9 COMMON APPARATUS

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We use many simple equipment and apparatus in a laboratory. Do we use them properly? Although they are simple to operate, improper use may lead to erroneous results and would also affect the performance of the apparatus.

In this chapter we will discuss the proper handling and use of some simple apparatus. Some apparatus can be brought to your working surface of the bench while others should be kept in a proper place without being carried around in order to ensure their good performance. However, you should keep the apparatus in a clean and safe environment having easy access to work.

Each and every apparatus, whether simple or complex, is given a *User Manual* which describes the components, maintenance and proper usage. These manuals may be specific to a certain model. Therefore, it is essential to read the manual before using the instrument however much you are familiar with similar apparatus.

Water Purification

Let us begin our discussion with the distillation apparatus, which is essential to purify water. Before we do so, we will consider water as a solvent. Water is widely used as a solvent in the laboratory. We cannot use tap water or water from any other source directly in the laboratory for experiments other than for washing and cleaning. The reason is that water from unknown sources is not chemically and biologically pure to be used in the laboratories. For most of the preparations we need purified water in the laboratory.

Purified water is water that is physically processed to remove impurities. Distilled water and deionized water have been the most common forms of purified water, but water can also be purified by other processes such as reverse osmosis, carbon filtration, micro-porous filtration, ultra filtration, ultraviolet oxidation, electro dialysis etc. In recent times, scientists combine two or more of the above processes to obtain water of high purity.

Distilled water is produced by a process of distillation and has an electrical conductivity of not more than 10 μS/cm (usually 0.5 – 3 μS/cm) and total dissolved

solids of less than 5-10 mg/L (ppm). Distillation involves boiling the water and then condensing the steam into a clean container. Distillation produces very pure water but also leaves behind a white or yellowish solid on the distillation apparatus, and therefore, the apparatus should be frequently cleaned. Distillation does not guarantee the absence of bacteria, unless the container is sterilized before being filled. Double-distilled water (abbreviated "ddH_2O", "Bidest. water" or "DDW") is prepared by double distillation of water.

Distillation and Deionization Apparatus

Distillation apparatus can be from very cheap and simple (Fig. 1) to expensive and complex (Fig.2).

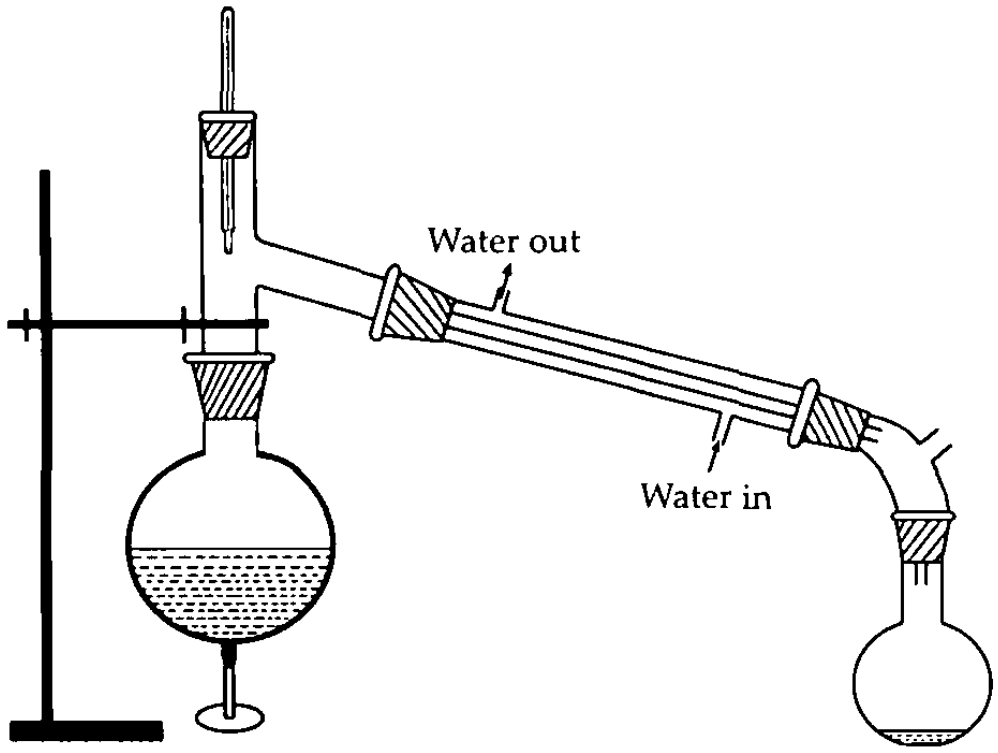

Figure 1. A simple distillation apparatus

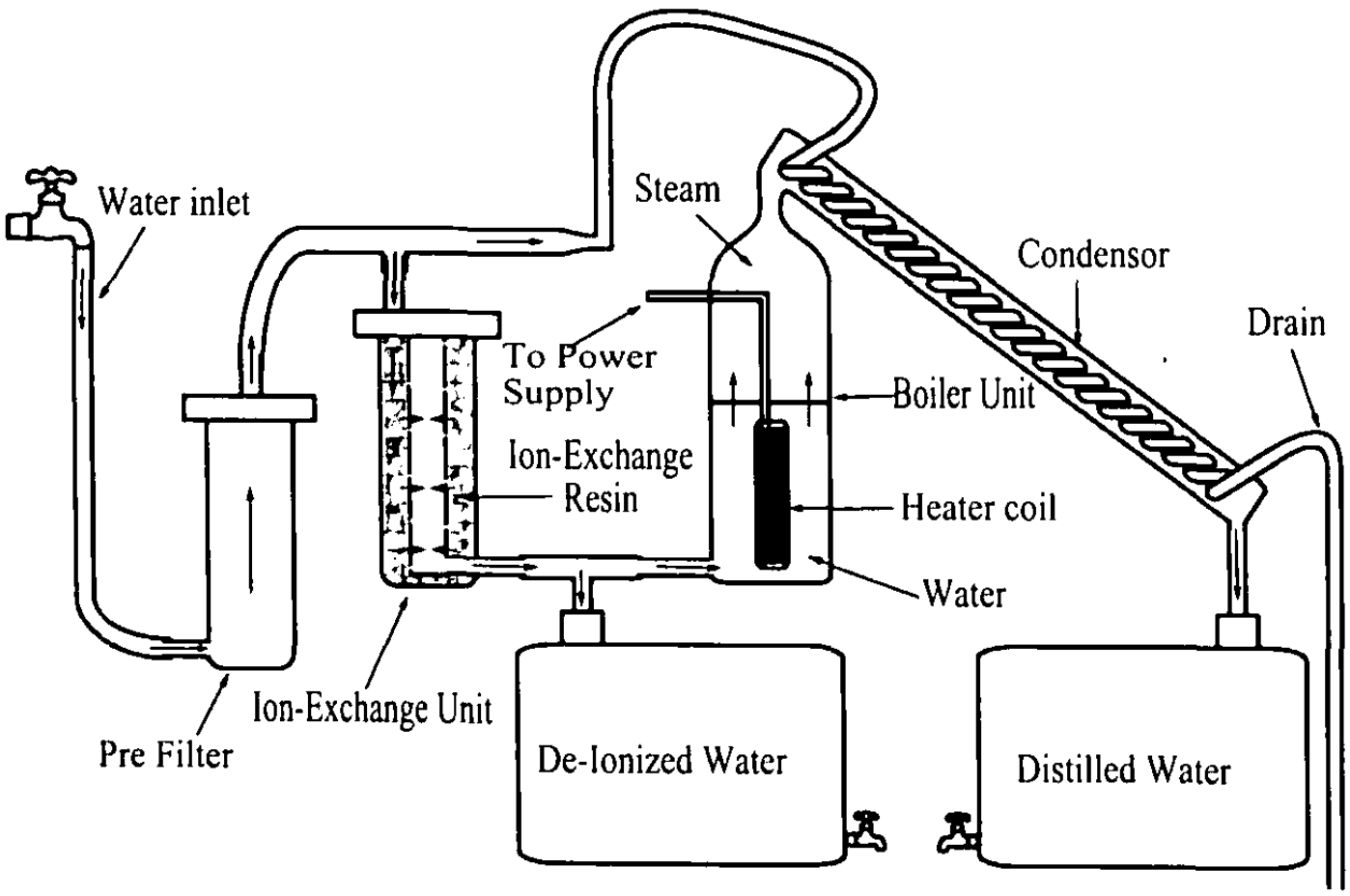

Figure 2. Schematic diagram of a modern distillation plant

The following steps should be carried out to ensure the expected quality of distilled water from the distillation plant in the laboratory.

1. Speed of water should be adjusted according to the instructions given in the manual.
2. Regular cleaning of the filters.
3. Regular checking of conductivity and maintenance of a chart of conductivity with the date.

In the laboratory we also use deionized water, also known as demineralized water. It is water from which the mineral ions are removed. Deionization is a process which uses specially-manufactured ion exchange resins by which the cations are exchanged with hydrogen ions and anions are exchanged with hydroxyl ions which together finally form H_2O (Fig.3). This process is quick. However, deionization does not significantly remove uncharged organic molecules, viruses or bacteria. The resins used can be regenerated and the regeneration process will depend on the type of the resin. Like distilled water, the quality of water has to be tested by checking the electrical conductivity. In order to maintain the efficiency of the deionization water plant, the same steps mentioned above for the distilled water plant should be carried out.

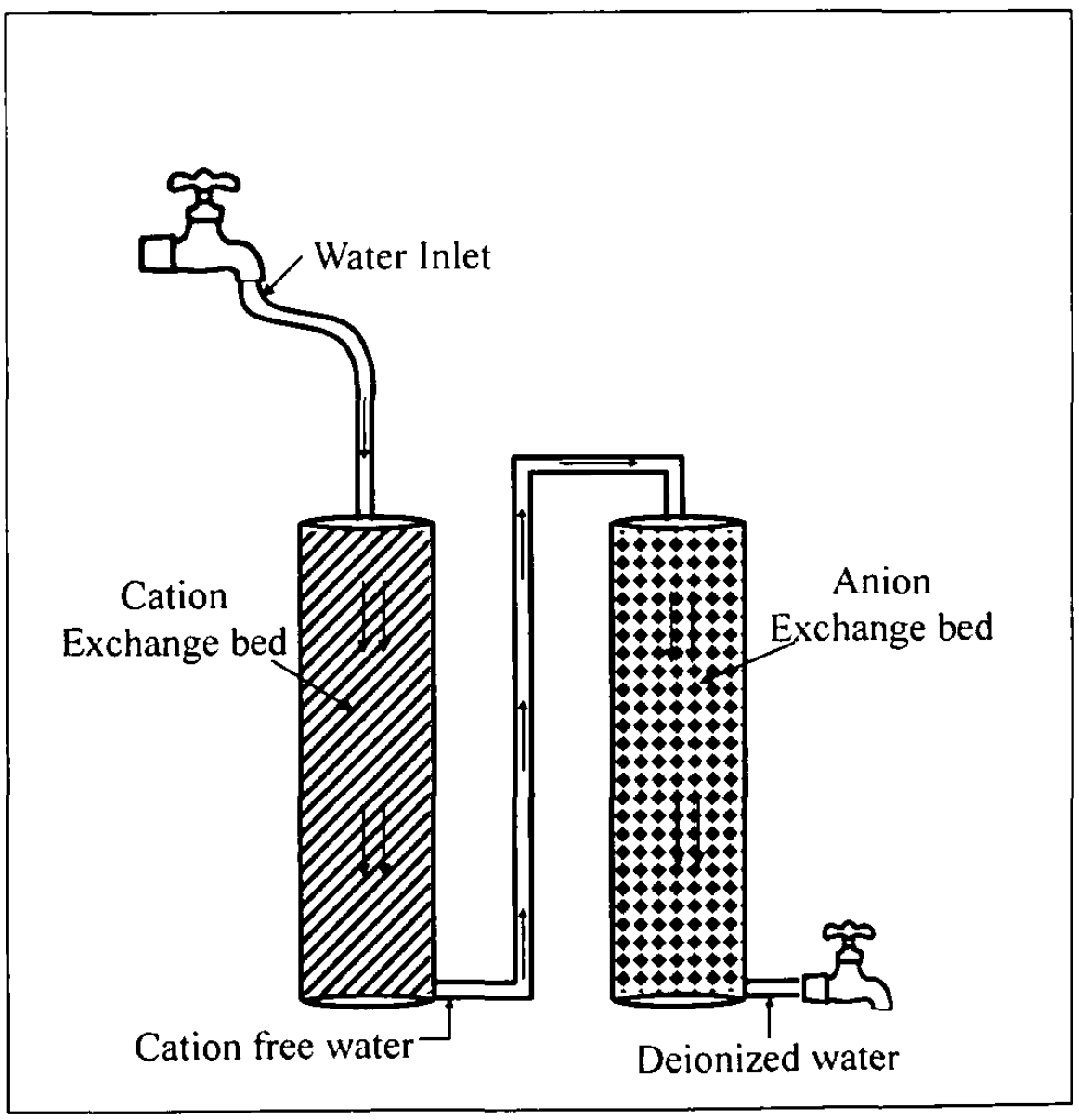

Figure 3. Deionizer

Heating Apparatus

Heating is required for different purposes in a laboratory. There is a variety of techniques and apparatus used. The technique and the apparatus you select will depend on the substance to be heated, the temperature required and the period of heating. The following are some of the apparatus used in a laboratory for heating.

Bunsen Burner

The common burner used in laboratories is the Bunsen burner. Usually the gaseous fuel used is Liquefied Petroleum Gas (LPG). Maximum temperature can be attained by adjusting the size of the air inlet hole and it is important to obtain a non-luminous blue flame. These burners should not be used to directly heat flammable liquids. Safety is extremely important when working with a Bunsen burner. The burner should be always placed on a stable fire proof surface (e.g. ceramic tile). Keep the ignited burner away from flammable materials, equipments, chemicals and overhead shelving. Make sure that the gas tube is undamaged and it fits securely and firmly to the gas valve and to the burner. A lighter with an extended nozzle should be used to ignite the burner (a match is not a safe option). Close the air holes of the burner (by turning the barrel/collar) before lighting up the burner. Before turning the gas on, ignite the lighter first and hold it near by the side of the open end of the Bunsen burner barrel. Once the gas is on, bring the lighter towards and little over the mouth of the burner. The yellow flame is not efficient. Therefore adjust the flame into a blue colour flame by opening the air holes. The height of the flame can be adjusted by regulating the gas flow. Never leave the burner alone when it is on. Keep in mind to close the gas valve once the work is completed.When heating a test tube over a Bunsen flame, leave the air hole half-open. Wave the test tube through the flame to avoid overheating and creating a hot-spot on the tube (Fig.5).

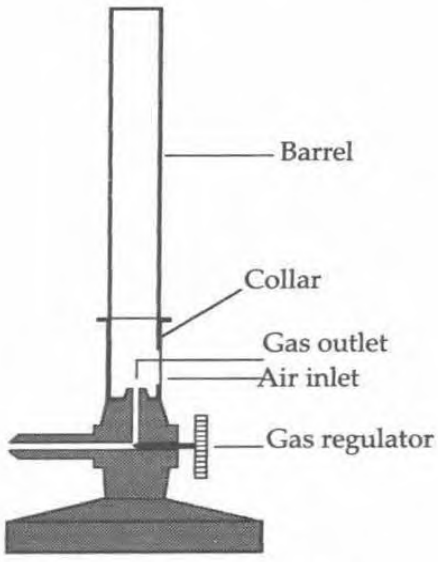

Figure 4. Bunsen burner

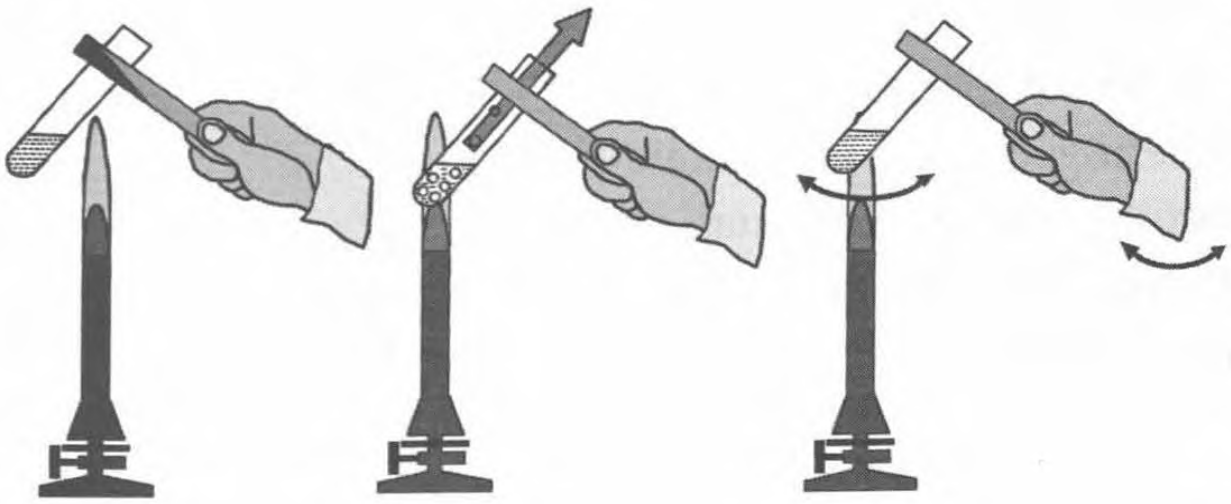

Figure 5. Using the Bunsen burner. (a) Holding a wooden test tube holder too close to the flame can burn it. (b) Overheating the base of the tube can cause the liquid to boil quickly and squirt out. (c) The test tube should be gently moved through the flame.

Hot Plates

Hot plates are electrically heated and are thus used to heat flammable liquids. Since there is a temperature and sometimes a time-control incorporated into the hot plate it is very much useful. A heat resistant sheet can be placed in between the plate and the vessel to be heated to provide even heating.

Electric Ovens

Ovens are also electrically heated and usually used for drying purposes. The maximum temperature range that can be obtained in a normal oven is between 250 ^{0}C – 300 ^{0}C and the temperature and time can be controlled. It is important to write the set temperature and the finishing time on a paper and paste on the oven to prevent another user from changing the setting. Since the oven is a common apparatus shared by all in a laboratory, it is advisable to cover (not fully but with a funnel) the substance you are drying to prevent any other substances from falling on to it.

Microwave Ovens

Microwaves are a form of energy similar to other waves. They are generated using electricity in a microwave oven. These can provide more heat in a short time and thus commonly used for rapid high temperature heating. Microwave-safe containers should be used for heating in microwave ovens.

Muffle Furnace

Muffle furnaces (Fig. 6) can be either gas or electrically heated and the maximum temperature that can be obtained is about 1200 ^{0}C. They are used to determine the inorganic constituents in a sample by burning away the organic material, digestion of material at high temperatures among other high temperature treatment of samples.

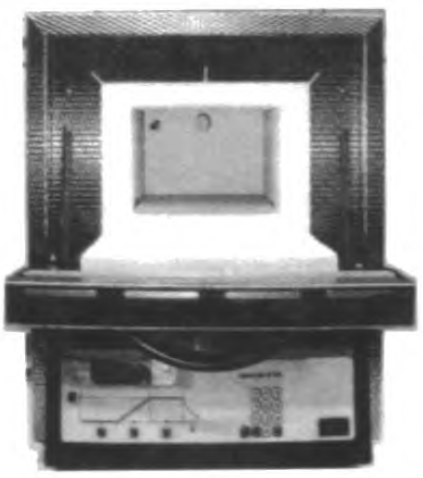

Figure 6. Muffle furnace

Heating Mantles

These are electrically heated and the electric wires are embedded within a strip of fabric that can hold round-bottom flasks, which have different diameters (Fig. 7). The heating element of a heating mantle is insulated from the container. Therefore containers may be placed in direct contact with the heating mantle, which provides even heating for long periods.

Figure 7. Heating mantle

Block Digesters

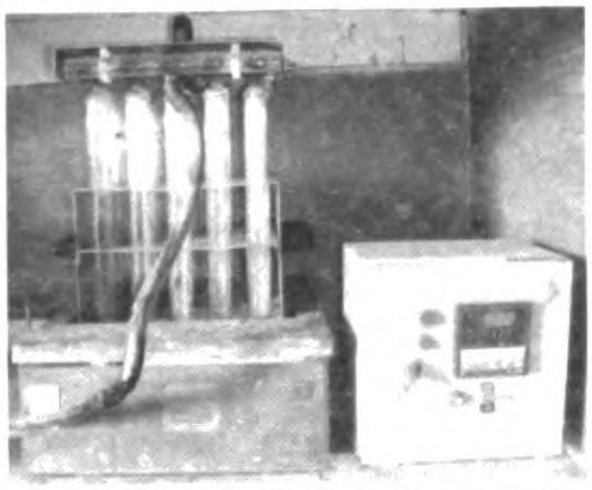

Figure 8. Block digester

It is a metal block which can be heated to higher temperatures; it has holes into which tubes can be inserted (Fig. 8), so that the tubes will be evenly heated for long times. Block digester is used for wet digestion of samples with strong acids. The tubes will be thermally stable over a wide range of temperatures and of different heights which can be selected according to the amount of sample and reagent used. Modern block digesters have more than about 20 temperature programmes and during the digestion the digester should be kept inside a fume hood. After usage, the digester should be cleaned with a dry cloth and the tubes should be thoroughly washed. Currently these block digesters are being replaced by microwave digesters. These are quite efficient and versatile.

Desiccator

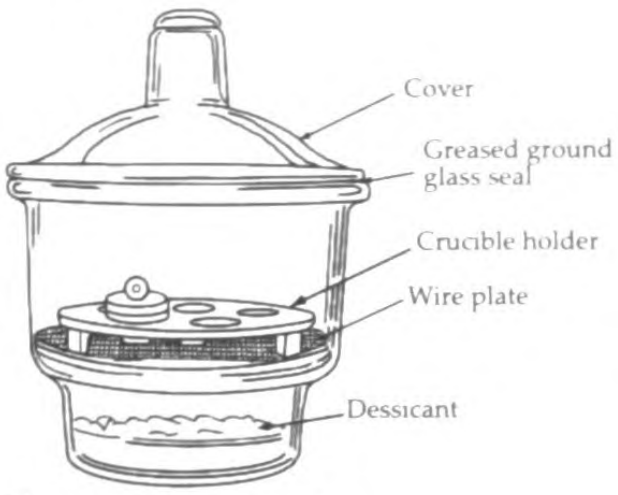

Figure 9. Desiccator

The desiccator is a covered glass sealed container to maintain a dry atmosphere. The drying agent (or desiccant) such as anhydrous calcium chloride, silica gel, activated alumina or anhydrous calcium sulphate is placed in the shallow part of a desiccator which is separated by a porous plate from the main chamber (Fig. 9). It is important to know the comparative efficiency of drying agents since the vapour pressure and the drying capacity (amount of water that can be removed by unit weight) affects the efficiency of drying. Some of these desiccants are coated with cobalt salt which gives a pink colour when sufficient amount of moisture is absorbed (therefore, cannot be used anymore) and blue when it is dry and capable of absorbing moisture. They can be regenerated by drying in an oven at suitable temperatures. The edge of the desiccator lid should be lightly coated with grease or Vaseline in order to make the inside air-tight. Before using a desiccator, it should be cleaned and the desiccant should be in the anhydrous state.

Stirring and Mixing Apparatus

Stirring or agitation is required to homogenize mixtures which are heterogeneous. It is also used during adding a substance to a solution that needs to be mixed or dissolved well immediately in the entire solution. These apparatus provide even stirring at different speeds and times.

Magnetic Stirrer

Stirring occurs by a small cylindrical rod (or bar) of iron sealed in Pyrex glass, polythene or Teflon, rotating in a rotating magnetic field. The speed should be controlled to avoid spilling of the liquid. After stirring is completed, the stirring rod should be removed using forceps or an encased magnet on a handle.

Mechanical Shaker

This is used when vigorous shaking is required for large volumes or when several samples have to be shaken under similar condition in a short time period. Shaking can differ in speed, pattern (circular, linear, reciprocal) and time. It is important to make sure that the containers will not fall off the shaker during shaking (Fig. 10). Soil samples are usually shaken on a linear shaker.

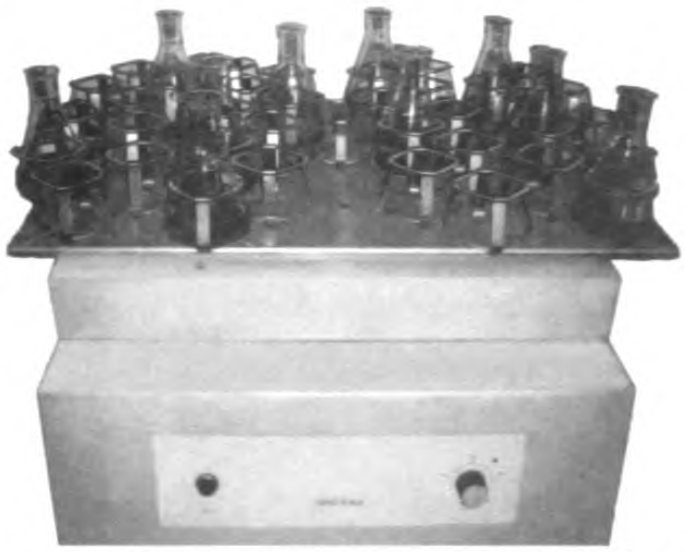

Figure 10. Mechanical shaker

Filtering and Separation Apparatus

Filtration is basically a separation technique. The filtering medium and the apparatus used will depend on the type of analysis, particle size of the residue, volatility of the liquid, and volume to be filtered.

Filter papers

Filter papers are made for qualitative and quantitative purposes. Quantitative filter papers have a smaller pore size to filter very fine particles for gravimetric analysis. The rate of filtration is hence slow. Qualitative filter papers are used for routine laboratory work.

Filter papers are available in different qualities. The quality will vary with the pore size, diameter, mechanical strength and amount of ash that is left behind if burnt. For quantitative analysis, where accurate weight of the precipitate is required after filtering and drying, filter papers are not suitable as a filtering media. This is because weight of only the filter paper is difficult to obtain accurately and quantitative transfer of the precipitate from the filter paper to a watch glass is not possible since the filter paper may adsorb some of the precipitate.

There are three types of filter papers:

Filter papers with different pore size

Although a smaller pore size is better in retaining the residue, it may slow down the filtration process. Therefore, using filter papers with a smaller pore size than necessary should be avoided. In quantitative analysis, the filter paper that should be used is usually indicated in the procedure. Filter papers are available in three textures: Large pore size (for gelatinous and coarse particles), Medium pore size (for average sized particles), and Small pore size (for fine particles).

Ash-less filter papers

These have very small ash content (a 11 cm circle the ash content is less than 0.0001 g). These are used for igniting the residue in gravimetry since the weight of ash of the filter paper after ignition is negligible when compared to the weight of the ignited residue. These are expensive and should not be used for ordinary filtering.

Acid hardened filter papers

These have a greater mechanical strength when wet and are more resistant to acids and alkali. They too have an extremely small amount of ash after ignition.

Folding filter papers

When a filter funnel is used as the filtering apparatus, it is necessary to fold the filter paper to fit in to the funnel. There are two ways of folding the filter paper: quadrant fold (Fig.11) and fluted fold (Fig. 12). The quadrant fold results in a part having three times the thickness of paper giving more mechanical strength to prevent any damage which might result when transferring the liquid. The advantage of using fluted filter papers (Fig. 12) is speed of filtering. Fluting facilitates the flow through the filter body, thus speeding the process. Fluted filter paper is used when we have to separate and retain the liquid and discard the solid.

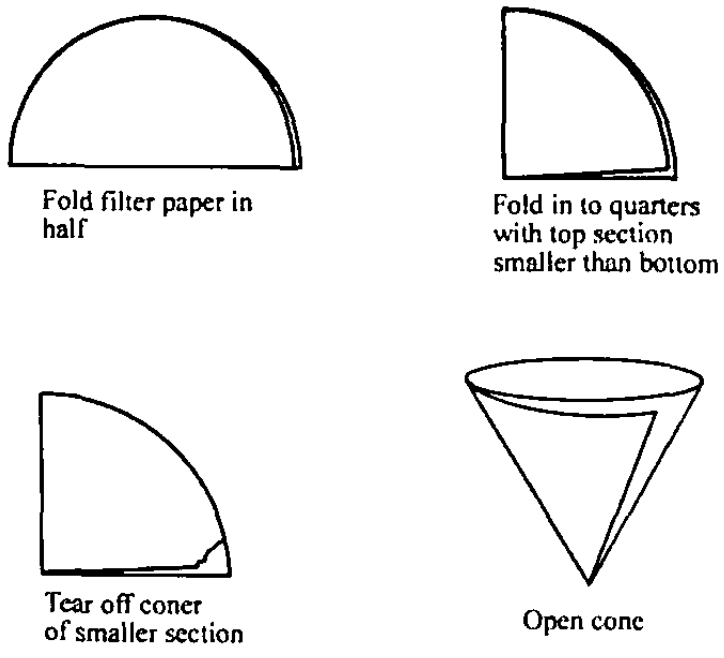

Figure 11. Quadrant folding

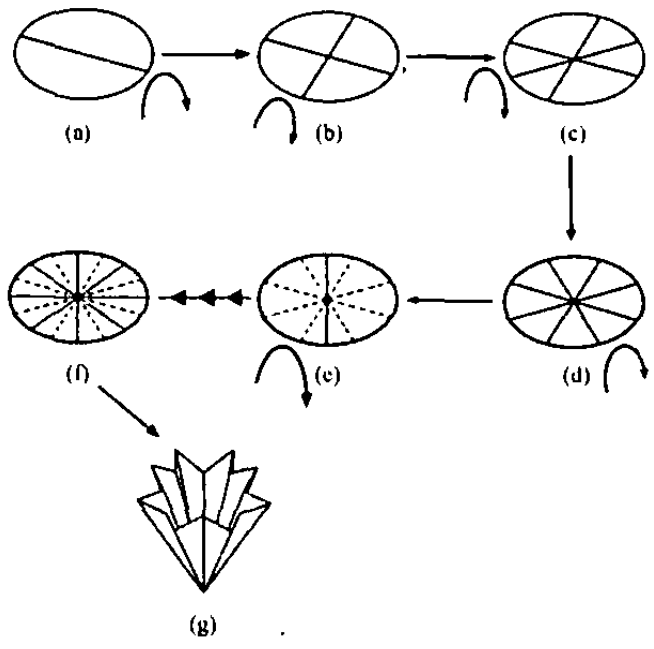

Figure 12. Fluted folding

Suction filtration

Suction filtration is a technique to separate a solid from a liquid. A specially designed filtering flask with a side arm and a Buchner funnel are needed for this process (Fig. 13).

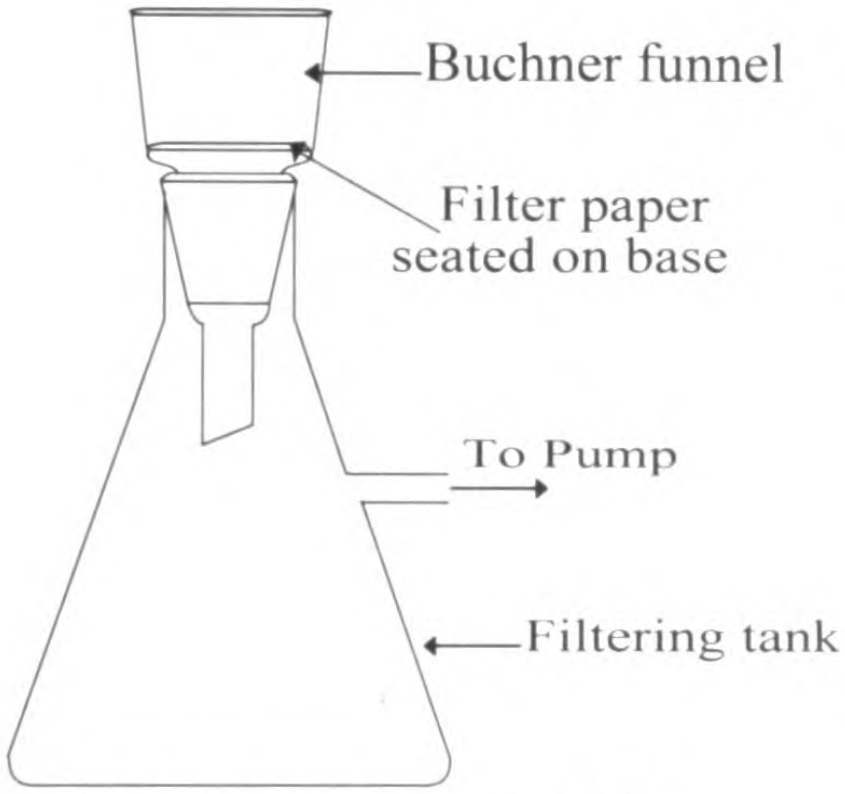

Figure 13. Buchner funnel and flask

The filter flask should be clamped safely to a stand. The Buchner funnel, with a rubber collar, should be fixed tightly on to the top of the flask ensuring there is a good seal between the funnel and the flask. A filter paper of correct diameter is placed in the funnel so that all the pores in the funnel are covered. The filter paper is moistened with a little solvent before beginning the filtration. The solution to be filtered is poured little by little along a glass rod onto the centre of the filter paper during filtration. Once the entire solution has been filtered, rinse the residue with clean solvent. After all the liquid has passed through, allow air to pass through the solid for some time to evaporate any remaining solvent in it. Remember to disconnect the filtering apparatus from the vacuum, before the funnel is removed from the flask.

Quantitative filtration

In most quantitative work, the residue or the precipitate is filtered, dried and then weighed (no ignition is involved). In such cases, it is better to use a filtering medium fitted to an apparatus which can be accurately weighed before filtering. The final weight will be with the residue and the difference will give the accurate weight of the residue. Crucibles with porous fritted plates made out of glass, silica or porcelain are such filtering devices. Sintered glass crucibles have a filter disc with different pore size indicated by a number from 0 (largest pore size) to 5 (smallest pore size) (Fig 14). These crucibles cannot be heated above 200 ^{0}C. Silica and Pyrex crucibles have the advantage of tolerating high temperatures. Since no filter paper is used, strongly acidic and weakly alkaline solutions can also be filtered. It is important to note that hydrofluoric acid, fluorides and strongly alkaline solutions **should not be used** for filtering since they attack silica. After the analysis is completed the crucible should

Figure 14. Sintered glass crucibles

be cleaned, first by immersing in tap water and then in hot 0.1 M tetra sodium salt of the ethylenediaminetetra-acetic acid (Na- EDTA) solution.

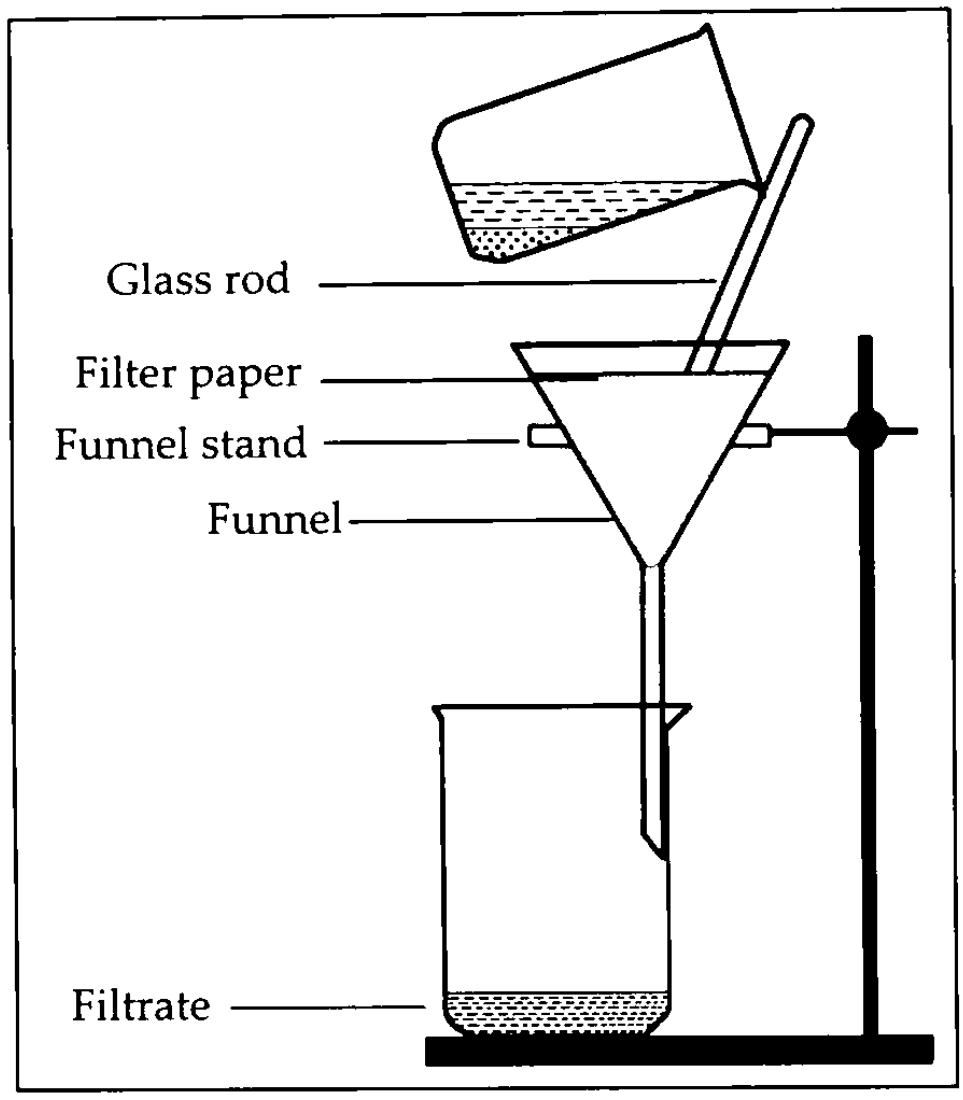

Figure 15. Quantitative filtration

Factors to be considered during quantitative filtration (Fig. 15).

1. Use a suitable filtering medium and a vessel of suitable size to collect the filtrate.
2. All the apparatus should be thoroughly cleaned.
3. A normal funnel should be placed on a funnel stand. If filtration is under reduced pressure, the flask, crucible and/or funnel should be well clamped.
4. If you are using a pump, check whether it works properly and whether the water pressure in the water supply line you intend using is adequate.
5. If you are using a filter paper, fold it appropriately and place on the funnel carefully making sure the upper portion tightly fits to the glass. There should be a gap between the top edges of the funnel and the filter paper cone (Fig. 15). Wet the paper with distilled water so that it will fit tightly for rapid filtration.
6. Place the collecting vessel against the stem of the funnel to avoid splashing and also to increase the rate of filtration. The tip of the funnel stem should always lie above the solution level of the filtrate.
7. Transfer the solution to the funnel with the aid of a glass rod to a side of the funnel (not into the apex and if the filter paper is quadrant folded, on to the part having three thickness of paper).

8. Fill the funnel with the solution slowly leaving at least 1 cm from the top without completely filling.
9. The last traces of the precipitate can be transferred with the washing liquid.
10. If the precipitate on the funnel is left over-night after filtration, cover the funnel loosely with a watch glass leaving space for the moisture to escape.

Centrifuge

Centrifuges are used to separate suspensions having light particles which are difficult to separate by filtering. Centrifuge separation is quick but cannot be applied for quantitative work. In a centrifuge there are sample tubes in metal buckets (Fig. 16). Only three quarters of the sample tubes should be filled to avoid any spillage and the two sides of the centrifuge should be balanced in weight by filling the two diametrically opposite sample tubes. The speed and the time of centrifuging can be selected and the lid of the centrifuge should be opened only when the centrifuge has stopped running completely. After using the centrifuge, any spillage should be cleaned.

Figure 16. Desktop centrifuge

Analytical Balances

A sensitive, accurately working balance is an essential instrument in the laboratory. An analytical balance (Fig. 17) gives the weight up to four decimal places and the accuracy is accepted for analytical work. The accuracy depends not only on the balance, but also on the ambient conditions (temperature, humidity, air movement), handling of the balance, and the weighing procedure among others. An unstable display of the weight can be due to fluctuations in the environmental conditions, evaporation effects on the sample, static electricity, magnetism etc. These can cause minute changes in the weight of the sample, which a high resolution balance is able to detect.

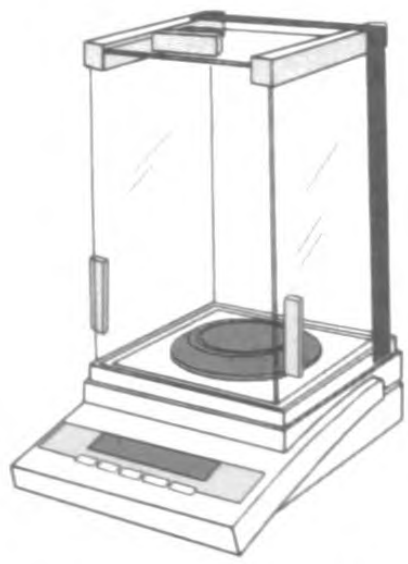

Figure 17. Analytical balance

Here are some Do's and Don't's when using the analytical balance

1. It should be placed in a chemical- and dust- free atmosphere with minimum interference from vibrations.

2. The balance should be kept on a solid surface which is free from vibrations such as a heavy table or concrete ledge.
3. The surface on which the balance is kept should be leveled. Adjust the feet of the balance until the air bubble is in the center of the indicator.
4. Never overload (note the maximum weight the balance is capable of weighing).
5. The pan of the balance should be cleaned with a soft brush before and after using the balance after it is switched off.
6. Do not touch samples or containers with your fingertips which leaves fingerprints (hygroscopic). Use gloves or long forceps.
7. Avoid turbulence close to the balance from air-conditioners, fans, open doors and windows, computer ventilation fans, lamps etc. A rapid change in temperature and air movement affects the read-out. Always close the draft shield of the balance before taking readings.
8. Do not use the balance table for writing or other tasks – the balance reacts to the slightest vibrations.

There are other mechanical balances which can be used to get a "rough" measurement of the mass. Although they are not as sensitive as an Analytical balance, they are useful when an accurate weight is not necessary.

Procedure for weighing of a salt

1. Clean a watch glass and a glass funnel.
2. Dry them in an oven at 105 °C.
3. Keep them in a desiccator until it reaches the room temperature (use a pair of tongs to handle the glassware and never use hands)
4. Bring the desiccator to the analytical balance.
5. Switch on the balance and wait until it is ready.
6. Place the watch glass on the pan using the pair of tongs.
7. "Tare" the weight of the watch glass using the proper key.
8. Transfer the pure solid, little at a time, using a clean spatula making sure you do not transfer more than the required weight (if the weight exceeds the required amount, never put the excess back to the storing bottle but discard.)
9. Record the weight accurately to four decimal places.
10. Take out the watch glass with the salt and cover with the cleaned and dried funnel and bring to the bench top you are working.

A glazed paper can be used to weigh and transfer small quantities of chemicals that do not react with the paper. A crease in the middle facilitates the weighed chemicals to be transferred (Fig. 18). For narrow necked vessels, a rolled glazed paper can be inserted into the neck of the vessel. Expired catalogues in the laboratory is a good source of glazed paper.

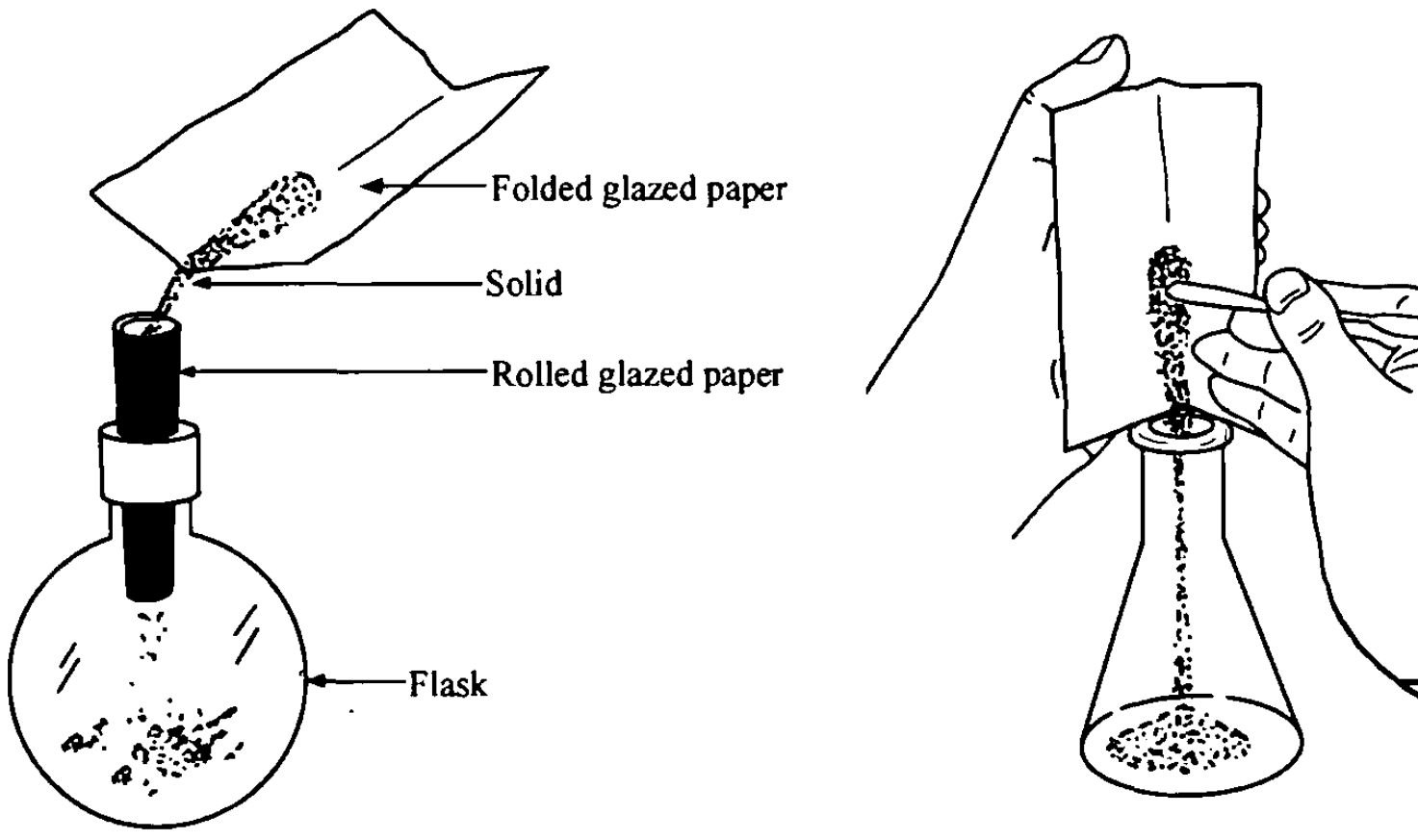

Figure 18. Transferring dry chemicals to glassware

pH Meter and pH Electrode

The pH (acidity or alkalinity) of a solution is measured by using a pH meter. It is an electronic devise consisting of an electrode connected to an electronic meter which displays the measured pH reading on its' screen. Most if not all modern pH meters are digital. The rod shaped pH electrode is the most essential and

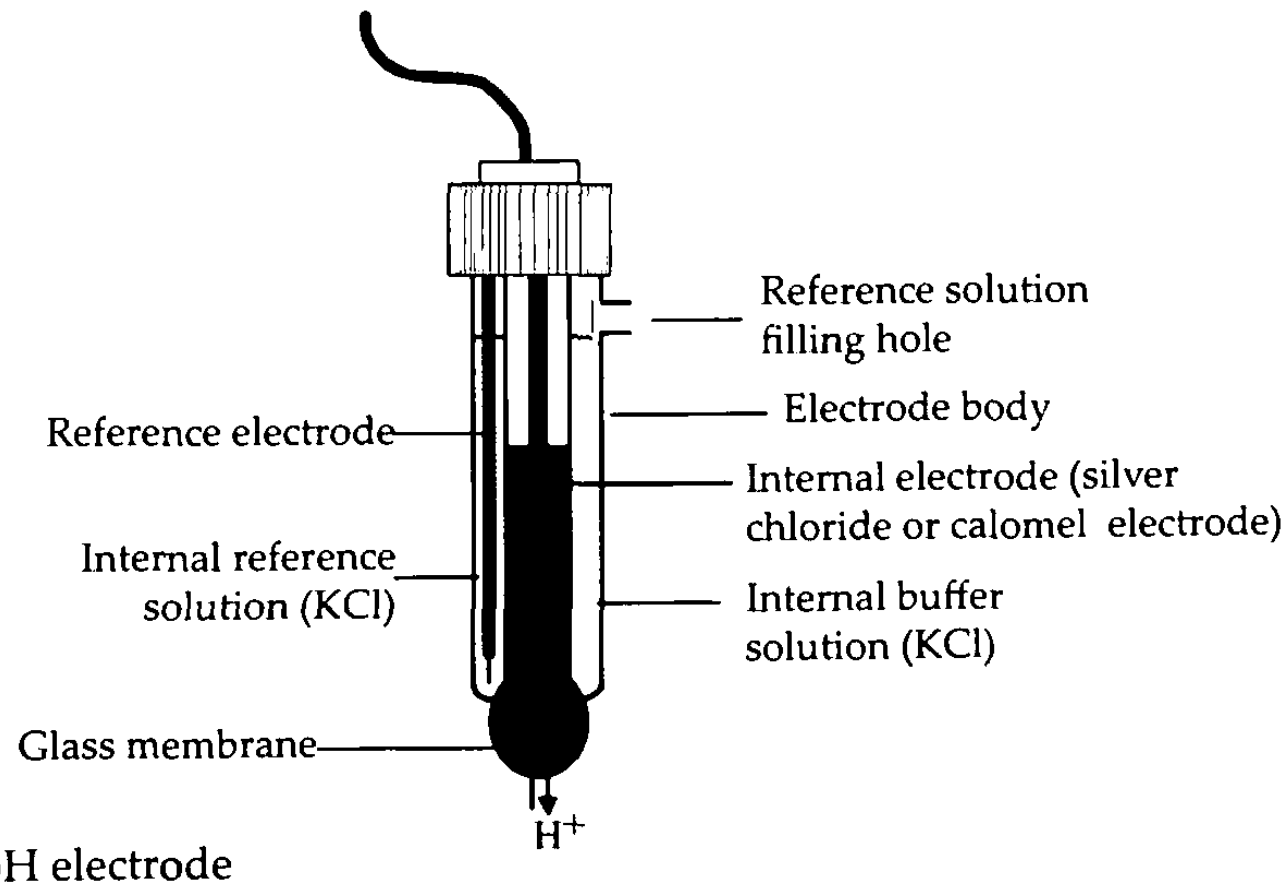

Figure 19. pH electrode

expensive component (Fig. 19). It is an ion-selective electrode. The body of the electrode is made of non-conductive glass or plastic. There is a very delicate glass bulb at the bottom of the electrode, which has a membrane that is sensitive to the H^+ concentration of the solution. The membrane of the pH electrode must not be allowed to dry off as this will damage the sensitivity of the membrane. Therefore the electrode membrane must be kept always immersed in the storage solution (usually a KCl solution) when it is not in use.

Steps for measuring pH of a solution

1. Switch on the pH meter at least 15 minutes before measuring to allow the instrument to be electronically stabilized.
2. Rinse the electrode with distilled water.
3. Remove water drops on the membrane by blotting them off with a dry tissue (the membrane must not be wiped or rubbed).
4. Calibrate the electrode using commercially available standard pH buffers (pH 4, 7 and 10 are the commonly used buffers).
5. Make sure that the membrane is fully immersed in the solution of which the pH is measured, when taking the readings.
6. Clean the electrode with distilled water after every usage.
7. Place the calibrated pH electrode in the test solution and read the pH.
8. After measurement, clean the electrode with distilled water and place it back in the storage solution.

References and Further reading

Practical Skills in Chemistry. J.R. Dean, A.M. Jones, D. Holmes, R. Reed, A. Jones and J. Weyers. 2nd edition. Pearson Education Limited 2011.