ENGINEERING CHEMISTRY
(20A51201P)

LAB MANUAL

I-B. TECH

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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	Engineering chemistry	L	T	P	C
20A51201P		0	0	3	1.5
Pre-requisite	lab manual	Semester			I & II

Course Objectives:
• Verify the fundamental concepts with experiments
• 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 using modern technology.

Course Outcomes (CO): After completion of the course, the student can able to
• Determine the cell constant and conductance of solutions
• Prepare advanced polymer materials
• Determine the physical properties like surface tension, adsorption and viscosity
• Estimate the Iron and Calcium in cement
• Calculate the hardness of water

VEMU INSTITUTE OF TECHNOLOGY

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DEPARTMENT OF HUMANITIES & SCIENCES

ENGINEERING CHEMISTRY LAB MANUAL

Name	
Register No.	
Branch/Section	
Academic year	

JAWAHARLAL NEHRU TECHNOLOGICAL UNIVERSITY ANANTAPUR

Syllabus for (R20 Regulations)

ENGINEERING CHEMISTRY LABORATORY (20A51201P)

(Common to Civil and Mechanical)

List of Experiments:

1. Determination of Hardness of a groundwater sample.
2. pH metric titration of (i) strong acid vs. strong base, (ii) weak acid vs. strong base
3. Determination of cell constant and conductance of solutions
4. Potentiometry - determination of redox potentials and emfs
5. Determination of Strength of an acid in Pb-Acid battery
6. Preparation of a polymer
7. Determination of percentage of Iron in Cement sample by colorimetry
8. Estimation of Calcium in port land Cement
9. Preparation of nanomaterials by precipitation.
10. Adsorption of acetic acid by charcoal
11. Determination of percentage Moisture content in a coal sample
12. Determination of Viscosity of lubricating oil by Redwood Viscometer 1 &2
13. Determination of Calorific value of gases by Junker's gas Calorimeter.

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 Jayaveera KN, SM Enterprises, Hyderabad, 2014.

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ENGINEERING CHEMISTRY LAB-List of Experiments

S.No	EXPERIMENTS
1.	Determination of Hardness of a ground water sample.
2	Determination of Viscosity of lubricating oil by Redwood Viscometer
3.	Estimation of Calcium in port land Cement
4.	Determination of percentage Moisture content in a coal sample
5.	Determination of percentage of Iron in Cement sample by colorimetry
6	pH metric titration of (i) 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 EMF using potentiometry
10	Preparation of Thiokol rubber
11	Preparation of nanomaterials by precipitation

Additional Experiments

12	Determination of Alkalinity of Water
13	Estimation of Dissolved oxygen by Winkler's method

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 practicals 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.
- To 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 materials (L2)
- Determine the physical properties like surface tension, adsorption and viscosity (L3)
- Estimate the Iron and Calcium in cement (L3)
- Calculate the hardness of water (L4)

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

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ENGINEERING CHEMISTRY LAB-SCHEME OF EVALUATION

S.No	EXPERIMENTS	Page. No	Marks awarded			
			Record (10M)	Observation (10M)	Viva (10M)	Total (30M)
1.	Determination of Hardness of a ground water sample.	7-10				
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8	Determination of cell constant and conductance of solutions	27-28				
9	Determination of EMF using potentiometry	29-34				
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Additional Experiments:						
12	Determination of Alkalinity of Water	38-40				
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Exp: 1 **Date:**

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 hand 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 Ca2+ 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 color.

Ca^{2+} + EBT $\longrightarrow$ [Ca^{2+} - EBT] Complex

Mg^{2+} + EBT $\longrightarrow$ [Mg^{2+} - EBT] Complex

(Hard water) (Indicator) (Weak wine-red color 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 color 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 ammoniacal buffer solution (NH_4Cl +NH_4OH).

[Ca^{2+}-EBT] Complex +EDTA $\longrightarrow$ [Ca^{2+}- EDTA] Complex +EBT

[Mg^{2+} -EBT] Complex +EDTA $\longrightarrow$ [Mg^{2+} - EDTA] Complex +EBT

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 ammoniacal buffer solution and 2 drops of Eriochrome Black – T indicator
- Then the solution turns wine red in color and then titrate against standard EDTA solution taken in the burette until the end point i.e., conversion of wine-red color 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 ammoniacal buffer solution and 2 drops of Eriochrome Black – T indicator.
- Then the solution turns wine red in color and then titrate against standard EDTA solution taken in the burette until the end point i.e., conversion of wine-red color 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.

Calculations:

STEP-1: **Standardization of EDTA** **Ind: EBT**

S. No	Volume of Standard hard water (V_1) in ml	Burette readings in (ml)		Volume of EDTA consumed (V_2) in ml
		Initial	Final	
1	20			
2	20			
3	20			

CALCULATIONS:

Volume of sample hard water V_1 = 20ml

Normality of hard water sample N_1 =_________?

Volume of EDTA V_2 = ____________ml

Normality of EDTA N_2 = __________N

According to the law of volumetric analysis

$$V_1N_1=V_2N_2$$

$$N_2 = \frac{V1N1}{V2}$$

=______________N

Normality of EDTA Solution = __________________ N

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

S. No	Volume of Hard water sample (V_3) in ml	Burette readings in (ml)		Volume of EDTA consumed (V_2) in ml
		Initial	Final	
1	20			
2	20			
3	20			

CALCULATIONS:

Volume of EDTA V_2= ____________ml

Normality of EDTA N_2= __________N

Volume of sample hard water V_3= 20ml

Normality of hard water sample N_3 =_________?

According to the law of volumetric analysis

$$V_2N_2 = V_3N_3$$

$$N_3 = \frac{V1N1}{V2} = \frac{V2N2}{V3}$$

=______________N

Total hardness of hard water sample = N_3 X 50 X 1000 =

=____________ppm.

RESULT: Amount of total hardness present in given water sample is ___________ ppm.

Exp: 2 **Date:**

DETERMINATIONOF 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 outward. 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 with the help of following equation.

The viscosity of the given oil sample with the help of Redwood viscometer at t ^{0}C

(i) Kinematic viscosity V=At-{B/t}

(ii) Absolute viscosity = Kinematic viscosity x density of the sample.

Were,

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 80^0c 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 are 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 ill 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 (40^0C, 50^0C, 60^0C, 70^0C and 80^0C) and the respective time of flow is noted.

RESULT: The given sample of lubricating oil has,

(i) Redwood viscosity at $t_1{}^0$C =____________ Redwood seconds
(ii) Kinematic viscosity at $t_1{}^0$C =___________ centistokes.
(iii) Absolute viscosity = ____________

OBSERVATIONS AND CALCULATIONS:

No. of observations	Temperature (^{0}C)	Time of flow (sec)redwood viscosity	Kinematic viscosity (centi stokes)
1	Room temperature		
2	40		
3	50		
4	60		
5	70		
6	80		

Exp.No:3 **Date:**

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.

PREPARATION OF SOLUTIONS:

1:1 HCl: To make 1 liter of 1mol HCl, we take 88 ml of the concentrated solution and add water to make a total of 1 liter.

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 100 ml 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) x 250 x w x 100.

=

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

Exp: 4 Date:

DETERMINATION OF % OF MOISTURE CONTENT IN 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:

1. Powdered coal sample.
2. Anhydrous $CaCl_2$

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 desiccator (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.

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^{0}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. HNO_3
3. Ammonium thiocyanate
4. Cement Sample
5. Ferrous ammonium sulphate
6. H_2SO_4
7. dil. $KMnO_4$
8. Distilled Water

APPARATUS REQUIRED:

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

PREPARATION S OF THE SOLUITIONS:

1.1:5 H_2SO_4: Add 640 ml of water to 100 ml of a solution of 37% HCl in water and mix them well, to achieve a final solution with volume of 740 ml whereby the HCl.

2. dil. $KMnO_4$: Dissolve 3.2 gms of $KMnO_4$ in 1000 ml of distilled water gives 0.02M .

3. 40% Ammonium Thiocyanate: Dissolve 7.8 gms of Ammonium Thiocyanate in 100 ml of distilled water and make the solution up to 1 liter wit distilled water.

PRINCIPLE:

When ammonium thiocyanate is added to Ferric ion, it produces blood red color. The color 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 color produced is measured by a photo colorimeter and the concentration of ferric iron is obtained from a standard calibration curve.

Equation

when ammonium thiocyanate is added to ferric chloride, since NH4SCN is weakly acidic due to the ammonium ion; it reacts with alkali hydroxides, such as sodium hydroxide or potassium hydroxide to form sodium thiocyanate or potassium thiocyanate, along with water and ammonia. The thiocyanate anion, specifically, reacts with ferric salts to form a deep-red ferric thiocyanate complex. So, when ammonium thiocyanate reacts with ferric chloride it forms a dark deep red color ferric thiocyanate complex.

$$FeCl_3 + NH_4SCN \rightarrow [Fe\ (SCN)_6]_3^{-} + NH_4^{+}$$

With this reaction iron (III) thiocyanate compound is formed. Ferric chloride is an orange to black brown solid. Ammonium thiocyanate is used in chemical analysis, in photography, as a fertilizer, and for many other uses. It can release ammonia vapors if mixed with a chemical base or with an acid.

PROCEDURE:

Step 1: Determination of standard calibration Curve:

1. Dissolve the given ferrous ammonium sulphate in 100ml of water in a conical flask and add 5ml of 1:5 H_2SO_4 and dil.$KMnO_4$ solution through burette until light pink color appears.
2. Dilute the solution to 1 liter 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 color 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.

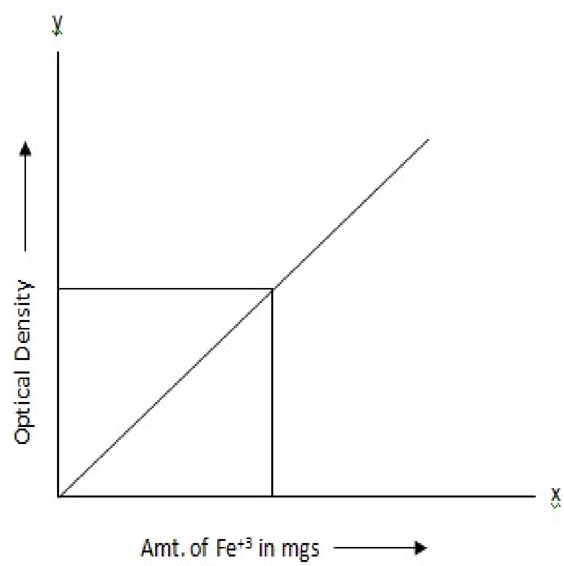
Step 2: Estimation of Iron present in the sample:

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 Wattmann 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 Fe^{+3} in the sample = $\frac{y \times 10 \times 10}{(w_1 - w_2)}$ mg =

RESULT: The percentage (%) of Fe^{+3} 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. pH meter
2. Burette
3. Burette stands
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.

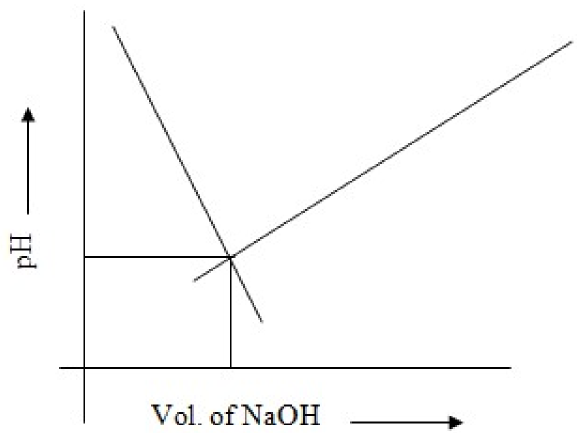
$$\mathbf{HCl\,(aq) + NaOH\,(aq) \longrightarrow NaCl(s) + H_2O(l)}$$

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.

Exp:7 **Date:**

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 color 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

$$N_1V_1 = N_2V_2$$

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

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

Exp: 8 **Date:**

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

APPARATUSREQUIRED

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 = $\frac{\text{Specific conductance (ohm}^{-1}\text{cm}^{-1}\text{) or conductivity}}{\text{Measured conductance(ohm}^{-1}\text{).}}$

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

PROCEDURE

- Calibrate the conductivity cel
- Measure the conductance of distilled water
- Dip the conductivity cell in 40ml 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}

Exp:9 **Date:**

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: $Hg^{+2} + 2Cl^{-} \rightarrow Hg_2Cl_2 + 2e^{-}$

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

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 OFCHEMICALS:

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

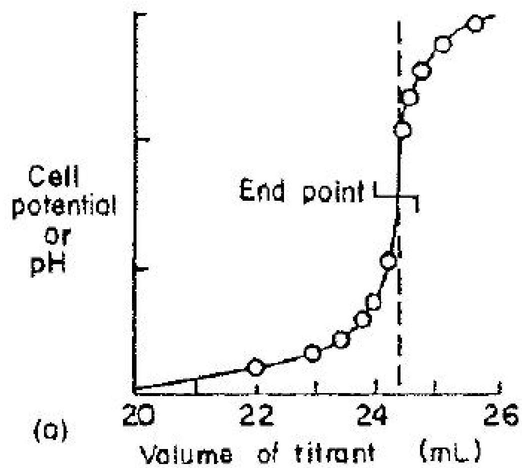
(-) Pt/ Hg(l), Hg_2Cl_2(s) /KCl(salt) //Fe^{3+}, Fe^{2+} /Pt (+)

- 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 points.

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:

Table-1

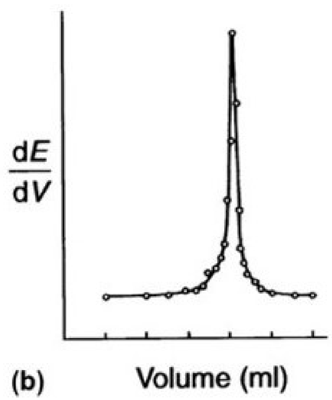
S.No.	Vol of $KMnO_4$ added (ml)	E_{cell} (EMF)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		

Table-2:

S.No.	Volume of $KMnO_4$ (ml)	E_{cell} (mv)	$\triangle E$	$\frac{\triangle E}{\triangle V}$

GRAPH 2:

Differential graph is drawn by plotting $\frac{\triangle E}{\triangle V}$ (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 ion presents in 1000 ml of the solution =------------------

= N×55.85g/L

=

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

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

= --------------------- gms.

EXP-10 **Date:**

Preparation of a polymer

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 stir until all the Sulphur has dissolved. The solution will turn from light yellow to dark brown when complete sodium Poly sulphide 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^{0}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.

EXP-11 **Date:**

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.

Exp.No:12 **Date:**

Estimation of Alkalinity of Water

Aim:

To determine the amount of alkalinity, present in the given water sample.

Apparatus Required:

1. Burette
2. Conical flask
3. Pipettes

Chemicals Required:

1. Water sample
2. Phenolphthalein indicator
3. Methyl orange indicator
4. Sulphuric acid (0.02 N)

Principle:

The alkalinity of water is a measure of its capacity to neutralize acids. It is measured volumetrically by titration with 0.01 N Sulphuric acid and is reported in terms of $CaCO_3$ equivalent.

For samples whose initial pH is above 8.3, the titration is conducted in two steps. In the first step, the titration is conducted until the pH is lowered to 8.2, the point at which phenolphthalein indicator turns from pink to colorless. This value corresponds to the point for conversion of carbonate to bicarbonate ion. The second phase of titration is conducted until the pH is lowered to 4.5, corresponds to methyl orange end point, which corresponds to the equivalence points for the conversion of bicarbonate ion to carbonic acid.

Procedure:

Part A: Phenolphthalein alkalinity or Partial Alkalinity:

- Pipette 20 ml of sample into a clean conical flask.
- Add two drops of phenolphthalein indicator & the color of solution becomes pink.
- Titrate against standard sulphuric acid in the burette, till the color just disappears.
- Note down the volume (V_1).

Part B: Methyl Orange Alkalinity or Total Alkalinity:

- Pipette 20ml of the sample water into a clean conical flask.
- Then add two drops of methyl orange indicator, the color turns yellow.
- Again titrate against acid, until the color turns to pink.
- Note down the volume (V_2).

Calculations:

Part A: Standard H_2SO_4 Vs Water Sample **Ind: Phenolphthalein**

S. No	Volume of water sample (V_2) in ml	Burette readings in (ml)		Volume of H_2SO_4 consumed (V_1) in ml
		Initial	Final	
1	20			
2	20			
3	20			

Calculation:

Normality of Std. H_2SO_4 solution = 0.02 N

Volume of Std. H_2SO_4 solution =____________ ml

Volume of water sample = 20 ml

$$\text{Partial Alkalinity} = \frac{\text{Vol. of } H_2SO_4 \times N_1 \times 50 \times 1000}{\text{Vol. of water sample}}$$

= ________________mg/lit

Part B: Standard H2SO4 Vs Water Sample **Ind: Methyl Orange**

S. No	Volume of water sample (V_2) in ml	Burette readings in (ml)		Volume of H_2SO_4 consumed (V_1) in ml
		Initial	Final	
1	20			
2	20			
3	20			

Calculation:

Normality of Std. H_2SO_4 solution = 0.02 N

Volume of Std. H_2SO_4 solution = ____________ ml

Volume of water sample = 20 ml

$$\text{Total Alkalinity} = \frac{\text{Vol. of } H_2SO_4 \times N_1 \times 50 \times 1000}{\text{Vol. of water sample}}$$

= ________________mg/lit

RESULT:

1. The amount of partial alkalinity present in given water sample = ______mg/lit(or) ppm
2. The amount of total alkalinity present in given water sample = ______mg/lit(or) ppm

Exp No: 13 **Date:**

Estimation of Dissolved Oxygen by Winkler's method

Aim:

To estimate the amount of dissolved oxygen (DO) in the given water sample by Winkler's method.

Apparatus Required:

1. Conical flask
2. Iodine flask/ BOD bottle
3. Pipette
4. Burette
5. Simple balance with weights

Chemicals Required:

1. Sodium thiosulphate
2. Potassium dichromate
3. Sulphuric acid
4. Potassium iodide
5. Starch
6. Sodium hydroxide
7. Manganese sulphate

Principle:

The determination of dissolved oxygen is based on the oxidation of potassium iodide by dissolved oxygen. The liberated iodine is titrated against standard sodiumthiosulfate solution using starch as an indicator. The dissolved molecular oxygen is unable to react with potassium iodide in water. Hence the carriers like manganese hydroxide carries oxygen and bring about the reaction between potassium iodide and oxygen. The manganese hydroxide is produced by manganese sulfate and potassium hydroxide.

$$Mn^{2+} + 2\ OH^- \longrightarrow Mn\ (OH)_2 \text{ (White precipitate)}$$

$$MnSO4 + 2\ KOH \longrightarrow Mn\ (OH)_2 + K_2SO_4$$

$$Mn\ (OH)_2 + O_2 \longrightarrow 2\ MnO\ (OH)_2 \text{ (Yellowish brown precipitate)}$$

(Basic manganic oxide)

$$2\ MnO\ (OH)_2 + H_2SO_4 \longrightarrow MnSO_4 + 2\ H_2O + (O)$$

$$2KI + H_2SO_4 + (O) \longrightarrow K_2SO_4 + I_2 + H_2O$$

$$2Na_2S_2O_3 + I_2 \longrightarrow Na_2S_4O_6 + 2NaI$$

(Disappearance of blue color)

Procedure:

Estimation of dissolved oxygen in the given water sample:

- Collect 100ml of water in a bottle avoiding as far as possible contact with the air.
- Immediately add 2ml of $MnSO_4$ solution and 2 ml of alkaline KI solution.
- Stopper the bottle and shake it thoroughly for 10-15 minutes and allow standing for few minutes to settle down the precipitate.
- Repeat the process of shaking and settling at least for times.
- Add 10ml of conc.H_2SO_4 to dissolve the precipitate. The contents of the flask is shaken well and keep aside in dark for 5 minutes.
- Titrate the contents of the flask against standardized hypo(0.1M) till the solution becomes yellow.
- Now add 2 ml of freshly prepared starch as indicator and continue the titration till the end point is reached which is disappearance of blue color.
- Repeat the titrations to get the concurrent values.

RESULT:

The dissolved oxygen content present in the given water sample = _________ mg/l

Calculations:

Estimation of dissolved oxygen **Ind: Starch**

S.No	Volume of Water sample (V_1) in ml	Burette readings in (ml)		Volume of hypo consumed (V_2) in ml
		Initial	Final	
1	20			
2	20			
3	20			

Calculation:

$$N_1V_1=N_2V_2$$

N_1= Normality of sodium thiosulphate solution = 0.01N

V_1= Volume of sodium thiosulphate solution = ____________ml

N_2= Normality of water sample =?

V_2= Volume of water sample = 100 ml

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

$$=$$

The Normality of water sample (N_2) =_________________N

Amount of dissolved oxygen in water sample = $N_2 \times 8 \times 1000$ mg/l

=

The amount of dissolved oxygen present in given water sample =_____________mg/l

RESULT:

The dissolved oxygen content present in the given water sample = _________ mg/l