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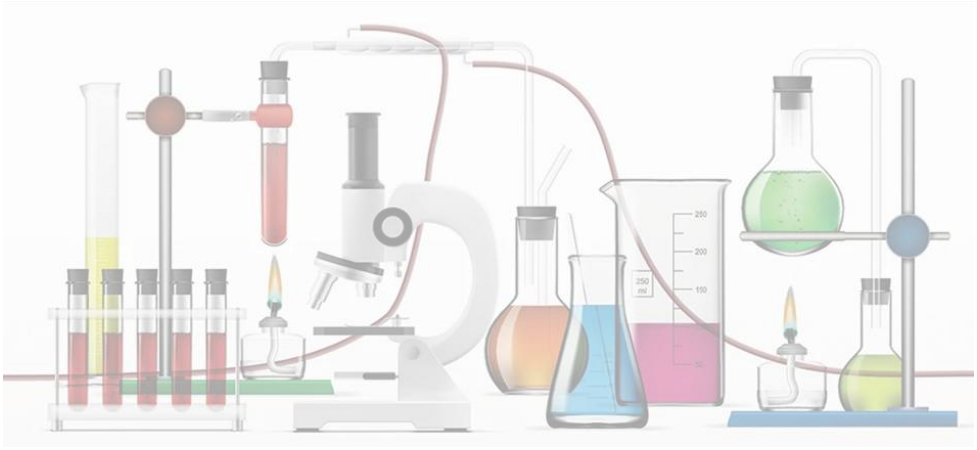
DEPARTMENT OF CHEMISTRY

LAB MANUAL

ENGINEERING CHEMISTRY
22CY1001

A. Y 2024-2025

III B. TECH (AI&DS, CSE, CSIT, ECE, ECS, IOT)



NAME:

Regd. ID:

Year & Branch:

LIST OF EXPERIMENTS

Exp. No.	Title of the Experiment	CO-Mapping
1	Introduction to engineering chemistry lab	CO5
2	Determination of the concentration of unknown solutions (HCl vs NaOH)	CO5
3	Determination of total alkalinity of a water sample	CO5
4	Estimation of hardness of water by EDTA method (Complexometry)	CO5
5	Determination of chloride content of water sample	CO5
6	Determination of the rate constant of a reaction.	CO5
7	Determination of Dissolved oxygen in the given water sample.	CO5
8	Estimation of amount of iron using potentiometric method	CO5
9	Conductometric titration of strong acid with strong base	CO5
10	Estimation of amount of an acid using pH metric method	CO5
11	Synthesis of a polymer/ drug.	CO5
12	Estimation of KMnO4 using standard oxalic acid	CO5
13	Estimation of ferrous and ferric ion using standard K2Cr2O7 solution.	CO5
14	Estimation of amount of Vitamin- C in the given sample.	CO5
15	Determination of Iron using colorimetry.	CO5

LAB CONTINUOUS EVALUATION PLAN

Note: Each experiment must be assessed, and marks must be awarded to each student as per the format given below.

Expt. No.	Date	Name of the Experiment	Marks					
			Record (5M)	Experiment (5M)	Inference, Analysis & Results (5M)	Viva Voce (5M)	Total marks (20M)	Sig. of Faculty
1		Determination of the concentration of unknown solutions (HCl vs NaOH)						
2		Determination of total alkalinity of a water sample						
3		Estimation of hardness of water by EDTA method (Complexometry)						
4		Determination of chloride content of water sample						
5		Determination of the rate constant of a reaction.						
6		Determination of Dissolved oxygen in the given water sample.						
7		Estimation of amount of iron using potentiometric method						
8		Conductometric titration of strong acid with strong base						
9		Estimation of amount of an acid using pH metric method						
10		Synthesis of a polymer/ drug.						
11		Estimation of $KMnO_4$ using standard oxalic acid						
12		Estimation of ferrous and ferric ion using standard $K_2Cr_2O_7$ solution.						
13		Estimation of amount of Vitamin- C in the given sample.						
14		Determination of Iron using colorimetry.						

Total:

NOTE: ***Faculty must maintain the same format in the attendance register of the said lab course***

Signature of the Faculty

INTRODUCTION TO CHEMISTRY LABORATORY

In any industry, quality control of the raw material and the final products is an essential and integral part of the industrial practice so as to achieve the standards, reputation and competence. Chemical analysis includes both qualitative and quantitative analysis. Quantitative analysis is frequently addressed.

Quantitative analysis is carried out by conventional methods as well as modern instrumental methods. The conventional methods include Volumetric analysis, Gravimetric analysis, etc. The modern methods involve use of electronic instruments like pH-meter, conductometer, colorimeter, spectrophotometer, polarograph, chromatograph, etc. among others.
The experiments described in this booklet aim at training the undergraduate engineering students of all branches in some analysis methods like volumetric analysis and some instrumental methods.

A few of the often-used terms are briefly explained below

Solute: In a solution, the substance present in relatively low quantity is called a solute.

Solvent: In a solution, the substance present in relatively high quantity is called a solvent.

Concentration: It is relative or absolute amount of the desired material in the company of others, especially in solution. The relative amounts of solute and solvent present in a solution is also called concentration of the solution. Concentration can also be defined as the number of solute particles present in unit volume of solution. Usually, normality or molarity is used to express the concentrations.

Normality (N): It is the number of gram equivalents of the substance in a liter of the given solution.

Molarity (M): It is the number of moles of the substance in a liter of the given solution.
Molarity= No. of moles/volume of the solution in liters.

Standard Solution: It is the solution for which the exact concentration is known. In general, graduated flasks are used to prepare standard solutions.

Molar Solution: If one gram molecular weight of a solute is dissolved in one liter of the solvent, it is called a Molar solution.

Standardizations: Determination of the concentration of a solution by any suitable analytical method is known as standardizations.

Primary standard substance: A primary standard is a reliable, readily quantified substance. Features of a primary standard include high purity, stability (low reactivity), low hygroscopic, efflorescence, high solubility and high equivalent weight. Some examples of primary standards are potassium hydrogen phthalate, sodium carbonate, sodium chloride, potassium chloride, potassium dichromate, etc.

Secondary standard substance: A secondary standard is a substance of low purity, low solubility, more hygroscopicity or high reactivity. The concentration of solution of such substance changes with time. Examples of secondary standards are $Na_2B_4O_7.10H_2O$, $CuSO_4.5H_2O$, etc. Hydrated salts do not make good standards because of difficulty of efficient drying.

Primary standard solution: If the concentration of a solution is known by dissolving a known mass of the desired substance into a known volume of solvent, then the solution is called a primary standard solution.

Secondary standard solution: If the concentration of a solution is determined by using a suitable primary standard solution, then the solution is called secondary standard solution.

Volumetric Analysis: Chemical procedure used for determining the concentration of a solution. A known volume of a solution of a unknown concentration is reacted with a known volume of a solution of known concentration (standard). The standard solution is delivered from a burette so the volume added is known. This technique is known as titration.

Titration: It is a common laboratory method of quantitative chemical analysis which can be used to determine the concentration of a known reactant. Because volume measurements play a key role in titration, it is also known as volumetric analysis. A reagent, called the titrant, of known concentration (a standard solution) and volume is used to react with a solution of the analyte (titrand), whose concentration is not known in advance. Using a burette to add the titrant, it is possible to determine the exact amount that has been consumed when the endpoint is reached.

Indicator: The completion of the titration is detected by the addition of an auxiliary reagent, known as an indicator. The indicator gives a clear visual change in color in the solution being titrated.

End point: The end point is the point at which the titration is complete, as determined by an indicator.

Equivalence point: It is the point at which the reaction between the titrant and analyte completes. It is the theoretical end point. In practice, a very small difference occurs between equivalence point and visible end point. This represents the titration error. The indicator and experimental conditions should be selected such that this error is as small as possible.

Types of titrations: Titrations can be classified by the type of reaction. Different types of titrations include:

1. Acid base titration is based on the neutralization reaction between the analyte and an acidic or basic titrant. They most commonly used as pH indicator, a pH meter, or a conductometer to determine the end point.

2. A redox titration is based on an oxidation reduction reaction between the analyte and titrant. They most commonly use a potentiometer or a redox indicator to determine the end point. Frequently either reactants or the titrant have a color intense enough that an additional indicator is not required.

3. A complexometric titration is based on the formation of a complex between the analyte and the titrant. The chelating agent EDTA is very commonly used to titrate metal ions in solution. These titrations generally require specialized indicators that form weaker complexes with the analyte. A common example is Eriochrome Black T for the titration of calcium and magnesium ions.

4. A precipitation titration is based on the formation of a product which is insoluble in solvent and forms precipitate, by the reaction between the analyte and the titrant. A classic example is the reaction between Ag^+ and Cl^- to form the very insoluble salt AgCl.

General procedure of a titration:

In order to perform a titration, the basic requirements are a burette, a pipette and a conical flask. Before starting the titration, wash all the glass apparatus with tap water. Collect distilled water into wash bottles provided and rinse all the glassware with distilled water. Collect the solutions required for the titration into beakers. Rinse the burette with the solution to be taken into it. Fill the burette with the respective solution slightly above the zero mark. Slowly open the stopper screw of the burette, allow the solution out slowly and makeup the burette reading to zero mark by the lower meniscus of the solution level coincided with the zero mark. Care is to be taken to avoid air bubbles in the solution of the burette. Fix the burette to the stand at an appropriate height. Rinse the pipette with the solution to be taken into the conical flask. Take the solution up to the mark of the pipette and transfer quantitatively into the conical flask. Add the required reagents and indicator as given in the prescribed procedure and place the conical flask on the glazed tile. Gently shake the conical flask with simultaneous addition of titrant from the burette until the end point is reached. After getting end point, take the burette out of the stand and note down the reading with out parallax error. Through out all the contents of conical flask, wash it with tap water and rinse with distilled water. Repetition of titration is must in order to conform the accurate reading. After completing the experiment, wash all the apparatus with tap water. Place all the apparatus and reagent bottles in their respective places after their use.

<u>Precautions</u>:

1. Do not measure acids with pipette.
2. Do not waste the valuable distilled water
3. Do not place bags, note books or manual on the working table.
4. Handle all the glass apparatus with care and avoid payment of breakage fees.
5. During the titration, observe the changes taking place in the contents of conical flask instead of the burette reading.

VIVA – VOCE

1. **What is molarity?**

The number of moles of solute present in one litre of given solution is called molarity.

$$Molarity(M) = \frac{Wt}{M.wt} \times \frac{1}{V(\text{in Litres})}$$

2. What is normality?

The number of gram equivalents of solute present in one litre of given solution is called normality.

$$Normality(N) = \frac{Wt}{Eq.wt} \times \frac{1}{V(\text{in Litres})}$$

3. What is molality?

The number of moles of solute present in one kg of solvent is called molality.

$$Molarity(m) = \frac{Wt}{M.wt} \times \frac{1}{V(\text{in Kg})}$$

4. What is mole fraction?

The ratio of number of moles of one component to total number of moles of components in a solution is called mole fraction.

$$\text{Mole fraction of A} = \frac{n_1}{n_1 + n_2} \quad \& \quad \text{Mole fraction of B} = \frac{n_2}{n_1 + n_2}$$

5. What is meant by oxidation?

The phenomenon of loss of electrons (or) removal of hydrogens (or) addition of oxygens is called oxidation.

6. What is meant by reduction?

The phenomenon of gain of electrons (or) removal of oxygens (or) addition of hydrogens is called reduction.

7. What is meant by oxidizing agent?

The substance which undergoes reduction is called oxidizing agent.

8. What is meant by reducing agent?

The substance which undergoes oxidation is called reducing agent.

9. What is normal solution?

A normal solution of any substance contains one gram equivalent of the substance in a litre of the solution.

Experiment. No: 1 **Date:**

DETERMINATION OF THE STRENGTH OF UNKNOWN SOLUTION

Learning objectives:

- Select a suitable reaction for the estimation of acid/base by volumetric analysis
- Understand the role of standard solution
- Explore the significance of indicator(s)

Aim: To determine the strength of given NaOH solution

Chemicals required: A standard solution of 0.02N Na_2CO_3 and unknown (approximately 0.01N) HCl solution, Phenolphthalein, Methyl Orange Indicators.

Apparatus required: Burette, Pipette, Beakers and Conical flask.

Theory:

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

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

Procedure:

Part A: Standardization of HCl solution: In this titration an unknown HCl solution is titrated against standard Na_2CO_3 solution.
Rinse and fill the burette with the given HCl solution. Take 10 ml of Na_2CO_3 in a conical flask with the help of pipette and add two drops of methyl orange indicator. The colour of solution turns pale yellow. Titrate it with the HCl solution till a reddish pink colour is obtained. Repeat the titrations to get successive concordant readings.

Observations:
Part A: Standardization of HCl solution:

S. No.	Volume of the solution taken in conical flask (ml)	Burette Readings		Volume of the HCl solution used. (ml)
		Initial reading (ml)	Final reading (ml)	
1.				
2.				
3.				

Volume of the acid used = ---------- ml

Use Normality equation and explain the terms clearly.
Normality of HCl solution =

Calculation:

N_1V_1 (Na_2CO_3) = N_2V_2 (HCl)

Normality of HCl (N_2) =

Volume of HCl (V_2)=

Normality of Na_2CO_3 (N_1) =

Volume of Na_2CO_3 (V_1)=

Part B: Estimation of Strength of NaOH solution:

The given NaOH normality can be determined by titrating it with HCl (Known) using phenolphthalein indicator.

Transfer 10 ml of NaOH solution in a clean conical flask with the help of pipette. Add two drops of phenolphthalein indicator. The solution turns to pink. Titrate NaOH solution with the HCl solution till the pink colour disappears. Repeat the titrations to get successive concordant readings.

Part B: Estimation of NaOH solution:

S. No.	Volume of the solution taken in conical flask (ml)	Burette Readings		Volume of the HCl solution used (ml)
		Initial reading (ml)	Final reading (ml)	
1.				
2.				
3.				

Volume of the acid used = ---------- ml

Calculation:

N_2V_2 (HCl) = N_3V_3 (NaOH)

Normality of HCl (N_2) =

Volume of HCl (V_2)=

Normality of NaOH (N_3) =

Volume of NaOH (V_3)=

Normality of NaOH solution =

Strength of NaOH solution = Normality of NaOH x Eq. wt of NaOH (40)

=

=

Result:

The strength of the given NaOH solution = ---------- g/L

Review Questions:

1. HCl is a primary standard or a secondary standard? Explain?
2. Why anhydrous Na_2CO_3 is considered as primary standard?
3. How an exact N/10 solution of NaOH can be prepared?
4. Give the structure of phenolphthalein and Methyl orange?
5. Apply modern quinoid theory to explain the action of phenolphthalein and methyl orange?

---oo0oo---

Experiment. No: 2 **Date:**

Determination of alkalinity of water sample

Learning Objectives:

- Explore the alkalinity of various water samples
- Understand the species responsible for alkalinity
- Interpret the function of methyl orange and phenolphthalein indicators.

Aim: To determine alkalinity of a given water sample.

Apparatus required: Burette, Pipette, Conical flask, Measuring cylinder

Chemical required: Water sample, Unknown HCl solution (approximately 0.02N), 0.02N Na_2CO_3 solution, Phenolphthalein indicator, Methyl orange indicator.

Theory:

The knowledge of alkalinity of water is necessary for controlling the corrosion, in conditioning the boiler feed water (internally), for calculating the amounts of lime and soda needed for water softening and also in neutralizing the acidic solution produced by the hydrolysis of salts.

The alkalinity of water is due to the presence of hydroxide ion (OH^-), carbonate ion (CO_3^{2-}) and bicarbonate ion (HCO_3^-) present in the given sample of water. These can be estimated separately by titration against standard HCl solution, using phenolphthalein and methyl orange indicators. The chemical reactions involved are shown below:

1. $[OH^-] + [H^+] \rightarrow H_2O$ — P
2. $[CO_3^{2-}] + [H^+] \rightarrow [HCO_3^-]$ — P
3. $[HCO_3^-] + [H^+] \rightarrow H_2O + CO_2$ — M

The titration of the water sample against a standard acid upto phenolphthalein endpoint shows the completion of reactions (1) and (2) only. This amount of acid consumed corresponds to hydroxide and half of the carbonate present. Further, titration of the water sample against a standard acid (HCl) to methyl orange endpoint marks the completion of reactions (1), (2) and (3). Hence, the amount of acid consumed after the Methyl Orange endpoint corresponds to bicarbonate produced and already present. The total amount of acid consumed (Phenolphthalein & Methyl Orange) represents the total alkalinity (due to hydroxide, bicarbonate and carbonate ions).

The possible combinations of ions causing alkalinity in water are:

(i) OH^- only,

(ii) CO_3^{2-} only

(iii) HCO_3^- only.

(iv) OH^- and CO_3^{2-} together

(v) CO_3^{2-} and HCO_3^- together

(vi) The possibility of OH^- and HCO_3^- ions together is not possible since they combine to form CO_3^{2-} ions.

$$OH^- + HCO_3^- \rightarrow CO_3^{2-} + H_2O$$

Procedure:

Part A: Standardization of HCl solution: In this titration an unknown HCl solution is titrated against standard Na_2CO_3 solution.
Rinse and fill the burette with the given HCl solution. Take 10 ml of Na_2CO_3 in a conical flask with the help of pipette and add two drops of methyl orange indicator. The colour of solution turns pale yellow. Titrate it with the HCl solution till a reddish pink colour is obtained. Repeat the titrations to get successive concordant readings.

Observations:
Part A: Standardization of HCl solution:

S. No.	Volume of the solution taken in conical flask (ml)	Burette Readings		Volume of the HCl solution used (ml)
		Initial reading (ml)	Final reading (ml)	
1.				
2.				
3.				

Volume of the acid used = ---------- ml

Use Normality equation and explain the terms clearly.

Calculation:

N_1V_1 (Na_2CO_3) = N_2V_2 (HCl)

Normality of HCl (N_2) =

Volume of HCl (V_2)=

Normality of Na_2CO_3 (N_1) =

Volume of Na_2CO_3 (V_1)=

Normality of HCl solution =

Part B: Titration of water sample with HCl:

Rinse and fill the burette with standard HCl solution (from part A). Take 20 ml of water sample into a conical flask with the help of pipette. Add 2 drops of phenolphthalein indicator. The solution turns to light pink color. Titrate the water sample with N/50 HCl till the pink color just disappears. Now add 1-2 drops of methyl orange indicator to the same solution. The solution turns to yellow color. Continue the titration until red color is appeared. Note down the reading and repeat the experiment to get concordant readings.

Observation tables for alkalinity:

Part B: Titration of water sample with HCl:

S. No.	Volume of water sample (ml)	Initial reading (ml)	Burette Readings (Phenolphthalein)	Burette Readings (Total/ Methyl Orange)
			Final reading (ml)	Final reading (ml)
1.	20	0		
2.	20	0		
3.	20	0		

Calculations of Alkalinity:

Phenolphthalein Alkalinity in terms of $CaCO_3$ equivalents:

Concentration of water: N_2V_2 (HCl) = N_3V_3 (water)

Phenolphthalein Alkalinity in terms of $CaCO_3$ equivalents = Normality of water x Eq. wt. of $CaCO_3$ =N3*100= X

Phenolphthalein Alkalinity **(P)** = ----X----- x 1000 mg/L or ---------- x 1000 ppm.

Total/Methyl Orange Alkalinity in terms of $CaCO_3$ equivalents:

Concentration of water: N_2V_2 (HCl) = N_3V_3 (water)

Total Alkalinity in terms of $CaCO_3$ equivalents = Normality of water x Eq. wt. of $CaCO_3$

=

Total Alkalinity **(M)** = --------- x 1000 mg/L or ---------- x 1000 ppm.

Interpretation of Results:

P- and M-Alkalinity	Hydroxyl (OH)	Carbonate (CO_3)	Bicarbonate (HCO_3)
P = O	0	0	M
P < ½ M	0	2P	M – 2P
P = ½ M	0	M	0
P > ½ M	2P – M	2(M – P)	0
P = M	M	0	0

Results:

The alkalinity of the given water sample is……ppm.

Review Questions:

1. What is the significance of determining alkalinity/acidity of water?
2. How phenolphthalein/methyl orange indicator does function in titration?
3. Why phenolphthalein is not a suitable indicator for titrating a weak base like NH_4OH against a strong acid?
4. Why methyl orange cannot be used as indicator for titrating weak acid, like acetic acid against a strong base?
5. What is the pH range for phenolphthalein/methyl orange indicator?
6. What are the demerits of alkalinity/acidity in industry perspective?
7. How will you determine the alkalinity/acidity of soil sample?

Experiment No: 3 **Date: ……..…….**

ESTIMATION OF HARDNESS OF WATER BY COMPLEXOMETRY

Learning Objectives:

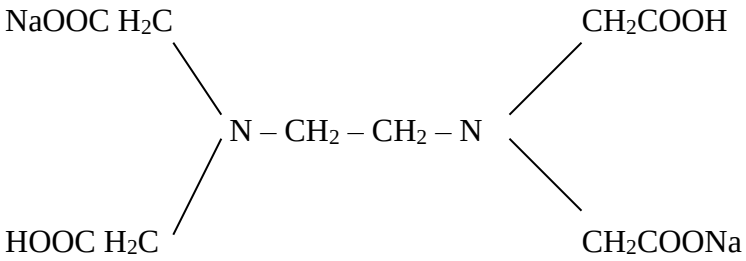
- Understand the concept of Complexometry
- Analyze different types of hardness
- Explore the importance of buffer solution & EBT indicator

Aim: To determine the hardness of water by complexometric titration.

Apparatus Required: Pipette, Burette, Conical flask, Beakers etc.,

Chemicals Required: 0.01M $ZnSO_4$, EDTA solution, Water sample, NH_3-NH_4Cl buffer solution, Erio-chrome Black T (EBT) indicator.

Theory: The method is based upon the fact that Ethylene diamine tetra acetic acid forms stable complex with all metals particularly with bi and polyvalent metals. For all practical purposes the hydrated disodium salt of the EDTA is used which has the following structure.



Initially when EBT indicator is added to the hard water sample in presence of buffer (pH 10) the solution turns wine red colour due to the formation of unstable Ca/Mg – EBT complex. Now this solution is titrated against standard EDTA solution until the blue colour appears (due to formation of stable complexes with Ca and Mg ions present in water).

M^{2+} + EBT ----------→ M- EBT
Wine- red complex

M^{2+} + EDTA ----------→ M- EDTA
Stable- colorless complex

M-EBT + EDTA ----------→ M-EDTA + EBT
Wine red Blue

(M = Ca or Mg)

Procedure: -

A) Standardization of EDTA:-

Transfer 10 ml of 0.01M $ZnSO_4$ solution into a conical flask with the help of pipette. Add 2 ml of Ammonia and Ammonium chloride buffer solution and 2 drops of EBT indicator. The solution turns wine red colour. Titrate with EDTA from the burette till the colour changes to blue. Note the final burette reading. Repeat the procedure until successive concordant readings are obtained.

Observations and Calculations: -

A) Standardization of EDTA: -

Molarity of $ZnSO_4$ solution $M_1 = 0.01M$

S. No.	Volume of $ZnSO_4$ solution (V_1)	Burette Readings		Volume of EDTA solution (b-a), V_2
		Initial (a)	Final (b)	
1.	10 ml			
2.	10 ml			
3.	10 ml			

Molarity of EDTA solution = $ZnSO_4$ = EDTA

$M_1V_1 = M_2V_2$

Calculation:

B) **Determination of total hardness of water**:-

Transfer 20 ml of water sample into a conical flask with the help of measuring cylinder. Add 2 ml of Ammonia and Ammonium chloride buffer solution and 2 drops of EBT indicator. The solution becomes wine red in colour. Titrate with EDTA solution from the burette till the solution colour changes to blue. Note the final burette reading. Repeat the procedure until successive concordant readings are obtained.

Determination of Total hardness of water:-

Molarity of EDTA = M_2=

S. No.	Volume of water sample V_3	Burette Readings		Volume of EDTA solution (b-a) V_2
		Initial (a)	Final (b)	
1.	20 ml			
2.	20 ml			
3.	20 ml			

EDTA = Water sample

$M_2V_2 = M_3V_3$

$M_3 = M_2V_2/ V_3$

Total Hardness of water = (1000 X M_3 X 100) = ---------- ppm of $CaCO_3$

Calculation:

C) **Determination of Permanent Hardness**:

Transfer 20 ml of boiled water sample into a conical flask with the help of measuring cylinder. Add 2 ml of Ammonia and Ammonium chloride buffer solution and 2 drops of EBT indicator. The solution becomes wine red in colour. Titrate with EDTA solution from the burette till the solution colour changes to blue. Note the final burette reading. Repeat the procedure until successive concordant readings are obtained.

Observations table for permanent hardness:

S. No.	Volume of water sample V_4	Burette Readings		Volume of EDTA solution (b-a) V_2
		Initial (a)	Final (b)	
1.	20 ml			
2.	20 ml			
3.	20 ml			

EDTA = Water sample

$M_2V_2 = M_4V_4$

$M_4 = M_2V_2 / V_4$

=

Permanent water Hardness of water = (1000 X M_4 X 100) = ---------- ppm of $CaCO_3$

Temporary hardness = Total hardness – Permanent hardness=

Results:

Total hardness of given water sample is ……..ppm of $CaCO_3$

Permanent hardness of the given water sample is……ppm of $CaCO_3$

Temporary hardness of the given water sample is……ppm of $CaCO_3$

Review Questions:

1. What is hardness of water?
2. What are soaps? Why hard water does not lather with soap?
3. What are carbonate and non-carbonate hardness?
4. How hardness of water is expressed?
5. What are the various units of hardness and how they are related to each other?
6. What is EDTA? In what form EDTA is used? Give its structure?
7. How does EBT acts as an indicator?
8. What are the disadvantages of hard water?

Experiment No: 4 **Date:**

DETERMINATION OF TOTAL RESIDUAL CHLORINE IN WATER

Learning Objectives:

- Understand the role of chlorine in water disinfection
- Identify the method to test the amount of free chlorine in the water

Aim: To determine the amount of total residual chlorine present in the given water sample

Apparatus Required: Burette, Pipette, Conical flask, Measuring cylinder, Beakers etc.,

Chemicals Required: CH_3COOH, KI, Starch indicator, $Na_2S_2O_3$ solution, 0.025 N $CuSO_4$ solution.

Theory:

Chlorination of water supply is done to destroy or deactivate disease-producing micro-organisms. It will also improve the quality of water by reacting with ammonia, iron, manganese, sulphide and some organic substances. The residual chlorine is maintained in water to promote the primary purpose of chlorination. This method of determination depends upon the oxidizing power of free and combined chlorine residuals. Chlorine will liberate free iodine from potassium iodide solution at pH 8 or less. The liberated iodine is titrated against standard sodium thiosulphate solution using starch indicator.

$$Cl_2 + 2KI \longrightarrow 2KCl + I_2$$
$$I_2 + 2Na_2S_2O_3 \longrightarrow Na_4S_4O_6 + 2NaI$$

Procedure:

PART-A: Standardization of $Na_2S_2O_3$: Take 10 ml of 0.025 N $CuSO_4$ in conical flask. Add 5ml of 10% KI and mix thoroughly till dark brown colour appears. Titrate with sodium thio sulphate solution until a pale yellow colour is obtained. Add 1mL of freshly prepared 1% starch solution and continue the titration until blue colour disappears. Repeat the experiment until successive concordant readings are obtained.

S. No.	Volume of the $CuSO_4$ solution V_1 (ml)	Burette Readings		Volume of $Na_2S_2O_3$ V_2 (ml)
		Initial (a)	Final (b)	
1.				
2.				
3.				

Calculation: $CuSO_4$ $Na_2S_2O_3$

$N_1V_1 = N_2V_2$

$N_2 = N_1V_1/V_2$

Normality of $Na_2S_2O_3$ solution (N_2) =

PART-B: Take 20 ml of sample in conical flask. Add 5 ml acetic acid to bring pH in the range 3–4. Add 5ml of 10% KI and mix thoroughly till dark brown colour appears. Titrate with standard sodium thio sulphate solution until a pale yellow colour is obtained. Add 1mL of freshly prepared 1% starch solution and titrate until the blue colour disappears. Repeat the experiment until to get successive concordant readings.

OBSERVATION AND CALCULATIONS:

Chlorinated sample vs. Standard sodium thiosulphate solution (0.025 N)

S.No.	Volume of the water sample (ml)	Burette Readings		Volume of titrant (ml)
		Initial (a)	Final (b)	
1.				
2.				
3.				

Calculation: Hypo solution Ware sample

$N_2V_2 = N_3V_3$

$N3 = N_2V_2/V_3$

Amount of Residual Chlorine= N_3 x 35.5 x 1000 = mg/L (or) ppm

Result:

Amount of total residual chlorine present in given water sample =……mg/L (or) ppm

Experiment. No: 5 **Date:**

DETERMINATION OF THE RATE CONSTANT OF A REACTION.

AIM:

To determine the rate constant of the hydrolysis of Ethyl acetate using an acid as a catalyst.

PRINCIPLE:

The hydrolysis of an ester occurs according to the equation

$$CH_3COOC_2H_5 + H_2O \longrightarrow CH_3COOH + C_2H_5OH$$

This reaction follows pseudo first order kinetics.

PROCEDURE:

100 ml of 0.5 N HCl is taken in a clean dry conical flask. 5 ml of ester is pipetted out into the conical flask and the mixture is immediately withdrawn into another dry conical flask. A stopwatch is started simultaneously. The reaction is then arrested by the addition of ice cubes and the mixture is titrated against 0.2 N NaOH using phenolphthalein as indicator. End point is the appearance of permanent pink colour. The volume of NaOH consumed in this titration is taken as V_0.

5 ml of acid – ester mixture is similarly withdrawn after 10, 20, 30, ..., 60 minutes respectively and titrated against NaOH using phenolphthalein as indicator. The volume of NaOH consumed for each of the above time intervals (t), is taken as V_t.

The contents are transferred into boiling tube with a cap and heated in a water bath for about 15 minutes. 5 ml of this mixture is withdrawn and titrated against NaOH to get V_∞.

CALCULATION:

The rate constant K is determined using the equation,

$$K = \frac{2.303}{t} \log \frac{(V_\infty - V_0)}{(V_\infty - V_t)}$$

Rate constant is also determined graphically by plotting $\log (V_\infty - V_t)$ Vs **time**.

TABULATION:

S.No.	Time Min	Volume of NaOH ml	$(V_\infty - V_t)$ ml	$log\ (V_\infty - V_t)$ ml	$K = \frac{2.303}{t} \log \frac{(V_\infty - V_0)}{(V_\infty - V_t)}$ min^{-1}
1	0				
2	10				
3	20				
4	30				
5	40				
6	50				
7	60				
8	∞				

RESULT:

The Rate Constant for the hydrolysis of an ester from

1. Calculated value =
2. Graphical value =

Experiment. No: 6 **Date:**

DETERMINATION OF DISSOLVED OXYGEN IN THE GIVEN WATER SAMPLE

Learning Objectives:

- Understand the concept of Iodometry
- Explore the importance of different reagents in Iodometry

Aim: To determine the amount of dissolved oxygen in the given water sample.

Apparatus: Burette, Pipette, Conical flask, Measuring cylinder, Beakers etc.

Chemicals: $Na_2S_2O_3$, $MnSO_4$ solution, 10% KI, Starch, Conc. H_2SO_4

Theory:

Dissolved oxygen (DO) is determined by Winkler's method or iodometric titration. The dissolved oxygen in water oxidizes KI and an equivalent amount of Iodine is liberated. This Iodine is titrated against a standard Hypo solution. However, since dissolved oxygen in water is in molecular state and is not capable of reacting with KI, therefore an oxygen carrier such as Manganese hydroxide is used.

$MnSO_4 + 2\ NaOH \longrightarrow Mn(OH)_2 + Na_2SO_4$

$Mn(OH)_2 + ½\ O_2 \longrightarrow MnO(OH)_2$

$MnO(OH)_2 + H_2SO_4 \longrightarrow MnSO_4 + (O) + 2H_2O$

$(O) + 2KI + H_2SO_4 \longrightarrow K_2SO_4 + H_2O + I_2$

$I_2 + I^- \longrightarrow I_3^-$

$S_2O_3^{2-} + I_3^- \longrightarrow 3I^- + S_4O_6^{2-}$

Procedure:

PART-A: Standardization of $Na_2S_2O_3$: Take 10 ml of 0.025 N $CuSO_4$ in conical flask. Add 5ml of 10% KI and mix thoroughly till dark brown colour appears. Titrate with sodium thio sulphate solution until a pale yellow colour is obtained. Add 1mL of freshly

prepared 1% starch solution and continue the titration until blue colour disappears. Repeat the experiment until successive concordant readings are obtained.

S. No.	Volume of the $CuSO_4$ solution (ml)	Burette Readings		Volume of $Na_2S_2O_3$ (ml)
		Initial (a)	Final (b)	
1				
2				
3				
4				

Calculation: $CuSO_4$ $Na_2S_2O_3$

$$N_1V_1 = N_2V_2$$

$$N_2 = N_1V_1/V_2$$

Normality of $Na_2S_2O_3$ solution (N_2) =

PART –B: Determination of Dissolved Oxygen of Water sample:

200 mL of water sample is transferred in Iodine flask. Add 1mL of $MnSO_4$ solution to it by means of pipette. Add 2mL of alkaline KI solution. Stopper the bottle and shake thoroughly. Allow the brown precipitate of manganic hydroxide formed, to settle down. Add 2 ml of conc. H_2SO_4 with the help of pipette and mix till the precipitate is completely dissolved. Transfer 50 mL of the above solution into a flask with the help of measuring cylinder. Titrate the liberated iodine with standardized sodium thiosulphate solution until the sample solution becomes pale yellow. Add 1 ml of freshly prepared 1% of starch solution. The solution will turn blue. Continue titration till the blue colour disappears. Repeat the titration until to get concordant readings.

Observation Table

S. No.	Volume of the solution taken in the titration flask (ml)	Burette Readings		Volume of the HYPO used
		Initial (a)	Final (b)	
1				
2				
3				
4				

Calculation: $Na_2S_2O_3$ Water sample

$N_2V_2 = N_3V_3$

$N_3 = N_2V_2/V_3$

Normality of water sample (N_3) =

Strength of dissolved oxygen in water = Concentration of water sample x eq. weight

= ---------- x 8 gms per litre ×1000

= ---------- ppm

Result:

The amount of dissolved oxygen in water = …… ppm

Review Questions:

1. State Henry's law for the solubility of a gas in a liquid? Outline the conditions under which the law is applicable?
2. Is dissolved oxygen necessary?
3. What is the effect of the presence of oxidizing impurities like NO_2^- and Fe^{3+} (if not removed) on the DO results?
4. How excess of Fe^{3+} can be removed?

5. How do reducing impurities such as Fe^{2+}, SO_3^{2-} and S^{2-} effect the DO determination?
6. Why starch solution is added near the end point?
7. Why is the $Na_2S_2O_3$ solution of N/40 strength selected for titration?
8. What are iodometric titrations?
9. Give the ionic equation showing reducing property of sodium thiosulphate?
10. With the help of above equation, find out the equivalent weight of sodium thiosulphate?

Experiment. No: 7 **Date:**

ESTIMATION OF AMOUNT OF IRON USING POTENTIOMETRIC METHOD

Learning objectives:

- **To Understand about potentiometer**
- **To Learn about electrode setup**
- **Redox reaction**

Principle: Potentiometric titrations depend on measurement of emf between reference electrode and an indicator electrode. When a solution of ferrous iron is titrated with a solution of p[otassium dichromate, following redox reaction takes place:

$$\mathbf{6Fe^{2+} + Cr_2O_7^{2-} + 14\ H^+ \rightarrow 6Fe^{2+} + Cr^{3+} + 7H_2O}$$

During this titration Fe^{2+} ion is converted into Fe^{+3}, whose concentration increases. At the end point, there will be sharp change due sudden removal of all Fe2+ ions.

The cell is set up by connecting this redox electrode with calomel electrode as shown below:

Pt, Fe^{2+}, Fe^{3+} // KCl, $HgCl_2$, Hg

A graph between emf measured against the volume of K2Cr2O7 added is drawn and the end point is noted from the graph.

Procedure: The given ferrous solution is made up in a 100ml standard flask. Standard potassium dichromate solution is filled in the burette up to the mark.

20 ml of ferrous solution is pipette out in to a 100ml beaker. 10 of dilute sulfuric acid and 20 ml of distilled water are added. A platinum electrode and a calomel electrode are dipped into this solution and connected to a potentiometer. Then 1 ml of $K_2Cr_2O_7$ is added to the solution and stirred well for 30 s. The emf is measured and titration is continued by adding $K_2Cr_2O_7$ in 1ml increments till 5 measurements after the end point.

A graph is drawn by plotting the emf against the volume of titrant added and the point range is fixed. A first derivative graph is also plotted between $\Delta E/\Delta V$ and volume of titrant added.

Observation table:

S.No	Volume of titrant (ml)	Potential (V)	ΔE/ΔV
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			

Calculation: N_1V_1 ($K_2Cr_2O_7$) = N_2V_2 (Fe^{2+})

Strength = Normality × equivalent weight of Fe

Result: The strength the Fe^{2+} is ……..gm/l

Experiment. No: 8 **Date:**

CONDUCTOMETRC TITRATION OF STRONG ACID WITH STRONG BASE

Aim: To find out the amount of strong acid (HCl) by conductometry.

Apparatus: Burette, Pipette, Conical flask, Beakers.

Chemicals: N/10-NaOH, HCl (unknown).

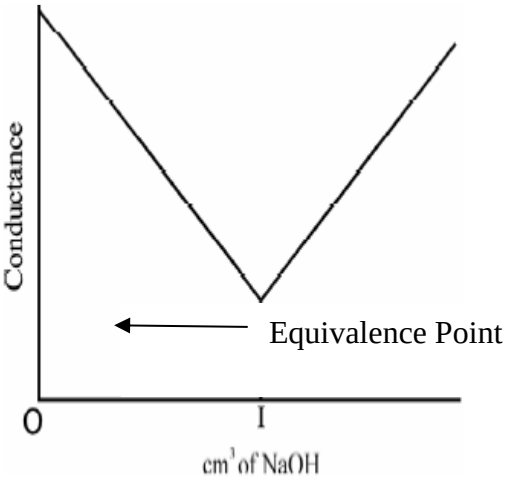
Instrument: Conductivity Bridge, Conductivity cell.

Principle: Conductometry can be used to detect the equivalence of a titration. This method is based upon the measurement of conductance during the course of titration. The conductance varies differently before and after the equivalence point. This is due to the reason that electrical conductance of a solution depends upon the ionic mobilities of ions present in the solution.

The conductance of acid solution is noted initially as well as after successive additions of small volume of NaOH solution. Conductance of acid solution in the beginning is high due to presence of highly mobile H^+ ions. On adding NaOH solution, the H^+ ions are replaced by slow moving of Na^+ ions decreasing the conductance of solution.

$$[H^+ + Cl^-] + [Na^+ + OH^-] \rightarrow Na^+ + Cl^- + H_2O$$

When neutralization is complete further addition of NaOH will cause the conductance to increase due to excess of highly mobile OH^- ions. The conductance is thus the minimum at the equivalence point. Thus if conductance values are plotted against the volume of NaOH added, "V" shape curve is obtained.



Procedure: Rinse and fill the burette with NaOH. Pipette out 50ml of given unknown HCl solution in to a 100 ml beaker. Immerse the conductivity cell in the solution so that the electrodes completely dip in solution. Select the proper conductance range and put the adjuster to the measurement position. Now note down the initial conductance of the solution. From the burette add 1 ml of 0.1 N NaOH, stir the solution with the help of glass rod carefully and note down the conductance of mixed solution. Keep on adding NaOH solution in 1 ml lots and note down the conductance value till 14 to 16 ml of solution is added. Plot the graph between observed conductance values along Y-axis against the volume of NaOH added along X-axis. The point of intersection gives the amount of NaOH required for complete neutralization of HCl.

Observation Table:

S.No	Volume of HCl taken (ml)	Volume of NaOH added (ml)	Observed conductance (ohm^{-1})
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			

Calculation: N_1V_1 **(NaOH) =** N_2V_2 **(HCl)**

Amount of HCl= N_2 **× 36.5=**

Result: The amount of HCl present in the given sample is……g/l.

Experiment. No: 9 **Date:**

ESTIMATION OF AMOUNT OF AN ACID USING pH- METRIC METHOD

Learning objectives:

- **To understand the concept of pH**
- **To understand how pH-meter works**
- **To learn about electrodes involved**

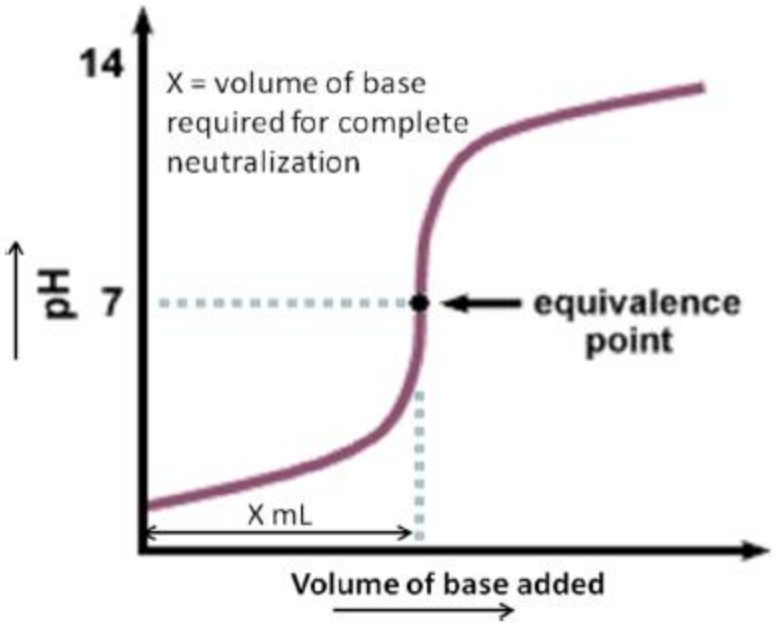
Apparatus: Electrodes, beaker, pipette, burette ctc.

Chemicals: Hydrochloric acid (HCl), sodium hydroxide (NaOH). buffer of pH = 4 and 9.2.

Instrumentation: pH-meter

Theory: Most of the chemical and biochemical processes are profoundly affected by the acidity or alkalinity of the medium in which the reaction takes place. All acid dissociate in aqueous solution to yield H^+ ions. Some acids like HCl, H_2SO_4, HNO_3 etc. are completely ionized in aqueous medium where as CH3COOH, HCOOH etc. ionize to a small extent only. The former is known as strong and the later as weak acid. pH of any solution is defined as ($-\log H^+$) and has values between 0–14. pH < 7 indicate acidic solution, pH > 7 indicate basic solution and pH = 7 means neutral solution. The pH of a solution can be measured accurately with the help of a pH meter. Measurement of pH is employed to monitor the cause of acid-base titration. The pH values of the solution at different stage of acid–base neutralization are determined and plotted against the volume of alkali added on adding a base to an acid, the pH rises slowly in the initial stages as the concentration of H^+ ion decreases gradually. But, at the equivalence point, it increases rapidly as at the equivalent point H^+ ion concentration is very small. Then it flattens out after the end point. The end point of the titration can be detected where the pH value changes most rapidly. However, the shape of the curve depends upon the ionisability of the acid and the base used and also

on the acidity of base and basicity of the acid.



Procedure:

0.1(N) NaOH solution is provided.

HCl solution of unknown strength is provided (100ml HCl).

Calibration of pH: Switch on the instrument and wait for 10–15 minutes so that machine gets warmed up. Prepare the buffer solution by adding buffer tablets of pH = 4 and pH = 9.2 in 100 mL of water separately. Wash the electrode with distilled water. Then, dip the electrode in the buffer solution (pH = 4) taken in a beaker, so that the electrode immersed to the solution properly. Measure the temperature of the solution and set the temperature compensate control accordingly. Set the pointer to pH = 7 exactly means of set = 0 control. Put the selector switch to proper pH range 0–7 (as the buffer pH = 4). So the pointers to the known pH value of the buffer by burning the set buffer control. Put back the selector at zero position. Wash the electrode with distilled water and standardize the pH meter using basic buffer solution pH = 9.2.

pH-metric titration: Clean the electrode with distilled water and wipe them with tissue paper or filter paper. Take 20 mL of HCl and about 40 ml distilled water in a 100 mL beaker and immerse the electrode in it. Set the burette with NaOH solution. The reading shown on the scale of pH meter is pH value of the HCl solution. Add NaOH solution drop wise from the burette (maximum 1.0 mL at a time), shake the solution well and note the corresponding

pH values. Near the end point, volume of NaOH added should be as small as possible because the acid is neutralized and there will a sharp increase in pH values. Further addition of even 0.01 mL of NaOH, increase the pH value to about 9–10. Now plot the graph between pH values and volume of titrant added. S shape curve is obtained. To calculate the exact equivalence point from the graph, derivative curve is plotted between ΔpH/ΔV and volume of titrant added.

Observation table:

S.No	Volume of NaOH added	pH values	ΔpH/ΔV
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			
18			

Calculation: N_1V_1 **(NaOH) =** N_2V_2 **(HCl)**

Amount of HCl= $N_2 \times 36.5=$

Result: The amount of HCl present in the given sample is……..g/l

Experiment. No: 10 **Date:**

SYNTHESIS OF POLYMER (UF RESIN)

Learning Objectives:

To learn about:

- Cross-linked polymers
- Monomers required for the preparation of U-F resin
- Condensation reaction

Aim:

To prepare Urea- formaldehyde resin.

Chemicals Required:

Formaldehyde (40%), Urea and Conc. H_2SO_4.

Theory:

Amino resins are condensation products obtained by the reaction of urea or melamine with formaldehyde. Commercially important amino resins are Urea-formaldehyde, Melamine formaldehyde which are formed by the condensation polymerization reaction of Urea with aqueous Formaldehyde and Melamine with formaldehyde respectively.

Reaction:

H_2N –CO-NH_2 + 2HCHO ----------→ U-F resin

Procedure:

Take 20 ml of 40% Formaldehyde solution in a 100 ml beaker. Add about 10 gms of urea while stirring until a saturated solution is obtained. Add a few drops of Conc. H_2SO_4 stirring continuously during the addition. All of a sudden a voluminous white solid mass appears in the beaker. When the reaction is complete wash the residue with water and dry the product and calculate the yield of the product formed.

Note:

The reaction is sometimes vigorous and it is better to be little away from the beaker while the addition of the Conc.H_2SO_4 and until the reaction is complete.

Result:

Yield = ……..

Review Questions:

1. What are resins?
2. What is Bakelite? What are the monomers of it?
3. Define thermoplastic polymer and thermosetting polymer?
4. What are the monomers of U-F resin?
5. Preparation of U-F resin undergoes what type of polymerization?

Experiment. No: 11 **Date:**

ESTIMATION OF $KMnO_4$ USING STANDARD OXALIC ACID (PERMANGANOMETRY)

Aim: To estimate the amount of Potassium permanganate using standard oxalic acid.

Requirements: Standard sodium carbonate solution, Oxalic acid solution, Potassium permanganate solution, methyl orange and 10% sulphuric acid solution.

Chemical Equations:

$$Na_2CO_3 + (COOH)_2 \rightarrow (COONa)_2 + CO_2 + H_2O$$

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

Theory:

Oxidometry involves oxidation – reduction reactions associated with transfer of electrons. In a reaction of this type, the oxidizing agent gains electrons and is reduced and the reducing agent loses electrons and is oxidized. This exchange of electrons leads to changes in the valence of the corresponding atoms or ions. The valence of an oxidized atom or ion is increased and the valence of a reduced atom or ion is decreased.

The permanganate method is based on reactions of oxidation by the permanganate ion. In acid solutions **$KMnO_4$ acts as an oxidizing agent**. The septivalent (+7) manganese in it is reduced to Mn^{2+} cations and manganese salt is formed.

Standard solution of oxalic acid is used to standardize potassium permanganate solution. When oxalic acid is titrated with potassium permanganate the reaction is

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

Here $C_2O_4^{2-}$ ions are oxidized as $C_2O_4^{2-} - 2e^- \rightarrow 2CO_2$

This reaction hardly proceeds at room temperature and so to make the reaction proceed at an adequate rate this titration should be performed at 60 to 70° C. No indicator is needed in permanganate titrations. The pink color of the Mn^{2+} ion will give the color change. When all the reducing agent has been titrated a single excess drop of permanganate colors the whole solution as distinct pink.

Procedure:

Part-I:

Standardization of Oxalic acid:

Pipette out 10ml of oxalic acid solution in to a conical flask. Add few drops of **methyl orange** indictor. The solution turns pink. Fill the burette with standard sodium carbonate solution. Titrate oxalic acid solution against sodium carbonate solution until the color of the solution changes from **pink to yellow**. This is the endpoint of the titration.

Part-II:

Estimation of potassium permanganate solution:

Pipette out 10ml of standard oxalic acid solution into a conical flask and add 5ml of 10% sulphuric acid. Heat the solution to about 70^0 C, (do not allow it to boil, because oxalic acid decomposes on boiling). Now, titrate this hot solution with $KMnO_4$ from burette. The first drop of $KMnO_4$ fades rather slowly. How ever as soon as a small amount of $MnSO_4$ (acts as catalyst in this reaction) is formed, subsequent fading is always instantaneous. The endpoint is obtained when one drop of permanganate colors the whole solution **pale pink which should remain for one minute**.

<u>Observations and Calculations</u>:

Part-1: Standardization of oxalic acid solution:

S.No.	Volume of oxalic acid solution (ml)	Burette reading (ml)		Volume of Sodium carbonate solution run down (ml)
		Initial	Final	

Concentration of oxalic acid solution is calculated from the expression,

$$\frac{V_1M_1}{n_1} = \frac{V_2M_2}{n_2}$$

Where

M_1 = Molarity of standard sodium carbonate solution = 0.02M

V_1 = Volume of standard sodium carbonate solution =

n_1 = No. of moles of standard sodium carbonate taken part in the reaction = 1

M_2 = Molarity of oxalic acid solution =?

V_2 = Volume of oxalic acid solution = 10ml

n_2 = No. of moles of oxalic acid taken part in the reaction = 1

.

$$\therefore M_2 = \frac{V_1M_1n_2}{n_1V_2}$$

=

=

Part2: Estimation of potassium permanganate:

S. No.	Volume of Oxalic acid solution (ml)	Burette reading (ml)		Volume of $KMnO_4$ Solution run down (ml)
		Initial	Final	

$$\frac{V_2 M_2}{n_2} = \frac{V_3 M_3}{n_3}$$

Where

M_2 = Molarity of oxalic acid solution =

V_2 = Volume of oxalic acid solution = 10ml

n_2 = No. of moles of oxalic acid solution taken part in the reaction = 5

M_3 = Molarity of potassium permanganate solution =?

V_3 = Volume of potassium permanganate solution =

n_3 = No. of moles of potassium permanganate taken part in the reaction = 2

$$\therefore M_3 = \frac{V_2 M_2 n_3}{n_2 V_3}$$

=

= –––––––– M

Amount of potassium permanganate present in one litre of solution

= Molarity (M_3) x Molecular weight

= x 158

= ––––––––––– g

Result:

Amount of potassium permanganate present in the given solution = _____ g/L

VIVA – VOCE

1. **Which type of titration is it?**
   This is a redox titration.
2. **What are the indicators used in this experiment?**
   In this experiment potassium permanganate it self acts as indicator, so it is also called as self indicator.
3. **What is the colour change and what is its reason?**
   The solution changes from colorless to pale pink which should retain for at least one minute. The extra drop of potassium permanganate after the end point gives pale pink colour.
4. **What are redox titrations?**
   The reactions which involve simultaneous oxidation and reduction are called redox reactions and titrations involving redox reactions are called redox titrations.
5. **What is permangnometry?**
   Redox titrations involving $KMnO_4$ as the oxidizing agent are called permangnometry.
6. **Why does $KMnO_4$ acts as self indicator?**
   $KMnO_4$ solution is purple in colour due to the presence of MnO_4^- ions. In presence of dil.H_2SO_4, it reacts with reducing agents and gets reduced to Mn^{2+} ions. So the colour disappears. At the end point, when all reducing agent has been oxidized, the excess drop of $KMnO_4$ added is not reduced and pink colour is observed in the solution.
7. **What is meant by auto catalyst and which substance acts as auto catalyst?**
   If one of the products it self acts as a catalyst then it is known as auto catalyst. Manganese sulphate ($MnSO_4$) acts as auto catalyst.
8. **What is the purpose of sulphuric acid?**
   Sulphuric acid is used to maintain the acidic medium, because redox titrations take place in acidic media.
9. **Why is dilute H_2SO_4most suitable as compared to HCl and HNO_3 in $KMnO_4$ titrations?**
   HCl reacts with $KMnO_4$ to liberate Cl_2 gas and consumes some $KMnO_4$. So, higher results are obtained. HNO_3 is a stronger oxidizing agent than $KMnO_4$. So it will oxidize Ferrous to ferric. So, lower results are obtained.
10. **What is an oxidizing agent?**
    The substance which undergoes reduction is called as oxidizing agent.
11. **What is a reducing agent?**
    The substance which undergoes oxidation is called as reducing agent.

Experiment. No: 12 **Date:**

ESTIMATION OF FERROUS AND FERRIC ION USING STANDARD K_2CrO_7

Aim: To estimate the amount of Ferrous ion in the given solution using potassium dichromate.

Requirements: Standard Mohr's salt solution, potassium dichromate solution, ferrous sulphate solution, 10% sulphuric acid and diphenylamine indicator.

Chemical Equation:

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

Theory:

Dichrometry titrations are based on oxidation reactions by dichromate ion. Its oxidizing action is due to conversion of $Cr_2O_7^-$ ions (containing Cr^{6+}) into Cr^{3+} ions.

$$Cr_2O_7^{2-} + 14H^+ + 6e^- \rightarrow 2Cr^{3+} + 7H_2O$$

Potassium dichromate acts as an oxidizing agent in the presence of sulphuric acid. It oxidizes Fe^{2+} ion and itself is reduced to green chromic salt.

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

To detect the end point, diphenylamine is used as indicator in the titration. Indicator used in oxidation – reduction titrations are known as oxidation – reduction or Redox indicators. Redox indicator change color when the oxidation potential of titrated solution reaches a define value. These indicators can be reversibly oxidized or reduced with different color in the oxidized and reduced forms.

$$\text{Indicator (ox)} + ne^- \rightarrow \text{Indicator (red)}$$

Diphenylamine changes from colorless to blue – violet between 0.73V and 0.79V. Effective potential range of Redox indicator must be within the limits of the sharp change of potential near the equivalence point.

Phosphoric acid is added to lower the Redox potential of Fe^{2+} - Fe^{3+} ion system so that abrupt potential change near the equivalence point nearly coincides with the potential range of the indicator.

Procedure:

Part-1:

Standardization of Potassium dichromate solution:

Pipette out 10ml of standard Mohr's salt solution into a conical flask and add 5ml of 10% H_2SO_4. Add **2 or 3 drops of diphenylamine**, titrate this solution with Potassium dichromate solution from a burette. With the titration the solution becomes green and just before the endpoint it is **bluish green** and at the endpoint it is **bluish violet**.

Part-2:

Estimation of Ferrous Ion:

Pipette out 10ml of ferrous sulphate solution in to a conical flask and add 5ml of 10% H_2SO_4 . Add **2 or 3 drops of diphenylamine** and titrate this solution with Potassium dichromate solution from a burette. With the titration the solution becomes green and just before the endpoint it is **bluish green** and at the end point it is **bluish violet**.

Observations and Calculations:

Part-1: Standardization of Potassium dichromate solution:

S.No.	Volume of standard Mohr's salt solution (ml)	Burette reading (ml)		Volume of $K_2Cr_2O_7$ Solution run down (ml)
		Initial	Final	

Concentration of potassium dichromate solution is calculated from the expression,

$$\frac{V_1M_1}{n_1} = \frac{V_2M_2}{n_2}$$

Where

M_1 = Molarity of standard Mohr's salt solution = 0.06M

V_1 = Volume of standard Mohr's salt solution = 10ml

n_1 = No. of moles of Mohr's salt taken part in the reaction = 6

M_2 = Molarity of potassium dichromate solution = ?

V_2 = Volume of potassium dichromate solution =

n_2 = No. of moles of potassium dichromate taken part in the reaction = 1

$$\therefore M_2 = \frac{V_1 M_1 n_2}{V_2 n_1} =$$

=

= ---------- M

Part2: Estimation of ferrous ion:

S.No.	Volume of ferrous sulphate solution (ml)	Burette reading (ml)		Volume of $K_2Cr_2O_7$ Solution run down (ml)
		Initial	Final	

$$\frac{V_2 M_2}{n_2} = \frac{V_3 M_3}{n_3}$$

Where M_2 = Molarity of potassium dichromate solution =

V_2 = Volume of potassium dichromate solution =

n_2 = No. of moles of potassium dichromate taken part in the reaction =

M_3 = Molarity of ferrous sulphate solution = ?

V_3 = Volume of ferrous sulphate solution = 10ml

n_3 = No. of moles of ferrous sulphate taken part in the reaction =

$$\therefore M_3 = \frac{V_2 M_2 n_3}{V_3 n_2} =$$

=

= ---------- M

Strength of ferrous ion present in one litre of solution

= Molarity (M_3) x Atomic weight

= x 56

=

Result:

Strength of ferrous ion present in the given solution = _____ g/L

VIVA – VOCE

1. **Which type of titration is it?**
   This is a redox titration.
2. **What is the indicator used in this experiment?**
   In this experiment Di phenyl amine acts as indicator and its formula is $(C_6H_5 - NH - C_6H_5)$.
3. **What is the colour change and what is its reason?**
   The solution changes from colorless to bluish violet. The redox potential of the indicator increases during the titration.
4. **What are redox titrations?**
   The reactions which involve simultaneous oxidation and reduction are called redox reactions and titrations involving redox reactions are called redox titrations.
5. **What is Dichrometry?**
   Redox titrations involving $K_2Cr_2O_7$ as the oxidizing agent are called Dichrometry.
6. **What is the purpose of sulphuric acid?**
   To maintain acidic medium, sulphuric acid is used because redox titrations are takes place in acidic media.
7. **What is the formula of Mohr's salt?**
   Mohr's salt: $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$

Experiment. No: 14 **Date:**

ESTIMATION OF AMOUNT OF VITAMIN- C IN THE GIVEN SAMPLE

Aim: Estimation of vitamin C in juices and real lemon using iodimetric method.

Requirements: Conical flask, burette, pipette, potassium iodide, potassium iodate, sulphuric acid, starch, vitamin C and Test solution.

Theory:

Many vegetables also contain large quantities of vitamin C, but ascorbic acid is commonly destroyed by many cooking processes, and hence citrus fruits are regarded as the most reliable source of vitamin C. Vitamin C can be determined in food by use of an oxidation-reduction reaction. The redox reaction is preferable to an acid-base titration because a number of other species in juice can act as acids, but relatively few interfere with the oxidation of ascorbic acid by iodine. The solubility of iodine is increased by complexation with iodide to form triiodide:

$$I_2(aq) + I^- \rightarrow I_3^-$$

Triiodide then oxidizes vitamin C to dehydro ascorbic acid:

$$C_6H_8O_6 + I_3^- + H_2O \rightarrow C_6H_6O_6 + 3I^- + 2H^+$$

The endpoint is indicated by the reaction of iodine with starch suspension, which produces a blue-black product. As long as vitamin C is present, the triiodide is quickly converted to iodide ion, and no blue-black iodine-starch product is observed. However, when all the vitamin C has been oxidized, the excess triiodide (in equilibrium with iodine) reacts with starch to form the expected blue-black color.

Procedure:

Part – 1:

Standardization of the iodine solution:

Add 10 ml of vitamin C solution into a conical flask. Add 10 drops of **1 % starch** solution. Rinse the burette twice with 5 -10 ml of iodine solution, and then fill it. Titrate the solution until the **solution turns to blue colour**. (That remains after at least 20 s of swirling). Record the final volume. Repeat this titration at least three times. Results should agree to 0.1 ml.

Part – 2:

Titration of test samples:

Add 10 ml of your beverage sample into a conical flask. Add 10 drops of **1 % starch** solution. Rinse the burette twice with 5 -10 ml of iodine solution, and then fill it. Titrate the solution until the **solution turns to blue colour**. (That remains after at least 20 s of swirling). Record the final volume. Repeat this titration at least three times. Results should agree to 0.1 ml.

Observations and Calculations:

Part-1: Standardization of iodine solution:

S.No.	Volume of vitamin C solution (ml)	Burette reading (ml)		Volume of iodine Solution run down (ml)
		Initial	Final	

$$\frac{V_1 M_1}{n_1} = \frac{V_2 M_2}{n_2}$$

Where

M_1 = Molarity of standard Vitamin C solution =0.0028M

V_1 = Volume of standard Vitamin C solution =10 ml

n_1 = No. of moles of standard Vitamin C taken part in the reaction =1

M_2 = Molarity of Iodine solution =?

V_2 = Volume of iodine solution =

n_2 = No. of moles of Iodine taken part in the reaction =1

. $\therefore M_2 = \frac{V_1 M_1 n_2}{n_1 V_2}$

=

=

Part-2: Estimation of vitamin C:

S. No.	Volume of test solution (ml)	Burette reading (ml)		Volume of iodine Solution run down (ml)
		Initial	Final	

$$\frac{V_2 M_2}{n_2} = \frac{V_3 M_3}{n_3}$$

Where

M_2 = Molarity of Iodine solution =

V_2 = Volume of iodine solution =

n_2 = No. of moles of Iodine taken part in the reaction =1

M_3 = Molarity of test Vitamin C solution =?

V_3 = Volume of test Vitamin C solution = 10ml

n_3 = No. of moles of test Vitamin C solution taken part in the reaction =1

$$\therefore M_3 = \frac{V_2 M_2 n_3}{n_2 V_3}$$

=

= –––––––– M

Amount of ascorbic acid present in the given sample = Molarity (M_3) x Mol. Wt (176)

= x

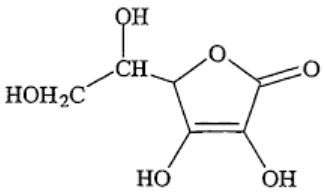
=

Result:

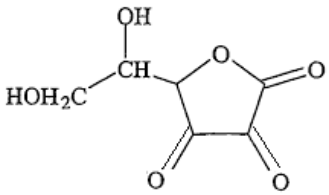
The amount of ascorbic acid present in the given sample = –––––––––– gm/lit

VIVA - VOCE

1. **What is the formula of Vitamin C?**
   The chemical name of vitamin C is Ascorbic acid. Its formula is $C_6H_8O_6$.
2. **What do you understand by iodimetric titrations?**
   Iodimetric titrations are defined as that iodine titration in which a standard iodine solution is used as an oxidant and iodine is directly titrated with a reducing agent.
3. **Which indicator is used in this titration and what is the colour at the end point?**
   Freshly prepared starch solution is used as an indicator. Appearance of blue colour is the end point of this titration.
4. **What is the reason for blue colour at the end point?**
   Blue colour is due to the formation of starch iodide complex.
5. **What is the formula of starch?**
   The chemical formula of starch is $(C_6H_{10}O_5)_n$.
6. **How do you prepare the iodine solution?**
   Iodine solution is prepared by dissolving KI, KIO_3 and H_2SO_4.
7. **What are the structures of ascorbic acid and dehydro ascorbic acid?**



Ascorbic Acid



Dehydroascorbic acid

Experiment. No: 15 **Date:**

DETERMINATION OF IRON BY A COLORIMETRIC METHOD USING THIOCYNATE AS REAGENT

Aim: To determine amount of iron present in a given solution using thiocyanate as reagent by a colorimetric method.

Requirements:
Colorimeter, Ammonium thiocyanate, Ferric ammonium sulpahte, Sulphuric acid, test tubes and Pipettes

Theory:

Iron is one of the many minerals required by the human body. It is used in the manufacture of the oxygen-carrying proteins, haemoglobin and myoglobin. A deficiency of iron in the body can leave a person feeling tired and listless and can lead to a disorder called anemia. Many of the foods we eat contain small quantities of iron.

In this analysis the iron present in an iron tablet (dietary supplement) or a sample of food is extracted to form a solution containing Fe^{3+} (ferric) ions. To make the presence of these ions in solution visible, thiocyanate ions (SCN^-) are added. These react with the Fe^{3+} ions to form a blood-red colored complex:

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

Principle:

Colorimeter measures the optical density of an absorbing substance where

optical density is defined as $O.D = \log \frac{I_0}{I}$ --------- (1)

Where I_0 = Intensity of incident light

I = Intensity of transmitted light

As per Beer's law, optical density of an absorbing substance is related to the concentration by the equation.

$$O.D = E.C.l$$

$$O.D = (E.l).C \text{ -------- (2)}$$

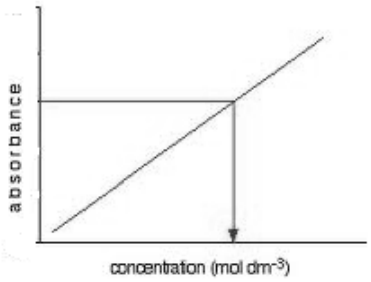
Where 'C' is the concentration of the substance, l is the path length, which represents the width of the cell used and is constant for a given cell used, E is the molar absorption coefficient and is a constant for given substance. Equation 2 may be written as $O.D \alpha C$ ------- (3)

Equation 3 represents the quantitative form of Beer's law, if the optical density of a substance is determined at varying concentration. A plot of O.D Vs C gives a straight line.

Graph:

A graph is drawn with concentration of ferric ions on X- axis and absorbance on Y-axis. A straight line passing through origin is obtained and it proves that it is in accordance with Beer-Lambert's law. Now the point corresponding to the absorbance of

unknown iron solution is marked in the graph and the concentration of iron ion corresponding to this absorbance is determined by drawing a vertical line to X-axis. The amount of iron in mg present in the given solution is calculated by using this concentration of iron ion noted from graph.



Procedure:

A standard solution of ferric ammonium sulphate is prepared and definite varying volumes of it are taken in a series of beakers to prepare sample solution of varying concentrations. Pipette out 10ml of each solution into series of boiling tubes and add 10 ml of ammonium thiocyanate solution to each iron solution taken in the boiling tubes, with 2 minutes time gap between each addition. These additions are carefully timed so that all samples react for the same period of time. The solutions are thoroughly mixed by swirling when a stable red colour will appear over the next few minutes. Take the solution in a cuvette after 15 minutes of adding thiocyanate and measure the absorbance at a wavelength of 490 nm for each colored solution. Repeat the procedure with all the samples in the test tubes. The measured absorbance of light is a direct measure of intensity of the colour of solution.

Now 10 ml of the test iron solution is accurately measured into a clean, dry boiling tube. This measurement is most accurately made using a 10 ml pipette. 10 ml of thiocyanate solution is added to this iron solution and transferred to the cuvette of the colorimeter and absorbance is measured by fixing the wavelength.

Observations:

Concentration of the standard iron solution =
Wavelength = 490 nm

S. No.	Concentration of ferric ion solution	Absorbance

Unknown iron solution O.D = ----------

Concentration of iron solution = ---------- M

Amount of iron present in one litre of given solution = Concentration x Atomic weight

= x 56

= ---------- g

Result:

Amount of iron present in the given solution = _____ g/L

VIVA – VOCE

1. **State Beer – Lambert's law.**
   The absorbance (A) is directly proportional to the molar concentration (C) as well as path length (l).
   i.e., A α Cl => A = ε Cl where ε is molar absorptivity co-efficient.
   Mathematically, the law can also be stated as $I_t = I_0 10^{-\varepsilon Cl}$
2. **What is colorimetry?**
   The method of analysis which involves the measurement of absorption of light radiations in the visible region of the spectrum is called colorimetry.
3. **What is the basis of colorimetry?**
   The variation of the colour of the solution with change in concentration of the ions forms the basis of colorimetry.

4. **What is the difference between a colorimeter and spectrophotometer?**
   A colorimeter determines the concentration of a substance by measurement of relative absorption of light w.r.t a known of a substance. In such instrument, the absorption in the visible region is generally employed.

   Spectrophotometer is an absorption meter, which employs by a much narrow band of wavelengths as produced by monochromatic source of light. Such an instrument can be made to operate in the UV, visible and I.R. region, using suitable source of radiation energy.
5. **What is the colour of the complex and what is the reason for the formation of complex?**
   Blood-red colored complex is formed due to the reaction of thiocyanate ions (SCN^-) with the Fe^{3+} ions.

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

6. **What is the purpose of adding thiocyanate?**
   To make the presence of Fe^{3+} (ferric) ions in solution visible, thiocyanate ions (SCN^-) are added.