

Laboratory Manual **Chemistry**

Class XI

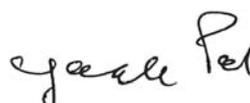


राष्ट्रीय शैक्षिक अनुसंधान और प्रशिक्षण परिषद्
NATIONAL COUNCIL OF EDUCATIONAL RESEARCH AND TRAINING

FOREWORD

The National Council of Educational Research and Training (NCERT) is the apex body concerning all aspects of refinement of School Education. It has recently developed textual material in Chemistry for Higher Secondary stage which is based on the National Curriculum Framework (NCF)–2005. NCF recommends that children's experience in school education must be linked to the life outside school so that learning experience is joyful and fills the gap between the experience at home and in community. It recommends to diffuse the sharp boundaries between different subjects and discourages rote learning. The recent development of syllabi and textual material is an attempt to implement this basic idea. The present laboratory manual will be complementary to the textbook of Chemistry for Class XI. It is in continuation to the NCERT's efforts to improve upon comprehension of concepts and practical skills among students. The purpose of this manual is not only to convey the approach and philosophy of the practical course to students and teachers but to provide them appropriate guidance for carrying out experiments in the laboratory. The manual is supposed to encourage children to reflect on their own learning and to pursue further activities and questions. Of course the success of this effort also depends on the initiatives to be taken by the principals and teachers to encourage children to carry out experiments in the laboratory and develop their thinking and nurture creativity.

The methods adopted for performing the practicals and their evaluation will determine how effective this practical book will prove to make the children's life at school a happy experience, rather than a source of stress and boredom. The practical book attempts to provide space to opportunities for contemplation and wondering, discussion in small groups, and activities requiring hands-on experience. It is hoped that the material provided in this manual will help students in carrying out laboratory work effectively and will encourage teachers to introduce some open-ended experiments at the school level.



21.5.08

PROFESSOR YASH PAL

Chairperson

National Steering Committee
National Council of Educational
Research and Training

New Delhi
21 May 2008

THE CONSTITUTION OF INDIA

PREAMBLE

WE, THE PEOPLE OF INDIA, having solemnly resolved to constitute India into a **SOVEREIGN SOCIALIST SECULAR DEMOCRATIC REPUBLIC** and to secure to all its citizens :

JUSTICE, social, economic and political;

LIBERTY of thought, expression, belief, faith and worship;

EQUALITY of status and of opportunity; and to promote among them all

FRATERNITY assuring the dignity of the individual and the unity and integrity of the Nation;

IN OUR CONSTITUENT ASSEMBLY this twenty-sixth day of November, 1949, do **HEREBY ADOPT, ENACT AND GIVE TO OURSELVES THIS CONSTITUTION.**

PREFACE

The development of the present laboratory manual is in continuation to the NCERT's efforts to improve upon comprehension of concepts and practical skills among the students. The present laboratory manual will be complementary to the textbook of Chemistry for Class XI.

The expansion of scientific knowledge and consequently the change in the system of education has led to the development of new methods of instructions. Today the stress is laid on the enquiry approach and discussion method instead of lecture method of teaching. Unfortunately, it is believed that study of chemistry means abstract thinking, writing long formulas and complex structures and handling complicated equipments. The reason behind such endeavour is that even well-endowed schools tend to give only the cosmetic importance to the laboratory work. Children's natural spirit of inquiry is often not nurtured.

The new syllabus of practical work in chemistry has been designed to cater to the needs of pupil who are desirous of pursuing science further. The fundamental objective of this course is to develop scientific attitude and desired laboratory skills required at this level. The practical syllabus includes content based experiments, which help in comprehension of the concepts.

The project work is expected to provide thrill in learning chemistry. It is expected to serve the real purpose of practical work, which means inculcating the ability to design an experiment, to make observations methodically and to draw conclusions out of experimental data. The real purpose of practical work should be to enable the students to represent the outcome of experiments logically to conclusion, with genuine appreciation of its limitation.

For each practical work, brief theory, material required, procedure, precautions and the questions for discussion are given in the book. The questions are aimed at testing learner's understanding of the related problems. However, teacher may provide help in case the problem is found to be beyond the capability of the learner. Precautions must be well understood by the learners before proceeding with the experiments and projects.

In order to provide some basic idea about the investigatory projects, a brief description of some investigatory projects is given in the book. However, this list is only suggested and not exhaustive. The students may select projects from subject area of chemistry, interdisciplinary areas or from the environment. While selecting a project, care should be taken to see that the facilities for carrying it out are available.

Appendices related to the chemical data and logarithmic tables are attached at the end of the book. International symbols for hazards and hazard warnings are given at several places in the book. It is expected that this will make the learners more careful about the environment and make them careful while dealing with the chemicals. Some non evaluative learning material has been given in the boxes to provide interesting information related to the practical work.

It is a pleasure to express my thanks to all those who have been associated at various stages of development of this laboratory manual. It is hoped that this practical book will improve teaching learning process in chemistry to a great extent. The learners will be able to understand the subject well and will be able to apply the acquired knowledge in new situations. I acknowledge with thanks the dedicated efforts and valuable contribution of Dr Alka Mehrotra, coordinator of this programme and other team members who contributed and finalised the manuscript. I especially thank Professor Krishna Kumar, *Director*, and Professor G. Ravindra, *Joint Director*, NCERT for their administrative support and keen interest in the development of this laboratory manual. I am also grateful to the participating teachers and subject experts who participated in the review workshop and provided their comments and suggestions which helped in the refinement of this manual and make it learner friendly. We warmly welcome comments and suggestions from our readers for further improvement of this manual.

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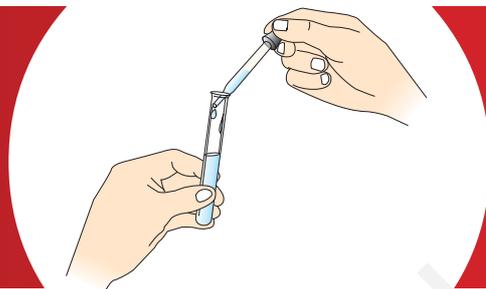
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UNIT-1

INTRODUCTION



LABORATORY work has special importance in the learning of science as scientific principles develop and grow on the basis of laboratory work. Chemistry is an experimental science; the concepts learned in the theory classes are better understood through experimentation. Laboratory work provides an opportunity to observe many of the chemical phenomena under controlled laboratory conditions and work out a problem through the method of inquiry. In other words, it provides you with ample opportunity to become a keen observer and to draw inferences and explain results.

The training in laboratory work helps to develop skills for handling apparatus and equipment and carry out experiments. In this way, the experimental work helps to promote scientific temper and adopt a cooperative attitude. Working in the laboratory provides a platform for trying novel and creative ideas and giving them concrete shape.

Before you become familiar with the scientific procedures and experimental skills and start working in the laboratory, you should be well acquainted with the chemistry laboratory. You should notice the facilities provided in the laboratory and on your working table.

You will notice that your table is provided with a water-tap, gas-tap, Bunsen burner spirit lamp/kerosene lamp, a reagent shelf and a bin for disposing waste material. You will find that some reagents are kept on the shelf fixed on the table while some reagents are kept on the shelf fixed on sidewalls. Reagents kept on the shelf of the table are frequently required while those on the shelf fixed on walls are less commonly used. Besides the facilities on the table, you will see that there are exhaust fans fitted on the upper portion of the wall opposite to the doors and windows and are placed close to the level of the ceiling. This facilitates the exhaustion of harmful fumes and circulation of fresh air in the laboratory. For this purpose, there are also enough number of windows in the laboratory. Keep these open while working. A fume cupboard is provided in the laboratory for performing those experiments in which fumes are produced.

It is strongly advised that you should become fully familiar with your chemistry laboratory, laboratory practices and procedures, and more importantly the precautions to be taken while working in the laboratory.

The environment in the chemistry laboratory is somewhat special in the sense that it can be a place of joy, discovery and learning. It can also become a place of frustration and danger. Frustration – if you come unprepared and neglect recording the important data properly, and danger – if you do not follow the precautions properly in conducting the experiments where potential danger exists.

In order to become proficient in basic principles underlying the laboratory work, you must learn to handle the equipment and familiarise yourself with the safety measures and good laboratory practices.

You should organise yourself before entering into the laboratory for work and be aware of the pre-laboratory preparation and experimental procedures so that your work is not haphazard. You should work individually unless the experiments require teamwork. Use your ingenuity and common sense while working. This attitude is the basic requirement to acquire scientific approach. Prepare reports of the experiments in the laboratory notebook. Do not use loose sheets or scraped papers for this purpose. Think and try to get answers of important questions that give you an understanding of the principles on which the experimental procedure is based.

Scientists learn much by discussion. In the same manner, you too may be benefitted by discussion with your teacher and classmates. Use books in case of any doubt because books are more reliable, complete and better source of information than classmates. Else consult your teacher.

Safety rules are designed to ensure that the work done in the laboratory is safe for you and your fellow students. Follow the safety rules and be aware of where the items like first-aid box, fire extinguisher etc., are kept.

Don't taste anything in the laboratory (Poisonous substances are not always so labelled in the laboratory) and never use laboratory as an eating-place. Never use glass apparatus of the laboratory for eating and drinking purposes. Never work in the laboratory alone. Seek the permission of your teacher for working for extra hours, in case it is needed.

1.1 Do's AND DON'TS IN A CHEMISTRY LABORATORY

The practices outlined below are designated to guide you in developing efficient laboratory techniques and to make your laboratory a pleasant place to work. You should follow the practices listed below:

- Wear safety glasses, lab coat and shoes while working in the laboratory.
- Check and read the label of the reagent bottle carefully before using its content.
- Read procedures and precautions carefully and follow them.
- It is a bad practice to leave the reagent bottles on the working table. Put the stoppers properly on the bottles and keep them on the shelf immediately after use.

- If a reagent bottle on your seat is empty, ask the laboratory attendant to fill it.
- If you require a reagent from the bottle kept on side shelf, take the test tube or the beaker to the shelf. Do not bring the bottle to your seat.
- **Avoid using excessive amounts of reagents** unless you are advised to do so.
- **Never return unused chemicals to the stock bottles.** If you commit a mistake in putting the material back into the correct bottle, experiments of other students will be spoiled.
- **Never mix the chemicals** unless it is required in the experiment. Failure in following this rule may result in serious accidents.
- **Use only properly cleaned droppers, spatulas or pipettes** etc. to take out the reagents from the stock solutions and reagent bottles.
- **Do not keep the stopper of the bottle on the table.** Impurities may stick to it and the content of the bottle may be contaminated. Whenever you require a chemical from the reagent bottle, pick up the bottle with one hand and remove or replace the stopper with the other hand and keep it on a clean glazed tile. To take out dry solid reagents use spatula and **keep it on watch glass, never use filter papers. Do not keep the reagent on your palm or touch it with your fingers.**
- **Never throw** used match sticks, litmus papers, broken glass apparatus, filter papers or any other **insoluble solid material into the sink** or on the floor. Dispose them off in the waste bin provided at your seat. Only waste liquids should be thrown in the sink while keeping the tap water running so that nothing stinks and sticks and the waste liquid is drained completely.
- **Do not waste water or gas.** Close the taps whenever they are not in use. Do not leave the lighted burner under the wire gauze when nothing is being heated. Extinguish it.
- Hot apparatus should not be placed on working table directly because it may spoil the working table. Place it on a glazed tile or a wire gauze.
- Do not heat the apparatus which is made of thick glass, e.g., graduated cylinder, bottles, measuring flasks etc., as these break on heating. Also heating distorts the glass and calibrations on the measuring apparatus may become invalid. Test tubes may break if they are heated above the level of the liquid filled in them. Crucibles may be heated to red-hot.

INTERNATIONAL HAZARD SYMBOLS



Harmful



Inflammable



Corrosive



Toxic



Explosive



Oxidising



Radioactive

Note : Look for these symbols on the labels of bottles of chemicals.

- Do not heat the test tube containing solution with its mouth towards yourself or your neighbour as the spurting of content may harm you or your neighbour (Fig 1.1). Do not heat the test tube continuously in one position. Keep it moving and shaking while heating so that heating is uniform.
- Clean every piece of apparatus as soon as the work is finished and keep these at proper place. A dirty seat and apparatus indicate careless habit and it hinders successful performance of the experiment.

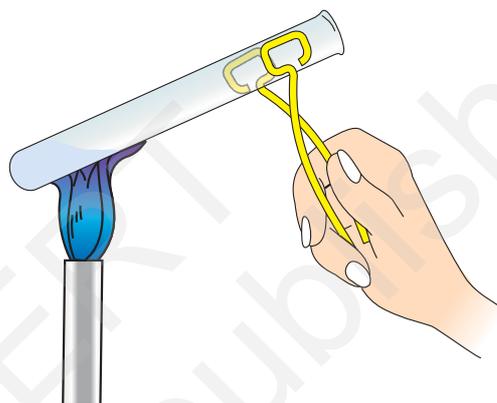


Fig. 1.1 : Correct method of heating solution in a test tube

The indication for cleanliness of glass apparatus is that after rinsing with water and holding it in position, water flows out readily and droplets do not stick to the surface. If water droplets stick to the glass surface it means that apparatus is greasy. In that case it should be washed with 5% NaOH solution or with soap and then thoroughly rinsed with water. If it still remains dirty or some stain remains sticking to it, then warm concentrated nitric acid can be used for cleaning. If some stain is still not cleaned then chromic acid, (also called chromosulphuric acid) can be used for cleaning. To prepare one litre of chromic acid solution, 100 g of potassium dichromate is dissolved in one litre of conc. sulphuric acid. **It is highly corrosive liquid and all care should be taken to avoid its contact with the skin and clothes.**

- Use fume cupboard for performing experiments in which poisonous and irritating fumes are evolved.
- Keep the doors and windows open and the exhaust fan on while working in the laboratory, so that poisonous vapours are quickly sucked out and flow of fresh air is facilitated.

- If you will follow the Do's and Don'ts outlined above, your experience of learning basic scientific techniques will surely be full of joy.

In the following pages you will be introduced to basic laboratory equipments, procedures and techniques required for working in a chemistry laboratory. Let us begin with the introduction to type of analytical methods used in chemistry.

1.2 ANALYTICAL METHODS

Elements and their compounds may be detected by their physical features such as physical state, colour, odour, lustre, melting point, boiling point, sublimation, colour imparted to the flame on heating, hardness, crystalline state or amorphous state, solubility in water and other solvents, etc., but sometimes it is impossible to identify the substance on the basis of physical properties only, therefore, chemical methods such as reaction with alkalies, acids, oxidising agents, reducing agents and other compounds are employed for the identification of substances. A substance is analysed to establish its qualitative and quantitative chemical composition. Therefore, analysis may be either qualitative or quantitative. **Qualitative analysis** is used to detect the elemental composition of the substance; it may involve detection of ions formed, and the type of molecules present in the substance. The methods of qualitative analysis are very diverse. They not only allow us to determine the elements which constitute the substances known on the Earth, but also the composition of celestial bodies which are away from the Earth. **Quantitative analysis** helps to establish the quantity of the constituents of substances. It helps in measurement of energy changes etc.

1.3 BASIC LABORATORY EQUIPMENT AND PROCEDURES

Heating, filtration, decantation, measuring volumes and weighing solids and liquids are some of the basic laboratory procedures, which are required frequently during the experimentations in the chemistry laboratory. Some of the specific equipment required for this purpose are shown in Fig. 1.2 and 1.3. You will learn about the use of these while performing experiments. Guidelines for using some of the common apparatus are as follows :

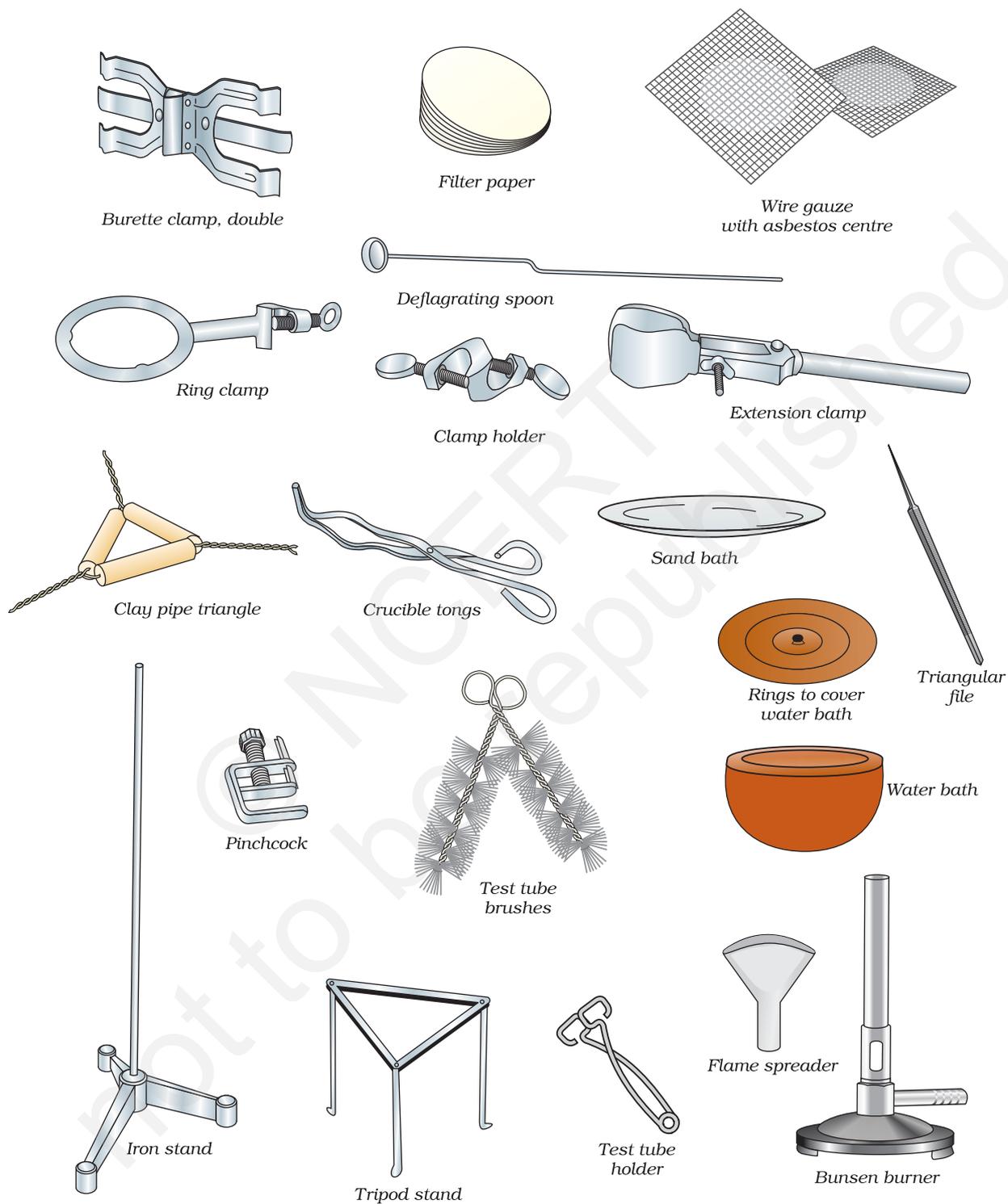


Fig. 1.2 : Common laboratory equipments

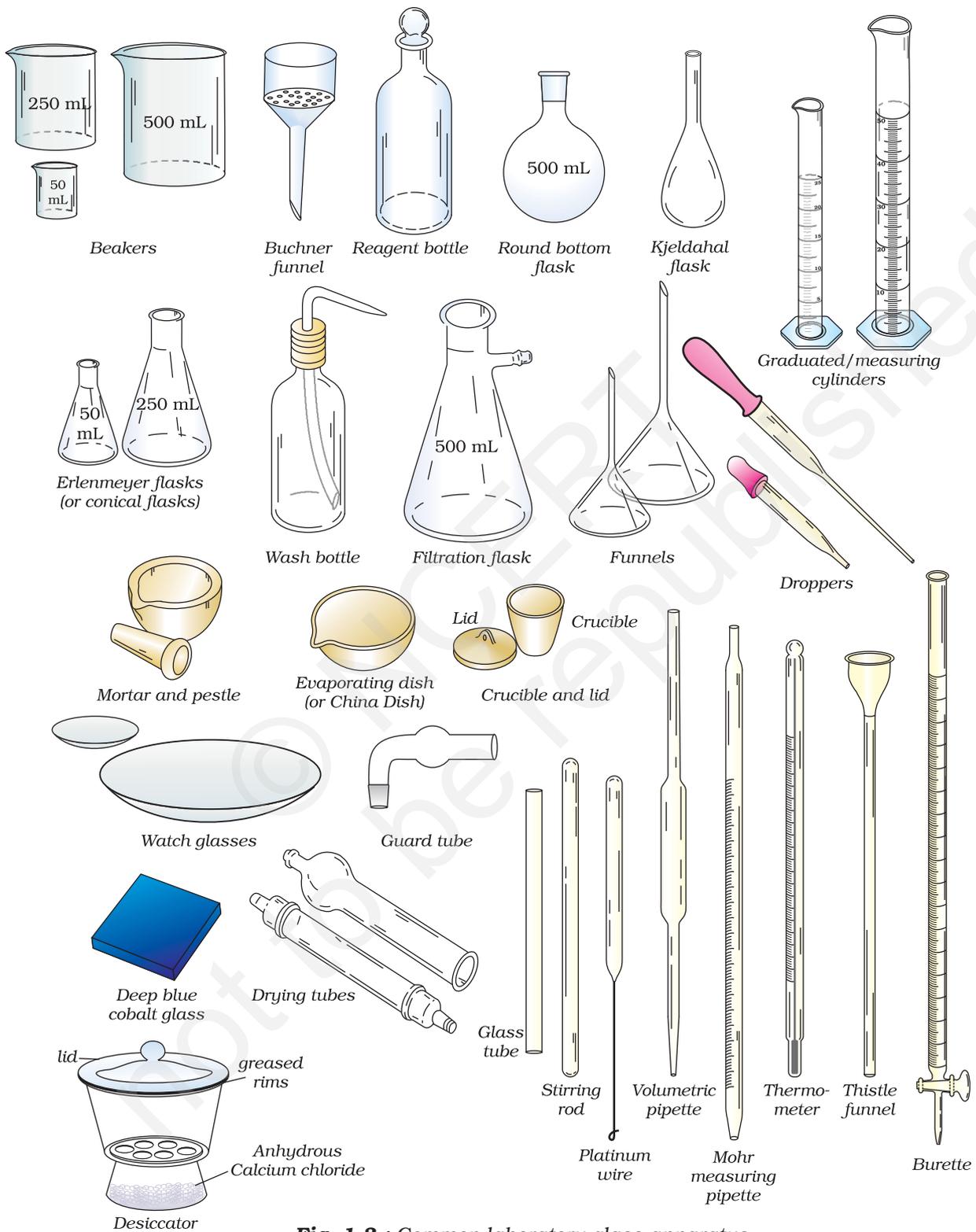


Fig. 1.3 : Common laboratory glass apparatus

Test Tubes

Test tubes of different volumes are available but usually for this level of chemistry practical work, test tubes of 125 mm (length) × 15 mm (diameter), 150 mm (length) × 15 mm (diameter) and 150 mm (length) × 25 mm (diameter) are used. Test tubes are available with or without rim around the mouth. Test tubes of smaller width are used for carrying out reactions, which do not require heating or when heating is required for a short period. **Only one third of the test tube should be filled while carrying out a reaction.** The test tube of bigger diameter is called **boiling tube**. It is used when large volume of solution is required to be heated. Test tube holder is used to hold a test tube while heating a mixture or solution in it. Test tube stand should be used to keep test tubes containing solutions in the upright position (Fig. 1.4).

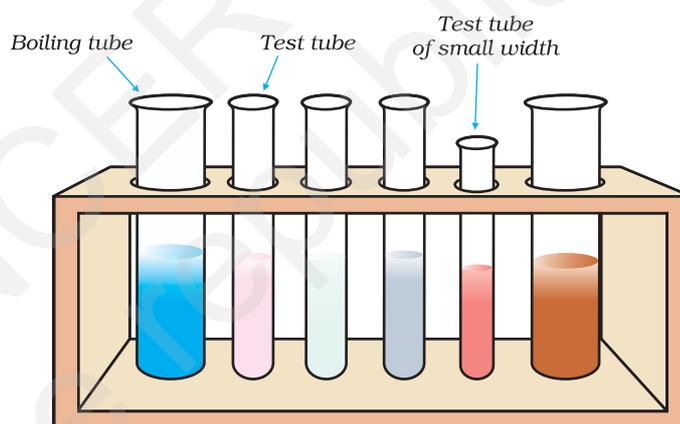


Fig. 1.4 : Stand carrying boiling tubes and test tubes of different sizes

Flasks

Mostly round bottom and conical flasks (also called Erlenmeyer flask) are used in chemistry laboratory. These are available in various capacities ranging from 5 mL-2000 mL. Choice of the size and type depends upon the amount of solution to be handled and the type of reaction to be carried out. Generally, for heating or refluxing a reaction mixture contained in a round bottom flask, direct flame / sand bath / water bath, is used. Conical flasks are employed for carrying out certain reactions at room temperature or lower temperatures. These are specially used for carrying out volumetric titrations.

Beakers

Beakers of various capacities ranging from 5 mL to 2000 mL are available and are employed for the purpose of preparing solutions, for carrying out precipitation reactions and for evaporation of solvents etc.

Separating Funnels

These are used for separating immiscible liquids. Separating funnels of various sizes and shapes are available (Fig. 1.5).

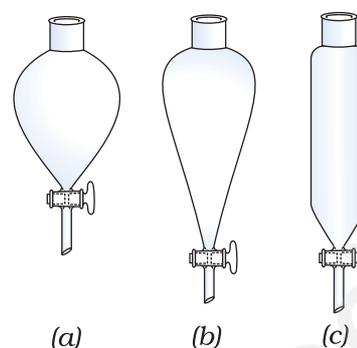


Fig. 1.5 : Separating funnels of various shapes

Condensers

Condensers are used to condense the vapour back to the liquid phase. Generally two types of condensers are used in the laboratory, (a) air condensers and (b) water condensers. Air condenser is shown in Fig. 1.6 (a). Length and diameter of the glass tube of air condensers vary. There is a rapid transfer of heat from the hot vapour to the surrounding air and vapour condenses.

Water condenser has an inner tube surrounded by an outer jacket [Fig. 1.6 (b)] with an inlet and an outlet for circulating water. Inlet is connected to the tap. Heat is transferred from hot vapour to the surrounding water.

For refluxing and distillation of solutions or liquids with high boiling point, air condensers are used. For low boiling liquids water condensers are used.

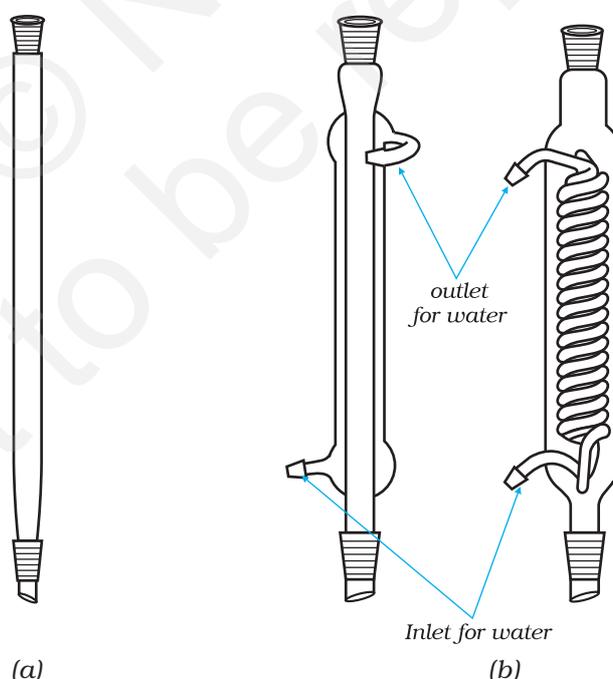


Fig. 1.6 : (a) Air condenser
(b) Water condensers

Ground Glass Joints

Now-a-days ground glass joints are fixed to inlets or outlets of the apparatus described above to minimize the use of corks. Apparatus with ground glass joints of various sizes are available.

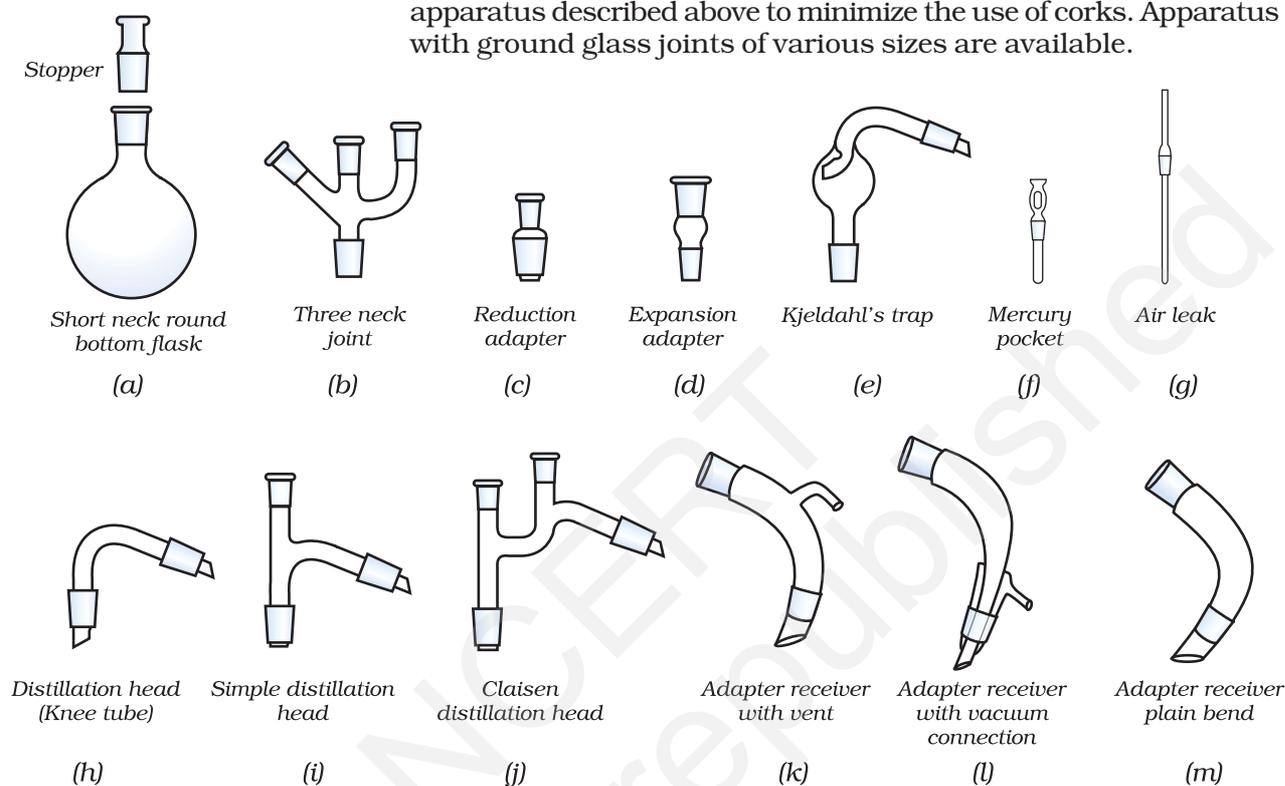


Fig. 1.7 : Apparatus with ground glass joints

1.4 HANDLING REAGENT BOTTLES

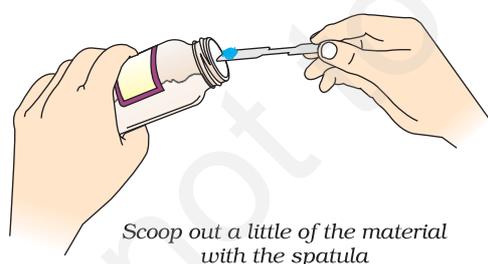


Fig. 1.8 : Method for taking out solid from the reagent bottle

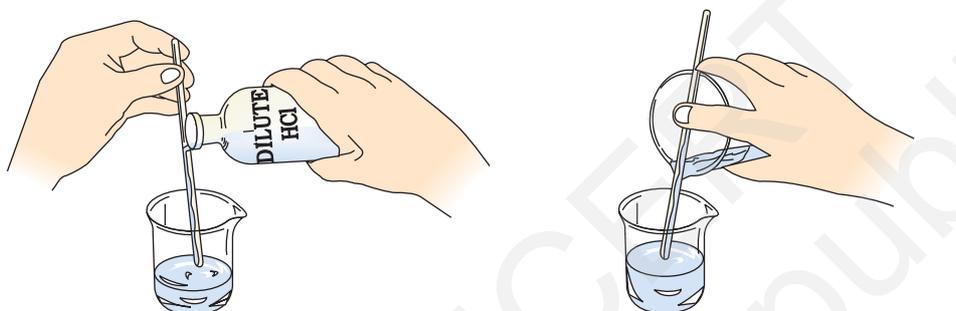
Correct methods for taking out solid and liquid reagents from reagent bottles are shown in Fig. 1.8 and 1.9 respectively. Before taking out any reagent from the bottle one should double-check the name written on label to make sure that correct reagent is being taken out. Liquid reagents are stored either in reagent bottle of small mouth with glass stoppers or dropping bottles. While using reagent bottles with stopper, put the stopper on a clean glazed tile. Never put the stopper on the table because dirt from the table may stick to it and contaminate the reagent. Replace the stopper immediately after taking out the reagent. The proper way of pouring liquid from the bottle is shown in Fig. 1.9. When adding liquids directly from bottles into the beaker, a glass rod is held against the mouth of the bottle so as to permit the flow of liquid along the rod without splashing.



First : Read the label twice

Second : Hold the stopper in and tilt the bottle until the content wets the stopper

Third : Transfer the liquid. The moistened neck and lip of the bottle prevent the first drop from gushing out



Pour the liquid down a glass rod.

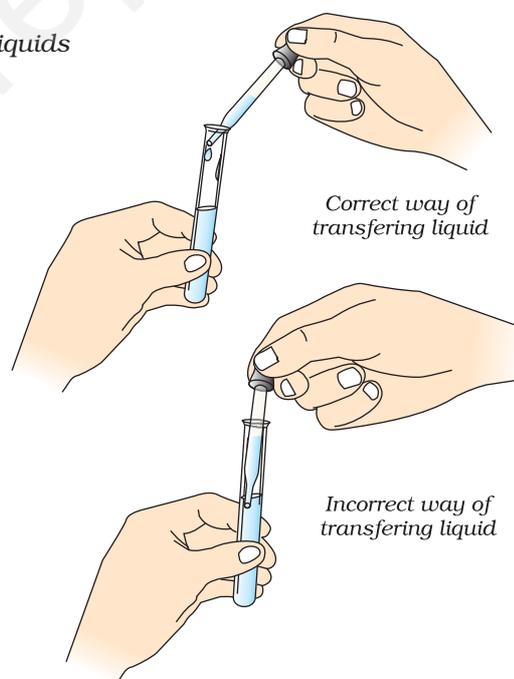
When pouring from a beaker, the stirring rod can be held in this manner.

Fig. 1.9 : Methods of pouring liquids

If dropper is used for transferring the liquid, it should not touch the content of the container while transferring the reagent. Correct way of transferring liquid with a dropper is shown in Fig. 1.10. Droppers of bottles should never be exchanged. Now-a-days, use of dropping bottles is considered more convenient and safe.

1.5 HEATING DEVICES

Heating during the laboratory work can be done with the help of a gas burner, spirit lamp or a kerosene lamp. The gas burner used in the laboratory is usually Bunsen burner (Fig. 1.11). Various parts of Bunsen burner are shown in Fig. 1.12. The description of these parts is as follows :



Correct way of transferring liquid

Incorrect way of transferring liquid

Fig. 1.10 : Transferring liquid through dropper

BUNSEN BURNER

(A) Parts of Bunsen Burner

1. The Base

Heavy metallic base is connected to a side tube called gas tube. Gas from the source enters the burner through the gas tube and passes through a small hole called *Nipple* or *Nozzle* and enters into the burner tube under increased pressure and can be burnt at the upper end of the burner tube.

2. The Burner Tube

It is a long metallic tube having two holes diametrically opposite to each other near the lower end which form the air vent. The tube can be screwed at the base. The gas coming from the nozzle mixes with the air coming through the air vent and burns at its upper end.

3. The Air Regulator

It is a short metallic cylindrical sleeve with two holes diametrically opposite to each other. When it is fitted to the burner tube, it surrounds the air vent of the burner tube. To control the flow of air through the air vent, size of its hole is adjusted by rotating the sleeve.

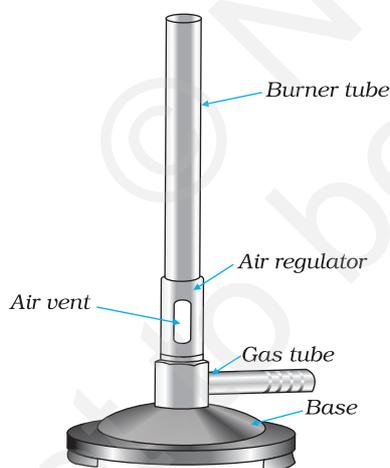


Fig. 1.11 : Bunsen burner

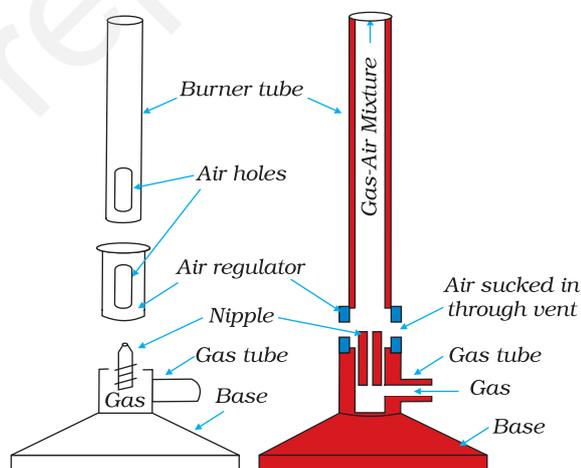


Fig. 1.12 : Parts of Bunsen burner

If the air vent is closed and the gas is ignited, the flame will be large and luminous (smoky and yellow in colour). The light emitted by the flame is due to the radiations given off by the hot carbon particles of partially burnt fuel. The temperature of the flame in this situation is low. If adjustment of sleeve on vent is such that gas mixed with air is fed into the flame, the flame becomes less

luminous and finally turns blue. When the flow of air is correctly adjusted, the temperature of the flame becomes quite high. This is called non-luminous flame. Various zones of flame are shown below in Fig. 1.13.

Three distinctly visible parts of the Bunsen flame are described below:

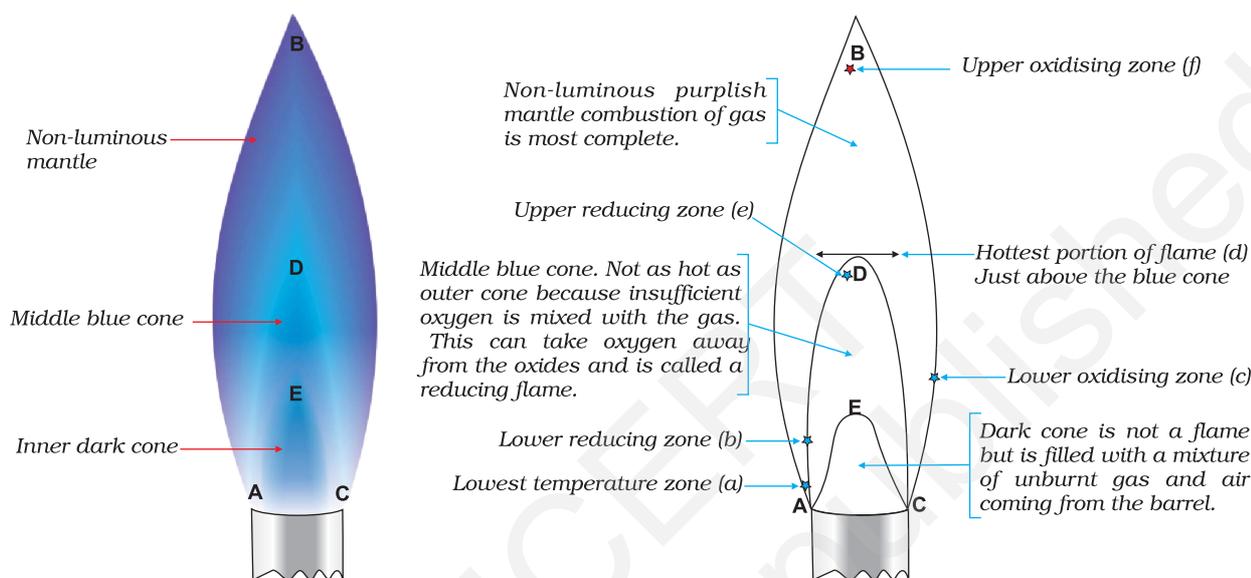


Fig. 1.13 : Zones of flame of Bunsen burner

(B) Principal Parts of Bunsen Flame

1. The Inner Dark Cone, A E C

This is innermost dark cone, which is just above the burner tube. It consists of unburnt gases. This zone is the coldest zone of the flame and no combustion takes place here.

2. The Middle Blue Cone, A D C E A

This is middle part of the flame. This becomes luminous when the air vent is slightly closed. Luminosity of this part is due to the presence of unburnt carbon particles produced by decomposition of some gas. These particles get heated up to incandescence and glow but do not burn. Since the combustion is not complete in this part, the temperature is not very high.

3. The Outer Non-luminous Mantle, A B C D A

This is purplish outer cone. It is the hottest part of the flame. It is in direct contact with the atmosphere and combustion is quite complete in this zone.

Bunsen identified six different regions in these three principal parts of the flame:

(i) The upper oxidising zone (f)

Its location is in the **non-luminous tip of the flame** which is in the air. In comparison to inner portions of the flame large excess of oxygen is present here. The temperature is not as high as in region (c) described below. It may be used for all oxidation processes in which highest temperature of the flame is not required.

(ii) Upper reducing zone (e)

This zone is at the **tip of the inner blue cone** and is rich in incandescent carbon. It is especially useful for reducing oxide incrustations to the metals.

(iii) Hottest portion of flame (d)

It is the fusion zone. It lies at about one-third of the height of the flame and is approximately equidistant from inside and outside of the mantle i.e. the outermost cone of the flame. Fusibility of the substance can be tested in this region. It can also be employed for testing relative volatility of substances or a mixture of substances.

(iv) Lower oxidising zone (c)

It is located on the outer border of the mantle near the lower part of the flame and may be used for the oxidation of substances dissolved in beads of borax or sodium carbonate etc.

(v) Lower reducing zone (b)

It is situated in the inner edge of the outer mantle near to the blue cone and here reducing gases mix with the oxygen of the air. It is a less powerful reducing zone than (e) and may be employed for the reduction of fused borax and similar beads.

(vi) Lowest temperature zone (a)

Zone (a) of the flame has lowest temperature. It is used for testing volatile substances to determine whether they impart colour to the flame.

(C) Striking Back of the Bunsen Burner

Striking back is the phenomenon in which flame travels down the burner tube and begins to burn at the nozzle near the base. This happens when vents are fully open. The flow of much air and less gas makes the flame become irregular and it strikes back.

The tube becomes very hot and it may produce burns on touching. This may melt attached rubber tube also. If it happens, put off the burner and cool it under the tap and light it again by keeping the air vent partially opened.

SPIRIT LAMP

If Bunsen burner is not available in the laboratory then spirit lamp can be used for heating. It is a device in which one end of a wick of cotton thread is dipped in a spirit container and the other end of the wick protrudes out of the nozzle at upper end of the container (Fig. 1.14). Spirit rises up to the upper end of the wick due to the capillary action and can be burnt. The flame is non luminous hence can be used for all heating purposes in the laboratory. To put off the lamp, burning wick is covered with the cover. **Never try to put off the lighted burner by blowing at the flame.**



Fig. 1.14 : The spirit lamp

KEROSENE HEATING LAMP

A kerosene lamp has been developed by National Council of Educational Research and Training (NCERT), which is a versatile and cheaper substitute of spirit lamp. It may be used in laboratories as a source of heat wherever spirit and gas burner are not available. Parts of kerosene lamp are shown in Fig. 1.15.

Working of the Kerosene Lamp

More than half of the container is filled with kerosene. Outer sleeve is removed for lighting the wicks. As the outer sleeve is placed back in position, the flames of four wicks combine to form a big soot-free blue flame.

The lighted heating lamp can be put off only by covering the top of the outer sleeve with a metal or asbestos sheet.

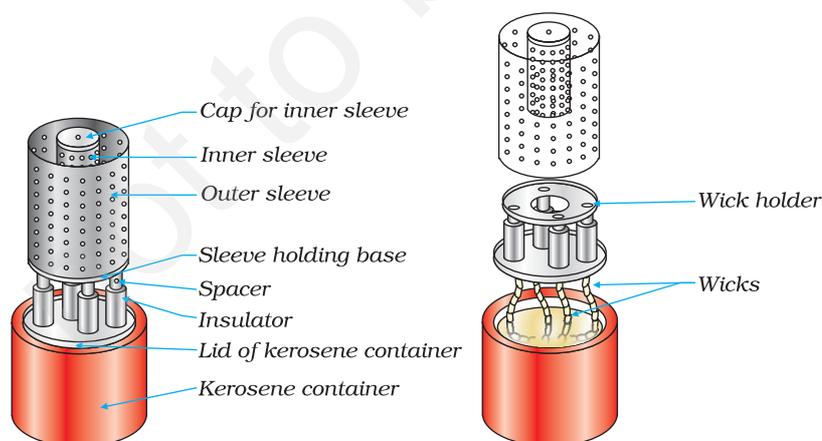
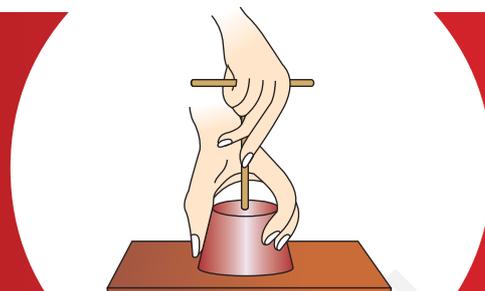


Fig. 1.15 : Parts of Kerosene Heating Lamp

UNIT-2

BASIC LABORATORY TECHNIQUES



THE laboratory apparatus for carrying out reactions, in general, is made up of glass. It is because glass is resistant to the action of most of the chemicals. Generally, two types of glass are used for making apparatus for laboratory work. These are soda-lime glass and borosilicate glass.

Soda-lime glass, which is made by heating soda, limestone and silica, softens readily at about 300-400°C in the burner flame. Therefore, on heating glass tubings made of soda-lime glass easily softens and can be bent. Coefficient of expansion of soda glass is very high, therefore on sudden heating and cooling, it may break. To avoid breaking, it should be heated and cooled gradually. Annealing by mild reheating and uniform cooling prevents breakage. Such glass should not be kept on cold surface while it is hot, since sudden cooling may break it.

Borosilicate glass does not soften below 700-800°C and requires oxygen-natural gas flame for working. Natural gas mixed with oxygen is burnt to get the oxygen-natural gas flame. Coefficient of expansion of this glass is low and apparatus made of this glass can withstand sudden changes in temperature. Therefore, apparatus used for heating purposes is made from borosilicate glass. On heating, glass apparatus made up of borosilicate glass does not distort.

In the following pages you will learn about some of the techniques of handling glass tubes and glass rods without injuring yourself. Also, you will learn the techniques of using laboratory apparatus and equipments.

2.1 CUTTING OF GLASS TUBE AND GLASS ROD

Material Required



- Soda-glass tube : 15 cm long
- Soda-glass rod : 15 cm long
- Triangular file : One

Procedure

- (i) Place the glass tube or the glass rod on the table and press it with your left hand.

- (ii) Keep the lower end of a triangular file with its sharp edge perpendicular to the tube to be marked and pull it towards you to make a single deep scratch on the glass tube or the glass rod at a desired length (Fig. 2.1 a).
- (iii) Keep thumbs of your hands on both sides, very close and opposite to the scratch as shown in Fig. 2.1 b and break the glass tube or rod by applying pressure from your thumbs in a direction away from you (Fig. 2.1 c). Break the tube/rod by holding it with a cloth so that hands are not harmed.
- (iv) If the glass tube does not break, make a deeper scratch at the point marked earlier and make a fresh attempt.
- (v) Trim any jagged edge by striking with a wire gauge (Fig. 2.2 a).
- (vi) Heat the freshly cut edge of the tube gently in the flame to make the edges round and smooth (Fig. 2.2 b). This is called fire polishing. For fire polishing, first continuously warm the cut end in the Bunsen flame and then rotate it back and forth until the edge is rounded. Too much heating may distort the rounded edge (Fig. 2.2 c).

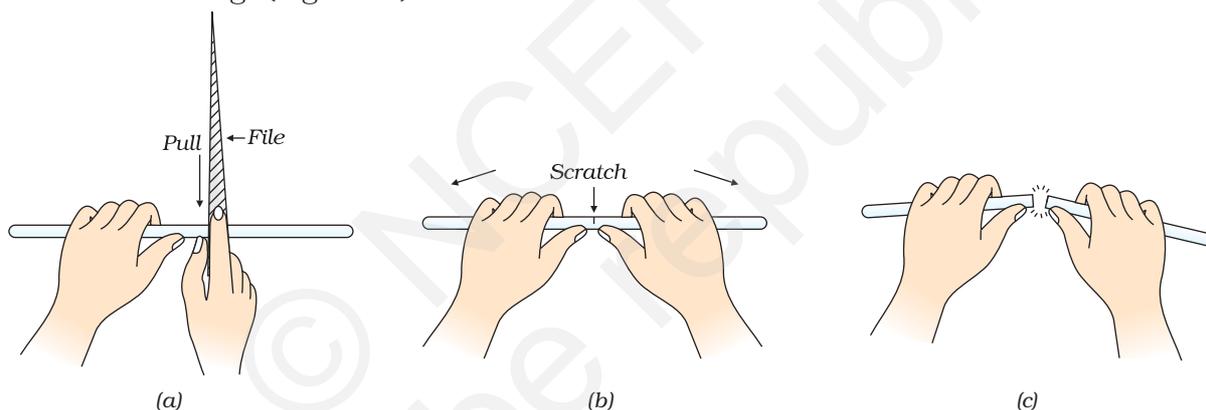


Fig. 2.1 : (a) Marking a glass rod or a glass tube
(b) Placing the thumbs together opposite to the scratch
(c) Breaking the glass rod or glass tube

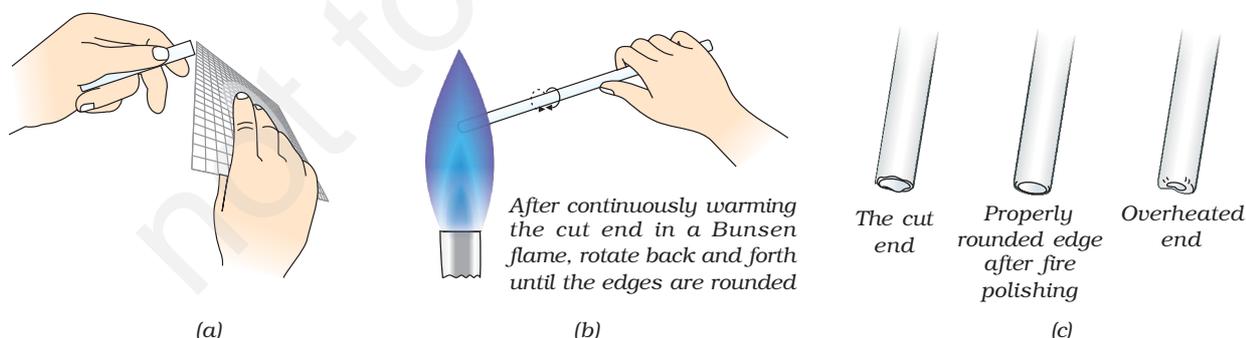


Fig. 2.2 : (a) Trimming the jagged edges
(b) Rounding the edges
(c) Properly and improperly rounded edges

Precautions

- Make a single deep scratch at the desired length with one stroke of the file.
- To avoid injury, carry out the filing and breaking of the glass tube/rod away from the face as far as possible and hold the glass tube / rod with the help of a piece of cloth to avoid injury to hands.



Discussion Questions

- Why does glass not possess a sharp melting point?
- Why is it required to round off the freshly cut edges of the glass tube or the glass rod?

2.2 BENDING OF A GLASS TUBE

Material Required



- Glass tube : 20-25 cm long
- Triangular file : One

Procedure

- Cut a tube of desired length with the help of a triangular file as described in section 2.1.
- Place the tube in the hottest zone of Bunsen burner flame and heat that portion from where it is to be bent (Fig. 2.3 a).
- While heating the tube in the flame keep it rotating slowly until the portion, which is to be bent, becomes red hot and soft and starts bending under its own weight. (Fig. 2.3 b).

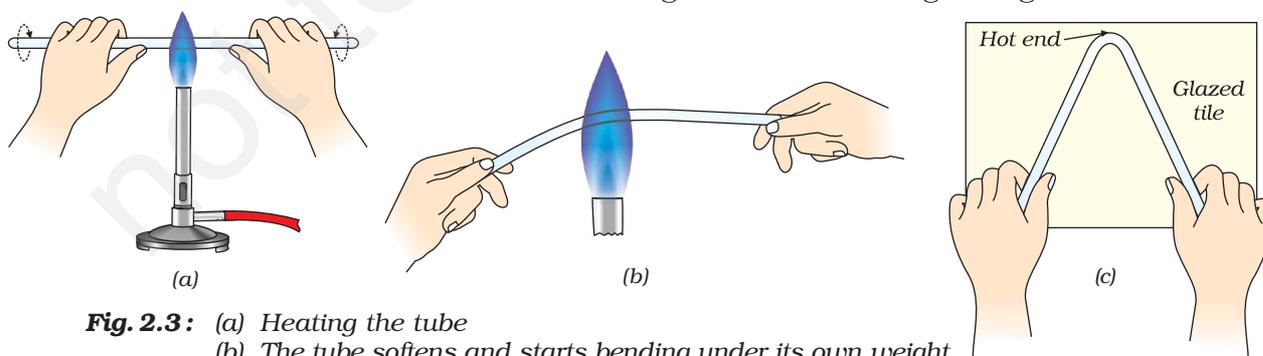


Fig. 2.3: (a) Heating the tube
 (b) The tube softens and starts bending under its own weight
 (c) Making the bend coplanar

- (iv) Remove the tube from the flame and bend it slowly at a desired angle by pressing it against a glazed tile to ensure the coplanarity of the bend (Fig. 2.3 c). Slow process of bending prevents flattening of glass tube (Fig. 2.4).
- (v) Cool it by placing on a glazed tile (Fig. 2.3 c).
- (vi) Bend the tubes at different angles as shown in Fig. 2.5.

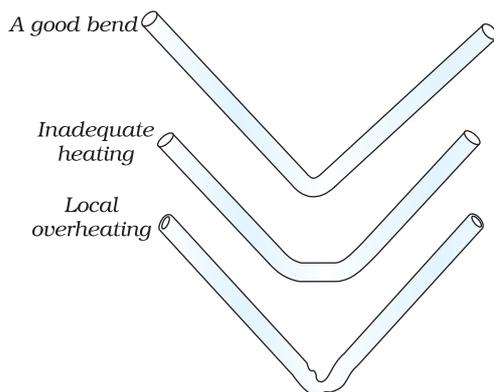


Fig. 2.4 : A glance of proper and improper bends

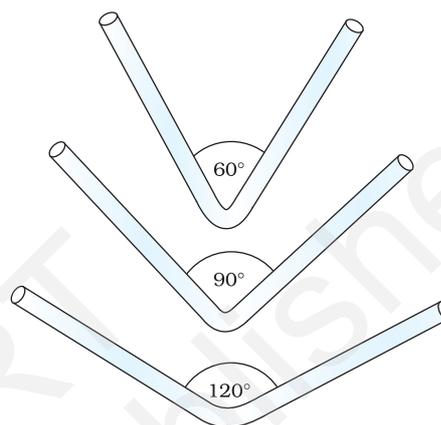


Fig. 2.5 : Bend drawn at various angles

Precautions

- Avoid heating the glass tube only on one side, rather rotate it while heating.
- Select a glass tube of appropriate length (nearly 30 cm long) to keep your hands safe from heat.
- To avoid flattening of the glass tube while bending, carry out the process slowly.

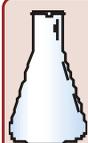


Discussion Questions

- Why should the tube be rotated while heating?
- Why is the red-hot tube bent slowly?

2.3 DRAWING OUT A JET

Material Required



- Glass tube : 20-25 cm long
- Triangular file : One
- Sand paper : As per need

Procedure

- (i) Select a glass tube of appropriate diameter for drawing a jet.
- (ii) Cut the glass tube of desired length with the help of a triangular file.
- (iii) Heat the tube in the hottest portion of the Bunsen burner flame by holding it at both the ends.
- (iv) Rotate the tube slowly until the portion, which is kept in the flame, becomes red hot and soft.
- (v) Remove the tube from the flame and pull the ends apart slowly and smoothly until it becomes narrow in the middle and then stretches into a fine jet as shown in Fig. 2.6 b.
- (vi) Cut the tube in the middle (Fig 2.6 c) and make the jet uniform and smooth by rubbing it with sand paper and by fire polishing.

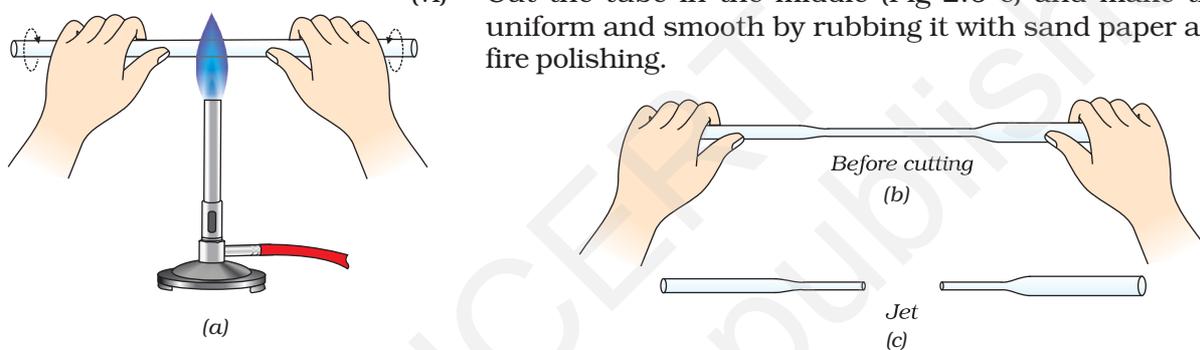


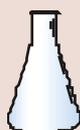
Fig. 2.6 : (a) Heating the tube to draw a jet (b) Before cutting (c) After cutting

Precaution

While drawing a jet, pull apart the two ends of the red-hot tube slowly so that it becomes thin uniformly.

**Discussion Questions**

- (i) What type of glass is preferred for drawing out a jet?
- (ii) Why is the glass tube of small diameter chosen for drawing out a jet?

2.4 BORING A CORK**Material Required**

- Rubber corks : As per need
- Cork borer set : One



- Glycerine solution (Shelf reagent) : As per need

Procedure

- (i) Mark the rubber cork on both its sides at the place where a hole is to be drilled (Fig. 2.7 a).
- (ii) Choose a borer of diameter slightly smaller in size than that diameter of the tube to be inserted in the hole (Fig. 2.7 b).
- (iii) Place the rubber cork on the table with its smaller end in the upward direction as shown in Fig. 2.7 c.
- (iv) Hold the cork in position with the left hand and put a suitable borer, lubricated by dipping in water or glycerine, at the place where the hole is to be drilled (Fig. 2.7 c). By lubricating the borer with water or glycerine a smooth hole is drilled.
- (v) Now hold and push the borer vertically in the downward direction, and drill the hole by rotating the borer and simultaneously apply the pressure gently.
- (vi) For making two holes in the same cork, keep appropriate distance between the holes and use borers of proper size.

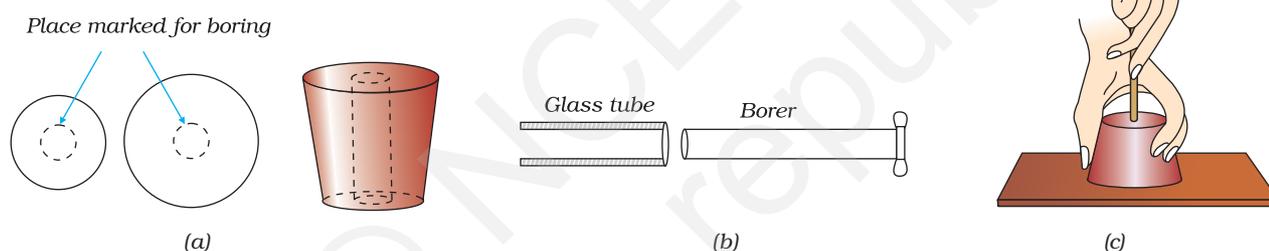


Fig. 2.7 : (a) Marked cork (b) Choosing the borer (c) Boring process

Precautions

- (a) Make a mark on both sides of the cork and select borer of proper size.
- (b) To obtain a smooth hole, drill half the hole from one side and another half from the other side of the cork.



Discussion Questions

- (i) What is the role of glycerine in the process of boring?
- (ii) Why should the diameter of the borer be less than the diameter of the tube to be inserted in the hole?

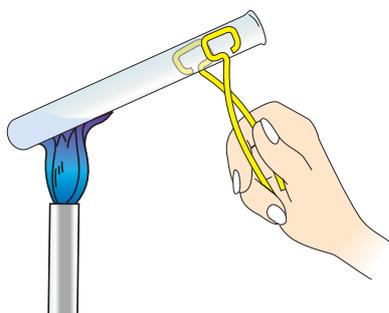


Fig. 2.8 : Heating solution in a test tube

2.5 HEATING SOLUTION IN A TEST TUBE

If a solution contained in a test tube is to be heated on a burner, hold the test tube with the help of a test tube holder at an angle and heat just below the surface of the liquid but not at the bottom (Fig. 2.8).

While heating, shake the test tube occasionally. If the test tube is heated at the bottom, a bubble may form causing the entire content to spill out of the test tube violently. This is called **bumping**. This can cause a serious accident, if the mouth of the test tube is pointing towards you or someone working near you. Therefore, when you heat a test tube over a burner, take care that its mouth does not point towards anyone. If content of the test tube is to be heated up to the boiling point, only one third of the test tube should be filled.

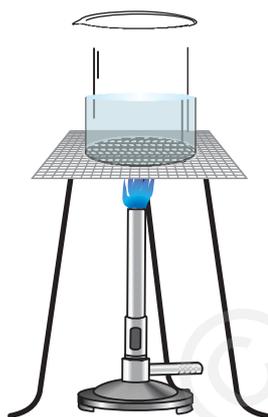


Fig. 2.9 : Heating solution in a beaker

2.6 HEATING SOLUTION IN A BEAKER OR A FLASK

If liquid is to be heated in a beaker or a flask, the beaker or the flask is placed on a wire gauze which in turn is placed on a tripod stand (Fig. 2.9).

For safe boiling, it is advisable to add a chip of broken china dish or carborundum/marble / a piece of capillary sealed at one end or any other non-reacting tiny material like pumice stone to avoid bumping.

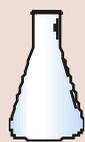
Note : (i) Never heat the apparatus with thick walls because it may break. Borosilicate glass apparatus is usually used for heating substances.

(ii) The apparatus, which is used for measuring volume, should also not be heated because heating may distort it and graduations may become invalid.

2.7 FILTRATION

Filtration involves separation of a solid from a liquid by passing the liquid through a porous material. In filtration, the porous filtering material can be a piece of cloth, paper, sintered glass, asbestos and so on. Filters of various pore sizes are available. If a filter paper has large pores, the liquid will pass through it more easily, and the filtration will be fast. However, solid particles of small size may also pass through the filter. Therefore, choice of the method of filtration and the filtering material depends on particle size of material to be retained on the filter paper.

Material Required



- Funnel : One
- Beaker : Two
- Funnel stand : One
- Glass rod : One
- Filter paper : As per need

Procedure

- (i) Fold the filter paper to fit in the funnel as shown in Fig. 2.10. For this, fold the circular filter paper in half, tear off a small piece of paper from the corner and once again fold it.
- (ii) Open the folded filter paper into a cone by keeping three folds on one side and one on the other such that the torn off corner is outside. Fit the cone into the funnel. Take care that filter paper cone fits in one cm below the rim of the funnel.

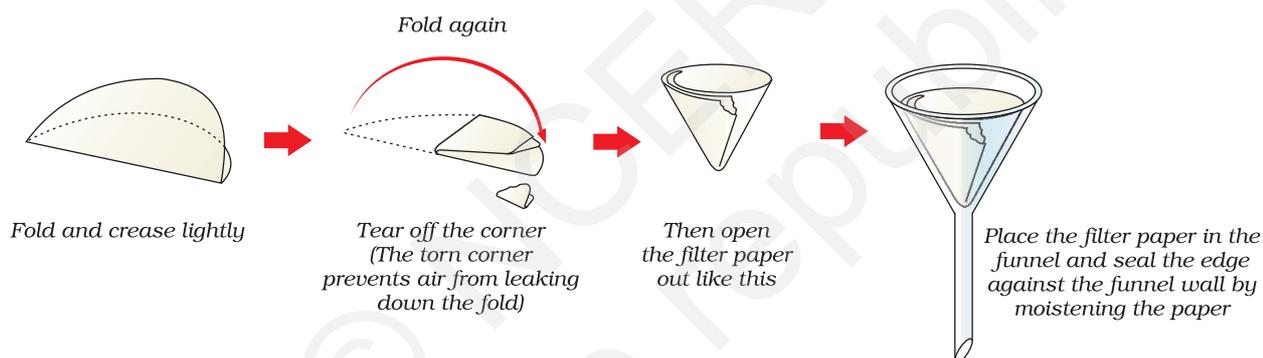


Fig. 2.10 : Folding the filter paper and placing it in the funnel

- (iii) Wet the paper with the solvent, which is usually water, and adjust it so that the entire cone tightly fits on the inner surface of the glass funnel and there is no air gap in between the paper cone and the glass.
- (iv) Add more water so that the stem of the funnel is filled with water. If the filter paper is fitted correctly, the filter paper will support a column of water in the funnel stem. The weight of this column of water produces a mild suction that expedites filtration (Fig. 2.11).

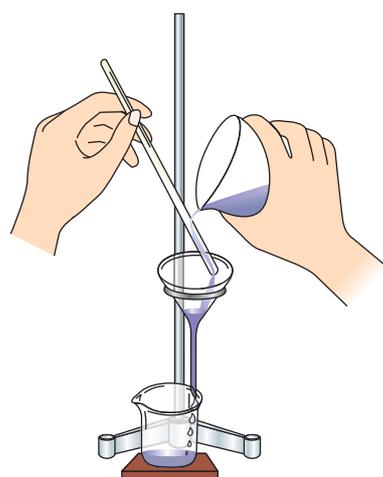


Fig. 2.11 : Process of filtration

Precautions

- The stem of the funnel should touch the side of the beaker in which filtrate is collected so that falling drops do not cause splashing.
- Filter paper cone should not be filled more than two-thirds. If level of the liquid being filtered rises above the cone, then some unfiltered solution may pass into the beaker kept below the funnel to collect the filtrate.

Note : (i) For quick filtration, a fluted filter paper may be used with advantage. The ordinary paper is folded into 6 or 16 folds instead of 4 and the folds are then turned alternately inwards and outwards. On opening the paper we get a cone of fluted filter paper with series of ridges meeting at the apex. Filtration is rapid due to the large surface available for filtration (Fig. 2.12).

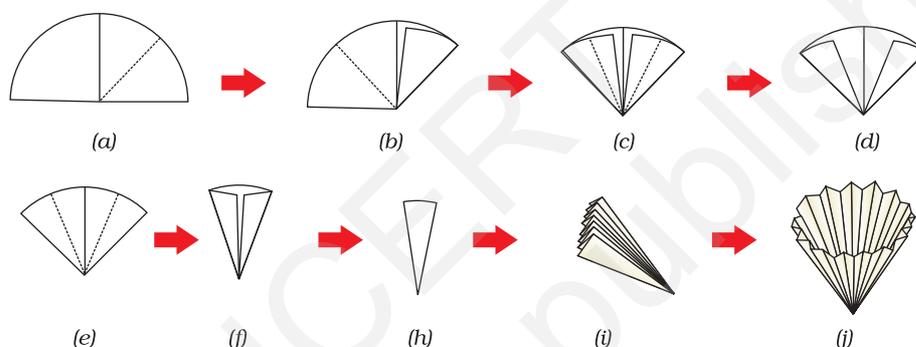


Fig. 2.12 : Folding the filter paper to get a fluted filter paper cone

- For separating the solid from the liquid, filtration should be done in two stages. First, almost whole of the liquid should be poured out carefully down a stirring rod (Fig. 2.11). When only a few millilitres of the mixture remain in the beaker, it should be poured into the funnel after swirling the beaker gently. The sides of the beaker are then rinsed with a stream of water and the content is again poured into the funnel. Rinsing is repeated till the beaker and the stirring rod is clean. It is better to pour down a solid liquid mixture along a glass rod (Fig. 2.11). However, care should be taken that paper is not punched by the stirring rod.

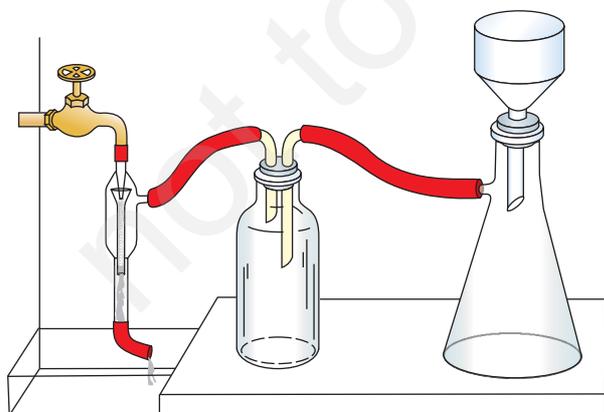


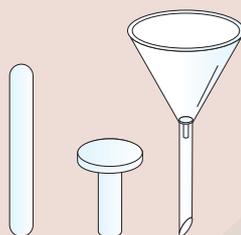
Fig. 2.13 : Suction filtration

Suction Filtration : Filtration in the above manner is a slow process. It can be speeded up by carrying out filtration under reduced pressure using suction, which can be applied by means of water aspirator (Fig. 2.13) or vacuum pump. Water aspirator can be fitted on to the tap through a rubber tubing. It uses fast stream of water to suck in air through the side arm. Suction is quite strong, therefore a special funnel called buchner funnel is used for filtration. It is fixed on to the mouth of the filtration flask through a rubber cork (Fig. 2.13).

IMPROVISATION

If you do not have a Buchner funnel or if you have very small amount of substance to be filtered, try following improvised apparatus for suction filtration. Take a glass rod and check that it passes through the stem of the funnel freely. Flatten one end of the glass rod by heating it in a bunsen burner flame and then pressing it against the glazed tile. The flat portion of the rod will now fit into the upper part of the stem of the funnel.

Cut the length of the rod to a small size so that it forms a button with a small stem. Fit the button in the funnel as shown in the figure below.



Cut a small circular piece of filter paper of the size such that it covers the flat button and just touched the sides of the funnel. Moisten the filter paper and use this improvised funnel in place of a Buchner funnel. Try using shirt button in place of this button.

2.8 MEASURING VOLUME OF LIQUIDS

Usually volumetric flasks, graduated cylinders, pipettes and burettes are used for measuring volume of liquids. Volumetric flasks and cylinders are graduated to measure volume of a liquid at a certain temperature. Pipettes and burettes are calibrated to deliver certain specific volume of a liquid at a specified temperature. The capacity mark is usually etched on the glass of the equipment.

Aqueous solutions wet the glass surface, therefore these form concave meniscus when filled in these equipments. Central part of the meniscus is rather flat (Fig. 2.14 a). Calibration of the

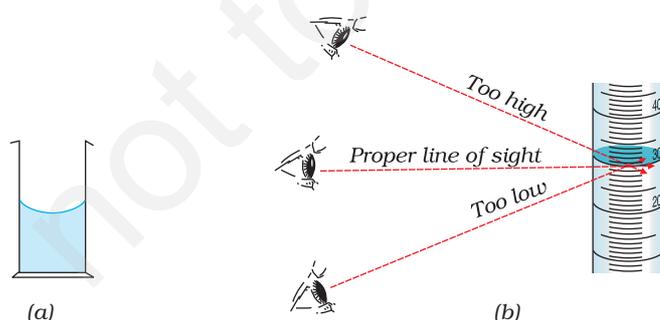


Fig. 2.14 : (a) Water forming curved surface in the glass apparatus
(b) Noting the reading

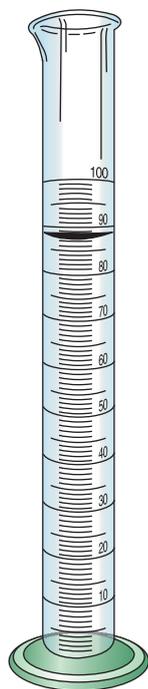


Fig. 2.15 : Measuring Cylinder

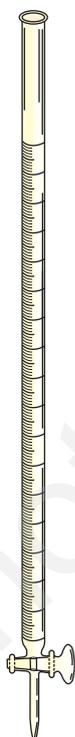


Fig. 2.16 : Burette

apparatus coinciding with this flat portion of the meniscus gives a measure of the volume of the liquid. Therefore, while making final adjustment of volume or noting the reading, the curved surface of the liquid should appear touching the etched mark when viewed by keeping the eye level aligned to the etched mark (Fig. 2.14 b). This helps in avoiding the parallax errors (error caused by the change in position of the observer). Note that if the liquid forms convex meniscus or is coloured and opaque e.g. KMnO_4 solution then reading coinciding with upward surface is noted. In flasks and pipettes capacity mark is etched on the narrow part of the equipment to minimize the error in noting the level of meniscus. Graduated cylinders are not used for very precise measurements, so they need not be narrow. Burettes and pipettes are used to measure the volume of a liquid accurately.

(a) Using Graduated Cylinder

Always a clean graduated cylinder (Fig. 2.15) should be used for measurement because dirt may chemically contaminate the substance being measured and it may deter accurate determination of volume. Dirty glassware does not drain properly and the volume delivered may not be equal to that indicated by calibration mark. Measuring cylinders of 5mL, 10mL, 25mL, 100mL, 250mL, 500mL, 1000mL and 2000mL capacity are available. Measuring cylinders used for delivering the volume actually contain slightly more than the volume read. This compensates for the film of liquid left on the walls when liquid is poured out.

(b) Using Burette

A burette is simply a long graduated tube of uniform bore with a stopcock or a pinchcock at one end (Fig. 2.16). It is used for measuring volume in a quantitative (titrimetric) estimation. The burette reading is noted before and after delivering the liquid. The difference between these two readings is the volume of the liquid delivered. The liquid should be delivered dropwise. If the liquid is allowed to run too fast, the walls of the burette will not drain properly and some liquid may remain sticking to the surface of the walls. This may lead to faulty reading. Measuring capacity of the burette usually used in the laboratory is 50 mL.

Before filling the solution to be used, the burette should be rinsed with the solution to be filled. For rinsing the burette, few millilitres of solution are taken into it and the whole inner surface of burette is wetted with the solution by rotating it. After rinsing, the solution is drained out of the nozzle of the burette (Fig. 2.17).

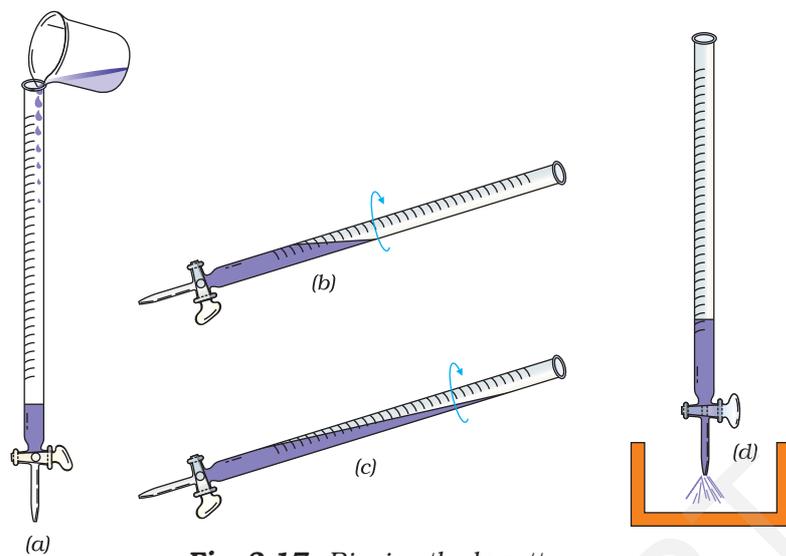


Fig. 2.17 : Rinsing the burette

After rinsing, the solution is filled in the burette with the help of a funnel above zero mark. Stopcock is then opened wide and the solution is allowed to run through the nozzle till there are no air bubbles in it (Fig. 2.18).



Fig. 2.18 : Filling the burette

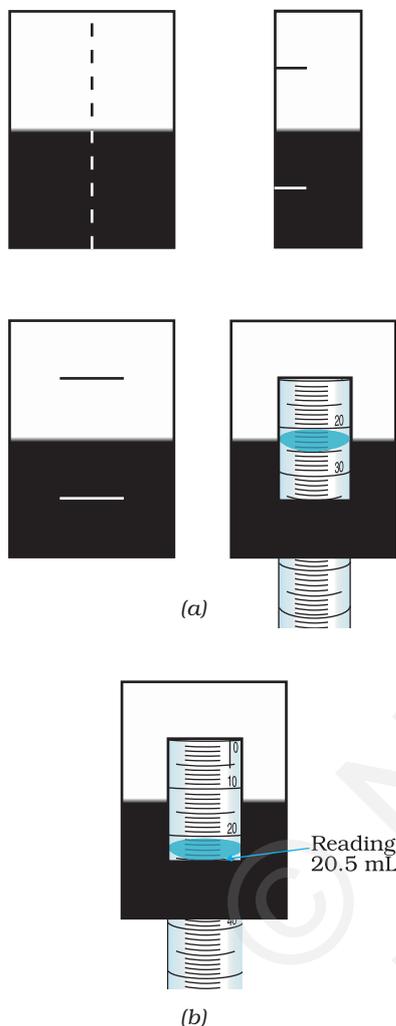


Fig. 2.19 : (a) Mounting anti parallax card on burette
 (b) Using anti parallax card for noting correct reading

In order to read the level of a liquid in the burette, hold a half blackened white card called **anti parallax card** behind the burette at the level of the meniscus, so that the black area appears to be just touching the meniscus of the liquid (Fig. 2.19 a, b). The eye must be levelled with the meniscus of the liquid to eliminate parallax errors. Read the graduation on the burette touching the black part of the card (Fig. 2.19 b). Always remember that for all transparent solutions in the burette, reading coinciding with the lower meniscus is noted and for all dark coloured solutions (e.g. potassium permanganate solution) reading coinciding with the upper meniscus is noted. Never forget to remove the funnel from the burette before noting the reading of the burette and ensure that the nozzle is completely filled. While noting the reading take care that no drop is hanging at the nozzle of the burette.

(c) Using Pipette

Normally pipettes of measuring capacity 1 mL, 2 mL, 5 mL, 10 mL, 20 mL, 25 mL etc. are used. Graduated pipettes are also used in the laboratory work (Fig. 1.3).

Pipette (Fig. 2.20 a) is used for measuring volumes of liquids, when these are to be transferred to a flask or some other apparatus. Liquids are sucked into the pipette by applying suction through mouth or by using a pipette filler bulb or a pipette filler pump. It is always safe to use pipette filler bulb or pipette filler pump to fill the pipette. When poisonous and corrosive solutions are to be drawn into the pipette, **never suck by mouth. Use a pipette filler bulb to draw the liquid up into the pipette.** Hold the pipette in one hand tightly, dip the jet of the pipette into the solution to be pipetted out, and squeeze the pipette bulb with the other hand (Fig. 2.20 b). Now loosen your grip on the bulb so that the liquid is sucked into the pipette. When the liquid is above the etched mark on the pipette, remove the bulb, and put the index finger of the hand at its place holding the pipette as shown in Fig. 2.20 c. Loosen the finger carefully to allow the excess liquid to flow out so that the curvature of the meniscus reaches the mark. Now carefully remove the finger and let the liquid run into the flask (Fig. 2.20 d). After emptying the pipette do not blow out the remaining liquid. Pipettes are so designed that the little amount of liquid, which remains untransferred, is not taken into calibration (Fig. 2.20 e).

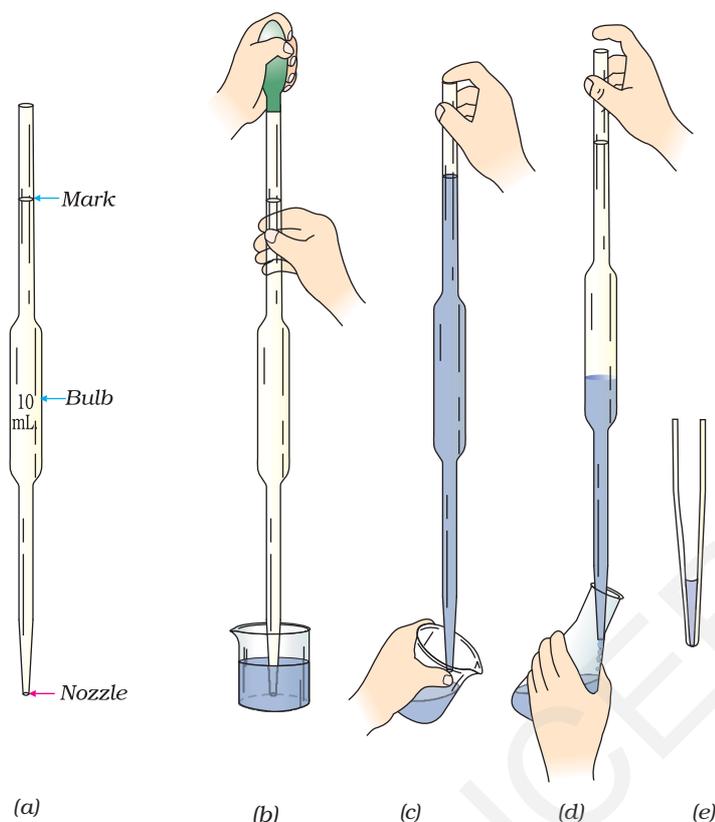


Fig. 2.20 : (a) Pipette
 (b) Using pipette filler bulb
 (c) Holding the pipette after removing the bulb
 (d) Transferring liquid to the flask
 (e) Nozzle of the pipette after measuring solution

To take out the maximum volume after complete transfer, simply touch the pipette to the side or base of the container into which the liquid is being transferred (Fig. 2.20 d).

The pipette should always be rinsed with the solution which is to be measured by it. For this fill the pipette with few millilitres of solution and wet the inner surface of the pipette with the solution by rotating it (Fig. 2.21). After rinsing, drain out the whole amount of solution taken in it through the nozzle. Now it is ready for measuring the solution. Note that while handling the pipette, hands should be dry so that pressure is regulated easily. Also, nozzle of the pipette being used should not be broken.

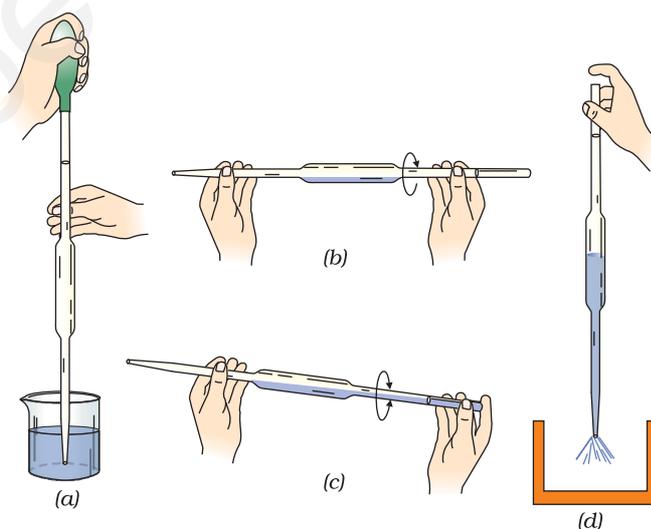


Fig. 2.21 : Rinsing the pipette

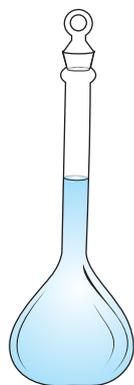


Fig. 2.22 : The measuring flask

(d) Using Measuring Flask

These are employed for making specific volumes of solutions. This is also called graduated flask or volumetric flask. It is a pear shaped vessel with a long narrow neck and flat bottom (Fig. 2.22). A thin circle etched around the neck indicates the volume of the liquid that it holds at a definite temperature.

The temperature and the capacity of the flask at that temperature are marked on the flask. The mark around the neck helps in avoiding errors due to parallax when making the final adjustment of meniscus. The lower edge of the meniscus of the liquid should be tangential to the graduated mark. While making final adjustment of the meniscus, the front and the back portion of the circular mark should be observable in single line. The neck of the flask is made narrow to reduce error in adjustment of the meniscus. In narrow space small change in volume makes large effect on the height of the meniscus.

Measuring flasks of various capacities are available. Usually at this level of experimentation flasks of capacity 50 mL, 100 mL and 250 mL are employed in the work. The method of preparation of solution by using measuring flask has been described in the Experiment 2.1 later in this unit.

2.9 WEIGHING TECHNIQUE

(a) Acquaintance with Analytical Balance (Chemical Balance)

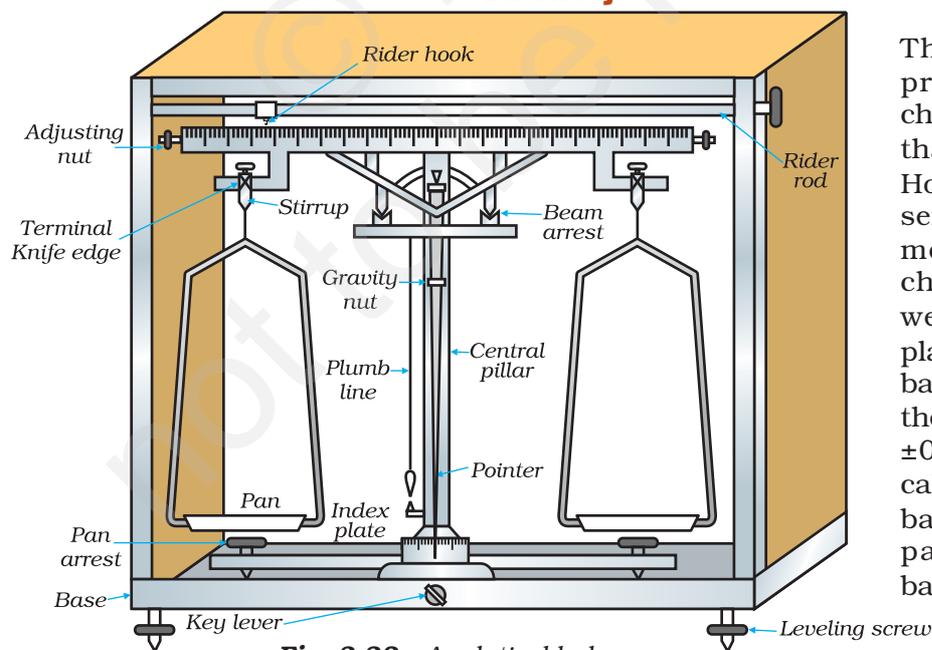


Fig. 2.23 : Analytical balance

The construction and principle of working of a chemical balance are same as that of a physical balance. However, due to its higher sensitivity, its accuracy is more. With the help of a chemical balance, one can weigh accurately up to 4 places of decimal. Analytical balance can be used to weigh the mass of a substance upto ± 0.0002 g accuracy. It is called the least count of the balance. A full view of a two-pan analytical chemical balance is shown in Fig. 2.23.

In this type of balance, the beam is made up of a hard but light weight material. The beam pivots at its centre on a knife-edge, which rests upon a plate made of very hard material such as agate or corundum. The plate is attached to the central beam support (central pillar). The two terminal agate knife-edges are fixed at equal distance from the central edge and each of these supports a suspension called stirrup from which the pans are hung. A sharp pointer is attached to the centre of the beam (Fig. 2.24 a). The pointer moves over a scale fixed at the bottom of the pillar and serves to point out the deflection of the beam from central position when the balance is in operation (Fig. 2.24 b). There are two adjusting screws on both sides of the beam, which are meant for adjusting the beam in the horizontal position. There are three leveling screws at the base of the balance to make it horizontal. A plumb line hangs near the central pillar, which helps in keeping the balance horizontal. In order to operate the balance there is a knob at the centre of the base.

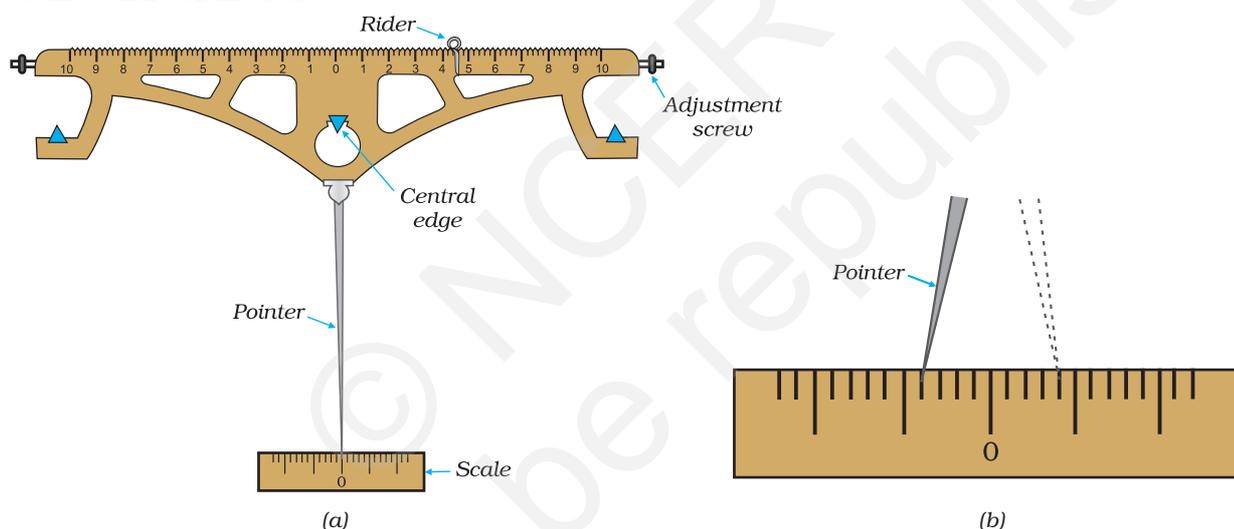


Fig. 2.24 : (a) Pointer attached to the beam
(b) Movement of pointer

(b) Weight Box Including Fractional Weights and Riders

The weight box of a chemical balance generally contains the following weights.

- Weights for weighing in grams : 100, 50, 20, 20, 10, 5, 2, 2, 1
- Weights for weighing in milligrams : 500, 200, 200, 100, 50, 20, 20, 10
- Rider : To weigh 0.2 mg to 10 mg.

The three categories of weights for weighing in a chemical balance are shown in Fig. 2.25. Materials used for making the weights are as follows :

Gram Weights : Made of copper and nickel with or without coating of chromium.

Milligram Weights : Made of Aluminium/German silver/Stainless steel.

Rider : A loop made of aluminum or platinum wire weighing 10.0 mg.

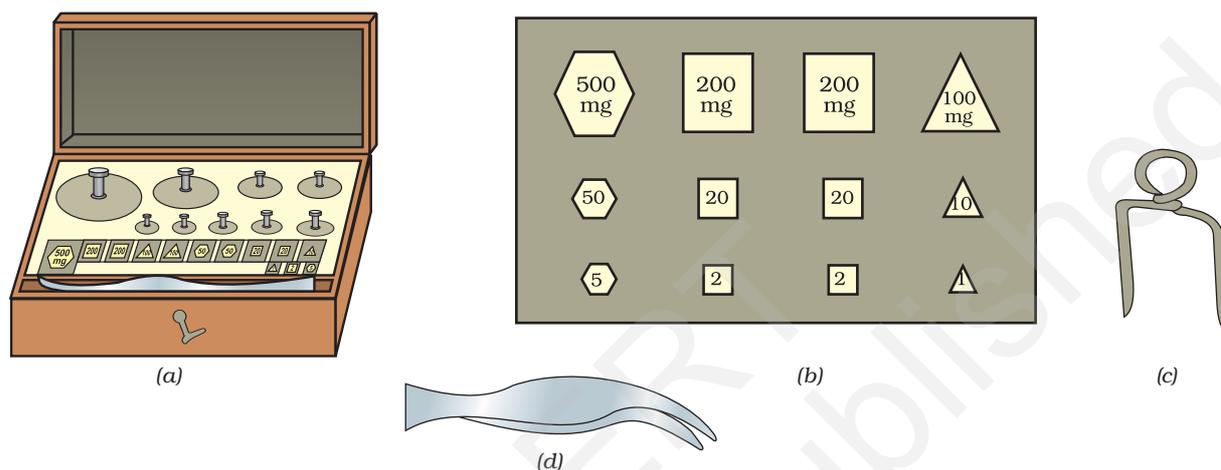


Fig. 2.25 : (a) Weight box (b) Fractional weights (c) Rider and (d) Forceps

(c) Setting of a Chemical Balance and Weighing

Material Required



• Chemical balance	:	One
• Weight box	:	One
• Set of fractional weights including rider	:	One
• Weighing bottle/watch glass	:	One

Procedure

Following steps are followed while using a chemical balance:

- (i) Level the balance with the help of leveling screws and plumb line.
- (ii) Ensure that the beam is horizontal. Adjust the pointer at zero point with the help of screws provided on both sides of the beam. If it is adjusted on releasing the beam arrest, the pointer moves equal divisions on both the sides of the zero of the base scale.

- (iii) Place a watch glass/weighing bottle on the left pan in which weighing material is kept. Put approximate weights from the weight box with the help of forceps on the right pan.
- (iv) Release the beam arrest slowly and note the movement of the pointer on the scale. If its weight is not appropriate, the pointer will move towards the lighter side. Add or remove weights according to the requirement after bringing the pans to rest by arresting the beam with the help of the knob located near the base. When weight on both the pans becomes equal, the pointer moves equal divisions on both sides of the zero of the base scale.
- (v) Use the rider for adjustment of weight below 10mg.

Using Rider

Maximum weight that can be weighed with the help of rider is 10 mg and the weight of the rider itself is 10 mg (i.e. 0.01g). It can be easily placed in the grooves of the balance beam (Fig. 2.26). When placed at the terminal position of the beam, which is marked 10, it will weigh 10 mg (i.e 0.01g). Principle of moment is applied for weighing by using the rider. Weight is equal to the arm length from the centre of the beam multiplied by the weight of the rider. Length of the beam from the centre to one side of the beam is taken as unit length.

On both sides from the centre, the balance beam is divided into ten equal parts through equidistant marks, each of which corresponds to $1/10$ of the length of the beam. Hence, each big division corresponds to $\frac{1}{10} \times 0.01\text{g} = 0.001\text{ g}$ or 1 mg weight. Each big division is further divided into five parts. Thus, each small division corresponds to only $1/5$ milligram i.e. 0.2 mg or 0.0002 g. Thus, the rider placed at the 4.2 mark will weigh 0.0044 g (i.e. $4 \times 0.001 + 2 \times 0.0002 = 0.0044\text{ g}$) (Fig. 2.26).

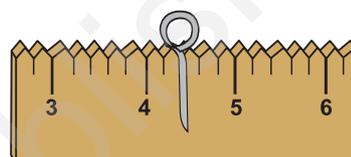


Fig. 2.26 : Rider resting on groove of balance beam

Precautions

- (a) Pans must be properly cleaned before and after weighing. Chemicals should never be placed directly on the pans for weighing.
- (b) Always release the beam gently.
- (c) Avoid overloading the balance.
- (d) Always transfer the weights from one place to another with the help of forceps.
- (e) Do not allow the weights to get spoiled by corrosion.
- (f) Never weigh a hot/cold object on the balance.
- (g) Always keep weights on the right pan and object on the left pan of the balance (if you are a right handed person).

- (h) Always make necessary adjustments in the balance before weighing.
- (i) To insert or remove weights and to keep the object on the pans always use the side doors. Never use the front shutter.
- (j) Keep the doors shut while releasing the beam arrest to note the movement of the pointer on the scale.



Discussion Questions

- (i) How is an analytical balance different from a physical balance?
- (ii) On what principle, is weighing by using rider based?
- (iii) What is the maximum weight that can be weighed on a chemical balance?
- (iv) Which weights are called fractional weights?
- (v) Why are forceps always used for handling the weights?
- (vi) The rider rests at a reading of 3.4 on the left side of the beam. What contribution does this make to the weight of the material being weighed when weights are placed on the right pan?
- (vii) Can you weigh 0.0023 g using chemical balance? Give reason for your answer.

EXPERIMENT 2.1

Oxalic acid

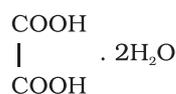


Aim

Preparation of 250 mL of 0.1M standard solution* of oxalic acid.

Theory

A solution of exactly known concentration is considered to be a standard solution. There are various ways of expressing the concentration of a standard solution. Standard solution of an acid/base is used to determine the unknown concentration of a solution of bases / acids by volumetric analysis. For example, a standard solution of oxalic acid can be used to determine the unknown concentration of an alkali solution. The strength of a standard solution is usually expressed in moles per litre. The formula of hydrated crystalline oxalic acid is



* Learn more about standard solution in Unit-6.

and its molar mass is 126 g. If 126 g of oxalic acid is present in one litre of the solution, it is known as one molar (1.0 M) solution.

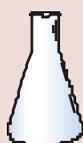
For the preparation of one litre of 0.1 M oxalic acid solution, we require $\frac{126}{10} = 12.6$ g of hydrated oxalic acid. Therefore, for preparing 250 mL of 0.1 M oxalic acid solution, we require:

$$\frac{12.6 \text{ g} \times 250 \text{ mL}}{1000 \text{ mL}} = 3.1500 \text{ g of hydrated oxalic acid.}$$

In general for preparing a solution of required molarity, the amount of substance to be weighed can be calculated by using the formula given below :

$$\text{Molarity (M)} = \frac{\text{Mass of solute in grams} \times 1000}{\text{Molar mass of solute} \times (\text{volume of solution to be prepared in mL})}$$

Material Required



- Measuring flask (250 mL) : One
- Funnel : One
- Weighing tube/Watch glass : One
- Wash bottle : One
- Iron stand with ring clamp : One



- Oxalic acid : As per need

Procedure

- (i) Weigh an empty, clean and dry watch glass/weighing tube accurately (Weight 1).
- (ii) Weigh 3.1500 g oxalic acid by placing it on the above watch glass/in a weighing tube (Weight 2). Always note weight up to the fourth decimal place and clean the balance before and after weighing the chemical.
- (iii) Transfer oxalic acid carefully from the watch glass/weighing tube into a clean and dry measuring flask using a funnel. Weigh the empty watch glass again (Weight 3) and find out the mass of oxalic acid transferred to the measuring flask by subtracting this mass (Weight 3) from the combined mass of watch glass and oxalic acid (Weight 2). Calculate the exact molarity of solution from this mass. Wash funnel several times with distilled water by using a wash bottle to transfer the sticking particles if any into the measuring flask. While washing the funnel, add water in small amounts so that its volume in the flask does not exceed $\frac{1}{4}$ th of the volume of the measuring flask as shown in Fig. 2.27 a, b.

Oxalic acid



- (iv) Swirl the measuring flask till solid oxalic acid is completely dissolved. Add more distilled water with shaking. Make up the volume with distilled water to the etched mark by adding last few mL dropwise. Stopper the flask and shake it thoroughly to make the solution uniform throughout (Fig. 2.27 c, d). Label it as 0.1 M oxalic acid solution.

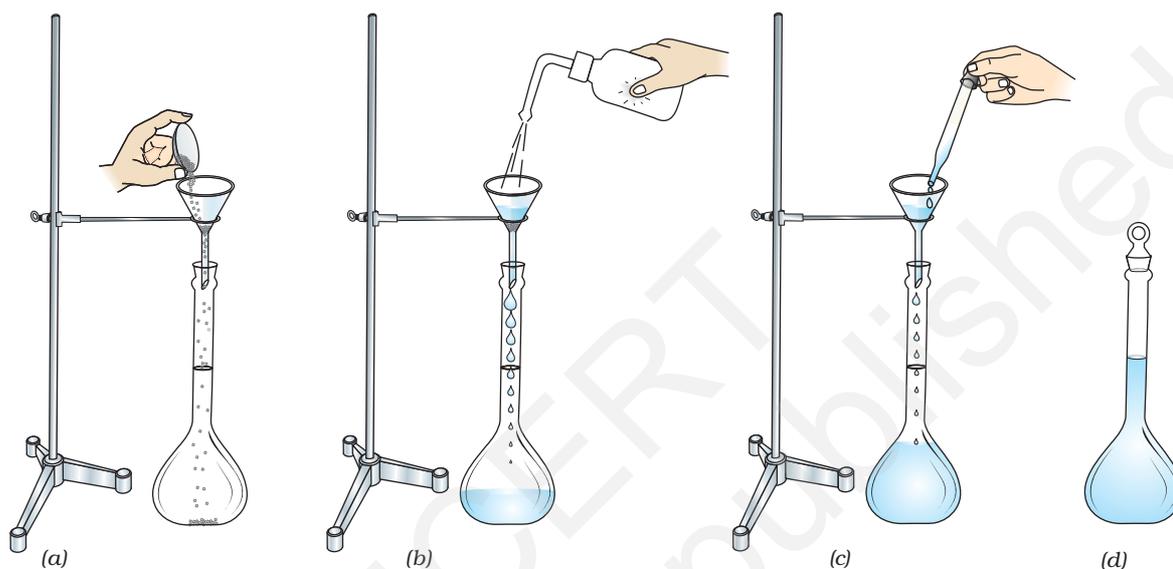


Fig. 2.27 : Making standard solution

(a) Transferring oxalic acid

(b) Diluting the solution

(c) Adding last few mL dropwise

(d) Standard solution

Precautions

- The pan of the balance should be cleaned before and after weighing.
- Never touch the weights with hand. Use forceps to transfer weights from the weight-box to the pan of the balance.
- Always use spatula to transfer the reagent from the bottle on to the watch glass.
- Stopper the reagent bottle immediately after withdrawing the substance.
- Always use distilled water to prepare the standard solution.
- Always check the adjustment of the balance before weighing the substance.
- Care should be taken while weighing the chemicals. These should not be spilled on the pan of the balance.
- Watch glass/weighing bottle and funnel should be washed several times by using small amounts of distilled water each time.
- While making the solution, water should be added carefully so that the lower part of the meniscus just touches the etched mark of the measuring flask.
- To ensure uniform composition of the solution, stopper the flask and shake it carefully and thoroughly.



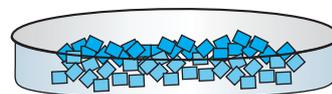
Discussion Questions

- (i) What is the formula and the basicity of hydrated oxalic acid and anhydrous oxalic acid?
- (ii) What do you mean by a molar solution?
- (iii) Why are the standard solutions always prepared in a volumetric flask?
- (iv) How will you prepare 250 mL of 0.05 M oxalic acid solution?
- (v) Can solid NaOH be used to prepare its standard solution?
- (vi) What type of substance can be used for preparing standard solution?
- (vii) What is meant by “weighing by transfer”? When is this used?

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UNIT-3

PURIFICATION AND CRITERIA OF PURITY



FOR the identification of a compound, qualitative analysis of pure substance is required. Therefore, first we have to purify the substance and then check its purity. There are many techniques namely, crystallisation, distillation, sublimation, chromatography etc. available for purification of a compound. In this unit you will learn about crystallisation as a technique for purification of a compound. The purity of a compound may be checked by determining its melting or boiling point. The technique for determination of melting and boiling points will also be described in this unit. Pure solid and liquid compounds possess sharp melting and boiling points. Therefore, melting and boiling points of a compound can be used as a criteria of purity.

EXPERIMENT 3.1

Aim

Purification of sample of any one of the following Potash alum, Copper sulphate or Benzoic acid by crystallisation.

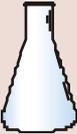
Theory

Crystallisation is one of the techniques for the purification of an impure compound particularly when the original crude material obtained after a reaction is in a very impure condition. First step of the process involves choosing a single solvent or a mixture of solvents, which dissolves the crude material readily when hot, but only to a small extent when cold. The crude substance is then dissolved in the minimum amount of boiling solvent to obtain a saturated solution. Insoluble impurities are removed by filtering the hot solution. It is then checked for crystallisation point and then cooled slowly when the solute crystallises out leaving the greater part of impurities in the solution. The crop of crystals is collected by filtration and the process is repeated until the crystals of pure substance are obtained. Sometimes during cooling minute quantity of the substance (solid which is being purified) is added to the solution to facilitate the initial crystallisation. This is called **seeding**. The added tiny crystal acts as a 'nucleus' for the growth of new crystals. Growth of crystals depends upon the conditions in which crystallisation is carried out. For obtaining good crystals, rapid cooling should be avoided because it results into small or disfigured crystals.

Purity of crystals is often judged from the colour of the crystals. For example, pure crystals of alum, copper sulphate and benzoic acid are white, blue and

greenish white respectively. Impurities impart colour to the crystals; therefore, impure crystals have a colour different from pure crystals.

Material Required

	• Beaker (250 mL)	:	One		• Potash alum, Copper sulphate and Benzoic acid	: As per need
	• Glass funnel	:	One			
	• Tripod stand	:	One			
	• Porcelain dish	:	One			
	• Glass rod	:	One			
	• Sand bath	:	One			

Procedure

- (i) Take 30-50 mL distilled water in a beaker and prepare a saturated solution of potash alum/copper sulphate in it at room temperature by adding the impure solid sample in small amounts with stirring. Stop adding the solid when it does not dissolve further. To prepare saturated solution of benzoic acid use hot water.
- (ii) Filter the saturated solution so prepared and transfer the filtrate into a porcelain dish. Heat it on a sand bath till nearly $\frac{3}{4}$ th of the solvent is evaporated. Dip a glass rod into the solution, take it out and dry it by blowing air from the mouth. If a solid film deposits on the rod, stop heating.
- (iii) Cover the porcelain dish with a watch glass and keep the content of the dish undisturbed for cooling.
- (iv) When crystals are formed, remove the mother liquor (liquid left after crystallisation) by decantation.
- (v) Wash the crystals of potash alum and copper sulphate, thus obtained first with very small quantity of alcohol containing small amount of cold water to remove the adhering mother liquor and then with alcohol to remove moisture. Wash the crystals of benzoic acid with cold water. Benzoic acid is soluble in alcohol. Do not use alcohol to wash its crystals.
- (vi) Dry the crystals between the folds of a filter paper.
- (vii) Store the dry crystals thus obtained at a safe and dry place.
- (viii) Repeat steps (ii-vii) for obtaining maximum amount of pure substance.

Copper sulphate



Precautions

- (a) Do not evaporate the entire solvent while concentrating the solution.
- (b) Do not disturb the solution while it is being cooled.
- (c) Use the washing liquid in 3-4 very small installments rather than in one installment.



Discussion Questions

- (i) Which one of the following formula is correct representation of potash alum(phitkari)? Explain.
 - (a) $K^+(H_2O)_6 Al^{3+} (H_2O)_6 (SO_4^{2-})_2$
 - (b) $K_2SO_4 \cdot Al_2(SO_4)_3 \cdot 24H_2O$
- (ii) What are isomorphous compounds?
- (iii) What is meant by the term, 'water of crystallisation'?
- (iv) Describe the effect of strong heating on each type of crystal prepared by you.
- (v) What do you understand by the term mother liquor?
- (vi) Which thermodynamic function favours the process of crystallisation?
- (vii) Explain the term-saturated solution?
- (viii) Why is the preparation of a saturated solution essential for making crystals?
- (ix) Name the processes involved in crystallisation?
- (x) What is Kipp's waste? How can we obtain crystals of ferrous sulphate from Kipp's waste?

EXPERIMENT 3.2

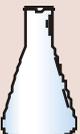
Aim

Determination of melting point of a solid organic compound.

Theory

The kinetic energy of molecules of a substance increases on heating. When it becomes high enough to overcome the attractive forces operating between the molecules, the lattice structure of the solid breaks, the solid melts and comes into the liquid state. Melting point of a substance is the temperature at which solid state of a substance begins to change into the liquid state, when the pressure is one atmosphere.

Material Required

	• Thiele's tube				• Liquid paraffin		
	/Kjeldhal's flask/beaker	: One			/Conc. H ₂ SO ₄	: As per need	
	• Thermometer	: One			• Organic Compound		
	• Capillary tubes	: As per need			(Naphthalene/ <i>p</i> -Dichlorobenzene/ <i>p</i> -Toluidine)	: As per need	
	• Iron stand with clamps	: One					

Procedure

- (i) Take a capillary tube of approximately 8 cm in length. Seal its one open end by heating it in a Bunsen flame. Rotate the capillary while sealing to ensure complete closure of the opening.
- (ii) Crush the desired substance (about 100 mg) into fine particles and fill the substance in the capillary tube up to nearly 1cm length. For filling the capillary, dip its open end in to the powder. Hold the sealed end between the index finger and the thumb and tap the upper end gently with the other hand so that solid particles are tightly packed and capillary is prevented from breaking.
- (iii) Moisten the capillary tube with liquid paraffin and stick it to the thermometer. It will stick to the thermometer by cohesive forces. See that the lower ends of the capillary tube and the thermometer bulb are at the same level. The thermometer is fitted into a rubber cork, which has a groove on its side for the escape of air and vapours.
- (iv) Take a Thiele's tube (Fig. 3.1 a) and fill it with 50 to 60 mL liquid paraffin so that it crosses the bent portion of the Thiele's tube. Alternatively, Kjeldahl flask's may be used in place of Thiele's tube.
- (v) Dip the thermometer along with the capillary tube in liquid paraffin and adjust the rubber cork in such a way that the thermometer bulb and the filled portion of the capillary is completely dipped in the liquid paraffin and the open end of the capillary remains in the air as shown in Fig. 3.1 a. The thermometer and the capillary tube should not touch the sides of the Thiele's tube.
- (vi) Now start heating the side arm of the Thiele's tube with a low flame from the side opposite to that of the capillary tube and note the temperature when the solid starts melting.

p-Dichlorobenzene



p-Toluidine



Naphthalene



Hazard Warning

- Avoid contact with skin and eyes and don't inhale vapours of these chemicals.

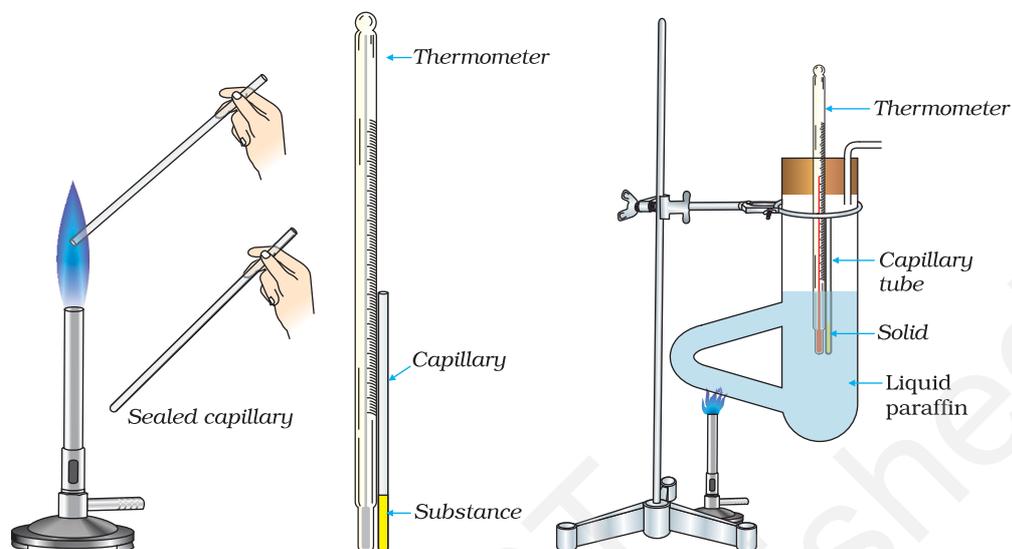


Fig. 3.1 : (a) Determination of melting point using Thiele's tube

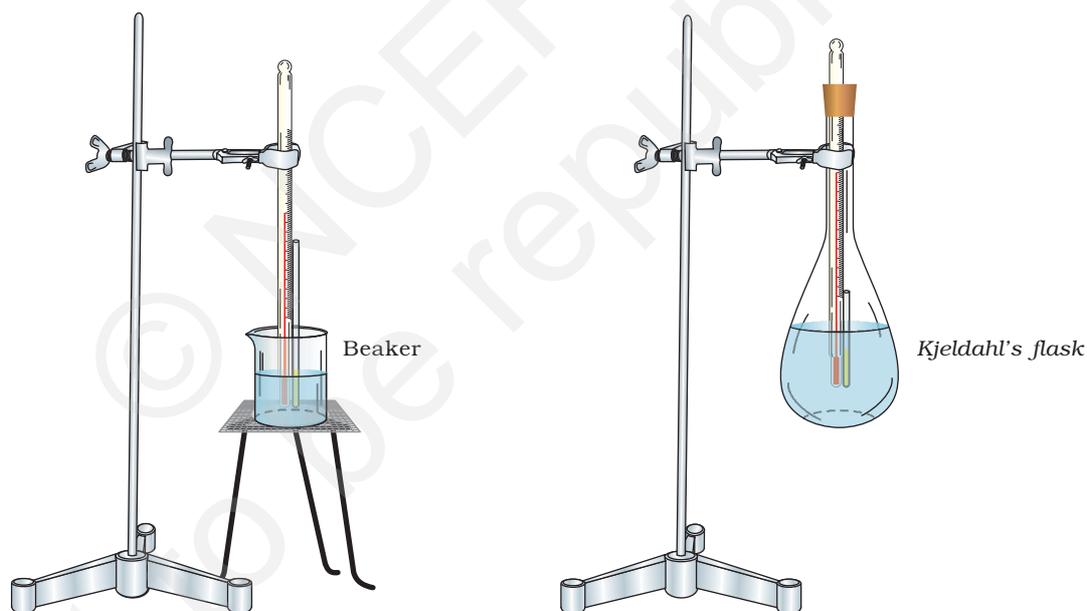


Fig. 3.1 : (b) Different apparatus used for determining melting point

This temperature is the melting point of the solid. If you have taken Kjeldahl flask, heat it by revolving the flame around the bottom of the flask to ensure uniform heating. For this, hold the burner in your hand and also keep a sand bath below the flask while heating. It will prevent spilling of acid in case of accident. Repeat the experiment with other solids.

Precautions

- (a) Keep the lower end of the capillary tube and the thermometer at the same level.
- (b) Capillary tube should not be very thick.
- (c) Packing of the powder should be uniform without any big air gaps in between the solid particles.
- (d) Thiele's tube should be heated at the side arm by using a low flame.
- (e) The cork of the Thiele's tube or Kjeldahl flask holding the thermometer should have a side groove so that vapours can escape through it during the process of heating to prevent bursting of the tube or flask.
- (f) Never fill the bulb of Kjeldahl flask's more than half.

Note : Paraffin can be safely heated upto 220°C. Therefore for determination of melting point of a substance possessing melting point higher than this, conc. H_2SO_4 may be used which can be heated upto 280°C. Sulphuric acid has been suggested for use but is not recommended. Silicone oils are most satisfactory liquids and can be used in place of sulphuric acid.



Discussion Questions

- (i) Why do pure solids possess sharp melting point?
- (ii) What is the effect of impurities on the melting point of a solid?
- (iii) Why is the melting point of benzamide more than acetamide?
- (iv) Can any other liquid be used in place of liquid paraffin to determine the melting point?
- (v) Can we heat the capillary directly for the determination of melting point?
- (vi) Why is liquid paraffin filled in the Thiele's tube/Kjeldahl's flask?
- (vii) Why is Thiele's tube heated at the side arm?

EXPERIMENT 3.3

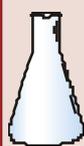
Aim

Determination of boiling point of a liquid organic compound.

Theory

The boiling point of a liquid is the temperature at which vapour pressure of the liquid becomes equal to the atmospheric pressure, which the surface of the liquid experiences. At 1.013 bar atmospheric pressure the boiling point of the liquid is termed as normal boiling point. Different liquids have different boiling point. The difference in the boiling points of liquids is essentially due to the difference in the intermolecular forces operating between the molecules of the liquid.

Material Required



- Thiele's tube/Kjeldahl's flask : One
- Thermometer 110°C or 360°C : One
- Iron stand with clamp : One
- Ignition tube : One
- Capillary tube : One



- Organic liquid : 1 mL
- Liquid paraffin/
Conc. H_2SO_4 : As per need

Concentrated H_2SO_4



Procedure

- (i) Fill Thiele's tube with the liquid paraffin so that it crosses the bent portion of the Thiele's tube.
- (ii) Take 1-2 drops of the given liquid in an ignition tube and tie the ignition tube with the thermometer with a rubber band as shown in Fig. 3.2. Note that the lower end of the ignition tube and the thermometer bulb are at the same level.
- (iii) Seal one end of the capillary tube of approximately 8 cm length by heating in the flame.
- (iv) Place the capillary tube with its open end dipped in the liquid present in the ignition tube.
- (v) Heat the side arm of Thiele's tube with a low flame.
- (vi) Observe the escape of bubbles at the lower end of the capillary dipped in the liquid organic compound. Note the temperature at which bubbles start coming briskly and continuously. This temperature is the boiling point of the liquid.

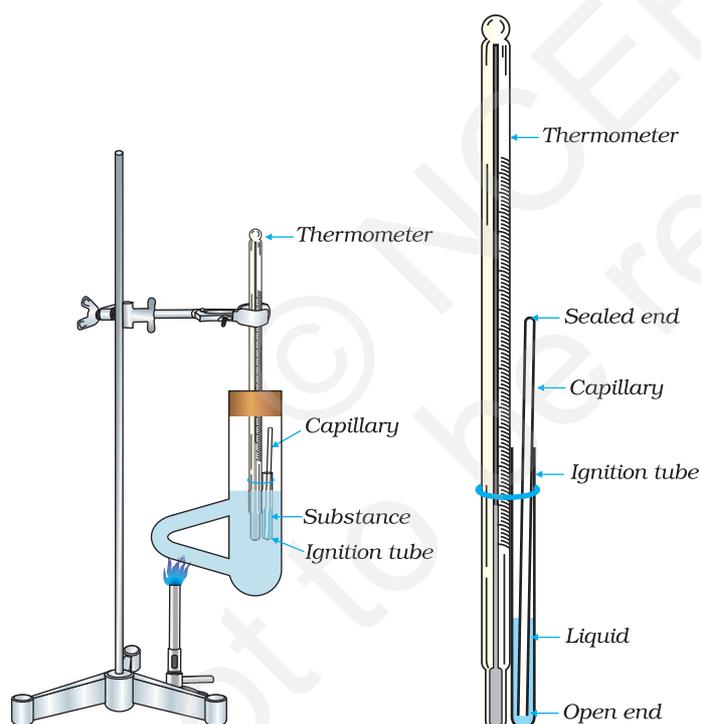


Fig. 3.2: Determination of boiling point

Note : For determination of boiling point of high boiling liquids, paraffin cannot be used as heating medium.

Precautions

- (a) Record the temperature as the boiling point at which brisk and continuous evolution of the bubbles starts from the lower end of the capillary dipped in the liquid organic compound.
- (b) Keep the lower end of the ignition tube and the thermometer bulb at the same level.
- (c) Heat the side arm of the Thiele's tube gently.
- (d) Boiling point of the liquid filled in Thiele's tube should be 50-60°C higher than that of the liquid, of which boiling point is to be determined.



Discussion Questions

- (i) Suggest a suitable liquid, which can be filled in the Thiele's tube for the determination of the boiling point of carbon tetrachloride?
- (ii) In place of liquid paraffin, can any other liquid be used for the purpose of determination of boiling point?
- (iii) Suppose boiling point of a liquid in Delhi is 100°C. At hill station, will it be the same or different? Give reasons.
- (iv) Why is food cooked more quickly in a pressure cooker?
- (v) How would the boiling point of water vary with the addition of equimolar quantities of urea, potassium chloride and potassium sulphate?
- (vi) Why do different isomers of alcohol represented by the formula $C_4H_{10}O$ differ in their boiling points?

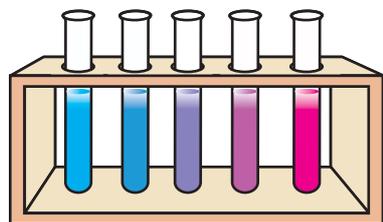
Do you know?

Learning technique of crystallisation is not only important from the point of view of purification of compounds but also from the point of developing large single crystals; because studies on single crystals have shown them to have many optical and electrical properties of great use. For example, slices from large crystals of silicon containing traces of certain impurities are used in making solar batteries used for the operation of instruments in satellites. Frequency control in radar, television and radio is done by making use of slices of some crystals. Also, use of crystals of some compounds is made in microphones and earphones. You can now realize how important it is for chemist to learn these techniques.

UNIT - 4

CHEMICAL EQUILIBRIUM

(IONIC EQUILIBRIUM IN SOLUTION)



CHEMICAL reactions can be classified into two categories; namely reversible and irreversible reactions. Reversible reactions take place in the same reaction vessel and can proceed in the forward and backward direction simultaneously under the same conditions of temperature and pressure. Further in the case of reversible reactions a state is reached, when the rate of the forward reaction becomes equal to the rate of the reverse reaction and it appears as though the reaction has come to a stand still. This state is referred to as the state of **dynamic equilibrium**. Consider the following simple reversible reaction at the given temperature, T .



According to the law of mass action, rate of forward reaction, r_1 , will be directly proportional to the product of concentrations of A and B and the rate of backward reaction, r_2 , will be directly proportional to the products of concentrations of C and D.

$$\text{Thus, } r_1 = k_1[A][B] \text{ and } r_2 = k_2[C][D]$$

where k_1 and k_2 are the rate constants for the forward and the backward reactions respectively and [A], [B], [C] and [D] are the molar concentrations of A, B, C and D respectively.

At equilibrium, r_1 will be equal to r_2

$$k_1[A][B] = k_2[C][D]$$

$$\Rightarrow \frac{k_1}{k_2} = \frac{[C][D]}{[A][B]}$$

Putting $\frac{k_1}{k_2} = K_c$ we have

$$K_c = \frac{[C][D]}{[A][B]}$$

K_c is called equilibrium constant. Its value is independent of initial concentration of reactants and is a function of temperature but remains constant at a constant temperature. At a given temperature, if the concentration of any one of the reactants or products is changed, then equilibrium is disturbed and according to Le Chatelier principle, reaction proceeds in that direction which counteracts the change in concentration, so as to maintain the equilibrium.

The state of equilibrium in any reaction is recognised by the constancy of an observable property (macroscopic property) like colour intensity of the solution. In this unit we will study about the shift in equilibrium in various reactions.

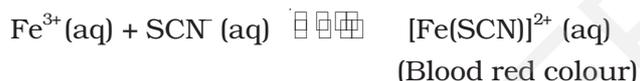
EXPERIMENT* 4.1

Aim

Study of shift in equilibrium in the reaction of ferric ions and thiocyanate ions by increasing the concentration of any one of these ions.

Theory

The equilibrium reaction between ferric chloride and potassium thiocyanate is conveniently studied through the change in the intensity of colour of the solution.



The equilibrium constant for the above reaction may be written as:

$$K = \frac{[\text{Fe}(\text{SCN})]^{2+}(\text{aq})}{[\text{Fe}^{3+}(\text{aq})][\text{SCN}^{-}(\text{aq})]}$$

Here K is constant at a constant temperature. Increasing the concentration of either Fe^{3+} ion or thiocyanate ion would result in a corresponding increase in the concentration of $[\text{Fe}(\text{SCN})]^{2+}$ ions. In order to keep the value of K constant, there is a shift in equilibrium, in the forward direction and consequently an increase in the intensity of the blood red colour which is due to $[\text{Fe}(\text{SCN})]^{2+}$. At equilibrium colour intensity remains constant.

Material Required



- Beakers (100 mL) : Two
- Beaker (250 mL) : One
- Boiling tubes : Six
- Burettes : Four
- Glass droppers : Two
- Test tube stand : One
- Glass rod : One



- Ferric chloride : 0.100g
- Potassium thiocyanate : 0.100g

* The very nature of the experiment is purely qualitative. therefore, preparation of solution in terms of molarity has not been stressed.

Ferric chloride

**Hazard Warning**

- Avoid contact with skin and eyes.

Procedure

- Dissolve 0.100 g ferric chloride in 100 mL of water in a beaker and 0.100 g potassium thiocyanate in 100 mL of water in another beaker.
- Mix 20 mL of ferric chloride solution with 20 mL of potassium thiocyanate solution. Blood red colour will be obtained. Fill this solution in a burette.
- Take five boiling tubes of same size and mark them as a, b, c, d and e.
- Add 2.5 mL of blood red solution to each of the boiling tubes from the burette.
- Add 17.5 mL of water to the boiling tube 'a' so that total volume of solution in the boiling tube 'a' is 20 mL. Keep it for reference.
- Now take three burettes and label them as A, B, and C.
- Fill burette A with ferric chloride solution, burette B with potassium thiocyanate solution and burette C with water.
- Add 1.0 mL, 2.0 mL, 3.0 mL and 4.0 mL of ferric chloride solution to boiling tubes b, c, d and e respectively from burette A.
- Now add 16.5 mL, 15.5 mL, 14.5 mL, and 13.5 mL of water to boiling tubes b, c, d and e respectively from burette C so that total volume of solution in each boiling tube is 20 mL.

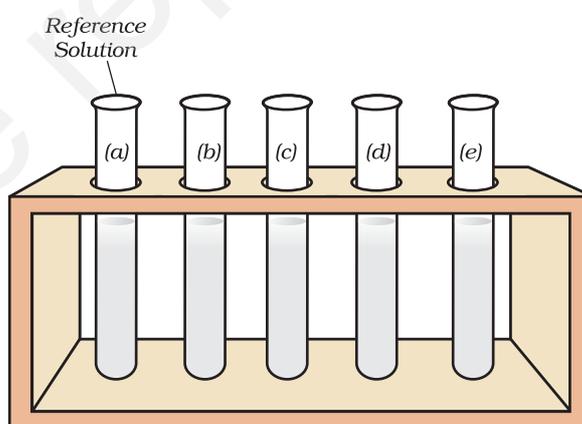


Fig. 4.1 : Set up of experiment for observing equilibrium, each boiling tube contains 20 mL solution

- Note :**
- Colour intensity of the solution will decrease very much on dilution. It will not be deep blood red colour.
 - Total volume in each test tube is 20 mL.
 - Each test tube has 2.5 mL equilibrium mixture.
 - Amount of FeCl_3 is increasing from test tubes 'b' to 'e'.

- (x) Compare the colour intensity of the solution in each boiling tube with the colour intensity of reference solution in boiling tube 'a'.
- (xi) Take another set of four clean boiling tubes. Add 2.5 mL of blood red solution to each of the boiling tubes from the burette. Repeat the experiment by adding 1.0 mL, 2.0 mL, 3.0 mL and 4.0 mL of potassium thiocyanate solution from burette B to the boiling tubes b', c', d', and e' respectively followed by addition of 16.5 mL, 15.5 mL, 14.5 mL and 13.5 mL of water respectively to these test tubes. Again compare the colour intensity of the solution of these test tubes with reference equilibrium solution in boiling tube 'a'.
- (xii) Record your results in tabular form as in Tables 4.1 and 4.2.
- (xiii) You may repeat the observations with different amounts of potassium thiocyanate and ferric chloride solution and compare with the reference solution.

Table 4.1 : Equilibrium shift on increasing the concentration of ferric ions

Boiling Tube	Volume of ferric chloride solution taken in the system in mL	Change in colour intensity as matched with reference solution in boiling tube "a"	Direction of shift in equilibrium
a	Reference solution for matching colour containing 2.5 mL blood red solution + 17.5 mL water (20 mL equilibrium mixture)		Equilibrium position
b	1.0		
c	2.0		
d	3.0		
e	4.0		

Table 4.2 : Equilibrium shift on increasing the concentration of thiocyanate ions

Boiling Tube	Volume of thiocyanate solution taken in the system in mL	Change in colour intensity as matched with reference solution in boiling tube "a"	Direction of shift in equilibrium
a	Reference solution for matching colour containing 2.5 mL blood red solution + 17.5 mL water (20 mL equilibrium mixture)		Equilibrium position
b'	1.0		
c'	2.0		
d'	3.0		
e'	4.0		

Precautions

- Use very dilute solutions of ferric chloride and potassium thiocyanate.
- Compare the colour of the solutions by keeping the boiling tube and the reference test tube side by side.
- To judge the change in colour of the solution in an effective manner, note the colour change in diffused sunlight.
- Use boiling tubes of the same size.



Discussion Questions

- Explain why representing the ionic reaction between ferric and thiocyanate ions as given in the text viz.



is more appropriate in the following form ?



- Does the constancy in colour intensity indicate the dynamic nature of equilibrium? Explain your answer with appropriate reasons.
- What is equilibrium constant and how does it differ from the rate constant?
- It is always advisable to carry out the present experiment with dilute solutions. Why?
- What will be the effect of adding solid potassium chloride to the system at equilibrium? Verify your answer experimentally.
- Why boiling tubes of same size are used in the experiment?

EXPERIMENT 4.2

Aim

Study of the shift in equilibrium in the reaction between $[\text{Co}(\text{H}_2\text{O})_6]^{2+}$ and Cl^{-} ions, by changing the concentration of any one of these ions.

Theory

In the reaction between $[\text{Co}(\text{H}_2\text{O})_6]^{2+}$ and Cl^{-} ions, the following displacement reaction takes place.



This reaction is known as ligand displacement reaction and the equilibrium constant, K , for this is written as follows:

$$K = \frac{[[\text{CoCl}_4]^{2-}]}{[[\text{Co}(\text{H}_2\text{O})_6]^{2+}][\text{Cl}^-]^4}$$

Since the reaction occurs in the aqueous medium, it is believed that concentration of H_2O is almost constant and is included in the value of K itself and is not shown separately in the expression for equilibrium constant.

Now if at equilibrium the concentration of either $[\text{Co}(\text{H}_2\text{O})_6]^{2+}$ ion or Cl^- ions is increased, then this would result in an increase in $[\text{CoCl}_4]^{2-}$ ion concentration thus, maintaining the value of K as constant. In other words we can say that equilibrium will shift in the forward direction and will result in a corresponding change in colour.

Hydrochloric acid



Acetone



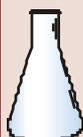
Alcohol



Hazard Warning

- Acetone and alcohol are inflammable, do not let the bottles open when not in use.
- Keep the bottles away from flames.
- Wash your hands after use.
- Wear safety spectacles.

Material Required



- Conical flask (100 mL) : One
- Beakers (100 mL) : Three
- Burettes : Three
- Test tubes : Six
- Test tube stand : One
- Glass rod : One



- Acetone/alcohol : 60 mL
- Concentrated hydrochloric acid : 30 mL
- Cobalt chloride : 0.6000 g

Procedure

- Take 60 mL of acetone in a 100 mL conical flask and dissolve 0.6000 g CoCl_2 in it to get a blue solution.
- Take 5 test tubes of same size and mark them as A, B, C, D and E. Add 3.0 mL of cobalt chloride solution in each of the test tubes from 'A' to 'E' respectively. Now add 1.0 mL, 0.8 mL, 0.6 mL, 0.4 mL and 0.2 mL of acetone respectively in these test tubes. Add 0.2 mL, 0.4 mL, 0.6 mL and 0.8 mL of water to test tubes B, C, D and E respectively, so that the total volume of solution in each of the test tubes is 4.0 mL.
- Note the gradual change in colour of the mixture from blue to pink with an increase in the amount of water.
- Take 10 mL cobalt chloride solution in acetone prepared above and add 5 mL distilled water to it. A solution of pink colour will be obtained.
- Take 1.5 mL of pink solution from step (iv) in five different test tubes labeled as A' B', C', D' and E'. Add 2.0 mL, 1.5 mL, 1.0 mL and 0.5 mL of water to the test tubes labelled

Note :

- In the first set of experiments concentration of chlorocomplex is constant and concentration of water is changing.
- In the second set concentration of aqua complex is constant and concentration of chloride ions is increasing.

from A' to D' and 0.5 mL, 1.0 mL, 1.5 mL, 2.0 mL and 2.5 mL concentrated HCl respectively in the test tubes A' to E' so that total volume of solution in the test tubes is 4 mL.

- (vi) Note the gradual change in colour of pink solution to light blue with increasing amounts of hydrochloric acid. Record your observations in tabular form (Tables 4.3 and 4.4).

Table 4.3 : Shift in equilibrium on adding water

Sl. No.	Test tube	Volume of acetone added in mL	Volume of CoCl_2 solution added in mL	Volume of water added in mL	Colour of mixture
1.	A	1.0	3.0	0.0	
2.	B	0.8	3.0	0.2	
3.	C	0.6	3.0	0.4	
4.	D	0.4	3.0	0.6	
5.	E	0.2	3.0	0.8	

Table 4.4 : Shift in equilibrium on adding Cl^- ions

Sl. No.	Test tube	Volume of conc. HCl added in mL	Volume of aquo complex solution added in mL	Volume of water added in mL	Colour of mixture
1.	A'	0.5	1.5	2.0	
2.	B'	1.0	1.5	1.5	
3.	C'	1.5	1.5	1.0	
4.	D'	2.0	1.5	0.5	
5.	E'	2.5	1.5	0.0	

Precautions

- Take all the precautions of experiment 4.1.
- Use distilled water for the experiment.
- Use burette or graduated pipette for adding water or solutions.

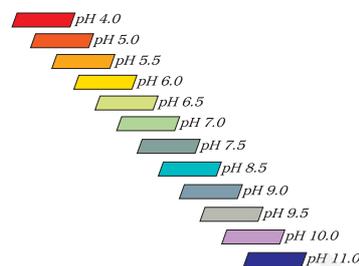


Discussion Questions

- What will be the effect of increasing the temperature of the reaction mixture at equilibrium?
- Can an aqueous solution of sodium chloride replace concentrated HCl? Verify your answer experimentally.
- Why should the total volume of the solution in each test tube be kept same?

UNIT-5

PH AND PH CHANGE IN AQUEOUS SOLUTIONS



You have already performed experiments on dynamic equilibrium between unionised salt and the ions produced by it on dissolving in a solvent. In this unit we will learn about shift in ionic equilibrium between unionised water molecules and H^+ and OH^- ions. The conductivity experiments prove that even pure water ionises to some extent although it has very low conductivity. On this basis it can be concluded that ionic equilibrium exists in pure water also. This ionic equilibrium can be represented as



Since H^+ ion cannot have independent existence in water because of its positive charge and small ionic radius, a better representation of this equilibrium is



This is **self ionisation** of water. Equilibrium constant for this chemical equation can be written as follows:

$$K = \frac{[H_3O^+][OH^-]}{[H_2O]^2}$$

Since water is in large excess, its concentration can be assumed to be constant and combining it with K provides a new constant K_w , which can be written as follows:

$$K_w = [H_3O^+][OH^-]$$

K_w is self ionisation constant of water or simply ionization constant of water. It remains constant at constant temperature. At $25^\circ C$ value of K_w is 1.0×10^{-14} . Thus, it is quite evident that at a given temperature in any aqueous solution, this product i.e. $[H_3O^+][OH^-]$ remains constant whether acidic, alkaline or neutral in nature. If dissolution of a substance shifts the equilibrium in such a way that at equilibrium the hydronium ion concentration is more than hydroxyl ion concentration then the solution is acidic in nature. If dissolution of a substance shifts the equilibrium in such a way that at equilibrium concentration of OH^- ions is greater than the concentration of hydronium ions, then the solution is alkaline in nature. Thus, concentration of hydronium ion in an aqueous solution can provide information about acidic, basic and neutral nature of the solution. The concentration of H_3O^+ ions in a solution is measured in terms of pH which is defined as the negative logarithm of hydronium ion concentration and is given by the following expression.

$$pH = -\log [H_3O^+]$$

At room temperature pH of neutral water is 7. A solution with pH less than this is acidic while the solution with pH greater than this is basic in nature.

EXPERIMENT 5.1

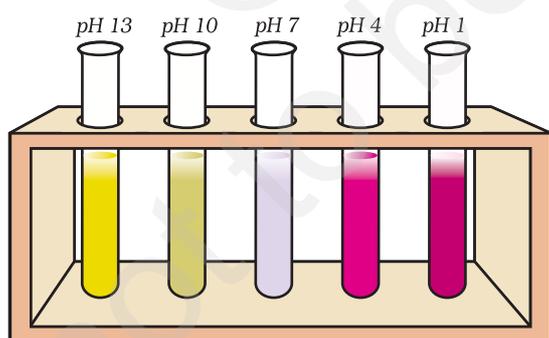
Aim

To determine the pH of some fruit juices.

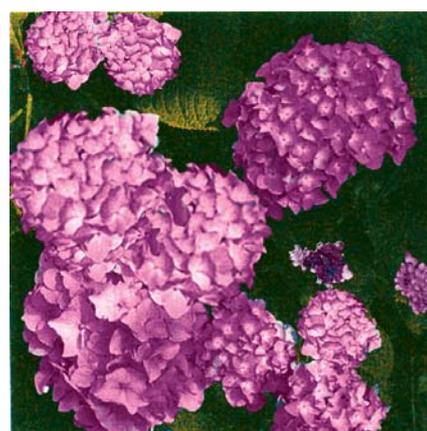
Theory

Several dyes show different colours at different pH. These act as acid-base indicators. Solution of a mixture of dyes can be used to obtain approximate pH value of a solution. A solution of a mixture of dyes can be obtained to measure pH values from zero to 14. It is called universal indicator. Some universal indicators can measure the pH change of even 0.5. In fact, dyes themselves are weak acids or bases. Colour change occurs as a result of change in the structure of dye due to acceptance or release of protons. Different forms of a dye have different colours and hence, colour change is observed when pH of the solution changes. A standard chart for the colour change of the universal indicator with pH is supplied with the indicator paper or solution and the comparison of observed colour change with the chart provides a good estimate of the pH of the solution.

Natural pH Indicators

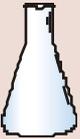


Red cabbage juice has vast pH range. It is a universal indicator of pH in aqueous solution.



The colour of these hydrangeas depends on the pH of the soil in which they grow. If pH of soil is acidic, flowers are blue and in alkaline pH, flowers are pink.

Material Required

	• Beakers (100 mL) : Four		• Fruit juice : Lemon, orange, apple, pineapple
	• Glass droppers : Four		
	• Test tubes : Four		• pH papers/universal indicator solution : As per need
	• pH chart : One		

Procedure

- Procure fresh juices of lemon, orange, apple and pineapple in separate beakers of 100 mL capacity each.
- Transfer nearly 2 mL of the fresh juice (20 drops) with the help of a separate dropper for each juice in four different test tubes marked 1, 2, 3 and 4 respectively.
- Add two drops of the universal indicator in each test tube and mix the content of each test tube thoroughly by shaking.
- Match the colour appearing in each test tube with the standard pH chart.
- Record your observations in Table 5.1.
- Repeat the experiment using pH papers to ascertain the pH of different juices and match the colour in each case with the one obtained with universal indicator.
- Arrange the pH value of the four juices in increasing order.

Table 5.1 : pH value of different fruit juices

Name of the Juice	Colour with universal indicator	pH	Inference
Lemon			
Orange			
Apple			
Pineapple			

Result

Increasing order of pH value of juices is _____.

Precautions

- Add equal number of drops of universal indicator to equal volumes of solutions in each of the test tubes.
- Match the colour of the solution with pH chart carefully.
- Store pH papers at a safe place to avoid contact with acidic and basic reagents kept in the laboratory.
- Use only fresh juice for the experiment.

**Discussion Questions**

- Out of the four juices, which one is least acidic? Explain.
- If we dilute each of the juices, what effect is likely to be observed on the pH values?
- On mixing any two juices, would the pH alter or remain the same? Verify your answer experimentally.
- How can you ascertain the pH of a soft drink?

EXPERIMENT 5.2**Aim**

To observe the variation in pH of acid/base with dilution.

Theory

Hydrogen ion concentration per unit volume decreases on dilution. Therefore, change in pH is expected on dilution of the solution.

Material Required

- Boiling tubes : Eight
- Glass droppers : Four
- Test tubes : As per need



- 0.1 M HCl solution : 20mL
- 0.1 M NaOH solution : 20mL
- 0.05 M H₂SO₄ solution : 20mL
- pH paper/universal indicator : As per need

Procedure

- Take four boiling tubes and mark them as A, B, C and D. (Fig. 5.1).
- Take 2mL of 0.1 M HCl in boiling tube A.

- (iii) Take 2mL of 0.1 M HCl in boiling tube B and add 18 mL water to it and mix thoroughly.
- (iv) Take 5mL of dilute HCl solution from boiling tube B in boiling tube C and add 15mL water to it.

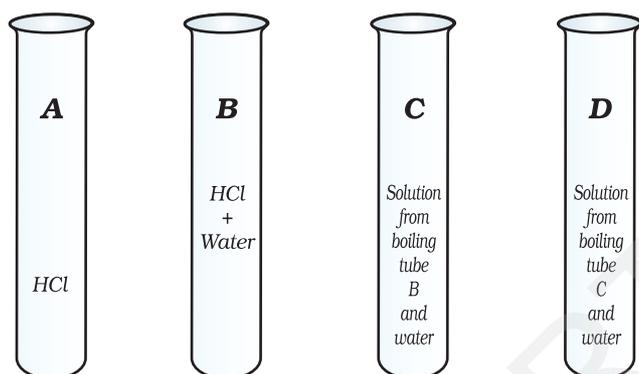


Fig. 5.1 : Set up for experiment 5.2

NaOH 

H₂SO₄ 

HCl 

Hazard Warning

- Never add water to the acid.
- For dilution add acid slowly into water.

- (v) Take 5mL of diluted HCl from boiling tube C in boiling tube D and add 15 mL water to it.
- (vi) Cut a pH paper into small pieces and spread these on a clean glazed tile.
- (vii) Take out some solution from boiling tube A with the help of a dropper and pour one drop on one of the pieces of pH paper kept on the glazed tile. Compare the colour of the pH paper with the standard chart.
- (viii) Similarly test the pH of solutions of boiling tubes B, C, and D respectively and record your results as in Table 5.2.
- (ix) Calculate the hydrogen ion concentration of solution B, C and D.
- (x) Take out 1mL of solution from each boiling tube and transfer in separate test tubes. Add 2 drops of universal indicator to each of these test tubes. Shake the test tubes well and match the colour of these solutions with the standard pH chart to estimate the pH.
- (xi) Similarly observe the change in pH of 0.05 M H₂SO₄ and 0.1M NaOH solution with dilution as detailed in steps (i) to (ix) above.
- (xii) Record your observations in Table 5.2.
- (xiii) Compare the result obtained by using universal indicator paper and that obtained by using universal indicator solution.

Table 5.2 : pH change on dilution

Boiling tube	HCl		H ₂ SO ₄		NaOH	
	Colour	pH	Colour	pH	Colour	pH
A						
B						
C						
D						

Result

- Concentration of solutions of test tube B, C and D are _____.
- Write your conclusion about the variation of pH with dilution.

Precautions

- Add equal number of drops of the universal indicator to equal amounts of solution in each of the boiling tubes.
- Match the colour of the solution with pH chart carefully.

**Discussion Questions**

- What trend is observed in the variation of pH with dilution for acidic as well as for basic solutions?
- How do you explain the results of variation in pH with dilution?
- If any two acidic solutions (say A and C) are mixed, what would happen to the pH of the mixture? Verify your answer experimentally.
- For each acidic solution, whether we use HCl or H₂SO₄, pH is same to a reasonably good extent, even though HCl is 0.1M, and H₂SO₄ is 0.05M. How do you explain this result?
- Will the pH of 0.1M acetic acid be the same as that of 0.1M hydrochloric acid? Verify your result and explain it?

EXPERIMENT 5.3**Aim**

To study the variation in pH by common ion effect in case of weak acids and weak bases.

Theory

It is a known fact that the ionisation in the case of either a weak acid or a weak base is a reversible process. This can be represented as:



The increase in concentration of A^- ions in case (1) and that of B^+ ions in case (2) would shift the equilibrium in the reverse direction thereby decreasing the concentration of H^+ ions and OH^- ions in cases (1) and (2) respectively so as to maintain the constancy of equilibrium constant K . This change either in H^+ ion concentration or OH^- ion concentration brings a change in the pH of the system, which can be judged either with the help of a pH paper or by using a universal indicator solution.

Material Required

	• Beakers (100 mL)	: Four		• Sodium ethanoate	: 2 g
	• Pipettes (25 mL)	: Two		• Ammonium chloride	: 2 g
	• Test tubes	: Four		• Ethanoic acid (1.0 M)	: 50 mL
	• pH chart	: One		• Ammonia solution (1.0 M)	: 50 mL
				• pH paper and universal indicator	: As per need

Procedure

- Take four 100 mL beakers and mark them as A, B, C and D.
- Transfer 25 mL of 1M ethanoic acid in beaker 'A' and 25 mL of (1M) ammonia solution in beaker 'B'.
- Similarly transfer 25 mL of (1 M) ethanoic acid in beaker 'C' and 25 mL of (1.0 M) ammonia solution in beaker 'D'. Now add 2 g sodium ethanoate in beaker 'C' and dissolve it. Likewise add 2 g of ammonium chloride in beaker 'D' and dissolve it by shaking the content of the beaker thoroughly.
- Take approximately 2 mL (20 drops) of the solution from beakers A, B, C and D respectively into test tubes marked as 1, 2, 3 and 4.
- In each of the test tubes add 2 drops of universal indicator solution. Shake the content of the test tubes well and match the colour in each case with the standard pH chart.
- Record your observations as given in Table 5.3.
- Compare pH of the solution in test tubes 1 and 3 and record the change in pH.
- Similarly compare pH of the solution in test tubes 2 and 4 and record the change in pH.

Ammonia solution



Ethanoic acid



Ammonium chloride



Table 5.3 : Comparison of pH of acid/base and its buffer

Sl. No. of Test Tube	Composition of the system	Colour of pH paper	pH
1	CH_3COOH in water		
2	NH_4OH (NH_3 in water)		
3	$\text{CH}_3\text{COOH} + \text{CH}_3\text{COONa}$		
4	$\text{NH}_4\text{OH} + \text{NH}_4\text{Cl}$		

Result

- pH of acetic acid is _____
- pH of buffer of acetic acid and sodium acetate is _____ than acetic acid.
- pH of ammonia solution is _____
- pH of buffer of ammonia solution and ammonium chloride is _____ than the ammonia solution.
- Common ion effect _____ ionization of acid/base.

Precautions

- Try only weak acid/weak base and its salt for the study of the common ion effect.
- Handle the bottle of ammonium hydroxide with care.
- Add equal number of drops of the universal indicator in each of the test tubes.
- Store pH papers at a safe and dry place.

**Discussion Questions**

- The addition of sodium acetate to acetic acid increases the pH whereas, the addition of NH_4Cl to aqueous NH_3 solution (NH_4OH) decreases the pH of the system. How do you explain these observations?
- Suggest suitable replacement for CH_3COONa for system 3 and NH_4Cl for system 4.
- Suggest other pairs of weak acid and its salt and weak base and its salt to carry out the present investigations.
- In salt analysis/mixture analysis, point out the situations where the variation in pH is carried out by common ion effect.
- How do buffer solutions resist change in the pH? Explain with a suitable example.

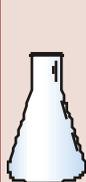
EXPERIMENT 5.4

Aim

To study the change in pH during the titration of a strong acid with a strong base by using universal indicator.

Theory

It is assumed that strong acids and strong bases are completely dissociated in solution. During the process of neutralisation, H^+ ions obtained from the acid combine with the OH^- ions produced by base and form water. Therefore, when a solution of strong acid is added to a solution of strong base or vice versa, the pH of the solution changes. As the titration proceeds, initially there is slow change in the pH but in the vicinity of the equivalence point there is very rapid change in the pH of the solution.

Material Required

- Burette : One
- Beakers (250 mL) : Two
- Conical flask (100 mL) : One
- Dropper : One
- pH chart : One



- Hydrochloric acid (0.1 M) : 25 mL
- Sodium Hydroxide solution (0.1 M) : 50 mL
- Universal indicator : As per requirement

Procedure

- (i) Take 25 mL hydrochloric acid solution (0.1 M) in 100 mL conical flask.
- (ii) Add five drops of universal indicator solution to it.
- (iii) Add (0.1 M) sodium hydroxide solution from the burette as given in Table 5.4.
- (iv) Shake the content of the flask well after each addition of sodium hydroxide solution. Note the colour of the solution in the conical flask each time and find out the pH by comparing its colour with the pH chart.
- (v) Note down your observations in Table 5.4.
- (vi) Plot a graph of pH vs. total volume of NaOH added.

Hydrochloric acid



Sodium hydroxide



Table 5.4 : pH change during the neutralisation of 25 mL of HCl (0.1 M) with NaOH (0.1 M) solution

Sl. No.	Volume of NaOH added in lots (mL)	Total volume of NaOH added to the solution in flask (mL)	pH
1.	0	0	
2.	12.5	12.5	
3.	10.0	22.5	
4.	2.3	24.8	
5.	0.1	24.9	
6.	0.1	25.0	
7.	0.1	25.1	
8.	0.1	25.2	
9.	0.1	25.3	
10.	0.1	25.4	
11.	0.5	25.9	

Precautions

- To get good results perform the reaction with solutions of strong acid and strong base of same concentration.
- Handle the bottle of acid and base with care.
- Use small amount of indicator.

Result

Write down your result on the basis of data.



Discussion Questions

- What trend of pH change will you observe in the neutralisation of strong acid with strong base?
- Do you expect the same trend of pH change for neutralisation of weak acid (acetic acid) with a strong base (sodium hydroxide)?
- In which pH range should the indicator show colour change if the hydrochloric acid is to be neutralised by sodium hydroxide? Give answer after looking at the graph of the experiment.
- Explain how does the study of pH change help in choosing the indicator for neutralisation reaction.

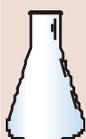
EXPERIMENT 5.5

Aim

To study pH of solutions of sodium chloride, ferric chloride and sodium carbonate.

Theory

Salts of strong acid and strong base form neutral solutions while salts of weak acid/base and strong base/acid are basic and acidic respectively in nature. Salts of weak acid/base with strong base/acid are hydrolysed in water while salts formed by neutralization of strong acid and strong base do not hydrolyse in solution. You have already learnt about this in your chemistry textbook.

Material Required

- Boiling tubes : Three
- Test tubes : Three
- Glass droppers : Three



- pH paper/universal indicator: As per need
- 0.1 M NaCl solution : As per need
- 0.1 M FeCl₃ solution : As per need
- 0.1 M Na₂CO₃ solution : As per need

Procedure

- (i) Take three boiling tubes and mark them as A, B and C.
- (ii) Take 20 mL of 0.1 M solution(s) of NaCl, FeCl₃ and Na₂CO₃ in boiling tubes A, B and C respectively.
- (iii) Cut the pH paper in small pieces and spread the pieces on a clean glazed tile.
- (iv) Test the pH of the solution in boiling tubes A, B and C as in the experiment 5.1.
- (v) Arrange three clean test tubes in a test tube stand.
- (vi) Number the test tubes as 1, 2 and 3.
- (vii) Pour 4 mL solution from boiling tube A in each of the test tubes.
- (viii) Add 5 mL, 10 mL and 15 mL water in the test tubes 1, 2 and 3 respectively.
- (ix) Note the pH of the solutions of test tubes 1, 2 and 3 with the help of pH paper and universal indicator.
- (x) Repeat the experiment with the solutions of boiling tubes B and C.
- (xi) Note your results in tabular form as in Table 5.5.

Table 5.5 : pH of NaCl, FeCl₃ and Na₂CO₃ solutions of different concentrations

Solution	pH of solution		
	Test tube 1	Test tube 2	Test tube 3
NaCl			
FeCl ₃			
Na ₂ CO ₃			

Result

Write down the result on the basis of your observations.

Precautions

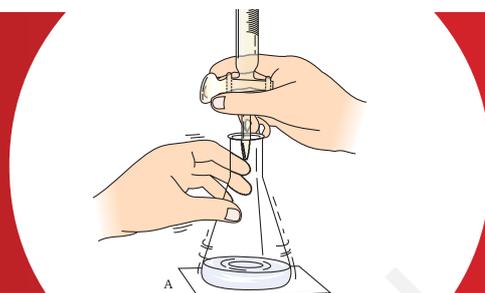
- Use freshly prepared solutions.
- Do not leave the bottles open after taking out salt.
- Use separate and clean test tube for each solution.
- Store the pH paper at a safe and dry place.

**Discussion Questions**

- Why are FeCl₃ and Na₂CO₃ solutions not neutral?
- Why are the salts of strong acid and strong base not hydrolysed? Explain.
- How is the phenomenon of hydrolysis useful in salt analysis?
- What is the effect of dilution on pH of salt solution? Verify and explain your results.

UNIT-6

TITRIMETRIC ANALYSIS



YOU are already aware that a substance is analysed to establish its qualitative and quantitative chemical composition. Thus, chemical analysis can be categorised as qualitative analysis and quantitative analysis. In this unit you will learn about determination of the amount of substance in solution. Depending upon the method adopted for determining the amount of chemical substances in solution, there are two methods of analysis namely, titrimetric analysis and gravimetric analysis. In titrimetric analysis measurement of only volumes is involved while in gravimetric analysis measurement of volumes as well as mass is involved.

Titrimetric analysis involves determination of the volume of a solution of accurately known concentration, which is required to react quantitatively with the measured volume of the solution of a substance, concentration of which is to be determined. The solution of accurately known concentration is called **standard solution**. The mass of the substance dissolved in the solution of unknown concentration is calculated from the volume of the standard solution used, the chemical equation and the relative molecular masses of the reacting compounds. The reagent of known concentration is called **titrant** and the substance being titrated is termed as **titrand**.

To carry out titrimetric analysis, standard solution is usually added from the long graduated tube called burette. The process of adding the standard solution to the solution of unknown concentration until the reaction is just complete is called **titration**. The point at which reaction is completed is called **equivalence point** or the **theoretical or stoichiometric end point**. It is not possible all the time to take standard solution in the burette. You will come to know about it later in this unit in the titration of sodium hydroxide with oxalic acid.

6.1 DETECTION OF END POINT

The end point is detected either by some physical change produced in the reaction mixture itself or by the addition of an auxiliary reagent, known as **indicator**; alternatively some other physical measurement may be used. At the completion of the reaction, the indicator shows a visible change e.g. (colour change or turbidity) in the solution being titrated. In an ideal titration, the visible end point coincides with the stoichiometric or theoretical end point; but in practice usually some small difference occurs. This represents **titration error**.

Indicator and the experimental conditions selected should be such that the difference between the visible end point and the theoretical end point is minimum.

6.2 REQUIREMENT FOR A REACTION IN THE TITRIMETRIC ANALYSIS

- (i) The substance of which amount is to be determined by titrimetric analysis must react completely and rapidly with the other reagent in stoichiometric proportion.
- (ii) The reaction should be fast and there must be alteration in physical or chemical property of the solution at the equivalence point, which can be detected by an indicator, or by measuring the potential difference or current etc.

6.3 ACIDIMETRY AND ALKALIMETRY

Titrimetric analysis can be carried out for various types of reactions. In this unit you will learn only about neutralisation reactions. These involve titrations of acids and bases. Standard solutions of acids (acidimetry) and bases (alkalimetry) are used in these titrations. In quantitative estimation through titrimetric analysis, concentration of solution is expressed in terms of molarity. It is number of moles of solute dissolved in 1 litre of solution.

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

Standard Solution

A solution of exactly known concentration is called standard solution. Any substance, which is stable at room temperature and does not react with solvent in which it is dissolved, can be directly weighed to prepare its standard solution. Description and preparation of these solutions is given below:

Primary and secondary standards

A **primary standard** is a compound of sufficient purity in which total amount of impurities does not exceed 0.01-0.02%. The standard solution can be prepared by direct weighing of a sample of primary standard followed by its dissolution in water (or solvent) to obtain a definite volume of solution. The substance to be used as a primary standard should also satisfy the following requirements:

1. It must be easily available in pure and dry form.
2. It should not undergo change in air i.e. it should not be hygroscopic, oxidised by air or affected by gases such as carbon dioxide present in the atmosphere or lose water of crystallization, so that it can be stored safely.
3. It should be easy to detect the impurities present in it.
4. It should have high **relative molecular mass** so that weighing errors are negligible.
5. Its reaction with another substance should be instantaneous and stoichiometric.
6. The substance should be readily soluble in water.

It is difficult to obtain an ideal primary standard. Therefore, substances having characteristics nearer to the primary standards are usually employed.

Unstable hydrated salts, as a rule, should not be used as primary standards. However, sodium carbonate, sodium tetraborate, potassium hydrogenphthalate, oxalic acid, ferrous ammonium sulphate etc. can be used as primary standards because of their sufficient stabilities.

A solution of secondary standard is the one which may be used for standardization after finding out its exact concentration by titration against a standard solution of primary standard.

A secondary standard cannot be used for preparing standard solution by direct weighing. Sodium hydroxide and potassium permanganate are examples of secondary standards.

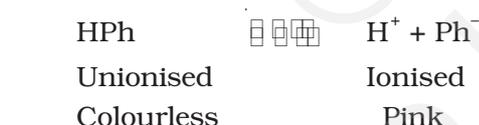
Before starting titrimetric analysis, you should be familiar with some techniques such as, weighing by using chemical balance, preparing standard solution, measuring volume by using burette and pipette.

6.4 INDICATORS IN ACID BASE TITRATION

Acid base indicators are sensitive to pH change. For most acid base titrations, it is possible to select indicators which exhibit colour change at pH close to the equivalence point. We will discuss here about only two indicators – phenolphthalein and methyl orange.

Phenolphthalein

Phenolphthalein is a weak acid, therefore it does not dissociate in the acidic medium and remains in the unionised form, which is colourless.



Ionised and unionised forms of phenolphthalein are given below :

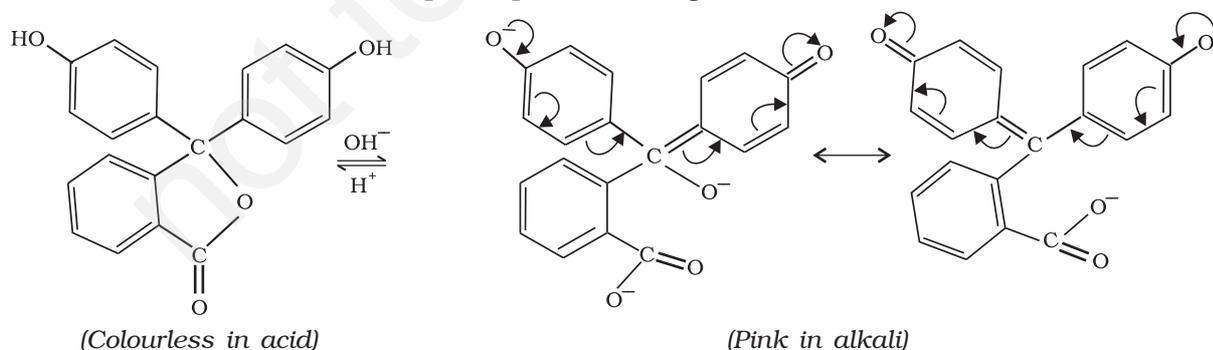
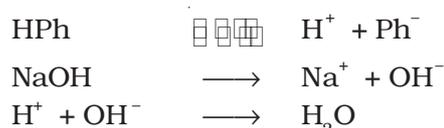


Fig. 6.1 : Phenolphthalein in acidic and basic medium

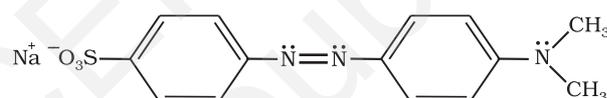
In the acidic medium, equilibrium lies to the left. In the alkaline medium, the ionisation of phenolphthalein increases considerably due to the constant removal of H^+ ions released from HPh by the OH^- ions from the alkali. So the concentration of Ph^- ion increases in the solution, which imparts pink colour to the solution.



For a weak acid vs strong alkali titration, phenolphthalein is the most suitable indicator. This is so because the last drop of added alkali brings the pH of the solution in the range in which phenolphthalein shows sharp colour change.

Methyl orange

Methyl orange is a weak base and is yellow in colour in the unionised form. Sodium salt of methyl orange is represented as follows:



The anion formed from the indicator is an active species, which on accepting a proton (i.e. acting as Bronsted Lowry base) changes from the benzenoid form to the quinonoid form. The quinonoid form is deeper in colour and thus is responsible for the colour change at the end point. This is illustrated in the following manner:

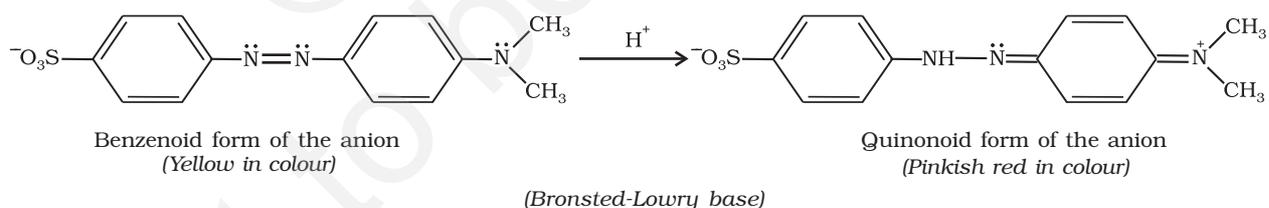


Fig. 6.2 : Structures of Methyl orange

Choice of Indicator

In the titration of strong acid and a weak base, methyl orange is chosen as indicator. When titration between strong base and weak acid is to be performed then phenolphthalein is a good indicator. In this case alkali is dropped from the burette and acid is taken in the titration flask. Colour of the solution taken in the titration flask

changes from colourless to pink. This change of colour is easily perceptible to the human eye. If we take alkali in the titration flask, the colour change will be from pink to colourless and accuracy in noting the colour change may be less. In the titration of strong acid versus strong base any one of the above indicators can be used. For the titration of weak acid vs weak base no indicator is available.

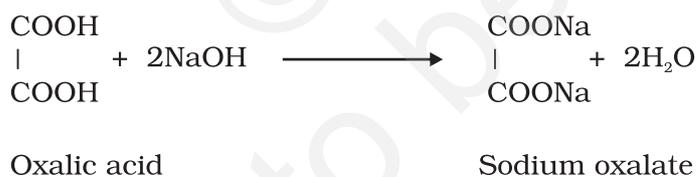
EXPERIMENT 6.1

Aim

Determination of the concentration (strength) of a given sodium hydroxide solution by titrating it against a standard solution of oxalic acid.

Theory

In the titration of a strong acid with a strong base, the amount of acid and base becomes chemically equivalent at the end point and the chemical reaction is called neutralization reaction. Near the end point there is a sudden change in the pH of the solution. If after end point even a small amount of base/acid is added the solution would become slightly alkaline or acidic respectively. In the titration between oxalic acid (weak acid) and sodium hydroxide (strong base), following reaction takes place:

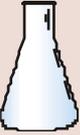


In this titration phenolphthalein (HPh) is used as an indicator. The concentration of unknown solution is calculated in g/L. Molarity of the solution can be calculated by using the formula

$$a_1 M_1 V_1 = a_2 M_2 V_2 \quad \dots(4)$$

where a_1 , M_1 , V_1 are respectively basicity, molarity and volume of acid used and a_2 , M_2 and V_2 are acidity, molarity and volume respectively of base used in the titration.

Material Required

	• Burette (50 mL)	:	One		• Oxalic acid	:	As per need
	• Pipette (10 mL)	:	One		• Sodium hydroxide solution	:	As per need
	• Conical flask (100 mL)	:	One		• Phenolphthalein indicator	:	As per need
	• Burette stand	:	One				
	• Funnel	:	One				
	• White glazed tile	:	One				
	• Measuring flask (100 mL)	:	One				

Procedure

Oxalic acid 

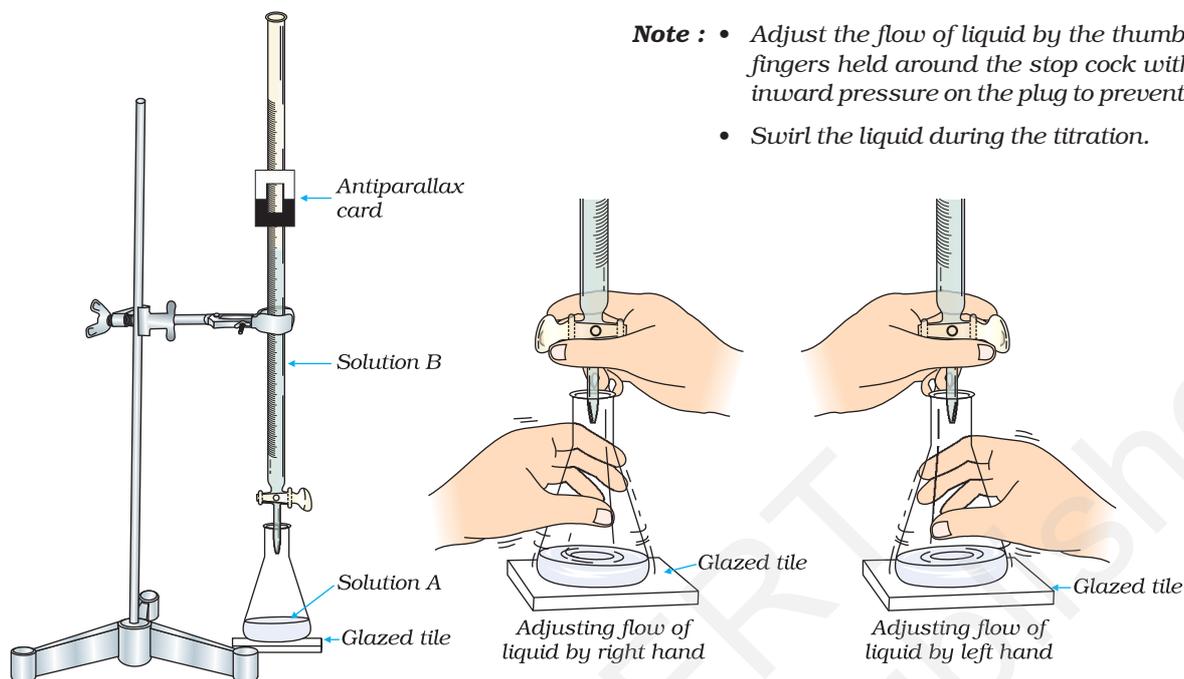
Sodium Hydroxide 

(A) Preparation of 0.1M Standard Solution of Oxalic Acid

Follow the procedure as described in Experiment No. 2.1.

(B) Titration of Sodium Hydroxide and Oxalic Acid Solution

- (i) Clean the burette thoroughly, wash it with distilled water and finally rinse it with sodium hydroxide solution. (Always rinse the burette (Fig. 2.17) with the solution, which is to be taken in it). Clamp the burette vertically in a burette stand.
- (ii) Fill sodium hydroxide solution into the burette through a funnel above the zero mark.
- (iii) **Remove the air gap, if any, from the nozzle** of the burette by running the solution forcefully from the burette nozzle.
- (iv) **Remove the funnel before noting initial reading of the burette.** Also while noting the reading, see that no drop of the liquid is hanging at the nozzle of the burette.
- (v) Note the initial reading by keeping the eye exactly at the same level as the meniscus of the solution.
- (vi) Pipette out 10 mL of oxalic acid solution in a washed and dried conical flask. Always wash the pipette with water and rinse (Fig. 2.21) with the liquid to be measured before pipetting out the liquid.
- (vii) Add 1-2 drops of phenolphthalein indicator to the conical flask. Place the flask over the glazed tile as shown in Fig. 6.3 Titrate the acid with sodium hydroxide solution till a very faint permanent pink colour is obtained. Add sodium hydroxide solution in small amounts initially and then dropwise.



- Note :**
- Adjust the flow of liquid by the thumb and two fingers held around the stop cock with a slight inward pressure on the plug to prevent leakage.
 - Swirl the liquid during the titration.

Fig. 6.3 : Titrating the solution

- (viii) Read the lower meniscus of the solution in the burette again and record it as final reading.
- (ix) Repeat the procedure until three concordant readings are obtained. Record your readings as in Table 6.1.

Table 6.1 : Titration of sodium hydroxide vs oxalic acid solution

Sl. No.	Volume of oxalic acid solution taken in conical flask each time V_1 mL	Burette readings		Volume of sodium hydroxide solution used V_2 mL = (y-x) mL	Concordant reading in mL
		Initial reading (x)	Final reading (y)		

Calculations

Molarity of NaOH solution can be calculated by using the equation:

Oxalic acid Sodium hydroxide

$$a_1 M_1 V_1 = a_2 M_2 V_2$$

where, M_1 and V_1 are the molarity and volume of the oxalic acid solution.

M_2 and V_2 are the molarity and volume of the sodium hydroxide solution.

a_1 and a_2 are respectively the basicity of oxalic acid and acidity of sodium hydroxide. In this case $a_1 = 2$ and $a_2 = 1$.

Also, Molar mass of oxalic acid, $(\text{COOH})_2 \cdot 2\text{H}_2\text{O} = 126 \text{ g mol}^{-1}$ and Molar mass of sodium hydroxide $(\text{NaOH}) = 40 \text{ g mol}^{-1}$

Calculate the concentration of sodium hydroxide solution in g/L by using the equation given below.

Concentration (strength) in g/L = Molarity \times Molar mass

Result

Concentration of NaOH solution is _____ g/L.

Precautions

- Always rinse the burette with the solution, which is to be taken in it.
- Remove the air gap if any, from the burette before titrating the solution. Make sure that the nozzle of burette is also filled.
- Never forget to remove the funnel from the burette before noting the readings of the burette and ensure that no drop is hanging from the nozzle of the burette.
- Always read the lower meniscus for all transparent solutions and upper meniscus for coloured solutions.
- To note the burette readings place the eye exactly at the level of the meniscus.
- Never hold the pipette at the bulb.
- Never use the pipette and burette with a broken nozzle.
- Never suck a strong acid or an alkali with the pipette.
- Always keep the lower end of the pipette dipped in the liquid while sucking the liquid.
- Do not blow out the last drop of the solution from the jet end of the pipette into the flask.
- The concentration (strength) of the solution must be calculated up to the fourth place of decimal.



Discussion Questions

- (i) Why are the burette and the pipette rinsed with the solution with which these are filled?
- (ii) What is an indicator? Which indicator is used in the titration of oxalic acid vs sodium hydroxide? Can the titration be performed by using some other indicator?
- (iii) Why should one read the lower meniscus in the case of colourless and transparent solutions and the upper meniscus for solutions of dark colour?
- (iv) Explain the term 'end point'?
- (v) What do you mean by 1.0 M solution?
- (vi) Why should the last drop of the solution not be blown out of a pipette?
- (vii) Explain the term basicity of an acid and acidity of a base?
- (viii) For titrating NaOH vs HCl, phenolphthalein and methyl orange, both are suitable indicators. Why?
- (ix) What is meant by the term, 'concordant readings'?
- (x) Can one take oxalic acid solution in the burette and sodium hydroxide solution in the titration flask? Point out the limitations of doing so if any.

KNOW THIS ALSO

The complete neutralisation is possible only when the amount of alkali is in equivalent proportion to the amount of acid. Therefore, at the end point equivalent mass of acid dissolved in volume V_1 of solvent should be equal to the equivalent mass of the base dissolved in Volume V_2 of solvent. If N_1 and N_2 are equivalent masses of acid and base respectively dissolved in per litre of solution then $N_1 V_1 = N_2 V_2$... (i)

Equivalent mass of acids and bases is given by the following expression:

$$\text{Equivalent mass of acid} = \frac{\text{molar mass}}{\text{basicity}} \quad \dots \text{ (ii)}$$

$$\text{Equivalent mass of base} = \frac{\text{molar mass}}{\text{acidity}} \quad \dots \text{ (iii)}$$

The number of gram equivalent mass of solute dissolved in one litre of the solution is called the normality of the solution. For acids and bases

$$\text{Normality (N)} = \frac{\text{Number of gram equivalent mass}}{\text{Volume of solution in litre (V)}} = \frac{w / \text{Equivalent mass}}{\text{Volume of solution in litre (V)}} \quad \dots \text{ (iv)}$$

Where w = mass of substance in grams

Relationship between Normality and Molarity

From definition of normality (N) [(equation (iv))]

$$\text{Equivalent mass} = \frac{w}{N \times V} \quad \dots \text{ (v)}$$

If acidity or basicity is 'a', then by definition of equivalent mass

$$\text{Equivalent mass} = \frac{\text{Molar mass}}{a} \quad \dots \text{(vi)}$$

From equations (v) and (vi) we can write

$$\frac{w}{N \times V} = \frac{\text{Molar mass}}{a}$$

Or
$$N = \frac{a(w/\text{Molar mass})}{V}$$

But $\frac{w/\text{Molar mass}}{V}$ is the molarity (M)

Therefore, $N = aM \quad \dots \text{(vii)}$

Equation (vii) is the expression for the relationship between normality and molarity.

By using equation (vii) in equation (i) we have $a_1 M_1 V_1 = a_2 M_2 V_2 \quad \dots \text{(viii)}$

Where a_1 and a_2 are basicity and acidity of acid and base respectively and M_1 and M_2 are the molar masses of acid and base respectively. Thus we see that equation (i) can also be used to calculate the strength of the solution. Equation (viii) can be used for making the solution by dilution. For solutions of the same substance $a_1 = a_2$. Therefore for using equation (viii) for dilution of solution –

$$M_1 V_1 = M_2 V_2 \quad \dots \text{(ix)}$$

Therefore, to obtain V_2 volume of a solution of molarity M_2 from a solution of molarity M_1 , the volume V_1 of the solution of molarity M_1 which is required for dilution, can be calculated from equation (ix). $(V_2 - V_1)$ volume of solvent will be required to be added to V_1 volume of the solution of molarity M_1 .

EXPERIMENT 6.2

Aim

Preparation of 0.1 M standard solution of sodium carbonate.

Theory

Sodium carbonate has characteristics nearer to the primary standards therefore its standard solution can be made by direct weighing.

To prepare 0.1 M Na_2CO_3 solution, 10.6000g of sodium carbonate should be dissolved per litre of the solution (Molar mass of sodium carbonate is 106 g mol^{-1}).

Therefore, to prepare 100 mL of 0.1M Na_2CO_3 solution 1.0600 g of sodium carbonate is dissolved in minimum quantity of water and the solution is diluted to exactly 100 mL by adding water to it.

Material Required



- Measuring flask (100 mL) : One
- Watch glass : One
- Funnel : One
- Wash bottle : One



- Sodium carbonate : As per need

Procedure

Follow the same procedure as in Experiment 2.1.

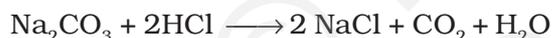
EXPERIMENT 6.3

Aim

Determination of the strength of a given solution of dilute hydrochloric acid by titrating it against a standard solution of sodium carbonate.

Theory

The strength of hydrochloric acid is determined by titrating it against a standard solution of sodium carbonate. The following reaction takes place:



In this titration, methyl orange, a weak base (yellow in the unionised form) is used as an indicator.

In this experiment also, the titration follows the usual course, i.e., the proton furnished by the addition of the acid first neutralises sodium carbonate solution. When the entire sodium carbonate solution is neutralised, the last drop of the acid added from the burette produces the pinkish red colour change, which is the end point.

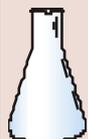
The concentration (strength) of the unknown solution is calculated in g/L. It is calculated from the molarity of the solution.

Here, the molarity equation is written as

$$\begin{array}{cc} \text{Base} & \text{Acid} \\ a_1 M_1 V_1 & = a_2 M_2 V_2 \end{array}$$

where, a_1 and a_2 are the acidity and basicity of the alkali and the acid respectively. M_1 and M_2 are the molarities, V_1 and V_2 are the volumes of the base and acid respectively used to neutralise each other.

Material Required



- Burette (50 mL) : One
- Pipette (10 mL) : One
- Conical flask (100 mL) : One
- Burette stand : One
- Funnel : One
- Glazed tile (white) : One
- Measuring flask (100 mL) : One



- Hydrochloric acid : As per need
- Sodium carbonate : As per need
- Methyl orange solution : As per need

Procedure

(A) Preparation of 0.1 M standard solution of sodium carbonate

Follow the procedure as described in Experiment 2.1.

(B) Titration of hydrochloric acid and standard sodium carbonate solution.

Follow the procedure as given in the Experiment 6.1.

In this case, hydrochloric acid is taken in the burette and sodium carbonate solution in the conical flask. Methyl orange is used as an indicator. The colour change at the end point will be from yellow to pinkish-red. Record your observations in Table 6.2.

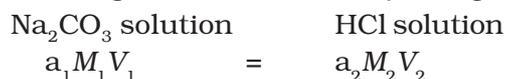
Hydrochloric acid 

Table 6.2 : Titration of Hydrochloric acid with standard sodium carbonate solution

Sl. No.	Volume V_1 of Na_2CO_3 solution taken in the conical flask each time in mL	Burette readings		Volume of HCl solution used V_2 mL = (y-x) mL	Concordant reading in mL
		Initial reading (x)	Final reading (y)		

Calculations

Calculate the strength of HCl solution by using the equation



where M_1 and V_1 are the molarity and volume of sodium carbonate solution respectively and a_1 is the number of moles of OH^- (aq) ions supplied by one mole of the base (i.e. the acidity of the Na_2CO_3 solution).

$$\therefore a_1 = 2$$

M_2 and V_2 are the molarity and volume respectively of hydrochloric acid solution.

a_2 is the number of moles of H^+ (aq) ions supplied by one mole of the acid (i.e. the basicity of HCl).

$$\therefore a_2 = 1$$

Molar mass of $\text{Na}_2\text{CO}_3 = 106 \text{ g mol}^{-1}$, Molar mass of $\text{HCl} = 36.5 \text{ g mol}^{-1}$,

$$\therefore \text{Concentration (Strength) of HCl solution in g/L} = \text{Molarity} \times \text{Molar mass}$$

Result

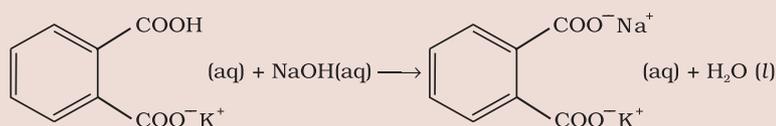
The concentration (strength) of the given HCl solution is _____ g/L.

Precautions

- Care should be taken while handling the acid and base.
- Always rinse the burette and the pipette with the solution which is to be taken in them.
- Remove the air gap if any, from the burette before titration.
- Never forget to remove the funnel from the burette before noting the initial reading of the burette and ensure that no drop is hanging from the nozzle.
- Always read the lower meniscus for all transparent solutions and upper meniscus for the coloured solutions.
- Never use burette and pipette with a broken nozzle.
- Never suck a strong acid or an alkali with the pipette, use pipette bulb.
- Always keep the lower end of the pipette dipped in the liquid while sucking the liquid.
- While transferring the solution to the flask, do not blow out the last drop of the solution from the jet of the pipette.
- The strength of the solution must be calculated up to the fourth decimal place.

KNOW THIS ALSO

Potassium hydrogenphthalate is primary standard for standardisation of sodium hydroxide solution. Formula of potassium hydrogen phthalate is $C_6H_5O_4K$. It behaves as a monobasic acid. Sodium hydroxide reacts with potassium hydrogenphthalate according to the following equation.



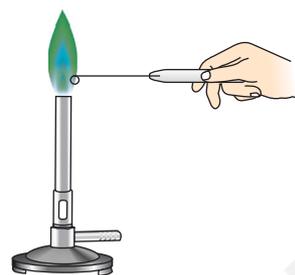
Phenolphthalein can be used as an indicator in this titration.

**Discussion Questions**

- (i) Which indicator is used in the titration of sodium carbonate against hydrochloric acid and what is the colour change at the end point?
- (ii) How will you prepare 250 mL of 0.05 M solution of sodium carbonate?
- (iii) Though sodium carbonate is a salt yet its aqueous solution is weakly alkaline in nature. Explain why?
- (iv) How can you determine the acidity of sodium carbonate solution?
- (v) Why is methyl orange not an Arrhenius base?
- (vi) How can you titrate a solution of the mixture of Na_2CO_3 and NaHCO_3 against HCl?
- (vii) What is the difference between an end point and an equivalence point?
- (viii) Can you directly prepare standard solution of HCl, HNO_3 and H_2SO_4 ?

UNIT-7

SYSTEMATIC QUALITATIVE ANALYSIS



ANALYSIS always does not mean breaking of substance into its ultimate constituents. Finding out the nature of substance and identity of its constituents is also analysis and is known as qualitative analysis. Qualitative analysis of inorganic salts means the identification of cations and anions present in the salt or a mixture of salts. Inorganic salts may be obtained by complete or partial neutralisation of acid with base or vice-versa. In the formation of a salt, the part contributed by the acid is called anion and the part contributed by the base is called cation. For example, in the salts CuSO_4 and NaCl , Cu^{2+} and Na^+ ions are cations and SO_4^{2-} and Cl^- ions are anions. Qualitative analysis is carried out on various scales. Amount of substance employed in these is different. In macro analysis, 0.1 to 0.5 g of substance and about 20 mL of solution is used. For semimicro analysis, 0.05 g substance and 1 mL solution is needed while for micro analysis amount required is very small. Qualitative analysis is carried out through the reactions which are easily perceptible to our senses such as sight and smell. Such reactions involve:

- Formation of a precipitate
- Change in colour
- Evolution of gas etc.

Systematic analysis of an inorganic salt involves the following steps:

- Preliminary examination of solid salt and its solution.
- Determination of anions by reactions carried out in solution (wet tests) and confirmatory tests.
- Determination of cations by reactions carried out in solution (wet tests) and confirmatory tests.

Preliminary examination of a salt often furnishes important information, which simplifies further course of analysis. Although these tests are not conclusive but sometimes they give quite important clues for the presence of certain anions or cations. These tests can be performed within 10–15 minutes. These involve noting the general appearance and physical properties, such as colour, smell, solubility etc. of the salt. These are named as dry tests.

Heating of dry salt, blow pipe test, flame tests, borax bead test, sodium carbonate bead test, charcoal cavity test etc. come under dry tests. Some of these tests are given later in this unit.

Solubility of a salt in water and the pH of aqueous solutions give important information about the nature of ions present in the salt. If a solution of the salt is acidic or basic in nature, this means that it is being hydrolysed in water. If the solution is basic in nature then salt may be some carbonate or sulphide etc. If the solution shows acidic nature then it may be an acid salt or salt of weak base and strong acid. In this case it is best to neutralise the solution with sodium carbonate before testing it for anions.

Gases evolved in the preliminary tests with dil. H_2SO_4 /dil. HCl and conc. H_2SO_4 also give good indication about the presence of acid radicals (see Tables 7.1 and 7.3). Preliminary tests should always be performed before starting the confirmatory tests for the ions.

EXPERIMENT 7.1

Aim

To detect one cation and one anion in the given salt from the following ions:

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

Anions - CO_3^{2-} , S^{2-} , SO_3^{2-} , SO_4^{2-} , NO_2^- , NO_3^- , Cl^- , Br^- , I^- , PO_4^{3-} , $\text{C}_2\text{O}_4^{2-}$,
 CH_3COO^- .

(Insoluble salts to be excluded)

Theory

Two basic principles of great use in the analysis are:

- (i) the Solubility product and
- (ii) the Common ion effect

When ionic product of a salt exceeds its solubility product, precipitation takes place. Ionic product of salt is controlled by making use of common ion effect which you have studied in the textbook of chemistry.

Material Required



- Boiling tube : As per need
- Test tubes : As per requirement
- Measuring cylinder : One
- Test tube stand : One
- Test tube holder : One
- Delivery tube : One
- Corks : As per need
- Filter paper : As per need



- Reagents : As per need

SYSTEMATIC ANALYSIS OF ANIONS

Step - I : Preliminary Test with Dilute Sulphuric Acid

In this test the action of dilute sulphuric acid (procedure is given below) on the salt is noted at room temperature and on warming.

Carbonate (CO_3^{2-}), sulphide (S^{2-}), sulphite (SO_3^{2-}), nitrite (NO_2^-) and acetate (CH_3COO^-) react with dilute sulphuric acid to evolve different gases. Study of the characteristics of the gases evolved gives information about the anions. Summary of characteristic properties of gases is given in Table 7.1 below.

Procedure

- (a) Take 0.1 g of the salt in a test tube and add 1–2 mL of dilute sulphuric acid. Observe the change, if any, at room temperature. If no gas is evolved, warm the content of the test tube. If gas is evolved test it by using the apparatus shown in Fig.7.1 and identify the gas evolved (See Table 7.1).

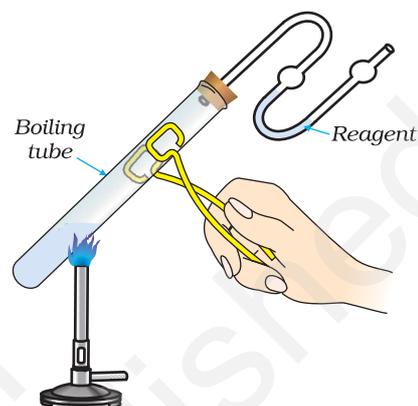


Fig. 7.1 : Testing a Gas

Table 7.1 : Preliminary test with dilute sulphuric acid

Observations	Inference	
	Gas evolved	Possible Anion
A colourless, odourless gas is evolved with brisk effervescence, which turns lime water milky.	CO_2	Carbonate (CO_3^{2-})
Colourless gas with the smell of rotten eggs is evolved which turns lead acetate paper black.	H_2S	Sulphide (S^{2-})
Colourless gas with a pungent smell, like burning sulphur which turns acidified potassium dichromate solution green.	SO_2	Sulphite (SO_3^{2-})
Brown fumes which turn acidified potassium iodide solution containing starch solution blue.	NO_2	Nitrite (NO_2^-)
Colourless vapours with smell of vinegar. Vapours turn blue litmus red.	CH_3COOH vapours	Acetate, (CH_3COO^-)

Confirmatory tests for CO_3^{2-} , S^{2-} , SO_3^{2-} , NO_2^- and CH_3COO^-

Confirmatory (wet) tests for anions are performed by using **water extract** when salt is soluble in water and by using sodium carbonate extract when salt is insoluble in water. Confirmation of CO_3^{2-} is done by using aqueous solution of the salt or by using solid salt as such because sodium carbonate extract contains carbonate ions. Water extract is made by dissolving salt in water. Preparation of sodium carbonate extract is given below.

Preparation of sodium carbonate extract

Take 1 g of salt in a porcelain dish or boiling tube. Mix about 3 g of solid sodium carbonate and add 15 mL of distilled water to it. Stir and boil the content for about 10 minutes. Cool, filter and collect the filtrate in a test tube and label it as sodium carbonate extract.

Confirmatory tests for acid radicals, which react with dilute sulphuric acid are given below in Table 7.2.

Table 7.2 : Confirmatory tests for CO_3^{2-} , S^{2-} , SO_3^{2-} , NO_2^- , CH_3COO^-

Anion	Confirmatory test
Carbonate (CO_3^{2-})	Take 0.1 g of salt in a test tube, add dilute sulphuric acid. CO_2 gas is evolved with brisk effervescence which turns lime water milky. On passing the gas for some more time, milky appearance disappears.
Sulphide (S^{2-})	Take 1 mL of water extract and make it alkaline by adding ammonium hydroxide or sodium carbonate extract. Add a drop of sodium nitroprusside solution. Purple or violet colouration appears.
*Sulphite (SO_3^{2-})	(a) Take 1 mL of water extract or sodium carbonate extract in a test tube and add barium chloride solution. A white precipitate is formed which dissolves in dilute hydrochloric acid and sulphur dioxide gas is also evolved. (b) Take the precipitate of step (a) in a test tube and add a few drops of potassium permanganate solution acidified with dil. H_2SO_4 . Colour of potassium permanganate solution gets discharged.
Nitrite (NO_2^-)	(a) Take 1 mL of water extract in a test tube. Add a few drops of potassium iodide solution and a few drops of starch solution, acidify with acetic acid. Blue colour appears. (b) Acidify 1 mL of water extract with acetic acid. Add 2-3 drops of sulphanilic acid solution followed by 2-3 drops of 1-naphthylamine reagent. Appearance of red colour indicates the presence of nitrite ion.

* Like CO_2 sulphur dioxide also turns lime water milky. But CO_2 is odourless gas and SO_2 has a characteristic smell.

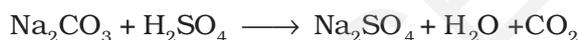
Acetate (CH_3COO^-)	<p>(a) Take 0.1 g of salt in a china dish. Add 1 mL of ethanol and 0.2 mL conc. H_2SO_4 and heat. Fruity odour confirms the presence of acetate ion.</p> <p>(b) Take 0.1 g of salt in a test tube, add 1-2 mL distilled water, shake well filter if necessary. Add 1 to 2 mL neutral** ferric chloride solution to the filtrate. Deep red colour appears which disappears on boiling and a brown-red precipitate is formed.</p>
---------------------------------------	--

** Preparation of neutral Ferric Chloride : Add dilute NaOH solution to ferric chloride solution drop by drop with shaking until a small but permanent precipitate of ferric hydroxide is obtained. Filter the precipitate and use the filtrate for analysis.

Chemistry of Confirmatory Tests

1. Test for Carbonate ion [CO_3^{2-}]

If there is effervescence with the evolution of a colourless and odourless gas on adding dil. H_2SO_4 to the solid salt, this indicates the presence of carbonate ion. The gas turns lime water milky due to the formation of CaCO_3 (Fig. 7.1)



If CO_2 gas is passed in excess through lime water, the milky appearance disappears due to the formation of calcium hydrogen carbonate which is soluble in water.

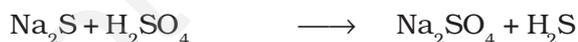


Hydrogen
sulphide



2. Test for Sulphide ion [S^{2-}]

- (a) With warm dilute H_2SO_4 a sulphide gives hydrogen sulphide gas which smells like rotten eggs. A piece of filter paper dipped in lead acetate solution turns black on exposure to the gas due to the formation of lead sulphide which is black in colour.

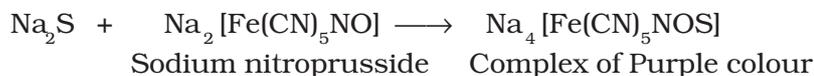


Lead sulphide

Black precipitate

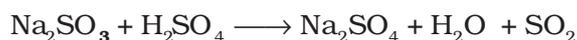
- (b) If the salt is soluble in water, take the solution of salt in water make it alkaline with ammonium hydroxide and add sodium nitroprusside solution. If it is insoluble in water take sodium carbonate extract and add a few drops of sodium nitroprusside solution. Purple or violet

colouration due to the formation of complex compound $\text{Na}_4[\text{Fe}(\text{CN})_5\text{NOS}]$ confirms the presence of sulphide ion in the salt.

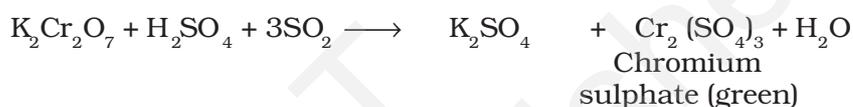


3. Test for Sulphite ion $[\text{SO}_3^{2-}]$

- (a) On treating sulphite with warm dil. H_2SO_4 , SO_2 gas is evolved which is suffocating with the smell of burning sulphur.



The gas turns potassium dichromate paper acidified with dil. H_2SO_4 , green.



Barium compounds



Potassium permanganate



- (b) An aqueous solution or sodium carbonate extract of the salt produces a white precipitate of barium sulphite on addition of barium chloride solution.



This precipitate gives following tests.

- (i) This precipitate on treatment with dilute HCl , dissolves due to decomposition of sulphite by dilute HCl . Evolved SO_2 gas can be tested.

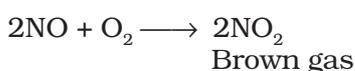
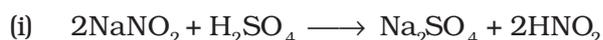


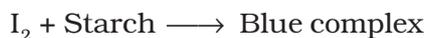
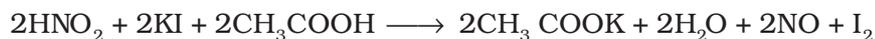
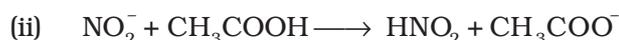
- (ii) Precipitate of sulphite decolourises acidified potassium permanganate solution.



4. Test for Nitrite ion $[\text{NO}_2^-]$

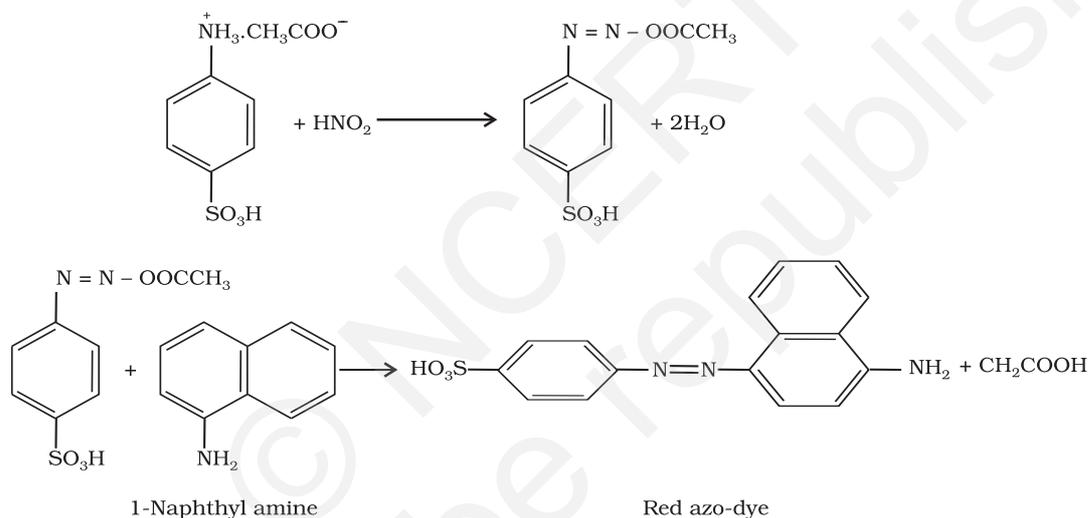
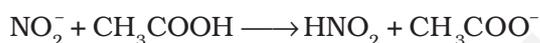
- (a) On treating a solid nitrite with dil. H_2SO_4 and warming, reddish brown fumes of NO_2 gas are evolved. Addition of potassium iodide solution to the salt solution followed by freshly prepared starch solution and acidification with acetic acid produces blue colour. Alternatively, a filter paper moistened with potassium iodide and starch solution and a few drops of acetic acid turns blue on exposure to the gas due to the interaction of liberated iodine with starch.





(b) Sulphanilic acid — 1-naphthylamine reagent test (Griss-Ilosvay test)

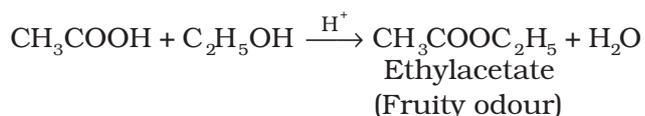
On adding sulphanilic acid and 1-naphthylamine reagent to the water extract or acidified with acetic acid, sulphanilic acid is diazotised in the reaction by nitrous acid formed. Diazotised acid couples with 1-naphthylamine to form a red azo-dye.



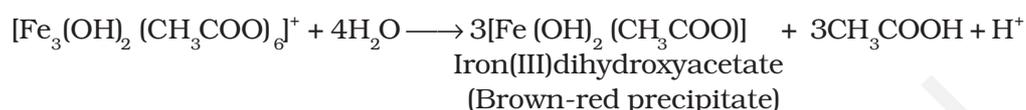
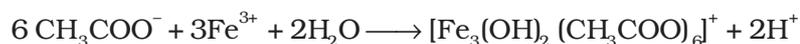
The test solution should be very dilute. In concentrated solutions reaction does not proceed beyond diazotisation.

5. Test for Acetate ion [CH_3COO^-]

(a) If the salt smells like vinegar on treatment with dil. H_2SO_4 , this indicates the presence of acetate ions. Take 0.1 g of salt in a china dish and add 1 mL of ethanol. Then add about 0.2 mL of conc. H_2SO_4 and heat. Fruity odour of ethyl acetate indicates the presence of CH_3COO^- ion.



- (b) Acetate gives deep red colour on reaction with neutral ferric chloride solution due to the formation of complex ion which decomposes on heating to give Iron (III) dihydroxyacetate as brown red precipitate.



Step-II : Preliminary Test with Concentrated Sulphuric Acid

If no positive result is obtained from dil. H_2SO_4 test, take 0.1 g of salt in a test tube and 3-4 drops of conc. H_2SO_4 . Observe the change in the reaction mixture in cold and then warm it. Identify the gas evolved on heating (see Table 7.3).

Table 7.3 : Preliminary examination with concentrated sulphuric acid

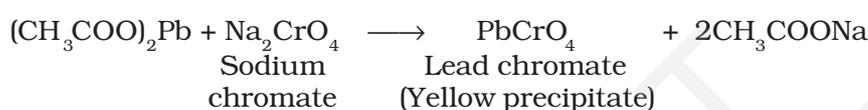
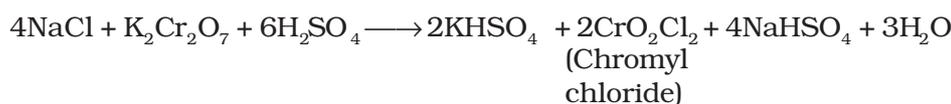
Observations	Inference	
	Gas/vapours evolved	Possible anions
A colourless gas with pungent smell, which gives dense white fumes when a rod dipped in ammonium hydroxide is brought near the mouth of the test tube.	HCl	Chloride, (Cl^-)
Reddish brown gas with a pungent odour is evolved. Intensity of reddish gas increases on heating the reaction mixture after addition of solid MnO_2 to the reaction mixture. Solution also acquires red colour.	Br_2 vapours	Bromide, (Br^-)
Violet vapours, which turn starch paper blue and a layer of violet sublimate is formed on the sides of the tube. Fumes become dense on adding MnO_2 to the reaction mixture.	I_2 vapours	Iodide, (I^-)
Brown fumes evolve which become dense upon heating the reaction mixture after addition of copper turnings and the solution acquires blue colour.	NO_2	Nitrate, (NO_3^-)
Colourless, odourless gas is evolved which turns lime water milky and the gas coming out of lime water burns with a blue flame, if ignited.	CO and CO_2	Oxalate, ($\text{C}_2\text{O}_4^{2-}$)

Confirmatory tests for the anions which react with concentrated sulphuric acid are given in Table 7.4.

Table 7.4 : Confirmatory tests for Cl^- , Br^- , I^- , NO_3^- and $\text{C}_2\text{O}_4^{2-}$

Anion	Confirmatory test
Chloride (Cl^-)	<p>(a) Take 0.1 g of salt in a test tube, add a pinch of manganese dioxide and 3-4 drops of conc. sulphuric acid. Heat the reaction mixture. Greenish yellow chlorine gas is evolved which is detected by its pungent odour and bleaching action.</p> <p>(b) Take 1 mL of sodium carbonate extract in a test tube, acidify it with dil. HNO_3 or take water extract and add silver nitrate solution. A curdy white precipitate is obtained which is soluble in ammonium hydroxide solution.</p> <p>(c) Take 0.1 g salt and a pinch of solid potassium dichromate in a test tube, add conc. H_2SO_4, heat and pass the gas evolved through sodium hydroxide solution. It becomes yellow. Divide the solution into two parts. Acidify one part with acetic acid and add lead acetate solution. A yellow precipitate is formed. Acidify the second part with dilute sulphuric acid and add 1 mL of amyl alcohol followed by 1 mL of 10% hydrogen peroxide. After gentle shaking the organic layer turns blue.</p>
Bromide (Br^-)	<p>(a) Take 0.1 g of salt and a pinch of MnO_2 in a test tube. Add 3-4 drops conc. sulphuric acid and heat. Intense brown fumes are evolved.</p> <p>(b) Neutralise 1 mL of sodium carbonate extract with hydrochloric acid (or take the water extract). Add 1 mL carbon tetrachloride (CCl_4)/chloroform (CHCl_3)/carbon disulphide. Now add an excess of chlorine water dropwise and shake the test tube. A brown colouration in the organic layer confirms the presence of bromide ion.</p> <p>(c) Acidify 1 mL of sodium carbonate extract with dil. HNO_3 (or take 1 mL water extract) and add silver nitrate solution. A pale yellow precipitate soluble with difficulty in ammonium hydroxide solution is obtained.</p>
Iodide (I^-)	<p>(a) Take 1 mL of salt solution neutralised with HCl and add 1 mL chloroform/carbon tetrachloride/carbon disulphide. Now add an excess of chlorine water drop wise and shake the test tube. A violet colour appears in the organic layer.</p> <p>(b) Take 1 mL of sodium carbonate extract acidify it with dil. HNO_3 (or take water extract). Add, silver nitrate solution. A yellow precipitate insoluble in NH_4OH solution is obtained.</p>

- (d) Mix a little amount of salt and an equal amount of solid potassium dichromate ($K_2Cr_2O_7$) in a test tube and add conc. H_2SO_4 to it. Heat the test tube and pass the evolved gas through sodium hydroxide solution. If a yellow solution is obtained, divide the solution into two parts. Acidify the first part with acetic acid and then add lead acetate solution. Formation of a yellow precipitate of lead chromate confirms the presence of chloride ions in the salt. This test is called **chromyl chloride test**.*



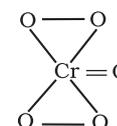
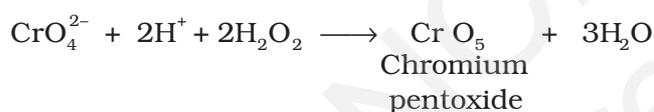
Chromyl chloride



Lead chromate



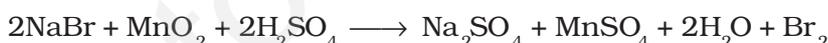
Acidify the second part with dilute sulphuric acid and add small amounts of amyl alcohol and then 1 mL of 10% hydrogen peroxide solution. On gentle shaking organic layer turns blue. CrO_4^{2-} ion formed in the reaction of chromyl chloride with sodium hydroxide reacts with hydrogen peroxide to form chromium pentoxide (CrO_5) (See structure) which dissolves in amyl alcohol to give blue colour.



Structure of chromium pentoxide

2. Test for Bromide ion (Br^-)

If on heating the salt with conc. H_2SO_4 reddish brown fumes of bromine are evolved in excess, this indicates the presence of Br^- ions. The fumes get intensified on addition of MnO_2 . Bromine vapours turn starch paper yellow.

Bromine
very toxic byinhalation
corrosive

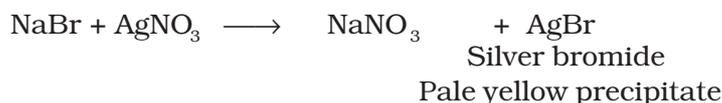
- (a) Add 1 mL of carbon tetrachloride (CCl_4)/chloroform ($CHCl_3$)** and excess of freshly prepared chlorine water dropwise to the salt solution in water or sodium carbonate extract neutralised with dilute HCl. Shake the test tube vigorously. The appearance of an orange brown colouration in the organic layer due to the dissolution of bromine in it, confirms the presence of bromide ions.



* Chromyl chloride test should be performed with minimum amount of substance to avoid pollution by Cr^{3+} ions.

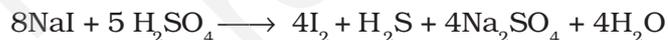
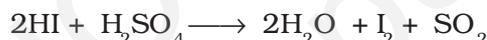
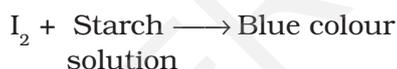
** In place of carbon tetrachloride or chloroform, carbon disulphide or dichloromethane (CH_2Cl_2) can also be used.

- (b) Acidify the sodium carbonate extract of the salt with dil. HNO_3 . Add silver nitrate (AgNO_3) solution and shake the test tube. A pale yellow precipitate is obtained which dissolves in ammonium hydroxide with difficulty.

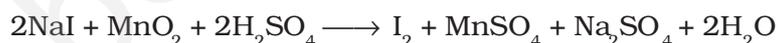


3. Test for Iodide ion (I^-)

- (a) If on heating the salt with conc. H_2SO_4 , deep violet vapours with a pungent smell are evolved. These turn starch paper blue and a violet sublimate is formed on the sides of the test tube, it indicates the presence of I^- ions. Some HI, sulphur dioxide, hydrogen sulphide, and sulphur are also formed due to the following reactions.



On adding MnO_2 to the reaction mixture, the violet vapours become dense.

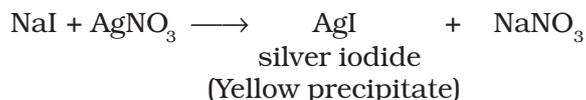


- (b) Add 1 mL of CHCl_3 or CCl_4 and chlorine water in excess to the salt solution in water or sodium carbonate extract neutralised with dil. HCl and shake the test tube vigorously. Presence of violet colouration in the organic layer confirms the presence of iodide ions.



Iodine dissolves in the organic solvent and the solution becomes violet.

- (c) Acidify sodium carbonate extract of the salt with dil. HNO_3 and add AgNO_3 solution. Appearance of a yellow precipitate insoluble in excess of NH_4OH confirms the presence of iodide ions.



Iodine,
harmful by
inhalation
and contact
with skin



Chlorine,
toxic by
inhalation

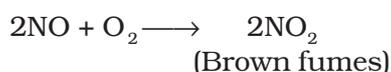
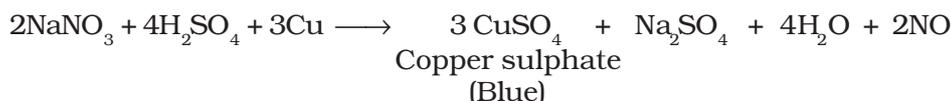


Chloroform,
harmful and
toxic by
inhalation



4. Test for Nitrate ion $[\text{NO}_3^-]$

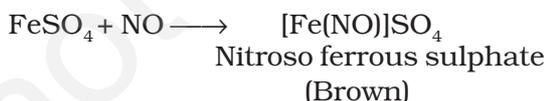
- (a) If on heating the salt with conc. H_2SO_4 light brown fumes are evolved then heat a small quantity of the given salt with few copper turnings or chips and conc. H_2SO_4 . Evolution of excess of brown fumes indicates the presence of nitrate ions. The solution turns blue due to the formation of copper sulphate.



- (b) Take 1 mL of an aqueous solution of the salt and add 2 mL conc. H_2SO_4 slowly. Mix the solutions thoroughly and cool the test tube under the tap. Now, add freshly prepared ferrous sulphate solution along the sides of the test tube dropwise so that it forms a layer on the top of the liquid already present in the test tube. A dark brown ring is formed at the junction of the two solutions due to the formation of nitroso ferrous sulphate (Fig. 7.2). Alternatively first ferrous sulphate is added and then concentrated sulphuric acid is added.

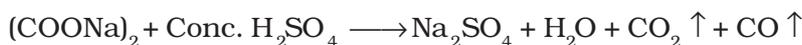


Fig. 7.2 : Formation of brown ring



5. Test for Oxalate ion $[\text{C}_2\text{O}_4^{2-}]$

If carbon dioxide gas along with carbon monoxide gas is evolved in the preliminary examination with concentrated sulphuric acid, this gives indication about the presence of oxalate ion.



Copper
sulphate



Nitric
acid

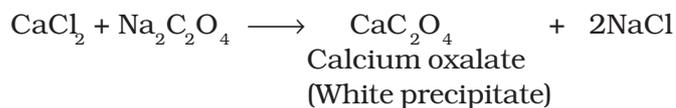


Oxalates



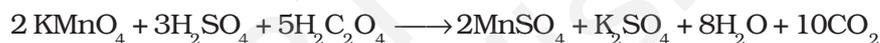
Oxalate is confirmed by the following tests:

- (a) Acidify sodium carbonate extract with acetic acid and add calcium chloride solution. A white precipitate of calcium oxalate, insoluble in ammonium oxalate and oxalic acid solution indicates the presence of oxalate ion.



- (b) KMnO_4 test

Filter the precipitate from test (a). Add dil. H_2SO_4 to it followed by dilute KMnO_4 solution and warm. Pink colour of KMnO_4 is discharged:



Pass the gas evolved through lime water. A white precipitate is formed which dissolves on passing the gas for some more time.

Step-III : Test for Sulphate and Phosphate

If no positive test is obtained in Steps-I and II, then tests for the presence of sulphate and phosphate ions are performed. These tests are summarised in Table 7.5.

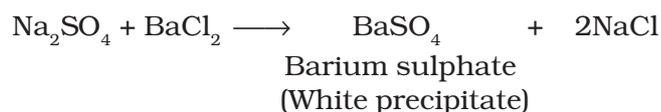
Table 7.5 : Confirmatory tests for Sulphate and Phosphate

Ion	Confirmatory Test
Sulphate (SO_4^{2-})	<p>(a) Take 1 mL water extract of the salt in water or sodium carbonate and after acidifying with dilute hydrochloric acid add BaCl_2 solution. White precipitate insoluble in conc. HCl or conc. HNO_3 is obtained.</p> <p>(b) Acidify the aqueous solution or sodium carbonate extract with acetic acid and add lead acetate solution. Appearance of white precipitate confirms the presence of SO_4^{2-} ion.</p>
Phosphate (PO_4^{3-})	<p>(a) Acidify sodium carbonate extract or the solution of the salt in water with conc. HNO_3 and add ammonium molybdate solution and heat to boiling. A canary yellow precipitate is formed.</p>

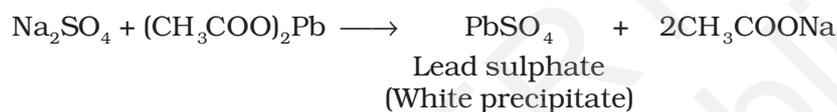
Chemistry of Confirmatory Tests

1. Test of Sulphate ions $[\text{SO}_4^{2-}]$

- (a) Aqueous solution or sodium carbonate extract of the salt acidified with acetic acid on addition of barium chloride gives a white precipitate of barium sulphate insoluble in conc. HCl or conc. HNO_3 .

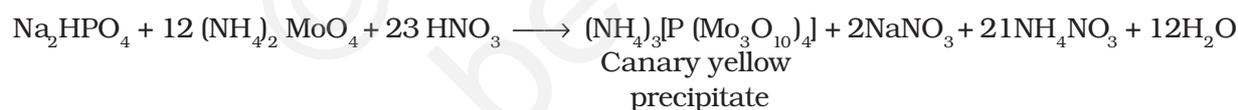


- (b) Sulphate ions give white precipitate of lead sulphate when aqueous solution or sodium carbonate extract neutralised with acetic acid is treated with lead acetate solution.



2. Test for Phosphate ion $[\text{PO}_4^{3-}]$

- (a) Add conc. HNO_3 and ammonium molybdate solution to the test solution containing phosphate ions and boil. A yellow colouration in solution or a canary yellow precipitate of ammonium-phosphomolybdate, $(\text{NH}_4)_3[\text{P}(\text{Mo}_3\text{O}_{10})_4]$ is formed. Each oxygen of phosphate has been replaced by Mo_3O_{10} group.



SYSTEMATIC ANALYSIS OF CATIONS

The tests for cations may be carried out according to the following scheme.

Step - I : Preliminary Examination of the Salt for Identification of Cation

1. Colour Test

Observe the colour of the salt carefully, which may provide useful information about the cations. Table 7.6 gives the characteristic colours of the salts of some cations.

Table 7.6 : Characteristic colours of some metal ions

Colour	Cations Indicated
Light green, Yellow, Brown	Fe^{2+} , Fe^{3+}
Blue	Cu^{2+}
Bright green	Ni^{2+}
Blue, Red, Violet, Pink	Co^{2+}
Light pink	Mn^{2+}

2. Dry Heating Test

- (i) Take about 0.1 g of the dry salt in a clean and dry test tube.
- (ii) Heat the above test tube for about one minute and observe the colour of the residue when it is hot and also when it becomes cold. Observation of changes gives indications about the presence of cations, which may not be taken as conclusive evidence (see Table 7.7).

Table 7.7 : Inferences from the colour of the salt in cold and on heating

Colour when cold	Colour when hot	Inference
Blue	White	Cu^{2+}
Green	Dirty white or yellow	Fe^{2+}
White	Yellow	Zn^{2+}
Pink	Blue	Co^{2+}

3. Flame Test

The chlorides of several metals impart characteristic colour to the flame because they are volatile in non-luminous flame. This test is performed with the help of a platinum wire as follows :

- (i) Make a tiny loop at one end of a platinum wire.
- (ii) To clean the loop dip it into concentrated hydrochloric acid and hold it in a non-luminous flame (Fig. 7.3).
- (iii) Repeat step (ii) until the wire imparts no colour to the flame.
- (iv) Put 2-3 drops of concentrated hydrochloric acid on a clean watch glass and make a paste of a small quantity of the salt in it.
- (v) Dip the clean loop of the platinum wire in this paste and introduce the loop in the non-luminous (oxidising) flame (Fig. 7.3).
- (vi) Observe the colour of the flame first with the naked eye and then through a blue glass and identify the metal ion with the help of Table 7.8.

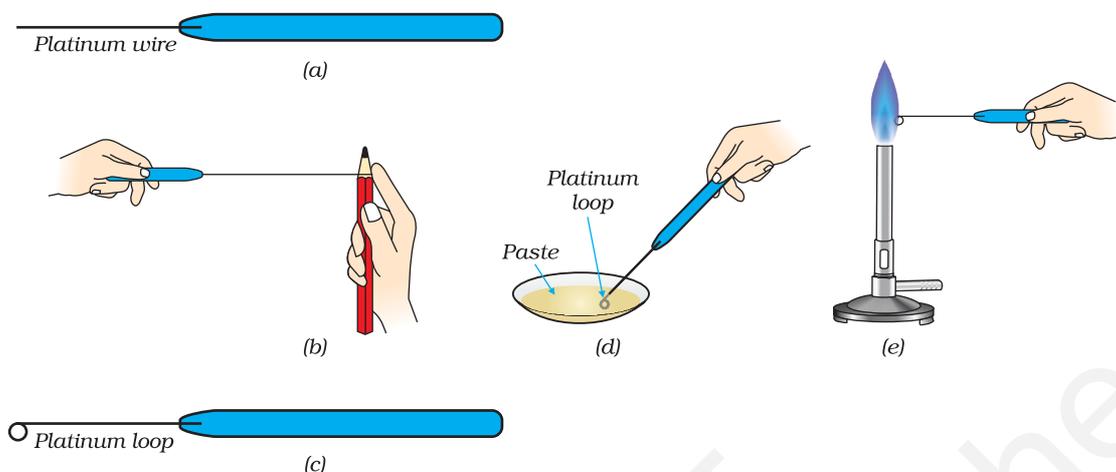


Fig.7.3 : Performing flame test

Table 7.8 : Inference from the flame test

Colour of the flame observed by naked eye	Colour of the flame observed through blue glass	Inference
Green flame with blue centre	Same colour as observed without glass	Cu^{2+}
Crimson red	Purple	Sr^{2+}
Apple green	Bluish green	Ba^{2+}
Brick red	Green	Ca^{2+}

4. Borax Bead Test

This test is employed only for coloured salts because borax reacts with metal salts to form metal borates or metals, which have characteristic colours.

- To perform this test make a loop at the end of the platinum wire and heat it in a flame till it is red hot.
- Dip the hot loop into borax powder and heat it again until borax forms a colourless transparent bead on the loop. Before dipping the borax bead in the test salt or mixture, confirm that the bead is transparent and colourless. If it is coloured this means that, the platinum wire is not clean. Then make a fresh bead after cleaning the wire.
- Dip the bead in a small quantity of the dry salt and again hold it in the flame.
- Observe the colour imparted to the bead in the non - luminous flame as well as in the luminous flame while it is hot and when it is cold (Fig. 7.4).
- To remove the bead from the platinum wire, heat it to redness and tap the platinum wire with your finger. (Fig. 7.5).

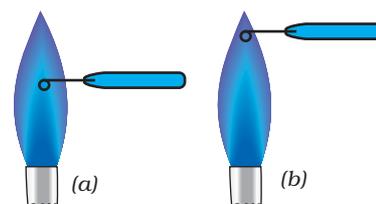
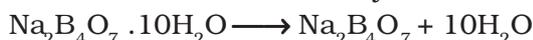
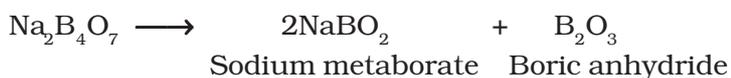


Fig. 7.4 : Borax bead test
(a) Heating in reducing flame (b) Heating in oxidising flame

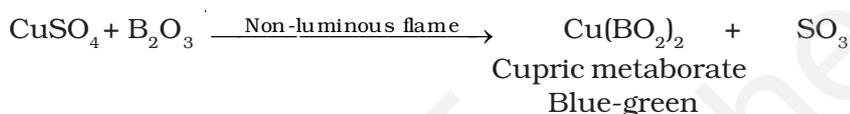
On heating, borax loses its water of crystallisation and decomposes to give sodium metaborate and boric anhydride.



Borax

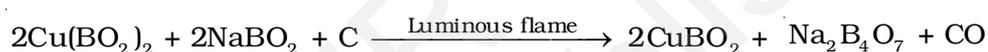


On treatment with metal salt, boric anhydride forms metaborate of the metal which gives different colours in oxidising and reducing flame. For example, in the case of copper sulphate, following reactions occur.



Two reactions may take place in the reducing flame:

(i) The blue $\text{Cu}(\text{BO}_2)_2$ is reduced to colourless cuprous metaborate as follows:



or (ii) Cupric metaborate may be reduced to metallic copper and the bead appears red and opaque.



The preliminary identification of metal ion can be made from Table 7.9.

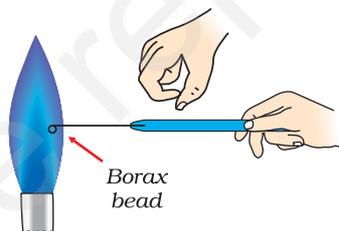


Fig. 7.5 : Removing borax bead

Table 7.9 : Inference from the borax bead test

Heating in oxidising (non-luminous) flame		Heating in reducing (luminous) flame		Inference
Colour of the salt bead		Colour of the salt bead		
In cold	In hot	In cold	In hot	
Blue	Green	Red opaque	Colourless	Cu^{2+}
Reddish brown	Violet	Grey	Grey	Ni^{2+}
Light violet	Light violet	Colourless	Colourless	Mn^{2+}
Yellow	Yellowish brown	Green	Green	Fe^{3+}

5. Charcoal Cavity Test

Metallic carbonate when heated in a charcoal cavity decomposes to give corresponding oxide. The oxide appears as a coloured residue in the cavity. Sometimes oxide may be reduced to metal by the carbon of the charcoal cavity. The test may be performed as follows:

- (i) Make a small cavity in a charcoal block with the help of a charcoal borer. Do not apply pressure otherwise it will crack [Fig. 7.6 (a)].
- (ii) Fill the cavity with about 0.2 g of the salt and about 0.5 g of anhydrous sodium carbonate.

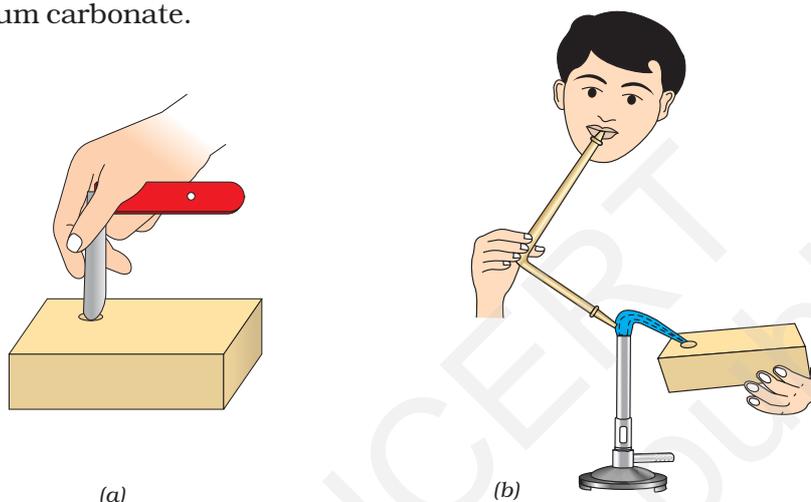
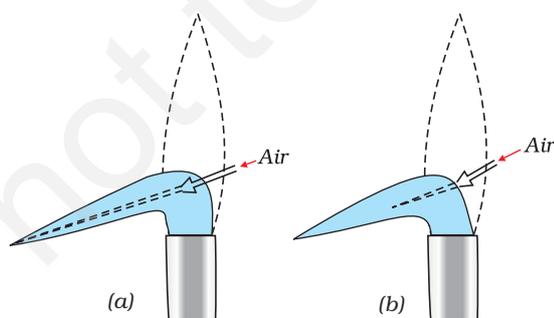


Fig. 7.6 : (a) Making charcoal cavity (b) Heating salt in the cavity

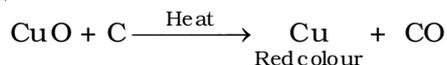
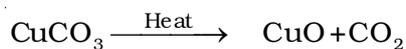
- (iii) Moisten the salt in the cavity with one or two drops of water, otherwise salt/mixture will blow away.
- (iv) Use a blowpipe to heat the salt in a luminous (reducing) flame and observe the colour of oxide/ metallic bead formed in the cavity both when hot and cold [Fig. (7.6 b)]. Obtain oxidising and reducing flame as shown in Fig. 7.7 a and b.
- (v) Always bore a fresh cavity for testing the new salt.



- Note :**
- To obtain oxidising flame hold the nozzle of the blowpipe about one third within the flame.
 - To obtain reducing flame place nozzle of the blowpipe just outside the flame.

Fig. 7.7 : Obtaining oxidising and reducing flame (a) Oxidising flame (b) Reducing flame

When test is performed with CuSO_4 , the following change occurs.



In case of ZnSO_4 :



Yellow when hot,
White when cold

The metal ion can be inferred from Table 7.10.

Table 7.10 : Inference from the charcoal cavity test

Observations	Inference
Yellow residue when hot and grey metal when cold	Pb^{2+}
White residue with the odour of garlic	As^{3+}
Brown residue	Cd^{2+}
Yellow residue when hot and white when cold	Zn^{2+}

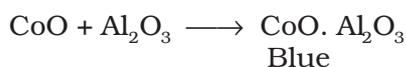
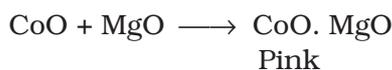
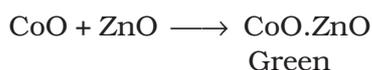
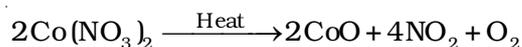
6. Cobalt Nitrate Test

If the residue in the charcoal cavity is white, cobalt nitrate test is performed.

- Treat the residue with two or three drops of cobalt nitrate solution.
- Heat it strongly in non-luminous flame with the help of a blow pipe and observe the colour of the residue.

On heating, cobalt nitrate decomposes into cobalt (II) oxide, which gives a characteristic colour with metal oxide present in the cavity.

Thus, with ZnO , Al_2O_3 and MgO , the following reactions occur:



Step-II : Wet Tests for Identification of Cations

The cations indicated by the preliminary tests given above are confirmed by systematic analysis given below.

The first essential step is to prepare a clear and transparent solution of the salt. This is called **original solution**. It is prepared as follows:

Preparation of Original Solution (O.S.)

To prepare the original solution, following steps are followed one after the other in a systematic order. In case the salt does not dissolve in a particular solvent even on heating, try the next solvent.

The following solvents are tried:

1. Take a little amount of the salt in a clean boiling tube and add a few mL of distilled water and shake it. If the salt does not dissolve, heat the content of the boiling tube till the salt completely dissolves.
2. If the salt is insoluble in water as detailed above, take fresh salt in a clean boiling tube and add a few mL of dil.HCl to it. If the salt is insoluble in cold, heat the boiling tube till the salt is completely dissolved.
3. If the salt does not dissolve either in water or in dilute HCl even on heating, try to dissolve it in a few mL of conc. HCl by heating.
4. If salt does not dissolve in conc. HCl, then dissolve it in dilute nitric acid.
5. If salt does not dissolve even in nitric acid then a mixture of conc. HCl and conc. HNO₃ in the ratio 3:1 is tried. This mixture is called aqua regia. A salt not soluble in aqua regia is considered to be an insoluble salt.

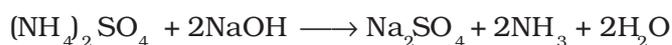
Group Analysis

(I) Analysis of Zero group cation (NH₄⁺ ion)

- (a) Take 0.1 g of salt in a test tube and add 1-2 mL of NaOH solution to it and heat. If there is a smell of ammonia, this indicates the presence of ammonium ions. Bring a glass rod dipped in hydrochloric acid near the mouth of the test tube. White fumes are observed.
- (b) Pass the gas through Nessler's reagent. Brown precipitate is obtained.

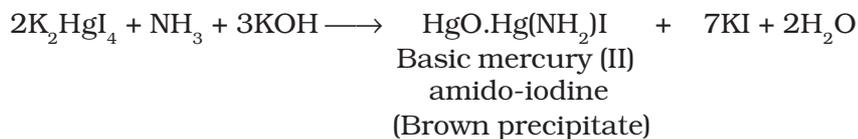
Chemistry of Confirmatory Tests for NH₄⁺ ion

- (a) Ammonia gas evolved by the action of sodium hydroxide on ammonium salts reacts with hydrochloric acid to give ammonium chloride, which is visible as dense white fume.



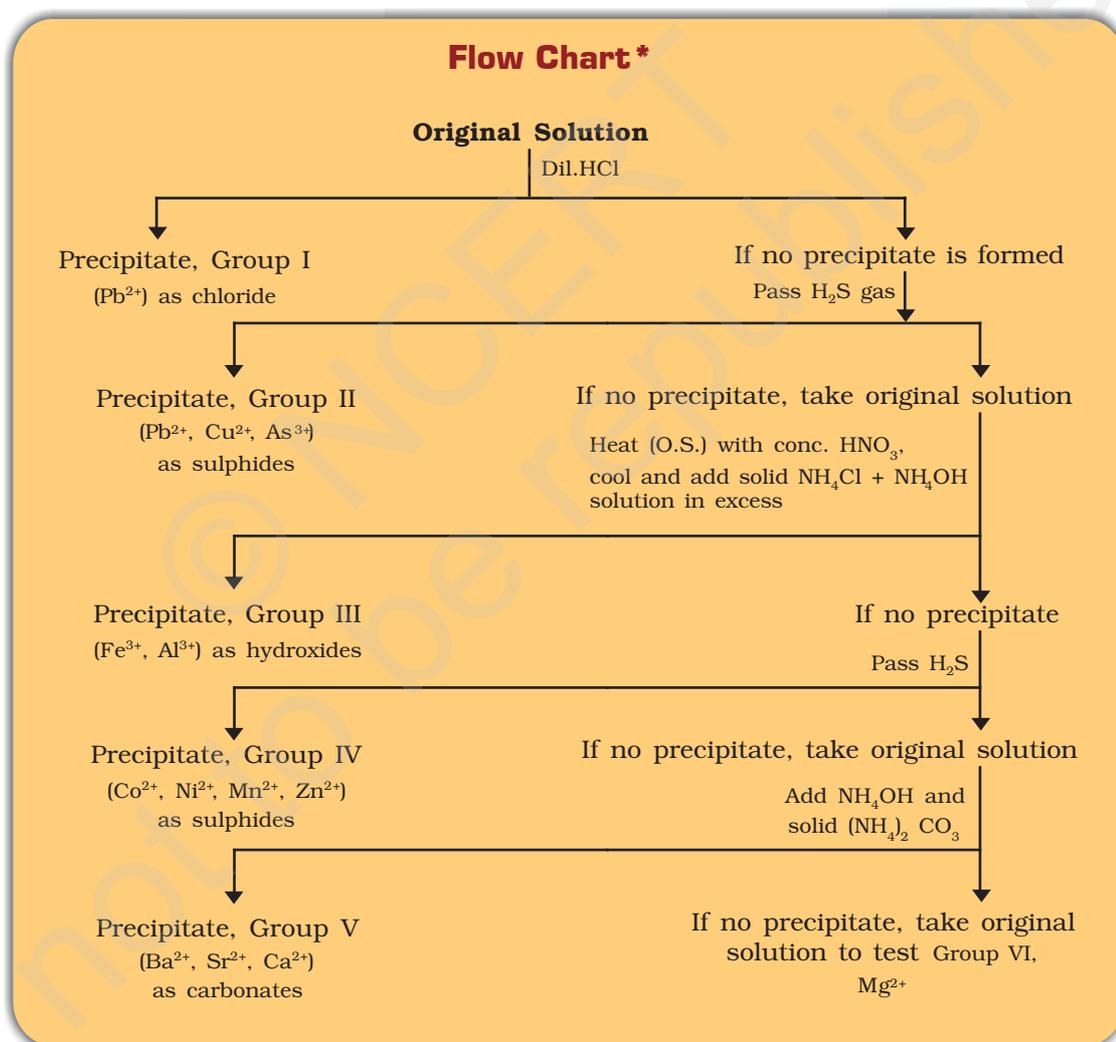
Mercury
Salts

On passing the gas through Nessler's reagent, a brown colouration or a precipitate of basic mercury(II) amido-iodine is formed.



For the analysis of cations belonging to groups I-VI, the cations are precipitated from the original solution by using the group reagents (see Table 7.11) according to the scheme shown in the flow chart given below:

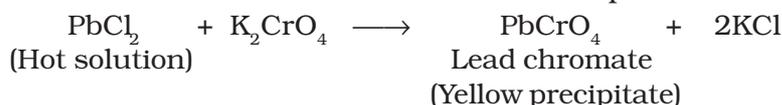
The separation of all the six groups is represented as below:



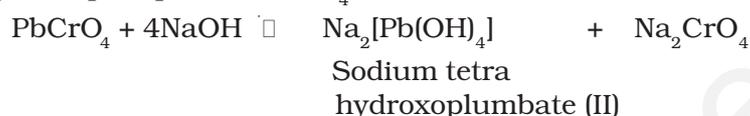
* This flow chart is for the detection of one cation only.
For detection of more than one cation modification will be required.

This yellow precipitate (PbI_2) is soluble in boiling water and reappears on cooling as shining crystals.

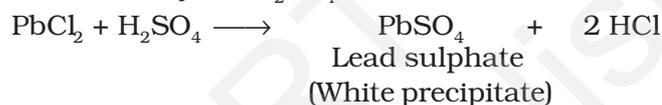
2. On addition of potassium chromate (K_2CrO_4) solution a yellow precipitate of lead chromate is obtained. This confirms the presence of Pb^{2+} ions.



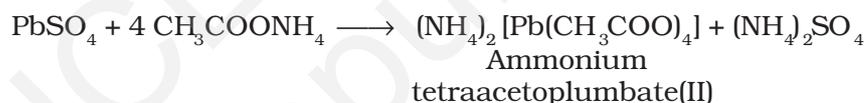
The yellow precipitate (PbCrO_4) is soluble in hot NaOH solution.



3. A white precipitate of lead sulphate (PbSO_4) is formed on addition of alcohol followed by dil. H_2SO_4 .



Lead sulphate is soluble in ammonium acetate solution due to the formation of tetraacetoplumbate(II) ions. This reaction may be promoted by addition of few drops of acetic acid.



Hydrogen
sulphide



(III) Analysis of Group-II cations

If group-I is absent, add excess of water to the same test tube. Warm the solution and pass H_2S gas for 1-2 minutes (Fig. 7.6). Shake the test tube. If a precipitate appears, this indicates the presence of group-II cations. Pass more H_2S gas through the solution to ensure complete precipitation and separate the precipitate. If the colour of the precipitate is black, it indicates the presence of Cu^{2+} or Pb^{2+} ions. If it is yellow in colour, then presence of As^{3+} ions is indicated.

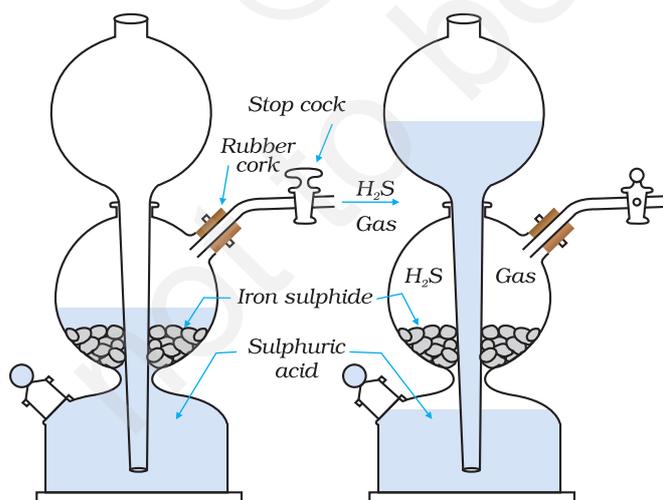


Fig. 7.8 : Kipp's apparatus for preparation of H_2S gas

Take the precipitate of group-II in a test tube and add excess of yellow ammonium sulphide solution to it. Shake the test tube. If the precipitate is insoluble, **group II-A (copper group)** is present. If the precipitate is soluble, this indicates the presence of **group-II B (arsenic group)**.

Confirmatory tests for the groups II A and II B are given in Table 7.13.

Table 7.13 : Confirmatory tests for Group-II A and II B cations

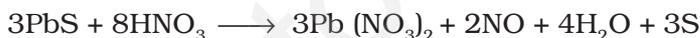
Black precipitate of Group II A ions (Pb^{2+} , Cu^{2+}) insoluble in yellow ammonium sulphide is formed.	If a yellow precipitate soluble in yellow ammonium sulphide is formed then As^{3+} ion is present.
Boil the precipitate of Group II A with dilute nitric acid and add a few drops of alcohol and dil. H_2SO_4 .	Acidify this solution with dilute HCl. A yellow precipitate is formed. Heat the precipitate with concentrated nitric acid and add ammonium molybdate solution. A canary yellow precipitate is formed.
<p>White precipitate confirms the presence of Pb^{2+} ions. Dissolve the precipitate in ammonium acetate solution. Acidify with acetic acid and divide the solution into two parts.</p> <p>(i) To the first part add potassium chromate solution, a yellow precipitate is formed.</p> <p>(ii) To the second part, add potassium iodide solution, a yellow precipitate is formed.</p>	<p>If no precipitate is formed, add excess of ammonium hydroxide solution. A blue solution is obtained, acidify it with acetic acid and add potassium ferrocyanide solution. A chocolate brown precipitate is formed.</p>

Group-II A (Copper Group)

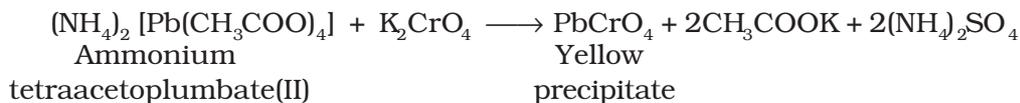
Alcohol

**Chemistry of confirmatory tests of Group-II A cations****1. Test for Lead ion (Pb^{2+})**

Lead sulphide precipitate dissolves in dilute HNO_3 . On adding dil. H_2SO_4 and a few drops of alcohol to this solution a white precipitate of lead sulphate appears. This indicates the presence of lead ions.

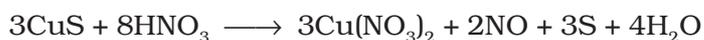


The white precipitate dissolves in ammonium acetate solution on boiling. When this solution is acidified with acetic acid and potassium chromate solution is added, a yellow precipitate of PbCrO_4 is formed. On adding potassium iodide solution, a yellow precipitate of lead iodide is formed.

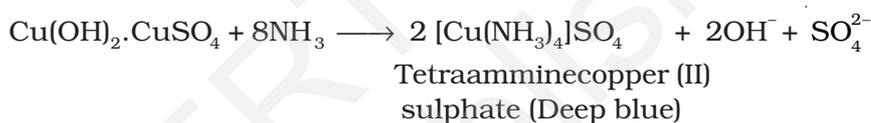
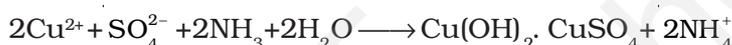
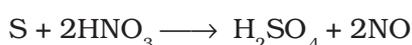


2. Test for Copper ion (Cu²⁺)

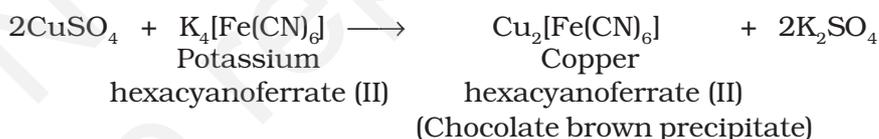
- (a) Copper sulphide dissolves in nitric acid due to the formation of copper nitrate.



On heating the reaction mixture for long time, sulphur is oxidised to sulphate and copper sulphate is formed and the solution turns blue. A small amount of NH₄OH precipitates basic copper sulphate which is soluble in excess of ammonium hydroxide due to the formation of tetraamminecopper (II) complex.

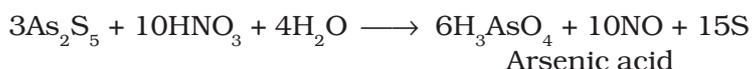
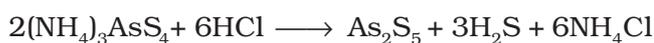
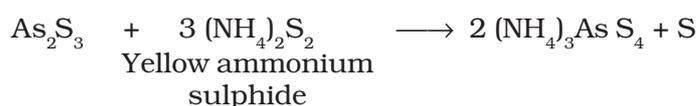


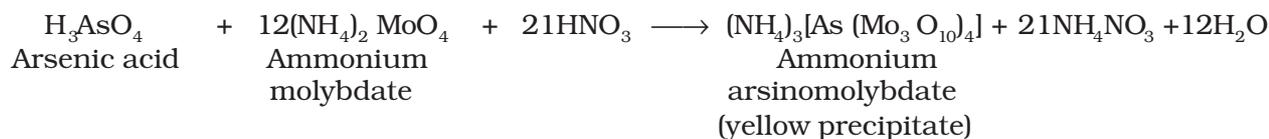
- (b) The blue solution on acidification with acetic acid and then adding potassium ferrocyanide [K₄Fe(CN)₆] solution gives a chocolate colouration due to the formation of copper ferrocyanide i.e. Cu₂[Fe(CN)₆].



Group-II B (Arsenic Group)

If group- II precipitate dissolves in yellow ammonium sulphide and the colour of the solution is yellow, this indicates the presence of As³⁺ ions. Ammonium thioarsenide formed on dissolution of As₂S₃ decomposes with dil. HCl, and a yellow precipitate of arsenic (V) sulphide is formed which dissolves in concentrated nitric acid on heating due to the formation of arsenic acid. On adding ammonium molybdate solution to the reaction mixture and heating, a canary yellow precipitate is formed. This confirms the presence of As³⁺ ions.





(IV) Analysis of Group-III cations

If group-II is absent, take original solution and add 2-3 drops of conc. HNO_3 to oxidise Fe^{2+} ions to Fe^{3+} ions. Heat the solution for a few minutes. After cooling add a small amount of solid ammonium chloride (NH_4Cl) and an excess of ammonium hydroxide (NH_4OH) solution till it smells of ammonia. Shake the test tube. If a brown or white precipitate is formed, this indicates the presence of group-III cations. Confirmatory tests of group-III cations are summarised in Table 7.14.

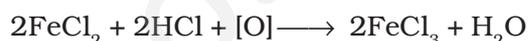
Observe the colour and the nature of the precipitate. A gelatinous white precipitate indicates the presence of aluminium ion (Al^{3+}). If the precipitate is brown in colour, this indicates the presence of ferric ions (Fe^{3+}).

Table 7.14 : Confirmatory test for Group-III cations

Brown precipitate Fe^{3+}	White precipitate Al^{3+}
Dissolve the precipitate in dilute HCl and divide the solution into two parts. (a) To the first part add potassium ferrocyanide solution [Potassium hexacyanoferrate (II)]. A blue precipitate/colouration appears. (b) To the second part add potassium thiocyanate solution. A blood red colouration appears.	Dissolve the white precipitate in dilute HCl and divide into two parts. (a) To the first part add sodium hydroxide solution and warm. A white gelatinous precipitate soluble in excess of sodium hydroxide solution. (b) To the second part first add blue litmus solution and then ammonium hydroxide solution drop by drop along the sides of the test tube. A blue floating mass in the colourless solution is obtained.

Chemistry of confirmatory tests of Group-III cations

When original solution is heated with concentrated nitric acid, ferrous ions are oxidised to ferric ions.

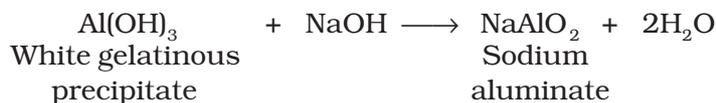


Third group cations are precipitated as their hydroxides, which dissolve in dilute hydrochloric acid due to the formation of corresponding chlorides.

1. Test for Aluminium ions (Al^{3+})

- (a) When the solution containing aluminium chloride is treated with sodium hydroxide, a white gelatinous precipitate of aluminium hydroxide is formed

which is soluble in excess of sodium hydroxide solution due to the formation of sodium aluminate.



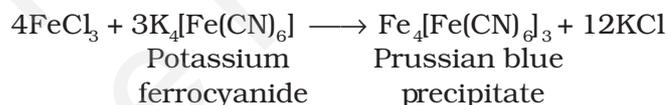
- (b) In the second test when blue litmus is added to the solution, a red colouration is obtained due to the acidic nature of the solution. On addition of NH_4OH solution drop by drop the solution becomes alkaline and aluminium hydroxide is precipitated. Aluminium hydroxide adsorbs blue colour from the solution and forms insoluble adsorption complex named 'lake'. Thus a blue mass floating in the colourless solution is obtained. The test is therefore called **lake test**.

2. Test for ferric ions (Fe^{3+})

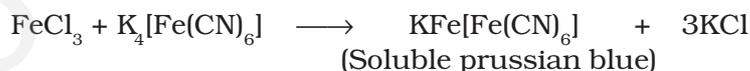
Reddish brown precipitate of ferric hydroxide dissolves in hydrochloric acid and ferric chloride is formed.



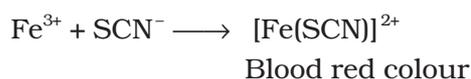
- (a) When the solution containing ferric chloride is treated with potassium ferrocyanide solution a blue precipitate/colouration is obtained. The colour of the precipitate is Prussian blue. It is ferric ferro-cyanide. The reaction takes place as follows:



If potassium hexacyanoferrate (II) (i.e. potassium ferrocyanide) is added in excess then a product of composition $\text{KFe}[\text{Fe(CN)}_6]$ is formed. This tends to form a colloidal solution ('soluble Prussian blue') and cannot be filtered.



- (b) To the second part of the solution, add potassium thiocyanate (potassium sulphocyanide) solution. The appearance of a blood red colouration confirms the presence of Fe^{3+} ions.



(V) Analysis of group-IV cations

If group-III is absent, pass H_2S gas in the solution of group-III for a few minutes. If a precipitate appears (white, black or flesh coloured), this indicates

the presence of group-IV cations. Table 7.15 gives a summary of confirmatory tests of group-IV cations.

Table 7.15 : Confirmatory test for Group-IV cations

White precipitate (Zn ²⁺)	Flesh coloured precipitate (Mn ²⁺)	Black precipitate (Ni ²⁺ , Co ²⁺)
<p>Dissolve the precipitate in dilute HCl by boiling. Divide the solution into two parts.</p> <p>(a) To the first part add sodium hydroxide solution. A white precipitate soluble in excess of sodium hydroxide solution confirms the presence of Zn²⁺ ions.</p> <p>(b) Neutralise the second part with ammonium hydroxide solution and add potassium ferrocyanide solution. A bluish white precipitate appears.</p>	<p>Dissolve the precipitate in dilute HCl by boiling, then add sodium hydroxide solution in excess. A white precipitate is formed which turns brown on keeping.</p>	<p>Dissolve the precipitate in aqua regia. Heat the solution to dryness and cool. Dissolve the residue in water and divide the solution into two parts.</p> <p>(a) To the first part of the solution add ammonium hydroxide solution till it becomes alkaline. Add a few drops of dimethyl glyoxime and shake the test tube. Formation of a bright red precipitate confirms the presence of Ni²⁺ ions.</p> <p>(b) Neutralise the second part with ammonium hydroxide solution. Acidify it with dilute acetic acid and add solid potassium nitrite. A yellow precipitate confirms the presence of Co²⁺ ions.</p>

Chemistry of confirmatory tests of Group-IV cations

Fourth group cations are precipitated as their sulphides. Observe the colour of the precipitate. A white colour of the precipitate indicates the presence of zinc ions, a flesh colour indicates the presence of manganese ions and a black colour indicates the presence of Ni²⁺ or Co²⁺ ions.

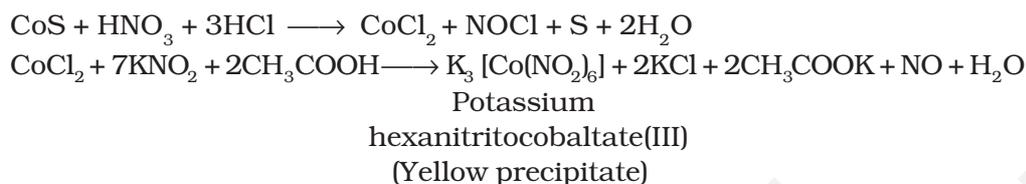
1. Test for Zinc ion (Zn²⁺)

Zinc sulphide dissolves in hydrochloric acid to form zinc chloride.



4. Test for Cobalt ion (Co^{2+})

Cobalt sulphide dissolves in aqua regia in the same manner as nickel sulphide. When the aqueous solution of the residue obtained after treatment with aqua regia is treated with a strong solution of potassium nitrite after neutralisation with ammonium hydroxide and the solution is acidified with dil. acetic acid, a yellow precipitate of the complex of cobalt named potassium hexanitritocobaltate (III) is formed.



(VI) Analysis of Group-V cations

If group-IV is absent then take original solution and add a small amount of solid NH_4Cl and an excess of NH_4OH solution followed by solid ammonium carbonate $(\text{NH}_4)_2\text{CO}_3$. If a white precipitate appears, this indicates the presence of group-V cations.

Dissolve the white precipitate by boiling with dilute acetic acid and divide the solution into three parts one each for Ba^{2+} , Sr^{2+} and Ca^{2+} ions. **Preserve a small amount of the precipitate for flame test.** Summary of confirmatory tests is given in Table 7.16.

Table 7.16 : Confirmatory test for Group-V cations

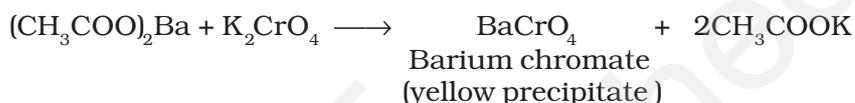
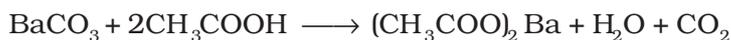
Dissolve the precipitate by boiling with dilute acetic acid and divide the solution into three parts one each for Ba^{2+} , Sr^{2+} and Ca^{2+} ions		
Ba^{2+} ions	Sr^{2+} ions	Ca^{2+} ions
(a) To the first part add potassium chromate solution. A yellow precipitate appears. (b) Perform the flame test with the preserved precipitate. A grassy green flame is obtained.	(a) If barium is absent, take second part of the solution and add ammonium sulphate solution. Heat and scratch the sides of the test tube with a glass rod and cool. A white precipitate is formed. (b) Perform the flame test with the preserved precipitate. A crimson-red flame confirms the presence of Sr^{2+} ions.	(a) If both barium and strontium are absent, take the third part of the solution. Add ammonium oxalate solution and shake well. A white precipitate of calcium oxalate is obtained. (b) Perform the flame test with the preserved precipitate. A brick red flame, which looks greenish-yellow through blue glass, confirms the presence of Ca^{2+} ions.

Chemistry of Confirmatory Tests of Group-V cations

The Group-V cations are precipitated as their carbonates which dissolve in acetic acid due to the formation of corresponding acetates.

1. Test for Barium ion (Ba^{2+})

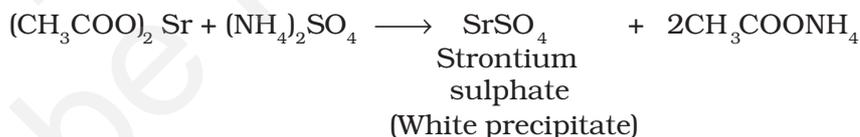
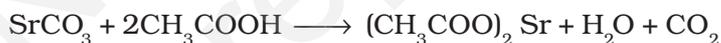
- (a) Potassium chromate (K_2CrO_4) solution gives a yellow precipitate of barium chromate when the solution of fifth group precipitate in acetic acid is treated with it.



- (b) **Flame test** – Take a platinum wire and dip it in conc. HCl. Heat it strongly until the wire does not impart any colour to the non-luminous flame. Now dip the wire in the paste of the (Group-V) precipitate in conc. HCl. Heat it in the flame. A grassy green colour of the flame confirms the presence of Ba^{2+} ions.

2. Test for Strontium ion (Sr^{2+})

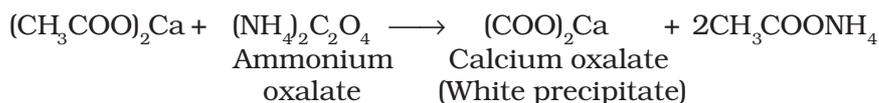
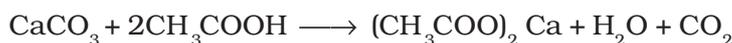
- (a) Solution of V group precipitate in acetic acid gives a white precipitate of strontium sulphate with ammonium sulphate, $(\text{NH}_4)_2\text{SO}_4$, solution on heating and scratching the sides of the test tube with a glass rod.



- (b) **Flame test** – Perform the flame test as given in the case of Ba^{2+} . A crimson red flame confirms the presence of Sr^{2+} ions.

3. Test for Calcium ion (Ca^{2+})

- (a) Solution of the fifth group precipitate in acetic acid gives a white precipitate with ammonium oxalate solution.



- (b) **Flame test** – Perform the flame test as mentioned above. Calcium imparts brick red colour to the flame which looks greenish-yellow through blue glass.

(VII) Analysis of Group-VI cations

If group-V is absent then perform the test for Mg^{2+} ions as given below.

Chemistry of Confirmatory Tests of Group-VI cations**Test for Magnesium ion (Mg^{2+})**

- (a) If group-V is absent the solution may contain magnesium carbonate, which is soluble in water in the presence of ammonium salts because the equilibrium is shifted towards the right hand side.



The concentration of carbonate ions required to produce a precipitate is not attained. When disodium hydrogenphosphate solution is added and the inner walls of the test tube are scratched with a glass rod, a white crystalline precipitate of magnesium ammonium phosphate is formed which indicates the presence of Mg^{2+} ions.



Magnesium ammonium
phosphate (White precipitate)

Note down the observations and the inferences of the qualitative analysis in tabular form as given in the specimen record given in pages 114-115.

Note :

Some times precipitate of magnesium ammonium phosphate appears after some time. So warm the solution and scratch the sides of test tube after adding sodium hydrogen phosphate solution.

Precautions

- Always use an apron, an eye protector and hand gloves while working in the chemistry laboratory.
- Before using any reagent or a chemical, read the label on the bottle carefully. Never use unlabelled reagent.
- Do not mix chemicals and reagents unnecessarily. Never taste any chemical.
- Be careful in smelling chemicals or vapours. Always fan the vapours gently towards your nose (Fig. 7.9).
- Never add sodium metal to water or throw it in the sink or dustbin.
- Always pour acid into water for dilution. Never add water to acid.
- Be careful while heating the test tube. The test tube should never point towards yourself or towards your neighbours while heating or adding a reagent.



Fig. 7.9 : How to smell a gas

- (h) Be careful while dealing with the explosive compounds, inflammable substances, poisonous gases, electric appliances, glass wares, flame and the hot substances.
- (i) Keep your working surroundings clean. Never throw papers and glass in the sink. Always use dustbin for this purpose.
- (j) Always wash your hands after the completion of the laboratory work.
- (k) Always use the reagents in minimum quantity. Use of reagents in excess, not only leads to wastage of chemicals but also causes damage to the environment.



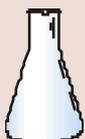
Discussion Questions

- (i) What is the difference between a qualitative and a quantitative analysis?
- (ii) Can we use glass rod instead of platinum wire for performing the flame test? Explain your answer.
- (iii) Why is platinum metal preferred to other metals for the flame test?
- (iv) Name the anions detected with the help of dilute H_2SO_4 ?
- (v) Why is dilute H_2SO_4 preferred over dilute HCl while testing anions?
- (vi) Name the anions detected by conc. H_2SO_4 .
- (vii) How is sodium carbonate extract prepared ?
- (viii) What is lime water and what happens on passing carbon dioxide gas through it?
- (ix) Carbon dioxide gas and sulphur dioxide gas both turn lime water milky. How will you distinguish between the two?
- (x) How will you test the presence of carbonate ion?
- (xi) What is the composition of dark brown ring which is formed at the junction of two layers in the ring test for nitrates?
- (xii) Name the radical confirmed by sodium nitroprusside test.
- (xiii) What is chromyl chloride test ? How do you justify that CrO_2Cl_2 is acidic in nature?
- (xiv) Why do bromides and iodides not give tests similar to chromyl chloride test?
- (xv) Describe the layer test for bromide and iodide ions.

- (xvi) Why is silver nitrate solution stored in dark coloured bottles?
- (xvii) How do you test the presence of sulphide ion?
- (xviii) Why does iodine give a blue colour with starch solution?
- (xix) What is Nessler's reagent?
- (xx) Why is original solution for cations not prepared in conc. HNO_3 or H_2SO_4 ?
- (xxi) Why cannot conc. HCl be used as a group reagent in place of dil. HCl for the precipitation of Ist group cations?
- (xxii) How can one prevent the precipitation of Group-IV radicals, with the second group radicals?
- (xxiii) Why is it essential to boil off H_2S gas before precipitation of radicals of group-III?
- (xxiv) Why is heating with conc. nitric acid done before precipitation of group-III?
- (xxv) Can we use ammonium sulphate instead of ammonium chloride in group-III?
- (xxvi) Why is NH_4OH added before $(\text{NH}_4)_2\text{CO}_3$ solution while precipitating group-V cations?
- (xxvii) Why do we sometimes get a white precipitate in group-VI even if the salt does not contain Mg^{2+} radical?
- (xxviii) What is aqua regia?
- (xxix) Name a cation, which is not obtained from a metal.
- (xxx) How can you test the presence of ammonium ion?
- (xxxii) Why are the group-V radicals tested in the order Ba^{2+} , Sr^{2+} and Ca^{2+} ?
- (xxxiii) Why does conc. HNO_3 kept in a bottle turn yellow in colour?
- (xxxiiii) Why should the solution be concentrated before proceeding to group-V?
- (xxxv) Why is the reagent bottle containing sodium hydroxide solution never stoppered?
- (xxxvi) What do you understand by the term common ion effect?
- (xxxvii) Why is zinc sulphide not precipitated in group-II?

SPECIMEN RECORD OF SALT ANALYSIS**Aim**

To analyse the given salt for one anion and one cation present in it.

Material required

- Boiling tubes, test tubes, test tube holder, test tube stand, delivery tube, corks, filter papers, reagents

Sl. No.	Experiment	Observation	Inference
1.	Noted the colour of the given salt.	White	Cu^{2+} , Fe^{2+} , Ni^{2+} , Co^{2+} , Mn^{2+} are absent.
2.	Noted the smell of the salt.	No specific smell.	S^{2-} , SO_3^{2-} , CH_3COO^- may be absent.
3.	Heated 0.5 g of the salt in a dry test tube and noted the colour of the gas evolved and change in the colour of the residue on heating and cooling.	(i) No gas was evolved. (ii) No particular change in colour of the residue is observed when heated and when cooled.	(i) CO_3^{2-} may be present, NO_3^- , NO_2^- , Br^- may be absent. (ii) Zn^{2+} may be absent.
4.	Prepared a paste of the salt with conc. HCl and performed the flame test.	No distinct colour of the flame seen.	Ca^{2+} , Sr^{2+} , Ba^{2+} , Cu^{2+} may be absent.
5.	Borax bead test was not performed as the salt was white in colour.	—	—
6.	Treated 0.1 g of salt with 1 mL dil. H_2SO_4 and warmed.	No effervescence and evolution of vapours.	CO_3^{2-} , SO_3^{2-} , S^{2-} , NO_2^- , CH_3COO^- absent.
7.	Heated 0.1 g of salt with 1 mL conc. H_2SO_4 .	No gas evolved.	Cl^- , Br^- , I^- , NO_3^- , $\text{C}_2\text{O}_4^{2-}$ are absent.
8.	Acidified 1 mL of aqueous salt solution with conc. HNO_3 . Warmed the contents and then added 4-5 drops of ammonium molybdate solution.	No yellow precipitate	PO_4^{3-} absent.

9.	Acidified water extract of the salt with dil. HCl and then added 2mL of BaCl ₂ solution.	A white ppt. is obtained which is insoluble in conc. HNO ₃ and conc. HCl.	SO ₄ ²⁻ present.
10.	Heated 0.1 g of salt with 2 mL NaOH solution.	Ammonia gas is not evolved.	NH ₄ ⁺ absent.
11.	Attempted to prepare original solution of the salt by dissolving 1g of it in 20 mL water.	Clear solution formed	Water soluble salt is present.
12.	To a small part of the above salt solution added 2 mL of dil. HCl.	No white precipitate formed.	Group-I absent.
13.	Passed H ₂ S gas through one portion of the solution of step 12.	No precipitate formed.	Group-II absent.
14.	Since salt is white, heating with conc. HNO ₃ is not required. Added about 0.2 g of solid ammonium chloride and then added excess of ammonium hydroxide to the solution of step 12.	No precipitate formed.	Group-III absent.
15.	Passed H ₂ S gas through the above solution.	No precipitate formed.	Group-IV absent.
16.	Added excess of ammonium hydroxide solution to the original solution and then added 0.5 g of ammonium carbonate.	No precipitate formed.	Group-V absent.
17.	To the original solution of salt added ammonium hydroxide solution, followed by disodium hydrogen phosphate solution. Heated and scratched the sides of the test tube.	White precipitate.	Mg ²⁺ confirmed.

Result

The given salt contains:

Anion : SO₄²⁻

Cation : Mg²⁺

PROJECTS

BACKGROUND INFORMATION ABOUT INVESTIGATORY PROJECTS

INTRODUCTION

The expansion of scientific knowledge and consequently the change in the system of education has led to a modification in the methods of instruction. Today the stress is laid on inquiry approach and discussion method instead of the age-old lecture method of teaching. A new dimension in the teaching of science has been added by including the project-work at the higher secondary level. Teaching through project work is an individualized instructional technique. It provides an opportunity to the student to define a problem, to plan his/her work, to search appropriate resources, to carry out his/her plans, and to draw conclusions. This way, students are exposed to the fundamental scientific principles, methods and processes and get a first hand knowledge about the various phases involved in a scientific investigation. Thus the project work helps to: (a) stimulate interest in science; (b) arouse scientific curiosity; (c) develop independent critical thinking; (d) provide experience of using various tools and techniques in the field of science and (e) develop self-confidence. The modern trend in the teaching of science therefore encourages more project work.

Any kind of investigation; formulated, designed and carried out in the library, laboratory, in the field, or at home constitutes an investigatory project. A project may be as simple as collection of samples of minerals and it may be as difficult as developing an original indigenous process for the production of a chemical. Some of the projects can be completely theoretical and involve only the library work. Others may involve the experimental work to be carried out in the laboratory. The experimental work in science exposes the students to a number of scientific instruments, tools, techniques and intellectual skills.

SELECTION OF PROJECTS

Projects should usually be selected by the students. The idea of a project originates from studying a subject in the classroom, reading the reports of various projects, science news, popular science articles from science journals etc. Sometimes the idea of a science project may strike during classroom discussion on topics, which may require testing, quantifying and interpreting. Some of the science journals for getting the ideas for a project are : (a) Journal of Chemical Education; (b) Chemistry Education; (c) Education in Chemistry; (d) New Scientist; (e) School

Science; (f) School Science Review; (g) Science; (h) Scientific American; (i) School Science Resource Letter, etc. Once the project work begins, it might provide new titles and ideas.

In spite of the availability of the above listed scientific literature, it should not be assumed that students can begin the work on projects spontaneously. Since majority of the science journals listed above are not available in Indian Schools. Students need the help and guidance from the teacher. If some student does not get an idea for the project, the teacher may provide a list of suggested projects or take the students to science fairs and exhibitions to show what other students are doing. A format for working on the projects is given below:

1. Title of Project
2. Objectives and importance of Project
3. A brief outline of work on Project

The title of the project should be written in such a manner that the objectives and importance of the project is clearly defined. In other words a project title and its objectives should create an interest and curiosity. A 'brief outline of working on a project' helps the student to initiate the work.

It may be debatable that this approach of suggesting ideas for the projects defeats one of the basic objectives of project work, namely originality, but providing guidelines to the students is completely scientific and desirable to encourage every student to begin and aim at something in the first attempt.

MANAGING TIME

Central Board of Secondary Education has allocated ten periods for the project work. A student can start the project right in the beginning of the academic year and can carry it out in phases and submit the report of the project at the end of the year.

TECHNICAL AND ACADEMIC GUIDANCE

This is an important factor in the smooth running of the project work. The student should plan for the project well in advance and discuss its design with the teacher. If improvisation of the apparatus or some instrument is needed or a chemical is not available in the laboratory. The help of teacher may be taken. If some technical or academic guidance is required help may be taken not only from the concerned teacher of chemistry but also from physics from and other science teachers as well.

LABORATORY FACILITIES

The selection of the project should be such that as far as possible, the material (apparatus, instruments, chemicals, etc.) needed for the project-work is easily available. Student may purchase the affordable chemical or an apparatus (improvised or original) if it is not available in the laboratory, and the student is very curious to take up the project and wants to do it. The students should be discouraged from taking highly expensive projects. An effective project-work requires an integrated approach rather than a subject specific approach.

The project work in the laboratory requires a bigger and separate space. Arrangements should be made in such a way that at a given time all the students are not involved in doing the laboratory work. Some students should carry out the work of collecting references in the library while others should design experiments.

Problems may arise in carrying out the long term experiments like corrosion, fermentation etc. in the laboratory. It is suggested to have a separate bench in the laboratory, where long term experiments can be set up. For storing the samples of certain chemicals and apparatuses relevant to the project-work, cardboard boxes, with student's name written on them can be used. Empty bottles, if available, can also serve the purpose of storing the chemicals.

RECORDING THE PROJECT WORK

Recording the actual observation in the project work is very essential. Students should be encouraged to record the negative results also. A general format for writing the project report is suggested below. It should involve the following points :

1. Title of project reflecting objectives
2. Principles used for Investigation
3. Apparatus and Chemicals required
4. Improvisation, if any
5. Procedure
6. Observations and Calculations
7. Conclusions and the logic upon which the conclusions are based
8. Precautions
9. Suggestions for further investigations, if any

To illustrate the format outlined above, a 'sample project' report is presented in the end. It may further be noted that sample project report serves merely as a guideline to the

students while writing their projects. It is by no means exhaustive and is open to further improvements. Brief outline of some projects is given below.

PROJECT 1

Title

To test the contamination of water by bacteria by checking the sulphide ion concentration and find out the cause of contamination.

Objective

To check the bacterial contamination in samples of water collected from different sources by determining sulphide ion concentration.

Background Information

Sulphide ions are present in water when anaerobic bacteria decompose organic matter or reduce sulphates. These are found in stagnant water. Generally pollutants from paper mills, gas works, tanneries, sewage works and other chemical plants are responsible for the growth of such bacteria.

Brief Procedure

Collection of Samples

Sulphides are readily oxidised, therefore care should be taken at the time of sampling to exclude air by flushing it with nitrogen or carbon dioxide. But the best way is to 'fix' the sample immediately after collection. This can be done by adding small volume of cadmium-zinc acetate solution. For this take 80 mL of water and add cadmium-zinc acetate solution 20 mL to obtain a total volume of about 100 mL. To make Cd-Zn acetate solution dissolve 50 g cadmium acetate and 50 g zinc acetate in 1.0 L of water. If collected sample is acidic in nature, then first neutralize it with little excess of alkali.

Titration of Fixed Solution

Take 100 mL fixed solution in a titration flask, add 20 mL 0.025 M iodine solution and immediately add 15 mL, (1:1) HCl and mix. Titrate the excess iodine against 0.05 M $\text{Na}_2\text{S}_2\text{O}_3$, adding starch solution as indicator towards the end point. Calculate the amount of sulphide ions in the original samples from the amount of iodine used in the reaction with H_2S . Subtract the values of blank titration if available from the calculated values.

PROJECT 2**Title**

To study the methods of purification of water.

Objectives

- To study level of purity achieved by using different methods of purification.
- To study advantages and disadvantages of using different method for purification.
- To know about specific uses of pure water.

Background Information

Purity of water obtained from different natural sources is different. The type of contamination and impurity present depends upon the source from which water is obtained. Besides drinking purposes, we require pure water for various other purposes e.g. in chemical analysis. There are various methods for the purification of water. These remove impurities and contamination to different extent. There are some advantages and disadvantages in using these methods. Comparison of various methods of purification will provide an idea about obtaining water of specific purity for a specific purpose.

Brief Procedure

Students may find out level of purity achieved by various techniques in use, for the purification of drinking water. They can survey the literature and visit industries etc. to find out uses of water of specific purity. Students may work in groups for the study of various aspects of the project.

Note : Another aspect of the project may be to study different methods of purification of water taken from different sources such as river, well, bore-well, municipality etc.

PROJECT 3**Title**

Testing the hardness, presence of iron, fluoride, chloride etc. in drinking water obtained from different regions and a study of the cause of presence of these ions above permissible limits.

Objectives

- To test the total hardness, iron, fluoride and chloride etc. in different samples of water.
- To collect information about local sources of above ions in water.
- To study the effect of these ions on health if present beyond permissible limits.
- To find out whether any such problem exists in the locality and around.

Background Information

Quality of drinking water has direct relationship with the human health and life. If iron, fluoride, chloride etc. are present in water above permissible limits, they may cause several health problems. For example, if fluoride is present above permissible limit, people of the region may suffer from fluorosis. Hardness of water is due to the presence of calcium and magnesium ions. It is well known fact that hard water is not fit for laundry purposes. Thus, it is very important to know the ions and their amount present in water.

Brief Procedure

Students may collect samples of water from different sources. They can detect the presence of different ions by usual methods of analysis. Total hardness of water can be estimated by standard procedure of complexometric titration. Estimation of Cl^- , F^- and Fe^{2+} is difficult at this level. Therefore existing data from approved labs can be taken for the purpose of investigation.

PROJECT 4

Title

Investigation of the foaming capacity of different washing soaps and the effect of addition of sodium carbonate on their foaming capacity.

Objective

To study the foaming capacity of soaps and the effect of addition of sodium carbonate on their foaming capacity.

Brief Procedure

Weigh 1 gram of a sample of soap and dissolve it completely in 100 mL of distilled water. Take 10 mL of the soap solution in a boiling tube close the mouth of the boiling tube with the help of a cork and shake the solution making 20 regular strokes so that foam increases uniformly. Measure the length of the boiling tube up to which the foam rises. Similarly, perform the experiment with other soap solutions.

Dissolve 0.5g of sodium carbonate in 50 mL of each of the above soap solutions separately. Now take 10 mL of a solution in a boiling tube and shake it equal number of times (e.g. 20 regular strokes). Measure the length up to which foam appears. Similarly, perform the experiment with other soap solutions. Record the observations in a tabular form.

Compare the height up to which foam produced rises in different soap solutions with and without the presence of Na_2CO_3 and draw conclusions.

PROJECT 5

Title

Study of the acidity of different samples of tea leaves and reasons for the variation in colour of tea prepared from these leaves.

Objective

To estimate the concentration of acids present in different tea samples and the effect of addition of acids or bases on the colour of tea extract.

Brief Procedure

(a) Estimation of Concentration of Acids Present in Tea

Weigh 10 grams of the sample of tea leaves and prepare the extract of each sample separately in 200 mL of distilled water. For this, boil different samples of tea leaves with distilled water for a fixed time period.

Take 5mL of tea extract in a conical flask and dilute it with 20 mL of distilled water. Shake the solution well for homogenous mixing and then titrate it against M/50 NaOH solution using phenolphthalein as an indicator. Similarly, titrate other tea

extracts with M/50 NaOH solution. Calculate the concentration of acids present in different samples of tea leaves in terms of molarity. If colour of the extract causes problem, then tea extract can be taken in the burette and sodium hydroxide solution in the conical flask. Phenolphthalein may be used as an indicator if sodium hydroxide solution is taken in conical flask. The colour change will be from pink to colourless.

(b) Effect of Acids and Bases on the Colour of Tea Extract

Take five filter paper strips and mark them as A, B, C, D and E. Dip all the strips in any one sample of tea extract and then take them out. Now put two drops of dilute HCl, acetic acid solution, NaOH solution and NH_4OH solution on strips A, B, C and D respectively. Compare the change in colour of these strips with reference to the colour of the strip E. Repeat this experiment with other samples of tea extract.

PROJECT 6

Title

Study the rates of evaporation of different liquids

Objective

To study the relationship between the rates of evaporation of different liquids and their chemical constitution.

Brief Procedure

Take five clean and dry weighing tubes and mark them as A, B, C, D and E. Weigh each weighing tube with its stopper. Now pour 10 mL of different liquids (ethanol, ether, tetrachloromethane, acetone etc.) in different weighing tubes. Weigh each weighing tube again and find the mass of the liquid taken in each weighing tube.

Remove the stoppers of the weighing tubes and keep them at room temperature for one hour. After exactly one hour, close the mouth of all the weighing tubes with their stoppers and weigh them again one by one.

Calculate the loss in mass of each liquid. The temperature and the surface area should be the same for the evaporation of each liquid. Determine the rate of evaporation of each liquid in grams of liquid evaporated per second. Relate the difference in rates of evaporation of liquids with their chemical constitution and variation in intermolecular/intra-molecular interactions.

PROJECT 7**Title**

Study the effect of acids and alkalies on the tensile strength of fibres

Objective

To study the effect of acids on the tensile strength of different types of fibers.

Brief Procedure

The tensile strength of a fibre is measured by noting the minimum weight required just to break the thread. It may be done as follows:

Take a thread of about 20 cm length and tie its one end with a ring fixed on the iron stand and the other end with a hanger which carries the weights. Increase the weight on the hanger and find out the minimum weight required to just break the thread. Repeat the experiment with threads of equal length and thickness but of different materials (eg. cotton, wool, silk, terylene etc.). These weights are the measure of tensile strength of the fibre.

The effect of acids and alkalies on the tensile strength of fibres can be determined by dipping them separately in dilute HCl or dilute NaOH solution of equal strengths for equal intervals of time. After a small but fixed interval of time, the fibres are removed from the solution, washed with water and dried. Then minimum weight required to just break these threads are determined. These weights are the measures of the tensile strengths of fibres after treatment with acid or alkali. Interpret your observations in terms of chemical constitution of the material of fibre.

PROJECT 8**Title**

Study of the acids and mineral contents of vegetables and fruits.

Objective

- (a) To determine the amount of acids present in various vegetables and fruits.
- (b) To detect the presence of iron, carbohydrate, protein and sugar etc. in vegetables and fruits.

Brief Procedure

(a) Acid Content

Take out the juice of a few fruits and vegetables (apple, orange, amla, lemon, raddish, cane sugar etc.) by crushing them. Keep the juice samples in different containers. Determine the pH of different samples of juices. Determine their acid content by titrating a known quantity of juice with M/100 potassium hydroxide solution using phenolphthalein as an indicator. In case of dark coloured juices, dilute them with enough distilled water to get sharp end point during titration.

Compare the acid contents of juices by comparing their acid values. Acid value of vegetables and fruits is the number of milligrams of potassium hydroxide required for neutralizing the acids present in one gram of vegetable/fruit.

(b) Tests for Iron, Carbohydrate (starch and sugar), Protein and Fats

Test the vegetables and fruit juices for the presence of iron. Heat the vegetable juice with concentrated HNO_3 for some time and perform the test for iron. Carbohydrate (starch, sugar), protein and fats can be tested by the usual tests.

SAMPLE PROJECT REPORT

Title

A study of the variation of viscosity of organic compounds of same homologous series with variation in the (a) molecular masses and (b) structures of carbon chains.

Background Information

Some liquids like honey or Mobil oil flow very slowly while others like water or kerosene flow rapidly. Liquids that flow slowly are known as viscous liquids while others that flow rapidly are known as non-viscous liquids. The resistance offered by a liquid to flow is known as viscosity. It is related to intermolecular forces existing in a liquid. Different liquids have different values of viscosity due to the existence of different magnitudes of intermolecular forces. The comparison of viscosities of various homologues and isomers in a particular homologous series would give an idea about the magnitude of intermolecular forces existing in them.

Objectives

The objectives of this project are to establish a relationship between (a) viscosity and molecular masses; and (b) viscosity and nature of carbon chain in organic compounds.

Principle Involved

The resistance to flow offered by a liquid is measured in terms of coefficient of viscosity which is defined as follows:

'Coefficient of viscosity of a liquid at a specified temperature is the steady force required to maintain a velocity difference of unity between two parallel layers of a liquid, a unit distance apart and having a unit area of contact'. Coefficient of viscosity is measured by Ostwald viscometer method. For two liquids whose coefficient of viscosity are η_1 and η_2 , the time of flow in seconds are t_1 and t_2 and densities are d_1 and d_2 respectively then the following relation holds:

$$\frac{\eta_1}{\eta_2} = \frac{d_1 \times t_1}{d_2 \times t_2}$$

Hence if the viscosity of one liquid is known, the viscosity of other can be determined.

Hazard Warning

- Acetone and alcohols are inflammable, do not let the bottles open when not in use.
- Keep the bottles away from flames.
- Wash your hands after use.
- Wear safety spectacles.

Material required

Ostwald viscometer, stop-watch, beaker (250 mL), pipette, graduated cylinder, kerosene, petrol, diesel, methyl alcohol, ethyl alcohol, propyl alcohol, isopropyl alcohol, butyl alcohol, isobutyl alcohol, tert butyl alcohol and amyl alcohol.

Brief Procedure

The viscometer was washed, rinsed with alcohol and dried. 10 mL of the liquid under investigation was filled in it and the time required for the flow of liquid between two marks of viscometer was noted with the help of a stop watch. These observation were recorded in Tables I and II. The viscosities of various liquids were calculated by the formula discussed under the heading principles involved.

Observations and Calculations

Room Temperature = 23°C.

A viscometer of different capacity was used for alcohols.

Table 1 : Data for the time of flow of various compounds

Sl. No.	Name of the compound	Time of flow (seconds)	Density (g/mL)	Viscosity (millipoise)
1.	Water	13.5	1	10.08
2.	Petrol	8.5	0.8	6.4
3.	Kerosene Oil	22.0	1	16.4
4.	Dieslel Oil	48.0	1	18.0

Table 2 : Data for the viscosity vs molecular mass relationship of various compounds

Sl. No.	Name of the compound	Molecular mass	Time of flow (Seconds)	Density in (g/mL)	Viscosity (millipoise)
1.	Water	18	180	1	10.08
2.	Methanol	32	136	0.79	7.6
3.	Ethanol	46	258	0.78	14.4
4.	Propan-1-ol	60	391	0.8	21.9
5.	Propan-2-ol	60	546	0.79	30.6
6.	Butan-1-ol	74	612	0.81	34.3
7.	Butan-2-ol	74	686	0.80	38.4
8.	2-Methlprapan-1-ol	74	1406	0.79	78.8
9.	Pentan-1-ol	88	784	0.817	43.9

Note : * If the homologues/isomers of alcohols are not available, other suitable compounds, which are available or are easily manageable can be used for this study.

** Time of flow recorded in the tables are specific for a viscometer and should not be taken as standard values.

Conclusion

As seen from the Table 1, the viscosities of various hydrocarbon fractions, i.e. petrol, kerosene and diesel oil are on an average 6.4, 16.4 and 18.0 respectively. Since the molecular mass of these fractions increases from petrol to diesel oil, this indicates that viscosity increases with increase in molecular mass. The intermolecular attractions tend to increase with increase in molecular mass.

In the case of alcohols, the viscosity of nine alcohols were determined and their values are given in Table-II. The viscosity of alcohols increases with an increase in the molecular mass as can be seen from the viscosities of methanol, ethanol, propan-1-ol, butan-1-ol, the viscosities are 34.3, 38.4 and 78.8 millipoise respectively. This shows that viscosity increases with the increase in molecular mass.

Precaution

The viscometer should be thoroughly cleaned and dried before use.

Suggestions for further investigation

A study of the variation of viscosity with intermolecular forces may be carried out using appropriate compounds.

References

KEENAN, C.W.; WOOD, J.H. *General Chemistry IVth Edition.*, Harper and Row Publishers Inc. New York.