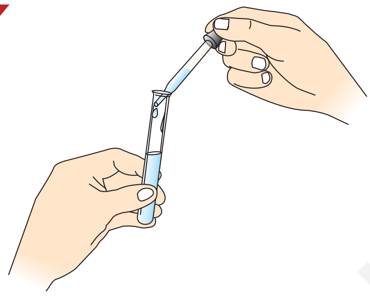
Unit-1

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 workout 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 exhaution 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 aquire 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.

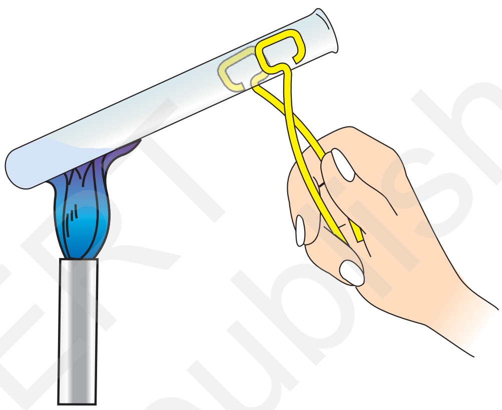


Fig. 1.1 : *Correct method of heating solution in a test tube*

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 :

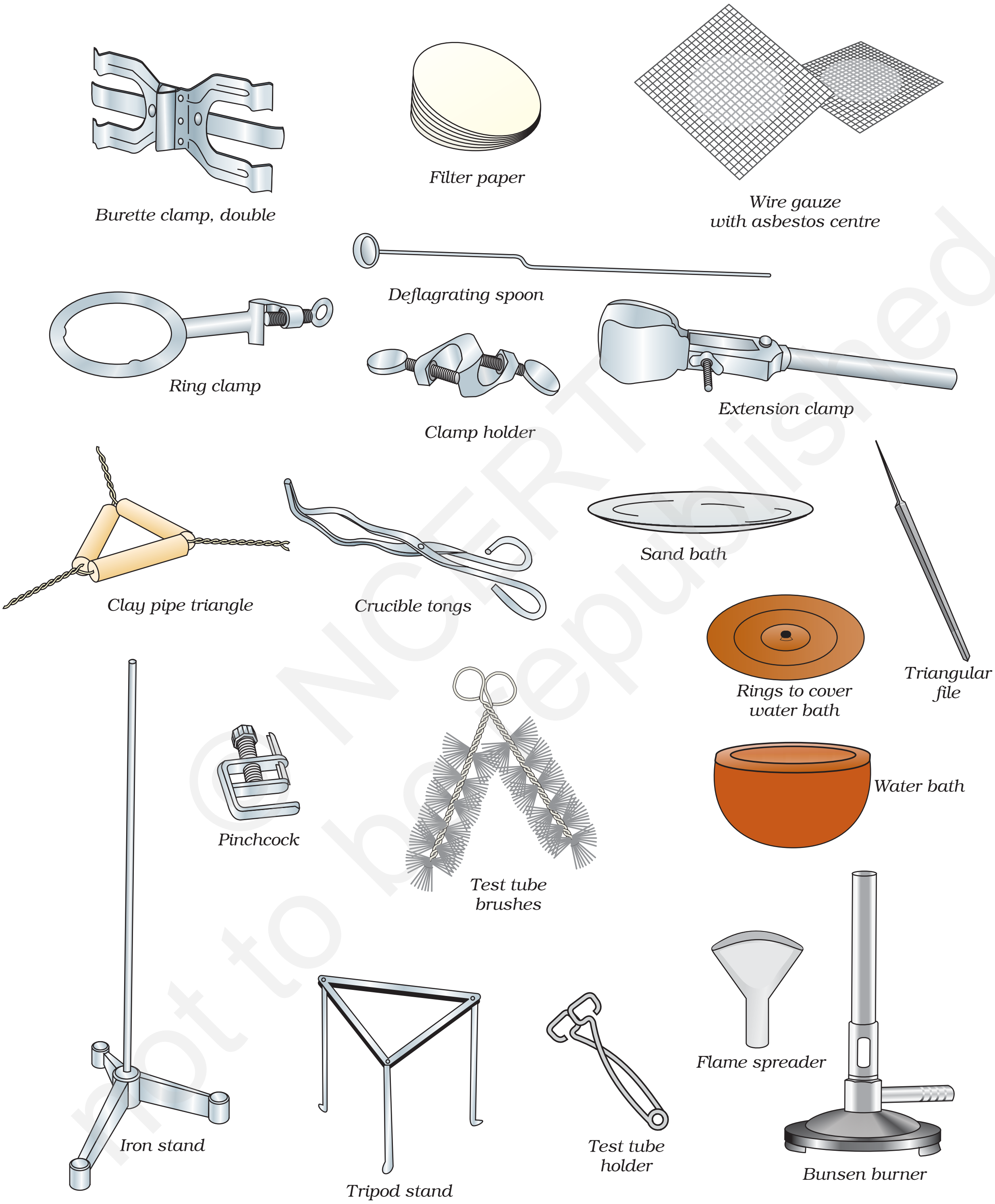


Fig. 1.2 : *Common laboratory equipments*

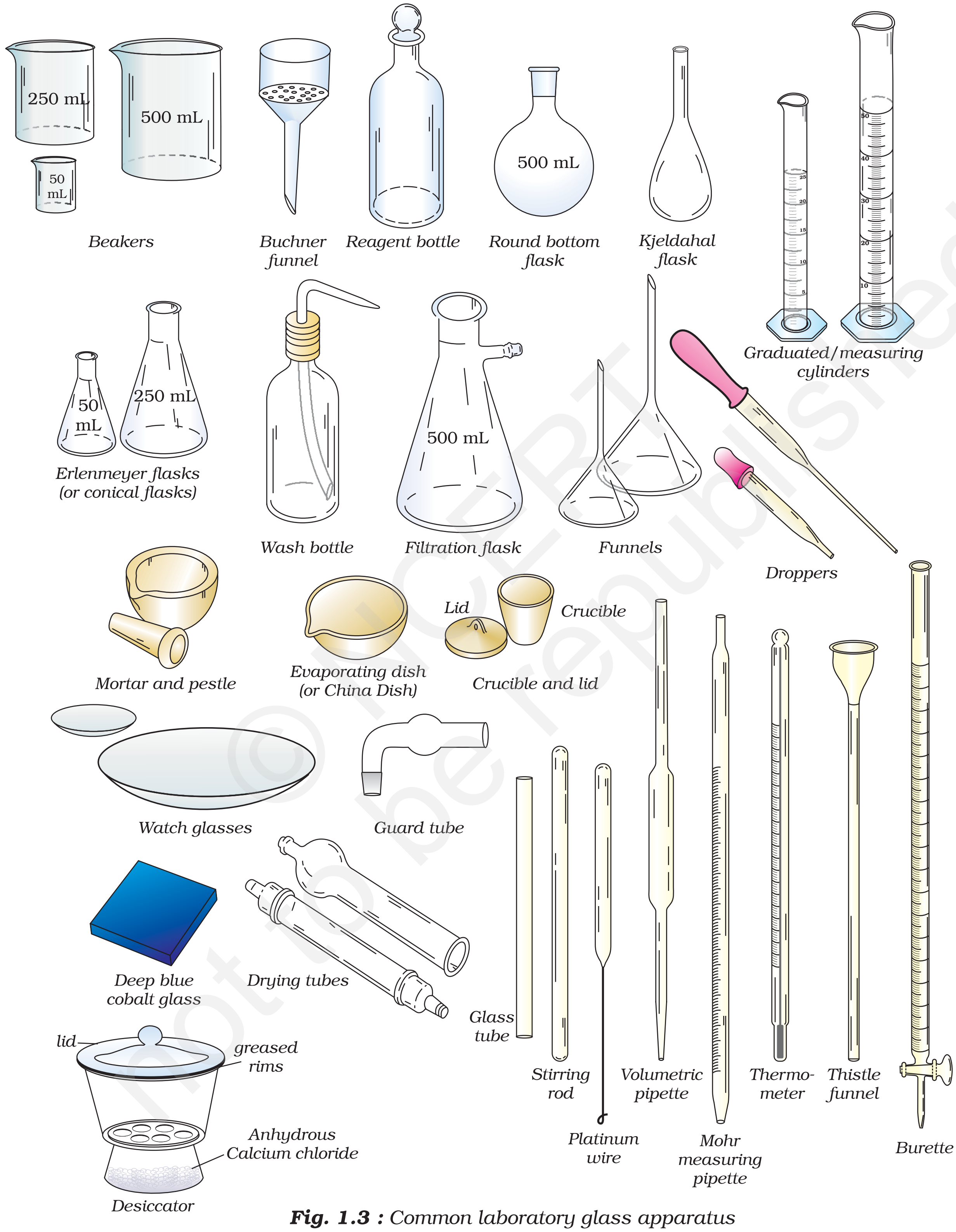


Fig. 1.3 : *Common laboratory glass apparatus*

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).

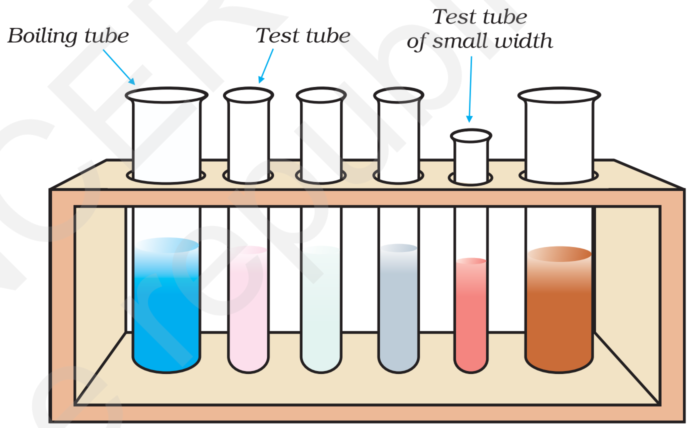


Fig. 1.4 : *Stand carrying boiling tubes and test tubes of different sizes*

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).

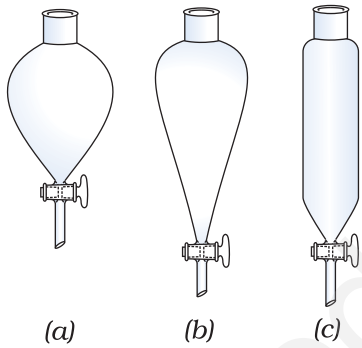


Fig. 1.5 : *Separating funnels of various shapes*

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.

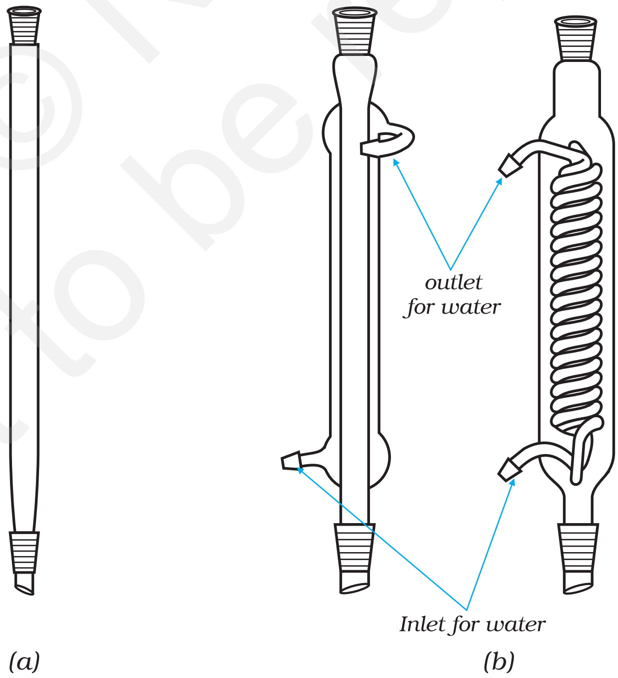


Fig. 1.6 : *(a) Air condenser*
(b) Water condensers

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.

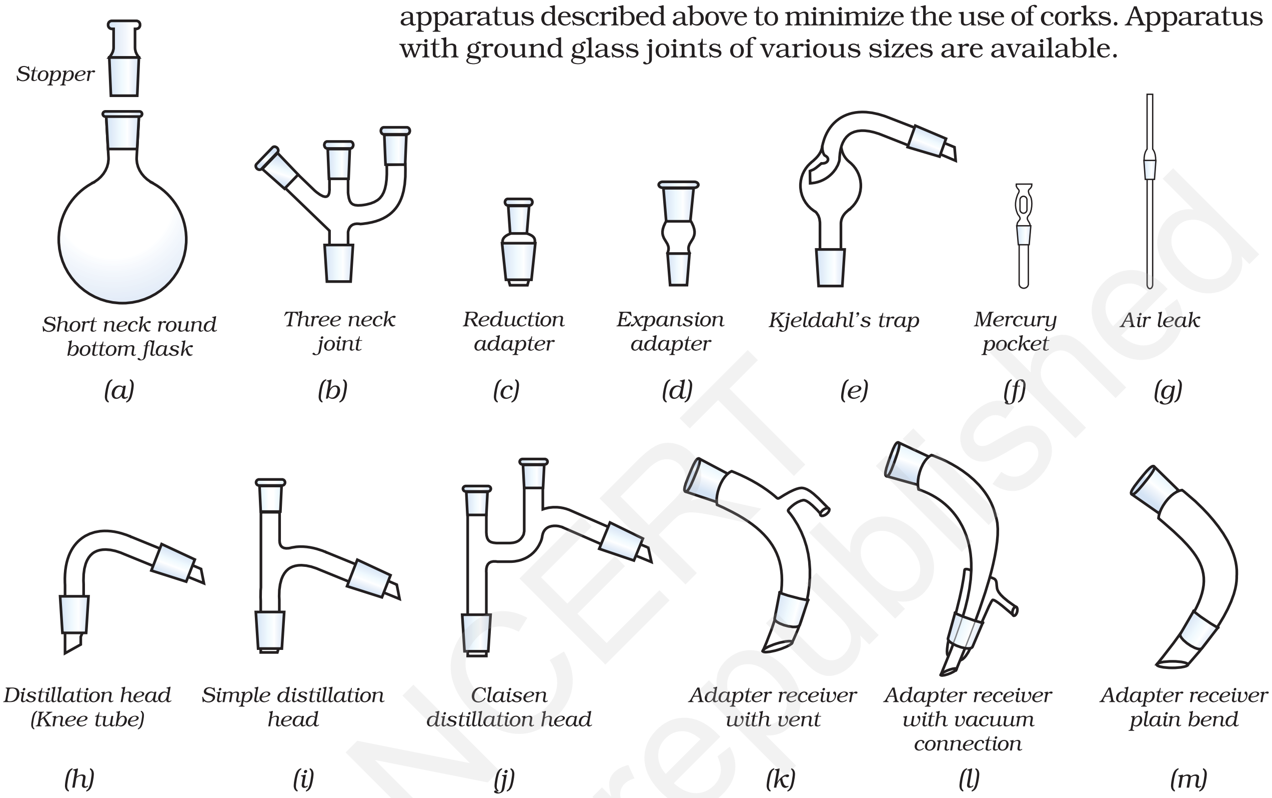


Fig. 1.7 : *Apparatus with ground glass joints*

1.4 HANDLING REAGENT BOTTLES

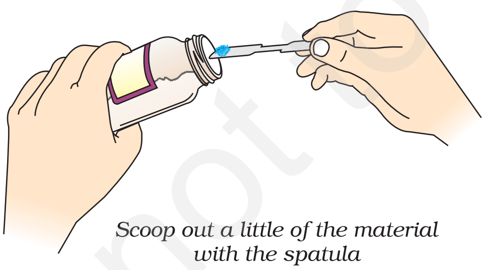
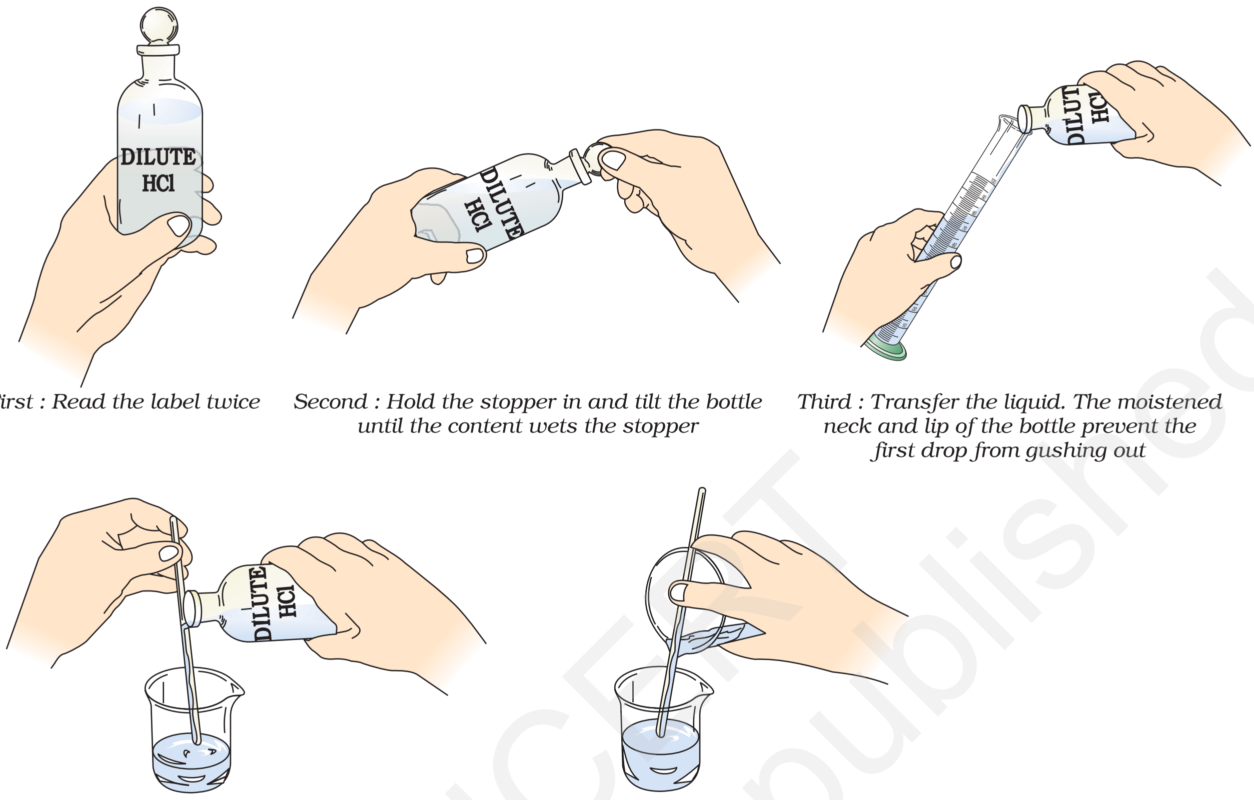


Fig. 1.8 : *Method for taking out solid from the reagent bottle*

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.

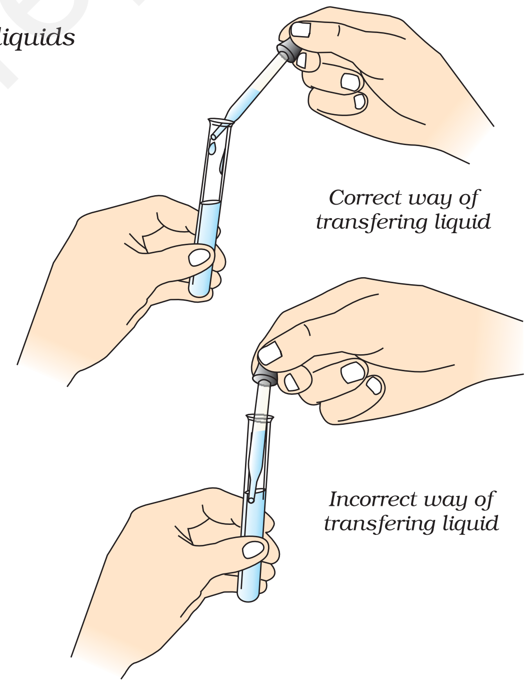


Fig. 1.10 : *Transferring liquid through dropper*

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 :

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.

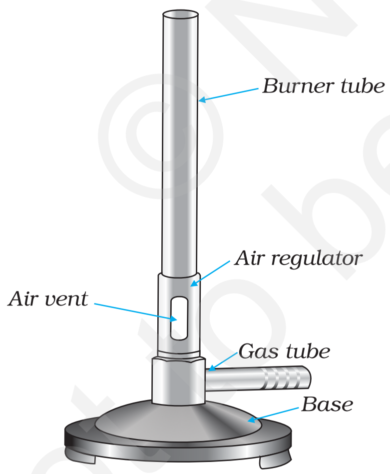


Fig. 1.11 : *Bunsen burner*

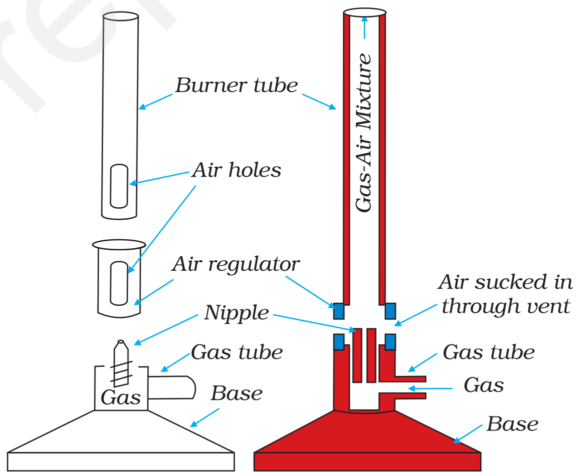


Fig. 1.12 : *Parts of Bunsen burner*

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:

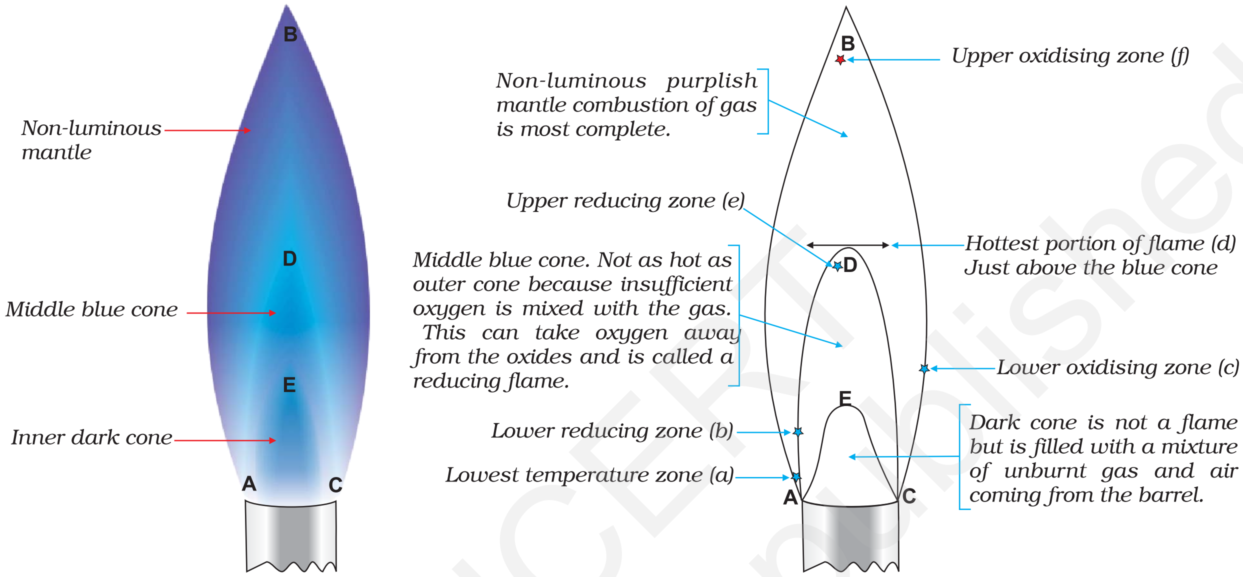


Fig. 1.13 : *Zones of flame of Bunsen burner*

(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 devise 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 upto 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**.

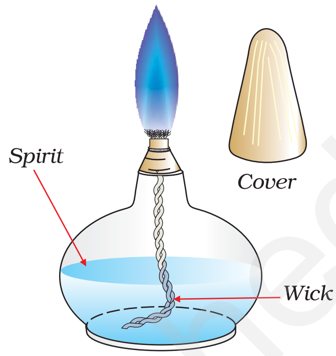


Fig. 1.14 : *The spirit lamp*

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 whereever 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.

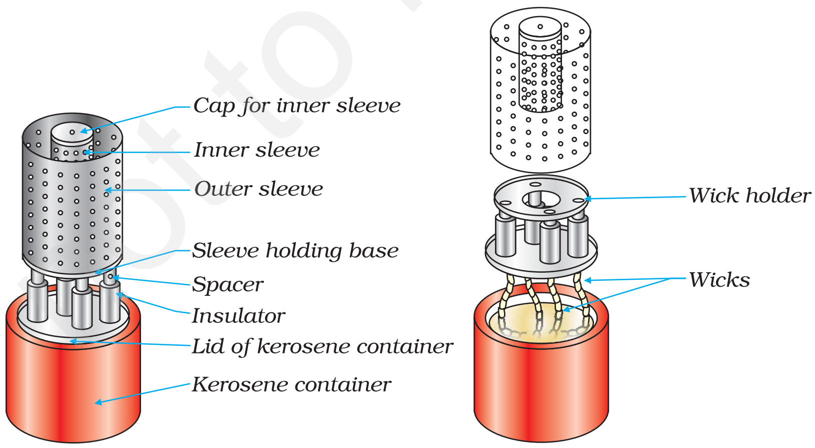


Fig. 1.15 : *Parts of Kerosene Heating Lamp*