GENERAL SAFETY MEASURES AND ACQUAINTANCE WITH BASIC LABORATORY TECHNIQUES

1. OBJECTIVES

After studying this unit, you should be able to,

- *Cut and bend glass rods / tubes ;*
- *Bore rubber cork ;*
- *Perform measurements of volume, mass and density ;*
- *Perform simple laboratory operations such as filtration, crystallisation and distillation ;*
- *Use burner properly in the laboratory.*

2. WHAT YOU SHOULD KNOW

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

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

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

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

2.1 The Do's

- Wear the lab coat / apron before starting the work.
- Light the burner with a match stick. Never use a piece of paper to light the burner.
- While heating a liquid in a test tube, always keep the mouth of test tube away from you and your neighbours.
- Always use a pair of goggles to protect your eyes during sodium ignition or such other operations.
- Always use minimum quantity of chemicals.
- To avoid contamination, use droppers for adding reagents.
- Carry out the reactions involving pungent or obnoxious fumes either in the open or in a fuming cup board.
- Wash your hands thoroughly with soap after the laboratory work.
- Keep the reagent bottles in their proper place.
- Always close the water and gas taps when not in use.

2.2 The Dont's

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

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

(a) Chemicals causing burn

Chemicals responsible for burn	Neutralizing wash
Acids like HCl, HNO_3 or H_2SO_4	Wash with 1 percent sodium bicarbonate solution or with 2M ammonium carbonate. Then, wash with enough water. Apply any soothing cream.
Alkalies like, KOH, NaOH etc.	Wash with 1M acetic acid, followed by enough water. Then apply vaseline or a soothing cream.
Sodium	Remove the solid piece immediately and wash with dilute acetic acid and enough water.

(b) Hazardous chemicals and their effects

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

Hazardous Chemicals	Effects
H_2S	Almost as poisonous as HCN. Exposure dulls the sense of smell.
H_2SO_4, HF, SO_2, NO_2, Cl_2, Br_2, I_2, HNO_3	All are dangerous. When concentrated they cause skin burns.
Salts of Ag, Ba, Hg, Ni, Pb, CrO_4^{2-} and MnO_4	These are harmful only when swallowed.
$AgNO_3$	Causes caustic burns.
Chlorinated alkene eg. $CHCl_3$, CCl_4	Most of these are narcotic and suspected carcinogens.
Benzene	Carcinogen, may cause cancer.

(c) Common Accidents in Chemistry Laboratory

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

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

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

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

3. ACQUAINTANCE WITH BASIC LABORATORY TECHNIQUES

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

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

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

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

3.1 To cut a glass tube of desired length

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

3.1.1 How to perform the experiment ?

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

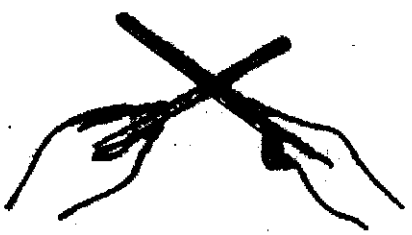

Fig.(i) : *Making a scratch on the* glass tube

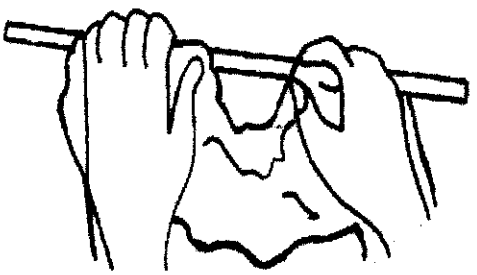

Fig.(ii) : *Holding & Breaking a glass tube.*

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

Normally, the freshly cut ends are sharp and may cause injury to the hands. Therefore,

round off the ends by rotating the tube in the blue zone of the flame for a short time as shown in the Fig. (iii). Keep your fingers at a safe distance from the flame.

3.1.2 Precautions

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

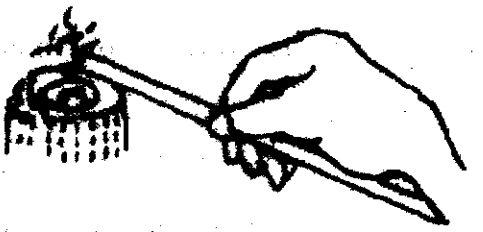

Fig. (iii): *Rounding off the edges of the broken*

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

3.2 Bending of a glass tube

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

3.2.1 How to perform the experiment

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

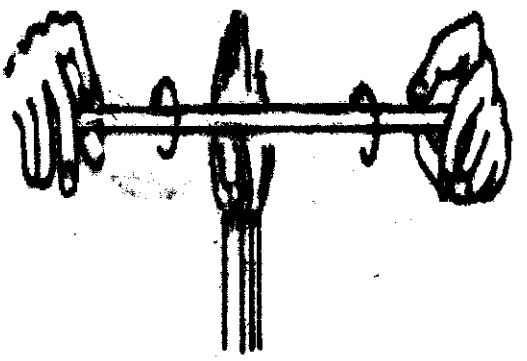

Fig. (iv) : *Heating the glass tube*

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

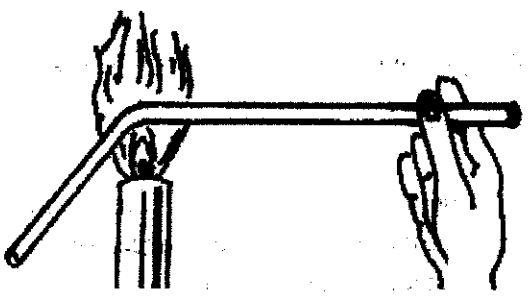

Fig (v): *Bending of glass tube under*

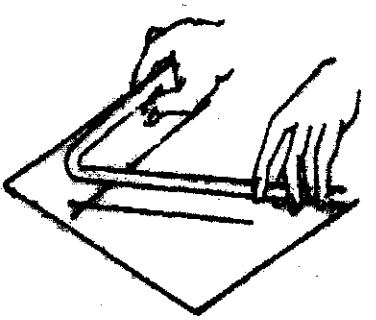

Fig. (iv) : *Making the bent limbs of*

3.2.2 Precautions

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

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

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

3.3.1 How to perform the experiment

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

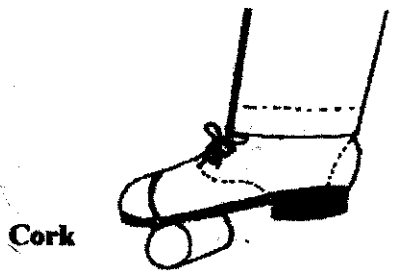

Fig. (vii) : Pressing the cork with shoe

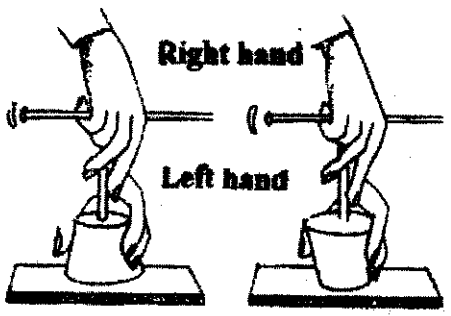

Fig. (viii) : *Boring of cark*

3.3.2 Precautions

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

3.4 Filtration

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

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

3.4.1 How to perform the experiment ?

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

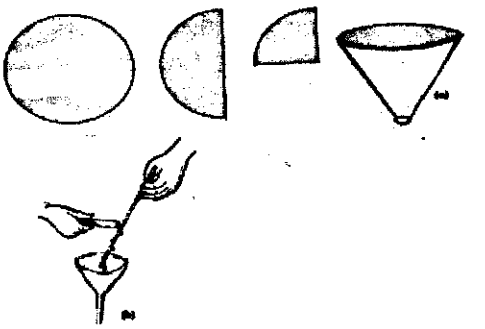

Fig. (ix) : *Filtration*

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

3.4.2 Precautions

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

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

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

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

3.5 Distillation

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

Materials Required :

(a) Apparatus : Conical flask / round bottomed flask, condenser, thermometer, beaker, heating mantle and oil bath.

(b) Chemicals : Impure solvents and tap water.

3.5.1 How to perform the experiment ?

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

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

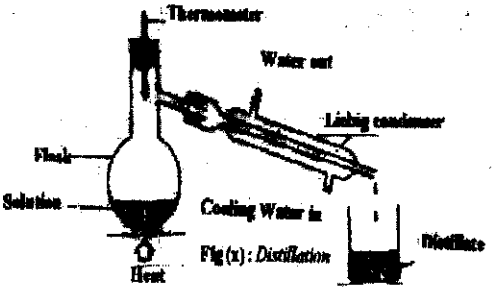

Fig (x) : *Distillation*

3.5.2 Precautions

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

3.6 Crystallization

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

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

3.7 Measurement of volume, mass and density

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

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

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

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

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

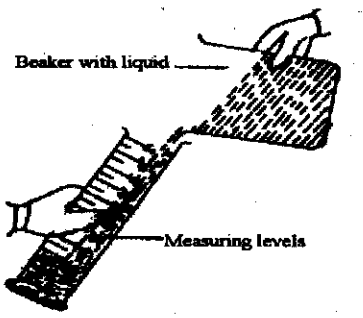

Fig. (xi): *Transfering liquid into the measuring vessel*

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

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

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

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

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

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

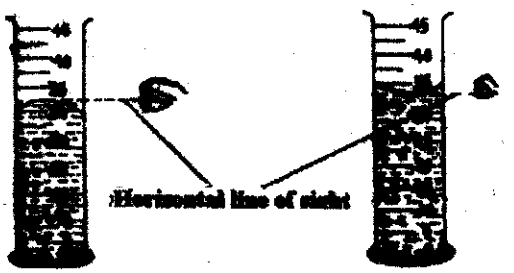

Fig. (xii) : *Taking reading of the (a) concave surface and (b) convex surface of liquid in vessel*

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

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

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

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

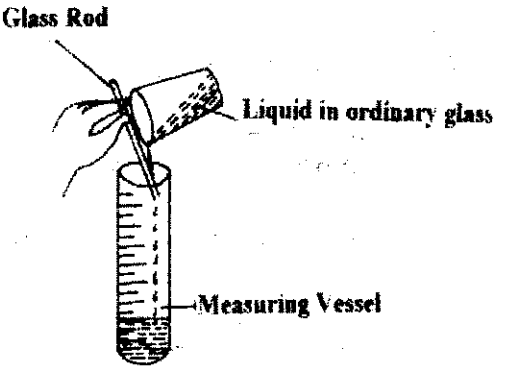

Fig. (xiii) : *Transfering liquid into a vessel*

3.8 Graduated cylinders

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

3.9 Pipettes

Graduated or volumetric pipettes can be used to accurately measure relatively small quantities of a liquid. Suction is required to draw the liquid into a pipette. However, suction by mouth is unwise because of the danger of drawing toxic or corrosive liquids into the mouth. An ordinary car syringe works quite well as a pipetting bulb. Another convenient pipetting-bulb assembly is operated as follows:

1. The top end of the pipette is inserted into the pinchcock valve.
2. The pinchcock is opened by pinching it at the glass bead and the bulb squeezed to eject the air.
3. The pipette tip is placed in the liquid and the pinchcock is squeezed open to fill the pipette to just above the calibration mark.

4. The bulb is removed from the narrow end of the dropper and the pinchcock is carefully opened until the liquid falls to the calibration mark.

5. The liquid is then delivered into another container by opening or detaching the pinchcock valve.

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

3.10 Syringes

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

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

3.11 Mass

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

3.12 Density of a liquid

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

Table: ***Densities of some liquids***

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

4. CHECK YOUR UNDERSTANDING

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

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

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

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

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5. NOTE FOR THE TEACHER

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

6. CHECK YOUR ANSWERS

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

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

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

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