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Government of Telangana

INTERMEDIATE
CHEMISTRY
PRACTICAL RECORD

TELANGANA OPEN SCHOOL SOCIETY, HYDERABAD

313

Intermediate (TOSS) Course

Senior Secondary Course

CHEMISTRY

4

LABORATORY MANUAL

Telangana Open School Society (TOSS), Hyderabad

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SENIOR SECONDARY COURSE
INTERMEDIATE (TOSS) COURSE
CHEMISTRY-ADDITIONAL CURRICULUM

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SENIOR SECONDARY COURSE
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CHEMISTRY-ADDITIONAL CURRICULUM

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A word with you

Dear Learner,

We welcome you to the Chemistry laboratory course. I hope that you will enter into the laboratory with lot of enthusiasm to learn and understand many chemical aspects.

The laboratory manual contains introduction, basic laboratory techniques, laboratory safety measures, 15 experiments and finally the appendices.

After doing experiments 1 to 7, you will learn how to prepare certain gases, dilute the concentrated acid, or concentrated solutions, compare the reactivity of metals, predict the pH value of the solutions and determine melting point of the substances. Experiment 8 will help you to find out effect of concentration and temperature on reaction rate. We are sure that you will really enjoy while doing separation of pigment of ink or grass juice in the experiment no. 9. Experiment 10 deals with detection of extra elements in organic compounds whereas the experiment no. 11 deals with reactions of carbohydrates which are really interesting to perform. After doing experiments no. 12, you can compare soaps which are available in the market by comparing their foaming capacity and cleaning effect.

Experiment No. 13 deals with following three major aspects:

i) Proper use of analytical balance.

ii) Preparation of standard solution by using primary standards, and

iii) Estimation of chemical substances by volumetric method.

Experiment no. 14 gives you an idea to identify unknown chemicals 1.e. salt (inorganic qualitative analysis) whereas experiment no. 15 deals with detection of functional groups of organic compounds(organic qualitative analysis).

We believe that after completing the experiments described in this manual, you will gain confidence to perform chemical reactions safely and efficiently.

The laboratory is a place to learn about the behavior of matter. You are suggested to forget preconceived notions about what is supposed to happen, follow instructions carefully, and see what actually happens. Be meticulous in recording the true observations even though you "know" something else should happen. Don't hesitate to seek advice from your teacher. Recall the ancient dictum "He who ask a question may appear ignorant for the moment, he who does not, remains ignorant forever".

We hope, you will find all the experiments described in the manual interesting and informative. In case, you face any problem while doing the experiments, you are suggested to consult your teacher all the success.

-WRITERS

CONTENTS

INTRODUCTION

INTRODUCTION

Chemical Science is developed on the basis of observations of natural events (happenings). These observations have given us bridge to new discoveries. These discoveries were realised by experimentation. Hence, experiments are essential, in chemical science. As we know, the objectives of practical chemistry are as follows:

1. To develop and inculcate laboratory skills and techniques.
2. To enable the students to understand the basic chemical concepts, and
3. To develop basic competence of analysing and synthesising chemical compounds and mixtures.

To meet these objectives three different types of laboratory experimetns are provided in the present practical course.

1) Experiments for developing laboratory skills / techniques.
2) Concept based experiments
3) Experiments based on preparation of compounds.

1. THE FORMAT OF THE MANUAL

One of the important features of this manual is that it is a self instructional material. Experiments are given in a standard format consisting of the following headings:

1.1 Objectives
1.2 What you should know
1.3 Materials required: (a) Apparatus, and (b) Chemicals
1.4 How to perform the experiment
1.5 Precautions
1.6 Observations
1.7 Conclusion
1.8 Check your understanding
1.9 Note for the teacher
1.10 Check your answers

Let us understand these one by one

1.1 Objectives: The objectives of an experiment give you an idea about the skills or the knowledge that you are expected to develop while performing the experiment. Certain objectives can be achieved even after going through the procedure.

1.2 What you should know: Brief discussion of theory of the experiment is given under this heading.

1.3 Materials required: It gives the essential list of apparatus and chemicals.

1.4 How to perform the experiment: The procedure to perform the experiment is described in this section.

1.5 Precautions: The precautions to be followed while performing the experiment are given. The learners are advised to follow these precautions to avoid accidents in the laboratory.

1.6 Observations: The observations should be recorded properly under this heading.

1.7 Conclusion: This section contains the conclusion drawn on the basis of observations made in the experiment.

1.8 Check your understanding: At the end of each experiment, a few questions have been incorporated to consolidate what has been done and to check your own understanding about it.

1.9 Note for the teacher: A note is given for the teacher where ever felt necessary. it gives important points about the experiment, the compounds/salts to be selected or special precautions to be taken by the students while performing the experiment.

1.10 Check your answers: In this section, the answers are given to those questions which are asked in the section to check your understanding. The students are advised to compare their answers. The corrections should be made wherever required.

2. CLEANING OF GLASS APPARATUS

It is essential to use clean glassware for performing an experiment.

- **i)** The dirt sticking to glass apparatus can be removed by rubbing it with a bottle brush (specially designed for it) or rubber tip attached to a glass rod.
- **ii)** Washing soda or any good detergent can be used for washing or a dilute solution of soap may be used.
- **iii)** For innovative purposes very clean glassware is to be used. For this, the glasswares are kept for some time inside the chromic acid bath and then washed with water. (Chromic acid is prepared by dissolving potassium dichromate in concentrated sulphuric acid.
- **iv)** After completing your experiment, clean all glass-ware, dry it and keep it in right place.

3. HANDLING OF GLASS APPARATUS

Glass apparatus is brittle in nature; therefore, proper precautions should be taken to avoid the breakage while handling the apparatus. It is also advised not to heat reagent bottles and any other bottle.

4. HANDLING REAGENT BOTTLES

While adding liquids directly from bottles, you should observe certain elementary precautions. First, double-check the label to ***make sure that you have the right reagent.*** Second, never put the stopper of the reagent bottle on the working table so as to contaminate its inner surface.

5. PERFORMING AN EXPERIMENT

1) Read the experiment carefully and understand the procedure to be followed.
2) Note down the instructions and precautions given by your teacher (and mentioned in the Manual).
3) Collect all the necessary apparatus and plan the work so as not to waste time.
4) Make sure that all the glass apparatus are clean.
5) Arrange the apparatus as instructed.
6) Carry out the experiment as said in the procedure carefully. Be observant and unprejudiced while making observations.
7) At the end of work and before leaving the laboratory:
    i. Wash your apparatus and place in right place.
    ii. Note down the instructions and precautions given by your teacher.
    iii. Check that the reagent bottles are placed at their proper places with proper lid.
    iv. Put off the gas and water.
    v. Close the locker-drawer after putting everything in it.

8) While coming for doing experiments in the laboratory, every student must bring the following items with her/him
    i. The laboratory manual for consultation
    ii. A note-book for recording observations.
    iii. A towel or limp-cloth for drying the glass apparatus and your wet hands.

6. PRACTICAL NOTE BOOK

The practical note book would be a complete original written record of data and the observations collected during practical work. This record is examined and evaluated by the examiner at the time of your final examination. Each student must have one's own hard cover bound Note book, according to the teacher's recommendation. Following points should be remembered while writing the laboratory record.

Throughout the practical note book you should follow the same style, and use the same writing ink, preferably a good fountain pen and a sharp black pencil. It is recommended that the right hand page should be written in blue ink and the left hand page with black pencil.

The recommended style of writing is shown below.

Left hand page	**Right hand page**
	Title of the experiment
Figures	Date
	Experiment No.
	Objective or Aim
	Apparatus
Observations and Calculations	Theory
	Procedure
	Precautions
	Results

1. Each experiment should start from a new page.
2. A neat, proportionate and well labeled line diagram of the apparatus used should be drawn.
3. Write chemical equations on the left hand page of the record note book, if any.
4. Record what was actually done and observed. Don't copy down the recommended procedure.
5. Under the heading procedure, write a detailed account of the procedure of doing the experiment in the past tense.
6. Write experiment, observations and inference in the tabular form, for qualitative analysis and functional group identification experiment.
7. If a mistake has been done, cross out the erroneous matter with a sinlge line so that it can still be read. Rewrite the correct statement in its place.
8. Do not tear off a page from the note-book.
9. Before leaving the laboratory, get the record signed by your teacher.

7. DISTRIBUTION OF MARKS FOR PRACTICAL EXAMINATION

You must be known that there will be practical examination of 20 marks and 3 hours duration. The distribution of marks is as given below.

		Experiment	Marks
1.		Salt (One cation + one anion)	4
2.		Volumetric Analysis	6
	i)	Write-up in which student may be asked to write brief method indicator, equations and end point	2
	ii)	Set-up of experiment	2
	iii)	Results	2
3.	i)	Detection of extra elements in an organic compound	
	ii)	Detection of functional group	
		OR	
		Setting up of one experiment from experiments at Serial number 2 to 10,12 and 13	4
4.		Viva-voce	3
5.		Record Book	3
		Total	**20**

GENERAL SAFETY MEASURES AND ACQUAINTANCE WITH BASIC LABORATORY TECHNIQUES

1. OBJECTIVES

After reading this lesson you will be able to:

- Cut and bend glass rods / tubes;
- Bore rubber cork;
- Seal the glass apparatus for leakage;
- Measure the volume, mass and density of a given substance;
- Perform simple laboratory operations such as filtration, crystallisation and distillation;
- Use burner properly in the laboratory.

2. WHAT YOU SHOULD KNOW

You can drive a car smoothly if you have proper training. Similarly, if you have to carry out any work, you need to have proper knowledge of the work in advance to do it successfully. Similary, if you have to start work in chemistry laboratory, you should know how to use chemicals and handle apparatus, cleaning of used appratus, disposing off used chemicals, use filter paper and broken glassware etc.

While performing experiments, the techniques such as cutting and bending of glass tube/rod, drawing out a glass tube, boring of a rubber cork, sealing of apparatus, calibration of certain **instruments** such as balance are frequently involved. Certain laboratory techniques, namely filtration, crystallisation and distillation are also involved in various experiments. In this unit, you will learn how to perform the required laboratory techniques.

In chemistry laboratory, we deal with different chemicals, glass apparatus, LPG gas and some instruments, etc. Many a times, in the laboratory, a number of accidents occur due to negligence and inadequate knowledge about the chemicals and apparatus being used. We can make chemistry laboratory a safer place by following **certain general safety measures with special reference to safe handling of chemicals.**

To avoid accidents, observe the following Do's and Don'ts in the chemistry laboratory.

2.1 The Do's

- Wear the lab coat / apron before starting the work.
- Light the burner with a match stick. Never use a piece of paper to light the burner.
- While heating a liquid in a test tube, always keep the mouth of test tube away from you and your neighbors.
- Pipette out harmful/corrosive liquids by using adaptor (used for sucking liquid by creating vacuum)
- Always use minimum quantity of chemicals.
- To avoid contamination, use droppers for adding reagents.
- Carry out the reactions involving pungent or obnoxious fumes either in the open or in a fuming cup board.
- Wash your hands thoroughly with soap after the laboratory work.
- Keep the reagent bottles & apparatus in their proper place. Always close the water and gas taps when not in use.

2.2 The Don'ts

- Don't wear loose and / or synthetic clothes while working in the laboratory.
- As a general rule, never taste any chemical in the laboratory not even sucrose or sodium chloride. They may be contaminated.
- Do not try to smell unknown chemicals.
- Don't pipette out harmful / corrosive liquids by sucking with mouth.
- Don't insert a thermometer or a glass tube forcibly through a cork.
- Don't inhale any gas deeply and directly. It may cause suffocation. Keep the test tube a little away from your nose and smell the gas by directing the gas away from you.
- Don't keep inflammable substances like organic solvents near the flame.
- Don't keep sodium in open (i.e. exposed to the atmosphere).
- Don't interchange droppers of different reagents and never keep the dropper on the table.
- Do not throw any solid material or corrosive liquids in the sink. The acids should be well diluted with water before discharging them.
- Don't add pumice stone to the boiling liquid.
- Don't touch electric switched with wet hands
- Don't perform an unknown experiment in the laboratory without the supervision of your teacher.

Inspite of taking all the precautions, accidents do happen. For such an eventuality, one must be fully equipped with the necessary first aids to remedy the injury. Apply following remedial measures in case of an accident.

a) Chemicals causing burn

Chemicals responsbile for burn	**Neutralizing wash**
Acids like HCl, HNO, or H_2SO. Alkalies like, KOH, NaOH etc. Sodium	Wash with 1 percent sodium bicarbonate solution or with 2M ammonium carbonate. Then, wash with enough water. Apply any soothing cream. Wash with 1M acetic acid, followed by enough water. Then apply vaseline or a soothing cream. Remove the solid piece immediately and wash with dilute acetic acid and enough

b) Hazardous chemicals and their effects

You should also know various hazardous chemicals and their effects. Do not expose yourself excessively to these chemicals. These chemicals and their effects are given below.

Hazardous Chemicals	**Effects**
H_2S	Almost as poisonous as HCN. Exposure dulls the sense of smell.
H_2SO_4, HF, SO_2, NO_2, Cl_2, Br_2, I_2, HNO_3	All are dangerous. When concentrated they cause skin burns
Salts of Ag, Ba, Hg, Ni, Pb CrO_4^{2-} and MnO_4^-	These are harmful only when swallowed
$AgNO_3$	Causes caustic burns.
Chlorinated alkanes e.g., $CHCl_3$, $CC1_4$, Benzene	Most of these are narcotics, and suspected carcinogens, Carcinogen, may cause cancer

c) Common Accidents in Chemistry Laboratory

i) Cuts : In chemistry laboratory, the common accidents are cuts from glasswares, which are broken while being used. Wash the cut with cold water. If bleeding does not stop, then apply pressure on the cut. Then apply antiseptic cream and a proper dressing. Consult the doctor, if necessary.

ii) Burns: These are caused by touching hot equipments. Wash the burns with cold water for about 10 min. (till burning sensation stops), then apply burnol.

iii) Fire: A fire in beaker is extinguished by covering the glass beaker with a watch glass or a metal plate. If clothes catch fire, then lie down on the floor and roll or cover the body with a blanket.

iv) Poisoning: If accidentally someone swallows a poisonous chemical then make him drink lot of fresh water. If the person is still unconscious seek medical help.

3. ACQUAINTANCE WITH BASIC LABORATORY TECHNIQUES

Experimentation is an integral part of any study of science including chemistry. It enables us to gain an insight into what we learn in theory. In this section of this course, we intend to acquaint you with the laboratory and some basic techniques like:

- Cutting and bending of glass tube, and Boring a cork
- Filtration, crystallization and distillation
- Measurement of volume, mass and density
- Using a burner and Cleaning of apparatus

While performing various experiments, we require glass tubes of different lengths for setting up of apparatus. It involves the cutting of glass tubes which are usually 4-6 min in diameter. In assembling an apparatus, bent glass tubes are required e.g. apparatus for preparing and collecting a gas. The corks available are without holes / bores. To use the corks in an apparatus such as preparing a gas, a glass tube is passed through the cork, which is fitted to a glass vessel. This fitting of glass tube in cork requires the hole of proper diameter. After the experiment, the glass apparatus should be cleaned thoroughly.

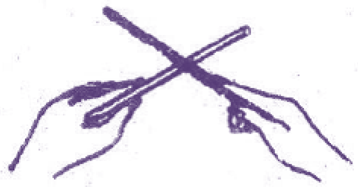
The basic laboratory techniques are given in the form of experiments.

3.1 To cut a glass tube of desired length

Materials Required: Glass tube / rod, ruler, small triangular file, gas burner / spirit lamp.

3.1.1 How to perform the experiment?

Take the given glass tube and measure the required length with help of ruler/measuring tape and put a mark on it with an ink pen or glass marking pencil. Hold it with one hand near the point where the cutting is to be done. Make a single straight scratch with the help of a triangular file. See Fig (i). Do not apply too much pressure on the tube otherwise the glass tube will break. Hold with a piece of cloth and press the tube gently outward. The tube will break into two pieces. Fig. (ii).



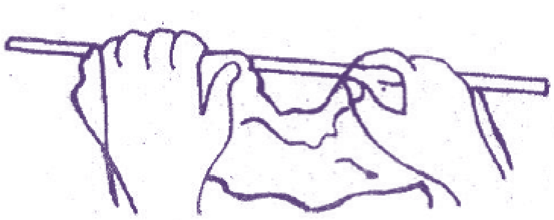


Fig. (i) *Making a scratch on the glass tube* **Fig ii)** : *Holding a Breaking a glass tube*

If the tube does not break, put the scratch again at the same point and try to break it by the same method.

Normally, the freshly cut ends are sharp and may cause injury to the hands. Therefore, round of the ends by rotating the rubs in the blue zone of the flame for a short time as shown in the Fig. (iii). Keep your fingers at the safe distance from the flame.

3.1.2 Precautions

1. The file should be moved always in backward down direction.
2. If difficulty is experienced in breaking the glass tube or rod, put another scratch on the point.

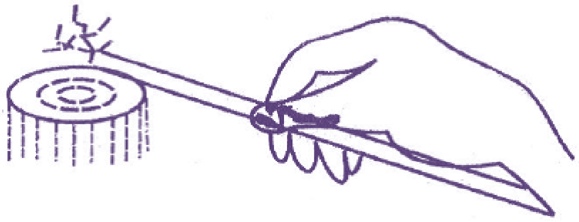


Fig (iii): *Rounding off the edges of the broken*

Note: To a cut glass rod same procedure should be used as done for the glass.

3.2 Bending of a glass tube

Materials Required: Glass tube, burner, file and asbestos sheet.

3.2.1 How to perform the experiment?

Hold the glass tube horizontally with the portion of the glass tube to be bent on the blue flame of the burner. Heat this portion of the glass tube with simultaneous rotation, in the blue flame of the burner, as shown in the Fig. (iv). Allow the tube to bend under its own weight (Fig. V).

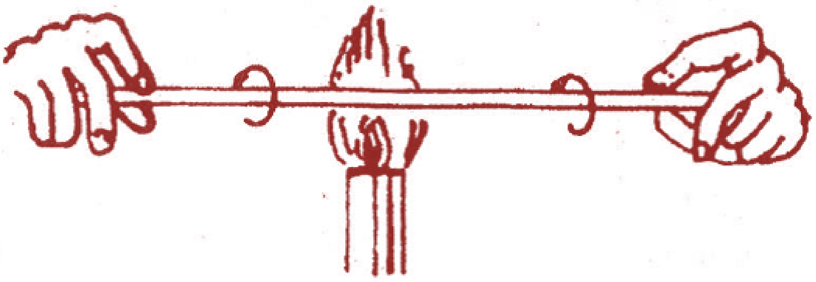


Fig. (iv): *Heating the glass tube*

When the tube becomes soft, bend the glass tube from its ends to the desired angle. Do not bend the tube abruptly. Maintain the desired angle by holding the glass tube ends with both hands. Press the bent limbs to make them coplanar by putting it on the asbestos sheet shown in Fig. (iv).

Fig (v): *Bending of glass tube under*

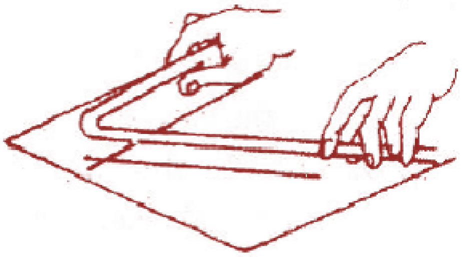


Fig (iv): *Making the bent limbs of*

3.2.2 Precautions

Do not touch the bent portion of the tube with fingers until it is cooled.

3.3 To bore a cork and fit a glass tube in it

Materials Required: A cork, cork borer set, glass tube.

3.3.1 How to perform the experiment?

The cork free from cracks. Checks that it fits tightsly in the mouth of apparatus for which it is needed. Wet the cork with water and soften it with the help of cork press. If it is not available, you may do it by rolling under your shoe as shown in Fig. (vii). Select a cork borer, whose diameter is slightly less than that of glass tube or rod, for which the hole is needed. Place the cork on the table with its narrow ends upwards and hold it with one hand, make around hole in its centre by pressing the borer gently inward with the other hands as shown in the Fig. (viii).

Fig. (vii) : *Pressing the cork with shoe*

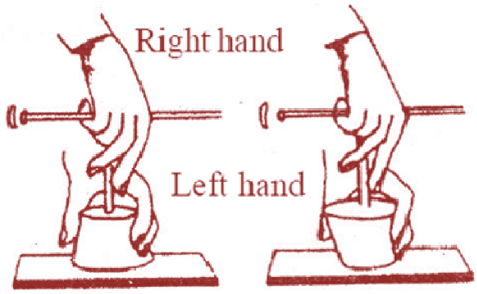


Fig. (viii) : *Boring of cark*

3.3.2 Precautions

1. While boring the cork, make sure that the hole is made in straight line.
2. While inserting the glass tube into the hole, the tube should be pushed gently with a rotatory motion.
3. The tube should be lubricated with water or soap solution to avoid accidental breakage.

3.4 Filtration

In filtration, insoluble small solid particles/dust/impurities etc. are separated from liquids by passing the mixture through filter paper which is affixed inside the funnel. The filter papers have small pores through which only the liquid (only particular particle size) can pass through.

Materials Required: Funnel, glass rod, beaker, filter paper, sugar solution with small amount of sand.

3.4.1 How to perform the experiment?

Filtration involves two steps. In first step, the filter paper is folded and fitted into the funnel. For this, take a clean filter paper (fix ix a) and cut it in a circle of required diameter with scissors. Fold it in half semi circle and then in quarters. Open one section. Place the filter paper into a clean funnel as shown in Fig. (ix). Wet the filter paper with distilled water/liquid which is part of the mixture. Place clean beaker/conical flask below the funnel to collect filtrate. Transfer the mixture to the funnel slowly by using a glass rod as shown in the Fig. (ix b).

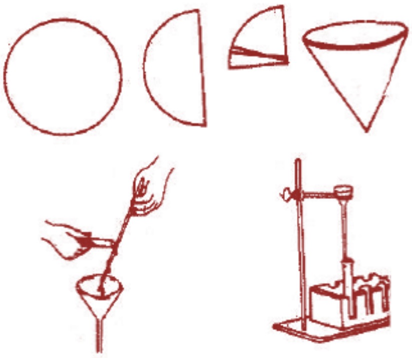


Fig. (ix): Filtration

Hold the glass rod at an angle over the funnel, the lower end of the glass rod should not touch the filter paper but should be just above it. Pour the mixture down the rod. The level of the mixture to be filtered should be not more than 3/4th the height the filter paper. Collect the filtrate in a beaker or test-tube or conical flask. For easy and quick filtration, first transfer the clear supernatant liquid of the mixture on the filter paper and then the remaining portion.

3.4.2 Precautions

i) Do not transfer all the mixture to the filter funnel at one time.

ii) The filter paper should be smaller in size than funnel.

iii) The mixture level should never be above 3/4th of the height of the filter paper

iv) The lower end of the funnel should just touch the inside wall of the beaker.

3.5 Distillation

Distillation is used to purify liquids such as water and organic solvents, and/or recycling used solvents. Distillation is the process in which the impure liquid is heated to its boiling point in a closed vessel. The vapour thus formed are cooled by passing through a air or water condenser. As a result, the pure liquid is obtained.

Materials Required:

a) **Apparatus:** Conical flask/round bottomed flask, condenser, thermometer, beaker, heating mantle and oil bath.
b) **Chemicals:** Impure solvents and tap water.

3.5.1 How to perform the experiment?

Take the impure solvent in the round bottomed (RB) flask. Fill the flask to about half of its capacity. Add a few pieces of pumice stone. Fit a cork with a hole in the mouth of the round bottom flask. In the hole, fit a thermometer as shown in Fig. (x). Fit a liebig condenser as shown in Fig. (x). Place the round bottom flask on the heating bath.

Heat the contents of the flask. At the boiling point of the liquid, it starts boiling and the emanating vapour pass through the condenser and get condensed into liquid. Collect the liquid at the end of the condenser. Collect the pure liquid at constant boiling point. Always leave some liquid in the round bottom flask.

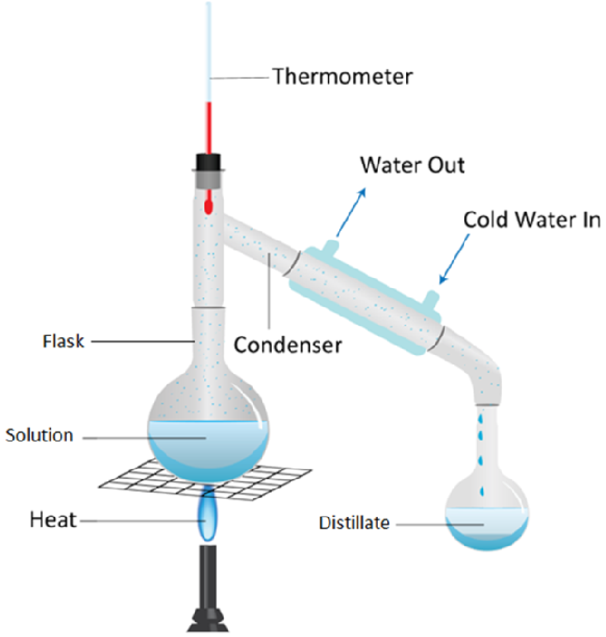


Fig. (x) : *Distillation*

3.5.2 Precautions

i) Slowly increase heating rate.
ii) Do not open the round bottom flask during heating.
iii) Stop distillation when a small amount of liquid is still left in the flask. Do not evaporate to dryness.
iv) Pumice stones should be added in the beginning itself.
v) In case of organic solvents, you have to watch the thermometer's temperature throughout the experiment to avoid overheating.

3.6 Crystallization

Crystallization is a process of obtaining crystals of a pure substance from a solution which may contain some impurities.

A hot saturated solution of impure substance is prepared in a suitable solvent. This is filtered to remove insoluble impurities and then allowed to cool slowly to room temperature. During the cooling period, the solution should not be disturbed. Crystals of pure substance are gradually formed. Then filter the crystals.

3.7. Measurement of volume, mass and density

The volume of liquids can be measured with the help of measuring cylinders, graduated beaker, pipette, burette and syringe. These measuring vessels can be chosen according to the need of accuracy of the experiment. Except syringe, measuring vessels are directly calibrated in cubic centimeter c.c. (or milliliters i.e. mL). These measuring vessels sometime may be wrongly graduated. Therefore, it is desirable to check the measuring vessel before using it.

Before you actually perform measurements for any liquid, you should take following precautions:

- Clean the measuring vessels with soap and water and dry them in air.
- Check the nature of the liquid. If it is an acid or some poisonous liquid, take care that your fingers do not get wet with it while handling the liquid.
- If the liquid is hot, then allow it to cool to room temperature.

i) Now, find out the least count (i.e. the least measurable volume) of each given measuring vessel. This is the value of the volume between two successive marks on the measuring vessel.

ii) Take the small measuring vessel and transfer the given liquid into it slowly. The rim of vessel containing liquid should be slightly inside the rim of the measuring vessel. Keep the measuring vessel slightly tilted from vertical as shown in Fig (xi), so tha the liquid falls down the wall smoothly. If it is not tilted, then the liquid falling from a height strikes the surface with splash and some of the liquid drops may sprinkle out of the measuring vessel.

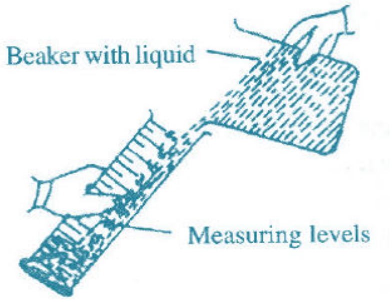


Fig. (xi) : *Transferring liquid into the measuring vessel*

iii) If the given liquid is less in volume than the total capacity of the measuring vessel, then after transferring the whole liquid put the measuring vessel on the table. Allow its level to become steady. Now take the reading of the level of the liquid in the vessel which gives the volume of the given liquid.

iv) If the quantity of the given liquid is more than the capacity of the measuring vessel, then fill the vessel to its capacity. Method to fill the vessel to capacity is as follows:

As the measuring vessel is being filled, it should also be moved to make it vertical, when it gets filled close to the uppermost mark corresponding to the capacity marked on the vessel. Use the dropper for transferring liquid drop by drop to fill the vessel exactly upto the uppermost mark. Transfer this liquid into an empty container. Again fill the measuring vessel with the remaining liquid and repeat the procedure.

The total volume (V) of the given liquid is now given by

Number of times the measuring vessel is filled to its capacity X capacity of the vessel + Level reading in the last transfer

v) if, however, the liquid is highly viscous like glycerine, then it would stick to the walls of the vessel. In such cases, in each transfer from the measuring vessel to the extra container, you have to wait till whole of the viscous liquid is tranferred slowly to the container and the measuring vessel becomes empty. But it may take too much time and you may use only the measuring vessel of capacity larger than the given liquid.

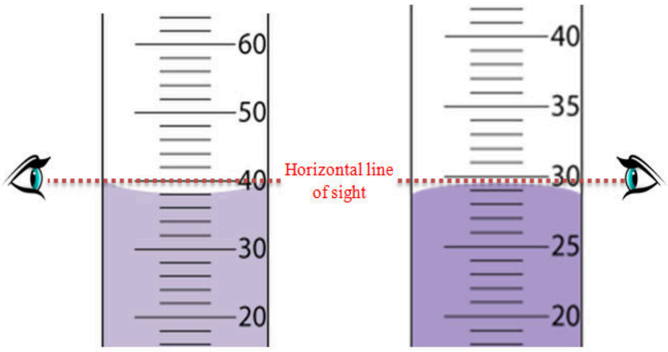


Fig. (xii) : *Taking reading of the (a) concave surface and (b) convex surface of liquid in vessel*

vi) The liquid meniscus in the measuring vessel is curved (concave or convex), therefore take the reading of the mark tangential to the lowest point of the curved surface in case of concave meniscus and uppermost point of the curved surface in case of convex mensiscus, as shown in the Fig. (xii).

vii) If the liquid is tranparent, then it should be properly illuminated by side light while taking the reading of its concave or convex surface.

viii) Take the readings of the level of liquid in the measuring vessel by keeping your eye in horizontal plane with the level, keeping the vessel on the table only, otherwise it may cause error due to parallax.

In case the vessel containing the given liquid does not have a rim like that of a beaker, then you can take help of a glass rod to transfer the liquid. For example, suppose the liquid is milk contained in a glass. Hold the glass rod in your left hand and lower it into the measuring vessel as shown in the Fig. (xii). Now transfer the milk from the glass into the measuring vessel by touching the rim of the glass to rod. The milk easily flows down the glass rod to the measuring vessel, without flowing down along the surface of tilted glass outside the measuring vessel. A given volume of a liquid can be measured using either a graduated cylinder, a pipette, or a syringe, depending upon the quantity and accuracy required. Burettes and volumetric flasks are also used to measure liquid volumes accurate.

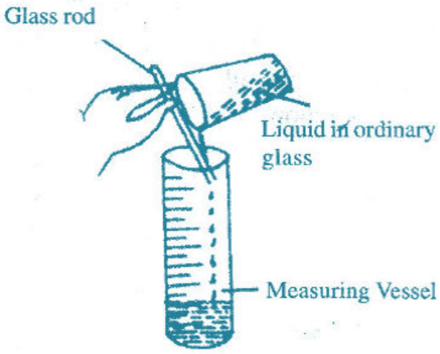


Fig. (xiii) : *Transfering liquid into a vessel*

3.8 Graduated Cylinders

Graduated cylinders are not highly accurate, but they are adequate for measuring specified quantities of solvents and wash liquids as well as liquid reactants that are present in excess. The level of the liquid should always be read from the bottom of the liquid meniscus.

3.9 Pipettes

Graduated or volumetric pipettes can be used to measure accurately relatively small quantities of a liquid. Suction is required to draw the liquid into a pipette. However, suction by mouth is unwise because of the danger of drawing toxic or corrosive liquids into the mouth. An adaptor should be used to pipette out harmful/corrosive liquids. It is used as follows:

1. Insert the adaptor into the top end of the pipette.
2. Dip the pipette tip in the liquid.
3. Press the adaptor to force the air out and then release to suck the liquid into the pipette.
4. Fill the pipette to just above the calibration mark.
5. Adjust the liquid up to the calibration mark and pipette out the liquid.
6. Remove the adaptor from the pipette.

Most volumetric pipettes are calibrated "to deliver" a given volume, meaning that the measured liquid is allowed to drain out by gravity, leaving a small amount of liquid in the bottom of the pipette. This liquid is not removed, since it is accounted for the calibration. Graduated pipettes are generally filled to the top (zero) calibration mark and then drained into a separate container until the calibration mark for the desired volume is reached. The remaining liquid is either discarded or returned to its original container. The maximum indicated capacity of some graduated pipettes is delivered by draining to a given calibration mark and of others by draining completely. It is important not to confuse the two, since draining the first type completely will deliver a greater volume than the indicated capacity of the pipette.

3.10 Syringes

Syringes are most often used for the precise measurement and delivery of very small volumes of liquid, as in gas-chromatographic analysis. A syringe is filled by placing the needle in the liquid and slowly pulling out the plunger until the barrel contains a little more than the required volume of liquid. Then the syringe is held with the needle pointed up and the plunger is pushed into eject the excess sample. Excess liquid is wiped off the needle with a tissue.

Syringes should be cleaned immediately after use, by rinsing them several times with a volatile solvent then reused.

3.11 Mass

In chemical laboratory, mass of chemicals can be determined by single pan balance or rough balance or a chemical balance. To make solutions for general purposes, one can use an ordinary balance, but when chemicals are needed to perform reactions, one should weigh them by the use of chemical balance. Therefore the mass of chemicals is very accurately needed. You can use chemical balance to measure mass of chemicals. The details of using chemical balance are given in experiment 13.

3.12 Density of a liquid

For determining the density of a liquid, the mass of the substances in divided its volume. The densities of few liquids are shown in the table given below:

Table: Densities of some liquids

Liquid	Volume in milliliters (ml)	Mass (g)	Density (g cm^{-3})
Water	100	100	1.00
Cooking oil	100	92	0.92
Glycerine	100	125	1.25
Whole milk	100	103	1.03

Precautions:

1. Don't use water to cool hot tube, beaker
2. Keep your hand dry and clean while performing the work.
3. Always follow precaution given in each section.

4. CHECK YOUR UNDERSTANDING

1. Why, while cutting a glass tube, one should not make multiple scratches ?

....

2. What is the right choise of a cork borer ?

....

3. Why is the crk moistened and softened before boring ?

....

4. How can you round off the edges of a freshly cut glass tube ?

....

5. NOTE FOR THE TEACHER

The Laboratory techniques such as cutting a glass tube, bending a glass tube are little difficult for the students. The teacher should help the students and should watch them throughout their practice.

6. CHECK YOUR ANSWERS

Ans. 1: Multiple scratches, if made on the glass tube will cause a rough and irregular cut.

Ans. 2: The outer diameter of the borer should be equal to the inner diameter of the tube to be inserted into the bore ?

Ans. 3: To avoid cracking of the cork and to get a smoother bore, it is moistened with water and pressed in a cork presser.

Ans. 4: The edges should be heated gently by rotating on a flame.

List of Experiments

EXPERIMENT-1

To prepare, collect and study some important physical and chemical properties of three gases, at least one each from the following groups:

a) Hydrogen / Oxygen
b) Carbon dioxide / hydrogen sulphide
c) Chlorine, hydrogen chloride and sulphur dioxide

1.1 OBJECTIVES

After reading this lesson you will be able to:

- Set up the apparatus for preparation of gases, namely, H_2, O_2, CO_2, H_2S, SO_2, HCl and Cl_2;
- Prepare and collect the gases mentioned above;
- Observe physical properties (e.g. colour, odour) of gases, and
- Identify various gases by their characteristic chemical tests.

1.2 WHAT YOU SHOULD KNOW

Gases, like hydrogen, oxygen, carbon dioxide, ammonia, hydrogen sulphide, chlorine, hydrogen chloride and sulphur dioxide can be prepared in the school laboratory and it is possible to identify (characterise) them by suitable tests. Identification of gases is an important step in chemical analysis as most of the substances give off some gas when heated alone or when treated with certain reagents such as acids, alkalies, etc.

In this experiment, you will learn to prepare some gases and study their important properties.

1.3 PREPARATION OF HYDROGEN (H_2) GAS

Hydrogen gas can be prepared in the laboratory by adding dilute hydrochloric acid to zinc granules. The reaction is

$$Zn(s) + 2HCl\ (aq) \longrightarrow ZnCl_2 + H_2(g)\uparrow$$

Materials Required

(1) Apparatus	(2) Chemicals
Woulf's bottle, Delivery tube,	i) Zinc metal granules (Zn)
Gas jar, bark corks,	ii) Dilute hydrochloric acid (HCl)
Thistle funnel, Water trough	iii) Distilled water

1.3.1 Procedure

Set up the apparatus as shown in the Fig. 1.1 Take 2-3 grams of zinc granules in Woulf's bottle. Fix a thistle funnel and delivery tube as shown in the diagram. Fix the two hole corks tightly over the mouth of the Woulf's bottle and make the joints airtight by sealing them with wax.

Now slowly add about 10mL of dilute hydrochloride acid from thistle funnel, so that Zinc pieces are covered with acid. As soon as the acid comes in contact with zinc pieces, the gas starts evolving.

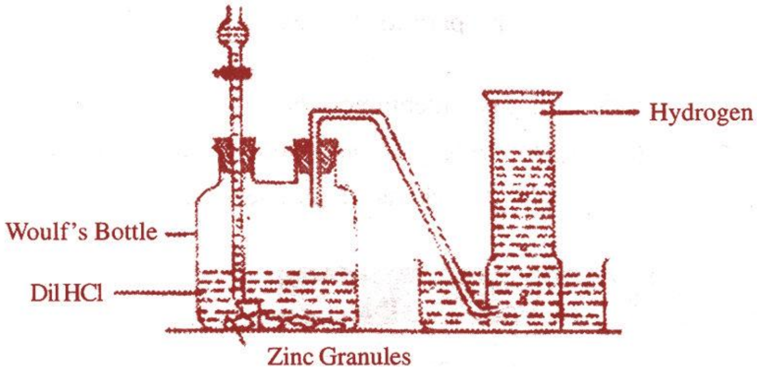


Fig. 1.1 : *Laboratory Preparation of Hydrogen gas*

Since the solubility of hydrogen in water is very less, it can be collected by downward displacement of water as shown in the Fig. 1.1

Fill a gas jar with H_2 gas. Examine the properties and perform the tests. Note your observations as follows:

i) Observe the colour of the gas; it is colourless.
ii) Carefully smell the gas; it is odourless.
iii) Collect the gas in a test tube, the way you did for collecting it in a jar. Bring a lighted match stick near the mouth of the test-tube. There is a sudden pop sound and the gas burns with a pale-blue flame.

1.4 PREPARATION OF OXYGEN (O_2) GAS

Oxygen gas is prepared by heating a mixture of potassium chlorate and manganese dioxide. Potassium chlorate is decomposed by heat while manganese dioxide acts as catalyst. If potassium chlorate was to be heated alone, a higher temperature would be required to decompose it. Since solubility of the gas in water is less, collect the gas by downward displacement of water as shown in the Fig. 1.2.

$$2KClO_3(s) \xrightarrow{MnO_2} 2KCl(s) + 3O_2(g) \uparrow$$

Materials Required

(1) Apparatus		(2) Chemicals
Round bottom flask (100mL)	(i)	Potassium chlorate ($KClO_3$)
Gas jar, Delivery tube, Water trough	(ii)	Manganese dioxide (MnO_2)

1.4.1 Procedure

Set up the apparatus as shown in the Fig. 1.2, Take approximately half a spatula of dry potassium chlorate in a watch glass and add a pinch of manganese dioxide. Mix the reagent and the catalyst. Take mixture in the boiling tube and fix the delivery tube in its mouth. Make sure that the cork is fitted by in the mouth of the tube. Seal it with wax, Heat the mixture gently and collect the gas in a jar kept inverted on a bee-hive shelf in a water through as shown in Fig. 1.2

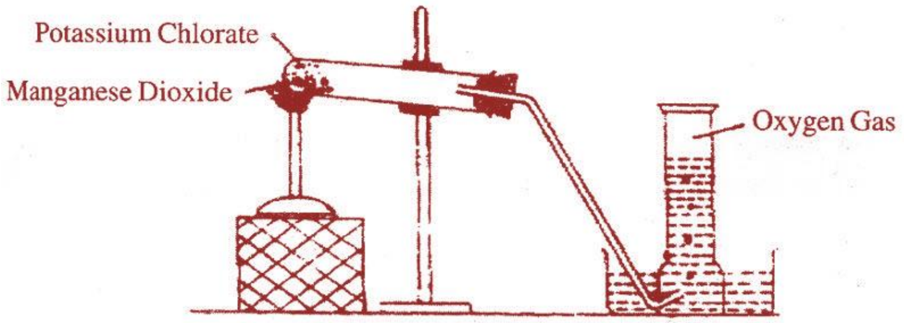


Fig. 1.2: *Preparation of Oxygen gas*

Care must be taken while heating the reagents, because potassium chlorate is a very powerful oxidising agent. If it is allowed to mix with carbon or any similar substance or dust, it can easily catch fire or even explode.

Fill two jars, examine the properties, perform tests that are listed and note your observations.

i) Observe the colour of the gas. It is colourless.

ii) Carefully smell the gas. It is Odourless.

iii) Light a splinter, and then blow it out, leaving the end glowing. Bring the glowing end of the splinter near the mouth of the jar. The splinter bursts into a flame. This shows the presence of oxygen inside the tube.

1.4.2 Physical Properties

- Oxygen is a colourless and odourless gas.
- It is heavier than air (Density 1.428g/cm^3).
- It is sparingly soluble in water.

1.4.3 Chemical Properties

i) The electronic arrangement of oxygen atom is 2,6. To attain eight electrons in its outer most shell, it has to gain two electrons. In doing so, it displays pronounced oxidising properties.

ii) **Reaction with metals:** Noble metals such as gold, platinum etc. are resistant to oxygen. But the reactive metals (Na, K etc.) react spontaneously, giving their oxides.

$$4Na + O_2 \longrightarrow 2Na_2O$$

Less reactive metals such as Mg, Cu react with oxygen on heating.

$$2Mg + O_2 \xrightarrow{\Delta} 2MgO$$

$$2Cu + O_2 \xrightarrow{\Delta} 2CuO$$

1.5 PREPARATION OF CARBON DIOXIDE (CO_2) GAS

Carbon dioxide gas is prepared in the laboratory by the action of dilute hydrochloric acid on marble (calcium carbonate).

$$CaCO_3(s) + 2HCl\ (aq) \longrightarrow CaCl_2\ (aq) + H_2O\ (l) + CO_2(g)$$

Materials Required

(1) Apparatus	(2) Chemicals

Round-bottom flask, Thistle funnel,	i) Calcium carbonate ($CaCO_3$)
Gas jar	ii) Dilute hydrochloric acid (HCl)

1.5.1 Procedure

Set up the apparatus as shown in the Fig. 1.3. Place a few small size marble pieces in a Woulf's bottle. Fix at Katle funnel so that its lower end dips in water contained in round bottom flask. Fix the two hole cork tightly in the mouth of the round bottom flask and make the joints airtight by sealing them with wax.

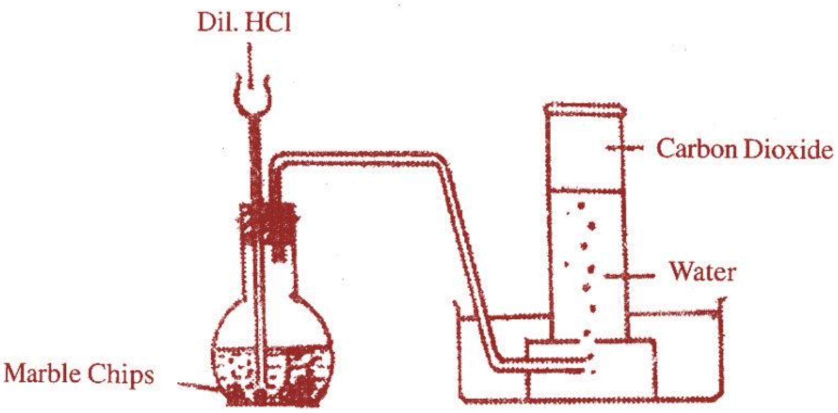


Fig. 1.3: *Preparation of Carbon-dioxide gas*

Add 2 to 3 mL of dilute hydrochloric acid from the thistle funnel. Immediately reaction takes place and carbon dioxide gas is produced. Allow some gas to escape as the initial release of carbon dioxide gas would displace air contained in gas jar. Collect the gas in a dry gas jar by the upward displacement of air.

To check, whether the gas is collected in the jar or not, bring a lighted match stick near the mouth of the gas jar. If it gets extinguished, the jar contains carbon dioxide gas. Place another jar at the delivery tube. Fill a few gas jars.

In order to examine the properties, perform the following tests and note your observations.

i. Note the colour of the gas; it is colourless,
ii. Carefully smell the gas; it is odourless.
iii. Bring a lighted match stick near the mouth of the test tube, it gets immediately extinguished,
iv. Add 2-3 ml of lime water to the gas jar. The lime-water turns milky. This is due to the formation of insoluble calcium carbonate.

$$Ca(OH)_2 + CO_2 \longrightarrow CaCO_3\downarrow + H_2O$$

1.5.2 Physical Properties

- Carbon dioxide is a colourless and odourless gas.
- It is denser than air.
- It is fairly soluble in water.

1.5.3 Chemical Properties

i) Carbon dioxide dissolves in water to form carbonic acid, which turns blue litmus red.

$$H_2O + CO_2 \longrightarrow \underset{\text{Carbonic acid}}{H_2CO_3}$$

ii) When carbon dioxide is passed through lime water, it turns milky due to the formation of insoluble calcium carbonate.

$$Ca(OH)_2 + CO_2 \longrightarrow CaCO_3\downarrow + H_2O$$

On passing more CO_2, the milkyness disappears due to the formation of soluble calcium bicarbonate.

$$CaCO_3 + H_2O + CO_2 \longrightarrow Ca(HCO_3)_2$$

1.6 PREPARATION OF HYDROGEN SULPHIDE (H_2S) GAS

Hydrogen sulphide is prepared in the laboratory by the action of dilute sulphuric acid on iron sulphide.

$$FeS(s) + H_2SO_4\ (Dil.) \longrightarrow FeSO_4\ (aq) + H_2S(g)$$

Materials Required

(1) Apparatus	(2) Chemicals
Round-bottom flask, Thistle funnel,	i) Ferrous sulphide
Gas jar, Delivery tube	ii) Dilute Sulphuric acid

1.6.1 Procedure

Fix up the apparatus as shown in the Fig. 1.4 take a few small size pieces of iron sulphide (ferrous sulphide) in a round bottom flask. Moisten them with water. Add 2-3ml. of dilute sulphuric acid into the bottle through thistle runnel. The reaction takes place immediately and hydrogen sulphide evolves. The gas is collected by an upward displacement of air. Collect the gas in gas jars and make the following observations.

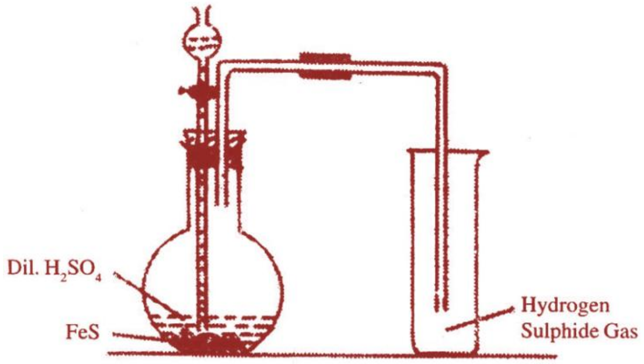


Fig. 1.4 : *Preparation of hydrogen sulphide gas*

i) Note the colour of the gas. it is colourless.
ii) Carefully smell the gas. The smell is that of rotten eggs.
iii) Add 2-3 mL of lead acetate solution to the gas jar. A black precipitate of PbS(s) is obtained.

$$H_2S + Pb\ (CH_3COO)_2 \longrightarrow \underset{\text{Lead Sulphide}}{PbS(s)} + 2CH_3COOH$$

1.6.2 Physical Properties

- Hydrogen sulphide is a colourless gas.
- It has the smell of rotten eggs.
- It is heavier than air. The density is 1.538g/cm^3
- It is sparingly soluble in water.
- Hydrogen sulphide is combustible but does not support combustion.

1.6.3 Chemical Properties

i) Hydrogen sulphide burns in air with a blue flame leaving a deposit of sulphur.

$$H_2S + O_2 \longrightarrow 2H_2O + 2S$$

ii) Hydrogen sulphide is soluble in water and gives a weakly acidic solution.

$$H_2S(g) + H_2O(I) \longrightarrow \underset{\text{Hydronium Ion}}{H_3O^+(aq)} + \underset{\text{Hydrogen Sulphide Ion}}{HS^-(g)}$$

$$HS^- + H_2O \longrightarrow H_3O^+ + \underset{\text{Sulphide ion}}{S^{2-}}$$

iii) Hydrogen sulphide gives two types of salts - (hydrogen sulphides and sulphides). When reacted with alkali like NaOH the following reactions take place. With excess of H_2S, Na_2S is the main product.

$$H_2S + NaOH \longrightarrow \underset{\text{Sodium hydrogen sulphide}}{NaHS} + H_2O$$

$$H_2S + NaHS \longrightarrow \underset{\text{Sodium sulphide}}{Na_2S} + 2H_2O$$

iv) Hydrogen sulphide reduces concentrated sulphuric acid to sulphur. The reaction takes place is two steps.

$$H_2SO_4 + H_2S \longrightarrow 2H_2O + SO_2$$

$$2H_2S + SO_2 \longrightarrow 2H_2O + 3S$$

v) Hydrogen sulphide reacts with bromine to form hydrogen bromide

$$\underset{\text{Reddish brown}}{Br_2} + H_2S \longrightarrow \underset{\text{coloulress}}{2HBr} + S$$

1.7 PREPARATION OF CHLORINE (Cl_2) GAS

Chlorine gas is prepared in the laboratory by heating concentrated hydrochloric acid with manganese dioxide.

$$4HCl\ (conc.) + MnO_2\ (s) \xrightarrow{\Delta} MnCl_2(aq) + 2H_2O(1) + Cl_2\ (g)$$

Materials Required

(1) Apparatus	(2) Chemicals
Round bottom flask (100 mL), Delivery tube, Gas jar, Burner	i) Cone, hydrochloric acid (HCl) ii) Manganese dioxide (MnO_2)

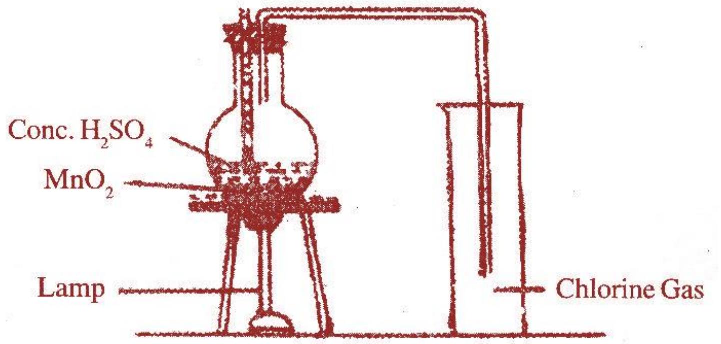


Fig. 1.5: *Preparation of Chlorine gas*

i. Note the colour of the gas. It is pale green in colour.

ii. Carefully smell the gas. It has pungent smell.

iii. When chlorine is passed in cold dilute sodium hydroxide solution containing 1 drop phenophthalein indicator, its pink colour disappears due to the formation of sodium chloride and sodium hypochlorite.

$$2NaOH(aq) + Cl_2(g) \longrightarrow NaCl\ (aq) + NaOCl(aq) + H_2O(1)$$

iv. It displaces bromine and iodine from their salt solutions.

a) $2KBr(aq) + Cl_2(g) \longrightarrow 2KCl\ (aq) + Br_2\ (g)$ (Brown)

b) $2KI(aq) + Cl_2(g) \longrightarrow 2KCl\ (aq) + I_2\ (g)$ (Violet)

1.7.2 Physical Properties

- Chlorine is a greenish-yellow gas with a strong pungent smell.
- It is soluble in water. Its water solution is known as chlorine water.
- It is heavier than air. Its density is 3.214 gm/cm^3.

1.7.3 Chemical Properties

i) Many metals react with chlorine gas and form respective metal chlorides.

$$Mg(s) + Cl_2(g) \longrightarrow MgCl_2(s)$$

ii) Chlorine water oxidises the vegetable colouring matter to colourless substances and is called bleaching agent. This is due to the formation of nascent oxygen in water:

$$Cl_2 + H_2O \longrightarrow 2HCl + \underset{\text{Nascent Oxygen}}{(O)}$$

iii) Chlorine has a high affinity for hydrogen.

iv) Chlorine readily reacts with phosphorous and gives phosphorous pentachloride.

$$P_4(s) + 10Cl_2(g) \longrightarrow 4PCl_5(s)$$

1.8 PREPARATION OF HYDROGEN CHLORIDE (HCl) GAS

Hydrogen chloride gas can be prepared by the reaction of conc. sulphuric acid on common salt (sodium chloride).

$$NaCl(s) + H_2SO_4 \longrightarrow NaHSO_4 + \underset{\text{Colourless}}{HCl(g)}$$

Materials Required

(1) Apparatus	(2) Chemicals
Round bottom flask, Thistle funnel, Gas jar, Delivery tube	i) Sodium chloride ii) Conc. sulphuric acid

1.8.1 Procedure

Set up the apparatus as shown in Fig. 1.6. Take 2-3 grams of common salt in 250 mL round bottom flask. Add 5 mL of concentrated sulphuric acid through the thistle funnel and heat the mixture gently. Collect the gas by the upward displacement of air.
Fill few gas jars with HCl gas, perform the following tests and note your observations.

i) Note the colour of the gas. It is colourless.
ii) Carefully smell the gas. It has pungent suffocating smell.
iii) Bring a glass rod dipped in ammonium hydroxide solution near the mouth of test tube.

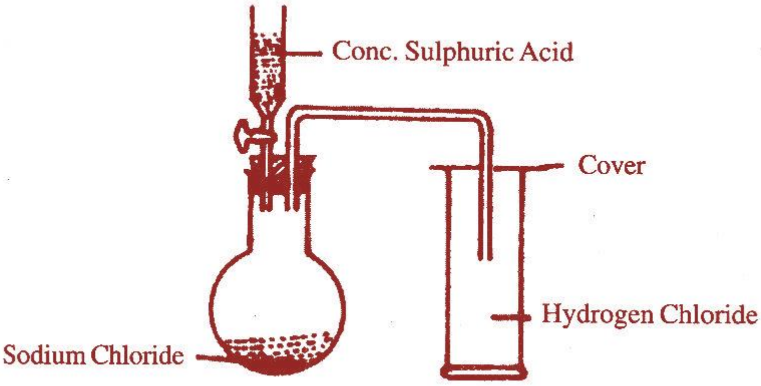


Fig. 1.6: *Preparation of Hydrogen Chloride gas*

Dense white fames of ammonium chloride are formed

$$HCl(g) + NH_3 \longrightarrow \underset{\text{Ammonium Chloride}}{NH_4Cl(s)}$$

1.8.2 Physical Properties

- Hydrogen chloride is a colourless gas.
- It has a pungent and chocking odour.
- It is heavier than air. The density is 1.639 g/cm^{-3}
- It fumes in moist air due to the formation of small droplets.

1.8.3 Chemical Properties

i) Hydrogen chloride gas is acidic in water. It turns blue litmus red. It reacts with ammonia and form ammonium chloride.

$$NH_3(g) + HCl(g) \longrightarrow NH_4Cl$$

ii) It reacts with silver nitrate solution and forms, a white curdy precipitate of silver chloride (AgCl).

$$Ag^+ + HCl \longrightarrow AgCl + H^+$$

iii) Water solution of hydrogen chloride reacts with magnesium to librate hydrogen gas.

$$Mg(s) + 2HCl\,(aq) \longrightarrow MgCl_2 + H_2\uparrow$$

1.9 PREPARATION OF SULPHUR DIOXIDE (SO_2) GAS

Sulphur dioxide gas can be prepared in the laboratory by the action of dilute hydrochloric acid on sodium sulphite.

$$Na_2SO_3(s) + 2HCl(l) \longrightarrow 2NaCl(s) + H_2O(l) + SO_2(g)$$

Materials Required

(1) Apparatus	(2) Chemicals
Round bottom flask (100 mL), Thistle tunnel, Gas jar, Delivery tube	i) Sodium Sulphite ii) Dilute hydrochloric acid

1.9.1 Procedure

Set up the apparatus as shown in Fig. 1.7. Take 1-2 grams of sodium sulphite in a 100 mL round bottom flask and add 5 mL hydrochloric acid from thistle funnel dropwise.

$$Na_2SO_3 + 2HCl \longrightarrow 2NaCl + SO_2 + H_2O$$

Fill few gas jars with SO_2 gas, perform the following tests and note your observations.

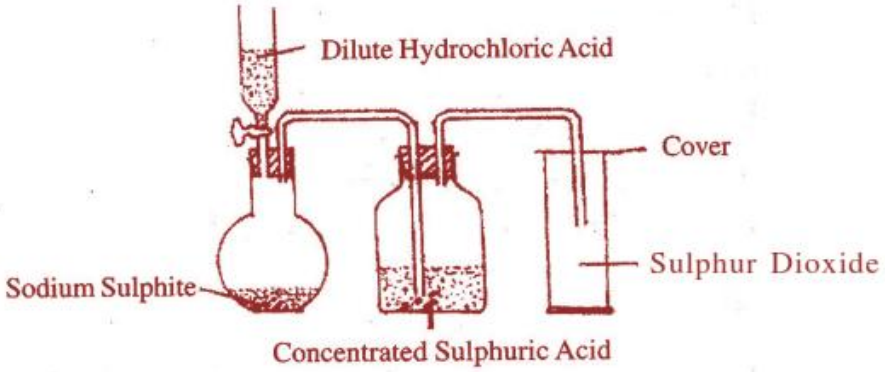


Fig. 1.7: *Preparation of Sulphur dioxide gas*

i. Note the colour of gas. It is colour less,
ii. Carefully smell the gas. It has smell of burning sulphur.
iii. Drop a piece of filter paper dipped in acidified potassium dichromate solution into jar. The filter paper turns green from organe.

$$K_2Cr_2O_7 + H_2SO_4 + 3SO_2 \longrightarrow K_2SO_4 + Cr_2(SO_4)_3 + H_2O$$

1.9.2 Physical Properties

- Sulphur dioxide is a poisonous, colourless gas.
- It has a characteristic choking smell of burning sulphur.
- It is denser than air. Its vapour density is 32 compared with that of air which is 14.2 unit.
- It is highly soluble in water and forms an acidic solution.

1.9.3 Chemical Properties

i) Sulphur dioxide is an acidic oxide. Its solution in water is highly acidic in nature.

$$SO_2 + H_2O \longrightarrow \underset{\text{Sulphurous acid}}{H_2SO_3}$$

Sulphur dioxide acts as a reducing agent as well as an oxidising agent.

ii) **Reducing Properties of Sulphur dioxide**

a) Sulphur dioxide reduces bromine to bromide ion

$$Br_2\,(aq) + SO_2\,(g) + 2H_2O \longrightarrow \underset{\text{Hydrogen bromide}}{2HBr} + H_2SO_4\,(aq)$$

b) Sulphur dioxide reduces iron (III) to iron (II) ions

$$\underset{\text{Iron (III) Chloride}}{2FeCl_3\,(aq)} + SO_2\,(aq) + 2H_2O \longrightarrow \underset{\text{Iron (II) Chloride}}{2FeCl_2\,(aq)} + H_2SO_4(aq) + 2HCl$$

c) Sulphur dioxide decolourises purple potassium permanganate (VII) solution in acidic medium, due to the reduction of permanganate ions, MnO^-_4, to manganese (II) ions, Mn^{2+} .Manganese is in +7 oxidation state in MnO^-_4 ion.

$$2MnO^-_4 + 8H^+ + 5e^- \longrightarrow Mn^{2+} + 4H_2O]\ x\ 2 \qquad \text{(i)}$$

$$SO_2 + 2H_2O \longrightarrow SO_4^{2-} + 2e^- + 4H^+]\ x\ 5 \qquad \text{(ii)}$$

On adding equation (i) and (ii) and cancelling the common species on both the sides. We get the overall equation as follows:

$$\underset{\text{Purple}}{2MnO^-_4} + 5SO_2 + 2H_2O \longrightarrow \underset{\text{Colourless}}{2Mn^{2+} + 5\,SO_4^{2-}} + 4H^+$$

d) When sulphur dioxide is bubbled through an acidified solution of dichromate ions, $Cr_2O_7^{2-}$, the colour of the solution changes from orange to green due to the formation of chromium (III) ions, Cr^{3+}. In $Cr_2O_7^{2-}$ ion, chromium is in +6 oxidation state.

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

$$SO_2 + 2H_2O \longrightarrow SO_4^{2-} + 4H^+ + 2e^-]\ x\ 3 \qquad \text{(iv)}$$

On adding equation (iii) and (iv) and cancelling the common species, on both the sides of arrow, the overall equation is

$$\underset{\text{Orange}}{Cr_2O_7^{2-}} + 2H^+ + 3SO_2 \longrightarrow \underset{\text{Green}}{2Cr^{3+}} + H_2O + 3SO_4^{2-}$$

iii) Oxidising Properties of Sulphur Dioxide

a) Suphur dioxide oxidises those substances which are stronger reducing agents than it, e.g. magnesium is oxidised to magnesium oxide.

$$2Mg(s) + SO_2\,(g) \longrightarrow 2MgO(s) + S(s)$$

b) When hydrogen sulphide is bubbled through a solution of sulphur dioxide, a pale yellow precipitate of sulphur is formed due to the oxidation of the hydrogen sulphide to sulphur.

$$2H_2S\,(g) + SO_2(aq) \longrightarrow 2H_2O(l) + 3S(s)\downarrow$$

1.10 GENERAL PRECAUTIONS

i. Wherever required, chemicals should be dried properly.
ii. Wherever water is to be used, distilled water should be used.
iii. Some of the reactions are explosive in nature; care should be taken while carrying out such reactions.
iv. Contamination of chemicals should be avoided.
v. All the reactions should be performed in a fume cupboard.

vi. In the preparation of hydrogen gas, there should be no flame near the apparatus because when air and hydrogen come in contact, explosion takes place.
vii. Fix the corks tightly in the mouth of the Woulfe's bottle or round bottom flask and make the joints airtight by sealing them with wax.
viii. Smell the gases by puffing with hand.
ix. Heat the reaction mixture gently, wherever required.

1.11 CHECK YOUR UNDERSTANDING

Q.1 What happens when CO_2 is bubbled through lime water?

....

Q.2 How would you show that carbon dioxide is acidic in nature?

....

Q.3 Name the physical property of chlorine gas which can help in its identification.

....

Q.4 What is the role of MnO_2 in the preparation of chlorine gas ?

....

Q.5 Describe any one method of laboratory preparation of hydrogen gas ?

....

Q.6 Compare the density of hydrogen gas with air and tell which one is heavier?

....

Q.7 Name the type of compound formed by the reaction of metal and hydrogen gas?

....

Q.8 What are the physical properties of oxygen gas?

....

Q.9 How is carbon dioxide prepared in the laboratory? Write the reaction?

....

Q.10 Can we use dilute H_2SO_4 in place of dil. HCl in the preparation of carbon dioxide? Give reason.

....

1.12 NOTE FOR THE TEACHER

When students perform these experiments, the teacher should ensure that the students are using proper set up of the apparatus. They should also see that students are using chemicals of required purity. While collecting gases, it should be properly collected as stated under individual gas preparation and the gas should not be allowed to leak in the laboratory

1.13 CHECK YOUR ANSWERS

Ans. 1: The lime water, Ca(OH) turns milky due to the formation of insoluble calcium carbonate, $CaCO_3$.

$$Ca(OH)_2 + CO_2 \longrightarrow CaCO_3 + H_2O$$

Ans. 2: To a jar containing CO_2 gas, put a piece of moise blue litmus. A change of colour from blue to red indicated acidic character of the gas.

Ans. 3: i) Greenish - yellow colour

ii) Pungent-suffocating odour

Ans. 4: It acts as a catalyst.

Ans. 5: Hydrogen gas is prepared in the laboratory when granules of zinc react with cold dilute sulphuric acid.

$$Zn + H_2SO_4 \longrightarrow ZnSO_4 + H_2 \uparrow$$

Ans. 6: i) Air is denser than hydrogen gas and air is heavier too.

Ans. 7: The reaction of metal and hydrogen gas gives the metal hydride.

Ans. 8: i) Oxygen is a colourless and odourless gas

ii) It is heavier than air.

iii) It is fairly soluble in water.

Ans. 9: Carbon dioxide is prepared in the laboratory by the action of dilute hydrochloric acid on marble chips.

$$CaCO_3 + \text{Dil. HCl} \longrightarrow CaCl_2 + CO_2 + H_2O(l)$$

Ans. 10: No, Sulphuric acid cannot be used because the reaction remains incomplete as the reaction between sulphuric acid and marble chips gives calcium sulphate which being insoluble, deposits, on the marble chips and hence the reaction is left incomplete.

$$CaCO_3 + H_2SO_4 \longrightarrow CaSO_4 + H_2O + CO_2\uparrow$$

EXPERIMENT-2

To prepare dilute solutions of given concentrations of hydrochloric acid, sulphuric acid and nitric acid from their stock solution.

2.1 OBJECTIVES

After performing this experiment, you should be able to:

- Explain the meaning of the terms saturated solution, standard solution and dilution;
- Use proper glassware to prepare solutions of different dilutions; and
- Prepare solutions of given concentrations by dilution.

2.2 WHAT YOU SHOULD KNOW

The most commonly used acids in laboratories are hydrochloric, sulphuric and nitric acids. The concentrations of these commercially available acids are 12M, 18M and 16M respectively. But expect a few cases; we need these acids in lower concentrations. The acids in their lower concentrations can be obtained by dilution of concentrated acids.

To make a solution of required concentration, we use the standard relation

$$M_1 V_1 = M_2 V_2$$

Where, M = molarity of concentrated acid, and V_1 = Volume of concentrated acid.

M_1 = Molarity of acid required, V_2 = Volume of acid required.

2.3 MATERIALS REQUIRED

Materials Required

(1) Apparatus	(2) Chemicals
Standard flask (100mL)	Hydrochloric acid (12M)
Funnel, Glass rod, Wash bottle	Sulphuric acid (18M), Nitric acid (16M)
Pipette and burette	and Distilled water.

2.4 HOW TO PERFORM THE EXPERIMENT

You are required to prepare

1. 100 mL of 1 molar solution of hydrochloric acid.
2. 100 mL of 0.5 molar solution of sulphuric acid and
3. 100 mL of 2 molar solution of nitric acid from the commercially available acid.

For preparing 100mL of 1 molar hydrochloric acid we use the equation, $M_1V_1 = M_2V_2$, We first calculate the volume of concentrated acid and water required.

M1 = Molarity of concentrated acid = 12M
V1 = Volume of concentrated acid = ?
M2 = Molarity of acid required = 100mL
V2 = Volume of acid required = 100mL

$$V_1 = \frac{M_2V_2}{M_1} = \frac{M_2V_2}{M_1} = 8.33\text{ml} \approx 8.3\text{ ml}$$

Take about 50mL of distilled water in a 100mL standard flask. Transfer 8.3 mL of concentrated acid from a burette to the standard flask. Shake the mixture gently. Add water to make up the volume of acid in the standard flask up to the mark. Close the flask with glass-stopper. Mix the solution by tilting the flask upside down.

For the preparation of dilute solution of sulphuric and nitric acids, follow the same procedure as given above.

2.5 PRECAUTIONS

i) The acids should be used with utmost care.

ii) All the three acids are corrosive to skin.

iii) Add concentrated acids slowly to water with constant stirring.

2.6 OBSERVATIONS

i) Volume of 12M HCl taken for preparing 1 M HCl, (100mL solution = 8.3 mL)

ii) Volume of 18M H_2SO_4, taken for preparing 0.5M, H_2SO_4 (100 mL solution) = ...

iii) Volume of 16M HNO_3, taken for preparing 2M, HNO_3 (100 mL solution) = ...

2.7 CONCLUSION

The solution of required concentrations may be prepared from their concentrated solution by the dilution method.

2.8 CHECK YOUR UNDERSTANDING

1. What are the components of a solution?

....

2. What do you understand by dilution?

....

3. What is molarity?

....

4. Calculate the volume of 8M HCl required preparing 250ml of 2M hydrochloric acid.

....

2.9 NOTE FOR THE TEACHER

All the three concentrated acids are highly corrosive. One should be careful while handling these acids.

The students need help in transferring acids from the commercial bottles into reagent bottles. The students should not be allowed to take acids from the commercial bottles.

2.10 CHECK YOUR ANSWERS

Ans. 1 There are two component in a solution (a) the solvent, and (b) solute.

Ans. 2 Dilution is a physical process involving addition of a solvent to the concentrated solution.

Ans. 3 Molarity is the number of moles of solute present in 1 litre of the solution.

Ans. 4 $M_1 V_1 = M_2 V_2$

$$V_1 = \frac{M_2V_2}{M_1}$$

$$V_1 = \frac{2 \text{ x } 150}{8} = 62.5 \text{ mL.}$$

EXPERIMENT-3

To study the interaction of metals with salt solutions of other metals and arrange them according to their reactivity. Metals and salts may be selected from the metals like Mg, Zn, Fe, Sn, Pb, Cu and Ag and their salts.

3.1 OBJECTIVES

After performing this experiment, you should be able to:

- identify the change that is taking place during reaction (colour change, change in colour intensity, etc.);
- interpret the observations in terms of the chemicals reactions taking place;
- write chemical equations of the reactions involved;
- differentiate between a less active metal from a more active metal;
- list the given metals in the order of their increasing activity; and
- predict the probable changes on reacting a known metal with a known salt solution.

3.2 WHAT YOU SHOULD KNOW

Compounds are ionic or covalent in nature. Salts are ionic compounds which dissolve in water to give cations (positively charged ions) and anions (negatively charged ions). When a metal piece is dipped in an aqueous solution of salt of another metal, the cation of the salt may be replaced by cation of the metal which is dipped in it if is more active. The displaced cation would change to metal atom and deposit on the dipped metal.

3.3 MATERIALS REQUIRED

(1) Apparatus	(2) Chemicals
Test tube stand, Test tubes, Burner Beaker, Tripod stand, Wire gauze, Glass rod	Mg, Zn, Fe, Sn, Pb and Cu metals and their water soluble salts. MgCl Mg $(NO_3)_2$, $Zn(NO_3)_2$, $ZnCl_2$, $Pb(NO_3)_2$, $CuSO_4$, $Cu(NO_3)_2$

3.4 HOW TO PERFORM THE EXPERIMENT

Take four test tubes and mark them (1), (2), (3), (4). Fill them half with 10% solution of a magnesium salt. Place them in the test tube stand as shown in Fig. 3.1

Add a small piece of magnesium ribbon to the first test tube, a small quantity of zinc granules to the second, iron filings to the third and copper filings to the fourth test tube. Observe the test tube carefully and record the observation in Table 3.1.

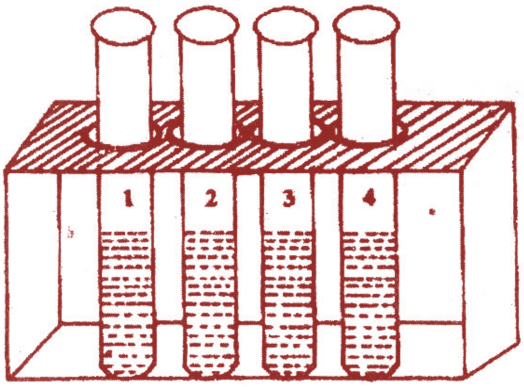


Fig. 3.1 : *Study of activity of different metals*

Repeat the same experiment using the solutions of pits of Cu, Fe (ferrous) and Zn respectively with the four metals as done earlier. In each case, observe carefully and record your observations in Table 3.1

Mark tick (✔) for the reaction taking place between the metal and the salt solution and cross (x) for the reaction not taking place by observing change in any property of solution.

3.5 PRECAUTIONS

1. The pieces of the metals should be clean.
2. Do not use very active metals like sodium, potassium, lithium or calcium.
3. Aluminium may be avoided as it forms a protective layer which makes the metal passive, whereas the metal is not passive.

3.6 OBSERVATIONS

Record your observations in the following table :

Table 3.1

Solid Metal	Metal ions in solution				Remarks if any
	Mg^{2+}	Cu^{2+}	Fe^{2+}	Zn^{2+}	
Mg	-				
Cu		-			
Fe			-		
Zn				-	

3.7 CONCLUSION

If the reaction takes place, it indicates that the metal added is more active than the metal in solution. We show it by marking (✔) sign. If the reaction does not take place, we conclude that the metal added is less reactive than the metal in solution. We show this by marking (x) in the table.A metal having maximum no. of (✔) marks is the most active. Number of (✔) marks shows the extent of acitivity.

Thus a list of metals in order of their decreasing activity may be prepared. The order of activity of the metals is _________> ________> _______> ________> _______

3.8 CHECK YOUR UNDERSTANDING

1. State a necessary condition for the reaction of a metal with the solution of salt of another metal.

....

2. State the hazard involved in using a very reactive metal like potassium in a displacement reaction.

....

3. List the probable observations from which you conclude that the reaction has taken place.

....

4. Write the chemical equations for the reactions taking place when magnesium ribbon is dipped in (i) zinc nitrate solution (ii) copper nitrate solution and (iii) iron sulphate solution.

1...

2..

3..

NOTE FOR THE TEACHERS

As far as possible the four metals and their salts should be carefully selected to show a difference of activity. Metals having very close activity should not be given. Highly active metals such as sodium should not be used in this experiment. Although the procedure mentions Cu, Fe, Zn and Mg metals, you may select any four out of the given list and suitably modify the procedure and observation table.

Students should be helped to draw conclusions on the basis of the recorded data. Projects can be assigned to students to record observations of such reactivities in daily life. The importance of such reactions in daily life should be highlighted.

3.10 CHECK YOUR ANSWERS

Ans. 1. The dipped metal should be more reactive of the two.

Ans. 2. Very active metals, such as sodium and potassium, react with water vigorously and may even catch fire with an explosion.

Ans. 3. a) Development of colour

b) Disappearance of colour

c) Change in colour intensity

Ans. 4.

$Mg(s) + Zn(NO_3)_2\,(aq) \rightarrow Mg(NO_3)_2\,(aq) + Zn(s)$

$Mg(s) + Cu\,(NO_3)_2\,(aq) \rightarrow Mg(NO_3)_2\,(aq) + Cu(s)$

$Mg(s) + FeSO_4\,(aq) \rightarrow MgSO_4\,(aq) + Fe(s)$

EXPERIMENT-4

a) To determine the pH of the following substances by using a universal indicator solution or pH paper.

i) Salt solution
ii) Acids and bases of different dilutions
iii) Vegetables and fruit juices

b) To study the pH change by common ion effect in case of weak acids and weak bases using universal indicator solution or pH paper.

4.1 OBJECTIVES

After performing this experiment, you should be able to:

- find the pH of solutions of different substances by using universal indicator solution or pH paper;
- classify various substances as neutral, acidic or alkaline;
- explain the nature of substances on the basis of their pH values;
- observe changes in pH of a weak acid when a compound containing common anion with the weak acid is added to it;
- observe change in pH of weak base when a compound containing common cation with the weak base is added to it; and
- explain the significance of buffer solution.

4.2 WHAT YOU SHOULD KNOW

Solutions of weak acids and bases in water have hydronium ions $[H_3O^+]$ and hydroxyl ions $[OH^-]$ in different 'Concentrations. Acidic solutions have $[H_3O^+]$ ions greater than 10^{-7} mol L^{-1} and alkaline solutions have $[OH^-]$ ions than 10^{-7} mol L^{-1} at 25^0C. A neutral solution, or pure water pure water has $[H_3O^+] = [OH^-]$. For expressing the concentration of H_3O^+ in a solution, the negative powers of 10 are involved. This inconvenient use of figures involving negative powers of 10 can be avoided by using a concept of pH scale. The pH of a solution is defined as the negative logarithm to the base 10 of the hydronium ion concentration.

$$\text{pH} = -\log [H_3O^+]$$

The pH acidic solutions is less than 7, that of basic solution, is greater than 7 and the neutral solutions have pH = 7.

pH of a basic solution can be calculated by using the relation,

$$\text{pH} = 14 \log_{10} [OH^-]$$

pH is accurately measured by a pH meter, but a reasonably good estimate can be made with the help of universal indicator solution or pH paper. These have characteristic colours depending upon the pH of the solution.

Now you would like to see the pH change of solution of weak acid or weak base, when their salt is added to the solution. Weak acids and bases do not dissociate completely. An equilibrium exists between the dissociated and undissociated molecules. For example, for a weak acid HA, you can write the equilibrium as follows,

$$HA \rightleftharpoons H^{+} + A^{-}$$

$$Ka = \frac{[H^{+}]\,[A^{-}]}{[HA]}$$

$$NaA \longrightarrow Na^{+} + A^{-}$$

This equilibrium is characterised by a constant, K_a. Addition of a salt containing a common ion, say NaA to its solutions causes the equilibrium to shift to the left (Le Chatterlier's principle). As a consequence, the concentration of hydrogen ions gets reduced and the pH gets increased. You would study this phenomenon (common ion effect) by using acetic acid as the weak acid and sodium acetate as its salt. Similar arguments hold true for the ionization of weak base (e.g. NH_4OH). You can check this adding NH_4Cl to NH_4OH solution (a weak base and its salt). Such mixtures of weak acid or weak bases with their respective salts are called as Buffer Solution. pH of an acidic buffer can be calculated by using the following equation

$$pH - pKa + \log \frac{[Salt]}{[Acid]}$$

K_a is the acid dissociation constant and pK_a is the negative logarithm of K_a.

pH for a basic buffer (a weak base and its salt) can be calculated by using the following equation.

$$pH = 14 - pKa - \log \frac{[Salt]}{[Base]}$$

K_b is the base dissociation constant and pK_b is the negative logarithm of K_b.

pK_a for acetic acid at $25^{0}C = 4.76$

pK_b for ammonium hydroxide at $25^0C = 4.75$

4.3 MATERIALS REQUIRED

(1) Apparatus	(2) Chemicals
Test tube - 6, Test tube stand Spatula, Measuring cylinder (25mL) and a Dropper.	Dilute acid and base solutions, Neutral solution (NaCl), Solution of hydrolysable salts like $CuSO_4$, NH_4Cl, CH_3COONa, fruit and vegetable juices, pH paper or universal indicator solution with colour chart, Acetic acid (0.1M), and sodium acetate.

4.4 HOW TO PERFORM THE EXPERIMENT

a) For determining the pH of a given solution proceed as follows :

Take six test tubes and label them 1 to 6. If you are using universal indicator solution, then take 3-4 mL each of the test solution (about one fourth of test tube) into separate labelled test tubes. To all the test tubes, add 4 to 5 drops of the universal indicator solution and observe the appearance of colour, if any. If you are using pH paper, add 1-2 drop each of the test solution with the help of a dropper on a pH paper strip. (Alternatively, you may dip the pH paper strips into each of the test solution). Observe the colour. Compare the colour of the solution in the test tubes or on the pH papers with the standard colour chart as provided to you to find the pH. Record your observations in Table 4.1 given in section 4.6. Classify the solutions as neutral, acidic or alkaline.

b) To study the pH change due to common ion effect proceed as follows :

Take 5 labeled test tubes and place them in a test tube stand. Take 5mL of given acetic acid solution in each of the labeled test tubes. Add 0.5g. 1.0g. 1.5g and 2.0 g of sodium acetate to test tube No. 2.3,4 and 5 respectively and dissolve to get clear solutions. Add 4-5 drops of the universal indicator solution to each of the test tubes. In case of pH paper, add 1-2 drops of the solutions to pH papers separately with the help of separate dropper. (Alternatively, you may dip the pH paper strips into each of the test solution). Compare the colours in test solution or on pH paper developed with the colour chart and record the corresponding pH values, in Table 4.2 of section 4.6. Calculate the pH of each buffer solution you have prepared using appropriate equation and list in the Table 4.2.

You may try the same procedure given above at (b) to study the pH in case of basic buffer using ammonium hydroxide as a base and ammonium chloride as its salt.

4.5 PRECAUTIONS

1. Compare the colour which appears on pH paper with the standard colour chart carefully.
2. Don't add too much of the indicator solution. You may follow the instruction given on the label of the indicator bottle.

4.6 OBSERVATIONS

Table 4.1 : pH Values of different test solutions

S.No.	Solution	pH Value
1.		
2.		
3.		
4.		
5.		
6.		

Table 4.2 : pH Values of different combinations of CH_3COOH and CH_3COONa

S.No.	Vol. of acetic acid (mL)	Amount of sodium	pH Observed	Calculated pH
1.	5.0	0.0		
2.	5.0	0.5		
3.	5.0	1.0		
4.	5.0	1.5		
5.	5.0	2.0		

4.7 CONCLUSIONS

a) pH of solution 1 is .... and therefore, it is acidic/basic/neutral
pH of solution 2 is .... and therefore, it is acidic/basic/neutral
pH of solution 3 is .... and therefore, it is acidic/basic/neutral
pH of solution 4 is .... and therefore, it is acidic/basic/neutral
pH of solution 5 is .... and therefore, it is acidic/basic/neutral
pH of solution 6 is .... and therefore, it is acidic/basic/neutral

b) pH of acetic acid solution increase / decreases / remains unchanged on adding increasing amounts of sodium acetate to it.

4.8 CHECK YOUR UNDERSTANDING

1. Calculate the pH of 10^{-2} M HCl solution and 10^{-2} M NaOH solution ?

....

2. The pH of an aqueous sodium chloride solution is 7. Explain, what does it mean ?

....

3. Why does copper sulphate solution (a slat solution) has a pH other than 7 ?

....

4.9 NOTE FOR THE TEACHERS

For acetic acid-sodium acetate experiment, dilute 3 mL of glacial acetic acid with water to get 100 mL solution in measuring cylinder.

4.10 CHECK YOUR ANSWERS

Ans. 1: pH = 2 for 10^{-2} M HCl pH = 2 for 10^{-2} M NaOH

Ans. 2: NaCl is a salt of strong acid (HCl) and a strong base (NaOH). In solution it dissociates completely to give sodium and dichloride ions which do not hydrolyse. In aqueous solutions the hydrogen ions come only from water and have a concentration of 10^{-7} M hence the pH = 7.

Ans. 3: $CuSO_4$ solution will have a pH which is less than 7, because in water solution, $CuSO_4$ will hydrolyse to give strong acid (H_2SO_4) which makes the solution acidic in nature.

EXPERIMENT-5

To determine the melting point of a solid substance having low melting point

5.1 OBJECTIVES

After performing this experiment, you should be able to:

- set up an apparatus to determine the melting point;
- determine the melting point of a given solid substance; and
- relate the melting point of a substance with its purity;

5.2 WHAT YOU SHOULD KNOW

The melting point of a solid is that constant temperature at which the solid and liquid phases of the substance are in equilibrium with each other.

This is an important physical constant used for establishing of the identity and determining the purity of a solid substance. A sharp melting point is usually indicative of high purity of a substance. The presence of impurities lower down the melting point. Thus, an impure solid substance has a lower melting point than that of its pure form

5.3 MATERIALS REQUIRED

(1) Apparatus	(2) Chemicals
Beaker (100 mL), Thermometer, Glass capillary tube (5-6 cm long) Burner, Iron stand, Clamp and boss, Trpod-stand, Wire gauze, Stirrer Spatula Glass tube (25-30cm), Watch glass, Cork with one bore.	Liquid paraffin (NmL) Naphthalene, Urea, Oxalic acid, Benzoic acid.

5.4 HOW TO PERFORM THE EXPERIMENT

1) Take a glass capillary tube of about 5-6 cm in length and seal one end of it. This is done by heating tip of tube with the side of the flame of the Bunsen burner. The end of the capillary tube melts and seals itself. It is indicated by the formation of a small glass bead at tip. Fig. 5.1

2) Take a small quantity of a solid (about 50mg) in watch glass, powder it by crushing it with the help of a spatula.

3) Fill the powdered substance in the capillary tube upto about 1 cm height. This is done by pushing the open-end of the capillary tube into the heap of powdered substance kept in a watch glass. Now drop the capillary tube with the seated end down through a glass- tube (25-30 cm length) kept vertically on a plane surface so that the powder reaches the closed end of the capillary.

4) Take a beaker of 100mL capacity. Fill it nearly half with paraffin oil. Place this beaker (paraffin bath) over a wire gauze kept on a tripod stand.

5) Take a thermometer and dip it in the paraffin bath so as to wet its lower end. Bring the capillary tube near to the wet end and spread a thin film of paraffin oil on one side of capillary tube. Bring the wet side of the capillary tube in contact with the thermometer. The capillary tube sticks to the thermometer due to the surface tension of paraffin. (Fig. 5.2). Now hang it on a clamp such that its bulb dips completely in the liquid paraffin but the open end of capillary is above it.

Fig. 5.1 : *Sealing of Capillary tube*

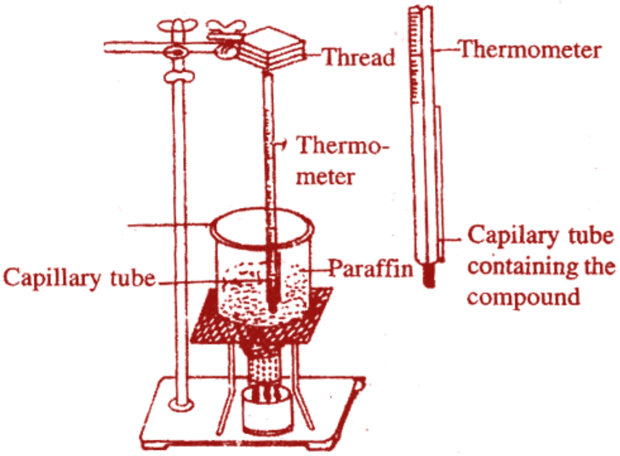


Fig. 5.2 : *Determination of melting point*

6) Heat the paraffin bath slowly with a burner and keep the oil stirring so as to maintain a uniform temperature.

7) Continue heating till the substance melts. Note down the temperature (t_1) at which the substance begins to melt. Observe carefully whether the substance melts at a fixed temperature or it melts over a range of temperature. A melting point is said to be sharp when the compound melts at a fixed temperature.

8) Remove the burner and allow the temperature to come down. Note the temperature (t_2) at which the solid just reappears.

9) The average of the readings ($t_1 + t_2$) is the correct melting point.

5.5 PRECAUTIONS

1. The substance whose melting point is to be determined should be completely dry.
2. The thermometer and the capillary should not touch the bottom or the sides of the beaker.
3. The heating of bath should be very slow with the help of a low flame so that the change in the state of the substance (solid to liquid) is clear.
4. The bath should be stirred gently to maintain a uniform temperature.

5.6 OBSERVATIONS

Temperature at which the substance begins to melt (t_1) = ... ^{0}C Temperature at which the substance resolidifies melts (t_2) = ... ^{0}C

Melting point (T_1) = ... ^{0}C

Note : In case of pure substance values t_1 and t_2 are almost the same or the difference between t_1 and t_2 is very small. In case of impure substance, t_1 and t_2 are different and the difference between t_1 and t_2 depends upon the amount of impurity.

5.7 CONCLUSION

i) The melting point of the substance is .... ^{0}C

ii) The melting point of the substance is sharp/not sharp. Hence the substance is of a pure/impure quality.

5.8 CHECK YOUR UNDERSTANDING

1. Define the melting point of a substance.

....

2. What will be the effect of impurity on melting point of a substance ?

....

3. How can you keep the temperature of the bath uniform while heating ?

....

4. What is the usefulness of determination of melting point ?

....

5. What is meant by sharp melting point ?

....

6. What will be the effect on melting point of the substance if the oil in the bath is not stirred regularly.

....

5.9 NOTE FOR THE TEACHER

Following substances may be given to the students for determination of melting point :

	Compound	M.P. (^{0}C)
1.	Naphthalene	80
2.	Citric Acid	100
3.	Oxalic Acid	101
4.	Benzoic Acid	121
5.	Urea	132

5.10 CHECK YOUR ANSWERS

Ans. 1 : Melting point of a substance is the temperature at which the solid and liquid phase of the substance are in equilibrium.

Ans. 2 : The presence of impurity lowers the melting point of a substance.

Ans. 3 : By stirring the paraffin oil gently.

Ans. 4 : To establish the identity and determining the purity of a solid substance.

Ans. 5 : The temperature at which a solid melts almost completely.

Ans. 6 : The lower part of the oil will have higher temperature than upper part and thus observed melting point will differ from the actual value.

EXPERIMENT-6

To study the solubility of solid substances in water at different temperatures and to plot the solubility curve.

6.1 OBJECTIVES

After performing this experiment, you should be able to:

- set up the apparatus for studying the solubility of different substances at different temperatures;
- determine the solubility of substances at different temperatures
- draw a solubility curve;
- read solubility value from the curve at different temperatures;
- interpret the variation of the solubility curve of different substances at different temperatures; and
- explain the basis of fractional crystallization

6.2 WHAT YOU SHOULD KNOW

Solubility of a substance is defined as the amount of the substance dissolved in 100g of the solvent so as to give a saturated solution at a given temperature. The variation of solubility with temperature is quite different for different substances. For some, salts, for example for NaCl, there is hardly any change in solubility with increase in temperature. For some, such as potassium nitrate, the change in solubility with temperature is very rapid, while for others such as potassium iodide, the change is very slow. This difference in solubility behaviour forms the basis for fractional crystallization of substances. This technique is used for the separation of impurities present in any substance.

It is quite convenient to express the solubility of substances at different temperatures by plotting a graph called solubility curve.

When a solid dissolves in a liquid, the process can be either endothermic or exothermic. For many ionic compounds, this process is endothermic. According to Le Chatelier's principle, the solubility of such salts will increase with increase in temperature. If the process of dissolution is exothermic, for example, for lithium sulphate (Li_2SO_4), the solubility decreases with increase in temperature. The variation of solubility with temperature of a few salt is depicted in Fig. 6.1

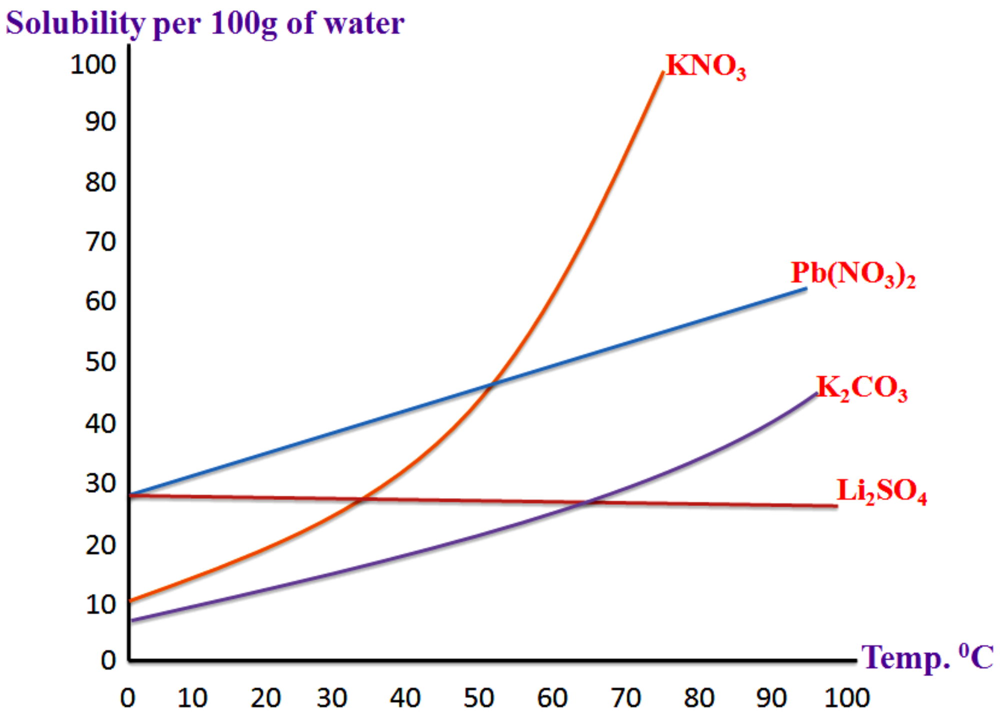


Fig. 6.1 : *Solubility Curves*

6.3 MATERIALS REQUIRED

(1) Apparatus	(2) Chemicals
Boiling tube, Glass rod, Glass water bath (glass beaker 250 mL). Thermometer, wire gauze, Tripod stand, Burner stand with clamp and boss, Spatula, stirrer	Potassium nitrate, Distilled water, Sodium nitrate

6.4 HOW TO PERFORM THE EXPERIMENT

1) Weigh 10.0 g of powdered sodium nitrate using a physical balance and pour it into the boiling tube.

2) With the help of a burette, add 7-8 mL of water into boiling tube. Stir it well with the help of stirrer. You will observe that a small amount of salt remains undissolved.

3) Now from burette, add water in very small volume say 0-1 mL, till all the salt dissolves. Stir well after each addition of water.

4) Now, add 1g of sodium nitrate to above boiling tube.

5) Set up the apparatus as shown in Figure (6.2). Note that the level of water in water bath

should be well above the level of solution in boiling tube.

6) Heat the water bath very slowly with constant stirring of solution. As soon as the salt dissolves, note down the temperature. Let us say, it is temperature, t_1.

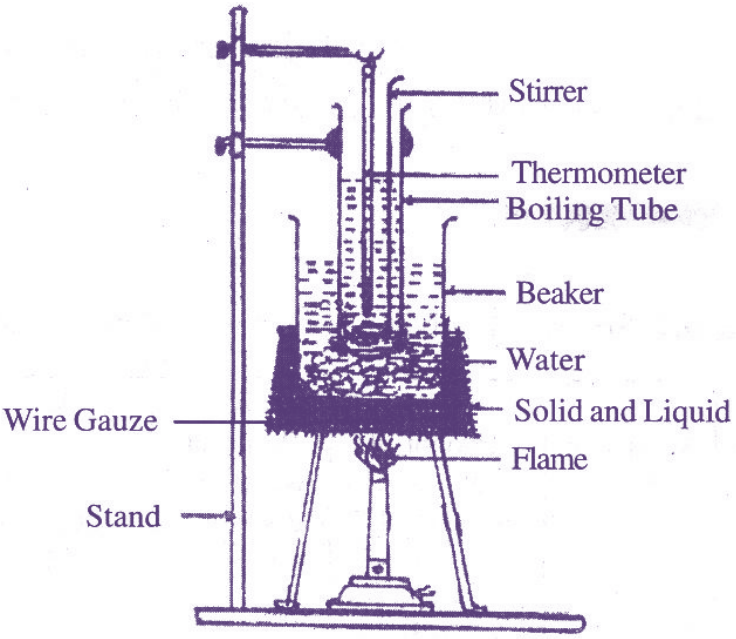


Fig. 6.2 : *Study of Solubility*

Note down the volume of water required for just dissolving the given mass of the salt. Also note down the temperature of solution. This is the solubility of salt at this particular temperature.

7) Now remove the burner, and let the solution to cool down. As soon as some solid reappears at the bottom of boiling tube, note down the temperature again. Let us say it is temperature, t_2.

8) Find out average of temperature t_1 and t_2 as $\left(\frac{t_1 + t_2}{2}\right)$. Add one more gram of sodium nitrate to the boiling tube. And repeat step number 6 and 7 again.

9) In a similar way find out solubility of sodium nitrate at 5 different temperatures.

10) Calculate the amount of $NaNO_3$ dissolved in 100mL of solvent at above recorded temperatures.

11) Record all your readings in the table 6.1 given below.

6.5 PRECAUTIONS

1) Powder the substance before use.
2) Weigh the salt as accurately as possible.
3) During heating and cooling, stir the solution gently.

4) Heat the water bath very slowly to control the rise in temperature.

6.6 OBSERVATIONS

Table 6.1 : Solubility of $NaNO_3$ at Different Temperatures

S. No.	I Quantity of $NaNO_3$ (mg)	II Volume of water (mL)	III Amount of $NaNO_3$ per 100 mL of water = solubility = (n/v) x 100g	IV Temp. at which salt dissolves t_1 (^0C)	V Temp. at which salt reappears t_2 (^0C)	VI Mean temp. $\left(\frac{t_1 + t_2}{2}\right)$ (^0C)
1.						
2.						
3.						
4.						

Plot a graph of the amount of sodium nitrate dissolved per 100mL of water versus temperature i.e. solubility as recorded in Column III and temperature as recorded in Column VI, taking solubility along x-axis and temperature along x-axis.

6.7 CONCLUSION

Solubility of sodium nitrate in water changes rapidly / slowly / does not change with temperature.

6.8 CHECK YOUR UNDERSTANDING

1. Define the term solubility

....

2. What is the advantage of solubility curves for separation of substances ?

....

3. Why do you heat the water bath slowy

....

6.9 NOTE FOR THE TEACHER

1) You can ask the students to perform this experiment with Potassium nitrate (KNO_3). They may compare the curve with that of Soidum nitrate or Ask some students to perform the experiment. With $NaNO_3$ and others with KNO_3 and compare their graphs.

2) Ask the students to find out the solubility of the substances at a given temperature from the graphs.

6.10 CHECK YOUR ANSWERS

Ans. 1 : Solubility of a substance is its amount dissolved in 100g of solvent so as to give a saturated solution at a specified temperature.

Ans. 2 : Solubility curves indicate the crystallization point at a particular temperature which can be used for separating two substances by the process of crystallization.

Ans. 3 : Rapid increase in temperature will not allow to read the temperature on the thermometer accurately.

EXPERIMENT-7

To study the shift in equilibrium between ferric ions and thiocyanate ions by increasing / decreasing the concentration of these ions.

7.1 OBJECTIVES

After peforming this experiment, you should be able to:

- perform dilution of the solutions using measuring cylinder;
- compare the intensity of colours in two solutions visually;
- predict the direction in which the equilibrium will shift if the concentration of any reactant is increased or decreased.
- predict the direction in which the equilibrium will shift, if the concentration of any product is increased or decreased.

7.2 WHAT YOU SHOULD KNOW

A reversible reaction never reaches the completion stage but results in an equilibrium state in which concentrations of all the reactants and products become constant. This equilibrium is dynamic in nature and it is the result of two opposite reactions occuring simultaneously and at the same rate. However, no 'net' change occurs in the system.

According to Le Chatelier's Principle *"when any system at equilibrium is disturbed by changing concentrations, temperature or pressure, the equilibrium shifts either in forward or backward direction and a 'net' change occurs in it so as to decrease the effect of the disturbing factor"*.

Consider the following equilibrium

$$A + B \rightleftharpoons C + D$$

The equilibrium constant for it is given by

$$K = \frac{[C]\,[D]}{[A]\,[B]}.$$

When concentration of A or B is increased, the equilibrium will shift in forward direction, so that some quantity of A and B is consumed and corresponding quantities of products C and D are formed. When concentration of A or B is decreased, (the equilibrium will shift in backward direction) Now same quantity of C and D is consumed and corresponding quantities of reactants A and B are produced. Similarly when concentration of products C or D is increased, the equilibrium will shift in the backward direction and when it is decreased it will shift in the forward direction.

Ferric ions react with thiocynate ions to form a blood red coloured complex, $[Fe(CNS)]^{2+}$, and the following equilibrium is established.

The effect of change in concentration of $Fe(aq)^{3+}$+ or CNS ions on this equilibrium can be studied easily. If this equilibrium shifts in forward direction, more quantity of the complex will be formed and the colour of the solution will become more intense.

Similarly, when the equilibrium shifts in backward direction, some quantity of the complex will break and the colour of the solution will become light.

7.3 MATERIALS REQUIRED

(1) Apparatus	(2) Chemicals
Test tube stand	0.02 M KCNS Solution
test tubes - 5	0.02 M Fe$(NO_3)_3$ Solution
Measuring cylinder - 10mL	
Measuring cylinder - 50 mL Wash bottle	
White glazed tile or a sheet of plain white paper.	

7.4 HOW TO PERFORM THE EXPERIMENT

1. Take five clean and dry test tubes, number them from 1 to 5 and keep them in a test tube stand. Mark the test tube number 3 as "Reference Tube".
2. Take 10mL Fe$(NO_3)_3$ solution (0.02 M) in a clean and dry 50mL measuring cylinder. Fill it up with distilled water upto 40mL mark and stir to mix. Take 5 mL of this diluted (0.005 M) Fe (N$_{O3)3}$ solution in each of the five test tubes.
3. In a clean and dry 10mL measuring cylinder take 10mL KCNS solution (0.02 M). Out of it, add 5mL solution to test tube, number 1.
4. The measuring cylinder now contains 5mL) of 0.02M KCNS solution. Add distilled water to make the volume upto 10mL mark in the measuring cylinder with a wash bottle and stir to mix. You get KCNS solution of 0.01M concentration. Pour 5mL of 0.01 M KCNS solution into test tube number 2.
5. Add distilled water in the remaining solution to make the volume upto 10mL which will give a solution 0.005 M and transfer 5 mL of this solution to test tube number 3.
6. Again add distilled water in the remaining solution in the test tube No. 1 to obtain a solution of 0.0025 M and transfer 5 mL of solution to test tube number 4.
7. Similarly, in order to set a solution of 0.00125 M upto 10mL in the first test tube by adding distilled water in it and transfer 5 mL of it to the fifth test tube.
8. Shake all the five test tubes to mix the solutions and keep them back on test tube stand.
9. Compare the intensity of red colour in test tube number 1 with that in 'Reference Tube' (Number 3). For this purpose, hold the two tubes against white background of a glazed tile or a plain white paper. Find out whether the colour intensity is more or less than the Reference tube and record it in Table 7.1 given in section 7.6.
10. Similary, compare the intensity of red colour in the remaining test tubes numbered 2, 4 and 5 and record the observations in Table 7.1
11. Similarly, we can study the effect of (Fe^{3+}) on the equilibrium by performing another experiment where we vary the concentration of Fe^{+++} while keeping the

concentration of (CNS^-) constant. For this, use KCNS solution in step 2 and $Fe(NO_3)_3$ solution in steps 3 to 8. Record your observations in Table 7.2 given in section 7.6

7.5 PRECAUTIONS

1. All glass apparatus (test tubes and measuring cylinders) should be thoroughly clean, otherwise the solutions will stick to their sides and not mix properly.
2. Volumes of solutions and distilled water should be measured carefully so that the solution of desired concentration can be obtained.
3. Mixing of solution and water or of two solutions should be done by gentle shaking and avoiding their splashing.

7.6 OBSERVATIONS

Table 7.1 : Effect of concentrations of CNS ion on the equilibrium

Test tube No.	Conc. of Fe $(NO_3)_3$ soln. used (mol^{-1})	Conc. of KCNS used sol. (mol^{-1})	Intensity of red colour as compared to the reference tube (more/less)	Shift in equilibrium (forward/ backward)
1.	0.005	0.02	....	....
2.	0.005	0.01	....	....
3.	0.005	0.005	Reference	....
4.	0.005	0.0025	....	....
5.	0.005	0.00125	....	....

Table 7.2 : Effect of concentrations of Fe^{3+} ion on the equilibrium

Test tube No.	Conc. of Fe $(NO_3)_3$ soln. used (mol^{-1})	Conc. of KCNS used sol. (mol^{-1})	Intensity of red colour as compared to the reference tube (more/less)	Shift in equilibrium (forward/ backward)
1.	0.02	0.005	....	....
2.	0.01	0.005	....	....
3.	0.005	0.005	Reference	....
4.	0.0025	0.005	....	....
5.	0.00125	0.005	....	....

7.7 CONCLUSIONS

1. When any reactant concentration is increased, the equilibrium shifts in ______ direction.
2. When any reactant concentration is decreased, the equilibrium shifts in ______ direction.

7.8 CHECK YOUR UNDERSTANDING

1. State Le Chalelier's Principle.

....

2. Why is the equilibrium reached by a reversible reaction called dynamic equilibrium ?

....

3. What will happen to the equilibrium by mixing Fe(NO)3 and KCNS solutions, if a few drops of $FeCl_3$ solution are added to it.

....

4. What will happen if some $[Fe(SCN)]^{2+}$ ions are removed from the above equilibrium ?

....

5. What will be the effect of addition of a few drops of dilute NH_4OH solution to the above equilibrium ? Give reasons.

....

7.9 NOTE FOR THE TEACHER

1. The teacher should help the students while doing the experiments.
2. Chemicals should be supplied in pure state.

7.10 CHECK YOUR ANSWER

Ans. 1: Le Chatelier's Principle states, when any system at equilibrium is disturbed by changing concentrations, temperature or pressure, the equilibrium shifts in a direction so as to nullify or reduce the effect of that change.

Ans. 2: It is because even after equilibrium is established, two opposite reactions continue to occur but at the same rate.

Ans. 3: The equilibrium will shift in forward direction as the concentration of reactant Fe^{3+} ion will increase on addition of FeCl .

Ans. 4: As $[Fe(SCN)]^{2+}$ ions (product) are removed the equilibrium will shift in forward direction

Ans. 5: The equilibrium will shift in back ward direction. It is because OH^- ions from NH OH will combine with some Fe^{3+} ions and form brown precipitate of $Fe(OH)_3$. Thus reactant (Fe^{3+} ions) concentration will decrease and the equilibrium will shift in backward direction.

EXPERIMENT-8

a) To study the effect of concentration on the rate of reaction between sodium thiosulphate and hydrochloric acid.

b) To study the effect of temperature on the rate of reaction between sodium thiosulphate and hydrochloric acid.

8.1 OBJECTIVES

After performing this experiment, you should be able to:

- observe the effect of concentration of reactants on rates of reactions;
- predict the effect of concentration of reactants on rates of reactions;
- observe the effect of temperature on rates of reactions;
- predict the effect of temperature on rates of reactions;

8.2 WHAT YOU SHOULD KNOW

Concentration and temperature are two important factors which affect the rate of a reaction. Effect of concentration on rate of a reaction is given by its rate law.

For a general reaction

$$aA + bB \longrightarrow \text{Products}$$

The rate law may be written as, rate = = $k[A]^a\ [B]^b$

Here, a and b are the orders of the reaction with respect to reactants A and B respectively. The overall order, n, is given by

$$n = a + b$$

And k is the rate constant of the reaction

Generally, a and b are positive integers. However, in some cases they may be fractions or zero, Thus generally speaking, on increasing the concentration of a reactant, the rate of reaction increases. The rate of reaction also depends upon the temperature. Arrhenius equation gives the relationship between the two

$$k = A.e^{-Ea/RT}$$

where, k is the rate constant of the reaction at temperature T. *A* and *Ea* are constants for a given reaction. While *A* is known as Arrhenius factor and *Ea* as activation energy. Activation energy is the minimum energy which the reactant molecules must possess over and above of their average energy in order to undergo chemical change. On raising the temperature, more reactant molecules possess sufficient energy required for reaction. Hence, the rate of the reaction increases.

The reaction between sodium thiosulphate and hydrochloric acid occurs according to the following equation,

$$Na_2S_2O_3\ (aq) + 2HCl(aq) \longrightarrow 2\ NaCl(aq) + H_2O(l) + SO_2\ (g) + S(s)$$

As the reaction proceeds, the amount of precipitated sulphur increase, the solution becomes increasingly turbid and at a certain stage it becomes opaque. This stage can be fairly judged accurately by a simple method. The flask containing the reaction mixture is kept on a sheet of white paper on which a cross is marked in ink and when the reaction mixture becomes opaque, the cross is no longer visible. Rate of the reaction is inversely proportional to the time interval between the start of the reaction and the disappearance of the cross mark.

Effect of concentration of each reactant can be studied by changing the concentration of one reactant while keeping that of the other constant. The time of disappearance of cross mark (X) is noted in each case. The inverse values of the time intervals are compared. Effect of temperature can be studied by starting the reaction with the same concentrations of both the reactants at different temperatures. The inverse values of the time intervals for the cross mark to disappear are compared.

8.3 MATERIALS REQUIRED

(1) Apparatus	(2) Chemicals
Four 150mL conical flasks, 50mL measuring cylinder, Thermometer - 10 to 110^{0}C, Stop watch, Bunsen burner, Tripod stand, Wire gauze, Sheet of plain white paper	Dilute HCl - 1M Sodium thiosulphate solution - 0.1M

8.4 HOW TO PERFORM THE EXPERIMENT

a) Effect of Concentration

Take three clean and dry conical flasks and mark them 1, 2 and 3. If necessary, clean the flasks with dilute nitric acid and then wash with water thoroughly before using them. With the help of measuring cylinder, take 30 mL-distilled water in the conical flask 1 and 20mL- each in flask 2 and flask 3. Rinse the measuring cylinder with the solution of sodium thiosulphate and add 10 mL of it each in flask 1 and flask 2 and 20mL-in the flask 3. Using ball point pen, mark a cross on a sheet of plain white paper, lay it on the table and keep the flask 1 on it, so that it covers the cross-mark. Make sure that the flask is absolutely dry from outside and base, before keeping it on paper. If necessary, wipe it dry with a piece of filter paper. Wash the measuring cylinder with water and rinse it with hydrocloric acid. Now add 10mL of hydrochloric acid to the flask 1, swirl it to mix the solutions and simultaneously start the stop watch. Keep the flask back on the white paper. Observe the cross mark through the solution and stop the watch as soon as it disappears. Record the time interval and its inverse in the Table 8.1 (given in section 8.5) and reset the zero of the stop watch for next reading. Repeat the procedure, first, using flask 2 and adding 20 mL hydrochloric acid to it and then with flask 3 and adding 10mL hydrochloric acid. Record the readings in Table 8.1

b) Effect of Temperature

Take three clean and dry conical flasks and mark them 1, 2 and 3. If necessary, before using, clean them with dilute nitric acid and wash with water thoroughly. Using a clean measuring cylinder take 30mL distilled water in each flask. Rinse the measuring cylinder with sodium thiosulphate solution and add 10mL of it in each of these three conical flasks. Using a ball- point pen, make a cross mark on a plain white sheet of paper and lay it on table. Wash the measuring cylinder with water and rinse with hydrocloric acid. Take another conical flask (other than the three marked flasks) and take 10mL hydrochloric acid in it using the measuring cylinder.

Heat both flasks (flask No. 1 containing $Na_2S_2O_3$ and the other containing HCl) to a temperature of about 40^0C separately on two tripod stands with wire gauze on them. Place the flask 1 on the sheet of white paper covering the cross mark. Quickly pour the hot HCl solution into the flask 1. Mix the solutions by swirling and start the stop watch simultaneously. Keep it back on the white sheet of paper. Measure the temperature of the mixture solution and record in table 8.2 (given in section 8.5). Observe the cross mark through the solution and stop the watch as soon as it disappears. Record the time interval and its inverse in Table 8.2. Reset the zero of the stop watch for next reading. Repeat this procedure two more times. First time use the flask 2, and heat the solutions to about 50^0C. Second time use flask 3 and heat the solutions to about 60^0C. Record the observations in table 8.2.

8.5 PRECAUTIONS

1. All conical flasks should be of same capacity and total volume of reaction mixture should be same for each observation.

2. Same sheet of white paper with cross marked on it should be used in all the observations.

3. Start the stop watch as soon as the solutions are mixed. Stop the stop watch as soon as the cross-mark just disappears.

4. While placing on white paper, the base and outer surface of the conical flask should be absolutely dry, else, the white paper or the cross marked on it would be spoiled.

5. Wash the conical flasks immediately after taking the reading with the help of dilute HNO_3 first and then with water. If not washed immediately, the sulphur starts depositing on the walls of the flask and makes the cleaning more difficult.

8.6 OBSERVATIONS

Table 8.1 : Effect of concentration on rate of reaction between HCl and $Na_2S_2O_3$

S.No.	Volume of				Initial Conc. of		Time	Time
	Water 0.1	M $Na_2S_2O_3$	1M HCl	Total	$Na_2S_2O_3$	HCl		inverse
	mL	mL	mL	mL	mol^{-1}	mol^{-1}	s	s^{-1}
1.	30	10	10	50	....	....	....	....
2.	20	10	20	50	....	....	....	....
3.	20	20	10	50	....	....	....	....

Table 8.1 : Effect of temperature on rate of reaction between HCl and $Na_2S_2O_3$

S.No.	Volume of				Temperature	Time	Time
	$Na_2S_2O_3$	Water	1M HCl	Total			inverse
	mL	mL	mL	mL	^0C	s	s^{-1}
1.	10	30	10	50	....	....	....
2.	10	30	10	50	....	....	....
3.	10	30	10	50	....	....	....

8.7 CONCLUSION

1. The rate of reaction increase/decreases/remains unchanged when concentration of either sodium thiosulphate or HCl is increased while keeping the other constant.

2. The rate the reaction increase/decrease/remains unchanged when it is carried out at higher temperature while keeping all other factors like concentration constant.

8.8 CHECK YOUR UNDERSTANDING

1. What are the initial concentrations of sodium thiosulphate and HCl in a solution obtained by mixing 10mL of 0.1 M $Na_2S_2O_3$, 20 mL of M HCl and 20 mL water. Assume that no reaction has started as yet.

2. While studying the effect of temperature on the rate of reaction, same volumes of sodium thiosulphate and HCl solutions are used in each experiment. Why it is so?

3. Why does increase in temperature generally increase the rate of a reaction ?

4. What will be the effect of decrease in temperature on rates of reactions which occur in biological processes ? Give one example where it is used.

5. Sometimes the solution of sodium thiosulphate starts becoming turbid even before adding HCl to it. What can be its reason ?

8.9 NOTE FOR THE TEACHER

Emphasize the importance of proper cleaning of conical flasks, especially after the flask has been used once. The students may be told that sulphur precipitated in the reaction sticks to the walls of the flask and is not easily removable. Therefore, washing should be done immediately after taking the reading. If simple washing by water does not remove the sulphur coating, washing may first be done with very dilute HNO_3, and then with water. Washing with water must be thorough so that HCl (used in the experiment) or HNO_3 (used for washing) are completely washed away. Otherwise, these will start the reaction in the next reading even before HCl is added.

8.10 CHECK YOUR ANSWERS

Ans. 1. Final concentrations can be calculated by using the relation $M_1 V_1 = M_2 V_2$ where M_1 and V_1 are the molarity and volume of the initial solution and M_2 and V_2 are the molarity and volume of the final solution obtained after dilution.

a) Molarity of $Na_2S_2O_3$

$M_1 = 0.1$ M $\quad\quad V_2 = 10 + 20 + 20 + 50$ mL

$V_1 = 10$mL $\quad\quad M_2 = ?$

$$M_2 = \frac{M_1V_1}{V_2}, = \frac{0.1 \text{ x } 10}{50} = 0.02 \text{ M}$$

b) Molarity of HCl

$M_1 = 0.1$ M $\quad\quad V_2 = 10 + 20 + 20 + 50$ mL

$V_1 = 20$ mL $\quad\quad M_2 = ?$

$$M_2 = \frac{M_1V_1}{V_2}, = \frac{0.1 \text{ x } 20}{50} = 0.4\text{ M}$$

Ans. 2. If different volumes of sodium thiosulphate or HC1 solutions are used besides temperature, their concentrations would also be different in different observations. Now the rate of the reaction would be affected by both the factors, and not only by temperature alone which is the aim of the study.

Ans.3. On raising the temperature, more reactant molecules possess the sufficiently high energy to overcome the activation energy barrier of the reaction. This increases the rate of the reaction.

Ans.4. It will decrease the rate of reactions occurring in biological processes. It is used in refrigeration or cold storage of food articles to increase their shelf life (or to keep them fresh for longer period of time) by slowing down the bacterial decay which occurs in them.

Ans. 5. It can be due to improper washing of conical flask with some HC1 (from the previous reading) or HNO_3 used for washing of flask) still remaining in it. The acid left in conical flask reacts with sodium thiosulphate and the solution becomes turbid due to the formation of sulphur.

EXPERIMENT-9

Separation of the coloured components of the following by paper chromatography and comparison of their R_f values.

a) Black ink or a mixture of red and blue inks

b) Juice of a flower or grass

9.1 OBJECTIVES

After performing this experiment, you should be able to:

- Separate the various constituents of inks, juice of a flower and / or grass, and
- calculate the R_f value of the various components of mixture.

9.2 WHAT YOU SHOULD KNOW

Chromatography is a technique by which a mixture of various substances can be (a) separated, (b) purified, and (c) identified

The term chromatography is applied to separation process based on the principle of distribution of a sample between two phases namely:

i) a stationary phase or a fixed phase; and
ii) a moving phase.

There are two variations in the types namely,

i) solid-liquid chromatography
ii) ii) liquid-liquid chromatography

In solid-liquid chromatography, the solid surface absorbs the substance which is loaded (charged) on the surface and the compounds are separated on the solid surface. It can also be classified as (i) adsorption chromatography, (ii) partition chromatography, (iii) and ion- exchange chromatography. Adsorption chromatography is based on selective adsorption of substances on the surface of an adsorbent. Column chromatography, thin layer chromatography, etc. are based on the adsorption phenomenon. In column chromatography, the stationary phase is an adsorbent and the mobile phase is a solvent.

Liquid-liquid chromatography involves the distribution of a substance between two immiscible solvents to different extents. Paper chromatography is an example of liquid-liquid chromatography. The stationary phase is supported by sheet of filter paper. The filter paper, known as chromatography paper, holds water which acts as a stationary phase. The mobile phase is another liquid which carries the substance on the paper along with it.

Every substance has a characteristic R_f value at a given temperature and for a given solvent. R_f stands for 'Ratio of Fronts' or the retention factor. It is the ratio of the distance travelled by the component to the distance travelled by the solvent, from the origin (Fig. 9.1).

$$R_f = \frac{\text{distance travelled by the substance from the initial line}}{\text{distance travelled by the solvent from initial line}}$$

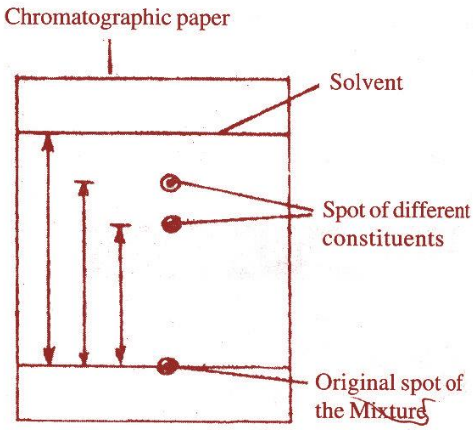


Fig. 9.1: *Measurement of R_f values*

$$\text{Rf of component A} = \frac{X}{Z} \text{ and Rf of component B} = \frac{Y}{Z}.$$

9.3 MATERIALS REQUIRED

(1) Apparatus	(2) Chemicals
Gas jar, Gas jar cover, Glass rod, Filter paper strip (whatmann No. 1), Fine capillary tube, Hook or clip for hanging the paper, Cork.	A mixture of red and blue inks, Alcohol,

9.4 HOW TO PERFORM THE EXPERIMENT

a) To separate the components of a mixture of red and blue inks and find out their R_f values.

Take the Whatmann filter paper strip (25 x 2cm). Draw a line with a pencil about 4 cm above the lower end as shown in Fig. 9.2

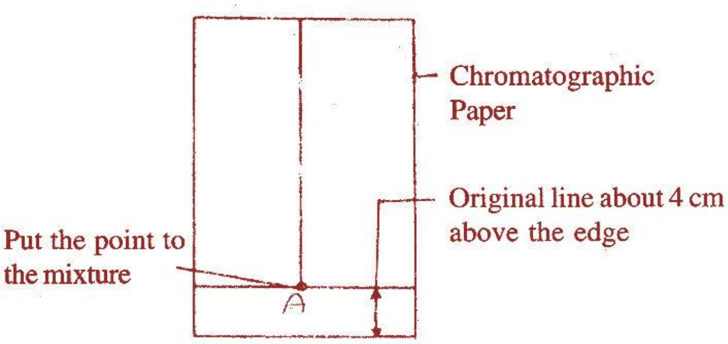


Fig. 9.2 : *Spotting the chromatographic paper*

Spotting: With the help of a fine capillary tube, put a drop of the mixture of red and blue inks at 'point A (Fig. 9.2) on the filter paper strip. Let the spot dry in air. Put another drop on the same spot and dry again. Repeat the process 2-3 times to make the spot rich in the mixture. Suspend the filter paper vertically in the gas jar containing the solvent mixture (50% alcohol). Hang the paper from the glass rod with the help of clips or hook in such a way that the spot remains about 2 cm above the solvent level. Alternatively cork with hook may be used.

Cover the jar with a glass cover and keep it undisturbed. Notice the rise of the solvent as it ascends the paper. After the solvent has risen about 20cm, you will see two different spots of red and blue colours on the filter paper. Take the filter paper out of the jar and mark the level of the solvent with a pencil. Dry the paper. Mark the centres of the blue and the red spots. Measure the distance of the two spots and that of the solvent from the initial line. Fig 9.2

Record your observations in Table 9.1 and calculate the R_f values of the red and blue spots.

b) To separate the coloured components present in grass / flower juice by paper chromatography and find the R_f values of the components separated out.

Material Required

(1) Apparatus	(2) Chemicals
Same as in experiment 9 (a)	Flower or grass juice as the sample. Solvent mixture (17 mL of petroleum ether + 3mL of acetone),

Note: The flower or grass juice is extracted by crushing the flowers or grass in a mortar with the help of a pastle and then extracting the juice by adding a little amount of acetone. Perform the experiment in the same way as in experiment 9 (a)

9.5 OBSERVATIONS

OBSERVATION FOR EXPERIMENT = 9 (B)

Record your observations and R_f in Table 9.1

Table 9.1

S. No.	Colour of the spot	Distance travelled by the spot from the initial line / cm	Distance travelled by the solvent from the initial line / cm	R_f
1.	Red	....	……………...............	....
2.	Blue	....	....	….........

9.6 PRECAUTIONS

1. Use very fine capillary tube for spotting. Otherwise the spot will become too big.
2. Allow the spot to dry before putting another drop. A good spot is small and does not contain too much of the mixture.
3. Do not press the capillary tube very hard, otherwise it may pierce the paper or break. If you make a hole in the paper then the compound will not rise.
4. Do not allow the paper to curl at the ends. It should hang straight, without touching the sides of the jar.
5. Keep the jar covered. Do not disturb the jar after handing the filter paper till the solvent has risen up to the desired level.
6. The spot should not dip in the solvent. Otherwise the compounds will dissolve in it.
7. Use distilled water for making the solvent mixture to be taken in the jar.

Precautions

Same as in experiment 9(a)

Observation for Experiment 9 (b)

Table 9.1

S. No.	Colour of the spot	Distance travelled by the spot from the initial line / cm	Distance travelled by the solvent from the initial line / cm	R_f
1.	Green (Chlorophyll)	....	....	....
2.	Yellow (xanthophyll)	....	....	....
3.	Red (Carotene)	....	....	....

The R_f of red ink = ...

The R_f of blue ink = ...

9.7 CONCLUSIONS

R_f of the green component (chlorophyll) = ...

R_f of the yellow component (xanthophyll) = ...

R_f of the red component (carotene) = ...

9.8 CHECK YOUR UNDERSTANDING

1. What is chromatography?

....

2. What is the basic principle of chromatography?

....

3. What is paper chromotography?

....

4. What is meant R_f value?

....

5. What is a chromatogram?

....

6. How are colouless compounds detected on a chromatogram?

....

7. What will happen if the paper strip is not vertical or it touches the side of the jar?

....

8. While spotting, if a hole is created on the paper, how will it affect the separation?

....

9.9 NOTE FOR THE TEACHER

i) An exercise can be given to separate a mixture of Ni^{2+}, CO^{2+} and Zn^{2+} by paper chromatography using the solvent mixture (85mL acetone + 5 mL water + 10mL conc. HCl).

ii) When students extract fruit juice or grass juice, the teacher should explain the method of extraction.

iii) Even high moisture content in the atmosphere will interfere in the solvent extraction.

Therefore the teacher should advice the students to perform in dry condition.

iv) The teacher should help students to use proper ratio of solvents, so that the required polarity can be maintained.

9.10 CHECK YOUR ANSWERS

Ans. 1. Chromatography is a technique by which mixtures of various substances can be (a) separated (b) purified, and (c) identified.

Ans. 2. Chromatography is based on the principle of distribution of a sample between two phases, namely a stationary phase and a moving phase.

Ans.3. Paper chromatography is a type of liquid - liquid chromatography. Here the water held on the cellulose of the paper acts as the stationary phase and the moving solvent acts as the moving phase.

Ans.4. R_f value is the ratio of distance travelled by the component from the original spot to the distance travelled by the solvent from the original spot. R_f is a constant for a given substance at a particular temperature and for a given solvent.

Ans.5. The colour spots appear on the paper at different levels after the chromatographics separation is over. This is called a chromatogram.

Ans.6. The colourless compounds can be made visible by spraying a reagent on the paper. The separated component on the paper reacts with the reagent and produces a coloured compound which becomes visible.

Ans.7. If the paper strip is not vertical or it touches the sides of the jar then solvent flow will not be uniform or separation will not be proper.

Ans.8. If a hole is created in the paper then the constituents of the mixture will not rise at all, but spread near the original point.

EXPERIMENT-10

To detect the presence of extra elements (nitrogen, sulphur and halogens) in an organic compound.

10.1 OBJECTIVES

After performing this experiment, you should be able to:

- detect the presence of nitrogen, sulphur and halogens (extra elements) present in a given organic compound;
- explain the reason for the necessity of fusing an organic compound with sodium metal in the detection of extra elements; and
- write chemical equations of various reactions taking place in the experiment.

10.2 WHAT YOU SHOULD KNOW

The determination of structure of an unknown organic compound requires the detection of the elements present in it. Generally all organic compounds contain carbon and hydrogen. In addition to these elements, they may also contain oxygen, nitrogen, sulphur and helium. The elements nitrogen, sulphur and halogens are known as extra elements.

For detecting the presence of elements in a compound, these have to be converted into ionic forms, This is done by fusing the compound with sodium metal. The elements, nitrogen, sulphur and halogens present in the compound are converted into soluble sodium salts according to the following equations.

$$\mathrm{Na + C + N \longrightarrow NaCN}$$

$$\mathrm{2Na + S \longrightarrow Na_2S}$$

$$\mathrm{Na + X \longrightarrow NaH}$$

(where X is Cl, Br or I)

If a compound contains both nitrogen and sulphur, sodium thiocyanate is formed on fusing with sodium. The soluble sodium salts are extracted with water. The extract is usually known as sodium fusion extract.

10.2.1 Test for Nitrogen

Nitrogen is present as sodium cyanide in sodium fusion extract. Sodium cyanide is converted to sodium ferrocyanide on treating with ferrous sulphate. On further treating it with ferric chloride, a Prussain blue complex, ferricferrocyanide is formed.

$$6NaCN + Fe\,SO_4 \longrightarrow Na\,[Fe(CN)_6] + Na_2\,SO_4$$

$$Na_4\,[Fe(CN)_6] + 4FeCl_3 \longrightarrow Fe_4\,[Fe(CN)_6)]_3 + 12NaCl$$

Ferricferrocyanide

(Prussain blue)

10.2.2 Test for Sulphur

i) Sulphur is present in sodium extract as sodium sulphide. It gives a purple colour with sodium nitroprusside due to the formation of sodium thionitroprusside.

$$Na_2S + Na_2\,[Fe(CN)_5NO] \longrightarrow Na_4\,[Fe(CN)_5\,NOS]$$

Sodium nitroprusside **Sodium thionitroprusside**

(Purple)

ii) A Sulphide reacts with lead acetate to yield lead sulphide as a black precipitate.

$$Na_2S + Pb\,(CH_3OO)_2 \longrightarrow PbS + 2CH_3COONa$$

Lead sulphide

(black)

iii) A sulphide reacts with silver nitrate to yield silver sulphide which is black in colour.

$$Na_2S + 2AgNO_3 \longrightarrow Ag_2S + 2NaNO_3$$

Silver Sulphide (Black)

10.2.3 Test for Nitrogen and Sulphur when present together

When both nitrogen and sulphur are present to gether in an organic compound, sodium thiocyanate is produced during sodium fusion. Sodium thiocyanate gives blood red colour with ferric chloride.

$$NaCNS + FeCl_3 \longrightarrow [Fe(CNS)^{2+} + Na^+ + 3Cl^-$$

(Blood red)

10.2.4 Test for Halogens

Halogens, present as halides in sodium extract, are tested as follows :

i) **Silver Nitrate test :** Chloride given white precpitate of silver chloride with silver nitrate. The precipitate is soluble in ammonium hydroxide.

$$NaCl + AgNO_3 \longrightarrow AgCl\downarrow + NaNO_3$$

$$AgCl + 2NH_4OH \longrightarrow [Ag(NH_3)_2]Cl + 2H_2O$$

Bromides yield yellow precipitate, sparingly soluble in ammonium hydroxide. Iodides yield dark yellow precipitate, insoluble in ammonium hydroxide.

ii) When a solution containing bromide or iodide is treated with chlorine water, the bromide and iodide are oxidised to the corresponding halogens. Being covalent, these halogens get dissolved in carbon tetrachloride CCl_4. Bromine imparts yellow to brown colour and iodine imparts purple colour in CCl_4 layer.

$$2NaBr + Cl_2 \longrightarrow 2NaCl + Br_2$$

$$2NaI + Cl_2 \longrightarrow 2NaCl + I_2$$

Chlorine being more electronegative, displaces bromides and iodides from solution.

10.3 MATERIALS REQUIRED

(1) Apparatus	(2) Chemicals
Test tubes - 6, test-tube stand,	Sodium metal, Ferrous sulphate,
Test tube holder,	Ferric chloride, Silver nitrate
Ignition tubes - 3, Spatula, Watch-glass,	Ammonium hydroxide, Chlorine water
Filter paper, Pair of tongs, Porcelain dish,	Carbon tetrachloride, Nitric acid
Funnel, Glass rod, Dropper, Wite gauze.	

10.4 HOW TO PERFORM THE EXPERIMENT

The experiment consists of two stages, (i) preparation of sodium fusion extract and (ii) to test for individial elements.

1) Preparation of Sodium Fusion Extract

Take out a small piece of sodium metal from a petri dish with the help of pair of forceps. Dry the sodium piece between the folds of filter papers. Put a small piece of sodium metal into a clean and dry ignition tube. Hold the tube with a pair of tongs and heat it gently over the flame so that sodium melts and makes a stirring ball. Remove the ignition tube away from the flame and immediately add a small amount of solid organic compound into it. Heat the tube to red hot. Again add a little of the compound into the tube and heat it again to red hot. Plunge the red hot ignition tube in about 10mL of distilled water taken in a porcelain dish. Immediately cover the porcelain dish with a wire gauze to avoid bumping of any unreacted sodium piece. Repeat the process of sodium fusion two more ignition tubes. Break the tubes into small pieces with a clean glass rod. Boil the mixture for about 2-3 minutes and filter.

(In case of liquid compounds, cover the molten sodium with solid sodium carbonate and then add the compound with a dropper).

Now process systematically and perform the following tests with the given sample (s), of organic compounds and identify the extra elements present in them. Record your observations and inferences in the table 10.1 in the following manner.

Table 10.1

S. No.	Experiment	Observations	Inference
1	**Test for nitrogen** Take about 1 mL of sodium fusion extract in a test tube and add a pinch of ferrous sulphate. Shake the contents and acidity with dilute hydrochloric acid, Boil the contents add a few drops of ferric chloride solution.	Prussain blue or Green precipitate or colour	Nitrogen present
2	**Test for sulphur** **i)** Take 1 mL of sodium fusion extract in a test tube and add a few drops of sodium nitroprusside to it and shake.	Purple colour	Sulphur present
	ii) Take 1 mL of sodium fusion extract in a test tube and acidify it with acetic acid. Add a few drops (4-5) of lead acetate solution to it	Black precipitate	Sulphur present
3	**Nitrogen and Sulphur present together** Take about 1 mL of sodium fusion extract in a test tube and acidify it with dilute hydrochloric acid. Add a few drops of ferric chloride solution.	Blood red colour present	Nitrogen and Sulphur
4	**Test for Halogens** **i)** Take about 1 mL of sodium fusion extract in a test tube and acidify it with dilute nitric acid. Add to it about 2mL of silver nitrate solution. Observe for the appearance for any precipitate and its colour.	**a)** A curdy white precipitate soluble in NH_4OH	Chlorine present
	Add 2-3 mL of ammonium hydroxide to the precipitate and shake the content. Observe the solubility of precipitate	**(b)** A light yellow precipitate partially soluble in NH_4OH	Bromine present
		(c) A pale yellow precipitate insoluble in NH_4OH	Iodine present
	ii) If chloride is absent, perform the following test of bromide and iodide. Take 2 mL of sodium fusion extract in a test tube and acidify it with dilute hydrochloric acid. Add 1 mL of carbon tetrachloride. being denser, CCl_4 forms the lower layer. Add excess of chlorine water and shake the mixture vigrously. Observe for appearance of any colour in CCl_4 layer.	a) Orange Colour	Bromine presence
		b) Violet	Iodine presence

10.5 PRECAUTIONS

1. Sodium metal is highly reactive. When exposed to air, it reacts even with the moisture present in the atmosphere. It also reacts with the sweat of hands. Hence, do not hold it with hands. Always use forceps handling sodium.
2. Before using sodium metal, press it within the folds of filter paper to remove oil.
3. Use dry ignition tubes for sodium fusion. Sodium reacts with water violently.
4. Put the unused sodium piece back in bottle. Do not throw it in to the sink.
5. Repeat the process of sodium fusion with three ignition rubes. This is to ensure that the fusion has taken place.
6. After immersing the red hot ignition tube in water, break it with a glass rod gently. Boil the contents for 2-3 minutes so as to extract the soluble sodium salts in water.

10.6 OBSERVATIONS

Table 10.1

S.No.	Experiment	Observations	Inference

The given sample responded positively to ____________________, ___________________ and ______________ tests. Therefore the extra elements present in the sample is ______________.

10.7 CONCLUSION

The given organic compound contains _____________________________ as extra elements.

10.8 CHECK YOUR UNDERSTANDING

1. Why is only freshly cut sodium metal used for fusion of an organic compound ?

....

2. Why is it necessary to carry out the fusion of an organic compound with sodium, more than one using different ignition tubes?

....

3. While testing for nitrogen why is it necessary to neutralize the sodium extract completely with an acid ?

....

4. Why is it necessary to fuse an organic compound with sodium for carrying out the tests for extra elements present in it ?

....

5. Why are bromide and iodide displaced from their salts in solution by chlorine water ?

....

6. What will happen if sodium extract is not completely neutralized before performing silver nitrate test for halogens ?

....

7. Why is it essential to use distilled water in the preparation of sodium extract ?

....

10.9 NOTE FOR TEACHER

Sodium metal is highly reactive. Ensure that students handle it carefully and take all precautions. In preparing sodium fusion extract, the low concentrations of ions in solution give only a faint colour or precipitate. It is advisable to use 2-3 ignition tubes during sodium fusion. In case of liquid compounds, the molten sodium metal is first covered with sodium carbonate. The liquid compound is added over it so that it is absorbed in sodium carbonate. The low boiling liquids tend to escape without undergoing reaction with sodium.

After the students have performed the tests of all the extra elements, they may be asked to identify the extra elements in given compounds and write full details in the experiments sheet.

10.10 CHECK YOUR ANSWERS

Ans. 1. Sodium is a highly reactive metal. Its surface gets a coating of its oxide due to its reaction with atmospheric oxygen. By taking a freshly cut sodium, the exposed surface is free from Oxide.

Ans. 2. A majority of organic compounds are low boiling and volatile. They tend to escape out of the ignition tube during heating. To ensure an adequate concentration of ions containing extra elements in test solution, it is preferable to carry out the sodium fusion 2-3 times.

Ans. 3. On adding ferrous sulphate to sodium extract, ferrous hydroxide forms a dirty green precipitate. To avoid this, the test solution is neutralized completely.

Ans. 4. The extra elements are present as covalently bonded atoms in organic compounds. To test them in solution, these are converted into ionic form by fusing the compound with sodium.

Ans. 5. Chlorine, being more electronegative displaces bromide and iodide in solution.

Ans. 6. A grey black precipitate of silver oxide is formed. It becomes difficult to identify the colour of silver halides.

Ans. 7. Ordinary water contains chloride ions. These interefere with the halide ions formed from halogen of the organic compound.

EXPERIMENT-11

Study of simple reactions of carbohydrates, fats and proteins and detection of their presence in given food stuffs.

11.1 OBJECTIVES

After performing this experiment, you should be able to:

- Test the presence of carbohydrates, proteins and fats in the given food sample;
- Establish whether given carbohydrate is reducing or non-reducing.
- Distinguish between saturated and unsaturated fats.

11.2 WHAT YOU SHOULD KNOW

11.2.1 Carbohydrates

Carbohydrates are polyhydroxy aldehydes or ketones and the substances which yield these on hydrolysis. Carbohydrates are classified as :

i)	Monosaccharides	-	glucose, fructose-($C_6H_{12}O_6$)
ii)	Disaccharides	-	sucrose, maltose-($C_{12}H_{22}O_{11}$)
iii)	Polysaccharides	-	starch, cellulose, glycogen.

All the carbohydrates, except cellulose, are hydrolysed in out body to glucose which on slow oxidation, in presence of oxygen, liberates large amount of energy. The oxidation of glucose can be represented as:

$$\underset{\text{Glucose}}{C_6H_{12}O_6} + 6O_2 \xrightarrow{\text{Respiration}} 6CO_2 + 6H_2O + \text{Energy}$$

Thus carbohydrates (sugars and starch) are the main source of energy in our body. The cellulose acts as a rough age. The carbohydrates which reduce Fehling's solution and Tollen's reagent are called reducing sugars. All the Mono-saccharides and most of the disaccharides are reducing sugars. Sucrose (cane-sugar) is a non-reducing sugar and hence does not reduce Fehling's solution.

Test for Carbohydrates

1. Molisch's Test (for all carbohydrates): When an aqueous solution or suspension of a sample containing a carbohydrates is treated with α-naphthol (Molish's reagent), followed by the addition of few drops of concentrated sulphuric acid along the sides of the test tube a red-voilet ring appears at the junction of two layers. This happens because concentrated sulphuric acid converts carbohydrates to furfural or its derivatives which reacts with α-naphthol to give a coloured (red-violet) product.

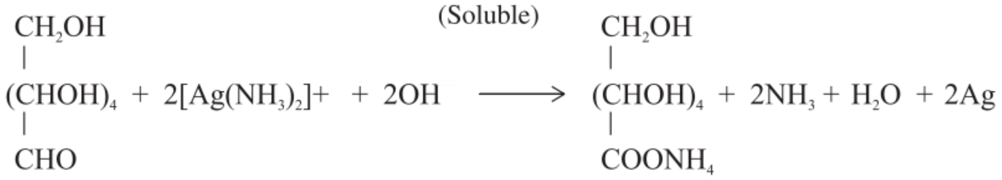
2. Tollen's Test (for reducing sugars): any aldose or ketose having a free carbonyl groups reduces Tollen's reagent to metallic silver. This test is commonly known as silver mirror test as a kind of mirror is obtained on the walls of the test tube.

The reactions involved in this test are as follows:

$$AgNO_3 + NH_4OH \longrightarrow NH_4NO_3 + AgOH$$

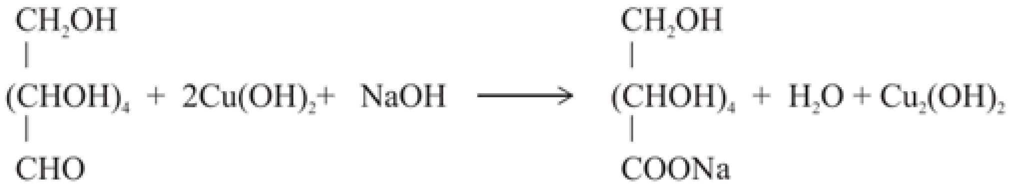
$$2\,AgOH \longrightarrow Ag_2O + H_2O$$

$$Ag_2O + 2NH_4OH \longrightarrow \underset{\text{(Soluble)}}{[Ag(NH_3)_2]\,OH^-} + H_2O$$



$$\begin{array}{l} CH_2OH \\ | \\ (CHOH)_4 \\ | \\ CHO \end{array} + 2[Ag(NH_3)_2]^+ + 2OH \longrightarrow \begin{array}{l} CH_2OH \\ | \\ (CHOH)_4 \\ | \\ COONH_4 \end{array} + 2NH_3 + H_2O + 2Ag$$

3. Fehling's Test (for reducing sugars): When equal quantities of Fehling's solution A and B are mixed with an aqueous solution of a reducing sugar and the mixture is heated on boiling water bath, a reddish precipitate of cuprous oxide is formed. The following reactions are involved in the test.



$$\begin{array}{l} CH_2OH \\ | \\ (CHOH)_4 \\ | \\ CHO \end{array} + 2Cu(OH)_2 + NaOH \longrightarrow \begin{array}{l} CH_2OH \\ | \\ (CHOH)_4 \\ | \\ COONa \end{array} + H_2O + Cu_2(OH)_2$$

$$Cu_2(OH)_2 \longrightarrow Cu_2O\downarrow + H_2O$$

4. Iodine test for Starch: Starch gives a purple-blue colour when treated with iodine. It is due to the adsorption of iodine one the surface of starch. The colour gets discharged on heating and reappears on cooling.

11.2.2 Fats

Fats are triesters of long chain fatty acids and glycerol. One of the function of fats in the body is to provide energy. Fats provide twice as much energy as that provided by the same amount of carbohydrates. Fats are insoluble in water. During the process of digest, fats are hydrolysed into simple substances, like fatty acids and glycerol which further break down into still simple substances. Sources of fats in our food are butter, ghee, cheese, milk, egg-yolk, meat, nuts, groundnut and soyabean etc.

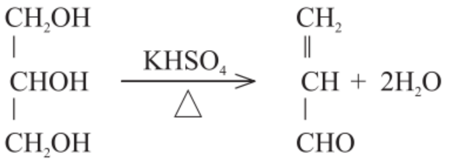
Test for Fats

1. Spot test: When a sample containing is pressed or rubbed on a clean white sheet of paper, a greasy spot (translucent spot) appears.

2. Solubility Test : This test is based on the fact that fats are soluble in organic solvents like chloroform, alcohol etc but are insoluble in water. A small amount of the sample is mixed with a few drops of water, chloroform and alcohol in different test tubes and inferences are drawn on the basis of the miscibility and immiscibility of the sample with the water or organic solvents.

3. Acrolein Test: When a sample containing fat is heated with solid potassium bisulphate ($KHSO_4$) in a test tube, a pungent irritating odour of acrolein is produced according to the following chemical reaction;

$$\text{Oil or fat} \xrightarrow{\text{Heat}} \text{Glycerol + fatty acid}$$

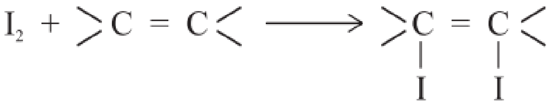


$$\begin{array}{l}CH_2OH\\ |\\ CHOH\\ |\\ CH_2OH\end{array} \xrightarrow[\triangle]{KHSO_4} \begin{array}{l}CH_2\\ \|\\ CH\\ |\\ CHO\end{array} + 2H_2O$$

Glycerol **Acrolein**

4. Huble's Test (for determination of unsaturation in fats): This test is based on the fact that greater the amount of unsaturation in a fat or oil, greater is the amount of Huble's solution required to produce a permanent violet colour.

For this equal amount of saturated fat (cotton seed oil) and unsaturated fat or oil (linseed oil) are taken in two different test tubes and Huble's solution is added to each test tube drop wise. The number of drops of Huble's solution required for getting a permanent colour is counted in each case. More the number of drops of Huble's solution, greater is the unsaturation in an organic compound. Iodine from Huble's solution adds up across the unsaturation sites of oil or fat giving a colourless product and a little more of iodine gives a permanent violet colour to the solution.



$$I_2 + {>}C=C{<} \longrightarrow {>}\underset{I}{\underset{|}{C}}=\underset{I}{\underset{|}{C}}{<}$$

11.2.3 Proteins

Proteins are complex nitrogenous organic compounds of high molecular mass. These help in the growth and maintenance of living body and in transmission of genetic information. Proteins are made up of-amino acids as the basic building blocks. In all, there are 20 different amino acids required by the body. Some of these are called essential amino acids as they cannot be synthesized by the organisms. These must form a part of our food.

Tests for Proteins

1) **Biuret Test:** When a sample of protein is treated with a solution of copper sulphate in alkaline medium, a violet colour appears. The intensity of the colour also indicates the relative amount of protein present in different samples.
2) **Ninhydrin Test:** When a few drops of a 0.1% alcoholic solution of ninhydrin, are added to a very dilute solution / suspension of protein and the contents are boiled for 1-2 minute, a red-purple colour appears.
3) **Xanthoprotein Test:** When an aqueous solution of protein is treated with a few drops of nitric acid and contents are allowed to stand; a deep yellow colour appears.
4) **Million's Test:** A few drops of milk are warmed with mercuric nitrate in presence of dil. Sulphuric acid and cooled. One drop of sodium nitrate solution is added and the mixture is again warmed. A red colour appears showing the presence of proteins in milk.

11.3 MATERIAL REQUIRED

(1) Apparatus	(2) Chemicals
Test tubes with stand, **Burner**	Fehling's solutions - A and B, Molish reagent (α-Naphthol, Silver nitrate, Ammonium hydroxide, Iodine solution, Ethyl alcohol, Huble's solution, Potassium bisulphate, dilute sulphuric acid, Mercuric nitrate, Sodium nitrate, Nitric acid, Ninhydrin solution (0.1% in alcohol), Sodium hydroxide, Copper sulphate, and some food samples like: milk, surcrose, glucose, wheat flour, vegetable oil, ghee, butter, cheese potato pieces, lemon, grapes, banana, eggs etc. 5% solution of egg white protein.

11.4 HOW TO PERFORM THE EXPERIMENT

11.4.1 Worksheet No. 1 : Test for Carbohydrates

Experiment	Observation	Inferences
For Carbohydrates **(i) Molish's Test :** Take 2-3 mL of aqueous solution or suspension of the sample. Add a few drops of Molish's reagent and shake it. Then add 5-6 drops of conc. H_2SO_4 slowly along the side of the test tube.		
(ii) Tollen's Test : Take a test tube and clean it thoroughly with NaOH solution. Then wash the tube with excess of water to remove NaOH. Take 2-3 mL of aqueous solution of carbohydrate in the test tube and add 2-3 mL of freshly prepared Tollen's reagent to it. Keep the test tube in a boiling water bath for about 10 minutes.		
(iii) Fehling Test : Take a small amount of crushed food items (Ig) or 2-3 mL of aqueous solution of the sample (nearly 5%). Add 2 mL each of Fehling's solution A and Fehling's solution B. Keep the test tube in a boiling water bath.		

(iv) Iodine Test for Starch : Take a small amount (1-2g) of the food sample in a test tube and add a few drops of dilute iodine solution.		

11.4.2 Worksheet No. 2: Test for Fats

Experiment	Observation	Inferences
For Fats **(i) Spot Test :** Take a food item to be tested and press or rub it on a clean white sheet of paper and hold it against light.		
(ii) Solubility Test : Take a small amount (50mg) of the given sample in a test tube. Add 2-3 mL water in and shake. Observe if it has dissolved in it. Similarly check the solubility of the compound in alcohol, and CCl_4 separately.		
(iii) Acrolein Test : Heat a small amount (2-3g) of sample with same amount (2-3g) of solid $KHSO_4$ in a test tube. Bring the mouth of the test tube near the nose and smell the gas evolved, if any, only superficially.		

(iv) Iodine Test for Starch : Take two test tubes labelled as 'A' and 'B'. Add about 3-4 mL of chloroform to each test tube. Put some sample (2-3g) of unsaturated fat (linseed oil) in test tube 'A' and saturated fat (cottonseed) in test tube 'B'. Add Ruble's solution to each test tube dropwise and keep on adding while counting the drops, until a permanent violet colour appears in each test tube.		

11.4.3 Worksheet No. 3: Test for Proteins

Experiment	Observation	Inferences
For Proteins **(i) Biuret Test :** Take one mL of milk or any other protein containing sample in a test tube. Make it alkaline by adding a few drops (4-5) of NaOH solution Then add 4-5 drops of dil. solution of $CuSO_4$.		
(ii) Ninhydrin Test : Take about 1mL of 5% solution of egg white in a test tube. Add to it 4-5 drops of 0.1% ninhydrin solution. Boil the contents for about one minute and cool it.		
(iii) Xanthoprotein Test : Take 2-3 grams of gluten in of wheat in a test tube. Add 2-3 mL of cone. HNO_3, to it. Shake the contents and keep aside.		

(iv) Million's Test : Take about 1mL of milk in a test tube. Add 1-2 drops of mercuric nitrate solution and one drop of dil.H_2SO_4. Boil and cool the contents and then add 1 drop of $NaNO_3$ solution. Warm the mixture again and cool it.		

11.5 PRECAUTIONS

1. Smell the gases coming out of reactions carefully.

Some compounds are highly corrosive. For example, phenol can cause blisters and burn your skin when it falls on it. Immediately resort to first aid.

Use a water bath for warning the contents. Do not heat organic the compounds on a direct flame, these can catch fire easily.

2. Neutral ferric chloride must be prepared fresh (procedure is given in appendix.)
3. Tollen's reagent must be freshly prepared (Procedure is given in the appendix)
4. Fehling's solution should be prepared by mixing equal amounts for Fehling's solution A and Fehling's solution B required for the test.

11.6 OBSERVATION

Record your observations in the blank worksheet in the following manner:

1. ______________________, ______________________, __________________ are found in the given food stuffs.

Table 11.1

Test	Glucose	Sucrose	Starch
1. Solubility in water			
2. Fehling's test			
3. Tollen's test			
4. Molisch's test			
5. Benedict's test			
6. Iodine test			

Table 11.2

S.No.	Test	Fat (Saturated)		Fat (unsaturated)
		Animal	Vegetable	
1.	Spot test			
2.	Solubility test (in org. solvent)			
3.	Acrolein test			
4.	Huble's test			

11.7 CONCLUSIONS

Following conclusions are drawn based on test for carbohydrate:

i) The given sample / does not / contain carbohydrates.
ii) The given sample contains reducing / non-reducing sugar.

For Fats:

i) The sample does not contain fat.
ii) The sample contains saturated / unsaturated fat.

For Proteins:

i) The given sample contains proteins.
ii) The given sample does not contain proteins.

Common:

i) The given sample contains fat and protein body.
ii) The given sample contains carbohydrats and protein.
iii) The given sample contains carbohydrates, fat and protein.

11.8 CHECK YOUR UNDERSTANDING

1. Why are carbohydrates known as 'hydrates of carbon' ?

   ....

2. Define carbohydrates.

   ....

3. What is the general formula of polysaccharides?

....

4. What is meant by the term reducing sugar?

....

5. Name the compound formed as red precipitate when a carbohydrate is treated with Fehling's solution.

....

6. Name the carbohydrate which is not digested in our body.

....

7. Which carbohydrate blue colour product when treated with iodine?

....

8. What are fats?

....

9. What products are obtained on hydrolysis of fats?

....

10. Write down the structural formula of Acrolein.

....

11. Name for sources of fats?

....

12. What is the difference in the physical state of saturated and unsaturated fats at room temperature?

....

13. A fat required more of Huble's solution of giving a permanent violet colour in the mixture? What inference will you draw from this observation?

....

14. Define proteins.

....

15. What is the main role of proteins in our body?

....

16. Name the bonding which unit amino acids to from a protein.

....

11.9 NOTE FOR THE TEACHER

1. Ensure that students work themselves with at least one sample of each kind of carbohydrates, (Glucose, Sucrose and starch), saturated and unsaturated fats.
2. After the students have performed the tests of all the samples, they may be asked to identify the unknown substance and write full details in the experiments sheet.
3. The reactions in all the experiments occur very easily. The students may be asked to perform slow addition and pay keen observation.

11.10 CHECK YOUR ANSWERS

Ans. 1. Carbohydrates are called as hydrates of carbon because proportion of hydrogen and oxygen in carbohydrates is similar to that in water.

Ans. 2. Carbohydrates are polyhydroxyaldehydes or ketones.

Ans. 3. $(C_6H_{14}O_5)_n$

Ans. 4. Sugars having free carbonyl group can reduce Tollen's reagent Fehling's solution to give metallic silver and Cu_2O respectively. Such sugars are called reducing sugars.

Ans. 5. Cu_2O (Cuprous oxide)

Ans. 6. Cellulose

Ans. 7. Starch

Ans. 8. Fats are triesters of long chain fatty acids and glycerol.

Ans. 9. Fatty acid and glycerol.

Ans. 10. $H_2C = CH - CHO$

Ans. 11. Cotton seed oil, coconut, milk, ghee

Ans. 12. Saturated fats are solids and unsaturated fats are liquids at room temperature.

Ans. 13. The given fat has more unsaturation.

Ans. 14. Proteins are complex nitrogenous organic compounds.

Ans. 15. Proteins are helpful in growth and maintenance of our body tissues and also regulate various physiological activities.

Ans. 16. Amino acids join through peptide bonds making a polypeptide chain (protein).

EXPERIMENT-12

To prepare soap by using different oils and its comparison with the market soap by determining the foaming capacity and cleaning effect.

12.1 OBJECTIVES

After performing this experiment, you should be able to:

- prepare soap using different vegetable oils;
- compare the foaming capacity of different brands of soaps;
- compare the cleaning effect of different brands of soap.

12.2 WHAT YOU SHOULD KNOW

Ordinary soaps are sodium salts of fatty acids. They are made by heating vegetable oils, animal fats with concentrated sodium hydroxide solution. Vegetable oils are the esters of glycerol with long chain largely, unsaturated carboxylic acid such as oleic acid ($C_{17}H_{33}COOH$) or linoleic acids ($C_{17}H_{31}COOH$). Fats are the esters, of glycerol with long chain saturate carboxylic acids such as stearic acid ($C_{17}H_{35}COOH$). The oils and fats undergo hydrolysis (saponification) when treated with sodium hydroxide. Sodium salt of carboxylyic acid (soap) and glycerol are obtained as the reaction products.

Cleansing action of Soap : Soap functions as a cleansing agent in a variety of ways. The soap molecule consists of two portions, the hydrocarbon portion (R^-) and the ionic portion ($COONa^+$). The hydrocarbon portion is soluble in oils and the ionic portion is soluble in water. When a soap solution is added to water, it forms a colloidal suspension and soap molecules cluster together. These clusters are known as micelles. The micelles remain suspended in water because the like charges are the end of the molecule.

When a soap solutions is added to water containing grease or oils, the micelles break up. The hydrocarbon portion becomes embedded in grease. Agitation now breaks grease into micelles with surfaces covered by negatively charged carboxylate groups. With a coating of negative charges enveloping the entire surface of each micelle, the grease droplets repel each other and remain suspended in wash water. In the end, the suspended droplets go down and drain. Thus, soap acts as an emulsifying agent to bring about the dispersion of oil or grease.

12.3 MATERIALS REQUIRED

(1) Apparatus	(2) Chemicals
Beaker (250mL), Glass tubes-2, Measuring cylinder (10mL), Glassrod, Boiling tubes - 2, Scale (30cm), Funnel, Filter paper, Spatula, Glass stirrer.	Vegetable oil or fat (castor, olive etc.), Sodium hydroxide, Common salt, Indigo

12.4 HOW TO PERFORM THE EXPERIMENT

Take 50mL of water in a 250 mL beaker and dissolve about 10 grams of sodium hydroxide in it. Use an ordinary balance for weighing sodium hydroxide. Take 10mL of castor oil (slightly more than half of a test tube) in a test tube and transfer it slowly into sodium hydroxide solution with continuous stirring.

Heat the mixture gently over the flame till it boils. Keep on stirring the mixture while heating. Boil the mixture for 2-3 minutes and then allow it to cool to room temperature. Break large pieces of solid, if any. Filter the mixture and wash the solid residue in the funnel with a little water and allow it to dry.

Comparison of Foaming Capacity : Take a sample of laboratory made soap and 2-3 soaps of different brands from market. Weigh one gram of each soap sample and dissolve it in 100g of water. Take 10mL soap solution in a boiling tube and shake the solution with a stirrer making twenty regular strokes, so that the foam increases uniformly. Measure the height of foam so produced with a scale. Repeat this experiment with every brand of soap. Record your observation in table 12.1

Dispersion Power : Take about 0.5 gm of indigo in a boiling tube and add to it 5 mL soap solution prepared as above. Shake and add 20 mL of distilled water. Allow to stand for a while and observe for any separation of indigo particles. Repeat the experiment using different brands of soap. Compare the dispersion power of soaps and assign a grade to it. Record it in Table 12.2

12.5 PRECAUTIONS

1. Stir the reaction mixture continuously so as to ensure thorough mixing of oil with sodium hydroxide solution.
2. While shaking the soap solution in a boiling tube, the strokes should be uniform and same in number for all the soap samples.
3. Compare the foaming capacity of all soap samples at room temperature as the foaming capacity varies with temperature.
4. Sodium hydroxide is highly corrosive. Use it carefully

12.6 OBSERVATIONS

Table 12.1 : Comparison of foaming capacity of different soap samples

Sl. No.	Name of Soap Brand	Height of foam produced in cm
1.	Lab made	
2.	Market Soap	
	a) Sample - 1	
	b) Sample - 2	
	c) Sample - 3	

Table 12.2 : Comparison of foaming capacity of different soap samples

Sl. No.	Name of Soap Brand	Grading for dispersion power
1.	Lab made	
2.	Market Soap	
	a) Sample - 1	
	b) Sample - 2	
	c) Sample - 3	

12.7 CONCLUSION

The grading of foaming capacity of soaps is in the following order as given in the table 12.3.

Table 12.3

Soap make	Grading (A/B/C/D)
1. Lab made	....
2. Market soap	
a)	....
b)	....
c)	....

The grading of dispersion power is indicated Table 12.3

Based on the dispersion power, the cleaning power of soap is in the following order as given in the Table 12.3 :

Table 12.4

Decreasing order of grading of dispersion	Soap Make
A B C D	

12.8 CHECK YOUR UNDERSTANDING

1. While preparing soap, it is preferred to add the oil/fat to NaOH solution and not-vice-versa. Explain giving reasons.

……………………………………………………………………………………………………

2. What is the chemical nature of soap ?

……………………………………………………………………………………………………

3. Why is it necessary to continuously stir the reaction mixture while preparing soap ?

……………………………………………………………………………………………………

4. Why should the soap prepared in laboratory be washed with water ?

……………………………………………………………………………………………………

12.9 NOTE FOR THE TEACHER

In the 12.1 and 12 2, the trade names of market soaps should not be given by students. They should indicate only sample number - 1, 2, 3 etc.

12.10 CHECK YOUR ANSWERS

Ans. 1. Sodium hydroxide breaks the oil/fat into glycerol and carboxylic acid. The reaction is very fast. When oil/fat is added to sodium hydroxide a large amount of heat is evolved and sodium hydroxide is in excess whereas when sodium hydroxide is added to oil, oil is in excess.

Ans. 2. Soaps are sodium or potassium salts of long chain fatty acids.

Ans. 3. The two reactants required for preparing the soap (oil and aqueous solution of sodium hydroxide) are immiscible. It is necessary to bring the molecules of both the reactants in contact with each other.

Ans. 4. The reaction mixture obtained in soap preparation contains unreacted sodium hydroxide. It is removed by washing the soap with water.

EXPERIMENT-13

a) Preparation of solution of oxalic acid and ferrous ammonium sulphate of known molarity by weighing (non-evaluative). Use of chemical balance to be demonstrated.

b) A study of (i) acid-base and (ii) redox titrations (single titration only). Both the solutions to be provided.

13.1 OBJECTIVES

After performing this experiment, you should be able to:

- handle chemical balance properly :
- weigh chemicals accurately using a chemical balance;
- handle burette and pipette properly;
- pipette out a given solution correctly with the help of a pipette,
- prepare standard solutions of oxalic acid and ferrous ammonium sulphate;
- detect the sharp end point;
- perform acid-base (oxalic acid and sodium hydroxide) and, redox, (ferrous ammonium sulphate and potassium permanganate) titrations;
- calculate the strength of the given solution by using appropriate formula.

13.2 WHAT YOU SHOULD KNOW

Volumetric analysis is quantitative analysis in which the results are expressed in a certain definite volume. It involves the use of at least one solution of known strength. It may be prepared by dissolving a definite amount of a solute in a solvent to get a known volume of solution. The volume of this solution which reacts with a volume of the solution of unknown strength of another substance is determined. The process is known as titration.

13.2.1 Handling the apparatus

In volumetric analysis, we deal with volumes of solutions. Therefore, these should be measured correctly. The glass apparatus must be clean and free from grease etc. Errors due to parallax in reading the level of a solution in burette, pipette and volumetric flask should be avoided.

To read the volume in the container shown in Figure (13.1) the point shown by the arrow is the correct position.

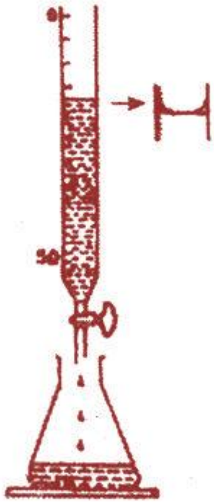


Fig. 13.1 : *Correct way to note down the burette readings*

13.2.2 Pipette

Pipettes are available in various capacities (volumes) and a pipette of required volume is used during titration for delivering a liquid out of pipette. It should be held vertically with its tip in contact with the wall of vessel. After the flow of liquid has ceased, a very small solution of the liquid remains in the lower portion of the pipette. It should not be blown out. For this, hold the bulb of the pipette in left hand palm with its upper tip closed with a finger. Touch the lower tip of pipette to the wall of the vessel. Handling of a pipette and correct way to drain out the solutions are shown in set of figures 13.2a and 13.2b. Measuring of solution with pipette is shown in Fig. 13.3.

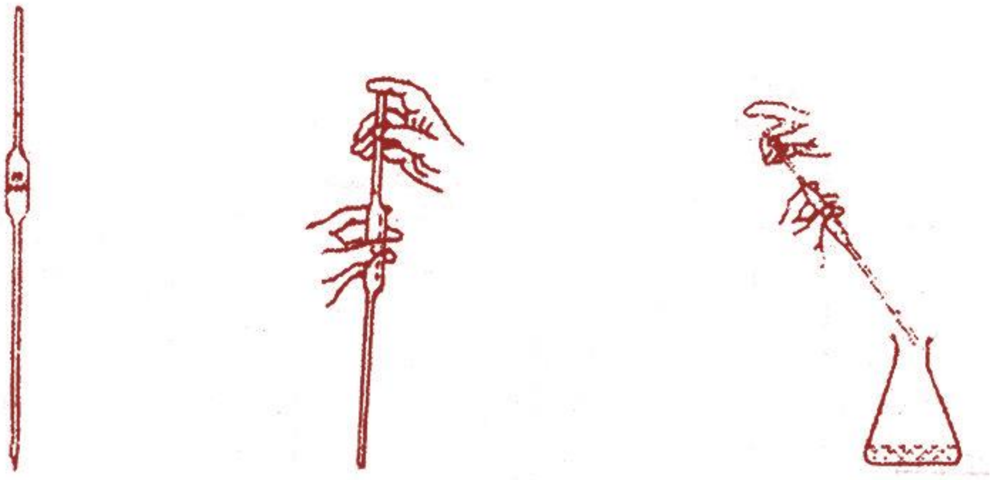


Fig. 13.2: *(a) Pipette (b) Handling of a pipette (c) Correct way to drain out the solution*

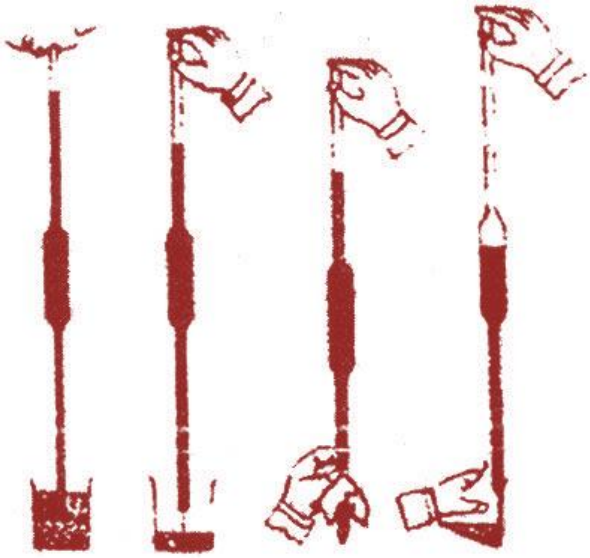


Fig. 13.3 : *Correct way to note down the pipette readings*

13.2.3 Chemical Balance Introduction

A chemical balance is commonly used in chemical laboratories to weigh an exact amount of the substance. The chemical reactions go to completion when exact mass of the substance are taken. Each molecule or atoms of the substance has its own importance. Hence it is necessary to use exact mass of the substance in each experiment. Therefore, the trip balance is not useful for reaction concerned. Chemical balance is used for weighing accurate mass of the substance.

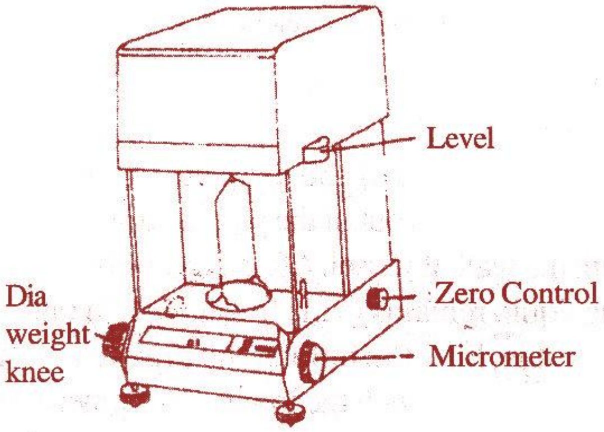


Fig. 13.4 : *Single Pan Balance*

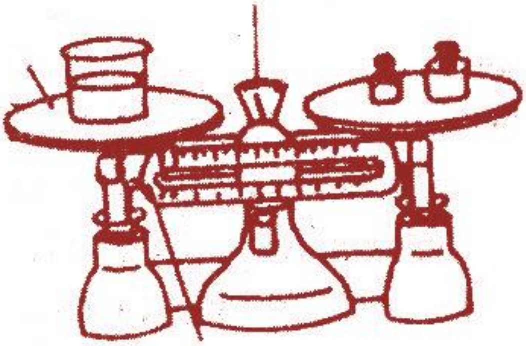


Fig. 13.5 : *Rough Balance*

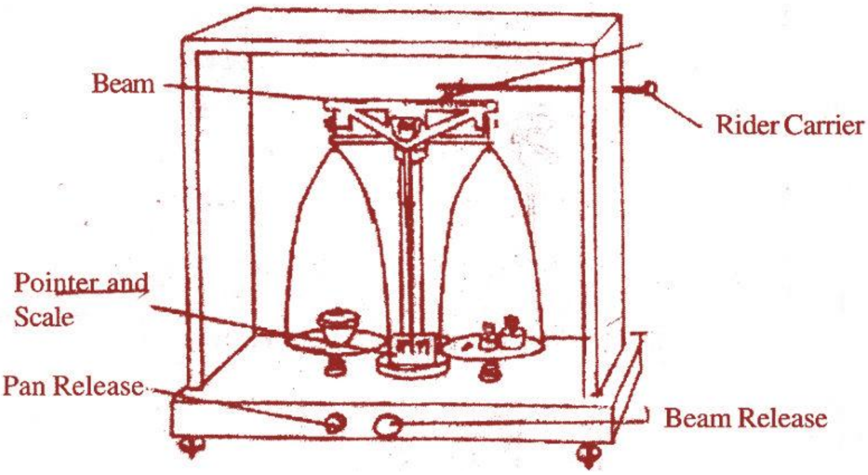


Fig. 13.6 : *Analytical Balance*

The analytcal balance is an instrument for determining mass of body by equilibrium of weights suspended from the opposite side of a bar having a fulcrum at its centre. It may be regarded as a rigid beam having a fulcrum at its centre and two arms of equal length. The chemical balance is shown in the Fig. (13.7).

A chemical balance consists of a light-weight beam having a mounted knife edge at its centre. It rests on a pure corundum plate attached to the pillar. There are two knife edges mounted on the two ends of the beam which are equidistant from the central knife-edge. The two ends of the beam also have adjusting screws for Zero-adjustment. The center of the beam is attached to a pointer which moves over the scale at the foot of the pillar. There are two leveling screws at the loose and one pumb-line suspended from the column. The beam is divided into 100 divisions, i.e. 50 divisions on each side of the fulcrum.

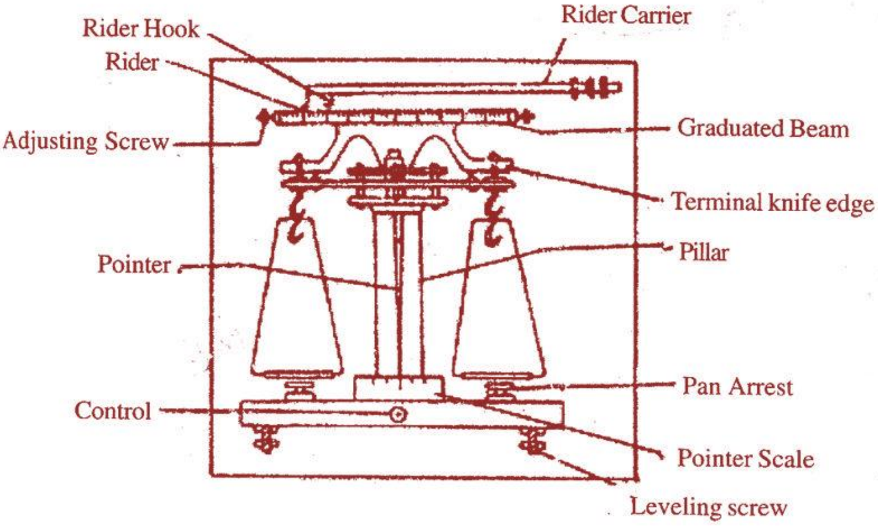


Fig. 13.7 : *A chemical Balance*

Weight - Box

A weight box is a wooden box having grooves of various sizes into which are placed different weights ranging from 1 to 100 as shown in Fig. 13, 8a. These weights are made up of brass coated with nickel or chromium. Each weight is nearly cylindrical having a knob at its one end with the help of which it can be lifted with forceps.

The following is the order of weights placed in a weight box.

100g, 50g, 20g, 20g

10g, 5g, 2g, 2g, 1g

The weights are places in the following sequence in a weight box.

Fractional weight box

Fractional weights are made up of aluminium or brass coated with chromium or nickel that range from 1 milligrams to 500 milligrams as shown Fig. 13.8b. The fractional weights less than 10 milligrams are not used, instead the use of rider is recommended.

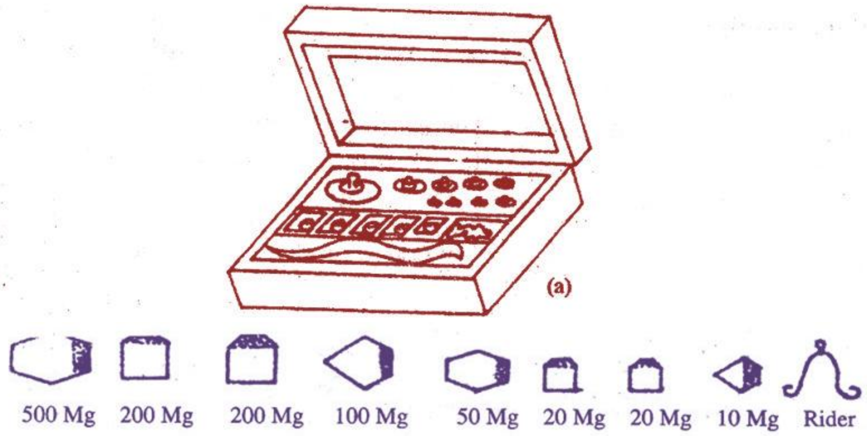


Fig. 13.8 : *(a) A weight box (b) Fractional weights*

Primary Standard

The standard solution of some substances can be prepared directly by weighing. These substances are available in their pure forms and do not undergo chemical change on storing. These are known as primary standards.

The important properties of primary standards are as follows:

i) They are easily available in pure and dry conditions.

ii) They should not undergo any chemical change with air, oxygen and carbon dioxide.

iii) They do not posses hygroscopic, deliquecent and efflorecent properties.

iv) They are easily soluble when added to the solvent (normally water).

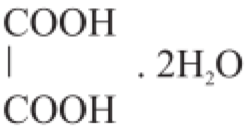
v) They normally possess high molecular mass so that weighing errors are negligible.

vi) The standard solution of these substances should react in a stoichiometric ratio with the volumetric titrants.

vii) They don't react with impurities which are present in the solution to be titrated.

Examples of Primary Standard Substances

Oxalic acid



$$\begin{array}{l}\mathrm{COOH}\\ |\\ \mathrm{COOH}\end{array} .\ 2H_2O$$

Mohr's salt $[FeSO_4(NH_4)_2SO_4\ .\ 6H_2O]$

Secondary Standards

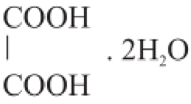
Many chemical substances do not posses the primary standard properties (properties given above). Therefore, they cannot be used to prepare standard solutions. However, the solution of this type of substance are first prepared of approximate strength and then standardised by titrating with a solution of a primary standard.

Example of Secondary Standard Substances

Sodium hydroxide (NaOH), potassium permanganate ($KMnO_4$).

13.2.4 Preparation of solutions of oxalic acid and ferrous ammonium sulphate of known molarity by weighing

Suppose you are asked to prepare 100 mL 1.0M solution of oxalic acid;
As a first step you have to calculate the mass of oxalic acid required for preparing 100 mL of solution.



$$\begin{array}{l}\mathrm{COOH}\\ |\\ \mathrm{COOH}\end{array} .\ 2H_2O$$

Molar mass = 126 g.

126 g. of oxalic acid is required to prepare 1 litre solution of molarity, 1 M.

To prepare 0.1 Molar 100 mL solution, you dissolve 1.26 of the oxalic acid in distilled water and make it upto 100mL. Similarly, to prepare 0.1 M solution of ferrous ammonium sulphate, 3.92 grams of ferrous ammonium sulphate is dissolved is distilled water so as to get 100 mL solution.

a) **Titration of oxalic acid solution against sodium hydroxide**

In this acid-base titration, oxalic acid is completely neutralised by the base (NaOH) according to the following reaction.

According to this reaction, two moles of sodium hydroxide neutralises one mole of acid for complete neutralisation. The end point is indicated by phenophthalein indicator. It is colourless in acid medium and pink in the basic medium.

Now using the empirical relation you can calculate the strength of sodium hydroxide

$$a_1 M_1 V_1 = a_2 M_2 V_2$$

where,

a_1 = Acidity of sodium hydroxide = 1

M_1 = Molarity of sodium hydroxide = ?

V_1 = Volume of sodium hydroxide consumed (burette reading)

a_2 = Basicity of oxalic acid = 2

M_2 = Molarity of oxalic acid = 0.1. M(known)

V_2 = Volume of oxalic acid taken for titration

Values of a_1, V_1, a_2, M_2, V_2 are known. By using the above relation you can calculate M_1.

$$M_1 = \frac{a_2 M_2 V_2}{a_1 V_1}$$

,

(b) Titration of Ferrous Ammonium Sulphate solution against Potassium Permanganate solution

Potassium permanganate is an oxidising agent, both in alkaline and acidic medium. Oxidation is always accompanied by reduction. In this titration, the ferrous ion is oxidised to ferric ion by permanganate ion, [Mn (VII)] and at the same time permanganate ion is reduced to manganese (II [Mn (II)].

The following reactions take place.

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

$$[Fe^{+2} \longrightarrow Fe^{+3} + e^-] \times 5$$

The overall reactions is

Oxidation

$$MnO_4^- + 5Fe^{+2} + 8H^+ \longrightarrow Mn^{+2} + 5Fe^{+3} + 4H_2O$$

Reduction

According to the stiochiometric relation, one mole of the oxidising agent, potassium permanganate, reduces 5 moles of reducing agent, ferrous ammonium sulphate.

Potassium permanganate is a self indicator; a pink colour appears and persists at the end point. To calculate the strength, the following relation is used:

$$a_2 M_1 V_1 = a_1 M_2 V_2$$

Here a_1 and a_2 are the change in oxidation number in oxidant and reductant respectively. For permanganate solution $a_1 = 5$, M, and V, are its molarity and volume respectively $a_2 = 1$ for ferrous ammonium sulphate, M_2 and V_2 are its molarity and volume respectively.

13.3 MATERIALS REQUIRED

(1) Apparatus	(2) Chemcials
Analytical balance,	Oxalic acid, Sodium hydroxide,
Weight-box, Measuring flask	Ferrous ammonium sulphate (Mohr's salt),
Beaker, Glass rod, Conical flask,	Phenolphthalein, Distilled water,
Funnel, Burette stand, Wire gauze,	Potassium permanganate,
Burette, Pipette	Sulphuric acid (dilute)

13.4 HOW TO PERFORM THE EXPERIMENT

To prepare standard solutions of oxalic acid and ferrous ammonium sulphate, the following steps are followed.

Weigh the required amount of substance (section 13.2) using an analytical balance. The substance should be weighed in a weighing bottle.

Transfer the weighed substance into a standard flask (100mL) and dissolve in minimum amount of distilled water. Make the volume of solution up the mark in the standard flask. Shake it by tilting the flask upside down. In case of ferrous ammonium sulphate, before making the solution upto the mark, dilute (sulphuric acid (-15mL) should be added to prevent the hydrolysis.

13.1.4 Acid - base titration

Take a, clean burette, rinse it with the given sodium hydroxide solution and clamp it vertically in a burette stand. Fill the burette with sodium hydroxide solution. Ensure that no air bubbles are there in the stop cock. Note the initial reading (V_1). Rinse a clean 20 mL pipette with standard oxalic acid solution and then pipette out 20mL of the given standard oxalic acid solution into a clean conical flask. Add a few drops phenolphthalein indicator. Hold the conical flask (as shown in figure 13.9) just below the nozzle of the burette and using your left hand, release sodium hydroxide solution into the conical flask drop by drop. Continuously swirl the contents of the conical flask smoothly and continue addition of NaOH solution dropwise til the appearance of pink colour. Note down the reading of the lower meniscus of NaOH solution. This is the final reading, (V). Repeat the titrations to get at least two concordant readings.

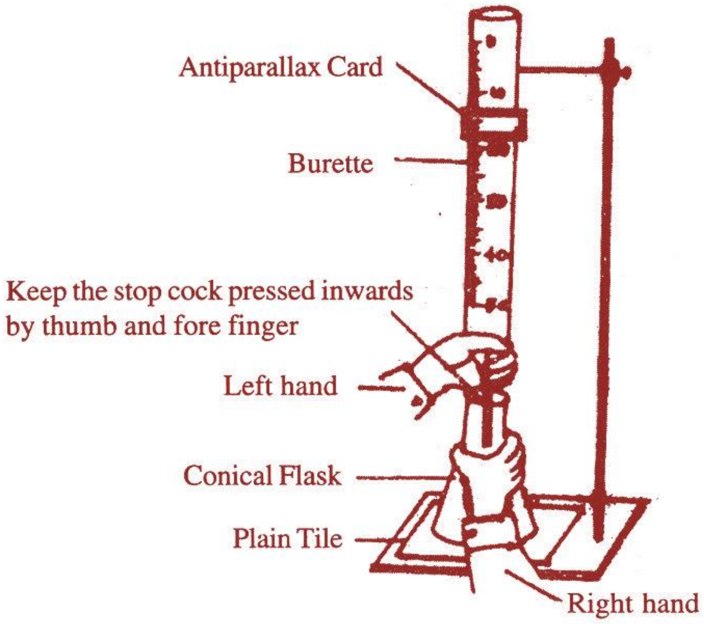


Fig. 13.9 : *Carrying out a titration*

13.4.2 Titration of ferrous ammonium sulphate and potassium permanganate

Take a clean and dry burette and rinse with the given potassium permanganate solution. Fit the burette on stand. Fill with potassium permanganate solution and remove the air bubbles.

Rinse a clean, dry pipette with given Mohr's salt solution. Then pipette out 20mL of Mohr's solution and transfer into a clean 250 mL conical flask. Add approximately 20 mL of dilute sulphuric acid. Titrate it by gently swirling the conical flask. (Refer the figure 13.9) A permanent pink colour appears at the end point. Repeat the titration to get at least two concordant readings.

13.5 PRECAUTIONS

i) Handle the Chemical Balance carefully.

ii) While preparing standard solutions add distilled water into the flask slowly. (Otherwise you will easily miss the mark).

iii) Chemicals should be pure (LR or AR).

iv) Apparatus used should be clean and dry.

v) Only 2 to 3 drops of indicator solution should be used.

vi) At least two concordant readings should be taken for calculations,

vii) While titrating, continuous swirling of the solution is required, to avoid incomplete reaction.

13.6 OBSERVATIONS

Preparation of standard solutions

i) Oxalic acid

Mass of empty weighing tube =

Mass of weighing tube + substance =

Mass of weighing tube after transfer the substance into volumetric flask =

Mass of substance =

ii) Ferrous ammonium sulphate

Mass of empty weighing tube =

Mass of weighing tube + substance =

Mass of weighing tube after transfer the substance into volumetric flask =

Mass of substance =

TITRATION

i) **Oxalic acid Vs. Sodium hydroxide.**

Burette Solution **:** sodium hydroxide.

End point **:** colourless to pink colour.

Burette reading **:**

Sl. No.	Initial reading	Final reading	Volume of NaOH used
1.			
2.			
3.			
4.			

Volume of oxalic acid solution taken in each titration, V = 20 mL

Molarity of oxalic acid = M_1 =

Volume of NaOH consumed (concurrent reading) = V_2

Molarity of Sodium hydroxide = M_2 = ?

The reaction

Acid		**base**
$a_2 M_1 V_1$	=	$a_1 M_2 V_2$
$\mathbf{a_2 = 1}$		$\mathbf{a_1 = 2}$

$$M_2 = \frac{a_2 M_1 V_1}{a_1 V_2}$$

Molarity of the given sodium hydroxide is .... mol $^{-1}$

ii) Ferrous ammonium sulphate *Vs*. potassium permanganate.

Burette solution **:** Potassium permanganate

End Point **:** Colourless to pink colour:

Burette readings:

S.No.	Initial readings	Final reading	Volume of $KMnO_3$ consumed (mL)
1.			
2.			
3.			

Relation of volume and molarity

$$a_2 \quad M_1 \ V_2 \ = a_1 \ M_2 \ V_2$$

where a_1 and a_2 changes in oxidation state (oxidation number), with respective species

Volume of Mohr's salt solution taken = V_1 = 20 mL

(Pipette readings)

Molarity of standard mohr's salt solution is M_1 =

Change in oxidation state of Mn in MnO^-_4 is a_2 = 5

Volume of $KMnO_4$ consumes = V

Molarity of $KMnO_4$ is M_2 = ?

By substituting a_1, a_2, M_1, V_1 and V_2, M_2 can be calculated.

Concentration of the given potassium permanganate solution is = ... mol 1^{-1}.

13.7 CONCLUSION

i) Strength of sodium hydroxide and potassium permanganate are determined by titrating them with oxalic acid and ferrous ammonium sulphate solutions respectively. Their respective molarities are ____________________ and __________________________.

13.8 CHECK YOUR UNDERSTANDING

1. State the need of rinsing the burette and pipette with respective solutions to be used in the titration.

   ....

2. Why should pure chemicals be used for preparing a standard solution?

   ....

3. Why only the lower meniscus should be taken into consideration while reading the level of solution.

   ....

4. How do last (final) drop of solution in pipette can be taken out?

   ....

5. Why do the secondary standard solutions cannot be used in the standard solution preparation?

   ....

13.9 NOTE FOR THE TEACHERS

i. Teacher should demonstrate to the students the method to handle an Analytical Balance.

ii. Teacher should ensure that the chemicals are pure enough and that distilled water is used in the titration. The appearance of the end point and the concept of concurrent readings should be made clear.

iii. The reaction which is occurring in the titration should be explained to the students.

13.10 CHECK YOUR ANSWERS

Ans. 1: To avoid error in the calculations

Ans. 2: The impurities may react and interfere in the reaction. Change in weights will lead to errors in the calculations.

Ans. 3: The solution level in the burette is always in the curved manner (concave). Therefore if we take the upper meniscus, there will always be a loss in the volume of the solution.

Ans. 4: After releasing all the solution just touch the edge of pipette on the sides of the conical flask.

Ans. 5: (a) Secondary standards are not available in the pure and dry forms.

(b) It may undergo reaction with solvents

(c) It may undergo hydration and alter its own weights during the process.

(d) It may react with air or oxygen and carbon dioxide.

EXPERIMENT-14

Qualitative analysis of a salt involving detection of one anion and one cation from the following (salts insoluble in hydrochloric acid excluded)

Anions:

CO_3^{2-} , S^{2-} , NO_2^- , Cl^- , Br^- , I^- , NO_3^- , SO_4^{2-} , PO_4^{3-}

Cations:

NH^{4+}, Pb^{2+}, Cu^{2+}, Fe^{3+}, Ni^{2+}, Zn^{2+}, Mn^{2+}, Ca^{2+}

14.1 OBJECTIVES

After performing this experiment, you should be able to:

- explain the meaning of Qualitative Analysis;
- handle properly the chemicals and reagent bottles in the chemistry laboratory;
- perform simple experimental operations such as heating, testing of gas evolved, filtration and 'washing of residue etc.;
- observe the appearance/disappearance of colour, precipitate, turbidity ect. in reaction mixture;
- acquire an elementary idea of the presence of an anion or a cation in the salt by carrying out dry test; and
- perform preliminary and confirmatory tests of anions and cations and analyse the given salt.

14.2 MATERIALS REQUIRED

Apparatus

Test tubes, Test tube holder, Funnel, Glass rod, Tripod stand, China dish Platinum wire /Nichrome wire, boiling tube, Brush, Breaker, Wire gauze, Spatula, Watch glass

Chemicals

Hydrogen Sulphide gas

Solution

Lime water
Starch solution
Ammonium hydroxide
Potassium sulphocyanide
Acetic acid (dilute)
Sodium hydroxide
(dilute and concentrated)
Hydrochloric acid
(dilute and concentrated)

Silver nitrate $AgNO_3$ (aq)
Sodium nitroprusside (aq)
Barium chloride (aq)
Potassium ferrocyanide (aq)
Nesseler's reagent
Nitric acid
(dilute and concentrated)
Dimethyl glyoxime solution

Lead acetate (aq)
Potassium iodide (aq)
Potassium chromate (aq)
Ammonium oxalate (aq)
Blue litmus solution
Sulphuric acid
(dilute and concentrated)

Solids

Ammonium chloride
Ammonium carbonate
Ammonium Molybdate
Potassium dichromate
Ferrous sulphate

14.3 WHAT YOU SHOULD KNOW

Qualitative analysis of unknown salt consists of the detection and identification of the constituent ions. The inorganic salts on dissolving in water dissociate completely into positively and negatively charged ions. An positively charged ion is called cation or a basic radical and the negatively charged ion is called an anion or an acid radical. A number of tests are carried out to identify the ions.

Qualitative analysis is done by carrying out two kinds of tests (i) dry tests, and (ii) wet tests.

The dry tests are performed on solid inorganic substances. These tests should be performed before proceeding with wet tests.

I. during a dry test, we note

i) Physical examination of salt such as colour, smell and density of the salt gives valuable clues regarding the nature of some of the basic and acidic radicals.

ii) Action of heat on dry salt.

II. The wet tests are carried out in solution. In a wet test, we note

i) The colour and smell of the gas evolved.

ii) Formation or disappearance of colour of solution,

iii) Formation or disappearance of a precipitate.

A test may be positive or negative. A positive test is the one which gives the result indicated in the theory and a negative test does not give the results as indicated in the theory.

For example, to test a carbonate ion, you add dilute sulphuric acid to the substance. The colourless gas evolved is passed through lime water which turns milky (a positive test). If the lime water does not turn milky it is a negative test.

To detect the anon and cation in a salt, certain chemicals are used. These chemicals are called **Reagents.** When reagents react with salts, new compounds are formed with some observable properties such as colour, smell and characteristic appearance of precipitate.

Before carrying out systematic analysis, it is necessary to know few important laboratory techniques.

14.3.1 Laboratory Techniques

To identify an anion and a cation in a salt, some techniques have to be used. The techniques are as follows:

1. Heating of salt or solution in a test tube

i) While heating a salt or a solution in a test tube, hold the test tube in such a manner that the mouth of the test tube is away from yourself or any other person working in adjacent place.

ii) Heat the test tube gently by placing its one side in the outermost zone of the flame. While heating, shake the test tube occasionally to avoid any spurting.

iii) Always heat the top layer of liquid in a test tube, so that it boils quickly. Never apply flame on the bottom of test tube, otherwise bumping will start. You may use a piece of porcelain to avoid bumping.

iv) While heating for a long time, use a test tube holder. Hold it between your thumb and the fingers if the volume in the test tube is less than half and you have to only warm the contents.

2. Use of a Reagent Bottle

Take out the desired reagent bottle from shelf. Remove the stopper and hold it in right hand. Hold the test tube between the thumb and first two fingers of the left hand. Now pour the required quantity of the reagent along the side of the test tube. Put the bottle back on the shelf and stopper it immediately. Never put the stopper on the table.

3. Testing of a gas evolved: On adding a reagent to a salt if effervescence are given out in cold or on heating it indicates the evolution of a gas.

The gas evolved can be tested in following manner:

i) Note the colour of gas evolved

ii) Smell the evolved gas by puff of hand. Never inhale a gas by placing the nose directly into the vapour.

iii) By brining a filter paper strip or a glass rod with its end dipped into the testing reagent near the mouth of the test tube.

iv) By passing the gas evolved for few seconds through a reagent taken in a test tube, shake the test tube well and observe the change.

4. Precipitation: Insoluble compound formed by addition of a reagent to a solution under the test or passing a gas through the solution under the test is called a precipitate and the process is called precipitation. As soon as a clear solution turns turbid, it is an indication that precipitate is formed.

In some cases the precipitate may dissolve in excess of precipitating reagent, in that case two observations should be taken, one on addition of precipitating reagent dropwise and second on adding excess of precipitating reagent.

5. Filtration: It is a process by which an insoluble compound is separated from a reaction mixture. To carry out filtration, first the filter paper cone is made. It is then opened in such a way that the three layers of paper are on one side, while the fourth layer is on the other side. By doing so a hollow cone of filter paper is obtained. The cone is then places in funnel and by wetting with water; it is fixed properly but gently in funnel. The liquid mixture is then poured over the filter paper funnel along the side of a rod. Never fill the filter paper cone more than 2/3 rd of its capacity. Note that there should be no space between filter paper cone and glass funnel. It reduces the rate of filtration. The liquid passes through the pores of filter paper and collects in the vessel kept below the stem of the funnel. The clear liquid so collected is called Filtrate. The insoluble compound or precipitate, which remains over the filter paper is called residue.

6. Washing and collection of perceptible: It is essential to wash the precipitate before carrying out any test on it. The washing is done by slowly pouring distilled water over the precipitate while it is still the runnel. After washing carefully remove the filter paper from funnel open up and spread it on a dry filter paper and remove the precipitate carefully with the help of a spatula and collect it on a watch glass. Use a small portion of it each time for carrying out various tests.

7. Dry test: A number of tests can be carried out using the powdered salt. The information thus obtained often provides a clue to the presence or absence of certain radicals. With their knowledge, the course of wet analysis may be shortened or modified. Some of the important dry tests are given below:

i) Physical examination: Examine the colour, smell and the density of salt.

S.No.	Observation	Inferences
1.	**The colour of solid** i) Blue ii) green (light or dark) iii) Yellow iv) Pink v) Buff vi) White	Copper salt may be present Copper, nickel and ferrous salt may be present Ferric salt may be present Manganese salt may be present Manganese salt may be present Salts of remaining cations may be present **(Ca^{2+}, NH_4^+, Pb^{2+}, Zn^{2+})**
2.	**Smell :** Rub a pinch salt with the help of a spatula on a watch glass smell of ammonia, smell of rotten eggs	Ammonium salt may be present Sulphide salt may be present
3.	**Density** Salt is light	Carbonates of zinc and calcium may be present

ii) Dry heating test: The salt is heated gently and then strongly in a clean and dry test tube.

S.No	Observation	Inferences
1.	Solid melts and resolidifies	Salts of calcium may be present
2.	Solid swells up	phosphate may be present
3.	Solid decrepitates (Craking sound)	Lead nitrate may be present
4.	Solid sublime and vapour are white in colour	Ammonium chloride may be presents

14.3.2 Precautions

i) Use a perfectly dry test tube for performing this test.

ii) Keep the mouth of the test tube away from yourself as well as from your neighboring students.

iii) During heating, do not heat the tube at one point but keep it rotating otherwise the tube may crack.

iv) Do not smell the gases evolved by placing the nose directly over the mouth of the tube.

v) Always smell the gas by puff of your hands.

List of anions: CO_3^{2}, S_2^-, NO_2^-, Cl^-, Br^-, I^-, NO_3^-, SO_4^{2-} & PO_4^{3-}

14.4.1 Preliminary tests

The method of detection of anions is not as systematic as that of basic radicals. It has not been possible to include acidic radicals (anions) into distinct groups as in the case of cations (basic radicals).

The process employed to detect an anion may be divided into two classes.

A) Those involving the identification of volatile products obtained on treatment with acids. This test can be further divided into the following two groups.

I) Action of dilute sulphuric acid

II) Action of concentrated sulphuric acid.

B) Those dependent on reaction in solution.

Before carrying out a confirmatory test in solution it is necessary to prepare a water solution or sodium carbonate extract of the salt.

i) Preparation of water solution for anion analysis: Take a pinch of given salt in a test tube. add 2-3 mL of water and shake well.

ii) Preparation of sodium carbonate of extract: If the salt is insoluble in water, preparation of sodium carbonate extract in necessary.

This is prepared as follows:

Mix the salt with nearly twice its weight of sodium carbonate. Add sufficient distilled water in a dish, boil and filter. The filtrate is known as sodium extract.

How to use sodium carbonate extract: Sodium carbonate extract always contains an excess amount of unreacted sodium carbonate which may interfere in the usual tests of acid radicals. It is absolutely necessary to destroy the excess of sodium carbonate. This is done by acidifying sodium carbonate extract with a suitable acid (choice depends upon the nature of the acid radical to be tested).

A) Action of Acids

I) Test with dilute sulphuric acid : The anions which can be detected by treating a slat with dilute H_2SO_4, are as follows :

CO_3^{2-}, S^{2-} and NO_2^-

A gas in given out on adding dilutes H_2SO_4 to salt. For observations and explanation follow the given table.

Anions	Observation	Explanation	Inferences
$CO^{2}_{3}{}^{-}$	Brisk effervescence a colourless odourless gas is given out	$MCO_3 + H_2SO_4$ $\rightarrow M\,SO_4 + H_2O + CO_2$ carbon dioxide (colourless)	CO_3^{2-}
S^{2-}	Colourless gas with Suffocating smell of rotton eggs is given out	$MS + H_2SO_4$ $\rightarrow M\,SO_4 + H_2S$ Hydrogen sulphide (colourless)	S^{2-} may be present
NO_2^-	A brown coloured gas is evolved	$2\,M\,NO_2 + H_2SO_4$ $\rightarrow M_2SO_4 + 2HNO_2$ Nitrous acid (colourless) $3MNO_2 \rightarrow H_2O + HNO_3 + NO$ $NO + O_2 \rightarrow 2NO_2$ Nitrogen dioxide (Reddish brown)	NO_2^- may be present

Do not boil the contents of the test tube. Over heating decomposes H_2SO_4 and gives sulphurdioxide.

II) Confirmatory tests of CO_3^{2-}, S^{2-} and NO_2^- ions

1) *Carbonate ion CO_3^{2-}* : On passing the gas through lime water, it turns milky due to the formation of calcium carbonate.

$$Ca(OH)_2 + CO_2 \rightarrow \underset{\text{Calcium carbonate}}{CaCO_3\downarrow} + H_2O$$

On passing excess of CO^{2+} the milkiness disappears and a clear solution is obtained.

$$CaCO_3 + CO_2 + H_2O \longrightarrow Ca(HCO_3)_2$$

2) *Sulphide ion, S^{2-} :*

a) Filter paper moistened with lead acetate turns black on exposure to gas.

$$Ph(CH_3COO)_2 + H_2S \longrightarrow 2CH_3COOH + PbS\downarrow$$

Black lead Sulphide

b) *Sodium Nitroprusside test* : A violet colouration is obtained on adding freshly prepared solution of sodium nitroprusside to salt solution.

$$Na_2S + Na_2\,[Fe(NO)(CN)_5] \longrightarrow Na_2\,Na_2\,[Fe(CN)_5NOS]$$

Sodium Nitroprusside Violet colouration

3) *Nitrite ion, $NO_2^{\ -}$* : When a filter paper soaked in a mixture of KI^+ starch solution is exposed to the gas, it turns blue or violet in colour.

$$2NO_2 + 2KI \longrightarrow 2KNO_2 + I_2$$

I_2 + Starch - Starch iodide complex, deep blue or violet.

III) test with concentrated sulphuric acid: This test is performed after performing the test with dilute H_2SO_4. Anions which give positive test with dilute H_2SO_4 will also react with concentrated H_2SO_4. On adding concentrated H_2SO_4 to a salt, if a gas is given out, follow the given table for drawing an inference.

Ion	Observations	Explanation	Inferences
Cl^-	A colourless gas with pungent smell Hydrogen Chloride	$MCl + H_2SO_4$ $MHSO_4 + HCl$ Hydrogen Chloride (colourless gas)	Cl^- may be present
Br^-	A brown gas with pungent smell Contents of the test solution turns reddish brown	$MBr + H_2SO_4 \rightarrow MHSO_4 + HBr$ $2HBr + H_2SO_4 \rightarrow H_2O + SO_4 + Br_2$ Bromine (reddish brown gas)	Br^- may be present
I^-	Violet fumes with pungent smell and Black specks appeared on the sides of the test tube	$MI + H_2SO_4 \rightarrow MHSO_4 + Hl$ $HI + 2H_2SO_4 \rightarrow 2H_2O + SO_2 + I_2$	Iodide may be present purple (violet)
NO_3^-	Brown coloured gas with pungent smell is evolved, the gas intensified on adding copper turning	$2MNO_3 + H_2SO_4 \rightarrow M_2SO_4 + 2HNO_3$ $4\,HNO_3 \rightarrow 2H_2O + O_2 + 4NO_2$ Nitrogen dioxide (light brown gas) $3Cu + 8HNO_3 \rightarrow 2Cu\,(NO_3)_2 + 2NO + 4H_2O$ Nitrogen oxide (colourless) $2NO + O_2 \rightarrow 2NO_2$ Nitrogen dioxide (Brown gas)	NO_3 may be present

Precautions

1. Do not boil the contents of test tube.

2. After the test is over, do not throw away the contents of test tube immediately into sink The reaction of concentrated H_2SO_4 with water is highly exothermic and generate a lot of heat.

3. Do not inhale the gas evolved during the test because they are corrosive in nature.

4. Concentrated H_2SO_4 should be handled carefully.

IV) Confirmatory tests of Cl^-, Br^-, I^- and NO_2^-

a) Chloride ion, (Cl^-)

i) Silver Nitrate test: A curdy white precipitate insoluble in conc. HNO_3 is obtained on adding silver nitrate to water solution Acidify with dil. HNO_3 before adding $AgNO_3$ in case sodium carbonate extract in used.

$$Ag^+ + Cl^- \rightarrow AgCl\downarrow$$

Curdy white precipitate is soluble in ammonium hydroxide.

$$AgCl + 2NH_4OH \rightarrow [Ag(NH_3)_2]^+ + 2H_2O$$

Diammine silver chloride (Colourless)

ii) Chromyl Chloride test

Yellow fumes of Chromyl Chlorides are obtained on heating a mixture of salt and solid $K_2Cr_2O_7$ and concentrated H_2SO_4 in a dry test tube.

$$2MCl_2 + K_2Cr_2O_7 + 3H_2SO_4 \rightarrow 2MSO_4 + K_2SO_4 + 2CrO_2Cl_2 + 2H_2O$$

Chromyl chloride (orangish red)

Chromyl chloride fumes turns dilute NaOH yellow

$$CrO_2Cl_2 + NaOH \rightarrow Na_2CrO_4 + 2HCl$$

Sodium Chromate (yellow)

A yellow precipitate of lead chromate, $PbCrO_4$ is formed on adding lead acetate to the yellow solution acidified with CH_3COOH

$$Na_2CrO_4 + Pb(CH_3COO)_2 \rightarrow PbCrO_4\downarrow + 2CH_3COONa$$

lead chromate (yellow precipitate)

(b) Bromide ion, Br^-

i) Silver nitrate test: A light yellow precipitate is obtained on adding silver nitrate to water solution (after acidifying the sodium carbonate extract with dil. HNO_3). The precipitate is partially soluble in ammonium hydroxide.

$$Ag^- + Br^- \rightarrow AgBr\downarrow$$

Silver bromide (light yellow)

$$AgBr + 2NH_4OH \rightarrow [Ag(NH_3)_2]Br + 2H_2O \text{ in } CCl_4$$

ii) Organic layer test: The addition of chlorine water to water solution containing bromide liberates free bromine which imparts orange red colour to the test solution. On shaking with carbon tetrachloride, a reddish brown colour is imparted in CCl_4 layer. The organic layer of CCl_4 is obtained below the colourless aqueous layer.

$$2NaBr + Cl_2 \rightarrow 2NaCl + Br_2$$

Bromine, reddish brown in CCl_4

c) Iodide ion, I^-

i) Silver nitrate test: Iodide ion (I^-) A yellow precipitate is obtained on adding $AgNO_3$ solution to acidified (with dil. HNO) water solution, which is insoluble in NH_4OH.

$$Ag^+ + I^- \rightarrow AgI$$

Silver iodide, yellow precipitate

ii) Organic layers test: The addition of chlorine water to test solution liberates free iodine, which colours the solution, violet, On shaking with carbon tetra chloride, a violet colour is imparted to the organic layer.

$$2NaI + Cl_2 \rightarrow 2NaCl + I_2$$

Iodine (Violet Colour in CCl4)

d) Nitrate ion, (NO_3^-) :

Ring test: On adding concentrated. H_2SO_4 gradually along the side of the test tube to a mixture of salt solution in water or its sodium carbonate extract, acidified with dil. H_2SO_4, and freshly prepared ferrous sulphate solution, a brown ring is obtained at the junction of two layers.

$$M(NO_3)_2 + H_2SO_4 \rightarrow MSO_4 + 2HNO_3$$

$$6FeSO_4 + 2HNO_3 + 3H_2SO_4 \rightarrow 3Fe_2(SO_4)_3 + 4H_2O + 2NO$$

$$X\,FeSO_2 + Y\,NO \rightarrow (FeSO_4)_x\,(NO)_Y$$

Nitrosoferrous sulphate,(brownring)

$$Fe^{2+} + 5H_2O + NO \rightarrow [Fe(H_2O)_5 + NO]^{2+}$$

B) Individual tests

a) Sulphate ion, SO_4^{2-} : On adding $BaCl_2$ solution to acidified (dil. HCl) salt solution or its sodium carbonate extract a white precipitate is obtained which is insoluble in concentrated HCl or concentrated HNO_3.

$$Ba^{2+} + SO4^{2-} \rightarrow BaSO4$$

Barium chloride white precipitate

(b) Phosphate ion, (PO_4^{3-}): salt or its water solution or its sodium carbonate extract is heated with concentrated, HNO_3 till the fumes no longer evolve. Contents of the test tube are diluted with water. A canary yellow precipitate of ammonium phosphomolybdate is formed on warming the above test solution with ammonium molybedate.

$$PO_4^{-3} + 12MoO_4^{-2} + 24H^+ + 3NH_4^+ \xrightarrow{\triangle} (NH_4)_3PO_4.12MogO_3.6H_2O$$

Ammonium phosphomolybdate
canary yellow precipirate

Precautions

1. Some times on adding $BaCl_2$ solution, a thin white precipitate is obtained, it may be due to formation of barium phosphate which is soluble in concentrate HCl.

2. During the test of phosphate ion, use a small porcelain piece or pumice stones while heating the salt or water solution with concentrated. HNO_3, to avoid bumping of contents of test tube.

3. Use cone HNO_3 carefully.

14.5 IDENTIFICATION OF CATIONS

For, identification of cations (excluding NH_4^+ ion belonging to group zero) have been divided into six groups depending upon the differences in solubility of chlorides, sulphides, hydroxides and carbonates. Of these ions, the cations are precipitated by adding some reagents called, group reagents.

The following table gives briefly about the classification of basic radicals (cations) into groups, Group reagents and form in which they precipitate.

Group	Cations	Group reagent	Form in precipitate which Cation precipitate
0	NH_4^+	Conc. NaOH	No precipitate obtained. A Colourless gas ammonia with pungent smell is evolved on worning
I	Pb^{2+}	dil. HCl	Chloride
II	Cu^{2+}	H_2S(g) in the presence of dil HCl	Sulphide
III	Fe^{3+}	excess of NH_4OH in presence of excess of NH_4Cl(s)	Hydroxide
IV	Zn^{2+}, Mn^{2+}, Ni^{2+}	H_2S(g) in the presence of NH_4Cl(s) and NH_4OH excess	Sulphide
V	Ca^{2+}	$(NH_4)_2CO_3$ soln. in the presence of NH_4Cl and NH_4OH	Carbonate

Before proceeding to detect the cations the solution of the salt should be prepared.

Preparation of original solution: Take a pinch of salt in a clean test tube. Add 2-3 mL of distilled water, shake well. If the solid is not soluble in cold water then heat the contents. If the salt is still not soluble in hot water add few drops of conc. HCl,

Precautions: Do not add excess of conc. HCl. This might create a problem during cation analysis.

a) If on addition of conc. HCl a gas is evolved, wait till the effervescence ceases and then add 2-3 drops more of conc. HCl.

b) Use only distilled water to prepare original solution.

General rules for the procedure involving the analysis of cations

1) The group reagent should be added in the systematic order.
2) Test for the higher group radicals are performed only when the radicals of a lower group are found absent.
3) Slight excess of the group reagent should be added to ensure complete precipitation of group radicals.
4) When a precipitate is obtained for a particular group, it is filtered and the residue is used for the analysis of the radicals of that group. Before carrying out the analysis, wash the residue two times with distilled water. Washing should be discarded.
5) To find out the presence of a cation in a particular group, a small portion of the solution is used which is known as the test portion.

14.5.1 Cation Analysis

(a) Group Zero, (Ammonium ion) NH_4^+

Ammonium ion, (NH_4^+): On warming with NaOH solution, a colourless gas with pungent smell of ammonia is evolved.

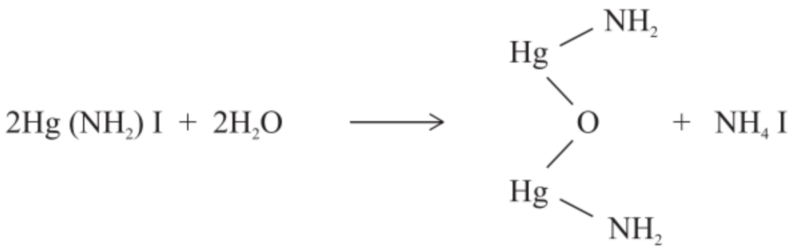
$$NH_4^+ + OH^- \xrightarrow{\triangle} \underset{\text{Ammonia (Colourless)}}{NH_3\uparrow} + H_2O$$

Confirmatory test:

On passing the gas through Nessler reagent a reddish brown precipitate is obtained.

$$\underset{\text{Nessler's reagent}}{K_2HgI_4} \rightarrow 2KI + HgI2$$

$$HgI_2 + 2NH_3 \rightarrow \underset{\text{(Amino - mercuric iodide)}}{Hg(NH_2)I + NH_4I}$$



Reddish brown, precipitate Iodide of millions base

(b) Group-I (Pb^{2+})

If a white precipitate is obtained on adding dilute HCl to salt solution in water or while preparing original solution in dilute HCl, it indicates the presence of Pb^{2+} ions. White precipitates dissolves again on warning and re-precipitates on cooling.

$$Pb^{2+} + Cl^- \rightarrow PbCl_2$$

Lead chloride (white precipitate)

1) KI test: On adding KI to hot solution of lead chloride, a yellow precipitate is obtained.

$$Pb^{2+} + 2I^- \rightarrow PbI_2 \downarrow$$ Lead iodide (Yellow precipitate)

2) Potassium chromate test: On adding K2CrO4 solution to hot-solution of lead chloride, a yellow precipitate is obtained.

$$Pb^{2+} + CrO_4^{2-} \rightarrow PbCrO_4\downarrow$$ Lead Chromate (Yellow precipitate)

(c) Group II: (Cu^{2+})

On passing hydrogen sulphide gas through acidified original solution obtained which indicates the presence of Cu^{2+} ions

$$M^{2+} + S^{2-} \rightarrow MS\downarrow$$

Metal sulphide

1) If no precipitate is obtained even on dilution of test portion, in that case gp II is absent.

2) To obtain a granular precipitate pass H2S for 2-3 minutes through hot solution.

Confirmatory test of Cu^{2+} ion: On heating black ppt of CuS with dilute HNO_3, a blue colour solution is obtained.

$$3CuS + 8HNO_3 \rightarrow 3Cu(NO_3)_2 + 3S + 2NO\uparrow + 4H_2O$$

Copper nitrate,
(blue colour)

1) On adding ammonium hydroxide, precipitate is obtained which is soluble in excess reagent to produce deep blue solution.

$$Cu^{2+} + 2OH^- \rightarrow Cu(OH)_2$$ Copper hydroxide (Blue precipitate)

$$Cu(OH)_2 + 4NH_3 \rightarrow [Cu(NH_3)_4]\ SO_4$$ Tetraammine cupric sulphate (deep blue colour)

2) On adding acetic acid followed by potassium ferrocyanide to blue solution, a chocolate brown precipitate is obtained.

$$2Cu^{2+} + K_4[Fe(CN)_6] \rightarrow Cu_2[Fe(CN)_6] + 4K$$

Copper ferrocyanide,
(chocolate brown precipitate)

d) Group III (Fe^{3+} ions)

Preliminary test: To the original solution add little concentrated HNO_3, and boil to oxidize Fe^{2+} to Fe^{3+} and then add excess of solid NH_4Cl followed by excess of ammonium hydroxide. White or reddish brown precipitate is obtained.

Reddish brown precipitate indicates the presence of Fe^{3+} ions.

Note: Addition of large excess of NH_4OH may start dissolving group III precipitates. Hence it should be avoided.

Confirmatory test of Fe^{3+} ion: Reddish brown precipitate of $Fe(OH)_3$ on dissolving in dilute HCl, produces yellow solution.

$$Fe(OH)_3 + 3HCl \rightarrow FeCl_3 + 3H_2O$$

Yellow solution

1) On adding K4[Fe(CN)6] to yellow solution, a Prussian blue precipitate is obtained.

$$4Fe^{3+} + 3K_4[Fe(CN)_6] \rightarrow Fe_4 [Fe(CN)_6]_3 + 12K$$

Pressian Blue

2) On adding KCNS to yellow solution a red coluration is obtained

$$Fe^{3+} + 3SCN^- \rightarrow [Fe(SCN)]3$$

Ferric thiocyanate
(red colour)

e) Group IV analysis (Zn^{2+}, Mn^{2+} & Ni^{2+} ions)

Preliminary test: Take the original solution and add NH_4OH to make it alkaline pass hydrogen sulphide gas (H_2S) through the solution, a precipitate is obtained.

Formation of white precipitate indicates the presence of Zn^{2+}

Formation of buff (flesh coloured) precipitate indicates the presence of Mn^{2+}

Formation of black precipitate indicates the presence of Ni^{2+}

$$M2+ + S^{2-} \rightarrow MS$$

Metal sulphide

Note: Hydrogen sulphide gas should always be passed slowly; otherwise black residue of FeS from kipps appratus enters the test solution and creates confusion.

2) Solution should smell of ammonia before passing H_2S.

Confirmatory test; Zn^{2+} ion

On dissolving white precipitate of ZnS. in dilute HCl, a colourless solution is obtained Boil off H_2S.

$$ZnS + 2HCl \rightarrow ZnCl_2 + H_2S\uparrow$$

1) On adding small amount of NaOH a white precipitate is obtained which is soluble in excess of NaOH

$$Zn_2+ + 2OH^- \rightarrow Zn(OH)_2$$

Zinc hydroxide, (white precipitate)

$$Zn(OH)_2 + 2OH^- \rightarrow [Zn(OH)_4]^{2-}$$

Tetrahydroxozincate (II) ion

2) Gray white precipitate is obtained on adding potassium ferrocyanide to solution of ZnS in dilute HCl.

$$2Zn^{2+} + K_4[Fe(CN)_6] \rightarrow Zn_2[Fe(CN)_6] + 4K$$

Grey white precipitate

Confirmatory test of Mn^{2+} Buff coloured precipitate of MnS dissolves in dilute HCl.

$$MnS + 2HCl \rightarrow MnCl_2 + H_2S$$

Boil off hydrogen sulphide gas. To the solution obtained above, on adding NaOH, a white precipitate of $Mn(OH)_2$ is obtained. The precipitate rapidly oxidises on exposure to air turning brown.

$$MN^{2-} + 2OH^- \rightarrow Mn(OH)_2$$

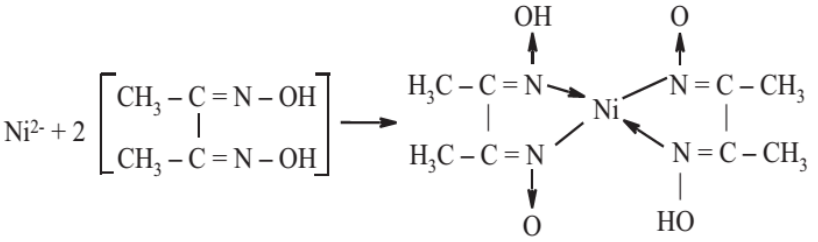
Manganese hydroxide (white precipitate)

$$Mn(OH)_2 + O_2 \rightarrow MnO_2 . H_2O$$

Hydrated Manganese dioxide, (brown precipitate)

Confirmatory test: *Ni*$^{2+}$ ion - Black precipitate of NiS is soluble in hot conc. HNO_3 giving light green solution. On adding NH.OH and dimethyl glyoxime reagent to above solution, a cherry red precipitate of nickel dimethylglyoxime is obtained.

$$3NiS + 2HNO_3 + 6H^+ \rightarrow 3Ni_2 + 2NO + 4H_2O + 3S\downarrow$$



Nickel dimethylglyoxime Cherry red precipitate

b) Group V (Ca^{2+} ion)

Preliminary test: A pinch of NH_4Cl is added to original solution. On adding excess of NH_4OH to make the solution alkaline then add freshly prepared $(NH_4)_2CO_3$ solution, a white precipitate is obtained.

$$M^{2-} + CO_3^{2+} \rightarrow MCO_3$$

Metal carbonate (white precipitate)

White precipitate is soluble in acetic acid

$$MCO_3 + 2CH_3COOH \rightarrow \underset{\text{Metal acetate (Colourless)}}{M(CH_3COO)_2} + H_2O$$

A white precipitate of calcium oxalate is formed on adding ammonium oxalate to above solution.

$$Ca(CH_3COO)_2 + (NH_4)_2C_2O_4 \rightarrow \underset{\text{(Clacium oxalate (white precipitate)}}{CaC_2O_4} + 2CH_3COO\,NH_4$$

14.6 HOW TO PERFORM THE EXPERIMENT

Tabular Presentation of qualitative test

14.6.1 Acid Radicals

i) Work sheet No. 1 Carbonate ion (CO_3^{2-}), Sulphide ion (S^{2-}) and Nitrite ion (NO_2^-)

Experiment	Observation	Inference
1) Carbonate ion, (CO_3^{2-}) Take a small in a clean test tube. Add 1/mL of dilute H2SO4. Lime water test Pass the gas evolved through 2-3mL of lime water and shake well	Brisk effervescence Colourless, odourless, Lime water turns milky	Carbonate, (CO_3^{2-}) may be present CO_3^{2-} confirmed
2) Sulphide ion, (S^{2-}) Take a small amount of given salt in a clean test tube. Add 1mL of dil H_2SO_4.	Colourless gas with suffocating smell of rotten egg is given out	Sulphide, S^{2-} may be present
i) Confirmative test : Bring a piece of filter paper moistened with lead acetate solution near the mouth of test tube	Paper turns black	Sulphide S^{2-} confirmed
ii) Sodium nitroprusside test: To 2 mL of Water solution of salt add 1 prepared sodium nitroprusside solution shake well	A red colouration is obtained	Sulphide S^{2-} confirmed
3) Nitrite ion, NO_2^- Take a small amount of give salt in a clean test tube. Add 1 mL of dilute H_2SO_4	A brown coloured gas with pungent smell is given out	Nitrite, (NO_2^-) may be present
Test the gas evolved in preliminary test with a filter paper soaked in mixture of KI and starch solution	Blue black or violet colour is obtained	Nitrite, (NO_2^-) ion confirmed

ii) Work sheet No. 2 : Chloride ion (Cl^-), Bromide (Br^-), Iodide ion (I^-) and Nitrate ion NO_3

Experiment	Observation	Inference
A) Chloride ion, (Cl^-) Take a small amount of given salt in clean and dry test tube. Add 2-3 drops of conc. H_2SO_4 carefully. Heat the test tube gently.	A colourless gas with pungent smell is evolved	Chloride ion (Cl^-) may be present
i) Chromyl Chloride test : Test a pinch of salt and solid potassium dichromate in a ratio of 1:3 in a dry test tube. Add 3-4 drops of concentrated H_2SO_4 and heat strongly.	Orange-yellow fumes are given are out	
ii) Pass the gas evolved through NaOH solution. Shake well.	A yellow solution is obtained	
iii) Acidify the soln. obtained above with acetic acid neutralize with excess of NaOH and then add lead acetate solution to it. Shake well.	A yellow precipitate is obtained	Cl^- confirmed
ii) Silver nitrate : (i) To 5mL of original solution add dil. HNO_3 to acidify it and then add 1 mL of $AgNO_3$ solution. Shake well	Crudy white precipitate is obtained	Cl^- may be present
ii) Filter the precipitate. After washing the precipitate add 2-3 mL of ammonium hydroxide solution to it. Shake well	A colourless precipitate is soluble	Cl^- confirmed
(B) Bromide ion, (Br^-) Take a small amount of given salt in a clean dry test tube Add 2-3 drops of concentrated H_2SO_4 . Heat the test tube carefully.	A brown gas with pungent smell is evolved. The contents of the test tube turns orange red	Bromide ion (Br^-) may be present
(i) $AgNO_3$ test : Acidify 2 mL of original soln. with dilute HNO_3 add silver nitrate solution.	A light yellow precipitate is obtained which is partially soluble in excess of NH_4OH.	Bromide ion (Br^-) confirmed
(ii) Organic layer test : To 2mL of water solution add 1/2 mL of CCl_4 and add 3 mL of Chlorine water. Shake well.	Reddish brown organic layer is obtained	Bromide ion (Br^-) confirmed

(C) Iodide ion, I $^-$ Take a small amount of given salt in a clean and dry test tube Add 2-3 drops of concentrated H_2SO_4 carefully. Heat the test tube gently.	Violet fumes with pungent smell evolved Black speacks appeared on the side of the test tube	Iodide ion (I^-) may be present
(i) $AgNO_3$ test : To 2 mL of water solution add dilute. HNO_3 to acidify it add 1mL at $AgNO_3$ solution. Shake well.	A yellow precipitate is obtained	
(ii) Add NH_4OH to the above solution ppt add 2-3 mL of ammonium hydroxide solution to it. Shake well	precipitate is insoluble	Iodide ion (I^-) confirmed
Take 2 mL of water solution Add 1/2 mL of CCl_4. Then add 3-4 mL of chlorine water. Shake well.	A violet organic layer is obtained	Iodide ion (I^-) confirmed
(D) Nitrate ion, NO_3^- Take a small amount of given salt in a clean and dry test tube add 2-3 drops of concentrated H_2SO_4 carefully. Heat the test tube gently.	Brown coloured gas coloured gas with pungent smell is evolved. The gas intensified on adding copper turnings, solution turns blue	Nitrate ion (NO_3^-) may be present
Take 2-3 mL of water solution in a test tube and add 2mL of freshly prepared solution of ferrous sulphate, shake well	A brown ring is obtained at the junction where the liquid surface meet each other	Nitrate ion (NO_3^-) confirmed
To this solution add few drops of concentrated H_2SO_4 gradually along the sides of test tube without disturbing the test tube.		

iii) Work sheet No. 3: Qualitative analysis of Sulphate ion (SO_4^{2-}) and Phosphate ion (PO_4^{3-})

Experiment	Observation	Inference
A) Sulphate ion, SO_4^{2-} i) Acidity the Na_2CO_3 extract or the salt solution in water with dil. HCl and then add $BaCl_2$ solution.	A white ppt insoluble in conc. HCl is obtained	SO_4^{2-} ion is confirmed
B) Prosphate ion, (PO_4^{3-}) Acidify the Na2CO3 extract with dil. NHO3 and then add solid ammonium molybdate warm the test tube gently.	A canary yellow precipitate or colour is obtained	Phosphate ion (PO_4^{3-}) confirmed

14.6.2 Basic Radicals

i) Work sheet No. 1 : Ammonium ion (NH_4^+)

Experiment	Observation	Inference
Take a pinch of salt in a test tube. Add 2-3 mL. of NaOH solution. Heat the test tube gently and then strongly (if necessary) smell the gas evolved.	A colourless gas with pungent smell is given out.	Ammonium ion (NH_4^+) may be present
Pass the gas evolved in the above test through Nessler's reagent for atleast 1 minute. Shake well.	A reddish brown precipitate is obtained.	Ammonium ion (NH_4^+) confirmed

ii) Work sheet No. 2: Group-1 : Lead (II) ion (Pb^{2+})

Experiment	Observation	Inference
Solubility in water		
Take a pinch of salt in clean test tube. Add 2-3 mL of water and then few drops of dilute HCl. Shake well.	A white precipitate is obtained.	Lead (II) ion (Pb2+) may be present
Carryout following tests with white precipitate obtained above.		
Wash the precipitate and dissolve a small amount of precipitate in 5-6 mL of hot water. Divide the soln. into two parts.	A clear solution is obtained	
(a) Potassium iodide test : To one part of hot soln. add 1mL Potassium iodide solution. Shake well.	A yellow precipitate is obtained (PbI_2)	Lead (II) ion (Pb^{2+}) confirmed
(b) Potassium Chromate test : To the second part of hot solution add 1mL of K_2CrO_4 solution and Shake well.	A yellow precipitate is obtained ($PbCrO_4$)	Lead (II) ion (Pb^{2+}) confirmed

ii) Work sheet No. 3 : Group-II : Copper (II) ion (Cu^{2+})

Experiment	Observation	Inference
Take 2mL of original solution in a test tube, add a few drops of dilute HCl. Pass H_2S gas through this solution.	black precipitate	(Cu^{2+}) may be present
Heat a small amount of precipitate in 1-2mL of dilute HNO3 and divide it into two parts.	A blue solution is obtained	(Cu^{2+}) may be present

i) To one part add NH4OH solution drop till it is in excess.	A blue precipitate is obtained which dissolved to produce deep blue solution.	(Cu^{2+}) confirmed
ii) Acidify another portion with 2 mL acetic acid and then add 1mL of potassium ferrocynide	A chocolate brown precipitated is obtained	(Cu^{2+}) confirmed

iv) Work sheet No. 4 : Group-III : Ferric Iron or Iron (III) ion (Fe^{3+})

Experiment	Observation	Inference
Take 2mL of original solution in a clean test tube. Add excess of NH_4Cl solid, shake well to dissolve it. Heat if necessary. To cold solution add NH_4OH drop by drop till it is in excess.	A reddish brown precipitate obtained	Fe^{3+} ion may be present
Iron (III) ion, (Fe^{3+}) if a reddish brown precipitate is obtained, it indicates the presence of Fe^{3+} ion. Dissolve the precipitate in 2-3 mL of dilute HCl. and divide it into two parts.	A yellow solution is obtained	
i) To one parts of solution add 1 mL potassium ferrocyanide. Shake well.	A prussian blue colour is obtained	Fe^{3+} confirmed
ii) To the second part of solution add 1 mL of potassium sulphocyanide	Blood red colour is obtained	Fe^{3+} confirmed

v) Work sheet No. 5 : Group-IV-Zinc (II) ion (Zn^{2+}), Manganese (II) ion (Mn^{2+}) and Nickel (II) ion (Ni^{2+})

Experiment	Observation	Inference
Take a 2-3 mL of original solution in a tube, add excess of NH_4OH to make the solution alkaline. Warm the contents of the test tube. Pass H_2S gas through it for 1/2 minute		
(A) Zinc (II) ion, Zn^{2+} :		
Formation of white or dirty white ppt indicates the presence of Zn^{2+} ions.	White precipitate is obtained	Zn^{2+} may be present
Dissolve the washed white precipitate in 1 mL of dilute HCl. Dilute it with 2-3 mL of water. Divide the solution into two parts.	A clear soln. is obtained	

To one part of the solution add dilute NaOH solution drop by drop till it is excess.	White precipitate is obtained which dissolves in excess to produce colourless soln.	Zinc (II) ion (Zn^{2+}) confirmed
To another part of solution add 1mL of potassium ferrocyanide. Shake well.	A grey white precipitate is obtained	Zinc (II) ion (Zn^{2+}) confirmed
(B) Manganese (II) ion (Mn^{2+})		
Formation of buff coloured precipitate indicates the presence of Mn^{2+} ions.	Buff precipitate obtained	Manganese (II) ion (Mn^{2+}) may be present
Dissolve the buff coloured precipitate into 1mL of dilute HCl. Dilute it with 2-3 mL of water.	A clear solution is obtained	
To this solution add dilute NaOH solution drop by drop till it is in excess.	A white precipitate is obtained which turns brown due to aerial oxidation.	(Mn^{2+}) ion confirmed
(C) Nickel (II) ion (Ni^{2+})		
Formation of black precipitate indicates the presence of Ni^{2+} ion.	Black precipitate is obtained	Ni^{2+} ion may be present
Take a small amount of washed precipitate in a china dish. Add 1-2 mL of concentrated HNO_3 to it. Boil it. Evaporate the contents of china dish to dryness.	A yellowish green residue is obtained	
Cool down the china dish Add 2-3 mL of water. Shake well. Transfer this solution to a clean test tube.	A light green solution is obtained	
Add excess of NH4OH solution and then 1mL of dimethyl glyoxime. Shake well.	A cherry red precipitate is obtained	Ni^{2+} ion is confirmed

vi) Work sheet No. 6 : Group- V : Calcium (II) ion. (Ca^{2+})

Experiment	Observation	Inference
Take 2mL of original solution in a clean test tube. Add a pinch of solid NH_4Cl and excess of NH_4OH. Solution and then 2mL of freshly prepared NH_4CO_3 solution, shake well.	A white precipitate is obtained	Calcium (II) ion (Ca^{2+}) may be present

Formation of white precipitate indicates the presence of Ca^{2+} ion.		
Take a small amount of white precipitate and add 1 mL of acetic acid.	A clear soln. is obtained	
To the above solution add 2mL of ammonium oxalate solution. Shake well.	A white precipitate slowly appears	Calcium (II) ion (Ca^{2+}) confirmed

An illustration of identification of cation and anion in a given sample of salt.

AIM : To find out the presence of an anion and a cation in the given salt.

Experiment	Observation	Inference
1. Physical Examination		
(i) Colour of salt	White	May contain salt of NH_4^+, Pb^{2+}, Zn^{2+}, Ca^{2+}
(ii) Heat small amount of salt in a clean dry test tube.	salt sublimates	May contain NH_4^+ ions
(a) Anion analysis		
2. A pinch of salt was taken in a clean test tube 2mL of dilute H_2SO_4 was added to it and heated first gently and then strongly.	No observable changes took place	CO_3^{2-}, S^{2-} and NO_2^- absent
3.A pinch of salt was taken in a clean and dry test tube. Add few drops of conc. H_2SO_4 to it, heat gently, few copper turnings were added and contents were heated again.	A brown gas with pungent smell is given out and solution turns brown	Nitrate ion (NO_3^-) may be present
4. Confirmation test of NO_2^- ion : 2mL original soln, were taken in test tube. To this 2mL of freshly prepared $FeSO_4$ (aq) was added. Shake well, add a few drops of concentrated H2SO4 along the side of test tube without disturbing the test tube.		
5. Take a pinch of salt in a clean test tube, add 2mL of concentrated NaOH solution to it. Warmed gently and then strongly.		

(b) Cation Analysis		
6. A pinch of salt was taken in a test tube. 2-3 mL water was added to it-followed by 3-4 drops of dil. HCl. Shake well.		
7. To the above solution pass H_2S gas		
8. 2mL of original solution was taken in a test tube. Solid NH_4Cl was added in excess, followed by addition of excess NH_4OH solution warm gently.		
9. Passed H_2S gas through group III solution		
10. 2 mL of original soln. was taken in a test tube. A pinch of NH_4Cl solid was added.		
Excess of NH_4OH solution was added followed by addition of 2mL frshly prepared $(NH_4)_2CO_3$ solution. shake well.		
Confirmation test of Cation		
11. Filtered and washed precipitate was dissolved in 2 mL of acetic acid.	ppt is soluble	Ca^{2+} may be present
12. 2mL of $(NH_4)_2CO_3$ soln. was added to above obtained solution.	White ppt is obtained	Ca^{2+} is confirmed

Result : The given salt contains

Anion - NO_3^-

Cation - Ca^{2+}

Precautions

1. Use acids carefully.
2. Avoid using large quantities of acids and salt.
3. Keep the materials ready for testing the gas evolved.
4. Performed the test on the gas evolved immediately. If for any reason the testing is delayed, then add a little amount of salt and test the gas immediately.
5. Addition of group reagents should be followed in correct order.
6. Analysis of anion should be carried out before cation analysis.
7. Pass H_2S gas through hot solution.
8. Always use a pinch of NH_4Cl in group V.

Observations

The students should record the observations and inference during the analysis of given salt (s).

14.7 CONCLUSION

The given salt contains

i) Acid radical ....

ii) Basic radical ....

The given salt is ....

14.8 CHECK YOUR UNDERSTANDING

1. What is meant by qualitative analysis ?

....

2. What is meant by the terms positive test and negative test ?

....

3. What happens when carbon dioxide is passed through $Ba(OH)_2$ solution instead of calcium hydroxide solution ?

....

4. What happens when an acid is over boiled ?

....

5. Why is it necessary to keep the test tube dry during chromyl chloride test ?

....

6. Why Bromine (Br_2) Iodine (I_2) (g) is more soluble in organic solvent such as CCl4 than in water ?

....

7. How will you distinguish between Br^- and NO_3 in qualitative analysis ?

....

8. Why can HNO_3 not be used to prepare original solution of salt ?

....

9. What is a group reagent ?

....

10. What chemicals are used in kipps apparatus to make hydrogen sulphide gas ?

....

11. What is the group V reagent ?

....

12. Why is it necessary to add concentrated H_2SO_4 along the sides of the test tube during ring test of NO_3 ion ?

....

13. Can group V precipitate dissolve in dilute HCl instead of acetic acid ?

....

14.9 NOTE FOR THE TEACHER

1. Teacher should explain important terms used during experiment.

2. teacher should demonstrate all the techniques explained in the experiment.

3. In qualitative analysis, students use concentrated acids, alkali and other laboratory reagents. The teacher should be cautious and ensure that all students follow proper precautions.

4. Freshly prepared soln. of **(a)** $FeSO_4$ **(b)** Sodium nitroprusside **(c)** $(NH_4)_2CO_3$ should be used.

5. Following combination of anions and cations should not be given

    i) Halide ions and Pb^{2+}

    ii) SO_4^2 ion and Pb^{2+}, Ca^{2+}

    iii) PO_4^3 and group III onwards

    iv) NO3 and group II

    v) S2 and group II and group IV cations

6. The teacher may ensure that the students carry out the analysis of given salts systematically.

7. Follow all the precautions given in this experiments and general precautions of a chemistry laboratory.

8. Get sufficient practice of analysis of salts. The students may use their notebooks to records the practicals.

14.10 CHECK YOUR ANSWERS

Ans. 1. Qualitative analysis is the scheme of the detection and identification of the constituent ions of an unknown salt.

Ans. 2. A positive test is the one which gives the observations, needed for that radical. A negative test does not give the required observations for a test.

Ans. 3. $Ba(OH)_2 + CO_2 \rightarrow BaCO_3 + H_2O$

Ans. 4. $2H_2SO_4 \rightarrow 2H_2O + 2SO_2 + O_2$

Ans. 5. If dry test tube is not taken. Concentrated H_2SO_4 gets diluted and chromyl chloride test cannot be performed properly.

Ans. 6. Being covalent in nature Br_2 or I_2 are more soluble in non-polar solvent such as CCl_4

Ans. 7. Bromide ions, (Br^-) gives reddish brown vapour of Br_2 and solution turns red in colours on treatment with concentrated H_2SO_4 NO_3^- ion gives brown fumes of NO_2 which intensified on heating with copper turning.

Ans. 8. HNO_3 is an oxidising agent. It oxidises H_2S to S (sulphur).

Ans. 9. The cations in their respective group are precipitated by adding certain chemicals under definite condition of acidic or alkanity. These chemicals are called group reagents.

Ans. 10. FeS and dilute H_2SO_4.

Ans. 11. Group V reagent $(NH_4)_2CO_3$solution in the presence of NH_4Cl and NH_4OH

Ans. 12. If the concentrated H_2SO_4 is added directly, then solution turn reddish brown.

Ans. 13. Ca^{2+} ion is precipitated as calcium oxalate, (CaC_2O_4) on adding ammonium oxlate $(NH_4)_2C_2O_4$ This precipitate is highly soluble in dilute HCl. Hence calcium (II) ion will not be precipitate out in presence of dilute HCl.

EXPERIMENT-15

To identify the Functional Groups present in the given organic compounds and perform:

i) Test for unsaturation

ii) Test for carboxylic, phenolic, aldehydic and ketonic groups.

15.1 OBJECTIVES

After performing this experiment, you should be able to:

- detect the presence of unsaturation in a given organic compound by simple tests;
- differentiate between saturated and unsaturated compounds;
- detect the presence of the following functional groups in the given organic compounds: carboxylic, phenolic, aldehydic and ketonic groups;
- differentiate between the pairs of compounds containing (a) carboxylic and phenolic groups and (b) aldehydic and ketonic groups;
- explain the acidic nature of carboxylic and phenolic groups;
- write the reactions involved in the various tests

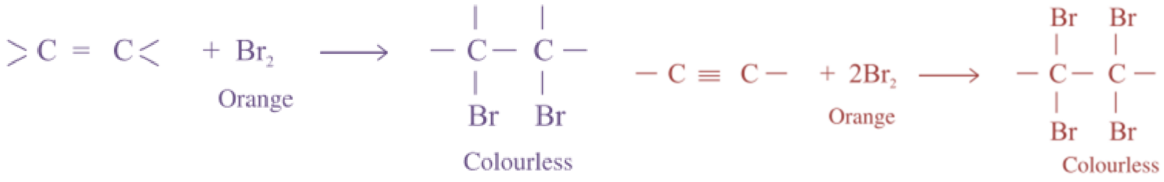
15.2 WHAT YOU SHOULD

In an organic compound, unsaturation is due to the presence of double (>C = C<) or triple (– C ≡ C –) bonds in the molecule. The presence of unsaturation is tested by the following two methods.

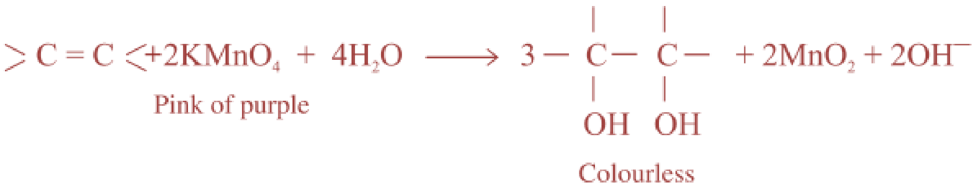
i) Bromine water test

ii) Baeyer's tst (using 1% alkaline potassium permanganate solution)

i) Bromine water test: The organic compound is treated with bromine water drop wise. The decolourisation of bromine water shows the presence of unsaturation in the organic compound.



ii) Baeyer's test: The organic compound is treated with an aqueous solution of alkaline potassium permanganate (Baeyer's reagent). The disappearance of the pink colour of potassium permanganate shows the presence of unsaturation.



$$>C=C<+2KMnO_4+4H_2O \longrightarrow 3-\underset{OH}{\overset{|}{\underset{|}{C}}}-\underset{OH}{\overset{|}{\underset{|}{C}}}-+2MnO_2+2OH^-$$

Pink of purple

Colourless

Precautions

1. Do not inhale the bromine water or bromine in carbon tetrachloride.
2. If the compound is soluble in water, use bromine water for testing. If the compound is insoluble in water, then dissolved 5 mL of the compound (liquid) or a pinch of the solid in 1 mL of carbon tetrachloride and test it with bromine in carbon tetrachloride solution.
3. Add the reagents drop wise.

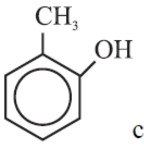
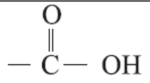
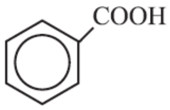
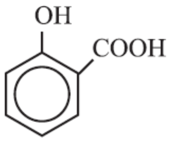
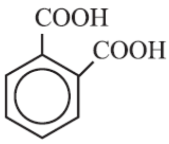
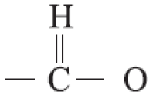
15.2.1 Functional Group

A functional group is that part of the compound which contains an atom or a group of atoms and shows specific reactions. It is the reactive part of a given compound whereas the hydrocarbon part is the non-reactive part. For example, in $CH_3–CH_2–CH_2–COOH$

$CH_3–CH_2–CH–$ is the non-reactive hydrocarbon chain, while- COOH is the functional group called carboxylic acid group.

All the reactions of CH_3 CH_2 CH_2 COOH are due to the presence of – COOH group.

In the following section, we shall discuss the chemistry of four functional groups, phenol, carboxylic (-COOH), aldehyde (-CHO) and ketone = O. The examples of compounds containing these groups are given in the following table.

Functional Group	**Class of the Compound**	**Specific Example**
C_6H_5-OH Phenolic	Phenols	C_6H_5-OH  cresol CH_3COOH
 $-\overset{O}{\overset{\|}{C}}-OH$ Ethanoic acid Carboxylic acid group	Carboxylic	CH_3COOH  Benzoic Acd  Salicylic Acid  Phthalic Acid
 $-\overset{H}{\overset{\|}{C}}-O$ Aldehydic	Aldehydes	CH_3CHO Ethanal C_6H_5CHO Benzaldehyde

$>C=O$ Ketonic	Ketones	CH_3COCH_3 Propanone  $COCH_3$

A) Test for Carboxylic Group

The carboxylic group can be tested by the tests as given below.

i) Litmus test: Put a drop of an aqueous solution of the compound on blue litmus paper. if the blue litmus turns red, acidic nature is indicated.

ii) Sodium hydrogen carbonate test: Add a saturated solution of sodium hydrogen carbonate to an aqueous solution of the compound. If brisk effervescence appear due to evolution of CO_2 gas, the carboxylic group is confirmed.

$$CH_3COOH + NaHCO_3 \longrightarrow CH_3COONa + CO_2 \uparrow + H_2O$$

iii) Esterification test: The given organic compound is mixed with absolute ethanol in equal amounts and a few drops of conc. sulphuric acid are added. The mixture is warmed on a water bath. If a fruity smell (pleasant sweet smell) is obtained, the presence of caboxylic group is confirmed.

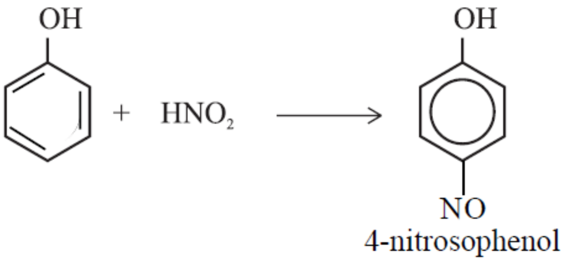
$$CH_3COOH + C_2H_5OH \xrightarrow[\text{Conc.}H_2SO_4]{\text{few drops of}} \underset{\text{warm ester (fruity smell)}}{CH_3COOC_2H_5 + H_2O}$$

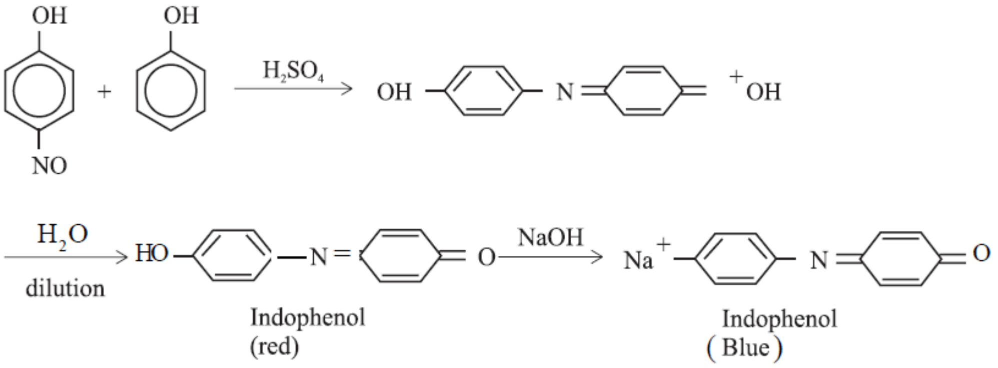
B) Tests for phenolic group

i) Ferric chloride Test: Dissolve a pinch of organic compound in water or alcohol. A few drops of this solution are mixed with neutral ferric chloride solution. If a red, blue or violet colouration appears, then the presence of phenolic group is confirmed.

For example, thus, red colouration is obtained with phenol while, with *o, m* and *p*-cresil, it is violet. With Resorcinol, gives blue colouration whereas catechol gives green colouration.

ii) Liberamann's Test: The organic compound is mixed with solid sodium nitrite (2-3 crystals) and gently warmed for a few seconds. The mixture is cooled and conc sulphuric acid is added. The solution appears red in colour on dilution with water and the colour changes to blue / green on treatment with sodium hydroxide. The above observation confirms the presence of phenolic group in the given compound.





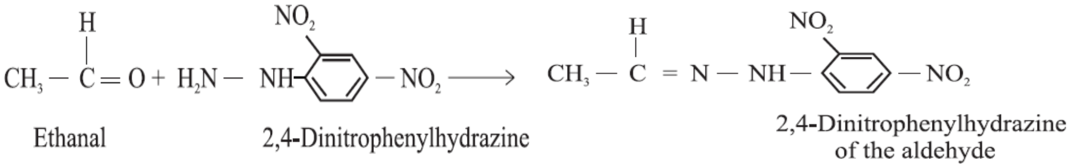
C) Tests for Aldehydes and Ketones

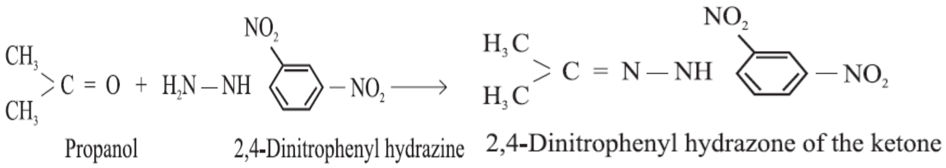
Both aldehydes and ketones contain the carbonyl group ($>C = O$). The carbonyl group can be confirmed by:

i) 2, 4- dinitrophenyl hydrazine (2,4-DNP test)

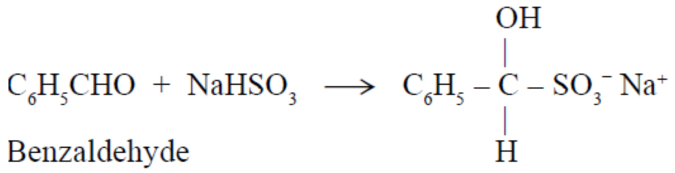
ii) Sodium bisulphite

i) 2, 4 - DNP Test: The organic compound is treated with 2,4-DNP reagent and warmed over a water bath. Formation of yellow or orange crystals indicates the presence of a carbonyl group (aldehydic or ketonic).





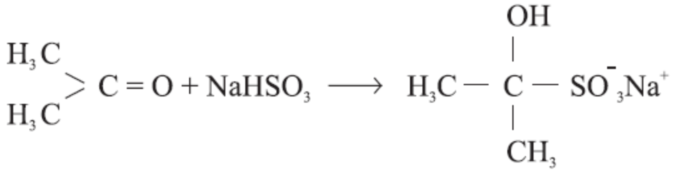
ii) Sodium bisulphite test: The organic compound is treated with a saturated solution of sodium bisulphite. The mixture is shaken well and left for 15 minutes. The formation of a crystalline precipitate indicates the presence of a carbonyl group.



$$C_6H_5CHO + NaHSO_3 \longrightarrow C_6H_5-\overset{\overset{\displaystyle OH}{|}}{\underset{\underset{\displaystyle H}{|}}{C}}-SO_3^-\,Na^+$$

Benzaldehyde

Addition product



$$\begin{matrix} H_3C \\ H_3C \end{matrix}\!\!> C = O + NaHSO_3 \longrightarrow H_3C-\overset{\overset{\displaystyle OH}{|}}{\underset{\underset{\displaystyle CH_3}{|}}{C}}-SO_3^-Na^+$$

Addition product

A) Tests for Aldehydes

i) Fehling's Test : The organic compound is treated with Fehling's solution and warmed over a water bath. Appearance of a red / orange precipitate confirms the presence of aldehyde group.

$$CH_3CHO + 2Cu^{2+} + 5OH^- \longrightarrow CH_3COO + Cu_2O\downarrow + 3H_2O$$

Acetaldehyde Fehlings Solution Red precipitate

The aldehyde reduces the Cu^{2+} (cupric ions) to cuprous state (Cu_2O cuprous oxide)

3. Tollen's Reagent test: To the organic compound, an equal amount of Tollen's reagent is added and the mixture is warmed on a water bath. Appearance of shinning silver mirror on the inner walls of the test tube confirms the presence of aldehydic group.

$$CH_3CHO + 2[Ag(NH_3)_2]^+ + 3OH^- \longrightarrow CH_3CO\bar{O} + 2Ag + 4NH_3 + 2H_2O$$

Tollen's Reagent Silver mirror

The Tollen's reagent (ammonical silver nitrate solution) contains silver ions. These are reduced by aldehydes to metallic silver.

B) Test for keton

Ketones do not give Fehling's and Tollen's tests. Ketones give the following two tests which are not given by aldehydes:

1. m-Dinitrobenzene test: To a mixture of finely powered m-dinitrobenzene and an equal amount of amount of organic compound, add dilute sodium hydroxide solution. The appearance of red colour indicates the ketonic group.

2. Sodium nitroprusside test: To the given organic compound, add sodium nitroprusside solution and a little sodium hydroxide solution. Appearance of red-violet colour confirms the presence of ketonic group.

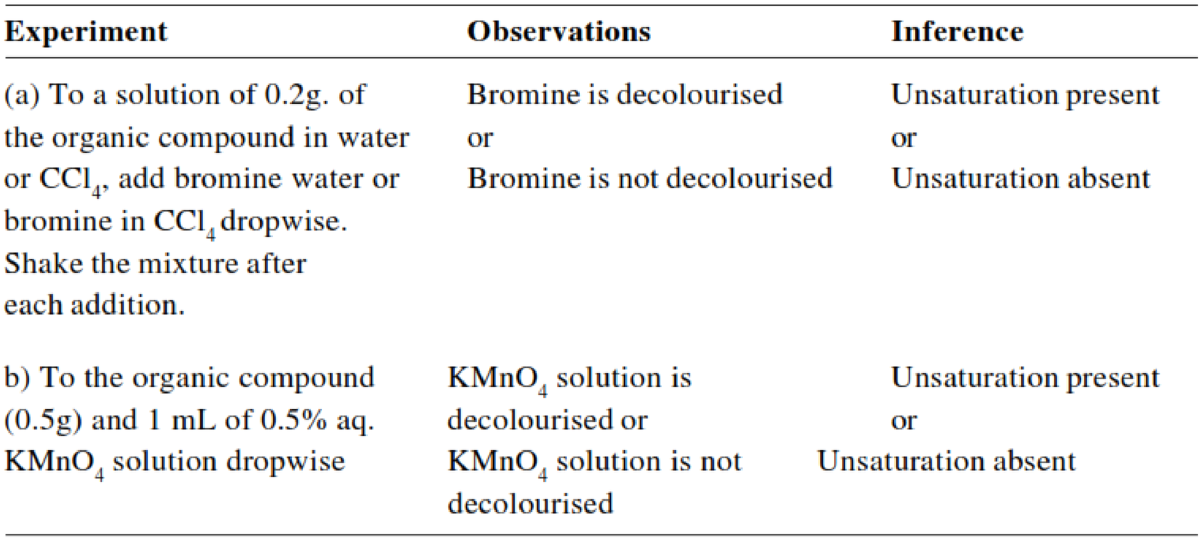
15.3 MATERIALS REQUIRED

(1) Apparatus	(2) Chemicals
Test tubes, Test tube stand, Test tube holder, Water bath, Tripod stand.	Samples of organic compounds. Bromine water. Alkaline potassium permanganate solution. Neutral ferric chloride solution, Sodium nitrite (solid). Litmus paper, Sodium hydrogen carbonate. Alcohol, Conc. sulphuric acid, 2, 4Dinitrophenyl hydrazine soltion, sodium bisulphate solution, m-Dinitrobenzene solid, Sodium nitroprusside, Sodium hydroxide (10% solution), Fehling's reagent, Schiffs reagent and Tollen's reagent.

15.4 HOW TO PERFORM THE EXPERIMENT

The tests should be performed systematically as described in the following table. All functional groups should be tested. The experiments performed should be recorded as shown below:

Table 1 : Tests for Unasaturation



Experiment	Observations	Inference
(a) To a solution of 0.2g. of the organic compound in water or CCl_4, add bromine water or bromine in CCl_4 dropwise. Shake the mixture after each addition.	Bromine is decolourised or Bromine is not decolourised	Unsaturation present or Unsaturation absent
b) To the organic compound (0.5g) and 1 mL of 0.5% aq. $KMnO_4$ solution dropwise	$KMnO_4$ solution is decolourised or $KMnO_4$ solution is not decolourised	Unsaturation present or Unsaturation absent

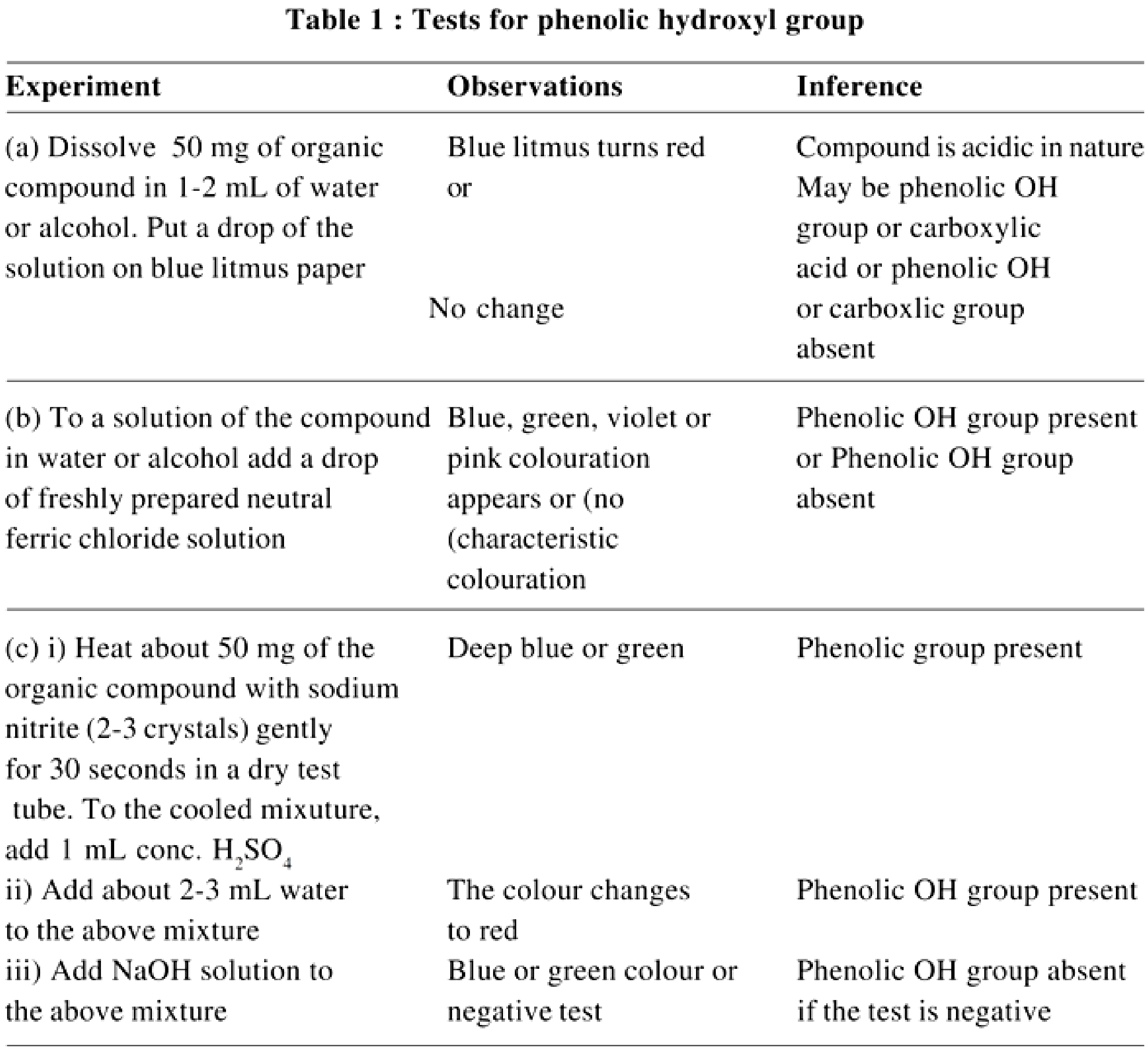


Table 1 : Tests for phenolic hydroxyl group

Experiment	Observations	Inference
(a) Dissolve 50 mg of organic compound in 1-2 mL of water or alcohol. Put a drop of the solution on blue litmus paper	Blue litmus turns red or No change	Compound is acidic in nature May be phenolic OH group or carboxylic acid or phenolic OH or carboxlic group absent
(b) To a solution of the compound in water or alcohol add a drop of freshly prepared neutral ferric chloride solution	Blue, green, violet or pink colouration appears or (no (characteristic colouration	Phenolic OH group present or Phenolic OH group absent
(c) i) Heat about 50 mg of the organic compound with sodium nitrite (2-3 crystals) gently for 30 seconds in a dry test tube. To the cooled mixuture, add 1 mL conc. H_2SO_4	Deep blue or green	Phenolic group present
ii) Add about 2-3 mL water to the above mixture	The colour changes to red	Phenolic OH group present
iii) Add NaOH solution to the above mixture	Blue or green colour or negative test	Phenolic OH group absent if the test is negative

Precautions

1. Phenol is highly corrosive. It causes blisters on the skin. Always handle it carefully.
2. Neutral ferric chloride should be freshly prepared, see Appendix for details.
3. Phenol turns blue litmus red. Carboxylic acids also give the test but phenol does not decompose sodium hydrogen carbonate.
4. Instead of phenol, naphthols may be given for test for phenolic group.

Table 3: Test for carboxylic acids

Experiment	Observations	Inference
(a) The aqueous solution of the organic compound is put on a blue litmus paper	Blue litmus turns red or No change	- COOH group may be present or - COOH group absent
(b) To the 0.2g organic compound add saturated solution of sodium hydrogen carbonate	Effervescences seen or No effervescences	- COOH group present or - COOH group absent

(c) Ester formation to 0.2 g of the organic compound add an equal amount of ethyl alcohol and a drop of conc. sulphuric acid. Warm the mixture on a water bath.	Fruity odour develops or No fruity odour	- COOH group present - COOH group absent

Table 4 : Tests for aldehydes and ketones

Experiment	Observations	Inference
(a) To the solution of the organic compound (1mL) and 5mL 2, 4-dinitrophenyl hydrazine reagent. Heat the solution in a water bath,	yellow of orange precipitate	carbonyl group present (-CHO or > CO)
(b) The organic compound (0.5g) is shaken with 2 mL of saturated sodium bisulphate solution.	white precipitate	-CHO, >C=O present
(c) To a solution of equal volumes offering's A and Fehling's B, add 0.2 g of the organic compound. Heat the mixture for 5 min. in a water bath.	Red precipitate of CuO_2	Aldehyde (-CHO) group present
(d) To 1 mL of AgNO3 solution add a drop of dil. NaOH solution. To this, add ammonium hydroxide dropwise fill the precipitate dissolves. To the resulting solution add 0.1g of the organic compound Heat for 5 min. in a water bath.	Silver mirror is formed on the inner side of the test tube.	Aldehyde (-CHO) group present
(e) Add 1 mL of sodium nitroprusside solution to 0.5 g of the organic compound. Shake the mixture and add NaOH solution dropwise	red colour	Ketone (>C = O) group present
(f) Mix 0.1g of the organic compound with 0.1 g m- dinitrobenzene. Add I mL dil. NaOH sol. add shake.	Violet colour fades slowly	Ketone (>C = O) group present

Precautions

1. Tollen's reagent should be freshly prepared.
2. Benzaldehyde reacts very slowly and a grey precipitate is generally obtained. Shake the tube vigorously to break the oily globules from time to time.
3. Wash the test tube thoroughly with warm sodium hydroxide solution followed by washing it with a large amount of water

Specific Example (This is not a part of the experiment)

Example: 1

Aim of the Experiment: To identify by chemical tests which one of the samples, A and B is benzaldehyde and which one is acetone.

Apparatus: Test-tubes

Chemicals: Silver nitrate solution, Ammonium hydroxide, Iodine solution and Sodium hydroxide.

Observations: Perform the following chemical tests with compounds A and B.

Experiment	Observation	
	Compound A	**Compound B**
1. Colour and physical state	Colourless liquid (turns yellow on standing)	Colourless liquid
2. Odour	Odour of bitter almonds	Pleasant smell
3. Solubility in i) Water ii) Sodium hydroxide solution iii) 5% $NaHCO_3$ Solution iv) Conc. HCl	 Immiscible Immiscible Immiscible Immiscible	 Miscible Immiscible Immiscible Immiscible
4. Chemical Test **i) Tollen's Test :** **a)** Place 1 mL of silver nitrate solution in a clean test-tube and add 1 drop of dil. NaOH solution. A brown ppt. is formed. Add ammonia solution drop wise until the brown ppt. just re-dissolves. To this add about 0.5 mL of liquid A and warm the test-tube in a beaker of boiling water for 5 minutes. **b)** Repeat this test with liquid B	A shining Silver mirror forms	
ii) Sodium nitroprusside test: Add 1 mL of soidum nitroprussdie to 0.5 mL of the liquid A and B in separate test tubes. Add NaOH Solution to both the test tubes and shake them.	No characteristic colour	No silver mirror Red colour formation
iii) (a) Mix 0.5 mL of the liquid B with 0.5g. of soild m-dinitrobenezene add NaOH and shake the contents	Violet colouration appears that fades slowly	
(b) Repeat the same with liquid A	No characteristic colour	

Example: 2

Aim of the Experiment: To identify by chemical tests which one of the samples, C and D is phenol and which one is benzoic acid

Apparatus: Test-tubes

Chemicals: Sodium nitrite, Sodium hydrogen carbonate

Observations:

Experiment	Observation	
	Compound A	Compound B
1. Colour and physical state	Colourless or pink crystalline solid	White solid
2. Odour	Carbolic	Odourless
3. Solubility in i) Water ii) Sodium hydroxide solution iii) 5% $NaHCO_3$ Solution iv) Conc. HCl	Sparingly soluble an emulsion insoluble insoluble insoluble	Sparingly soluble Soluble Soluble Soluble Soluble
4. Blue litmus test	Turns red	Turns red
5. Chemical Test **i) Liebermann's test** **a)** Perform Liebermann's test with portion of compound C. For procedure, see experiment in table **b)** Repeat this test with a portion of compound D	Deep blue of green colour	
ii) Sodium hydrogen carbonate test **(a)** Place 3 mL of 10% $NaHCO_3$, sol. in a clean test-tube and add a portion or the compound C. Warm it.	No effervescences	No coloration
(b) Repeat this test with a portion of compound D.		Strong effervescences

Conclusion

The given organic compound was found to contain ________________ functional group.

15.5 CHECK YOUR UNDERSTANDING

1. Name the functional group (s) presence in the following compounds.
   a. Phenol, benzoic acid, sallicylic acid and acetone.

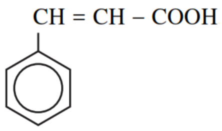
   ....
2. How will you distinguish the following pairs of compounds?
   a) Acetic and acetaldehyde. b) Acetic acid and phenol.

   ....
3. Write the reaction for bromine water test on C_6H_5 - CH = CH2

   ....

4. What tests would be given by the following compound?

....



....

5. What is Bayer's reagent?

....

6. Why should Tallen's reagent be freshly prepared ?

....

7. Which compound is more acidic? (Phenol or benzoic acid)

....

8. How will you differentiate between phenol and cresols?

....

9. How will you infer the presence of carbonyl group in a compound?

....

10. Give an example of aromatic compound which is known methyl ketone.

....

15.6 NOTE FOR THE TEACHER

1. The students may be given enough number of samples of the same functional group for proper practice.
2. Phenol is highly corrosive. Test for phenolic groups, the students may be given-Naphthol, β - Naphthol, or o-, m- p- cresols etc. Avoid giving phenol directly to the students without proper precautions of handling.
3. Students should be told to wear lab coats and use gloves when corrosive substances are being handled.
4. The students and the assistant should be instructed to keep inflammable liquids away from the burner.
5. A sample worksheet is given in this section. The students may be asked to record the experiment in this format.

15.7 CHECK YOUR ANSWERS

Ans. 1: The functional groups present in the following compounds are:

Phenol - Phenolic group		Benzoioc acid - Carboxylic acid group

Salicylic acid - Phenolic group and carboxylic group		Acetone - Ketonic group.

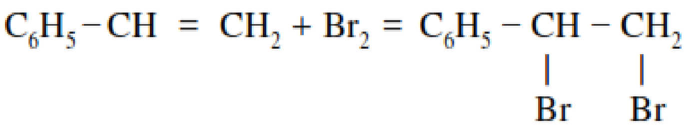
Ans. 2: Acetone and Acetaldehyde can be distinguished by any of the following tests.

i) Fehling's test   ii) Tollen'stest   iii) Sodium nitroprusside test   iv) *m*-dinitrobenzene test

The changes observed are tabulated below:

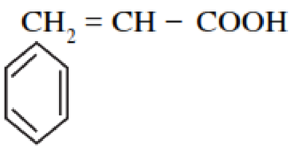
S.No.	Test performed	Acetaldehyde	Acetone
a.	Fehling's test	red colouration appears	x
b.	Tollen's test	Silver mirror is formed	x
c.	sodium nitroprusside test	x	Violest colouration
d.	m-dinitrobenzene test	x	red colouration

Ans. 3:



$$C_6H_5-CH = CH_2 + Br_2 = C_6H_5-\underset{\displaystyle Br}{\underset{|}{CH}}-\underset{\displaystyle Br}{\underset{|}{CH_2}}$$

Ans. 4:



will give the following test :

i) Positive test for unsaturation due to the double bond,

ii) Positive test for carboxylic groups.

The compound will therefore, give positive test with bromine water and positive test with sodium hydrogen carbonate.

Ans. 5: Baeyer's reagent is an alkaline solution of potassium permanganate, (Please see appendix for details)

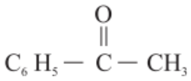
Ans. 6: Tollen's reagent is a solution of ammonical silver nitrate. It should be freshly prepared because it is unstable as it can easily decompose to Ag_2O and finally to silver, on standing.

Ans. 7: Benzoic acid is a stronger acid than phenol. Benzoic acid will give brisk effervescences with sodium hydrogen carbonate solution due to the liberation of carbon dioxide gas. Phenol does not give positive test with sodium hydrogen carbonate as it is a weak acid.

Ans. 8: Phenol and cresois can be differentiated by neutral ferric chloride test. Phenol will give red colouration. o-m- and p-cresol will give voilet colouration.

Ans. 9: Carbonyl compounds give 2,4-dinitrophenylhydrazine test and soiumbisulphite test. Crystalline precipitates are formed in both cases.

Ans. 10: Acetophenone is an example of methyl ketone. It has the formula.



$$C_6H_5 - \overset{\overset{\displaystyle O}{\|}}{C} - CH_3$$

15.8 CHECK YOUR ANSWERS

Ans. 1: Multiple scratches, if made on the glass tube will cause a rough and irregular cut.

Ans. 2: The outer diameter of the borer should be equal to the inner diameter of the tube to be inserted into the bore.

Ans. 3: To avoid cracking of the cork and to get a smoother bore, it is moistened with water and pressed in a cork presser.

Ans. 4: The edges should be heated gently by rotating on a flame.

Appendix

Preparation of Common Laboratory Reagents

1. **Bromine water:** Shake 5 ml of liquid bromine with 100 ml of distilled water in a conical flask. Decant off the clean solution into a bottle and stopper it.
2. **Bromine in CCl_4:** Shake 4 ml of liquid bromine with 100 ml of carbon tetrachloride and stopper the bottle.
3. **Alkaline potassium permanganate solution:** (Baeyer's Reagent) Dissolve 1g of solid potassium permanganate in 100ml of distilled water, then add 10g of anhydrous sodium carbonate. Shake the mixture to dissolve it and stopper the bottle.
4. **Neutral ferric chloride:** Place 2ml ferric chloride solution in a clean test tube. Add ammonium hydroxide until a brown precipitate just appears. Now add the original ferric chloride solution until the precipitate just disappears. (Prepare fresh for a day).
5. **2,4-dinitrophenyl hydrazine (2,4-DNP):** Dissolve 2g of 2,4-DNP in 100ml methanol to which 4ml of conc. Sulphuric acid is added. Filter if necessary.
6. **Sodium bisulphate solution:** Dissolve 30g of sodium bisulphite in 100ml of distilled water.
7. **Schiff's reagent:** Dissolve 0.1g of fuchsine (p-rosaniline hydro chloride) in 100ml water. Pass sulpher dioxide gas until its red color is discharged. Filter and use the filtrate.
8. **(a) Fehling's solution A:** Dissolve 35g of crystalline copper sulphate in 500ml water. Add 3ml of concentrated sulphuric acid to it.

   (b) Fehling's solution B: Dissolve 173g of Rochelle salt (sodium potassium tartarate) and 60g of sodium hydroxide in 500ml of water.

   Note: (i) Fehling's solution A and Fehling's solution B are available in the market also, (ii) Mix equal volumes of A and B solutions just before performing the test.
9. **Tollen's Regent:** Place 2ml of silver nitrate solution in a clean test tube Add two drops of sodium hydroxide solution (10% solution). A brown precipitate is formed. Now add dilute ammonium hydroxide solution drop wise until the brown precipitate of silver oxide just re-dissolves. *(freshly prepare the solution at the time of test)*
10. **Conc. sulphuric acid** 18M as supplied may be used.
11. **Conc. hydrochloric acid** 12M as supplied may be used.
12. **Conc. nitric acid** 16M as supplied may be used.
13. **Acetic acid** (glacial) 17M as supplied may be used.
14. **Dil. Hydrochloric acid 12M:** Pour 166.6 mL of conc. hydrochloric acid slowly and with constant stirring in 500ml of distilled water. Cool and make up the volume to 1 liter.
15. **Dil. Hydrochloric acid 4M:** Add 333.3ml of conc. HCl in the distilled water and make up the volume to 1 liter.

16. **Dil. nitric acid 4M:** Add 250ml of cone.HNO_3 in the distilled water make up the volume to liter.
17. **Dil. Acetic acid 2M:** Add 117.6 mL of glacial acetic acid in distilled water and make up the volume to 1 liter.
18. **Ammonia solution** 15M as supplied may be used.
19. **Dil. Ammonia solution 4M:** Pour 266.6ml of the conc. ammonia solution in distilled water and make up the volume to 1 liter.
20. **Ammonium chloride 4M:** Dissolve 214g of the salt in one liter of water.
21. **Ammonium carbonates 2M:** Dissolve 193.6g of ammonium carbonate in 1 liter distilled water.
22. **Ammonium acetate 3M:** Dissolve 231g of the salt in a litre of distilled water.
23. **Ammonium sulphate IM:** Dissolve 132g of the salt in 1 litre of distilled water.
24. **Ammonium oxalate 0.5M:** Dissolve 71g of the salt in 1 litre of distilled water.
25. **Ammonium molybdate 0.5M:** Dissolve 88g of the salt in a mixture of 120ml of conc. ammonia solution and 80ml of distilled water. Add 240g of ammonium nitrate and dilute it to 1 liter water.
26. **Barium chloride 0.2M:** Dissolve 48.8g of the salt in distilled water and make up the volume to litre.
27. **Chlorine water:** Take one liter distilled water and saturate it with chlorine gas and keep the solution in a dark coloured bottle.
28. **Calcium chloride 0.2M:** Dissolve 29.402g of the salt in distilled water and make up the volume to 1 litre.
29. **Copper sulphate 0.5M:** Dissolve 124.75g of the salt in distilled water. Add a few drops of dilute acetic acid and make up the volume to 1 litre.
30. **Cobalt nitrate 0.15M:** Dissolve 43.65g of the salt in distilled water and make up the volume to 1 litre.
31. **Dimethyl glyoxime 1%:** Dissolve 10g of the solid in ethyl alcohol and make up the volume to 1 litre with distilled water.
32. **Ferric chloride 0.5M:** Dissolve 135g of the salt in distilled water. Add 20ml of conc. Hydrochloric acid and make up the volume to 1 litre.
33. **Ferrous sulphate 0.5M:** Dissolve 138.5g of the salt in distilled water containing 10ml of conc. sulpuric acid make up the volume to 1 litre.
34. **Iodine solution 0.05M:** Dissolve 12.7g of iodine crystals in distilled water containing 20g of potassium iodide and then dilute it to 1 litre.
35. **Lime water:** Dissolve some amount of calcium oxide in distilled water, filter the solution after sometime and keep it in a reagent bottle.
36. **Litmus solution (Blue):** Dissolve 10g of litmus in distilled water and make up the volume to 1 litre.
37. **Litmus solution (Red):** To the blue litmus solution add about 10 drops of dilute hydrochloric acid.
38. **Lead acetate 0.1M:** Dissolve 37.9g of the salt in 500mL of distilled water containing a little acetic acid and make up the volume to 1 litre.
39. **Methyl orange:** Dissolve 1g. of the solid in distilled water and make up the volume to 1 litre.

40. **Mercuric chloride 0.1M:** Dissolve 27.2g of the salt in the water and make up the volume to 1 litre.
41. **Nesslers reagent:** Dissolve 23g. of mercuric iodide and 16g of potassium iodide in distilled water and make up the volume to 100mL. Add 150mL of 4M NaOH solution. Allow it to stand for 24 hours and decant the solution. Solution should be stored in a dark coloured bottle.
42. **Potassium chromate 0.2M:** Dissolve 38.8g of the salt in distilled water and make up the volume to 1litre.
43. **Potassium dichromate 0.1M:** Dissolve 29.4g of the salt in distilled water and make up the volume to 1 litre.
44. **Potassium iodide 0.2 M:** Dissolve 33.2g. of the salt in water and make up the volume to 1litre.
45. **Potassium thiocyanate 0.2M:** Dissolve 19.4g of the salt in distilled water an make up the volume to 1 litre.
46. **Potassium permanganate 0.02M:** Dissolve 6.32g of the salt in distilled water and make up the volume to 1litre. Heat the solution and filter it.
47. **Potassium ferrocyanide 0.1M:** Dissolve 42.2g of the salt in distilled water and make up the volume to 1 litre.
48. **Potassium ferricyanide 0.2 M:** Dissolve 65.8g of the salt in distilled water and make up the volume to 1 litre.
49. **Pheuolphthalein 0.1%:** Dissolve 0.25g of the solid in 125mL of ethyl alcohol and then add 125mL of distilled water.
50. **Sodium hydroxide 4M:** Dissolve 160g of the sodium hydroxide pellets in distilled water and make up the volume to 1 litre.
51. **Silver nitrate 0.1 M:** Dissolve 17g of the salt in distilled water and make up the volume to 1 litre and store it in a brown coloured bottle.
52. **Starch:** Prepare paste of about 1g. of starch in cold water and pour it in 100 mL of boiling water with constant stirring. Allow it to cool.
53. **Disodium hydrogen phosphate 0.1M:** Dissolve 35.8g of the salt in distilled water and make up the volume to 1 litre.
54. **Sodium nitroprusside 0.03M:** Dissolve 1g. of the solid in 100mL of distilled water.
55. **Sodium cobaltinitrate 0.16M:** Dissolve 64.64g of the solid in distilled water and make the volume to 1 litre.
56. **Stannous chloride 0.5M:** Dissolve 113g of salt in 200mL of concentrated hydrochloric acid by heating (if necessary). Add several pieces of metallic tin and make up the volume to 1 litre.
57. **Yellow ammonium sulphide:** Take about 200mL of concentrated ammonia solution in a bottle and saturate it with H_2S gas. Add 10g of flowers of sulphur and shake well until sulphur is completely dissolved. Dilute the solution to one litre with distilled water.

CURRICULUM FOR PRACTICAL WORK IN CHEMISTRY

Objectives of the present course in practical work are as follows:

1. To develop and inculcate laboratory skills and techniques
2. To enable the student to understand the basic chemical concepts.
3. To develop basic competence of analysing and synthesing chemical compound and mixtures.

To meet these objectives three different types of laboratory experiments are provided in the present practical course.

1. Experiment for developing laboratory skills/techniques
2. Concept based experiments
3. Traditional experiments (for analysing and synthesing chemicals)

List of Practicals

I. Identify the anion and cation present in the given salt and report correct salt and record the systematic procedure adopted in analyzing the salt.

a) $(NH_4)_2CO_3$
b) NH_4Cl
c) $(NH_4)_2SO_4$
d) NH_4Br
e) $Al_2(SO_4)_3$
f) $Al(NO_3)_3$
g) $CaCl_2$
h) $Pb(NO_3)_2$
i) $CuSO_4$
j) $MnCl_2$
k) $FeSO_4$
l) $ZnSO_4$

II. Estimate the amount of solute present in the given Solution.

SECTION - A

1. Estimate the amount of NaOH present in 250 ml of a given solution 0.2 m Hydrochloric acid solution is supplied.
2. Estimate the amount of HCl present in 500 ml of given solution 0.2 m Sodium hydroxide solution is supplied.

SECTION - B

3. Estimate the amount of Na_2CO_3 present in 100 ml of given solution 0.2 m HCl solution is supplied.
4. Estimate the amount of HCl present in 250 ml of given solution 0.1 m Na2CO3 is supplied.

SECTION-C

5. Estimate the amount of H_2SO_4 present in 1000 ml of a given solution 0.2 m NaOH is supplied.
6. Estimate the amount of NaOH present in 500 ml given solution 0.1 m H_2SO_4 solution is supplied.

SECTION - D

7. Estimate the amount of Na_2CO_3 present in 250 ml of given solution 0.1 m H_2SO_4 is supplied.
8. Estimate the amount of H_2SO_4 in a 100 ml of given solution 0.1 m Na_2CO_3 solution is supplied.

SECTION - E

9. Estimate the amount of Ferrous Ammonium Sulphate present in 250 ml of given solution 0.02m $KMnO_4$ solution is supplied.
10. Estimate the amount of $KMnO_4$ present in a 100 ml of given solution 0.1 m Mohr Salt solution is supplied.

SECTION - F

11. Estimate the amount of Oxalic acid present in a 500 ml of given solution 0.02 m $KMnO_4$ solution is supplied.
12. Estimate the amount of $KMnO_4$ present in 250 ml of given solution 0.1 m Oxalic acid is supplied.

III. a. Detection elements in the given organic compound

i. Aniline ($C_6H_5NH_2$)
ii. Chloroform ($CHCl_3$)
iii. Ether (C_2H_5-O-C_2H_5)

b. Identify the functional group in the given organic compound

i. Acetone
ii. Acetaldehyde
iii. Phenol
iv. Acetic acid

OR

III. Perform any one experiment from the following

a.

i. Prepare the 2M standard solution of H_2SO_4 from on stock solution of H_2SO_4
ii. Prepare 1 M solution of HCl
iii. Prepare 5 M solution of HNO_3

b. Determine the p^H of the following substance using universal indications of p^H papers.

i. Given salt solution
ii. Acid solutions with different dilution
iii. Alkali solutions with different dilutions
iv. Fruit juice

c. Determine the melting point (*m.p.*) of a given solid substance with the help of capillary tube.

d. Prepare 250 ml, 0.1 M Ferrous Ammonium sulphate solution by weighting (using analytical balance)

e. Study the solubility of a given solid substances in water at different temperature and plot the graph (tempt *vs* solubility)

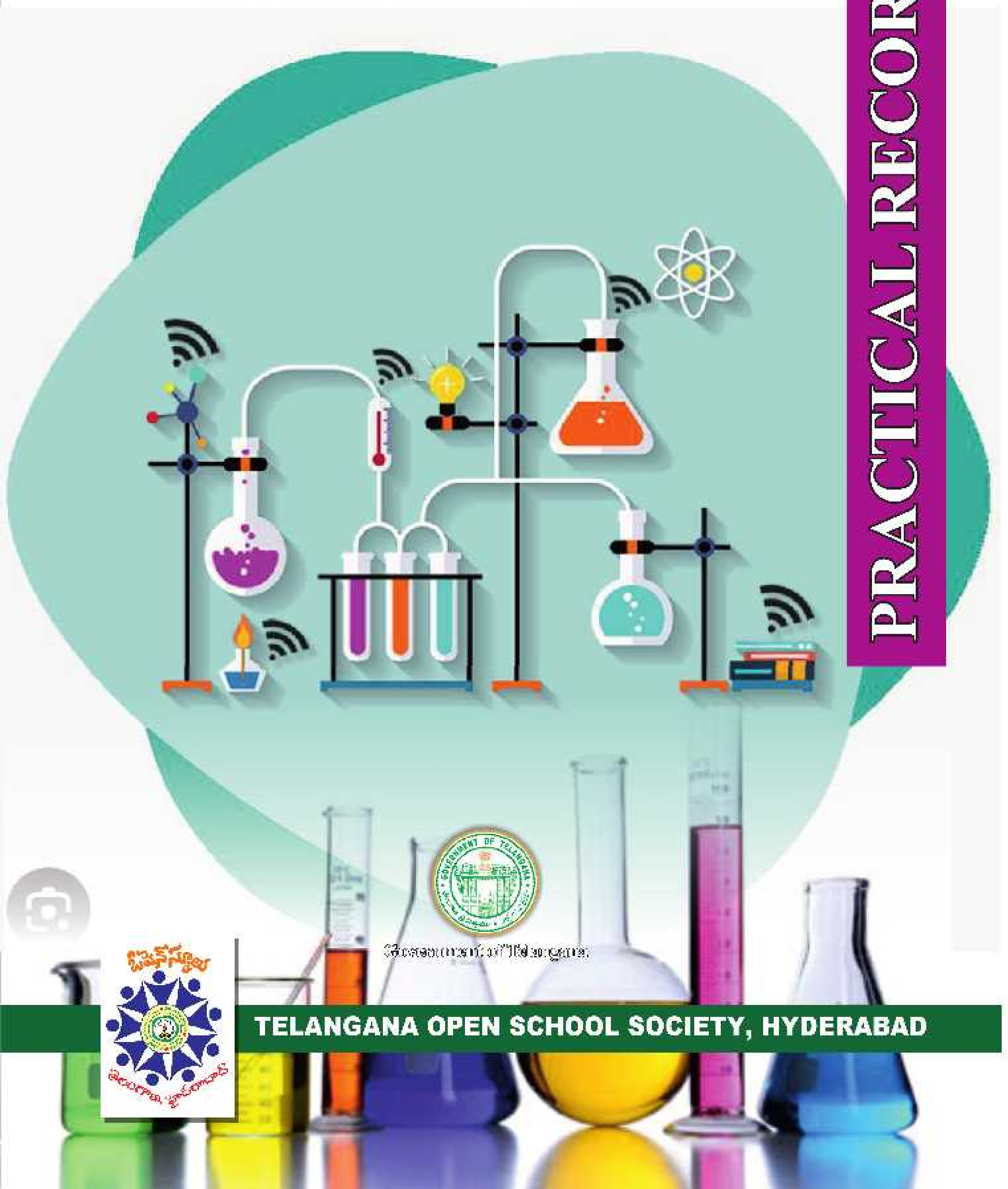
IV. Viva - Voice

V. Record

INTERMEDIATE

CHEMISTRY

PRACTICAL RECORD



Government of Telangana

TELANGANA OPEN SCHOOL SOCIETY, HYDERABAD