LABORATORY MANUAL

for

5-Year Integrated *(B.Sc. - M. Sc.)* Botany

Fundamentals of Chemistry

DEPARTMENT OF BOTANY

UNIVERSITY OF KASHMIR, NORTH CAMPUS, DELINA, BARAMULLA

1.1.OBJECTIVES

This course is designed to ensure that all the students regardless of their educational backgrounds are competent in necessary laboratory skills. These skills include but are not limited to the use of an analytical balance, volumetric glassware, various pipettes, performing titrimetric and learning the proper use and calibration of microscopes and centrifuges. Students should also become familiar with using laboratory apparatus.

Use the proper laboratory techniques to do the following;

- Pour liquids from a glass-stopper bottle.
- Transfer solids from a bottle.
- Heat liquids in a beaker.
- Heat liquids in a test tube.
- Light and adjust a Bunsen burner.
- Measure to 0.1 cm with a metric ruler.
- Use a graduated cylinder to measure volume.
- Use an analytical balance to measure mass.

1.2. *INTRODUCTION*

Introduction to basic laboratory techniques and procedures necessary for competent performance. Topics will include laboratory Reagents, apparatus, Glassware laboratory safety, volumetric and gravimetric measurements, titrations, critical evaluation of data, laboratory mathematics, preparing solutions and dilutions. The purpose of this experiment is to introduce several of the tools andtechniques necessary for success in this course.

Chemistry is an experimental science, and the laboratory is where you learn about “how we know what we know about it.” The laboratory deals with the processes of scientific inquiry that organic chemists use. It demonstrates the experimental basis of what your textbook presents as fact. The primary goal of the laboratory is to help you understand how Chemistry is done by actually doing it. Learning how to obtain and interpret experimental results and draw reasonable conclusions from them is at the heart of doing science. Your laboratory work will give you the opportunity to exercise your critical thinking abilities, to join in the process of science.

1.3. *LOCATION OF LABORATORY EQUIPMENT*

(a) Chemicals and Solvents Organic and Inorganic: Acids and Bases - under hood Solvents - on shelves at end of benches

(b) Ovens and Refrigerators: Each oven is designated for a specific purpose. Do not place any plastic items in the ovens. All samples must be clearly labelled with the identity of compound, your name and date. Ovens will be cleared weekly and improperly labelled samples will be removed. Refrigerators. Samples must be clearly labelled.

(c) Balances: Abuse of balances and littering of the area will not be tolerated.

1.4. LABORATORY NOTE BOOK

1.4.1. General Guideline:

1. Use a ballpoint pen (press hard if duplicate pages). Write on one side only.
2. Do not erase or use whiteout. If you make a mistake, draw a single line through the error and write the correct entry on the top or side of it.
3. Do not remove an original page. If the entire page is incorrect, draw a single diagonal line through the page and state the reason for this line.
4. Record all data and results (with units) directly into your notebook.
5. Do not record data on scrap paper, your hand, etc., to be transferred later.
6. Start a new page for each new experiment.
7. Write the title of the experiment, date, and your name at the top of each page.
8. Indicate if a page is continued from the previous page.
9. Never skip a space for later additions.

1.4.2. COMPONENTS

A. Pre-Lab – a detailed plan of the work that you will be doing:

1. Brief statement of purpose.

2.Paragraph discussion of the safety and environmental issues (ex. waste generation).

3.Step-by-step procedure in your own words. Be concise and complete, but do not cop the lab manual. Use diagrams and sketches when necessary. Reference all sources of Information.

Note: The lab manual may not be brought into the laboratory or consulted during the laboratory session. However, the appendices are allowed.

B. Factual Record – what to record:

Keep a running account of all procedures carried out and observations made during experimental work.

1. Record observations such as physical appearance, colour, odour, and physical properties.
2. Sketch apparatuses and label parts.
3. Use a table to record all information about reactants (see below).
4. Record all data and results, including the crude yield of products and mixtures. Use Tables when possible.
5. All of the reactants must be accounted for in the factual record. For example, if you started with 1.0 mol of Reactant 1, you must account for the fate of all 1.0 moles at the end of the reaction. Simply describing the isolated 0.25 mol of product at the end (for example) will not be acceptable.
6. For calculations, show the formula and a sample calculation. If the calculation is repeated; use a table to report your results.

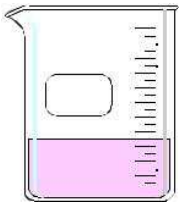
Data Analysis/Conclusions:

Examine and discuss the accuracy and precision of your data. Is the precision reasonable? Discuss possible systematic and random errors. Summarize the key results and provide a conclusion. Describe any difficulties that you had. Discuss which results are poor and provide explanations. Provide suggestions for improvement.

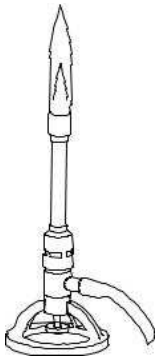
LABORATORY APPARATUS AND OPERATION

A. Common Laboratory Apparatus:

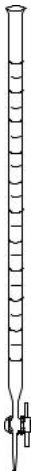
Beakers are useful as a reaction container or to hold liquid or solid samples. They are also used to catch liquids from titrations and filtrates from filtering operations.



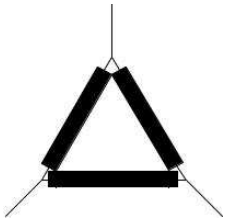
Bunsen Burners are sources of heat.



Burettes are for addition of a precise volume of liquid. The volume of liquid added can be determined to the nearest 0.01 ml. with practice.

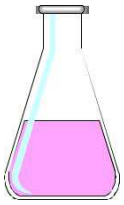


Clay Triangles are placed on a ring attached to a ring stand as a support for a funnel, Crucible, or evaporating dish.



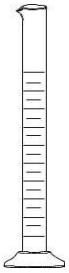
Droppers are for addition of liquids drop by drop

Erlenmeyer Flasks are useful to contain reactions or to hold liquid samples. They are also useful to catch filtrates.

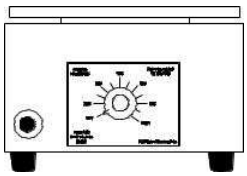


Glass Funnels are for funnelling liquids from one container to another or for filtering when equipped with filter paper.

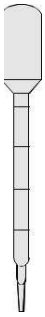
Graduated Cylinders are for measurement of an amount of liquid. The volume of liquid can be estimated to the nearest 0.1 m with practice.



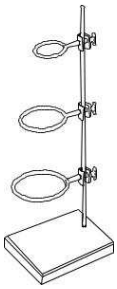
Hot Plates can also be used as sources of heat when an open flame is not desirable.



Pipets are used to dispense small quantities of liquids.

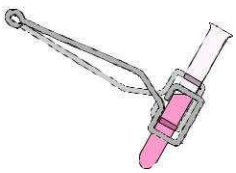


Ring stand with Rings are for holding pieces of glassware in place.



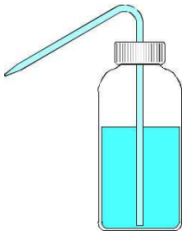
Test Tubes are for holding small samples.

Test tube holders are for holding test tubes when tubes should not be touched



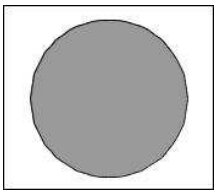
Volumetric Flasks are used to measure precise volumes of liquid or to make precise dilutions.

Wash bottles are used for dispensing small quantities of distilled water.



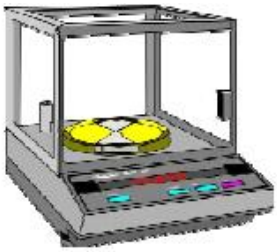
Watch glasses are for holding small samples or for covering beakers or evaporating dishes.

Wire Gauze on a ring supports beakers to be heated by Bunsen burners are used to determine the mass of a reagent or object.



B. Laboratory equipment:

Balances are used to determine the mass of a reagent or object.



Spectrophotometers are used to measure the absorbance or transmittance of a liquid sample.

Fume Hoods are used to ventilate noxious or harmful gases

LABORATORY SAFETY

GENERAL SAFETY RULES:

1. The safe way is the right way to do your job. Plan your work. Follow instructions. If you do not know how to do the experiment safely, ask your teaching assistant.
2. Be able to use all safety devices and protective equipment provided for your use and *know their location* (eyewash fountain, shower, fire blanket, fire extinguisher).
3. Safety goggles must be worn at all times.
4. *Do not* eat or drink in the laboratory (and do not store food in the refrigerators). Smoking in the laboratory is absolutely forbidden.
5. Horseplay in any form is dangerous and prohibited. Do not run in laboratory areas.
6. Report to your TA all unsafe conditions, unsafe acts, and "near misses" that might cause future accidents. Report any accident or fire, no matter how trivial, to the TA.

Hazardous Chemicals:

a) Be especially mindful of fire hazards when you or your lab neighbors are working with flammable liquids.
b) Hazardous Substances: Know common explosive, toxic, and carcinogen materials and use them only with adequate safeguards.
c) Never leave a reaction or experiment running unattended, unless you have told your lab partners enough about it to deal with potential hazards while you are away.
d) Keep hood and bench top areas clean and workable space maximized.

Disposal of solvents, chemicals and other materials:

Never pour solvents or reactive chemicals down a drain. Such careless handling of flammable or toxic liquids presents a serious hazard in the laboratory. Also, never keep an open beaker of such solvents outside a hood. Chlorinated solvents are poured into solvent waste containers kept inside the hoods. When in doubt about how to dispose of something, ask a TA. If drain disposal is necessary and acceptable, always flush the drain before, during, and afterwards with a lot of water, always using the drains in the hoods. All glass must be discarded in the specially designed containers. A dustpan and brush for broken glass can be checked out of Lab Supplies. Spilled mercury is a special safety hazard and should be reported to your TA for clean-up.

EXERCISE I

Methods of Expressing Concentration of Solution

Concentration of solution is the amount of solute dissolved in a known amount of the solvent or solution. The concentration of solution can be expressed in various ways as discussed below,

(1) **Percentage:** It refers to the amount of the solute per 100 parts of the solution. It can also be called as parts per hundred (pph). It can be expressed by any of following four methods,

(i) **Weight to weight percent**

% w/w = Wt of solute/ Wt of solution × 100

(ii) **Weight to volume percent**

% w/v = Wt of solute/ Volume of solution × 100

(iii) **Volume to volume percent**

% v/v= Volume of solute /Volume of solution × 100

(iv) **Volume to weight percent**

% v/w = Volume of solute / Wt of solution × 100

(2) Parts per million (ppm) and parts per billion (ppb):

When a solute is present in trace quantities, it is convenient to express the concentration in parts per million and parts per billion. It is the number of parts of solute per million (10^1) or per billion (10^9) parts of the solution. It is independent of the temperature.

$$Ppm = \frac{\text{mass of solute component}}{\text{Total mass of solution}} \times 10^1$$

$$Ppb = \frac{\text{mass of solute component}}{\text{Total mass of solution}} \times 10^9$$

(3) Normality (N)

It is defined as the number of gram equivalents (equivalent weight in grams) of a solute present per litre of the solution. Unit of normality is gram equivalents litre^{-1}. Normality changes with temperature since it involves volume. When a solution is diluted *x* times, its normality also decreases by times. Solutions in term of normality generally expressed as,

N= Normal solution; 5N= Penta normal,

10N= Deca normal;N/2= semi normal

N/10= Deci normal; N/5= Penti normal

N/100 or 0.01N= centinormal,

N/1000 or 0.001= millinormal

Mathematically normality can be calculated by following formula

$$\text{Normality (N)} = \frac{\text{Number of gm eq. of solute}}{\text{Volume of solution (l)}}$$

(* 1 equivalent = 1000 mill equivalent or meq.)

(4) Molarity

The number of moles of solute per litter of solution OR the molar concentration of a solution usually expressed as the number of moles of solute per litre of solution. It is also known as molar concentration, is the number of moles of a substance per litter of solution. Solutions libelled with the molar concentration are denoted with a capital M. A 1.0M solution contains 1 mole of solute per litre of solution.

$$\text{Molarity (M)} = \frac{\text{Mole of solute}}{\text{Litres of solution}}$$

Molarity - M → moles per litter solution

(5) Molality

The number of moles of solute per kilogram of solvent. It is important the mass of solvent is used and not the mass of the solution. Solutions labelled with molal concentration are denoted with a lower-case m. A 1.0 m solution contains 1 mole of solute per kilogram of solvent

$$\text{Molality (m)} = \frac{\text{Mole of solute}}{\text{Kg of solvent}}$$

Molality - m → moles per kilogram solvent

Experiment 1: Preparation and Standardization of 1M Oxalic Acid:

Oxalic Acid - M.Wt - 126.07

Preparation of 1M Oxalic Acid Solution: Dissolve an accurately weighed amount of 126.07 gmof Oxalic acid in sufficient amount of water to give or to produce 1000 ml.

Principle: It is an example of alkalimetry. When a strong base is titrated with a weak acid, the saltproduced in the reaction is not completely hydrolyzed and the pH of the resultant solution at theend-point is exactly 7.0. Oxalic Acid, a weak acid, is standardized by titration with a strong base,NaOH. The following reaction takes place when NaOH is titrated with Oxalic Acid.

In this titration, for detecting the end-point Phenolphthalein solution is used as indicator.

Procedure:

Preparation of 1M NaOH Solution: Dissolve an accurately weighed amount of 40 gm of NaOHin sufficient amount of water to give or to produce 1000 ml.

Procedure for Standardization of 1M Oxalic Acid: Take 10 ml of 1M NaOH solution into aconical flask and add 2 or 3 drops of phenolphthalein indicator. Titrate the contents of the flaskagainst Oxalic acid solution until the pink colour disappears. Repeat the titration to get concordantvalues. Enter the values in a tabular form...

Titration of 1M NaOH solution with Oxalic acid solution

The Molarity of Oxalic Acid is calculated using the formula:

M1V1=M2V2

Where, V1 = Volume of 1M NaOH solution = 10 ml

M1 = Molarity of NaOH solution = 1M

V2 = Volume of Oxalic acid solution rundown (Average Burette Reading)

M2 = Molarity of Oxalic acid =?

Therefore,

$$M_2 = \frac{M_1 V_1}{V_2}$$

Experiment 2: Preparation and Standardisation of 0.1M Oxalic Acid:

Oxalic Acid - M.Wt - 126.07

Preparation of 0.1M Oxalic Acid Solution: Dissolve an accurately weighed amount of 12.607 gm of Oxalic acid in sufficient amount of water to give or to produce 1000 ml.

Principle: It is an example of alkalimetry. When a strong base is titrated with a weak acid, the salt produced in the reaction is not completely hydrolysed and the pH of the resultant solution at the end-point is exactly 7.0. Oxalic Acid, a weak acid, is standardised by titration with a strong base, NaOH. The following reaction takes place when NaOH is titrated with Oxalic Acid.

In this titration, for detecting the end-point Phenolphthalein solution is used as indicator.

Procedure:

Preparation of 0.1M NaOH Solution: Dissolve an accurately weighed amount of 4 gm of NaOH in sufficient amount of water to give or to produce 1000 ml. Procedure for Standardisation of 0.1M Oxalic Acid: Take 10 ml of 0.1M NaOH solution into a conical flask and add 2 or 3 drops of phenolphthalein indicator. Titrate the contents of the flask against Oxalic acid solution until the pink colour disappears. Repeat the titration to get concordant values. Enter the values in a tabular form...

Titration of 0.1M NaOH solution with Oxalic acid solution

S.No.	Volume of 1M NaOH solution (ml)	Burette Reading		Vol. of Oxalic Acid rundown (ml)
		Initial	Final	
1	10	0		
2	10			
3	10			

The Molarity of Oxalic Acid is calculated using the formula:

$M_1V_1=M_2V_2$

Where, V1 = Volume of 0.1M NaOH solution = 10 ml

M1 = Molarity of NaOH solution = 0.1M

V2 = Volume of Oxalic acid solution rundown (Average Burette Reading)

M2 = Molarity of Oxalic acid =?

Therefore,

$$M_2 = \frac{M_1V_1}{V_2}$$

Experiment 3: Preparation and Standardization of 1N Oxalic Acid:

Oxalic Acid - M.Wt - 126.07

Preparation of 1N Oxalic Acid Solution: Dissolve an accurately weighed amount of 63.035 gms of Oxalic acid in sufficient amount of water to give or to produce 1000 ml.

Principle: It is an example of alkalimetry. When a strong base is titrated with a weak acid, the salt produced in the reaction is not completely hydrolyzed and the pH of the resultant solution at the end-point is exactly 7.0. Oxalic Acid, a weak acid, is standardized by titration with a strong base, NaOH. The following reaction takes place when NaOH is titrated with Oxalic Acid.

$$2(COOH) + 2NaOH \longrightarrow 2(COONa) + 2H_2O$$

In this titration, for detecting the end-point Phenolphthalein solution is used as indicator.

Procedure:

Preparation of 1N NaOH Solution: Dissolve an accurately weighed amount of 40 gm of NaOH in sufficient amount of water to give or to produce 1000 ml.

Procedure for Standardization of 1N Oxalic Acid: Take 10 ml of 1N NaOH solution into aconical flask and add 2 or 3 drops of phenolphthalein indicator. Titrate the contents of the flask against Oxalic acid solution until the pink color disappears. Repeat the titration to get concordant values. Enter the values in a tabular form...

Titration of 1N NaOH solution with Oxalic acid solution

S.No.	Volume of 0.1M NaOH solution (ml)	Burette Reading		Vol. of Oxalic Acid rundown (ml)
		Initial	Final	
1	10	0		
2	10			
3	10			

The Normality of Oxalic Acid is calculated using the formula:

$N_1V_1=N_2V_2$

Where, V_1 = Volume of 1N NaOH solution = 10 ml

N_1 = Normality of NaOH solution = 1N

V_2 = Volume of Oxalic acid solution rundown (Average Burette Reading)

N_2 = Normality of Oxalic acid =?

Therefore,

$$N_2 = \frac{N_1V_1}{V_2}$$

Experiment 4: Preparation and Standardization of 0.1N Oxalic Acid:

Oxalic Acid - M.Wt - 126.07

Preparation of 0.1N Oxalic Acid Solution: Dissolve an accurately weighed amount of 6.3 gm of Oxalic acid in sufficient amount of water to give or to produce 1000 ml.

Principle: It is an example of alkalimetry. When a strong base is titrated with a weak acid, the salt produced in the reaction is not completely hydrolyzed and the pH of the resultant solution at the end-point is exactly 7.0. Oxalic Acid, a weak acid, is standardized by titration with a strong base, NaOH. The following reaction takes place when NaOH is titrated with Oxalic Acid.

$$2(COOH) + 2NaOH \longrightarrow 2(COONa) + 2H_2O$$

In this titration, for detecting the end-point Phenolphthalein solution is used as indicator.

Procedure:

Preparation of 0.1N NaOH Solution: Dissolve an accurately weighed amount of 4 gms of NaOH in sufficient amount of water to give or to produce 1000 ml.

Procedure for Standardization of 0.1N Oxalic Acid: Take 10 ml of 0.1N NaOH solution into a conical flask and add 2 or 3 drops of phenolphthalein indicator. Titrate the contents of the flask against Oxalic acid solution until the pink color disappears. Repeat the titration to get concordant values. Enter the values in a tabular form...

Titration of 0.1N NaOH solution with Oxalic acid solution

S.No.	Volume of 1N NaOH solution (ml)	Burette Reading		Vol. of Oxalic Acid rundown (ml)
		Initial	Final	
1	10	0		
2	10			
3	10			

The Normality of Oxalic Acid is calculated using the formula:

$N_1V_1=N_2V_2$

where, V_1 = Volume of 0.1N NaOH solution = 10 ml

N_1 = Normality of NaOH solution = 0.1N

V_2 = Volume of Oxalic acid solution rundown (Average Burette Reading)

N_2 = Normality of Oxalic acid =?

Therefore,

$$N_2 = \frac{N_1V_1}{V_2}$$

Experiment 5: Preparation and Standardization of 1M Sodium Hydroxide:

Sodium Hydroxide - M.Wt - 40

Preparation of 1M Sodium Hydroxide Solution: Dissolve an accurately weighed amount of 40 gm of Sodium Hydroxide in sufficient amount of water to give or to produce 1000 ml.

Principle: It is an example of acidimetry. When a weak acid is titrated with a strong base, the salt produced in the reaction is completely hydrolyzed and the pH of the resultant solution at the end-point is more than 7.0 (Alkaline). Sodium hydroxide, a strong base, is standardized by titration with a weak acid, Oxalic acid. The following reaction takes place when Oxalic Acid is titrated with

$$2(COOH) + 2NaOH \longrightarrow 2(COONa) + 2H_2O$$

In this titration, for detecting the end-point Phenolphthalein solution is used as indicator.

Procedure:

Preparation of 1M Oxalic Acid Solution: Dissolve an accurately weighed amount of 126.07 gmsof Oxalic Acid in sufficient amount of water to give or to produce 1000 ml.

Procedure for Standardization of 1M Sodium Hydroxide: Take 10 ml of 1M Oxalic Acidsolution into a conical flask and add 2 or 3 drops of phenolphthalein indicator. Titrate the contentsof the flask against Sodium Hydroxide solution until a permanent pink color is obtained. Repeatthe titration to get concordant values. Enter the values in a tabular form...

Titration of 1M Oxalic Acid solution with NaOH solution

S.No.	Volume of 0.1N NaOH solution (ml)	Burette Reading		Vol. of Oxalic Acid rundown (ml)
		Initial	Final	
1	10	0		
2	10			
3	10			

The Molarity of NaOH is calculated using the formula:

$M_1V_1=M_2V_2$

Where, V_1 = Volume of 1M Oxalic Acid solution = 10 ml

M_1 = Molarity of Oxalic Acid solution = 1M

V_2 = Volume of NaOH solution rundown (Average Burette Reading)

M_2 = Molarity of NaOH =?

Therefore,

$$N_2 = \frac{N_1V_1}{V_2}$$

Experiment 6: Preparation and Standardization of 0.1M Sodium Hydroxide:

Sodium Hydroxide - M.Wt - 40

Preparation of 0.1M Sodium Hydroxide Solution: Dissolve an accurately weighed amount of 4gm of Sodium Hydroxide in sufficient amount of water to give or to produce 1000 ml.

Principle: It is an example of acidimetry. When a weak acid is titrated with a strong base, the salt produced in the reaction is completely hydrolyzed and the pH of the resultant solution at the end-point is more than 7.0 (Alkaline). Sodium hydroxide, a strong base, is standardized by titration with a weak acid, Oxalic acid. The following reaction takes place when Oxalic Acid is titrated with

$$2(COOH) + 2NaOH \longrightarrow 2(COONa) + 2H_2O$$

In this titration, for detecting the end-point Phenolphthalein solution is used as indicator.

Procedure:

Preparation of 0.1M Oxalic Acid Solution: Dissolve an accurately weighed amount of 12.607 gms of Oxalic Acid in sufficient amount of water to give or to produce 1000 ml.

Procedure for Standardization of 0.1M Sodium Hydroxide: Take 10 ml of 0.1M Oxalic Acid solution into a conical flask and add 2 or 3 drops of phenolphthalein indicator. Titrate the contents the flask against Sodium Hydroxide solution until a permanent pink color is obtained. Repeat the titration to get concordant values. Enter the values in a tabular form.

Titration of 0.1M Oxalic Acid solution with NaOH solution

S.No.	Volume of 1M Oxalic Acid solution (ml)	Burette Reading		Vol. of NaOH rundown (ml)
		Initial	Final	
1	10	0		
2	10			
3	10			

The Molarity of NaOH is calculated using the formula:

$M_1V_1=M_2V_2$

Where, V_1 = Volume of 0.1M Oxalic Acid solution = 10 ml

M_1 = Molarity of Oxalic Acid solution = 0.1M

V_2 = Volume of NaOH solution rundown (Average Burette Reading)

M_2 = Molarity of NaOH =?

Therefore,

$$M_2 = \frac{M_1V_1}{V_2}$$

Experiment 7: Preparation and Standardization of 1N Sodium Hydroxide:

Sodium Hydroxide -M.Wt - 40

Preparation of 1N Sodium Hydroxide Solution: Dissolve an accurately weighed amount of 40gm of Sodium Hydroxide in sufficient amount of water to give or to produce 1000 ml.

Principle: It is an example of acidimetry. When a weak acid is titrated with a strong base, the salt produced in the reaction is completely hydrolyzed and the pH of the resultant solution at the end-point is more than 7.0 (Alkaline). Sodium hydroxide, a strong base, is standardized by titration with a weak acid, Oxalic acid. The following reaction takes place when Oxalic Acid is titrated with

$$2(COOH) + 2NaOH \longrightarrow 2(COONa) + 2\,H_2O$$

In this titration, for detecting the end-point Phenolphthalein solution is used as indicator.

Procedure:

Preparation of 1N Oxalic Acid Solution: Dissolve an accurately weighed amount of 63.035 gms of Oxalic Acid in sufficient amount of water to give or to produce 1000 ml.

Procedure for Standardization of 1N Sodium Hydroxide: Take 10 ml of 1N Oxalic Acid solution into a conical flask and add 2 or 3 drops of phenolphthalein indicator. Titrate the contents of the flask against Sodium Hydroxide solution until a permanent pink color is obtained. Repeat the titration to get concordant values. Enter the values in a tabular form...

Titration of 1N Oxalic Acid solution with NaOH solution

S.No.	Volume of 1N Oxalic Acid solution (ml)	Burette Reading		Vol. of NaOH rundown (ml)
		Initial	Final	
1	10	0		
2	10			
3	10			

The Normality of NaOH is calculated using the formula:

$N_1V_1=N_2V_2$

Where, V_1 = Volume of 1N Oxalic Acid solution = 10 ml

N_1 = Normality of Oxalic Acid solution = 1N

V_2 = Volume of NaOH solution rundown (Average Burette Reading)

N_2 = Normality of NaOH =?

Therefore,

$$N_2 = \frac{N_1V_1}{V_2}$$

Experiment 8: Preparation and Standardization of 0.1N Sodium Hydroxide:

Sodium Hydroxide - M.Wt - 40

Preparation of 0.1N Sodium Hydroxide Solution: Dissolve an accurately weighed amount of 4gm of Sodium Hydroxide in sufficient amount of water to give or to produce 1000 ml.

Principle: It is an example of acidimetry. When a weak acid is titrated with a strong base, the salt produced in the reaction is completely hydrolyzed and the pH of the resultant solution at the end-point is more than 7.0 (Alkaline). Sodium hydroxide, a strong base, is standardized by titration with a weak acid, Oxalic acid. The following reaction takes place when Oxalic Acid is titrated with

$$2(COOH) + 2NaOH \longrightarrow 2(COONa) + 2\,H_2O$$

In this titration, for detecting the end-point Phenolphthalein solution is used as indicator.

Procedure:

Preparation of 0.1N Oxalic Acid Solution: Dissolve an accurately weighed amount of 6.3 gms of Oxalic Acid in sufficient amount of water to give or to produce 1000 ml.

Procedure for Standardization of 0.1N Sodium Hydroxide: Take 10 ml of 0.1N Oxalic Acid solution into a conical flask and add 2 or 3 drops of phenolphthalein indicator. Titrate the contents of the flask against Sodium Hydroxide solution until a permanent pink color is obtained. Repeat the titration to get concordant values. Enter the values in a tabular form...

Titration of 0.1N Oxalic Acid solution with NaOH solution

S.No.	Volume of 0.1N Oxalic Acid solution (ml)	Burette Reading		Vol. of NaOH rundown (ml)
		Initial	Final	
1	10	0		
2	10			
3	10			

The Normality of NaOH is calculated using the formula:

$N_1V_1=N_2V_2$

Where, V1 = Volume of 0.1N Oxalic Acid solution = 10 ml

N1 = Normality of Oxalic Acid solution = 0.1N

V2 = Volume of NaOH solution rundown (Average Burette Reading)

N2 = Normality of NaOH =?

Therefore,

$$N_2 = \frac{N_1V_1}{V_2}$$

Experiment 9: Preparation and Standardization of 1M Hydrochloric Acid:

Hydrochloric Acid - M.Wt - 36.5

Preparation of 1M Hydrochloric Acid Solution: Dissolve an accurately measured volume of 85 ml of Hydrochloric Acid in sufficient amount of water to give or to produce 1000 ml.

Principle: It is an example of alkalimetry. When a strong acid is titrated with a strong base, the salt produced in the reaction is not hydrolyzed and therefore the pH of the resultant solution at the end-point is 7.0. The following reaction takes place when sodium carbonate is titrated with HCl.

$$Na_2CO_3 + 2\ HCL \longrightarrow 2NaCl + H_2O + CO_2$$

In this reaction, for the detection of the end-point methyl orange is used as indicator.

Procedure:

Preparation of 1M Na_2CO_3 Solution: Dissolve an accurately weighed amount of 286.15 gm of Na_2CO_3 in sufficient amount of water to give or to produce 1000 ml.

Procedure for Standardization of 1M HCl: Pipette out exactly 10 ml of 1M Na_2CO_3 solution into a clean conical flask and add 2 or 3 drops of methyl orange indicator. Titrate the contents of the flask with 1M HCl until red color is obtained. Repeat the titration for concordant values. Record the values in the tabular form...

Titration of 1M Na_2CO_3 solution with HCl solution

S.No.	Volume of 1M Na_2CO_3 solution (ml)	Burette Reading		Vol. of HCl rundown (ml)
		Initial	Final	
1	10	0		
2	10			
3	10			

The Molarity of HCl is calculated using the formula:

$M_1V_1=M_2V_2$

Where, V_1 = Volume of 1M Na_2CO_3 solution = 10 ml

M_1 = Molarity of Na_2CO_3 solution = 1M

V_2 = Volume of HCl solution rundown (Average Burette Reading)

M_2 = Molarity of HCl =?

Therefore,

$$M_2 = \frac{M_1V_1}{V_2}$$

Experiment 10: Preparation and Standardization of 0.1M Hydrochloric Acid:

Hydrochloric Acid - M.Wt - 36.5

Preparation of 0.1M Hydrochloric Acid Solution: Dissolve an accurately measured volume of 8.5 ml of Hydrochloric Acid in sufficient amount of water to give or to produce 1000 ml.

Principle: It is an example of alkalimetry. When a strong acid is titrated with a strong base, the salt produced in the reaction is not hydrolyzed and therefore the pH of the resultant solution at the end-point is 7.0. The following reaction takes place when sodium carbonate is titrated with HCl.

$$Na_2CO_3 + 2\ HCL \longrightarrow 2NaCl + H_2O + CO_2$$

In this reaction, for the detection of the end-point methyl orange is used as indicator.

Procedure:

Preparation of 0.1M Na_2CO_3 Solution: Dissolve an accurately weighed amount of 28.62 gms of Na_2CO_3 in sufficient amount of water to give or to produce 1000 ml.

Procedure for Standardization of 0.1M HCl: Pipette out exactly 10 ml of 0.1M Na2CO3 solution into a clean conical flask and add 2 or 3 drops of methyl orange indicator. Titrate the contents of the flask with 0.1M HCl until red color is obtained. Repeat the titration for concordant values. Record the values in the tabular form...

Titration of 0.1M Na2CO3 solution with HCl solution

S.No.	Volume of 0.1M Na_2CO_3 solution (ml)	Burette Reading		Vol. of HCl rundown (ml)
		Initial	Final	
1	10	0		
2	10			
3	10			

The Molarity of HCl is calculated using the formula:

$M_1V_1=M_2V_2$

Where, V1 = Volume of 0.1M Na2CO3 solution = 10 ml

M1 = Molarity of Na2CO3 solution = 0.1M

V2 = Volume of HCl solution rundown (Average Burette Reading)

M2 = Molarity of HCl =?

Therefore,

$$M_2 = \frac{M_1V_1}{V_2}$$

Experiment 11; Preparation and Standardization of 1N Hydrochloric Acid:

Hydrochloric Acid - M.Wt - 36.5

Preparation of 1N Hydrochloric Acid Solution: Dissolve an accurately measured volume of 85ml of Hydrochloric Acid in sufficient amount of water to give or to produce 1000 ml.

Principle: It is an example of alkalimetry. When a strong acid is titrated with a strong base, the salt produced in the reaction is not hydrolyzed and therefore the pH of the resultant solution at the end-point is 7.0.

The following reaction takes place when sodium carbonate is titrated with HCl.

$$Na_2CO_3 + 2\ HCL \longrightarrow 2NaCl + H_2O + CO_2$$

In this reaction, for the detection of the end-point methyl orange is used as indicator.

Procedure:

Preparation of 1N Na2CO3 Solution: Dissolve an accurately weighed amount of 53 gm of Na_2CO_3 in sufficient amount of water to give or to produce 1000 ml.

Procedure for Standardization of 1N HCl: Pipette out exactly 10 ml of 1N Na_2CO_3 solution into a clean conical flask and add 2 or 3 drops of methyl orange indicator. Titrate the contents of the flask with 1N HCl until red color is obtained. Repeat the titration for concordant values. Record the values in the tabular form...

Titration of 1N Na_2CO_3 solution with HCl solution

S.No.	Volume of 1N Na_2CO_3 solution (ml)	Burette Reading		Vol. of HCl rundown (ml)
		Initial	Final	
1	10	0		
2	10			
3	10			

The Normality of HCl is calculated using the formula:

$N_1V_1=N_2V_2$

Where, V1 = Volume of 1N Na_2CO_3 solution = 10 ml

N = Normality of Na2CO3 solution = 1N

V2 = Volume of HCl solution rundown (Average Burette Reading)

N2 = Normality of HCl =?

Therefore,

$$N_2 = \frac{N_1V_1}{V_2}$$

Experiment 12: Preparation and Standardization of 0.1N Hydrochloric Acid:

Hydrochloric Acid -M.Wt - 36.5

Preparation of 0.1N Hydrochloric Acid Solution: Dissolve an accurately measured volume of 8.5 ml of Hydrochloric Acid in sufficient amount of water to give or to produce 1000 ml.

Principle: It is an example of alkalimetry. When a strong acid is titrated with a strong base, the salt produced in the reaction is not hydrolyzed and therefore the pH of the resultant solution at the end-point is 7.0.

The following reaction takes place when sodium carbonate is titrated with HCl.

$$Na_2CO_3 + 2\ HCL \longrightarrow 2NaCl + H_2O + CO_2$$

In this reaction, for the detection of the end-point methyl orange is used as indicator.

Procedure:

Preparation of 0.1N Na_2CO_3 Solution: Dissolve an accurately weighed amount of 5.3 gms of Na_2CO_3 in sufficient amount of water to give or to produce 1000 ml.

Procedure for Standardization of 0.1N HCl: Pipette out exactly 10 ml of 0.1N Na2CO3 solution into a clean conical flask and add 2 or 3 drops of methyl orange indicator. Titrate the contents of the flask with 1N HCl until red color is obtained. Repeat the titration for concordant values. Record the values in the tabular form...

Titration of 0.1N Na2CO3 solution with HCl solution

S.No.	Volume of 0.1N Na_2CO_3 solution (ml)	Burette Reading		Vol. of HCl rundown (ml)
		Initial	Final	
1	10	0		
2	10			
3	10			

The Normality of HCl is calculated using the formula:

$N_1V_1=N_2V_2$

Where, V1 = Volume of 0.1N Na2CO3 solution = 10 ml

N = Normality of Na2CO3 solution = 0.1N

V2 = Volume of HCl solution rundown (Average Burette Reading)

N2 = Normality of HCl =?

Therefore,

$$N_2 = \frac{N_1V_1}{V_2}$$

Experiment no: 13

Determination of total hardness of water using EDTA titration

Theory

Water samples that do not readily form lather with soap or deposit scales on the walls of the container when there is appreciable change in temperature are called hard water. Hardness is caused by the presence of some metallic ions viz. Ca^{2+}, Mg^{2+} and some other metallic ions Fe^{2+}, Mn^{2+} and Sr^{2+}. But these are usually present in small quantities. The portion of hardness that can be removed by boiling is known as temporary hardness. Boiling converts the soluble bicarbonates present into insoluble carbonates and hydroxides which can be removed by filtration. The portion of hardness that cannot be removed by boiling is termed as permanent hardness. Hardness is generally reported in terms of CaCO3 equivalent and is calculated by general formula:

Hardness (in mg/l) = M^{2+} (in mg/l) × (50/Equivalent wt of M^{2+})

50 being the equivalent weight of $CaCO_3$ and M^{2+} is any divalent metallic ion.

Unit of Hardness

Parts per million (ppm) is the most commonly used unit of hardness.

1 ppm = 1 part CaCO3 equivalent in 106 parts of water

Degree of hardness

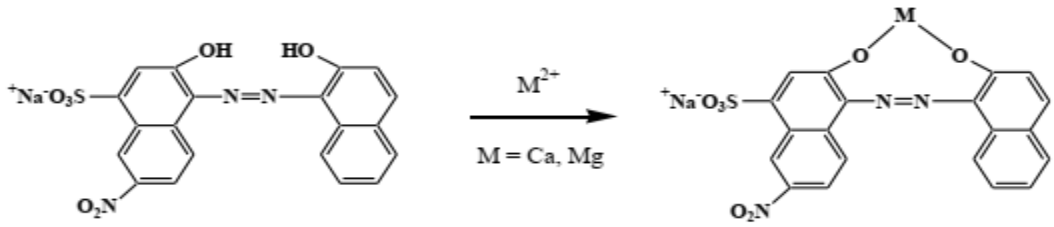
Very soft: 0-70 ppm; Soft: 70-140 ppm

Slightly hard: 140-210 ppm; moderately hard: 210-320 ppm

Hard: 320-530 ppm; Very hard> 530 ppm

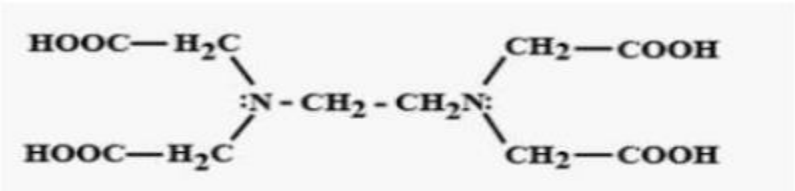
Equation

Between pH 7 to 11, Erichrome Black-T (EBT) indicator is blue in color. Addition of metallic salt in this pH range results in a brilliant change in color from blue to wine red due to formation of metal ion-EBT complex (C1). When the wine red complex C1 is treated with the disodium salt of EDTA, the colorless metal ion-EDTA complex (C_2) is formed rapidly. This is indicated by the change of the wine-red color of the metal ion-EBT complex (C1) to the blue color of the free indicator EBT. The Full form of $EDTA^{2-}$ is ethylene diamine tetracetate ion.



EBT(In) **C1**

C1	+	$EDTA^{2-}$	=	MEDTA	+	In
(wine red)				**(C2)**		(Free Indicator)
				(Metal-EDTA complex)		(blue)



Structure of ethylenediaminetetraacetic acid

Chemicals required

1. EDTA (N/50) Solution

2. Buffer ($NH_4Cl + NH_4OH$) [pH 10]

3. Erichrome Black T indicator

Apparatus

1. Burette

2. Pipette

3. Conical flask

4. Measuring Cylinder

Procedure

50 ml of hard water sample is pipetted out and transferred to a conical flask. 2 mL of buffersolution and 2 drops of EBT indicator are added to it. The solution is then titrated againststandard EDTA solution running from a burette till the wine-redcolor of the solution isturned to blue. Three such titrations are performed and the average value is recorded. Thebuffer solution is used to maintain the pH of the solution. Ammonium hydroxide-Ammoniumchloride buffer is used for the reaction.

No. of observations	Burette Reading (ml)		Volume of EDTA used (ml)	Average Volume of EDTA (ml)	Strength of EDTA Solution	Volume of water sample taken (ml)
	Initial	Final				
1	0	2.6	2.6	2.53	(N/50)	50
2	3	5.5	2.5			
3	0	2.5	2.5			

Calculation

1000 ml of 1(M) EDTA ≡ 100 gms $CaCO_3$

1 ml of 1(M) EDTA ≡ (100/1000) gms $CaCO_3$

1 ml of 1(M/50) EDTA ≡ (100/1000) × (1/50) gms $CaCO_3$

≡ (100/1000) × (1/50) × 1000 mg $CaCO_3$

≡ 2 mg $CaCO_3$

2.53 ml of EDTA required for titration

So, 2.53 ml (M/50) EDTA ≡ 2 × 2.53 = 5.06 mg $CaCO_3$

Now Volume of water taken = 50 ml

So,

50 ml water contains = 5.06 mg CaCO3

1000 ml water contains = [5.06/50 × 1000] mg CaCO3 = 101.2 ppm or mg/l

Precautions

1. Distilled water was used for washing and rinsing of glass apparatus
2. Same amount of indicator was added each time
3. pH 10 was maintained during the titration by adding buffer
4. End point was observed carefully

EXERCISE II

PREPARATION OF BUFFER

THEORETICAL INTRODUCTION TO THE EXERCISE

2.1. Buffer mixtures

Buffer mixtures are mixtures of weak bases or acids with their salts, e.g. CH_3COOH + CH_3COONa or NH_4OH + NH_4Cl and mixtures of salts of weak polyprotonic acids with different degrees of neutralization, e.g. NaH_2PO4 + Na_2HPO4 or Na_2HPO4 + Na_3PO_4.

Buffer mixtures have a certain pH, the value of which changes slightly when some excess ions H_3O^+ or OH^- are introduced into the solution. In other words, these mixtures have a buffering effect", that is, they prevent a sudden change in the pH of the solution. Similarly, diluting or increasing the concentration of buffer solutions has no effect on their pH value.

For an acidic buffer, e.g. CH_3COONa + CH_3COOH, the concentration of hydronium ions [H3O+] is calculated from the formula

$$[H_3O^+] = K_a \frac{C_a}{C_s}$$

Where:

K_a – weak acid dissociation constant,

C_a – acid concentration,

C_s – Salt concentration.

The mechanism of action of the buffering solution is as follows: after adding an acid to the buffer mixture – the anion of the salt contained in the buffer with the H_3O^+ ion creates a weakly dissociated acid, while after adding a base – the hydronium ion of the acid contained in the mixture forms with the OH^- ion poorly dissociated water molecules. Due to the formation of poorly dissociated acid and water particles, the pH of the solution changes slightly.

After adding CH_3COOH and CH_3COONa of hydrochloric acid to the acetate buffer, the CH_3COO^- anion from sodium acetate forms with H_3O^+ acetic acid

$$CH_3COO^- + H_3O^+ \rightleftharpoons CH_3COOH + H_2O$$

After the addition of e.g. sodium hydroxide, NaOH, a neutralization reaction takes place between the hydronium ions from acetic acid and the hydroxyl ions from the base.

$$CH_3COOH + H_2O \rightleftharpoons CH_3COO^- + H_3O^+$$
$$H_3O^+ + OH^- \rightleftharpoons 2H_2O$$

The introduction of acid to the buffer mixture causes an increase in the concentration of weak acid, while the introduction of a base – an increase in the concentrationof the appropriate salt. Changes in the concentration of the components of the buffer mixture have a slight effect on the pH of the solution.

In the case of an alkaline buffer, e.g. $NH_4OH + NH_4Cl$, the equation is used to calculate the concentration of hydronium $[H_3O^+]$

$$[H_3O^+] = \frac{10^{-14} \cdot C_s}{K_b \cdot C_b}$$

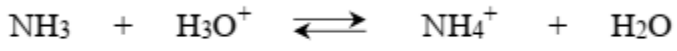
Where:

Cs – salt concentration,

Cb – base concentration,

Kb – The weak base dissociation constant of a given buffer.

The basic buffer (NH_4OH, NH_4Cl) mechanism of action is as follows: Dissociation has occurred in the solution and ions NH_4^+, Cl^- are present and the molecule NH_3. By introducing a small amount of hydronium ions from the acid, the reaction will take placewhich confirms that the pH does not change.



If a strong base is introduced into the buffer mixture, the ammonium ion will reactaccording to the equationand in this case the pH of the solution did not change.

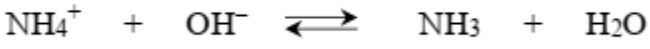


Table 1

Exemplary buffer mixtures

Buffer name	The composition of the buffer with a concentration of 1 : 1	pH
ammonium	ammonium hydroxide NH_4OH, ammonium chloride NH_4Cl	9.3
benzoate	benzoic acid C_6H_5COOH, sodium benzoate C_6H_5COONa	4.2
phosphate	sodium hydrogenorthophosphate Na_2HPO_4, sodium dihydrogenorthophosphate NaH_2PO_4	6.8
formate	formic acid $HCOOH$, sodium formate $HCOONa$	3.7
acetate	acetic acid CH_3COOH, sodium acetate CH_3COONa	4.7

Experiment 1 – Preparation of ammonium chloride and ammonium hydroxide buffer mixtures.

Materials and reagents: 50 cm^3 beakers, measuring cylinder, solutions: ammonium chloride (0.1 M NH4Cl), ammonium hydroxide (0.1 M NH4OH), ammonium chloride (0.2 M NH4Cl), ammonium hydroxide (0.2 M NH4OH), solution of the universal indicator.

Performance:

Using 0.1 M and 0.2 M solutions, prepare buffer mixtures by mixing them at the following volumetric ratios:

Ammonium Chloride	1	1	10
Ammonium Hydroxide	10	1	1

Then add 5 drops of the universal indicator solution to every single buffer mixture and determine the pH using Yamada Table: Color change of the universal indicator solution according to Yamada depending on the exponent of the concentration of the hydronium ion (depending on the pH)

pH	Colour of the indicator
4.0	red
5.0	orange
6.0	yellow
7.0	green
8.0	blue
9.0	dark blue
10.0	purple

Elaboration of the results:

Provide the obtained results in the table

Compound	The volume ratio of the solutions		
0.1 M ammonium chloride	1	1	10
0.1 M ammonium hydroxide	10	1	1
pH value			
0.2 M ammonium chloride	1	1	10
0.2 M ammonium hydroxide	10	1	1
pH value			

AIM: To prepare the buffer at required pH.

PRINCIPLE:

The pH meter measures at electrical potential developed by pair of electrode pins in a solution. For measurement of pH, an electrode system sensitive to change in H^+ ion concentration of solution is taken. The electrode system consists of sequence of electrode whose potential raise with pH (H^+ concentration of the solution).

PROCEDURE:

1. ACETIC ACID- SODIUM ACETATE BUFFER:

REAGENTS REQUIRED: Acetic Acid 0.2M: 1.5 ml of glacial acetic acid is made up to 100ml with distilled water. Sodium Acetate Solution**:** 0.64 gms of sodium acetate or 2.72gm of sodium acetate trihydrate is dissolved in 100ml Distilled water.

PROCEDURE:

Pipette out exactly 36.2ml of sodium acetate solution into 100ml of standard flask and add 14.8ml of glacial acetic acid, make the volume 100ml using distilled

water using distilled water. This gives 0.2 M of acetic acid and sodium acetate buffer. The pH is measured with pH meter. The pH meter is first standararised with pH buffer. Wash electrode with distilled water and introduced into 0.2M acetic acid-sodium acetate buffer prepared, the pH of solution is 4.6.

RESULT:

36.2ml Sodium acetate and 14.8 ml glacial acetic acid were mixed and buffer was prepared. pH was measured initial reading observed was 4 which made up to 4.6 with 5N NaOH.

Experiment 3 – Testing the pH of buffer solutions

Materials and reagents:

Beakers, measuring cylinder, solutions: ammonium chloride (0.1M NH4Cl), ammonium hydroxide (0.1M NH_4OH), sodium acetate (0.1M CH_3COONa), acetic acid (0.1M CH_3COOH), indicators: litmus paper, indicator papers, universal indicator.

Performance:

Pour 4 cm^3 of ammonium chloride solution (0.1M NH_4Cl) and 8 cm^3 of ammonium hydroxide solution (0.1M NH_4OH) into the first beaker with a capacity of 50 $cm^{3.}$ Pour 4 cm^3 of sodium acetate (0.1M CH_3COONa) and 8 cm^3 of acetic acid (0.1M CH_3COOH) into the second beaker. Then, in both beakers, determine the color and pH of the prepared solutions using the indicators given in Table, and finally measure the pH with a pH-meter.

Summary of results for experiment 3

No.	Method	Beaker 1		Beaker 2	
		Buffer solution		Buffer solution	
		Colour	pH/reaction	Colour	pH/reaction
1.	Litmus paper				
2.	Indicator papers with a selected range, 1 – 7 and 7 – 14				
3.	Universal indicator (alcoholic solution)				
4.	Measurement – with a pH meter				
5.	Calculation of the pH value				

Elaboration of the results

1. Determine the type of buffer solution in beakers 1 and 2.
2. Enter the colors and pH values obtained into Table 8.
3. Calculate the pH of the tested buffer solutions using the following formulas for calculatingthe concentration of hydronium ions: acidic buffer (acetate buffer)

$$[H_3O^+] = K_a \frac{C_a}{C_s},$$

$$[H_3O^+] = \frac{10^{-14} \cdot C_s}{K_b \cdot C_b}; \quad pH = -\log[H_3O^+]$$

Alkalinebuffer (ammonium buffer)

4. Compare the methods used to determine the pH of buffer solutions.