

CHEMISTRY LABORATORY

For I Semester B.E., / B.Tech., Students Common to All Branches
(CBCS Pattern)
Regulation 2021



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N.Karthika

ENGINEERING CHEMISTRY LAB MANUAL

**(I SEMESTER B.E/B.TECH STUDENTS COMMON TO ALL
BRANCHES)**

REGULATION – 2021

AUTHOR:

Mrs. N. KARTHIKA, M.Sc, M.Phil.,

GENERAL GUIDELINES AND SAFETY INSTRUCTIONS

1. Sign in the log register as soon as you enter the lab and strictly observe your lab timings.
2. Strictly follow the written and verbal instructions given by the teacher / Lab Instructor. If you do not understand the instructions, the handouts and the procedures, ask the instructor or teacher.
3. **Never work alone!** You should be accompanied by your laboratory partner and / or the instructors / teaching assistants all the time.
4. It is mandatory to come to lab in a formal dress and wear your ID cards.
5. Do not wear loose-fitting clothing or jewels in the lab. Rings and necklaces are usual excellent conductors of electricity.
6. Mobile phones should be switched off in the lab. Keep bags in the bag rack.
7. Keep the labs clean at all times, no food and drinks allowed inside the lab.
8. Intentional misconduct will lead to expulsion from the lab.
9. Do not handle any equipment without reading the safety instructions. Read the handout and procedures in the Lab Manual before starting the experiments.
10. Do your wiring, setup, and a careful circuit checkout before applying power. Do not make circuit changes or perform any wiring when power is on.
11. Avoid contact with energized electrical circuits.
12. Do not insert connectors forcefully into the sockets.
13. **NEVER** try to experiment with the power from the wall plug.
14. Immediately report dangerous or exceptional conditions to the Lab instructor / teacher: Equipment that is not working as expected, wires or connectors are broken, the equipment that smells or “smokes”. If you are not sure what the problem is or what's going on, switch off the Emergency shutdown.
15. Never use damaged instruments, wires or connectors. Hand over these parts to the Lab instructor/Teacher.
16. Be sure of location of fire extinguishers and first aid kits in the laboratory.
17. After completion of Experiment, return the bread board, trainer kits, wires, CRO probes and other components to lab staff. Do not take any item from the lab without permission.
18. Observation book and lab record should be carried to each lab. Readings of current lab experiment are to be entered in Observation book and previous lab experiment should be written in Lab record book. Both the books should be corrected by the faculty in each lab.

**I SEMESTER B.E/B.TECH STUDENTS COMMON
TO ALL BRANCHES**

(DEPARTMENT OF SCIENCE AND HUMANITIES)

REGULATION – 2021

**BS 3171 - ENGINEERING CHEMISTRY
LABORATORY**

Mrs.N.KARTHIKA,M.Sc,M.Phil.,

Assistant Professor/Chemistry

Annai Mira College of Engineering and Technology

Ranipet – 632 517

PREFACE

This book on “**ENGINEERING CHEMISTRY LABORATORY MANUAL (I semester B.E/B.TECH students common to all branches)**” covers the complete syllabus prescribed by the Anna University, Chennai , course under **Outcome Based Education Credit System with the new regulation 2021**.

This book deals with both volumetric analysis and physical chemistry experiments, has been presented systematically and lucidly.

We hope that this book will be useful to both teachers and students. Finally we would request the readers to kindly send their valuable comments and suggestions towards the improvement of the manual and the same will be gratefully acknowledge.

Any suggestion from the reader for the betterment of this book can be dropped into karthikarajanmsc@gmail.com.

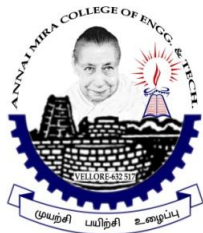
Mrs.N.KARTHIKA,M.Sc,M.Phil.,

ACKNOWLEDGEMENT

We are thankful to and fortunate enough to get constant encouragement, support and guideline from Chairman **Thiru.S.Ramadoss Ayya**, Secretary & Treasurer **Mr.G.Thamotharan** for his blessings to complete the book successfully.

We would not forget to remember our Principal **Dr.T.K.Gopinathan** for his constant assistance in preparing this book.

ANNAI MIRA COLLEGE OF ENGINEERING AND TECHNOLOGY



I SEMESTER B.E/B.TECH STUDENTS COMMON TO ALL BRANCHES

DEPARTMENT OF SCIENCE AND HUMANITIES

LAB MANUAL (Regulation - 2021)

Subject Code / Name : BS3171 / Engineering Chemistry Lab
Semester/Year : I / I – (B.E / B.Tech Common to All branches)

PREPARED BY

Mrs.N.KARTHIKA,M.Sc,M.Phil.,
Assistant Professor / Chemistry

APPROVED BY

K. SURESH. M.Sc,M.Phil.,Ph.D
HOD / S&H

I SEMESTER B.E/B.TECH STUDENTS COMMON TO ALL BRANCHES

Department of Science and Humanities

BS3171 ENGINEERING CHEMISTRY LABORATORY

List of Experiments

1. Preparation of Na_2CO_3 as a primary standard and estimation of acidity of a watersample using the primary standard
2. Determination of types and amount of alkalinity in water sample.
3. Determination of total, temporary & permanent hardness of water by EDTA method.
4. Determination of DO content of water sample by winkler's methods.
5. Determination of chloride content of water sample by argentometric method.
6. Estimation of copper content of the given solution by Iodometry
7. Determination of strength of acids in a mixture of acids using conductivity meter.

TOTAL = 45 PERIODS

INDEX

S.NO	NAME OF EXPERIMENT	PAGE NO.
1	Preparation of Na_2CO_3 as a primary standard and estimation of acidity of a water sample using the primary standard	1
2	Determination of types and amount of alkalinity in water sample.	5
3	Determination of total, temporary & permanent hardness of water by EDTA method.	15
4	Determination of DO content of water sample by winkler's methods.	21
5	Determination of chloride content of water sample by argentometric method.	25
6	Estimation of copper content of the given solution by Iodometry	29
7	Determination of strength of acids in a mixture of acids using conductivity meter.	33

TABLE OF CONTENTS

S.NO.	DATE	NAME OF THE EXPERIMENT	SIGNATURE
1		Preparation of Na_2CO_3 as a primary standard and estimation of acidity of a water sample using the primary standard	
2		Determination of types and amount of alkalinity in water sample.	
3		Determination of total, temporary & permanent hardness of water by EDTA method.	
4		Determination of DO content of water sample by Winkler's methods.	
5		Estimation of copper content of the given solution by iodometry	
6		Determination of chloride content of water sample by argentometric method.	
7		Determination of strength of acids in a mixture of acids using conductivity meter.	

Calculation of Molarity of Na_2CO_3

Weight of salt + bottle (W_1) = gm

Weight of empty bottle (W_2) = gm

Weight of salt $W = (W_1 - W_2) = \dots\dots\dots$ gm

$$\begin{aligned} \text{The Molarity of standard solution } M_1 &\equiv \left(\frac{W}{\text{Mol.weight}} \right) \times \left(\frac{1000}{\text{volume of solution}} \right) \\ &= \left(\frac{W}{106} \right) \times \left(\frac{1000}{100} \right) \\ &= \underline{\hspace{2cm}} \text{ M} \end{aligned}$$

1. PREPARATION OF Na₂CO₃ AS A PRIMARY STANDARD AND ESTIMATION OF ACIDITY OF WATER SAMPLE USING THE PRIMARY STANDARD

AIM:

- a) To prepare Na₂CO₃ solution as a primary standard using anhydrous Na₂CO₃ crystal.
- b) To estimate the acidity of given water sample using a standard Na₂CO₃ solution.

MATERIALS REQUIRED:

1. Burette 2. Measuring Jar, Conical Flask, Weighing Bottle, Std. Flask, Pipette, Burette, Electronic Balance.

PRINCIPLE:

Acidity is generally measured by titration with sodium carbonate to an accepted pH value. Hydrogen ions present, in a sample as a result of dissociation (or) hydrolysis of solutes, reacts with additions of standard alkali (Na₂CO₃). Acidity thus depends on end point of the indicator use. The colour change of phenolphthalein indicator is close to pH at 25°C corresponds to stoichiometric neutralization of carbonic acid to bicarbonate.



PROCEDURE:

A) Preparation of standard Solution:

Take approximately 1.06gm of the Na₂CO₃ salt in a weighing bottle and weigh it accurately by using digital balance. Now, transfer the salt into a 100 mL standard flask through funnel and dissolve the salt with minimum amount of distilled water. Then make-up the solution up to the mark of the standard flask and shake well to get uniform concentration. Later, the Molarity of Na₂CO₃ solution (M₁) can be calculated.

TITRATION

Water sample Vs standard Na_2CO_3

S.No.	Volume of water sample (mL) V_3	Burette reading (mL)		Concordant value (mL)		
		Initial	Final		[M] V_1	[P] V_2
			At methyl orange endpoint [M]	At phenolphthalein endpoint [P]		
1						
2						
3						

CALCULATION

1. Mineral acidity

Volume of Na_2CO_3 (V_1) (M) = mL

Normality of Na_2CO_3 (N_1) = N

Volume of water sample (V_3) = mL

Strength of water sample (acidity) (N_2) = N

$$\text{Acidity } (N_2) = V_1 N_1 / V_3$$

Mineral acidity = N

1. Total acidity

Volume of Na_2CO_3 (V_2) (P) = mL

Normality of Na_2CO_3 (N_1) = N

Volume of water sample (V_3) = mL

Strength of water sample (acidity) (N_2) = N

$$\text{Total acidity } (N_2) = V_1 N_1 / V_3$$

Total acidity = N

B) Estimation of acidity of water:

Burette is washed with distilled water and rinsed with standard Na_2CO_3 solution. Then the burette is filled with the same solution up to the zero mark without any air bubbles.

20 mL of water sample is pipetted out into a clean conical flask then few drops of methyl orange indicator is added to the conical flask. The solution is titrated against Na_2CO_3 solution till the colour changes from orange red to yellow. The volume consumed by water sample is noted. The 2 to 3 drops of phenolphthalein indicator is added to the same solution. The titration is continued till the colour of water sample is turned to pale pink. The end point is noted. The titration is repeated until the two concordant values are obtained.

RESULT:

i) Molarity of the Na_2CO_3 solution = M

ii) The given sample contains

1. Mineral acidity =
2. Total acidity =

Titration – I

HCl Vs Std. NaOH

S.No.	Volume of NaOH (mL)	Burette reading (mL)		Concordant volume of HCl (mL)	Indicator
		Initial	Final		
1	20	0			Phenolphthalein
2	20	0			
3	20	0			

Volume of NaOH $V_1 = 20\text{ml}$

Normality of NaOH $N_1 = \dots\dots\dots\text{N}$

Volume of HCl $V_2 = \dots\dots\dots\text{ml}$

Normality of HCl $N_2 = \dots\dots\dots\text{N}$

According to the law of volumetric analysis,

$$V_1 N_1 = V_2 N_2$$

$$N_2 = V_1 \times N_1 / V_2$$

$$= 20 \times \dots\dots\dots\text{N} / \dots\dots\dots\text{ml}$$

$$= \dots\dots\dots\text{N}$$

TITRATION – II

Water sample – I Vs Std. HCl

S.NO.	Volume of water sample (ml)	Burette reading (ml)		Concordant volume of HCl (ml)	
		Phenolphthalein end point [P]	Methyl orange end point [M]	[P]	[M]
1	20				
2	20				
3	20				

2. DETERMINATION OF TYPES OF AND AMOUNT OF ALKALINITY IN WATER SAMPLE

AIM

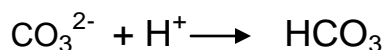
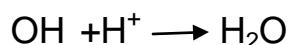
To determine the types and amount of alkalinity present in the given water sample. A standard solution of sodium hydroxide of strength N and a known solution of hydrochloric acid are provided.

PRINCIPLE

Alkalinity caused by the presence of hydroxide, carbonate and bicarbonate. These are five alkalinity conditions possible in a given sample of water, hydroxide only, carbonate only, bicarbonate only, combination of hydroxide and carbonate or carbonate and bicarbonate. The various alkalinities can be determined by titrating with a standard acid using phenolphthalein and methyl orange indicators successively.

1. Phenolphthalein end point

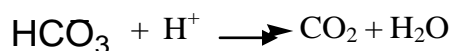
When alkaline water is titrated with acid using Phenolphthalein indicator, hydroxide alkalinity is completely neutralized.



2. Methyl orange end point

After the phenolphthalein end point, Methyl orange indicator is added and titrated with acid.

Bicarbonate neutralization occurs.



From the two titre values the different alkalinities are calculated.

When, $P=M$, \rightarrow Hydroxide

alkalinity $2P=M$, \rightarrow Carbonate

alkalinity

$P=0$, \rightarrow Bicarbonate alkalinity

CALCULATION

I. If the data satisfied the condition $P > \frac{1}{2} M$

(i) Volume HCl required for $[\text{OH}^-]$ Alkalinity = $2[P] - [M]$

$$= 2 \dots\dots\dots - \dots\dots\dots$$

$$= \dots\dots\dots \text{ ml}$$

(ii) Volume of HCl required for $[\text{CO}_3^{2-}]$ alkalinity = $2[M] - 2[P]$

$$= 2 \times \dots\dots\dots - 2 \times \dots\dots\dots$$

$$= \dots\dots\dots \text{ ml}$$

(iii) HCO_3^- is not present.

1. Calculation of OH^- alkalinity

Volume of HCl $V_1 = \dots\dots\dots \text{ ml}$

Strength of HCl $N_1 = \dots\dots\dots \text{ N}$

Volume of water sample $V_2 = 20 \text{ ml}$

Strength of water sample } $N_2 = ?$
 (OH⁻ alkalinity)

According to the law of volumetric analysis,

$$V_1 N_1 = V_2 N_2$$

$$N_2 = V_1 \times N_1 / V_2$$

$$= \dots\dots\dots \text{ ml} \times \dots\dots\dots \text{ N} / 20 \text{ ml}$$

$$= \dots\dots\dots \text{ N}$$

\therefore Amount of OH^- content present in 1 litre }
 of water sample, in terms of CaCO_3 equivalent } = Strength of water sample x equ. wt. of CaCO_3

i.e., OH^- alkalinity in terms of CaCO_3 equivalent = $\dots\dots\dots \text{ N} \times 50 \times 1000 \text{ ppm}$

Alkalinity due to OH^- ions = $\dots\dots\dots \text{ ppm}$.

$P < \frac{1}{2}M$, → Carbonate and bicarbonate alkalinity

$$CO_3^{2-} = 2P$$

$$HCO_3^- = M - 2P$$

$P > \frac{1}{2}M$ → Carbonate and hydroxide alkalinity

$$CO_3^{2-} = 2(M - P)$$

$$OH^- = 2P - M$$

Alkalinity values are expressed in terms of milligrams per litre as calcium carbonate.

TABLE

Titre value and different alkalinities

S.NO.	Result of titration of [P] and [M]	Hardness causing ions		
		OH^-	CO_3^{2-}	HCO_3^-
1	[P] = 0	0	0	[M]
2	[P] = [M]	[P] or [M]	0	0
3	$[P] = \frac{1}{2}[M]$	0	2[P] or [M]	0
4	$[P] > \frac{1}{2}[M]$	2[P] - [M]	2[M] - 2[P]	0
5	$[P] < \frac{1}{2}[M]$	0	2[P]	[M - 2[P]]

2. Calculation of CO_3^{2-} alkalinity

Volume of HCl $V_1 = \dots\dots\dots$ ml

Strength of HCl $N_1 = \dots\dots\dots$ N

Volume of water sample $V_2 = 20$ ml

Strength of water sample } $N_2 = ?$
 (CO_3^{2-} alkalinity)

According to the law of volumetric analysis,

$$V_1 N_1 = V_2 N_2$$

$$N_2 = V_1 \times N_1 / V_2$$

$$N_2 = \dots\dots\dots \text{ml} \times \dots\dots \text{N} / 20 \text{ ml}$$

$$= \dots\dots\dots \text{N}$$

\therefore Amount of CO_3^{2-} content present in 1 litre of water sample, in terms of CaCO_3 equivalent } = \text{Strength of water sample} \times \text{equ. wt. of } \text{CaCO}_3

i.e., CO_3^{2-} alkalinity in terms of CaCO_3 equivalent = $\dots\dots\dots$ N $\times 50 \times 1000$ ppm

Alkalinity due to CO_3^{2-} ions = $\dots\dots\dots$ ppm.

TITRATION – III

Water sample – II Vs Std. HCl

S.No.	Volume of water sample(ml)	Burette reading (ml)		Concordant volume of HCl (ml)	
		Phenolphthale in end point [P]	Methyl orange end point [M]	[P]	[M]
1	20				
2	20				
3	20				

PROCEDURE

TITRATION – I

Standardisation of HCl

The burette is washed well with distilled water and rinsed with the small amount of hydrochloric acid solution. It is then filled with the same solution up to the zero mark without air bubbles. 20 mL of the standard sodium hydroxide solution is pipette out in a clean conical flask. 2-3 drops of phenolphthalein indicator is added, the colour turn to pink colour. It is then titrated against hydrochloric acid taken in the burette. The end point is disappearance of pink colour. The titration is repeated to get concordant values, the normality of HCl is calculated.

TITRATION – II

Water sample-I Vs Std.HCl

The pipette is rinsed with the small amount of water sample then 20 mL of the same water sample is pipette out in a clean conical flask. A drop of phenolphthalein indicator is added. Pink colour is observed. This solution is titrated against standard hydrochloric acid taken in the burette, until pink colour is disappeared. The end point is noted. This titre value corresponds to phenolphthalein end point (P). Few drops of methyl orange is added to the same solution after the phenolphthalein end point. The titration continued until the solution become red orange. The total titre value is noted. This titre value corresponds to methyl orange end point (M). The titration is repeated to get concordant values. From the titre value the amount of alkalinity present in the given water sample is calculated.

TITRATION – III

Water sample-II Vs Std.HCl

20 mL of the water sample is pipette out in a clean conical flask. A drop of phenolphthalein indicator is added. Pink colour is observed. This solution is titrated against standard hydrochloric acid taken in the burette, until pink colour is disappeared. The end point is noted. This titre value corresponds to phenolphthalein end point (P). Few drops of methyl orange is added to the same solution after the phenolphthalein end point. The titration continued until the solution become red orange. The total titre value is noted. This titre value corresponds to methyl orange end point (M). The titration is repeated to get concordant values. From the titre value the amount of alkalinity present in the given water sample is calculated.

CALCULATION

II. If the data satisfied the condition $P < \frac{1}{2} M$

(i) Volume HCl required for $[\text{CO}_3^{2-}]$ Alkalinity = $2[P]$

$$= 2 \dots\dots\dots$$

$$= \dots\dots\dots \text{ ml}$$

(ii) Volume of HCl required for $[\text{HCO}_3^{2-}]$ alkalinity = $[M] - 2[P]$

$$= \dots\dots\dots - 2 \times \dots\dots\dots$$

$$= \dots\dots\dots \text{ ml}$$

(iii) OH^- is not present.

1. Calculation of CO_3^{2-} alkalinity

Volume of HCl $V_1 = \dots\dots\dots \text{ ml}$

Strength of HCl $N_1 = \dots\dots\dots \text{ N}$

Volume of water sample $V_2 = 20 \text{ ml}$

Strength of water sample } $N_2 = ?$
 $(\text{CO}_3^{2-} \text{ alkalinity})$

According to the law of volumetric analysis, $V_1 N_1 = V_2 N_2$

$$N_2 = V_1 \times N_1 / V_2$$

$$= \dots\dots\dots \text{ ml} \times \dots\dots\dots \text{ N} / 20 \text{ ml}$$

$$= \dots\dots\dots \text{ N}$$

\therefore Amount of CO_3^{2-} content present in 1 litre }
of water sample, in terms of CaCO_3 equivalent } = \text{Strength of water sample} \times \text{equ. wt. of } \text{CaCO}_3

i.e., CO_3^{2-} alkalinity in terms of CaCO_3 equivalent = $\dots\dots\dots \text{ N} \times 50 \times 1000 \text{ ppm}$

Alkalinity due to CO_3^{2-} ions = $\dots\dots\dots \text{ ppm}$.

2. Calculation of HCO_3^- alkalinity

Volume of HCl $V_1 = \dots\dots\dots \text{ml}$

Strength of HCl $N_1 = \dots\dots\dots \text{N}$

Volume of water sample $V_2 = 20 \text{ ml}$

Strength of water
sample (HCO_3^-
alkalinity) } $N_2 = ?$

According to the law of volumetric analysis,

$$V_1 N_1 = V_2 N_2$$

$$N_2 = V_1 \times N_1 / V_2$$

$$N_2 = \dots\dots\dots \text{ml} \times \dots\dots \text{N} / 20 \text{ ml}$$

$$= \dots\dots\dots \text{N}$$

\therefore Amount of HCO_3^- content present in 1
litre of water sample, in terms of CaCO_3
equivalent } = \text{Strength of water sample} \times \text{equ. wt. of } \text{CaCO}_3

i.e., HCO_3^- alkalinity in terms of CaCO_3 equivalent = $\dots\dots\dots \text{N} \times 50 \times 1000 \text{ ppm}$

Alkalinity due to HCO_3^- ions = $\dots\dots\dots \text{ppm}$.

RESULT

1. Water sample-I contain the following alkalinity

(i) Hydroxide alkalinity = _____ppm

(ii) Carbonate alkalinity = _____ppm

(iii) Total alkalinity = _____ppm

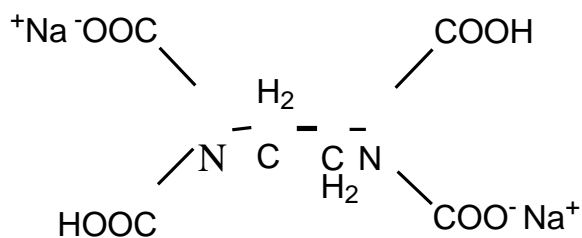
2. Water sample-II contain the following alkalinity

(i) Carbonate alkalinity = _____ppm

(ii) Bicarbonate alkalinity = _____ppm

(iii) Total alkalinity = _____ppm

Structure of disodium salt of EDTA



STANDARDISATION OF EDTA

TITRATION – I

Standard Hard water Vs EDTA

S.No.	Volume of Standard hardwater (mL)	Burette reading (mL)		Concordant volume of EDTA(mL)	Indicator
		Initial	Final		
1	20	0			
2	20	0			
3	20	0			

CALCULATION

Volume of standard hard water $V_1 = 20$ mL

Strength of standard hard water $N_1 = \dots\dots\dots$ N

Volume of EDTA $V_2 = \dots\dots\dots$ mL

Strength of EDTA $N_2 = \dots\dots\dots$ N

According to the law of volumetric analysis

$$V_1 N_1 = V_2 N_2$$

$$N_2 = V_1 \times N_1 / V_2$$

$$= 20 \times \dots\dots\dots N / \dots\dots\dots \text{mL}$$

Strength of EDTA $N_2 = \dots\dots\dots$ N

3. DETERMINATION OF TOTAL, TEMPORARY AND PERMANENT HARDNESS OF WATER BY EDTA METHOD

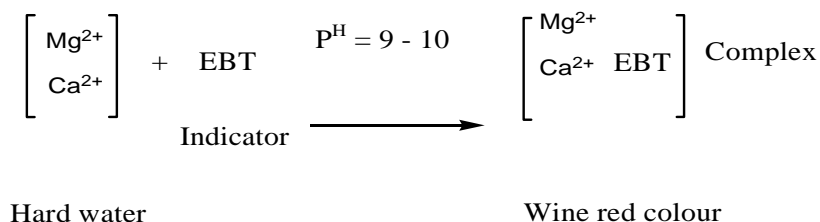
AIM:

To determine the total, temporary and permanent hardness in the given sample of hard water by EDTA method. A standard hard water of N and an EDTA solution are provided.

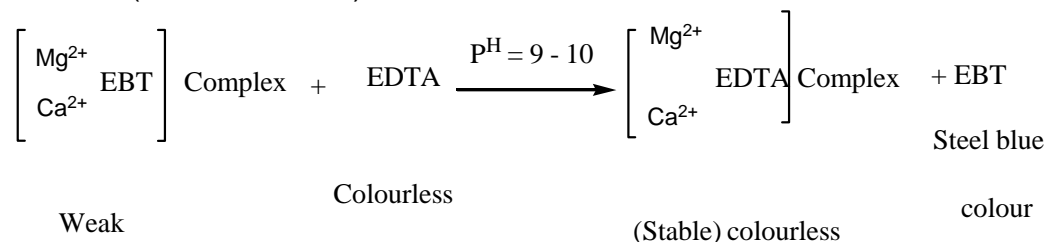
PRINCIPLE:

Disodium salt of Ethylene Tetra Acetic acid (EDTA) is a well known complexing agent.

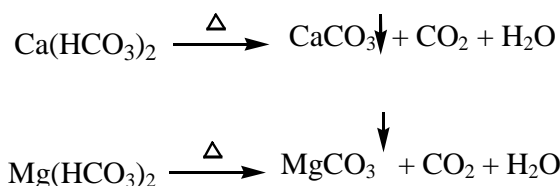
Disodium salt of EDTA is used to estimate the various hardness of the given hard water containing Ca^{2+} and Mg^{2+} ions. When EDTA is added to hard water, it reacts with the calcium and magnesium ions present in the hard water to form stable metal EDTA complex. From the volume of EDTA consumed the hardness can be calculated. Eriochrome Black - T is used as an indicator. The EBT forms weak complex with metal ions present in the hard water and gives wine red colour.



When EDTA is added into the hard water, the metal ions form a stable metal EDTA complex by leaving the indicator. When all the metal ions are taken by EDTA from the EBT metal ions complex, the wine red colour change into steel blue, which denotes the end point. The metal EDTA complex is stable at pH 8-10. This pH range can be maintained by adding ammoniacal buffer ($\text{NH}_4\text{Cl} + \text{NH}_4\text{OH}$).



The filtrate is collected in the conical flask, EBT indicator and buffer solutions are added. It is then titrated against the EDTA.



DETERMINATION OF TOTAL HARDNESS OF HARD WATER SAMPLE

TITRATION – II

Hard water sample Vs Std. EDTA

S.No.	Volume of sample hardwater (mL)	Burette reading (mL)		Concordant volume of EDTA(mL)	Indicator
		Initial	Final		
1	20	0			
2	20	0			
3	20	0			

CALCULATION

Volume of EDTA $V_1 = \dots \dots \dots$ mL

Strength of EDTA $N_1 = \dots \dots \dots$ N

Volume of the hard water sample $V_2 = 20$ mL

Strength of hard water sample $N_2 = \dots \dots \dots$ N

According to the law of volumetric analysis

$$V_1 N_1 = V_2 N_2$$

$$N_2 = V_1 \times N_1 / V_2$$

$$= \dots \dots \dots \times \dots \dots \dots / 20$$

$$= \dots \dots \dots \text{ N}$$

Total hardness of hard water sample $= \dots \dots \dots \text{ N} \times 50 \times 1000$

$$= \dots \dots \dots \text{ ppm}$$

$$\text{Temporary hardness} = \text{Total hardness} - \text{Permanent hardness}$$

MATERIALS REQUIRED

1. EDTA Solution
2. Standard water
3. EBT indicator
4. Buffer solution
5. Burette
6. Pipette
7. Conical flask
8. 250 mL beaker
9. 100 mL standard flask
10. Sample hard water.

PROCEDURE

TITRATION – I

Standardization of EDTA

The burette is washed well with distilled water and rinsed with a little amount of the given EDTA solution and then filled with the same EDTA solution up to the zero level without air bubbles. Initial burette reading is noted. 20 mL of standard hard water sample is pipetted out in a clean conical flask. 5 mL of ammonia buffer solution and then 2 drops of EBT indicator are added. The solution turns wine red colour and it is then titrated against EDTA taken in the burette. The change of wine red to steel blue colour is the end point. The final reading in the burette is noted. The difference in the burette reading gives the volume of EDTA solution. The titration is repeated to get concordant values.

TITRATION - II

Determination of total hardness of hard water sample

20 mL of the given hard water sample is pipetted out in a conical flask then added 5 mL of ammonia buffer solution and 2 drops of EBT indicator are added. The solution turns wine red in colour. This solution is titrated against EDTA solution taken in the burette. The change of wine red into steel blue colour is the end point. The titration is repeated to get concordant values.

TITRATION – III

Determination of permanent hardness

100 mL of the given sample of hard water is taken in a clean 250 mL beaker and boiled for 10 – 15 minutes. It is then cooled and filtered. The filtrate is collected in a 100 mL standard flask and made up to the mark using distilled water. 20 mL of this made up solution is pipetted out into a clean flask. 5 mL of ammonia buffer solution and 2 drops of Eriochrome Black – T indicator are added. The solution turns wine red in colour. This solution is titrated against EDTA taken in the burette.

DETERMINATION OF PERMANENT HARDNESS OF HARD WATER SAMPLE

TITRATION – II

Boiled hard water sample Vs Std. EDTA

S.No	Volume of boiled hard water (mL)	Burette reading (mL)		Concordant volume of EDTA(mL)	Indicator
		Initial	Final		
1	20	0			
2	20	0			
3	20	0			

CALCULATION

Volume of EDTA $V_1 = \dots\dots\dots$ mL

Strength of EDTA $N_1 = \dots\dots\dots$ N

Volume of the hard water sample $V_2 = 20$ mL

Strength of hard water sample $N_2 = \dots\dots\dots$ N

According to the law of volumetric analysis

$$V_1 N_1 = V_2 N_2$$

$$N_2 = V_1 \times N_1 / V_2$$

$$= \dots\dots\dots \times \dots\dots\dots / 20$$

$$= \dots\dots\dots \text{N}$$

Permanent hardness of hard water sample $= \dots\dots\dots \text{N} \times 50 \times 1000$

$$= \dots\dots\dots \text{ppm}$$

Calculation of the temporary hardness of the hard water

Temporary hardness = Total hardness – Permanent hardness

$$= \dots\dots\dots \text{ppm.}$$

The change in colour from wine red into steel blue colour is the end point. The titration is repeated to get concordant values.

Determination of Temporary hardness

Temporary hardness of the water sample is calculated by subtracting permanent hardness from total hardness.

$$\text{Temporary hardness} = \text{total hardness} - \text{Permanent hardness}$$

RESULT

1. Amount of total hardness of the given sample of water =Ppm
2. Amount of permanent hardness of the given sample of water = Ppm
3. Amount of temporary hardness of the given sample of water =Ppm

4. DETERMINATION OF DISSOLVED OXYGEN CONTENT TO WATER SAMPLE BY WINKLER'S METHOD

AIM:

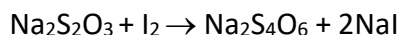
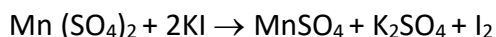
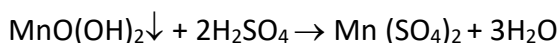
To determine the amount of dissolved oxygen in the given water sample by Winkler's method, a standard solution of $K_2Cr_2O_7$ of strength ___N is given

PRINCIPLE:

Oxygen is dissolved in water to the extent of 7 – 9 mgs/lit at a temperature range of 25° - 35° C. The estimation of dissolved oxygen in water is useful in studying corrosion effects of boiler feed water and in studying water pollution. The amount of dissolved oxygen in water is estimated using Winkler's reagent (Potassium bromide + Potassium bromate). Water sample is collected carefully avoiding aeration/deaeration in ground stoppered flask. Initially manganous sulphate and alkali-iodide reagents are added and the reactions occur as follows



The precipitate dissolves in concentrated sulphuric acid liberating iodine and the liberated iodine is titrated against $Na_2S_2O_3$.



MATERIALS REQUIRED

1. Sodium thiosulphate solution
2. Std. Potassium dichromate solution
3. Dil H_2SO_4 .
4. 5% KI
6. Starch indicator
7. Manganese sulphate
8. Alkali iodide mixture
9. Con. H_2SO_4
10. Burette
11. pipette
12. iodine flask.

STANDARDISATION OF SODIUM THIOSULPHATE

TITRATION – I

Std. Potassium dichromate Vs Sodium thiosulphate

S.No.	Volume of Potassium dichromate (mL)	Burette reading (mL)		Concordant volume of Sodium thiosulphate (mL)	Indicator
		Initial	Final		
1	20	0			Starch
2	20	0			
3	20	0			

CALCULATION

Volume of Potassium dichromate $V_1 = 20\text{mL}$

Strength of Potassium dichromate $N_1 = \dots \dots \dots \text{N}$

Volume of Sodium thiosulphate $V_2 = \dots \dots \dots \text{mL}$

Strength of Sodium thiosulphate $N_2 = \dots \dots \dots ? \dots \text{N}$

According to the law of volumetric analysis

$$V_1 N_1 = V_2 N_2$$

$$N_2 = V_1 \times N_1 / V_2$$

$$= \dots 20 \dots \times \dots \dots \dots / \dots \dots \dots$$

Strength of sodium thiosulphate $N_2 = \dots \dots \dots \text{N}$

PROCEDURE

TITRATION – I

Standardization of Sodium Thiosulphate

The burette is washed and rinsed with sodium thiosulphate solution. Then the burette is filled with the given sodium thiosulphate solution upto zero mark. 20 ml of potassium dichromate solution is pipetted out into a clean conical flask. To this, 5 ml sulphuric acid and 15 ml of 5% potassium iodide solution are added. This is titrated against sodium thiosulphate solution. When the solution becomes straw yellow colour, starch indicator is added and then titration is continued. The end point is disappearance of blue colour and appearance of light green colour. The titration is repeated to get concordant value.

TITRATION – II

Determination of Dissolved Oxygen

100-150 ml of the water sample is taken in the iodine flask, 2ml of manganese sulphate and 2 ml of alkali-iodide mixture are added. The stopper is replaced and the flask is inverted and shaken several times for the rough mixing of reagents. The flask is left aside for some time. When half of the precipitate settles down, the stopper is removed and 2 ml of concentrated sulphuric acid is added.

The stopper is replaced and the flask is inverted several times for complete dissolution of the precipitate to get a clear yellow solution. 20 ml of this solution is pipetted out in a clean conical flask and titrated against standardized sodium thiosulphate solution. When the solution becomes light yellow, starch indicator is added. The titration is continued until the blue colour disappears. From the titre value the strength of dissolved oxygen is calculated and hence the amount of dissolved oxygen in the water sample is calculated.

RESULT

Amount of dissolved oxygen in sample water = _____ mgs/lit.

DETERMINATION OF DISSOLVED OXYGEN

TITRATION – II

Water sample Vs Sodium thiosulphate

S.No.	Volume of water sample(mL)	Burette reading (mL)		Concordant volume of Sodium thiosulphate (mL)	Indicator
		Initial	Final		
1	20	0			Starch
2	20	0			
3	20	0			

CALCULATION

Volume of Sodium thiosulphate $V_1 = \dots\dots\dots$ mL

Strength of Sodium thiosulphate $N_1 = \dots\dots\dots$ N

Volume of water sample $V_2 = 20$

mL Strength of water sample } (or)

Strength of dissolved oxygen in water } $N_2 = \dots\dots\dots? \dots\dots$ N

According to the law of volumetric analysis

$$V_1 N_1 = V_2 N_2$$

$$N_2 = V_1 \times N_1 / V_2$$

$$= \dots\dots\dots \times \dots\dots\dots / 20$$

Strength of sodium thiosulphate $N_2 = \dots\dots\dots$ N

Amount of dissolved oxygen in one litre of tap water = Normality x Eq.weight of oxygen x 1000 mgs

$$= \dots\dots\dots N \times 8 \times 1000$$

Amount of dissolved oxygen in one litre of tap water = $\dots\dots\dots$ mgs/lit.

TITRATION – I

Standard NaCl Vs AgNO₃

S.No.	Volume of NaCl (mL)	Burette reading (mL)		Concordant volume of AgNO ₃ (mL)	Indicator
		Initial	Final		
1	20	0			K ₂ CrO ₄
2	20	0			
3	20	0			

CALCULATION

Volume of Sodium chloride $V_1 = 20\text{mL}$

Strength of Sodium chloride $N_1 = \dots \dots \dots \text{N}$

Volume of Silver Nitrate $V_2 = \dots \dots \dots \text{mL}$

Strength of Silver Nitrate $N_2 = \dots \dots \dots \text{N}$

According to the law of volumetric analysis

$$V_1 N_1 = V_2 N_2$$

$$N_2 = V_1 \times N_1 / V_2$$

$$= 20 \times \dots \dots \dots / \dots \dots \text{ml}$$

$$= \dots \dots \dots \text{N}$$

Strength of Silver Nitrate = $\dots \dots \dots \text{N}$

5. DETERMINATION OF CHLORIDE CONTENT OF WATER SAMPLE BY ARGENTOMETRIC METHOD

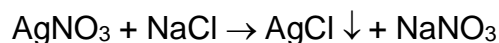
AIM

To determine the amount of chloride present in 100 mL of the given water sample, being supplied with standard solution of sodium chloride of strength _____N and approximately N/20 solution of silver nitrate.

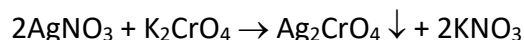
PRINCIPLE

Generally water contains chloride ions in the form of NaCl, KCl, CaCl₂ and MgCl₂. The concentration of chloride ions in water, more than 250 ppm, is not desirable for drinking purpose. The total chloride ions can be determined by argentometric method (Mohr's method).

In this method chloride ion solution is directly titrated against AgNO₃ using potassium chromate as an indicator.



At the end point, when all the ions are removed. The yellow colour of chromate changes into reddish brown due to the following reaction.



MATERIALS REQUIRED

- 1) Std. NaCl solution
2. AgNO₃ solution
3. 2% K₂CrO₄ indicator
4. Burette
5. Pipette
6. Conical flask.

PROCEDURE

TITRATION – I

Standardisation of silver nitrate

The burette is washed with distilled water and rinsed with the small amount of AgNO₃ solution.

It is then filled with the same solution up to the zero mark without air bubbles.

The pipette is washed with water and rinsed with same amount of standard NaCl solution. 20 mL of this solution is pipette out in a clean conical flask. 1 mL of 2% K₂CrO₄ indicator is added and titrated

TITRATION – I

Water sample Vs Std. AgNO_3

S.No.	Volume of Water sample(mL)	Burette reading (mL)		Concordant volume of AgNO_3 (mL)	Indicator
		Initial	Final		
1	20	0			K_2CrO_4
2	20	0			
3	20	0			

CALCULATION

Volume of Water sample $V_1 = 20\text{mL}$

Strength of Water sample $N_1 = \dots \dots \dots \text{N}$

Volume of Silver Nitrate $V_2 = \dots \dots \dots \text{mL}$

Strength of Silver Nitrate $N_2 = \dots \dots \dots \text{N}$

According to the law of volumetric analysis

$$V_1 N_1 = V_2 N_2$$

$$N_1 = V_2 \times N_2 / V_1$$

$$= \dots \dots \dots \times \dots \dots / 20\text{ml}$$

Strength of Water sample (Cl^- ions) = $\dots \dots \dots \text{N}$

CALCULATION OF AMOUNT OF THE CHLORIDE

Amount of the chloride ion present in
ion 1 litre of the water sample $\left\{ \begin{array}{l} \text{Equivalent weight of chloride} \\ \times \text{Strength of the chloride ions} \end{array} \right\}$

$$= 35.46 \times \dots \dots \dots \text{N}$$

$$= \dots \dots \dots \text{mgs}$$

Amount of the chloride ions present in 100 ml of the given water sample = $\dots \dots \dots \text{gms} \times 100/1000$

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

against AgNO_3 solution taken in the burette. The end point is change of colour from yellow to reddishbrown. The titration is repeated for concordant values.

TITRATION - II

Determination of chloride ions

20 mL of the given water sample is pipette out in a clean conical flask and then 1 ml of 2% K_2CrO_4 indicator is added and titrated against AgNO_3 solution taken in the burette. The end point is change of colour from yellow to reddish brown. The titration is repeated for concordant values.

RESULT

(i) Amount of chloride ion present in the whole of the given water sample = _____mgs

(ii) Amount of chloride ion present in the 100 ml of the given water sample= _____mgs

STANDARDISATION OF SODIUM THIOSULPHATE

TITRATION – I

Standard Potassium dichromate Vs Sodium thiosulphate

S.No	Volume of Standard Potassium dichromate (mL)	Burette reading (mL)		Concordant volume of Sodium thiosulphate (mL)	Indicator
		Initial	Final		
1	20	0			Starch
2	20	0			
3	20	0			

CALCULATION

Volume of Potassium dichromate $V_1 = 20\text{mL}$

Strength of Potassium dichromate $N_1 = \dots \dots \dots \text{N}$

Volume of Sodium thiosulphate $V_2 = \dots \dots \dots \text{mL}$

Strength of Sodium thiosulphate $N_2 = \dots \dots \dots ? \dots \text{N}$

According to the law of volumetric analysis

$$V_1 N_1 = V_2 N_2$$

$$N_2 = V_1 \times N_1 / V_2$$

$$= \dots 20 \dots \times \dots \dots \dots / \dots \dots \dots$$

Strength of sodium thiosulphate $N_2 = \dots \dots \dots \text{N}$

6. ESTIMATION OF COPPER CONTENT OF THE GIVEN SOLUTION BY IODOMETRY

AIM

To estimate the amount of copper present in the given solution by iodometric titration. You are provided with standard N $K_2Cr_2O_7$ solution.

PRINCIPLE

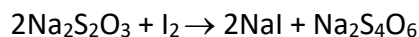
Copper ion occurs naturally in drinking water and is a micronutrient required for the metabolism of living beings. But the presence of copper in water, in quantities more than 1.3 mg/l, will cause stomach ache, intestinal distress and digestive problems. High concentration of copper will also impart a metallic bitter taste to water. Occurrence of copper ions in drinking water may be due to corrosion in plumbing materials and faulty water treatment processes. Copper is also known to cause toxicity to aquatic organisms.

Titrimetric estimation of copper is done through a redox reaction in which stoichiometric quantity of iodine is liberated on reaction with potassium iodide. The liberated iodine can be titrated against standardized sodium thiosulphate solution.

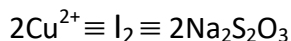
When KI is added to the copper ion solution, copper ions react with KI and liberate I_2 .



The liberated I_2 is titrated against sodium thiosulphate, using starch indicator.



Sodium tetrathionate



ESTIMATION OF COPPER CONTENT OF THE GIVEN SOLUTION

TITRATION – II

Copper ion solution Vs Std.Sodium thiosulphate solution

S.No.	Volume of copper ion solution (mL)	Burette reading (mL)		Concordant volume of Sodium thiosulphate (mL)	Indicator
		Initial	Final		
1	20	0			Starch
2	20	0			
3	20	0			

CALCULATION

Volume of Sodium thiosulphate $V_1 = \dots\dots\dots$ mL

Strength of Sodium thiosulphate $N_1 = \dots\dots\dots$ N

Volume of the given copper ion solution $V_2 = 20$ mL

Strength of the given copper ion solution $N_2 = \dots\dots\dots?$ N

According to the law of volumetric analysis

$$V_1 N_1 = V_2 N_2$$

$$N_2 = V_1 \times N_1 / V_2$$

$$= \dots\dots\dots \times \dots\dots\dots / 20$$

Strength of sodium thiosulphate $N_2 = \dots\dots\dots$ N

CALCULATION OF AMOUNT OF COPPER PRESENT IN THE GIVEN SOLUTION

Amount of copper present in 1000 ml of the solution = Strength x Eq.wt. of Copper

$$= \dots\dots\dots \text{N} \times 63.5$$

$$= \dots\dots\dots \text{gms/lit.}$$

PROCEDURE

TITRATION - I

Standardisation of sodium thiosulphate

20 ml of standard potassium dichromate solution is pipetted out into a clean conical flask. About 10 ml of dilute H_2SO_4 and 10 ml of 10% KI are added to it. The liberated iodine is immediately titrated against sodium thiosulphate solution taken in the burette. When the solution turns pale yellow, about 1 ml freshly prepared starch is added and the titration is continued. The end point is the disappearance of blue colour. Titration is repeated for concordant values.

TITRATION - II

Estimation of copper ion content of the given solution

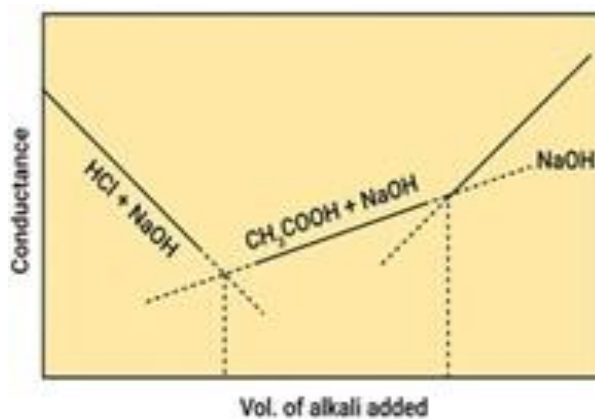
20 ml of the given copper ion solution is pipetted out into a clean conical flask. About 10 ml of dil. H_2SO_4 and 10 ml of 10% KI solutions are added to this solution and the liberated iodine is titrated against standardized sodium thiosulphate taken in the burette. When the solution turns pale yellow, about 1 ml of freshly prepared starch is added and the titration is continued. The disappearance of blue colour is the end point. The titration is repeated for concordant values.

RESULT

1. Strength of the given copper ion solution = N
2. The amount of copper in the given copper ion solution = g/lit

TABLE**Titration of mixture (HCl + CH₃COOH) Vs NaOH**

S.No	Volume of NaOH added (ml)	Conductance (mho)	Remarks
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			
15.			

**Model graph mixture of acid with strong base**

7. DETERMINATION OF STRENGTH OF ACIDS IN A MIXTURE OF ACIDS USING CONDUCTIVITY METER

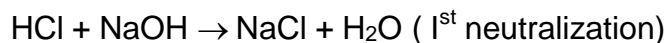
AIM

To determine the strength and amount of a strong acid and a weak acid (HCl and CH₃COOH) present in one litre of the given mixture of acid solution by conductometric titration. You are provided with standard NaOH solution of N.

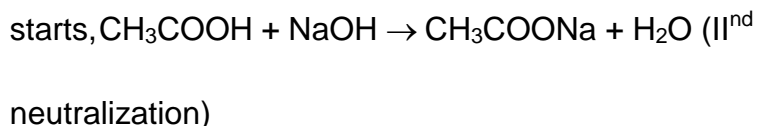
PRINCIPLE

Solution of electrolyte conducts electricity due to the presence of ions. Since specific conductance of a solution is proportional to the concentration of ions in it, conductance of the solution is measured during titration.

When the sodium hydroxide is added slowly from the burette to the solution, HCl (strong acid) gets neutralized first. Since the fast moving H⁺ ions are replaced by slow moving Na⁺ ions, decrease in conductance take place until the end point is reached.



After the complete neutralization of all HCl, the neutralization of CH₃COOH



Since CH₃COONa is stronger electrolyte than CH₃COOH, conductivity slowly increases until all

CH₃COOH is completely neutralized. When the endpoint is reached, addition of NaOH will cause sudden increase in the conductance. This is due to the presence of fast moving OH⁻ ions.

MATERIALS REQUIRED

1. Conductivity bridge 2. Conductivity cell 3. 100 ml beaker 4. Standard NaOH 5. Given HCl & CH₃COOH mixture 6. Burette, pipette, glass rod etc., 7. distilled water.

PROCEDURE

The given mixture of acids (HCl+ CH₃COOH) is transferred into 100 ml standard flask and made up to zero mark using distilled water. 20 ml of the made up solution is pipetted out into a clean 100 ml beaker.

CALCULATION OF STRENGTH OF HCL

Volume of the mixture (HCl), $V_1 = 20$ ml

Strength of the mixture (HCl), $N_1 = ?$

Volume of the NaOH, $V_2 = \dots\dots\dots(A)$ ml **(Ist titre value)**

Strength of the NaOH, $N_2 = \dots\dots\dots N$

According to the law of volumetric analysis,

$$V_1 N_1 = V_2 N_2$$

$$N_1 = V_2 N_2 / V_1$$

Strength of Hydrochloric acid = $\dots\dots\dots N$

CALCULATION OF AMOUNT OF HCL

The amount of HCl present in 1000 ml of the given solution = Strength x Eq.wt. of HCl

$$= \dots\dots\dots N \times 36.45$$

The amount of HCl present in 1000 ml of the given solution = $\dots\dots$ gms

The conductivity cell is placed in it and then diluted to 50 ml by adding conductivity water, so that the electrodes are well immersed in the solution. The two terminals of the cell are connected with a conductivity bridge.

The burette is washed well with water and rinsed with the given standard NaOH solution. It is then filled with NaOH solution upto the zero level.

Now 1 ml of NaOH from the burette is added to the solution, taken in the beaker, stirred for some time and then conductivity is measured. (The conductivity is going on decreasing upto the end point). This process is repeated until atleast five readings are taken after the end point (A) has been reached.

After the end point, again NaOH is gradually added, which causes increase in conductance. This increase in conductance is observed until the end point (B) is reached.

After the second end point, sudden increase in conductance is observed on further addition of NaOH. The reading (conductivity) is continuously measured for each addition of NaOH and are tabulated. Now the graph is plotted between the volume of NaOH Vs conductivity. From the graph the first end point (A) and the second end point (B) are noted. From the end points the strength and amount of HCl and CH₃COOH present in 1 litre of the mixture of solution are calculated.

RESULT

1. Strength of HCl present in 1 litre of the given solution = N
2. The amount of HCl present in 1 litre of the given solution = gms
3. Strength of CH₃COOH present in 1 litre of the given solution =N
4. The amount of CH₃COOH present in 1 litre of the given solution =gms

CALCULATION OF STRENGTH OF CH₃COOH

Volume of the mixture (CH₃COOH), V₁ = 20 ml

Strength of the mixture (CH₃COOH), N₁ = ?

Volume of the NaOH, V₂ =(B-A) ml (IInd titre value)

Strength of the NaOH, N₂ =N

According to the law of volumetric analysis,

$$V_1 N_1 = V_2 N_2 \quad N_1 = V_2 N_2 / V_1$$

Strength of CH₃COOH =N

CALCULATION OF AMOUNT OF CH₃COOH

The amount of CH₃COOH present in 1000 ml of the given solution = Strength x Eq.wt. Of CH₃COOH

$$= \dots\dots\dots N \times 60$$

$$= \dots\dots\dots \text{gms}$$