CHEMISTRY (20A51101P)

LAB MANUAL

I-B.TECH

Prepared by:

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VEMU INSTITUTE OF TECHNOLOGY

(Approved By AICTE, New Delhi and Affiliated to JNTUA, Anantapur)

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JAWAHARLAL NEHRU TECHNOLOGICAL UNIVERSITY ANANTAPUR
(Established By Govt.of A.P., Act No.30 of 2008)
ANANTHAPUARAM-515 002 (A.P) INDIA
Humanities & Sciences

Course Code	chemistry	L	T	P	C
20A51101P		0	0	3	1.5
Pre-requisite	chemistry lab manual	Semester			I & II

Course Objectives:
• Verify the fundamental concepts with experiments
Course Outcomes (CO): After completion of the course, the student can able to
CO-1 Determine the cell constant and conductance of solutions
CO-2: Prepare advanced polymer Bakelite materials
CO-3: Measure the strength of an acid present in secondary batteries
CO-4 Analyse the IR of some organic compounds

VEMU INSTITUTE OF TECHNOLOGY

P.KOTHAKOTA,NEAR PAKALA, CHITTOOR-517112, A.P., India

DEPARTMENT OF HUMANITIES & SCIENCES

CHEMISTRY LAB MANUAL

Name	
Register No.	
Branch/Section	
Academic year	

JAWAHARLAL NEHRU TECHNOLOGICAL UNIVERSITY ANANTAPUR

Syllabus for (R20 Regulations)

CHEMISTRY LABORATORY (20A51101P)

(CSE, AI & DS, CSE (AI), CSE(IoT), CSE (Data Science),

CSE (AI & ML), IT, ECE, EEE and IT)

List of Experiments:

1. Measurement of 10Dq by spectrophotometric method

2. Models of potential energy surfaces

3. Conductometric titration of (i) strong acid vs. strong base, (ii) weak acid vs. strong base

4. Determination of cell constant and conductance of solutions

5. Potentiometry - determination of redox potentials and emfs

6. Determination of Strength of an acid in Pb-Acid battery

7. Preparation of a Bakelite and measurement of its mechanical properties (strength.).

8. Verify Lambert-Beer's law

9. Thin layer chromatography

10. Identification of simple organic compounds by IR.

11. Preparation of nanomaterials by precipitation

12. Estimation of Ferrous Iron by Dichrometry

13. pH metric titration of (i) strong acid vs. strong base, (ii) weak acid vs. strong base

14. Potentiometric determination of iron using standard potassium dichromate

References:

1. Vogel's Text book of Quantitative Chemical Analysis, Sixth Edition – Mendham J et al, Pearson Education, 2012.

2. Chemistry Practical– Lab Manual, First edition, Chandra Sekhar KB, Subba Reddy GV and Jayaweera KN, SM Enterprises, Hyderabad, 2014.

CHEMISTRY LAB-LIST OF EXPERIMENTS

S. No	EXPERIMENTS
1.	Estimation of Ferrous Iron by Dichrometry
2	Preparation of nanomaterials by precipitation
3.	Determination of emf by Potentiometry
4.	Conductometric titration of (i) strong acid vs. strong base
5.	Conductometric titration of weak acid vs. strong base
6	Determination of cell constant and conductance of solutions
7	Determination of Strength of an acid in Pb-Acid battery
8	Preparation of a Bakelite
9	Verify Lambert-Beer's law
10	Thin layer chromatography

Additional Experiments:

11	pH metric titration of strong acid vs strong base
12	Estimation of iron by potentiometric titration

LABORATORY INSTRUCTIONS

- Engineering Chemistry is an experimental science.
- The main aim of engineering chemistry is to give fundamental knowledge of science and technology for engineering students.
- The study of Engineering Chemistry emphasizes the application of basic scientific principles to the design of equipment, which includes electronic and electro-mechanical systems, for use in measurements, communications, and data acquisition.
- The theory that is presented in lectures has its origins in, and is validated by, experimental measurement.
- The practical aspect of Chemistry is an integral part of the subject.
- The laboratory practical's take place throughout the semester in parallel to the lectures.

They serve a number of purposes:

- It is an opportunity, as a student, to test theories by conducting meaningful scientific experiments.
- It is useful to enrich and deepen understanding of physical/chemical concepts presented in lectures.
- It is helpful to develop experimental techniques, in particular skills of data analysis, the understanding of experimental uncertainty, and the development of graphical visualization of data.
- Students are advised to thoroughly go through this manual rather than only topics mentioned in the syllabus as practical aspects are the key to understanding and conceptual visualization of theoretical aspects covered in the books.

Course Objectives: The objective of the laboratory is learning.

- To learn practical understanding of the redox reaction
- To learn the preparation and properties of synthetic polymers and other material that would provide sufficient impetus to engineer these to suit diverse applications.
- To also learn the hygiene aspects of water would be in a position to design methods to produce potable water using modern technology.
- Verify the fundamental concepts with experiments

Course Out comes:

- At the end of the course, the students will be able to
- Determine the cell constant and conductance of solutions (L3)
- Prepare advanced polymer Bakelite materials (L2)
- Measure the strength of an acid present in secondary batteries (L3)
- Analyse the IR of some organic compounds (L3)

INSTRUCTIONS TO THE STUDENTS:

The following instructions must be followed by the students in their laboratory classes.

1. Students are expected to be punctual to the lab classes. If they are late, they will be considered absent for that particular session.

2. Students should strictly maintain the dress code.

3. Students must bring their observation note, record note (completed with previous experiment) and the calculator, scales, pencils to every lab class without fail.

4. Students are advised to come with full preparation for their lab sessions by

5. Reading the detailed procedure of the experiment from the laboratory manual.

6. Data entry in the observation note book must be by pen only.

7. Bring necessary graph papers for each of experiment. Learn to optimize on usage of graph papers. Graphs should be neatly drawn with pencil. Always label graphs and the axes and display units.

8. If you finish early, spend the remaining time to complete the calculations and drawing graphs.

9. Students should complete their calculations for their experiments and get it corrected on the same day of that experiment.

10. Students who miss observation, record note they have to do the experiment once again and get it corrected.

11. Internal marks for each experiment are based only on their performance in the laboratory.

12. Record note has to be completed then and there and get corrected when the students are coming for the next lab class.

13. Students must strictly maintain silence during lab classes.

14. If any of the students is absent for the lab class for genuine reasons, he/she will be permitted to do the experiment during the repetition class only.

15. If any student is found causing damage to the lab equipments, he/she shall replace the same with a new.

Good Luck for your Enjoyable Laboratory Sessions

CONTENTS

CHEMISTRY LAB-SCHEME OF EVALUATION

S.No	EXPERIMENTS	Page. No	Marks awarded			
			Record (10M)	Observation (10M)	Viva (10M)	Total (30M)
1.	Estimation of Ferrous Iron by Dichrometry	6-7				
2	Preparation of nanomaterials by precipitation	8				
3.	Determination of emf by Potentiometry	9-14				
4.	Conductometric titration of (i) strong acid vs. strong base	15-17				
5.	Conductometric titration of weak acid vs. strong base	18-20				
6	Determination of cell constant and conductance of solutions	21-22				
7	Determination of Strength of an acid in Pb-Acid battery	23-24				
8	Preparation of a Bakelite	25-26				
9	Verify Lambert-Beer's law	27-30				
10	Thin layer chromatography	31-32				
11	pH metric titration of strong acid vs strong base	33-35				
12	Estimation of iron by potentiometric titration	36-40				

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.
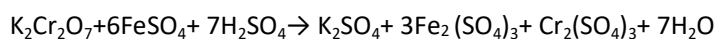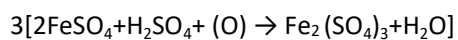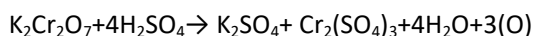
APPARATUS REQUIRED:

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

CHEMICALS REQUIRED:

1. $K_2Cr_2O_7$
2. Diphenylamine(DPA)
3. Conc. H_2SO_4
4. Ferrous ammonium sulphate(FAS)
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.

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

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

$$K_2Cr_2O_7 + 6FeSO_4 + 7H_2SO_4 \rightarrow K_2SO_4 + 3Fe_2(SO_4)_3 + Cr_2(SO_4)_3 + 7H_2O$$

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 a 100 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 acid with 300ml of concentrated H_2SO_4 in a reagent bottle and stopper it.

3. **Preparation of Diphenylamine:** Dissolve 1gm of Diphenylamine in 100ml of concentrated H_2SO_4.

PROCEDURE:

- Rinse and fill the burette with standard $K_2Cr_2O_7$ solution.
- Pipette out 20ml of ferrous ammonium solution (FAS) 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(ml)	Final(ml)	
1				
2				
3				

CALCULATIONS:

Normality of $K_2Cr_2O_7$ = N_1 =   N

Volume of $K_2Cr_2O_7$ = V_1 =   ml

Normality of FAS   = N_2 =   N

Volume of FAS   = V_2 =   ml

$$N_1V_1 = N_2V_2$$

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

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

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

PREPARATION OF NANOMATERIALS BY PRECIPITATION

Aim: To prepare nanomaterials by precipitation process.

Chemicals Required:

  (i) Zinc Nitrate

  (ii) Potassium Hydroxide

Apparatus Required:

  (i) Centrifuge

  (ii) Beaker

  (iii) Glass rod

PRINCIPLE:

Procedure:

 ZnO nano particles were synthesized by direct preparation method using Zinc nitrate and KOH as precursors. In this process the aqueous solution of Zinc nitrate (0.2M) and solution of (0.04M) of KOH were prepared with deionized water.

  The KOH solution is slowly added into Zinc nitrate solution at room temperature under vigorous stirring which resulted in the formation of a white suspension. The white product was centrifuged at 5000 rpm for 20 minutes and washed three times with distilled water and washed with absolute alcohol at last. The obtained product was calcinated at 500^0C in air atmosphere for 3hrs.

Result:

  In this experiment, ZnO nano particles were successfully synthesized by direct precipitation method using Zinc nitrate as Zinc source and KOH as precipitating agent in aqueous solution. The size range of the generated ZnO powder was approximately _____nm.

Determination of EMF using Potentiometry

Aim: Determination of EMF 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(l), Hg_2Cl_2(s /KCl (salt) //Fe $^{3+}$, Fe^{2+} /Pt (+)

Cell Reaction: Anode: Hg^{+2} + 2Cl$^-$ → Hg_2Cl_2 + 2e$^-$

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

Cell e.m.f.: E_{cell} = $E^o_{(Fe^{3+}/Fe^{2+})}$ + (2.303RT/ F) log ($_{Fe^{3+}/Fe^{2+}}$) - E_{SCE}

E_{cell} =Electrode potential of cell

$E^o_{(Fe^{3+}/Fe^{2+})}$ =Std. Electrode potential

T=absolute temperature

F=Faraday constant

E_{SCE}= Std. Electrode potential of Calomel Electrode

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.

PREPARATION OF CHEMICALS:

1.PREPARATION OF STANDARD FAS SOLUTION:

- Weigh 0.98 gms of Mohr's salt (FAS) 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.

2. PREPARATION OF $KMnO_4$:

- Weigh 3.2 gms of $KMnO_4$ and transfer the salt into standard flask with the help of funnel and add little distilled water to dissolve salt completely.
- Make up the solution up to 1000 ml and the normality of $KMnO_4$ is 0.02N.

3. PREPARATION OF dil. H_2SO_4 (6N):

- In order to prepare 1 liter of 6.0 Normal acid solution, add 300.2 grams of 98% purity sulfuric acid. Mix gently and add enough deionized water to make it exactly 1 liter.

Procedure:

Step 1: 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 color which persists for at least a minute as end point. Note the titre value.
- Repeat the process till concurrent titre values are obtained.
- Calculate the normality of $KMnO_4$ solution

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

- The burette is washed with water and rinsed and filled with given $KMnO_4$ solution up to zero mark.
- Take 20ml of the prepared standard FAS solution into a beaker and add 5-6 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(l), Hg_2Cl_2(s) /KCl(salt) //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 increase(inflexion) in EMF is observed. Then take about 6 to 8 readings after inflexion in 1 ml intervals.
- From the titrations approximate volume of $KMnO_4$ required is calculated.

GRAPH:1

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

Model Graph:
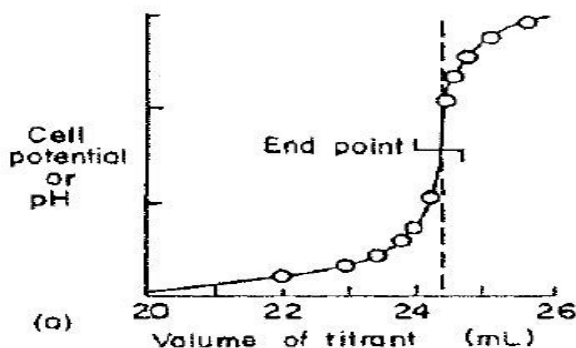
CALCULATIONS:

Step 1: **Standardization of FAS solution.**

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

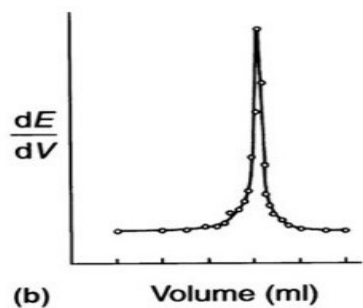$$N_1V_1 = N_2V_2$$

$$N_2 = \frac{N_1V_1}{V_2} = \text{-----------} N$$

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

S.No.	Vol of KMnO$_4$ added (ml)	E$_{cell}$ (EMF)
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)	$\triangle E$	$\dfrac{\triangle E}{\triangle V}$

GRAPH 2:

Differential graph is drawn by plotting $\dfrac{\triangle E}{V \triangle}$ (y-axis) **Vs** Volume of KMno₄ (X-axis) to get a sharp peak, which corresponds to the precise equivalence point of titration.

(b)

RESULT:

The amount of ferrous ion present in 1000 ml of the solution =-----------------

$$= N \times 55.85 \text{g/L}$$

$$=$$

The amount of ferrous ion present in 100 ml of the given solution=-----------------

$$= N \times 55.85 \times \dfrac{100}{1000}$$

$$= \text{---------------------- gm}$$

CONDUCTOMETRIC TITRATION OF i) STRONG ACID Vs STRONG BASE

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

APPARAUTS REQUIRED:-

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

CHEMICALS REQUIRED:-

1. Standard NaOH solution
2. HCl solution

REPARATION OF CHEMICALS:-Standard NaOH Solution:- Weigh 4 gms of NaOH pallets and dissolve in 1000 ml of distilled water gives 0.1 N

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 neutralization of H^+ ions of acid 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 addition of free Na^+ and OH^- of the excess NaOH solution.

$$HCl + NaOH \rightarrow NaCl + H_2O$$

$$H^+ + Cl^- + Na^+ + OH^- \rightarrow Na^+ + Cl^- + H_2O$$
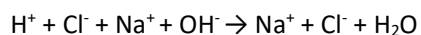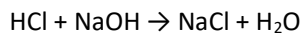
PROCEDURE:

- 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 diluted Hydrochloric acid (HCl) solution and measure the value of conductance.
- From the burette, add 0.5 ml of NaOH solution into HCl solution with shaking to get uniform solution and note down the observed conductance.
- Repeat the above procedure and record the observed conductance.
- Plot the graph between the observed conductance and volume of NaOH added and find out the volume of NaOH is required for complete neutralization.

GRAPH:

Plot a graph between measured conductances against 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).

cm³ of NaOH
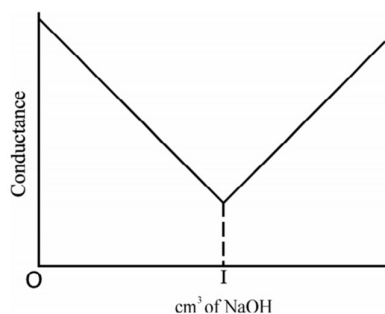
OBSERVATIONS:

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

$$N_1V_1 = N_2V_2$$

$$N_1 = \frac{N_2V_2}{V_1} =$$

RESULT: The strength of unknown HCl sample is _____N

Exp:5   **Date:**

CONDUCTOMETRIC TITRATION OF WEAK ACID VS. STRONG BASE

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

APPARAUTS REQUIRED: -

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

CHEMICALS REQUIRED: -

1. Standard NaOH solution
2. CH_3COOH solution

REPARATION OF CHEMICALS: - Standard NaOH Solution: - Weigh 4 gms of NaOH
  pellets and dissolve in 1000 ml of distilled water gives 0.1 N

PRINCIPLE: -

The conductance of the acid initially is very low because of low ionization of weak acetic acid. With the constant addition NaOH, the conductance's keep on increasing, as the number of ions in solution is increasing. But the conductance is slow initially 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.

$$CH_3COOH + NaOH \rightarrow CH_3COO^- + Na^+ + H_2O$$
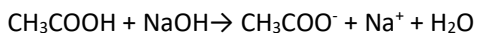
In case of weak acid (like CH_3COOH) against strong base (NaOH), the curve shape is found different. The point of intersection tells the exact volume of NaOH used for complete neutralization.

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, add 0.5 ml an of NaOH solution into CH_3COOH solution with shaking to get the uniform solution and note down the observed conductance.
- Repeat the above procedure and record the observed conductance.
- Plot the graph between the observed conductance and volume of NaOH mixed and find out the volume of NaOH   required for complete neutralization.

GRAPH:
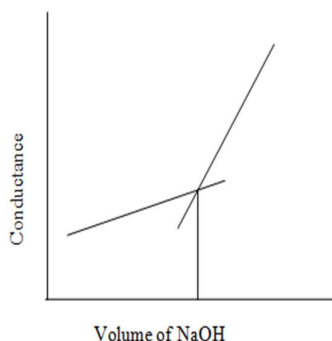
OBSERVATION: -

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 $CH_3COOH = N_1 = \quad$ N

Volume of $CH_3COOH \quad = V_1 = \quad$ ml

Normality of NaOH $\quad = N_2 = \quad$ N

Volume of NaOH $\quad = V_2 = \quad$ ml

$$N_1V_1 = N_2V_2$$

$$N_1 = \frac{N_2V_2}{V_1} =$$

RESULT: The strength of unknown CH_3COOH sample is _____N

DETERMINATION OF CELL CONSTANT AND CONDUCTANCE OF SOLUTIONS

AIM: To determine the cell constant and conductance of the solutions.

Chemicals Required:

1. Potassium chloride
2. Distilled water

APPARATUS REQUIRED

1. Conductance cell
2. Beakers
3. Standard flask
4. Glass rod

PREPARATION OF SOLUTIONS:

Preparation of 0.1N KCl solution: Weigh 0.745 gms of KCl and dissolve it in 100ml of distilled water.

Preparation of 0.01N KCl solution: Take 10ml of 0.1N KCl solution and dilute to 100ml with distilled water.

PRINCIPLE:

Conductivity: Conductivity can be defined as the ability of a Solution to conduct electrical current. Conductivity (mho or $ohm^{-1)}$ of any solution depends on the nature of ions present in it.

Cell Constant: The cell constant is defined as the ratio of distance between the electrodes which is divided by the area of the cross-sectional of the electrode

The specific conductance is measure of conductance of solutions with electrodes of unit area that are place 1 cm apart. Conductance is the inverse (or reciprocal) of electrical resistance, represented as 1/R.

The specific conductance (K) is given by

$K = (1/R)(l/a)$ as $1/R = C$ i.e. Conductance

$K = C(1/a)$

$l/a = K/C$ as $1/a$ is cell constant

Cell Constant = Specific conductance ($ohm^{-1}cm^{-1}$) or conductivity**/**

           Measured conductance(ohm^{-1}).

Thus the unit of cell constant is $cm^{-1.}$

PROCEDURE

- Calibrate the conductivity cell
- Measure the conductance of distilled water
- Dip the conductivity cell in 40ml of 0.1N KCl and record the conductance observed.
- Note down the temperature.
- Wash the conductivity cell with distilled water and measure the conductance for 0.01N KCl
- Tabulate the readings.

OBSERVATIONS:

Conductance of distilled water =(A)_____ohm^{-1}

Conductance of 0.1N KCl   =------------------

Conductance of 0.01N KCl   = ----------------

TABLE:

Concentration of KCl	Conductivity at room temp.(ohm^{-1} cm^{-1} (A)	Conductance(ohm^{-1}) (B)	Cell constant (cm^{-1})

0.1N	0.012		
0.01 N	0.0014		

RESULT:

0.1N Cell constant = ---------------- cm^{-1}

0.01N Cell constant =   cm^{-1}
                           _____

DETERMINATION OF STRENGTH OF AN ACID IN Pb-ACID BATTERY

AIM: To determine the strength of a sulfuric acid solution in Pb-acid battery by titration with

standard NaOH solution.

CHEMICALS REQUIRED:

1. H_2SO_4
2. NaOH
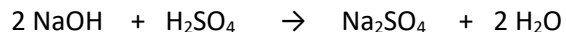3. Phenolphthalein indicator

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 it to 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 a clean conical flask.

2. Add 2 drops of phenolphthalein indicator to solution.

3. Rinse and Fill the burette with standard sodium hydroxide solution.

4. Titrate the sulfuric acid against standard NaOH solution, till pink colour appears.

5. Note down the volume (V_1) of NaOH consumed.

6. Repeat the titration for concurrent readings.

Calculations

S.No	Volume of H_2SO_4(V_2) ml	Burette Readings		Volume of NaOH consumed (V_1) ml
		Initial	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

$$N_1V_1 = N_2V_2$$

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

RESULT: Strength of Sulphuric acid in Pb-acid battery is = ------------------N

PREPARATION OF BAKELITE

AIM: To prepare phenol formaldehyde resin (Bakelite).

CHEMICALS REQUIRED:

1. Glacial acetic acid or 2N NaOH
2. 40% formaldehyde solution
3. Phenol
4. Concentrated H_2SO_4/HCl

APPARATUS REQUIRED:

1. Glass rod
2. beakers
3. Funnel
4. Measuring cylinder
5. Dropper and filter paper

Preparation of Chemicals: -

40% formaldehyde solution: *40% Formalin* (*Formaldehyde* aqueous solution) i.e., 1 part of the stock formalin with 9 parts water, preferably distilled.

PRINCIPLE:

Phenol formaldehyde resins (PFs) are condensation polymers and are obtained by condensing phenol with formaldehyde in the presence of an acidic or alkaline catalyst. They were first prepared by *Backland*, an American Chemist who gave them the name as *Bakelite.* These are thermosetting polymers.

PREPARATION: -

PFs are prepared by reaction of phenol with formaldehyde in the presence of acidic or basic catalyst. The process may be carried out as follows

A mixture of phenol and formaldehyde are allowed to react in the presence of a catalyst. The process involves formation of methylene(-CH_2-) bridges in *ortho, para* or *both ortho* **and** *para* positions. This result first in the formation of linear polymer (Called *NOVALAC*) and Formation of Novolac takes place from hydroxy benzyl alcohol and a water molecule is removed from the process. Bakelite is formed from novolac as to cross-linked polymer called phenol-formaldehyde resin or *Bakelite.*
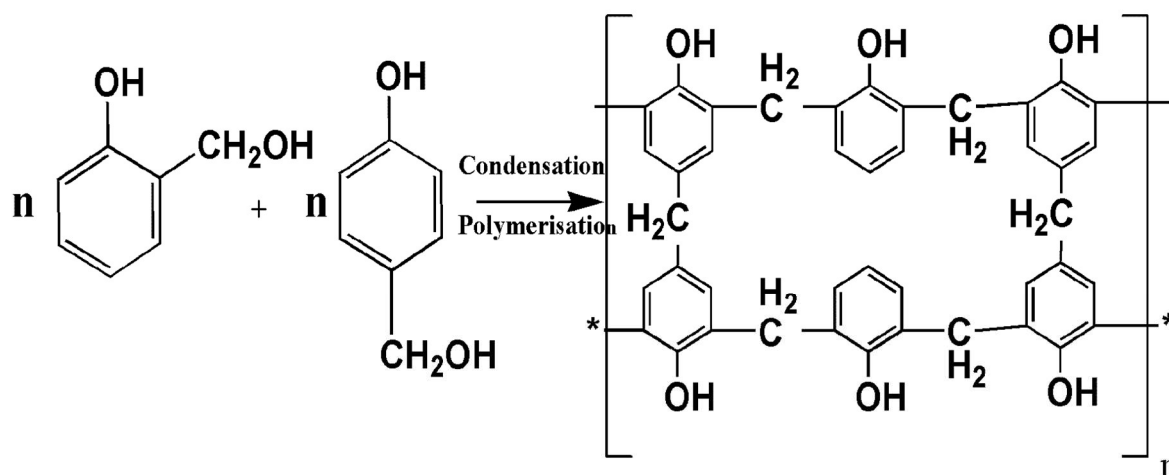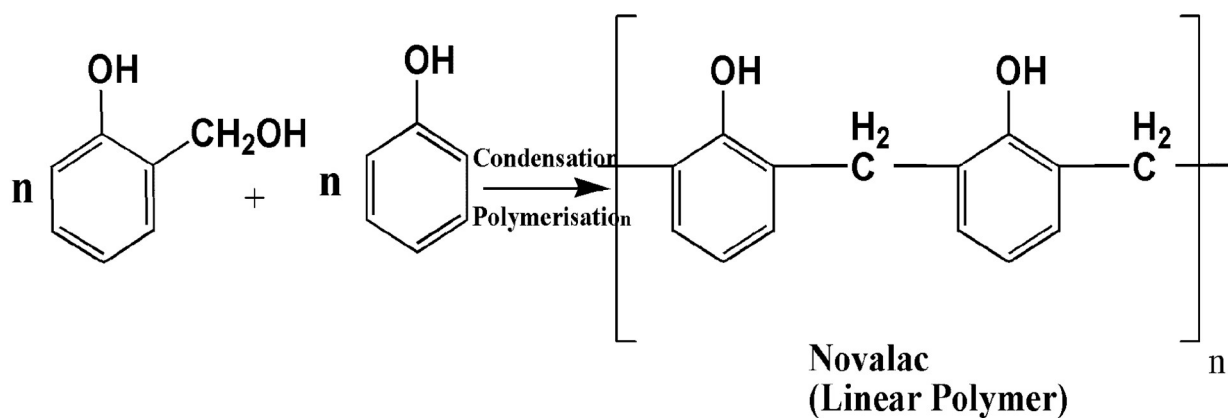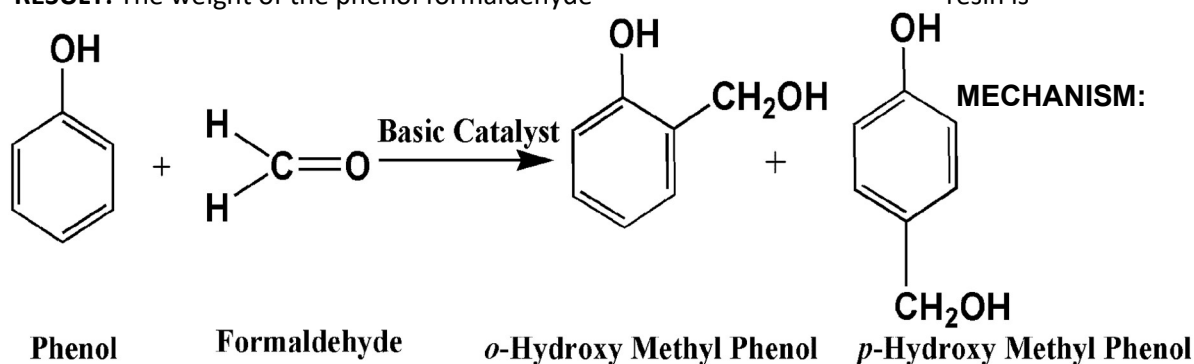
PROCEDURE:

1. Place 5ml of glacial acetic acid (or 2N NaOH) and 2.5ml of 40% formaldehyde solution in a 500ml beaker.
2. Add 2 grams (or approximately 2ml) of phenol.
3. Add few ml of conc.H_2SO_4/HCl into the mixture carefully.

4. Within 5 min. a large mass of pink color plastic is formed.
5. The residue obtained is washed with distilled water
6. Filtered product is dried and yield is calculated.

PRECAUTIONS:

1. The reaction is sometimes vigorous and it is better to be a few feet away from the beaker while adding the H_2SO_4 and until the reaction is complete.
2. The experiment should be preferably carried out in fume cupboard.

RESULT: The weight of the phenol formaldehyde resin is ------------------g.

MECHANISM:

Phenol   Formaldehyde   o-Hydroxy Methyl Phenol   p-Hydroxy Methyl Phenol

Novalac
(Linear Polymer)

Bakelite
(Cross-linked Polymer)

VERIFICATION OF BEER-LAMBERT'S LAW

AIM: To verify Beer-Lambert's law

CHEMICALS REQUIRED:

1. $KMnO_4$
2. Ferrous Ammonium Sulphate (Mohr's salt)
3. Sulphuric acid
4. Distilled water

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

PRICIPLE:

Beer and Lambert Law states that, when a monochromatic light is passed 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

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

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

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.

PREPARATION OF CHEMICALS:

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 10ml dil.H_2SO_4 and make the homogeneous solution up to the mark.

PREPARATION OF $KMnO_4$:

- Weigh 3.2 gms of $KMnO_4$ and transfer the salt into standard flask with the help of funnel and add little distilled water to dissolve salt completely.
- Make up the solution up to 1000 ml and the normality of $KMnO_4$ is 0.02N.

PROCEDURE

STEP 1: 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.

STEP 2: ESTIMATION OF CONCENTRATION OF $KMnO_4$ BY COLORIMETRY & VERIFICATION OF BEER-LAMBERTS LAW:

- Take 10ml of $KMnO_4$ 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 into 10 test tubes respectively.
- Make up every solution up to 10ml with distilled water. (Dilution is done as given in Table 2.of the experiment.)
- Calibrate the colorimeter with blank solution.
- Select the filter no:52
- Calibrate the colorimeter by taking distilled water as blank and adjust OD to zero.
- Fill the cuvvete 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.
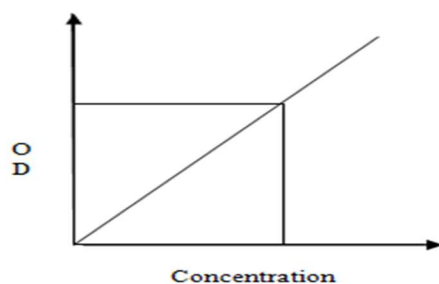
CALCULATIONS:

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 KMnO₄ solution.

S.No	Volume of FAS solution (V_1 ml)	Burette readings		Volume KMnO₄ solution V_2 (ml)
		Initial(ml)	Final(ml)	
1				
2				
3				
4				

Normality of FAS   = N_1 =   N

Volume of FAS = V_1 =   ml

Normality of KMnO₄   = N_2 =   N

Volume of KMnO₄   = V_2=   ml

$$N_1V_1 = N_2V_2$$

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

Normality of KMnO₄ solution N_2= --------------N

Part C: Verification OF Beer-Lambert's law

S.No	Volume of Std. KMnO₄ sol. + Distilled Water	Concentration	Optical Density(OD)
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		

Result: Beer-Lambert's law is verified.

THIN LAYER CHROMATOGRAPHY

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

CHEMICAL REQUIRED: -

1. Chloroform
2. Alcohol
3. Silica gel
4. Spinach leaves

APPARATUS REQUIRED: -

1. Chromatographic plates
2. Chromatography chambers with lids
3. Tongs
4. Capillary tubes
5. Beaker
6. Filter paper
7. Pestle mortar
8. Reagent bottles with lid
9. Glass rods

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 colored. 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 reagent bottle with constant swirling motion; the bottles should be tightly Stoppard.

2. Thin layer of adsorbent is prepared by holding the two cleaned and dried glass plates together, dipping them in the slurry of adsorbent and removing the plates quickly.

3. 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.

4. 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. Then filter the contents are filtered and the filtrate is taken as the 'extract of leaves.

5. 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.

6. Place the glass plate in chromatography jar in such a way that it does not touch the sides of the jar.

7. Measure the distance travelled by the spots and the developing agent.

8. 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 cleaned and dried.
*   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   =   m

Distance travelled by solvent   =   m

Rf value of component   =

pH METRIC TITRATION OF STRONG ACID Vs STRONG BASE

AIM: To perform pH metric titration of a strong acid with a strong alkali and determine the strength   of acid.

APPARATUS REQUIRED:

1. pH meter
2. Burette
3. Burette stand
4. Wash bottle
5. Pipette
6. Plastic funnel
7. Glass rod
8. Beaker

CHEMICALS REQUIRED:

1. 0.05M HCl solution

2. 0.1 M NaOH solution

3. Distilled water.

PRINCIPLE:

In an acid-base titration, the strength of acid is determined with the help of equivalence point. It is the point at which an equal amount of acid has been neutralized by equal amount of base and vice versa. If only acid is present in the sample, the pH value will be low. With the addition of base, neutralization takes place and pH value increases. At the end point, the addition of very slight amount of base increases the pH very sharply indicating the completion of neutralization process.

The number of moles of acid in the test sample can be calculated by determining the equivalence point by using the molarity of the base and volume of the base added.

$$\text{HCl (aq)} + \text{NaOH (aq)} \longrightarrow \text{NaCl(s)} + \text{H}_2\text{O(l)}$$
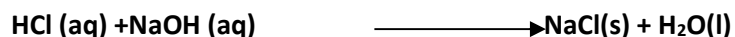
PROCEDURE:

- Clean the electrode with distilled water and wipe with tissue paper or filter paper.

- Take 25ml of HCl solution in a 50ml beaker and immerse the electrode in it. Note down the pH. The reading shown on the scale of pH meter is pH value of the HCl solution.

- Rinse and fill the burette with standard NaOH solution.

- Add NaOH solution drop wise from the burette (maximum 0.2ml at a time), mix the solution well with the help of glass rod and note the corresponding pH values.

- Continue the addition of NaOH until the pH increases continuously for five values.

- Plot the graph by taking Volume of NaOH on X-axis and pH on Y-Axis with the values obtained from titration, extend the lines and note the point if intersection which indicates the volume of base needed for complete neutralization of acid.

SAMPLE GRAPH:
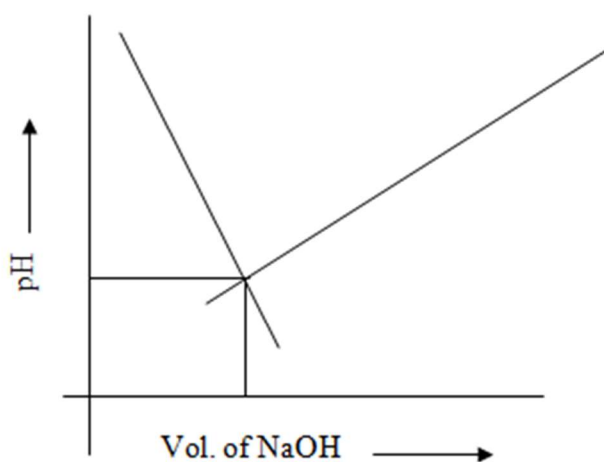
Result: The strength of given acid sample is _____N.

CALCULATIONS:

S.NO	Volume of HCl taken ($v_1$ml)	Volume of NaOH added ($v_2$ml)	pH
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			

Volume of acid Taken $= V_1 =$ _____ml

Molarity of acid taken $= M_1 =$ _____M

Volume of NaOH consumes $= V_2 =$ _____ml

Molarity of NaOH $= M_2 = $ _____M

$$V_1M_1 = V_2M_2$$

$$M_1 = \frac{M_2V_2}{V_1} = $$

Result: The strength of given acid sample is _____M.

ESTIMATION OF IRON BY POTENTIOMETRIC TITRATION

AIM: To estimate Fe^{2+} ion present in the given solution.

CHEMICAL REQUIRED: -

1. FAS,
2. dil.H2SO4,
3. standard K2Cr2O7

APPARATUS REQUIRED:

1. Potentiometer assembly,
2. 50ml burette,
3. 10ml pipette,
4. 250ml beaker,
5. magnetic stirrer,
6. calomel and platinum electrodes

PRINCIPLE: -

The method usually used for the measurement of the emf of a cell is known as Poggendorff method. The instrumental technique is called potentiometry. The electromotive force (emf) of a galvanic cell is usually measured by comparing the emf of the experimental cell with that of a standard cell. The emf is measured using a potentiometer when zero current is drawn. The comparison cell is standard in the sense that its emf is accurately known and is constant. (Emf of Weston Cadmium cell is 1.0186 V at 250C) Emf of Calomel electrode is 0.2422 V at 250C.

REDOX TITRATION

This is a typical Redox Titration using potentiometer experimental set up. For a redox titration, an electrochemical cell has to be setup by taking a known quantity of an aqueous solution of the reducing agent (here FAS) in a beaker provided with an indicator electrode – **Platinum electrode** and a reference electrode – **Calomel electrode** connected to a potentiometer. The potential developed by the cell is measured. The electrode potential of the reference electrode is constant and the potential of the indicator electrode depends on the concentration of the reacting iron. From the Nernst equation,

$$E = E^0 + 0.0591/n \log [Ox]/[Red]$$

The aqueous solution of the standard oxidizing agent (potassium dichromate solution) taken in the burette is added in small increments to the reducing agent in the beaker. The change in potential for each addition is noted. The rate of change of potential is maximum at the end-point.

A plot of Emf vs. volume of oxidizing agent added (potential – volume curve) gives the S – shaped curve in which a large variation in emf at equivalence point is obtained.

The cell is shown as follows:

$$Pt|Fe^{2+}|Fe^{3+}|Cr_2O_7^{2-}|KCL|HgCl_2|Hg$$
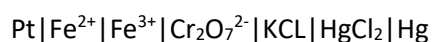
LOCATION OF END-POINT:

Generally, S-shaped curve is obtained when EMF vs volume of titrant added (in ml) is plotted. When the curve shows a very clearly marked steep portions, the approximate volume (EMF in millivolts) of the end-point may be located from the mid-point of the steep portion of the curve.

PROCEDURE:

STEP 1: STANDARDIZATION OF $K_2Cr_2O_7$ SOLUTION:

- Rinse and fill the burette with $K_2Cr_2O_7$ solution.

- Take 20ml of the prepared standard FAS solution into a clean conical flask.

- Add 10 ml of dilute H_2SO4 (6N) to provide acidic medium.

- Titrate the solution against $K_2Cr_2O_7$ taken in burette until the solution acquires pale pink color which persists for at least a minute as end point. Note the titer value.

- Repeat the process till concurrent titer values are obtained.

- Calculate the normality of $K_2Cr_2O_7$ solution.

PART – B: DETERMINATION OF REDOX POTENTIAL OF Fe^{2+} / Fe^{3+}:

- 20ml of ferrous ammonium sulphate solution is pipette out into a 100ml beaker.

- 20ml of dil. H_2SO4 is added to it and stirred well.

- The platinum electrode and the reference electrode (calomel electrode) are connected to the instrument and placed into the beaker.

- From the burette dichromate solution is added in the increments of 1ml. Stir the contents and measure the EMF every time.

- The approach of the equivalent point is noted by the rapid change of E.M.F.

- The solution in the beaker is discarded.

- 20ml of ferrous ammonium sulphate and 20ml of dil H_2 SO4 solutions are freshly taken again into a 100ml beaker.

- It is titrated potentiometrically against dichromate solution. Dichromate solution is added in the increments of 0.1ml.

- Contents of the beaker are stirred well and the EMF is noted. The potential difference corresponds to ΔE. This increases rapidly near the equivalence point and it is indicated by the maximum value of ΔE.

- A graph is drawn between EMF (E) on Y-axis and volume of $K_2Cr_2O_7$ solution on X-axis. 'S' shape curve is obtained. The maximum value on the curve corresponds to equivalence point

GRAPH:1

Draw a graph of E_{cell}(EMF) Vs volume of $K_2Cr_2O_7$ added; the inflexion point gives an approximate equivalence point.

Model Graph:
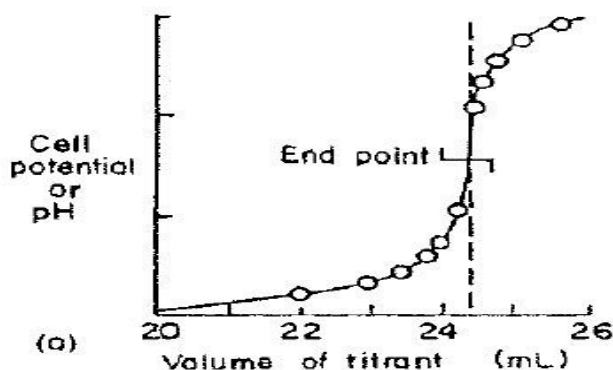
RESULT

The amount of Iron present in the given solution   =------------g

CALCULATIONS:

Step 1: **Standardization of FAS solution.**

S. No.	Volume of FAS (V$_2$ ml)	Burette Reading(ml)		Vol. of K$_2$Cr$_2$O$_7$ (ml)
		Initial	Final	
1				
2				
3				
4				

Volume of FAS   (V$_1$)   =   ml

Volume of K$_2$Cr$_2$O$_7$ (V$_2$)   =   ml

Normality of K$_2$Cr$_2$O$_7$ (N$_1$)   =   N

Normality of FAS (N$_2$)   = ?

$\quad\quad$ N$_1$V$_1$ =N$_2$V$_2$

$\quad\quad\quad$ N$_2$ = $\dfrac{N_1V_1}{V_2}$ = -----------N

$\quad\quad\quad\quad\quad$ =

The amount of Iron present in the given solution   = N$_2$ x 55.85 x 1000/10

$\quad\quad\quad\quad\quad\quad\quad\quad\quad\quad$ = ---------------mg

Table1: FAS VS $K_2Cr_2O_7$ (Pilot Titration)

S. No.	Vol. of $K_2Cr_2O_7$ (ml)	EMF (milli volts)

Table   FAS VS K$_2$Cr$_2$O$_7$ (Fair Titration)

S. No.	Vol. of K$_2$Cr$_2$O$_7$ (ml)	EMF (Milli volts)	ΔE (milli volts)	ΔV (ml)	ΔE/ ΔV (milli volts/ml)

GRAPH:1 Draw a graph of E_{cell}(EMF) Vs volume of KMnO4 added; the inflexion point gives an approximate equivalence point.

(b)   Volume (ml)
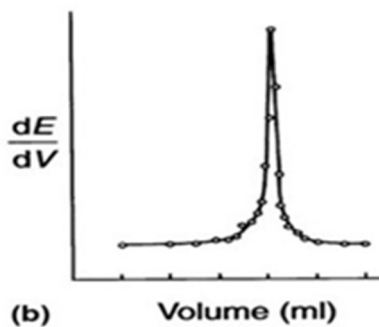
RESULT

The amount of Iron present in the 100 ml of given solution   =-------------g