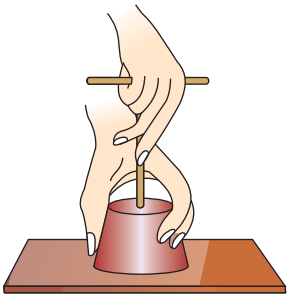
UNIT-2

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).

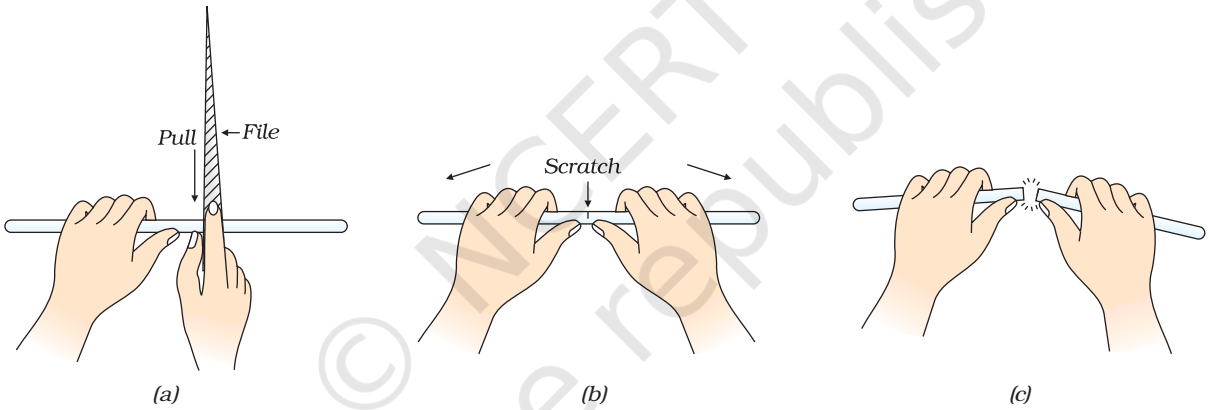


Fig. 2.1 : *(a) Marking a glass rod or a glass tube*
(b) Placing the thumbs together opposite to the scratch
(c) Breaking the glass rod or glass tube

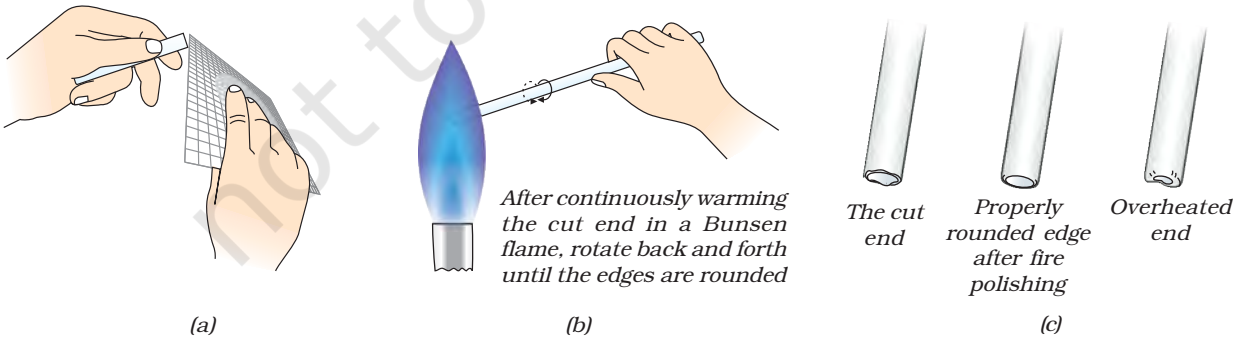


Fig. 2.2 : *(a) Trimming the jagged edges*
(b) Rounding the edges
(c) Properly and improperly rounded edges

Precautions

(a) Make a single deep scratch at the desired length with one stroke of the file.

(b) 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

(i) Why does glass not possess a sharp melting point?

(ii) 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

(i) Cut a tube of desired length with the help of a triangular file as described in section 2.1.

(ii) 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).

(iii) 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).

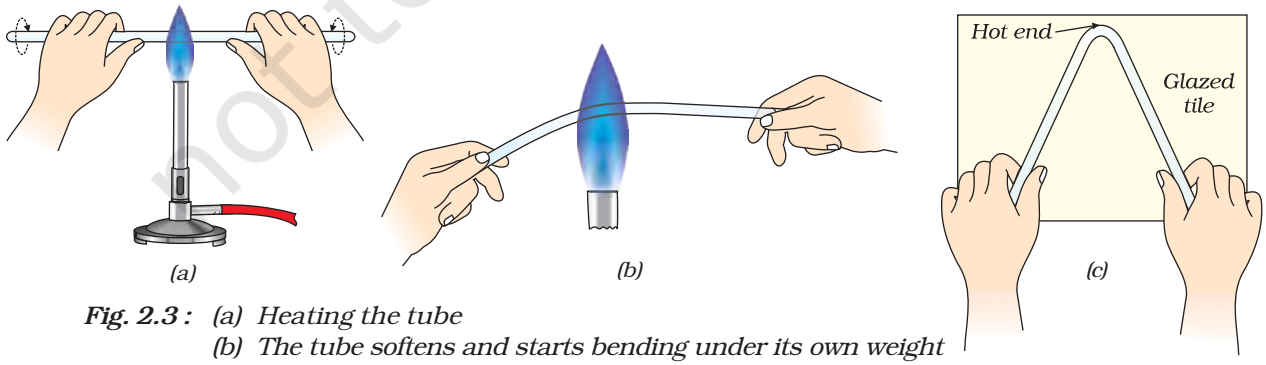


Fig. 2.3 : (a) Heating the tube
(b) The tube softens and starts bending under its own weight
(c) Making the bend coplanar

(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.

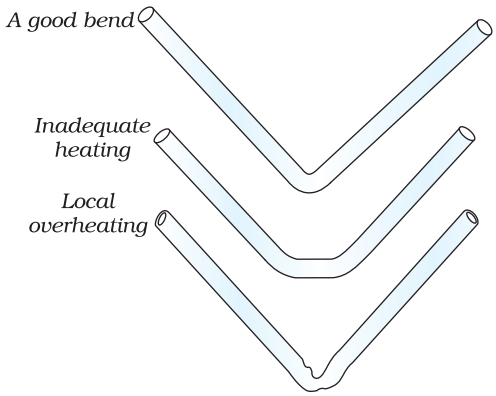


Fig. 2.4 : A glance of proper and improper bends

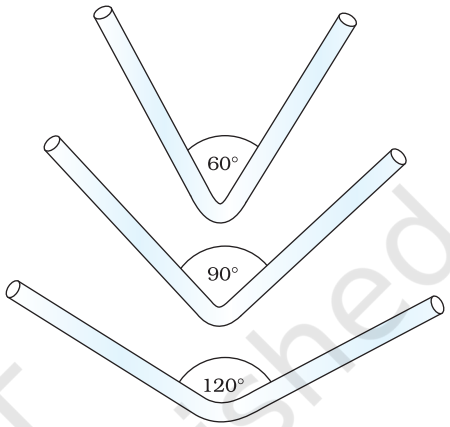


Fig. 2.5 : Bend drawn at various angles

Precautions

(a) Avoid heating the glass tube only on one side, rather rotate it while heating.

(b) Select a glass tube of appropriate length (nearly 30 cm long) to keep your hands safe from heat.

(c) To avoid flattening of the glass tube while bending, carry out the process slowly.

Discussion Questions

(i) Why should the tube be rotated while heating?

(ii) 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.

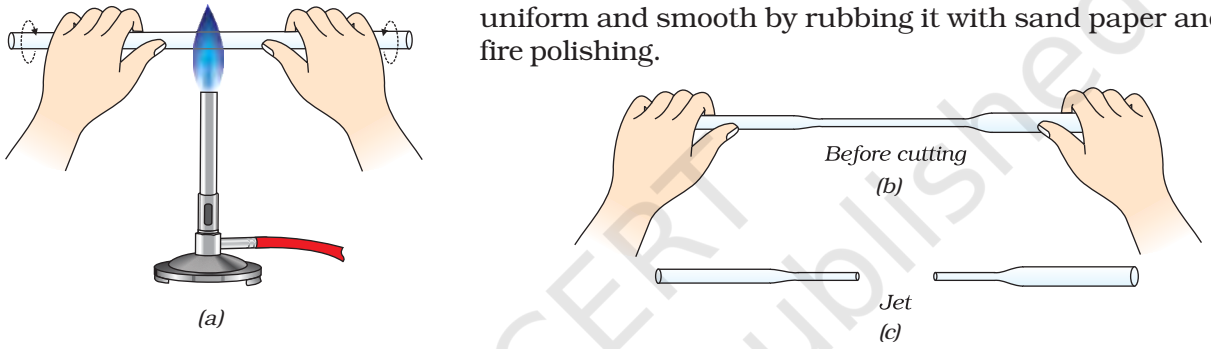


Fig. 2.6 : (a) Heating the tube to draw a jet (b) Before cutting (c) After cutting

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.

(v) For making two holes in the same cork, keep appropriate distance between the holes and use borers of proper size.

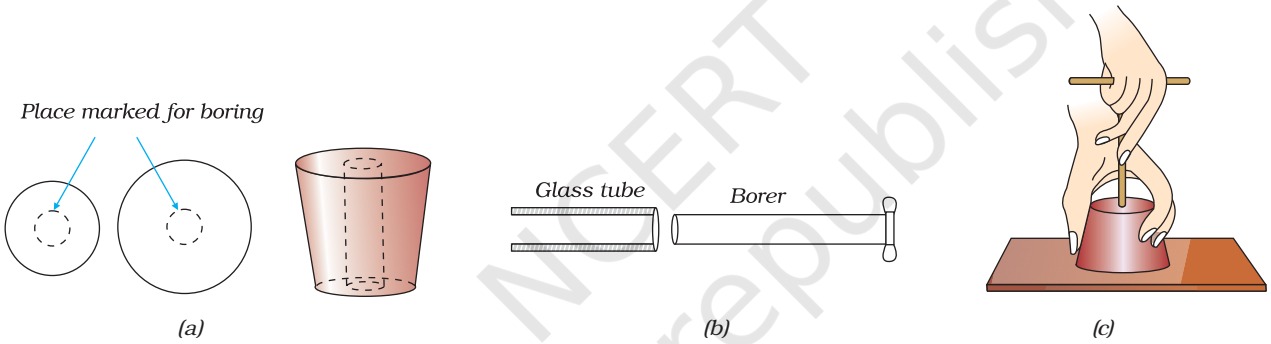


Fig. 2.7 : (a) Marked cork (b) Choosing the borer (c) Boring process

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?

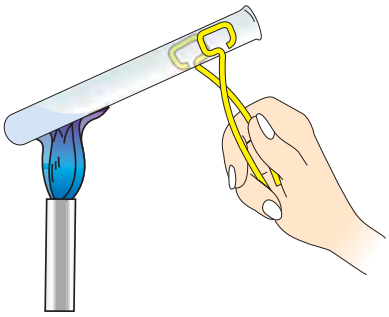


Fig. 2.8 : Heating solution in a test tube

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.

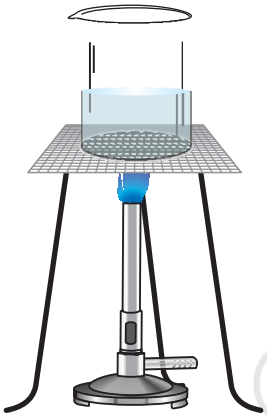


Fig. 2.9 : Heating solution in a beaker

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.

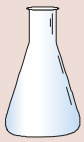
Note : *(i) Never heat the apparatus with thick walls because it may break. Borosilicate glass apparatus is usually used for heating substances.*

(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.

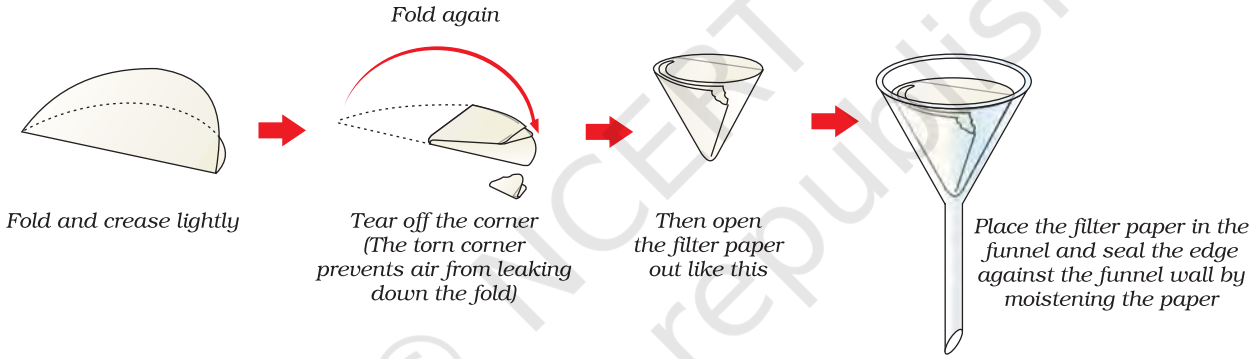


Fig. 2.10 : *Folding the filter paper and placing it in the funnel*

(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).

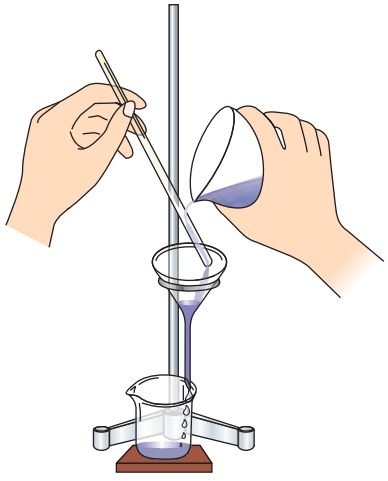


Fig. 2.11 : *Process of filtration*

Precautions

(a) 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.

(b) 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).*

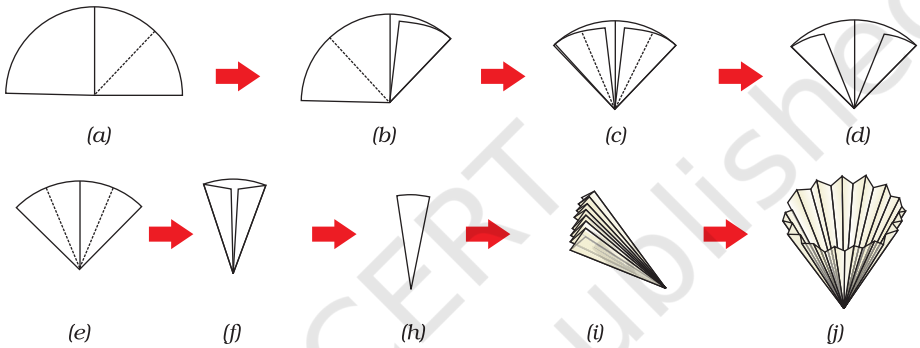


Fig. 2.12 : *Folding the filter paper to get a fluted filter paper cone*

(ii) 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.

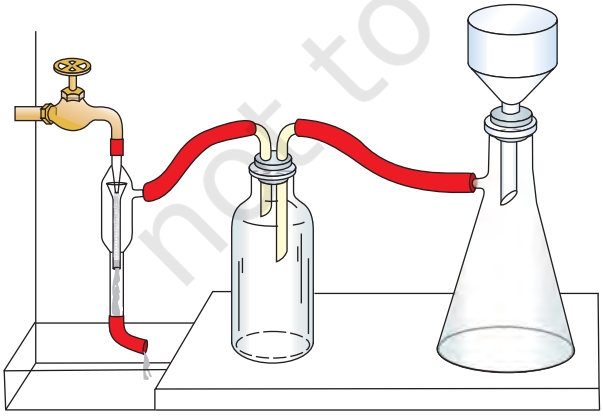


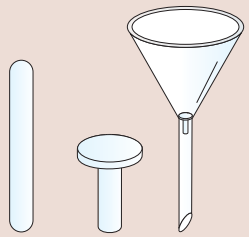
Fig. 2.13 : *Suction filtration*

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

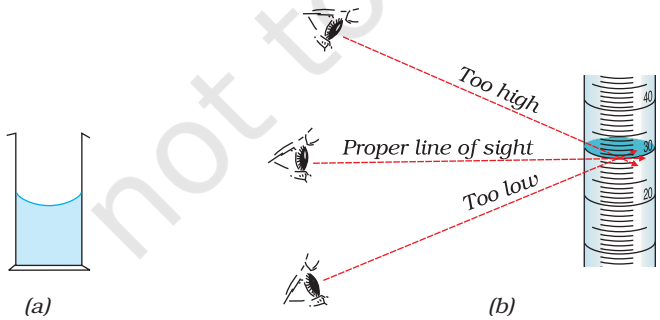


Fig. 2.14 : (a) Water forming curved surface in the glass apparatus
(b) Noting the reading

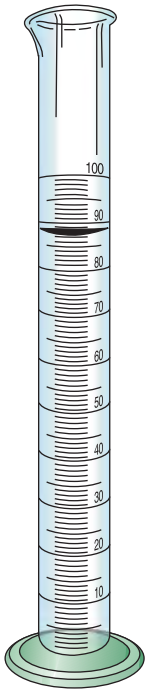


Fig. 2.15 : Measuring Cylinder

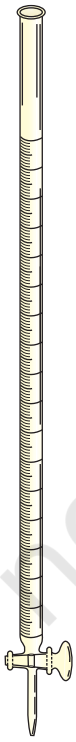


Fig. 2.16 : Burette

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).

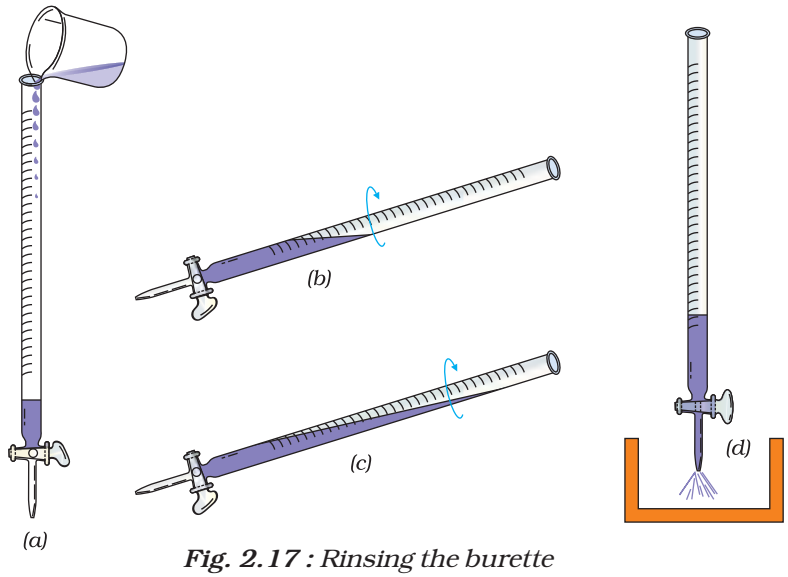


Fig. 2.17 : *Rinsing the burette*

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).

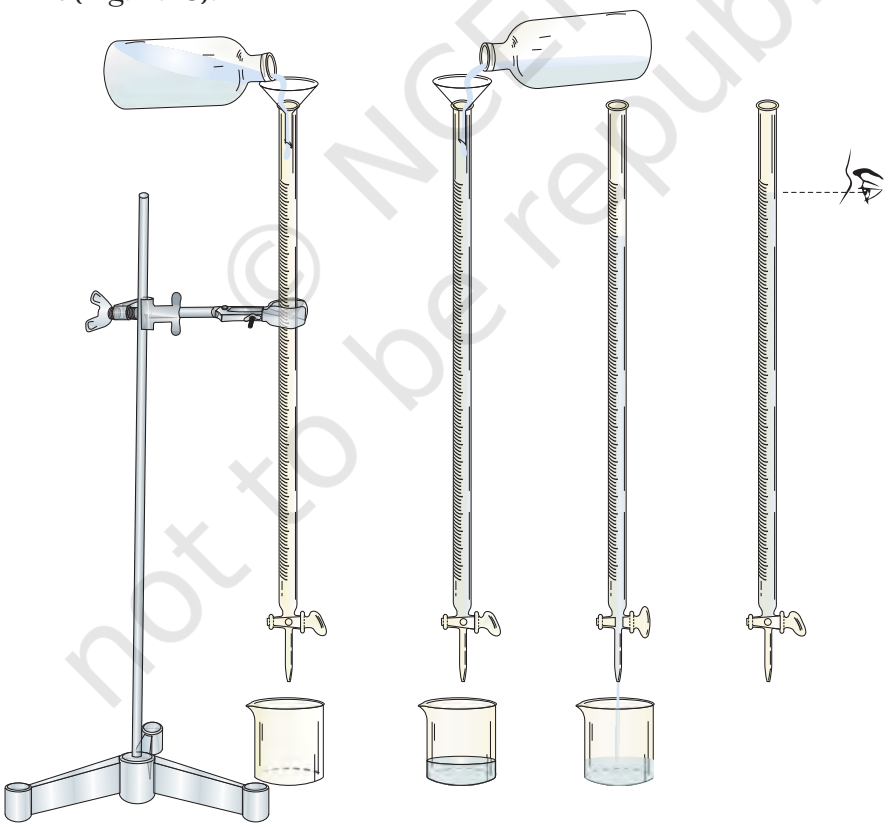


Fig. 2.18 : *Filling the burette*

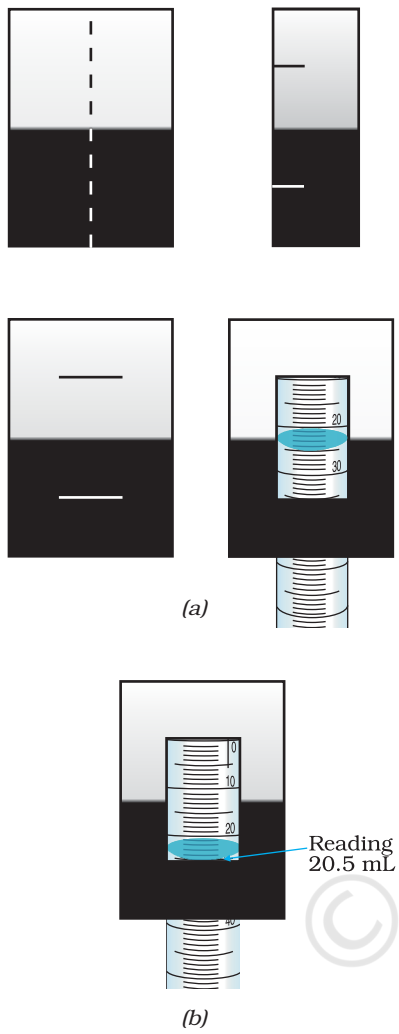


Fig. 2.19 : (a) Mounting anti parallax card on burette
(b) Using anti parallax card for noting correct reading

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).

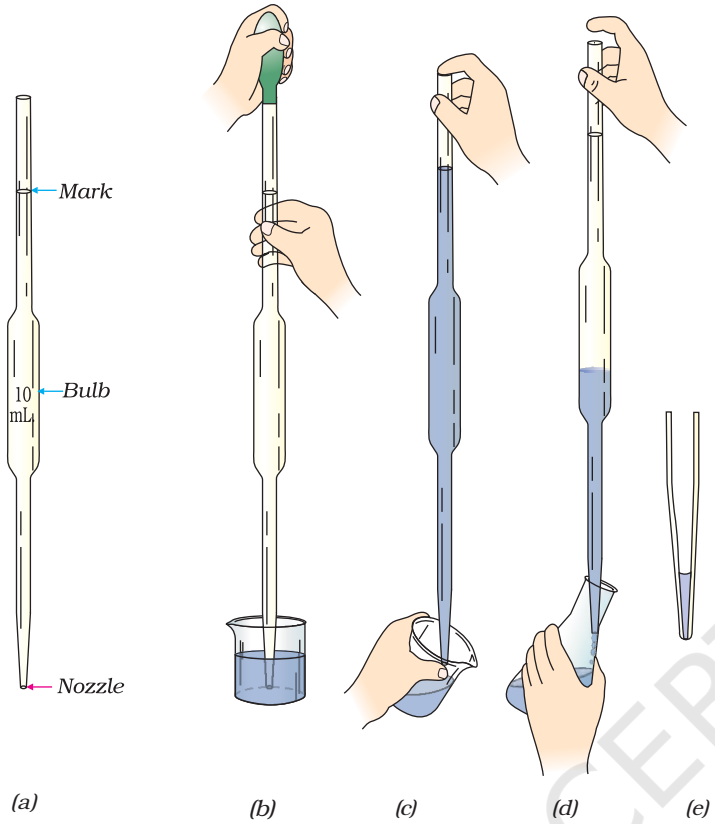


Fig. 2.20 : *(a) Pipette*
(b) Using pipette filler bulb
(c) Holding the pipette after removing the bulb
(d) Transfering liquid to the flask
(e) Nozzle of the pipette after measuring solution

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.

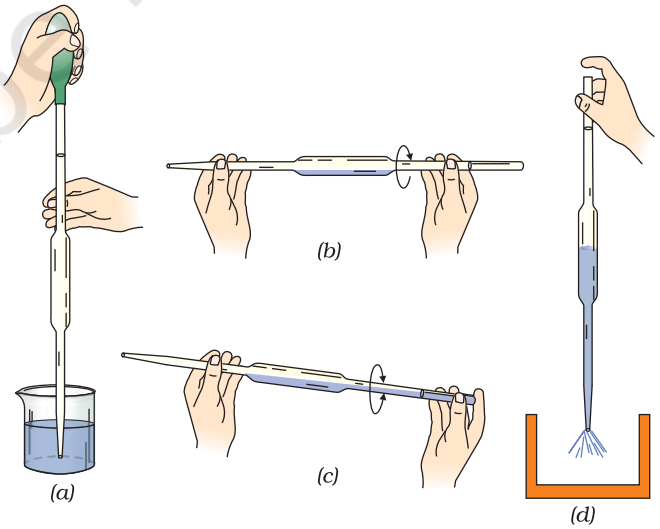


Fig. 2.21 : *Rinsing the pipette*

(d) Using Measuring Flask

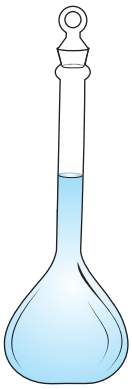


Fig. 2.22 : The measuring flask

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)

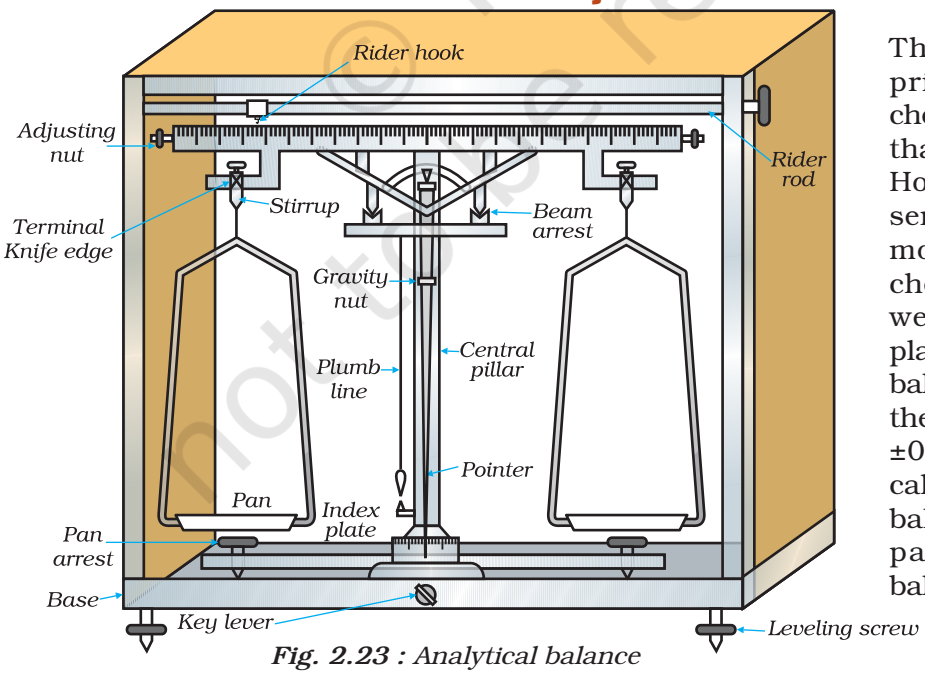


Fig. 2.23 : Analytical balance

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.

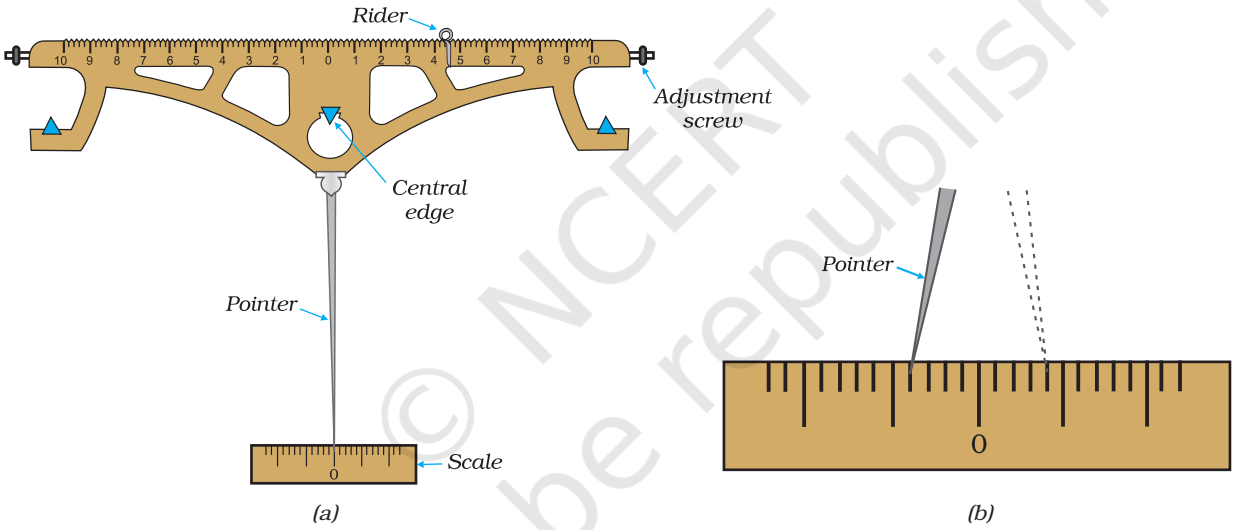


Fig. 2.24 : *(a) Pointer attached to the beam*
(b) Movement of pointer

(b) Weight Box Including Fractional Weights and Riders

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

(a) Weights for weighing in grams :100, 50, 20, 20, 10, 5, 2, 2, 1

(b) Weights for weighing in milligrams : 500, 200, 200, 100, 50, 20, 20, 10

(c) 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.

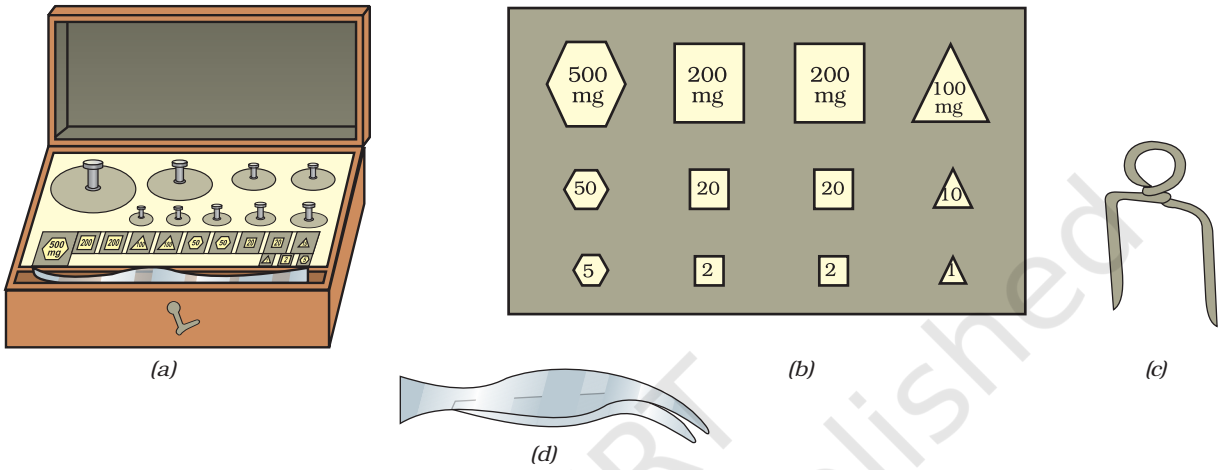


Fig. 2.25 : (a) Weight box (b) Fractional weights (c) Rider and (d) Forceps

(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.

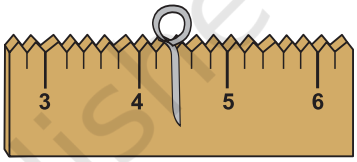


Fig. 2.26 : Rider resting on grove of balance beam

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 $\frac{1}{10} \times 0.01\text{g} = 0.001$ 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 0.001 + 2 0.0002 = 0.0044 g) (Fig. 2.26).

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 relasing 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

$$\begin{matrix}\text{COOH}\\|\\\text{COOH}\end{matrix} \cdot 2H_2O$$

** 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 $\frac{126}{10} = 12.6\text{g}$ 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 :

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

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).

Oxalic acid 

(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 substracting 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 th of the volume of the measuring flask as shown in Fig. 2.27 a, b.

(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.

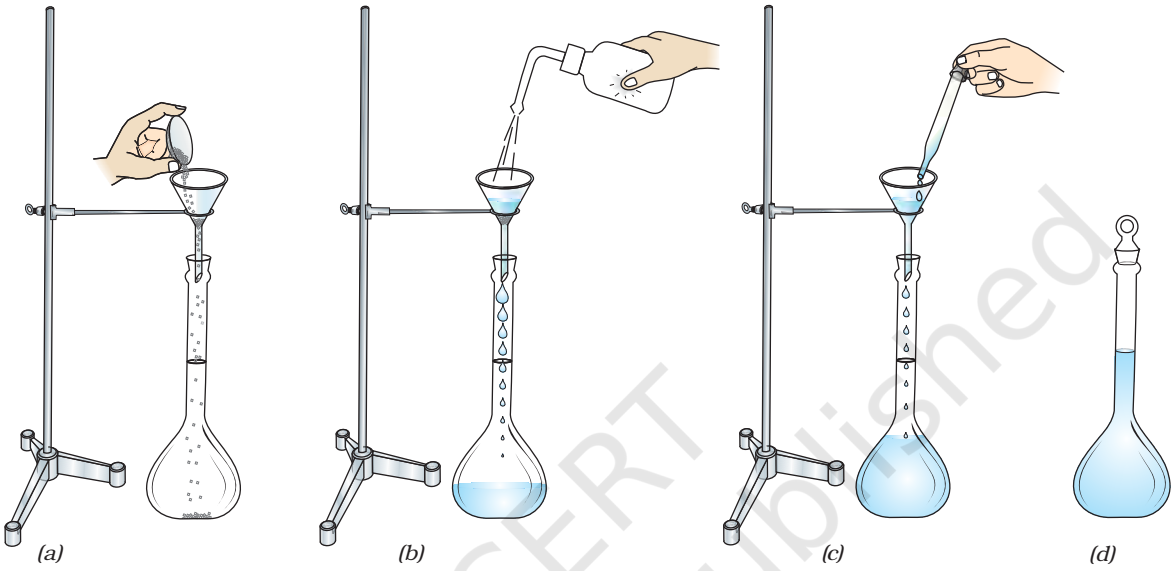


Fig. 2.27 : *Making standard a solution*
(a) Transfering oxalic acid (b) Diluting the solution
(c) Adding last few mL dropwise (d) Standard solution

Precautions

(a) The pan of the balance should be cleaned before and after weighing.
(b) Never touch the weights with hand. Use forceps to transfer weights from the weight-box to the pan of the balance.
(c) Always use spatula to transfer the reagent from the bottle on to the watch glass.
(d) Stopper the reagent bottle immediately after withdrawing the substance.
(e) Always use distilled water to prepare the standard solution.
(f) Always check the adjustment of the balance before weighing the substance.
(g) Care should be taken while weighing the chemicals. These should not be spilled on the pan of the balance.
(h) Watch glass/weighing bottle and funnel should be washed several times by using small amounts of distilled water each time.
(i) 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.
(j) 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?