A HANDBOOK ON
ENGINEERING CHEMISTRY LABORATORY

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ON
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LABORATORY

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(For B. Tech. first-year students)

1ˢᵗ Edition

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PREFACE

This book has been written for engineering students for strengthening their fundamental knowledge in the chemistry laboratory. It aims to explore the needs and requirements of hands-on laboratory work and to extend the knowledge of laboratory fundamentals in chemistry in an organized manner.

Initially, the students will get a flavor of different laboratory equipments, glass goods and also the do's and don's of the laboratory. Each experiment is clearly written along with explanation of theory behind it.

We express our heartfelt thanks to Publisher, Ideal International E-Publication, for their support for publication of this book. We express our sincere gratitude to the college authority for stimulating encouragement and unflinching support which are a source of great strength to us. We also convey our sense of appreciation to our beloved students whose inquisitive questions provided valuable inputs in this book. We collected different simple images from different digital sources for the sake of the students which help them to understand clearly about laboratory equipments.

We expect this book will be helpful and give proper guidance to the students and also be supportive to the teachers.

Any comments and suggestions of this book will be highly acknowledged.

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CONTENTS

GENERAL INSTRUCTIONS FOR CHEMICTRY LABORATORY

1. Keep your bags outside the laboratory in the proper place.

2. Maintain all rules as instructed by the teachers.

3.Use the apparatus and chemicals carefully. Glass apparatus should be handled with special care.

4. During lab work, always keep your hands away from your face.

5. Laboratory waste must be kept in the proper place.

6. Wash your hands with plenty of water well before leaving the laboratory.

7. Safety is an integral part of working in chemistry laboratory, so learning about safety is a part of your education

8. Know the place and learn how to use the Fire extinguisher.

9. Learn about reagents before using them.

10. Read the lab manual thoroughly and discuss with the instructor before doing any experiment.

DOS AND DON'TS OF A CHEMISTRY LAB

DOs

- Always wear a Lab coat on entering a Chemistry Laboratory

- Wear covered shoes

- Girls should properly tie back their hair

- Appropriately label chemical containers

- Use only the required amount of chemicals as given in manual

- Clean glassware thoroughly before and after using

- Use tongs to hold glassware during heating

- For heating liquids use boiling chips

- Always add acid to water and never water to acid. Stir the solution slowly and constantly while adding the acid and let it cool down before using

- Dispose of chemicals and other wastes according to the given guidelines

- Dispose of broken glassware appropriately

- For chemical spills in tabletops or floor use gloves, neutralize acid/base, absorb using paper towels and discard them properly

- Keep working table clean

- Know the location of emergency equipment (fire extinguishers, safety showers etc) and also how to use them and how to summon assistance in the event of an emergency

DON'Ts

- Don't wear apparel that may caught on equipment

- Don't leave naked flames unattended in a laboratory

- Don't heat flammable liquids in Bunsen burner

- Don't leave equipment running unattended

- Don't work in the laboratory without an instructor

- Don't weigh a hot substance

- Don't place chemicals directly on the balance pan while weighing

LABORATORY EQUIPMENTS

A. <u>Weighing balance</u>

Top-loading balances: These will weigh to an accuracy of ± 1 mg and are suitable for most weighing of amounts that are specified to only two or three significant figures.

Analytical balances: These will weigh to an accuracy of ± 0.1 mg and must be used whenever you desire four or more significant figure accuracy. This will be the case when you weigh out samples of unknown, primary standards or when taking crucibles to constant weight. Directions for the use of these balances are posted in the balance room.

Top-loading balance   **Analytical balance**

Rules for analytical balances:

1. Always close the balance door, while weighing.

2. Do not handle objects to be weighed with bare hands.

3. To weigh accurately, all objects must be at room temperature.

4. Use containers such as beakers, flasks and weighing bottles.

5. The use of weighing paper must be strictly avoided when using an analytical balance.

6. Before weighing anything on this analytical balance, it needs to be "tared," or recalibrated to read 0.0000 g. When first turned on, or when left by the previous user, the balance may indicate something other than 0.0000 g. The Tare button needs to be pressed and released to effect this recalibration.

7. An analytical balance is so sensitive that it can detect the mass of a single grain of a chemical substance. Thus, if a method of direct weighing is used, the substance ought to be added to the tared container which will hold it, NEVER directly to the pan or even to weighing paper placed on the pan.

8. The container used should be completely dry and at room temperature, never at an elevated or reduced temperature.

9. Even slight temperature differences can produce APPARENT changes in mass of the container. Finally, the container ought to be completely dry, inside and out.

B. <u>Hot air oven</u>

• Hot air ovens are very common laboratory equipment which uses dry hot air mixture for drying and sterilization of laboratory equipment.

• The material which is to be placed inside the oven should not melt, catch fire or change its form during heating at high temperature.

• The general temperature range required for operation varies from 5°C to 450°C as per need.

Hot air oven

C. <u>Shaker</u>

• A shaker is a piece of laboratory equipment used to mix, blend, or agitate substances in a tube or flask.

- A shaker contains an oscillating board that is used to place the flasks, beakers, or test tubes.

Shaker

D. Magnetic stirrer cum hot plate

- This equipment is specially designed to meet up the lab requirements when stirring is needed along with uniform heating

- Both the stirring speed and temperature can be read from the digital display.

Magnetic stirrer cum hot plate

E. Bunsen burner

- The Bunsen burner, named after Robert Bunsen a common laboratory equipment for producing hot, soot less, non-luminous flame.

- By manipulating the Bunsen burner, both the size and temperature of the flame can be controlled.

Bunsen burner

F. <u>Volumetric flask</u>

- Volumetric flasks measure one specific volume, marked by the etched line on the neck.
- Easy to mix solutions, as they have glass stoppers and are designed to be insertedmultiple times to mix. Careful not to fill above the etched mark, see reading the meniscus.

Volumetric flask

G. <u>Burette</u>

- A volumetric burette is a long graduated glass tube with a stopcock at one end, for delivering known volumes of a liquid during titrations.

- To fill the burette close the stopcock and use a funnel to pour the solution into the burette.

- Fill the solution upto the zero mark.

- Check for any air gap left in the column.
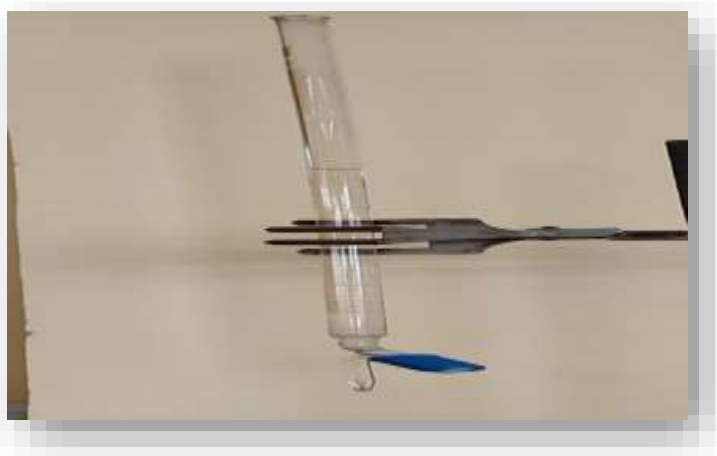
Burette

H. Pipette

- Pipette is a common laboratory tool used to transfer measured volume of liquid.

- Liquids are taken up in a pipette by suction.

I. Graduated measuring cylinder

- Used for measuring many different volumes.

- The spout lines up nicely with the rim of other glassware for easy pouring.
- Carefully measure the required amount, see reading the meniscus.

J. <u>Erlenmeyer flask</u>

An Erlenmeyer flask is also known as conical flask or titration flask, is a common laboratory glass container flat bottom, conical body, and cylindrical neck. They are usually made up of borosilicate glass in a wide range of volumes and may be graduated.

K. <u>Beaker</u>

- A beaker is a common cylindrical laboratory container with a flat bottom.

- They usually come in different capacities and have a small spout which helps in pouring.

- Beakers are commonly made up of borosilicate glass and sometimes of polythene or polypropylene.

L. <u>Funnel</u>

- Laboratory funnels are used to pass liquids or finely powdered chemicals into a laboratory ware with a narrow opening.

M. <u>Weighing bottle</u>

- Weighing bottles are glass laboratory equipment used for accurate weighing of solid substances.

- They are usually made up of non borosilicate glass.

N. <u>Other laboratory equipments</u>

TEST BUBE & TEST TUBE STAND	REAGENT BOTTLE

ROUND BOTTOM FLASK	DROPPER	GLASS ROD

O. Distillation unit
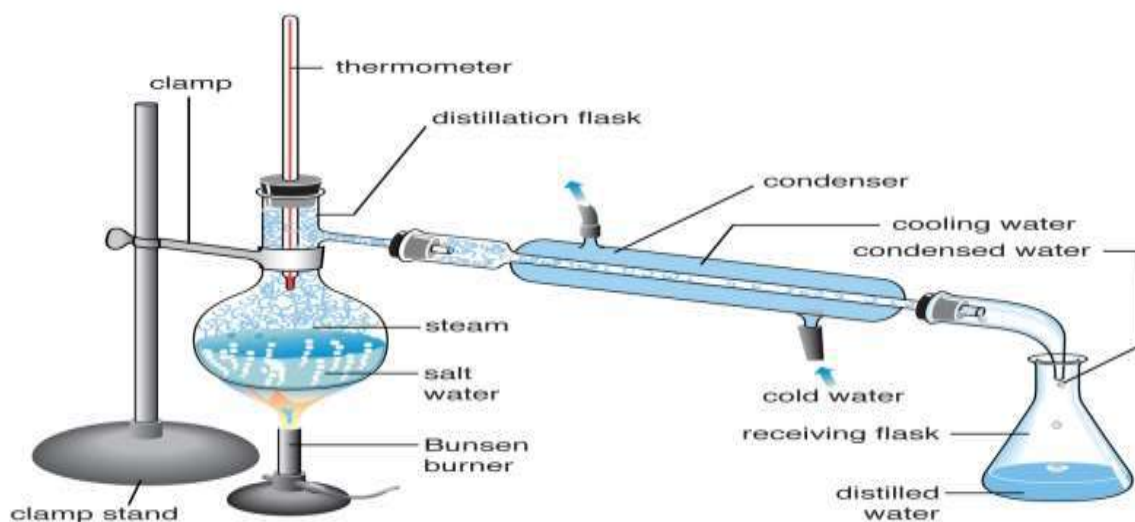
Preparation of distilled water

- Tap water is heated in a big round bottom flask. The water is heated to a rolling boil, which causes evaporation and steam production

- Steam leaves the boiling chamber and reaches the condenser

- Steam condenses in the condenser and the pure water collects in the receiving flask

VOLUMETRIC ANALYSIS

It involves the estimation of the strength of substance in solution by neutralization, precipitation, oxidation or reduction by means of another solution of accurately known strength. Example: Titration

<u>Terms associated with volumetric analysis</u>

Standard Solutions: A standard solution is a solution whose strength is known.
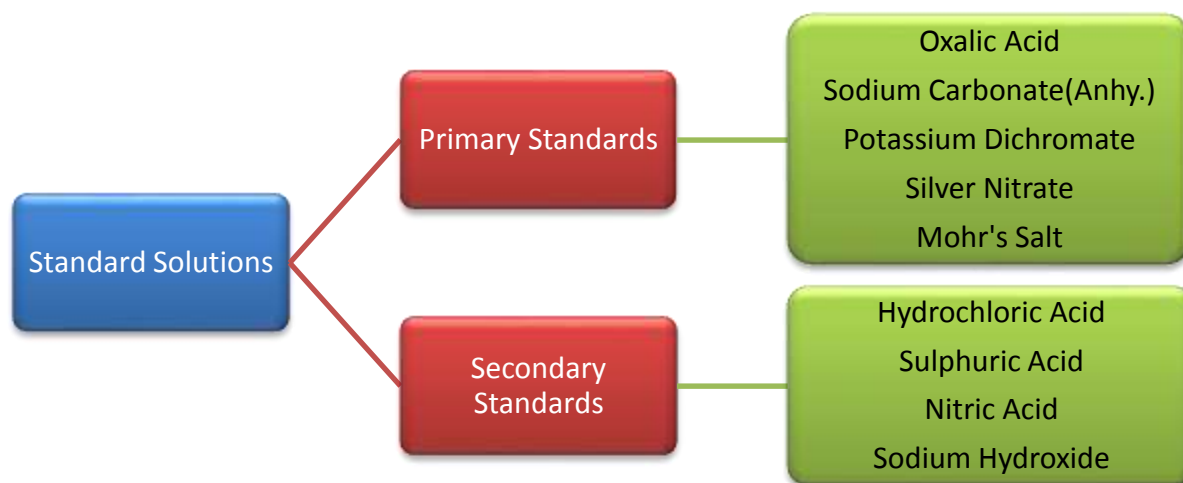
Primary Standard solutions

These are prepared by direct weighing method. Accurate amount of the substance are weighed and dissolved in required volume of water.

Substances used for preparation of Primary Standard solutions are called primary standards.

Those substances should have the following properties:

(i)   Exist in highly purified state

(ii)   Stable in nature

(iii)   Soluble in water

(iv)   Preferably with large equivalent weight

Secondary Standard solutions

They are prepared by indirect method. The solution is prepared by dissolving the substance (approximate weight) in required volume of water. Exact strength of the solution is determined by titrating it against a primary standard solution.

Expressions of Concentration of Solutions

The concentration or strength of solution means the amount of solute present in a given amount of the solution. The concentration can be expressed as;

Normality (N) and normal solution: Normality is defined as the number of gram equivalents of the solute present in 1 litre (1000 ml) of the solution.

i.e. if W g of solute of equivalent weight E is present in V mL of the solution, the normality of the solution is given by:

$$Normality = \frac{W \times 1000}{N \times V}$$

A solution containing one gram equivalent weight of the solute dissolved per litre is called a **Normal solution** e.g. when 40 g of NaOH are present in one litre of NaOH solution, the solution is known as normal (N) solution of NaOH.

Molarity (M) and molar solution: It is defined as the number of moles of the solute present in 1 litre (or 1000 mL) of the solution. i.e. if W g of solute of Molecular weight M is present in V mL of the solution, the molarity of the solution is given by:

$$Molarity = \frac{W \times 1000}{M \times V}$$

One molar solution contains 1 mole of the solute dissolved in 1 litre of the solution.

Molality (m): It is defined as the number of moles of solute dissolved in 1000 g of the solvent. One molal solution contains one mole of the solute dissolved in 1000 g of the solvent.

$$Molality = \frac{No. of\ moles\ of\ solute}{Mass\ of\ solvent\ in\ Kg}$$

Formulae used in solving numerical problems on volumetric analysis:

Normality formula, $N_1V_1 = N_2V_2$

$$S_1V_1 = S_2V_2$$

Where N_1, N_2 → Normality of titrant and titrate respectively

S_1, S_2 → Strength of titrant and titrate respectively

V_1, V_2 → Volume of titrant and titrate respectively

Preparation of some reagents

1. 1 N Hydrochloric Acid solution : 86 ml of 12(N)HCl and diluted to 1000 ml

2. 5 n Hydrochloric Acid solution : 430 ml of 12(N)HCl and diluted to 1000 ml

3. 1 N Sulphuric Acid solution : 27.80 ml of 36(N) H_2SO_4 and diluted to 1000 ml

4. 5 N Sulphuric Acid solution : 139 ml of 36(N) H_2SO_4 and diluted to 1000 ml

5. 1 N Nitric Acid solution: 61.8 ml of 16.2 (N) HNO_3 and diluted to 1000 ml

6. 5 N Nitric Acid solution: 309 ml of 16.2 (N) HNO_3 and diluted to 1000 ml

7. 1 N Acetic Acid solution : 57.4 ml of 17.4(N)99.5% A.R. Glacial Acetic Acid diluted to 1000 ml

8. 5 N Acetic Acid solution: 287 ml of 17.4 (N) 99.5% A.R. Glacial Acetic Acid diluted to 1000 ml

9. 0.1 N Oxalic acid solution: Dissolve 1.575 gm of Oxalic Acid (A.R. grade) in a volumetric flask of 250 ml and make up the volume upto 250ml withdistilled water.

10. 0.1 N Sodium Hydroxidesolution: Dissolve 4 gm of NaOH (A.R. grade) in 1000ml distilled water.

11. 0.01 M EDTAsolution: Dissolve 3.72gm$Na_2EDTA.2H_2O$ in deionised water and dilute to 1000ml.

12. 0.02N Silver Nitrate solution: Dissolve 3.398 gm A.R. grade solid $AgNO_3$ in distilled water and dilute to 1000 ml.

13. 0.1N Sodium Thiosulphate solution: Dissolve 24.82gm A.R. grade $Na_2S_2O_3.5H_2O$ in distilled water and dilute to 1000 ml.

14. 0.1N Potassium dichromate: Dissolve 4.9 gm pure $K_2Cr_2O_7$in distilled water and dilute to 1000 ml.

15. 0.1N Potassium Permanganate solution: Dissolve 3.1607 gm of $KMnO_4$ in distilled water and dilute to 1000 ml. Boil the solution and cool if require.

16. 0.1 M Potassium iodide: Dissolve 16.6 gm if iodate free A.R. grade KI in distilled water and dilute to 1000 ml.

17. NH_4Cl – NH_4OH buffer(pH 10) : Dissolve 1.5gm of A.R. NH_4Cl in 142 ml concentrated NH_3

Indicators

Types of Indicators

- Acid-Base Indicator

- Natural acid-base indicators

- Indicators of oxidation-reduction reactions (redox)

- Adsorption (precipitation) indicators

- Metallochromic or complexometric indicators

Acid–base indicators are compounds that change color when they become protonated or deprotonated. This color change occurs over a specific pH range. Example Phenolphthalein is colourless in acid medium and pink in basic medium and its pH range is 8.3 to 10.

Natural Indicators are few naturally occurring substances which show different colors in acidic and basic medium. Some examples of natural indicators are Litmus, red cabbage juice, turmeric etc. Red cabbage juice is purple in colour and turns brown in acid medium and green in basic medium.

Oxidation/reduction or Redox indicators are colorimetric reagents which show a distinct color change at a specific electrode potential. These are all organic compounds exhibiting reversible redox reactions. Examples include anilinic acid, diphenylamine, methylene blue etc.

Complexometric indicators are dyes that undergoes a definite color change in presence of specific metal ions. They form a weak complex with the ions present in the solution, which has a significantly different color from the form existing outside the complex.

Common indicators are organic dyes like Eriochrome Black T, Eriochrome Red B, Patton Reeder etc.

Indicator Solutions

1. Phenolphthalein indicator: Dissolve 1 gm in 100 ml of ethyl alcohol and then add 100 ml of distilled water with constant stirring.

2. Potassium Chromate (0.5N): Dissolve 5 gm in 100 ml distilled water

3. Eriochrome Black T(EBT): Dissolve 0.5 gm of the dye in 100 ml of rectified spirit. This solution can be stored for 1 month.

4. Calcon indicator: Dissolve 0.4 gm of the dye in 100 ml of methyl alcohol

5. Starch indicator: Add 1gm powder starch in 100 ml distilled water and boil the mixture with constant stirring until a clear solution is obtained. This solution cannot be stored.

BASIC FIRST AID IN CHEMISTRY LABORATORY

- Small Burns Burn from hot labware: wash with cold water, dry and apply ointment

- Serious Burn from hot labware : Contact medical help immediately

- Chemical burns: Immediately wash with plenty of water

- Acid Burn: wash with water, apply very dilute solution of sodium carbonate and wash with water again

- Base: wash with water, apply acetic acid and again wash with water

- Minor Cut from broken glassware: wash with soap apply antiseptic and sterile bandage

- Major Cut from broken glassware: Control bleeding by applying pressure with sterile pads

Essentials of a first aid kit

- Burn Cream

- Antiseptic wipes

- Antiseptic creams,

- Adhesive bandages

- Sterile pad/gauze/cotton

- Medicines (analgesic or painkillers)

- Pain relief spray

- Blunt tip Scissor

- Tweezers

- Instant ice pad

- ✓ First Aid Box should be kept in an accessible place

- ✓ A person should be made responsible for maintaining a list of the contents and discarding the expired medicines and replacement with fresh stocks.

EXPERIMENTAL

DETAILS

1. Determination of total alkalinity in a given sample of water

Theory:

The knowledge of alkalinity of water is necessary for controlling the corrosion, in conditioning the boiler feed water, for calculating the amounts of lime and soda needed for water softening and also in neutralizing the acidic solution produced by the hydrolysis of salts. The alkalinity of water is due to the presence of hydroxide ion (OH^-) as $NaOH$ or KOH, carbonate ion (CO_3^{2-}) as Na_2CO_3 or K_2CO_3 and bicarbonate ion (HCO_3^{2-}) as $NaHCO_3$ or $KHCO_3$ or $Ca(HCO_3)_2$ or $Mg(HCO_3)_2$.

For the estimation of alkalinity of water, a known volume of the given water sample is titrated against a standard acid like $N/50$ H_2SO_4 solution using methyl orange indicator. The end point is yellow to pink colour.

Chemical reactions involved:

a) For hydroxides:

$2NaOH + H_2SO_4 = Na_2SO_4 + 2H_2O$

$2KOH + H_2SO_4 = K_2SO_4 + 2H_2O$

b) For carbonates:

$K_2CO_3 + H_2SO_4 = K_2SO_4 + H_2O + CO_2$

$Na_2CO_3 + H_2SO_4 = Na_2SO_4 + H_2O + CO_2$

c) For bicarbonates:

$Mg(HCO_3)_2 + H_2SO_4 = MgSO_4 + 2H_2O + 2CO_2$

$Ca(HCO_3)_2 + H_2SO_4 = CaSO_4 + 2H_2O + 2CO_2$

$2NaHCO_3 + H_2SO_4 = Na_2SO_4 + 2H_2O + 2CO_2$

$2KHCO_3 + H_2SO_4 = K_2SO_4 + 2H_2O + 2CO_2$
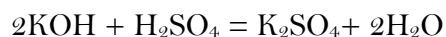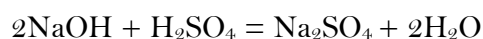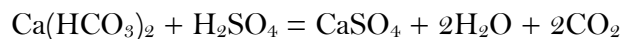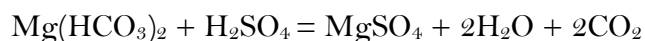
Apparatus:

Burette, Pipette, Conical flask, Burette stand

Chemicals:

Standard $N/50$ sulphuric acid solution, Methyl orange indicator, Sample water

Procedure:

i) After washing, rinse and fill the burette with N/50 H_2SO_4 solution.

ii) Wash the conical flask with distilled water and add 10 ml of the sample solution (to be analyzed for alkalinity).

iii) Add 3 drops of methyl orange indicator. The solution will acquire yellow colour.

iv) Note the initial reading of the burette and start adding the acid till the addition of the last drop changes the colourfrom yellow to pink.

v) Repeat the titration at least thrice.

Observation table:

Sl no.	Volume of water sample (ml)	Burette reading		Volume of acid consumed (ml)	Mean volume (ml)	Alkalinity of water sample
		Initial	Final			
1	10					
2	10					
3	10					

Calculation:

In the titration of water against N/50 H_2SO_4 solution,

Strength of sulphuric acid: N/50

Volume of water: 10 ml

Volume of sulphuric acid (titre value): V ml

Strength of water (Alkalinity w.r.t $CaCO_3$):

$$\frac{\text{Strength of sulphuric acid X Volume of sulphuric acid}}{\text{Volume of water}}$$

= (1/50 X V) X 1/10 X 50 (Equivalent of $CaCO_3$ = 50) gm/lit

= V/10 gm/lit

= V/10 X 1000 mg/lit

= 100 V mg/lit (ppm)

So the total alkalinity of the given water sample = 100 V ppm.

Precautions:

1. The pipette, burette, conical flask should be properly cleaned and calibrated before use.

2. The indicator solution should not be kept open as it contains alcohol which tends to evaporate.

3. Care should be taken while handling sulphuric acid and especially if the dilute acid is made from a concentrated one.

2. Determination of chloride ion in a given water sample by Argentometric method

Theory:

When silver nitrate solution is added to a given solution of chloride-containing few drops of indicator (K_2CrO_4), white silver chloride is precipitated initially. The moment all the chloride ions have been precipitated out, even a drop of silver nitrate added in excess gives a brick-red precipitate of silver chromate. This indicates the end point. Because $K_{sp}(AgCl) < K_{sp}(Ag_2CrO_4)$, hence as long as the chloride ions are available, the soluble silver chloride is precipitated. As soon as all the chloride ions have been precipitated out, even a slight excess of Ag^+ produce insoluble silver chromate which is red in colour.

$$Ag^+ \ + \ Cl^- \ \rightarrow AgCl \text{ (White ppt.)}$$
$$2\ Ag^+ \ + CrO_4{}^{2-} \rightarrow \ \ Ag_2CrO_4 \text{ (Brick ppt)}$$
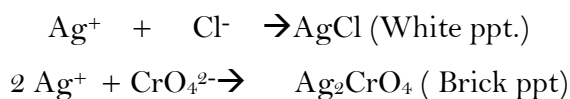
Apparatus:

Burette, Pipette, Beaker, conical flask

Chemicals:

$AgNO_3$ solution (N/50), K_2CrO_4 indicator

Procedure:

1. Fill the washed and rinsed burette with $AgNO_3$ solution.

2. Wash the pipette with water and then rinse it with chloride solution. Pipette out 10ml of chloride solution in a clean titration flask.

3. Add 6 drops of K_2CrO_4 indicator.

4. Add AgNO3from burette, shaking the flask constantly. A white precipitate of AgCl is obtained. After addition of a few ml of $AgNO_3$, a red colour appears in the flask but disappears quickly upon shaking.

5. Continue the addition drop by drop till a permanent reddish brown colour is obtained. Take three concordant readings.

Observation table:

Sl. No	Volume of sample taken (ml)	Burette readings			Average volume of $AgNO_3$ (ml)	Amount of chloride ion
		Initial	Final	Difference		
1						
2						
3						

Calculation:

S_1 = Strength of sample taken(strength of chloride ion)

S_2 = Strength of $AgNO_3$ solution

V_1 = Volume of sample taken

V_2 = Volume of $AgNO_3$

From $V_1 S_1 = V_2 S_2$

$$S_1 = \frac{V2\ S2}{V1} = V_2 \ X \frac{(N)}{50} \ X \frac{1}{10} \quad = \quad \frac{V2}{500}(N)$$

Amount of chloride ions present   = S_1 X Equivalent wt of chloride ions

$$= \frac{V2}{500} \ X \ 35.5 \ gms \ / \ L$$

$$= \frac{V2}{500} \ X \ 35.5 \ X \ 1000 \ mg/L$$

$$= V_2 \ X \ 35.5 \ X \ 2 \ ppm$$

$$= V_2 \ X \ 71$$

Result:

The amount of chloride ion present in given water sample = ……. ppm.

Precautions

1. All apparatus should be cleaned and calibrated before use.

2. The flask should be constantly swirled during titration.

3. The silver nitrate solution should be added dropwise to get a sharp end point.

THE

3. Determination of calcium and magnesium hardness of a given water sample separately

The experiment has two parts:

1. Estimation of total hardness (calcium and magnesium hardness) of water by EDTA titration.

2. Estimation of calcium hardness of water by EDTA titration.

Theory:

The total hardness of water is due to dissolved calcium and magnesium salts and expressed as parts per million (ppm) of calcium carbonate equivalent. The total hardness is conveniently determined by titrating the calcium and magnesium present in an aliquot of the sample at pH 10 with EDTA (Ethylenediamine tetra acetic acid) using Eriochrome Black T (EBT) indicator. Disodium salt of EDTA is used to estimate the Ca^{+2} and Mg^{+2} ions of the sample.

When Eriochrome Black T is added to the hard water at pH 10, it gives wine red colored unstable complex with Ca^{+2} and Mg^{+2} ions of the sample water. Now when this wine red colored complex is titrated against EDTA solution (of known strength), the color of the complex changes from wine red to blue showing the endpoint.

The structure of EDTA is the following

EDTA, disodium salt
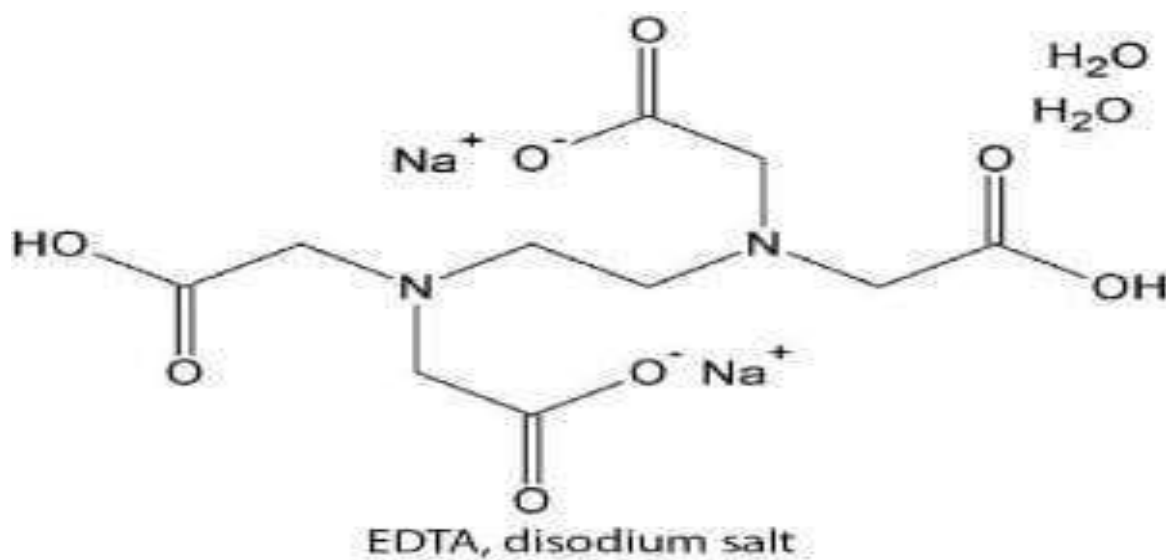
In the aqueous solution, EDTA ionizes to give Na^+ ions and a strong chelating agent.

For estimation of calcium hardness, Mg^{+2} in hard water is precipitated as $Mg(OH)_2$ by adding diethylamin which raises the pH of the solution to about 12.5. Ca^{+2} can then be titrated with EDTA using calcon indicator. The colour changes from pink to blue at the end point. The difference between the two titre values corresponds to the magnesium hardness.

Apparatus:

Burette, Pipette, Beaker, conical flask

Chemicals:

0.01 M EDTA solution, buffer of pH 10, EBT indicator, Diethyl amine, Calcon indicator.

Procedure:

1. Total hardness: Pipette out 10ml of the given water sample in a clean conical flask. To this add 3ml buffer solution of pH 10 and then 2-3drops of EBT indicator. Thus titrate against standard EDTA solution from burette until the colour changes from wine red to blue with one drop of EDTA solution .Repeat this titration thrice.

2. Ca^{+2} hardness: Pipette out 10ml of the given water sample in a clean conical flask. Add 2ml of diethyl amine and 4-6 drops of calcon indicator. Shake the flask and titrate it against standard EDTA solution until the colour changes from pink to pure blue.

Table 1

(Determination of total hardness)

No of observation	Vol. of water sample taken(ml)	Burette reading	(ml)		Mean volume of EDTA solution (V1)	Total hardness (ppm)
		Initial	Final	Difference		
1						
2						
3						

Table 2

(Determination of calcium hardness)

No of observation	Vol. of water sample taken (ml)	Burette reading			Mean volume of EDTA solution (ml)	Calcium hardness (ppm)
		Initial	Final	Difference		
1						
2						
3						

Calculation:

1. <u>Total Hardness</u>:

1000 ml 1(M) EDTA solution: = 100 gm $CaCO_3$

V_1 ml   0.01(M) EDTA solution: $=\dfrac{100 \text{ x V1 x } 0.01}{1000}$ gm $CaCO_3$

=X mg CaCO3

So 10 ml hard water contains: = X mg CaCO3

1000 ml hard water contains: = Y mg CaCO3

So the total hardness of the given water sample: =…………..ppm.

2. <u>Calcium Hardness</u>:   Same as earlier

Calcium hardness of the given water sample: =…… ppm.

3. <u>Magnesium Hardness</u>:   Total Hardness — Calcium Hardness

= ……… ppm

Conclusion:

The calcium hardness of the given water sample = …..ppm

The magnesium hardness of the given water sample = ……. Ppm

Precautions:

1. All apparatus should be cleaned thoroughly with distilled water before use as any contamination of calcium or magnesium salts could give erroneous reading.
2. The pH should be maintained during titration.
3. Freshly prepared indicator solutions must be used.

4. Determination of partition coefficient of acetic acid between n-butanol and water
(Heterogeneous equilibrium)

Theory:

When a system consists of parts that have different physical properties and are separated by boundary surface, the system is said to be a heterogeneous one. The Nernst distribution law states that at constant temperature, when different quantities of a solute are allowed to distribute between two immiscible solvents in contact with each other, then at equilibrium the ratio of the concentration of the solute in two solvent layers is constant.

When a solute is shaken in two immiscible liquids, then the solute is found to be distributed between the liquids in a definite manner if the solute is soluble in each of the solvent. According to the distribution law, the distribution Coefficient at a particular temperature is given by

$K = C_1 / C_2$ where C_1 and C_2 represent the concentration of solute in solvent 1 and 2 respectively.

Consider a liquid – liquid system, say water and n-butanol (immiscible solvents), to which a little quantity of acetic acid is added. Acetic acid will dissolve partly in water and partly in n-butanol. The two solutions of acetic acid will separate into two layers at equilibrium. The concentration of acetic acid in two layers is different but their ratio is fixed at a constant temperature.

Apparatus:

Stoppered bottle, volumetric flask, conical flask, burette, pipette

Chemical required:

Pure n-butanol, Glacial acetic acid, Sodium hydroxide solution (N/2), phenolphthalein indicator.

Procedure:

1. Take two stoppered bottles and mark as bottle 1 and bottle 2. Add the following materials in bottle 1 and bottle 2.

BOTTLE 1	BOTTLE 2
Butanol: 50 ml	Butanol: 75 ml
Water: 50 ml	
Acetic acid solution (2N): 50 ml	Acetic acid solution (2N): 75 ml

2. Stoppered bottle are shaken for one hour and allowed to stand till the liquids layers are separated.

3. Pipette out 5 ml aqueous layer in a conical flask. Add 20 ml water and 2-3 drops of phenolphthalein indicator. Shake the mixture and titrate against standard NaOH solution.

4. Pipette out 5 ml organic layer into a conical flask. Add 20 ml water and 2-3 drops of phenolphthalein indicator. Shake the mixture and titrate against standard NaOH solution.

5. Calculate C_1 and C_2

6. Find out the ratio C_1 / C_2 in each case.

Results:

Table 1

(Temperature details)

Temperature before the experiment (^{0}C)	Temperature after the experiment (^{0}C)	Average temperature (^{0}C)

Table 2

(Standardization of aqueous layer and n-butanol by NaOH solution)

Bottle no.	Layer taken	Volume of layer (ml)	Burette reading (ml)	Partition Coefficient $(K=C_1/C_2)$	Mean Partition Coefficient

1	Organic (C1)				
	Aqueous (C2)				
2	Organic (C1)				
	Aqueous (C2)				

Conclusion:

The partition coefficient of acetic acid between n-butanol and water at ……..⁰C is ……….. .

Precautions:

1. Pipette, volumetric flask used in the experiment must be properly cleaned and calibrated.
2. While withdrawing aliquots, one layer must not be contaminated with others.
3. Temperature should be noted at the beginning and at the end of the experiment.
4. n-butanol is less polar solvent with respect to water, so it will form homogeneous solution with acetic acid. Hence vigorous shaking is essential for at least one hour to make the system heterogeneous so that acetic acid can be distributed properly.

5. pH metric titration for determination of strength of a given acid solution against a standard NaOH solution

Theory:

pH meters have provisions of standardizing the glass electrode in a buffer solution of known pH. This is necessary because different electrodes have different electrode potentials. Once the adjustment has been made, the meter registers correctly the known pH of the buffer solution and the instrument gives the pH of other solution without any calculation.

Measurement of pH is also employed to monitor the course of acid base titrations. The pH values of the solution at different stages of acid base neutralization are determined and are plotted against the volume of alkali added. On adding a base to an acid, the pH rises slowly in the initial stages then it changes rapidly at the end point. Then it flattens out. The end point of the titration can be detected where the pH changes most rapidly. However, the shape of the inflexion of the acid and the base used and on the basicity of the acid and the acidity of the base.

Apparatus:

pH meter, beaker, burette, conical flask, pipette.

Chemicals:

Buffer solution of pH 4 and 9, oxalic acid, sodium hydroxide solution

Procedure:

A. Standardization of the pH meter:

a) Switch on the instrument. Wait 10-15 minutes so it gets warmed up.

b) Prepare the buffer solutions having pH 4 and 9 (by using buffer tablets of BDH or E-Mark)

c) Wash the combined electrode with distilled water.

d) Take the buffer solution (pH =4) in a clean beaker. Lower the electrode so that it is immersed in the solution to a depth of about one inch.

e) Measure the temperature of the solution by a thermometer and set temperature compensate control to this value.

f) Set the pointer to pH exactly by means of Set Zero Control.

g) Put the selector switch to proper pH range 0-7 (because pH of buffer solution is 4).

h) Set the pointer to the known pH value of the buffer by turning the Set Buffer Control. Now the standardization is done.

i) Put back the selector to zero position.

Wash the electrodes with distilled water and standardize the pH meter by using a buffer solution of pH = 9. Same procedure is followed except now the selector switch is put to a pH range of 7-14.

B. Procedure for pH -metric titration:

a) Standardize the pH meter as described above.

b) Clean the electrodes by distilled water and wipe them with tissue paper.

c) Take 40 ml oxalic acid solution in a 100 ml beaker and immerse the electrodes.

d) Set up a burette containing standard sodium hydroxide solution.

e) Measure the temperature of the solution and set temperature compensate to this value.

f) Put the selector to the expected range.

g) The reading shown on the scale of the meter is the pH value of the unknown solution.

h) Add sodium hydroxide solution gradually from the burette. Note the corresponding value from the meter. Near the end point add small amount of sodium hydroxide solution because change in pH will be very much appreciable when the acid is neutralized.

i) Put back the selector to zero position after pH measurement and before removing the electrodes from solution.

j) Plot a graph between pH and drops of sodium hydroxide added. From this graph determine the volume of NaOH required for complete neutralization of oxalic acid.

Results:

Temperature of the solution = 27°C

Volume of oxalic acid taken = 40 ml

Observation table:

No of observations	Drops of NaOH	pH of the solution
1		
2		
3		

4		
5		
6		
7		
8		
9		
10		
11		
12		

A graph has been plotted against drops of NaOH (x-axis) vs pH of the solution (y-axis). From graph, required volume of NaOH at the equivalence point would be determined.

Calculation:

Total volume of NaOH required = V1 ml (from burette reading)

Volume of 1 drop of NaOH = V1 / Total no. of drops = V2 ml

Volume of NaOH required for complete neutralization of oxalic acid

  = No. of drops (from graph) X V2 ml   = V3 ml

Now,

$$\text{Strength of oxalic acid} = \frac{\text{Volume of NaOH} \times \text{Strength of standard NaOH}}{\text{Volume of oxalic acid}}$$

$$= \frac{V3 \times 0.213}{40} \; (\text{N})$$

$$= \dots\dots\dots (\text{N})$$
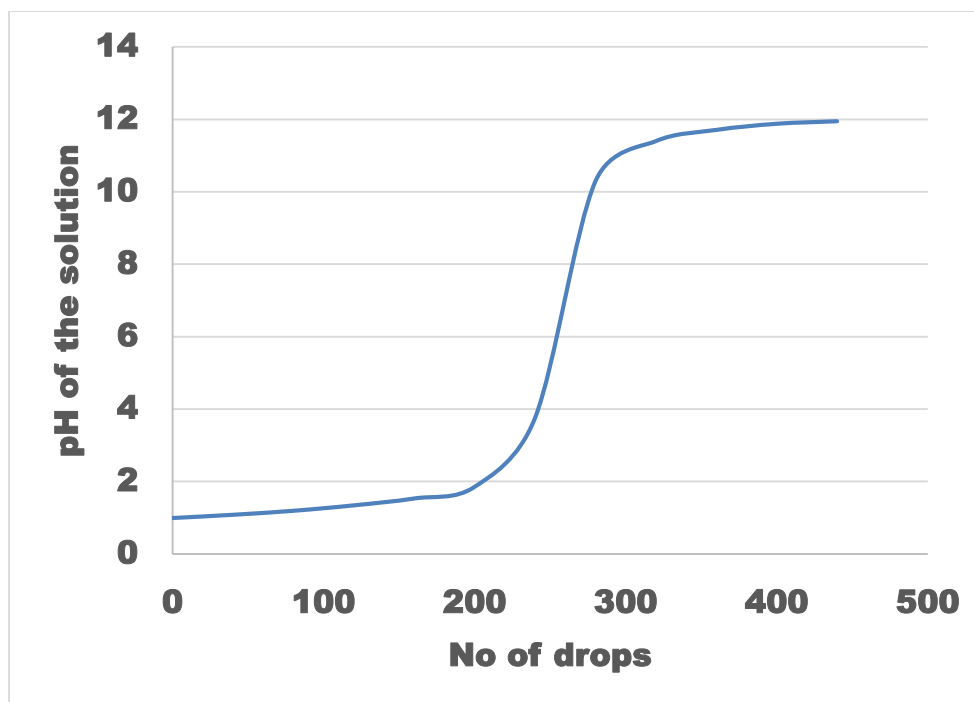
Sample plot of pH metric titration

Precautions:

1. Electrodes must be immersed properly in the solution and sufficient time to be allowed for the electrodes to assume the temperature of the solution.

2. The temperature of the solution should not change appreciably during the measurement.

3. If possible magnetic stirrer may be employed or the solution should be stirred mechanically from time to time.

4. After the determination of pH value, back the selector to zero position and before removing the electrodes from solution. Leave the selector in zero position when not in use.

6. Conductometric titration for determination of strength of a given HCl solution by titration against a standard NaOH solution

Theory:

Conductometry can be used to detect the equivalence point (end point) of a titration. This method is based upon a measurement of conductance during the course of titration. During ionic reactions, the conductance may either increase or decrease depending upon the nature of the electrolyte involved. The substitution of ions of one conductivity by the ions of another conductivity is the basis of conductometric titrations.

HCl being a strong electrolyte undergoes complete ionization and produce a large number of cations and anions in solution. The initial conductance of the solution will be very high as the ionic conductance of ionic mobility of H^+ ion is highest.

When is HCl, taken in a beaker, is gradually titrated against a base NaOH, being added from burette, the following reaction will occur.

$$H^+ + Cl^- + Na^+ + OH^- \quad \rightarrow \quad Na^+ + Cl^- + H_2O$$

The salt NaCl remains in the solution in completely ionized form while water being a weak electrolyte remains partially ionized. The H^+ ions are gradually replaced by equivalent amount of Na^+ ions which have relatively much lower ionic conductance than that of H^+ ions. Thus the conductance of resulting solution will decrease. This trend will continue until all H+ ions are replaced by Na^+ ions, i.e. equivalence point is reached. At the equivalence point, only solution of NaCl is present. Beyond equivalence point, further addition of alkali increases the number of Na^+ and OH^- ions which remain unutilized in the solution. As a result, conductance of the solution increases. Thus the conductance will minimum at the equivalence point.

When conductance values are plotted against volume of titrant added, two straight lines are obtained; the point of intersection of the lines give the end point corresponding to the minimum conductance and consequently equivalence point. The volume of alkali added corresponding to the equivalence point represent the volume of alkali required for neutralization of acid.

Apparatus:

Conductivity bridge, conductivity cell, beaker, burette, pipette, volumetric flask, conical flask, funnel.

Chemical required:

0.1 (N) standard oxalic acid solution, 0.1 (N) NaOH solution, 0.1 (N) HCl solution, and phenolphthalein indicator.

Procedure:

A) Prepare 0.1 (N) 250 ml standard oxalic acid solution.

B) Standardize 0.1 (N) NaOH solution (supplied) against oxalic acid solution using phenolphthalein indicator.

C) Rinse the conductivity cell with deionized water.

D) Pipette out 50 ml 0.1(N) HCl solution (supplied) into the conductivity cell (both the electrodes should be completely immersed within the solution). Measure the conductivity of the solution carefully.

E) Add NaOH solution from a burette drop wise (3 to 5 drops) to the beaker with constant a gentle stirring and measure the conductance of the solution after each addition. Repeat the process until 5 point beyond the endpoint have been recorded.

F) Plot a graph between observed conductance values along y-axis against the volume of alkali added along x-axis. The point of intersection gives the amount of alkali required for neutralization of acid. Calculate the strength of HCl solution.

G) Check the strength of HCl solution by titration against standard NaOH solution using phenolphthalein indicator.

Results:

The temperature of the solution: ……………^{0}C

Table 1

(Preparation of standard oxalic acid solution)

Initial weight	Final weight	Weight taken (W)	Weight to be taken	Strength
			1.575	

Strength of oxalic acid solution = $\dfrac{W}{1.575}$ x (N/10) = M (N/10)

Table 2

(Standardization of NaOH solution by standard oxalic acid solution)

No of observation	Vol. of oxalic acid (ml)	Burette reading (ml)			Mean volume of NaOH (Y ml)	Strength of NaOH solution
		Initial	Final	Difference		
1						
2						
3						

Strength of NaOH solution = $\dfrac{\text{Volume of oxalic acid solution X Strength of oxalic acid solution}}{\text{Volume of NaOH solution}}$

$$= \frac{10 \text{ X M (N/10)}}{Y} = Z \text{ (N/10)}$$

Table 3

(Conductometric titration of HCl solution against a standard NaOH solution)

Volume of NaOH added (ml)	Conductance of the solution (mho)	Volume of NaOH added (ml)	Conductance of the solution (mho)
0			
2			
4			
6			
8			

A plot of conductance of solution (y-axis) vs volume of NaOH (x-axis) has been drawn and from the graph, exact volume of NaOH was obtained.

Table 4

(Determination of strength of HCl solution by standard NaOH solution)

No of observation	Vol. of HCl taken (ml)	Burette reading (ml)			Mean volume of NaOH (ml)	Strength of HCl solution
		Initial	Final	Difference		
1						
2						
3						

Volume of NaOH = A ml (from graph)

Strength of NaOH = Z (N/10)

Volume of HCl = 10 ml.

So, the strength of HCl solution = (AxZ) / 10 = B (N)

Table 5

(Comparative study)

Method	Strength	Ratio
Conductometric titration		
Titration using indicator		
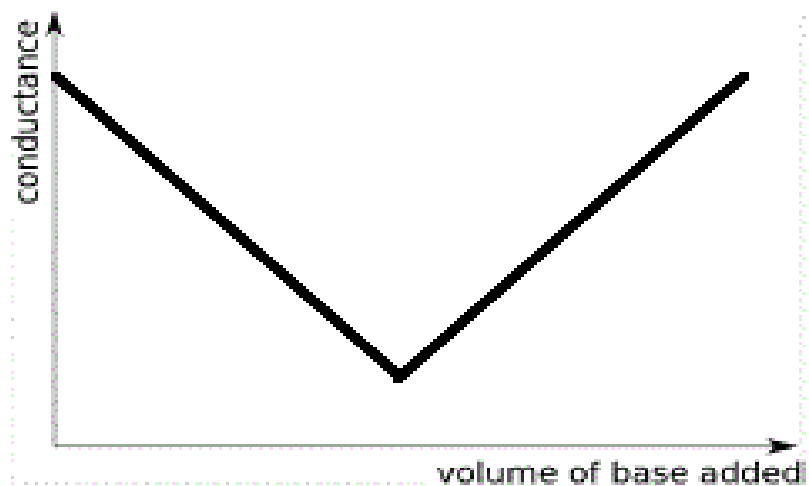
Sample plot of Conductometric titration

Precautions:

1.   Temperature of the experiment should be noted.

2.   Conductivity cell must be thoroughly washed with de-ionised water.

3.   Ensure that there is no air bubble in the burette.

4.   During titration, the titration flask should be constantly swirled.

5.   For each titration, use same no of drops of indicator.

7. Determination of viscosity coefficient of a given liquid with Ostwald's viscometer

Theory:

The viscosity is the characteristic property related to the flow of fluid. In case of streamline flow of liquid through a tube, the liquid layer adjacent to the wall of the tube remains stationary. The velocity of flow of consecutive layers towards the center of the tube increases gradually, being maximum at the center. Thus a layer, moving with another slower adjacent layer experiences a drag acting opposite to the direction of motion. This backward drag arises due to internal friction between two consecutive layers. The property of a liquid by virtue of which it opposes a relative motion of its layer is called viscosity.

In 1844, Hagen–Poiseuille deduced the expression for viscosity coefficient (η). This equation is called Poiseuille's equation and is expressed as

$$\eta = \frac{\pi r^4 P t}{8 V L} \qquad (1)$$

Where t is the time of flow of liquid, V is the volume of the liquid, P is the hydrostatic pressure and L is the distance travelled by the liquid during time t. The unit of viscosity is called the Poise and SI unit for absolute viscosity is kg/m s (or Pascal-seconds, Pa s).

Viscosity can be measured using a viscometer. Different types of viscometer are used like Ostwald viscometer, Falling sphere viscometer, Falling piston viscometer, Oscillating piston viscometer, Vibrational viscometers, Rotational viscometers and Bubble viscometer

Ostwald viscometer is a commonly used viscometer, which consists of a U-shaped glass tube held vertically. For more accurate measurements, it is held in a controlled temperature bath. It is also known as a glass capillary viscometer. A liquid is allowed to flow through its capillary tube between two etched marks and the time of flow of the liquid is measured using a stopwatch.

In an Ostwald viscometer, the distance travelled by the liquid (L), the radius (r) and the volume of liquid (V) will be constant. So, the equation (1) can then be simplified to:

$$\eta = K\,P\,t \text{ ----------(2)}$$

where K is a constant and

$$K = \frac{\pi\,r^4}{8\,V\,L}$$

Let equal volume of two liquids of coefficient of viscosity η_s and η_w flow down the same viscometer in time t_s and t_w respectively, then

$$\frac{\eta_s}{\eta_w} = \frac{P_s.\,t_s}{P_w.\,t_w} \text{ ----------(3)}$$

Again, the pressure is directly proportional to the density (ρ) of the liquid, so

$$P_s = h\rho_s\,g \quad \text{and} \quad P_w = h\,\rho_w\,g$$

Now, equation (3) becomes…

$$\frac{\eta_s}{\eta_w} = \frac{\rho_s.\,t_s}{\rho_w.\,t_w}$$

$$\text{or,}\ \eta_s = \frac{\rho_s\,.ts}{\rho_w.\,tw} .\,\eta_w$$

Apparatus:

Ostwald's viscometer, Specific Gravity bottle, Clamp, Stopwatch

Chemical required:

Sample liquid, water

Procedure:

1.   Determine the specific gravity of the given solution by using specific gravity bottle.

2.   Take 10 ml of water in the wider arm of a clean viscometer and clamp it vertically. Suck water into the other arm above the upper mark.

3.   Allow to fall a definite volume of water between two definite marks. Start the stopwatch when the liquid touches the upper mark of the bulb and stop the watch when the liquid touches the lower mark of the bulb. Note the time of flow.

4.   Wash the viscometer thoroughly with distilled water, rinse with the given liquid and repeat the procedure with the supplied solution.

Results:

Temperature = ……………….°C

Table 1: Specific gravity of the given liquid

Weight of empty specific gravity bottle (W_1 g)	Weight of empty specific gravity bottle + water (W_2 g)	Weight of empty specific gravity bottle + experimental liquid/ solution (W_3 g)	Sp. Gravity of the experimental liquid $\mathrm{ds} = \dfrac{W_3 - W_1}{W_2 - W_1}$

Table 2: Determination of coefficient of viscosity of the given solution

Liquid	Time of Flow in sec	Mean time of Flow (sec)	Coefficient of viscosity (centipoise) $\eta_s = \dfrac{\rho_s \times t_s}{\rho_w \times t_w} \cdot \eta_w$

Water			
Given solution			

Calculation:

Coefficient of viscosity of water at t°C = 1 centipoise

Coefficient of viscosity of the given solution = ……. centipoise

Conclusion:

The importance of the experiment can be understood from the following examples.

(i) The knowledge of coefficient of viscosity of organic liquids is used to determine their molecular weights.

(ii) The knowledge of coefficient of viscosity and its variation with temperature helps us to choose a suitable lubricant for specific machines. In light machinery thin oils (example, lubricant oil used in clocks) with low viscosity is used. In heavy machinery, highly viscous oils (example, grease) are used.

N.B.

Density = Mass / Volume.

Specific gravity is the density of a substance divided by the density of water.

Since water has a density of 1 g/cm³ (at sea level, and 4°C) specific gravity is usually very close to the same value as density (but without any units).

A sample viscometer

Upper mark indicating where sample is moved to by suction

Lower mark representing original position which sample returns to when suction force is released

Capillary tube which tests samples viscosity by reducing the diameter of tube which oil flows in
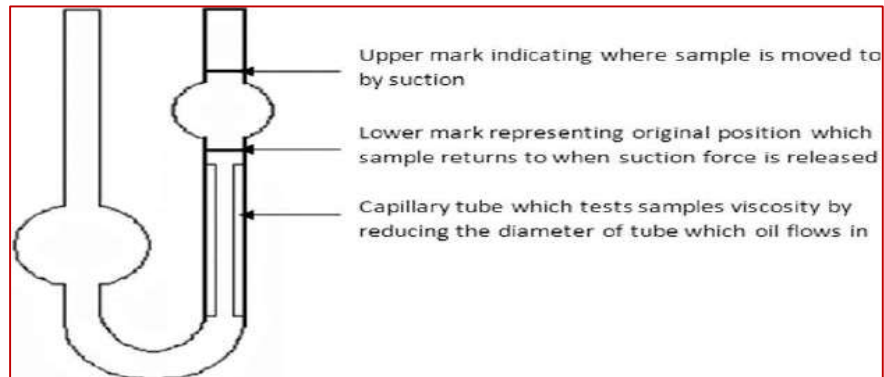
Precautions:

1. The Ostwald,s viscometer should be held in vertical positions during the flow of liquids.

2. While sucking the liquids in the viscometer, care should be taken to avoid the inflow of any air bubble into the viscometer.

3. The viscometer should be cleaned well before use.

8. Determination of saponification value of a given oil sample

Theory:

The saponification value of an oil or fat is the number of milligrams of KOH required to neutralize the fatty acids resulting from complete hydrolysis of one gm of oil or fat. When a known weight of oil is boiled with excess of alcoholic KOH solution,the triglycerides hydrolyse and glycerol and soap are formed. The alkali consumed by this hydrolysis is a measure of the saponification value.The oil is saponified completely according to the following equation:

$$
\begin{array}{ll}
CH_2-O-COR & CH_2OH \\
\;\;\;| & \;\;\;| \\
CH-O-COR \;\; + \;\; 3KOH \;\; \rightarrow \;\; CHOH \;\; + \;\; 3\;RCOOK \\
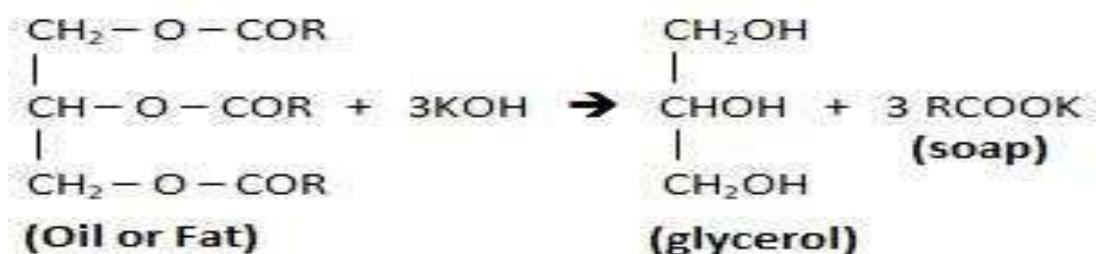\;\;\;| & \;\;\;| \qquad\qquad\quad (soap) \\
CH_2-O-COR & CH_2OH \\
(Oil\ or\ Fat) & (glycerol)
\end{array}
$$

Materials required:

0.5 mL KOH solution, 0.5 mL HCl solution, phenolphthalein indicator, 250 mL conical flask, reflux condenser, burette.

Procedure:

1. Accurately weigh 2 gm of coconut oil or any other supplied oil into a 250 mL conical flask with a ground glass joint and add 25 mL of KOH solution.

2. Attach a reflux condenser and heat the flask contents on a steam bath for 1 hour with occasional shaking.

3. Titrate the excess KOH under hot condition with 0.5 N HCl using phenolphthalein indicator. Record the titrant as V_1mL.

4. Titrate 25 mL of KOH with 0.5 N HCl using phenolphthalein indicator which serves as blank. Record the titrant as V_2mL.

Results:

No. of Obs	Volume of HCL for sample (V_1 mL)	Volume of HCL for blank (V_2 mL)	$(V_2 - V_1)$ mL	Average $(V_2 - V_1)$ mL
1				

2			
3			

Calculation:

$$\text{Saponification Value} = \frac{(V_2 - V_1) \times 0.5 \times 56.1}{\text{Wt. of oil (gm)}}$$

$$= \ldots\ldots\ldots \text{ mg KOH/gm}$$

Things to know:

1. Fats and oils are the principle stored forms of energy in many organisms. They are derivatives of fatty acids.

2. Fatty acids are carboxylic acids with hydrocarbon chains of 4 to 36 carbons, which can be saturated or unsaturated.

3. The simplest lipids constructed from fatty acids are triacylglycerols or triglycerides. Triacylglycerols are composed of three fatty acids each in ester linkage with a single glycerol.

4. Saponification literally means "soap making".

5. It is important for an industrial user to know the amount of free fatty acid present, since this determines the large measure of refining loss.

Precautions:

1. All glassware used must be absolutely clean and oil free.

2. The indicator solution should not be kept open as it contains alcohol which tends to evaporate.

9. Preparation of silver nanoparticles

Theory:

Silver nano particles are nanoparticles of silver between 1 nm and 100 nm in size. Numerous shapes of nanoparticles can be constructed depending on the application. Commonly used are spherical silver nanoparticles but diamond, octagonal and thin sheets are also popular.

Their extremely large surface area permits the coordination of a vast number of ligands. Introduction of silver into bacterial cells induces a high degree of structural and morphological changes, which can lead to cell death. As the silver nanoparticles come in contact with the bacteria, they adhere to the cell wall and cell membrane. This property of silver nanoparticles led to their increasing application in antimicrobial coatings, many textile, keyboards, wound dressings and biomedical devices.

At the macro scale, silver always look like silver, but solution of silver nanoparticles may have colour. In the silver nano particles, electrons oscillate collectively. The oscillation affects how light interact with the nanoparticles. The specific oscillation depends on the particle size and shape. So particles of different size have different colours. Colours can be measured by UV-Spectro-photometer.

Two most popular method of synthesizing silver nanoparticles are

1. Turkevich method (1951): Silver reduced by trisodium citrate
2. Brust method (1994) : Silver reduced by sodium borohydride

Here, we are going to use Turkevich method, since the materials are less hazardous.

Procedure:

1. Boil 60 ml 1(M) silver nitrate solution, covered with watch glass on a hot plate.
2. Stir the solution with a magnetic stir bar.
3. Once boiling, add 6ml 10 (M) trisodium citrate drop wise, about one drop per second.
4. Again cover with watch glass.
5. Wait for solution to change to a light golden colour.
6. Carefully removed beaker from hot plate and let the solution cool.

Reactions:

Silver nitrate   Trisodium citrate   AgNPs
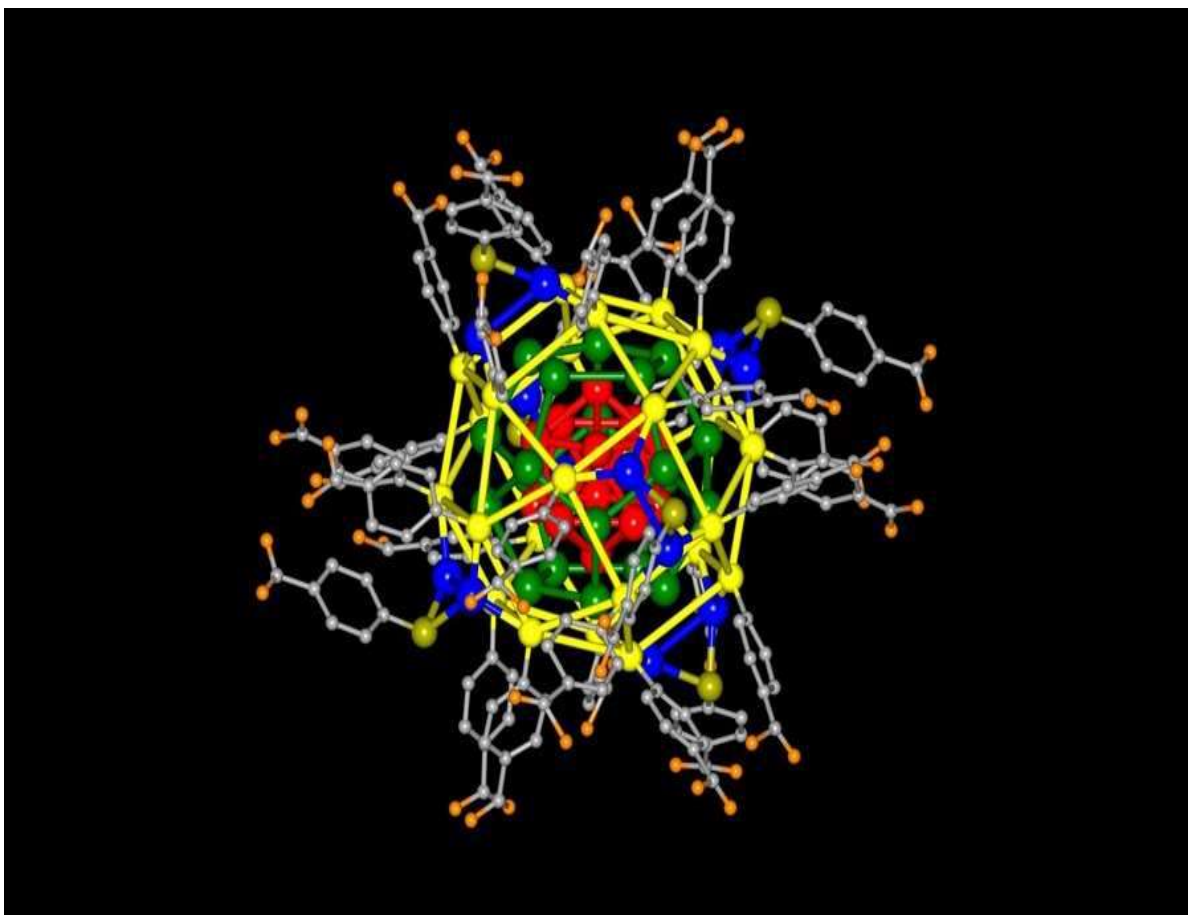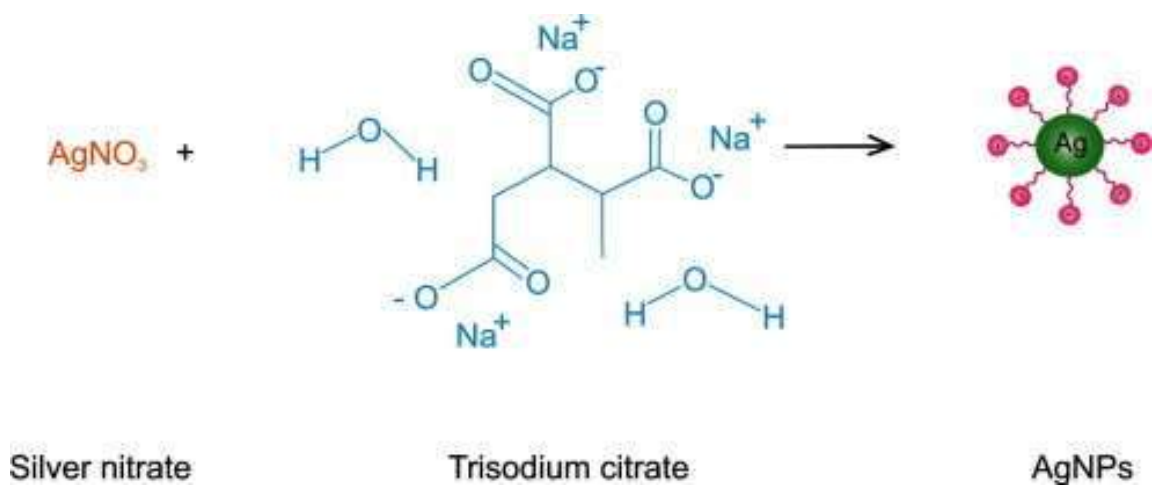
Silver nanoparticles

10. Determination of rate constant of a 1st order reaction

Theory:

For a first-order reaction, the rate of reaction is directly proportional to the concentration of one of the reactants.

Differential Rate Law: r = k [A]

The rate constant, k, has units of sec^{-1}.

A first-order reaction depends on the concentration of only one reactant (a unimolecular reaction). Other reactants can be present, but each will be zero-order. The rate law for an elementary reaction that is first order with respect to a reactant A is

$$r = -\frac{d[A]}{dt} = k[A]$$

k is the first order rate constant, which has units of time^{-1}.

The integrated first-order rate law is

$$\ln [A] = -kt + \ln [A]_0$$

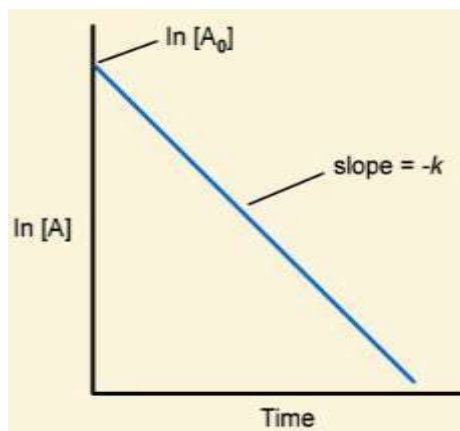A plot of ln[A] vs. time t gives a straight line with a slope of − k.

The half life of a first-order reaction is independent of the starting concentration and is given by:

$$t_{\frac{1}{2}} = \frac{\ln (2)}{k}$$

The hydrolysis of methyl acetate is a 1st order reaction. In water the hydrolysis is very slow and is catalysed by H^+ ions.

$$CH_3COOCH_3 \ + \ H_2O \xrightarrow{\quad [H^+] \quad} CH_3COOH \quad + \quad CH_3OH$$

Methyl acetate   Acetic acid   methyl alcohol

The progress of the reaction can be studied by withdrawing measured volume of reaction mixture from time to time followed by titration with alkali. The volume of alkali required at any stage is equivalent to the sum of acetic acid produced along with acid catalyst (constant quantity).

If V_1 and V_n and $V\alpha$ are the volume of alkali required at the time t_1, t_n & $t\alpha$ respectively.

A graphical plot of $\log (V\alpha - V_1) / (V\alpha - V_n)$ vs Δt will give a straight line passing through the origin and k can be determined from the slope.

This confirms the reaction is 1st order.

Apparatus:

Burette, Pipette, Reagent bottles, Conical flask, Volumetric flask, Stop watch

Chemicals required:

Methylacetate, N/2 HCl, N/10 NaOH, Phenolphthalein

Procedure

1. Pipette out 50 ml of N/2 HCl in a 250 ml dry conical flask
2. Pipette out 5 ml of methyl acetate into the flask with constant swirling and start a stop watch at the time of half discharge from the pipette. Shake the mixture thoroughly.
3. After 5 minutes pipette out 2 ml aliquot from the reaction mixture in a separate conical flask containing 50 ml ice-cooled distilled water.
4. Note the time at half discharge.
5. Titrate the solution with N/10 NaOH using phenolphthalein as indicator.
6. Repeat steps 3, 4 & 5 for 6 times at an interval of 5 minutes.

7. Heat the rest of the solution at about 60°C in a water bath for 30 minutes. Cool to room temperature & repeat steps 3, 4 & 5 to get $V\alpha$.

Results:

Room Temperature = _____°C

No. of Obs	Time in secs	$\Delta t = t_n - t_1$	Volume of aliquot (ml)	Volume of NaOH (ml)	$V\alpha$ (ml)	$(V\alpha - V_1)$ ml	$(V\alpha - V_n)$ ml	Log $\dfrac{(V\alpha - V_1)}{(V\alpha - V_n)}$

Graph Plot:

Plot Log $\dfrac{(V\alpha - V_1)}{(V\alpha - V_n)}$ vs. Δt for each set and calculate k from the slope of the graph.

k = Slope × 2.303   = …………………….sec^{-1}

Precaution:

1. The aliquot should be placed in ice cooled distilled water after pipetting out.

2. The time gap should be maintained as mentioned.

SAMPLE QUESTIONS FOR VIVA-VOCE

1. Define pH

2. Name the instrument used for pH metric titration

3. Mention at least two advantages of pH metric titration.

4. How the pH meter is calibrated?

5. Mention the pH of the buffer solutions used for calibration of a pH meter.

6. Which electrode is used in pH meter?

7. What is the shape of the plot of pH metric titration?

8. Do we need to record the temperature in this experiment?

9. Describe in brief the procedure of pH metric titration.

10. How the plot of a monobasic acid differs from a dibasic acid in pH metric titration.

11. Describe the procedure of determination of chloride content from water?

12. What is the brick red precipitate in the determination of chloride content from water?

13. Define solubility product.

14. What is argentometric method?

15. What is the unit of chloride content & conductance

16. Name the indicators used in the following experiments?

i.   Determination of chloride ion

ii.   Total Hardness

iii.   Calcium Hardness

iv.   Partition coefficient

v.   Hydrolysis of methyl ester

vi.   Conductometric titration

17. What is the full form of EBT & EDTA?

18. Show the structure of EDTA.

19. What are the captions and anions responsible for hardness of water?

20. Why hardness is measured with respect to calcium carbonate.

21. Why the wine red colour is develops during measuring total hardness of water?

22. Describe in brief the procedure for measuring Calcium hardness of water

23. Why do you observe blue colour at the end point while estimating total hardness of water?

24. What is the unit of hardness of water, chloride content & conductance

25. What is Nernst Distribution law of Partition coefficient?

26. Is measurement of temperature important for viscosity & partition coefficient?

27. Define Viscosity. What is its unit?

28. What is Poisulie's equation?

29. Mention the working formula used in Determination of coefficient of viscosity by Ostwald's Viscometer.

30. How the working formula is derived?

31. Describe Ostwald's Viscometer.

32. Name some viscometers.

33. What is the unit of conductance?

34. What is the major difference between the plot of conductometric titration and pH metric titration?

35. What are the advantages of conductometric titration?

36. What are the disadvantages of conductometric titration?

37. Define nanomaterial.

38. What are the colours of silver nanoparticles?

39. Why colour varies in silver nanoparticles.

40. Define the order of a reaction.

41. What are factors that affect reaction rates?

42. What do you mean by the rate of a reaction?

43. Mention the importance of partition coefficient.

44. What happens if shaking is not in partition coefficient experiment?

45. What is primary standard? Give examples.

46. What kind of compounds are selected as primary standards.

47. What are secondary standard solutions? Give examples.

48. How will you prepare a 250 ml (N/10) Oxalic acid solution?

49. How will know the exact strength of a NaOH solution prepared in your laboratory.

50. Define rate constant.

ABOUT AUTHORS

Dr. Sumit Nandi did B.Sc. (Hons.) in Chemistry, B.Tech. (Chemical Technology), M.Tech. (1st class 1st & Gold Medalist) in Chemical Technology and Ph.D. (Tech.) from Calcutta University. After that he completed MBA with dual specialization in finance and marketing. He also completed one year Post Graduate Diploma in Environmental Management from Kalyani University. Dr. Nandi has been teaching for the last seventeen years at Narula Institute of Technology under JIS Group and presently working as Head and Associate Professor, Department of Basic Science of Humanities in the same institute. Dr. Nandi has been teaching as a Visiting faculty in the Department of Chemical Technology, Calcutta University for the last twenty one years. He has several years' industrial experience also. His main research area is enzyme kinetics, environment-friendly biofuels, mathematical modelling in environmental issues, characterization of blended polymers etc. Several prestigious projects from DBT and UGC have also added many feathers in his cap. He has six patents and published one book on "Engineering Chemistry Simplified" for the B.Tech. students. He guided several research scholars also. He has got best paper presentation award several times in different international conferences in India and abroad. Dr. Nandi contributes more than 100 research publications in reputed international journals and conferences. He visited different countries like Germany, China, Poland, Czec Republic, Hungary, Bulgaria and Lithuania for invited talk and attending international conferences. Dr. Nandi acted as Coordinator in the World Bank funded TEQIP project. He conducted several International and National conferences, Workshops, Faculty Development Programmes, Webinars etc. at Narula Institute of Technology

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