



# LORDS INSTITUTE OF ENGINEERING AND TECHNOLOGY

(Approved by AICTE, Accredited by NAAC 'A' Grade & NBA, Affiliated to Osmania University)

(AUTONOMOUS)

Department of Sciences & Humanities



## ENGINEERING CHEMISTRY LAB MANUAL

B.E- I Year

(WITH EFFECT FROM ACADEMIC YEAR 2023-2024)

DEPARTMENT OF CHEMISTRY



Student Name:	
Roll no:	
Course:	
Year/Sem:	
Branch:	

### **GENERAL PRECAUTIONS TO BE TAKEN IN THE LABORATORY:**

1. Never work in the laboratory unless a demonstrator or teaching assistant is present.
2. Do not throw waste such as match stems filter papers etc. into the sink. They must be thrown into the waste jars.
3. Keep the water and gas taps closed expect when these utilities are needed.
4. Never taste any chemical unless instructed to do so and don't allow chemicals to come in contact with your skin.
5. While working with gases, conduct the experiment in a fume hood.
6. Keep all the doors and windows open while working in the laboratory.
7. You should know about the hazards and properties of every chemical which you are going to use for the experiment. Many chemicals encountered in analysis are poisonous and must be carefully handled.
8. Sulphuric acid must be diluted only when it is cold .This should be done by adding it slowly to cold water with stirring, and not vice versa.
9. Reagent bottles must never be allowed to accumulate on the work bench. They should be placed back in the shelves as and when used.
10. Containers in which reaction to be performed a little later should be labelled.

### **CODE OF CONDUCT:**

1. Students should report to the concerned lab as per the time-table schedule.
2. Students who turn up late to the labs will in no case be permitted to perform the experiment scheduled for the day.
- 3 After completion of the experiment, certification of the concerned staff in charge in the observation book is necessary.
4. Students should bring a note book of about 100 pages and should enter the readings / observations into the note book while performing the experiment.
5. The record of observations along with the detailed experimental procedure of the experiment performed in the immediate last session should be submitted and certified by the staff member in-charge.
6. The group-wise division made in the beginning should be adhered to, and no mix up of students among different groups will be permitted later.
7. The components required pertaining to the experiment should be collected from the lab assistants.
8. When the experiment is completed, students should clean the apparatus, carefully dispose the leftover chemicals and disconnect the setup made by them, and should return all the components/instruments taken for the purpose.
9. Any damage to apparatus that occurs during the experimentation should be brought to the notice of lab in-charge, consequently, the cost of the repair or new apparatus should be brought by the students.
10. Students should be present in the labs for the total scheduled duration.
11. Students are required to prepare thoroughly to perform the experiment before coming to Laboratory.
12. Procedure sheets/data sheets provided to the students' groups should be maintained neatly and to be returned

### **INSTRUCTIONS FOR RECORD WRITING:**

1. Write on the right side of page in the following order:
  - a) Serial number and date of performance (in the margin)
  - b) Name and number of the experiment as given in the list.
  - c) Aim of the experiment.
  - d) Description of the apparatus.
  - e) Procedure including sources of error and precautions taken to eliminate or to minimize them.
  - f) Inference or Result.
  - g) Explanation, if necessary, of any divergence in the expected result.
2. Left side of page should contain the following in their proper places.
  - a) Neat diagram of the main apparatus.
  - b) Observation in tabular form.
  - c) Calculation in tabular form.
  - d) Graph sheets and other papers to be attached.
3. Students should submit a record of the previous experiments when they come for practical work.
4. An experiment is deemed to be complete when it is satisfactorily performed and recorded.

### **VISION OF THE INSTITUTE:**

Strive continuously for excellence in professional education through Quality, Innovation, Team Work and Value creation, to emerge as a premier Institute in the State and the Nation.

### **MISSION OF THE INSTITUTE:**

1. To impart quality professional education that meets the needs of present and emerging technological world.
2. To strive for student achievement and success, while preparing them for life, career and leadership.
3. To produce graduates with professional ethics and responsibility towards the development of industry and the society and for sustainable development.
4. To ensure abilities in the graduates to lead technical and management teams for conception, development and management of projects for industrial and national development.
5. To forge mutually beneficial relationships with government organizations, industries, society and the alumni.

## PROGRAM OUTCOMES (POs)

### ENGINEERING GRADUATES WILL BE ABLE TO:

S.No.	Program outcomes (POs):
1.	<b>Engineering knowledge:</b> Apply the knowledge of mathematics, science, engineering fundamentals, and an engineering specialization to the solution of complex engineering problems.
2.	<b>Problem analysis:</b> Identify, formulate, review research literature, and analyze complex engineering problems reaching substantiated conclusions using first principles of mathematics, natural sciences, and engineering sciences.
3.	<b>Design/Development of solutions:</b> Design solutions for complex engineering problems and design system components or processes that meet the specified needs with appropriate consideration for the public health and safety, and the cultural, societal, and environmental considerations.
4.	<b>Conduct investigations of complex problems:</b> Use research-based knowledge and research methods including design of experiments, analysis and interpretation of data, and synthesis of the information to provide valid conclusions.
5.	<b>Modern tool usage:</b> Create, select, and apply appropriate techniques, resources, and modern engineering and IT tools including prediction and modelling to complex engineering activities with an understanding of the limitations.
6.	<b>The engineer and society:</b> Apply reasoning informed by the contextual knowledge to assess societal, health, safety, legal and cultural issues and the consequent responsibilities relevant to the professional engineering practice.
7.	<b>Environment and sustainability:</b> Understand the impact of the professional engineering solutions in societal and environmental contexts, and demonstrate the knowledge of, and need for sustainable development.
8.	<b>Ethics:</b> Apply ethical principles and commit to professional ethics and responsibilities and norms of the engineering practice.
9.	<b>Individual and team work:</b> Function effectively as an individual, and as a member or leader in diverse teams, and in multidisciplinary settings.
10.	<b>Communication:</b> Communicate effectively on complex engineering activities with the engineering community and with society at large, such as, being able to comprehend and write effective reports and design documentation, make effective presentations, and give and receive clear instructions.

11.	<b>Project management and finance:</b> Demonstrate knowledge and understanding of the engineering and management principles and apply these to one's own work, as a member and leader in a team, to manage projects and in multidisciplinary environments.
12.	<b>Life-long learning:</b> Recognize the need for, and have the preparation and ability to engage in independent and life-long learning in the broadest context of technological change.

## COURSE OUTCOMES

### ENGINEERING CHEMISTRY LAB (U23CH1L1)

The experiments will make the student gain skill on:

C116.1	Estimate the amount of Ferrous ions.
C116.2	Calculate the total hardness and alkalinity of water.
C116.3	Identify the mobility of ions in strong acids and weak acids using conductivity meter.
C116.4	Analyse cell potential of a given solution and determine the strength of solution.
C116.5	Apply the principles of Colorimetry and estimate the rate constant.
C116.6	Demonstrate and understand the chemical kinetics of chemical reaction and determine the amount of synthesized drug.

## MAPPING

### COURSE ARTICULATION MATRIX:

#### MAPPING OF COURSE OUTCOMES (CO) WITH PROGRAM OUTCOMES (PO)

COURSE TITLE : ENGINEERING CHEMISTRY LAB	YEAR & SEMESTER : I YEAR I/II SEM
COURSE CODE : U22CH1L1	YEAR : 2022-23

The experiments will make the student gain skill on:

Course Outcomes (CO)	Program Outcomes (PO)											
	1	2	3	4	5	6	7	8	9	10	11	12
Estimate the amount of Ferrous ions.	3	3	2			3	3					
Calculate the total hardness and alkalinity of water.	3	3	2			3	3					
Identify the mobility of ions in strong acids and weak acids using conductivity meter.	3	3	2			3	3					
Analyse cell potential of a given solution and determine the strength of solution.	3	3	2			3	3					
Apply the principles of Colorimetry and estimate the rate constant.	3	3	2			3	3					
Demonstrate and understand the chemical kinetics of chemical reaction and determine the amount of synthesized drug.	3	3	2			3	3					
AVERAGE	3	3	2			3	3					

# INDEX

S.NO	NAME OF THE EXPERIMENT	DATE	PAGE NO	FACULTY SIGN
1	<b>VOLUMETRIC ANALYSIS:</b> Determination of strength of Ferrous solution by Permanganometry.			
2	Determination of strength of Ferrous solution by Dichrometry			
3	<b>Water Analysis:</b> Determination of hardness of water by Complexometric method using EDTA.			
4	Determination of strength of Carbonates and bicarbonates in a given mixture .			
5	<b>INSTRUMENTAL ANALYSIS</b> <b>Conductometry</b> Determination of strength of given HCl solution			
6	Determination of strength of HCl & CH <sub>3</sub> COOH in given mixture			
7	<b>Potentiometry</b> Determination of strength of HCl solution			
8	Determination of strength of Ferrous solution			
9	<b>Colorimetry:</b> Verification of Beer-Lambert's law and determination of Permanganate.			
10	<b>Drug Synthesis</b> Synthesis of Drug (Aspirin / Paracetamol).			



## VOLUMETRIC ANALYSIS

### BASIC CONCEPTS OF VOLUMETRIC ANALYSIS

Chemical analysis of the compounds is carried out in two ways

1. Qualitative analysis.
2. Quantitative analysis.

Qualitative analysis shows what element a given contains. Quantitative analysis determines the quantity of a particular component present in substance. It is carried out in two ways

1. Gravimetric analysis.
2. Volumetric analysis.

Gravimetric analysis involves the DETERMINATION of the amount of a given compound from the results of weighing. Volumetric analysis is based on the measuring the volume of the solution of a substance.

**Terms involved in volumetric analysis:**

1. **Titration:** The process of finding out the volume of one of the solution required to react completely with a definite volume of one the other solution of known concentration is called titration
2. **Titrant:** The solution of known strength is called titrant.
3. **Titrate:** The solution whose concentration is to be estimated.
4. **Indicator:** The reagent which indicates the endpoint or equivalent point of the titration.

**The strength of concentration of a solution is expressed in the following ways:**

- 1) **NORMALITY:** Number of gram equivalents of the substance dissolved per litre of the solution is called Normality. It is denoted by N

$$N = \frac{\text{weight of solute}(w)}{\text{Gram equivalent weight of solute(GEW)}} \times \frac{1000}{\text{Volume of Solvent }(V)(ml)}$$

$$\text{Equivalent weight} = \frac{\text{Molecular weight}}{n}$$

where 'n' denotes the number of H<sup>+</sup> ions in an acid, OH<sup>-</sup> ions in a base and charge of the ions in a salt.

- 2) **MOLARITY:** Number of gram moles of a solute dissolved per liter of solution is called Molarity. It is denoted by M

$$\text{Molarity} = \frac{\text{weight of solute}(w)}{\text{Gram molecular weight of solute(GMW)}} \times \frac{1000}{\text{Volume of Solvent }(V)(ml)}$$

## VOLUMETRIC ANALYSIS

### CHEMICAL ANALYSIS

#### 1. Qualitative Analysis

Ex. Salt analysis

#### 2. Quantitative Analysis

- a. Conventional Methods (Volumetric and Gravimetric)
- b. Modern Method

#### **INTRODUCTION:**

Volumetric analysis or Titrimetry analysis, just as gravimetric analysis gives a quantitative DETERMINATION of species. It involves a measurement of the volume of a solution known as concentration (standard solution) that is required to react completely with the species to be estimated. This method is applicable to fast reaction in solutions. Its advantages include: simple apparatus, simple methods and lesser time with a high accuracy as compared to many other techniques.

The Chemical Analysis which includes both, Qualitative and Quantitative, can be done using instruments.

Since two reacting species would react in the same number of equivalents, the following equation is established:

$$N_1 X V_1 = N_2 X V_2$$

Where N and V refer to normality and volume respectively and the subscripts refer to species 1 and 2.

Knowing the normality of one of the solutions (the standard solution) and the two reacting volumes, the normality N of the solution under test can be determined.

#### **DEFINITIONS:**

**i) Titration:** The overall procedure of determining stoichiometric or equivalence point is called titration or titrimetry .

**ii) Titrant:** The solution added in a titration.

**iii) Titrand:** The solution to which the titrant is added.

**iv) End point:** A point in the progress of the reaction which may be precisely located (almost coincident with stoichiometric or equivalence point).

**v) Indicator:** A reagent used to indicate when the end point is reached. (In some cases, one of the reactants serves as its own indicator (self-indicator) as in the case of titrations involving KMnO<sub>4</sub>)

# EXPERIMENTS

THE INSTITUTE OF  
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## **LAB EQUIPMENTS**



## EXPERIMENT (1)

**PREPARATION OF STANDARD MOHR'S SALT SOLUTION, STANDARDISATION OF POTASSIUM PERMANGANATE ( $KMnO_4$ ) & DETERMINATION OF FERROUS IONS:**

### **PART A: PREPARATION OF STANDARD FERROUS AMMONIUM SULPHATE**

$[FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O]$  SOLUTION.

### **PART B: STANDARDIZATION OF $KMnO_4$**

**Titration of Std. F.A.S. vs.  $KMnO_4$**

S. No	Volume of Standard FAS (ml)	Burette Readings		Volume of $KMnO_4$ Consumed (ml)
		Initial Reading (ml)	Final Reading (ml)	
1.	20			
2.	20			
3.	20			

#### Calculations:

$$N_1 V_1 = N_2 V_2$$

Where,  $N_1$  = Normality of Standard F.A.S = \_\_\_\_\_ N

$V_1$  = Volume of Standard F.A.S = 20 ml

$N_2$  = Normality of  $KMnO_4$  = \_\_\_\_\_ ?

$V_2$  = Volume of  $KMnO_4$  = \_\_\_\_\_ (Burette reading)

$$N_1 V_1 = N_2 V_2$$

$$N_2 = \frac{N_1 V_1}{V_2} = \text{_____} = N$$

$$\text{Normality of } KMnO_4 (N_2) = \text{_____ N}$$

## VOLUMETRIC ANALYSIS

### EXPERIMENT (1)

#### **PREPARATION OF STANDARD MOHR'S SALT SOLUTION, STANDARDISATION OF POTASSIUM PERMANGANATE (KMnO<sub>4</sub>) & DETERMINATION OF FERROUS IONS:**

**AIM:** To standardize KMnO<sub>4</sub> solution using Std. FAS solution.

**APPARATUS:** Conical flask, Burette, Pipette, Burette stand and Dropper

**CHEMICALS:** H<sub>2</sub>SO<sub>4</sub>, KMnO<sub>4</sub>, FeSO<sub>4</sub> (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>.6H<sub>2</sub>O (Mohr's Salt)

**PRINCIPLE:** Potassium Permanganate oxidizes Ferrous Sulphate present in the Mohr's salt in the presence of dil. H<sub>2</sub>SO<sub>4</sub> to Ferric Sulphate according to following chemical reaction.



#### **PROCEDURE:**

##### **PART-A:**

##### **PREPARATION OF STANDARD FERROUS AMMONIUM SULPHATE FeSO<sub>4</sub> (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>.6H<sub>2</sub>O SOLUTION**

Exactly \_\_\_\_\_ g of FAS is weighed out in a clean weighing bottle. It is transferred to a 100ml volumetric flask through a funnel, and 10 drops Conc. H<sub>2</sub>SO<sub>4</sub> is added. Add some distilled water and shake well to make it homogenous. The solution is made up to the mark with distilled water.

##### **PART-B:**

##### **STANDARDIZATION OF GIVEN KMnO<sub>4</sub> SOLUTION:**

Pipette out 20 ml of the prepared std. Ferrous Ammonium Sulphate solution into a clean conical flask. Titrate with KMnO<sub>4</sub> (taken in the burette) to a faint pink colour which persists for at least a minute. This is the end point. (Last addition of KMnO<sub>4</sub> should be drop wise with particular care to allow each drop to become decolorized before the next is added). Repeat the process till at least two concurrent values are obtained.

##### **PART-C:**

**DETERMINATION OF Fe<sup>2+</sup> IN THE GIVEN TEST SOLUTION:** Pipette out 20 ml of the given Ferrous Ammonium Sulphate solution into a clean conical flask and titrate with KMnO<sub>4</sub> (taken in a burette) to a faint Pink colour which persists for at least a minute. This is the end point. The process is repeated till two concurrent titre values are obtained. From the titration data the amount of Fe<sup>2+</sup> in the given test solution is estimated.

**PART C:****DETERMINATION OF Fe<sup>2+</sup> IN THE GIVEN TEST (FAS) SOLUTION**Titration: Fe<sup>2+</sup> (Test Solution of FAS) Vs KMnO<sub>4</sub>

S. No	Volume of unknown / test FAS solution) (ml)	Burette Readings		Volume of KMnO <sub>4</sub> Consumed(ml)
		Initial (ml)	Final (ml)	
1.	20ml	0		
2.	20ml			
3.	20ml			

Calculations:**Test solution (FAS) VS KMnO<sub>4</sub>**

For Neutralization,

$$N_2 V_2 = N_3 V_3$$

Where,

N<sub>2</sub> = Normality of KMnO<sub>4</sub> \_\_\_\_\_ N (from 2<sup>nd</sup> Step)V<sub>2</sub> = Volume of KMnO<sub>4</sub> \_\_\_\_\_ (Burette reading)N<sub>3</sub> = Normality of Test solution (FAS) \_\_\_\_\_ ?V<sub>3</sub> = Volume of Test solution (FAS) \_\_\_\_\_

$$N_3 = \frac{N_2 V_2}{V_3} = \text{_____ N}$$

Normality [strength] of Test FAS solution = \_\_\_\_\_ N

Therefore,

Amount of iron present in the given Test solution

N<sub>3</sub> X Equivalent weight of Fe<sup>2+</sup>N<sub>3</sub> X 27.92 = \_\_\_\_\_ g/litre

**RESULT:**

Strength of Test Solution (FAS) \_\_\_\_\_ N

Amount of Ferrous Ions ( $\text{Fe}^{+2}$ ) present in the given Test FAS solution \_\_\_\_\_ gms / litre



## **EXPERIMENT-2**

### **DETERMINATION OF FERROUS ION BY DICHROMETRY:**

#### **PART A: PREPARATION OF STANDARD FERROUS AMMONIUM SULPHATE [FeSO<sub>4</sub> (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>.6H<sub>2</sub>O] SOLUTION**

#### **PART B : STANDARDISATION OF K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> SOLUTION**

**Standard FAS vs K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>**

S.No.	Volume of FAS solution[ml]	Burette reading		Volume of K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> consumed [ml]
		Initial	Final	
1				
2				
3				

***Calculations and Observation:***

Normality of Standard FAS (N<sub>1</sub>)= \_\_\_\_\_ N

Volume of Standard FAS (V<sub>1</sub>) = \_\_\_\_\_ ml

Normality of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> (N<sub>2</sub>) = \_\_\_\_\_ N

Volume of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> (V<sub>2</sub>) = \_\_\_\_\_ ml [burette reading from table 1]

$$N_1 V_1 = N_2 V_2$$

$$N_2 = \frac{N_1 V_1}{V_2}$$

Normality [strength] of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution = \_\_\_\_\_ N

## **EXPERIMENT-2:**

Determination of strength of Ferrous solution by Dichrometry:

**AIM:** To Determine the amount of ferrous ion present of the whole solution with the help of standard solution of potassium dichromate [oxidizing agent]

**APPARATUS:** Beaker, burette, burette stand, pipette, conical flask, volumetric flask.

**CHEMICALS:** Potassium dichromate [ $K_2Cr_2O_7$ ], Diphenylamine indicator, Conc. $H_2SO_4$ , and Ferrous ammonium sulphate [Mohr's salt]

**THEORY AND PRINCIPLE:** Ferrous ion is oxidized to ferric ion by potassium dichromate in acid solution. The completion of the oxidation of reaction is marked by the appearance of blue violet color of the diphenylamine which is used as internal indicator.



### **PROCEDURE:**

#### **PART A: PREPARATION OF STANDARD FERROUS AMMONIUM SULPHATE $FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ SOLUTION.**

Exactly \_\_\_\_\_ g of FAS is weighed out in a clean weighing bottle. It is transferred to a 100ml standard Volumetric flask through a funnel, add 10 drops Conc.  $H_2SO_4$ . Add some distilled water and shake well to make it homogenous. The solution is made up to the mark with distilled water.

**PART B: STANDARDISATION OF  $K_2Cr_2O_7$  SOLUTION:** The burette is washed with distilled water & rinsed with  $K_2Cr_2O_7$  and then filled with  $K_2Cr_2O_7$  without any air bubbles up to zero level. Pipette out 20ml of Std FAS solution in to a clean 250ml conical flask and add 2 drops of diphenylamine indicator. Then titrate the resulting solution with potassium dichromate [ $K_2Cr_2O_7$ ] taken in the burette till blue violet color is obtained as end point. Repeat the titration to get concurrent values.

#### **Part C: Standardization of Ferrous ion:**

Rinse the burette with standard  $K_2Cr_2O_7$  solution and take  $K_2Cr_2O_7$  solution up to the zero mark of the burette. Pipette out 20ml of ferrous solution in to a 250ml conical flask and add 2 drops of diphenylamine indicator. Titrate the resulting solution with potassium dichromate taken in the burette till blue violet color is obtained as end point. Repeat the titration to get concurrent values.

**PART C: DETERMINATION of Fe<sup>2+</sup>in the given Test FAS solution:**

Standard K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> vs given unknown [Fe<sup>2+</sup>] solution

S.No.	Volume of FAS [Fe <sup>2+</sup> ] solution[ml]	Burette reading		Volume of K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> consumed [ml]
		Initial	Final	
1				
2				
3				

**Calculation and observation:**

Normality of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> [N<sub>2</sub>] = \_\_\_\_\_ N

Volume of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> [V<sub>2</sub>] = \_\_\_\_\_ ml

Normality of Ferrous solution [N<sub>3</sub>] = \_\_\_\_\_ N

Volume of Ferrous solution[V<sub>3</sub>] = \_\_\_\_\_ ml

For Neutralization,

$$N_2 V_2 = N_3 V_3$$

$$N_3 = \frac{N_2 V_2}{V_3} = \text{_____ N}$$

Normality [strength] of given test FAS solution = \_\_\_\_\_ N

Therefore,

Amount of Ferrous ions present in the given test FAS solution

N<sub>3</sub> X Equivalent weight of Fe<sup>2+</sup>

$$N_3 \times 27.92 = \text{_____ g/litre}$$

**RESULT:**

Normality of FAS \_\_\_\_\_ N

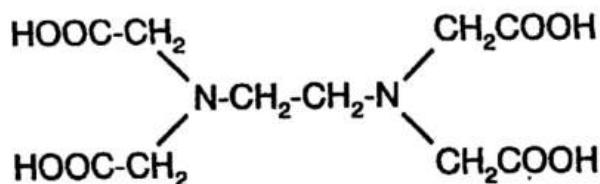
The amount of ferrous ion  $[Fe^{+2}]$  in the given solution is \_\_\_\_\_ g/litre



### **EXPERIMENT: (3)**

#### **DETERMINATION OF HARDNESS OF WATER BY EDTA METHOD:**

##### **STRUCTURE OF EDTA**



##### **CALCULATIONS AND OBSERVATIONS**

###### **PATR A: PREPARATION OF STANDARD MgSO<sub>4</sub>**

###### **PART-B: STANDARDISATION OF EDTA**

S.NO	VOLUME OF STANDARD HARD WATER SAMPLE (ml)	BURETTE READING		Volume Of EDTA Consumed(ml)
		INITIAL	FINAL	
1	20			
2	20			
3	20			

##### **CALCULATIONS:**

M<sub>1</sub> = Molarity of standard Hard water = 0.01M

V<sub>1</sub>=Volume of standard water = 20ml

M<sub>2</sub> = Molarity of EDTA = .....? V<sub>2</sub> = Volume of EDTA = Burette Reading =

For Neutralization, M<sub>1</sub>V<sub>1</sub> = M<sub>2</sub>V<sub>2</sub>

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

## WATER ANALYSIS

### **EXPERIMENT: (3)**

#### **Determination of hardness of water by Complexometric method using EDTA**

**AIM:** To determine the hardness of water in given sample of water by EDTA method.

#### **APPARATUS:**

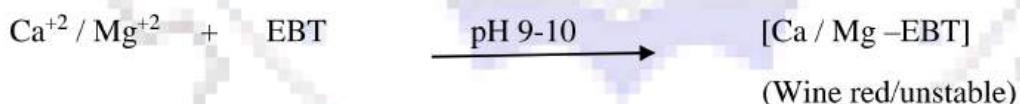
1. Conical flask
2. Burette
3. Pipette
4. Burette stand
5. Spatula

#### **CHEMICALS:**

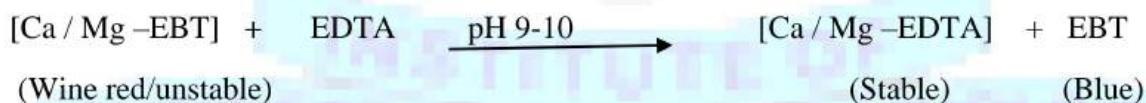
1. Buffer solution
2. Eriochrome Black-T indicator (EBT)
3. EDTA Solution

#### **PRINCIPLE:**

EDTA forms colorless, stable complexes with  $\text{Ca}^{2+}$  &  $\text{Mg}^{2+}$  ions present in water at pH 9-10. To maintain pH at 9-10,  $\text{NH}_4\text{Cl}$  &  $\text{NH}_4\text{OH}$  buffer is used. Eriochrome Black-T (EBT) is used as Indicator. Hard water sample with EBT indicator forms unstable, wine red coloured complexes with  $\text{Ca}^{2+}$  &  $\text{Mg}^{2+}$  present in water.



The metal indicator complex is less stable than Metal-EDTA complex. So, this metal indicator complex is titrated with std. EDTA solution. Now, colourless Metal-EDTA complex is formed by releasing EBT indicator which is blue colour. So, the colour change from wine red to blue indicates the end point.



### **PART-C: DETERMINATION OF HARDNESS OF WATER**

S.No	VOLUME OF TEST WATER SAMPLE (ml)	BURETTE READING		VOLUME OF EDTA CONSUMED (ml)
		INITIAL (ml)	FINAL (ml)	
1				
2				
3				

#### **CALCULATIONS:**

$M_2$  = Molarity of EDTA = (from 2<sup>nd</sup> STEP)

$M_3$  = Molarity of water sample = .....?

$V_2$  = Volume of EDTA = Burette Reading =

$V_3$  = Volume of water sample = 20ml

For Neutralization,

$$M_2 V_2 = M_3 V_3$$

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

$$M_3 = \dots\dots\dots M$$

$$\text{Total hardness} = M_3 \times 100 \times 1000 \text{ mg/l or ppm} = \dots\dots\dots \text{ppm.}$$

**PROCEDURE:**

**PART A: PREPARATION OF STANDARD MgSO<sub>4</sub> SOLUTION.**

Exactly \_\_\_\_\_ g of MgSO<sub>4</sub> is weighed out in a clean weighing bottle. It is transferred to a 100ml standard Volumetric flask through a funnel, dissolved with Distilled water. The solution is made up to the mark with distilled water. It is shaken well to make homogenous.

**PART -B: STANDARDISATION OF EDTA:**

Pipette out 20 ml standard hard water into a conical flask. To this add 3ml of buffer solution followed by addition of 3 to 4 drops of EBT indicator. Then the solution turns to wine red in colour. Now, titrate the wine-red solution against the EDTA taken in the burette until the solution turns to blue colour. Note down the burette reading. Repeat the titration to get concurrent values.

**PART C: DETERMINATION OF TOTAL HARDNESS OF SAMPLE WATER:**

Take 20 ml of sample water into a conical flask. To this add 5ml of buffer solution followed by addition of 3 to 4 drops EBT indicator. Then titrate against with EDTA which is taken in burette. Continue the titration till wine red colour to blue colour. Repeat this titration until two concurrent values are obtained.

**RESULT:**

Total hardness in given sample is ..... ppm.

**EXPERIMENT: (4)****DETERMINATION OF CARBONATE AND BICARBONATE ALKALINITY IN WATER****PART A:****PREPARATION OF 100 ml of 0.02 N Std. Na<sub>2</sub>CO<sub>3</sub> SOLUTION:**

$$N_{\text{Na}_2\text{CO}_3} = \frac{\text{weight}}{\text{Equivalent weight}} \times \frac{1000}{V_{ml}}$$

Weight of Na<sub>2</sub>CO<sub>3</sub> = \_\_\_\_\_ g

**PART B: STANDARDISATION OF HCl**

S.No	Volume of Na <sub>2</sub> CO <sub>3</sub> (ml)	Burette Reading		Volume of HCl consumed (ml)
		Initial (ml)	Final (ml)	
1.	20			
2.	20			
3.	20			

**CALCULATION:****Na<sub>2</sub>CO<sub>3</sub> Vs HCl**

Normality of Na<sub>2</sub>CO<sub>3</sub> (N<sub>1</sub>) = \_\_\_\_\_

Volume of Na<sub>2</sub>CO<sub>3</sub> (V<sub>1</sub>)

Normality of HCl (N<sub>2</sub>) = \_\_\_\_\_ ?

Volume of HCl (V<sub>2</sub>) consumed, burette reading

As per volumetric analysis,

$$N_1 V_1 = N_2 V_2$$

$$N_2 = \frac{N_1 V_1}{V_2}$$

$$= ..... N$$

Normality (strength) of HCl (N<sub>2</sub>) = \_\_\_\_\_ N

**EXPERIMENT: (4)**

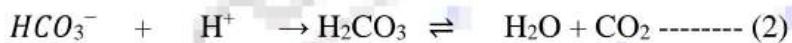
Determination of strength of Carbonates and bicarbonates in a given mixture

**AIM:** To Determine the strength of Carbonate and Bicarbonate alkalinity in water

**APPARATUS:** Burette, Pipette, beaker, volumetric flask, conical flask, dropper

**CHEMICALS:** Standard HCl, Phenolphthalein indicator, Methyl Orange indicator

**PRINCIPLE:** The reaction taking place may be represented by the following equations.



The volume of acid run down up to phenolphthalein end-point[P] corresponds to the completion of equation (1) given above, while the volume of acid run down after [P], corresponds to the completion of equation (2).

The total amount of acid used from the beginning of the experiment, i.e., [M] corresponds to the total alkalinity and represents the completion of reactions shown by equations (1) and (2). Alkalinity is generally expressed as parts per million (ppm) in terms of  $CaCO_3$ .

#### **PROCEDURE:**

##### **PART A:PREPARATION OF Std. $Na_2CO_3$ SOLUTIONS**

Weigh out accurately about -----g of (A.R.)  $Na_2CO_3$  and transfer it to a clean 100 ml. standard volumetric flask through a funnel, dissolve it in distilled water, shake well to make it homogeneous and make up the solution to the mark .

##### **PART B: STANDARDISATION OF HCl**

Pipette out 20ml of the prepared Std.  $Na_2CO_3$  solution into a clean conical flask. Add 2 drops of **methyl orange** indicator. The solution becomes **yellow**. Titrate with the given HCl (taken in the burette) to a initial **light pink** appears, this is the **end point**. Repeat the process of titration till at least two concurrent values are obtained.

## PART :C

### DETERMINATION OF CARBONATE AND BI-CARBONATE ALKALINITY IN WATER:

Volume of Water sample	BURETTE READING				Volume of HCl consumed	
	Phenolphthalein		Methyl Orange		V <sub>P</sub>	V <sub>M</sub>
	Initial (ml)	Final (ml)	Initial (ml)	Final (ml)		

### CALCULATION:

**Phenolphthalein alkalinity (P):** It is due to complete OH<sup>-</sup> and  $\frac{1}{2}$  CO<sub>3</sub><sup>2-</sup> present in water

H<sub>2</sub>O Vs HCl,

$$N_{H_2O} V_{H_2O} = N_{HCl} V_{HCl}$$

$$N_{H_2O} = \frac{N_{HCl} V_{HCl}}{V_{H_2O}}$$

$$N_{H_2O} = \dots\dots\dots N$$

**Phenolphthalein alkalinity (P):**  $N_{H_2O} \times 50 \times 1000 =$

(Where, 50= Equivalent weight of CaCO<sub>3</sub>)

**Methyl orange alkalinity (M):** It is due to all the ions present in water OH<sup>-</sup> &  $\frac{1}{2}$  CO<sub>3</sub><sup>2-</sup>, HCO<sub>3</sub><sup>-</sup>, hence methyl orange alkalinity is equal to total alkalinity of water)

$$N_{H_2O} V_{H_2O} = N_{HCl} V_{HCl}$$

$$N_{H_2O} = \frac{N_{HCl} V_{HCl}}{V_{H_2O}} = \dots\dots\dots N$$

**Methyl orange (M):**  $N_{H_2O} \times 50 \times 1000 =$

S.No	Observations	Alkalinity due to -		
		Hydroxide (OH <sup>-</sup> )	Carbonat CO <sub>3</sub> 2- )	Bicarbonate (HCO <sub>3</sub> - )
1	P = 0			M
2	P = M	P or M		
3	P = $\frac{1}{2}$ M		2P	
4	P > $\frac{1}{2}$ M	2P-M	2(M-P)	
5	P < $\frac{1}{2}$ M		2P	M-2P

**PART C:****DETERMINATION OF  $CO_3^{2-}$  &  $HCO_3^-$  ALKALINITY IN TEST SOLUTION:**

Make up the given test solution up to the mark by adding distilled water. Make the solution homogeneous. Pipette 20 ml of test solution containing both  $CO_3^{2-}$  &  $HCO_3^-$  in a clean conical flask.

- i) Add 2 drops of phenolphthalein indicator, and titrate the sample with standard HCl. Until the pink color just disappears-note the titre value as phenolphthalein end point [P].
- ii) Add 2 drops of methyl orange indicator to the same solution and continue the titration until color changes from yellow to red or pink. Note the titre value as [M] end point

**RESULT:**

Alkalinity of Carbonate in given water sample = \_\_\_\_\_ ppm

Alkalinity of Bicarbonate in given water sample = \_\_\_\_\_ ppm

**EXPERIMENT: (5) DETERMINATION OF STRENGTH OF GIVEN HCl BY CONDUCTOMETRIC TITRATION**

**PART A: Standardisation of NaOH**

S.No	Volume of HCl	Burette Readings		Volume of NaOH
		Initial(ml)	Final (ml)	
1.	20			
2.	20			
3.	20			

As per volumetric analysis,  $N_1V_1 = N_2V_2$

Normality of NaOH ( $N_2$ ) = ..... ; Volume of NaOH consumed, ( $V_2$ ) = ..... ml

Normality of HCl ( $N_1$ ) = .....? Volume of HCl ( $V_1$ ) = 20 ml

$$N_1V_1 = N_2V_2 \Rightarrow N_2 = \frac{N_1V_1}{V_2} = ..... N$$

**PART B: DETERMINATION OF HCl**

**CALCULATIONS & OBSERVATION**

S.NO	VOLUME OF NaOH	CONDUCTANCE
1.		
2.		
3.		
4.		
5.		
6.		
7.		
8.		
9.		
10.		
11.		
12.		
13.		
14.		
15.		
16.		
17.		
18.		

## **EXPERIMENT : (5)**

### **Determination of strength of given HCl solution.**

**AIM:** To determine the strength of hydrochloric acid solution by titrating against standard sodium hydroxide solution by conductometrically.

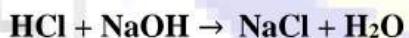
**APPARATUS:** Conductivity meter, conductivity cell, burette, beaker, glass rod.

**CHEMICALS:** HCl & NaOH

**PRINCIPLE:** Conductometric titration is the volumetric analysis based upon the measurement of the conductance during the course of titration. The number of free ions , charge on the free ions and mobility of the ions affects the

Conductance of an aqueous solution. When one electrolyte is added to another electrolyte , the change in number of free ions cause a change in the conductance.

For example when a strong acid (HCl) is titrated against a strong base (NaOH), before NaOH solution is added from the burette, the acid solution has high conductivity due to the highly mobile  $H^+$  ions. When NaOH is added to the acid, the conductivity of the acid solution decreases due to the neutralization of highly mobile  $H^+$  ions of the acid with  $OH^-$  ions of the base.



Thus, the conductance of the solution continues to decrease until the equivalent point is reached. Further addition of NaOH solution will increase the conductance by highly mobile hydroxyl ions.

### **PROCEDURE:**

#### **PART: A-STANDARDISATION OF NaOH**

Pipette out 20 ml of given 0.1N HCl solution in a clean conical flask, add phenolphthalein indicator and titrate with NaOH taken in burette till light pink color appears. Repeat the process till concurrent values are obtained.

#### **PART: B - DETERMINATION OF HCl**

1. Fill the burette with standard NaOH solution.
2. Take 20 ml of given HCl solution in 100 ml beaker and dip the conductivity cell in it and measure the conductance initially.
3. Now add 1 ml NaOH from the burette drop wise.
4. After each addition, stir the solution gently by using glass rod and note down the change in conductance.
5. Measured conductance values are recorded in the table.

### **CALCULATION:**

Normality of NaOH ( $N_2$ ) =

Volume of NaOH consumed, end point from graph ( $V_2$ ) = .....ml

Normality of HCl ( $N_3$ ) = .....?

Volume of HCl ( $V_3$ ) = 20 ml

For Neutralization,

$$N_2 V_2 = N_3 V_3$$

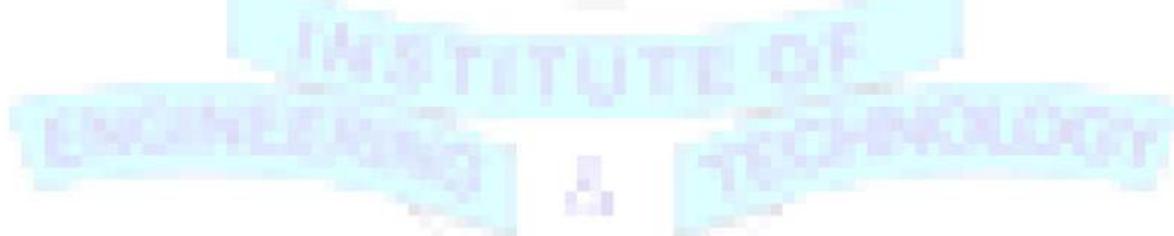
$$N_3 = \frac{N_2 V_2}{V_3} = \text{_____} N$$

Normality (strength) of HCl ( $N_3$ ) = .....N

The amount of HCl in the given solution =  $N_3 \times$  Eq. Wt of HCl

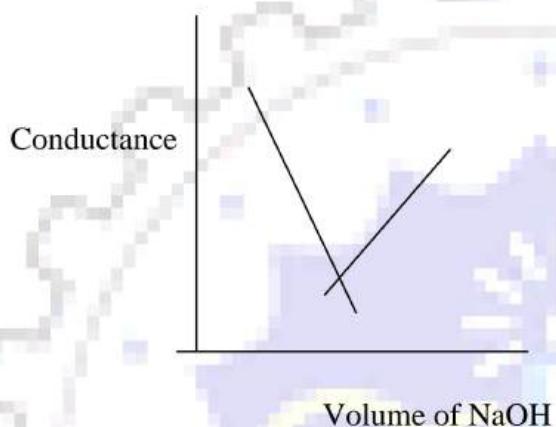
$$= N_3 \times 36.5 \text{ gms/l}$$

$$= \text{.....gms/lt.}$$



**GRAPH:**

Plot the graph between conductance against volume of base added, which consists of 2 straight lines. The intersection of 2 straight lines gives the end point as shown in the graph below



**RESULT:**

1. The end point of the titration = .....ml
2. The strength (Normality) of given solution = .....N.
3. The amount of HCl present in the given solution = .....gms/litre

## **EXPERIMENT-(6)**

### **DETERMINATION OF HCl AND ACETIC ACID IN GIVEN MIXTURE BY CONDUCTOMETRY:**

#### **PART A: STANDARDISATION OF NaOH:**

S.No	Volume of HCl	Burette Readings		Volume of NaOH Consumed (ml)
		Initial (ml)	Final (ml)	
1.	20			
2.	20			
3.	20			

As per neutralization formula

$$N_1 V_1 = N_2 V_2$$

Normality of NaOH ( $N_2$ ) = ..... ; Volume of NaOH consumed, ( $V_2$ ) = ..... ml

Normality of HCl ( $N_1$ ) = .....? Volume of HCl ( $V_1$ ) = 20 ml

$$N_1 V_1 = N_2 V_2 \Leftrightarrow N_2 = \frac{N_1 V_1}{V_2} = ..... N$$

#### **PART B : DETERMINATION OF HCl AND ACETIC ACID**

S.NO	VOLUME OF NaOH	CONDUCTANCE
1.		
2.		
3.		
4.		
5.		
6.		
7.		
8.		
9.		
10.		
11.		
12.		
13.		
14.		
15.		
16.		
17.		
18.		
19.		

## **EXPERIMENT-(6)**

### **DETERMINATION OF HCl AND ACETIC ACID IN GIVEN MIXTURE BY CONDUCTOMETRY**

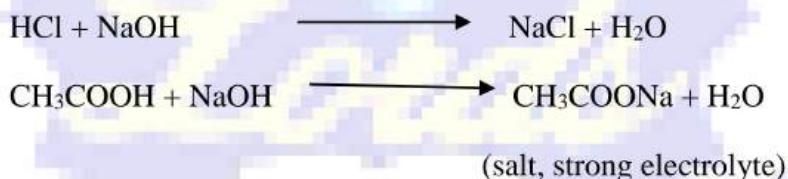
**AIM:** To determine the end point of the titration of a mixture of acids with strong base by conductometric method and DETERMINATION of the mixture of strong and weak acids.

**APPARATUS:** Burette, beaker, burette stand, glass rod, conductivity meter &conductivity cell

**CHEMICALS:** NaOH, HCl & CH<sub>3</sub>COOH

**PRINCIPLE:** Conductometric titration is the volumetric analysis based upon measurement of the conductance during the course of titration.

When a mixture containing strong acid(HCl) and a weak acid(CH<sub>3</sub>COOH) is titrated against a strong base(NaOH), strong acid will be neutralized first. The neutralization takes place in the following way. First strong acid react with the strong base and then weak acid reacts with strong base.



The conductance of the mixture falls on the addition of NaOH due to the neutralization of highly mobile H<sup>+</sup> ions with a base till the strong acid neutralized. Then the conductivity rises due to the neutralization of the weak acid to the salt which is a strong electrolyte. Finally the conductivity raised rapidly as the alkali is introduced in excess after the neutralization of the weak acid. This is due to the presence of fast moving OH<sup>-</sup> ions.

#### **PROCEDURE:**

##### **PART A: STANDARDISATION OF NaOH**

Pipette out 20 ml of given 0.1N HCl solution in a clean conical flask, add phenolphthalein indicator and titrate with NaOH taken in burette till light pink color appears. Repeat the process till concurrent values are obtained.

##### **PART B: DETERMINATION OF HCl AND ACETIC ACID**

1. Fill the burette with standard 0.1N NaOH solution.
2. Pipette out 10ml of given HCl and 10ml of CH<sub>3</sub>COOH solution into clean 100ml beaker.
3. Wash the conductivity cell with distilled water and dip it in a mixture of acids taken in the beaker
4. Connect the conductivity cell to the conductivity meter and measure initial conductance.

## **CALCULATIONS:**

### **HCl vs NaOH:**

Normality of NaOH ( $N_2$ ) =

Volume of NaOH consumed, end point from graph ( $V_2$ ) = .....ml

Normality of HCl ( $N_3$ ) = .....?

Volume of HCl ( $V_3$ ) = 20 ml

For Neutralization,

$$N_2 V_2 = N_3 V_3$$

$$N_3 = \frac{N_2 V_2}{V_3} = \text{_____} N$$

Normality (strength) of HCl ( $N_3$ ) = .....N

The amount of HCl in the given solution =  $N_3 \times$  Eq. Wt of HCl

=  $N_3 \times 36.5$  gms/lt

= .....gms/lt.

### **CH<sub>3</sub>COOH vs NaOH:**

Normality of NaOH ( $N_2$ ) =

Volume of NaOH consumed 2<sup>nd</sup> end point from graph (q-p) ( $V_2$ ) = .....ml

Normality of CH<sub>3</sub>COOH ( $N_4$ ) = .....ml

Volume of CH<sub>3</sub>COOH ( $V_4$ ) = 10ml

As per volumetric analysis

$$N_4 V_4 = N_2 V_2$$

$$N_4 = \frac{N_2 V_2}{V_4} = \text{_____} N$$

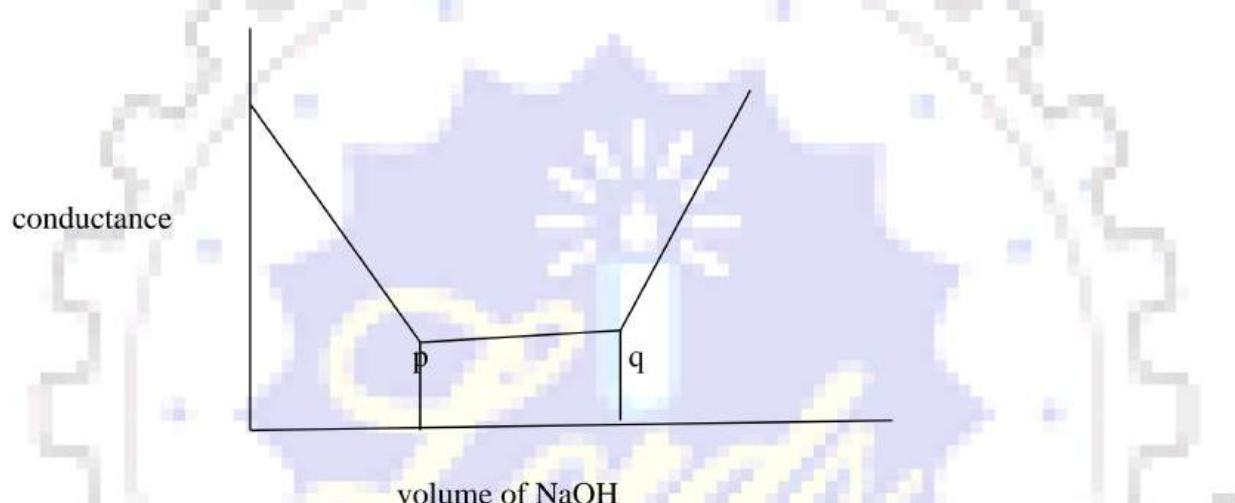
Amount of CH<sub>3</sub>COOH in the given solution =  $N_4 \times$  Eq.Wt. of CH<sub>3</sub>COOH

=  $N_4 \times 60$

= .....g/L

- Now add NaOH solution from the burette drop wise i.e. 1ml addition. After each addition stir the solution by glass rod and note down the conductance.
- The measured conductance values are tabulated in the table.

**GRAPH:** Plot the graph between conductance along Y-axis and volume of NaOH added along X-axis. The conductometric titration curve will be by two breaks. The first one corresponds to the equivalent point of HCl and second to equivalent point of  $\text{CH}_3\text{COOH}$ . The graph obtained is shown below.



Equivalent point of HCl = p

Equivalent point of  $\text{CH}_3\text{COOH} = q - p$

#### CALCULATIONS:

#### RESULTS:

- The equivalent point HCl = \_\_\_\_\_ ml
- The normality of HCl = \_\_\_\_\_ N
- The amount of strong acid(HCl) in given solution = \_\_\_\_\_ g/l
- The equivalent point of  $\text{CH}_3\text{COOH} =$  \_\_\_\_\_ ml
- The normality of  $\text{CH}_3\text{COOH} =$  \_\_\_\_\_ N
- The amount of weak acid ( $\text{CH}_3\text{COOH}$ ) in the given solution = \_\_\_\_\_ g/l

## **EXPERIMENT (7): DETERMINATION OF HCl BY POTENTIOMETRY**

### **PART A: STANDARDISATION OF NaOH**

S.No	Volume of HCl (ml)	Burette Readings		Volume of NaOH Consumed (ml)
		Initial (ml)	Final (ml)	
1.	20			
2.	20			
3.	20			

**As per neutralization formula**

$$N_1 V_1 = N_2 V_2$$

Normality of NaOH ( $N_2$ ) = ... ; Volume of NaOH consumed, ( $V_2$ )=Burette Reading= ml

Normality of HCl ( $N_1$ ) = ...? Volume of HCl ( $V_1$ ) = 20 ml

$$N_1 V_1 = N_2 V_2 \implies N_2 = \frac{N_1 V_1}{V_2} = \dots \text{N}$$

### **PART B : DETERMINATION OF HCl**

S.NO	Volume Of NaOH	EMF	$\Delta E$	$\Delta V$	$\Delta E/\Delta V$
1.					
2.					
3.					
4.					
5.					
6.					
7.					
8.					
9.					
10.					
11.					
12.					
13.					
14.					
15.					
16.					

## **EXPERIMENT(7):**

### **DETERMINATION OF HCl BY POTENTIOMETRY**

**AIM:** To determine the strength of hydrochloric acid solution by titrating against standard sodium hydroxide solution by using potentiometer.

**APPARATUS:** Potentiometer , platinum electrode, saturated calomel (reference ) electrode, beaker, glass rod, burette and burette stand

**CHEMICALS:** HCl, NaOH & Quinhydrone.

**PRINCEPLE:** Potentiometric titration depend on measurement of emf between reference electrode and an indicator electrode.

The quinhydrone electrode (indicator electrode) is prepared by taking saturated solution of quinhydrone in a known quantity of strong acid whose concentration to be determined. This electrode is combined with the calomel electrode to make the cell. The cell representation is as shown below.



When the HCl is titrated against NaOH solution, the EMF is going on decreasing as the  $\text{H}^+$  ion concentration decreases. After equivalent point is reached the EMF values increases due to presence of  $\text{OH}^-$ .

The reaction between the HCl and NaOH is as shown below.



### **PROCEDURE:**

#### **PART- A: STANDARDISATION OF NaOH**

Pipette out 20 ml of given 0.1N HCl solution in a clean conical flask, add phenolphthalein indicator and titrate with NaOH taken in burette till light pink color appears. Repeat the process till concurrent values are obtained.

#### **PART -B: DETERMINATION OF HCl:**

1. Fill the burette with standard sodium hydroxide solution(NaOH) after rinsing the burette with NaOH upto zero mark.
2. Take 20 ml of given HCl solution into a clean beaker , and pinch of quinhydrone power to the solution until get Saturated. Dip the platinum electrode into the solution and combine it to saturated calomel electrode by using a salt bridge.
3. Connect the platinum electrode to the positive terminal and calomel electrode to the negative terminal in the potentiometer.
4. Measure the EMF value initially.
5. Now add NaOH from the burette drop wise i. e., 1 ml for each addition. After each addition, stir the solution by using a glass rod and note down the EMF values.
6. The measured EMF values are tabulated in the table.

### **CALCULATION:**

Normality of NaOH ( $N_2$ ) =

Volume of NaOH consumed, end point from graph ( $V_2$ ) = .....ml

Normality of HCl solution ( $N_3$ ) = .....?

Volume of HCl solution ( $V_3$ ) = 20 ml

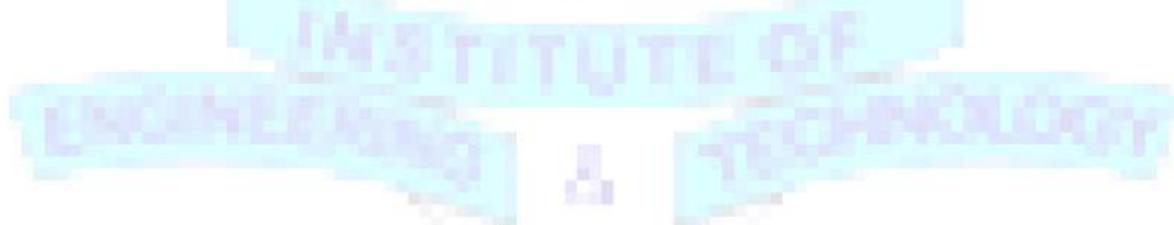
**As per neutralization formula**

$$N_3 V_3 = N_2 V_2$$

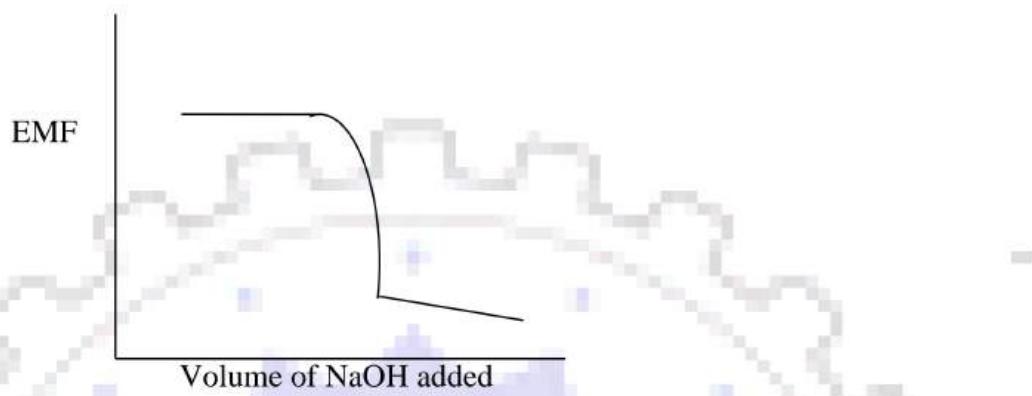
$$N_3 = \frac{N_2 V_2}{V_3} = \text{_____} = N$$

Normality (strength) of HCl solution ( $N_3$ ) = .....N

The amount of HCl in the given solution  
=  $N_3 \times$  Eq. Wt of HCl  
=  $N_3 \times 36.5$  gms/lt. =  
= ..... gms lt.

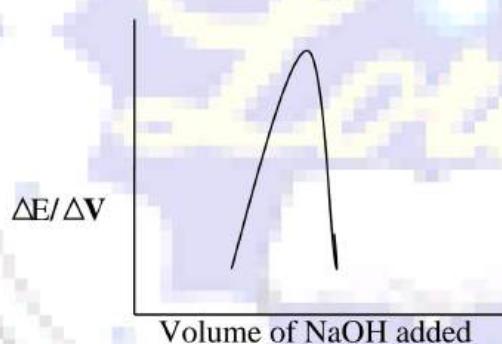


**GRAPH:** Plot a graph between EMF against the volume of NaOH added and a curve is obtained as shown below.



The point of inflexion (where the curve changes its curvature) in the curve gives the equivalent point.

The accurate value can be obtained by plotting a graph between  $\Delta E/\Delta V$  against the volume of the NaOH added. The graph obtained is shown as below.



### **RESULT:**

1. The normality of HCl solution = .....N
2. The amount of HCl in the given solution = .....gms/lt.

## **EXPERIMENT (8): Determination of Fe<sup>+2</sup> by Potentiometry using KMnO<sub>4</sub>**

### **Part A: Standardization of KMnO<sub>4</sub>**

#### **Titration of Std. F.A.S. Vs. KMnO<sub>4</sub>**

S.No	Volume of FAS (ml)	Burette Readings		Volume of KMnO <sub>4</sub> Consumed
		Initial (ml)	Final (ml)	
1.	20			
2.	20			
3.	20			

**As per neutralization formula**

$$N_1 V_1 = N_2 V_2$$

Where,  $N_1$  = Normality of Std F.A.S \_\_\_\_\_ N     $V_1$  = Volume of Std F.A.S = 20 ml

$N_2$  = Normality of KMnO<sub>4</sub> \_\_\_\_\_ ?     $V_2$  = Volume of KMnO<sub>4</sub> \_\_\_\_\_ Burette reading

$$N_1 V_1 = N_2 V_2$$

$$N_2 = \frac{N_1 V_1}{V_2} = \frac{\text{_____}}{\text{_____}} = N$$

Normality of KMnO<sub>4</sub> ( $N_2$ ) = \_\_\_\_\_ N

### **PART B: DETERMINATION OF KMnO<sub>4</sub>**

S.NO	VOLUME OF KMnO <sub>4</sub>	EMF	Δ E	Δ V	ΔE/ΔV
1.					
2.					
3.					
4.					
5.					
6.					
7.					
8.					
9.					
10.					
11.					
12.					
13.					
14.					
15.					
16.					
17.					
18.					

## **EXPERIMENT (8):**

### **Determination of Fe<sup>+2</sup> by Potentiometry using KMnO<sub>4</sub>**

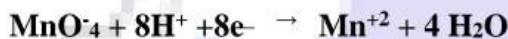
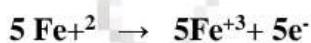
**Aim:** To estimate the amount of ferrous ion present in the given solution by using potentiometer

**Apparatus:** Potentiometer, platinum electrode, saturated calomel (reference ) electrode, beaker, glass rod, burette and burette stand

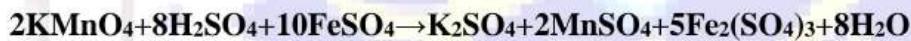
**Chemicals:** KMnO<sub>4</sub>, FAS(ferrous ammonium sulphate)

#### **Principle:**

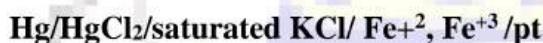
Ferrous ion oxidizes to ferric ion whose concentration increases with progressive addition of potassium permanganate and the observed EMF generally increases



The total reaction



The cell is constructed as



Addition of potassium permanganate increases the observed EMF. At the end point , there is sharp increases in EMF due to complete oxidation of ferrous ion to ferric ion

#### **PROCEDURE:**

##### **PART A : STANDARDIZATION OF GIVEN KMnO<sub>4</sub> SOLUTION**

Pipette out 20 ml of the prepared std. Ferrous Amm. Sulphate solution into a clean conical flask. Titrate with KMnO<sub>4</sub> (taken in the burette) to a faint pink color which persists for at least a minute. This is the end point. (Last addition of KMnO<sub>4</sub> should be drop wise with particular care to allow each drop to become decolorized before the next is added). Repeat the process till at least two concurrent values are obtained

##### **PART B: DETERMINATION OF Ferrous ions**

- Fill the burette with standard potassium permanganate KMnO<sub>4</sub> after rinsing the burette with KMnO<sub>4</sub> upto zero mark.
- Take 20 ml of given FAS solution into a clean beaker , and dip a platinum electrode and combine it to saturated calomel electrode by using a salt bridge.
- Connect the platinum electrode to the positive terminal and calomel electrode to the negative terminal in the potentiometer.
- Measure the EMF value initially.
- Now add from the burette KMnO<sub>4</sub> drop wise i. e., 1 ml for each addition. After each addition, stir the solution by using a glass rod and note down the EMF values.
- The measured EMF values are tabulated in the table.

### **CALUCLATION:**

Normality of KMnO<sub>4</sub> (N<sub>2</sub>) =

Volume of KMnO<sub>4</sub> consumed, end point from graph (V<sub>2</sub>) = .....ml

Normality of FAS solution (N<sub>3</sub>) = .....?

Volume of FAS solution (V<sub>3</sub>) = 20 ml

**As per neutralization formula**

$$N_3 V_3 = N_2 V_2$$

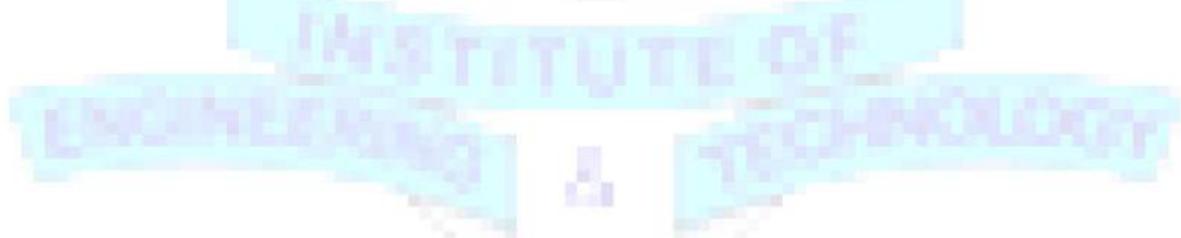
$$N_3 = \frac{N_2 V_2}{V_3} = \text{_____} = N$$

Normality (strength) of FAS solution (N<sub>3</sub>) = .....N

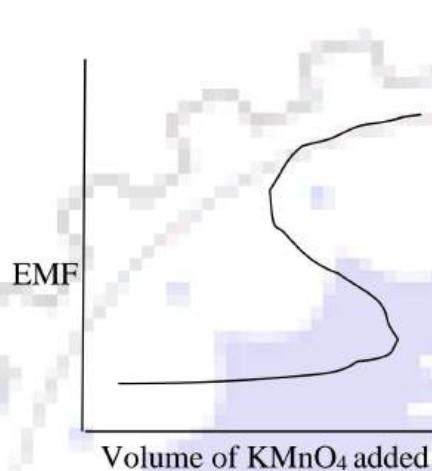
The amount of Fe+<sup>2</sup> in the given solution = N<sub>3</sub> x Eq. Wt of Fe+<sup>2</sup>

$$= N_3 \times \text{gms/lt.}$$

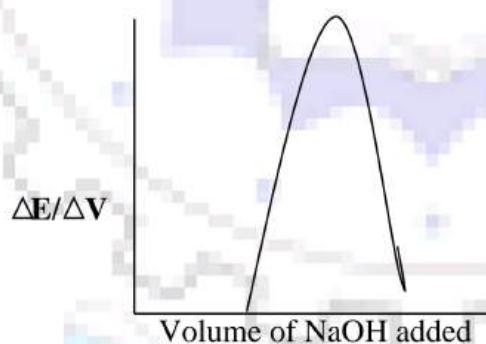
$$= \text{..... gms lt.}$$



**GRAPH:** Plot a graph between EMF against the volume of KMnO<sub>4</sub> added and a curve is obtained as shown below.



The point of inflection (where the curve changes its curvature) in the curve gives the equivalent point. The accurate value can be obtained by plotting a graph between  $\Delta E/\Delta V$  against the volume of the KMnO<sub>4</sub> added. The graph obtained is shown as below.



#### **RESULT:**

1. The normality of FAS solution = .....N
2. The amount of FAS in the given solution = .....gms/lit.

## **EXPERIMENT:9**

### **DETERMINATION OF KMnO<sub>4</sub> BY COLORIMETRIC METHOD**

#### **Part A: Standardization of KMnO<sub>4</sub>**

**Titration of Std. F.A.S. Vs. KMnO<sub>4</sub> :**

S.No	Volume of FAS (ml)	Burette Readings		Volume of KMnO <sub>4</sub> consumed
		Initial (ml)	Final (ml)	
1.	20			
2.	20			
3.	20			

**As per neutralization formula , N<sub>1</sub>V<sub>1</sub>= N<sub>2</sub>V<sub>2</sub>**

Where, N<sub>1</sub> = Normality of Standard F.A.S = \_\_\_\_\_ N

V<sub>1</sub> = Volume of Standard F.A.S = 20 ml

N<sub>2</sub> = Normality of KMnO<sub>4</sub> = \_\_\_\_\_ ?

V<sub>2</sub> = Volume of KMnO<sub>4</sub> = \_\_\_\_\_ (Burette reading)= ml

$$N_2 = \frac{N_1 V_1}{V_2} = \text{_____} = N$$

#### **PART B : DETERMINATION OF MANGANESE IN KMnO<sub>4</sub>**

S.No.	Volume of Distilled water	Volume of KMnO <sub>4</sub>	Concentration	Absorbance (or) OD Values
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				

## **EXPERIMENT:9**

### **DETERMINATION OF PERMANGANATE BY COLORIMETRIC METHOD**

**AIM:** To Determine the amount of Permanganate present in KMnO<sub>4</sub> Solution by colorimetry method.

**APPARTUS:** Photo Colorimeter, beaker, glass rod, pipette, burette, test tubes and cuvettes.

**CHEMICALS:** KMnO<sub>4</sub>, Distilled water.

**PRINCIPLE:** Colorimeter is an instrument used to measure the intensity of light absorbed by a substance. The relation between absorbance (A) and concentration of the solution(C) is given by Beer-Lambert's law.

$$\log \frac{I_0}{I} = A = \epsilon CX$$

Where,

I<sub>0</sub> = Intensity of incident light

I = Intensity of transmitted light

$\epsilon$  = Molar absorption coefficient (formerly called molar extinction coefficient)

X = Thickness of cell

C = Concentration of the solution

From the equation, it is seen that, the absorbance (A) is directly proportional the molar concentration and thickness of the cell.

#### **PROCEDURE:**

##### **PART A:**

##### **STANDARDIZATION OF GIVEN KMnO<sub>4</sub> SOLUTION:**

Pipette out 20 ml of the prepared std. Ferrous Ammonium Sulphate solution into a clean conical flask. Titrate with KMnO<sub>4</sub> (taken in the burette) to a faint pink color which persists for at least a minute. This is the end point. (Last addition of KMnO<sub>4</sub> should be drop wise with particular care to allow each drop to become decolorized before the next is added). Repeat the process till at least two concurrent values are obtained.

##### **PART B:**

##### **1. Preparation of various standard KMnO<sub>4</sub> solutions**

Now prepare a series of solutions of concentration 0.001N, 0.002N, 0.003N, 0.004N, 0.005N, 0.006N, 0.007N, 0.008N and 0.009N by transferring 1ml, 2ml, 3ml, 4ml, 5ml, 6ml, 7ml, 8ml, 9ml, 10ml stock solution respectively into the test tubes and make up to 10ml using distilled water.

**ROUGH WORK**



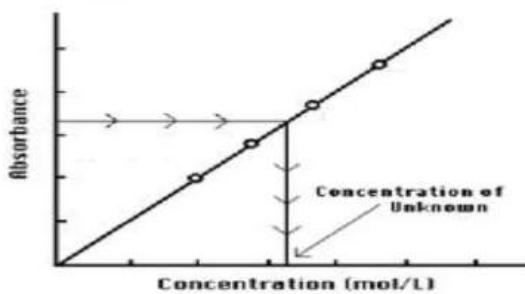
## **2. Colorimetric method**

- The transmittance of colorimeter is adjusted to 100% using distilled water i.e. set it zero
- Check the absorbance of highest concentrated solution (0.1N) by using different filters.
- Select the filter which is giving highest absorbance for the given solution and set it to zero by using blank (distilled water).
- Now measure the absorbance of all the solutions
- In the similar way measure the absorbance of given unknown solution

### **Calibration graph**

Plot a graph between the concentration along the X-axis and absorbance on the Y-axis to obtain the straight line. From the graph the concentration of unknown solution is measured.

The graph which is obtained is shown a



### **RESULT:**

The concentration of unknown sample is \_\_\_\_\_

The Amount of Permanganate in given Solution \_\_\_\_\_

**ROUGH WORK**



## **EXPERIMENT – 10**

### **PREPARATION OF ASPIRIN :**

**AIM :** To prepare a pure sample of Aspirin

**APPARATUS** 1. Measuring jar 2. Beaker 3. Conical flask 4. Glass rod 5. Watch glass 6. Funnel

### **CHEMICALS**

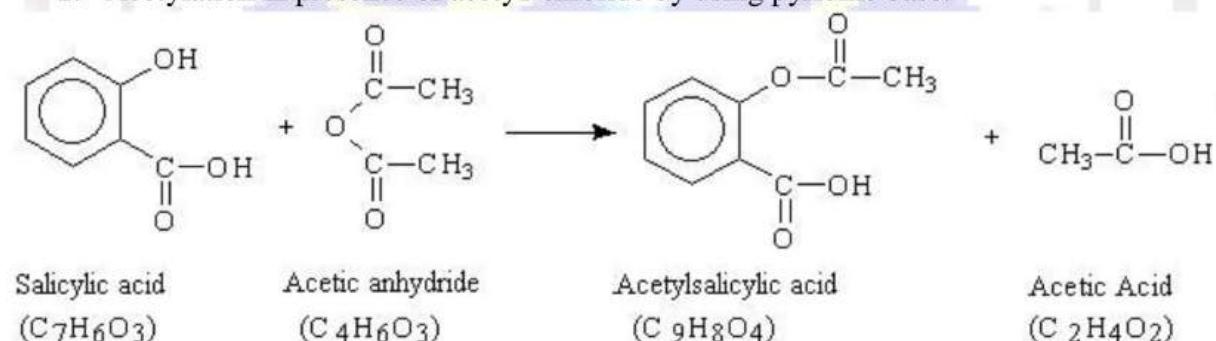
1. Salicylic acid 2. Acetic anhydride 3. Conc. Sulphuric acid

### **BASIC PRINCIPLE:**

Aspirin is an acetyl derivative of salicylic acid and prepared by the acetylation of salicylic acid.

the acetylation of salicylic acid is carried out in two ways.

1. Acetylation with acetic anhydride in presence of an acid.
2. Acetylation in presence of acetyl chloride by using pyridine base.



### **Procedure:**

Take 5 gms of salicylic acid in a 100 ml conical flask add about 10 ml of acetic anhydride: to this mixture 1-2 ml of conc .sulphuric acid . Shake the solution well and the temperature of the reaction mixture raises to 70° – 80 °C because of exothermic reaction. Maintain the temperature of the reaction at 70° – 80 °C for about 20 minutes by heating it on water bath. Allow the solution to cool to room temperature and pour the solution into 100 ml of cold water taken in a 250 ml beaker with stirring (addition to water is to destroy excess of reagent). Scratch the sides of the beaker with a glass rod to induce crystallisation and stir the solution till complete precipitation. Filter the colourless, crystalline solid and wash it with cold water. Spread the solid aspirin on a filter paper and dry.

Recrystallise the crude aspirin sample from a mixture of ethanol and water ( 1 : 1 ). Find the melting point of the sample.

The melting point of aspirin reported is 130° – 135 ° C.

**ROUGH WORK**



**Precautions:**

1. Take acetyl chloride in excess as it can act as acetylating reagent and solvent.
2. Make sure that all salicylic acid is dissolved in acetylating mixture.
3. The presence of unreacted salicylic acid is checked by adding a drop of 1% ferric chloride solution to the reaction mixture and observing the colour. Formation of intense colour indicates the presence of unreacted salicylic acid in that case recrystallisation is compulsory.

**Pharmaceutical applications of Aspirin**

**RESULT:**

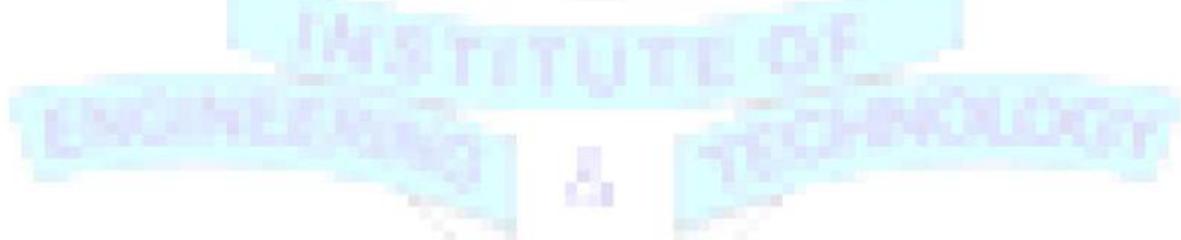
Amount of aspirin obtained: \_\_\_\_\_ gms

## **EXPERIMENT - 11**

### **DETERMINATION OF RATE CONSTANT OF ACID CATALYSED HYDROLYSIS OF METHYL ACETATE:**

#### **Observation**

s.n o	Time (min )	Volu me of soluti on taken	Burette reading		Volume of NaOH consume d (ml)	$(V_{\infty} - V_t)$ (ml)	$\log \frac{V_{\infty} - V_t}{V_t}$	$2.303 \frac{V_{\infty} - V_0}{v_{\infty} - vt}$ : (min <sup>-1</sup> )	$\log$
			Initi al	final					



## **EXPERIMENT - 11**

### **DETERMINATION OF RATE CONSTANT OF ACID CATALYSED HYDROLYSIS OF METHYL ACETATE :**

#### **Aim :**

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

#### **Basic principle :**

The hydrolysis of ester occurs according to the equation



The reaction follows pseudo first order kinetics

In the presence of an acid, this reaction should be of second order, since two molecules are reacting. But, it is found to be first order. This may be explained in the following way :

The rate of the reaction is given by

$$dx/dt = k' [\text{CH}_3\text{COOCH}_3] [\text{H}_2\text{O}]$$

where  $k'$  is the rate constant (or specific rate constant). Since water is present in large excess, its active mass (molar concentration) virtually remains constant during the course of the reaction. Therefore, its active mass gets included in the constant, and the above equation reduces to :

$$dx/dt = k_1 [\text{CH}_3\text{COOCH}_3]$$

Thus, the rate of the reaction is determined by one concentration term only (that is, by a single power of the concentration term only). Hence, the reaction is first order.

Such reactions are also referred to as pseudo first order reactions. The progress of the reaction (hydrolysis of ester) is followed by removing a definite volume of the reaction mixture, at definite intervals of time, cooling it in ice, and titrating the acetic acid formed against alkali, which has already been standardized. The amount of alkali used is equivalent to the total amount of hydrochloric acid present originally and the amount of acetic acid formed in the reaction. The amount of acetic acid formed ( $x$ ), at definite intervals of time ( $t$ ), can be obtained. The amount of acetic acid formed, at the end of the reaction, is equivalent to the initial concentration ( $a$ ) of the ester. Suppose the volumes of the sodium hydroxide solution (titre value) required for neutralization of 5 ml of the reaction mixture are :

- (i) at the commencement of the reaction is  $V_0$
- (ii) after time ( $t$ ) is  $V_t$
- (iii) at the end of the reaction is  $V_\infty$

Then

$x$  (amount of acetic acid formed after time  $t$ ) is proportional to  $(V_t - V_0)$

$a$  (initial concentration of ester) is proportional to  $(V_\infty - V_0)$

$[a - x]$  (concentration of ester present after time  $t$ ) is proportional to

**ROUGH WORK**



$$(V_{\infty} - V_0) - (V_t - V_0) = (V_{\infty} - V_t)$$

The first order rate expression given by :

$$k_1 = 2.303 \log \frac{a}{a-x}$$
 would correspond to :

$$k_1 = 2.303 \log \frac{V_{\infty} - V_0}{V_{\infty} - V_t}$$

### **Procedure:**

#### **DETERMINATION OF RATE CONSTANT (K1) FOR THE ACID-CATALYZED HYDROLYSIS OF METHYL ACETATE.**

1. 100 ml of given HCl (whose strength is determined in step II) solution is taken in a stoppered reagent bottle.
2. 5 ml of methyl acetate solution is added to the HCl solution. Note the time when half of the methyl acetate solution is added. The mixture is shaken well.
3. Pipette out 5 ml of the reaction mixture and discharge it into 50 ml of ice cold water kept in a conical flask.
4. Titrate the reaction mixture against NaOH solution using phenolphthalein as indicator. This titre value corresponds to  $V_0$ .
5. Steps 3 and 4 are repeated at intervals of 5, 10, 15, 20, 30, 45, 60 minutes. Each titre value corresponds to  $V_t$ .
6. The remaining solution is taken in a stoppered conical flask and heated to 60°C, and kept at this temperature for 5 minutes.
7. The solution is allowed to cool to room temperature.
8. Repeat Steps 3 and 4. This titre value corresponds to  $V_{\infty}$  till concurrent values are obtained.
9. **Plot a graph** of  $\log(V_{\infty} - V_t)$  versus time ( $t$ ) and determine the slope.
10. Report the theoretical and graphical value of rate constant ( $k_1$ ).

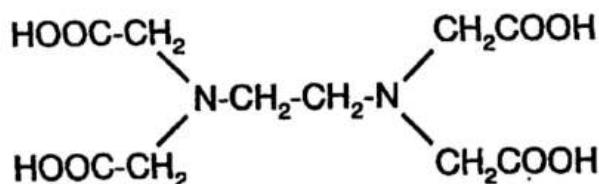
### **RESULT:**

Rate Constant ( $k_1$ ) for the acid-catalyzed hydrolysis of methyl acetate ( $\text{CH}_3\text{COOCH}_3$ ) at ....  
°C = \_\_\_\_\_ (theoretical). = \_\_\_\_\_ (graphical).

## **CHEMISTRY VIVA QUESTIONS**

1. What is EDTA? Write the structure of EDTA.

ANS: Ethylene Diamine Tetra Acetic acid



2. What is EBT?

ANS: Eriochrome black-T, used as an indicator in EDTA experiment.

3. What is pH? Write the formula for it. What is its range?

Ans: pH is to find out whether the solution is acid or basic (or) neutral.

pH = -log<sub>10</sub>[H<sup>+</sup>] The pH range is 0 to 14.

4. What is a buffer solution? Give examples.

Ans: Buffer solution is the one which maintains the pH of a solution constant.

Ammonia buffer = Ammonia chloride and liquid ammonia.

5. What the pH range is to be maintained around 10 in EDTA titration?

Ans: In order to get the end point, steel blue colour. It is the colour of the indicator EBT.

6. On what principle the colour changes from wine red to steel blue?

Ans: Hard water + EBT Metal ion –indicator complex(wine red colour) Metal ion-indicator complex + EDTA Metal ion – EDTA complex + Indicator

7. What is alkalinity?

Ans: A measure of acid neutralizing ability of a substance

8. Which ions impart alkalinity to the natural water?

Ans : Carbonate, bicarbonate and hydroxide ions.

9. How many types of alkalinity are possible?

Ans: 1. Carbonate alkalinity 2. Bicarbonate alkalinity and 3. Hydroxide alkalinity

10. What is a standard solution ?

Ans: A solution whose strength is known is called a standard solution.

11. What is a normal solution ?

Ans: A solution containing one gram-equivalent mass of the solute per litre of the solution is called a normal solution.

12.What is the equivalent mass of KMnO<sub>4</sub> when it acts as oxidizing agent in acidic medium ?  
Answer. KMnO<sub>4</sub> loses 5 electrons per molecule, when it acts as oxidizing agent in the presence of acids. Therefore, its equivalent mass is one-fifth of its molecular mass.

$$\text{Eq. mass} = \frac{\text{Mol. mass}}{5} = \frac{158}{5} = 31.6.$$

13.What is redox reaction?

Ans: A chemical reaction in which both oxidation and reduction reaction takes place.

14.What is Oxidation?

Ans: Oxidation is

- a) Loss of Electrons
- b) Increase in Oxidation number
- c) Gain of Oxygen
- d) Loss of Hydrogen

15.What is Reduction?

Ans:-Reduction is

- a) Gain of Electrons
- b) Decrease in Oxidation number
- c) Loss of Oxygen
- d) Gain of Hydrogen

16. What is the indicator used in this reaction?

Ans: No indicator is used. KMnO<sub>4</sub> acts as a self indicator.

17. What is the color change in this reaction?

Ans: Colorless to pale pink.

## EXPERIMENT – 6 (DETERMINATION OF pH)

1. What is pH?

Ans:- pH is the negative logarithm of hydrogen ion concentration  
 $pH = -\log [H^+]$

2. Write the pH range?

Ans:- pH range is from 0 to 14.

3. What happens when a base is added to an acid?

Ans:- The pH value increases.

4. What is buffer solution?

Ans:- The solution which resists the change in the pH even on the slight addition of acid or base.

5. Name the electrode used in the determination of pH.

Ans:- Glass combined electrode.

6. Why you need to calibrate a pH meter?

Ans:- The characteristic of a pH electrode will change with time due to electrode coating and aging. And even a pH electrode would be stable over time, pH electrodes cannot be produced with identical characteristics.

In practice the response of a real pH sensor does not exactly follow the Nernst equation. This difference between the theoretical and actual behavior of a pH electrode must be compensated for. A calibration is required to match the pH meter to the current characteristics of the used pH sensor.

7. What is pH of blood?

Ans:- 7.35 – 7.45.

8. What is pH of pure water?

Ans:- At 27°C, pH = 6.83, at 25°C = 7.00, at 100°C = 6.14

9. What is pH of soil?

Ans:- 6-8



## EXPERIMENT - 7 & 8 (CONDUCTOMETRIC TITRATIONS)

1. When strong acid combines with a strong base what type of reaction occurs?

Ans:- Acid and a base combines to form salt and water. The reaction is called as neutralization reaction.

2. Name the apparatus used for this method?

Ans:- Conductivity meter with a conductivity cell.

3. How conductance is related to the concentration of the ions?

Ans:- The specific conductance is proportional to the concentration of ions in it.

4. How the end point for a particular reaction is calculated using this, titration method?

Ans:- On plotting a graph between conductance and volume of the base, the point of intersection of the straight lines gives the end point.

5. Why conductance decreases on addition of NaOH to HCl ?

Ans:- 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.

6. What is the unit for conductance?

Ans:- The unit for conductance is mho or Siemens.

7. State Ohm's law?

Ans:- Ohm's law states that the current through a conductor between two points is directly proportional to the potential difference across the two points. ( $V = IR$ )

8. What is specific conductivity?

Ans:- It is the measure of the ability of the electrolytic solution to conduct electricity.

9. What is equivalent conductivity?

Ans:- The conductance of that volume of solution containing one equivalent of an electrolyte is known as equivalent conductivity. It is denoted by  $\Lambda$ .

10. What is molar conductivity?

Ans:- The conductance of that volume of solution containing one mole of an electrolyte is known as molar conductivity. It is denoted by  $\Lambda_m$  or  $\mu$ .

11. What is conductance?

Ans:- It is the tendency of a material to allow the flow of current through it. It is the reciprocal of resistance.

12. Name the conductivity which is measured during conductometric titrations?

Ans:- Equivalent conductance.

13. The electrical conductivity of a solution depends upon \_\_\_\_\_.

Ans:- The number of ions present in the solution and their mobility.

## **EXPERIMENT - 9 (POTENTIOMETRIC TITRATIONS)**

1. What are the electrodes used in the potentiometric titrations?

Ans:- Reference Electrode – Calomel electrode  
Indicator electrode – Platinum electrode

2. What is the Principle of Potentiometric titration?

Ans:- Nernst equation

3. What is the indicator electrode?

Ans:- The electrode with which we measure the potential of the test solution is called indicator electrode.

4. What is reference electrode? Give examples.

Ans:- The potential of unknown electrode can be measured by coupling it with another electrode called reference electrode whose potential is already known or arbitrarily zero. Example: calomel electrode, standard hydrogen electrode.

5. What is calomel electrode?

Ans:- It is a secondary reference electrode containing mercury, mercurous chloride and solution of KCl.

6. Write the  $E^\circ$  values for a calomel electrode.

Ans:-  
1. In 1N KCl solution = 0.2800V  
2. In saturated KCl solution = 0.2422V  
3. In 0.1N KCl solution = 0.3338V

7. What is the advantage of potentiometric titrations?

Ans:- they are particularly useful with colored or turbid solutions and for detecting the presence of unsuspected species.

## **EXPERIMENT - 10 (Colorimetry)**

1. What is Beer's law?
2. What is Lambert's law?
3. What is the principle involved in colorimetry?
4. What is the wave length region of visible region?
5. Which is the source of light in colorimetry?

Ans:- Tungsten lamp.

Apart from above..generalized questions will be asked.