

VEMU INSTITUTE OF TECHNOLOGY

P.KOTHAKOTA, CHITTOOR DIST – 517 112



DEPARTMENT OF HUMANITIES & SCIENCES

ENGINEERING CHEMISTRY

**LABORATORY MANUAL FOR I-Year B.Tech
(*CE,ME*)**

DEPARTMENT OF HUMANITIES & SCIENCES

ENGINEERING CHEMISTRY LAB MANUAL

VEMU INSTITUTE OF TECHNOLOGY

P.KOTHAKOTA, CHITTOOR DIST – 517 112

Name	
Register No.	
Branch/Section	
Academic year	

SYLLABUS

(CE,ME)

ENGINEERING CHEMISTRY LABORATORY: EXPERIMENTS

1. Determination of total hardness of water sample.
2. Determination of viscosity of the oils using Redwood Viscometer.
3. Estimation of calcium in Portland cement.
4. Determination of % of moisture content in coal sample.
5. Determination of percentage of iron in cement sample.
6. pH metric titration of strong acid Vs strong base.
7. Determination of strength of an acid in Pb-acid battery
8. Determination of cell constant and conductance of solutions.
9. Determination of redox potentials using Potentiometry.
10. Preparation of Thiokol rubber.

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ENGINEERING CHEMISTRY LAB

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DETERMINATION OF TOTAL HARDNESS OF WATER SAMPLE

AIM: To determine the hardness present in the ground water sample by EDTA method.

CHEMICALS REQUIRED:

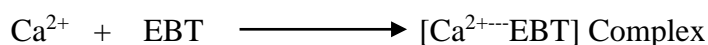
1. EDTA solution
2. Standard hard water
3. EBT indicator
4. Buffer solution
5. Sample hard water

APPARATUS REQUIRED:

1. Burette,
2. Pipette
3. Conical flask
4. 250 ml beaker
5. 100 ml standard flask

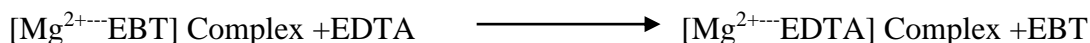
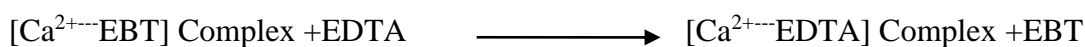
PRINCIPLE:

Disodium salt of Ethylene Diamine Tetra Acetic acid (EDTA) is a well known complexing agent. Disodium salt of EDTA is used to estimate the various hardness of the given hard water containing Ca^{2+} and Mg^{2+} ions. When EDTA is added to hard water, it reacts with Calcium and Magnesium ions present in hard water to form stable EDTA metal complexes. From the volume of EDTA consumed the hardness can be calculated. Eriochrome Black-T indicator is used as an indicator. This indicator forms a weak, unstable complex with the metal ions present in the hard water and gives wine red colour.



(Hard water) (Indicator) (Weak wine red colour complex)

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



From the volume of EDTA consumed in the reaction, total hardness can be calculated.

PROCEDURE:

STEP 1: Standardization of EDTA:

- Wash the burette with distilled water and rinse well with the standard EDTA solution.
- Fill the burette with standard EDTA solution up to the zero level without air bubbles.
- Note the initial reading of the burette.
- Take 20ml of standard hard water solution into a clean conical flask.
- Add 5ml of ammonical buffer solution and 2 drops of Eriochrome Black – T indicator
- Then the solution turns wine red in colour and then titrate against standard EDTA solution taken in the burette until the end point i.e conversion of wine red colour to deep blue.
- Note the volume of EDTA consumed.
- Repeat the titration to get concordant values.

Step II: Determination of Hardness of water sample:

- Take 20ml of hard water sample into a clean conical flask.
- Add 5ml of ammonical buffer solution and 2 drops of Eriochrome Black – T indicator.
- Then the solution turns wine red in colour and then titrate against standard EDTA solution taken in the burette until the end point i.e conversion of wine red colour to deep blue.
- Note the volume of EDTA consumed.
- Repeat the titration to get concordant values.

RESULT: Amount of hardness present in the given water sample = _____PPM.

STEP-1: STANDARDISATION OF EDTA:

S.NO	Volume of standard Hard Water (ml)	Burette readings		Volume of EDTA (ml)
		Initial	Final	
1				
2				
3				

CALCULATIONS:

Volume of standard Hard water solution (V_1) = 20ml

Normality of standard Hard water solution (N_1) = _____ N.

Volume of EDTA (V_2) = _____ ml

Normality of EDTA (N_2) = _____ N.

According to law of volumetric analysis

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 N_1}{V_2}$$

$$= 20 \times \text{_____} \text{ N.} =$$

$$= \text{_____} \text{ N}$$

Normality of EDTA Solution= _____ N

Step-2: Determination of total hardness of hard water sample

S.NO	Volume of sample hard water(ml)	Burette readings		Volume of EDTA (ml)
		Initial	Final	
1.				
2.				
3.				

CALCULATIONS:

Volume of EDTA $V_1 = \underline{\hspace{2cm}}$ ml

Normality of EDTA $N_1 = \underline{\hspace{2cm}}$ N

Volume of sample hard water $V_2 = 20$ ml

Normality of hard water sample $N_2 = \underline{\hspace{2cm}} ?$

According to the law of volumetric analysis

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 N_1}{V_2} =$$
$$= \underline{\hspace{2cm}} \text{ N}$$

Total hardness of hard water sample = $N_2 \times 50 \times 1000 =$

$$= \underline{\hspace{2cm}} \text{ ppm.}$$

RESULT: Amount of hardness present in the given water sample = $\underline{\hspace{2cm}}$ PPM.

Exp: 2

Date:

DETERMINATION OF VISCOSITY OF THE OILS USING REDWOOD VISCOMETER

AIM: To determine the viscosity of lubricating oil by Redwood viscometer.

APPARATUS REQUIRED:

1. Redwood viscometer
2. Thermometer
3. Stopwatch
4. Kohlrausch flask.

MATERIALS REQUIRED:

Given sample of lubricating oil.

DESCRIPTION OF THE APPARATUS

1. Oil cup:

It is 90 mm in height and 46.5 mm in diameter silver plated brass cylinder. Its upper end is open. Its lower end is fitted with an agate jet having bore of diameter 1.62 mm and length 10 mm. The jet can be opened and closed by a valve rod. The valve rod is a small silver plated brass ball fixed to a stout wire. There is a pointer to indicate the level to which the cylinder is to be filled with oil. The pointer is fixed on the inner side of the cylinder. The cover of the cup is fitted with a thermometer to indicate the temperature of the oil.

2. Heating bath:

There is a cylindrical copper bath which surrounds the oil cup. This copper bath contains water. It is provided with an out let tap to let out water from it and a long side tube projection outwards. This is needed to heat the bath water by means of a burner. There is a thermometer to indicate the temperature of water.

CALCULATIONS

The ratio of absolute viscosity to density for any fluid is known as its Kinematic viscosity. Since the instruments used are of standard dimension, Kinematic viscosity of the oil in Centistokes (CS) can be calculated from the time taken by the oil to flow through the standard orifice of the instrument the help of following equation.

The viscosity of the given oil sample with
The help of Redwood viscometer at t^0_e

- (i) Kinematic viscosity $V = At - \{B/t\}$
- (ii) Absolute viscosity = Kinematic viscosity x density of the sample.

Where,

V = Kinematic viscosity of the oil in centistokes

t = Time of flow in seconds

A & B are instrument constants. The value of

A = 0.264 and B = 190, when t = 40 to 85 seconds
B = 0.247 and B = 65, when t = 85 to 2000 seconds.

3. Stirrer:

The heating bath is provided with stirrer which stirs the water in the heating bath for maintaining uniform desired temperature. The stirrer is sealed at the top to prevent water rushing into the oil cylinder.

4. Spirit level:

The cover of the cup is provided with a spirit level for vertical leveling of the jet.

5. Leveling screws:

The entire apparatus rests on three legs provided at the bottom with leveling screws.

6. Kohlrausch flask:

This flask receives the oil from jet outlet. Its capacity is 50 ml up to mark in its neck.

THEORY

Viscosity is one of the most important properties of any lubricating oil. This indicates us about the suitability of the oil for lubricating purpose. A lubricant must reduce friction between sliding parts of any machine. This avoids the direct metal to metal contact. The main criteria of a lubricant are that it should be sufficiently viscous under high temperature and pressure exerted by the machine to adhere to the surface. If the viscosity is low then a thin film of lubricant cannot adhere to the sliding surfaces. In case the viscosity is high there will be excessive friction. The absolute viscosity of fluid can be determined by measuring the rate of flow of the oil through a capillary tube kept at a uniform temperature. But in case of lubricating oil specific viscosity is generally determined by measuring the time taken for a given quantity of oil to flow through an orifice or jet of standard dimension under standard conditions.

Measurement of viscosity of lubricating oil is made with the help of an apparatus called Redwood viscometer of thin lubricating oils.

Types of Redwood Viscometers

There are two types of Redwood viscometers.

- (a) Redwood viscometer No.1
- (b) Redwood viscometer No.2.

(a) Redwood viscometer No.1

Redwood viscometer No.1 is used for low viscosity oils. It will correctly indicate the viscosity of a liquid having time flow between 30 seconds to 2000 seconds. If the time flow measured with this apparatus for any oil exceeds 2000 seconds, the test should be repeated with Redwood viscometer No.2.

(b) Redwood viscometer No.2

Redwood viscometer No.2 will give the correct value of viscosity for such highly viscous oil.

Viscosity measured by RED WOOD viscometers is reported as n seconds of RED WOOD I or II as the case may be at a given temperature.

PROCEDURE

The oil cup is washed thoroughly with a suitable solvent. It is then dried and cleaned in such a way that it contains no any residue. The bath is mounted on a stand and is filled with water to

determine the viscosity of an oil at 80°C and below. The brass ball is kept in a position so as to seal the orifice.

The sample oil is now carefully poured into the oil cup up to the mark. A Kohlrausch flask in the position below the jet. A thermometer and a stirrer is inserted and allowed to stirrer the water in the bath and oil in the cup. The temperature of the bath is adjusted until the oil attains the desired constant temperature. The ball valve is now lifted and simultaneously the stop watch is started. The oil is allowed to fill Kohlrausch flask up to 50 ml mark. Stop watch is stopped and time in seconds is noted.

The ball valve is replaced in the position to seal the cup to prevent overflow of the oil. Oil cup is again refilled up to mark and the experiment is repeated to get nearly reproducible results. The experiment is repeated for five different temperature (40°C , 50°C , 60°C , 70°C and 80°C) and the respective time of flow is noted.

RESULT: The given sample of lubricating oil has,

- (i) Redwood viscosity at $t_1^{\circ}\text{C}$ = _____ Redwood seconds
- (ii) Kinematic viscosity at $t_1^{\circ}\text{C}$ = _____ centistokes.
- (iii) Absolute viscosity = _____

OBSERVATIONS AND CALCULATIONS:

No. of observations	Temperature (°C)	Time of flow (sec) redwood viscosity	Kinematic viscosity (centi stokes)
1	Room temperature		
2	40		
3	50		
4	60		
5	70		
6	80		

ESTIMATION OF CALCIUM IN PORTLAND CEMENT

AIM: To estimate the calcium content in the given cement sample.

APPARATUS REQUIRED:

1. Crucible
2. Desiccators
3. Beaker
4. Water Bath
5. No 40 Wattmann Filter Paper
6. Measuring Jar
7. Electric Bunsen Burner

CHEMICALS REQUIRED:

1. Sample of Portland cement
2. 1:1HCl
3. 15%NaOH solution
4. Ammonium Chloride
5. Ammonia
6. Ammonium Oxalate.

PROCEDURE:

1. Weigh 2gm of cement and transfer into a crucible and add 2ml of water to prevent lumping.
2. Add 10ml of 1:1HCl to this and allow to digest for 5min.
3. Then add 50ml of water to transfer the contents into a beaker and add 50ml of 1:1HCl to beaker to make the acidic medium.
4. Filter the solution through No 40 Wattmann filter paper and collect the filtrate into a beaker and make it to 250ml with distilled water.
5. Pipette out 100ml of the above solution into a beaker and boil it.
6. To this add 2gm of ammonium chloride to avoid the precipitation of calcium compounds.
7. Add 20ml of ammonia to the boiling solution to make the solution alkaline.
8. Boil the solution for 5min until a pale brown jelly precipitate is formed.
9. Cool and filter the solution and then heat the collected filtrate.
10. Dissolve 1gm of ammonium oxalate in 10ml of boiling water and add to the boiling filtrate. A white precipitate of oxalate is formed during the above process and filter it.
11. Incinerate the pre-weighed crucible and cool it in desiccators and find the amount of calcium content from the difference in weight.

RESULT: The amount of calcium content was found to be _____ gm.

CALCULATIONS:

Weight of Cement taken (w) = _____ gm.

Weight of empty crucible (a) = _____ gm.

Weight of crucible + Calcium (b) = _____ gm.

% of calcium content = $(b-a) \times 250 \times w \times 100$.

=

RESULT: The amount of calcium content was found to be _____ gm.

Exp: 4

Date:

DETERMINATION OF % OF MOISTURE CONTENT IN A COAL SAMPLE

AIM: To determine the moisture content of a given sample of coal.

APPARATUS:

1. Silica Crucible With Vented Lid
2. Electric Oven
3. Muffle Furnace
4. Spatula
5. Desiccators
6. Pair Of Tongs
7. Weighing Balance
8. Long Legged Tongs

CHEMICALS REQUIRED: Powdered coal sample.

PROCEDURE:

- Weigh empty and clean silica crucible.
- Transfer about 1g of powdered, air dried coal sample into a previously weighed silica crucible.
- Place the open crucible with sample in an electric oven and heat it at about 105-110⁰c for an hour.
- Take out the crucible after one hour from the oven cool it in a desiccators (containing absorbing anhydrous calcium chloride).
- Then weigh the crucible with sample and repeat the process of heating, cooling& weighing till constant weight is obtained.
- Finally calculate the loss in weight.

RESULT: The percentage of moisture content in the given coal sample is _____%

CALCULATIONS:

Weight of empty crucible (W_1) = _____ g.

Weight of crucible + coal sample (W_2) = _____ g.

Weight of coal sample before heating = ($W_2 - W_1$) = W_3 = _____ g.

Weight of crucible + Sample after heating for 1hr at $105-110^{\circ}\text{C}$ = W_4 _____ g.

Weight of coal sample after heating = ($W_4 - W_1$) = W_5 = _____ g.

Loss in weight of sample due to moisture = $W_5 - W_3$

=

RESULT: The percentage of moisture content in the given coal sample is _____%

Exp: 5

Date:

DETERMINATION OF PERCENTAGE OF IRON IN CEMENT SAMPLE

AIM: To estimate the amount of iron present in the cement sample using ammonium thiocyanate.

CHEMICALS REQUIRED:

1. HCl
2. HNO₃
3. Ammonium thiocyanate
4. Cement Sample
5. Ferrous ammonium sulphate
6. H₂SO₄
7. Dil.KMnO₄
8. Distilled Water

APPARATUS REQUIRED:

1. Burette
2. Pipette
3. Conical flask
4. 250 ml beaker
5. 100 ml standard flask
6. Colorimeter
7. Cuvets

PRINCIPLE:

When ammonium thiocyanate is added to Ferric ion, it produces blood red colour. The colour produced is stable in presence of nitric acid. The intensity of color produced is directly proportional to the concentration of ferric ion present in the sample. The intensity of colour produced is measured by a photo colorimeter and the concentration of ferric iron is obtained from a standard calibration curve.

PROCEDURE:

Step 1: Determination of standard calibration Curve:

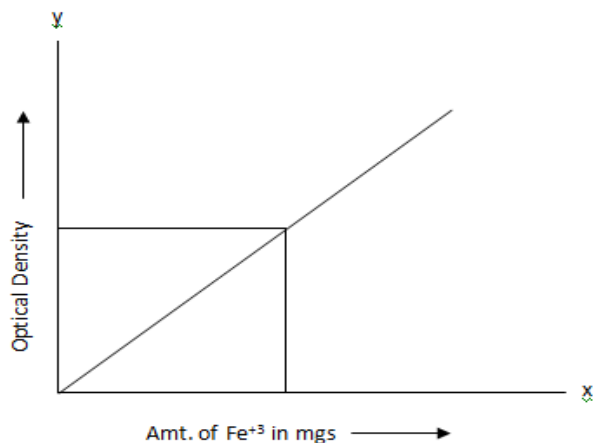
1. Dissolve the given ferrous ammonium sulphate in 100ml of H₂O in a conical flask and add 5ml of 1:5 H₂SO₄ and dil.KMnO₄ solution through burette until light pink colour appears.
2. Dilute the solution to 1 litre such that 1ml of solution contains 0.1mg of Fe⁺³.
3. From the above solution take separately 1, 2, 3, 4, 5 ml into five 100ml standard volumetric flasks.
4. Add 1ml of nitric acid and 5ml of 40% ammonium thiocyanate solution to all the above samples to get blood red colour and make up the solutions to the mark by adding distilled water.
5. Now measure the optical densities of all the solution using photo colorimeter.
6. Plot a graph by taking amount of ferrous iron on X-axis and optical density on Y-axis.
7. The curve obtained is called standard calibration curve.

Step 2: Estimation of Iron present in the sample:

1. Weigh about 1gm of cement sample accurately and transfer into a clean and dry 250ml beaker.
2. Add about 5ml of water to moisten the sample.
3. Place a glass rod and cover the beaker with a watch glass and add about 5ml of Conc. HCl drop wise and heat the solution till the sample dissolves.
4. Heat the beaker on small flame and evaporate the solution to almost dryness to expel the excess acid.
5. Add about 20ml of the distilled water to the beaker to dissolve the contents.
6. Then filter the solution through Whatmann no. 40 filter paper into 100ml standard volumetric flask.
7. Wash the funnel with 10ml portions of distilled water into the beaker, remove the funnel and make up the solution to 100ml with distilled water.
8. Shake the flask well for uniform concentration.
9. Pipette out 10ml of prepared solution into a 100ml standard volumetric flask and add 1ml of conc. HNO_3 .
10. From the burette add 5ml of 40% NH_4SCN and make up the solution to 100ml with distilled water and shake the flask well for uniform concentration.
11. Measure the optical density of the solution by using photo colorimeter and determine the concentration of iron from the standard calibration curve.

RESULT: The percentage of Fe^{+3} present in 1gm of cement _____

STANDARD CALIBRATION CURVE



CALCULATIONS:

S.NO	Conc. Of Fe ³⁺ in mgs	Optical density
1	0.05	
2	0.10	
3	0.15	
4	0.20	
5	0.25	
6	0.30	
7	0.35	
8	0.40	

Weight of bottle + cement sample = $w_1 =$ _____ g.

Weight of empty bottle = $w_2 =$ _____ g.

Weight of cement = $w_1 - w_2 =$ _____

% of Fe³⁺ in the sample = $\frac{y \times 10 \times 10}{(w_1 - w_2)}$ mg = _____

RESULT: The percentage of Fe³⁺ present in 1 gm of cement _____

Exp: 6

Date:

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. P^H meter
2. Burette
3. Burette stand
4. Wash bottle
5. Pipette
6. Plastic funnel
7. Glass rod
8. Beaker

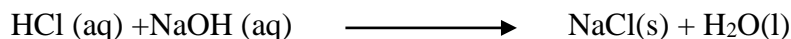
CHEMICALS REQUIRED:

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.

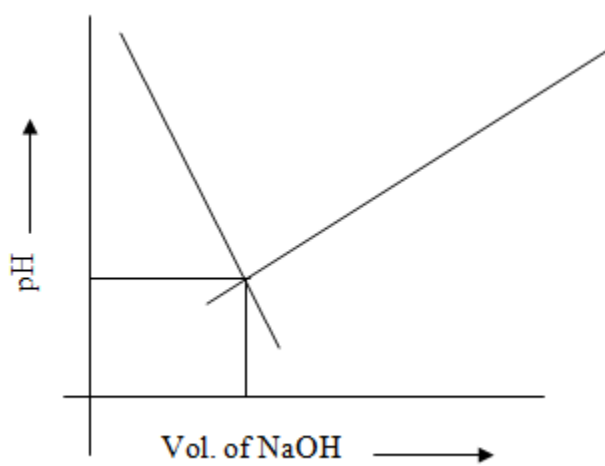
The number of moles of acid in the test sample can be calculated by determining the equivalence point by using the molarity of the base and volume of the base added.



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 P^H meter is P^H 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 pH on X-axis and Volume of NaOH on Y-Axis with the values obtained from titration, extend the lines and note the point of intersection which indicates the volume of base needed for complete neutralization of acid.

SAMPLE GRAPH:



Result: The strength of given acid sample is _____M

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 = V₁= _____ml

Molarity of acid taken = M₁= _____M

Volume of NaOH consumes =V₂= _____ml

Molarity of NaOH = M₂= _____M

$$V_1M_1=V_2M_2$$

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

Result: The strength of given acid sample is _____M

EXP:7

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:

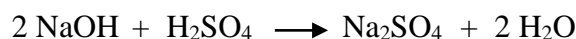
1. Sulfuric acid
2. NaOH
3. Phenolphthalein indicator

APPARATUS REQUIRED:

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

PRINCIPLE:

The strength of acid present in Pb-Acid battery is determined by titration against standard NaOH solution using Phenolphthalein Indicator. When Phenolphthalein indicator is added to acid, it is colour less. When the acid sample is titrated against standard base, the pH increases and then the solution colour turns to pink which indicates the end point of the reaction.



PROCEDURE:

1. Rinse and fill the burette with standard NaOH solution.
2. Pipette out 20 ml of given Sulphuric acid into clean conical flask.
3. Add 2 drops of phenolphthalein indicator to sample.
4. Read the initial volume on the burette and record the value.
5. Titrate against the standard NaOH solution, till pink colour appears.
6. Note down the volume of base consumed.
7. Repeat the titration for concordant values.

RESULT: The strength of acid in the given Pb-Acid battery is _____M

CALCULATIONS:

S.No	Volume of H ₂ SO ₄ (V ₂) ml	Burette Readings		Volume of NaOH (V ₁) ml
		Initial	Final	
1				
2				
3				

Normality of NaOH solution = N₁ = N

Volume of NaOH solution = V₁ = ml

Volume of H₂SO₄ solution = V₂ = 20 ml

Normality of H₂SO₄ = N₂ = ?

$$V_1 N_1 = V_2 N_2$$

$$N_1 = \frac{N_2 V_2}{V_1} =$$

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

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	

EXPERIMENT-9

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 + Hg₂Cl₂ 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), Hg₂ Cl₂(s) /KCl(salt) //Fe³⁺, Fe²⁺ /Pt (+)

Cell Reaction: Anode: - 2 Hg + 2Cl⁻ → Hg₂Cl₂ + 2e⁻

Cathode: - 2Fe⁺³ + 2e⁻ → 2Fe⁺²

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

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. H₂SO₄ 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 KMnO₄ solution:

- Rinse and fill the burette with KMnO₄ solution.
- Take 20ml of the prepared standard FAS solution into a clean conical flask.
- Add 10 ml of dilute H₂SO₄ (6N) to provide acidic medium.
- Titrate the solution against KMnO₄ 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 KMnO₄ solution

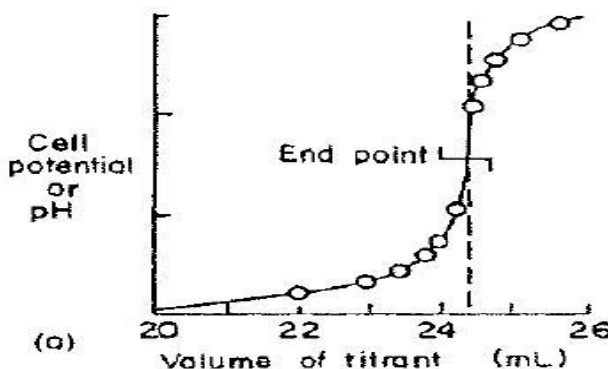
PART C: Estimation of Fe⁺² in the given test solution:

- The burette is washed with water and rinsed and filled with given KMnO₄ solution upto zero mark.
- Take 20ml of the prepared standard FAS solution into a beaker and add 10 ml of H₂SO₄ 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), Hg₂ Cl₂(s) /KCl(sat) //Fe³⁺, Fe²⁺ /Pt (+)
- Add KMnO₄ from burette in 1 ml portions to the ferrous solution, stir it and note the EMF.
- Continue the titration till a sudden inflexion in EMF occurs. Then take about 6 to 8 readings after inflexion in 1 ml intervals.
- From the titrations approximate volume of KMnO₄ required is found out.
- The titration is repeated with addition of KMnO₄ 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 = W₁ = ---- g.

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

Wt. of FAS = $W_1 - W_2$ =

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

Part B: Standardization of FAS₄ solution.

Titration of Std. FAS. Vs. KMnO_4

S. No.	Volume of FAS (V_2 ml)	Burette Reading		V_{KMnO_4} (V_1) 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

$$N_1 V_1 = N_2 V_2$$

$$N_2 = \frac{N_1 V_1}{V_2} =$$

V_2

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

Table. 1

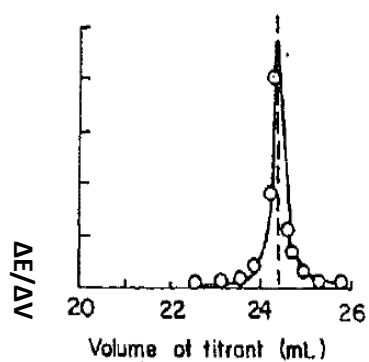
S.No.	Vol of KMnO₄ added (ml)	E_{cell} (mv)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		

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 = -----NX55.85 $\frac{100}{1000}$

= -----gms

PREPARATION OF THIOKOL RUBBER

AIM: To prepare Thiokol rubber

CHEMICALS REQUIRED:

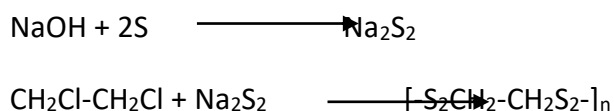
1. Sodium Hydroxide
2. Ethylene Dichloride
3. Sulphur

APPARATUS REQUIRED:

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

PRINCIPLE:

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

**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 stir 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°C 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.

