PRINCIPLES FOR SAFETY IN THE CHEMICAL LABORATORY

Safe practices in the chemical laboratory are of prime importance. A student should consider it an essential part of his or her educational experience to develop safe and efficient methods of operation in a lab. To do this, one must acquire a basic knowledge of properties of materials present in the lab, and one should realize the types of hazards that exist and the accidents and injuries that can result from ignorance or irresponsibility on the part of the student or a neighbor.

Regulations

1. Report all accidents to the instructor or lab assistant immediately.
2. NEVER eat, drink, chew, or smoke in the laboratory.
3. NEVER leave an experiment unattended. Inform the lab assistant if you must leave the lab.
4. After the experiment is completed, turn all equipment off, making sure it is properly stored, and clean your area. Failure to comply with these regulations is cause for immediate dismissal from lab.

Precautions

1. Approach the laboratory with a serious awareness of personal responsibility and consideration for others in the lab.
2. Become familiar with the location of safety equipment, such as acid-base neutralizing agents, eye wash, fire extinguisher, emergency shower, and fire blanket.
3. Pay strict attention to all instructions presented by the instructor. If something is not clear, do not hesitate to ask the instructor or lab assistant.
4. Clean up all chemical spills immediately.
5. Be aware of all activities occurring within a reasonable proximity of yourself since you are always subject to the actions of others.
6. To avoid contamination of community supplies, do not use personal equipment such as spatulas in shared chemicals and replace all lids after use.
7. Avoid unnecessary physical contact with chemicals; their toxic properties may result in skin irritation.
8. Use all electrical and heating equipment carefully to prevent shocks and burns.
9. NEVER handle broken glassware with your hands; use a broom and a dust pan.
10. Wash your hands at the end of the laboratory.

Personal Attire

Choice of clothing for the laboratory is mainly left to the discretion of the student. Because of the corrosive nature of chemicals, it is in your best interest to wear comfortable, practical clothing. Long, floppy sleeves can easily come into contact with chemicals.

A lab coat is suggested to help keep clothes protected and close to the body. Accessories also need consideration. Jewelry can be ruined by contact with chemicals. Open toed shoes do not adequately protect one against chemical spills. If hair is long enough to interfere with motion or observation, it should be tied back. Remember that your clothes are worn to protect you.

Assembling Equipment

Equipment should be assembled in the most secure and convenient manner. Utility clamps are provided to fasten flasks, etc., to the metal grid work located at the center of each bench. This keeps top-heavy or bulky equipment away from the edge where it can be knocked easily off the bench. Consider the safe location of the hot plate. Keep it near the grid work to minimize chances of contact with the body. If the aspirator is being used, locate your apparatus near the sink for convenience.

Handling Glassware

Laboratory glassware is usually fragile, and if it is not properly handled, serious injuries may result do not force glass tubing or thermometers into a rubber stopper. Lubricate the tubing or thermometer with glycerol or water, wrap it in a towel, and gently insert it into the stopper by using pressure in a lengthwise direction while rotating it. Always grasp the tubing near the stopper. When removing the tubing, remember to protect your hands with a towel. If there are difficulties with this procedure, ask for the instructor's assistance. Apparatus that can roll should be placed between two immobile objects away from the edge of the bench. Chipped or broken glassware cannot be used. There are special receptacles near each bench for these waste materials. After the experiment is completed, all glassware should be emptied, rinsed, and cleaned.

Acids and Bases

In this lab sequence, you will come in contact with several acids and bases. As with all chemicals, caution must be taken to prevent contact with the skin. When handling these chemicals, keep hands away from the eyes and face until they have been thoroughly washed. If an acid or base comes in contact with your skin, flush the area with large quantities of clean, cold water. Eyes are extremely sensitive.

Use the eye wash provided in the laboratory, or wash with water for at least 10 minutes. Again, the instructor must be notified immediately. To insure your safety, neutralize acid or base spills before cleaning

them up. Boric acid solution is available to neutralize base spills, and carbonate powder is provided to neutralize acids.

Guidelines to be followed:

1. Always bring your notebook with you to lab. You will be graded on the completeness of your previous note taking and your preparation for the current experiment. You may use your notebook during a lab quiz.
2. Number the pages sequentially and reserve space at the beginning for a table of contents.
3. Take your notebook to the balance room, etc. and record values directly in it - not on loose scraps of paper.
4. Specify each measured quantity by name and include the units.
5. If you make a mistake in your notebook, simply draw a solid line through the error and write the correction nearby.
6. Tables greatly simplify data entry; they should be set up before coming to lab.
7. Write down all observations such as color and phase changes - don't rely on your memory.
8. Save time by doing trial calculations in your notebook before filling out any report sheets.
9. Save time by making preliminary sketches of graphs on the ruled lines in your notebook.

A.UNIVERSITY PRESCRIBED LAB EXPERIMENTS

EXPERMINET NO: 1 **Date:**

INTRODUCTION TO CHEMISTRY LABORATORY

In all chemistry laboratories chemical analysis is carried out. Chemical analysis is the resolution of a chemical compound into its proximate or ultimate parts. It is divided into two types.

1. **Qualitative Analysis**: It deals with identification and conformation of the nature of a substance present in a given sample.
2. **Quantitave Analysis**: It deals with the determination of how much of each component is present in a given sample. Quantitative Analysis is further divided into two types.
   - **Volumetric Analysis:** It is based on measuring the volume of the solution of a substance.
   - **Gravimetric Analysis**: It is based on estimation of the amount of a given compound from the results of weighing.

Terms used in Volumetric Analysis:

(i) **Titration**: It is a process of adding one solution from the burette to another in the conical flask in order to complete the chemical reaction.

(ii**) Titrant**: The solution of known strength is called as titrant.

(iii) **Titrat**e: The solution which contains the substance to be estimated is called as titrate.

STANDARD SOLUTION:

A solution whose concentration is known is called a standard solution.

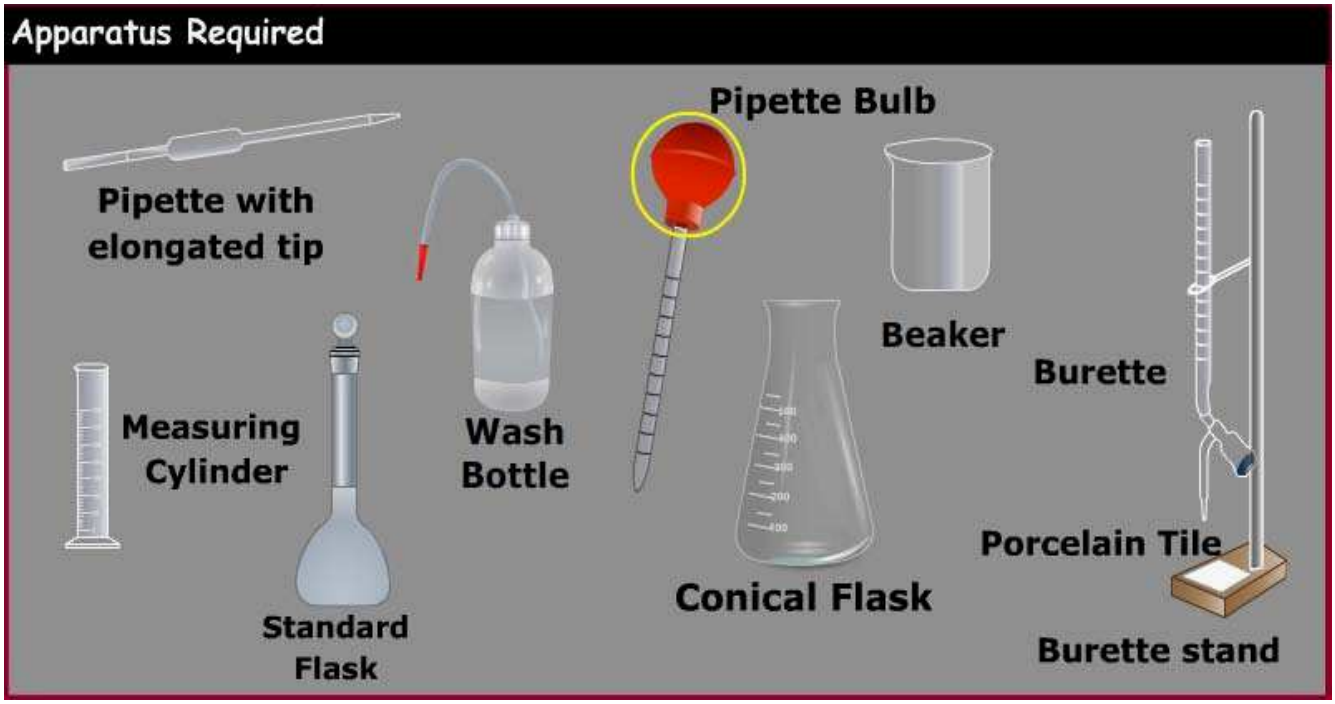
It is of two types

(iv) **Primary standard**: The substance whose standard solution can be prepared by direct weighing is known as Primary standard. They provide a reference to determine unknown concentrations or to calibrate analytical instruments and the composition of its solution should not change on standing or during storage.It is non-hygroscopic.

Examples: Oxalic acid, Potassium dichromate, Zinc sulphate, sodium carbonate etc.

(v) **Secondary Standards**: A secondary standard is a standard, their solutions are not prepared directly by weighing and the exact strength is determined by titrating against a primary standard and the process is called standardisation.It should change on standing or during storage.It is hygroscopic.

Example: NaOH,KOH,$KMnO_4$,HCl,H_2SO_4 etc.



(vi) **Indicator**: Indicator is a substance which indicates the completion of reaction in a titration by color change.

(vii) Error: "error" is not the same as a "mistake." It does not mean that you got the wrong answer. The error in measurement is a mathematical way to show the uncertainty in the measurement.

(viii)Percent of Error:

Error in measurement may also be expressed as a **percent of error**. .

$$\% \text{ of Error} = \frac{[\text{Actual value} - \text{Measured value}]}{\text{Actual value}} \times 100$$

(ix)Accuracy

Accuracy is how close a measured value is to the **actual (true) value**. The word **accuracy** means correctness. It means that there are no errors. The **accuracy** of a measurement system is the degree of closeness of measurements of a quantity to that quantity's actual (true) value.

(x) Precision

Precision is how close the measured values are **to each other**. The **precision** of a measurement system, also called reproducibility or repeatability, is the degree to which repeated measurements under unchanged conditions show the same results. Precision measurements are those which are repeatable - so all measurements are clustered around the same value.

Concentration of a standard solution is generally expressed as:

1. Morality : It is the no of gram molecules of solute present in one liter of solution

$$\text{Molarity M} = \frac{\text{weight of the substance}}{\text{Gram molecular weight}} \times \frac{1000}{\text{volume in ml}}$$

2. Normality: It is defined as the no of gram equivalents of solute present in one liter of solution.

$$\text{Normality N} = \frac{\text{weight of the substance}}{\text{Gram equivalent weight}} \times \frac{1000}{\text{volume in ml}}$$

$$\text{Equivalent weight of an acid} = \frac{\text{molecular weight of an acid}}{\text{Number of replaceable } H^+ \text{ ions}}$$

$$\text{Equivalent weight of a base} = \frac{\text{molecular weight of a base}}{\text{Number of replaceable } OH^- \text{ ions}}$$

$$\text{Equivalent weight of an oxidizing or reducing agent} = \frac{\text{molecular weight of a substance}}{\text{No.of electrons gained or lossed}}$$

3. Molality: It is defined as the number of the moles of the solute present in 1 kg or 1000ml of the solvent,

$$\text{Molality m} = \frac{\text{no. of moles of solute}}{\text{Gram molecular weight}} \times \frac{1000}{\text{volume in ml}}$$

Types of Volumetric Titrations :

1) Acid-Base Titrations
2) Redox titrations
3) Complexometric Titrations

1) Acid-Base Titrations:

Titration is a process of neutralization.This method is used for determining an acid with alkali or base with an acid to produce salt and unionized water.

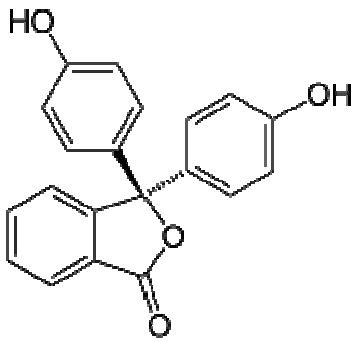
$$NaOH + HCl \rightarrow NaCl + H_2O$$

An indicator is (often) a weak acid or weak base that is placed into the unknown solution to determine the endpoint of the titration.

Phenolphthalein is a weak acid .it gives pink colour in alkaline medium and colourless in acidic medium and the Ph range is 8.0 – 9.6

$$H\text{-}phph_{(aq)} \rightleftharpoons H^+_{(aq)} + phph^-_{(aq)}$$

Colourless in acidic medium (H^+) attachéd **pink in basic media (H^-) ion is removed**

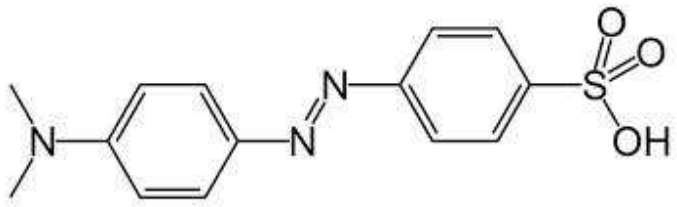


Structure of phenolphthalein indicator

Methyl orange indicator: It is a weak base. It gives yellow colour in basic media and red colour in acidic media and the pH range 3.1-4.4.

$$H\text{-}Meor_{(aq)} \rightleftharpoons H^+_{(aq)} + Meor^-_{(aq)}$$

Red Colour in acidic medium (H^+) attach **yellow colour in basic media (H^-) removed**



Structure of Methyl orange indicator

2) Red-ox titration:

It is also known as Oxidation – Reduction reaction.Titration of a reducing agent (loosing of electrons)by an oxidizing agent (gaining of electrons) or titration of an oxidizing agent by a reducing agent.is known as Redox titration. The common of the redox titrations are

1.Permanganometry : $KMnO_4$ is the oxidizing agent and titrated against a reducing agent like Fe^{2+}(ferrous ammonium sulphate) etc.

2. Dichrometry: $K_2Cr_2O_7$ is the oxidizing agent and titrated against a reducing agent like Fe^{2+}(ferrous ammonium sulphate) etc.

3. Iodometry: It is based on oxidation by the action of free iodine generated from KI.

Type of Redox Indicators:

Self-Indicators: Many a times the titrant itself may be so strongly coloured that after the equivalence point, a single drop of the titrant produces an intense color in the reaction mixture.

Eg: potassium permanganate. Such Indicators are called self indicators. Self indicators generally are strongly colored as a result of charge transfer transitions in them.

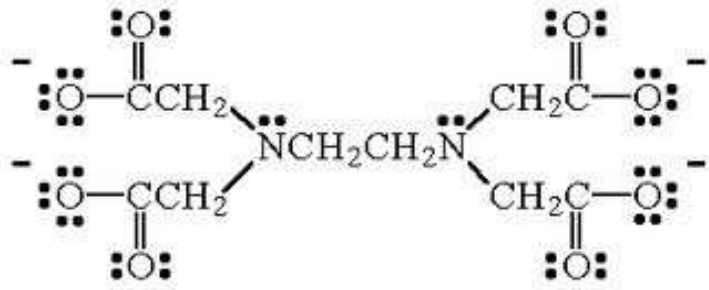
Internal indicator : Such indicators are added into the reaction mixtures Such indicators always have reduction potential values lower than the analyte system so that they react with the titrant only when whole of the analyte has been consumed, producing a readily detectable color change.

External indicator : In case a suitable redox indicator is not available for a given system, an indicator may be employed which will indicate the completion of reaction by physically or chemically reacting with the analyte (not through redox reaction). This reaction between indicator and the analyte may sometimes be an irreversible one and in some cases may even lead to precipitation. In those case indicators are not added to the reaction mixture on the whole, rather used externally on a grooved tile. Such indicators are called external indicators.

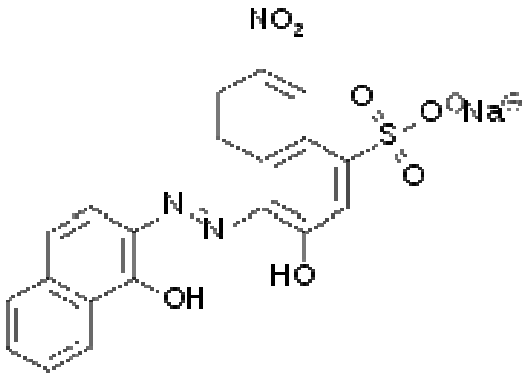
3) Complexometric Titrations:

The technique involves titrating metal ions with a complexing agent or chelating agent (Ligand) and is commonly referred to as complex metric titration. Ligand used widely in

complexometric titrations is EDTA (ethylene diamine tetra acetic acid),because it forms stable complexes with a number of metal atoms at a definite pH range.The indicator used in this titration is EBT(eriochrome blck T)



Structure of EDTA



Structure of EBT

Applications of Complexometric Titrations:

1. Complexometric titrations have been employed with success for determination of various metals like Ca, Mg, Pb, Zn, Al, Fe, Mn, Cr etc.
2. Determination of total hardness of water by Complexometric method.

Conclusion:

1. CO1 to CO6 and PO1, PO2, PO7, PO9 is attained
2. Students understand the terms- volumetric analysis, morarity, molality normality and redox titration.

VIVA QUESTIONS

1. What is meant by Volumetric Analysis?

Answer: It is based on measuring the volume of the solution of a substance.

2. What is meant by Standard solution?

Answer: A solution whose concentration is known is called a standard solution.

3. What is meant by Primary Standard solution?

Answer: The substance whose standard solution can be prepared by direct weighing is known as Primary standard.

4. What is meant by Secondary Standard solution?

Answer: A secondary standard is a standard, their solutions are not prepared directly by weighing and the exact strength is determined by titrating against a primary standard.

5. What is meant by Normality, Morality & Molality?

Answer: Morality : It is the no of gram molecules of solute present in one liter of solution

$$\text{Molarity } M = \frac{\text{weight of the substance}}{\text{Gram molecular weight}} \times \frac{1000}{\text{volume in ml}}$$

Normality: It is defined as the no of gram equivalents of solute present in one liter of solution.

$$\text{Normality } N = \frac{\text{weight of the substance}}{\text{Gram equivalent weight}} \times \frac{1000}{\text{volume in ml}}$$

$$\text{Equivalent weight of an acid} = \frac{\text{molecular weight of an acid}}{\text{Number of replaceable } H^+ \text{ ions}}$$

$$\text{Equivalent weight of a base} = \frac{\text{molecular weight of a base}}{\text{Number of replaceable } OH^- \text{ ions}}$$

Equivalent weight of an oxidizing or reducing agent = molecular weight of a substance

No.of electrons gained or lossed

Molality: It is defined as the number of the moles of the solute present in 1 kg or 1000ml of the solvent,

$$\text{Molality , m} = \frac{\text{no. of moles of solute}}{\text{Gram molecular weight}} \times \frac{1000}{\text{volume in ml}}$$

6. What is meant by Acid-Base titration?

Answer: Titration is a process of neutralization.This method is used for determining an acid with alkali or base with an acid to produce salt and unionized water.

$$NaOH + HCl \rightarrow NaCl + H_2O$$

7. What is meant by Redox titration?

Answer: It is also known as Oxidation – Reduction reaction.Titration of a reducing agent (loosing of electrons)by an oxidizing agent (gaining of electrons) or titration of an oxidizing agent by a reducing agent.is known as Redox titration

8. What is meant by Complexometric Titration?

Answer: The technique involves titrating metal ions with a complexing agent or chelating agent (Ligand) and is commonly referred to as complex metric titration.

9. What is meant by Indicator?

Answer: It imparts colour to the colourless solution.

10.What are the Applications of Complex metric titration?

Answer:

1. Complex metric titrations have been employed with success for determination of various metals like Ca, Mg, Pb, Zn, Al, Fe, Mn, Cr etc.
2. Determination of total hardness of water by Complexometric method

Date:

Experiment -2

Determination of HCl using standard Na_2CO_3 Solution

Aim:-

a) Standardization of HCl using standard solution of 0.1N Na_2CO_3

b) Calculate the amount of HCl present in 100ml of the solution

Apparatus:-

Burette, pipette, burette stand, conical flask, wash bottle, glazed tile

Chemicals required:-

0.1N Na_2CO_3, HCl, Methyl orange indicator, distilled water.

Principle:-

HCl reacts with Na_2CO_3 the following manner

$$2HCl + Na_2CO_3 \rightarrow 2NaCl + H_2O + CO_2$$

Na_2CO_3 is a weak base; it can be estimated by titrating with a strong acid like HCl using methyl orange as indicator.

Procedure:-

a) Standardization of HCl solution:-

Burette: - The burette is cleaned first with distilled water and filled with HCl, without any air bubbles.

Conical Flask: - Conical flask is washed with distilled water and then pipette out 20ml of Na_2CO_3 Solution into the conical flask and add 2 or 3 drops of Methyl Orange indicator.

Indicator: - Methyl Orange

End Point: - pale yellow to pale pink

The conical flask is placed under the burette on a glazed tile, then the HCl present in the burette is slowly rundown by shaking the conical flask in clockwise direction continuously, the titration is continued until a

color change yellow to red which is the endpoint. The experiment is repeated till the concurrent readings are obtained.

S.No	Volume of Na_2CO_3 Solution in ml	Burette Readings		Volume of HCl rundown in ml(b-a)
		Initial (a)	Final (b)	

Calculations:-

Normality of HCl is calculated using the equation

$$N_1V_1 = N_2V_2$$

Na_2CO_3

N_1,Normality of Na_2CO_3 = 0.1 N

V_1,Volume of Na_2CO_3 solution = 20ml

HCl

N_2, Normality of HCl = ?

V_2, Volume of HCl = ml (burette readings)

Normality of HCl (N_2) = N_1V_1/ V_2

=

The amount of HCl present in 100ml of the given solution: x

$$= \frac{N_2 \text{ X Eq.Wt Of HCl(36.5) X } 100}{1000}$$

= gm

Report:-

The Normality of HCl (N_2) = N

The amount of HCl present in 100 ml of given solution = gm

Conclusion:

1. CO1 to CO6 and PO1, PO2, PO7, PO9 is attained
2. Students acquire the knowledge to calculate the strength of HCl using Normality equation.
3. Students acquire the skill to perform the Acid-Base titration in the real lab.
4. Students understand the apparatus used for a titration.

Precautions:

1. Pipptteing as to be accurate in order to avoid excess addition of the titrating agent
2. The flask containing the indicator must be shaken well while acid is added to it.
3. The acid should be added drop wise.
4. Excess of indicator should not be used.
5. The acid is added drop wise should be removed as soon as the indicator changes the colour.
6. Reading should be taken to avoid the parallax error.
7. The titration should be placed on white paper to identify properly the color change at the end point.
8. All the glass apparatus should be washed thoroughly with distilled water before use.
9. They should not be any leakage in the burette.

VIVA QUESTIONS

1. To what Category of Titration does this Experiment Belong?

Answer: It is an acid-base titration or neutralization titration in which strong acid and weak base are participating.

2. Which Indicator Is Used In this Titration?

Answer: methyl orange

3. Why did you choose Methyl Orange?

Answer: The pH range of this titration is between 3.1-4.4. Under such condition, methyl orange exhibits color change.

4. What is the Standard Solution in this Experiment?

Answer: A solution of sodium carbonate.

5. Is it a Primary Standard Solution?

Answer: yes

6. Why Should You Carry Out the Titration for 3-4 Times?

Answer: To get accurate values.

Date:

Experiment – 3

Determination of alkalinity of a sample containing Na_2CO_3 and NaOH

Aim:-

a) Standardization of H_2SO_4 standard solution of 0.1N Na_2CO_3

b) Determine the amount of alkalinity present in 100ml of the solution.

Apparatus:-

Burette, pipette, burette stand, conical flask, wash bottle, glazed tile

Chemicals required:-

0.1N H_2SO_4, 0.1N Na_2CO_3 , distilled water, Phenolphthalein indicator, methyl orange indicator

Principle:-

H_2SO_4, reacts with Na_2CO_3 the following manner

$$\mathbf{H_2SO_4 + Na_2CO_3 \rightarrow Na_2SO_4 + H_2O + CO_2}$$

Na_2CO_3 is a weak base; it can be estimated by titrating with a strong acid like H_2SO_4 using methyl Orange as indicator

Procedure:-

a) **Standardization of H_2SO_4 using standard solution of 0.1N Na_2CO_3**

Burette: - The burette is cleaned first with distilled water and filled with H_2SO_4, without any air bubbles.

Conical Flask: - Conical flask is washed with distilled water and then pipette out 20ml of Na_2CO_3 solution into the conical flask and add 2 or 3 drops of Methyl Orange indicator.

Indicator: - Methyl Orange

End Point: - yellow to red

The conical flask is placed under the burette on a glazed tile, then the H_2SO_4,present in the burette is slowly rundown by shaking the conical flask in clockwise direction continuously, the titration is continued until a color change yellow to red which is the endpoint. The experiment is repeated till the concurrent readings are obtained.

S.No	Volume of Na_2CO_3 Solution in ml	Burette Readings		Volume of H_2SO_4 rundown in ml (b-a)
		Initial (a)	Final (b)	

Calculations:-

Normality of H_2SO_4,is calculated using the equation

$$N_1V_1 = N_2V_2$$

Na_2CO_3

Normality of Na_2CO_3 N_1 = 0.1N

Volume of Na_2CO_3 solution V_1= 20ml

H_2SO_4

Normality of H_2SO_4 N_2 =

Volume of H_2SO_4 V_2 = ml (burette readings)

Normality of H_2SO_4 (N_2) = N_1V_1/ V_2

=

= N

b) **Determine the amount of alkalinity present in 100ml of the solution**

Burette: - The burette is cleaned first with distilled water and filled with H_2SO_4,

Conical Flask: - Conical flask is washed with distilled water and then pipette 20ml of sample Solution into the conical flask and add 2 or 3 drops of Phenolpthalein indicator.

Indicator: - Phenolpthalein indicator, methyl orange indicator

End Point: - pink to colourless, yellow to red

The conical flask placed under the burette on a glazed tile, then the H_2SO_4, present in the burette is slowly rundown by shaking the conical flask in clockwise direction continuously, the titration is continued Until a color change pink to colourless then 2 drops of indicator was addedand the titration is continued Until a color changes from yellow to red. The experiment is repeated till the concurrent readings are obtained.

S.No	Volume of sample Solution in ml	Burette Readings for Phenoiphthalein end point		Volume of H_2SO_4 rundown in ml (b-a)
		Initial (a)	Final (b)	

S.No	Volume of sample Solution in ml	Burette Readings for Methyl orange end point		Volume of H_2SO_4 rundown in ml (b-a)
		Initial (a)	Final (b)	

Calculations:-

Phenoiphthalein alkalinity (as $CaCO_3$)mg/l = $\frac{V_1 X \text{ Normality of } H_2SO_4 \text{ X } 1000 \text{ X } 50}{\text{Volume of sample taken}}$

=

= gm

Where V_1= volume of the acid consumed for Phenoiphthalein end point

Total alkalinity (as $CaCO_3$)mg/l = $\frac{V_2 X \text{ Normality of } H_2SO_4 \text{ X } 1000 \text{ X } 50}{\text{Volume of sample taken}}$

=

= mg/l

= gm

Where V_2= volume of the acid consumed for Methyl orange end point

Report:-

The amount of alkalinity present in a given solution = gm

Conclusion:

1. CO1 to CO6 and PO1, PO2, PO7, PO9 is attained.
2. Students understand the apparatus used for a titration.
3. Students acquire the skill to perform the Alkalinity of a sample in the real lab.
4. Students acquire the knowledge to calculate the strength of Alkalinity of a given sample.

Precautions:

1. Pipptteing as to be accurate in order to avoid excess addition of the titrating agent.
2. Excess of indicator should not be used.
3. Reading should be taken to avoid the parallax error.
4. The titration should be placed on white paper to identify properly the color change at the end point.
5. All the glass apparatus should be washed thoroughly with distilled water before use.
6. They should not be any leakage in the burette.

VIVA QUESTIONS

1. To what Category of Titration does this Experiment Belong?

Answer: It is an acid-base titration or neutralization titration in which strong acid and weak base are participating.

2. Why did you choose Methyl Orange?

Answer: The pH range of this titration is between 3.1-4.4. Under such condition, methyl orange exhibits color change

3. What is the Standard Solution in this Experiment?

Answer: A solution of sodium carbonate.

4. Is it a Primary Standard Solution?

Answer: yes

5. Why Should You Carry Out the Titration for 3-4 Times?

Answer: To get accurate values.

6. Why do you choose Phenolphthalein?

Answer: It gives pink colour in alkali medium and colorless in Acidic medium and the P^H range is 8.0-9.6.

Experiment – 4

Date:

Determination of $KMnO_4$ using standard oxalic acid solution

Aim:-

a) Determine the concentration of $KMnO_4$ using a standard 0.1N oxalic acid solution

b) Calculate the amount of $KMnO_4$ in a given 100ml of unknown solution.

Apparatus:-

Burette, pipette, burette stand, conical flask, wash bottle, glazed tile, Bunsen burner

Chemicals required:-

$KMnO_4$, 0.1 N Oxalic acid, distilled water, Dil. H_2SO_4

Principle:-

KMnO4 is an oxidizing agent in the presence of dil. H_2SO_4; $KMnO_4$ oxidizes oxalic acid to CO_2 and water. Oxalic acid acts as an oxidizing agent.

$$2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5(O)$$

$$5H_2C_2O_4 + 5(O) \rightarrow 5H_2O + 10CO_2$$

$$2KMnO_4 + 3H_2SO_4 + 5H_2C_2O_4 \rightarrow K_2SO_4 + 2MnSO_4 + 8H_2O + 10CO_2$$

Procedure:-a) Determine of concentration of $KMnO_4$ using a standard oxalic acid solution

Burette: - The burette is washed with distilled water, and then fills it with $KMnO_4$ without air bubbles.

Conical Flask: - Conical flask is washed with distilled water and then pipette out 20ml of oxalic acid solution into the conical flask.

Indicator: - $KMnO_4$ acts as a self indicator.

Endpoint: - Colorless to pale pink

To the conical flask containing oxalic acid add an equal amount of 20ml dil.H_2SO_4 and then heat it. Now the conical flak is placed under the burette on a glazed tile, then the $KMnO_4$ present in the burette is slowly rundown by shaking the conical flask in clockwise direction continuously, the titration is continued until the color changes from colorless to pale pink which is the endpoint. The experiment is repeated until concurrent readings are occurred.

S.No	Volume of oxalic acid in ml	Burette readings		Volume of $KMnO_4$ Solution in ml (b-a)
		Initial (a)	Final (b)	

Calculations:-

Normality of $KMnO_4$ is calculated using the formula $N_1V_1 = N_2V_2$

Oxalic Acid

Normality of oxalic acid solution (N_1) = 0.1 N

Volume of oxalic acid solution (V_1) = 20ml

$KMnO_4$

Normality of $KMnO_4$ solution (N_2) =

Volume of $KMnO_4$ solution (V_2) = ml

Normality of $KMnO_4$ (N_2) = N_1V_1/ V_2

=

= N

Amount of $KMnO_4$ present in 100ml of solution = N_2 X Eq.Wt of $KMnO_4$ (31.6) X 100

1000

=

= gm

Report:-

The Normality of KMnO4 (N_2)= N

The amount of $KMnO_4$ in 100 ml solution = gm

Conclusion:

1. CO1 to CO6 and PO1, PO2, PO7, PO9 is attained.

2. Students acquire the knowledge to calculate the strength of $KMnO_4$ using molarity equation.

3. Students understand the purpose of addition of dil. H_2SO_4 and the purpose of heating of oxalic acid before titration.

4. Students acquire the skill to prepare standard solutions of oxalic acid

Precautions:

1. Pipptteing as to be accurate in order to avoid excess addition of the titrating agent..
2. Reading should be taken to avoid the parallax error.
3. The titration should be placed on white paper to identify properly the color change at the end point.
4. All the glass apparatus should be washed thoroughly with distilled water before use.
5. They should not be any leakage in the burette.

VIVA QUESTIONS

1. What Is Oxidation or reducing agent?

Answer: Gain of oxygen or loss of electrons.

2. What is an Oxidizing Agent or reduction?

Answer: Gain of electrons or loss of oxygen.

3. Name some common Oxidizing Agents?

Answer: $KMnO_4$ and $K_2Cr_2O_7$, $KMnO_4$.

4. Which Compound is Oxidized in this Experiment?

Answer: oxalic acid is oxidized to co_2

5. To what Compound, $Kmno_4$ is Reduced?

Answer: $MnSO_4$

6. How can you say that $Kmno_4$ is reduced?

Answer: The oxidation state of Mn in $KMnO_4$ is +7 while in $MnSO_4$ is +2. So, Mn has gained five electrons in the acid medium. Thus Mn got reduced.

7. Which Indicator is used in this Titration?

Answer: No external indicator is used in this reaction. Color changes of $KMnO_4$ itself indicates the end point.

8. Why it is called as Redox Titration?

Answer: Because, both oxidation and reduction takes place in this reaction.

9. Why do we Heat the Contents in this Reaction?

Answer: As the reaction between $KMnO_4$ and oxalic acid is slow, to ensure the completion of the reaction, the contents are heated.

10. What happens when heated to Higher Temperatures?

Answer: At higher temperatures, oxalic acid decomposes to CO_2

11. Why do you have to use only H_2SO_4 and why not HCl Or HNO_3 ?

Answer: we cannot use HCL. Because, $KMnO_4$ can oxidize HCl to Cl_2 as per the following equation.

$$16HCL+2KMnO_4 \rightarrow 2MnCL_2 + 2KCL + 5CL_2 + 8H_2O$$

Next, we cannot use HNO_3 as it is also an oxidizing agent. In the presence of H_2SO_4 only $KMnO_4$ reduced to MnO_2.

Experiment – 5

Date:

Determination of Ferrous iron using standard $K_2Cr_2O_7$ solution

Aim:-

a) Standardization of $K_2Cr_2O_7$ solution using standard 0.1 N Mohr's salt solution.

b) Calculate the amount of ferrous iron present in 100ml of the solution.

Apparatus:-

Burette, pipette, burette stand, conical flask, wash bottle, glazed tile

Chemicals required:-

0.1N Mohr's salt solution, $K_2Cr_2O_7$ solution, Acid mixture, Barium diphenylamine Sulphonate Indicator (BDAS)

Principle:-

Mohr's salt being a reducing agent is estimated by titration with an oxidizing agent like $K_2Cr_2O_7$ in the presence of sulphuric acid and orthophosphoric acid mixture using BDAS as a redox indicator. BDAS indicator must be performed in the presence of phosphoric acid because the efficiency of indicator increased and also lowers the oxidation potential of the solution only under this conditions only the indicator change color.

$$K_2Cr_2O_7 + 4H_2SO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3(O)$$

$$3[2FeSO_4 + H_2SO_4 + (O) \rightarrow Fe_2(SO_4)_3 + H_2O]$$

$$K_2Cr_2O_7 + 6FeSO_4 + 7H_2SO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 3Fe_2(SO_4)_3 + 7H_2O$$

Procedure:-

a) Standardization of potassium dichromate solution:-

Burette: - The burette is washed with distilled water, and then fills it with $K_2Cr_2O_7$ without air bubbles.

Conical Flask: - Conical flask is washed with distilled water and then pipette out 20ml of standard Mohr's salt solution, add 10ml of acid mixture and add 4 to 5 drops of BDAS indicator into the conical flask.

Indicator: BDAS indicator

Endpoint: - Colorless to violet color.

To the conical flask containing Mohr's salt, acid mixture, BDAS indicator is placed under the burette on a glazed tile, then the $K_2Cr_2O_7$ present in the burette is slowly rundown by shaking the conical flask in Clockwise direction continuously, the titration is continued until the color changes from colorless to violet color Which is the endpoint. The experiment is repeated until concurrent readings are occurred.

S.No	Volume of standard Mohr's salt solution taken	Burette Readings		Volume of $K_2Cr_2O_7$ rundown
		Initial	Final	

Calculations:-

Normality of $K_2Cr_2O_7$ solution can be calculated from the equation, $N_1V_1 = N_2V_2$

Mohr's salt

Normality of standard Mohr's salt solution $N_1 = 0.1N$

Volume of Mohr's salt solution $V_1 = 20ml$

$K_2Cr_2O_7$

Normality of $K_2Cr_2O_7$ solution $N_2 =$ N

Volume of $K_2Cr_2O_7$ solution $V_2 =$ ml

Normality of $K_2Cr_2O_7$ solution $N_2 = N_1V_1/ V_2$

=

= N

b) Estimation of given Ferrous iron solution in 100 ml volumetric flask:-

The Mohr's salt solution given in the 100ml volumetric flask is dilute up to the mark and mix well to make the uniform solution.

Burette: - The burette is washed with distilled water, and then fills it with $K_2Cr_2O_7$ without air bubbles.

Conical Flask: - Conical flask is washed with distilled water and then pipette out 20ml of Mohr'ssalt solution from the 100ml volumetric flask, add 10ml of acid mixture and add 4 to 5 drops of BDAS indicator into the conical flask.

Indicator BDAS **Endpoint: -** colorless to violet colour.

To the conical flask containing Mohr's salt and other solutions is placed under the burette on a glazed tile, then the $K_2Cr_2O_7$ present in the burette is slowly rundown by shaking the conical flask in Clockwise direction continuously, the titration is continued until the color changes from colorless to violet which is the endpoint. The experiment is repeated until concurrent readings are occurred

S.No	Volume of unknown Mohr's salt solution taken	Burette readings		Volume of $K_2Cr_2O_7$ rundown
		Initial	final	

Calculations:-

$K_2Cr_2O_7$

Normality of $K_2Cr_2O_7$ solution $N_2 = 0.1N$

Volume of $K_2Cr_2O_7$ solution $V_2 = 20ml$

Mohr's salt

Normality of Mohr's salt solution N_3 =

Volume of Mohr's salt solution V_3 = ml

$N_3 = N_2V_2/V_3$

=

= N

The amount of Ferric iron present in the given 100ml solution is

$$= \frac{N_3 \text{ X Eq.Wt. Ferric iron (55.85) X 100}}{1000}$$

=

= gm

Result:-

The amount of ferric iron present in given 100ml solution = gm

Report:-

S.No	Given	Obtained	% of error = Given-Obtained/Given X100

Conclusion:

1. CO1 to CO6 and PO1,PO2,PO7,PO9 is attained.
2. Students acquire the knowledge to calculate the strength of Ferric iron using Normality equation.
3. Students acquire the skill to perform the redox-titration in the real lab
4. Students acquire the skill to prepare standard solutions of Mohr's salt.

Precautions:

1. Pipptteing as to be accurate in order to avoid excess addition of the titrating agent..
2. Reading should be taken to avoid the parallax error.
3. The titration should be placed on white paper to identify properly the color change at the end point.
4. All the glass apparatus should be washed thoroughly with distilled water before use.
5. They should not be any leakage in the burette.

VIVA QUESTIONS

1. Name Some Common Reducing Agents?

Answer: Metals, metal oxides,H_2S, lithium aluminum hydride ($LiAlH_4$)

2. What Is Electrode Potential?

Answer: The difference in the potential developed when a metal is dipped in its salt solution.

3. What Do You Mean By Reduction Potential?

Answer: It is the measure of the tendency of a chemical species to acquire electrons. It is measured in volts (V), or mill volts (mv). It can also be defined as the potential developed at an electrode at which the reduction takes place.

4. Which Indicator Is Used In The Estimation Of Ferric Iron Using Standard Solution Of Potassium Dichromate?

Answer: Diphenyl amine or diphenyl amine sulphonate.

5. What Is Equivalent Mass Of Potassium Dichromate?

Answer: 49

6. How can you arrive at that Value?

Answer: $cr_2o_7^{2-}{}_{(aq)} + 14H^+ + 6e^- \rightarrow 2Cr^{3+}{}_{(aq)} + 7H_2O$

It is evident from the above reaction that six electrons are involved in this process. So the equivalent mass of reducing agent is equal to molecular mass divide by number of electrons involved in the process. So Equivalent weight of potassium dichromate = 294.18/6 = 49

7. What Is The End Point Of This Titration?

Answer: the end point of this titration is marked by the appearance of blue violet color.

Date:

Experiment – 6

Determination of Copper using standard $K_2Cr_2O_7$ solution

Aim:-

a) Standardization of $Na_2S_2O_3$ (Hypo) with standard $K_2Cr_2O_7$ solution.

b) Determination of Copper (II) ion using standard $Na_2S_2O_3$ solution

Apparatus:-

Burette, pipette, burette stand, conical flask, wash bottle, glazed tile

Chemicals required:-

$Na_2S_2O_3$ (Hypo), 0.1N $K_2Cr_2O_7$ solution,Starch,KI,HCl,$NaHCO_3$, CuSO4,Ammonium solution,Acetic –acid.

Principle:-

In acid medium $K_2Cr_2O_7$ liberate Iodine from KI. The liberated Iodine can be titrated against a solution of sodium thiosulphate. Thus a solution of Hypo can be standardized by titrating the Iodine liberated from KI by a known volume of standard $K_2Cr_2O_7$ using starch as indicator.

$$K_2Cr_2O_7 + 14HCl \rightarrow 2KCl + 2CrCl_2 + 7H_2O + 3Cl_2$$

$$3Cl_2 + 6KI \rightarrow 6KCl + 3I_2$$

$$3I_2 + 6Na_2S_2O_3 \rightarrow 3Na_2S_2O_4 + 6NaI$$

Procedure:-

a**) Standardization of $Na_2S_2O_3$ (Hypo) with standard $K_2Cr_2O_7$ solution**.

Burette: - The burette is washed with distilled water, and then fills it with hypo without air bubbles.

Conical Flask: - Conical flask is washed with distilled water and then pipette out 20ml of $K_2Cr_2O_7$ solution into the conical flask, add 2 gm of KI,5ml of Con.HCl,1gm of $NaHCO_3$.The mixture is allowed to stand in the dark for 5 min. the liberated Iodine dissolves in the excess of KI present and a brown solution is formed. The solution is diluted to about 50ml.

Indicator: - Starch Indicator.

Endpoint: - blue color just turns into light green

Now the conical flak is placed under the burette on a glazed tile Hypo is rundown from the burette into the conical flask with a constant shaking until the brown color changes to greenish yellow (straw green), then 1-2 drops of starch is added. The solution turns to blue. Now Hypo is added drop by drop until the blue color just turns into light green. The titrations are repeated until consecutive readings are obtained.

S.No	Volume of $K_2Cr_2O_7$ (V_2)	Burette readings		Volume of Hypo rundown (V_1)
		Initial	Final	

Calculations:-

Normality of Hypo is calculated using the formula $N_1V_1 = N_2V_2$

Hypo

Normality of Hypo solution N_1 =0.1N

Volume of Hypo solution V_1 = ml

$K_2Cr_2O_7$

Normality of $K_2Cr_2O_7$ solution N_2 =
Volume of $K_2Cr_2O_7$ solution V_2 =20ml

Normality of Hypo $N_1 = \frac{N_2V_2}{V_1}$

=

= N

b) Determination of Copper (II) ion using standard $Na_2S_2O_3$ solution

Principle:-

Neutral or weakly acid solution of cupric salt reacts with KI forming cuprous Iodide (Cu_2I_2) and Iodine.

$$2CuSO_4 + 4KI \rightarrow 2CuI_2 + 2K_2SO_4$$

$$2CuI_2 \rightarrow Cu_2I_2 + I_2$$

$$I_2 + 2\ Na_2S_2O_3 \rightarrow Na_2S_4O_6 + 2NaI$$

Procedure:-

Burette: - The burette is washed with distilled water, and then fills it with hypo without air bubbles.

Conical Flask: - Conical flask is washed with distilled water and then pipette out 20ml of $CuSO_4$ solution

into the conical flask, . Ammonium solution is added drop by drop until a light blue permanent precipitate is formed. To this 5 ml of acetic acid is added, followed by 2 gm of KI. Iodine is liberated, which dissolves in the excess of KI and a brown solution is formed, then the mixture is allowed to stand in the dark for 5 min.

Indicator: - Starch Indicator.

Endpoint: - blue color just turns into cream or white.

Hypo is rundown from the burette into the flask until brown color becomes pale yellow. Then 2 ml of starch solution is added. The solution turns blue. Titration is continued from the burette into the flask until the solution turns to white or cream. The titrations are repeated until concurrent readings are obtained.

S.No	Volume of $CuSO_4$ in ml	Burette readings		Volume of Hypo rundown
		Initial	Final	

Calculations:-

Normality of copper sulphate solution is calculated using the formula $N_4V_4 = N_3V_3$

$CuSO_4$

Normality of $CuSO_4$ solution N_3 =

Volume of $CuSO_4$ solution V_3 = 20ml

Hypo

Normality of Hypo solution N_4 = 0.1N

Volume of Hypo solution V_4 = ml

Normality of $CuSO_4$ N_3 = $\frac{N_4V_4}{V_3}$

=

= N

Amount of $CuSO_4$ present in 100 ml of the given solution = $\frac{\text{Normality of } CuSO_4 \text{ X } 249.5 \text{ X } 100}{1000}$

=

= gm

Amount of Cu^{+2} ion present in 100 ml of the given solution = $\frac{\text{Normality of } CuSO_4 \text{ X } 63.5 \text{ X } 100}{1000}$

=

= gm

Result:-

The amount of $CuSO_4$ present in given 100ml solution = gm

The amount of Cu^{+2} ion present in given 100ml solution = gm

Report:-

S.No	Given	Obtained	% Of Error = $\frac{\text{Given-Obtained}}{\text{Given}}$ X 100

Conclusion:

1. CO1 to CO6 and PO1, PO2, PO7, PO9 is attained.
2. Students acquire the knowledge to calculate the strength of cuprous ion using Normality equation.
3. Students acquire the skill to perform the Iodometric titration in the real lab
4. Students acquire the skill to prepare standard solutions of sodiumthiosulphate.

Precautions:

1. Pipptteing as to be accurate in order to avoid excess addition of the titrating agent..
2. Reading should be taken to avoid the parallax error.
3. The titration should be placed on white paper to identify properly the color change at the end point.
4. All the glass apparatus should be washed thoroughly with distilled water before use.
   They should not be any leakage in the burette.

VIVA QUESTIONS

1. What Is Electrode Potential?

Answer: the difference in the potential developed when a metal is dipped in its salt solution.

2. What Do You Mean By Reduction Potential?

Answer: it is the measure of the tendency of a chemical species to acquire electrons. It is measured in volts (V), or mill volts (mv). It can also be defined as the potential developed at an electrode at which the reduction takes place.

3. Which Indicator is used In the Estimation Of Copper?

Answer: freshly prepared starch solution.

4. What is Starch?

Answer: starch is naturally occurring bio molecule

5. To what category of Bio Molecule does Starch Belong?

Answer: carbohydrates

6. What are the constituents of Starch?

Answer: Amylase and amylopectin.

7. What is Amylase?

Answer: Amylase is a polymer of α-D-glucose.

8. What do you mean by Standard Solution?

Answer: a solution whose concentration is known.

9. What is the Primary Standard Solution in this Titration?

Answer: a solution of potassium dichromate.

10. What is Hypo?

Answer: sodium thiosulphate is called as hypo.

11. What is the Formula of Hypo?

Answer: $Na_2S_2O_3.5H_2O$

12. What Is The End Point In The Standardization Of Hypo?

Answer: Appearance of violet blue color.

13. Which Oxidizing Agents are used in this Titration?

Answer: potassium dichromate and cupric salts.

14. Why should be the Mixture be kept in Dark?

Answer: in order to prevent the unwanted side reaction.

15. Why should we add Sodium Carbonate to the Reaction mixture in the Conical Flask?

Answer: in order to flush the oxygen from the conical flask thereby preventing the reaction mixture from exposing to the oxygen.

16. Why should you add Ammonium Hydroxide to Copper Solution?

Answer: to neutralize any acid present.

17. Why Should You Add Minimum Quantity Of Acetic Acid To The Conical Flask Containing Copper Solution?

Answer: actually we add ammonium hydroxide to neutralize any acid present. In the process we may add little excess ammonium hydroxide. So to neutralize excess ammonium hydroxide minimum quantity of acetic acid should be added.

Experiment – 7 **Date:**

Determination of temporary and permanent hardness of water using standard EDTA solution

Aim:-

a) Standardization of the EDTA solution using standard Zinc sulphate solution.

b) Determination of the temporary and permanent hardness of given water sample.

Apparatus:-

Burette, pipette, burette stand, glazed tile, conical flask,

Chemicals required:-

0.1M Standard zinc sulphate solution, EDTA solution, Ammonia buffer, Erichrome Black T indicator.

Principle:-

Metal ions form a complex with EDTA according to the equation

M^{2+} + EBT → M-EBT complex

Wine -red

M-EBT complex + EDTA → M-EDTA complex + EBT

STABLE complex-blue

The completion of the reaction between M^{+} and EDTA is detected by the use of metal ion indicator namely Eriochrome Black T. Initially, when the P^{H} of the medium maintained at P^{H}=7to11 the metal ion combine with indicator to form metal indicator complex which appears as a wine red color. Near the end point, EDTA breaks the metal indicator complexation, resulting in the formation of metal-EDTA complex. Hence at the end point, the liberated free indicator yields a blue color to the solution. Thus the end point is a fine, sharp change from wine red to blue color.

Procedure:-

a) **Standardize the EDTA solution using standard zinc sulphate solution**.

Burette: - The burette is washed with distilled water, and then fills it with EDTA without air bubbles.

Conical Flask: - Conical flask is washed with distilled water and then pipette out 20ml of Zinc sulphate solution into the conical flask and then add 3.0 ml of ammonia buffer solution, 3-4 drops of Erichrome Black T indicator.

Indicator: - Erichrome Black T indicator.

Endpoint: - wine red to blue color.

To the conical flask containing Zinc sulphate, ammonia buffer solution, Erichrome Black T indicator is placed under the burette on a glazed tile, then the EDTA present in the burette is slowly rundown by shaking the conical flask in clockwise direction continuously, the titration is continued until the color changes from wine red to blue color which is the endpoint. The experiment is repeated until concurrent readings are obtained.

S.no	Volume of Zinc Sulphate solution (V_1) in ml	Burette readings		Volume of EDTA solution rundown (V_2) in ml
		Initial	Final	
1.				
2.				

Calculations:-

The Molarity of EDTA solution can be calculated from the formula $V_1M_1 = V_2M_2$

Zinc sulphate

Volume of zinc sulphate solution V_1 = 20.0 ml

Molarity of zinc sulphate solution M_1 =0.1N

EDTA

Volume of EDTA solution V_2 = ml

Molarity of EDTA solution M_2 = M

Molarity of EDTA $M_2 = \frac{M_1V_1}{V_2}$

= M

b) Determination of the Total hardness of given water sample.

Burette: - The burette is washed with distilled water, and then fills it with EDTA without air bubbles.

Conical Flask: - Conical flask is washed with distilled water and then pipette out 20ml of Zinc sulphate solution into the conical flask and then add 3.0 ml of ammonia buffer solution, 3-4 drops of Erichrome Black T indicator.

Indicator: - Erichrome Black T indicator.

Endpoint: - wine red to blue color.

To the conical flask containing water sample, ammonia buffer solution, Erichrome Black T indicatoris placed under the burette on a glazed tile, then the EDTA present in the burette is slowly rundown by shaking the conical flask in clockwise direction continuously, the titration is continued until the color changes from wine red to blue color which is the endpoint. The experiment is repeated until concurrent readings are obtained.

S.No	Volume of Water Sample (V_1 ml)	Burette readings		Volume of EDTA solution rundown (V_2) in ml
		Initial	Final	

Calculation:

The concentration of water solution can be calculated from the equation, $V_1 M_1 = V2 M_2$

Water sample
V_3 = Volume of Water sample (20.0ml)
M_3 = Molarity of water sample (?)

EDTA
V_2 = Volume of EDTA Solution
M_2 = Molarity of EDTA Solution
Therefore $M_3 = \frac{V_2 M_2}{V_3} =$ M

Hardness of water is expressed in terms of equivalent of calcium carbonate as ppm (parts per million)

Total Hardness of Water = M_3 X 100g/lit

= M_3 X 100 X 1000 mg/lit or ppm

= ppm

C) Determination of the temporary and permanent hardness of given water sample.

Burette: - The burette is washed with distilled water, and then fills it with EDTA without air bubbles.

Conical Flask: - Take 100ml of water sample in large beaker boil it till the volume is reduced to about 20ml (when all the bicarbonates are decomposed to insoluble to $CaCO_3$+$Mg(OH)_2$) .Filter ,wash the precipitate with distilled water ,collecting filtrate and washing in 100ml measuring flask finally make up the volume to 100ml with distilled water. Then pipette out boiled water sample into the conical flask and then add 3.0 ml of ammonia buffer solution, 3-4 drops of Erichrome Black T indicator.

Indicator: - Erichrome Black T indicator.

Endpoint: - wine red to blue color.

To the conical flask containing water sample, ammonia buffer solution, Erichrome Black T indicatoris placed under the burette on a glazed tile, then the EDTA present in the burette is slowly rundown by shaking the conical flask in clockwise direction continuously, the titration is continued until the color changes from wine red to blue color which is the endpoint. The experiment is repeated until concurrent readings are obtained.

S.No	Volume of Water Sample (V_1 ml)	Burette readings		Volume of EDTA solution rundown (V_2) in ml
		Initial	Final	

Calculation:

The concentration of water solution can be calculated from the equation, $V_1 M_1 = V2 M_2$

Water sample

Volume of Water sample V_3 = (20.0ml)

Molarity of water sample M_3 = (?)

EDTA

Volume of EDTA Solution V_4 =

Molarity of EDTA Solution M_4 =

Therefore $M_3 = \frac{M_4 V_4}{V_3}$ = M

Hardness of water is expressed in terms of equivalent of calcium carbonate as ppm (parts per million)

Permanent Hardness of Water = M_3 X 100g/lit

= M_3 X 100 X 1000 mg/lit or ppm

= ppm

Temporary hardness = Total hardness-Permanent hardness

= ppm

Result:

Temporary hardness present in the given water sample = ppm

Permanent hardness present in the given water sample = ppm

Conclusion:

1. CO1 to CO6 and PO1, PO2, PO7, PO9 is attained.

2. Students acquire the knowledge to calculate the strength of Temporary and Permanent hardness of a given water sample.

3. Students acquire the skill to perform the Total Hardness in the real lab

4. Students acquire the skill to perform the quality of raw water in the real lab

Precautions:

1. Pipptteing as to be accurate in order to avoid excess addition of the titrating agent..
2. Reading should be taken to avoid the parallax error.
3. The titration should be placed on white paper to identify properly the color change at the end point.
4. All the glass apparatus should be washed thoroughly with distilled water before use.
5. They should not be any leakage in the burette.

VIVA QUESTIONS

1. (A) What Is Hardness?

(B) Why It Is Due To?

(C) Why Soaps Are Not Affective Hard Water?

Answer: (a) sample of water that does not give lather with soap is hard water.

(b) It is due to the presence of dissolved salts of calcium and magnesium and other heavy metals

(c) Soaps form insoluble precipitates with calcium and magnesium salts.

2. What Is the Difference between Temporary and Permanent Hardness?

Answer: temporary hardness is due to the bicarbonates of calcium, magnesium, while the permanent hardness is due to the chlorides, sulphates and carbonates of calcium, magnesium and other heavy metals.

3. What is the full form of EDTA & EBT Indicators?

Answer: Ethylene diamine tetra acetic acid & Eriochrome Black-T.

4. What is the Principle in EDTA titrations?

Answer: Ca^{+2} or Mg^{+2} can form complex with EDTA (also with EBT).

5. Give The Structures Of EDTA & EBT?

Answer: refer theory and principle.

6. Why EBT Indicator should be used in Basic Medium?

Answer: it can exhibit color changes in basic medium.

7. Which Buffer is used in this Titration?

Answer: Ammonium buffer.

8. What is the Composition of the Buffer?

Answer: a mixture of ammonium chloride and ammonium hydroxide.

9. What is the Composition Of The Buffer?

Answer: 67.5gm of ammonium chloride and 570ml of ammonium hydroxide.

Date:

Experiment – 8

Determination of Copper using standard E.D.T.A solution

Aim:

a) Standardization of the EDTA solution using standard Zinc sulphate solution.

b) Determine the amount of copper present in a given solution by using E.D.T.A

Apparatus:-

Burette, pipette, burette stand, glazed tile, conical flask,

Chemicals required:-

0.1M zinc sulphate solution, EDTA solution, Ammonia buffer, Erichrome Black T indicator, Copper sulphate pentahydrate, distilled water, fast sulfan balck F indicator

Principle:-

Metal ions form a complex with EDTA according to the equation

M^{2+} + EBT → M-EBT complex

Wine -red

M-EBT complex + EDTA → M-EDTA complex + EBT

STABLE complex- blue

The completion of the reaction between M^{+} and EDTA is detected by the use of metal ion indicator namely Eriochrome Black T. Initially, when the P^{H} of the medium maintained at P^{H}=7to11 the metal ion combine with indicator to form metal indicator complex which appears as a wine red color. Near the end point, EDTA breaks the metal indicator complexation, resulting in the formation of metal-EDTA complex. Hence at the end point, the liberated free indicator yields a blue color to the solution. Thus the end point is a fine, sharp change from wine red to blue color.

Procedure:-

a) Standardize the EDTA solution using standard zinc sulphate solution.

Burette: - The burette is washed with distilled water, and then fills it with EDTA without air bubbles.

Conical Flask: - Conical flask is washed with distilled water and then pipette out 20ml of Zinc sulphate solution into the conical flask and then add 3.0 ml of ammonia buffer solution, 3-4 drops of Erichrome Black T indicator.

Indicator: - Erichrome Black T indicator.

Endpoint: - wine red to blue color.

To the conical flask containing Zinc sulphate, ammonia buffer solution, Erichrome Black T indicatoris placed under the burette on a glazed tile, then the EDTA present in the burette is slowly rundown by shaking the conical flask in clockwise direction continuously, the titration is continued until the color changes from wine red to blue color which is the endpoint. The experiment is repeated until concurrent readings are obtained.

S.No	Volume of Zinc Sulphate solution (V_1) in ml	Burette readings		Volume of EDTA solution rundown (V_2) in ml
		Initial	Final	

Calculations:-

The Molarity of EDTA solution can be calculated from the formula $M_1V_1 = M_2V_2$

zinc sulphate

volume of zinc sulphate solution V_1 = 20.0 ml

Molarity of zinc sulphate solution M_1 =

EDTA

volume of EDTA solution V_2 =

Molarity of EDTA solution M_2 =

Molarity of EDTA $M_2 = \frac{M_1V_1}{V_2}$

= M

b) Estimate the amount of copper present in a given solution using fast sulfan black -F indicator

The copper ion solution given in the 100ml volumetric flask is dilute up to the mark and mix well to make the uniform solution

Burette: - The burette is washed with distilled water, and then fills it with EDTA without air bubbles.

Conical Flask: - Conical flask is washed with distilled water and then pipette out 20ml of copper ion solution into the conical flask, add 20ml of distilled water, 5ml of concentrated ammonia solution and add 5-6 drops of the indicator.

Indicator: - fast sulfan black -F

Endpoint: - blue to a dark green

To the conical flask containing copper ion solution,distilled water, ammonia solution, fast sulfan black -F indicator is placed under the burette on a glazed tile, then the EDTA present in the burette is slowly rundown by shaking the conical flask in clockwise direction continuously, the titration is continued until the color changes from blue to a dark green color.

S.No	Volume of Copper Sulphate solution (V_3) in ml	Burette Readings		Volume of EDTA solution rundown (V_2) in ml
		Initial	Final	

Calculations:-

The copper ion can be determined by using the following formula: $M_2V_2 = M_3V_3$

Copper sulphate

Volume of copper sulphate solution V_3 = 20 ml

Molarity of copper sulphate solution M_3 = ?

EDTA

volume of EDTA solution V_2 = ml

Molarity of EDTA solution M_2 = M

The Molarity of copper sulphate solution, $M_3 = \frac{M_2V_2}{V_3}$

= M

The amount of copper ion present in a given unknown solution

$$= \frac{M_3 \text{ X gram atomic weight of } Cu^{+2} (63.54) \text{ X } 100}{1000}$$

= gm

Result:- The amount of copper ion present in given 100ml solution = 0.6354 gm.

Report:

S.No	Given	Obtained	% of error $\frac{\text{Given-Obtained}}{\text{Given}}$ X 100

Conclusion:

1. CO1 to CO6 and PO1,PO2,PO7,PO9 is attained.
2. Students understand the apparatus used for a titration.
3. Students acquire the skill to perform the Complexometric-titration in the real lab.
4. Students acquire the skill to determine the concentration of metals in the real lab.

Precautions:

1. Pipptteing as to be accurate in order to avoid excess addition of the titrating agent..
2. Reading should be taken to avoid the parallax error.
3. The titration should be placed on white paper to identify properly the color change at the end point.
4. All the glass apparatus should be washed thoroughly with distilled water before use.
5. They should not be any leakage in the burette.

VIVA QUESTIONS

1. Which Indicator Is Used In the Titration of EDTA V/S Copper Solution

Answer: Fast sulphone Black F

2. What Do You Mean By Chelating Effect?

Answer: the increased affinity of poly dentate ligands towards a particular metal when compared to mono dentate ligand is called chelating effect.

3. How The End Point Is Indicated In The Estimation Of Copper?

Answer: conversion of purple color to green

4. How the End Point Is Indicated In the Standardization of EDTA?

Answer: conversion of Wine red color to blue.

5. What Is a Ligand?

Answer: An electron rich species capable of donating electron pair to the vacant d-orbitals of metal atoms.

6. What Is a Polydentate Ligand?

Answer: a ligand with many bonding sites.

7. What Is A Chelate Complex?

Answer: ring complex formed between the metal and polydentate ligand.

Date:

Experiment – 9

Determination of Iron by colorimetric method using thiocynate as reagent

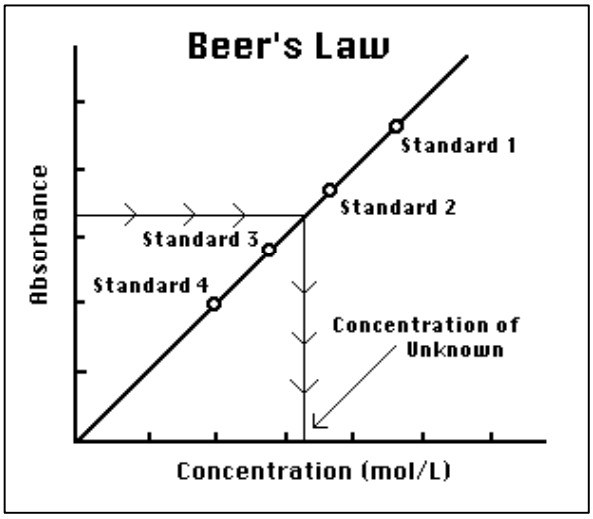
Aim: To estimate the Iron(III) using colorimeter

Apparatus: cuvettes, Spectrophotometer, beaker, volumetric flask, burette,

Chemicals: Distilled water, 0.1N Potassium thiocyanate, 0.1NFerric Ammonium sulphate.

PRINCIPLE

White light from a tungsten lamp passes through a condenser lens to give a parallel beam which falls on the filter that is positioned to select radiation of specific wavelength to impinge on a glass cuvette containing the solution. As the light is passing through the solution, some part of it is absorbed by the sample component, while the part that is not absorbed is transmitted, and detected by a photo electric cell (detector). In order to measure the absorbance of a solution, the meter reading is first adjusted to 100% transmittance (zero absorbance) with a blank solution. The sample is then inserted in place of the blank and the absorbance is read directly. The concentration corresponding to the absorbance of the sample is then obtained from the standard or calibration graph.

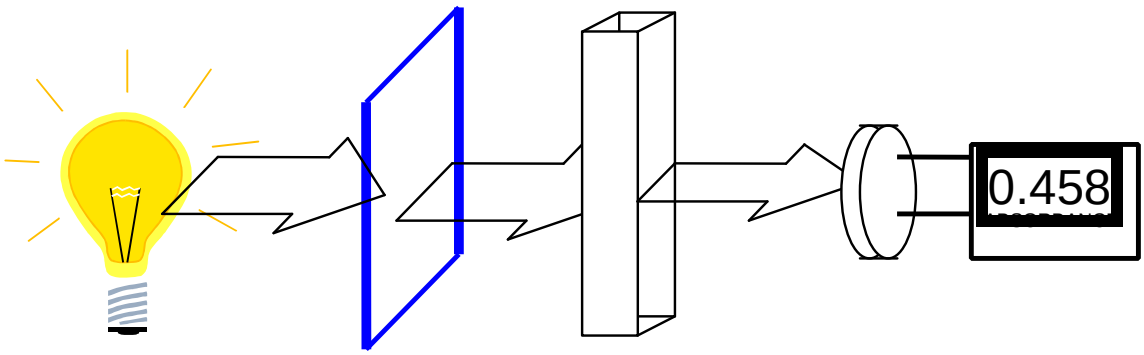


The amount of light absorbed is **directly proportional to the concentration of the ions** in the solution as defined by Beer in 1852, Beer discovered that the transmittance of light decreases exponentially in proportion to the concentration of the species absorbing the light. Therefore, **Beer's Law** can be stated most simply as:

$$A = k \times C$$

A is the Absorbance of light by the sample.

k is the constant depends on the path length through the sample (diameter of the container),



LIGHT SOURCE. MONOCHROMATOR CUVETTE DETECTOR. DISPLAY.

the wavelength of the light used, and the type of absorbing sample.

C is the concentration of the sample

1. LIGHT SOURCE: white light of many colors.

2 MONOCHROMATOR or FILTER: The filter to remove unwanted colors.

3. CUVETTE: Cuvette consists of sample solution which absorbs some colors depending on concentration.

4. DETECTOR: Responds to light falling on it and gives an electrical output corresponding to the amount of light.

5. DISPLAY: Amount of absorbance can be displayed.

Wavelength, nm	Color (light absorbed)	Complementary color (light transmitted)
380-435	violet	yellow-green
435-480	blue	yellow
480-490	green-blue	orange
490-500	blue-green	red
500-560	green	purple
560-580	yellow-green	violet
580-595	yellow	blue
595-610	orange	green-blue
610-750	red	blue-green

Principle:

Thiocyanate ions react with iron(III) ions in solution to form an intense red coloured complex ion:

$Fe^{3+}(aq) + SCN^{-}(aq) \rightarrow [FeSCN]^{2+}(aq)$

[Fe(H2O)6]3+(aq) + SCN−(aq) → [Fe(H2O)5SCN]2+(aq) + H2O(l)

Method:

1. Fill three burettes, one with the potassium thiocyanate solution containing 250 ppm thiocyanate, one with deionised water and one with iron(III) chloride solution.
2. Add 0.0, 2.0, 4.0, 6.0, 8.0 and 10.0 cm3 of the potassium thiocyanate solution to six 100 cm3 volumetric flasks A-F. Add deionised water to bring the volume in each flask to about 80 cm3.
3. To each flask add 10 cm3 iron(III) chloride solution and then add deionised water to bring volume to 100 cm3. Mix the solutions thoroughly.
4. Measure the absorbance of each solution using a colorimeter.
5. Plot a graph of absorbance (*y* axis) against thiocyanate concentration (in ppm thiocyanate) (*x* axis) for the six solutions.
6. Add 10 cm3 of the solution of unknown thiocyanate concentration to a 100 cm3 volumetric flask and add deionised water to bring the volume in the flask to about 80 cm3.
7. Add 10 cm3 iron(III) chloride solution to the flask and then add deionised water to bring volume to 100 cm3 and mix the solution thoroughly.
8. Measure the absorbance of the solution using a colorimeter at 480nm
9. Use the graph to find the concentration of thiocyanate ions as ppm thiocyanate in the unknown solution.

test tube No	**0.05 M copper concentration**	**Vol 0.05M standard Copper (mL)**	**Volume water (mL)**	**Vol 1M ammonia solution (mL)**	**Vol Unknown solution (mL)**	**Total Volume (mL)**	**Concentration In moles/lit Vol.of cu^{+2} X 0.05M / 30**
1	**0 (Blank)**	0	25 5	5		3 0	
2	**0.05**	5	20	5		3 0	

3	**0.05**	10	15	5		30	
4	**0.05**	15	10	5		30	
5	**0.05**	20	5	5		30	
6	**0.05**	25	0	5		30	
7	**Unknown Solution**	0	0	5	25	30	

REPORT

Sample	Absorbance	Concentration

Conclusion:

1. CO1 to CO6 and PO1,PO2,PO7,PO9 is attained.
2. The students would be aware of instrumental methods of chemical analysis.
3. Students acquire the skill to determine the concentration of Fe^{+2} ions in the colored solution using. **Beer's Law** in the real lab.

Precautions:

1. Gentle handling of the Instrument
2. Clean the cuvettes with distilled water.

VIVA QUESTIONS

1. What do you mean by Colorimetry?

Answer: as the temperature increases, the EMF of the solution also increases.

2. What is a colorimeter?

Answer: Colorimeter is a device useful to determine the concentration of a solution by measuring its absorbance at a specific wavelength of light.

3. What is Beer`s Law?

Answer: According to Beer`s law the absorbance of transmitted light is directly proportional to the concentration of a solution. The plot absorbance versus concentration, the resulting graphy yields a straight line passing through origin.

4. What do you mean by colorimetry?

Answer: as the temperature increases, the EMF of the solution also increases.

Date:

EXPERIMENT: 10

Determination of p^H of the given unknown solution using p^H meter

Aim:

a) Determine the pH of a given unknown solution by using pH meter.

b) Determine the pH of a given unknown solution by using pH paper.

Apparatus:

1. A pH meter with glass and reference electrode
2. Beaker.
3. pH papers.

Chemicals Required:

pH tablets like 7, 9,4.

Principle:

pH may be measured accurately using a pH meter. The degree of acidity or alkalinity of a solution is expressed by the pH scale. The concept of pH was put forward by **Sorenson** in 1909. It is expressed as the negative logarithm of the hydrogen ion concentration in moles/liter at a given temperature. The pH scale extends from 0 (very acidic) to 14 (very alkaline) with 7 corresponding to exact neutrality at 25°C. pH is used in the calculation of carbonate, bicarbonate and CO_2, corrosion, stability index etc.

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

Example: The pH of a given water sample measures its hydrogen ion concentration and indicates whether the sample is acidic, neutral or basic.

Procedure:

Using pH meter:

1. Switch on the instrument and allow the instrument to warm up for about 10 minutes.
2. Prepare any two buffer solutions (take a clean beaker and place buffer tablet or powder in it.now add 100ml distilled water and cover to avoid CO_2 mixing).
3. Adjust the temperature to room temperature.

4. Dip the electrode in the buffer solution of known pH and wait for reading to be stabilized. Adjust the SLOPE knob so that display reads the pH of the buffer.
5. In this way calibrate the electrodes with two standard buffer solutions of pH 4.0, 7.0 and 9.0.
6. Now wash the electrodes with distilled water and wipe with tissue paper
7. Then Immerse the electrodes in the beaker containing unknown solution (whose pH is to be determined) and wait up to one minute for steady reading.
8. Note down the pH reading.
9. Now wash the electrodes with distilled water and wipe with tissue paper.
10. Immerse the electrode in the beaker containing distilled water.
11. Switch off the instrument.

Using pH paper

1. Dip the paper in the sample.

2. Compare the color with that of the color given on the wrapper of the pH paper..

Results

Sample	pH meter	pH paper

Conclusion:

1. CO1 to CO6 and PO1, PO2, PO7, PO9 is attained.
2. The students would be aware of instrumental methods of chemical analysis.
3. Students acquire the skill to determine the concentration of H^+ ions for a given water sample using. **p^H Meter** in the real lab.
4. The students would be aware of problems associated with acidic water.

Precautions:

1. Gentle handling of the Instrument
2. The electrode is always dipped in distilled water when not in use.
3. The standard buffer solutions are kept in the seal tight bottles and avoid contamination.

VIVA QUESTIONS

1.What is the reason for acidity in natural waters?

Answer: due to the presence of dissolved non metal oxides in the atmosphere.

2.What Is the Concept of $Caco_3$ Equivalent For Expressing Acidity?

Answer: irrespective of acid present, the acidity can be expressed uniformly.

3.What Is the Range Of Acidity In Lake, River Waters?

Answer: around 7.

4.Give two Examples Strong And Weak Acids?

Answer: strong acids: HCL,H_2SO_4

Weak acids: CH_3COOH,$C_2H_2O_4$

5.What Is the pH Range of the Acidic Solutions?

Answer: 0 to 6.9

6.What Is the pH Range of Basic Solutions?

Answer : 7.1-14.

7.Which Ph has a Higher Numerical Value, Acids Or Bases?

Answer: bases (14)

8. How The Ph Varies With H^+ Ion Concentration?

Answer: if H^+ Ion concentration increases, the pH value also increases.

9. Though Water Contains H^+ and OH^- Ions Why is it Neutral?

Answer: Because there are equal number of H^+ and OH^- ions in water . As they neutralize, the pH will be 7 i.e., neutral.

10. What Is The pH of Pure Water?

Answer: 7

11. What Is the Importance of pH In Biological Systems?

Answer: Enzymatic reactions take place only at a particular pH range.

12. How Would Lakes and Rivers Affected By the Changes in Ph?

Answer: change in pH drastically affects the aquatic life.

Conductometric titrations

Introduction:

Basic principle is the changes in conductivity of a solution when no.of ions that are the responsible for the conduction of electricity is changing. Conductometer is the measure of conductivity of a solution due to mobility of cations and anions towards Respective electrodes. Conductivity is inversely proportional to resistance(R) of a solution.

C=1/R.mho called conductivity.

Conductivity of the solution based on

1. The number of free ions,
2. Charge on the free ions
3. Mobility of the ions in the solution.

The electrical conductivity is entirely due to the movement of ions. During the titration, the number of free ions in the solution changes and identity of the ions also changes. Due to this, conductance of the solution also change.

Conductometric titration is a method of volumetric analysis based on the change in conductance of the solution, at the equivalence point (or end point during titration).when the mobility of ions decreases the conductivity also decreases. A titration in which electrical conductance of a solution is measured during the course of titration known as conductometric titration.

Advantages over volumetric titrations:

1. More accurate results are obtained because the end point is determined graphically.
2. Titration can be carried out along with volored solution.
3. These are successful even in weak acids and strong bases.
4. This method which gives accurate results even with weak acids against weak bases.

Experiment – 11

Date:

Determination of Conductometric titration between strong acid and strong base

Aim:-

To determine the concentration of strong acid using strong base

Apparatus and Chemicals required:-

Analytical balance, weight box, 100ml volumetric flask, funnel, pipette, wash bottle, oxalic acid Sodium hydroxide, HCl distilled water.

Theory:-

Conductivity of the solution based on

1. The number of free ions,
2. Charge on the free ions
3. Mobility of the ions in the solution.

The electrical conductivity is entirely due to the movement of ions.At first the conductivity of acid solution is high but on titrating strong acid like HCl with strong base like NaOH ,the mobility of H^+ ions decreases, conductivity decreases and finally H^+ ion reacts with OH^- ion forms water,which is a nonconducting molecule ,this point can be called as equivalence point (the point at which neither acid nor base).on further addition of NaOH the OH^- concentration increases, there is a mobility of ions in the solution the conductivity increases. The conductance plotted against the volume of solution and the end point is determined graphically in case of HCl and NaOH the neutralization of HCl is

$HCl + NaOH \rightarrow NaCl + H_2O$

In this reaction the fast moving H^+ Ions are replaced by slow moving Na+ ions. Therefore conductance between decreases sharply. All the HCl is neutralized. The conductance increases sharply due to OH^- ions.

Part-1:

Procedure:-

Standardization of NaOH solution using 0.1N Oxalic acid:-

Burette: - The burette is cleaned first with distilled water and filled with NaOH, without any air bubbles.

Conical Flask: - Conical flask is washed with distilled water and then pipette out 10ml of oxalic acid solution into the conical flask.

Indicator: - 2 or 3 drops of Phenolphthalein indicator.

End Point: - colorless to pale pink

The conical flask is placed under the burette on a glazed tile, then the NaOH present in the burette is slowly rundown by shaking the conical flask in clockwise direction continuously, the titration is continued until a color change colorless to pale pink which is the endpoint. The experiment is repeated till the concurrent Readings are obtained.

S.No	Volume of oxalic acid Solution in ml	Burette Readings		Volume of NaOH rundown in ml (b-a)
		Initial (a)	Final (b)	

Calculations:-

Concentration of NaOH Solution is calculated using the equation

$$N_1V_1 = N_2V_2$$

Oxalic acid

Concentration of Oxalic acid $N_1 = 0.1N$

Volume of Oxalic acid solution $V_1 = 10ml$

NaOH

Concentration of NaOH $N_2 =$

Volume of NaOH $V_2 =$ ml (burette readings)

Concentration of NaOH $(N_2) = N_1V_1 / V_2$

=

= N

Part-2:

Determination of strength of HCl using standard solution of NaOH

Procedure:-

Burette: - The burette is cleaned first with distilled water and filled with NaOH, without any air bubbles.

Beaker- Beaker is washed with distilled water and then add 5 ml of HCl, 5ml of distilled water.

The beaker containing solutions is titrated against NaOH solution on conductometer observe the Conductance values for every 0.5 ml addition of 0.1N NaOH solution

Draw the graph, plotting on x-axis –volume of NaOH and on y-axis – conductance. Using the Equivalence point , calculate the Concentration of HCl.

S.No	Volume of sodium hydroxide (ml)	Conductance Ω-1	S.No	Volume of sodium hydroxide (ml)	Conductance Ω-1
1.	0.5		**13.**	6.5	
2.	1.0		**14.**	7.0	
3.	1.5		**15.**	7.5	
4.	2.0		**16.**	8.0	
5.	2.5		**17.**	8.5	
6.	3.0		**18.**	9.0	
7.	3.5		**19.**	9.5	
8.	4.0		**20.**	10.0	
9.	4.5		**21.**	10.5	
10.	5.0		**22.**	11.0	
11.	5.5		**23.**	11.5	
12.	6.0		**24.**	12.0	

Calculations:-

HCl

Concentration of HCl N_3 = N

Volume of HCl solution V_3 = 10 ml

NaOH

Concentration of NaOH N_2 = N

Volume of NaOH V_2 = ml

Concentration of HCl solution (N_3) = N_2V_2/ V_3

=

= N

The strength of HCl solution = N_3 X Equivalent weight of HCL(36.5 g/lit)

=

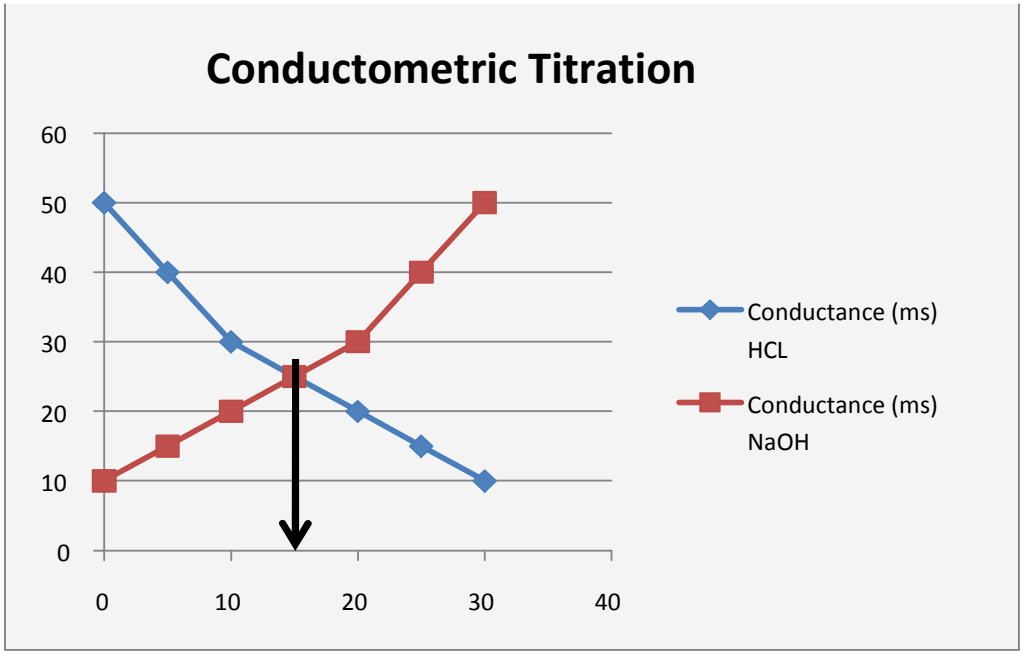
= gm

Report:-

The Concentration of HCl (N_3) = N

The strength of HCL solution = gm

Strong Acid – Strong Base



Conclusion:

1. CO1 to CO6 and PO1, PO2, PO7, PO9 is attained.
2. The students would be aware of instrumental methods of chemical analysis.
3. Students acquire the skill to determine the conductivity of acid base using conductivity meter in the real lab.

Precautions:

1. Gentle handling of the Instrument
2. The electrode is always dipped in distilled water when not in use.

VIVA QUESTIONS

1. Explain Conductivity, Molar Conductivity, And Specific Conductivity & Equivalent Conductivity?

Answer: the reciprocal of resistance is called conductance or **conductivity**. **Electrical conductivity** or **specific conductance** is a measure of a material's ability to conduct an electric current. When an electrical potential difference is placed across a conductor, its movable charges flow giving rise to an electric current**.**

Molar conductivity is defined as the conductivity of an electrolyte solution divided by the molar concentration of an electrolyte, and so measures the efficiency with which a given electrolyte conducts electricity in solution. Its units are Siemens per meter per morality, or Siemens meter-squared per mole.The usual symbol is capital lambda, Λ or Λ_m.

Equivalent conductivity: Property of an electrolyte, equal to the specific conductance divided by the number of gram equivalents of solute per cubic centimeter of solvent.

2. What do you Mean by Cell Constant?

Answer: the ratio of distance between conductometric titration electrodes to area of the electrodes, measured from the determined resistance of a solution of known specific conductance.

3. What Is the Principle of this Experiment?

Answer: determination of volume of the base required conduct metrically to find the concentration of given acid sample.

4. What Could Be the Shape of the Graph?

Answer: v-shape.

5. What are the Units of Conductivity In Cgs and Si Systems?

Answer: Ohm^{-1} or mho, Siemens.

6. What Is the Equivalent Mass Of Oxalic Acid?

Answer: 63

7. How Can You Arrive at that Value /

Answer: its molecular mass is 126. Basicity is 2 So

Equivalent weight = (molecular weight) / basicity.

Equivalent weight = 126 / 2

= 63

8. When Strong Acid Combines With A Strong Base. What Type Of Reaction Occurs?

Answer: acid and base combines to form salt and water. The reaction is called as neutralization reaction.

9. Name the Apparatus used for this Method?

Answer: the conductivity meter with a conductivity cell.

10. How Conductance is related to the concentration of the Ions?

Answer: the specific conductance is proportional to the concentration of ions in it.

11. How the End Point for particular Reason Is Calculated using this Titration Method?

Answer: on plotting a graph between conductance and volume of the base, the point of intersection of the straight lines gives the end point.

12. Why Conductance Decreases On Addition Of NaOH And HCl ?

Answer: during the titration the fast moving hydrogen ions are replaced by the slow moving Sodium ions, as a result the conductance of the solution decreases.

13. What Is the Unit for Resistance?

Answer: the unit for resistance is Ohm.

14. What is the Equivalent Mass of Hcl?

Answer : Equivalent mass of HCL is 36.5

15. What is the Equivalent Mass of NaOH ?

Answer: equivalent mass of NaOH is 40.

Date:

Experiment – 12

Determination Conductometric titration between strong acid and weak base

Aim:-

To determine the concentration of weak base using strong acid

Apparatus and Chemicals required:-

Analytical balance, weight box, 100ml volumetric flask, funnel, pipette, wash bottle, oxalic acid sodium hydroxide,HCl,NH_4OH distilled water.

Theory:-

The initial conductivity is high because strong acid completely dissociate into H^+ ions and ionic conductivity is 350when ammonium hydroxide is added as titrant, the OH^- ions and H^+ Reacts to produce water and the number Of H^+ ions decreases and conductivity gradually decreases after every 0.5ml addition after the end point.

When all the H^+ Ions are reacted further addition of poorly ionized ammonium hydroxide does not cause appreciable change in the conductance due to weak base.

$$HCl + NH_4OH \rightarrow NH_4Cl + H_2O$$

Part-1:

Procedure:-

Standardization of HCl solution using 0.1N Na_2CO_3 :-

Burette: - The burette is cleaned first with distilled water and filled with HCl, without any air bubbles.

Conical Flask: - Conical flask is washed with distilled water and then pipette out 20ml of Na_2CO_3 Solution into the conical flask and add 2 or 3 drops of Methyl Orange indicator.

Indicator: - Methyl Orange

End Point: - yellow to red

The conical flask is placed under the burette on a glazed tile, then the HCl present in the burette is slowly rundown by shaking the conical flask in clockwise direction continuously, the titration is continued until a color change yellow to red which is the endpoint. The experiment is repeated till the concurrent readings are obtained.

S.No	Volume of Na_2CO_3 Solution in ml	Burette Readings		Volume of HCl rundown in ml(b-a)
		Initial (a)	Final (b)	

Calculations:-

Normality of HCl is calculated using the equation

$$N_1V_1 = N_2V_2$$

Na_2CO_3

N_1,Normality of Na_2CO_3 = 0.1 N

V_1,Volume of Na_2CO_3 solution = 20ml

HCl

N_2, Normality of HCl = ?

V_2, Volume of HCl = ml (burette readings)

Normality of HCl (N_2) = N_1V_1/ V_2

=

Part-2:

Determination of strength of NH_4OH using standard solution of HCl

Procedure:-

Burette: - The burette is cleaned first with distilled water and filled with **NH_4OH**, without any air bubbles.

Beaker- Beaker is washed with distilled water and then add 5 ml of HCl, 5ml of distilled water.

Indicator: - 2 or 3 drops of Phenolphthalein indicator

End Point: - colorless to pale pink

The beaker containing solutions is titrated against NaOH solution on conductometer observe the conductance values for every 0.5 ml addition of NH_4OH solution and calculate the conductance. Draw the graph, plotting on x-axis –volume of NH_4OH and on y-axis –corrected conductance. using the Equivalence point, calculate the concentration of HCl.

S.No	Volume of Ammonium hydroxide (ml)	Conductance Ω-1	S.No	Volume of Ammonium hydroxide (ml)	Conductance Ω-1
1.	0.5		**13.**	6.5	
2.	1.0		**14.**	7.0	
3.	1.5		**15.**	7.5	
4.	2.0		**16.**	8.0	
5.	2.5		**17.**	8.5	
6.	3.0		**18.**	9.0	
7.	3.5		**19.**	9.5	
8.	4.0		**20.**	10.0	
9.	4.5		**21.**	10.5	
10.	5.0		**22.**	11.0	
11.	5.5		**23.**	11.5	
12.	6.0		**24.**	12.0	

Calculations:-

HCl

Concentration of HCl N_3 = N

Volume of HCl solution V_3 = ml

NH_4OH

Concentration of NH_4OH N_4 = ? N

Volume of NH_4OH V_4 = 10 ml (burette readings)

Concentration of NH_4OH solution (N_4) = N_3V_3/ V_4

=

= N

The strength of HCL solution = N_3 X Equivalent weight of HCL(36.5 g/lit)

=

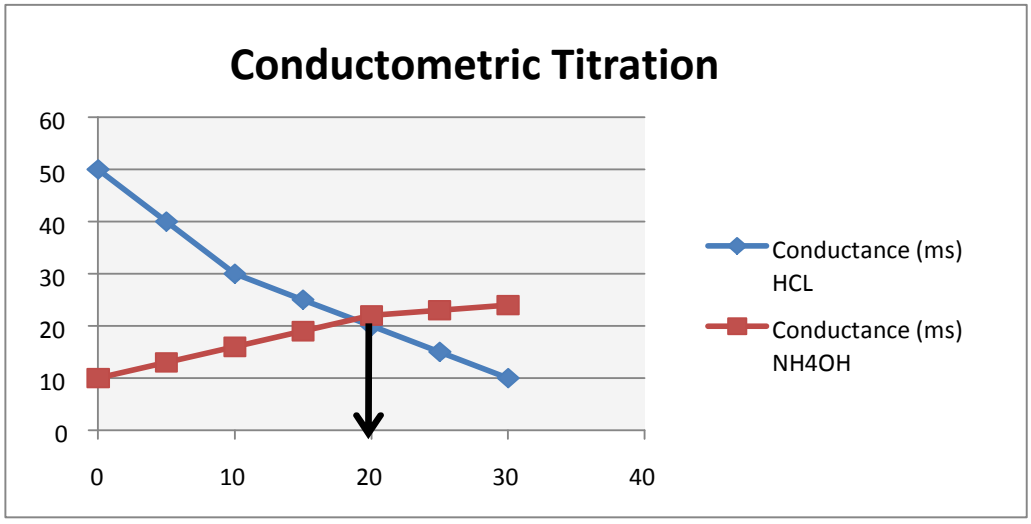
= gm

Report:-

The Concentration of HCl (N_3) = N

The strength of HCL solution = gm

Strong Acid-Weak Base



Conclusion:

1. CO1 to CO6 and PO1, PO2, PO7, PO9 is attained.
2. The students would be aware of instrumental methods of chemical analysis.
3. Students acquire the skill to determine the conductivity of acid base using conductivity meter in the real lab.

Precautions:

1. Gentle handling of the Instrument
2. The electrode is always dipped in distilled water when not in use.

VIVA QUESTIONS

1.Explain Conductivity, Molar Conductivity, And Specific Conductivity & Equivalent Conductivity?

Answer: the reciprocal of resistance is called conductance or **conductivity**. **Electrical conductivity** or **specific conductance** is a measure of a material's ability to conduct an electric current. When an electrical potential difference is placed across a conductor, its movable charges flow giving rise to an electric current**.**

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Equivalent conductivity: Property of an electrolyte, equal to the specific conductance divided by the number of gram equivalents of solute per cubic centimeter of solvent.

2. What do you Mean by Cell Constant?

Answer: the ratio of distance between conductometric titration electrodes to area of the electrodes, measured from the determined resistance of a solution of known specific conductance.

3. What Is the Principle of this Experiment?

Answer: determination of volume of the base required conduct metrically to find the concentration of given acid sample.

4. What Could Be the Shape of the Graph?

Answer: v-shape.

5. What are the Units of Conductivity In Cgs and Si Systems?

Answer: Ohm^{-1} or mho, Siemens.

6. What Is the Equivalent Mass Of Oxalic Acid?

Answer: 63

7. How Can You Arrive at that Value /

Answer: its molecular mass is 126. Basicity is 2 So

Equivalent weight = (molecular weight) / basicity.

Equivalent weight = 126 / 2= 63

8. When Strong Acid Combines With A Strong Base. What Type Of Reaction Occurs?

Answer: acid and base combines to form salt and water. The reaction is called as neutralization reaction.

9. Name the Apparatus used for this Method?

Answer: the conductivity meter with a conductivity cell.

10. How Conductance is Related to the concentration of the Ions?

Answer: the specific conductance is proportional to the concentration of ions in it.

11. How the End Point for aParticular Reason Is Calculated Using this Titration Method?

Answer: on plotting a graph between conductance and volume of the base, the point of intersection of the straight lines gives the end point.

12. Why Conductance Decreases On Addition Of NaOH And Hcl ?

Answer: during the titration the fast moving hydrogen ions are replaced by the slow moving Sodium ions, as a result the conductance of the solution decreases.

13. What Is the Unit for Resistance?

Answer: the unit for resistance is Ohm.

14. What is the Equivalent Mass of Hcl?

Answer : Equivalent mass of HCL is 36.5

15. What is the Equivalent Mass of NaOH ?

Answer: equivalent mass of NaOH is 40.

Potentiometric Titrations

Introduction

When a metal chloride is dipped in a solution containing its own ions, there are two opposite process which takes place at the metal solution junction. The metal atoms have a tendency to pass into the solution inthe form of metal ions. For a mono valent metal `M` the process can be represented as

$$M - e \rightarrow M^{+} \quad \text{or} \quad M \rightarrow M^{+} + e$$

At the same time, the metal ions M^{+} present in the solution have a tendency to get discharged on the surface of the metal chloride.This process is known as ion discharged process and can be represented as

$$M^{+} + e^{-} \rightarrow M$$

When a metasl is placed in the solution of its ions, ion formation of and ion discharge process takes place out at different rates .But after some time the rate of formation of ions become equal to the rate of their discharge.

The electrode is then said to be in equilibrium and the potential is required under such condition is referred to as its reversible potential which is shown by `E` is given by

$$\mathbf{E = E^{0} + (RT/nF)\ ln M^{n+}}$$

Where E^{0} = Standard electrode potential

n = valency of the metal ion

F = faraday

R= Gas constant

T= temperature

M^{n+} = Activity of metal ion in solution

Date:

Experiment – 13

Determination of Potentiometric titration between Strong acid and Strong base

Aim:-

To determine the concentration of strong acid hydrochloric acid using standard solution of strong base sodium hydroxide using electrode.

Apparatus and Chemicals required:

Sodium hydroxide solution (0.1N), oxalic acid solution (0.1N), Hydrochloric acid (0.1N)

Principle:-

The potentiometric titrations are the titrations which involve the measurement of electrode potentials with the addition of titrant.The potential of an electrode depends upon the concentration of the ion .During in a itration with titrant there is a change in ionic concentration which can be followed by measuring the potential of a suitable indicator electrode.

A simple arrangement for a manual potentiometric titration is a reference electrode, indicator electrode. Which are connected to an electronic voltmeter which is measured in milli volts. The emf of the indicator electrode changes gradually with the charge of concentration of ions caused by the addition of titrant from the burette .The equivalence point is indicated by a sharp change in electrode potential. The reference electrode gas a constant value.

The equivalence point can be find by plotting a graph between the cell emf and the volume titarnt added frome the burette.the steepest portion of the curve indicates the equivalence.

Procedure:-

1. Standardize the sodium hydroxide solution using standard oxalic acid solution with phenolphthalein as indicator.
2. Standardize the stock solution of hydrochloric acid using standard sodium hydroxide solution and Phenolphthalein as indicator.
3. Prepare 100ml 0.1N HCl solution from the stock solution by exact dilution.
4. Take 10ml of 0.1N HCl solution into a 100ml beaker using a burette or a pipette.Add 40ml of distilled water with measuring jar.

5. Introduce the electrode into the solution and connect them to the terminals of the potentiometer.
6. Note down the emf of the cell against 0.00 volume of sodium hydroxide.
7. Fill the burette with standard NaOH.

Perform a pilot titration by adding NaOH to the experimental solution in one ml portions And noting down the corresponding emf values for each addition .make sure that the solution is uniform After each addition by stirring it well using a glass rod or magnetic stirrer.Locate the endpoint where there is a large change in the emf of the cell.

Now an accurate titration can be performed by repeating qall the steps above but adding only 0.1ml portions of sodium hydroxide in the volume range where jump in cell potential has occurred in the previous pilot titration.

Tabulate the results in the format given

VOLUME OF NaOH added in ml (V)	POTENTIAL OF THE ELECTRO CHEMICAL CELL against SCE,Milli volts(E)	VOLUME OF NaOH added in ml (V)	POTENTIAL OF THE ELECTRO CHEMICAL CELL against SCE,Milli volts(E)
0.0		**11.0**	
1.0		**12.0**	
2.0		**13.0**	
3.0		**14.0**	
4.0			
5.0			
6.0			
7.0			
8.0			
9.0			
10.0			

Draw a sigmoid titration plot of cell potential against volume of sodium hydroxide added.Note down the volume of NaOH `X` ml corresponding to the end pint where there is aq large change in the cell emf.

volume of NaOH `X` ml corresponding to the end pint where there is aq large change in the cell emf.

Calculations:

Calculate the strength of HCl using the formula $V_1N_1 = V_2N_2$

HCl

Concentration of HCl N_1= ? N

Volume of HCl solution V_1 = 10ml

NaOH

Concentration of NaOH N_2 = 0.1 N

Volume of NaOH V_2 = 10 ml

Concentration of HCl (N_1) = N_2V_2/ V_1

=

= N

The strength of HCL solution = N_3 X Equivalent weight of HCL(36.5 g/lit)

= 0.1X36.5

= gm

Report:-

The Concentration of HCl (N_3) = N

The strength of HCL solution = gm

Conclusion:

1. CO1 to CO6 and PO1, PO2, PO7, PO9 is attained..
2. The students would be aware of instrumental methods of chemical analysis.
3. Students acquire the skill to determine the potential of acid base using potentiometer in the real lab.

Precautions:

3. Gentle handling of the Instrument
4. The electrode is always dipped in distilled water when not in use.

VIVA QUESTIONS

1. **What is Potentiometric Titration**?

Answer: the volumetric titration which involves the determination of strength of given solution by measuring electrode potentials is called potentiometric titrations.

2. What is the Electrode Potential?

Answer: the electrode potential is the tendency of an electrode to lose or gain electrons when dipped in a solution of its own ions.

3. What is the Reduction Potential?

Answer: the reduction potential is the tendency of an electrode to gain electrons when it is dipped in a solution of its own ions.

4. What is the Oxidation Potential?

Answer: the oxidation potential is the tendency of an electrode to lose electrons when it is dipped in a solution of its own ions.

5. What is Reference Electrode?

Answer: the electrode whose potential is already known (or arbitrarily fixed) and is used as reference to find out the electrode potential of another electrode is called reference electrode.

6. **What is the Indicator Electrode**?

Answer: the electrode with which EMF is measured by connecting it to standard reference electrode is called indicator electrode.

7. What is the Effect of Temperature on Emf of an Unknown Solution?

Answer: as the temperature increases, the EMF of the solution also increases.

Date:

Experiment – 14

Determination of Potentiometric titration between Strong acid and Weak base

Aim:-

To determine the concentration of strong acid hydrochloric acid using standard solution of weak base ammonium hydroxide using electrode.

Apparatus and Chemicals required:

Ammonium hydroxide solution (0.1N),oxalic acid solution (0.1N), Hydrochloric acid (0.1N)

Principle :-

The potentiometric titrations are the titrations which involve the measurement of electrode potentials with the addition of titrant.The potential of an electrode depends upon the concentration of the ion .During in a titration with titrant there is a change in ionic concentration which can be followed by measuring the potential of a suitable indicator electrode.

A simple arrangement for a manual potentiometric titration is a reference electrode, indicator electrode. Which are connected to an electronic voltmeter which is measured in milli volts. The emf of the indicator electrode changes gradually with the charge of concentration of ions caused by the addition of titrant from the burette .The equivalence point is indicated by a sharp change in electrode potential. The reference electrode gas a constant value.

The equivalence point can be find by plotting a graph between the cell emf and the volume titarnt added frome the burette.the steepest portion of the curve indicates the equivalence.

Procedure:-

1. Standardize the Ammonium hydroxide solution using standard oxalic acid solution with Phenolphthalein as indicator.
2. Standardize the stock solution of hydrochloric acid using standard ammonium hydroxide solution and Phenolphthalein as indicator.
3. Prepare 100ml 0.1N HCl solution from the stock solution by exact dilution.
4. Take 10 ml of 0.1N HCl solution into a 100ml beaker using a burette or a pipette. Add 40ml of distilled water with measuring jar.

5. Introduce the electrode into the solution and connect them to the terminals of the potentiometer.
6. Note down the emf of the cell against 0.00 volume of ammonium hydroxide.
7. Fill the burette with standard NH_4OH.

Perform a pilot titration by adding NH_4OH to the experimental solution in one ml portions And noting down the corresponding emf values for each addition .make sure that the solution is uniform

After each addition by stirring it well using a glass rod or magnetic stirrer. Locate the endpoint where there is a large change in the emf of the cell. Now an accurate titration can be performed by repeating qall the steps above but adding only0.1ml portions of ammonium hydroxide in the volume range where jump in cell potential has occurred in the previous pilot titration.

Tabulate the results in the format given

VOLUME OF NH_4OH added in ml (V)	**POTENTIAL OF THE ELECTRO CHEMICAL CELL against SCE,Milli volts(E)**	**VOLUME OF NH_4OH added in ml (V)**	**POTENTIAL OF THE ELECTRO CHEMICAL CELL against SCE,Milli volts(E)**
0.0		**11.0**	
1.0		**12.0**	
2.0		**13.0**	
3.0		**14.0**	
4.0			
5.0			
6.0			
7.0			
8.0			
9.0			
10.0			

Draw a sigmoid titration plot of cell potential against volume of Ammonium hydroxide added.Note down the volume of NH_4OH `X` ml corresponding to the end pint where there is aq large change in the cell emf.

Calculation

Calculate the strength of HCl using the formula $V1N1 = V_2N_2$

HCl

Concentration of HCl N_1 = ? N

Volume of HCl solution V_1 = 10ml

NH_4OH

Concentration of NH_4OH N_2 = N

Volume of NH_4OH V_2 = ml

Concentration of HCl (N_1) = N_2V_2/ V_1

= N

The strength of HCL solution = N_3 X Equivalent weight of HCL(36.5 g/lit)

= gm

Report:-

The Concentration of HCl (N_3) = N

The strength of HCL solution = gm

Conclusion:

1. CO1 to CO6 and PO1,PO2,PO7,PO9 is attained.
2. The students would be aware of instrumental methods of chemical analysis.
3. Students acquire the skill to determine the potential of acid base using potentiometer in the real lab.

Precautions:

1. Gentle handling of the Instrument
2. The electrode is always dipped in distilled water when not in use.

VIVA QUESTIONS

1. **What is Potentiometric Titration**?

Answer: the volumetric titration which involves the determination of strength of given solution by measuring electrode potentials is called potentiometric titrations.

2. What is the Electrode Potential?

Answer: the electrode potential is the tendency of an electrode to lose or gain electrons when dipped in a solution of its own ions.

3. What is the Reduction Potential?

Answer: the reduction potential is the tendency of an electrode to gain electrons when it is dipped in a solution of its own ions.

4. What is the Oxidation Potential?

Answer: the oxidation potential is the tendency of an electrode to lose electrons when it is dipped in a solution of its own ions.

5. What is Reference Electrode?

Answer: the electrode whose potential is already known (or arbitrarily fixed) and is used as reference to find out the electrode potential of another electrode is called reference electrode.

6. **What is the Indicator Electrode**?

Answer: the electrode with which EMF is measured by connecting it to standard reference electrode is called indicator electrode.

7. What is the Effect of Temperature on Emf of an Unknown Solution?

Answer: as the temperature increases, the EMF of the solution also increases.

Date:

Experiment – 15

Determination of Zinc using standard EDTA solution

Aim:-

a) Standardization of the EDTA solution using standard Zinc sulphate solution.
b) Determination of zinc using standard EDTA.

Apparatus:-

Burette, pipette, burette stand, glazed tile, conical flask,

Chemicals required:-

0.1M Standard zinc sulphate solution, EDTA solution, Ammonia buffer, Erichrome Black T indicator.

Principle:-

Metal ions form a complex with EDTA according to the equation

M^{2+} + EBT → M-EBT complex

Wine -red

M-EBT complex + EDTA → M-EDTA complex + EBT

STABLE complex blue

The completion of the reaction between M^{+} and EDTA is detected by the use of metal ion indicator namely Eriochrome Black T. Initially, when the P^{H} of the medium maintained at P^{H}=7to11 the metal ion combine with indicator to form metal indicator complex which appears as a wine red color. Near the end point, EDTA breaks the metal indicator complexation, resulting in the formation of metal-EDTA complex. Hence at the end point, the liberated free indicator yields a blue color to the solution. Thus the end point is a fine, sharp change from wine red to blue color.

Procedure:-

b) **Standardize the EDTA solution using standard zinc sulphate solution**.

Burette: - The burette is washed with distilled water, and then fills it with EDTA without air bubbles.

Conical Flask: - Conical flask is washed with distilled water and then pipette out 20ml of Zinc sulphate solution into the conical flask and then add 3.0 ml of ammonia buffer solution, 3-4 drops of Erichrome Black T indicator.

Indicator: - Erichrome Black T indicator.

Endpoint: - wine red to blue color.

To the conical flask containing Zinc sulphate, ammonia buffer solution, Erichrome Black T indicator is placed under the burette on a glazed tile, then the EDTA present in the burette is slowly rundown by shaking the conical flask in clockwise direction continuously, the titration is continued until the color changes from wine red to blue color which is the endpoint. The experiment is repeated until concurrent readings are obtained.

S.No	Volume of Zinc Sulphate solution (V_1) in ml	Burette readings		Volume of EDTA solution rundown (V_2) in ml
		Initial	Final	

Calculations:-

The Molarity of EDTA solution can be calculated from the formula $V_1M_1 = V_2M_2$

Zinc sulphate

Volume of zinc sulphate solution V_1 = 20.0 ml

Molarity of zinc sulphate solution M_1 =0.1M

EDTA

Volume of EDTA solution V_2 = ml

Molarity of EDTA solution M_2 =?

Molarity of EDTA $M_2 = \frac{M_1V_1}{V_2}$

=

= M

b) **Determination of Zinc present in the solution**

The zinc solution given in the 100ml volumetric flask is dilute up to the mark and mix well to make the uniform solution

Burette: - The burette is washed with distilled water, and then fills it with EDTA without air bubbles.

Conical Flask: - Conical flask is washed with distilled water and then pipette out 20ml of Zinc sulphate solution into the conical flask and then add 3.0 ml of ammonia buffer solution, 3-4 drops of Erichrome Black T indicator.

Indicator: - Erichrome Black T indicator.

Endpoint: - wine red to blue color.

To the conical flask containing Zinc sulphate, ammonia buffer solution, Erichrome Black T indicator is placed under the burette on a glazed tile, then the EDTA present in the burette is slowly rundown by shaking the conical flask in clockwise direction continuously, the titration is continued until the color changes from wine red to blue color which is the endpoint. The experiment is repeated until concurrent readings are obtained.

S.No	Volume of Zinc Sulphate solution (V_3) in ml	Burette readings		Volume of EDTA solution rundown (V_4) in ml
		Initial	Final	

Calculations:-

The Molarity of EDTA solution can be calculated from the formula $V_3M_3 = V_4M_4$

Zinc sulphate

Volume of zinc sulphate solution V_3 = 20.0 ml

Molarity of zinc sulphate solution M_3 = 0.1M

EDTA

Volume of EDTA solution V_4 =

Molarity of EDTA solution M_4 =

Molarity of EDTA $M_3 = \frac{M_4V_4}{V_3}$

=

=

Amount of zinc ion present in 100 ml of the given unknown solution = $\frac{M_3 \text{ X } 65.38 \text{ X } 100}{1000}$ g

= gm

Result:-

The amount of zinc ion present in given 100ml solution = gm.

S.No	Given	Obtained	% of error = $\frac{\text{Given-Obtained}}{\text{Given}}$ X 100

Conclusion:

1. CO1 to CO6 and PO1,PO2,PO7,PO9 is attained.
2. Students understand the apparatus used for a titration.
3. Students acquire the skill to perform the Complexometric-titration in the real lab.
4. Students acquire the skill to determine the concentration of metals in the real lab.

Precautions:

1. Pipptteing as to be accurate in order to avoid excess addition of the titrating agent..
2. Reading should be taken to avoid the parallax error.
3. The titration should be placed on white paper to identify properly the color change at the end point.
4. All the glass apparatus should be washed thoroughly with distilled water before use.
5. They should not be any leakage in the burette.

VIVA QUESTIONS

1. What is the full form of EDTA & EBT Indicators?

Answer: Ethylene diamine tetra acetic acid & Eriochrome Black-T.

2. What is the Principle in EDTA titrations?

Answer: Ca^{+2} or Mg^{+2} can form complex with EDTA (also with EBT).

3. Give The Structures Of EDTA & EBT?

Answer: refer theory and principle.

4. Why EBT Indicator should be used in Basic Medium?

Answer: it can exhibit color changes in basic medium.

5. Which Buffer is used in this Titration?

Answer: Ammonium buffer.

Experiment – 16

Date:

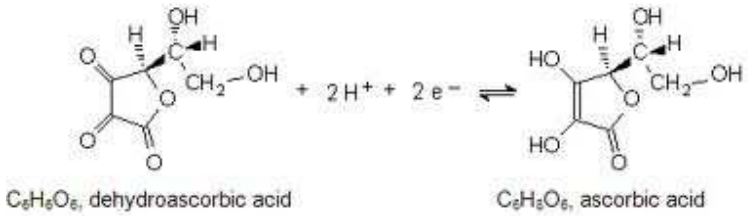
Determination of Vitamin-C

Aim:- To determine the Vitamin – C concentration in fruit juices.

Introduction:-

Vitamin – C or ascorbic acid is a water soluble antioxidant that plays a vital role in protecting the body from infection and disease. It is an essential antioxidant needed by the human body. It is not synthesized by the human body and therefore must be acquired by the human body and therefore must be acquired from dietary sources – primarily fruits and vegetables.

The chemical structure and antioxidant action of ascorbic acid are illustrated in the redox half equation below



$C_6H_6O_6$, dehydroascorbic acid $C_6H_8O_6$, ascorbic acid

This method determines the Vitamin – C concentration in a solution by a redox titration using iodine. As the iodine is added during the titration, the ascorbic acid is oxidized o dehydroascorbic acid, while the iodine is reduced to iodine ions.

Ascorbic acid + I_2 → $2I^-$ + dehydroascorbic acid

Once all the ascorbic acid has been oxidized, the excess iodine is free to react with the starch indicator, forming the blue-black starch-iodine complex. This is the endpoint of the titration. The method is suitable for use with Vitamin – C tablets, fresh or packaged fruit juices and solid fruits and vegetables.

Chemicals required:-

0.005 M Iodine solution

Weigh 2 gm of potassium iodide into a 100 ml beaker. Weigh 1.3 gm of iodine and add it to the beaker. Add a few ml of distilled water and swirl for a few minutes until iodine is dissolved. Transfer iodine solution to a 1 L volumetric flask, making sure to rinse all traces of solution into the volumetric flask

using distilled water. Make the solution up to 1 L mark with distilled water. The concentration of the prepared iodine solution can be more accurately determined by titration with a standard solution of potassium thiosulphate using starch indicator.

0.5% starch indicator solution

Weigh out 0.25 gm of soluble starch and add it to 50 ml of near boiling water in a 100 ml conical flask. Stir to dissolve and cool before using.

Sample preparation:-

For Vitamin C tablets: Dissolve a single tablet in 100 ml of distilled water

For fresh fruit juice: Strain the juice through cheese cloth to remove seeds and pulp which may Block pipettes.

For packaged fruit juice: This may also need to be strained through cheese cloth if it contains a lot of Pulp or seeds.

For fruits and vegetables: Cut a 100 gm sample into small pieces and grind in a mortar and pestle. Add 10 ml portions of distilled water several times while grinding the sample, Each time decanting off the liquid extract into a 100 ml volumetric flask.

Finally, strain the ground fruit/ vegetable pulp through cheese cloth, rinsing. The pulp with a few 10 ml portions of water and collecting all filtrate and washings in the volumetric flask. Make the extracted solution up to 100 ml with distilled water.

Procedure:-

Pipette 20 ml aliquot of the sample solution into a 250 ml conical flask and add about 150 ml of distilled water and 1 ml of starch indicator solution. Titrate the sample with 0.005 M iodine solution. The endpoint of the titration is identified as the first permanent trace of a dark blue-black colour due to the starch-iodine complex. Repeat the titration with further aliquots of average volume of iodine solution used from concordant titers.

The average titre volume should ideally be in the range of 10-30 ml. if the titre required for a 20 ml aliquot of sample solution is well outside this range then a larger or smaller aliquot volume should be chosen. If the volume of the titre is too low, dilute the standard. If the titre volume is too high, dilute the sample.

Calculations:-

Volume of the iodine solution consumed in ml = _____

Molarity of iodine solution (M) = _____

Volume of sample solution taken (x ml) = _____

Molarity of sample solution = $\frac{\text{Molarity of iodine X Volume of iodine}}{\text{Volume of the sample solution}}$

Amount of Vitamin C in 100 ml = $\frac{\text{Molarity X Eq.Wt. Of ascorbic acid}}{10}$

= ……..

Then calculate the concentration of Vitamin C in percentage of weight.

Report:-

Sample Name: …….

The concentration of Vitamin C in sample (fruits or fruit juices) = ……. %

Conclusion:

1. CO1 to CO6 and PO1,PO2,PO7,PO9 is attained.
2. Students understand the apparatus used for a titration.
3. Students acquire the skill to determine the Vitamin – C concentration in fruit juices in the real lab.

Precautions:

1. Pipptteing as to be accurate in order to avoid excess addition of the titrating agent..
2. Reading should be taken to avoid the parallax error.
3. The titration should be placed on white paper to identify properly the color change at the end point.
4. All the glass apparatus should be washed thoroughly with distilled water before use.
5. They should not be any leakage in the burette.

VIVA QUESTIONS

1. What is the other name for the Vitamin-C?

Answer: Ascorbic Acid

2. What is meant by Ascorbic Acid?

Answer: It is an antioxidant

3. Deficiency of Vitamin-c lead to what type of disease?

Answer: Scurvy.

4. What are the characteristics of the scurvy?

Answer: Abnormalities in the bones and teeth.

5. What are main sources of Vitamin-C?

Answer: Raw citrus fruits and their juices.

6.How can you determine the vitamin –C in food?

Answer: By redox titration.

7.What is meant by redox titration?

Answer: Any chemical reaction which involves oxidation and reduction.

B.ADVANCED EXPERIMENTS

Date:

1. VISCOSITY

Aim: To determine the viscosity of oil by red wood viscometer

Apparatus: Red wood viscosity meter, stop watch, kohlrush flask.

Chemicals required: Given sample of lubricant.

Principle:

Viscosity of liquid is generally determined using poiseuille's equation. The viscosity of oil is determined by measuring the time taken for a given quantity of oil to flow through an orifice of standard dimensions the viscometers commonly used are saybolt viscometer and red wood viscometer.

Procedure:

1. Clean the viscometer cup properly dry it to remove any traces of solvent.
2. Level the viscometer with the help leveling screws.
3. Fill the outer bath for determining the temp at 80˚C and below.
4. Place the ball valve on the jet to close it and pour the test oil into the cup up to the tip of indicator.
5. Place the clean dry kohl rush flask immediately below and directly in line with discharging jet.
6. Insert a clean thermometer and a stirrer in the cup and cover it with lid.
7. Heat the water filled in the slowly with constant stirring. When the oil in the cup attains a desired temperature, stop the heating.
8. Lift the ball valve and start the stop watch oil from the jet flows into the flask.
9. Stop the stop watch when the lower meniscus of the oil reaches the mark on the neck of receiving flask
10. Record the time taken for the 50ml of the oil collect in the flask.
11. Repeat the experiment to get more readings.

Tabular form:

S.No	Sample Oil	Viscosity(seconds, Red wood viscometer)

Result: Viscosity of given oil sample

Conclusion:

1. CO1 to CO6 and PO1,PO2,PO7,PO9 is attained.
2. The students able to determine the Viscosity of oils in the real lab.

Precautions:

1. Always a fresh portion of the oil sample should be used.
2. The thermometer bulb should dip into the oil
3. Fill the outer bath with water.

2.FLASH POINT AND FIRE POINT

Date:

Aim: To find the flash point and fire point of the given oil sample by using Cleveland`s apparatus

Definition:

Fire Point is the lowest temperature at which application of test film causes the material to ignite and burn at least for 5 secs under specified conditions to the test.

Apparatus:

Cleveland`s apparatus, thermometer, Oil sample (petrol,diesel and kerosene etc)

Procedure:

The oil cup is filled with sample , so that the minscus is exactly at the filling line at room temperature.Care is taken that no sample is above the filling line or on the out side of the apparatus.The sample is heated by adjusting the energy regulator so that the raise in temperature doesnot exceed 17˚C/min till the temperature reaches approximately 37.7˚C lessthan the flash point of the sample.There after the rate of the heating is decreasing for atleast the last 28˚C below the flash point is reached it shall not be lessthan 5˚C/min.However if the flash point of the given oils are lower than 65˚C,the rate of heating should give 2˚C/min rise in the beginning and 0.5˚C/min in final stage.The test flame is applied and the flash point is obtained.After determine the flash point heating is continued so that the rise in temperature is maintained at the specific rate and rise point is obtained .The experiment with oil is continue until the successive minimum temparatres are equal.

Observations:

S.No	Sample used	Flash Point	Fire point

Result: The fire and flash point of the given oil sample

Conclusion:

1. CO1 to CO6 and PO1, PO2, PO7, PO9 is attained.
2. The students able to determine the ignition temperature of oils.

Precautions:

1. As moisture effects the flash point,all the parts of the cup and its accessories should be dried before placing oil in the cup.
2. Always a fresh portion of the oil sample should be used.
3. A second determination on the same portion of oil shows a higher flash point.
4. The thermometer bulb should dip into the oil.
5. For applying the test flame, the slide should be drawn open slowly and closed quickly.
6. Stirring should be discontinuing during the application of the test flame.