MAHATMA GANDHI INSTITUTE OF TECHNOLOGY

(Sponsored by Chaitanya Bharathi Educational Society, Estd: 1997)

Accredited Six UG Programs 3 times by NBA and NAAC by 'A' Grade, Affiliated to JNTUH, Hyderabad

DEPARTMENT OF PHYSICS & CHEMISTRY

LABORATORY MANUAL

of

ENGINEERING CHEMISTRY LAB.

for

I B.TECH FOR THE ACADEMIC YEAR: 2018-2019 – I & II SEM

Student Name	
Roll No	
Branch	
Section	

Engineering Chemistry Lab

Course Objectives:

- ❖ The course consists of experiments related to the principles of chemistry required for engineering student. The student will learn:

- ❖ Estimation of hardness and chloride content in water to check its suitability for drinking purpose.

- ❖ To determine the rate constant of reactions from concentrations as a function of time.

- ❖ The measurement of physical properties like adsorption and viscosity.

- ❖ To synthesize the drug molecules and check the purity of organic molecules by thin layer chromatographic (TLC) technique.

Course Outcomes:

- ❖ The experiments will make the student gain skills on:

- ❖ Determination of parameters like hardness and chloride content in water.

- ❖ Estimation of rate constant of a reaction from Concentration – Time relationships.

- ❖ Determination of physical properties like adsorption and viscosity.

- ❖ Calculation of Rf values of some organic molecules by TLC technique.

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GENERAL PRECAUTIONS TO BE TAKEN IN THE LABORATORY

❖ Never work in the laboratory unless a demonstrator or teaching assistant is present.

❖ Do not throw waste such as match stems filter papers etc. into the sink. They must be   thrown into the Dustbins.

❖ Keep the water and gas taps closed except when these utilities are needed.

❖ Never taste any chemical unless instructed to do so and don't allow chemicals to come in   contact with your skin.

❖ While working with gases, conduct the experiment in a fume hood.

❖ Keep all the doors and windows open while working in the laboratory.

❖ Hazards and properties of every chemical used in the experiment should be known. Many chemicals encountered in analysis are poisonous and must be carefully handled.

❖ 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.

❖ Reagent bottles must never be allowed to accumulate on the work bench. They should be placed back in the shelves as and when used.

❖ Containers in which reaction to be performed a little later should be labelled. Working space should be cleaned immediately.

INSTRUCTIONS FOR RECORD WRITING

1. Write on the right hand page the following order:

- ❖ Serial number and date of performance (in the margin)

- ❖ Name and number of the experiment as given in the list.

- ❖ Aim of the experiment.

- ❖ Description of the apparatus.

- ❖ Procedure including sources of error and precautions taken to eliminate or to minimize them.

- ❖ Inference or Result.

- ❖ Explanation, if necessary of any divergence in the expected result.

2. Left hand page should contain the following in their proper places.

- ❖ Neat diagram of the main apparatus.

- ❖ Observation in tabular form.

- ❖ Calculation in tabular form.

- ❖ 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.

KEEP THE RECORD BOOK NEAT IT FETCHES MARKS

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 substance 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 estimation 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 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 equivalence point of the titration.
The strength of concentration of a solution is expressed in the following ways.

NORMALITY:

Number of gram equivalents of the substance dissolved per liter of the solution is called Normality.
It is denoted by N   N= $\frac{wt . of solute}{Eq. wt}$ x $\frac{1000}{vol. of sol. in ml}$

Normality = Wsolute/Esolute × 1/Vsovent (in lit) Where E is Gram equivalent weight

MOLARITY: Number of grams moles of a solute dissolved per liter of solution is called Molarity.

It is denoted by M   M = $\frac{wt . of solute}{Mol. W.t of solute}$ x $\frac{1000}{vol.of sol. in ml}$

Molarity = Wsolute/Msolute × 1/Vsolvent (in lit) Where M is Gram molecular weight

MOLALITY: It is the number of moles of the solute dissolved in 1kg of the solvent it is denoted by (m).

Molality = Wt. of solute/Mol. Wt of solute × 1/Wt. of solvent (in kg)

EXPERIMENT NO: 1

ESTIMATION OF HARDNESS OF WATER BY COMPLEXOMETRIC METHOD USING EDTA
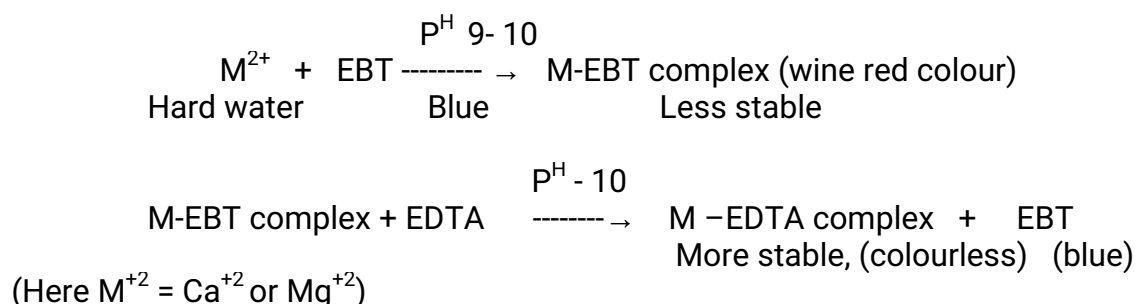
AIM: To determine the total hardness of water by EDTA method.

APPARATUS: Standard Flask, Pippette, Burette, Conical Flask, Beakers, Weighing Bottle, Funnel etc.

CHEMICALS REQUIRED: Epsom Salt ($MgSO_4 . 7H_2O$) , EDTA solution. (Ethylene Diamine Tetra Acetic acid), EBT indicator (Eriochrome black-T), Buffer solution ($NH_4Cl + NH_4OH$) (P^H = 9-10) & Hard water sample.

PRINCIPLE:

The hardness of water is generally due to the presence of calcium and magnesium ions in water. The chlorides, Nitrates and sulphates of calcium & magnesium are responsible for permanent hardness, while bicarbonates of calcium & magnesium are responsible for temporary hardness. EDTA forms a stable complex of Ca-EDTA or Mg-EDTA with hardness causing ions of Ca and Mg present in hard water at P^H 9-10. EBT is used as the indicator to detect the completion of the reaction. When a small amount of EBT is added to hard water sample, it reacts with Mg^{2+} or Ca^{2+} to produce wine red coloured solution due to the formation of a complex. This wine red coloured solution is titrated with EDTA till the colour of the solution changes blue, as EBT is released free in the solution and the stable Mg-EDTA or Ca-EDTA complex is formed. End point is determined by the change in colour from wine red to blue.

$$P^H \ 9\text{-}10$$
$$M^{2+} \ + \ EBT \ \text{---------} \rightarrow \ \text{M-EBT complex (wine red colour)}$$
Hard water   Blue   Less stable

$$P^H \text{-} 10$$
$$\text{M-EBT complex + EDTA} \ \text{--------} \rightarrow \ \text{M }-\text{EDTA complex} \ + \ EBT$$
       More stable, (colourless)   (blue)

(Here M^{+2} = Ca^{+2} or Mg^{+2})

Procedure: It consists of 3 parts-

PART – I: <u>Preparation of standard Epsom salt ($MgSO_4.7H_2O$) Solution:</u>

Weigh out accurately the given Epsom salt ($MgSO_4.7H_2O$) and transfer it into clean 100ml standard flask using a funnel. Dissolve the substance in minimum quantity of distilled water and make it up to the mark with distilled water. Shake the solution thoroughly for uniform concentration.

PART – II: Standardization of EDTA solution using standard $MgSO_4.7H_2O$ solution:

Pipette out 20ml of $MgSO_4.7H_2O$ solution. into a clean conical flask and add 5ml of buffer solution of (mixture of NH_4OH + NH_4Cl) followed by 4 to 5 drops of EBT-indicator. Titrate this solution against the EDTA solution taken in the burette, until the wine red colour of the solution turns blue (end point). Repeat the titration for at least two concurrent titre values.

PART – III: Estimation of Total hardness of water

Pipette out 20 ml of the given hard water sample into a clean conical flask and add 5ml of the buffer solution (NH_4OH + NH_4Cl) followed by 4 to 5 drops of EBT indicator. Titrate this solution against the standardized EDTA solution, until the wine red colour changes blue (end point). Repeat the titration for atleast two concurrent titre values.

CALCULATIONS:

PART – I: Preparation of standard Magnesium Sulphate Solution

Weight of the weighing bottle + Epsom salt (W_1)   = ------ gms

Weight of the empty weighing bottle   (W_2)   = ------ gms

Weight of the Epsom sal   ($W_1 - W_2$)   = ------ gms

Volume of Solution   =100 ml

Gram equivalent Weight of Epsom salt($MgSO_4.7H_2O$) = 123.25 gm)

Normality of Epsom salt solution. (N_1) = $\dfrac{(W_1 - W_2) \times 1000}{\text{Gr. eq. wt} \times V \text{ (in ml)}}$

PART – II: Standardization of EDTA solution using standard Epsom Salt Solution.

S.No	Vol. of Epsom salt Pipette out(ml)	Burette readings		Vol. of EDTA required (ml)
		Initial	Final	
1				
2				
3				

Epsom salt solution   **EDTA solution**

EDTA solution

Normality of Epsom salt sol.(N_1) =------ N   Normality of EDTA sol.(N_2) = ----- N

Volume of Epsom salt sol. (V_1)= ------ml   Volume of EDTA sol. (V_2) = ------ml

$$\text{Normality of EDTA solution } (N_2) = \frac{N_1V_1}{V_2} = \text{------------- N}$$

PART – III: Estimation of Total Hardness of water

S.No	Volume of Hard Water Pipette out(ml)	Burette readings		Volume of EDTA required (ml)
		Initial	Final	
1				
2				
3				

     EDTA Solution   **Hard water**

Normality of EDTA solution (N_2) =------N;   Normality of hard water (N_3)   =------- N

Volume of EDTA solution (V_2) =------ml;   Volume of hard water (V_3)   =------- ml

$$\text{Normality of hard water } (N_3) = \frac{N_2V_2}{V_3} = \text{----------- N}$$

Total hardness of water in terms of $CaCO_3$ equivalents   = $N_3 \times 50 \times 1000$ mg/lit

               =--------------- mg/lit or ppm

RESULT:

1) Normality of EDTA solution (N_2) =-------------N

2) Normality of Hard Water in terms of CaCO3 equivalents (N_3)   = ------------ N

3) Total hardness of water in terms of $CaCO_3$ equivalents =--------------- ppm

Viva – Questions

1. Mention the salts causing temporary and permanent hardness.
2. How is hardness of water tested?
3. How hardness is expressed? What are its units?
4. What is Normality? How can we calculate the weight of solute using normality of the solution?
5. What is the indicator used in this experiment?
6. What are the equivalent weights of Epsom salt ($MgSO_4.7H_2O$) and Calcium Carbonate?
7. What is a standard solution?
8. Calculate the weight of $MgSO_4.7H_2O$ required to prepare 100ml of 0.01N solution?
9. What is a buffer solution?. And which buffer is used in this titration?
10. What is the colour of the solution at the end point in this titration?
11. What is the colour of metal indicator complex & M-EDTA complex?
12. Draw the structures of EDTA.

## EXPERIMENT NO: 2

DETERMINATION OF CHLORIDE IN WATER SAMPLE

AIM: To determine the chloride content in the given water sample by argentometric method (Mohr's method).

APPARATUS: Burette, pipette, conical flask, beaker, burette stand.

CHEMICALS: Silver nitrate, sodium chloride, potassium chromate, water sample.

PRINCIPLE: Chlorides are widely distributed in nature as salts of sodium (NaCl), potassium (KCl), and calcium ($CaCl_2$). These salts of Chlorides are widely used in the production of different industrial chemicals such as Sodium Chloride is used for the production of caustic soda, chlorine, sodium chlorite, and sodium hypochlorite. Potassium chloride is used in the production of fertilizers.

Chloride ion in a water sample can be determined by Argentometric method. This method determines the chloride ion concentration of a solution by titration with silver nitrate. As the silver nitrate solution is slowly added, a precipitate of silver chloride forms.

$$Ag^+(aq) + Cl^-(aq) \rightarrow AgCl(s)$$
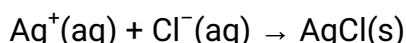
The end point of the titration occurs when all the chloride ions are precipitated. Then additional silver ions react with the chromate ions of the indicator, potassium

chromate, to form a red-brown precipitate of silver chromate.

$$2\,Ag^+(aq) + CrO_4^{2-}\,(aq) \rightarrow Ag_2CrO_4(s)\,(red\,precipitate)$$

PROCEDURE: It consists of 3 steps.

Step – I: Preparation of sodium chloride solution: 0.585gm of sodium chloride is weighed, transferred to a 100ml standard flask and dissolved in a little amount of distilled water. Then the solution is made upto 100ml with distilled water.

Step – II: Standardisation of silver nitrate solution: 20ml of sodium chloride solution is taken into a 250ml conical flask using pipette and 2 drops of potassium chromate indicator is added and titrated with silver nitrate solution taken in a burette till the colour changes from yellow to red. The experiment is repeated till concurrent readings are obtained and the normality of silver nitrate is calculated.

S.No	Vol of NaCl solution	Burette Readings		Vol of AgNO3 solution run down
		Initial	Final	
1	20			
2	20			
3	20			

Concentration of $AgNO_3$ solution is calculated using the formula,

$$N_1V_1 = N_2V_2$$

N_1 is normality of NaCl = 0.1N

V_1 is volume of NaCl solution = 20ml

N_2 is normality of $AgNO_3$ =

V_2 is volume of $AgNO_3$ solution rundown =

$$N_2 = N_1V_1/V_2$$

Step – III: Determination of chloride in water sample:

Titration with distilled water(blank solution): Pipette out 20 ml of the distilled water in a conical flask and add 3-4 drops of potassium chromate solution. Slowly add standard silver nitrate solution from the burette and shake the solution well till light yellow colour changes to red colour. The titration is repeated until a concurrent volume V1 is obtained.

Titration with the Sample Water: Pipette out 20 ml of the given water sample in a

conical flask and add 3-4 drops of potassium chromate solution. Slowly add standard silver nitrate solution from the burette and shake the solution well till light yellow colour changes to red colour. The titration is repeated until a concurrent volume V2 is obtained.

Observations and Calculations:

Titration with the Blank solution

S.No	Vol of distilled water	Burette Readings		Vol of AgNO3 solution run down(V1)
		Initial	Final	
1	20			
2	20			
3	20			

Titration with the Sample Water

S.No	Vol of sample water	Burette Readings		Vol of AgNO3 solution run down(V2)
		Initial	Final	
1	20			
2	20			
3	20			

Normality of water sample is calculated using the formula,

$$N_2 V_2 = N_3 V_3$$

N_2 is normality of silver nitrate solution =

V_2 is Volume of $AgNO_3$ solution consumed = $V_2 - V_1$

V_3 is Volume of water sample = 20ml

N_3 is normality of water sample =

$$N_2 V_2 / V_3$$

Amount of chloride ion = N_3 **x** 35.5 x 1000 **mg/lit**

Result: The amount of chloride content in the given water sample = _____mg/lit.

EXPERIMENT NO: 3

ESTIMATION OF HCl BY CONDUCTOMETRY
(STRONG ACID VS STRONG BASE)

AIM: To estimate the amount of HCl present in the given solution using NaOH solution by conductometric titration.

APPARATUS: Conductometer, conductivity cell, beakers, glass rod, conical flask, burette, standard flask, weighing bottle, funnel, pipette, Wash bottle etc...

CHEMICALS: Oxalic acid ($H_2C_2O_4.2H_2O$), NaOH solution, HCl solution, phenolphthalein indicator, and distilled water.

THEORY: The electrical conductance of any electrolytic solution depends on the number of ions and their mobilities. In the titration of HCl vs NaOH, before the addition of NaOH, HCl shows high conductance, due to the presence of highly mobile H^+ ions. As NaOH solution is added, the H^+ ions combine with OH^- and form feebly ionisable H_2O molecules.

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

Hence the conductance of the solution decreases gradually by the addition of NaOH solution. When all the H^+ ions have been neutralized, the conductance starts increasing with the further addition of NaOH, due to the increase in no. of OH^- ions. When a graph is drawn between the volume of NaOH added and conductance, the point of intersection gives the end point.

PROCEDURE: It consists of 3 parts

PART –I: <u>Preparation of standard solution of oxalic acid</u>

Weigh accurately a sample of oxalic acid and transfer it into a clean 100ml standard (volumetric) flask using a funnel. Dissolve the substance in minimum quantity of distilled water. Make the solution up to the mark with distilled water and shake well for uniform concentration.

PART- II: <u>Standardization of given NaOH solution</u>:

Pipette out 20ml of standard oxalic acid solution into a clean conical flask and add 2 - 3drops of phenolphthalein indicator. Titrate the contents of the conical flask against the NaOH solution taken in the burette until a pale pink colour (end point) is obtained. Repeat the titration for at least two concurrent titre values.

PART-III: <u>Estimation of the amount of HCl using conductometer.</u>

Pipette out 10 ml of HCl solution in a clean 100ml standard flask and add distilled water tom it upto the mark. Pipette out 40ml of the above HCl solution into a well cleaned   100 ml beaker, dip the conductivity cell in it. Then press the push button and stir the solution with glass rod, measure the initial conductance. Add 1ml of NaOH solution from the burette and note down the corresponding conductances after stirring the solution. Take a minimum of 20 readings.

CALCULATIONS:

PART-I: <u>Preparation of standard solution of oxalic acid</u>

Weight of weighing bottle + oxalic acid (W1)   =--------   gms

Weight of empty weighing bottle   (W2)   =----------   gms

Weight of oxalic acid taken   (W1- W2)   = ---------   gms

Equivalent weight of Oxalic Acid   = Mol. wt. / Basicity = 63

Volume of Solutions   = 100ml

Normality of oxalic acid (N_1)   = $\dfrac{(W1-W2)}{63} \times \dfrac{1000}{100}$ =   ---------N

PART –II: <u>Standardization of NaOH solution</u>

S.No	Vol. of Oxalic acid Pipette out(ml)	Burette readings		Vol. of NaOH Required (Initial-Final) (ml)
		Initial	Final	
1				
2				
3				

Oxalic acid Solution   **NaOH Solution**

N_1 = -------- N N_2 = -------- N

V_1 = 20 ml V_2 = ------- ml

Normality of NaOH solution (N_2)= $\dfrac{N_1 V_1}{V_2}$ = --------- N

PART-III: Estimation of amount of HCl

S.No.	Volume of NaOH (ml)	Conductance x range(ms)

1	0	
2	1	
3	2	
4	3	
5	4	
6	5	
7	6	
8	7	
9	8	
10	9	
11	10	
12	11	
13	12	
14	13	
15	14	
16	15	
17	16	
18	17	
19	18	
20	19	
21	20	

GRAPH: Plot a graph taking volume of NaOH added, on X-axis and corresponding conductance, on Y-axis. It gives two straight lines. Intersection of the two straight lines indicates the neutralization point.
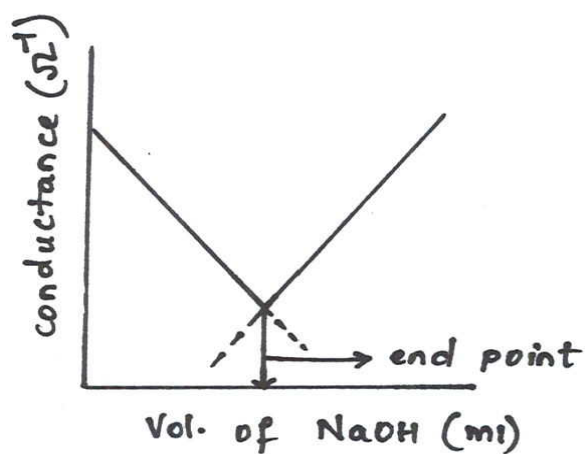
PART-III: Determination of amount of HCl

NaOH Solution	HCl Solution

N_2 = --------- N N_3 =-------- N

V_2 = ---------ml(from graph) V_3 = 40ml

 Normality of HCl solution (N_3)= $\underline{N_2V_2}$ =   -------------N

                                                          V_3

Amount of HCl in the given solution = N_3 X 36.5/10

                                                    = ----------gm/100ml

Result:

1. Normality of NaOH solution (N_2) =-------N

2. End point of the HCL (from graph) =-------- ml

3. Normality of HCl solution (N_3)=----------N

4. The amount of HCl present in the given solution = ---------   gms/100ml

Viva – Questions

1. What is Conductometric Titration?

2. What is conductance? What are its units?

3. Define specific conductance and equivalent conductance? What are its units?

4. How does specific and equivalent conductances vary with dilution?

5. What is cell constant and what are its units?

6. Define Kholraush Law?

7. Is Conductivity cell an electrolytic or an electrochemical cell?

8. How does the conductance vary in this titration ?

9. How do you detect the neutralization point in conductometric titration?

10. What are the advantages of conductometric titration over volumetric titrations?

11. Draw a rough graph for this experiment?

12. What is the effect of temperature on the conductance of the electrolyte ?

EXPERIMENT NO: 4
ESTIMATION OF ACETIC ACID BY CONDUCTOMETRY

AIM: To estimate the amount of CH_3COOH present in the given solution, using NaOH solution by conductometric titration.

APPARATUS: Conductometer, conductivity cell, beaker, glass rod, conical flask, burette, standard flask, funnel, pipette, and wash bottle. etc...

CHEMICALS: Oxalic acid ($H_2C_2O_4.2H_2O$), NaOH solution, Acetic acid solution, phenolphthalein indicator and distilled water.

PRINCIPLE: When a weak acid is titrated with a strong base.

$$CH_3COOH + NaOH \longrightarrow CH_3COONa + H_2O$$
$$\text{(Salt, Strong electrolyte)}$$
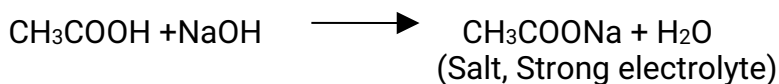
The conductance of Acetic acid(Weak acid) is low it raises on the addition of NaOH due to the formation of a salt (CH_3COONa), which is a strong electrolyte. Finally the conductivity rises rapidly due to increase in OH^- ions. When a graph is drawn between the volume of NaOH (X-axis) added and conductance (Y-axis), the point of intersection gives the end point.

PROCEDURE: It consists of 3 parts

PART –I: Preparation of standard solution of oxalic acid:

Weigh accurately a sample of oxalic acid and transfer it into a clean 100ml standard (volumetric) flask using a funnel. Dissolve the substance in minimum quantity of distilled water. Make the solution up to the mark with distilled water and shake well for uniform concentration.

PART- II: Standardization of given NaOH solution:

Pipette out 20ml of the standard oxalic acid solution into a clean conical flask and add 1-2 drops of phenolphthalein indicator. Titrate the contents of the conical flask against the NaOH solution taken in the burette until a pale pink colour (end point) is obtained. Repeat the titration for least two concurrent titre values.

PART-III: Estimation of the amount of CH_3COOH using Conductometer.

Pippete out 10 ml of given CH_3COOH solution in a 100ml standard flask and add distilled water to it upto the mark. And pipette out 40ml of the CH_3COOH solution into a well cleaned 100 ml beaker, dip the conductivity cell in it, stir the solution and measure the initial conductance. Add 1ml of NaOH solution from the burette and note down the corresponding conductance values after stirring the solution. Take minimum of 20 readings.

CALCULATIONS:

Part-I: Preparation of standard solution of oxalic acid

Weight of weighing bottle + oxalic acid (w_1)   =   -------   gms

Weight of empty weighing bottle   (w_2)   =   --------   gms

Weight of oxalic acid taken   (w_1- w_2)   =   --------   gms

Volume of Solutions   = 100ml

Normality of oxalic acid (N_1)   = $\dfrac{(w_1-w_2)}{63} \times \dfrac{1000}{100}$ = ----------N

PART –II: Standardization of NaOH solution

S.No	Vol. of Oxalic acid (ml)	Burette readings		Vol. of NaOH required (FBR – IBR) (ml)
		IBR	FBR	
1				
2				
3				

Oxalic acid Solution   **NaOH Solution**

N_1 =--------- N N_2 =-------- N

V_1 = 20 ml V_2 =--------ml

Normality of NaOH solution (N_2) = $\dfrac{N_1V_1}{V_2}$ =- ---------N

PART-III: Estimation of amount of CH_3COOH

S.No.	Volume of NaOH (ml)	Conductance x range(ms)
1.	0	
2.	1	
3.	2	

4.	3	
5.	4	
6.	5	
7.	6	
8.	7	
9.	8	
10.	9	
11.	10	
12.	11	
13.	12	
14.	13	
15.	14	
16.	15	
17.	16	
18.	17	
19.	18	
20.	19	
21.	20	

Graph: Plot a graph taking volume of NaOH added on X-axis and corresponding conductance on Y-axis. Intersection of two straight lines drawn along the points indicates neutralization point.

(b) weak acid vs strong base
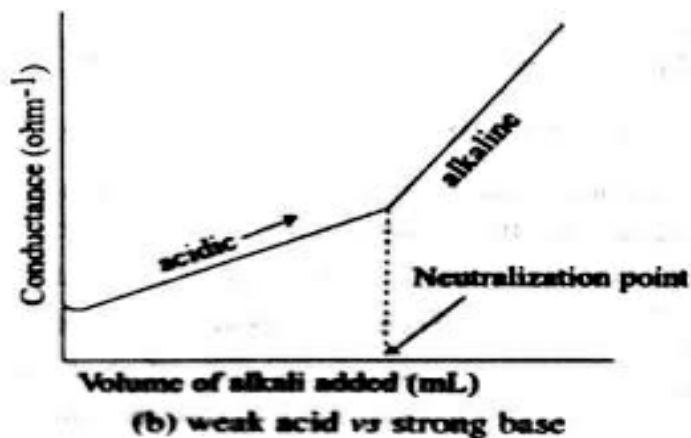
PART-III: Determination of amount of CH₃COOH

NaOH Solution	CH₃COOH Solution
N_2 =--------- N	N_3 =---------- N
V_2 = x ml (from graph)	V_3 = 40ml

Normality of CH₃COOH solution (N_3)= $\dfrac{N_2 V_2}{V_3}$ =--------N

Amount of CH₃COOH in given solution = N_3 X 60/10 =------gm/100ml

Result:

      1. Normality of NaOH solution (N_2) =-------N

      2. End point of Acetic Acid(from graph)= ---------- (x)ml

      3. Normality of CH₃COOH solution (N_3) = -------N

      4. The amount of CH₃COOH present in the given solution = ——— gm/100ml

VIVA QUESTIONS

1. Define an electrolyte.

2. Define strong and weak electrolytes.

3. How does conductance vary in this experiment?

4. What are strong and weak electrolytes in this experiment.

5. Write the reaction between Oxalic acid & NaOH solution.

6. Write the chemical reaction involved in the experiment.

7. What is P^H? Give the P^H range of phenolphthalein indicator.

8. Define basicity. What is the basicity of CH_3COOH?

9. Draw the rough graph for this experiment.

10. Define Conductometric titration.

EXPERIMENT NO: 5
ESTIMATION OF HCl BY POTENTIOMETRY

AIM: To estimate the amount of HCl present in the given solution using NaOH by potentiometric titration.

APPARATUS: Potentiometer, saturated calomel electrode, platinum electrode, salt bridge, beakers, glass rod, conical flask, burette, standard flask, weighing bottle, funnel, pipette.etc...

CHEMICALS: Oxalic acid ($H_2C_2O_4.2H_2O$), NaOH solution, HCl solution, phenolphthalein indicator, saturated KCl solution, quinhydrone powder, and distilled water.

THEORY:

It involves potentiometric measurement of EMF of a cell constructed using the test solution-HCl which is used for determining the end point in acid-base titration. The cell consists of a saturated calomel electrode (SCE) as reference electrode and quinhydrone electrode (Q_1+ QH_2) as the indicator electrode, the EMF of which depends on the H^+ ion concentration of test solution. The quinhydrone is a powdered organic substance having equimolar mixture of quinone (Q) and hydroquinone (QH_2). When a pinch of quinhydrone is added to the acid solution in contact with platinum wire/electrode, quinone, hydroquinone and H^+ ions form a reversible redox system.

$$Q + 2H^+ + 2\,e^- \leftrightarrow QH_2$$
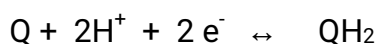
Initially, before titration, the EMF measured will be high due to high H^+ concentration (low $P^{H)}$ and on addition of NaOH solution, it decreases due to decrease in concentration of H^+ as a consequence of neutralization to form H_2O.

$$HCl + NaOH \rightarrow NaCl + H_2O$$
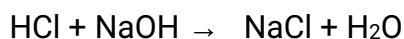
After neutralization, it decreases even below zero as indicated by −ve value. The cell used can be represented as **Pt, Hg(l) / Hg$_2$Cl$_2$(S), KCl(sat.soln) // H$^+$(C=?),Q / QH$_2$, Pt.** Oxidation takes place on the calomel electrode and reduction takes place at quinhydrone electrode. Thus EMF of the cell is calculated as

E_{cell} = E_{right} - E_{left} = E_Q - $E_{calomel}$

= $[0.6994-0.0591P^H]$ − 0.2422

E_{cell} = $0.4572 − 0.0591P^H$

PROCEDURE: It consists of 3 parts

PART −I: <u>Preparation of standard solution of oxalic acid</u>

Weigh accurately a sample of oxalic acid and transfer it into a clean 100ml standard (volumetric) flask using funnel. Dissolve the substance in minimum quantity

of distilled water. Make the solution up to the mark with distilled water and shake well for uniform concentration.

PART- II: Standardization of the given NaOH solution

Pipette out 20ml of standard oxalic acid solution into a clean conical flask and add 1- 2 drops of phenolphthalein indicator. Titrate the contents of the conical flask against the NaOH solution taken in the burette until a permanent pale pink colour (end point) is obtained. Repeat the titration to get at least two concurrent titre values.

PART-III: Estimation of the amount of HCl in the given solution using potentiometer

Pippete out 10 ml of given HCl solution into a clean 100ml standard flask and add distilled water to it upto the mark. Pipette out 20 ml of above HCl solution into a clean 100ml beaker and add a pinch of quinhydrone powder. Dip the platinum electrode into it and connect it to the potentiometer. Dip the calomel electrode into saturated KCl solution provided in another beaker. Connect these two solutions with salt bridge and measure the initial EMF. Now add 1ml of NaOH solution from burette and stir it with glass rod. Note down the corresponding EMF. Take about 20 readings. Note the volume of NaOH where there is a steep change in EMF (+ve EMF to −ve EMF), this indicates the neutralization point.

CALCULATIONS:

PART-I: Preparation of standard solution of oxalic acid

Weight of weighing bottle + oxalic acid (w_1)   = -------- gms

Weight of empty weighing bottle (w_2)   = ------- gms

Weight of oxalic acid taken   ($w_1 - w_2$)   = -------- gms

Volume of Solutions   = 100ml

Normality of oxalic acid (N_1) $= \dfrac{(w_1 - w_2)}{63} \times \dfrac{1000}{100} =$ --------N

PART −II: Standardization of NaOH solution

S.No	Vol. of Oxalic acid (ml)	Burette readings		Vol. of NaOH required (FBR - IBR) (ml)
		IBR	FBR	
1				
2				
3				

Oxalic acid Solution   NaOH Solution

$N_1 =$-------- N $N_2 =$--------N

$V_1 = 20$ ml $V_2 =$ -------- ml

Normality of NaOH solution (N_2) = $\underline{N_1 V_1}$ =   ----------N
                                                                V_2

PART −III: Determination of amount of HCl

S.No.	Volume of NaOH (ml)	EMF (mv)	ΔE=E₁-E₂	ΔV=V₂-V₁	ΔE/ΔV
1	0				
2	1				
3	2				
4	3				
5	4				
6	5				
7	6				
8	7				
9	8				
10	9				
11	10				
12	11				
13	12				
14	13				
15	14				
16	15				
17	16				
18	17				
19	18				
20	19				
21	20				

GRAPHS: Plot a graph between volume of NaOH on X-axis and corresponding EMF on Y-axis the volume of NaOH at which (+ve) EMF changes to (−ve) EMF gives the end point (approximate) which has been consumed to neutralize 20ml of HCl taken in the beaker.

Plot another graph taking volume of NaOH on X-axis and ΔE/ΔV on Y-axis, the highest inflection point gives the exact end point of HCl solution.
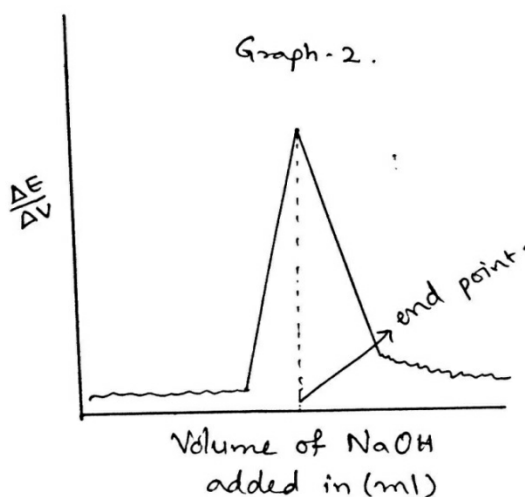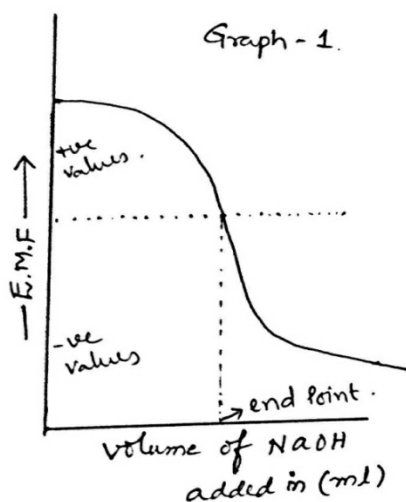
Graph - 1.

Graph - 2.

HCl Solution

$N_2 = $ -------- N

$V_2 = $ --------ml (from graph -2)

Normality of HCl solution (N_3) = $\dfrac{N_2V_2}{V_3}$ = --------- N

NaOH Solution

$N_3 = $ --------- N

$V_3 = 20$ ml

Amount of HCl in given solution= $N_3 \times 36.5/10$ gms/100ml = -----------gm/100ml

Results:

1. Normality of NaOH solution (N_2) =---------N
2. End point of HCL (from graph -2) = ---------ml
3. Normality of HCl solution (N_3) = ---------N
4. The amount of HCl present in the given solution = --------- gm/100ml

VIVA questions

1. Define Potentiometri titration.

2. Define an electrochemical cell?

3. What is SCE? Give its EMF values.

4. What is the role of quinhydrone electrode?

5. What is Cell notation ? Write the cell notation of the cell used in the experiment.

6. Write Nernst's equation.

7. How do you get EMF of a cell consisting of two electrodes?

8. Give the relation between P^H and EMF of quinhydrone electrode?

9. What are the advantages of potentiometric titrations over conductometric titrations?

10. Define single electrode potential?

11. What is the role of salt bridge?

EXPERIMENT NO: 6
ESTIMATION OF FERROUS IRON (Fe^{+2}) POTENTIOMETRY

AIM : To estimate the amount of Fe^{2+} present in given solution Potentiometrically using KMnO$_4$ solution.

APPARATUS: Potentiometer, saturated calomel electrode, platinum electrode, salt bridge, beakers, glass rod, conical flask, burette, standard flask, weighing bottle, funnel, pipette.etc...

CHEMICALS: Mohr's salt (Ferrous ammonium sulphate), Conc. H$_2$SO$_4$, 6N H$_2$SO$_4$, KMnO$_4$, saturated KCl solution and distilled water.

THEORY: Potentiometric measurement of EMF is used for locating the end point in the redox titrations. The cell consists of a saturated calomel electrode (SCE) as reference electrode and the platinum electrode. The EMF of the electrode dipped in Fe^{2+} (Test) solution as an indicator electrode depends upon the ratio,(Fe^{+2} / Fe^{+3})

The cell can be represented as: **(-) Pt, Hg/Hg$_2$Cl$_2$(s)/ KCl (sat) // Fe^{3+}/ Fe^{2+}, Pt (+).**

The EMF of the cell is given by E$_{cell}$ =

$$E^0_{(Fe3+, Fe2+)} + \underline{2.303R}\,T\,\log\left[\frac{Fe^{3+}}{Fe^{2+}}\right] - E_{(SCE)}$$

Initially , the conc. of Fe^{3+} in the solution is very low and the EMF measured will be low and on addition of KMnO$_4$ solution to Fe^{2+} solution, the concentration of Fe^{3+} increases (due to oxidation of Fe^{2+}) and the EMF increases. Finally, at the end point, the EMF increases sharply and an inflection in the titration curve can be observed due to sharp decrease in Fe^{2+} concentration.

$$5Fe^{2+} + MnO^-_4 + 8H^+ \rightarrow 5Fe^{3+} + Mn^{2+} + 4H_2O$$

PROCEDURE: It consists of 3 parts

PART −I: Preparation of standard solution of Mohr's salt(FeSO$_4$(NH$_4$)$_2$SO$_4$.24H$_2$O):

Weigh accurately a sample of Mohr's salt and transfer it into a clean 100ml standard (volumetric) flask using a funnel. Add about 50ml of distilled water followed by 8 drops of conc.H$_2$SO$_4$ and shake the flask to dissolve the substance.. Make the solution up to the mark with distilled water and shake well for uniform concentration.

PART- II: Standardization of KMnO$_4$ solution:

Pipette out 20ml of standard Mohr's salt solution into a clean conical flask and add about 20ml of 6N H$_2$SO$_4$ solution. Titrate the contents of the conical flask against the KMnO$_4$ solution taken in the burette until a permanent pale pink colour (end point) is obtained. Repeat the titration for at least two concurrent titre values.

PART-III: <u>Estimate the amount of Fe^{2+} in the given solution:</u>

Take 10 ml of 0.5N Fe^{2+} solution into clean 100ml standard flask, make the solution up to the mark with distilled water and shake the solution thoroughly. Pipette out 20ml of Fe^{2+} solution into a clean beaker and add a 20ml 6N H_2SO_4 to it. Dip the platinum electrode into the same solution and connect it to the potentiometer. Dip the calomel electrode into saturated KCl solution provided in another beaker. Connect these two electrodes with a salt bridge and measure the initial EMF. Now titrate the solution by adding 1ml of $KMnO_4$ each time and note down the corresponding EMF. Take about 21 readings in the following tabular column. (Note the volume of $KMnO_4$ where there is a steep change in EMF. This volume is the approximate volume of $KMnO_4$ required for oxidation of Fe^{2+}.

CALCULATIONS:

PART-I: <u>Preparation of standard solution of Mohr's salt</u>

Weight of weighing bottle + **Mohr's salt** (W_1) =--------- gm

Weight of empty weighing bottle (W_2)   = -------- gm

Weight of **Mohr's salt** taken (W1-W2)   = --------- gm

Volume of Solution   =100 ml

Normality of Mohr's salt (N_1) = $\dfrac{(W1-W2) \times 1000}{GEW \times V}$ **= ———N**

Where , GEW = 392,

PART −II: <u>Standardization of $KMnO_4$ solution</u>

S.No	Vol. of Mohr's salt (ml)	Burette readings		Vol. of $KMnO_4$ (ml)
		Initial	Final	
1				
2				
3				

<u>Mohr's salt Sol.</u>   **<u>$KMnO_4$ sol.</u>**

N_1 = N_2 =--------N

V_1 = 20 ml V_2 = titre value-- ml

Normality of $KMnO_4$ solution = N_2 = $\dfrac{N_1V_1}{V_2}$ = ----------N

PART −III: <u>Estimation of Fe^{2+} (iron) by EMF measurement</u>

TABLE:

S.No.	Volume of KMnO$_4$ in (ml)	EMF (mv)	ΔE=E$_2$-E$_1$	ΔV=V$_2$-V$_1$	ΔE/ΔV
1	0				
2	1				
3	2				
4	3				
5	4				
6	5				
7	6				
8	7				
9	8				
10	9				
11	10				
12	11				
13	12				
14	13				
15	14				
16	15				
17	16				
18	17				
19	18				
20	19				
21	20				

GRAPHS: Plot a graph between volume of KMnO$_4$ on X-axis and corresponding EMF on Y-axis. Plot another graph (differential graph) taking the volume of KMnO$_4$ on X- axis and ΔE/ΔV on Y-axis. From graph − 1, the volume of KMnO$_4$ at which steep change in EMF is noted. This gives the approximate volume of KMnO$_4$ needed for oxidation of given ferrous solution. From graph − II (inflexion point), the exact titre value can be known.

EMF — Volume of KMnO₄ (ml)

$\dfrac{\Delta E}{\Delta V}$ — Volume of KMnO₄ (ml
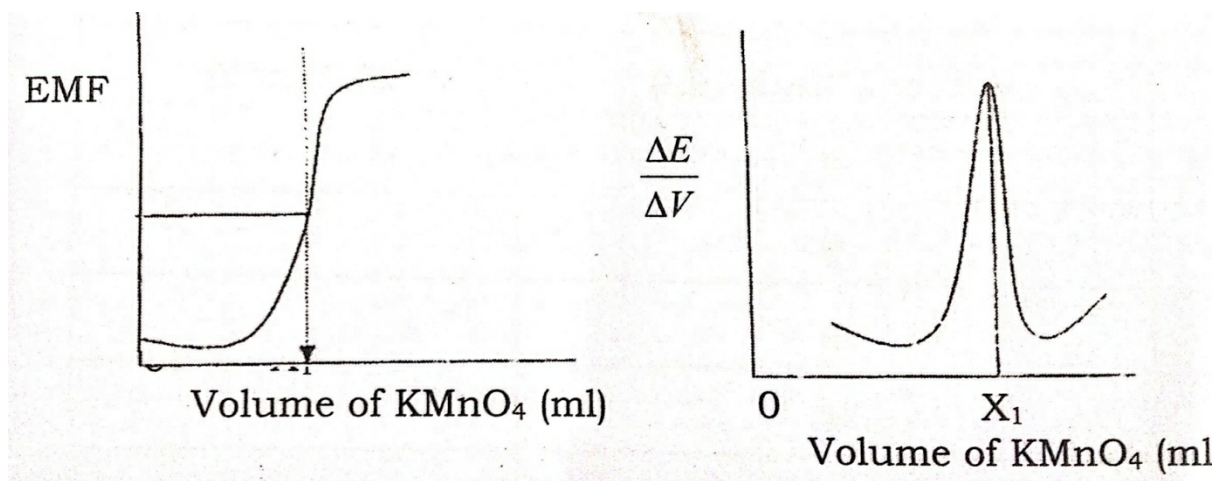
KMnO₄ Solution

N_2 = From step 2

V_2 = ------- ml(from graph – 2)

Fe²⁺ Solution

N_3 = ---------N

V_3 = 20 ml

Normality of KMnO₄ solution = $N_3 = \dfrac{N_2 V_2}{V_3}$ = -----------N

Amount of Ferrous ion in given 100 ml solution = $\dfrac{N_3 \times 56}{10}$ = -----------gms.

RESULT:

1) Normality of KMnO₄ solution(N_2) = ------- N

2) End point of Fe²⁺ solution (from graph -2) = -------- ml

3) Normality Fe²⁺ solution(N_3) = --------- N

4) Amount of Ferrous iron in given solution = --------- g/100ml

Viva questions

1. What is the indicator used in this experiment?

2. What type of titration is involved in this experiment?

3. What are the molecular and equivalent weights of KMnO₄?

4. Write the chemical reactions involved in this experiment?

5. What is the equivalent weight of iron in this experiment?

6. What is the Molar ratio of KMnO₄ and FeSO₄?

7. What is Oxidation & Reduction?

8. How do you detect the end point?

9. Define an oxidising and a reducing agent.

10. What are oxidising & reducing agents in this experiment?

EXPERIMENT NO: 7
HYDROLYSIS OF METHYL ACETATE

Aim: To prove that the hydrolysis of Methyl Acetate follows first order chemical kinetics.

Apparatus: Iodination flask, conical flasks, Burette, Pipette, Beaker and Test tube.

Chemicals: Methyl Acetate, 0.5M HCl solution, 0.5M NaOH solution, Phenolphthalein indicator, Ice cold water.

Principle: Hydrolysis of Methyl Acetate is catalyzed by mineral acids. The acetic acid formed in the reaction is titrated at various intervals of time with standard alkali to know the progress of the reaction. The amount of alkali consumed at any time corresponds to the quantity of ester decomposed up to that time. The rate constant can be calculated by the following expression.

$$K = \frac{2.303}{t} \log \frac{(V\alpha - Vo)}{(V\alpha - Vt)}$$

Where Vo is the initial titre value (zero time), Vt is the titre value at any time and Vα is the titre value at the completion of reaction, t is the time interval.

A constant value of K shows that the above reaction follows first order kinetics (Pseudo unimolecular). A plot of log $\frac{(V\alpha - Vo)}{(V\alpha - Vt)}$ Vs. Time, gives a straight line passing through origin with a slope equal to K/2.303.

Procedure: Take 10ml of Methyl Acetate in a dry test tube and 100ml of HCl solution in an Iodination flask then keep in water bath to attain constant temperature. Fill the burette with the given NaOH solution and fix it to the stand. Take nearly 30ml of ice cold water in a conical flask and add 2-3 drops of Phenolphthalein indicator.

Now add 10ml of methyl Acetate to the iodination flask containing HCl solution and shake it once and simultaneously start the stop clock. Immediately pipette out 10ml of this reaction mixture into the conical flask containing ice cold water. Titrate it against NaOH solution taken in the burette until a pale pink colour appears, indicating the end point. Stop the titration and note down the titre value as Vo.

Clean the conical flask then take around 30ml of ice cold water and add 2-3 drops of phenolphthalein indicator and keep it ready for the next titration. Exactly at the 10th minute pipette out 10ml of reaction mixture into ice cold water, titrate it against standard NaOH solution taken in the burette until a pale pink colour appears .Note down

the titre value as V_{10}.

Repeat the above titration for every 10mins and note the titre values corresponding to $V_{20,}$ V_{30} and ………..up to V_{60}. Tabulate the reading and calculate the first order rate constant.

Calculation for Vα:

Take 20ml of the reaction mixture into another conical flask and heat it on water bath for about 20 mins. Then cool the conical flask under tap water and pipette out 10 ml of the solution into a conical flask and add 2-3 drops of phenolphthalein indicator and titrate it against NaOH present in the burette until pale pink colour appears. Note down the titre value as Vα.
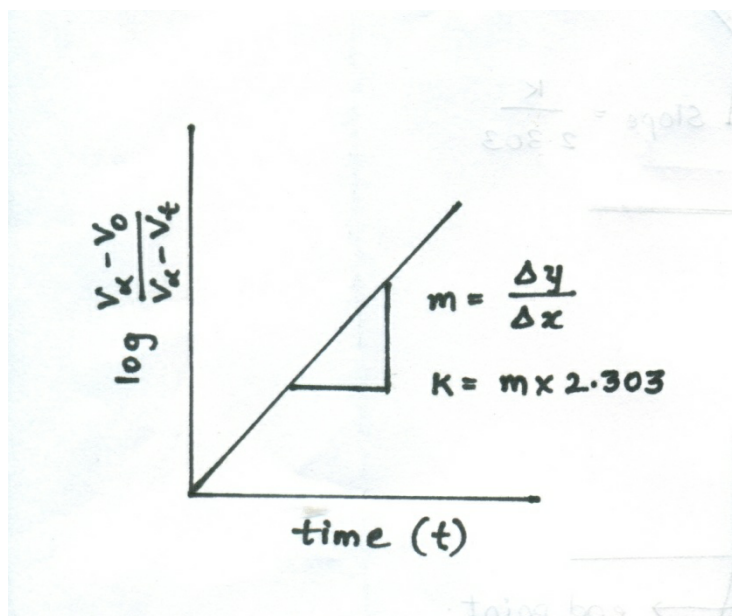
A graph is drawn between Log $\frac{(Vα - Vo)}{(Vα - Vt)}$ Vs Time. A straight line graph passing through the origin is obtained. Slope is equal to K/2.303

Calculations: Tabular form

S.No	Time (Mins.)	Vol.of NaOH (Vt)	Vα − Vt	$\frac{(Vα - Vo)}{(Vα - Vt)}$	Log $\frac{(Vα - Vo)}{(Vα - Vt)}$	K = $\frac{2.303}{t}$ log $\frac{(Vα - Vo)}{(Vα - Vt)}$
1	0					
2	10					
3	20					
4	30					
5	40					
6	50					
7	60					
8	α					

Graphical Representation

$$m = \frac{\Delta y}{\Delta x}$$

$$K = m \times 2.303$$

Result: The rate constant obtained from experiment (K) = _____ min^{-1}

The rate constant obtained from graph (K) = _____ min^{-1}

HYDROLYSIS OF METHYL ACETATE

Viva – Questions

1. What is hydrolysis?

2. What is the role of HCl?

3. Why do we need to add ice?

4. Write the equation of the first order reaction?

5. What is the order of a reaction?

6. What is molecularity?

7. Why do we heat the reaction mixture?

8. What are the units for rate constant of a reaction?

9. What are the units of first order rate constant?

10. Draw a graph between log $\frac{(V\alpha - V_o)}{(V\alpha - Vt)}$ Vs Time.
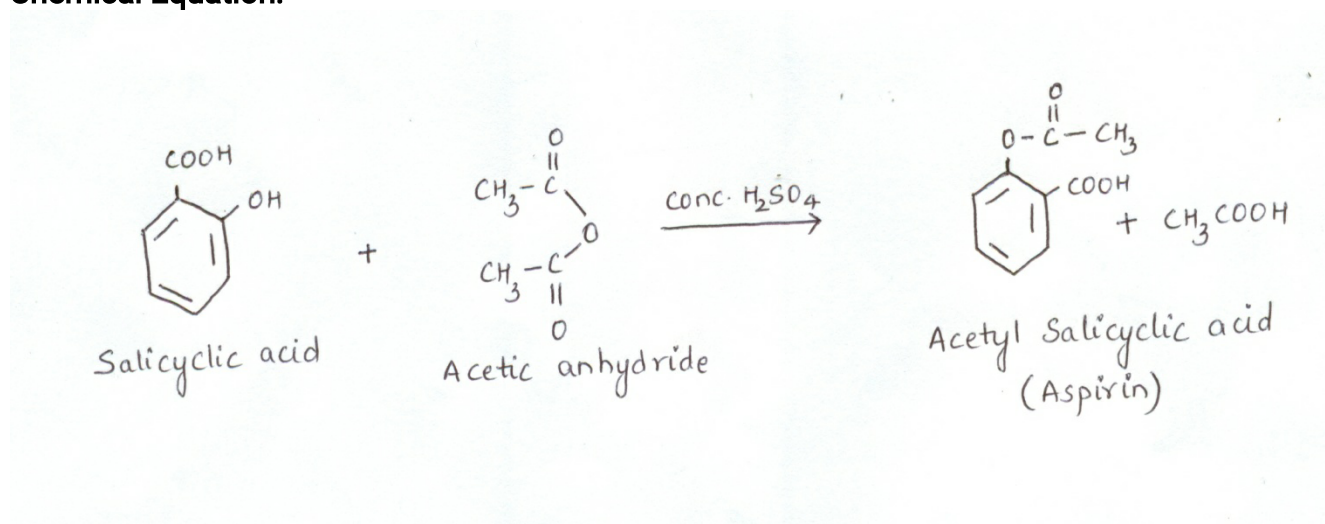
EXPERIMENT NO: 8

PREPARATION OF ASPIRIN

Aim: To prepare a pure sample of Acetyl Salicylic Acid i.e. Aspirin.

Apparatus: Conical Flask, Beaker, Glass Rod, Water bath and Funnel.

Chemicals required: Salicylic Acid (2 gms), Acetic Anhydride (8 ml), Conc.H_2SO_4,

Chemical Equation:

Procedure: Take 2 gms of Salicylic Acid in a clean and dry 250ml conical flask and add 8ml of Acetic Anhydride followed by 2 drops of Conc.H_2SO_4. Stir well and warm it on a water bath at 50°C to 60°C with occasional stirring for about 30 minutes. Pour the reaction mixture into a beaker containing 30ml of cold water and stir it with glass rod. Filter and wash the crude Aspirin with cold water.

Recrystallization: The crude aspirin is purified by dissolving in 30ml of hot boiling water with charcoal and then filter it; white needles of aspirin will be obtained in the filtrate.

Report: Yield obtained is 2.5 gms

Melting Point: $128^{o}C$ -$135^{o}C$

PREPARATION OF ASPIRIN

Viva – Questions

1. Write the chemical name of Aspirin?

2. What is the use of Aspirin?

3. Write down the chemical reaction involved for preparation?

4. Give the name of reaction used for the preparation?

5. What is a condensation reaction?

6. What is the catalyst used in the reaction?

7. Define Anti-pyretic.

8. Define Analgesic.

9. How do we distinguish between salicylic acid & acetyl salicylic acid?
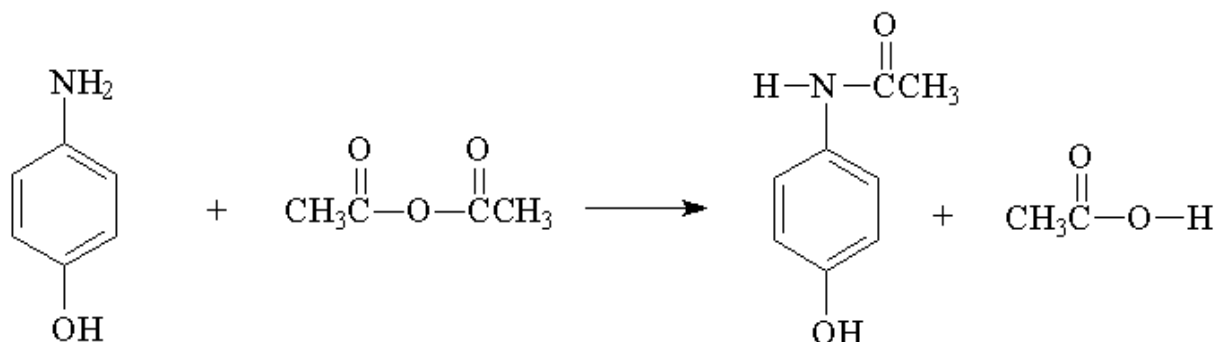
10. Define melting point?

EXPERIMENT NO: 9
PREPARATION OF PARACETAMOL

Aim: To prepare a pure sample of Paracetoaminophenol.

Apparatus: Conical Flask, Beaker, Glass Rod, Water bath and Funnel.

Chemicals required: Acetic Anhydride (8 ml), Conc.H_2SO_4,

Chemical Equation:

Procedure: Place 2gr of the 4-aminophenol into a 250ml conical flask, add 15ml of water and stir the suspension vigorously (by using magnetic stirrer) for a few minutes. Add 2.5ml of ethanoic anhydride and continue stirring until the suspension dissolves and a precipitate (Paracetamol) eventually forms. After 10 minutes filter the precipitate (using a buchner funnel), washing with small amounts of cold, distilled water.

Recrystallization: After drying, the crude product should be placed in a clean 100ml beaker and heat until it just dissolves in approximately 20ml of water. Cool the beaker in ice until crystals of the purified paracetamol appear. Filter the crystals. Repeat recrystalisation process to achieve a more pure product

Report: Yield obtained is 2.5 gms

Melting Point: 170°C)

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Viva – Questions

</div>

1. Write the chemical name of paracetamol?

2. List out the uses of paracetamol.

3 . Write down the chemical reaction involved for preparation?

4. Give the name of reaction used for the preparation?

5. What is a condensation reaction?

6. What is the catalyst used in the reaction?

7. Define Anti-pyretic.

8. Define Analgesic.

<div align="center">

EXPERIMENT: 10

CALCULATION OF RF VALUES BY THIN LAYER CHROMATOGRAPHY

</div>

AIM: To separate the components present in the given mixture by means of TLC

CHEMICALS: o-nitrophenol, p-nitrophenol, silica gel, n-hexane, ethyl acetate.

APPARATUS: TLC slide, beaker, watch glass

PRINCIPLE: Thin layer chromatography is mainly based on adsorption chromatography technique. The compound spotted on thin layer of silica gel coated on a glass plate gets adsorbed onto the thin layers(stationary phase) and gets partitioned between the thin layer and the mobile phase, when the mobile phase percolates through the thin layer. As adsorption and partition takes place simultaneously, the compound will move along the mobile phase. When more than one components are present, movement of components is according to their differential migration rates. The component which has grater affinity towards the stationary phase will move later than the one which has relatively lesser affinity towards the stationary phase.

PROCEDURE:

Step – I: Preparation of Slurry:

A solvent system consisting of 7ml of n-hexane and 3ml of ethyl acetate is prepared. 35gm of silica gel is weighed and transferred it into a bottle. To this the solvent mixture is added and mixed thoroughly such that a uniform silica slurry is obtained.

Step – II: Preparation of TLC slides:

Two clean and dry TLC slides are dipped together in the slurry carefully and removed. A uniform layer of silica gel gets adsorbed on slides along with little solvent. Then allow the solvent to evaporate by keeping the slides aside for few minutes.

Step – III: Development of plate:

2-3 ml of the solvent is taken in a glass beaker and covered with a lid and kept aside for few minutes so that the beaker gets saturated with solvent vapours. A saturated solution of given mixture is prepared in acetone or methanol in a test tube. A small amount of the solution is taken with a capillary tube and is spotted on TLC slide just 5mm above the bottom. The solution from the capillary tube now comes out and adsorbed on silica plate. The point at which the compound is spotted on the plate is called loading point. Now place the plate gently inside the beaker in a slant position and covered with a lid. Observe that the loading point is above the solvent. Allow the solvent to ascend on the plate by capillary action. The compound spotted move upward direction along with the solvent. Then remove the slide from the beaker after the solvent reaches to the maximum height. Then allow the solvent to evaporate. If there are any colored spots, circle them lightly with a pencil. Note down the distance travelled by the component from loading point

Before development   After development
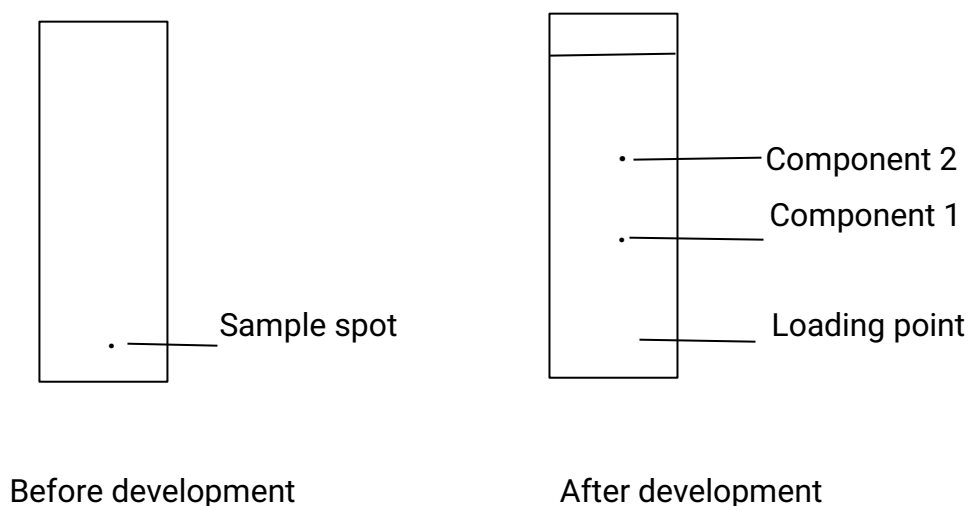
Step – IV: Calulation of Rf value

The retention factor, or Rf, is defined as the distance traveled by the compound divided

by the distance traveled by the solvent.

$$Rf = \frac{\text{Distance travelled by the compound}}{\text{Distance travelled by the solvent front.}}$$

Result: Rf of component 1 is _____.

Rf of component 2 is _____.

EXPERIMENT: 11

DETERMINATION OF ACID VALUE OF COCONUT OIL

AIM: To determine the acid value of coconut oil using potassium hydroxide solution.

APPARATUS: Burette, beaker, conical flask, measuring cylinder and 100ml standard flask.

CHEMICALS: Coconut oil, NaOH solution, fat solvent (mixture of 100ml ethanol and 100ml diethyl ether) and phenolphthalein indicator.

THEORY: Acid value is expressed as the percentage of free fatty acid present in an oil or fat. It is defined as the number of milligrams of caustic potash or caustic soda required to neutralize the acid in 1 gm of the sample. Fatty acids are normally found in the triglyceride form, but hydrolysis of these fatty acids accounts to the decreased quality of the oil. The normal acid value for most samples lies within 0.5. A high acid value indicates a stale oil or fat stored under improper conditions.

PROCEDURE: It consists of 3 steps.

Step-I: Preparation of Standard oxalic acid solution

About 0.63gm of oxalic acid di hydrate is weighed, transferred to a standard flask and dissolved in small amount of distilled water and made up to the mark in 100ml of volumetric flask.

Normality of Oxalic acid = Wt/Eq.wt x 1000/volume

Step – II: Standardization of NaOH Solution

Pipette out 20 ml of Oxalic acid into a conical flask, then add two drops of phenolphthalein indicator and titrate it against sodium hydroxide taken in a burette till the colour changes to pale pink. The experiment is repeated till concurrent readings are obtained.

S.No	Vol of Oxalic acid	Burette Readings		Vol of NaOH run down
		Initial	Final	
1	20			
2	20			
3	20			

Vol of Oxalic acid Solution V_1 = 20 ml

Normality of Oxalic Acid N_1 =

Vol of NaOH Solution V_2 = ---- ml

Normality of NaOH N_2 = -----N

$$N_1V_1 = N_2V_2 \Rightarrow N2 = \frac{N_1 V_1}{V_2} \rightarrow$$

$\therefore$ Normality of NaOH N_2 = ----------------N

Step – III: Determination of acid value:

5ml of oil is weighed and transferred into 250 ml conical flask. To this 50ml of neutralized alcohol solution and 2 drops of phenolphthalein indicator are added and titrated against NaOH solution till the color changes to pale pink.

S.No	Weight of oil	Burette Readings		Vol of NaOH run down
		Initial	Final	
1	5			
2	5			
3	5			

CALCULATION

Acid value = Volume of NaOH x Normality of NaOH x Eq.Wt of NaOH

Weight of Oil sample

Equivalent weight of NaOH = 40

Acid Value = _____.

RESULT: The acid value of the coconut oil is _____.

EXPERIMENT NO: 12

ADSORPTION OF ACETIC ACID ON ACTIVATED CHARCOAL

AIM: To calculate the amount of acetic acid adsorbed on activated charcoal and also to verify Freundlich adsorption isotherm.

APPARATUS: Burette, pipette, conical flask, funnel, reagent bottles, volumetric flasks, beakers, glazed filter papers.

CHEMICALS: 0.5N acetic acid, 0.1N NaOH, activated charcoal, phenolphthalein indicator.

PRINCIPLE: Adsorption is the accumulation of a gas or liquid solute (the adsorbate) on a surface of a solid or a liquid (the adsorbent) forming a molecular or atomic film. Adsorption is operative in most natural physical, biological, and chemical systems, and is widely used in industrial applications such as activated charcoal, synthetic resins and water purification. As adsorption is a surface phenomenon the amount adsorbed will depend on the specific area of the adsorbent. The surface area available per gram of an adsorbent is known as its specific area.

The amount of gas adsorbed by a given amount of an adsorbent also depends on temperature and pressure. At a given temperature, the variation of the amount of solute adsorbed with change in pressure is given by Freundlich adsorption isotherm as,

$$x/m = KC^{1/n}$$

where 'x' is weight of solute adsorbed

'm' is weight of an adsorbent

'C' is equilibrium concentration of the solute in solution.

'K' and 'n' are constants whose values depend on temperature and nature of adsorbate and adsorbent.

Taking logarithms of the above equation we get,

$$Log\ x/m = logK + 1/n\ logC$$

Freundlich adsorption isotherm is said to be verified only when a straight line having an intercept is obtained by plotting a graph between log x/m versus log C.

PROCEDURE:

Take six clean and dried reagent bottles and label them as 1, 2, 3, 4, 5 and 6. Take two burettes and fill one with distilled water and the other with 0.5N acetic acid and prepare six samples as follows:
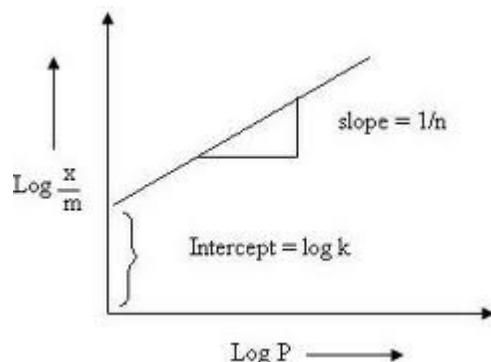
	1	2	3	4	5	6
Acetic acid(ml)	50	40	30	20	10	0
Distilled water(ml)	0	10	20	30	40	50

Mix the solutions well. Then weigh six different 2gm quantity of charcoal on glazed papers and add to each of the six flasks. Shake the solutions thoroughly for about 30 minutes shaking the contents from time to time. Then filter the solutions and reject about 5 ml of the initial filtrate in each case and collect the remaining filtrates in separate beakers. Then pipette out 10 ml of solution from each beaker into six conical flasks add 2 drops of phenolphthalein indicator and titrate against standard sodium hydroxide taken in a burette till pale pink color is obtained. Repeat the titration to get concurrent values. Let the titre values for all the flasks be V_1, V_2, V_3, V_4, V_5 and V_6. Then calculate the amount of acetic acid adsorbed(x) and plot a graph between log x/m and log C.

OBSERVATION TABLE:

		Volume	Initial concentrati	Equilibrium concentration(af	Amount			

Flask No.	of NaOH reqd for 10 ml solution	on of acetic acid in moles/lit(C_i)	ter adsorption) of acetic acid in moles/lit(C_e)	adsorbed $x = C_i - C_e/20$	x/m m is mass of charcoal.	Log x/m	Log C_e
1							
2							
3							
4							
5							
6							

GRAPH:

RESULT:

Amount of acetic acid adsorbed = _____ gm.

From the graph the Freundlich adsorption isotherm is said to be _____.

EXPERIMENT NO:12
DETERMINATION OF VISCOSITY

AIM: To determine the viscosity of a given liquid using Ostwald viscometer.

APPARATUS: Ostwald viscometer, beaker, pipette, rubber tube, stand.

CHEMICALS REQUIRED: Butanol and distilled water.

PRINCIPLE: It is the property of fluids. It is defined as the resistance to the flow of a liquid. It measures the resistance to the flow shown by a layer of fluid when another adjacent parallel layer tends to flow past the first layer. When a liquid flows through a narrow tube, the velocity of flow depends on the force that produces the flow. The

same amount of various liquids flowing through the same capillary tube doesn't take the same time to flow. This is due to the difference in their viscosities. If suppose two liquids with viscosities η_1, η_2 having densities d_1, d_2 and their time of flow t_1, t_2 respectively then,

$$\frac{\eta_1}{\eta_2} = \frac{d_1 t_1}{d_2 t_2}$$

PROCEDURE: Clean the viscometer with chromic acid and then with distilled water. Dry the apparatus and clamp it in vertical position to the stand. Now pipette out 7 ml of distilled water and introduce into the viscometer to bulb A side. Suck the liquid by means of rubber tubing a little beyond level A of the bulb B. allow the liquid to flow through the capillary tube under its own weight. When the liquid reaches level A start the stop clock and when the liquid reaches level B, stop the stop clock. Note down the time taken for the flow of water. Repeat the above procedure for 3 times and find the mean time flow for distilled water. Let it be t_1.

Repeat the above procedure for organic liquid after cleaning and drying the viscometer. Note down the mean time flow for organic liquid. Let it be t_2.
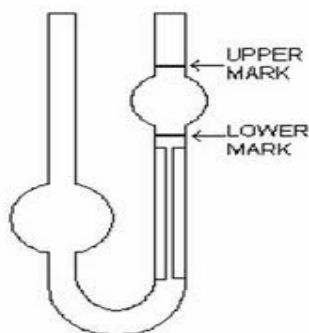
OBSERVATION TABLE

S.No.	Time taken for the flow of water(t_1) sec.	Time taken for the flow of organic liquid(t_2) sec.
1		
2		

3		
	Average time taken =	Average time taken =

$$\frac{\eta_1}{\eta_2} = \frac{d_1 t_1}{d_2 t_2}$$

η_1 = Viscosity of distilled water = 8.90 millipoise.

η_2 = Viscosity of organic liquid = ------------- millipoise.

d_1 = Density of water = 1 gm/cm^3.

d_2 = Density of organic liquid = 0.81 gm/cm^3.

t_1 = time taken for the flow of water = -------------- sec.

t_2 = time taken for the flow of organic liquid = ---------- sec.

Therefore viscosity of organic liquid, $\eta_2 = \dfrac{\eta_1 \times d_2 t_2}{d_1 t_1}$

RESULT: Viscosity of the given organic liquid is -------------------- millipoise.

Viva – Questions

1. Define Viscosity.

2. Define absolute Viscosity.

3. Define kinematic Viscosity.

4. Give the mathematical formula for viscosity.

5. Give the relation between absolute and kinematic viscosity.

6. What is the importance of viscosity? Where is this phenomenon used.

7. Give the relation between viscosity and density of lubricants.

8. Give the relation between viscosity and temperature of lubricants.

EXPERIMENT-14

DISTRIBUTION OF ACETIC ACID BETWEEN N-BUTANOL AND WATER

Aim: To determine the distribution coefficient of acetic acid between n- butanol and water.

Chemicals: Water, acetic acid , n-butanol.

Apparatus: Reagent bottles, pipette, conical flask, burette. etc.

Principle:

If a solid or liquid (i.e. solute) is added to a mixture of two immiscible liquids, in both of which it is soluble, it will distribute itself between the two liquids according to the partition law.

The Partition Law states that "a solute "X" distributes itself between two immiscible solvents in such a way that the ratio of the concentrations in the two solvents is constant at a fixed temperature " (provided that the solute is in the same molecular state in both solvents).

i.e., $\dfrac{\text{Concentration of X in solvent A}}{\text{Concentration of X in solvent B}}$ = a constant = K

Let C_1 be the concentration of X in solvent A, C_2 be the concentration of X in solvent B then C_1/C_2 = K. This constant, which varies with temperature, is known as partition coefficient or distribution constant.

Procedure:

In a clean distribution (Reagent) bottle, take 50 ml of n-butanol and add 50 ml of 0.5 N acetic acid solution to it. Cork the bottle and shake it well (without spilling the solution) for about 5 minutes. Open the cork and keep the bottle aside with intermittent shaking so that the two layers separate out. Take (0.1 N) standard NaOH solution into a clean burette and fix it to the stand. Pipette out 10 ml of the upper layer (organic layer) into a clean conical flask and to that add 2 drops of phenolphthalein indicator and titrate this solution against standard NaOH solution until light permanent pink colour is obtained. (While titrating, the solution must be shaken vigorously). Now, note down the titre value (V_{org}). Similarly, Pippete out 10 ml of the lower layer (water layer) into a clean conical flask and add two drops of phenolphthalein indicator and titrate it against standard NaOH solution until the colour changes to light pink. Note down the titre value obtained (V_{aq}).

Now 10 ml of n-butanol and 10 ml of distilled water are added to the same distribution bottle and the solution is shaken thoroughly for 5 minutes. The titrations are repeated for both the layers as above. In the same way, five readings are taken and the data obtained is tabulated and distribution constant "K" is calculated both from the table as well as from the graph.

Table:-

Volume of both organic layer and aqueous layer taken for titrations is 10 ml

each.

Sl. No.	Titre value of organic layer			Titre value of aqueous layer			$K=V_{org}/V_{aq}$
	Intial	Final	V_{org}	Intial	Final	V_{aq}	
1							
2							
3							
4							
5							
6							

Graph:

When a graph is plotted taking V_{aq} on X-axis and V_{org} on Y-axis, an ascending straight line is obtained. It passes through the origin "O". Slope of the straight line in the above graph is "K"/ "K" is partition coefficient.
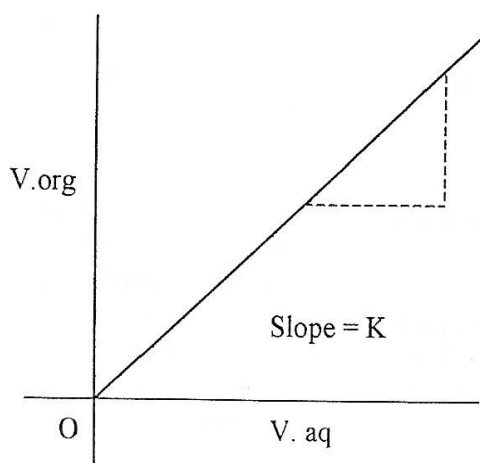
$$K = \frac{V.org}{V\,aq}$$

$$V.org = K \times V.aq$$

$$Y = m\,x$$
$$Slope = m = K$$

RESULT: The partition coefficient of Acetic acid between n-butanol and water K is = --------

Distribution co efficient of Acetic acid in n-Butanol and Water
Viva questions

1. What is Partition co-efficient (K)?

2. Define Nernst Distribution law.

3. What do you understand by the term immiscible liquids?

4. Why organic solution formed as upper layer?

5. What are the applications of distribution law?

6. Write the limitations of Nernst Distribution law.

7. What are the factors effecting Partition co-efficient (K)

8. Why butanol does not dissolve in water?

EXPERIMENT: 15

DETERMINATION OF SURFACE TENSION OF A LIQUID

Aim: To determine the surface tension of a given liquid using stalagmometer.

Chemicals: Water, test liquid (liquids like benzaldehyde, aniline, acetophenone, soap solution etc.)

Apparatus: Stalagmometer.

Principle: The force in dynes acting along the surface of the liquid at right angles to any line 1cm in length is known as surface tension. The units of surface tension are dynes cm^{-1}. The number of drops formed by constant volume of a liquid at the flat end of the stalagmometer depends on the surface tension of the liquid. One can determine the relative surface tension of liquid to water by counting the number of drops formed by the liquid and water of the same volume.

Let γ_L be the surface tension of the liquid

Let γ_w be the surface tension of the water (γ_w = 72 dynes/cm)

Let n_L be the number of drops formed by liquid

Let n_w be the number of drops formed by water

Let d_L be the density of liquid

Let d_w be the density of water

These are related by: $\gamma_L / \gamma_w = n_w d_L / n_L d_w$

Procedure: Clean the stalagmometer and fill it with distilled water by sucking it while immersing the lower end into the distilled water. Bring the level of water to upper mark. Open the pinch cock; adjust it so that the rate of flow of liquid is about 12-15 drops per minute. Allow the drops to fall into a weighing bottle (whose weight has already been taken) till water level reaches the lower mark. Count the number of drops and take the weight of bottle along with water. Repeat the same with the given liquid.

Calculations:

Weight of empty weighing bottle (W_1) = g

Weight of weighing bottle + Dist. Water (W_2) = g

Weight of empty weighing bottle + liquid (W_3) = g

$m_L = W_3 - W_{1} = \text{---------} g$

$m_W = W_2 - W_{1} = \text{---------} g$

$d_L = m_L/m_W \times d_w$

Density of distilled water i.e d_W = 1 gr/ml

Surface tension of sample liquid (γ_L) is given by

$$\gamma_L / \gamma_w = n_w \, d_L / n_L \, d_w$$

S.No.	Volume of solution	No. of Drops	Density of solution(gm/cc)	Surface Tension
1	50ml Distilled water	$n_w =$	d_w = 0.99 gm/cc	U_w=72dynes/cm
2	50 ml Soap Solution	$n_s =$	$d_s =$	
3	25 ml Soap Solution+25 ml Distilled Water	$n_s =$	$d_s =$	
4	25ml above Soap Solution+25 ml Distilled Water	$n_s =$	$d_s =$	

Result: The surface tension of sample liquid is = -------------------- dynes/cm

Viva Questions

1. Define Surface tension

2. Give the C.G.S & S.I units of surface tension

3. What method do you use in laboratory for determination of surface tension?

4. What is the shape of liquid drop falling from capillary?

5. Why liquid drops are spherical in shape?

6. Which instrument is used for measuring the density of a liquid?

7. What is the effect of the rise in temp on the surface tension of the liquid?

8. Why surface tension decreases with the rise in temperature

9. What formula is used for calculating the surface tension of a liquid?

10. What is the Reference sample used for measuring the surface tension of any liquid and
    what is its value at room temp?