes and cool. Filter the contents. Filtrate is sodium carbonate extract. Residue is used for cations.

Identification of Anions

S.N.	Experiment	Observation	Inference
1.	Add dil HCl to a pinch of mixture A-20.	Brisk effervescences seen with evolution of a colourless and odourless gas	CO_3^{2-} suspected
2.	Pass the evolved gas into lime water.	It turns turbid (milky)	CO_3^{2-} confirmed
3.	Add conc. H_2SO_4 to the mixture A-20 heat and it gently.	A colourless gas with pungent odour	The gas is HCl Cl^- suspected
4.	Bring a moist blue litmus paper near the mouth of the test tube.	It turns red.	
5.	Neutralise Na_2CO_3 extract with dil HNO_3 . Add to it a few drops of AgNO_3 soln.	White ppt.	
6.	Add NH_4OH to the above contents.	The ppt. dissolves readily.	Cl^- confirmed

Preparation of O.S.

Residue of Sodium Carbonate extract is dissolved in dil HCl. Warm it to prepare O.S. It is used for identification of cations.

Identification of Cations

S.No.	Experiment	Observation	Inference
1.	To a part of O.S. add dil HCl.	No change	Group I is absent
2.	Pass H ₂ S gas into the above contents	Black ppt.	Group II is present
3.	Filter the contents. Retain the filtrate for subsequent groups. Dissolve the residue in minimum amount of 33% HNO ₃ . Divide the soln. into two parts.		
4.	To first part add NH ₄ OH soln.	Deep blue ppt	Cu ²⁺ suspected
5.	To the second part add K ₄ [Fe(CN) ₆] soln.	Chocolate brown ppt.	Cu ²⁺ confirmed
6.	Boil off H ₂ S from the filtrate. Add a pinch of NH ₄ Cl + excess NH ₄ OH	No change	Group III is absent
7.	Pass H ₂ S into the above soln.	No change	Group IV is absent
8.	Boil off H ₂ S. Add excess NH ₄ OH and divide the soln. in two parts	No change	Group V is absent
9.	To one part add (NH ₄) ₂ CO ₃ soln.		
10.	To the other part add di-sodium hydrogen phosphate soln. Scratch the sides of the test tube with a glass rod.	Fine crystalline white ppt.	Mg ²⁺ confirmed Group VI is present

Result. The given mixture contains the following ions:

Cations	Copper	Cu ²⁺
	Magnesium	Mg ²⁺
Anions	Carbonate	CO ₃ ²⁻
	Chloride	Cl ⁻

Experiment No. 14

You are provided with a mixture A-21 containing two anions and two cations. Identify all four ions.

It is necessary to prepare Sodium Carbonate extract for the identification of anions and original soln. for cations.

Dry tests are not considered as confirmatory tests.

Preparation of sodium carbonate extract. To a pinch of A-21 add Na₂CO₃ in the ratio 1:3. Add 10 ml water, boil and cool. Filter the contents. Filtrate is used for anion identification

Identification of Anions

S.No.	Experiment	Observation	Inference
1.	Add dil. HCl to a pinch of substance A-21 in a clean test tube	Light brown gas is evolved with mild effervescence.	Nitrite ions suspected
2.	Pass the evolved gas through starch iodide soln.	The soln. turns blue black	Nitrite ions confirmed

3.	Neutralise Sodium Carbonate extract of A-21 with dil HNO_3 . Add BaCl_2 soln. to it.	White ppt.	SO_4^{2-} suspected
	Add dil HCl to the above	The ppt. insoluble	is SO_4^{2-} confirmed

Preparation of original soln. Residue of sodium carbonate extract is dissolved in dil HCl . The soln. obtained is original soln. It is used for the identification of cations.

Detection of Cations

S.No.	Experiment	Observation	Inference
1.	Add few drops of dil HCl to one portion of O.S.	No change	Group I is absent
2.	Pass H_2S gas into the above soln.	No change	Group II is absent
3.	To a fresh portion of O.S. add 1 ml conc. HNO_3 . Boil, cool and add pinch of NH_4Cl and excess NH_4OH .	Reddish brown ppt	Group III is present Fe^{3+} suspected
4.	Filter the contents. Retain the filtrate. Dissolve residue in dil HCl and divide it in two parts. To the first part add pot. ferrocyanide soln.	A deep blue coloured soln.	Fe^{3+} confirmed
	To the second part and KCNS solu.	Intense blood red colour	Fe^{3+} confirmed
5.	To the filtrate of group III and excess NH_4OH and pass H_2S gas.	Black ppt.	Group IV is present
6.	Filter the contents. Dissolve the residue in aqua regia. Divide it in two parts (a) To one part add NaOH soln.	Green ppt.	Ni^{2+} suspected
	(b) To the other part add NH_4OH and dimethyl glyoxime soln.	A scarlet red ppt.	Ni^{2+} confirmed

Result. The given substance A-21 contains the following ions:

Cations	Ferric	Fe^{3+}
	Nickel	Ni^{2+}
Anions	Nitrite	NO_2^-
	Sulphate	SO_4^{2-}

Experiment No. 15

Analyse the given substance A-22 for two cations and two anions. Identify all four ions. It is necessary to prepare Sodium Carbonate extract for anions and original soln. for cations. In case of anions describe the colour and odour of the gas/gases evolved. Give confirmatory tests for all ions.

Identification of Anions

Preparation of Sodium Carbonate extract : To one part of A-22 add three parts of solid sodium carbonate. To this add 15 ml of distilled water and boil. Cool and filter. The filtrate is the required extract. Retain the residue for O.S. preparation.

S.No.	Experiment	Observation	Inference
1.	To a portion of A-22 in a clean test tube add dil HCl.	Brisk effervescence with colour less and odour less gas	CO_3^{2-} suspected CO_3^{2-} confirmed
2.	Bring a moist blue litmus paper	It turns red	
3.	Pass the evolved gas into lime water	It turns milky	
4.	To a portion of A-22 add conc. H_2SO_4 and heat	Colourless gas evolved with pungent odour	Cl^- suspected
5.	Bring a moist blue litmus paper near the gas	It turns red	
6.	Bring a glass rod moistened with NH_4OH near the gas	Dense white fumes are formed	Cl^- suspected
7.	Neutralise nearly 5 ml of sodium carbonate extract with dil. HNO_3 and add 1 ml of AgNO_3 soln.	Curdy white ppt is formed	Cl^- confirmed
8.	Add NH_4OH to the contents of the above test tube	The precipitate dissolves	

Identification of Cations

Preparation of original soln.: Dissolve the residue of Sodium Carbonate extract in dil. HCl.

S.No.	Experiment	Observation	Inference
1.	Since the O.S. is prepared in dil HCl group I is absent.		Group I is absent
2.	To a portion of O.S. pass H_2S . Reject the contents of the test tube.	No Change	Group II is absent
	Take fresh portion of O.S. To this add conc. HNO_3 . Boil and cool. Add solid NH_4Cl followed by excess NH_4OH .	Gelatinous green ppt.	Cr^{3+} suspected
	Filter the contents. Retain filtrate for next group.		
	Dissolve the residue in minimum quantity of dil HCl and add NaOH soln.	Gelatinous green ppt.	
	Add NaOH soln. in excess.	The above ppt. dissolves	
	Divide this soln. in two parts.	Formation of a green ppt.	
	(i) Boil the first part in a test tube.		
	(ii) To the second portion add Bromine water and boil. To this add small amount of acetic acid and lead acetate.	Yellow ppt. formed	Cr^{3+} confirmed
	Take the filtrate retained for next group.		
	To a portion of it add a pinch of solid NH_4Cl followed by excess NH_4OH . Pass H_2S gas.	No change	Group IV absent

Reject the above contents of the test tube. Take the remaining filtrate in a test tube and boil it to reduce its volume. Cool it and add solid NH_4Cl followed by excess NH_4OH . Add to it excess ammonium carbonate soln.	White ppt.	Gp V suspected
Filter the contents. Reject the filtrate and take residue for this group. Dissolve it in minimum quality of hot dil. acetic acid. To a portion of it add Potassium Chromate soln.	Yellow ppt.	Ba^{2+} confirmed

Result : The given substance A-22 contains

Anions	CO_3^{2-} Cl^-
Cations	Cr^{3+} Ba^{2+}

Experiment No. 16

Analyse the given substrate A-24 for two cations and two anions. Identify all four ions.

Prepare sodium carbonate extract for anions. Describe the colour and odour of gas/gases evolved.

For cations describe the preparation of original soln. and give formal group analysis. Give confirmatory test for each ions you have identified.

Identification of Anions

Preparation of Sodium Carbonate extract: To a pinch of A-24 add solid Na_2CO_3 in the ratio 1 : 3. Add 15 ml of distilled water. Boil, cool and filter. The filtrate is sodium carbonate extract.

S.No.	Experiment	Observation	Inference
1.	To the given substance A-24, added some dil HCl	A light brown coloured gas evolved with pungent smell	NO_2 suspected
2.	Bring a moist blue litmus paper near the mouth of the test tube.	It turns red.	
3.	Bring a starch iodide paper near the mouth of the test tube	It turns blue.	NO_2 is confirmed
4.	To a pinch of A-24 add conc. H_2SO_4 and heat.	Dark brown coloured gas with strong pungent odour	Br^- suspected
5.	Neutralise a portion of sodium carbonate extract with dil. HNO_3 and add AgNO_3 soln.	A pale yellow ppt formed.	Br^- suspected
6.	Add NH_4OH to the above test tube.	The pale yellow ppt dissolves with difficulty	Br^- confirmed

Chapter

4

Preparation of Inorganic Compounds

Preparation of Inorganic compounds

- (i) Preparation of Potash alum/mohr's salt
- (ii) Preparation of crystalline $\text{FeSO}_4/\text{CuSO}_4$.

A **salt** is an electrovalent or ionic compound formed by the partial or complete replacement of the replaceable (ionisable) hydrogen atoms of an acid by a metallic ion or ammonium ion.

A normal salt is formed by the complete replacement of the replaceable hydrogen atoms or an acid molecule by a metallic or ammonium ion.

A double salt is formed by the combination of two simple salts in simple molecular ratio, They are obtained by mixing the saturated solutions of two simple salts followed by the crystallisation of the resultant saturated solution.

Examples. Alums, Mohr's salt etc.

The name alum is given to all double salts of the type $\text{M}_2\text{SO}_4 \cdot \text{R}_2(\text{SO}_4)_3 \cdot 24 \text{H}_2\text{O}$ where M may be Li, Na, K, NH_4 , Rb, Cr, etc., while R may be Al, Fe, Cr, Mn Co, Rh, etc.

Preparation of Potash Alum ($\text{K}_2\text{SO}_4 \cdot \text{Al}_2(\text{SO}_4)_3 \cdot 24\text{H}_2\text{O}$)

Steps

1. Take a clean beaker of 250 ml capacity and add to it 100 ml distilled water. Weigh accurately 17.4 g potassium sulphate and slowly dissolve it in this distilled water using a glass rod to get 1 M solution.
2. In another 250 ml beaker take 100 ml distilled water and add to it 34.2 g anhydrous aluminium sulphate using a glass rod to get 1 M solution.
3. In a third 250 ml beaker add 100 ml of 1 M solution of potassium sulphate and add to it equal volume of 1 M solution of aluminium sulphate.
4. Transfer this mixture to a China dish and heat the contents gently to reduce the bulk.
5. Cool the contents of China dish to crystallise.
6. Decant the uncrystallised liquid from the dish and transfer the crystals of potash alum on to a filter paper. Press the octahedral crystals between the folds of filter paper to get it dry. This is final sample of potash alum.

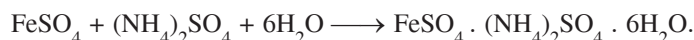


Preparation of Mohr's salt $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$

Steps

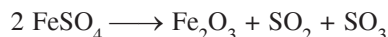
1. In a 250 ml clean beaker prepare a 1 M solution of iron (II) sulphate crystals by dissolving 27.800 g of it in 100 ml distilled water. Add few drops of dil. sulphuric acid to check hydrolysis.

2. In another 250 ml beaker prepare a 1 M solution of ammonium sulphate by dissolving 13.200 g of it in 100 ml distilled water.
3. In a 250 ml China dish transfer 100 ml of 1 M iron (II) sulphate solution followed by same volume of 1 M ammonium sulphate solution.
4. Using a tripod stand and a wire gauge heat this China dish gently over a Bunsen burner flame to evaporate excess water and to get a saturated solution.
5. Cool the contents to crystallise the salt.
6. Pale green crystals of ammonium iron (II) sulphate are obtained. This is commonly called as Mohr's salt.



Iron (II) Sulphate Crystals ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$)

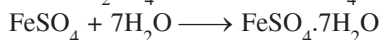
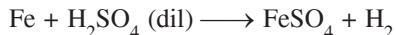
Ferrous sulphate is the most important ferrous salt. As septa hydrate it is commonly called as green-vitriol. When heated in vacuum at 140°C , the white monohydrate ($\text{FeSO}_4 \cdot \text{H}_2\text{O}$) is formed. This is stable in air. When heated at 300°C in a current of dry hydrogen, a white amorphous anhydrous ferrous sulphate is obtained. On heating to redness it liberates SO_2 and SO_3 .



Laboratory Preparation of Iron (II) Sulphate Crystals

Steps :

1. In a 250 ml beaker place about 100 ml of dil. sulphuric acid and heat it gently.
2. Add to it iron filings or iron (II) sulphide in small amount and shake the contents with a glass rod. Now add more iron filings or FeS till no more dissolves. Stir the mixture continuously till the effervescence ceases.
3. Filter the contents. Reject the residue.
4. Collect the filtrate in a beaker and transfer it to an evaporating dish.
5. Heat the evaporating dish to reduce the bulk of filtrate.
6. Cool this condensed mass to get pale green monoclinic crystals.
7. Dry the crystals by pressing them between the folds of filter paper.
8. Store them in an air tight container as the crystals are efflorescent.



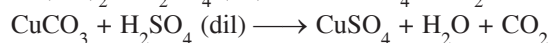
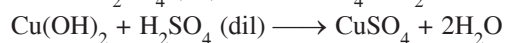
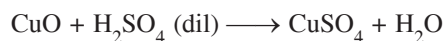
Copper (II) Sulphate Crystals ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$)

Cupric sulphate (CuSO_4) generally known as copper sulphate which crystallises from water in large blue triclinic crystals $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ is called blue vitriol. The penta hydrate crystals at 100°C crumble to a bluish white powder of monohydrate $\text{CuSO}_4 \cdot \text{H}_2\text{O}$. At 240°C in a current of dry air it becomes anhydrous mass which decomposes completely at 736°C to liberate sulphur trioxide leaving behind a black residue to copper (II) oxide.

Laboratory Preparation of Copper (II) Sulphate Crystals ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$)

1. In a 250 ml beaker take 100 ml of dil. sulphuric acid and gently heat it.
2. To this warm and dilute sulphuric acid add small amount of black copper (II) oxide or blue copper hydroxide or emerald green copper carbonate. Stir the contents using a glass rod. Add more of the copper salt till no more dissolves.

3. Filter the contents and collect the filtrate in a beaker. Reject the residual mass.
4. Transfer the filtrate in an evaporating dish and heat it gently to reduce the bulk to get a saturated solution.
5. Allow the saturated solution to cool for crystallization.
6. Blue crystals of penta hydrated copper sulphate appear in the dish.
7. Drain off the uncrystallized liquid and transfer the crystals on to a filter paper. Press the crystals between the folds of filter paper to get dry mass.
8. Store the blue crystals in an air tight container since the crystals are efflorescent.



Chapter

5

Separation Technique by Paper-Chromatography

The separation technique of chromatography is perhaps one of the most important aspects of modern chemical analysis. The credit of bringing the technique to perfection goes to Russian Botanist Mikhail Tswett. In the Course of his investigation of the pigments in plants, he performed a simple experiment to separate the two pigments from chlorophyll.

Paper Partition Chromatography

If we gently heat some pieces of dry filter paper in a dry test tube, the water drops condense at the cooler part of the test-tube. This proves that the dry paper contains water which is upto twelve per cent. This water acts as stationary phase in paper partition chromatography and behaves as immiscible liquid towards another liquid which passes over the paper.

The basic principle of chromatography can be easily understood by means of a simple experiment. Take a strip of blotting paper and about an inch from one side place a series of drops of coloured ink. Then make a mixture of two or more of these inks and put a spot of the mixture on the blotting paper. Now keep the blotting paper up-right in a dish of water about a quarter of an inch deep, with the raw of spots at the bottom.

The water is slowly drawn up the blotting paper by capillary attraction, and as its level in the paper reaches and passes through the raw of spots, it tends to washout the coloured matters and carry them up towards the top of the sheet.

In about half an hour, when the water level has nearly reached the top of the paper the experiment is complete.

It is observed that the red ink has given rise to two streaks of colour indicating that the original ink contains more than one dye. Similarly, your spot of mixed inks will separate more or less completely into its original colours.

Green ink will travel quickly up the paper revealing a small amount of yellow pigment, travelling less quickly. A mixture of all these three inks shows clear evidence of separation into yellow, red and green.

The patterns of the coloured bands obtained during the process of chromatography are called as **chromatograms**.

In the process of chromatography, the compounds can be characterised by their R_f values. It is the distance travelled by component divided by the distance travelled by the solvent front.

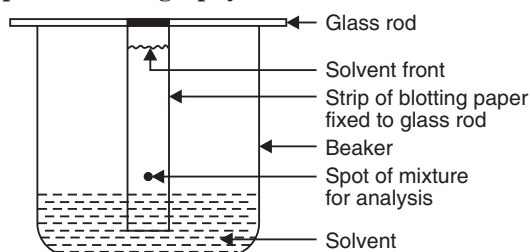
$$\text{Retention Factor, } R_f = \frac{\text{distance moved by the solute}}{\text{distance moved by the solvent}}$$

R_f value depends on a number of factors, such as :

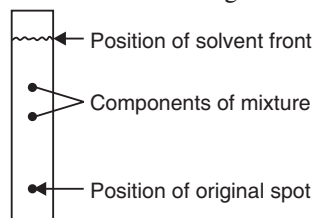
- (i) Choice of adsorbent
- (ii) Choice of solvent

- (iii) Size of sample
- (iv) distance travelled by the solvent front.

A typical set-up for paper chromatography :



Set the experiment as shown in the figure. As the solvent travels up the paper, it carries the components of the mixture with it. The final chromatogram is shown in the figure given below:



The identity of the components can be discovered by measuring their R_f values.

Task to Perform in the Laboratory.

Task Number 1

Place a spot of ink with the help of a dropper in the centre of a circular whatman number - 1 filter paper. Dry this ink spot. Now place drops of water on this ink spot and leave it to dry. Repeat the addition of water drops on the central ink spot and drying of the filter paper. After some-time it is observed that the coloured rings are obtained during the process. This pattern of coloured rings is termed as chromatogram.

Such technique of paper chromatography is known as circular paper chromatography.

Task Number 2

Repeat the above experiment with extracts of leaves and flowers using ethyl alcohol as solvent.

The extract of leaves and flowers can be prepared by crushing the leaves and flowers with ethyl alcohol in a mortar and filtering the contents.

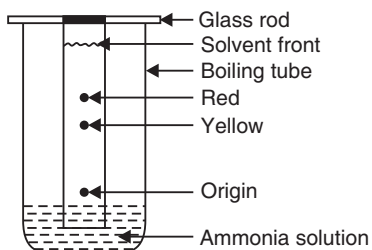
Task Number 3

Take a boiling tube containing a glass rod at its mouth, with the help of glue or fevistik fix the upper end of the filter paper with the glass rod. The length of the filter paper should be equal to the length of boiling tube but the width should be less than the diameter of the boiling tube so that it will suspend freely in the boiling tube.

Make a pencil mark on the paper about 3 cm from one end and place on the mark a small drop of a mixture of methyl orange and phenolphthalein. Dry the spot. Add another drop of this indicator mixture on the same place and dry again. Place 5 cm³ of conc. ammonia solution into the boiling tube and suspend the strip of filter paper with the precaution that it does not touch the wall of the boiling tube and the dried spot is above the surface of ammonia solution. Leave the tube standing until the solvent front has risen nearly to the glass rod.

It is observed that phenolphthalein travels faster than the methyl orange. Thus the two indicators forming chromatogram consisting of a red patch followed by yellow patch

The ratio of the distance moved by a solute in a given time to the distance moved by the solvent front is the retention factor (R_f) of the solvent for the solute.



Since the solvent travels upward, the method is called ascending paper chromatography. If the solvent travels downward on the filter paper the chromatography is termed as descending paper chromatography.

APPENDICES

I. Short Answer Questions Based on Practical Chemistry

Q.1. What is the principle of volumetric analysis?

Ans. The principle of volumetric analysis is to take a certain known volume of a solution of unknown strength and to this a solution of suitable reagent of known strength is added to get the end point and the strength of the unknown solution is calculated.

Q.2. What is standard solution?

Ans. Standard solution is defined as a solution in which a known amount of substance is present in a known volume of solution.

Q.3. What is an indicator?

Ans. An indicator may be defined as a substance which during the course of a titration makes the end point visible, either by appearance or disappearance of a colour or turbidity.

Q.4. How would you make a choice for indicator?

Ans. For strong acid and strong base - Phenolphthalein or Methyl orange

For strong acid and weak base - Methyl orange

For weak acid and strong base - Phenolphthalein

For weak acid and weak base - No suitable reagent (generally not titrated)

Q.5. Why is conc. Hydrochloric acid used in flame test?

Ans. Because chlorides easily vaporise by heat and some chloride salts form line in visible spectrum. Another reason is that chlorides ionise at low temperature which is in - reach of a burner.

Q.6. Why is platinum wire used in flame test?

Ans. Platinum wire does not melt at the temperature of Bunsen burner, nor is it oxidised. It is passive to conc. Hydrochloric acid and does not give any characteristic colour to the flame.

Q.7. Why is it necessary to prepare solution of a salt before performing wet test?

Ans. Since inorganic reactions are ionic, it is necessary to dissolve the salts to get their ions.

Q.8. When does a salt dissolve?

Ans. When solvation energy of a salt exceeds the lattice energy, it dissolves.

Q.9. Why is the chromyl chloride test given this name?

Ans. Because in this test dark brown vapours of chromyl chloride (CrO_2Cl_2) form.

Q.10. Why is it necessary to use a dry test-tube for chromyl chloride test?

Ans. Because vapours of chromyl chloride formed will dissolve in water present in the wet test-tube its hydrolysis will produce HCl and H_2CrO_4 .

Q.11. In the test for nitrate, a brown ring is formed. What is the brown ring due to?

Ans. The brown ring is due to nitroso ferrous sulphate ($\text{FeSO}_4\cdot\text{NO}$).

Q.12. Why do we use a freshly prepared ferrous sulphate solution in the "ring-test" for the identification of NO_3^- .

Ans. Because on standing ferrous ion oxidises readily to ferric ion which is not a suitable reagent for the test.

Q.13. For the precipitation of II group why it is necessary to add dilute hydrochloric acid before passing H_2S gas?

Ans. For the precipitation of II group small amount of dil HCl is added before passing H_2S gas. The S^{2-} ion concentration in the solution is greatly decreased due to the common ion effect caused by H^+ ions obtained from the acid. At the decreased concentration of the S^{2-} ions the ionic product of the III and subsequent group metal sulphides become less than their solubility product, therefore, they do not precipitate out. The IIInd group metal sulphides have a low solubility product hence the ionic product of their sulphides exceeds the solubility product and they precipitate out.

Q.14. Hydroxides of IVth and subsequent groups do not precipitate out in III group, explain?

Ans. To the filtrate of II group (after removing H_2S gas), when excess NH_4OH is added, in presence of NH_4Cl , the NH_4^+ ions obtained from NH_4Cl cause common ion effect hence the ionisation of NH_4OH is suppressed, causing the concentration of the OH^- ions to be greatly reduced. At the decreased concentration of OH^- ions, only the metal hydroxides of very low solubility product like $\text{Al}(\text{OH})_3$, $\text{Cr}(\text{OH})_3$, $\text{Fe}(\text{OH})_3$ are precipitated. The hydroxides of IV and subsequent groups cations do not precipitate out as their solubility product is very high.

Q.15. Why are only two drops of the indicator used in titration?

Ans. Their addition in greater amount will change the concentration of the solutions used in analysis as the indicators themselves are acids or bases. Methyl orange is a weak base and phenolphthalein is a weak acid.

Q.16. Why is it necessary to rinse the burette with the solution to be filled in it?

Ans. A burette is washed very well with water in order to remove the traces of solution sticking to its wall which was filled before in it. After this if it is not rinsed with the solution, the water drops sticking to it will affect the volume of the solution.

Q.17. But the conical flask is only washed with water, not rinsed with the solution. Why?

Ans. Since known volume of the solution measured with pipette is taken in the conical flask, water sticking to the flask will not make any change. It is rinsed with the solution, the volume will increase.

Q.18. When the pipette filled up to mark is emptied in to the conical flask for titration, a little solution remains behind in its top. Should it be blown out?

Ans. It should not be blown out as it is extra.

Q.19. How does a molar solutions differ from a molal soln? In both the cases in molecular weight of the solid dissolved in solvent, but in molar soln the solvent is one litre and in molal, it is one kg.

Q.20. Is it correct to call flame test a dry test?

Ans. In fact it is not the right name because a paste with HCl, not dry salt, is used in the experiment. However, we call it dry test because in the usual group analysis tests which are known as wet tests, solution of the salt is used.

Q.21. Why is flame test not applicable in all cations?

Ans. Since chlorides of all cations are not easily volatile over a Bunsen Burner Flame, it is not applicable in all the cases.

Q.22. Why is sulphuric acid unfit for the preparation of original soln?

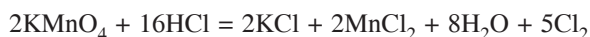
Ans. Some salts react with sulphuric acid to form their sulphates which are insoluble, hence this acid not used

Q.23. A hot original solution to be for the detection of cations develops turbidity when allowed to cool. What does it indicate?

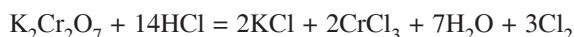
- Ans.** It indicates the presence of lead ions as lead chloride solution prepared in hot conc. HCl throws out the insoluble lead chloride when cooled.
- Q.24.** Some white salts turn light yellow when heated but turns white when cooled. What does it indicate?
- Ans.** It indicates the presence of zinc ions because zinc oxide has this property of changing colour.
- Q.25.** Silver Nitrate solution is filled in coloured bottles only. Why?
- Ans.** Since silver nitrate changes to metallic silver readily when exposed to sunlight, coloured bottles are used to protect the solution from sunrays.
- Q.26.** Lime water bottles should not be left unstoppered. Why?
- Ans.** Lime water when exposed to air absorbs CO_2 to turn it turbid due to the formation of CaCO_3 , and the turbid solution will not work in the test of carbonate, the bottle should not be left unstoppered.
- Q.27.** Why are big pieces of ferrous sulphide used not small or powder?
- Ans.** Small pieces or powder will not rest in the middle spherical bulb but fall down into the lowest one. The apparatus will then work like a woulfes bottle and the reaction will not stop when gas is not used.
- Q.28.** Why should Cork be tight in the middle bulb?
- Ans.** If not, the pressure of the gas will throw it out following a continuous reaction and the apparatus will fail to work.
- Q.29.** What will happen if the cork in the lowest bulb becomes loose?
- Ans.** The acid filled in the apparatus will flow out disturbing the arrangement.
- Q.30.** Commercial sample of ferrous sulphide is responsible for impurities in gas. Why is it used in this form?
- Ans.** Because it is Cheap.

II. Important Chemical Equations

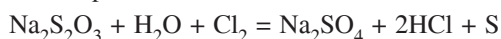
1. When potassium permanganate crystals react with conc. hydrochloric acid, potassium chloride manganese (ii) Chloride, water and chlorine are obtained.



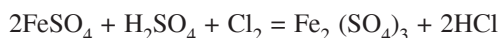
2. When potassium dichromate crystals react with hydrochloric acid the chlorides of potassium and chromium are formed along with water and chlorine



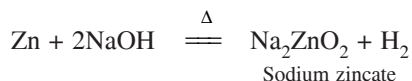
3. Sodium thiosulphate reacts with chlorine in presence of water to form sodium sulphate, hydrochloric acid and sulphur.



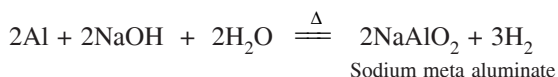
4. Ferrous sulphate reacts with chlorine in presence of sulphuric acid (dil) to form ferric sulphate and hydrochloric acid.



5. Caustic soda soln. reacts with zinc to form sodium zincate and hydrogen.



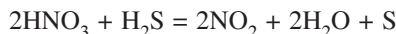
6. Aluminium reacts with caustic soda soln to form sodium meta aluminate and hydrogen



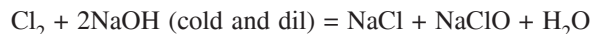
7. Ferric chloride soln is reduced by hydrogen sulphide to ferrous chloride, sulphur and hydrochloric acid



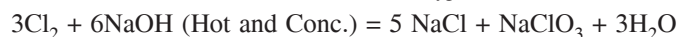
8. Nitric acid reacts with hydrogen sulphide to form colloidal sulphur and yellowish brown coloured nitrogen dioxide gas



9. Chlorine reacts with caustic soda soln. The products depend upon whether caustic soda is cold and dilute or hot and concentrated

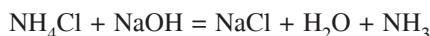


Sodium hypochlorite



Sodium chlorate

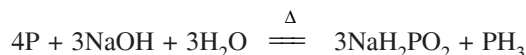
10. Ammonium chloride reacts with caustic soda soln when heated, to produce sodium chloride, ammonia and water



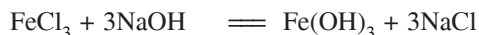
11. Sulphur dissolves in hot concentrated soln of caustic soda to form sodium sulphide, sodium thiosulphate and water



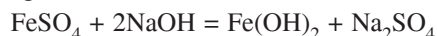
12. Yellow phosphorus dissolves in caustic soda soln to form sodium hypophosphite and phosphine gas



13. Ferric chloride soln. reacts with caustic soda soln to form reddish brown ppt. of ferric hydroxide and sodium chloride



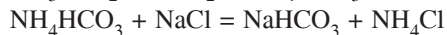
14. Ferrous sulphate soln. reacts with caustic soda soln to form dirty green coloured ferrous hydroxide and sodium sulphate



15. Magnesium reacts with hot & Con. sulphuric acid to produce magnesium sulphate, water and sulphur dioxide



16. When ammonia gas and carbon dioxide gas are passed into an aqueous soln. of sodium chloride the following reactions take place

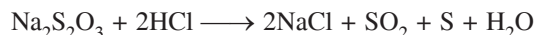


17. Sodium bicarbonate on heating produces sodium carbonate, water and CO_2 gas.



These are the main reactions of manufacture of washing soda by solvay process.

18. When sodium thiosulphate reacts with dil HCl, colloidal sulphur is provided as given below



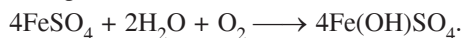
Sulphuric acid behaves in the same manner



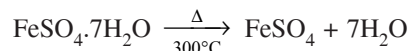
19. When ferrous sulphate is heated, ferric oxide, sulphur dioxide and sulphur trioxide are produced



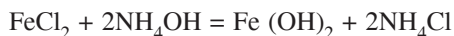
20. Ferrous sulphate, when exposed to moist air slowly turns yellow by partial oxidation to basic iron (iii) sulphate



21. Ferrous sulphate crystals when heated at 300°C lose the molecules of water of crystallisation seven in number



22. When ferrous chloride soln. reacts with ammonium hydroxide, dirty green coloured ferrous hydroxide is formed



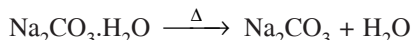
23. Crystalline form of sodium carbonate exists as decahydrate ($\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$)

On heating it loses nine molecules of water of crystallisation, leaving behind one molecule attached to it



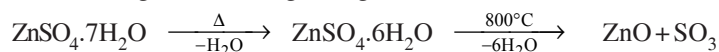
On further heating, one molecule also is given out leaving it anhydrous

The anhydrous salt is stable towards heat and melts without decomposition at 852°C.



24. In crystalline form zinc sulphate exists as hepta hydrate ($\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$)

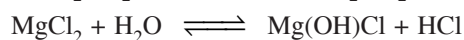
When heated it undergoes following changes



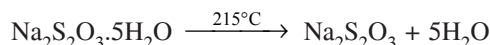
25. ZnSO_4 soln. reacts with sodium bi carbonate as shown



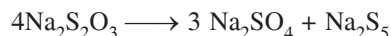
26. When magnesium chloride hexa hydrate crystals are heated first four molecules are lost and then it undergoes partial hydrolysis with out producing the anhydrous salt



27. When sodium thiosulphate penta hydrate crystals are heated at 215°C, five molecules of water of crystallisation are lost



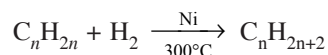
The anhydrous salt when heated above 223°C it decomposes to form sodium sulphate and sodium penta sulphide



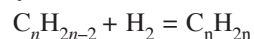
III. Organic Reactions

(i) Sabatier Senderen's Reaction

Conversion of an alkene into alkane

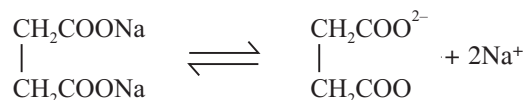


Conversion of an alkyne in to alkene

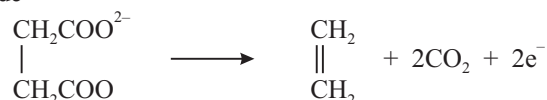


(ii) Kolbe's Reaction

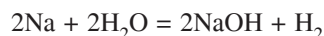
Preparation of ethylene by the electrolysis of an aqueous soln. of sodium or potassium succinate



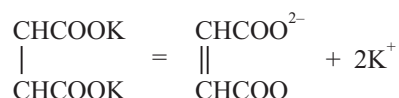
At the anode



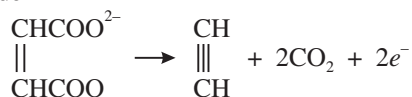
At the cathode $2\text{Na}^+ + 2e^- = 2\text{Na}$



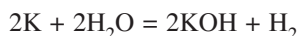
Same way acetylene can be prepared by an aqueous soln of sodium or potassium fumarate



At the anode



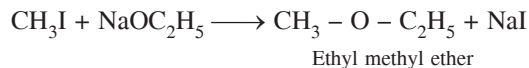
At the cathode $2\text{K}^+ + 2e^- = 2\text{K}$



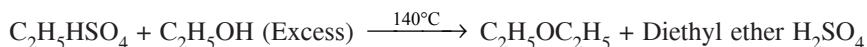
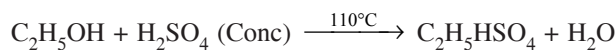
(iii) Wurtz Reaction



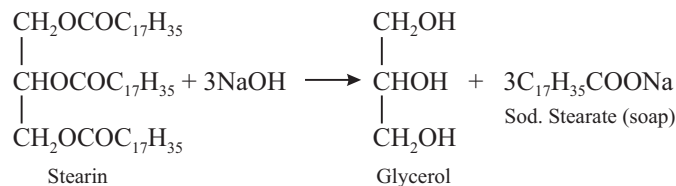
(iv) Williamson's Reaction



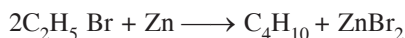
Williamson's Continuous etherification reaction



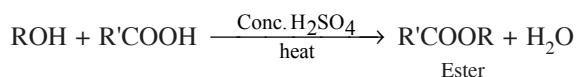
(v) Saponification Reaction



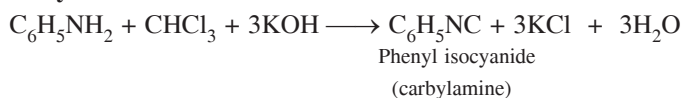
(vi) Frankland Reaction

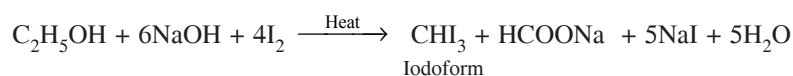
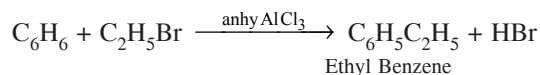
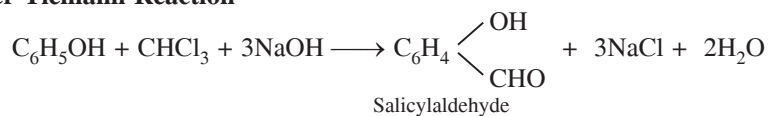


(vii) Esterification



(viii) Carbylamine Reaction



(ix) Haloform Reaction**(x) Friedel Craft's Reaction****(xi) Reimer Tiemann Reaction****IV. Common Abbreviations in Chemistry**

abs.	Absolute	hyd.	hydrated
anhyd.	anhydrous	<i>i.e.</i>	that is
approx.	approximately	insol.	insoluble
aq.	aqueous	liq.	liquid
b.p.	boiling point	max.	maximum
conc.	concentrated	min.	minimum
concn.	concentration	m.p.	melting point
const.	constant	p.d.	potential difference
crit.	critical	ppt.	precipitate
cryst.	crystalline	r.a.m.	relative atomic mass
d.	decomposed	r.m.m.	relative molecular mass
decomp.	decomposition	sol.	soluble
dil.	dilute	soln.	solution
dist.	distilled	sp.	specific
<i>e.g.</i>	for example	s.t.p.	standard temperature and pressure
e.m.f.	electro motive force		
eqn.	equation	temp.	temperature
expt.	experiment	t.t.	test tube
fig.	figure (diagram)	vac.	vacuum
f.p.	freezing point	v.d.	relative vapours density
		wt.	weight
		o	degree (celsius)

V. Important Constants

Absolute zero of temperature is $^{\circ}\text{K}$ or -273°C

Avogadro constant, L or $N_A = 6.02 \times 10^{23} \text{ mole}^{-1}$

1 Calorie = 4.18 Joules

Charge on an electron = $1.6 \times 10^{-19}\text{C}$

1 electron - volt (1eV) = $1.6 \times 10^{-19}\text{J}$

Faraday constant $F = 9.65 \times 10^4\text{C mol}^{-1}$

Ionic product of water $K_w = 1.008 \times 10^{-14} \text{ mol}^2\text{dm}^{-6}$

(at 298K or 25°C) mass of electron, ($9.11 \times 10^{-31}\text{kg}$).

Molar gas constant, $8.314 \text{ JK}^{-1} \text{ Mol}^{-1}$

Ratio of masses, neutron/electron, 1840

Ratio of masses, proton/electron, 1840

s.t.p. = standard temperature and pressure which is 1.00 atmosphere or 760 m.m. of mercury or 101 KPa and 273K or 0°C.

Temperature of the triple point of water is 273.16K volume of 1 mole of gas at s.t.p. is 22.4 dm³ (molar volume).

VI. Some Common Elements, their Symbols, Atomic Numbers and Atomic Weights.

Element	Symbol	Atomic number	Atomic weight
Aluminium	Al	13	26.981
Argon	Ar	18	39.948
Barium	Ba	56	137.34
Bromine	Br	35	79.909
Calcium	Ca	20	40.08
Carbon	C	6	12.011
Chlorine	Cl	17	35.453
Chromium	Cr	24	51.996
Copper	Cu	29	63.546
Hydrogen	H	1	1.0079
Iodine	I	53	126.904
Iron	Fe	26	55.847
Lead	Pb	82	207.19
Magnesium	Mg	12	24.312
Manganese	Mn	25	54.938
Mercury	Hg	80	200.59
Nitrogen	N	7	14.006
Oxygen	O	8	15.999
Phosphorus	P	15	30.973
Potassium	K	19	39.102
Silver	Ag	47	107.870
Sodium	Na	11	22.989
Sulphur	S	16	32.064
Tin	Sn	50	118.69
Zinc	Zn	30	65.37

VII. Behaviour of Salts with Water

Soluble	Insoluble
Lead Nitrate	Manganese (IV) Oxide
Copper Chloride	Mercury (II) Oxide
Copper Nitrate	Lead (II) Oxide
Copper Sulphate	Lead (IV) Oxide
Ferrous Sulphate	Tri Plumbic Tetroxide
Ferric Chloride	Lead Carbonate
Zinc Nitrate	Copper Oxide
Potassium Chloride	Copper Carbonate
Sodium Chloride	Zinc Oxide
Sodium Carbonate	Zinc Carbonate
Caustic Soda	
Ammonium Chloride	
Ammonium Dichromate	

VIII. Formulae and Colours of Some Salts

Name of Compound	Chemical Formula	Colour
Mercury (II) oxide	HgO	Red or Yellow
Lead (II) oxide	PbO	Yellow
Lead (IV) oxide	PbO ₂	Dark Brown
Tri plumbic tetroxide	Pb ₃ O ₄	Scarlet
Lead carbonate	PbCO ₃	White
Lead nitrate	Pb(NO ₃) ₂	White
Copper (II) oxide	CuO	Black
Copper (II) carbonate	CuCO ₃	Bright green
Copper (II) carbonate dihydrate	CuCO ₃ ·2H ₂ O	Emerald green
Copper (II) chloride	CuCl ₂	Dark Brown
Copper (II) nitrate tri hydrate	Cu(NO ₃) ₂ ·3H ₂ O	Deep Blue
Copper (II) sulphate pentahydrate	CuSO ₄ ·5H ₂ O	Blue
Copper (II) sulphate	CuSO ₄	White
Copper hydroxide	Cu(OH) ₂	Pale Blue
Ferrous sulphate septa hydrate	FeSO ₄ ·7H ₂ O	Green
Anhydrous Ferrous sulphate	FeSO ₄	Dirty yellow
Ferric chloride	FeCl ₃	Black
Zinc oxide	ZnO	White
Zinc carbonate	ZnCO ₃	White
Zinc nitrate	Zn(NO ₃) ₂	White
Potassium chloride	KCl	White
Sodium chloride	NaCl	White
Sodium carbonate decahydrate	Na ₂ CO ₃ ·10H ₂ O	White
Ammonium chloride	NH ₄ Cl	White
Ammonium dichromate	(NH ₄) ₂ Cr ₂ O ₇	Orange
Manganese (IV) oxide	MnO ₂	Black

IX. Molecular and Equivalent Weights of Some Common Substances

Name	Formula	Molecular Weight	Equivalent Weight
Hydrated copper sulphate	$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$	249.71	249.71
Hydrated ferrous amm. sulphate.	$\text{FeSO}_4(\text{NH}_4)_2^-$		
	$\text{SO}_4 \cdot 6\text{H}_2\text{O}$	392.10	392.10
Hydrochloric acid	HCl	36.5	36.5
Iodine	I_2	253.84	126.92
Nitric acid	HNO_3	63.0	63.0
Oxalic acid	$(\text{COOH})_2 \cdot 2\text{H}_2\text{O}$	126.08	63.04
Potassium bicarbonate	KHCO_3	100.00	100.00
Potassium dichromate	$\text{K}_2\text{Cr}_2\text{O}_7$	294.21	49.03
Potassium hydroxide	KOH	56.0	56.0
Potassium permanganate	KMnO_4	158.03	31.6
Sodium bicarbonate	NaHCO_3	84.0	84.0
Sodium carbonate	Na_2CO_3	106.0	53.0
Sodium hydroxide	NaOH	40.0	40.0
Sodium thiosulphate	$\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$	248.20	248.20
Sulphuric acid	H_2SO_4	98.0	49.0

X. Strength of Aq. Solution of the Common Acids and of Aq. Ammonia

	Approximate			Volume required to make 1 litre of approximate
Reagent	Percent by Weight	Specific gravity	Normality	N soln in ml.
HCl	35	1.18	11.3	89
HNO_3	70	1.42	16.0	63
H_2SO_4	96	1.84	36.0	28
H_3PO_4	85	1.69	41.1	23
CH_3COOH	99.5	1.05	17.4	58
Aq. NH_3	27	0.90	14.3	71

XI. Preparation of Reagents

S.No.	Name of chemical	Volume/mass dissolved per litre of distilled water
1.	Dil. hydrochloric acid	430 ml
2.	Dil. sulphuric acid	140 ml
3.	Dil. nitric acid	310 ml
4.	Acetic acid	300 ml
5.	Caustic soda	220 gm
6.	Lime water	Saturated soln of slaked lime should be filtered to get a clear soln.
7.	Lead acetate	95 gm
8.	Pot. per manganate	3.2 gm
9.	Pot. dichromate	15.0 gm

10.	Silver nitrate	17 gm
11.	Barium chloride	61 gm
12.	Amm. hydroxide	340 ml
13.	Amm. carbonate	160 gm in 140 c.c. conc. amm. hydroxide and dilute to one litre
14.	Pot. ferrocyanide	53 gm
15.	Pot. iodide	83 gm
16.	Pot. chromate	49 gm
17.	Pot. ferricyanide	55 gm
18.	Pot. thiocyanate	49 gm
19.	Amm. oxalate	35 gm
20.	Methyl orange	1 gm
21.	Ferric chloride	135 gm in 1 litre (containing 20 ml conc. hydrochloric acid)
22.	Iodine soln.	12.7 gm mixed with 20 gm of KI in 30 ml distilled water and dilute to one litre
23.	Sodium nitroprusside	5 gm

XII. Preparation of Some Common Indicators

Phenolphthalein Solution

2.5 gm of solid phenolphthalein is dissolved in 25 ml of rectified spirit. This is diluted by adding 250 ml of distilled water with constant stirring. Any suspension if left may be filtered out to get clear filtrate.

Methyl Orange Solution

0.5 gm of solid methyl orange is dissolved in 500 ml. of distilled water and heated. Filter to get clear filtrate.

Blue Litmus Solution

5 gm of solid blue litmus is dissolved in 500 ml of distilled water.

Red Litmus Solution

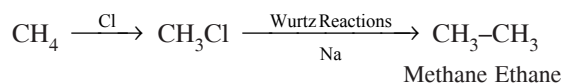
This can be prepared easily by adding a few drops of conc. HCl to the blue litmus soln. prepared above. (Acid should be added till the solution acquires a red colour)

Methyl Red Solution

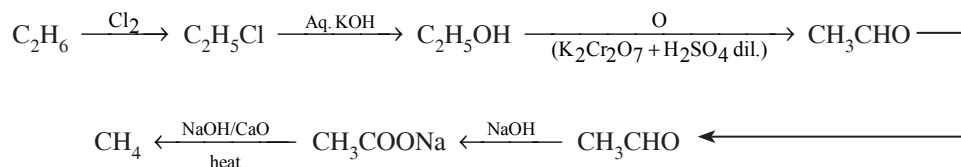
0.5 gm of solid is dissolved in 250 ml of ethanol and then diluted by adding 250 ml of distilled water to it.

XIII. Organic Conversions

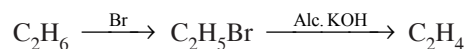
Conversion of methane to ethane

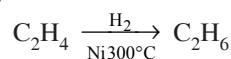
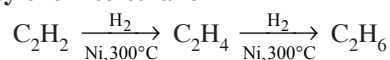
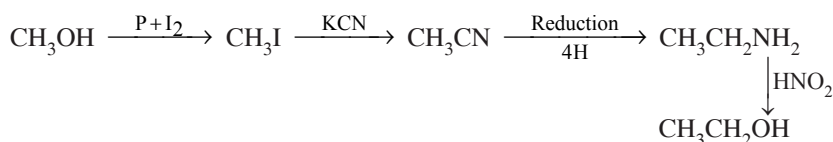
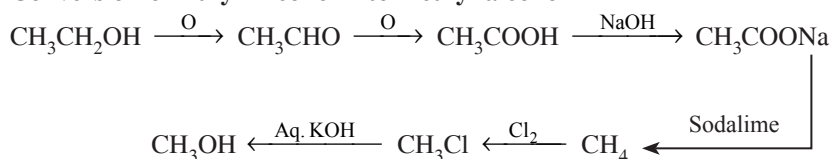


Conversion of ethane to methane



Conversion of ethane into ethylene



Conversion of ethylene into ethane**Conversion of ethane to acetylene****Conversion of acetylene into ethane****Conversion of methyl alcohol into ethyl alcohol****Conversion of Ethyl Alcohol into methyl alcohol****XIV. Distinctions of Aliphatic Compounds****How would you distinguish between**

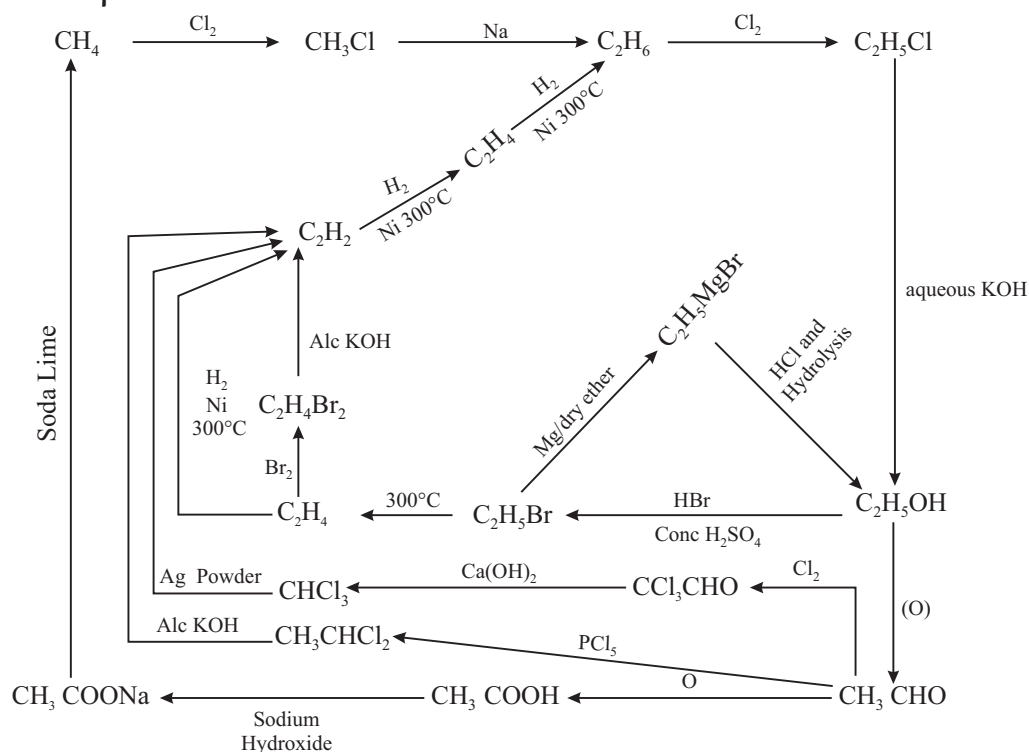
- Ethyl alcohol and di methyl Ether
- Glycerol and Ethanol
- Ethane and Ethylene
- Ethylene and acetylene
- Methanol and Ethanol

(i)	Treated with	Ethyl Alcohol	Ether
	(a) sodium metal	Hydrogen gas evolves	No reaction
	(b) Acetic acid and conc. sulphuric acid	Fruity odour due to formation of Ethyl acetate	No reaction
	(c) I ₂ + Alkali	Yellow crystals of iodoform	No reaction
(ii)	Treated with	Glycerol	Ethanol
	(a) KHSO ₄ and heated	Disagreeable odour produced due to formation of acrolein.	No reaction
	(b) I ₂ + Alkali	No reaction	Yellow crystals of iodoform
(iii)	Treated with	Ethane	Ethylene
	Bromine water	No change	Bromine water Decolourises
(iv)	Treated with	Ethylene	Acetylene
	(a) Ammonical AgNO ₃ soln.	No Change	White ppt. of silver acetylide
	(b) Ammonical Cu ₂ Cl ₂ soln.	No Change	Red precipitate of cuprous acetylide
(v)	Treated with	Methyl Alcohol	Ethyl Alcohol
	(a) Salicylic acid	Methyl salicylate formed	No change
	(b) I ₂ + Alkali	No change	Yellow crystals of Iodoform.

DISTINCTION OF INORGANIC COMPOUNDS (IONS)**XV. How would you distinguish between**

- (i) Cl^- , Br^- and I^-
- (ii) CO_3^{2-} and SO_3^{2-}
- (iii) SO_3^{2-} and SO_4^{2-}
- (iv) Pb^{2+} and Zn^{2+}
- (v) Pb^{2+} and Hg^{2+}
- (vi) Ca^{2+} and Zn^{2+}
- (vii) Fe^{2+} and Fe^{3+}

(i) Treated with (a) Conc. H_2SO_4 and heated (b) AgNO_3 soln.	Cl^- Colour less fumes White ppt soluble in excess NH_4OH	Br^- Brown fumes Pale yellow ppt. which dissolves in NH_4OH with difficulty	I^- Violet fumes Yellow ppt which is insoluble in excess NH_4OH
(ii) Treated with dil. H_2SO_4	CO_3^{2-} Colourless odourless gas (CO_2)	SO_3^{2-} Colour less gas with burning sulphur odour (SO_2)	
(iii) Treated with BaCl_2 Soln/ Ba $\text{Ba}(\text{NO}_3)_2$ Soln.	SO_3^{2-} white ppt. which is soluble in dil HCl	SO_4^{2-} White ppt which is insoluble in dil HCl	
(iv) Treated with NH_4OH	Pb^{2+} White ppt which is insoluble in excess NH_4OH	Zn^{2+} White ppt which is soluble in excess NH_4OH	
(v) Treated with KI Soln.	Pb^{2+} yellow ppt. which does not dissolve in excess KI	Hg^{2+} Scarlet ppt which dissolves in excess KI soln.	
(vi) Treated with NaOH soln.	Ca^{2+} White ppt. formed which is insoluble in excess NaOH Soln.	Zn^{2+} White ppt formed which is soluble in excess NaOH solution.	
(vii) Treated with NaOH soln. KCNS soln.	Fe^{2+} Dirty green ppt. No change	Fe^{3+} Reddish brown ppt. Blood red colour	

XVI. Aliphatic Conversion Chart**XVII. First Aid**

In case of accidental injuries, the first aid treatment should be given to the students as given below. But if the injury is of a very serious nature, a competent doctor should be consulted immediately.

Nature of accident**Treatment required****Burns**

(i) From fire, steam and hot objects.

Treat the affected area with cool water thoroughly and apply a mixture of linseed oil and lime water in equal amounts or cold cream. Bandage lightly.

(ii) From acids

Wash the affected area with plenty of running water and then with dilute solution of sodium hydrogen (bi) carbonate.

Note: In case of sulphuric acid, do not use water but wash with sodium hydrogen carbonate solution directly.

(iii) Alkalies

Wash with plenty of water followed by dilute acetic acid.

(iv) From phenol

Wash with alcohol and apply soframycine skin ointment.

(v) From Bromine

Wash with dilute solution of carbonate and apply alcohol.

Cut	Stop bleeding by washing with alum solution. Remove any glass pieces if visible.
Eye injuries acids and alkalies	If drops of acid or alkali fall into eye, wash with plenty of running water. Then wash with a 3% solution of sodium hydrogen carbonate in case of acid and 2% boric acid soln. in case of alkali.
Fire	
(i) From inflammable liquids	Throw plenty of sand on it - preferably moist.
(ii) Clothes	Allow the student to lay down on the floor such that the burning side of the clothes remain upwards check the supply of air to the burning portion.
Inhalation of poisonous fumes and gases	
(i) Chlorine gas	Allow the student to inhale liquor ammonia.
(ii) Carbon monoxide gas	Expose the student to fresh air.
(iii) Chloroform	Expose the student to fresh air and arrange for artificial respiration.
(iv) Hydrogen sulphide gas	Allow inhalation of ammonia and arrange for artificial respiration.
(v) Nitrous oxide gas	Expose student to fresh air and allow to inhale steam.
Poisoning	Allow the student to drink plenty of water
Acid	Give the student two table spoons-ful of Digene or any other anti acidal suspension.
Alkali	Give plenty of water followed by one table spoon-ful of vinegar or lime juice.
Chemicals on dress	
Acids	Wash with ammonium hydroxide or ammonium carbonate soln. followed by plenty of water.
Strong alkalies	Apply dil. acetic acid or lime juice wash with water.

