ENGINEERING CHEMISTRY LABORATORY EXPERMENTS

B.Tech I YEAR

EXP...No. 1

TITRIMETRY

AIM: Estimation of hardness of water by EDTA method

APPARATUS: - Burette, Pipette, beakers, Standard flask, Conical flask ………etc.

CHEMICALS: - 0.1 M EDTA, Eriochrome, Blacke-T, water sample buffer solution.

THOERY:- Estimation of hardness by EDTA method is based on the following principles.

1. First, the indicator Eriochrome Black-T, which is a blue colored dye, forms an unstable complex with calcium or Magnesium ions in hard water at P^H of 9 to 10. The complex is wine red in color.

$$Mg^{2+}/Ca^{2+} + \text{Eriochrome Black-T} \rightarrow [\text{Mg/Ca—Eriochrome black-T})$$
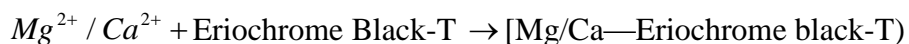
Unstable Complex (Wine Red)

2. As this solution is titrated against EDTA, the free Ca^{2+} and Mg^{2+} tones in water from stable metal ion EDTA $\rightarrow$ [Mg/Ca-EDTA] Stable Complex. Colorless

   Once the free metal ions are complexed, the EDTA replaces Ca and Mg ions from the unstable indicator complex also, to form a stable complex, with the result, the indicator is set free. Since the free indicator is blue in colour at the above mentioned PH, the end point is the appearance of blue color

   [Mg/Ca-Eriochrome Black-T] +EDTA $\rightarrow$ [Mg/Ca-EDTA] + Eriochrome Black-T(Blue)

   The temporary hardness is removed by boiling and after the removal of precipitate by filtration; the permanent hardness in the filtrate is determined by titration with EDTA as above.

Procedure:-

1 .Rinse and fill the burette with EDTA solution.

2. Pipette out 25 ml of hard water into volumetric flask and add 20 ml of distilled water and
    10 ml of buffer solution

3. Add 2-3 drops of Eriochrome Black-T indicator.

4. Titrate against 0.01M EDTA solution till the color of the solution changes from wine red to
    Blue.

5. Note the final reading and repeat to get three concordant readings.

Observation Table:

Sl.No	Volume of the solution taken in the titration flask (nl)	Burette Readings		Volume of the titrant used $V_2 - V_1$ nl
		Initial Reading(V_1nl)	Final Reading (V_2nl)	

$\rightarrow$ Volume of hard water taken for each titration = 25 ml

$\rightarrow$ Volume of 0.01 M EDTA used [from titration] = V_1 ml

Calculation: - 100 ml of 1 M EDTA = 100 gm of $CaCo_3$ = 1 mg of $CaCo_3$

1 ml of 0.01M EDTA = $100 \times \dfrac{1}{1000} \times \dfrac{1}{100\,gm}$ of $CaCo_3$ = 1 mg of $CaCo_3$

V ml of 0.01M EDTA = I × V Mg of $CaCo_3$ = x mg of $CaCo_3$

25 ml of hard water contains=x mg of $CaCo_3$

1000 ml of hard water contains = x/25×100 mg of $CaCo_3$ = y Mg/L

RESULT: The Total hardness of water= y Mg/L

Precautions:-

1. The burette and pipette, conical Flask should be rinsed with distilled water.

2. The color change near the end point is very slow and this should be observed carefully.

EXPERIMENT NO.2

DETERMINATION OF Cu^{2+} THROUGH IODOMETRIC TITRATION

AIM: Determination of Cu percentage in Copper one (Brass)

APPARATUS: Burette, Pipette, Beakers, Titration flask

CHEMICALS: Copper One, Hypo, KI, Br_2 Solution, Acetic Acid, Nitric and HCl, H_2So_4

INDICATOR: Starch solution

END POINT Blue to Colourless

PROCEDURE:

Preparation of Solution of ore

1. Take about 1 g of the finely ground copper ore in a beaker and warm with 15-20 ml of conc. HNO3 until the copper passes into solution.

2. Evaporate to a volume of 5 ml and add 10 ml of conc. HCl and equal volume of 1: 1 H_2SO_4.

Again evaporate till dense fumes of SO_3 appear indicating complete removal of nitrogen oxides.

3. Heat the mixture with 15 ml of bromine solution and an equal volume of water to complete oxidation of any As and Sb to the Pentavalent state.

4. Boil off excess of bromine and dilute the resulting solution to 100 ml in a measuring flask.

Titration:

1. Rinse and fill the burette with 0.1 N Na2S2O4 solution

2. Measure 20 ml of the ore solution into a conical flask

3. Neutralize any free acid present by adding Na2CO3 solution drop by drop till a faint permanent precipitate remains even on shaking.

4. Add dil. Acetic acid drop wise until precipitate just dissolves

5. Add 1 gm of solid Kl, cover the mouth of conical flask by filter paper and allow the mixture to stand for 2 to 5 minutes in the dark the solution becomes brown due to liberated iodine

6. Titrate the liberated iodine with hypo solution added from the burette, the brown colour of iodine becomes fainter as the addition by hypo solution proceeds and when only a faint yellow colour remains add 2 ml starch solution. This immediately form a deep blue iodo-starch complex

7. Now add hypo solution further, drop by drop when only milky white colour remains.

Observation Table:

Sl.No	Volume of the solution taken in the titration flask(ml)	Burette Readings		Volume of the titrant used (final-Initial reading((ml)
		Initial Reading	Final Reading	
1. 2. 3.				

Calculations:

(Ore Soln.)   (Na$_2$S2O$_3$)

$N1V1 \quad = \quad N_2V_2$

$N_1 \times 20 \quad = \quad 0.1 \times V_2$

$N_1 \quad = \quad \times \dfrac{63.5}{Strength of Solution} \times 100 \quad \dfrac{0.1 \times V_2}{20}$

$N(Cu^{2+}) \quad = \quad \dfrac{V_2}{200}$ Strength of $Cu^{2+} = \dfrac{V_2}{200} \times 63.5$ g/L

Percentage of Cu in ore solution $= \dfrac{V_2}{200} \times \dfrac{63.5}{Strength of Solution} \times 100$

RESULT: The amount of Cu present in copper ore solution is …….

EXPERIMENT NO.4

COLORIMETRY

ESTIMATION OF COPPER BY COLORIMETRIC METHOD

AIM: Estimation of Copper by Colorimetry

APPARATUS: - Colorimeter, Test tubes, Burettes.

PRINCIPLE: - Colorimeter measures the optical density of an absorbing substance where optical

density (O.D) is defined as $O.D = \log\dfrac{I_o}{I} \rightarrow 1$

Where I_o = Intensity of incident light

$\qquad$ I = Intensity of transmitted light

As per beers law, optical density of an absorbing substance is related to the concentration by the

equation. $\qquad\qquad O.D = E.C.l$

$$O.D = (E.l).C \rightarrow 2$$

Where 'C' is the concentration of the substance, l is the path length, which represents the width

of the cell used and is constant for a given cell used, E is the molar absorption coefficient and is

a constant for given substance. Equation 2 may be written as $O.D. \alpha \ C \rightarrow 3$

Equation 3 represents the quantitative form of Beer's law . if the optical density of a substance is

determined at varying concentration. A plot of O.D.vs C gives a straight line.

PROCEDURE:- take the sample solution of CuSo4 and prepare the following 10 sample

solutions in test tubes as 1 to 10

1. $\qquad$ 1 ml 0.1 m $CuSo_4$ + 9 ml Distilled water
2. $\qquad$ 2 ml 0.1 m $CuSo_4$ + 8 ml Distilled water
3. $\qquad$ 3 ml 0.1 m $CuSo_4$ + 7 ml Distilled water
4. $\qquad$ 4 ml 0.1 m $CuSo_4$ + 6 ml Distilled water
5. $\qquad$ 5 ml 0.1 m $CuSo_4$ + 5 ml Distilled water
6. $\qquad$ 6 ml 0.1 m $CuSo_4$ + 4 ml Distilled water
7. $\qquad$ 7 ml 0.1 m $CuSo_4$ + 3 ml Distilled water
8. $\qquad$ 8 ml 0.1 m $CuSo_4$ + 2 ml Distilled water
9. $\qquad$ 9ml 0.1 m $CuSo_4$ + 1 ml Distilled water
10. $\qquad$ 10 ml 0.1 m $CuSo_4$ + 0 ml Distilled water

The ten sample solutions prepared above have a varying concern from 0.01m to 0.1m, choose the filter in the colorimeter with maximum absorbance. Tabulate the result of filter and O.D with a given CuSo$_4$ sample solutions.

After selecting filter, determine the O.D. of the above mentioned ten sample solutions and tabulate the results.

Observation:

Table-1

Filter No	λ-Range (nm)	Peak (nm)	Optical Density(OD)
1			
2			
3			
4			
5			
6			
7			
8			

Table-II   **Selected Filter--------------------**

Sl.No	Volume of $CuSo_4$ (ml)	Volume of H_2O (ml)	Concentration of $CuSo_4$	Optical Density
1	10	0		
2	9	1		
3	8	2		
4	7	3		
5	6	4		
6	5	5		
7	4	6		
8	3	7		
9	2	8		

10	1	9		

GRAPH: - Plot a graph of O.D Vs [CuSo4]. A straight line passing through origin is obtained the slope of which gives El, E is the molar absorption coefficient.

$$\text{Slope} = \frac{y_2 - y_1}{x_2 - x_1}$$

$$slope = El \Rightarrow E = \frac{slope}{l}$$

$$E = -mole^{-1}cm$$

RESULT: - Amount of copper present in given $CuSo_4$ solution is _____grams?

EXP.NO.5

CONDUCTOMETRY

Aim: - To determine strength of given HCl Solution Conductometrically

APPARATUS: - Conductivity meter, conductivity cells, Beakers, Pipette, Burette

CHEMICALS:- Oxalic acid Solution (0.1N), NaOH Solution, HCl, Phenolphthalein Indicator.

Preparation of Standard Oxalic acid Solution (o.1N)
About 1.6g of Oxalic acid dehydrate is weight accurately and dissolved in small amount of distilled water and made up to the mark in 250 ml volumetric flask.

PRINCIPLE: - The conductivity of HCl is very high due to mobility of H+ ions in it. When a solution of strong alkali is added to the solution of strong acid, the highly conducting H^+ ions are replaced by Na^+ ions is considerably smaller than H^+ and hence addition of alkali to the acid always accompanied by a decrees in conductivity. When all the H^+ are replaced that is after the end point, further addition of NaOH results in increase in conductivity. from the plot of the conductive Vs Volt NaOH added, the end point can be calculated

PROCEDURE: - 20 ML OF Oxalic acid is pipette out into a conical flask and is diluted to 30 ml with distilled water. To the solution, two drops of phenolphthalein indicator is added and the titrated against sodium hydroxide taken in a burette. The endpoint colorless to pale pink. The experiment is replaced till correct reading are obtained

Determination of Strength of Given HCl Solution:-

The given unknown HCl solution is 100 ml volumetric flask is made up tot the mark and then 10ml of given unknown HCl Solution is taken in a 100 ml beaker. To this 40ml of distilled water is added, the electrode of the cell is dipped in the solution. The conductivity of solution is noted before adding the alksli. The standardized sodium hydroxide is added from a burette with 1ml of increment and by shaking thoroughly, the contents of the beaker the conductivity values are noted. The values of observed conductivities are plotted against volume of sodium hydroxide The concentration of hydrochloric acid is determined using the end point from the graph.

PRECAUTIONS: - after 2 each addition of titrant from the burette the solution should be thoroughly stirred and then the reading should be taken.

RESULT: - The Strength of Hydrochloric Acid is _____ g/lit.

Calculation:-

Standardization of Sodium Hydroxide Solution

S.No	Volume of Oxalic Acid (V_1ml)	Burette Readings		Volume of NaOH Solution (V_2 =F-I)
		Initial	Final	
1	20			
2	20			
3	20			

WE know that $V_1N_1 = V_2N_2$

V1 is Volume of Oxalic Acid = 20ml

N1 is Normality O Oxalic Acid= 0.1N

V2 is Volume of Sodium Hydroxide=- -ml

N2 is Normality of Sodium Hydroxide – N

$$N2 = \frac{V_1 N_1}{V_2}$$

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

Determination of Strength of Hcl:-

Sl.No	Volume of NaOH	Conductivity (ohm^{-1})
1		
2		
3		
20		
40		

Volume of NaOH Solution from graph Ph V2 = -ml   GRAPH:

Normality of NaOH Solution N2= -N

Volume of Hudrochloric acid solution V3= 10ml

Normality of HCl in mixture N3= -----N.

$V2N2 = V3N3$

$$N3 = \frac{V2N2}{V3}$$

Strength of HCl Solution = Normality of HCl Solution ×eg.Wt of HCl

$$= N3 \times 36.5$$

$$= n \underline{\hspace{2cm}} g/Lit$$

EXP.NO.6

POTENTIOMETRY

TITRATION OF STRONG ACID Vs STRONG BASE

AIM:- To Determine the concentration of a strong acid by a Potentiometry titration given a strong base of known concentration.

APPARATUS: - Potentiometer, Pt-electrode, Calomel electrode, Salt bridge, 150 ml beakers, Stirrer, Pipette, Burette.

CHEMICALS: - 0.1m NaOH, 0.1m HCL, Quinhydrone solid, KCl Solution.

PRINCIPLE: - The quinhydrone electrode is prepared by taking saturated solution of quinhydrone in a known quantity of strong acid whose concentration is to be determined. This electrode is combined with the calomel electrode to make the cell. Pt, Hg, $Hg_2Cl_2(s)$/Saturated//H^+/QH_2/Pt

In acid medium quinhydrone exist in two forms.

As NaOH is added to a solution of H+ ions with quinhydrone the H+ ions are consumed steadily and the relative concentration of QH2 and Q changes with the titration. At the end point where all the ions are consumed, QH2 gets completely converted to Q. the electrode potential of the electrodes is given as per the nearest equation.

$$EQH_2/Q = E^0 QH_2/Q - \frac{0.059}{\cap} 10g \frac{aQ.a^2 Qh^+}{aQH_2}$$

$$= E^0 QH_2/Q - \frac{0.118}{n} \log aH \qquad [\because aQ \text{ and } aCH_2 = 1]$$

At the endpoint, which $aH^+ \square o$ there is a sudden fall in the cell emf $E_{cell} = E_{calomel} + E_{aH_2/Q}$

Since calomel is constant the variation of E_{cell} is a measure of the variation of $E_{aH_2/Q}$. The endpoint is the titration is the point where there is a maximum full in potential with a small addition of NaOH.

Procedure: - Pipettes out 10 ml of the given HCl solution into a clean100ml beaker add a pinch of quinhydrone and stir it well to make a saturated solution of quinhydrone is H+. dip a platinum electrode. Take a second beaker with saturated KCl solution in it. Dip the calomel electrode in it. Connect two beakers with a salt bridge; connect the terminals of the potentiometer with the two electrodes. Note the emf of the cell. fill a burette with 0.1 NaOH, add 0.5ml of NaOH into the beakers containing acid, stir well for equilibrium to establish. Note the emf of the cell once again. The variation of emf by successive addition of 0.5 ml of NaOH. Each time will be about 10-20mv, as the endpoint approaches, the fall in the emf would increase from 10-20 mv to 40-50 mv. At this stage add NaOH in batches of 0.2 ml and note emf after each addition. At the end point the emf falls suddenly by about 100-150 mv. Continue the titration with 0.2 ml of NaOH and note 5-6 emf values. Now the variation is emf gets smaller and takes 4-5 emf readings, after the endpoint by adding batches of 1ml of NaOH each time, tabulate the results.

GRAPH: - Plot a graph of emp Vs volume of NaOH added and a second graph of $\Delta E/\Delta V$ Vs Volume of NaOH added. Note the point of Neutralization from both the graph and calculate the cone of given HCl using the concern of given NaOH as 0.1m

RESULT: - At the Neutralization point 10 ml of given HCl =? And concern of given HCl=_____

Sl.No	Volume of Alkali (ml)	Emf (mv)	$\xrightarrow{\text{Poly'n}} \dfrac{\Delta E}{\Delta V}$
1			
2			
3			

. . . 30			

CALCULATION: -

HCl   NaOH

$V_1 = 10ml$ $V_2 = \underline{\qquad}$ (From Graph)

$N_1 = ?$ $N_2 = 0.1$

$$V_1 N_1 = V_2 N_2 \quad \Rightarrow \quad N_1 = \frac{V2 N2}{V_1}$$

$\therefore$ Concentration of given HCl (N_1) = _____N.

EXPERIMENT – 9

IDENTIFICATION OF FUNCTIONAL GROUPS IN ORGANIC COMPUNDS
SYSTEMATIC PROCEDURE

EXPERIMENT   **OBSERVATION**   **INFERENCE**

PHYSICAL CHARACTERSTICS:

a)Physical state   **i) Liquid (or) Solid)**

                            ii) Amorphous (or)

                               crystalline

b) Odour   i) Pale yellow   Nitro compounds

                            ii) Reddish orange   Nitro amines

	iii) Brownish yellow	Aniline
	iv) Pale pink	Phenol
	v) Colourless	Carbohydrates, aldehydes, ketones, carboxylic acids, esters and some simple hydrocarbons.
c) Odour	i) Odourless	Carbohydrates, aromatic acids
	ii) Bitteralononds	Nitrobenzene, benzaldehyde are absent
		Aromatic amines
	iii) Fishy smell	Acetamide
	iv) Mouse like smell	Formaldehyde, formic acid
	v) Pungent smell	Esters
	vi) Pleasant fruites	Phenols
	(Phenolic) Carbolic smell	Acetic acid
	Vinegar like Cinamon like	Cinnamaldehyde

d) Melting point/ Boiling point

II. Solubility : Take a small amount of compound in a test tube and add 3-ml of solvent

a)Water	Soluble in cold water	i) Carbohydrates, glucose, Fructose
		ii) Amides, urea acetamide
b) 5% NaOH	Soluble in 5% NaOH and	May be week acid like phenols
	regerated with HCl	

c) 5% NaHCO$_3$	Soluble in 5% NaHCO$_3$ and	May be strong acids,
		benzolic,
	regenerated with HCl	salicylic acids.
d) 5% HCl	Soluble in 5% HCl regenerated	May be amines like aniline
	with NaOH.	
e) Ether	Soluble in ether	May be nautral compounds
		like, chlorobenzene,
		Aldehydes, Ketones,
		Nitrobenzene etc.

III. Ignition Test :

Heat a small amount Of given compounds On spatula	1) Burns with non-luminous and non-smoky flame.	Aliphatic compound
	2) Burns with Luminous And smokes flame	Aromatic compound
	3) Burns with sugar smell	Carbohydrates

DETECTION OF FUNCTIONAL GROUPS

TEST FOR PHENOLS

i)With neutral FeCl$_3$ test :

| The compound to dissolved In alcohol and add 1 (or) 2 Drops of 5% Fec | Violet (or) Blue colour is observed | Phenol is present |

$$6C_6H_5OH + FeCl_3 \rightarrow H_3[Fe(OC_6H_5)] + 3HCl$$

ii) Liebermann's nitroso reaction:

| Take the compound in a dry Test tube add sodium nitrite And conc sulphuric acid(1ml) Mix well and heat gently | A blue colour is obtained the solution turns red on dilution with water and blue on basification with dil Sol of NaOH | Phenol is present |

Test for Carboxylic Acids:

1.NaHCo3 Test

To a saturated solution	Brisk effervescence	Carboxylic acid is present
Of sodium bicarbonate	is observed	
in water (1ml) add the		
given compound		

$$R-COOH + NaHCO_3 \rightarrow RCOONa + H_2O + CO_2 \uparrow$$

ii) Ester Test:

Heat the mixture	Fruity smell is observed	Carboxylic acid is present
Of the acid, ethanol (1ml)		
And concentrated sulphuric acid		
in a dry test-tube in a water bath.		
Pour the reaction mixture carefully into a		
Beaker containing Na_2Co_3 Solution.		

$$R-COOH + R^1OH \xrightarrow[Heat]{Conc.H_2SO_4} RCOOR^1 + H_2O$$

iii) Fe Cl$_3$ Test:

Neutralise0.5 gm of the	i) A buff (or) brownish coloured	Aromatic acids
Acid with excess of ammonia	ppt is obtained	
In a boiling test tube. Boil the	ii) An intense yellow colour is	α-Hydroxy acids
Solution to remove excess	obtained	
Of ammonia, cool and add a few	iii) A violet (or) fleshy colour is	β-substituted aromatic
Drops of neutral ferric chloride	obtained	hydroxy acid
Solution.		

Test for Carbonyl Compounds:

I) Reaction with 2,4 DNP1

Dissolve the carbonyl compound	a yellow or orange colour	may be a
(100 mg (or) 1-2 drops) in ethanol (2-3ml)	ppt is formed	carbonyl compound
To this add an alcoholic solution of		
2,4 – di nitro-phenyl hydrazine (2 ml)		
and Shake the mixture well.		

ii) Tollen's Test:

To the tollen's reagent (1ml) Add a solution of the aldehyde (2 drops (01) 50 mg) dissolved In aldehyda free alcohol (-2-3 ml) and warm the solution in a hot water bath.	a grey black ppt(or) silver mirror deposits on the inner walls of a test tube	May be aldehyde is present

iii) Fehling Test:

Add Fehling solution (2-3 ml By mixing equal amounts of Fehling A+ Fehling B0 to the Organic compound .and warm the mixture	Red precipitate of cuprous oxide is formed	may be aldehyde is present

$$-CHO + 2Cu(OH)_2 \rightarrow COOH + Cu_2O \downarrow +2H_2O$$

$$-CHO + 2Cu(OH)_2 \rightarrow COOH + Cu_2O \downarrow +2H_2O$$
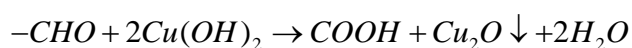
    Fehling Solution   Red

iv) Schiff's Test

|		
To a solution organic Compound add Schiff's Reagent (2-3 drops) and Shake the contents	Violet (or) purple colour (pink) is observed	may be aldehyde is present

v) Iodo form test:

Dissolve the given compound (2-3 ml (or) 100 mg) in water (2-3ml) in a test tube and add 2-3ml NaOH (10%). To this add A saturated solutaion of iodine-in Kl with stirring until the dark colour of iodine persists. Heat the solution in a boiling water bath for 1-2 minutes.	A yellow ppt lodo form is observed	may be ketone

$$CH_3COR \xrightarrow{I_2+NaOH} Cl_3COR \xrightarrow{NaOH} CHl_3 + RCOONa$$ Iodo form yellow solid
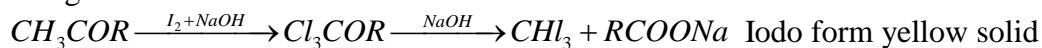
Test for Carbohydrates:

i).Molisch's Test:

Add an alcoholic solution of α -NaPhthol (10%) to an aqueous Solution of substance. Then add Conc H_2So_4 (1ml) carefully along the sides of the test tube allow to stand for 2 minutes.	A violet ring is observed at the junction of two layers	may be carbohydrate

ii).Barfoed Test:

Heat the aqueous solution   i) red colour precipitate of Cu2O   carbohydrate is present
of the compound (iml) with   is formed   present
barfoed reagent (1ml) in
boiling water bath for 1-2 minutes

iii).Test with Benedict solution:

Add Benedict solution (2ml) to the
Aqueous solution of organic substance   Red ppt is observed   Carbohydrate is present
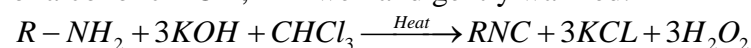(reducing suguar)

IV). TEST FOR $[1^0, 2^0, 3^0]$ AMINES:

1. Carbylamine test:

To the compound add few ml   a foul smell is observed   may be 1^0 amine
Of chloro form and then add 2 ml
of alcoholic KOH; mix well and gently warmed.

$$R - NH_2 + 3KOH + CHCl_3 \xrightarrow{Heat} RNC + 3KCL + 3H_2O_2$$

2. Azodye Test:

Take 2 ml of compound, add 2 ml   orange red dye is formed   presence of 1^0 amine
Of HCl, coll in ice, and then add
2 ml of ice cold 10% Aq NaNo2
solution. Add to it cold solution of 0.4 gms
of 2-Naphthol in 4 ml of 5% NaOH solution is added.

EXPERMENT NO.10

Preparation of Aspirin

AIM   :   To Prepare Aspirin from salicylic Acid

APPARATUS: Conical flask, Water bath, beakers, glass rod

CHEMICALS: Salicylic acid, acetic anhydride, sulphuric acid, alcohol

PROCEDURE:

1. Take 5 gm of salicylic acid in a 100 ml conical flask. To this add about 10 ml of acetic-
   anhydride and 1-2 ml of con.H_2So_4.

2. 2Shake the contents to 800C, because of exothermic reaction. Maintain the tem at 60-70^0
   for about 15 minutes, keeping on a water bath.

3. Allow the solution to come to room temperature and the pour it in 100 ml cold water taken in a 500 ml beaker with stirring (water is added to destroy the excess acetic anhydride which gets converted to acetic acid.

4. To induce crystallization scratches the sides of the flask with glass rod.

5. Filter the solid and wash it with cold water. Press it in between the folds of filter paper.

6. Spread the solid on the filter paper to dry

7. To obtain colorless crystals, recrystallization can be done using equal volumes of ethanol and water solvent system.

RESULT: - Yield=

Precautions:-1 Take acetic anhydride in excess as if acts as acetylating agent as well as
               Solvent.
             2. Make sure that all the salicylic acid is dissolved.

CHEMICAL EQUATION:

Experiment No.11
KINETICS:

To determine the rate constant of hydrolysis of methyl acetate catalysed by an acid also the activation energy.

AIM: To determine the rate constant of hydrolysis of methyl acetate catalysed by two different concentrations of acids.

APPARATUS: - Conical Flask, Beakers, Reagent bottles, Burette, Pipette.

CHEMICALS: - Methyl acetate, Hydrochloric acid (1M, 2M)
Oxalic acid solution (0.2N), NaOH, Phenol phthalein indicator

<u>Preparation of Slandered oxalic acid solution (0.2N)</u>

About 3.15 g of oxalic acid di hydrate is weight accurately and dissolved in small amount of distilled water and made up to the mark in 250ml of volumetric flask.

$$N = \frac{W}{E2.Wt} \times \frac{1000}{250}$$

Principle: - The hydrolysis of methyl acetate in presence of an acid may be represented as

$$CH_3COOCH_3 + H_2o \rightarrow CH_3COOH + CH_3OH$$
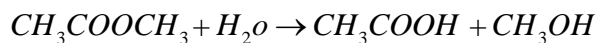
The rate of reaction is given by

$$\frac{dx}{dt} = \frac{2.303}{t} \log\left(\frac{a}{a-x}\right) \rightarrow (First\ Order)$$

As acetic acid is producing during the reaction, the reaction may be studied by the titration of unknown concentration of reaction mixture with a standard alkali at suitable of time.

Standardization of NaOH Solution-

20 ml of Oxalic acid is pipette out into a conical flask and is diluted to 30 ml with distilled water. To the solution two drops of phenolphthalein indicator is added and then titrated against sodium hydroxide taken in a burette. The cold point is colorless to pale pink. The experiment is repeated till concurrent readings are obtained.

PROCEDURE:

From standardized 1N and 2N HCl , p0 ml of IN HCl taken in a iodination flask and 10 ml of pure methyl acetate is taken a test tube. Both the iodination flask and test tubes are placed in water bath to bring them into room temperature. Measure exactly 5 ml of methyl acetate and add to 90 ml 1N HCl in a iodination flask. When exactly half of the amount in the pipette is been discharged, the stop watch is started. Mixture is shaken well and maintain at a constant temperature. Now titrated with NaOH solution + using phenolphthalein indicator. The volume of alkali required for titration is noted and is taken as "V_O". Similarly 10 ml of reaction mixture is taken out in successive intervals of time 10, 20, 30, 40, 50 minutes. To obtain compute hydrolysis the reaction mixture kept in a water bath maintained at for about half an hour.

After the hydrolysis completed and it is cooled at room temperature and titrated the 10 ml of reaction mixture with NaOH solution and is taken as "V_∞".

Method of Calculation:-

The initial volume of titrate; NaOH is V_O at T_0 ^{0}C is proporsonal to the amount of HCl present in the reaction mixture at Zero time. When there is an acetic acid present as the hydrolysis proceeds $V_\infty - V_0$ directly proportional to initial concentration of ester (a), at successive intervals of time $V_\infty - V_t$ is proportional the concentration of esters at time 't'(a-x).

$$K_1 = \frac{2.803}{t} 10g \left(\frac{V_\infty - V_O}{V_\infty - V_t} \right) 80C^{-1}$$

Same procedure and calculation is repeated for 2N HCl solution.

Relative strength of two acids is given by $\dfrac{K_2}{K_1} =$

Calculation of Activation Energy:-

$$\log \frac{K_2}{K_1} = \frac{Ea}{2.303 \times R} \times \frac{T_2 - T_1}{T_2}.$$

$$Ea = 10g \frac{K_2}{K_1} \times 2.3.3 \times R \times \frac{T_2}{T_2 - T_1}$$

Result:-Rate Constant for hydrolysis of methyl acetate by 1N HCl are $K_2 =$ ------, $K_2 =$ ----

Activation Energy $Ea =$ --------------------

Observation Table:-

Table-1   Standardization of NaOH:-

| Sl.No | Vol of Oxalic acid (V1M1) | Burette Readings | | Vol of NaOH (V2) |
		Initial	Final	V2= F-I
1	20			
2	20			
3	20			

Vol of Oxalic acid Solution V_1 = 20 ml

Normality of Oxalic Acid N1 = 0.2 N

Vol of NaOH Solution V2 = ---- M1

Normality of NaOH N2= -----N

$N_1V_1 = N_2V_2 \Rightarrow N2 = \dfrac{N_1V_1}{V_2} \rightarrow$

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

Calculations for determine rate constants

For 1N Hcl

Sl.No	Time (t) Minutes	Vol of NaOH (Vt)	$V_\alpha - V_t$	$\dfrac{V_\alpha - V_0}{V_\alpha - V_t}$	$10g\dfrac{V_\alpha - V_0}{V_\alpha - V_t}$	$K_1 = \dfrac{2.303}{t}\log\dfrac{V_\alpha - V_0}{V_\alpha - V_t}$
1	0					
2	10					
3	20					
4	30					
5	40					
6	50					
7	60					
8	$V\alpha$					
9						

Average K_1 =

$V_0 = -- ml$ GRAPH:

$V_\alpha = ---$

$V_\alpha - V_0 = ----ml$

From Graph

$Slope = \dfrac{y2 - y1}{x2 - x1} = \underline{\qquad}$

$Slope = \dfrac{K1}{2.303}$

$\Rightarrow K1 = 2.303 \times Slope$

For 2N HCl:

Sl.No	Time (t) Minutes	Vol of NaOH	$V_\alpha - V_t$	$\dfrac{V_\alpha - V_0}{V_\alpha - V_t}$	$10g\dfrac{V_\alpha - V_0}{V_\alpha - V_t}$	$K_2 = \dfrac{2.303}{t}\log\dfrac{V_\alpha - V_0}{V_\alpha - V_t}$

		(Vt)				
1	0					
2	10					
3	20					
4	30					
5	40					
6	50					
7	60					
8	$V\alpha$					
9						

Average K_2 =

$V_0 = -- ml$ GRAPH:

$V_\alpha = ---$

$V_\alpha - V_0 = ---- ml$

$$Slope = \frac{y2 - y1}{x2 - x1} = \underline{\hspace{2cm}}$$

From Graph $Slope = \dfrac{K_2}{2.303}$

$$\Rightarrow K_2 = 2.303 \times Slope$$

EXPERIMENT -12

AIM: - Preparation of Thiokol Rubber

Operators: - Beakers, Glass Rod, Funnel, Filter Paper

**Chemicals:** - Ethylene Dichloride, $Na_2 S_2$ (Sodium Ploy Sulphide)

Procedure: -

1. Place about 10 ml. of Ethylene dichloride solution in 100 m). beaker
2. Add about 5 gr. of sodium polysulphide and Heated to form poly sulphide rubber (solid)
3. Wash the above solid with water and dry it in the folds of filter paper
4. Calculate the yield of the product

Result:

The yield of Thiokol Rubber is _____ gr.

CHEMICAL EQUATION:

$$nCl\text{-}CH_2\text{-}CH_2\text{-}Cl + Na_2S_2 \xrightarrow{\textit{Poly'n}} \text{-----}[CH_2 - CH_2\text{-}S\text{-}S\text{-}]_n$$
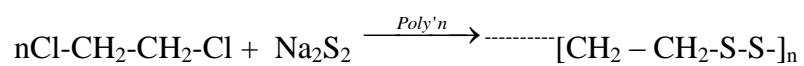
(Ethylene Chloride) (Sodium Poly Sulphide) (Poly Sulphide Rubber (or) Thiokol)