VEMU INSTITUTE OF TECHNOLOGY

P.KOTHAKOTA, CHITTOOR DIST – 517 112

DEPARTMENT OF HUMANITIES & SCIENCES

CHEMISTRY

LABORATORY MANUAL FOR I-Year B.Tech
(CSE)

DEPARTMENT OF HUMANITIES & SCIENCES

CHEMISTRY LAB MANUAL

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Name	
Register No.	
Branch/Section	
Academic year	

SYLLABUS

(CSE)

CHEMISTRY LABORATORY: EXPERIMENTS

1. Estimation of Ferrous iron by Dichrometry
2. Conductometric titration of i) strong acid Vs. strong base ii) Weak acid Vs. Strong base.
3. Preparation of a polymer (Thiokol Rubber)
4. Verification of Beer-Lambert's law
5. Potentiometry- Determination of Redox potentials and emfs
6. Determination of cell constant and conductance of solutions
7. Thin layer Chromatography
8. Determination of strength of an acid in Pb-acid battery
9. Identification of simple organic compounds by IR and NMR.
10. HPLC method in separation of gaseous and liquid mixtures.

CONTENTS

CHEMISTRY LAB

Exp:1 **Date:**

ESTIMATION OF FERROUS IRON BY DICHROMETRY

AIM: To estimate the amount of ferrous iron present in the solution with the help of standard solution of potassium dichromate.

APPARATUS REQUIRED:

1. Beaker
2. Burette
3. Pipette
4. Conical Flask
5. Volumetric Flask

CHEMICALS REQUIRED:

1. $K_2Cr_2O_7$
2. Diphenylamine
3. Conc. H_2SO_4
4. Ferrous ammonium sulphate
5. Distilled water

PRINCIPLE:

Ferrous iron is oxidized to ferric iron by potassium dichromate in presence of acid solution. The completion of the oxidation of reaction is marked by the appearance of blue violet color of the diphenylamine which is used as an internal indicator.

$$K_2Cr_2O_7 + 4H_2SO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3(O)$$

$$3[2FeSO_4 + H_2SO_4 + (O) \rightarrow Fe_2(SO_4)_3 + H_2O]$$

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

The equivalent weight of iron is its atomic weight i.e.55.86 since one equivalent of potassium dichromate oxidizes one equivalent of iron.

PREPERATION OF CHEMICALS:

1. **Preparation of standard potassium dichromate:** weigh out accurately about 0.49gms of potassium dichromate into a100 ml standard flask and dissolve the solid in a small quantity of distilled water. Make up the resulting solution with distilled water up to the mark and shake the flask well for uniform concentration.
2. **Preparation of acid mixture:** Mix up 100 ml of phosphoric with 300ml of concentrated H2S04 in a reagent bottle and stopper it.
3. **Preparation of diphenylamine:** Dissolve 1gm of diphenylamine in 100ml of concentrated H2S04.

PROCEDURE:

- Rinse and fill the burette with standard $K_2Cr_2O_7$ solution.
- Pipette out 20ml of ferrous solution into a 250ml conical flask and add 5ml of acid mixture and 2drops of diphenylamine indicator.
- Titrate the solution against potassium dichromate taken in the burette till blue violet color is obtained as end point.
- Repeat the titration to get concurrent values.

RESULT: Amount of ferrous iron present in the given solution = ……..gms/100ml.

OBSERVATIONS:

S.No	Volume of Ferrous Solution (ml) V_2	Burette reading		Volume of $K_2Cr_2O_7$ solution (ml) V_1
		Initial	Final	
1				
2				
3				

CALCULATIONS:

Normality of $K_2Cr_2O_7$ $= N_1 =$ N

Volume of $K_2Cr_2O_7$ $= V_1 =$ ml

Normality of ferrous iron $= N_2 =$ N

Volume of ferrous iron $= V_2 =$ ml

$$N_1V_1 = N_2V_2$$

$$N_2 = \frac{N_1V_1}{V_2} =$$

Amount of iron (II) present in 100 ml of the given solution= $\frac{N_2 X\ 55.5}{10}$ =

RESULT: Amount of ferrous iron present in the given solution = ……..gms/100ml.

Exp:2 **Date:**

CONDUCTOMETRIC TITRATION OF i) STRONG ACID Vs STRONG BASE

AIM:- To determine the strength of given strong acid (HCl) solution by titrating it against standard base (NaOH) solution conductometrically.

APPARAUTS REQUIRED:-

1. Beaker
2. Pipette
3. Burette
4. Conductometer
5. Conductivity cell

CHEMICALS REQUIRED:-

1. Standard NaOH solution
2. HCl solution

PRINCIPLE:

According to Kohlrausch's law, electrical conductivity of a solution depends on the number of mobile ions present in it. In the titration of strong acid HCl with strong base NaOH solution, before addition of basic solution there will be high conductance due to presence of large number of mobile ions (H^+). Gradual addition of NaOH solution decreases the conductance due to combination of H^+ and OH^- ions of the base to form undissociated water molecules. The conductance of the solution decreases till neutralization point and increases quickly after neutralization due to free Na^+ and OH^- of the added excess NaOH solution.

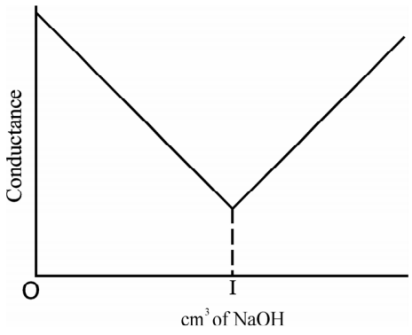
$$HCl + NaOH \rightarrow NaCl + H_2O$$
$$H^+ + Cl^- + Na^+ + OH^- \rightarrow Na^+ + Cl^- + H_2O$$

PROCEDURE:

- Take 20 ml of given HCl solution in a beaker. Dilute the solution so that conductivity cell dips in the solution.
- Wash the conductivity cell with distilled water and connect it to Conductometer.
- Dip the cell in acetic acid and find out the conductance.
- From the burette, mix 0.5 ml an of NaOH solution into HCl solution with shaking and note down the observed conductance.
- Repeat the above procedure on recording the observed conductance.
- Plot the graph between the observed conductance and volume of NaOH mixed and find out the volume of NaOH required complete neutralization.

GRAPH:

Plat a graph between measured conductance vs. volume of base added. The intersection of two straight lines gives the end point. Calculate the strength of given acid (HCl) from the known strength of given strong base (NaOH).



OBSERVATIONS:

S.No	Volume NaOH added (ml)	Observed conductance (Siemens)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		

CALCULATIONS:

Normality of HCl = N_1 = N

Volume of HCl = V_1 = ml

Normality of NaOH = N_2 = N

Volume of NaOH = V_2= ml

$$N_1V_1 = N_2V_2$$

$$N_2 = \frac{\mathbf{N_IV_I}}{\mathbf{V_2}} =$$

RESULT: The strength of unknown HCl sample is _____N

II) CONDUCTOMETRIC TITRATION OF (II) WEAK ACID VS. STRONG BASE

AIM:- To determine the strength of given weak acid (CH_3COOH) solution by titrating it against standard base (NaOH) solution conductometrically.

APPARAUTS REQUIRED:-

1. Beaker
2. Pipette
3. Burette
4. Conductometer
5. Conductivity cell

CHEMICALS REQUIRED:-

1. Standard NaOH solution
2. CH_3COOH solution

PRINCIPLE:-

The conductance of the acid initially is very low because of low ionization of weak acetic acid. With the addition of more and more NaOH, the conductance keeps on increasing, as the number of ions in solution increasing. But the increase is slow due to low mobility of CH_3COO^- ions. After the complete neutralization of CH_3COOH, further addition of NaOH results in increase in the conductance of the solution due to increase in number of high mobile Na^+ and OH^- ions.

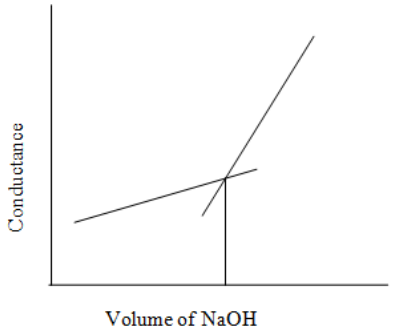
$$CH_3COOH + NaOH \rightarrow CH_3COO^- + Na^+ + H_2O$$

In case of weak acid (like CH_3COOH) against strong base (NaOH), the curve shape is found different. The point of intersection tells the exact volume of NaOH used for complete neutralization.

PROCEDURE:-

- Take 20 ml of given CH_3COOH solution in a beaker. Dilute the solution so that conductivity cell dips in the solution.
- Wash the conductivity cell with distilled water and connect it to Conductometer.
- Dip the cell in acetic acid and find out the conductance.
- From the burette, mix 0.5 ml an of NaOH solution into CH_3COOH solution with shaking and note down the observed conductance.
- Repeat the above procedure on recording the observed conductance.
- Plot the graph between the observed conductance and volume of NaOH mixed and find out the volume of NaOH required complete neutralization.

GRAPH:



OBSERVATION:-

S.No	Volume NaOH added (ml)	Observed conductance (Siemens)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		

CALCULATIONS:

Normality of CH_3COOH $= N_1 =$ N

Volume of CH_3COOH $= V_1 =$ ml

Normality of NaOH $= N_2 =$ N

Volume of NaOH $= V_2 =$ ml

$$N_1V_1 = N_2V_2$$

$$N_2 = \frac{N_IV_I}{V_2} =$$

RESULT: The strength of unknown CH_3COOH sample is _____N

Exp:3 **Date:**

PREPARATION OF THIOKOL RUBBER

AIM: To prepare Thiokol rubber

CHEMICALS REQUIRED:

1. Sodium Hydroxide
2. Ethylene Dichloride
3. Sulphur

APPARATUS REQUIRED:

1. Hot Plate
2. Beaker
3. Funnel
4. Forceps

PRINCIPLE:

Thiokol rubber is prepared by the condensation polymerization between sodiumpolysulphide and 1,2-Dichloroethane. Sodiumpolysulphide is prepared by adding Sulphur to NaOH at boiling point.

$$NaOH + 2S \longrightarrow Na_2S_2$$

$$CH_2Cl\text{-}CH_2Cl + Na_2S_2 \longrightarrow [\text{-}S_2CH_2\text{-}CH_2S_2\text{-}]_n$$

PROCEDURE:

- Dissolve 3.0 gm of NaOH in 50 ml of distilled water and heat the solution to the boiling point. Place a stirrer rod in the solution to prevent bumping.
- Add 6 gm of sulphur to NaOH solution and stirr until all the sulphur has dissolved. The solution will turn from light yellow to dark brown when complete sodium polysulphide is formed.
- After 5 minutes, allow the solution to cool and decant the dark brown liquid from undissolved sulphur. If much of the sulphur remains undissolved it can be more effectively removed by filtration through filter paper.
- Add 15ml of ethylene dichloride (1,2- Ethylene dichloride) to the solution and warm the mixture up to 70^0C with continuous stirring, while stirring a rubbery polymer will be formed at the interface between the two immiscible liquids and will collect as lump at the bottom of the beaker.
- Wash the product under tap water and dry within the folds of filter paper.
- The yield will be 2.2 gms

PRECAUTIONS:

1. Wear the lab coat and goggles while working in the lab. Rubber Gloves should be used while performing this experiment.
2. Handle ethylene dichloride with high care as it is a strong irritant of eyes and skin.
3. Sulfur can catch fire easily in powdered form and it also irritates the skin and nose hence care must be taken while using it.

RESULT: Thiokol rubber is prepared with the given reagents.

Exp:4 **Date:**

VERIFICATION OF BEER-LAMBERT'S LAW

AIM: To verify Beer-Lambert's law

CHEMICALS REQUIRED:

1. $KMnO_4$
2. Ferrous Ammonium Sulphate
3. Sulphuric acid
4. Distilled water

APPARATUS REQUIRED

1. Colorimeter
2. Cuvettes
3. 25 ml Standard Flasks

PRICIPLE: Beer and Lambert Law states that, when a monochromatic light passes through a transparent medium, the rate of decrease in light intensity with the concentration and thickness of the medium is directly proportional to the intensity of the light.

The relation between absorbance A, the transmittance T and the molar absorption coefficient (ε) is given

$$OD = \varepsilon bc = \log (I_0/I)$$

$$T= I/I_0,\ A= -\log T = -\log (I/I_0)$$

Where I_0 = Intensity of incident light

I = Intensity of transmitted light

ε= Molar absorption coefficient

c = Concentration in mol L^{-1}

b = Path length of absorbing solution in cm^{-1}

The above is the fundamental equation of colorimetry& spectrophotometry, and is often termed as the Beer-Lambert's Law. The Conditions for this Law to be applicable are:

1) Solution should be colored.
2) Incident radiation should be monochromatic.
3) Solution should be homogeneous
4) Solution should be dilute.
5) Each molecular or ion species should absorb independently.

PROCEDURE

PART A: PREPARATION OF STD. FERROUS AMMONIUM SULPHATE SOLUTION:

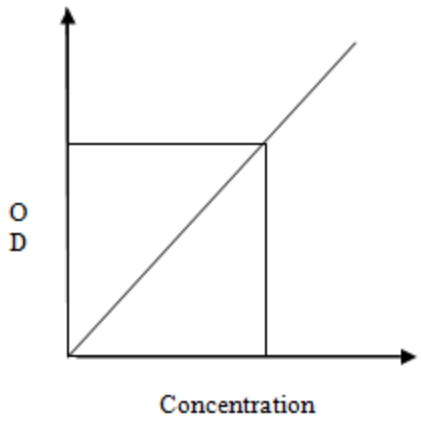
- Weigh accurately about 0.98gm of Mohr's salt and transfer it into a clean 100 ml flask using funnel.
- Dissolve it in distilled water, add-10 ml dil H_2SO_4 and make the solution homogeneous up to the mark.

PART B: STANDARDIZATION OF $KMnO_4$ USING MOHR'S SALT:

- Rinse and Fill the burette with $KMnO_4$ solution.
- Pipette out 20 ml of Mohr's salt solution in a clean conical flask.
- Add 2 ml of dil H_2SO_4 and titrate the contents of flask with $KMnO_4$ till a light pink color appears.
- From titration data, calculate the Normality of $KMnO_4$ solution.

PART C: ESTIMATION OF $KMnO_4$ BY COLORIMETRY & VERIFICATION OF BEER-LAMBERTS LAW:

- Take 10ml of KMnO4 in to a standard volumetric flask and dilute it up to 100 ml. Make up the volume up to the mark to get a homogenous stock solution.
- Pipette out 1 to 10 ml of stock solution in to 10 test tubes respectively.
- Make up the solution up to 10ml with distilled water. (Dilution is done as given in Table 2.of the experiment.)
- Calibrate the colorimeter with blank solution.
- Measure the OD of stock solution for each filter from No. 45 to 67.
- Select the filter No. which gives maximum OD. (For $KMnO_4$, the filter No.52 gives maximum OD.
- Fix the selected filter no for $KMnO_4$.
- Calibrate the colorimeter by taking distilled water blank and adjust OD to zero.
- Fill the cuvette with each of the ten sample solutions prepared, one after the other and measure the OD.
- Plot a graph between optical densities against concentration of $KMnO_4$. It gives a straight line passing through origin. It is called calibration graph.
- The graph passing through origin is a proof for verification of Beer-Lambert's Law.
- This graph can be used for estimation of the concentration of a given species ($KMnO_4$ in this case) in any Test solution.
- Molarity of test solution can be calculated using calibration graph.



CALCULATIONS:

Part A: Preparation of Std. Ferrous Ammonium Sulphate Solution:

Weight of weighing bottle + FAS =W_1= gms

Weight of the empty bottle = W_2 = gms

Wt. of FAS = W_1-W_2 = gms

Part B: Standardization of $KMnO_4$ solution.

S.No	Volume of Ferrous ion solution (V_1 ml)	Burette readings		Volume $KMnO_4$ solution (V_2 ml)
		Initial	Final	
1				
2				
3				
4				

Normality of FAS = N_1 = N

Volume of FAS = V_1 = ml

Normality of $KMnO_4$ = N_2 = N

Volume of $KMnO_4$ = V_2= ml

$$N_1V_1 = N_2V_2$$

$$N_2 = \frac{N_IV_I}{V_2} =$$

Normality of $KMnO_4$ solution N_2=

Part C: Verification Beer-Lambert law

S.No	Filter No.	OD
1		
2		
3		
4		
5		
6		

S.No	**Volume of Std. $KMnO_4$ sol. + Distilled Water**	**Concentration**	**Optical Density**
1	1+9		
2	2+8		
3	3+7		
4	4+6		
5	5+5		
6	6+4		
7	7+3		
8	8+2		
9	9+1		
10	10+0		
11	Test Solutions		

Result: Beer-Lambert's law was verified and the Concentration of unknown $KMnO_4$ solution is

_____N

Exp:5 **Date:**

Determination of Redox Potential using Potentiometry

Aim: Determination of Redox Potential of the given sample using Potentiometry.

Apparatus Required:

1. Saturated Calomel Electrode
2. Platinum Electrode
3. Potentiometer
4. Beakers
5. Pipette
6. Stirrer
7. Salt Bridge

Chemicals Required:

1. Ferrous ammonium sulphate solution
2. Potassium permanganate solution

PRINCIPLE:

The reference electrode used here is saturated calomel electrode (SCE). It consists of mercury metal covered with a paste of Hg + Hg_2Cl_2 in contact with saturated KCl solution and Pt wire for electrical contact. The reduction potential of this electrode is 0.242V. This saturated calomel electrode functions as anode. The Indicator electrode is a platinum electrode which responds rapidly to oxidation- reduction couples and senses the potential which depends upon the concentration ratio of the reactants & products of redox reactions. Here, the Pt electrode is in contact with a Ferrous-Ferric couple. This electrode functions as cathode.

Cell Representation: (-) Pt/ Hg(1), Hg_2 Cl_2(s) /KCl(salt) //Fe^{3+}, Fe^{2+} /Pt (+)

Cell Reaction: Anode: - 2 Hg + $2CI^-$ → Hg_2Cl_2 + $2e^-$

Cathode: - $2Fe^{+3}$ + 2e- → $2Fe^{+2}$

Cell e.m.f.: Ecell = $E^o_{(Fe3+ / Fe2+)}$ + (2.303RT/ F) log $\frac{Fe3+}{Fe2+}$ - ESCE

The cell potential is measured during the course of reaction and graphs are plotted. From the graphs, end point of the titration is located and concentration is calculated.

PROCEDURE: PART-A: PREPARATION OF STANDARD F.A.S. SOLUTION:

- Weigh the given 0.98 gms of Mohr's salt (F.A.S.) accurately in to a clean weighing bottle and transfer it into a clean 100 ml standard flask through a funnel.
- Dissolve it in 10 ml of dil. H_2SO_4 and make up the solution up to the mark with distilled water.

- Shake the solution thoroughly to make it homogeneous. From the weight of FAS, calculate the Normality of Standard solution.

PART-B: Standardization of $KMnO_4$ solution:

- Rinse and fill the burette with $KMnO_4$ solution.
- Take 20ml of the prepared standard FAS solution into a clean conical flask.
- Add 10 ml of dilute H_2SO_4 (6N) to provide acidic medium.
- Titrate the solution against $KMnO_4$ taken in burette until the solution acquires pale pink colour which persists for at least a minute as end point. Note the titre value.
- Repeat the process till concordant titre values are obtained.
- Calculate the normality of $KMnO_4$ solution

PART C: Estimation of Fe^{+2} in the given test solution:

- The burette is washed with water and rinsed and filled with given $KMnO_4$ solution upto zero mark.
- Take 20ml of the prepared standard FAS solution into a beaker and add 10 ml of H_2SO_4 to it.
- A platinum electrode is dipped into the solution. This electrode is then coupled with a saturated calomel electrode and the cell is introduced into Potentiometric circuit.
- The two solutions are connected by means of salt bridge to form the Galvanic cell

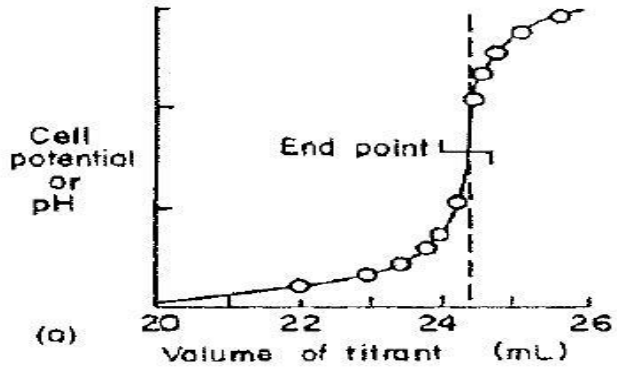
(-) Pt/ Hg(1), Hg_2Cl_2(s) /KCl(sat) //Fe^{3+}, Fe^{2+} /Pt (+)

- Add $KMnO_4$ from burette in 1 ml portions to the ferrous solution, stir it and note the EMF.
- Continue the titration till a sudden inflection in EMF occurs. Then take about 6 to 8 readings after inflexion in 1 ml intervals.
- From the titrations approximate volume of $KMnO_4$ required is found out.
- The titration is repeated with addition of $KMnO_4$ in 0.1 ml.

GRAPH:1

Draw a graph of E_{cell} Vs volume of KMnO4 added; the inflexion point gives an approximate equivalence point.

Model Graph:



CALCULATIONS:

Part A: Preparation of Std. Ferrous Ammonium Sulphate Solution:

weight of bottle + FAS =W_1= ---- g.

Weight of empty weighing bottle = W_2 =----------g.

Wt. of FAS = W_1-W_2 =

$N_{(std)}$ FAS = $\frac{Wt}{eq.wt}$ X$\frac{1000}{V}$ =---

Part B: **Standardization of FAS_4 solution.**

Titration of Std. FAS. Vs. $KMnO_4$

S. No.	Volume of FAS (V_2 ml)	Burette Reading		V_{KMnO4} (V_2) ml
1		Initial	Final	
2				
3				
4				

Normality of $KMnO_4$ = N_1 = N

Volume of $KMnO_4$ = V_1 = ml

Normality of FAS = N_2 = N

Volume of FAS = V_2= ml

$$N_1V_1 = N_2V_2$$

$$N_2 = \frac{\mathbf{N_IV_I}}{\mathbf{V_2}} =$$

PART-C: Estimation of Fe^{+2} in the given test solution:

Table. 1

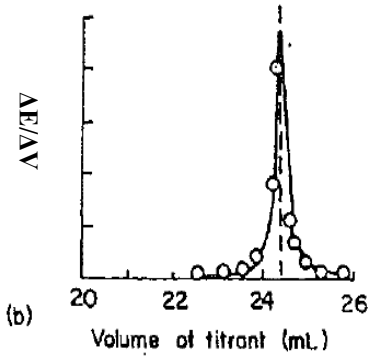
S.No.	Vol of $KMnO_4$ added (ml)	E_{cell} (mv)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		

Table. 2

S.No	Volume of $KMnO_4$(ml)	E_{cell} (mv)	ΔE	ΔE/ ΔV
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				`
13				
14				
15				

GRAPH.2

Differential graph is drawn by plotting $\Delta E/\Delta V$ (Y-axis) Vs volume of KMnO4 (X-axis) to get a sharp peak, which corresponds to the precise equivalence point of titration.



RESULT:

The amount of ferrous iron present in 1000 ml of the solution = ----------NX55.85 g/L

The amount of ferrous iron present in 100 ml of the given solution = --------NX55.85X$\frac{100}{1000}$

= ----------gms

Exp:6 **Date:**

DETERMINATION OF CELL CONSTANT AND CONDUCTANCE OF SOLUTIONS

AIM: Measurements of the cell constant and determination of the conductance of deionized and tap water, the conductance of an electrolyte as a function of concentration for strong electrolyte: HCl or KNO_3

Chemicals Required

1. 0.02 N KCl (specific conductance, κ, at 25°C: 0.002767 $ohm^{-1}cm^{-1}$)
2. 0.02 N HCl
3. Succinic acid $(HOOC)_2(CH_2)_2$ (Although it is a dibasic acid, it is considered a weak electrolyte with an ionization constant $K_1 = 6.4 \times 10^{-5}$ at 25°C).

APPARATUSREQUIRED

1. Conductance cell
2. Beckman Conductivity Bridge
3. Barnstead Water Purity Meter

PROCEDURE

1. Read the cell constant, x, for the cell used in your experiments from the conductometer. $x = \kappa R$ where κ is the conductivity and R is the resistance of Conductometer by immersing the conductivity cell into a beaker, filled with deionized (distilled) water. Set the knob to red line and adjust the scale.
2. Rinse the conductivity cell with 0.02 M KCl, then fill a beaker with 100 ml of this solution and immerse the conductivity cell.
3. Keep the conductivity cell steady in the solution and adjust the knob to "conductance". Read and record the value from the upper scale (in units of μmhos).
4. Take 25 ml of the 0.02 M KCl solution into a beaker add 75 ml distilled water. Repeat steps 2-3 with this solution.
5. Prepare 0.02 M KCl solutions at 1/16 and 1/64 dilutions in the same way explained in step 4 and repeat steps 2-3 with these solutions. Serial dilutions are strongly encouraged. Repeat steps 5-6 with solutions of 0.02 M Potassium acitate (KAc), 0.02 M HCl and 0.05 M Acetic acid (HAc).

DATA SHEET:

KCl	µS/cm	HCl	µS/cm	KAc	µS/cm	HAc	µS/cm	H2O	µS/cm
0.02 M		0.02 M		0.02 M		0.02 M		0.02 M	
1/4		1/4		1/4		1/4		1/4	
1/16		1/16		1/16		1/16		1/16	
1/64		1/64		1/64		1/64		1/64	

Exp:7 **Date:**

THIN LAYER CHROMATOGRAPHY

AIM:- To separate green leaf pigments by thin layer chromatography and determine their R_f values.

CHEMICAL REQUIRED:-

1. Chloroform
2. Acetone
3. Benzene
4. Silica gel
5. Spinach leaves

APPARATUS REQUIRED:-

1. Glass plates
2. Wide mouthed bottles with stoppers
3. Glass jars with lids
4. Tongs
5. Capillary tubes
6. Beaker
7. Filter paper

THEORY:-

In thin layer chromatography where the stationary phase is a polar adsorbent and the mobile phase can be a single solvent or combination of solvents. A thin layer of adsorbent usually silica gel or activated alumina on a smooth surface is used as a stationary phase and chromatogram is developed by upward capillary movement of the solvent through the thin layer of adsorbent.

PRINCIPLE:-

The different components will have different solubility and different strengths of adsorption to the adsorbent. When the components are placed on a thin layer chromatography gel on smooth surfaces, some components will be carried farther up on gel than others depending on their adsorption capacity. Based on the distance travelled by the components, various components can be separated and identified using thin layer Chromatography. The compounds that are separated can be clearly visualized if the compounds are coloured. Various compounds on the developed TLC plates can be identified through their R_f values. R_f stands for Retention factor or Ratio of Fronts.

R_f = Distance travelled by the compound/ Distance travelled by solvent

PROCEDURE:-

1. Prepare the adsorbent slurry by mixing about 30 g of silica gel in 100 ml of chloroform in a wide mouth bottle with constant swirling motion; the bottles should be tightly stopper.

2. Thin layer of adsorbent can be prepared by holding the two cleaned and dried glass plates together with tong, dipping them in the slurry of adsorbent and removing the plates quickly.
3. Do not dip the top and end of the plates.
4. Now allow the slurry to drain by holding the top edges, separate the two plates and allow them to dry in air by placing these plates on a filter paper with slurry side up wards. These plates are known as chromatographic plates.
5. Prepare the 'extract of leaves' by dipping few crushed leaves of spinach in a little quantity of alcohol for 30 minutes in a beaker stirring with a glass rod the contents are filtered and the filtrate is taken as the 'extract of leaves'.
6. Apply a drop of leaf extract in the center of the chromatographic plate with the help of a capillary tube. Allow the drop dry in air.
7. Place the glass plate in chromatography jar in such a way that it does not touch the sides of the jar.
8. Measure the distance travelled by the spots and the developing agent.
9. Calculate the R_f values of the spots corresponding to different components.

By using the relation

$$R_f = \frac{\text{Distance travelled by the component}}{\text{Distance travelled by the solvent}}$$

PRECAUTIONS:-

- The glass plate used must be thoroughly clean and dry.
- Fine capillary tube should be used for applying a spot of solution.
- The glass plate should be kept erect.
- During the experiment, keep the glass jar always covered and undisturbed.
- The slurry bottle must be tightly Stoppard.
- The spots of the solution must not dip in the developing solvent.

RESULT:-

Distance travelled by component 1 = m

Distance travelled by component 2 = m

Distance travelled by component 3 = m

Distance travelled by developing liquid = m

Rf value of component 1 =

Rf value of component 2 =

Rf value of component 3 =

Exp:8 **Date:**

DETERMINATION OF STRENGTH OF AN ACID IN Pb-ACID BATTERY

AIM: To determine the concentration of a sulfuric acid solution in Pb acid battery by titration with standard NaOH solution.

CHEMICALS REQUIRED:

1. H_2SO_4
2. NaOH,
3. Phenolphthalein indicator

APPARATUS Required:

1. Beaker,
2. Burette,
3. Pipette,
4. Conical Flask,
5. Volumetric Flask.

PRINCIPLE:

Titration is the process, of determining the concentration of a substance in solution (the analyte) by adding to it a standard reagent of known concentration (the titrant) in carefully measured amounts until a reaction of definite and known proportion is completed, as shown by a color change or electrical measurement, and then calculating the unknown concentration.

In this experiment, sulfuric acid is titrated with sodium hydroxide.

$$2\ NaOH + H_2SO_4 \rightarrow Na_2SO_4 + 2\ H_2O$$

The titration is done in the presence of phenolphthalein indicator that is colorless in acid solution but turns pink in basic solution. At the equivalence point, all of the analyte has reacted, and only a tiny excess of titrant has been added, just enough to change the color of the indicator.

Procedure:

1.Pipette out 20 ml of given Sulphuric acid into clean conical flask.

2.Add 2 drops of phenolphthalein indicator to flask.

3.Fill the burette with sodium hydroxide solution, and then clamp the burette to the ring stand.

4.Read the initial volume on the burette and record the value. Titrate the sulfuric acid against standard NaOH solution, till pink colour appears. Note down the volume (V_1)

5.Repeat the titration for concurrent readings.

Calculations

Titration of Std NaOH solution against H_2SO_4

S.No	**Volume of H_2SO_4 (V_2) ml**	**Burette Readings**		**Volume of H_2SO_4 consumed (V_1) ml**
		Inial	Final	
1				
2				
3				
4				

Normality of NaOH = N_1 = N

Volume of NaOH = V_1 = ml

Normality of H_2SO_4 = N_2 = N

Volume of H_2SO_4 = V_2= ml

$$N_1V_1 = N_2V_2$$

$$N_2 = \frac{N_IV_I}{V_2} =$$

RESULT: Strength of Sulphuric acid in lead acid battery is =_____

Exp: 9 **Date:**

IDENTIFICATION OF SIMPLE ORGANIC COMPOUNDS BY IR & NMR

AIM: To identify a simple organic compound by IR & NMR spectroscopy.

PRINCIPLE: In order to determine the structure of organic molecules on the basis of their spectra all the structural information available in the spectra is to be used.

IDENTIFICATION BY IR SPECTRUM:

1. Determination of nature of carbon skeleton (aliphatic/aromatic):

(i) C-H stretching: The =C-H stretch in aromatics is observed at 3100-3000$cm^{-1.}$ where as –C-H stretching frequencies for standard aliphatic hydrocarbons is below 3000cm^{-1}.

(ii) C-C ring sreching vibrations: The aromatic hydrocarbons show C-C stretching vibrations in the regions 1600-1585cm^{-1} and 1500-1400cm^{-1}.

(iii) Out of plane C-H bending vibrations: These are observed in the region 900-675cm^{-1} and provide information about the substitution pattern of aromatic compounds

Thus a weak absorption in the region 3080-3030cm^{-1} accompanied by medium absorption in the ring vibration region indicates the presence of an aromatic ring. A signal around 1605 cm^{-1} is quite a good indicator of an aromatic molecule, occasionally splits into a doublet. The out of plane bending vibrations are also very important. A lack of strong absorption band in the 900-650 cm^{-1} region generally indicates a non aromatic structure.

(B)DETERMINATION OF CHARACTERISTIC FREQUENCIES OF FUNCTIONAL GROUPS:

(i) The C=C bond usually gives rise to a moderate band in the region 1680 – 1640 cm^{-1}

(ii) The – C = C – Stretch appears as a weak band from 2260 – 2100 cm^{-1}

(iii)The bending vibration of the = C - H group are observed in the 1000 – 650 cm^{-1} region.

(iv)The terminal – C = C – H stretch is observed as a strong, narrow band in the range 3330 – 3270 cm^{-1}

(v)The – C = C – H bending vibration is observed in 700 – 600 cm^{-1}

NMR SPECTRUM:

(i)A combination of two proton quartet and a three proton triplet are suggestive of an ethyl group. Similarly, a six proton doublet and a one proton septet (or) multiplet is characteristic of an isopropyl group.

(ii)The broadened signels in the spectrum indicate towards the presence of – OH (or) – NH protons.

(iii)The absorptions in the range of 7-8 ppm suggest the presence of an aromatic ring; Benzene absorbs at 7.27ppm. The aromatic absorptions are further downfield than -7.27, indicate the presence of electron withdrawing substituent.

(iv) The absorptions in the region of = 2.1 to = 2.5 are indicative of the protons adjacent to a carbonyl group (or) an aromatic ring.

EXP -10 **Date:**

HPLC METHOD IN SEPERATION OF GASES AND LIQUID MIXTURES

Aim: To separate gaseous and liquid mixtures by using HPLC method

Principle: Some molecules take longer time than other to pass through Chromatography column. This depends on the affinity of the molecules with the mobile phase (liquid (or) gas) and stationary phase (solid (or) liquid). HPLC is used in various industries to analyze different products by their separation into its compound.

Procedure: HPLC Stands for High Performance Liquid Chromatography. This is an analytical chemistry technique. A liquid mixture (first phase) is pumped through a column that contain a packing of small porous particles with a second phase bound to the surface. The different solubility of the sample components in the two phases because the components to move through the column with different average velocities, thus creating a separation of these components.

The pumped solution is called mobile phase, while the phase in the column is called the stationary phase.

There are several modes of liquid Chromatography depending upon the type of stationary and mobile phase employed. This experiment uses reversed-phase Chromatography, where the stationary phase is non-polar and the mobile phase is polar.

During HPLC experiment a high pressure pump takes the mobile phase from a reservoir through an injector. It then travels through a reverse phase C-18 packed column for component separation. Finally the mobile phase moves into a detector cell, where the absorbance is measured at 220nm and ends in a waste bottle. The amount of time taken for a component to travel from injector port to the detector is called retention time.

In reverse phase HPLC, the column stationary phase packing is usually either a C4, C8 or C18 packing. The C4 columns are primarily for proteins with large molecular weights, where as the C18 columns are for peptides and basic samples with molecular weights.

This experiment uses a single mobile phase and pump, which is called an isocratic mobile phase. For samples that are difficult to separate, a gradient mobile phase is used. This is when the initial mobile phase is primarily an aqueous one and over time, a second organic mobile phase gradually added to the overall mobile phase. This method raises the polarity of this phase over time, which lowers the retention time of the components and works similarly to a temperature gradient on a gas chromatography. There are some instances where the column is

heated (usually at 40^0C), which takes away any retention time errors associated with a change of ambient temperature.