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Programme Revision Committee

Prof. S.S. Hasan (Retd.)
School of Sciences, IGNOU

Dr. Bharati Sarkar (Retd.)
Maitreyi College, University of Delhi

Dr. Swadesh Taneja (Retd.)
School of Sciences, IGNOU

Dr. Balaram Pani
Principal,
Bhaskaracharya College of Applied Sciences,
University of Delhi

Dr. Parveen Garg
Principal (in charge)
Swami Shradhanand College, University of Delhi

Dr. R. Venkataraman (Retd.)
K.M. College, University of Delhi
Senior Consultant in Physics

School of Sciences, IGNOU
School of Sciences, IGNOU

Prof. Vijayshri, Director

Prof. S.C. Garg

Prof. B.S. Saraswat

Prof. Neera Kapoor

Prof. S.R. Jha

Prof. Amrita Nigam

Prof. Lalita S. Kumar

Programme Coordination Committee:

Prof. Neera Kapoor

Prof. S.R. Jha

Prof. Lalita S. Kumar (Convener)

Course Revision Committee

Dr. Chandana Mukherjee
Gargi College, University of Delhi

Dr. Balaram Pani,
Principal,
Bhaskaracharya College of Applied Sciences,
University of Delhi

Ms. Shadab Bano, PGT Chemistry
Syed Abid Hussain Senior Secondary School,

Jamia Millia Islamia, New Delhi
School of Sciences, IGNOU

Prof. Vijayshri, Director

Prof. B.S. Saraswat

Prof. Sunita Malhotra

Prof. Bharat Inder Fozdar

Prof. Javed A. Farooqi

Prof. Lalita S. Kumar

Dr. Sanjiv Kumar

Block Preparation

Prof. B.S. Saraswat (Experiment 5 and 9) (Editor)

Dr. Sanjiv Kumar (Experiments 1, 2, 3, 4, 6, 7, 8 and 10)

Course Coordinator: Prof. Javed A. Farooqi

Production

Mr. Sunil Kumar
Assistant Registrar (Pub.)

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**The following Expert Committees designed and developed the first edition of the
Programme/Course in the year 2000.**

Programme Design Committee

COMMONWEALTH

SECRETARIAT, LONDON:

Dr. Ved Goel

Chief Programme Officer

Bangladesh Open University, Gazipur, Bangladesh:

Prof. Mofiz Uddin Ahmed

School of Science and Technology

Dr. A.K.M. Alamgir

Medical Officer

Md. Anwarul Islam

School of Education

Distance Education Centre, Nepal National Commission for UNESCO, Kathmandu, Nepal:

Mr. Sagar Nath Upreti, Director

Allama Iqbal Open University, Islamabad, Pakistan:

Dr. Shahida Naeem

Department of Basic Sciences

Mr. Muhammad Iqbal Shah

Department of Science Education

The Open University of Sri Lanka, Nawala, Nugegoda, Sri Lanka:

Prof. Uma Coomaraswamy

Department of Botany

Dr. K.W.S. Kularatne

Department of Chemistry

Dr. W.L. Sumathipala

Department of Physics

INDIA:

Prof. Janardan Jha

(Former Pro-Vice-Chancellor,
Indira Gandhi National Open
University)

Dr. B.R. Ambedkar Open University, Hyderabad, Andhra Pradesh:

Dr. M. Ramachandraiah

Department of Botany

Dr. G. Ramachandraiah

Department of Chemistry

Y.C.M. Open University Nasik, Maharashtra:

Dr. R.V. Vadnere

Department of Science and
Technology

S.R.T. Marathwada University, Nanded, Maharashtra:

Prof. Suresh Chandra

(Former Faculty Member in
Physics, IGNOU)

Indira Gandhi National Open University:

International Cell:

Dr. V.V. Raghavan

(Former Reader in Life Sciences,
IGNOU)

Regional Services Division:

Dr. Neeta Kapai

SCHOOL OF SCIENCES:

CHEMISTRY:

Prof. B. S. Saraswat

(Former Director)

Prof. S. Kannan

Dr. Sunita Malhotra

Dr. Bharat Inder Fozdar

Dr. Javed A. Farooqi

Dr. Lalita S. Kumar

Life Sciences:

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Dr. Swadesh Taneja

Dr. Geeta Kaicker

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Dr. Pushplata Tripathi

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Ms. Bano Saidullah

Late Dr. Tosha Bhan

Physics:

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Dr. Vijayshri

Dr. S. R. Jha

Dr. Shubha Gupta

Mr. S.S. Dubey

Mathematics:

Prof. R.K. Bose

(Former Director)

Dr. Poornima Mital

Dr. Sujatha Varma

Course Design Committee

SCHOOL OF SCIENCES:

Dr. K.W.S. Kularatne

The Open University of Sri Lanka
Nawala, Nugegoda,
Sri Lanka

Prof. Md. Anwarul Islam

Bangladesh Open University,
Gazipur, Bangladesh

Dr. G. Ramachandraiah

Dr. B.R. Ambedkar Open
University, Hyderabad,
Andhra Pradesh

Mr. Muhammad Iqbal Shah

Allama Iqbal Open University,
Islamabad, Pakistan

School of Sciences

Prof. B.S. Saraswat

Dr. Sunita Malhotra

Dr. Bharat Inder Fozdar

Dr. Javed A. Farooqi

Block Preparation

Prof. B.S. Saraswat (Course Coordinator)

Dr. Bharat Inder Fozdar

IMPORTANT

- **Attendance** is compulsory in the Laboratory Course work held generally at the Study Centre.
- The Laboratory Course is worth **2 credits** to be completed over **5 days** duration:
 - **4 days** of **Guided** Laboratory work
 - **1 day** for the **Unguided** Laboratory work
- To successfully complete the laboratory course you will have to pass (at least **35% marks**) in the Guided and Unguided components separately.

BASIC EXPERIMENTS IN CHEMISTRY : INTRODUCTION

In the CLT-103 course you have learnt in detail about the apparatus and the techniques used in a Chemistry laboratory for the experimental work. This course is designed to put that learning into use. There are ten experiments in this course, which you would be performing in a Chemistry laboratory at your study centre under the supervision and guidance of your counsellor. These experiments are based on the topics covered in the CLT-103 course. In fact these experiments are in the form of activities, which would involve your working on the bench in a Chemistry Lab. The overall objective is to provide you with an experience of the type of activities done by a laboratory assistant in a Chemistry laboratory of a school or a college. The expected learning outcomes of the course are as under.

Expected Learning Outcomes

After going through the contents of this course and performing the experiments described herein, you should be able to:

- state and follow the measures to be taken for safe working in a Chemistry laboratory,
- identify the common pieces of apparatus and equipment used in a Chemistry laboratory,
- clean, safely handle, store and assemble apparatus and equipment in a Chemistry laboratory,
- perform simple laboratory operations like, boring a hole in a cork, cutting and bending a glass tube, servicing a Bunsen burner, using and maintaining an analytical balance,
- prepare distilled and deionised water,
- prepare solutions and laboratory reagents and perform dilutions, and
- perform simple experiments based on preparation, separation and purification techniques.

Study Guide

As stated above, the experiments in this course are designed according to the topics covered in the CLT-103 course. Therefore, you are advised to have a relook at the relevant portions of the theory course before performing the experiments. These are suitably indicated in the experiments. You are expected to study the experiments and the relevant theory portions before going to the laboratory. Due care has been taken to ensure that you are clear about the theory and the procedure of the experiment by reading through the instructions. However, in case you have any doubt, you may discuss with the fellow colleagues or seek the help of your counsellor in resolving the same.

As in a Chemistry laboratory you come across variety of dangerous chemicals and handle different glass apparatus and instruments, you are required to follow appropriate safety measures. These have been suitably indicated in the beginning of the first experiment. Further, you are supposed to maintain a record of all the experiments and activities performed in the course. These are to be submitted daily to the counsellor for correction and evaluation. The performance of the assigned activities and experiments in the lab and evaluation of the laboratory records carry 50% of the marks of the course.

Best of luck!

SESSION-WISE SCHEDULE OF THE EXPERIMENTS

Day	Session	Experiment
1	I	Introduction
	II	Experiment 1
2	III	Experiment 2
	IV	Experiment 3
3	V	Experiment 4 and 5
	VI	Experiment 6
4	VII	Experiment 7 and 8
	VIII	Experiment 9 and 10
5	IX	Unguided experiment (Term End Examination)

EXPERIMENT 1

INTRODUCTION TO CHEMISTRY LABORATORY

Structure

1.1	Introduction	1.4	Glassware and its Cleaning
	Expected Learning Outcomes		Cleaning the Glassware
1.2	Inside a Chemistry Laboratory	1.5	Simple Laboratory Operations
1.3	Safety Measures in the Chemistry Laboratory		Boring a Hole in a Cork
	Do's and Don'ts in a Chemistry Laboratory		Cutting a Glass Rod/Tube
	Handling Glass Apparatus		Bending a Glass Rod/Tube
	Handling Hazardous Chemicals and Volatile Liquids		Drawing a Glass Jet
			Servicing a Bunsen Burner
		1.6	Summary

1.1 INTRODUCTION

You would have been to a Chemistry laboratory in your school or may have had an opportunity to be in a Chemistry laboratory elsewhere. In this course you are going to perform certain experiments/activities in a Chemistry laboratory. Therefore, we would like to formally introduce you to a Chemistry laboratory. We are sure that you would like to be safe in the laboratory and would agree that the safety of others in the laboratory is equally important. Therefore, we will begin the first experiment of this course by introducing you to a Chemistry laboratory and share with you the precautions to be followed while working in a Chemistry laboratory so that you are safe during your stay in the laboratory. These have been dealt in detail in the course CLT-101. We would briefly recall the first aid procedures also that may be useful in case of an accident while working in the laboratory.

You might have used some of the common glassware in Chemistry laboratory. We would make you have a fresh look at some of them again. Then we would explain the importance of clean apparatus and describe the procedure to clean the apparatus. In this experiment, you would be doing some simple activities like, boring hole in a cork, cutting and bending a glass tube, drawing a jet and servicing a Bunsen burner. In the next experiment you would learn about weighing an object by using analytical balance. Let us lay down the expected learning outcomes of this experiment.

Expected Learning Outcomes

After going through the contents of this experiment and performing the activities described herein, you should be able to:

- ❖ state the measures to be taken for safe working in a Chemistry laboratory;
- ❖ follow the safety measures while working in the laboratory;
- ❖ outline the precautions to be taken while handling laboratory glassware;
- ❖ state the importance of clean glassware and describe methods of cleaning glass apparatus;
- ❖ take necessary precautions while handling hazardous chemicals and volatile liquids;
- ❖ bore a hole of appropriate size in a cork;
- ❖ cut a glass tube/rod and bend it at a given angle;
- ❖ draw a jet from glass tube; and
- ❖ service a Bunsen burner.

1.2 INSIDE A CHEMISTRY LABORATORY

You may have been to a Chemistry laboratory but the laboratory where you would work and perform the experiments of this course may be a totally new setup for you. Therefore, in order to work effectively, you need to get familiar with it. You need to get familiar with the location of the common chemicals, reagents, acids, glass apparatus, gas supply, balances, safety equipment, fire extinguishers and first aid box etc. Your counsellor would take you around the laboratory and show you all the items mentioned above. Listen carefully to her/his instructions. Do clarify if you have any doubts.

When you enter the laboratory you will notice rows of working benches equipped with burners and washbasins along with water taps. This is the place where you are going to perform all your experimental work. The benches may have some shelves with certain chemicals of common use on them. On the sidewalls of the laboratory you may find shelves carrying a number of reagents etc. commonly used by all the students. You may also find a small room called as preparation room adjoining the laboratory. This is where the laboratory staff does all the prior preparations related to the experiments. Fig. 1.1 gives a picture of a modern Chemistry laboratory.

Having got familiar with the laboratory and before actually getting on to the work it is worthwhile to know about the safety measures to be followed in the laboratory.

1.3 SAFETY MEASURES IN THE CHEMISTRY LABORATORY

A Chemistry laboratory is a place, where an accident can occur if proper safety measures are not followed. Most of these accidents are a consequence of carelessness and lack of knowledge about chemicals and the apparatus



Fig. 1.1: A modern Chemistry laboratory showing working benches.

being used. We can minimize them by observing suitable precautions. Yet, the accidents can happen even in the most well run laboratories. You have learnt in detail about the issues related to the possible hazards and the safety measures required while working in a laboratory in the CLT-101 course. Here we are briefly restating some general precautions to be followed while working in a Chemistry laboratory. Let us begin with the general Do's and Don'ts in a Chemistry laboratory.

1.3.1 Do's and Don'ts in a Chemistry Laboratory

- Always wear a lab coat or an apron while working in the laboratory.
- Never wear loose clothes and tie your hair, if you carry long hair.
- Carefully follow the instruction of the counsellor; clarify in case of a doubt.
- Work only during scheduled laboratory hours.
- Put paper trash and broken pieces of glass in dustbins.
- Always clean your work area and put away all glassware before leaving the laboratory.
- Always close the gas and water taps before leaving the laboratory.
- Always label the containers having chemicals or solutions of any kind.
- Know the location of fire extinguishers, and the nearest exit from the laboratory.
- Never perform an unauthorized experiment.
- Never taste or smell chemicals; treat all chemicals as dangerous.
- Use a matchstick and not paper to light the burner.
- Check glassware before using it; do not use broken or cracked glassware.
- Do not eat, drink, chew gum, apply cosmetics, take medications or smoke in the laboratory.
- Don't pipette out corrosive liquids with mouth.

- Never put the used reagents back into their container.
- Avoid contact of chemicals with skin.
- Never keep flammable liquids near an open flame.
- Don't touch electrical switches with wet hands.

In spite of following the precautions mentioned above, the accidents can still occur. Therefore, one has to be prepared for some immediate actions to be performed in case of an accident before calling a doctor, if required. These actions are the **first aid procedures**. You have learnt in detail about these in Unit 11 of the CLT-101 course. These have been briefly given again as an appendix to this course (see p. 73).

1.3.2 Handling Glass Apparatus

A number of accidents can be avoided by proper handling of glass apparatus.

Mishandling of glassware is probably one of the most common causes of accidents in the lab. Accidents and breakage can happen either during assembly/dismantling or during use of glass apparatus. Observing the following precautions while handling glassware can go a long way in minimising laboratory accidents.

- Always use two hands to hold any glassware and position one hand under it for support.
- Use correct type of glassware for a particular application.
- Keep glassware away from the edge of the working bench.
- Tighten the clamps properly. If these are loose, the apparatus may fall and if too tight the glass may crack.
- Do not put too much pressure on the glassware while assembling the apparatus.
- Use properly fitting rubber tubing.
- In case you need to heat the glassware, ensure that the glass can withstand the desired temperature.
- Do not use scratched or cracked glassware as this may pose a risk of flying glass due to explosion or implosion in the apparatus.
- Properly grease the ground glass joints, if any, in the apparatus.
- Take due care to ensure that there is no suck back in the apparatus.
- In case you are using a heating mantle or a steam bath, the glassware or the clamps used to hold glassware might become hot enough to cause a thermal burn on your skin. Handle them carefully.
- Never use worn out cleaning brushes as these can cause scratches on the glassware.
- Always wear closed-toed shoes. These provide protection to the feet not only from the dropped glassware, but also from broken pieces of glass that may be on the floor from a previous lab session.

1.3.3 Handling of Hazardous Chemicals and Volatile Liquids

You need to be aware of the potential danger of the chemicals being handled and also the precautions to be taken in handling them while working in the Chemistry laboratory. The following general precautions should be observed while handling chemicals and volatile liquids in the laboratory.

- Carefully study the label on the container of the chemical as it indicates the nature of the chemical. For example, whether the substance is flammable, explosive, oxidizing, toxic, corrosive or irritant.
- The label also has a risk phrase that underlines and explains the general nature of the risk, and a safety phrase, which gives advice about precautions to be taken while using it.
- Use fume cupboard if the reaction uses or produces harmful gases, dusts or vapours that pose a risk of inhalation.
- The bulk supplies of all flammable liquids should be kept in a solvent store well away from main laboratory building.
- The bottles of flammable liquids must not be placed in direct sunlight as this may result in a fire.
- Never keep flammable liquids near an open flame.

1.4 GLASSWARE AND ITS CLEANING

You have learnt about different glassware used in the Chemistry laboratory in the Units 1 and 2 of the CLT-103 course. Some of the more commonly used glassware is shown in Fig. 1.2. You are advised to acquaint yourself with these.

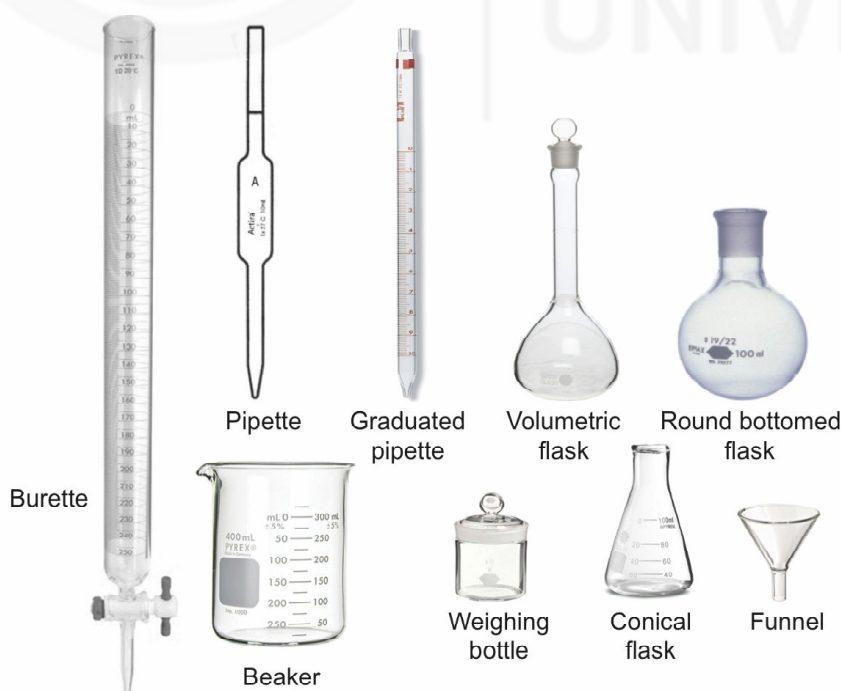


Fig. 1.2: Common laboratory glassware.

Clean glassware is important in every experiment. It is exceptionally important that glassware used for volumetric experiment (burettes, pipettes and volumetric flasks) is highly clean; otherwise there would be an error in volume determination which would affect the results of the experiment. The impurity in the unclean apparatus may also interfere with the experiment. Thus, the reliability of the results of an experiment depends on the cleanliness of the glassware used. The level of cleanliness required depends on the type of the experiment being carried out. Let us learn about the general approach to cleaning the glassware.

1.4.1 Cleaning the Glassware

The general sequence of steps to be followed for cleaning the glassware is as under.

- Generally, the glassware can be cleaned using water and a common laboratory detergent. For this, the apparatus to be cleaned is scrubbed with a little of detergent and washed with plenty of water. Sometimes, a brush may be required to reach the inaccessible portions of the apparatus.
- After washing with tap water, the glassware should be washed with a little distilled water and/or a small amount of acetone and be placed in a cabinet to dry.
- If the stains are not removed by this treatment, the glassware should be immersed in a solution of an oxidizing agent e.g. aqueous 1% potassium permanganate – this is particularly useful for volumetric glassware. Any brown stains remaining after treatment with potassium permanganate solution may be removed with a little concentrated hydrochloric acid.
- Many a times, organic residues or greasy/oily material adhering to the walls of the apparatus do not get removed with detergent. In such cases you may use an organic solvent like acetone to clean. For this, the apparatus is washed with a small quantity of acetone. The washing of glassware with acetone is followed by washing with soap and water as described above.
- Sometimes you may come across some apparatus that does not get cleaned even with an organic solvent like acetone. In such cases, the apparatus can be cleaned by using a solution of chromic acid. However, we would not advice you to try to use it yourself. You may request the laboratory attendant/assistant to exchange your apparatus with a better one.

One of the important things about cleaning glassware is that whatever method is used, apparatus should be cleaned as soon as it has been used.

The acetone used for washing should be kept in a used acetone bottle so that it can be recovered by distillation.

In general, solutions of alkalis should not be used for cleaning volumetric glassware. However, if they are used, all stoppers and taps must be removed before immersing the glassware in the alkali, otherwise they may become fused in place.

1.5 SIMPLE LABORATORY OPERATIONS

In Chemistry laboratory the laboratory assistant needs to perform certain general operations that are the preliminary steps in the experiments to be

performed by the students. Let us learn about and perform a few activities based on these operations:

Activity 1: To Bore a Hole in a Cork/Rubber Cork

While working in a Chemistry laboratory, you will often be required to insert a glass tube or a glass thermometer into a cork. For doing this, you will have to first bore a hole in the cork/rubber cork.

Expected Learning Outcomes

After performing this activity, you should be able to:

- bore a smooth hole of desired size in a cork;
- insert a glass tube or a thermometer into the cork; and
- remove the glass tube or thermometer from the cork.

Requirements

- 1) Cork : 1
- 2) Set of cork borers : 1
- 3) Glass tube/thermometer
- 4) Glycerol or soap solution

Procedure

1. Take a cork of proper size. Soften it by wrapping it in a filter paper and rolling it under foot. (You need not do so for a rubber cork.)
2. Put a mark on both the sides of the cork at the place where the hole is required.
3. Take a borer of a size slightly smaller than the size of the hole required and lubricate it with glycerol or soap solution. The size of the hole required equals the diameter of the glass tube or thermometer to be inserted.
4. Hold the cork with left hand keeping the small side up and the wider base against a firm surface, Fig. 1.3. **Do not use the palm of the hand for this purpose.** Place the borer at the mark, press gently and rotate it clockwise into the smaller end of the cork till it reaches about half way down.

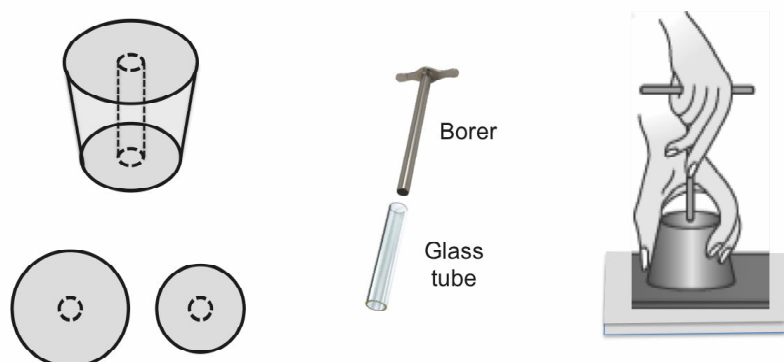


Fig. 1.3: Boring a cork/rubber cork.

5. Remove the borer from the cork by rotating it anticlockwise and pulling it. Then invert the cork and start boring from the other end until the two holes meet. Remove the borer and make sure that the sides of the hole are smooth.

To fit the glass tube or thermometer into the hole in the cork:

1. Dip the end of the cork through which the tube or thermometer is going to be inserted in water/soap solution/glycerin so as to ensure easy insertion of the tube or thermometer into the cork.
2. Hold the cork in left hand and the tube in the right hand and insert the tube into the bored hole of the cork using a rotatory motion.
3. In order to remove the glass tube or thermometer, hold it from close to the cork and pull it gently while rotating anticlockwise.

Hold the tube or thermometer from closer to the cork else it may break and hurt you.

Activity 2: Cutting a Glass Rod/Glass Tube

Glass rods and tubes of different dimensions are routinely required in a Chemistry laboratory. In order to get the glass tube/rod of the desired dimensions, we need to cut it from a larger tube/rod. When we cut or break a glass rod or tube it generally fractures in a highly unpredictable way. In order to produce the right kind of break, we need to form the crack in the required direction as described in the procedure given below. In this activity you would learn about the cutting of glass tube/rod of up to a diameter of 8.0 mm.

Remember that it is better to waste a cork by cutting it rather than to struggle to take out a struck glass rod as it may hurt you.

Expected Learning Outcomes

After performing this activity, you should be able to:

- cut a glass rod/tube of the desired size;
- remove the tongue edges from the cut glass tubes/rods;
- smoothen the edges by fire polishing; and
- state precautions to be taken while cutting a glass rod/tube.

Requirements

- 1) Glass rods or tubes of diameters less than 8 mm and of different lengths
- 2) A piece of cloth
- 3) A triangular file

Procedure

The procedure for cutting a glass rod/tube depends on its diameter. The procedure given below is for the glass rod/tube of a diameter up to 8.0 mm. The procedure for the glass rod/tubes of higher diameter is beyond the scope of this exercise.

1. Take a clean glass rod (or tube) of about 15-20 cm length and place it flat on the workbench.
2. Measure the length of rod required and put a mark for cutting by using a triangular file. For this, keep the glass rod/tube flat on the bench and hold



Triangular file

it firmly with your left hand so as to prevent it from rolling. Place the triangular file at the desired place in such a way that its lower end with a sharp edge is exactly at the place where you want to put the mark.

3. Now, press the file and pull it towards yourself so as to put a single scratch on the tube/rod. You need to do it only once. (Remember you need to put a mark and not cut it as with a saw.)
4. Hold the glass rod/tube with your thumbs in such a way that the mark is facing away from you as shown in the Fig. 1.4. Now break the glass rod by applying pressure away from your body with your thumbs. *It is advisable to wrap the glass rod/tube loosely in a piece of cloth while breaking it.*
5. In case the rod does not break due to insufficient crack developed in the first attempt. Repeat the whole process again by putting the file at the same place and pressing it harder while making the mark.
6. The glass rod breaks neatly into two pieces. However sometimes you may have tongued edges. These can be smoothened by rubbing them against a wire gauge.
7. The cut ends of the glass rod/tube are then made smooth by placing and rotating them gently in the flame by a process called fire polishing.

Caution: In order to avoid injury the breaking of rod should be done as far away from the face as possible.

Heating the sharp edges in the flame to make them smooth is called **fire polishing**.

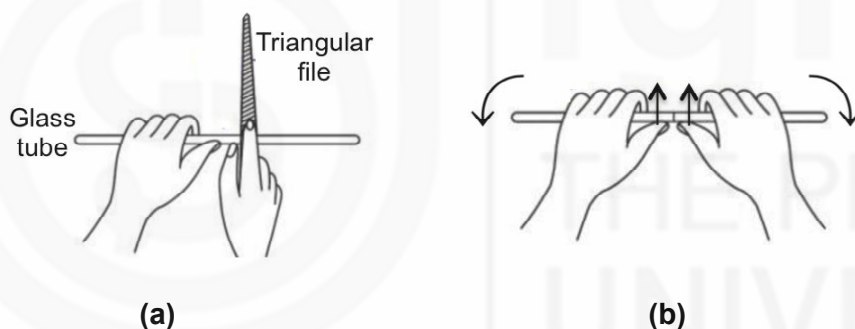


Fig. 1.4: Cutting a glass rod/tube: (a) putting the mark (b) breaking the rod/tube.

Activity 3: Bending a Glass Rod/Glass Tube

Glass rods and tubes of different shapes are routinely required in a Chemistry laboratory. In order to get the glass tube/rod of the desired shape we need to bend it at a suitable angle. In this activity you would learn about bending of a glass tube/rod at a desired angle.

Expected Learning Outcomes

After performing this activity, you should be able to:

- bend a glass rod/tube at the desired angle.

Requirements

- 1) Glass rod and/or tube
- 2) Asbestos sheet or a glazed tile
- 3) Bunsen burner

Procedure

1. Take a clean glass rod/tube of about 20 cm length, hold it from the corners with your both the hands as shown in Fig. 1.5, and heat the portion of the tube where it is to be bent by placing it in the hottest zone of the flame of Bunsen burner.
2. Keep rotating the tube slowly till the portion to be bent becomes soft and red hot. It would start bending by itself.
3. Take the tube out of the flame and apply gentle pressure to bend it to desired angle.
4. Keep the hot bent tube flat on a glazed tile or an asbestos sheet and press it gently to make it coplanar.
5. Allow the tube to cool while it is still on the tile/asbestos sheet.



Fig. 1.5: Bending a glass rod/tube.

Activity 4: Drawing a Glass Jet

In some of the experiments we need a very fine glass tube called a jet. You would be using one of these in the last experiment of this course. The jet can be obtained by heating a glass tube to softness and carefully pulling out to get a narrow tube, which can then be cut, into two to get a pair of jets.

Expected Learning Outcomes

After performing this activity, you should be able to:

- heat the glass tube to softness;
- pull the softened glass tube to narrow the tube;
- cut the pulled glass tube to get the jets; and
- fire polish the sharp edges of the jets.

Requirements

- | | | | |
|----|-----------------|---|---|
| 1) | Glass tube | : | 1 |
| 2) | Bunsen burner | : | 1 |
| 3) | Triangular file | : | 1 |

Procedure

1. Take a glass tube of about 20-25 cm length and 5 mm diameter. Hold it with both the hands and place it in the Bunsen flame as shown in the Fig. 1.6.

2. Slowly rotate the tube clockwise/anticlockwise to ensure uniform heating. Continue heating till the glass tube becomes soft.
3. Take the tubing out of the flame and gently pull the two ends in the opposite direction such that the middle portion of the tube has a diameter of about 1-2 mm.

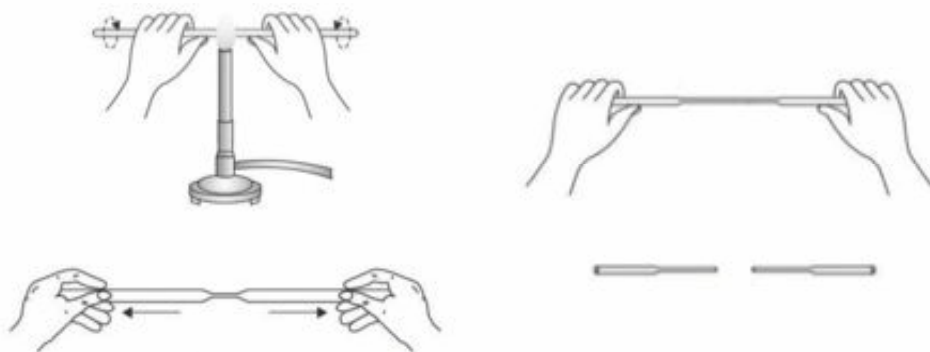


Fig. 1.6: Drawing a jet from a glass tube.

4. Allow the tube to get cool and cut the drawn out portion with a triangular file as described above. This would provide you with two jets.
5. Round off the sharp edges of the jets by placing them in flame for a few moments.

Activity 5: Servicing a Bunsen Burner

You are aware that the Bunsen burner is the most common means of heating in a lab. It is generally neglected because it keeps working even on careless handling. However, due to extensive use it may sometimes get blocked/choked. Therefore, it is desirable that the Bunsen burner is checked and serviced regularly. In Unit 1 of CLT-103 course, you have studied about servicing of Bunsen burners. In this activity, you will be required to actually service a non-working Bunsen burner.

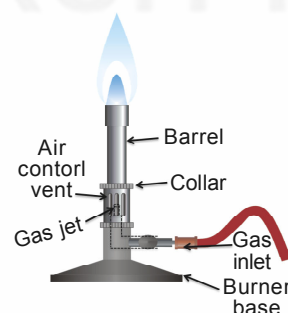
Expected Learning Outcomes

After performing this activity, you should be able to:

- explain the need to service a Bunsen burner;
- dismantle a Bunsen burner;
- clean different parts of a Bunsen burner; and
- reassemble and test a Bunsen burner.

Requirements

- | | | |
|--------------------------------------|---|---|
| 1) Bunsen burner | : | 1 |
| 2) Stove pin or a piece of thin wire | : | 1 |
| 3) Emery cloth or sand paper | | |
| 4) Acetone | | |
| 5) Dilute hydrochloric acid | | |
| 6) Dryer | | |



Bunsen burner

Procedure

The servicing of a Bunsen burner involves three steps as follows:

- I. Dismantling the burner
- II. Cleaning the burner
- III. Reassembling the burner

Perform these steps as per the procedure outlined below:

I. Dismantling the Bunsen Burner

1. Remove the rubber tube of the gas supply — cut off the portion that has developed cracks.
2. Unscrew the gas inlet pipe from the burner base.
3. Unscrew the barrel from the threaded collar in the burner base. This is generally very tight; try to open it carefully. Do not distort the barrel tube by harsh handling, as it would affect the functioning of the burner.
4. Once the barrel has been removed, slide the collar out of the tube.
5. Unscrew the jet from the threaded collar in the burner base.

II. Cleaning the Bunsen Burner

1. Open the blockage in the jet, if any, using a stove pin or a piece of wire.
2. Clean thick deposits, if any, using a fine grade sand paper or emery cloth.
3. Wash the jet in water or with a little dilute hydrochloric acid. Organic deposits may be removed with acetone.
4. Blow the jet dry with compressed air or a dryer.
5. Clean the gas inlet tube in the same way as the jet and wipe the base clean with a dry lint-free cloth. Remove any rubber if present on the tube grip.
6. Remove any deposits from the barrel and collar using a fine grade of emery cloth, and then wash the barrel and collar with water.
7. Thoroughly wipe and blow-dry the tube and the sleeve.
8. Apply a small amount of grease to the inside of the collar to ensure that it moves freely on the barrel.

III. Reassembling the Bunsen Burner

The procedure for reassembling of the burner is the reverse of the dismantling procedure. However, you should note the following:

- It should not be necessary to use grease or sealants when refitting the jet, the gas inlet tube, or the barrel.

- Retighten the barrel to the extent you can do with hand. Do not over tight it else it may get distorted.
- If the gas supply tube is cracked or broken in any way, replace it and tie it with a wire so that it is not easily removed.

Once you have reassembled the burner test it before returning it to store.

1.6 SUMMARY

In the first experiment of this course you have been formally introduced to a Chemistry laboratory and have been apprised of the Do's and Don'ts while working in a Chemistry laboratory. We also introduced you to common laboratory glassware and its cleaning etc. besides taking up the issue of handling of apparatus and chemicals. In addition, you have learnt about and performed five simple activities that the laboratory staff needs to do on a regular basis. These were boring a hole in the cork, cutting and bending a glass tube/rod, drawing a jet and servicing a Bunsen burner.



EXPERIMENT 2

WEIGHING AN OBJECT USING ANALYTICAL BALANCE

Structure

2.1	Introduction	Using an Analytical Balance
	Expected Learning Outcomes	Observations and Calculations
2.2	Types of Balances	Results
2.3	Analytical Balance and its Usage	Precautions
	Principle	2.4 Weighing by Difference
	Requirements	2.5 Summary

2.1 INTRODUCTION

Weighing, the measurement of mass, is one of the most common activities performed in a Chemistry laboratory. It is done with the help of a balance and the type of balance to be used for the measurement of mass depends on the required accuracy. For example, in order to prepare a derivative of an organic compound the required starting material can be weighed on a rough or top loading balance, a balance we see at the grocery shop or at supermarket, as we do not need high accuracy for the mass. On the other hand, while preparing a standard solution in a titrimetric experiment we need to know the mass of the solute accurate upto fourth decimal place. For such accurate measurements of mass, you would be required to weigh the solid using an analytical balance. In this experiment you would learn about different types of balances used in a laboratory and their accuracy. You would then learn about using an analytical balance and actually weigh objects using the same. As most of the errors in the weighing are a result of incorrect handling, you would also learn about the precautions to be taken while weighing with analytical balance.

Expected Learning Outcomes

After going through the contents of this experiment and performing the experiment, you should be able to:

- ❖ list different types of balances used in the Chemistry laboratory and state their accuracy;
- ❖ identify the type of balance required for weighing depending on the accuracy required for a given experiment;

- ❖ identify different parts of an analytical balance;
- ❖ explain the principle of analytical balance;
- ❖ state the importance of leveling of the analytical balance and level an analytical balance;
- ❖ define and determine the zero point of an analytical balance;
- ❖ weigh an object using an analytical balance;
- ❖ use fractional weights and a 'rider' during weighing; and
- ❖ state and follow the precautions while using an analytical balance.

2.2 TYPES OF BALANCES

All measurements of mass (weighing) in the Chemistry laboratory are done with a balance. In a Chemistry laboratory we use different types of balances having varying degrees of sensitivity and accuracy. The choice of suitable balance is made on the basis of the accuracy required for a given experiment. The different types of balances used are:

- Double pan balance
 - Standard laboratory balance
 - Analytical balance
 - Electronic balance
- **Double pan balance** (or rough balance): This is used for preparative work where the required accuracy in mass is of the order of 0.1 g. It is also used for an initial approximate weighing for a more accurate mass determination.
- **Standard laboratory balance** (or top loading balance): This type of balance is also used for an approximate weighing as it has accuracy of the order of 0.02 g. Further, as these balances are not covered the air currents make them less precise also.
- **Student analytical balance** (or chemical balance): It is the most commonly used balance for an accurate determination of mass in quantitative experiments. This has an accuracy of the order of 0.0002 g.
- **Electronic balance**: It is the most sophisticated of the types of balances used in Chemistry laboratories. These are very sensitive and can have an accuracy of 0.00001 g. These are quite expensive and are normally not used routinely by students at school or college level.



Double pan balance



Standard laboratory balance



Student analytical balance



Electronic balance

2.3 ANALYTICAL BALANCE AND ITS USAGE

An analytical balance commonly used in the Chemistry laboratories is shown in Fig. 2.1 with the various parts of the balance suitably labeled. As mentioned

above, an analytical balance is an accurate piece of instrument and can measure mass upto fourth decimal place and has an accuracy of ± 0.0002 g. It is important that you familiarize yourself with the different parts of the analytical balance.

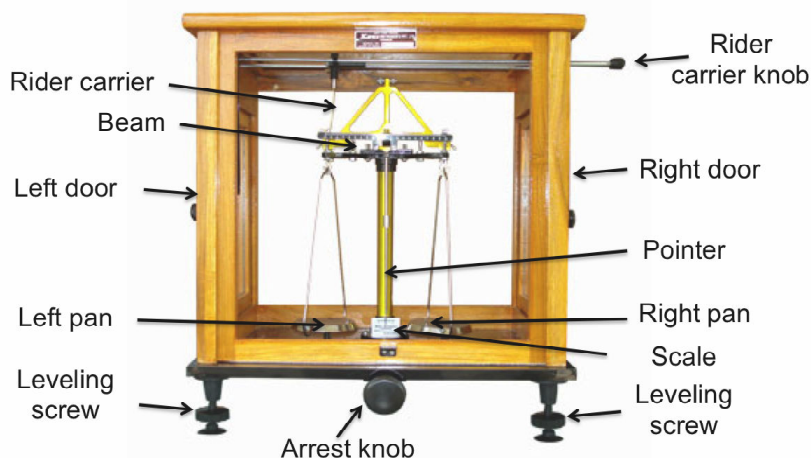


Fig. 2.1: Analytical balance and its different parts.

2.3.1 Principle

A typical balance is a comparison instrument as it determines the mass of an object by comparing it with that of a standard or known mass. Different balances use different ways to do this comparison. We would not get into details of this. We would discuss the principle of the analytical balance, which you are going to use in this experiment. Analytical balance is a two-pan balance; the pans hang at equal distance from the centre of the central beam that pivots (hinges) on a hard plate made of agate or corundum. The object to be weighed is kept on the left hand pan and the standard weights are placed on the right hand pan. The balance is indicated by the pointer that is attached to the centre of the beam and moves over a scale fixed at the bottom of the pillar.



Standard weight box

In Chemistry laboratory for measurement of mass we use weights from a standard weight box. This consists of the weights in the denomination of 1, 2, 5, 10, 20, 50 and 100 g. In addition, a standard weight box contains weights of lesser than 1 g. These are called as **fractional weights**. The fractional weights are of 10, 20, 50, 100, 200 and 500 mg denomination. The fractional weights also have a **rider**, which is used for measurements of mass of less than 10 mg. The way to use rider is explained later.

2.3.2 Requirements

- | | | | |
|----|---------------------|---|---|
| 1) | Analytical balance | : | 1 |
| 2) | Weighing bottle | : | 1 |
| 3) | Watch glass | : | 1 |
| 4) | Standard weight box | : | 1 |

2.3.3 Using an Analytical Balance

In order to minimize the errors in the measurement of mass by using analytical balance, we need to ensure that the balance is stable and properly

leveled. In fact using analytical balance to weigh an object involves three steps, the actual weighing being the third step. These are:

1. Leveling the balance
2. Determining the zero point of the balance
3. Weighing the object

The procedure for these steps is given as under :

Step 1: Leveling the balance

The first and foremost requirement to get correct results from the analytical balance is to ensure that it is leveled. The leveling of the balance is achieved with the help of leveling screws located under the balance. These are on the legs on which the balance rests. You are required to adjust the screws in such a way that the plumb line (adjacent to the central pillar of the balance) is exactly aligned with the pointer on the base behind the scale. You may also make use of a spirit level for the same purpose. Some balances have an inbuilt spirit level or you may use an external one. For this keep the spirit level horizontally on the top of the balance box in such a way that the spirit tube runs parallel to the box. The bubble in the spirit level floats on the top of the tube; adjust the leveling screws and monitor the bubble. The leveling is achieved when the bubble is in the middle of the lines on the tube.



Spirit level.

Step 2: Determining the zero point of the balance

For this purpose, close the side doors of the balance and gently turn the arrest knob anti-clockwise; the pointer starts swinging around the centre of the scale. Carefully observe the extreme positions of the swing and note down the average mean position. This is referred to as the **zero point**. To arrive at the mean position the swing to the right is taken as positive while the one to the left is taken as negative. Further the swings on either side of the scale are first averaged separately and then the average of the two averages is taken to get the zero point. If the zero point is quite far from the middle of the scale you may request your counsellor to adjust it; very small deviations however can be ignored.

Avoid jerks as they may disturb the setting of the balance.

After leveling the balance and determining its zero point, we are equipped to take up the actual weighing. Let us learn about the steps involved in weighing.

Step 3: Weighing the object

- i) To begin with, the object to be weighed is placed on the left side pan by opening the left side door of the balance and the door is closed again. You may use any object like a coin, an eraser, a ring etc., however, we would suggest that you use a weighing bottle or a watch glass for the purpose.
- ii) To get an approximate mass you may weigh the weighing bottle on a rough balance before placing it in the analytical balance.
- iii) Close both the doors and raise the pans of the balance by turning the arrest knob to the left and observe the movement of the pointer.



Weighing bottle.

To get an approximate mass you may weigh the weighing bottle on a rough balance.

- iv) If the pointer moves more to the left, then the weights transferred are in excess of the mass of the bottle. In that case some weights have to be removed. On the other hand, if the pointer moves to the right, then the added weights are not sufficient and we need to add more weights.
- v) Arrest the movement of the beam by turning the knob fully towards the right. Open the right side door to add or remove weight, as the case may be and close the door again.
- vi) Recheck the position of the pointer every time after changing the weights on the right hand pan. Continue this process till the addition of 1 gram weight makes the right hand pan heavier while its removal makes it lighter, e.g., if the weight is say 17.5 g then 17g weight would be lighter and 18 g weight would be heavier.
- vii) Remove the one-gram weight i.e., in the above example put the weight equal to 17 g on the pan and add the fractional weights. These are added in the order of decreasing weights; checking the pointer position every time, till the two pans are balanced. Do not use fractional weights of less than 10 mg; you should use a rider in such cases.

Using a rider

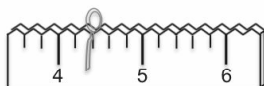
A rider is a thin metallic wire suitably bent so that it can be placed on the beam of the balance. It is normally put on the right hand side of the beam with the help of the rider carrier. By varying the position of the rider on the beam, the rest point is found, i.e., when the two pans are balanced. The beam scale has got markings from 0-10 on either side. It is calibrated in such a way that each main division is numerically equal to mass in milligram, when the rider is put on it. Each main division is further divided into 5 subdivisions and each subdivision is equivalent to 0.2 mg. Thus the accuracy of such an analytical balance can be only up to 0.2 mg.

The mass of an object can be calculated using the following formula:

$$\begin{aligned}
 \text{Mass of the object} = & (\text{Weights added in grams}) \\
 & + (\text{Fractional weights added} \times 0.001) \text{ g} \\
 & + (\text{Main division of the rider position} \times 0.001) \text{ g} \\
 & + (\text{Subdivision of the rider position} \times 0.0002) \text{ g}
 \end{aligned}$$

Example

Let us illustrate the use of this formula. Suppose that while weighing an object, the weights added to the right side pan are 17g, 500 mg, 20 mg, and 10 mg; the rider position be 4th main division and 2nd subdivision on the right hand side.



$$\begin{aligned}
 \text{The mass of the object} &= 17.00 \text{ g} + (530 \times 0.001) \text{ g} + (4 \times 0.001) \text{ g} \\
 &\quad + (2 \times 0.0002) \text{ g} \\
 &= 17.5344 \text{ g}
 \end{aligned}$$

2.3.4 Observations and Calculations

Observations

Mass of weighing bottle (Object 1)

Table 1: Details of the standard weights added (in g)

Standard weight	Number of standard weights used	Total mass of the standard weights (in g)
50 g		
20 g		
10 g		
5 g		
2 g		
1 g		
Total mass of standard weights (g)		

Table 2: Details of the fractional weights added (in mg)

Fractional weight	Number of fractional weights used	Total mass of the fractional weights (in mg)
500 mg		
200 mg		
100 mg		
50 mg		
20 mg		
10 mg		
Total mass of fractional weights (mg)		

Position of the rider:

Main division: _____

Subdivision: _____

Mass of watch glass (Object 2)**Table 3: Details of the standard weights added (in g)**

Standard weight	Number of standard weights used	Total mass of the fractional weights (in g)
50 g		
20 g		
10 g		
5 g		
2 g		
1 g		
Total mass of standard weights (g)		

Table 4: Details of the fractional weights added (in mg)

Fractional weight	Number of fractional weights used	Total mass of the fractional weights (in mg)
500 mg		
200 mg		
100 mg		
50 mg		
20 mg		
10 mg		
Total mass of fractional weights (mg)		

Position of the rider:

Main division: _____

Subdivision: _____

Calculations:

The mass of an object can be calculated using the following formula:

$$\begin{aligned}
 \text{Mass of the object} = & (\text{Mass of the standard weights added}) \text{ g} \\
 & + (\text{Mass of the fractional weights added (in mg)}) \times 0.001 \text{ g} \\
 & + (\text{Main division of the rider position} \times 0.001) \text{ g} \\
 & + (\text{Subdivision of the rider position} \times 0.0002) \text{ g}
 \end{aligned}$$

Mass of the weighing bottle (object 1) =

Mass of the watch glass (object 2) =

2.3.5 Results

The mass of the weighing bottle (Object 1) is:

The mass of the watch glass (Object 2) is:

2.3.6 Precautions

The following precautions must be observed to obtain accurate results of weighing by analytical balance:

- Ensure that your balance is in good condition. In case there is any malfunctioning, it may be reported to the counsellor.
- Keep the pans clean.
- Never release the arrest knob abruptly as this may disturb the setting of the balance and you may not get reliable results.
- Arrest the beam before adding or removing either the sample or the weights.
- Never place the hot objects for weighing on the balance. An object must be cooled to attain the room temperature before weighing.
- Always shut the doors of the balance before raising the pans using the arrest-knob.
- Handle the sample with a spatula. The weights must be picked with forceps only.
- Avoid spilling of chemicals on the pan. Clean immediately in case of a spill.

2.4 WEIGHING BY DIFFERENCE

The measurement of mass of an object can be done in two ways. In the commonly used method, the mass of the empty container e.g., a paper, watch glass, or weighing bottle is measured and recorded. The object/sample is then taken in the container and the combined mass of the object/sample and the container is measured and recorded. The mass of the container is then subtracted from the combined mass to get the mass of the object/sample.

The above method works well for routine measurements of mass of objects. However, in titrimetric experiments where we need to know the mass of the solute transferred for making the solution, the routine method has certain limitations. In such cases, it is advisable to use the '**weighing by difference method**'. For this, we first obtain the approximate mass of the weighing bottle (container). Then the substance to be accurately weighed is placed in the weighing bottle. The substance is first weighed a little more than required on a rough balance before putting into the weighing bottle. The weighing bottle containing the substance is then weighed (m_1 , g) accurately and recorded. The

substance is then transferred into the volumetric flask through a funnel and the bottle is weighed (m_2 g) accurately again. The difference of the two masses, i.e., $(m_1 - m_2)$ g gives the exact amount of the substance transferred.

2.5 SUMMARY

In this experiment you have learnt about the measurement of mass by using an analytical balance. In this process you learnt about different types of balances and the criteria for choosing a suitable balance for a given mass determination. You have also learnt in detail about analytical balance and made measurements using the same.



EXPERIMENT 3

PREPARATION OF SOLUTIONS AND REAGENTS

Structure

3.1	Introduction	Requirements
	Expected Learning Outcomes	Procedure
3.2	Preparation of a Standard Solution	Observations and Calculations
	Principle	Result
	Requirements	3.4 Preparation of Some Laboratory Reagents
	Procedure	Procedure
	Observations and Calculations	Result
	Result	3.5 Summary
3.3	Preparation of a Stock Solution and its Dilutions	
	Principle	

3.1 INTRODUCTION

Preparation of solution is an integral part of a science laboratory. The laboratory assistant is required to prepare different types of solutions and reagents on a regular basis. In the previous experiment you have learnt about determination of mass i.e., weighing by using analytical balance. In this experiment you would learn about preparation of different types of solutions. In this experiment you would put the learning from the previous experiment to use. You would also learn about and perform dilutions of the stock solution. There are three parts of this experiment. In the first part you would learn about and prepare a standard solution. In the second part you would learn about and perform the dilutions of the stock solution. In the third part you would prepare some common laboratory reagents.

You have learnt about solutions in detail in the Unit 4 of the CLT-103 course. You are advised to revisit Sections 4.3 and 4.4 of the Unit before attempting this experiment.

Expected Learning Outcomes

After learning about the contents of this experiment and performing the experiment, you should be able to:

- ❖ define a standard solution and state its types;
- ❖ calculate the amount of solute required to prepare a given standard solution;
- ❖ prepare a standard solution of the desired concentration;
- ❖ define the terms stock solution and dilution;
- ❖ perform the desired dilution of a given stock solution; and
- ❖ prepare some laboratory reagents.

3.2 PREPARATION OF A STANDARD SOLUTION

You have learnt in the Unit 4 of CLT-103 course that a standard solution is defined as the one whose concentration (strength) is known accurately. It is prepared by dissolving an accurately weighed, pure, stable solid substance called solute in an appropriate solvent to prepare a known volume of the solution. There are two types of standard solutions. These are called primary and secondary standard solutions, respectively. A primary standard solution can be prepared by accurately weighing a quantity of the solute (a primary standard) directly, followed by its dissolution in a solvent to give a definite volume of the solution. On the other hand the secondary standard solutions are the ones, which can be prepared by direct weighing but may not have the desired concentration due to the substance being impure or unstable; the solution prepared may also not be stable. For such solutions, the concentration of active substance is found by titrating it against a primary standard solution. The process is called **standardization** and the solution so obtained is called a secondary standard solution. You would learn about this process in the next experiment.

3.2.1 Principle

The preparation of a standard solution involves three distinct steps and all are crucial for the accuracy and reliability of the solution prepared. These steps are:

- Calculation of the mass of solute required for preparing the given volume of the standard solution of the desired concentration.
- Accurate weighing of the solute using a suitable balance.
- Dissolving the solute in sufficient amount of the solvent so as to get the desired volume of the solution.

In order to prepare $V \text{ cm}^3$ of a standard solution of molarity M (mol dm^{-3}), the mass m (g) of the solute of molar mass M_m (g mol^{-1}) required is calculated by the following formula:

$$m \text{ (in g)} = \frac{M \text{ (mol dm}^{-3}\text{)} \cdot V \text{ (cm}^3\text{)} \cdot M_m \text{ (g mol}^{-1}\text{)}}{1000} = \frac{MVM_m}{1000}$$

The solute is then weighed accurately by difference method explained in the previous experiment and is transferred into the volumetric flask with the help of a funnel. It is then dissolved in just sufficient amount of distilled water by carefully swirling the flask. The solution is then made up to the mark by carefully adding more distilled water.

3.2.2 Requirements

Apparatus			Chemicals
1)	Volumetric flask	: 1	Oxalic acid
2)	Weighing bottle	: 1	Distilled water
3)	Funnel	: 1	
4)	Standard weight box	: 1	
5)	Wash bottle	: 1	
6)	Pipette/dropper	: 1	

3.2.3 Procedure

The procedure given below can be used to prepare 100 cm³ of standard solution of about 0.05 *M* oxalic acid.

- i) The amount of oxalic acid required to prepare 100 cm³ of its 0.05 *M* solution can be calculated by using the given formula as:

$$m(\text{g}) = \frac{MVM_m}{1000}$$

Substituting the values in the formula, we get,

$$m(\text{g}) = \frac{0.05 \text{ (mol dm}^{-3}\text{)} \cdot 100 \text{ (cm}^3\text{)} \cdot 126 \text{ (g mol}^{-1}\text{)}}{1000} = 0.63 \text{ g}$$

Thus, a solution prepared by dissolving 0.63 g of oxalic acid in water and making the volume up to 100 cm³ would give a 0.05 *M* solution.

- ii) Weigh a little more than the calculated mass of oxalic acid on a rough weighing balance and transfer it to a clean and dry weighing bottle.
- iii) Weigh the weighing bottle containing oxalic acid accurately. Record the observations.
- iv) Transfer the oxalic acid to a clean volumetric flask of 100 cm³ capacity through a glass funnel.
- v) Weigh the weighing bottle containing oxalic acid left (if any) and find the exact mass of oxalic acid transferred by subtracting this mass from the mass of the weighing bottle plus oxalic acid.

- vi) Dissolve oxalic acid in about 30-40 cm³ of distilled water taken in the volumetric flask. Once dissolved, make the volume up to the mark with distilled water. This can be done initially by pouring distilled water with a wash bottle till the level reaches the neck of the flask and then with the help of a pipette or a clean dropper.
- vii) Stopper the flask tightly and shake well to obtain a homogeneous solution.
- viii) Calculate the actual concentration (molarity) of the solution by using the mass of oxalic acid actually transferred in the formula.

$$M_{\text{Oxalic acid}} = \frac{m \times 1000}{126 \times 100} = \frac{10m}{126} = \dots\dots\dots M$$

(Note: The molarity may be close to 0.05 but may not be exactly 0.05. It is important to be accurate than to be exact.)

Do not discard this solution. You would be using it in the next experiment.

3.2.4 Observations and Calculations

Preparation of standard solution of oxalic acid

Mass of oxalic acid required for preparing 100 cm³ of 0.05M solution as calculated before = 0.63 g

Mass of weighing bottle + oxalic acid = m_1 g = ...

Mass of weighing bottle (after transferring oxalic acid) = m_2 g = ...

Amount of oxalic acid transferred = $(m_1 - m_2)$ g = m g = ...

Molar mass (M_m) of oxalic acid = 126 g mol⁻¹

Volume of oxalic acid solution prepared = 100 cm³

Molarity of oxalic acid solution = $M_{\text{Oxalic acid}} = \frac{m \times 1000}{126 \times 100} = \frac{10m}{126} = \dots\dots\dots M$

(Substitute the value of m and get the molarity of oxalic acid)

3.2.5 Result

The molarity of the standard solution of oxalic acid prepared = ...

3.3 PREPARATION OF A STOCK SOLUTION AND ITS DILUTIONS

You would have noticed that the packaged fruit juices and squashes come as concentrates, which are suitably diluted before consumption. These in fact are stock solutions of the juices. We may define a **stock solution** as a solution

having a relatively high concentration, which can be suitably diluted to some lower concentration for use.

Suppose we need a number of solutions of different concentrations of a given solute, then instead of preparing all the solutions individually, it would be convenient to prepare a stock solution of fairly high concentration (may be ten times or more) and then dilute it appropriately to get the solutions of desired concentrations. This process saves time and also reduces the requirement of storage space. The preparation of stock solutions improves the accuracy also if we need to prepare very dilute solutions. Can you think how?

Yes, in case we need to prepare a very dilute solution the mass of solute required would be very small and there may be errors in its measurement. On the other hand, a stock solution of fairly high concentration can be prepared quite accurately as the measurement of mass in such a case is less likely to be inaccurate.

3.3.1 Principle

The preparation of stock solution is same as explained above for preparation of a standard solution. A standard solution of a given concentration is essentially a stock solution. In order to prepare a solution of concentrations lower than the stock solution, we take a calculated volume of the stock solution and add sufficient volume of the solvent so as to get the desired dilute solution. The process is called dilution. The concentrations and volumes of the stock solution and the desired dilute solution are related as

$$C_1V_1 = C_2V_2$$

Where, C_1 and V_1 refer to the concentration and volume of the stock solution whereas C_2 and V_2 are for the desired dilute solution. The volume of the stock solution required, V_1 for a given dilution could be calculated by using the following formula.

$$V_1 = \frac{C_2V_2}{C_1}$$

The expression given above is a generalised expression for all types of concentration expressions. However, we need to use the same concentration expressions for the stock as well as for the dilute solution. In case we are using concentration in terms of molarity then the corresponding equations would be

$$M_1V_1 = M_2V_2 \text{ and } V_1 = \frac{M_2V_2}{M_1}$$

Let us take an example. Suppose we wish to prepare 100 cm³ of 0.01 M solution of oxalic acid by using the stock solution of 0.05 M oxalic acid prepared above, then the volume of the stock solution required would be

$$V_{0.05M} = \frac{0.01 M \times 100 \text{ cm}^3}{0.05 M} = 20 \text{ cm}^3$$

This means that when 20 cm³ of the 0.05 M stock solution of oxalic acid is diluted to 100 cm³, its concentration would be 0.01 M.

You can take the solution of oxalic acid prepared above and prepare its different dilutions. However, we would suggest that you prepare a stock solution of 0.5 % (w/V) sodium chloride and prepare its dilutions as given under the procedure.

3.3.2 Requirements

Apparatus			Chemicals
1)	Volumetric flask (250 cm ³)	1	Sodium Chloride
2)	Volumetric flask (100 cm ³)	4	Distilled water
3)	Weighing bottle	1	
4)	Funnel	1	
5)	Weight box	1	
6)	Wash bottle	1	
7)	Pipette / dropper	1	
8)	Burette	1	
9)	Burette stand and clamp	1	

3.3.3 Procedure

The procedure given below is for the preparation of 250 cm³ of 0.5 % (w/V) stock solution of sodium chloride and its dilution to prepare 100 cm³ each of 0.1, 0.2, 0.3 and 0.4 % (w/V) solutions of sodium chloride. Essentially, there are two parts of the experiment:

- Preparation of 250 cm³ of 0.5 % (w/V) stock solution of sodium chloride
- Dilution of the stock solution to prepare 100 cm³ each of 0.1, 0.2, 0.3 and 0.4 % (w/V) solutions of sodium chloride.

1. Preparation of the Stock Solution of NaCl

- Calculate the amount of sodium chloride required to prepare 250 cm³ of 0.5 % (w/V) solution. You know that 0.5 % (w/V) solution refers to 0.5 g of NaCl dissolved in water to make 100 cm³ of its solution. The expression to find the mass of solute is given as :

$$\text{Mass of solute} = \frac{[\% (w / V) \times \text{Vol of Solution (cm}^3\text{)}]}{100}$$

Substituting the values in the formula we get,

$$\text{Mass of sodium chloride} = \frac{[0.5 (g / 100 \text{ cm}^3) \times 250 (\text{cm}^3)]}{100} = 1.25 \text{ g}$$

Thus 250 cm³ of a solution obtained by dissolving 1.25 g of NaCl in water would have a concentration of 0.5 % (w/V).

- Accurately weigh 1.25 g of dry sodium chloride by using an analytical balance.

Note: In this case you need to weigh an exact amount of the solute; the weighing by difference may not be a convenient method in this case. You can take a clean and dry weighing bottle and weigh it accurately. Now add more weights equal to the required mass (1.25 g) of sodium chloride on the right hand pan and add sufficient sodium chloride in the weighing bottle to balance it.

The right hand pan would have weights equal to the mass of the weighing bottle plus the mass of NaCl required.

- iii) Transfer the sodium chloride to a clean volumetric flask of 250 cm³ capacity through a glass funnel. Ensure that all the sodium chloride is transferred to the volumetric flask. You may need to rinse the weighing bottle 2-3 times and carefully transfer the washings to the volumetric flask.
- iv) Dissolve sodium chloride in about 30-40 cm³ of distilled water taken in the volumetric flask. Once dissolved, make the volume up to the mark with distilled water. This can be done initially by pouring distilled water with a wash bottle till the level reaches the neck of the flask and then with the help of a pipette or a clean dropper.
- v) Stopper the flask tightly and shake well to obtain a homogeneous solution.
- vi) Label the flask as 0.5 % (w/V) NaCl stock solution.

2. Preparation of Different Dilutions of the Stock Solution

- i) Take four volumetric flasks of 100 cm³ capacity each and label them as 0.1 % (w/V) NaCl, 0.2 % (w/V) NaCl, 0.3 % (w/V) NaCl and 0.4 % (w/V) NaCl respectively.
- ii) The volume of stock solution of 0.5 % (w/V) NaCl required to prepare the desired dilutions can be calculated as follows using the formula

$$V_1 = \frac{C_2 V_2}{C_1}$$

For 0.1 % (w/V)

$$V_1 = \frac{0.1 \% \times 100 \text{ (cm}^3\text{)}}{0.5 \%} = 20 \text{ cm}^3$$

For 0.2 % (w/V)

$$V_1 = \frac{0.2 \% \times 100 \text{ (cm}^3\text{)}}{0.5 \%} = 40 \text{ cm}^3$$

For 0.3 % (w/V)

$$V_1 = \frac{0.3 \% \times 100 \text{ (cm}^3\text{)}}{0.5 \%} = 60 \text{ cm}^3$$

For 0.4 % (w/V)

$$V_1 = \frac{0.4 \% \times 100 \text{ (cm}^3\text{)}}{0.5 \%} = 80 \text{ cm}^3$$

Thus, to prepare 0.1 % (w/V) NaCl, 0.2 % (w/V) NaCl, 0.3 % (w/V) NaCl and 0.4 % (w/V) NaCl from a stock solution of 0.5 % (w/V) NaCl, we need to take 20 cm³, 40 cm³, 60 cm³ and 80 cm³ of the stock solution, respectively and dilute each one of them to 100 cm³. These volumes are indicated in the Observation Table 1.

- iii) Take a clean and dry burette, rinse it with the given stock solution, mount on the burette stand and fill it with the stock solution.
- iv) Take the 100 cm³ volumetric flask marked as 0.1 % (w/V) NaCl and carefully transfer 20 cm³ of 0.5 % (w/V) NaCl stock solution from the burette.
- v) Add distilled water to make up the volume in the flask. This can be done initially by pouring distilled water with a wash bottle till the level reaches the neck of the flask and then with the help of a pipette or a clean dropper.
- vi) Stopper the flask tightly and shake well to obtain a homogeneous solution.
- vii) Repeat step (iv), (v) and (vi) for the rest of the three volumetric flasks of 100 cm³ and add stock solutions as calculated above and indicated in the Observation Table 1.

3.3.4 Observations and Calculations

A) Preparation of Stock Solution

The amount of sodium chloride required to prepare 250 cm³ of 0.5 % (w/V) solution = 1.25 g

Mass of empty weighing bottle = m_1 g = ...g

Mass of weighing bottle + sodium chloride = m_2 g = ... g

Amount of sodium chloride used = $(m_2 - m_1)$ g = m g
= g

Concentration of sodium chloride stock solution prepared =

$$[\% (w / V)] = \frac{\text{Mass of solute (in g)} \times 100}{\text{Vol of Solution (in cm}^3\text{)}} = \dots \% (w/V)$$

B) Preparation of Different Dilutions of the Stock Solution

Observation Table 1: Volume of stock solution (0.5 % (w/V) of sodium chloride required to prepare the desired dilutions

Concentration of the desired dilute solution, C_2	Volume of the desired dilute solution required, V_2 (cm ³)	Volume of the stock solution required, V_1 (cm ³) = $\frac{C_2 V_2}{C_1}$
0.1 % (w/V)	100	20
0.2 % (w/V)	100	40
0.3 % (w/V)	100	60
0.4 % (w/V)	100	80

3.3.5 Result

Prepared a stock solution of 0.5 % (*m/V*) NaCl and made four solutions of 0.1 % (*w/V*), 0.2 % (*w/V*), 0.3 % (*w/V*) and 0.4 % (*w/V*) from it by dilution.

3.4 PREPARATION OF SOME LABORATORY REAGENTS

In this experiment so far you have learnt about the preparation of a standard solution, a stock solution and its dilutions. In these activities you have taken a solute, weighed and dissolved the same in the required amount of solvent to make a solution. As mentioned in the introduction, a laboratory assistant has to prepare different types of solutions on a regular basis. Many of these are termed laboratory reagents. These are prepared and made available to the students working in the laboratory to perform their experiments. Let us learn about and prepare a few laboratory reagents. You would prepare three reagents. These are:

- Fehling solution A
- Fehling solution B
- Starch solution

3.4.1 Procedure

Follow the procedure given below to prepare the required reagents.

Fehling solution A

It is one of the common laboratory reagents prepared by dissolving 69.2 g of copper sulphate pentahydrate crystals in distilled water and making the volume up to 1 dm³ (litre). If the solution is cloudy, 1 cm³ of concentrated sulphuric acid is added to it before making up the volume.

This is the typical procedure for preparing 1 litre of Fehling solution A. You can work in a group of four students and prepare 100 cm³ of the solution. Reduce the amount of the solute required accordingly, i.e., you should take 6.92 g of copper sulphate pentahydrate.

Note: This is not a quantitative solution, so you can weigh copper sulphate pentahydrate on a double pan or a top loading balance. You need not use a volumetric flask. The solution can be prepared in a measuring cylinder or a graduated beaker.

Similarly you can prepare Fehling solution B whose procedure is given below.

Fehling solution B

It is prepared by dissolving 346 g of potassium sodium tartrate (Rochelle Salt) and 140 g of sodium hydroxide in water and making the volume of the solution up to 1 dm³ (litre).

This is the typical procedure for preparing 1 litre of Fehling solution B. You can work in a group of four students and prepare 100 cm³ of the solution. Reduce

the amount of the solute required accordingly. You should take 34.6 g of potassium sodium tartrate and 14.0 g of sodium hydroxide. Weigh them as mentioned above for copper sulphate pentahydrate.

Note: You may note here that in this case there are two solutes. You need to weigh and dissolve both the solutes in water before making up the volume.

Starch solution

The method depends on the type of starch being used and the application of the solution.

- i) Dissolve 5 g of soluble starch in cold water, pour the solution into 2 dm³ of water and boil for a few minutes. Keep the solution in a glass stoppered bottle. This solution is used as an indicator in iodometric titrations. You may use 0.5 g of soluble starch to prepare 200 cm³ of starch solution.
- ii) Alternatively, make a thin paste of the starch (other than soluble) with cold water, and then stir in 200 times its weight of boiling water and boil for a few minutes. A few drops of chloroform is added to the solution as a preservative.

3.4.2 Result

Prepared Fehling solution A, Fehling solution B and Starch solution.

3.5 SUMMARY

In this experiment you have learnt about preparation of solutions for laboratory work as the laboratory assistant is required to prepare different types of solutions and reagents on a regular basis. We started by defining a standard solution and described in detail the procedure for preparing the same. Then we took up the second important aspect of solution preparation i.e., dilution of the stock solutions. In the end we have described the preparation of three common laboratory reagents.

EXPERIMENT 4

SIMPLE ACID-BASE TITRATION

Structure

4.1	Introduction	Requirements
	Expected Learning Outcomes	Procedure
4.2	Titrimetry: Basic Aspects	Observations and Calculations
4.3	Determination of Strength of given Solution of NaOH by Acid-Base Titration	Result
	Principle	4.4 Summary

4.1 INTRODUCTION

In the previous experiment you have learnt about and have acquired the skills of preparing standard solution, stock solution, performing dilution and preparing laboratory reagents. In this experiment you would learn about one of the most common analytical experiments called titration. You would apply the skills of standard solution preparation learnt in the previous experiment and learn newer skill of performing a titration. We would begin by discussing the basic aspects of titrimetry-analytical determinations using titrations. In this context we would define and describe a titration and give a brief account of the type of indicators used in titrations. Thereafter we would explain the determination of the strength of a given solution of NaOH by acid-base titration. You would be required to titrate the standard solution of oxalic acid prepared in the previous experiment with the given solution of NaOH to determine its strength.

Expected Learning Outcomes

After learning the contents of this experiment and performing the tasks described herein, you should be able to:

- ❖ define a titration and describe the process of titration;
- ❖ explain the terms like, titrant, titrand and titre value;
- ❖ define and differentiate between the equivalence point and end point;
- ❖ explain the role of an indicator in a titration;

- ❖ prepare a standard solution of oxalic acid;
- ❖ titrate the given solution of sodium hydroxide with oxalic acid; and
- ❖ calculate the strength of the given solution of sodium hydroxide on the basis of the titration.

4.2 TITRIMETRY: BASIC ASPECTS

Titrimetry is a type of quantitative chemical analysis method in which we perform an operation called a **titration**. A titration involves the determination of the volume of a solution of accurately known concentration (a standard solution), required to react quantitatively with a known volume of the solution of the substance to be determined. The volume and the concentration of the standard solution used are then utilised to determine the concentration of the given solution.

In a typical titration, the given or unknown solution, taken in a burette, is usually added in small lots to a known volume of the standard solution of a suitable reagent taken in a conical flask (it may be reverse also). The solution taken in the burette is called **titrant** and the one taken in the conical flask is referred to as the **titrand**. The solution from the burette is added slowly to the solution in the conical flask till the reaction is just complete. At this stage, called **equivalence point**, the amounts of the titrant and titrand are equivalent. We use a reagent, known as **indicator** to determine the equivalence point. The indicator changes color at or around equivalence point and the stage, at which we observe the change in colour of the indicator, is called **end point** of the titration. The volume of the titrant used in the titration is called **titre value**. As volume measurements play a key role in titration, the analysis involving a titration is also known as **volumetric analysis**.

The stage at which the amounts of titrand and the titrant are equal is called **equivalence point**. The point of a titration indicated by a change of colour of an indicator is called **End point**.

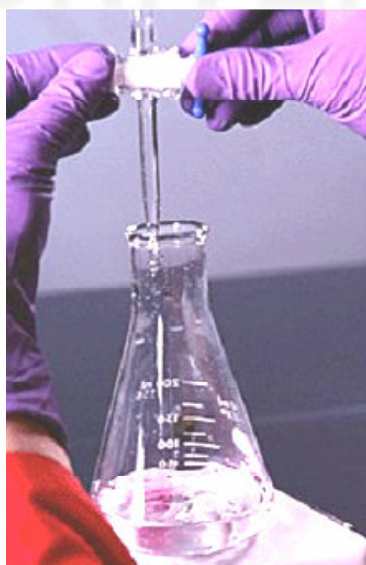


Fig. 4.1: In a titration, a solution of unknown concentration taken in a burette is added to a known volume of the standard solution taken in a conical flask or vice-versa.

There are different types of titrations depending on the type of chemical reaction occurring between the titrant and the titrand. In this experiment you would be performing an **acid-base** or **neutralization titration**. In such titrations solutions of acid and base react to neutralize each other.

An indicator may be defined as a chemical substance that indicates the completion of the reaction between the titrant and the titrand in terms of a colour change. The basic and essential requirement for an indicator is that it should have distinctly different colours before and after the equivalence point in the titration. The type of indicator, to be used in a given titration, would depend on the nature of the chemical reaction involved between the two reacting solutions. Accordingly there are different types of indicators. In this experiment you would be using an acid-base indicator.

The **acid-base indicators** are weak acids or bases and change colour depending on the pH of the solution. Phenolphthalein is a suitable indicator in the titrations of strong bases against strong or weak acids while methyl orange is suitable indicator for the titrations of strong acids against strong and weak bases.

In an alternative way the indicators are classified on the basis of the way these are used. Accordingly, we have the following types:

Internal indicators: These indicators are added directly into the reaction solution and change colour on the basis of the physical property change occurring in the titration. Some of the common examples are: phenolphthalein, methyl orange, diphenylamine, etc.

External indicators: As is evident from the name these indicators are used externally and are not added into the reaction solution. In such cases the indicator is kept out on a grooved porcelain tile. A drop of the solution from titration mixture is taken out with the help of a glass rod and is mixed with a drop of the indicator taken in a groove. A change in colour indicates the end point. Potassium ferricyanide used in the redox titration between ferrous ions and potassium dichromate is one such example.

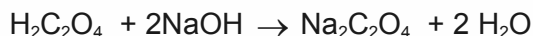
Self indicator: Sometimes either the titrand or the titrant changes its colour at the end point and acts as a self-indicator. Potassium permanganate used in the determination of different reducing agents is one such example. It becomes colourless at the end point.

4.3 DETERMINATION OF STRENGTH OF GIVEN SOLUTION OF NaOH BY ACID-BASE TITRATION

As mentioned above, in an acid-base titration the solutions of acid and base react quantitatively to neutralize each other. As in the present experiment, we need to determine the strength of the given solution of a base (sodium hydroxide) by acid-base titration we would need a standard solution of an acid. You would use oxalic acid prepared in the previous experiment as the primary standard solution in this titration.

4.3.1 Principle

Oxalic acid reacts with sodium hydroxide as per the following equation :



In this reaction one mole of oxalic acid reacts with two moles of sodium hydroxide. The molarities and volumes of NaOH and Oxalic acid for this reaction are related by the following molarity equation.

$$\frac{M_O V_O}{M_N V_N} = \frac{1}{2}$$

Where, M_O and V_O are the molarity and volume of oxalic acid whereas M_N and V_N are the molarity and volume of sodium hydroxide. This equation can be rearranged as follows:

$$M_N = \frac{2M_O V_O}{V_N}$$

Thus, the molarity and hence the strength of sodium hydroxide solution can be obtained by determining the volume (V_O) of M_O molar oxalic acid solution required to neutralize the $V_N \text{ cm}^3$ of the solution of sodium hydroxide.

In this titration we are using a primary standard solution to determine the strength of a secondary standard (NaOH). This process is called **standardisation**.

4.3.2 Requirements

Apparatus			Chemicals
1)	Volumetric flask (100 cm ³)	1	Oxalic acid
2)	Burette (25 or 50 cm ³)	1	Sample solution
3)	Pipette (20 cm ³)	1	(~ 0.1 M NaOH)
4)	Burette stand with clamp	1	Phenolphthalein indicator
5)	Conical flasks	2	
6)	Funnel	1	

4.3.3 Procedure

This experiment has two distinct steps:

- A. Preparation of a standard solution of oxalic acid.
- B. Titration of standard solution of oxalic acid with given solution of sodium hydroxide.

A Preparation of a standard solution of oxalic acid

As mentioned before, here you would be using the standard solution of oxalic acid prepared in the previous experiment. In case you wish to prepare a fresh solution please refer to the procedure given in the Experiment 3.

B. Titration of standard solution of Oxalic acid with given solution of sodium hydroxide

- Take a clean and dry burette and rinse it with the given solution of NaOH.
- Fill up the burette with the given solution of sodium hydroxide by using a funnel and mount it on the burette stand. Note the reading on the burette and record it in the Observation Table 4.1 under the initial reading column.
- Carefully pipette out 20 cm³ of the standard oxalic acid solution and transfer it to a clean 100 cm³ conical flask. Add one drop of phenolphthalein indicator to it.
- Titrate the colorless solution with constant swirling against a white background (you may use a porcelain tile for the purpose) till a pink colour appears which does not fade away on swirling the solution. Record the burette reading in the Observation Table 4.1 under the final reading column.
- Repeat the titration to get at least two concordant readings and record the same in the Observation Table 4.1.

Two consecutive identical readings of a measurement are called concordant readings.

4.3.4 Observations and Calculations**a) Preparation of standard solution of Oxalic acid**

Molarity of oxalic acid solution prepared in the previous experiment

= M

In case you have prepared a fresh solution; record your observations as under Experiment 3.

b) Titration of sodium hydroxide solution with standard Oxalic acid

Volume of standard oxalic acid solution taken, $V_O = \dots \text{cm}^3$

Solution in the burette: NaOH

Indicator used: phenolphthalein

Observation Table 4.1: Titration of Oxalic acid and sodium hydroxide

Sl. No.	Volume of Oxalic acid (cm ³)	Burette reading		Titre value (cm ³) (Final – initial reading)
		Initial	Final	
1	20			
2	20			
3	20			
Concordant reading				

The concentration of the given sodium hydroxide solution can be determined as follows :

The reaction involved: $\text{H}_2\text{C}_2\text{O}_4 + 2\text{NaOH} \rightarrow \text{Na}_2\text{C}_2\text{O}_4 + 2\text{H}_2\text{O}$

Molarity equation: $2M_O V_O = M_N V_N$

$$M_N = \frac{2M_O V_O}{V_N}$$

As the volume of oxalic acid taken was 20 cm^3 , we get: $M_N = \frac{2M_O \times 20}{V_N}$

Substituting the values of M_O , and V_N the molarity of sodium hydroxide =

$$M_N = \frac{2 \times \dots \times 20}{\dots} M$$

The molarity of given sodium hydroxide solution is = ...M

4.3.5 Result

The molarity of the given solution of sodium hydroxide is found to be
= M

4.4 SUMMARY

In this experiment you have learnt about titrations – an important technique of analytical chemistry. You studied the basic aspects of titrimetry in terms of its purpose, common terms involved, the principle of acid-base titration and procedural details for the titration of oxalic acid with a solution of NaOH.



EXPERIMENT 5

PREPARATION OF HYDROGEN SULPHIDE GAS USING A KIPP'S APPARATUS

Structure

5.1 Introduction

Expected Learning Outcomes

5.2 Principle

5.3 Requirements

5.4 Procedure

5.5 Summary

5.1 INTRODUCTION

In qualitative analysis, hydrogen sulphide gas is used as a group reagent for precipitation of the cations of analytical groups 2 and 4. On passing hydrogen sulphide gas into their solution, the cations are precipitated as their sulphides. The laboratory technicians are required to set up the Kipp's apparatus for generating hydrogen sulphide gas, which is then used by the students for qualitative analysis. Therefore, the objective of this experiment is to demonstrate the setting up and working of a Kipp's apparatus for generating hydrogen sulphide gas.

Expected Learning Outcomes

After performing this experiment, you should be able to:

- ❖ set up a Kipp's apparatus for generating hydrogen sulphide or hydrogen or carbon dioxide gas;
- ❖ explain the working of the Kipp's apparatus; and
- ❖ take appropriate safety measures while handling hydrogen sulphide gas.

5.2 PRINCIPLE

In laboratory, hydrogen sulphide is prepared by the action of dilute sulphuric acid on iron sulphide in a Kipp's apparatus:



Fig. 5.1 shows the essential features of a Kipp's apparatus. It is made up of glass or of plastic. Iron sulphide is placed in the middle bulb and the apparatus is filled up with dilute sulphuric acid, with the tap open, so that the solid is just covered. The gas is generated and collected in the middle bulb. When the tap is opened, pressure in the central bulb falls as the gas escapes allowing the acid to rise up into this bulb from the bottom one. This causes the liquid in the top reservoir to flow down the central tube. More gas is produced in the central bulb by the action of the acid on iron sulphide. When the tap is closed, the build up of pressure caused by the evolution of the gas forces the liquid back into the lower reservoir and up the central tube to the upper reservoir. Iron sulphide in the central bulb dries up and no more gas is produced.

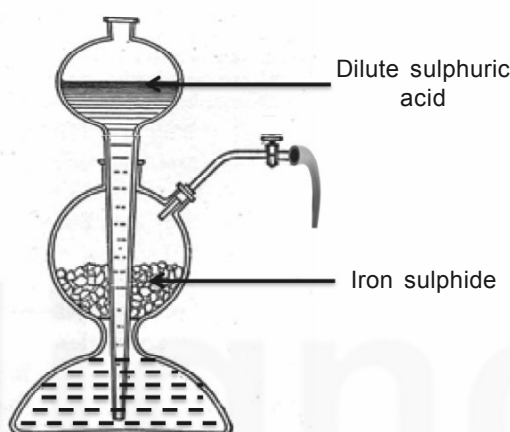


Fig. 5.1: Kipp's Apparatus.

5.3 REQUIREMENTS

<i>Apparatus</i>	<i>Chemicals</i>
1) Kipp's apparatus	Iron sulphide
2) Funnel	Dilute sulphuric acid

5.4 PROCEDURE

Take a clean Kipp's apparatus and fix the upper bulb into the central bulb. Place sufficient amount of iron sulphide into the central bulb and fix the tap (stopcock) into it. With the help of a funnel, add dilute sulphuric acid into the upper bulb till the lower bulb is completely filled and the iron sulphide is just covered with acid. The acid will react with iron sulphide to produce hydrogen sulphide gas which will fill up the middle bulb. The gas can be delivered by opening the tap.

Hydrogen can be generated similarly by putting granulated zinc on top of some glass marbles in the central bulb and filling the apparatus with 50% commercial hydrochloric acid, with the tap open, so that the solid is just covered. As very pure zinc does not react readily with acid, it is necessary to add a few drops of copper(II) sulphate solution to the acid.



Carbon dioxide can be made by putting several lumps of calcium carbonate (marble) on top of glass marbles in the central bulb and filling the lower reservoir, through the top of the upper one, with 50% commercial hydrochloric acid, with the tap open. The acid level should just cover the solid in the central bulb.



Safety Measures

Hydrogen sulphide is a hazardous gas. It is as toxic as hydrogen cyanide by inhalation. Hydrogen sulphide, therefore, should be handled in a fuming cupboard. If a fuming cupboard is not available, it may be kept in an open space outside the laboratory.

Hydrogen sulphide rapidly desensitizes the sense of smell, so that one may be misled into believing that the concentration is diminishing. In high concentrations, it may cause immediate unconsciousness followed by respiratory paralysis; at lower concentrations it causes irritation of all parts of the respiratory system and eyes, headache, dizziness and weakness.

In case of inhalation of the gas, the person should be immediately taken out to fresh air. Provided breathing has not stopped and the person is conscious, there is little else that needs to be done except to maintain careful observation on his/her condition. If breathing has stopped, artificial respiration should be administered until it resumes naturally or medical attention becomes available.

5.5 SUMMARY

In this experiment, you would have performed the following:

- set up a Kipp's apparatus for generating hydrogen sulphide, hydrogen and carbon dioxide gas,
- taken appropriate safety measures while handling the hydrogen sulphide gas.

EXPERIMENT 6

PREPARATION OF DISTILLED/ DEIONIZED WATER AND DETERMINATION OF ITS CONDUCTANCE

Structure

6.1	Introduction	6.4	Preparation of Deionized Water
	Expected Learning Outcomes		Principle
6.2	Determination of Boiling Point		Requirements
	Principle		Procedure
	Requirements		Result
	Procedure	6.5	Measurement of Conductance of Water Sample
	Result		Principle
6.3	Preparation of Distilled Water		Requirements
	Principle		Procedure
	Requirements		Observations and Calculations
	Procedure		Result
	Result	6.6	Summary

6.1 INTRODUCTION

In the previous experiment you have learnt about the preparation of H_2S and a few other gases using Kipp's apparatus. In this experiment you would learn about preparation of distilled and deionized water in the laboratory. This is an important activity that is to be performed by the laboratory staff on a regular basis. A lot of water is required on a daily basis in the laboratory for preparing solutions and reagents. As you are aware that the water supplied through the taps contains dissolved solids and gases and cannot be used as such for preparing solutions etc. We need to purify the water either by distillation or by

deionisation. One of the indicators of the purity of water is its conductance. You would also learn about the measurement of the conductance of a sample of water. You have learnt about distillation and boiling point determination in detail in Unit 5 of CLT-103 course. You are advised to have a relook at the same before taking up this experiment.

In this experiment you would perform four activities. These are :

- determination of boiling point of a liquid,
- study the working of a distillation still/distillation apparatus,
- study the working of a deionisation plant,
- measure the conductivity of a water sample.

In the next experiment, you would learn about preparation of buffer solutions and determination of their pH value.

Expected Learning Outcomes _____

After learning the contents of this experiment and performing the activities described herein, you should be able to:

- ❖ define boiling point;
- ❖ assemble the quick fit apparatus for the determination of boiling point of a liquid;
- ❖ determine the boiling point of a liquid by quick-fit distillation apparatus;
- ❖ determine the boiling point of a liquid by Siwoloboff's method;
- ❖ explain the working of distillation still/water distillation apparatus;
- ❖ use distillation still/water distillation apparatus to prepare distilled water;
- ❖ explain the working of deionisation plant and use the same to prepare deionised water; and
- ❖ use a conductometer and determine the conductance of a water sample.

6.2 DETERMINATION OF BOILING POINT

Boiling point is an important physical characteristics of a liquid. It can be used to ascertain the purity of the liquid and is utilised in the laboratory to identify and characterise a liquid. In this activity you would learn about the boiling point of a liquid and its determination.

6.2.1 Principle

You have learnt in Unit 5 of CLT-103 course that every liquid at a given temperature has certain vapor pressure and the vapour pressure of a liquid increases with an increase in the temperature. The temperature, at which the

vapour pressure of a given liquid equals the atmospheric pressure, is called its boiling point. The boiling point of a liquid can be determined by using a quick-fit boiling point apparatus as shown in Fig.6.1.

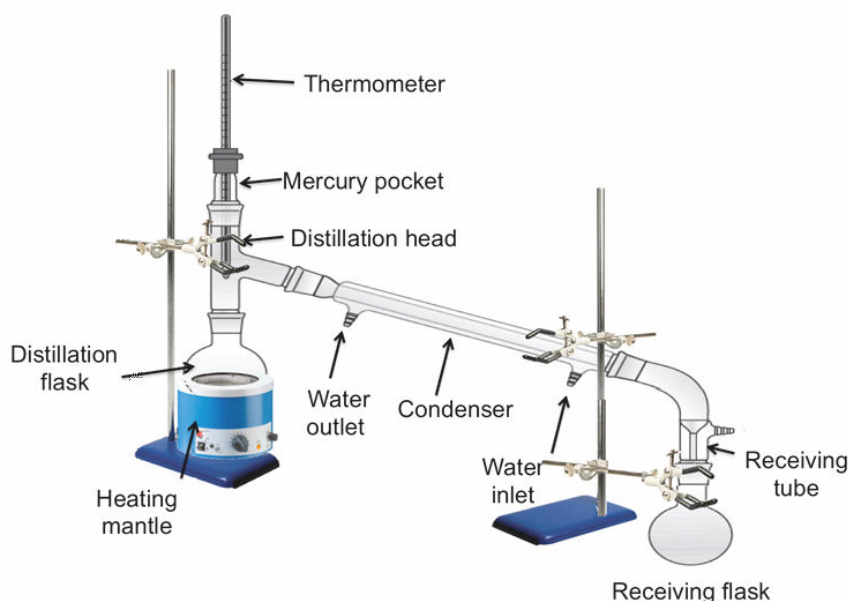


Fig. 6.1: Distillation setup for determination of boiling point of a liquid.

In order to determine the boiling point of a liquid, it is taken in the distillation flask such that the flask is about half full. The flask is clamped on an iron stand and the apparatus is assembled as shown in the Fig. 6.1. The water is supplied to the condenser and the distillation flask is heated continuously using a heating mantle. The temperature of the vapours is monitored with the help of the thermometer in the mercury pocket. When the temperature becomes constant and the distilled liquid falls continuously from the receiving tube the temperature is recorded. It is the boiling point of the liquid.

Siwoloboff's method is the method of choice when the volume of the liquid is very small



Alternatively, the boiling point of a liquid can be determined by a simpler, though less accurate method called **Siwoloboff's method**. In this method, a small volume ($\sim 0.5 \text{ cm}^3$) of the liquid is taken in a micro test tube or an ignition tube and a capillary tube with one sealed end is inserted in it in such a way that the open end is pointing downwards. The test tube containing the liquid and capillary tube is then attached to a thermometer with the help of a rubber band. This assembly is then dipped into a beaker containing paraffin or heating oil as shown in the figure. The beaker is then heated. Initially the trapped air escapes the capillary tube as a stream of bubbles. After some time a fresh rapid and continuous stream of bubbles escapes from the capillary tube. At this stage the heating is stopped and the bubbles slow down and stop. When they stop, the liquid sample is drawn into the capillary tube. At this stage the temperature is measured and recorded as the boiling point.

6.2.2 Requirements

Apparatus			Chemicals
1)	Boiling point apparatus (Quick-fit)	1	Given liquid
2)	Thermometer	1	Mercury

3)	Heating mantel	1	Heating oil / Paraffin
4)	Beaker	1	
5)	Micro test tube / ignition tube	1	
6)	Capillary tube	1	
7)	Tripod stand	1	
8)	Bunsen burner	1	
9)	Wire gauze	1	

6.2.3 Procedure

As discussed above, there are two simple methods of determination of the boiling point of a liquid. The procedure of both the methods are provided over here. You may use any one of these methods as per the availability of the materials in the lab where you are working.

Method 1: Distillation method

- Take a clean distillation flask and fill it about half full with the given liquid and add a few pieces of pumice stone to it
- Clamp the flask properly on an iron stand and insert the distillation head and mercury pocket containing mercury. Ensure that the joints of the distillation head and the mercury pocket are properly greased.
- Now attach the water condenser to the distillation head in such a way that the water outlet is closer to the distillation head and the water inlet is at the farther end. Also tilt the condenser such that the joint for the receiving tube is at lower height than the distillation head (See Fig. 6.1).
- Attach the receiving tube and the receiving flask.
- Provide the water supply from the water tap by using rubber tubing. Attach the tubing to the water inlet. Attach another water tube to the water outlet and place its other end in the washbasin.
- Start heating the distillation flask uniformly with the help of a heating mantel. You would observe that the liquid vapours rise, condense in the distillation head and fall back. The temperature reading on the thermometer starts increasing.
- Continuously monitor the temperature at the thermometer. At certain stage the temperature becomes constant and a continuous stream of distilled liquid is obtained in the receiver. This is the boiling point of the liquid.
- Note and record the boiling point of the liquid.

The pumice stone is added to prevent bumping of the liquid.

The water condenser can be used only if the expected boiling point of the liquid is up to 170-180°C. For high boiling liquids you need to use an air condenser.

Method 2: Siwoloboff's method

The procedure for Siwoloboff's method for the determination of boiling point is as under:

- Take about 0.5 cm³ of the liquid in a micro test tube or an ignition tube and insert a capillary tube with one sealed end in it in such a way that the open end is pointing downwards.
- Attach the test tube containing the liquid and capillary tube to a thermometer with the help of a rubber band. Ensure that the rubber band is tied somewhat away from the bulb of the thermometer.
- Dip this assembly into a beaker containing paraffin or heating oil. Ensure that neither the thermometer nor the test tube containing the liquid touches the beaker.
- Heat the beaker gradually and observe.
- Stop heating when a rapid and continuous stream of bubbles escapes from the capillary tube.
- Note and record the temperature when the liquid sample is drawn into the capillary tube. This is the boiling point of the liquid.

6.2.4 Result

The boiling point of the given liquid by distillation/Siwoloboff's method is found to be: _____ °C. (Indicate the method used by you.)

6.3 PREPARATION OF DISTILLED WATER

As mentioned above, the tap water supplied in the laboratory is not of suitable quality to be used in the preparation of solutions and reagents. In this activity you would learn how distilled water is prepared in the laboratory. In some of the laboratories a distillation still is used for the production of distilled water whereas some laboratories use glass distillation apparatus to do so. In this activity you would observe the working of a distillation still or the glass water distillation apparatus as per its availability in the laboratory in which you are working. Your counsellor would explain the same to you. You are required to observe carefully and understand the operation.

6.3.1 Principle

As regards the principle involved in preparation of distilled water, it is same for both the methods. A distillation basically is a process of purifying a liquid containing non-volatile impurities. The impure sample is heated in the distillation apparatus as described under determination of boiling point above. The hot liquid vapours get condensed in the condenser and the pure liquid is collected in the receiving flask through the receiving tube. The non-volatile impurities are left behind in the distillation flask.

6.3.2 Requirements

Distillation still or Distillation apparatus

6.3.3 Procedure

Your counsellor would demonstrate the preparation of distilled water in the laboratory by Distillation still or Distillation apparatus as the case may be.

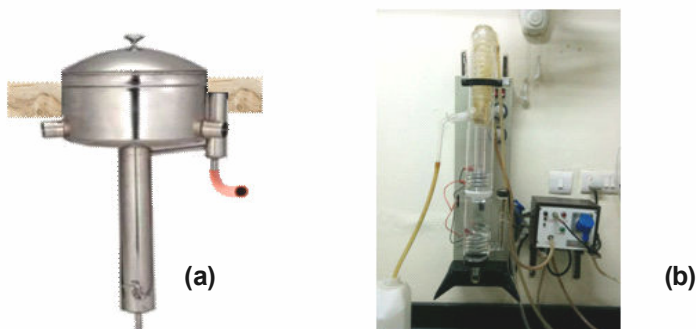


Fig. 6.2: (a) Distillation still; (b) Apparatus for distillation of water.

6.3.4 Result

Observed the working of a distillation still / distillation apparatus.

6.4 PREPARATION OF DEIONIZED WATER

In the previous activity you have learnt about the preparation of distilled water from impure tap water. In preparing distilled water lot of energy is consumed in the process of heating the impure water to make it boil. Also a lot of water is used to pass through the condenser to condense the hot vapours of water. In other words it is an expensive process. For quantitative purposes we do need water of distilled water quality. However, for many of the activities in the laboratory, involving qualitative determinations, it is sufficient to remove the ionic impurities from the tap water. This can be achieved by using a deioniser.

In this activity you would observe the working of a deioniser, if available, in the laboratory at which you are working. Your counsellor would explain the same to you. You are required to observe carefully and understand the deionisation of water and recharging operations of deionizer.

6.4.1 Principle

A deioniser assembly is based on the principle of ion exchange. A typical deioniser may consist of two ion exchange columns. One of these contains cation exchange resin while the other one has an anion exchange resin in it. The tap water containing ionic impurities is passed first through a cation exchanger and then through an anion exchanger. In the cation exchanger the cationic impurities in the water sample are replaced by H^+ ions. The anionic impurities are replaced by OH^- ions in the anion exchanger. Thus, the water coming out of the anion exchange columns is free of both the cationic and anionic impurities. This water can be used for most of the laboratory work.

It may be noted that the deionizers of different brands work on the same principle but may have different operating instructions. You can study the instruction manual of the deionizer to understand its operation.

In some cases the deioniser consists of a single column of a mixed bed ion exchanger. In this case the cationic as well as the anionic impurities in the impure water are removed in a single step and we get a reasonably pure sample of water.

When the ion exchange resins get exhausted, i.e., their capacity to replace the ions in impure water decreases, then these are recharged by passing concentrated solutions of HCl and NaOH, respectively through the cation and anion exchange columns.

6.4.2 Requirements

Deionisation assembly

6.4.3 Procedure

Your counsellor would demonstrate the preparation of deionised water in the laboratory by using deioniser and explain the procedure for recharging of the deioniser.

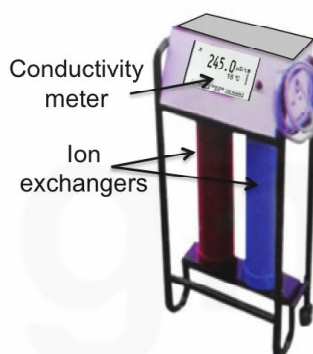


Fig. 6.3: A typical laboratory deioniser.

6.4.4 Result

Observed the working of a deioniser and understood the procedure for recharging of the deioniser.

6.5 MEASUREMENT OF CONDUCTANCE OF WATER SAMPLE

As mentioned above, the tap water supplied in the laboratory contains ionic impurities. Therefore, it has high conductance. The distilled water or the deionised water is freed of these impurities to a good extent and is expected to have lower conductance. Thus, the conductance of a water sample is a measure of its purity or suitability for laboratory usage. A water sample having a conductivity of less than 30 micro siemens is acceptable to be used in the laboratory. In this activity you would learn about and measure the conductivity of a given sample of water. You can do this activity with two water samples. One of these could be the tap water and the other obtained after the distillation or deionisation of the tap water.

6.5.1 Principle

The electrical conductance of a solution is a measure of its current carrying capacity and is determined by the type and concentration of ions present in the

solution. The conductivity of a given solution is determined with the help of an instrument called **conductometer**, Fig. 6.4.

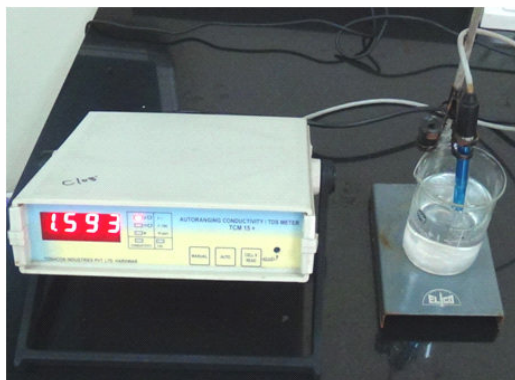


Fig. 6.4: A typical laboratory conductometer.

For this the solution is taken in a beaker; a **conductivity cell** is dipped in it and the conductance is measured. The conductivity cell consists of two platinum electrodes coated with platinum black. These electrodes have an area of cross section equal to 'A' and are separated by a distance 'l'. The ratio of l to A is called **cell constant**.

The conductivity of the solution is given by the following formula:

$$\text{Conductivity} = \text{Measured conductance} \times \text{Cell constant}$$

In a typical conductivity measurement, a solution of known conductivity is taken and its conductance is measured. This provides the value of cell constant as follows:

$$\text{Cell constant} = \frac{\text{Conductivity of the standard solution}}{\text{Measured conductance of the standard solution}}$$

The solution whose conductivity is to be determined is then taken and the conductance is measured. We can then determine the conductivity of the given solution by using the following equation

$$\text{Conductivity of the given solution} = \text{Measured Conductance of the given solution} \times \text{Cell constant}$$

6.5.2 Requirements

Apparatus			Chemicals
1)	Conductometer	1	KCl solution (0.01 M)
2)	Conductivity cell	1	Water samples
3)	Beakers (50 cm ³)	2	

6.5.3 Procedure

There are a large number of manufacturers of conductometers used in the laboratory. All these instruments used in the laboratories have their own operation / instruction manual. You are required to take the instruction manual of the conductometer being used and study its operation. Your counsellor would help you in learning the way to measure conductance with the

conductometer. Follow the following general procedure for the determination of conductivity of the given water sample:

- Clean and connect the conductivity cell with the conductometer; switch on the conductometer and allow it to stabilize for about 15 minutes.
- Wash the conductivity cell and place it in the 0.01 M solution of KCl taken in a 50 cm³ beaker. Ensure that the electrodes of the conductivity cell are completely dipped in the solution.
- Gently swirl the solution in the beaker and measure its conductance as explained in the instructional manual and record your reading.
- Take out the conductivity cell and wash it well with distilled water and place it in the given sample of the water taken in a 50 cm³ beaker. Again ensure that the electrodes of the conductivity cell are completely dipped in the sample.
- Measure the conductance and record.
- Calculate the conductivity of the given sample of water as explained above

6.5.4 Observations and Calculations

Temperature of the laboratory: _____ °C

The measured conductance of 0.01 M solution of KCl = _____ S

Conductivity of 0.01 M KCl (known standard value) = _____ S cm⁻¹

(Your counsellor would provide you the standard value of the Conductivity of 0.01 M KCl at the temperature of measurement)

$$\text{Cell constant} = \frac{\text{Conductivity of 0.01 M KCl (S cm}^{-1}\text{)}}{\text{Measured conductance (in S)}} = \text{_____ cm}^{-1}$$

The measured conductance of the given sample of water = _____ S

The conductivity of the water sample = Measured conductance $\times$ Cell constant

→ The conductivity of the water sample = _____ S $\times$ _____ cm⁻¹ = _____ S cm⁻¹

6.5.5 Result

The conductivity of the water sample is found to be = _____ S cm⁻¹

6.6 SUMMARY

In this experiment you have learnt about the purification of tap water by distillation and by deionisation so that it can be used in the laboratory. In this process we have explained the principles and importance of these techniques. We took this opportunity to introduce the concept of boiling point and explained the determination of boiling point of a liquid. Further, as conductance is one of the criteria for the purity and usability of a water sample in the laboratory, we explained the determination of conductance of a water sample by using conductometer.

EXPERIMENT 7

PREPARATION OF BUFFER SOLUTIONS AND DETERMINATION OF THEIR pH VALUES

Structure

7.1	Introduction	7.3	Measurement of pH of a Solution
	Expected Learning Outcomes		Principle
7.2	Preparation of Buffer Solutions		Requirements
	Principle		Procedure
	Requirements		Observations
	Procedure		Result
	Observations	7.4	Summary
	Result		

7.1 INTRODUCTION

In the previous experiment, you have learnt about the purification of water by distillation and deionisation. In addition, you have also learnt about the determination of boiling point of a liquid and the determination of conductivity of a water sample. In this experiment you would learn about buffer solutions, their preparation and determination of their pH values.

A number of experiments in the laboratory make use of buffer solutions. These are solutions that resist change in their pH on addition of small volumes of acids or bases or on dilution. This experiment consists of two simple activities. These are

- Preparation of a series of buffer solutions and
- Determination of pH of the prepared buffer solutions

In the next experiment you would learn about purification by recrystallization.

Expected Learning Outcomes

After learning the contents of this experiment and performing the activities described herein, you should be able to:

- ❖ define a buffer solution;
- ❖ prepare a series of buffer solutions of different pH values;
- ❖ define pH;
- ❖ write Henderson-Hasselbalch equation for the pH of a buffer solution;
- ❖ explain the principle of determination of pH of a solution;
- ❖ operate a pH meter; and
- ❖ determine the pH of a given solution using a pH meter.

7.2 PREPARATION OF BUFFER SOLUTIONS

As mentioned above a buffer solution can be defined as a solution that resists change in its pH value on addition of small volumes of acids or bases or on dilution. Let us learn about the principle of preparation of a buffer solution.

7.2.1 Principle

The buffer solutions are the mixtures of weak acids or weak bases and their salts. There are two types of buffer solutions. These are acidic buffers and the basic buffers. The acidic buffers are obtained by mixing the aqueous solutions of a weak acid and its salt with a strong base. On the other hand, the basic buffers are prepared by mixing the aqueous solutions of a weak base and its salt with a strong acid. The pH of a buffer solution is given in terms of the following Henderson-Hasselbalch equation.

$$\text{pH} = \text{pK}_a + \log \frac{[\text{Conjugate base}]}{[\text{acid}]}$$

This implies that the pH of the buffer solution depends on the ratio of the concentration of the salt (conjugate base) to that of the undissociated acid. In other words we can vary the pH of the buffer solution by varying this ratio. You are going to prepare acetic acid-sodium acetate buffer by mixing their solutions in different ratios. In the next activity you would learn about and determine the pH values of the prepared buffer solutions.

7.2.2 Requirements

Apparatus			Chemicals
1)	Beakers (100 cm ³)	6	0.2 M Acetic acid solution*
2)	Burettes	2	0.2 M Sodium acetate solution*
3)	Funnels	2	Distilled water
4)	Iron stand and clamp	2	
5)	Wash bottle	1	

* These solutions would be prepared by your counsellor.

7.2.3 Procedure

Follow the procedure given below to prepare a series of buffer solutions of different pH values:

- Take a clean and dry burette and rinse it with the given solution of acetic acid. Fill the burette with acetic acid solution by using a funnel and clamp the burette on the stand. Label the burette as acetic acid.
- Similarly take another clean and dry burette, rinse it with the given solution of sodium acetate. Fill the burette with sodium acetate solution by using a funnel and clamp the burette on the stand. Label the burette as sodium acetate.
- Take four beakers of 100 cm³ capacity and label them as 1 to 4.
- Carefully transfer 16 cm³ of acetic acid and 4 cm³ of sodium acetate from the respective burettes to beaker number 1 and mix the solution.
- Similarly transfer acetic acid and sodium acetate to the other three beakers as per the Observation Table-1 given under observations and stir each solution.

7.2.4 Observations

Observation Table 1 : Volumes of 0.2 M acetic acid and 0.2 M sodium acetate required for preparing the buffer solutions of different pH values.

S.No.	Volume of 0.2 M acetic acid (in cm ³)	Volume of 0.2 M sodium acetate (in cm ³)
1	16	04
2	12	08
3	10	10
4	06	14

7.2.5 Result

Four buffers of different pH values prepared by varying the concentrations of acetic acid and sodium acetate.

(Save these solutions to be used in the next activity)

7.3 MEASUREMENT OF pH OF A SOLUTION

The pH is an important characteristics of an aqueous solution and is defined as

$$\text{pH} = -\log [\text{H}^+]$$

It depends on the concentration of the hydrogen ions in the solution and is a measure of the acidity / alkalinity of the solution. The pH scale varies from 0-14. A solution having a pH of 7.0 at 25 °C is said to be neutral and the solutions with a pH < 7 are acidic while the ones having a pH > 7.0 are

alkaline. The pH of a solution can be measured either by using Universal pH paper or with the help of an instrument called pH meter.

The measurement of pH of a solution with a universal pH paper is very straight forward. We take a small piece of the Universal pH paper and keep it on a porcelain tile. To this we add a drop of the test solution using a glass rod or a fine dropper. The colour developed on the pH paper is compared with the reference colour chart supplied with the pH paper to know the pH. It gives an approximate value of the pH. In order to get the correct value we need to use a pH meter. Let us learn about the principle of a pH meter.

7.3.1 Principle

The measurement of pH of a solution with pH meter is based on the measurement of the potential of an electrode that is sensitive to the hydrogen ion concentration (and hence pH) of the solution. A combination glass electrode is most commonly employed for this purpose. The electromotive potential of the glass electrode is measured by the pH meter and is electronically converted to the pH of the solution.



Fig. 7.1: A typical laboratory pH meter.

Typically the pH measurement by pH meter involves the calibration of the pH meter using standard buffer solutions of known pH values. Once calibrated, then we can directly read the pH of the given solution by dipping the glass electrode in it.

7.3.2 Requirements

Apparatus			Chemicals
1)	pH meter	1	Standard buffer solutions (pH= 4.0 and pH= 7.0)
2)	Glass electrode	1	Sample for pH measurement*
3)	Beakers (50 cm ³)	2	
4)	Wash bottle	1	

* You would use the buffer solutions prepared in the previous activity.

7.3.3 Procedure

There are a large number of manufacturers of pH meters used in the laboratories. All the pH meters used in the laboratory have their own operation / instruction manual. You are required to take the instruction manual of the pH meter being used and study its operation. Your counsellor would help you in learning the way to measure pH with the pH meter available in your laboratory.

Follow the following general procedure for the determination of pH of the buffer solutions prepared by you in the first activity.

- i. Connect the glass electrode to the pH meter; switch on the pH meter and allow it to stabilize for about 15 minutes.
- ii. Take the standard buffer solution of pH=7.0 in a clean beaker and place the glass electrode connected to the pH meter in this solution. Swirl the solution and note the pH as described in the operational manual. It is expected to be 7.0.
- iii. If it is not seven adjust it to 7.0 with the calibration knob.
- iv. Repeat the same process with the standard buffer of pH 4.0.
- v. Repeat step (iii) and (iv) till you get consistent values with the standard buffer solutions. You may take the help of your counsellor or the laboratory assistant for this.
- vi. Measure the pH of the buffer solutions prepared in the previous activity and record the values in the Observation Table given below.

7.3.4 Observations

Observation Table: The measured pH values of the buffer solutions

S.No.	measured pH
1	
2	
3	
4	

7.3.5 Result

The pH of the buffer solutions prepared were measured with pH meter and found to be ____, ____, ____ and ____, respectively.

7.4 SUMMARY

In this experiment you have learnt about and performed two important activities done in the laboratory. These were the preparation of buffer solutions and the determination of their pH value using Universal indicator paper and also by using a pH meter.

EXPERIMENT 8

PURIFICATION OF BENZOIC ACID BY RECRYSTALLIZATION

Structure

8.1	Introduction	Requirements
	Expected Learning Outcomes	Procedure
8.2	Purification by Recrystallization	Result
	Principle	8.3 Summary

8.1 INTRODUCTION

In the previous experiment you have learnt about the preparation of buffer solutions and the determination of their pH values. In this experiment you would learn about one of the important laboratory operations for purification of organic compounds by recrystallization. We would take the example of impure benzoic acid and show that how can we purify it by recrystallization. In the next experiment you would learn about the preparation of potash alum– an important inorganic substance.

Expected Learning Outcomes

After learning the contents of this experiment and performing the same, you should be able to:

- ❖ state the need for purification of organic compounds;
- ❖ define recrystallization;
- ❖ state the criteria for choosing a suitable solvent for recrystallization; and
- ❖ recrystallize an impure sample of benzoic acid.

8.2 PURIFICATION OF BENZOIC ACID BY RECRYSTALLIZATION

One of important types of experiments performed in the Chemistry laboratories is synthetic experiment. In these experiments we start with reactants and

perform certain reactions to get a new product. Typically the products obtained in these reactions are not pure and need to be purified. Recrystallization is one of the methods of purification of solid compounds.

8.2.1 Principle

The basic principle of recrystallization is the difference in the solubility of the desired compound in a given solvent at its boiling point and at room temperature. A suitable solvent for recrystallization of a solid compound is the one in which the desired compound is fairly soluble at its boiling point and is much less soluble at room temperature or the temperatures of ice-water mixture.

The impure compound is dissolved in the suitable solvent at or around its boiling point. The solution so obtained is then filtered to remove any undissolved impurity. The hot filtrate is then allowed to gradually cool down. On cooling, the pure compound separates out as a crystalline solid that can be separated by filtration.

The benzoic acid is soluble in boiling water but does not dissolve in cold water. Therefore, we would use water as a solvent for recrystallization of benzoic acid. We would use a sample of benzoic acid that has been deliberately made impure by mixing with small amount of sand.

8.2.2 Requirements

Apparatus			Chemicals
1)	Beakers (100 cm ³)	3	Impure sample of benzoic acid
2)	Conical flasks (100 cm ³)	2	
3)	Funnels	2	Distilled water
4)	Bunsen burners	2	
5)	Filter paper	Few	
6)	Glass rod	2	
7)	Wash bottle	1	
8)	Tripod stand	1	
9)	Wire gauze	1	

8.2.3 Procedure

- Take approximately 2 g of the given sample of impure benzoic acid in a 100 cm³ beaker and add about 20 cm³ of distilled / deionized water to it and heat it on a burner or a hot plate. Simultaneously keep another beaker containing about 50 cm³ of distilled / deionized water for heating.
- Keep stirring with the help of a glass rod to ensure the dissolution of benzoic acid. Allow the solution to heat upto boiling.

**Filtration**

- iii) Observe whether all the benzoic acid is dissolved. If not, add a little more water and continue. You have to ensure that all the benzoic acid is dissolved in minimum amount of water. Add more water as required but do not add too much of water.
- iv) Arrange for the filtration of the solution by placing a funnel on a conical flask and place fluted filter paper on the funnel.
- v) Add a little of hot water on the filter paper to make it wet and the funnel also becomes slightly warm.
- vi) Quickly pour the hot benzoic acid solution on the funnel to filter it.
- vii) Some of the benzoic acid may crystallise on the filter paper or on the neck of the conical flask. Add hot boiling water from the other beaker in small lots to ensure that all the crystals are dissolved and you get a clear filtrate.
- viii) The impurity of sand would separate out as the residue on the filter paper.
- ix) Add about 10 cm³ of hot distilled / deionized water to the filtrate, cover it with a watch glass and keep it undisturbed for some time. Allow it to cool gradually.
- x) Needle shaped shining crystals of benzoic acid would separate out.
- xi) Set up the filtration apparatus again and filter the crystallised benzoic acid.
- xii) Submit the sample to your counsellor.

8.2.4 Result

Impure sample of benzoic acid was purified by recrystallization and needle shaped crystals of pure benzoic acid were obtained.

8.3 SUMMARY

In this experiment you have learnt about and performed one of very important laboratory operations namely purification of organic compounds by recrystallization. The experiment also involved identification of a suitable solvent for recrystallization and filtration operation.

EXPERIMENT 9

PREPARATION OF POTASH ALUM

Structure

9.1	Introduction	9.5	Observations
	Expected Learning Outcomes	9.6	Calculations
9.2	Principle	9.7	Result
9.3	Requirements	9.8	Summary
9.4	Procedure		

9.1 INTRODUCTION

Potash alum is used in purification of water. Potash alum, $\text{K}_2\text{SO}_4 \cdot \text{Al}_2(\text{SO}_4)_3 \cdot 24\text{H}_2\text{O}$, is an example of a double salt. A double salt is made up of two single salts which crystallize as a single substance, but ionise as two distinct salts when dissolved in water. Ferrous ammonium sulphate, $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$, which is commonly known as the Mohr's salt, is another example of a double salt, which is used in redox titrations. In this experiment, you will learn how to prepare and purify potash alum.

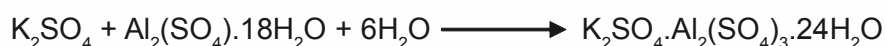
Expected Learning Outcomes

After performing this experiment, you should be able to:

- ❖ prepare a double salt such as potash alum;
- ❖ perform crystallisation; and
- ❖ calculate percent yield of potash alum.

9.2 PRINCIPLE

When a hot concentrated solution of an equimolar mixture of potassium sulphate and aluminium sulphate is allowed to cool, potash alum crystallises out as colorless crystals.



When dissolved in water, the alum furnishes potassium ions (K^+), aluminium ions (Al^{3+}) and sulphate ions (SO_4^{2-}). The names and gram formula weights of main compounds involved in this experiment are listed below.

Name	Formula	Gram Formula Weight $g\ mol^{-1}$
Potassium sulphate	K_2SO_4	174.25
Aluminium sulphate	$Al_2(SO_4)_3 \cdot 18H_2O$	666.42
Potash alum	$K_2SO_4 \cdot Al_2(SO_4)_3 \cdot 24H_2O$	948.76

9.3 REQUIREMENTS

Apparatus	Chemicals
1) Beakers ($150\ cm^3$) 2	Potassium sulphate
2) Bunsen burner 1	Aluminium sulphate
3) China dish 1	Sulphuric acid
4) Glass funnel 1	Distilled water
5) Funnel stand 1	
6) Glass rod 1	
7) Measuring cylinder 1	
8) Porcelain plate 1	
9) Tripod stand 1	
10) Watch glass 1	
11) Wire gauze 1	
12) Filter paper Few circles	

9.4 PROCEDURE

- I) Weigh 5.0 g aluminium sulphate and powder it. Transfer it to a $150\ cm^3$ beaker. Add in $15\ cm^3$ distilled water containing $1\ cm^3$ concentrated sulphuric acid. Warm to dissolve and add more sulphuric acid if necessary to get a clear solution.
- II) Weigh 1.5 g potassium sulphate and powder it. Transfer it to another $150\ cm^3$ beaker. Add $10\ cm^3$ distilled water. Stir to dissolve. Warm if necessary.
- III) Filter the two solutions through a previously moistened paper into a China dish. Heat the solution on a wire gauge and concentrate the solution to the crystallisation point, which can be checked as follows. After every two minutes take some solution on a glass rod and blow out air on it. When a solid crust is formed on the end of the glass rod, this

indicates that the solution has been concentrated to the crystallisation point.

- IV) Cover with a watch glass and allow the solution to crystallise. When the crystallisation is complete, decant the mother liquor and wash the crystals with 5 cm³ ice cold distilled water. Dry the crystals by pressing them gently between pads of filter papers. Allow to dry on a porous plate. Weigh the crystals and record the yield. Calculate the percentage yield.

9.5 OBSERVATIONS

- i) Mass of potassium sulphate taken = ... g
- ii) Mass of aluminium sulphate taken = ... g
- iii) Mass of potash alum formed = ... g
(Experimental yield)

9.6 CALCULATION OF PERCENT YIELD

Your objective should be to prepare as high a yield of potash alum as possible. To measure the efficiency of the procedure and your own performance, you should calculate the percent yield, where

$$\text{Percent yield} = \frac{\text{experimental yield}}{\text{theoretical yield}} \times 100\%$$

The theoretical yield is the calculated maximum amount of product which might be obtained under ideal conditions from the starting materials. In an experiment, the theoretical yield is seldom, if ever, reached. In this experiment, the theoretical yield will be the maximum amount of potash alum which might be obtained from the specified amounts of starting materials.

In calculating the theoretical yield, you should first calculate the moles of each reactant and then find the limiting reagent. The limiting reagent determines the theoretical yield of the product. The reactant that is consumed completely in a chemical reaction is called the limiting reagent.

$$\begin{aligned}\text{No. of moles of Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O taken} &= \frac{\text{g of Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O taken}}{\text{gram formula weight}} \\ &= \frac{5.0}{666.2} = 0.0075 \text{ mol}\end{aligned}$$

$$\begin{aligned}\text{No. of moles of K}_2\text{SO}_4 \text{ taken} &= \frac{\text{g of K}_2\text{SO}_4 \text{ taken}}{\text{gram formula weight}} \\ &= \frac{1.50}{174.25} = 0.0085 \text{ mol}\end{aligned}$$

Since, aluminium sulphate and potassium sulphate react in equimolar ratio, the former is the limiting reagent because it is present in less amount. Thus, the theoretical yield of potash alum is 0.0075 mol. The theoretical yield in gram can be calculated by multiplying the yield in mol by the molecular weight.

$$\text{Theoretical yield} = 0.0075 \times 948.26 = 7.1 \text{ g}$$

$$\text{Percent yield} = \frac{\text{experimental yield}}{7.1} \times 100\%$$

9.7 RESULT

The percent yield of potash alum = ... %

9.8 SUMMARY

In this experiment, you have performed the following:

- prepared potash alum,
- crystallized potash alum, and
- calculated the percent yield of potash alum.



EXPERIMENT 10

SEPARATION OF PIGMENTS FROM LEAVES BY PAPER CHROMATOGRAPHY

Structure

10.1 Introduction

Expected Learning Outcomes

Procedure

Observation and Calculations

10.2 Separation of Pigments by Paper Chromatography

Result

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10.3 Summary

10.1 INTRODUCTION

In the previous experiment you have learnt about the preparation of potash alum. In this last exercise of the course you would learn about paper chromatography—an important separation technique. You would use this technique to separate the pigments from the extract of leaves (or flowers).

Expected Learning Outcomes

After learning the contents of this experiment and performing the activities described herein, you should be able to:

- ❖ define chromatography;
- ❖ explain the principle of paper chromatography;
- ❖ set up apparatus to perform paper chromatographic experiment;
- ❖ prepare a leaf / flower extract;
- ❖ mark a spot of the leaf extract on the chromatographic paper; and
- ❖ develop a chromatogram and determine the R_f values of different components separated on the chromatogram.

10.2 SEPARATION OF PIGMENTS BY PAPER CHROMATOGRAPHY

You would have noticed that the leaves of plants change colour with change in season. In the summer season these are green due to the pigment chlorophyll that is responsible for photosynthesis. In autumn the colour of leaves changes due to formation of newer pigments of yellow, orange or red colour. The extraction and separation of different compounds from natural sources is an important class of experiments performed in the laboratory. In this experiment you are going to extract some pigments from green plant leaves (or flowers) and separate them by paper chromatography.

10.2.1 Principle

The chemical nature of the pigments present in natural material is such that these are soluble in organic solvents like acetone. Therefore, to extract these pigments we crush the leaves and mix with acetone so that the pigments get extracted into acetone. The leaf extract contains a number of pigments that can be separated by paper chromatography.

In Unit 6 of CLT-103 course you have learnt about chromatography, its mechanism and types. You are advised to revisit Unit 6 before working on this experiment. You would recall that paper chromatography is a type of partition chromatography. Different components get separated on the basis of their distribution between the stationary and mobile phases (discussed in Unit 6). In a typical extraction the leaves are crushed and mixed with some acetone. The extract so obtained is separated by paper chromatography. The positions of different spots obtained on the chromatogram are characterised in terms of their R_f value which is defined as

$$R_f = \frac{\text{Distance travelled by a given component}}{\text{Distance travelled by the solvent}}$$

and is illustrated in the Fig. 10.1.

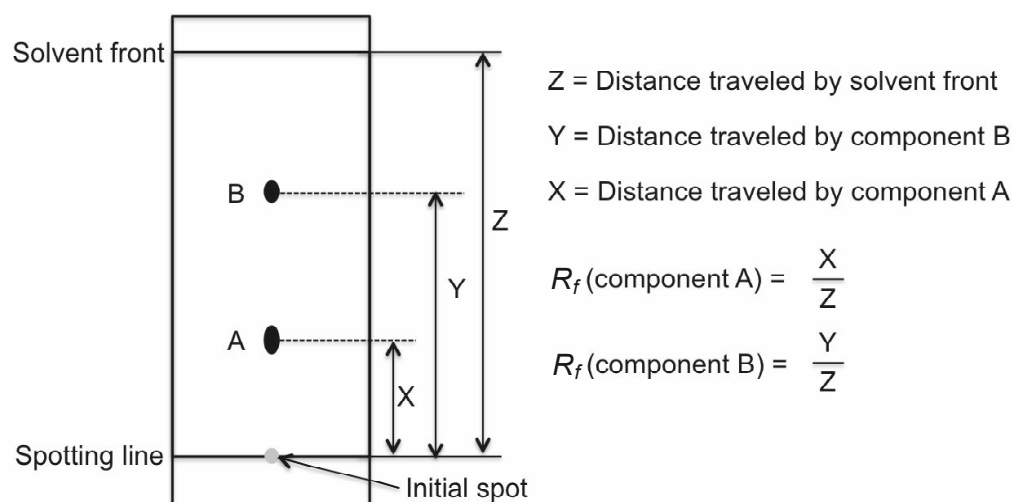


Fig. 10.1: Illustrating R_f value.

10.2.2 Requirements

Apparatus			Chemicals
1)	Pestle and mortar	1	Green leaves / flowers
2)	Chromatographic paper	1 strip	Acetone
3)	Chromatographic jar	1	Sand
4)	Watch glass	1	
5)	Pencil	1	
6)	Scale	1	
7)	Jet / capillary tube	1	
8)	Scissors	1	
9)	Dropper	1	

10.2.3 Procedure

- Take a strip of chromatography paper (this would be cut by the laboratory staff from a sheet of chromatography paper) and draw a line, using a pencil, about 2 cm from the bottom end of the strip.
- Take a measuring cylinder or a chromatography jar and pour acetone into it such that the level is about 1.0 cm from the bottom and cover the jar with a watch glass.
- Now, take some fresh green leaves (you may take some coloured flowers instead); cut them into small pieces with scissors and place them in a mortar.
- Add a pinch of sand and about 1-2 cm³ of acetone (propanone) using a dropper and grind the mixture with a pastel till you get a paste.
- You would observe that the acetone would acquire green colour due to extraction of pigments from the leaves. This is called the **leaf extract**.
- With the help of a fine jet or a capillary tube, take the leaf extract and make a small spot of the leaf extract on the centre of the line drawn on the chromatography paper.
- Allow the spot to dry, make another spot just on the top of the previous spot. Allow it also to dry. Repeat the process 4-5 times so as to get a reasonably concentrated spot on the paper.
- Hang the chromatography paper in the chromatography jar such that its lower end dips in acetone. *Make sure that the level of acetone is below the spot.*
- Cover the chromatography jar again and leave it for some time so that the acetone rises on the chromatography paper till about the top of the paper.



The solvent (acetone) rises due to capillary action.

- x) Mark the level, to which the solvent (acetone) rises, with the help of a pencil.
- xi) Allow the paper to dry.
- xii) Measure the distance moved by the solvent and different coloured spots separated from the line drawn on the chromatographic paper.
- xiii) Determine the R_f value of different pigments separated on the chromatogram.

10.2.4 Observations and Calculations

Calculate the R_f values of the separated components by using the formula

$$R_f = \frac{\text{Distance travelled by a given component}}{\text{Distance travelled by the solvent}}$$

R_f value of component 1=

R_f value of component 2=

10.2.5 Results

- a) The pigments in a sample of green leaves were extracted with acetone and these were separated by paper chromatography.
- b) The R_f values of the separated pigments is found to be:

R_f value of component 1=

R_f value of component 2=

(You are required to submit the paper chromatogram developed by you to the coordinator).

10.3 SUMMARY

In this last experiment you have learnt about an important separation technique called paper chromatography. The experiment involved an activity of extraction of pigments from a natural source (plant leaves) followed by their separation by paper chromatography. You also determined the R_f values of the separated components of the leaf extract.

APPENDIX FIRST AID PROCEDURES

You would agree that we can minimize the accidents in a laboratory by following the precautions but we can't eliminate them. There is always a probability of accidents to occur. It is essential, therefore, that necessary preparations should be there to cope up with any eventuality. The relevant first aid procedures for the common accidents likely to occur in a Chemistry laboratory are given below. These are not the treatments for the accidents but are mere procedures that would minimize the possible damage from the accident. A timely first aid can, at times, provide more relief than a delayed medical aid.

I. Chemical Burns

These are caused by accidental exposure to acids, bases and other corrosive chemicals. The long-term effect of these burns is scarring and, depending on the site of the burn, can be quite harmful/dangerous. Phenols are exceptionally dangerous. These are highly caustic and are absorbed very rapidly through the skin.

First aid

- The first step in such a situation is to remove the offending agent (chemical) at the earliest.
- Remove contaminated clothes, longer they stay on the body, greater is the damage.
- Remove rings, watches and other ornaments in the affected area as these may trap the contaminants.
- Wash the affected portion under running water for about 10 - 15 minutes to limit the tissue destruction.
- Take care that the washings do not go onto the unaffected portion of the skin. Ensure your safety also.
- Avoid using neutralising solutions. These generate heat, which increases damage.

In case the corrosive chemical falls in the eye, it is a case of an ocular burn. The following procedure is to be observed in such cases:

- Remove the contact lens, if it is there, as the chemical can get in between the lens and cornea and cause damage. Do not let the patient rub his / her eyes.
- Wash the eye gently with running water from the tap or an eyewash station for 10 to 15 minutes.
- Open the eyelids and ensure that water drenches the eyeball.
- All eye injuries must be seen by a doctor; preferably within an hour.

II. Thermal Burns

There can be two types of burns. The burns caused by direct contact with open flame or contact with a hot object like a hot flask etc., are called contact burns. In such a case the injury is confined to the point of contact. On the other hand the burns that result from contact with hot liquids are called scalds.

First aid

- Remove the victim from the source of the burn. Cool the burn using large amounts of cool water.
- Do not use ice except in small superficial burns. It may cause body heat loss.
- Once the burn has been cooled, cover the burn using dry, sterile dressing or the cleanest cloth available.
- Refer patients with critical burns to a hospital burn centre for further management.

III. Cuts and Wounds from Broken Glass

Cuts and wounds are probably the most likely accidents in a Chemistry laboratory. These result from using broken or cracked glassware; forcing glass tubes or rods through corks, or accidental breaking of apparatus etc. The injury may vary from a minor cut to a profusely bleeding wound.

First aid

- In case of a minor cut or abrasion, clean the wound and surrounding skin with soap and water.
- To avoid contamination, wash away from the wound and not towards it.
- Pat the wound dry, with a sterile pad or the cleanest cloth available and, apply a suitable bandage as required.
- For a major wound involving profuse bleeding, apply firm pressure over and around the wound with a sterile pad or the cleanest cloth available, to reduce bleeding as much as possible.
- Make the victim lie down with bleeding part raised higher than rest of her / his body.
- Apply a tight bandage and call for the physician.
- Meanwhile keep the victim warm and give her / him (if conscious) a lot of nonalcoholic liquid to drink.

IV. Ingestion of Chemicals

Incidents of ingestion of chemicals are far less common than the ones involving other routes of exposure. However, significant quantities of toxic chemicals can be ingested e.g. in a mouth-pipetting experiment.

First aid

- If the ingested corrosive material is confined to mouth only then repeated washing of the mouth is probably the best way.
- However, if the chemical has been ingested, give about 250 cm³ of water to dilute the contents.

- Do not try to neutralise the chemical as the heat generated may cause additional injury.
- Do not try to induce vomiting. This may cause injury to the delicate tissues of the esophagus.
- Arrange for the medical help, better transfer the victim to the hospital.

V. Inhalation of Gases and Vapours

Inhalation of toxic gases and obnoxious vapours of certain volatile liquids is another common situation in a laboratory that demands prompt first aid. Inhalation of gases and vapours can cause varied effects ranging from irritation of eyes and mucous membrane to drowsiness to unconsciousness.

First aid

- Move the victim immediately to fresh air.
- If the victim is unconscious, place in a face down position and check for the breathing.
- On the first sight of difficulty in breathing or stopped breathing apply artificial respiration immediately, (mouth to mouth method).
- Keep the patient warm and still till the help arrives.
- Call for the medical help. If needed transfer the victim to the hospital.

VI. Chemical Splashes

The splashing of chemicals on the skin or in the eyes is quite common in a Chemistry laboratory. No chemical can be considered totally hazard free, though some are relatively safer. It is good, therefore, to regard all chemicals as hazardous and proceed for the first aid.

First aid

- Wash the affected portion immediately under running water for about 10-15 minutes. Take care that the washings do not go onto the unaffected skin.
- Remove contaminated clothes, rings, watches and other ornaments in the affected area.
- In such cases the quickness with which the splashed chemical is removed really matters. However, do not get affected in the process.
- If situation is serious, a medical advice should be sought.

For the splashing of the chemical into the eye, proceed as instructed under chemical burns.